Instruction Manual

Software HAAKE RheoWin 3

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International / Germany

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info.mc.de@thermo.com
www.thermo.com

The following specifications should be given when product enquiries are made:

- Software title
- Version number
- Serial number

Software License Agreement

3. Software Licence Agreement

between

Thermo Electron (Karlsruhe) GmbH Dieselstraße 4, 76227 Karlsruhe (hereinafter referred to as "Thermo")

the buyer (hereinafter referred to as "customer").

- The customer automatically acknowledges this licence agreement by installing the HAAKE software. Thermo grants the undersigned customer a non-exclusive, nontransferable single-user system licence to use the software hereunder in conjunction with a computer and a HAAKE rheometer from Thermo. Software and documentation are to be treated as confidential and may not be disclosed to any unauthorized third party. The customer may not make copies of the whole or any part of said software and/or documentation without written consent from Thermo. No transfer of confidential information may be made to a third party. If such confidential information is disclosed to a third party by the unauthorized action of the customer. Thermo will, for each separate instance of wrongful disclosure, be entitled to claim damages to the amount of € 10.000.- in addition to a conventional claim for compensation. The customer is contractually liable for payment of these amounts to Thermo.
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4. Law

Any dispute will be settled before a competent Karlsruhe court of law.

4. Installation

4.1 Hardware

The software calls for certain minimum requirements from the computer and its peripheral equipment in order to function properly.

For effective working choose the recommended configuration.

4.1.1 Recommended Configuration

- IBM or IBM-compatible computer with processor Pentium IV or later
- minimum 256 MB RAM
- CD-ROM drive
- Hard disk with minimum 2GB of free memory (10GB for image acquisition wiht HAAKE RheoScope 1)
- Graphics adapter, resolution 1024 x 768
- Serial interface (RS232C) for measuring instrument (if available second Serial interface (RS232C) for Temperature control assembly)
- Microsoft[®] or PS/2[®]mouse

4.1.2 Minimum requirements

- IBM or IBM-compatible computer with processor Pentium II, 300Mhz
- 128 MB RAM
- CD–ROM drive
- Hard disk with minimum 500 MB of free memory
- Graphics adapter, resolution 800 x 600
- Serial interface (RS232C) for measuring instrument (if available second Serial interface (RS232C) for Temperature control assembly)
- Microsoft[®] or PS/2[®]mouse

4.1.3 Operating systems

- Microsoft WinXP.
- Microsoft Windows2000, Servicepack 1
- Microsoft WinNT 4.0, Servicepack 6, Internetexplorer 6

4.2 HAAKE RheoWin_Installation

The installation must be carried out with administrator rights. Administrator rights are not required in order to start the HAAKE RheoWin Software.

New installation

The software cannot be run from the data—carrier and must be installed before it can be used. Before installation starts it is advisable to reboot the computer. Place the CD in the drive and the installation should start automatically; if it does not, start Setup.exe from the CD.

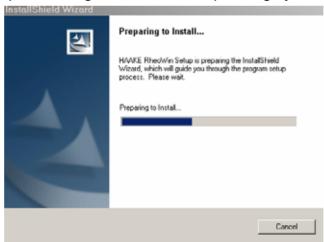
When the installation starts, the first step is to choose the language for the installation process.

As soon as this has been done the InstallShield Wizard is started.



The installation will now be prepared.

This can take a relatively long time, depending on the computer configuration and the operating system.



Entry of the Keys (Registration)

For the installation of HAAKE RheoWin 3 it is absolutely essential to enter a valid key for the measuring instrument that is to be installed. If this entry is not made the installation will not be carried out.

The fields of Module 1 (CFR part 11) can be left empty. HAAKE RheoWin 3 will then be installed without Module CFR part 11.

They Keys for the measuring instrument and the module are on the CD.

Key for measuring instrument

For the installation the key for the measuring instrument must be entered in the upper group of RheoWin3.

The company name, the serial number, and the key must be entered in the relevant fields. The entry of the company name and the serial number is case—sensitive.

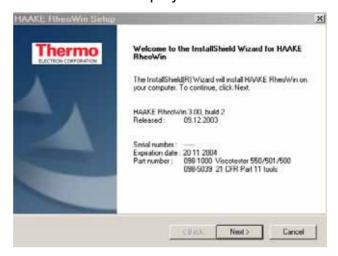


Key Module CFR part 11 (optional)

The Module CFR part 11 can only be used in conjunction with a measuring instrument.

Use HAAKE RheoWin 3 Installation / CFR Part 11 to install Module CFR part 11, "Operating instructions".

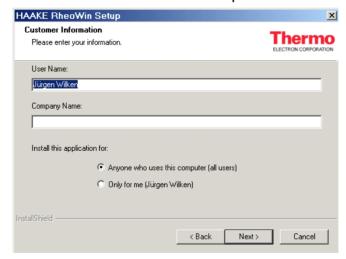
the information display:



HAAKE RheoWin Version, Serial number Measuring instrument Options Expiry date (of the demo versions)

Select whether the HAAKE RheoWin software is to be installed only for the user registered with the operating system or is to be accessible to all users who log in on this computer.

Standard: All users of this computer



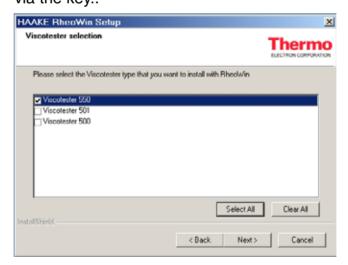
Select the folder into which HAAKE RheoWin is to be installed. The folder selection can be opened with the "Search" button.

Standard: C:\Programmes\RheoWin3



If the HAAKE RheoWin software has already been installed, the existing folder will be suggested as the standard installation folder.

Select the measuring instruments that are to be installed. This selection dialogue does not usually appear because exactly one type of instrument that is being installed is defined via the kev..



A further selection can be made in the case of certain types of instrument such as the HAAKE Viscotester. The instruments that are to be installed must be highlighted with the mouse button on the left of the name of the instrument. A check—mark (tick) is then visible.

If all instruments are to be installed, the "Select all" button can be pressed.

Select the measuring equipment.

Standard: All items of measuring equipment will be installed.

If "Select measuring equipment" is highlighted, groups of instruments can be selected via the selection dialogue.

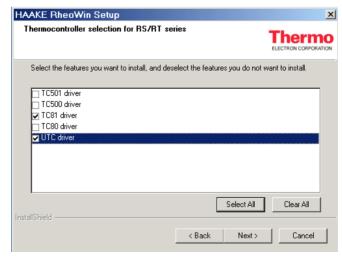


t is advisable to install all the measuring instruments because any that are not needed can be removed later in the programme.

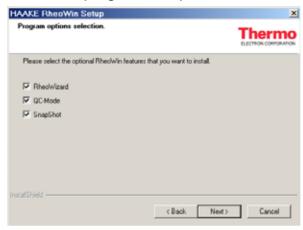
Select the thermo-controllers that are to be installed.

Here the thermo-controllers can be highlighted for which this measuring instrument is to be used.

The "Select all" button can be used to highlight all the thermo-controllers that are to be installed.



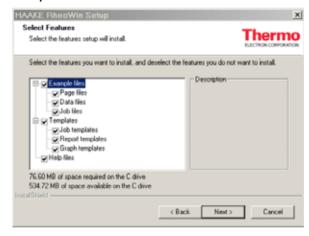
Select the programme options:



RheoWizard: Intelligent user guidance for creating a job sequence.

QC–Mode: Evaluation of analysis results.

SnapShot: Quick information on an unknown substance.



Here you can select the templates, examples, and help files that are to be installed.



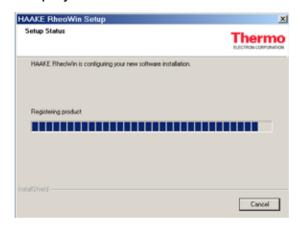
Select the programme file in which links are to be created to the HAAKE RheoWin programmes.

Standard: RheoWin3

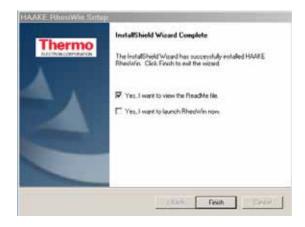


Select whether programme links are to be created to the HAAKE RheoWin Programmes on the desktop or in the task bar

Display the installation status.



Complete the installation.



Select and read the "ReadMe" file and start the HAAKE RheoWin Software as soon as it has been successfully installed.

Introduction



5. Introduction

5.1 HAAKE RheoWin User Manager

The User Manager contains the user administration. It serves as access control for the individual users. The administration of user rights is handled by an autonomous management console that is independent of the relevant application (Job or Data Manager).

Define users

The Administrator can

- open, copy, delete, and alter user accounts,
- allocate and withdraw users' rights.
- re–activate accounts (after an automatic deactivation), and deactivate them permanently.

Define groups

To simplify the creation of user accounts the Administrator can define groups of rights and allocate them to these users.

Rules for passwords

Certain rules can be laid down for the choice of passwords such as a maximum length or duration of validity.

Account parameters

Parameters can be defined for user accounts such as automatic deactivation after a failed attempt to log in.

5.2 HAAKE RheoWin Job Manager

Measuring sequences, the so-called jobs, are defined in the Job Manager. Job sequences, i.e. the starting and stopping of measurements as well as the administration of jobs e.g. storage functions or the creation of job templates are organized here.

The RheoWizard can also be called up and a snapshot made of a test.

How is a job defined in the Job Manager?

The "Job Editor" is opened when the "File – New Job" command is entered. Jobs are defined using so-called elements in the Job Editor. A job can consist of one or more elements. Elements are available under the headings "General", "Measurement" and "Evaluation". The headings can be represented graphically as a window at the user interface.

The elements can easily be dragged into their position by the mouse ("drag & drop") in the Job Editor and can be



Introduction

edited there (right mouse button). Once a job is completely defined, it can be started using the "Start" button. When the job is finished, i.e. all elements have been completed in sequence, a dialog commences and the results can be saved (file format *.rwd).

Using predefined templates and jobs

There are two possibilities to use predefined sequences in the RheoWin Job Manager.

A completely defined job, i.e. the fixed sequence of edited elements, can be saved so that it can be used again for future applications. When the Job Editor window is open, a name can be determined (file format *.rwj) using the command "File – Save". Jobs saved in this way will be displayed in the window "Jobs".

Jobs can also be saved as templates (file format *.rwt). In order to do this a sequence of elements in the job is determined but the elements are not edited. A list of available templates for selection is displayed with the command "File – New". The template "Blank job" corresponds to an empty Job Editor.

Unit configuration and information on the sample

There are three buttons in the Job Editor, with which unit and sequence—specific parameters and/or information on the sample can be entered. All specifications which are made under these three points belong to the job. You can also be added as elements to the job (see "General elements").

Introduction



5.3 HAAKE RheoWin Data Manager

The Data Manager is an evaluation program in which the saved measuring data can be separately displayed and processed.

Loading and viewing data

Measuring data (file format *.rwd) can be loaded using the command "File – Open". The options "Graph", "Table" or "Graph/Table" can be selected as the display form. The number of files can be selected for individual representation using the commands "Add file" or "Remove file".

The type and number of the desired measuring variables can be selected for both the graph or table display. The symbols and their colors as well as the lines and their colors can be individually edited in the graph display.

Evaluating data mathematically and rheologically

There are various mathematical and rheological models as well as analytical processes available in the RheoWin Data Manager with which flow and viscosity curves can be evaluated. For example there is the evaluation according to Casson for yield value determination or thixotropy calculation.

Regression results can be obtained in table or graph form.

Filtering data

Various mathematical filters can be used for representing and evaluating data in order to determine the number and distribution of measuring values individually.

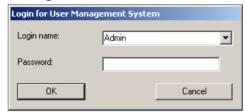
Outputting data

Measuring data and regression results can be obtained to a connected printer in the RheoWin Data Manager. Rheo-Win data and graphs can be embedded and further processed in Microsoft standard applications such as e.g. "Word" or "Excel" (for example via the clipboard).

6. HAAKE RheoWin User Manager

6.1 Settings for user administration

The administrator uses the programme UserManCfg.exe and logs in as "Admin". The following dialogue appears:

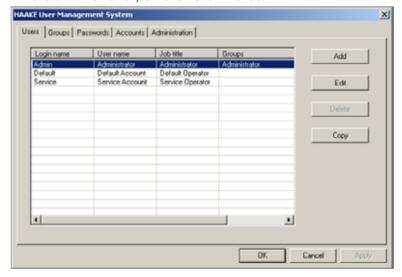


Each registration card of this administration dialog is described in the following Sections separately:

- Users
- Groups
- Passwords
- Accounts
- Administration

6.1.1 Users

When the UMS is installed there are already three users in it: "Admin", "Service", and "Default". The user "Admin" is the administrator, and she or he can:



- create, copy, delete, and alter user accounts
- grants or withdraw administrator privileges to other users
- activate user accounts, e.g. after an automatic bar, and deactivate them.

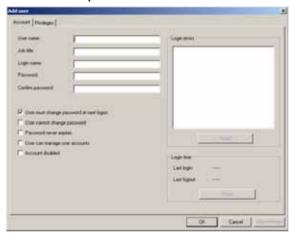
The Admin account can neither be barred nor deactivated; the three predefined accounts cannot be deleted nor automatically barred on account of non—use.

If the default account is activated the logging—in dialogue is automatically by—passed, i.e. there is no individual log—in and all users have the same rights, namely those of the default account. The administrator can restrict these if necessary.

Account

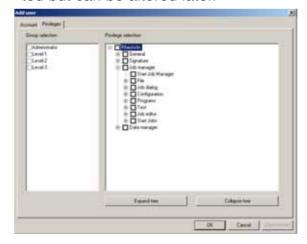
The log-in name is not case-sensitive: "Smith" is the same as "SMITH" or "smith".

In contrast to this, user IDs are case—sensitive; "Password" and "password" are not treated in the same way.



Rights

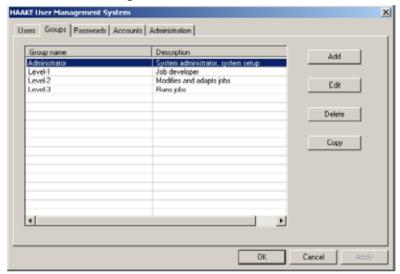
The rights that a user is to have to an application are allocated to her or him individually when the account is created but can be altered later.



To simplify the allocation of the same rights to several users, the administrator can define groups (see Groups).

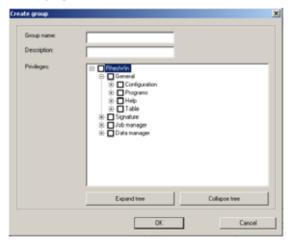
6.1.2 Groups

To simplify the creation of user accounts the administrator can define groups of rights and grant them to individual users. The effective rights of users result from the combination of the rights of all the groups to which they belongs and their own rights.



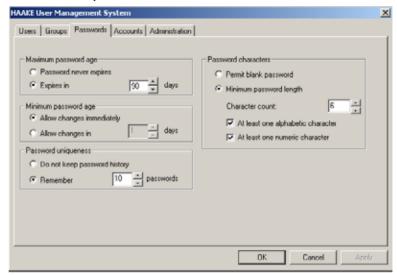
If an individual right is withdrawn that had been granted to her or him through membership of a group, that user ceases to be a member of the group but retains all her or his other rights, as do the remaining members of the group.

A group can be given different rights for different applications.



6.1.3 Passwords

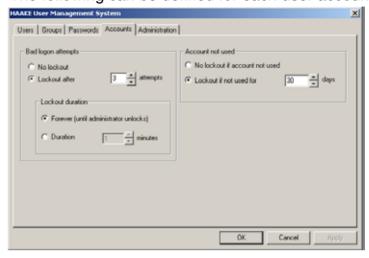
Each account is given a password. Rules can be laid down for passwords:



- Maximum duration of validity (the password becomes invalid after n days).
- Minimum age (the password cannot be changed until at least *n* days have elapsed).
- Uniqueness (at least *n* different passwords have to be selected before the first can be reused).
- An empty password is permissible.
- Minimum length of password (the password must be at least n characters long).
- Complexity rules (the password must contain at least one letter / one number).

6.1.4 Accounts

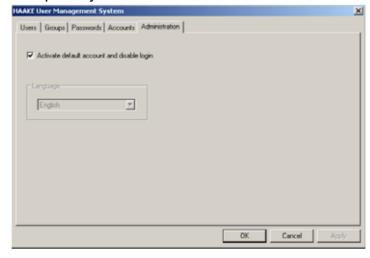
The following can be defined for each user account:



- whether it is to be barred after n invalid log-ins have been attempted,
- whether this bar is limited or unlimited (i.e. until the administrator reactivates the account),
- whether it is to be barred after remaining unused for n days (and can then only be reactivated by the administrator).

6.1.5 Administration

This is the position of the central switchboard that ensures the compatibility of the UMS with the rules of CFR21 Part 11:



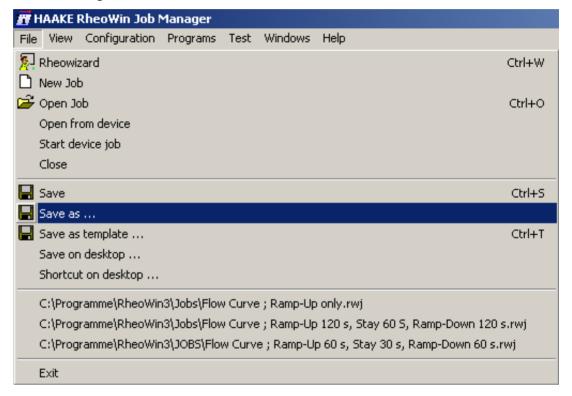
If the "default" account is activated, no individual log-in is made to the application. All users have the same rights, namely those of the default account, but the administrator can still limit them.

If the configuration dialogue is started from an application it is carried out in the language of the application, Otherwise the language can also be selected here.

7. HAAKE RheoWin Job Manager

7.1 Menu structure

This section describes the menu structure of the Job Manager.



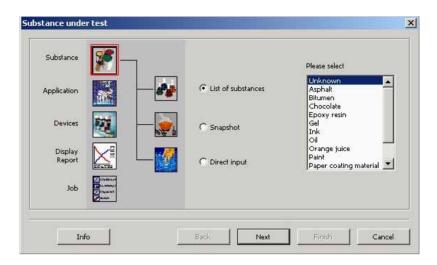
7.1.1 File Menu

Rheowizard

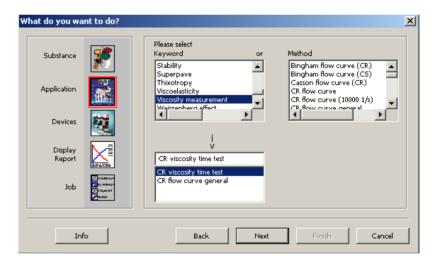
The RheoWizard helps in setting up a new job.



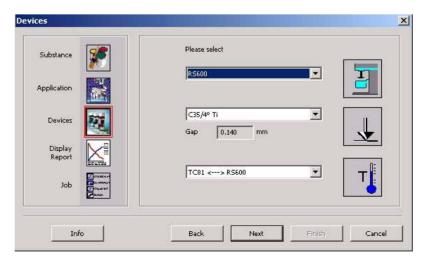
It provides a substance list from which the material to be examined can be selected. As an alternative a snapshot can be made or the material characterised by a direct input.



After this a job can be created from lists of key words and measuring methods.



The sensor system and a thermo-controller can also be selected at the same time.

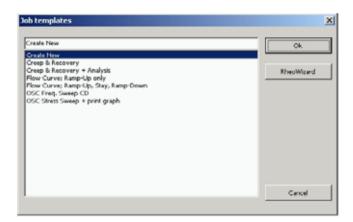


New job

A "job templates" window opens on selecting the function "File", New job. The selection "Make new" opens a blank job editor which can be configured by the user (see Chapter 7.3 "Creating jobs").

Furthermore, templates are already available containing a pre-defined sequence of elements corresponding to typical measuring sequences. It is only still necessary for the user to configure these elements (entering measuring parameters).

Templates have the filename extension *.rwt (RheoWin template). Templates can also be created by the user as *.rwt files and are then available under the command "File", New job.



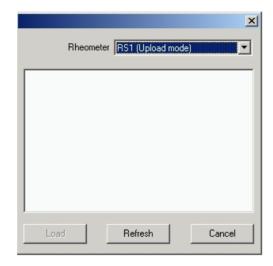
Open job

An operating system dialog window opens on selecting the function "File", Open job. A specified job (filename extension *.rwt) can be opened in this window. The default path "...\RheoWin3\Jobs\" is shown. The path can be changed by the user.



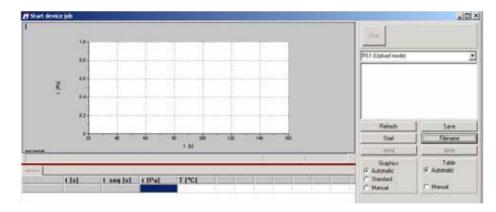
Open from device

When the "Open from device" function is activated the "Jobs" are opened that have been defined for the measuring mode described below (Upload mode).



Start device job

When the "Start device job" function is activated.



Close

The currently active window is closed on selecting the function "File", Close. An active window may be a job or an element template.

Save

The currently active window is saved on selecting the function "File", Save. The currently active window can only be a

job or a job template. If a filename already exists, the window is saved under this filename.

This function is disabled if there are only element templates on the desktop.

(This function is also accessible with the right mouse button.)

Save as

The currently active window is saved on selecting the function "File", Save as. This function always opens the save dialog window.

Save as template

A user-created job can be saved as template (filename extension *.rwt) by selecting the function "File", Save as template.

Templates created in this manner are normally kept on the path "...\RheoWin\Templates".

(This function is also accessible with the right mouse button.)

Save to desktop

A user-created job can be saved as icon on the Windows desktop by selecting the function "File", Save to desktop.

Shortcut an desktop

This enables the user to start jobs directly from the Windows user interface by pressing a button, without having to start the program.

This function can be used as SAFETY FUNCTION. Jobs can be started in the sense of various user levels because the program is password–protected (see also Section 7.2, "Configuration", Password).

(This function is also accessible with the right mouse button.)

Job signieren

Only appears in connection with the optional "CFR part 11" module.

Exit

When the "Exit" function is activated the Job Manager programme is terminated. A warning message appears if any jobs have been altered but not saved.

7.1.2 View Menu



Elementes

The element window is displayed if the box is checked. This is where the so-called elements are to be found for inclusion in jobs.

General elements (see chapter 8.1)

Measurement elements (see chapter 8.2)

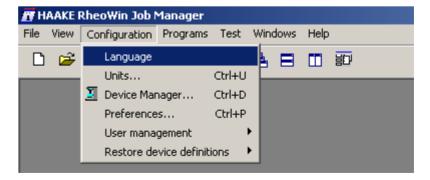
Evaluation elements (see chapter 8.3)

Jobs

If the box is checked the "Jobs" window is displayed. This is where the file names of the predefined jobs are shown and the jobs that the user has defined. They can be called up (in accordance with "File – Open job") and also deleted.

7.1.3 Configuration Menu

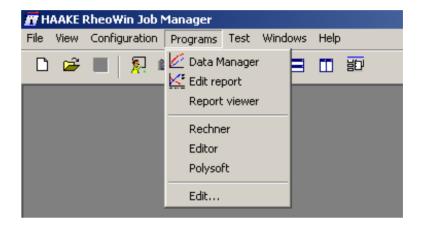
The main menu "Configuration" allows selecting and changing hardware, software and file management settings. From within the Configuration menu the device manager (see chapter 6) is accessible. For further information about the device manager see chapter 7.2.3.



A detailed description of all sub-points of the menu "Configuration" is in chapter 7.2.

7.1.4 Programs Menu

The main menu "Programs" offers the possibility to launch other applications, such as other parts of the RheoWin software, Windows applications or other HAAKE programs. (HAAKE RheoSoft)



Data-Manager

Start the RheoWin-Data Manager. A description can be found in capter 9.

Edit report

The "Edit report" command is used to open the Report files dialogue box. The dialogue box contains several report template files (Extension *.ist). After selecting a report template file and clicking OK, the report editor is opened.

The report is available as an evaluation element of the Rheo-Win ProJob Manager. The "List & Label Designer" instruction shows in detail how to create and modify the report template.

Report viewer

Start the "List & Label Designer".

Edit

The "Edit" command is used to define and modify a short cut to another program.

Short cuts to the **calculator** and **editor** of the Windows operating system as well as to the Thermo Haake software "HAAKE RheoSoft" are already defined. (Please note, that "HAAKE RheoSoft" is not a standard component of the RheoWin). Other links can be added.

7.1.5 Test Menu



Snapshot

The snapshot is a routine for quickly characterising a substance under which readings are taken in rotation and possibly oscillation. A protocol is produced at the end of the routine.

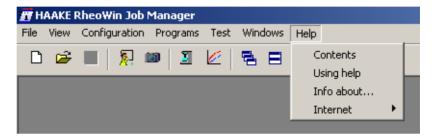
7.1.6 Window Menu

The element and job template windows can be arranged on the desktop surface in the main menu point "Window". This function is accessible in the same way as with other Windows programmes.



7.1.7 Help Menu

RheoWin contains a Online—help system that can be used by the operator for getting help when working with the software for the first time, or for getting a quick answer to a special question.



Contents

With the "Contents" command various help themes can be displayed onscreen. Several topics can be chosen from the Help window. The search for a specific word is also possible.

Using help

The "Using help" command offers a "help for the help system" from the current operating system, in the course of which the most common help functions will be explained.

Info about

The "Help" Info option shows information about version and serial number of the RheoWin software.

Internet

The "Help" Internet command offers a direct connection to the Internet, where Thermo Electron information and services are on offer. Available are

"Home page": direct link to the Thermo Electron Webside

"RheoWin update": The newest version of HAAKE RheoWin can be downloaded from the Thermo Electron Home page

"RheoWin Feedback": An e-mail with commentary and suggestion can be send directly to Thermo Electron (Karlsruhe) GmbH.

To use the internet–function of RheoWin, it is necessary to have a connection to the Internet.

7.1.8 Tool Bar

Certain functions of the main menu bar can be performed directly via the tool bar.



The following functions are available:

New Job (see chapter 7.1.1)

Open Job (see chapter 7.1.1)

Save (see chapter 7.1.1)

Job signieren (see chapter 7.1.1)

RheoWizard (see chapter 7.1.1)

Snapshot (see chapter 7.1.5)

Device–Manager (see chapter 7.2.3)

Data-Manager (open the Data Manager)

Cascade (see chapter 7.1.6)

Titel horizontal (see chapter 7.1.6)

Titel vertical (see chapter 7.1.6)

Default arrangement (see chapter 7.1.6)



7.2 Configuration

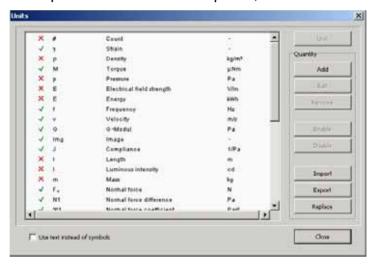
7.2.1 Language

With the "Language" command from the Configuration menu the dialogue language can be changed. English, German, French, Spanish, Polnisch, Portugiesisch, Swedish, and Japanese, Chinese (simplifies and traditionally) as well as Korean available (status February. 2004).

Whenever the dialogue language has been changed, all windows already opened will not be changed concerning their language.

7.2.2 Units

With the "Unit" command" all variables relevant for rheological measurements can be configurated. To change the unit of a variable, select the desired variable from the list, and click "Unit" to select another unit. For example, the variable "Torque" can have the unit "µNm", "Ncm" etc.



New values can be registered under "Add".

With the "Edit" command units can be changed and new units can be added to the list by defining a mathematical relation between the new unit and the basic unit.

With the "Disable" command the selected quantity will be put in brackets. These quantities are not displayed in quantity selection dialogues and other parts of RheoWin. With the "Enable" command the "Disable" command is cancelled.

With the help of the two commands "Export" and "Import" all quantities and their units can be stored in and retrieved from an Unit Data Files (*.udf). This feature can be used to transfer the quantities/unit settings from one computer to another.

"Replace" replaces the current set of dimensions completely with the definition from another file (Device Data Files *.udf).

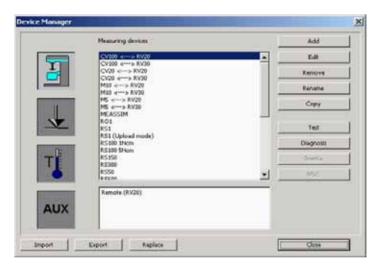
The set of dimensions loaded as standard can be found in the path "0\RheoWin3\Devices\All_device.UDF2.

7.2.3 Device Manager

7.2.3.1. Introduction

This chapter will discuss the possibilities based on the viscometer HAAKE Viscotester[®] 550 and rheometer HAAKE RheoStress 600.

The RheoWin Device Manager is the center for adapting the viscometer and rheometer to the software environment. Depending on the devices selected during installation there are different choices and options available. All items not highlighted are not active for the chosen option either because not selected or not applicable.



The main menu of the device manger allows to select one of the 4 main categories on the left hand side.



Sensors

Thermocontroller

Aux

The right side offers the possible actions to be executed in this category.

It is important that, for all the numbers with a decimal point, the full stop should be used as the decimal separator. Otherwise the digits behind the decimal point will be cut off in

HAAKE RheoWin and the entry will be set at zero.

33



Add

New settings can be generated with the "Add" button.

Edit

The existing settings can be confirmed or altered with the "Edit" button. All windows and menus can be opened here and the settings altered.

Remove

The selected device is removed from the list of devices.

Rename

A different name can be entered for the device selected in the list of devices. No other settings are affected when this is done.

Copy

The settings for the device selected are copied. The copy can be found in the list of devices under the name of "Copy of..."

Test

The "Test" command tests the communications between the computer and the device using the settings parameters of the COM interface. If the test is successful the connection parameters will be displayed. An error message will show that there is a problem such as a defective or missing cable or the wrong COM interface.

Diagnosis

During the diagnosis the status of the connected devices is read and the EPROM version number and other items of equipment information displayed. The device can still work both as a rheometer with a direct data display and in monitor device.

Inertia

Inertia can be active for /Devices/ or for /Sensors/; in both positions it is possible to automatically determine the inertia.

Position Device

Here, it is a must to remove all attached sensors (rotating parts) from the rheometer, because the intention is to measure the inertia of the rheometer itself. This value is then used to calculate the inertia of a sensor when a measurement is done with /Inertia/ in position sensor.

Inertia value = inertia value measured

Position Sensor

When the inertia measurement is selected in this position the value is calculated as:

Inertia value = inertia value measured - inertia value of rheo-

meter

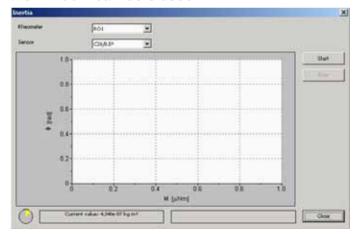
This shows the importance of a correct inertia value for the rheometer.

Implementation:

To proceed with the inertia determination, click on the inertia button.

The test method used is an oscillating stress sweep with the inertia calculation based on the slope of the deformation amplitude vs. torque preset. The procedure is predefined depending on the selection of device and sensor.

At the top of the graph the actual settings are listed for verification and with /Start/ the measurement beginns. At the end of the test a window appears with the newly calculated inertia value. Next to it is the old one shown as reference. If the deviation between the two values is very big without a good reason like different materials for the connected sensor (aluminium is lighter than titanium which is lighter than steel) the test should be repeated. When the result has been accepted the window can be closed.

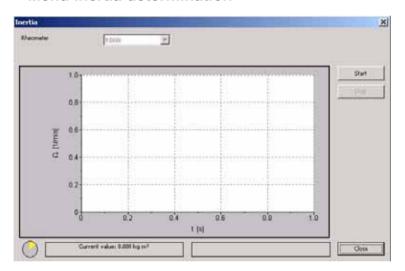


The method changes according to the connected measuring device:

A) HAAKE RheoStress reometers

A torque is set for the motor (or the motor with rotor) and it is recorded how long it takes to reach the final speed .From the measured data the inertia of masses can be calculated.

Menu inertia determination

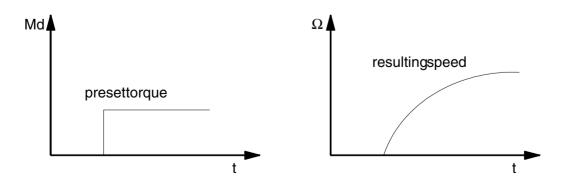


The inertia can be described directly by the following equation:

$$\begin{array}{rcl} \text{Md}(t) = I \cdot \dot{\Omega}(t) + f \cdot \Omega(t) \\ I &= & \text{moment of inertia} \\ \Omega &= & \text{angular velocity} \\ \dot{\Omega} &= & \text{first derivative of angular} \\ && \text{velocity} \\ && \text{Md} &= & \text{torque} \\ && t &= & \text{time} \end{array}$$

One solution of this differential equation can be found using a step/jump experiment with constant torque Md.

$$\Omega(t) = \frac{Md}{f} (1 - e^{-f/I \cdot t})$$



Based on such an experiment the values of f and I can be found with a regression analysis:

y = a (1-e^{-bx})
with:
$$y = \Omega$$
 angular velocity
 $x = t$ time
 $a = Md/f$

b = f/I

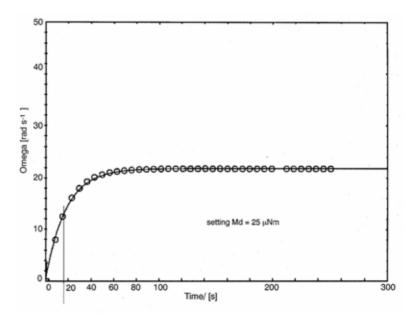
A test performed using a torque value of 25 μ Nm gives the following result:

This inertia value I represents not only the inertia of the rotor C35/4 in this example, but the total inertia consisting of the motor and sensor. Calculating the polar inertia using the standard cylinder, pyramid and other geometrical equations results in a value of $3.09 \cdot 10^{-6} \, \text{kgm}^2$. The rotating motor part of the Rheometer has consequently an inertia of approx.:

$$I_{RS/RT} = I_{Total} - I_{P35}$$

 $I_{RS/RT} = 19.8 - 3.09 = 16.7 \cdot 10^{-6} \text{ kg} \cdot \text{m}^2$

Test results: Determination of inertia using the C35/4 rotor



B) HAAKE RheoStress reometers

During a dynamic measurement of constant frequency and increasing voltage (torque) the characteristic curve of motor (motor plus rotor) is recorded and the inertia of masses is calculated.

The calculation:

Oscillation measurements are severely affected by the measuring system's angular momentum at high frequencies. It is especially important that this amount is determined preciselv.

If the "Newtonian oil" in the motor sensor of a mechanical system is periodically excited via a sine-wave shaped torque that has an amplitude M_{d 0} and frequency f, then the following is valid for the amplitude angle Φ_0 :

$$\Phi_0 = \frac{M_{d,0}}{I} \cdot \frac{1}{\omega \sqrt{\omega^2 + \left(\frac{\eta}{G \cdot I}\right)^2}} \tag{1}$$

Whereby I is the angular momentum, η the viscosity of the

$$G = \frac{A_{Sensor}}{M}$$

 $G = \frac{A_{Sensor}}{M_{Sensor}}$ and $\omega = 2\pi \cdot f$. For air oscillations the sensor Newtonian oil and tions, $\eta \to 0$, so that with sufficiently high frequencies the second term under the root in (1) can be disregarded as opposed to the first term. The equation (1) will then become:

$$\Phi_0 = \frac{M_{d,0}}{\omega^2 \cdot I}$$

If the resulting amplitude angle is now measured by different torque amplitudes (OSC-Stress-Sweep) and the measurement results are displayed as $\Phi_0 = f(M_{d,0})$, the angular momentum I can be determined from the ascending gradient

$$\frac{d\Phi_0}{dM_{d,0}} \text{ of } \qquad \qquad \text{this} \qquad \qquad \text{function.}$$

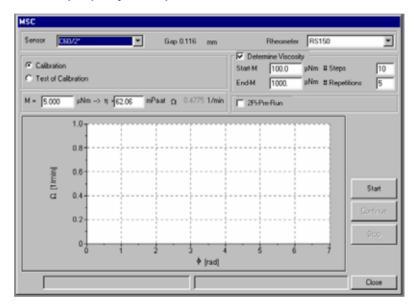
$$\frac{d\Phi_0}{dM_{d,0}} = \frac{1}{\omega^2 I} \qquad \qquad \text{or} \qquad \qquad I = \frac{1}{4\pi^2 \cdot f^2} \left(\frac{d\Phi_0}{dM_{d,0}}\right)^{\!\!-1}$$

These procedures are implemented in the HAAKE RheoWin software. In order to increase the accuracy it is recommended that the determining of the angular momentum for each

rotor (singular) should be repeated five times and the average value of the individual angular momentum is then manually entered in the sensor file.

MSC measurement

Micro Stress Control can improve the test results by a factor of 10 if properly set up.



The selected environment is shown (sensor and device) for verification and some options for the measurement offered.



MSC is the torque correction for physical positions of the rotor position based on a reference measurement. Therefore, the zero position is determined during powering up the rheometer and stored. It will correct the imperfectness of an individual bearing based on a measurement with standard liquid. The operator has two options; one is to include all deviations of the expected result in the MSC table and the other one to correct only for bearing errors.

Include all deviations in the table:

In this mode, the software has to know the viscosity of the test fluid which can be determined by a measurement. The test parameter can be set accordingly.

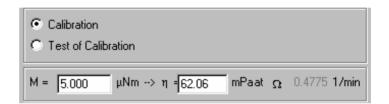


The MSC table will show relatively high correction values in the neighbourhood of 1 to 2 micro Nm and most of the time no extra offset values are required.

Include bearing imperfectness only

To record only the bearing properties and correct the remaining deviations with the separate offset factor (/Sensors/MSC/) the /Determine Viscosity/ option has to be unchekked.

To start the MSC measurements the settings for your system have to be optimized with a 2 PI pre—run before the real test starts.



Select /Calibration/ to determine the MSC table or /Test of Calibration/ to verify the newly determined data. A good torque value for MSC is 5 micro Nm which results in a certain viscosity value for a given sensor when a speed of about 0.48 rpm is desired. One can enter the torque value and adjust the viscosity with the variation of the temperature or enter the viscosity and accept the torque value as displayed.

Torque values below 1 and above 10 micro Nm are not very effective and should be avoided. High viscosity values and low torque values together result in very long test times. In this case it is a good idea to set parameters close to the default values.

At the end of the test run the speed and torque are shown on the screen which represents the 2 PI image of the air bearing. These data are now used (if MSC is activated) to correct steady shear and creep measurements.

The buttons for "Import", "Export", and "Replace" are located in the lower part of the dialogue window. In the case of "Import" the existing definition is augmented by an additional one, but in the case of "Replace" the old definition is removed and over—written by the new one.

7.2.3.2. Measuring Devices

Depending on the device(s) installed, one or more instruments can be selected.

The possible settings differ from one device to another. In the following pages the equipment is described by taking the example of the HAAKE Viscotester[®] 550 viscometer and the HAAKE RheoStress 600 rheometer.

HAAKE Viscotester® 550



Add devices

This function is very helpful if several instruments are connected to the same computer/software and need to be differentiated from each other. An "ALIAS NAME" can be given to each device installed like >VT550 with cylinder sensors<. To define the special parameters press "OK" to continue.



Edit General

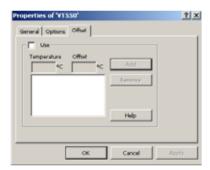
The important setting is the selection of the COM port to communicate with the viscometer. Check which Com port is physically connected and select it from the list box. The baud rate is set to 9600 and cannot be altered.



Edit – Options

The "Options" registration card allows the choice to be made of an automatic decimal—point correction in order to compensate for any torque shift. If the automatic torque compensation function is switched off the readings are used without any correction. This is recommended in the case of samples with a flow limit.

A communications protocol (between the computer and the measuring device) under "Other", which makes searching easier in the event of a possible error occurring.



Edit – Devitation

The PT100 temperature sensor can be adjusted to the measuring device in the "Deviation" registration card with the aid of a manually entered reference table. The deviations at given temperatures can be entered but at least two temperature settings are necessary; the software interpolates the intermediate temperatures.

If only one temperature is entered with its deviation, no interpolation will be possible. In order to obtain an interpolation with the highest level of approximation the relevant temperature range should be described at intervals of 10°C. Click on the "Use" control box in order to activate the correction.

Communikation Record

This function has been installed for service use. If this function is activated, all commands between the measuring device and the PC are recorded in a file named Driver.log (RV1.log, RS1.log, RW1.log) and are managed in the directory \rheowin\driver\.

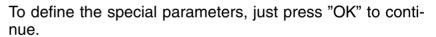
HAAKE RheoStress reometers

The option RS600 offers almost all possibilities in the device manager and can be used as information for all HAAKE RheoStress rheometers.

Add

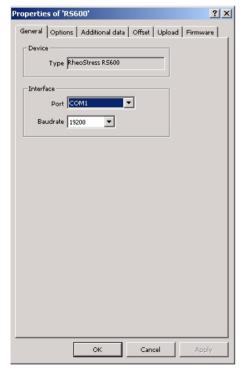
This function is very helpful, if several instruments are connected to the same computer/software and need to be differentiated from each other. An "ALIAS NAME" can be given to each device installed like >RS600 with Peltier<.

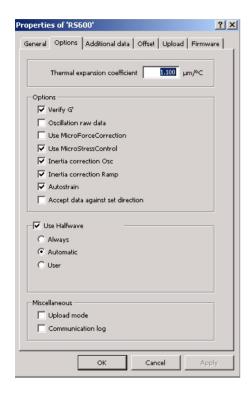




Edit General

With /Edit/ the actual settings for the RS150 called >RS150 with Peltier< can be individually set to preferred values such as the OM Port and the baud rate. Choose the correct port and the maximum baud rate possible e.g. 19200.





Edit – Options

Thermal expansion coefficient:

When measurements are performed at different temperatures (e.g. temperature ramp), the thermal expansion of the system must be taken into consideration.

The default value of 1.1 microns per °C can be changed if required but according to our experience it is good for all steel and titanium rotors with and without ceramic shafts.

Data calculation at low rpm:

In the range from maximum speed down to about 5 rpm the speed signal is refreshed fast enough to get good data. Below 5 rpm it takes quite a while to get a valid speed signal. Therefore an optional procedure for the speed determination can be selected based on the basic equation: speed = d(distance) / d(time). An internal buffer collects pairs of data for position and time and the derivative is the speed value.

Verify G'

The calculation of G' from an dynamic experiment is based on the phase shift angle between stress and strain and the signal amplitude (G*). The calculation of the phase angle is correct but the value is in certain ranges dominated by the inertia of the rheometer and not related to the test material. This influence is significant when non–elastic materials are tested such as calibration oils which theoretically should not show any elastic component. When G' is checked the phase angle is set to 90° if the elastic response measured is very close to the effect created by the inertia of the rheometer. If the option is not checked the data are calculated with the normal inertia correction.

Oscillation raw data

When this option is off there are no sine wave data transmitted from the rheometer except the amplitude ratios and the phase angle. The data evaluation is completely done within the rheometer. If the box is checked, then the individual data of both sine waves (stress and strain) are transmitted to the application software and displayed on—line in a small window below the results graph. The advantage is that the shape of the sine waves gives additional information about the quality of the data with the disadvantage that it slows down the testing speed.

Use Micro Stress Control

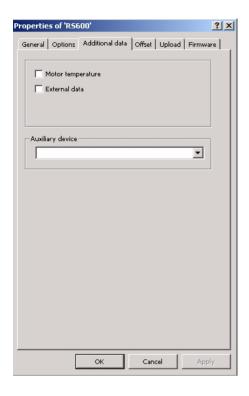
Every rheometer has a certain dependency of the response on the actual position of the rotor in reference to a full rotation (2 PI). This effect can be a symmetrical response like a sine wave dependant speed (viscosity) when a constant stress value is applied or an asymmetric effect due to certain mechanic tolerances. Both responses can only be evaluated if this special rheometer's behavior is known by the software. If the response has been determined with the "MSC Test" all future measurements can be corrected based on this test. MSC is active in all tests except dynamic experiments.

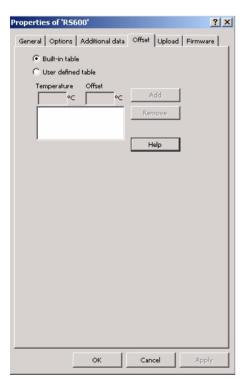
Activate MSC: Check MSC and close the device manager. To make sure that this information is activated at the HAAKE RheoStress 600 switch the instrument off and on again. The rotor will rotate for several turns to search for the reference mark on the encoder disk in the measuring drive unit. This reference mark will be activated as zero position until the instrument is switched off or MSC deactivated.

Inertia correction Osc
Inertia correction Rampe
Use Halfwave

Communikation Record

This function has been installed for service use. If this function is activated, all commands between the measuring device and the PC are recorded in a file named Driver.log (RV1.log, RS1.log, RW1.log) and are managed in the directory \rheowin\driver\.





Edit - Additonal data

Motor temperature

For service reasons the motor temperature can be monitored. This is advisable only if measurements are performed for longer times at the limits of the upper torque range. Overheating is prevented by an automatic cut off at 65°C.

Normal force

The newer rheometers RT20, RS75, RS80 and RS150 can measure the normal force by using the air bearing position as a reference signal. In normal mode without pressure applied to the air bearing the position is "0". If there is a force applied in normal direction the air bearing is pushed out of this "0" position and measured by an optical sensor. The relationship between position and force is calibrated for an air pressure of 2.5 bars; other values require corrections.

External data: Current gap

Edit - Offset

In /Read Offset/ the temperature probe PT100 can be adjusted by a reference table entered manually. The known temperatures with their deviations can be entered for different temperature values to allow the interpolation of offsets in a measurement between the set data. If only one temperature with offset is entered an interpolation is not possible. The most accurate way is to describe the interesting temperature range with steps of 10°C for accurate interpolation. Check the /Use/ box to activate the read offset.

7.2.3.3. Sensors

In sensors the calculation factors and geometry settings for selected sensors can be altered, new sensors added or obsolete ones removed.



All sensors available in the software are listed because the device manager does not know yet which instruments are or will be connected. Add, Edit and Remove are active choices as previously described (see capter 7.2.3.2).

Edit General

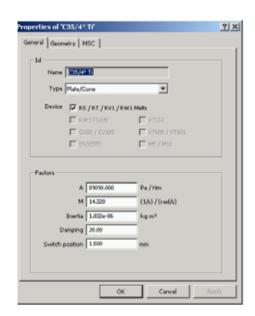
With /Edit/ an options menu is opened to view or alter the present data. The values shown are used for controlling and mathematical calculations.

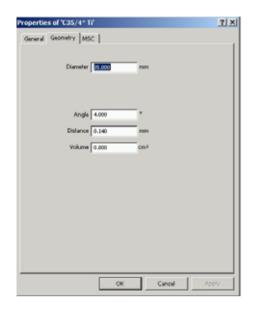
The <u>factors A and M</u> are used to calculate shear stress and shear rate.

Inertia is used in inertia correction during oscillation and ramps. The inertia value is usually determined with the /Inertia/ measurement.

Damping can be a value between 10 and 90 with 90 meaning a very slow CR control loop used only with inhomogeneous, low viscosity fluids and 10 only for very high viscous materials. Intermediate values of 20 to 40 are recommended.

Switch position is the gap distance when the lift speed is reduced before touching the sample. Values of 1 to 1.5 mm are recommended.





Edit Geometry

Here it is possible to change the geometry factors of the selected sensor. The important value /Distance/ is the gap between the lower part and the upper part of a sensor. The other data are for documentation and identification purposes.



Edit MSC

Each RS600 rheometer connected is identified by the serial number because RheoWin can do multitasking with more than one device. The serial number identifies the MSC settings for this sensor. If more than 1 rheometer is/has been connected, choose the correct device for the actual environment.

Default: is the standard setting with an MSC table for the rheometer without sensor active. This is the minimum correction possible besides deactivating MSC in /Devices/ or resetting the table to zero values.

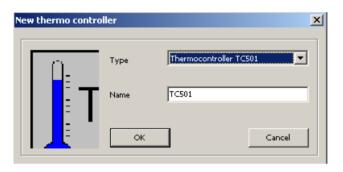
Offset: Is only a constant torque value which is added to any output torque value

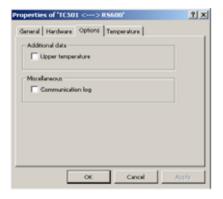
Offset and table: allows to correct the bearing for a 2 PI distance with an offset and a data table which is usually generated in /MSC/. Data can be inserted or removed for manual adjustment.

7.2.3.4. Thermocontroller

The same philosophy can be applied to this section as to the previous device section. Thermocontroller can be connected to a viscometer/rheometer (Port = Remote) or to a port at the host computer.

Example: HAAKE Thermocontroller TC501 connected to the HAAKE RheoStress 600







More Thermocontrollers can be added with /ADD/ or deleted with /REMOVE/. Test and Diagnose verify the Thermocontroller and display its status.

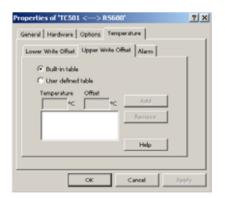
Because it is connected directly to the HAAKE RheoStress 600 control box the port /REMOTE/ has been selected and as master unit /RS600/. More options are different COM ports or other rheometers. Choose the appropriate settings for your installation.

Hardware

Select in the hardware section which items are connected to the TC501 controller such as cylinder, plate or cone heater with base plate which activates the corresponding PID control factors. Wrong settings result in bad temperature contol.

Choose the correct cooling media to improve cooling rates and temperature overshoots.





Options

Additional data

The temperature of the cone heater (upper heating zone) can be monitored when checked for service purposes.

Communikation Record

This function has been installed for service use. If this function is activated, all commands between the measuring device and the PC are recorded in a file named Driver.log (RV1.log, RS1.log, RW1.log) and are managed in the directory \rheowin\driver\.

Temperature

The temperature section defines the temperature settings, alarms and offset tables.

Use /Default/ means to set the entered temperatures when powered up and leave as last temperature setting the /shut-down/ value. This value is kept until a new value is transmitted to the TC501 controller.

Offsets and alarms

Enter offsets and alarms if needed or desired.

Lower write offset: the set value is entered as control temperature for the lower plate /cylinder plus offset or the interpolated offset.

Lower read offset: the measured value is entered in the software for the lower plate /cylinder plus the offset or the interpolated offset.

Alarm: Allows to enter alarm temperature values for lowest and highest temperature tolerable.

7.2.3.5. AUX divice

For hygrometers the COM interface can be configured under "General", and the communication protocol can be activated under "Options".



For manometers such as, for instance, in the utilization with the pressure measurement cell D400/300 (up to 400 bar and 300°C) the COM interface can be configured under "General".

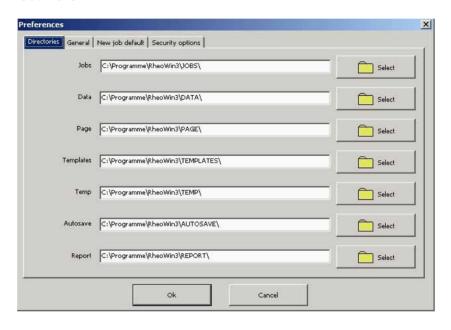




7.2.4 Preferences

7.2.4.1. Directories

The respective standard registers for jobs, data, pages, patterns, temporary data files, auto—save and reports can be set here.



7.2.4.2. General

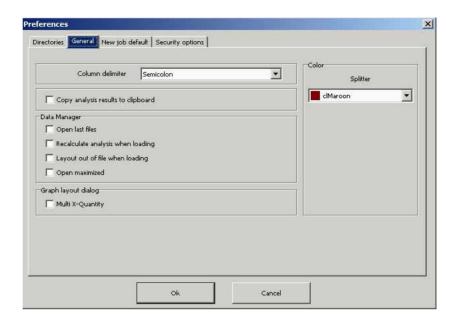
The space character, semicolon, comma or the tabulator can be selected as column separator.

It can be selected whether to insert analysis results in the interim memory.

For the data evaluation it can be determined whether the data file loaded last should be opened, whether the analyses should be updated during loading, whether the page layout from the data files should be used when opening the files and whether the window should be opened at maximum.

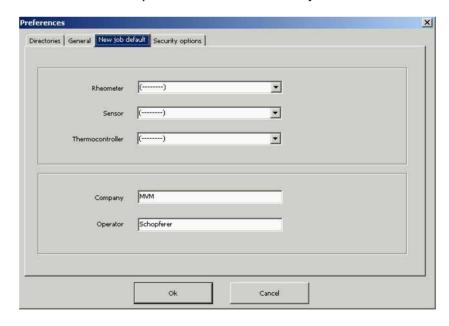
For the graphic dialog one can select with the option "Multi X-Quantity".

The color of the separator bars in the measuring window can be selected under "Splitter".



7.2.4.3. New job default

Here the used Rheometer, the Senorsystem and a Thermocontroller can be put as a model for new jobs.



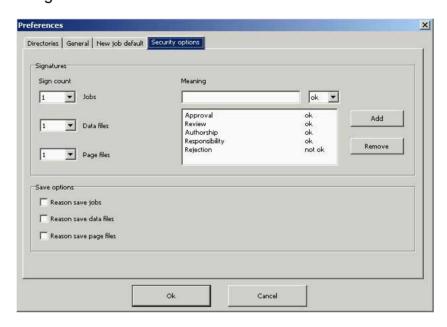
The company name and the processor's name can be preset.

7.2.4.4. Security options

Only appears in connection with the optional "CFR part 11" module. The number of the signatures for jobs, data files and data pages can be set between one and five.

The meaning of the terms approval, review, authorship, re-

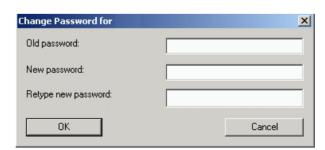
sponsibility and rejection can be set between "ok" and "not ok". Other terms can be added or deleted. The function "Reason" can be set under "Memory Settings" when storing jobs, data files and pages. If the function is activated, a window requesting a reason from the user is opened prior to saving.



7.2.5 User management (see capter 6.)

7.2.5.1. Password modification

The pass word for the actual user can be changed here. For this purpose, the old pass word must be entered. The new pass word must conform to the rules determined by the Admin in the User–Manager.



7.2.5.2. User–Manager (see capter 6.)

7.2.6 Restore equipment definitions

Automatic back-up file

With this menu item the equipment definitions which were set prior to potential modifications are restored after confirmation.

From file...

The equipment definitions saved in a RheoWin Device File (Data file ending in *.flp) are read in and used for further work with RheoWin. The standard definitions are located in the data file "DRIVERS.FLP" in the RheoWin file "...\RheoWin3\Drivers".

7.3 Creating a Job

Jobs can consist of one or more elements. A Job can contain general elements (such as Lift control, file loading, printing a protocol etc.), measurement –and evaluation elements. It is possible to define a job that only performs evaluations on stored measurement data. The Jobs window lists some predefined job files. They can be loaded, changed and saved under a new file name.

Every element is identified by an icon, that facilitates the access to the function of the element. By moving the coursor over the icon a short explanation appears. At the example shown below, the note pad with pen indicates the possibility to take some notes on the sample identification.

Drag and Drop



To draw the elements from the element window to the job window, the element is clicked on with the left mouse key and drawn into the job window by keeping the key depressed. The sequence of the elements in the job window can be changed in this manner as well.

Some elements must be edit or correctly defined, otherwise they will be displayed as shown below.

If such elements are retained within the job, RheoWin will not execute them. The job is still carried out.

Press the right mouse button in order to edit the elements (the middle mouse button is not supported).

Elements can be deleted from a job by drawing them into the trash bin.

Every job can consist of measurement elements, evaluation elements and general elements. It is possible to insert new elements, to remove or change elements into a predefined job. Especially within the measurement elements it will be necessary to define the exact measurement parameters (for example: ramp up to, number and distribution of data points.)

The temperature for all elements can now be set/changed at once in one dialog. This dialog is accessed by clicking on the button with a fuchsia coloured T. In the case the Job contains one or more temperature elements of some kind, the dialog offers the possibility to shift the end temperature according the the change in the start temperature.

7.3.1 Display

In the "Display" window, the settings for the onscreen display during the measurement can be selected, such as a graph, protocol, and/or table.



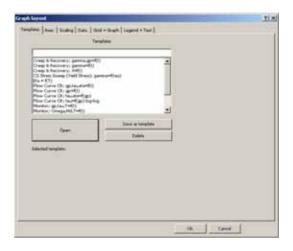
In the example shown above, the graph and the table will be shown during the measurement.

In the settings for the protocol one can select which information is to be displayed.

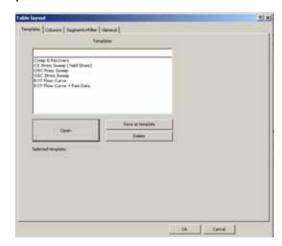


The "manual graph" option offers the possibility to choose a graph layout from a list of given templates (here a flow— and viscosity curve as a function of the shear rate).

All formats of illustration (such as, e.g. the scaling of the axes or symbols for the curves) can be varied and supplemented as well. A modified pattern can be saved.



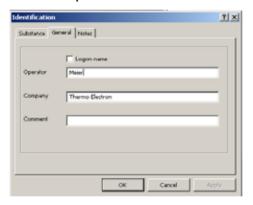
In the manual table setting one can also select from given patterns. Other quantities can be entered into the table and the format of the numbers can be determined. A modified pattern can be saved.

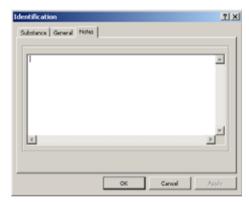


7.3.2 Identification



The "identification" window can set to be opened automatically at the beginning of a job. It is used to enter the name of the operator, some notes and all information about the sample substance.

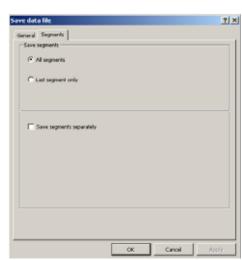




7.3.3 File name

Other options are automatic overwriting, automatically replacing invalid symbols, saving the table in ASCII-format, selection of the segments to be saved and DO NOT save data.





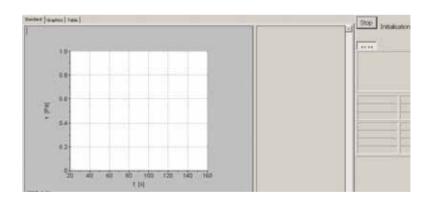
7.4 Job execution (Start)

The execution of a job is started with the key "Start". Now the individual elements are processed in sequence. First the window of the JobController is displayed. This is the measuring window where all information regarding the measuring defined in the job is displayed. After the initializing of the measuring device the measuring position is started up which is illustrated by an animation in the JobController.

7.4.1 Standard

In this display all information, previously set under "View", is displayed simultaneously. The measuring values are graphically displayed in the top left, below are all measuring values in the form of a table. The information regarding the "Identification", the selected measuring system as well as the individual elements of the job, is displayed in the center. The buttons for the control of the job are located all the way on the right hand side. The job can be interrupted with "Stop". With a click on the green arrow one can jump to the processing of the next element. A click on the icon with the diskette saves the previous measuring values.

7.4.2 Graphics



In the tab "Diagram" the measuring values, previously defined under "View", are graphically displayed.

A right—click on the diagram surface produces a **context menu** containing the following options:

Load template

Predefined layouts can be loaded in the same way as under "Graphic layout – templates".

Save as template

After any changes have been made the current layout is stored as a future template.

Print

Print graph only

Save

The current graph can be saved under one of the following formats: "Meta File" (*.wmf), "Enhanced Meta File" (*.emf), or "Device Independent Bitmap" (*.dib).

Copy

Copies the graph onto the Notepad, from where it can be inserted into other programmes.

Restore scaling

Within the drawing area the presentation can be enlarged by generating an area with the left mouse button onto this area. The original scale can be presented by selecting the menu point "Restore scale".

Cross hair

Display appropriate picture

This works only in connection with measurements taken with the HAAKE RheoScope1.

Add text...

A text box is generated that can be positioned anywhere required and then edited or deleted with a right-click.

Add clipboard text...

A text box is generated that contains text from the Windows Notepad. It can be edited or deleted with a right—click.

Graph Layout

It opens a dialog window with more setting options. This can also be achieved with a double click on the diagram surface. With a right click on one of the axes one can switch between linear and logarithmic scaling. A right click on the lowest or highest number of an axis permits the modification of the respective minimum or maximum value of the axis.

The legend can be moved to a different position with a left click and by drawing the mouse. A double click on the diagram surface opens a dialog window with more setting options.

All options of this tab are also available in the display "Standard". However, modifications in one display have no effect on the other display.

A detailed description of all sub-points in the "Graph Layout" menu can be found in capter 10.

7.4.3 **Table**

In the tab "Table" all measuring values, previously defined under "View", are displayed in a table.

If more values were defined than columns fitting into the display window, the other values can be displayed with the scroll bar at the bottom. The number of "fixed columns" is always displayed in the illustration. With a right click on a column the value displayed there can be changed or a value can be added. The width of the columns can be modified by clicking and drawing of the separating lines with the left mouse key.

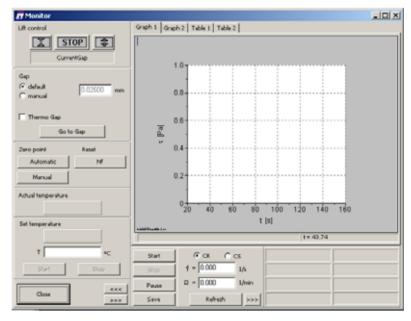
All options of this tab are also available in the view "Standard". However, modifications in one display have no effect on the other display.

A detailed description of all sub-points in the "Table" menu can be found in capter 10.

7.5 Manal control

With a click on "Manual operation" a monitor window opens which permits the control of the Rheometer and a manual measurement.

With "Close" the manual operation is terminated. Clicks on the double arrows expand or reduce the display of the monitor window.



7.5.1 Lift control

With the graphic symbols "Approach together", "Stop" and "Approach separately" the lift of the Rheometer can be controlled. With "Approach together" the lift travels to the top stop, with "Approach separately" it travels to the first bottom switch—off position. Another shut—down (if necessary, to the swivel out position) is possible only with the control buttons on the Rheometer.

With "Approach gap" the measuring device approaches the position of the measuring gap. With "Automatic" or with the selected measuring device parameters the position of the job definition is determined.

With "manual" the height of a gap can be set. The option "Thermogap" assures that the gap is maintained with activated tempering. (See Chapter 7.2.3.2).

The zero point which must be re-determined after each change of the measuring equipment can be set here automatically or manually. With an automatic setting the equipment approaches the top stop together and then sets the gap to zero. With a manual setting, the gap is set to zero in the current position.

7.5.2 Thermocontrollerbedienung

The current temperature is displayed in the window "Actual temperature".

If a tempering unit has been selected in the Job-Editor, a temperature can be entered under "Target temperature" which is approached by the tempering unit after clicking on "Start". It must be observed that the probe has sufficient time to assume the set temperature. With a click on "Stop" the tempering is terminated.

7.5.3 Manal Messung

When selecting "CR", a value can be set for the shear rate (Gamma point) or the angular frequency (Omega), when selecting "CS", the shear stress (tau) or the torque (M) can be set. Then the measurement can be started via "Start" with these specifications.

A click on "Pause" interrupts the measurement, another click continues the measurement.

With "Save" the data is stored in a RheoWin–Datafile (*.rwd). The measured values are displayed as a diagram or as a table depending on the selection. The layout of diagram or table can be set analogously to the description in the chapters 10.2 and 10.3.

7.6 Jobs in the Upload-Mode

The rheometers can be operated by the software RheoWin in "direct mode" or it can be loaded by special functions measuring programs in the store of the measuring device. By that, the measuring device gets independent of the PC and it is only connected for programming or data transfer. The measuring programs ("JOBs") are started after the corresponding programming with the display control unit.

For programming or data transfer the Rheometer must be connected to the PC via RS232 cable as usual and Thermo Haake software RheoWin must be loaded.

After installing Series 1 Rheometer two versions per model can be found in the device manager. The device e.g.HAAKE *RotoVisco1* is running in the software direct mode as usual. Model *RV1* (*Upload mode*) has already been pre–defined as independent device.

The characteristic "Upload Mode" is transferred to the device driver on the following card which can be found under "Edit". So you can give the device driver the characteristic "Upload" or "Software" mode. For practical reason it is advisable to define a new device for each operation.

Other options on the device record card are:

Torque Compensation

When the torque compensation is activated, the present value of the torque display is set on zero "0" right before the measurement so that possible faults (offset faults) can be reduced. This is always advisable when samples without "yield point" are to be measured. In case of doubt it is measured first without "Torque Compensation" and after that it is decided whether the function can be used.

Inertia of Masses Correction Ramp

If fast speed ramps (< 180 s) are driven, the dampening set at the sensors (usually 30%) and also the inertia of the masses of the measuring device (motor and rotor) enter the result. Fast ramp speeds make up a hysteresis curve which does not come from the sample but from the test conditions and the measuring device. The influence can be seen qualitatively in the following diagram. It is recommended to drive measurements with ramp times of less than 120s without correction only in special cases. With correction, the influences of the inertia of masses are compensated. This can be tested individually at examples relating to practice.

Communikation Record

This function has been installed for service use. If this function is activated, all commands between the measuring device and the PC are recorded in a file named Driver.log (RV1.log, RS1.log, RW1.log) and are managed in the directory \rheowin\driver\.

Upload

Under "Upload" you can find the present data for user, sample and sensor. These data are entered with RheoWin and are read out. In the display unit these exact terms can be selected. If you are connected to the measuring device at the running time the present level of the store is read out with "READ" and is displayed. Changes can be made. They are transferred to the measuring device while leaving the menu and are available now. If the device has not been programmed yet (state at delivery) all lists are empty.

Now the three record cards can be filled with information. With the mouse and the cursor the first position is clicked and e.g. a name is entered. After that the second line can be clikked and other entries can be made.

When all names of possible users are entered, the names of the samples which are to be measured can be entered. Only these names can be recalled from a selection list.

In principle the same is valid for the sensors of the rheometer. It is recommended to enter only the sensors which are really in existence from the list of all variants to keep the display clearly organized.

The sensors of the right window are transferred to the measuring device if you leave the menu with <OK>. With Cancel the existing adjustments are kept. If the factors are changed, e.g. after a calibration, the values of the sensors can be renewed with <READ> after the reading of the store.

Drawing up JOBS for Uploading

JOBs or measuring and evaluation procedures are drawn up with the RheoWin Job manager. The distinction takes place before the drawing up of a JOB at the selection of the measuring device. If existing JOBs are changed afterwards unexpected reactions can take place. The following proceeding is recommended:

First the measuring device is selected. After that the procedure is drawn up.

After the selection of the measuring device, the sensor and a temperature control unit are selected. If no temperature control unit is selected, no temperature control can take place. No selection (——)also means that the temperature control unit is switched off.

The measuring process is now composed according to the requirements of the application or of the user just as in the

PC mode. There are some restrictions of the length of names and the selection of elements. These restrictions are visible only at editing.

After the drawing up of a JOB it can directly be started for a test or it can be uploaded to the measuring device. On this occasion a list gets visible that shows an empty list or already existing JOBs. The order or position of the list corresponds to the reservation in the measuring device. So you can make an allocation indirectly. Empty positions have to be avoided, because otherwise the following entries would be ignored.

Uploaded JOBs can be called in via the display control unit and can be carried out directly.

Reading back uploaded JOBs from the measuring device

JOB's can be read back from the measuring device with the RheoWin function /File/Open Job of Measuring Device/. This function corresponds to the reading of the hard disk.

If this function is selected, all installed unit drivers are called in and it is tested if a connection can be made. Drivers that are not installed correctly cause error messages and should be cancelled from the device list (device manager). If a measuring device with the possibility of managing JOBs responds it is selected and displayed immediately.

In the white area the existing JOBs are listed for selection.

Measuring result

The measuring result is a chart with measuring and preset values according to the measuring definition. It can be transferred, if defined in the JOB, directly to a printer. The result is then shown as selected. Segments can be printed or ignored. The selection of the column contents takes place via the list menus:

Importing the measuring result in RheoWin

The last measuring result can also be imported directly to the RheoWin software (measuring device connected to PC and RheoWin active). For this the file in the DATA manager is opened.

From the following list the device of your choice is selected for reading out the data. This is necessary because the software RheoWin can drive and read several units by multitasking.

After the selection the data are transferred via RS232 interface from the measuring device to the PC/RheoWin software. The data are treated like comparable values of the local hard disc or of the network.

8. Job Elements

8.1 General elements

General elements such as "Load data file", "Save", "Print" as well as "Identification / Notes" can be integrated in automatic measuring processes. This is particularly advantageous and time saving for routine measurements, since the manual evaluation in the data manager can then be dispensed with. The utilization of the general elements also permits the preparation of straight evaluation jobs, which do not include any measuring elements.

In the case of RT/RS-equipment with electronic distance setting, the motion sequence of the equipment can be defined in a job via the installation of one or several lift elements ("Lift control").

Some examples follow:

1. Example: Immediately following the measurement, the diagram, the protocol and the value table should be printed out. It is recommended to enter the data file name and the probe identification at the beginning of the job. It is reasonable to manually set the values to be shown for the diagram as well as for the table.

In this case the diagram, table and the protocol are printed on different pages. If it is desired to print out one page only with the complete information, the print out of a report is to be recommended. The report element can be found with the measuring elements, and a printing element must be selected again afterwards.



8.1.1 Identification / Notes

The options "General", "Substance" and "Notes" can be activated. The activated options are read during the processing of the job. Information which was already entered in the Job–Editor under "Identification" is later displayed as a specification in the respective fields.



8.1.2 Message

A text message can be entered under "General" which is displayed on the screen when processing this element. With the activation of "Continue automatically to" and the entering of a time, the window will close automatically after the expiration of this time. The option "Always in the foreground" prevents a covering up of the message by other windows. An audio data file in WAVE format (*.wav) can be selected under "Acoustic signal". It is activated via "Use". The data file can be played after the selection via "Test".



8.1.3 File load

This element permits the selection or the fixed specification of an evaluation data file (data file ending: *.rwd). If "Select" has been activated, the data file must be indicated during the processing of the job via a selection dialog. If "Fixed data file" has been activated, the data file can be specified via "Load data file".



8.1.4 Save datafile

Under data file name it can be set, whether the question for the data file name is to be displayed at the beginning or at the end of the job. Optionally, fixed or variable data file names can also be generated. For variable data file names a fixed part can be specified. In addition, various different name parts can be generated. For instance, those are the time or the batch number.

Other options include automatic overwrite, replace invalid symbols automatically, save table in ASCII format, do NOT save selection of segment and data to be saved.



8.1.5 Data export

This element allows the saving of the data in an ASCII data file. Like in "Save data", fixed or variable data file names can be generated. The information to be saved can be selected under the tab "Select". The option "Report" opens the report preview. The job is then continued only after the termination of the job preview.



8.1.6 Graphic / Table definition

When reaching this element, the display of the diagram or the table is in accordance to the settings in this element. The original display options which were set in the Job Editor under "Display" are now overwritten.



8.1.7 Show data window

Displays the measuring window and waits for manual clikking of "Continue". Only then the job is further processed.



8.1.8 Report

Via this element a report pattern can be loaded and certain settings can be adjusted in the report pattern (e.g. title).



8.1.9 Print

Depending on the option activated, a separate page is printed for "Diagram", "Table" or "Protocol".



8.1.10 Lift control

The options "Zero point", "Measuring position", "Standby" and "Lift separate" can be selected. If a message is issued after the reaching of the position, the job is continued only after the confirmation of the message. If the measuring position is not reached within the time indicated under "Options", the job is terminated immediately. The option "Start measurement without correct gap (PK)" prevents the same. The measuring position can also be approached with normal force control in devices which have the option normal force. In addition, the ThermoGap is activated in the lift element.



8.1.11 Temperature setting

A temperature can be entered which is approached by the connected Thermocontroller. For this purpose, a maximum variation and a maximum waiting period can be defined. If one of these / or conditions has been reached, the job is continued with the next element. During the tempering a window appears which displays the current temperature and the specifications. With "Accept" one can immediately jump to the next element.



8.1.12 Camera control

This element is only reasonable in connection with the HAAKE RheoScope1.

The settings of the camera control can be entered from a specification data file.

During the processing of the element the settings can be modified. Modified settings can be saved and other settings can be loaded. The settings data files are located in the Rheo-Win3–Register /Templates/.



8.1.13 Reset

The previously expired global time is immediately reset to zero or with the start of the next element. The time of the element (tseg) continues to run.

This is often helpful when too much time in relation to the overall time is passing between the preparation of the probe (load probe, approach measuring position, first measuring points) and the actual measuring. With that, every user can determine when the measuring time begins to run.

In addition to the time, the normal force N can be set to zero.



8.1.14 Data dump

The data of the job can be saved as an ASCII data file or issued as a report. Depending on the selection, the name of the ASCII data file will be prompted during the processing of the element or a name can be set and supplemented through an automatically generated number.

The data can also be printed out as a report in a portrait or landscape format.

The data to be issued must be activated under the tab "Select". The issuing of the table of the measuring values is not activated as a standard.



8.1.15 Graphic definition

This element is reasonable only when working with the measuring and evaluation unit KB1 which is available for the equipment of the series 1 and the HAAKE RheoStress 600. The diagram of the KB1 can be defined utilizing this element.

8.2 Measuring elements

8.2.1 General

CR measurements (Controlled Rate) are possible with the Viscotesters.

In addition to this, CS (Controlled Stress) and oscillation measurements are possible with the Series 1 rotational rheometers and the HAAKE RheoStress 600 rheometers.

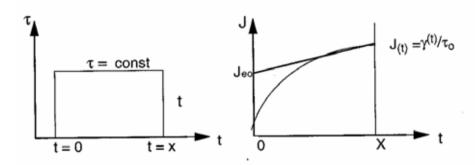
IMPORTANT: The function THERMOGAP must be activated before measuring with "Temperature Steps" or "Temperature Ramp" and cone-and-plate system! Otherwise unit parts can be damaged due to the thermal expansion of the measuring shaft!

(see capter 8.1.10)



8.2.2 CS Creep-Test

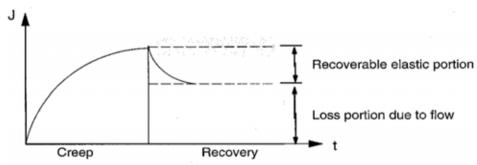
This measurement procedure provides important information regarding the viscoelastic properties of a substance. A constant shear stress is applied instantaneously. The resulting deformation is measured as a function of time. An analysis of the creep—curve enables the determination.of.for.example the zero—shear viscosity h_0 , the elastic deformation g_{e0} and the steady—state compliance $J_{(e0)}$.





8.2.3 CS Recovery-Test

Usually a recovery segment is linked to a creep segment. The shear stress is set to zero. With this measurement the recoverable elastic portion of the deformation can be determined.

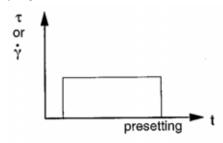




8.2.4 CS/CR-Rotation Time Curve

In this measurement procedure a constant shear stress/ shear rate and a constant temperature are defined for a fixed time

The resulting shear rate (shear stress) is measured and displayed.

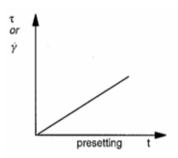


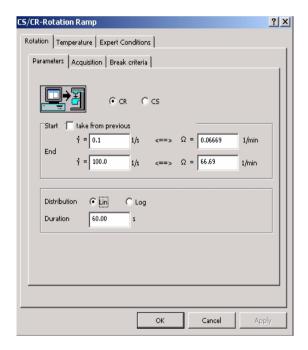


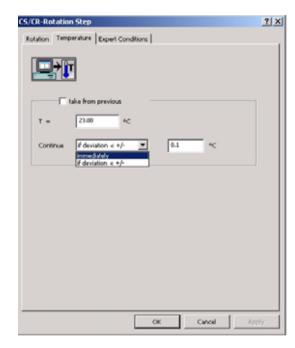
8.2.5 CS/CR-Rotation Ramp

In this measurement procedure the preset shear stress/ shear rate is continuously altered – either continuously increased or continuously decreased.

The resulting shear stress/shear rate is measured and displayed without waiting for equilibrium conditions.











8.2.6 CS/CR-Rotation Steps

The measurement of the flow curve helps to classify a substance according to one of the possible characteristics: Newton; Pseudoplastic; Dilatant; Bingham; Plastic; etc.

With the steady state flow curve in the CR-mode a certain shear rate is selected. Now, it has to be waited until the steady state conditions have stabilized; i.e. the resulting shear stress does not change any more. The result will be a data point for the viscosity.

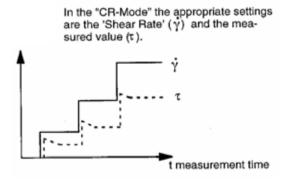
Then, the selected shear rate is increased or decreased in order to obtain again a constant shear stress value after a certain waiting time. Normally, the data points are taken over a wide shear rate range in order to obtain a steady state flow curve.

Those flow curves can also be obtained in the CS-mode. Analog to the procedure described above here the shear stress values are preset and the resulting shear rate values have to stabilize.

In the box "Data acquisition mode" the user can now choose from two different modes:

The mode "Take average of" and the mode "Integration time", RheoWin will take the average of all the (raw) data points measured during that time.

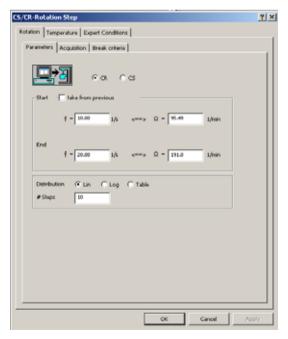
The Step mode options are independent of the Data acquisition mode options.

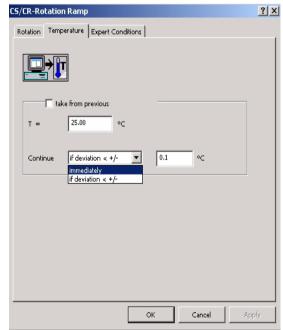


In the box Step mode the user can now choose from tow different modes:

The mode "Wait for equilibrium at each step" and "Fixed duration for each step" each step will take exactly as long as defined by the Step duration. The actual data acquisition takes place either over a given volume of raw data or over a given integration time.





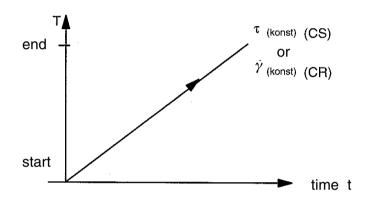




8.2.7 CS/CR-Rotation Temperature Ramp

In this measurement procedure the temperature is increased or decreased continuously and all other measurement conditions are kept constant.

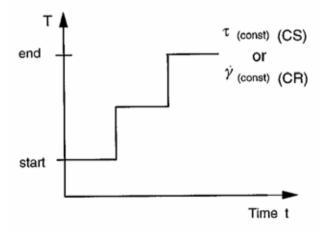
The temperature changes directly proportional to time from a starting value to an end value.





8.2.8 CS/CR-Rotation Temperature Steps

In this measurement procedure the temperature is raised step by step and all other measurement conditions are kept constant. The result is the dependency between viscosity and temperature.







8.2.9 Oscillation Time Sweep

The Oscillation Time Sweep is the ideal tool to observe how material changes over time. The initiator for this change can be multiple. It might be just aging, but it also can be started by an UV light impulse, by a chemical initiator or by quenching the material to test temperature.

You need to make the following settings:

Stress (CS mode): The stress value you pick needs to be in the linear visco-elastic range, which is determined by the Stress Sweep. It is recommended that this value correlates with a torque, that is at least 5times higher than the specified minimum torque. It also needs to create a reasonable deformation. Check the angular displacement ϕ , which should be approx. 10 to 100 times the angular resolution specified.

To be in accordance with the linear visco—elastic range requirement becomes quiet tricky, due to the fact that from juvenile to final stage the material changes can be very drastic. It is possible to split the whole test procedure in several segments with varying conditions. You will need to run several pre—tests to work out a suitable JOB procedure.

E.g., it can be anticipated that higher stress values will be necessary for an ongoing curing reaction, as the deformation becomes smaller and smaller with the material getting harder and harder. Therefore you may use the break criteria tool. If deformation falls below a certain value the measurement element will be stopped and the test proceeds with the next element, which is defined the same way as the previous but with a 10 times higher stress value. The same thing can be accomplished with the CD mode, but the usage of the break criteria is much faster!

Note: It might be not worth putting to much effort into the subject of linear visco-elasticity, as the comparison of results measured under the same conditions probably contains the necessary information.

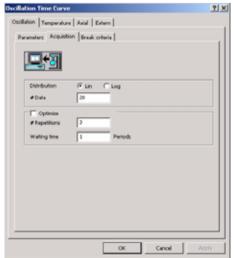
Deformation (CD mode): The instrument will detect the necessary stress value to accomplish the requested deformation automatically.

You may need to check that a reasonable stress value occurred. Do so by looking for the torque M, which needs to be at least 5 times the minimum torque specified.

Note, this mode is not recommended for very fast changing materials, as the control loop can not follow up adjusting the test parameter rapidly enough!

Frequency: Values of 1 to 10Hz are common. The frequency should be high enough, so that the measurement of a single data point is fast compared to the change of the material.

Duration: As long as it takes. Maybe 10 or 15 minutes are a good starting value. If you notice that the material functions



G' and G" etc. haven't reached a plateau you probably want to lengthen the time.

Acquisition: Repetition should be set to 1 and waiting time to 0 periods in order to obtain data at maximum speed.

The time curve hosts a unique feature named trigger found under the

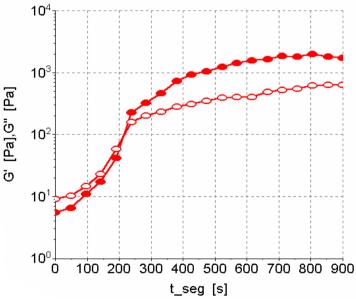


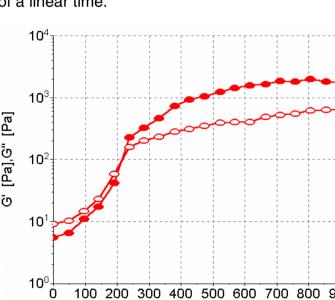
Activate the trigger function by check marking it. The trigger function will switch on/off a relais for the set duration time, until this specific measurement element is finished or until the entire Job is finished (indefinite).

This is e.g. used to trigger an UV light impulse, but can be used to trigger any external accessory, which can be started by an electric impulse.

What do we get?

Commonly G' and G" are presented logarithmically as function of a linear time.





Several points can be evaluated.

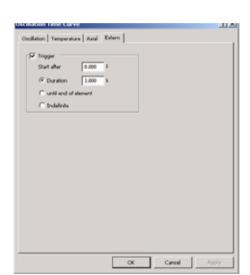
The time when aging, gelation, curing.... sets in.

That will be the point where the material functions start raising/falling.

- The crossover time, where G' and G" are equal. It can be questioned if that point has any physical meaning - regardless, this point is easy to spot and indicates a significant change in the material properties, whatsoever.

The time when the final stage has been reached.

Some material will turn out not to come to a finite condition!





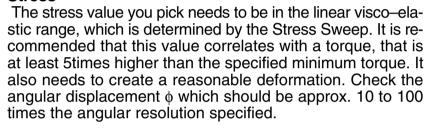
8.2.10 Oscillation Frequency Sweep

The Frequency Sweep is used to characterize test materials. The sample is not supposed to change its properties throughout the test time.

The principles of oscillation testing are described in the chapter "Introduction to Oscillation Testing".

You may use the test parameter given in the example as a first attempt:

Stress



It might be necessary to split the frequency sweep into a few steps, which will allow you to set varying numbers of repetitions without creating an unacceptable test time and you may set different stress values for the individual frequencies to be in accordance with the linear visco—elastic range presumption.

Start Frequency

In most cases 0.1Hz should be a suitable start frequency. Lower values require a longer measurement time. Each data point has an estimated test time of the reciprocal of the actual frequency, multiplied by the number of cycles running through. Each data point requires at least 2 cycles – one prerun and one test run repetition.

If applicable you need to prevent your material from drying out, as this will change the materials properties.

End Frequency

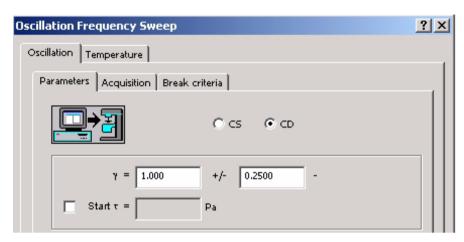
14Hz (approx. 100rad/s) is a suitable value. Higher frequencies may run into problems in terms of correction for inertia and collecting enough data points for a decent sine wave. The problem gets worse the lower the viscosity is. Having a smooth curve turning bizarre probably indicates the effect. To improve the data quality you may select several repetitions.

Data points

The software will set a number of logarithmically equidistant frequencies per decade according to your selection. 6 points per decade is the most common choice.

When using the CD mode you will be asked for a deformation instead of a stress value.





Deformation

The instrument will detect the necessary stress value to accomplish the requested deformation automatically.

Note: This will take some time and can be very time consuming at very low frequencies, because the machine has to run through a full cycle before it can detect if the selected strain was reached.

You may need to check that a reasonable stress value occurred. Do so by looking for the torque M, which needs to be at least 5 times the minimum torque specified.

Click the Acquisition tab to set a few more parameter.



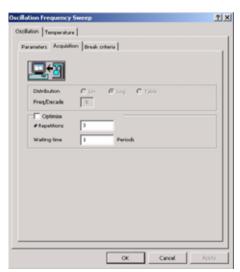
For data point improvement you may run several repetitions, from which an average is build. Above frequencies of 1Hz you may use 5 to 10 repetitions. For time saving reasons you should use less repetitions on lower frequencies.

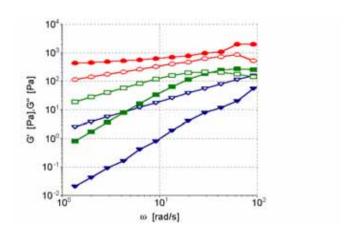
Waiting time

This sets the numbers of pre-cycles, before data are collected.

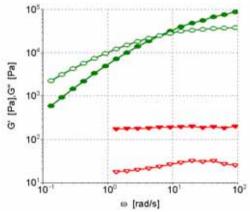
What do we get?

The frequency sweep tells us about the structural conditions of the sample. It is possible to distinguish between a particle solution, an entangled solution (paste) or a three–dimensional network (gel) simply by the shape of G' and G" curves.

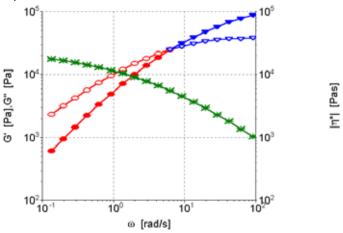




A gel can even be the "softer" material compared to the paste.



The material function curves can also be used to interpret unusual flow behavior. PDMS is an often cited material which flows even under gravity but behaves elastic under a sudden impact. This behavior can not be described by viscosity measurements, but it can be predicted from an Oscillation frequency sweep.



At low frequencies the viscous behavior reflected by G" is dominant whereas at high frequencies the elastic behavior reflected by G' is outweighing.

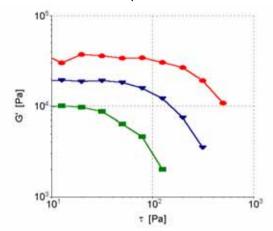


8.2.11 Oscillation Stress Sweep

The Oscillation Stress Sweep is first of all used to determine a material's linear visco-elastic range, which is to say that the measurement parameters are set in this manner that stress and strain amplitude have a linear relationship which can be described by the following equation:

$$\tau_0 = G^* \cdot \gamma_0$$

A more practical way to identify the linear visco—elastic range is to look for the region where the material functions as e.g. G' and G" are independent of the stress / strain value.

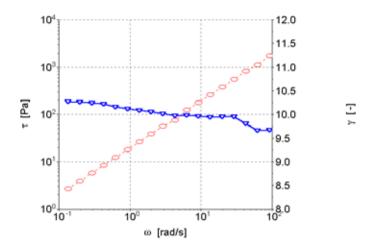


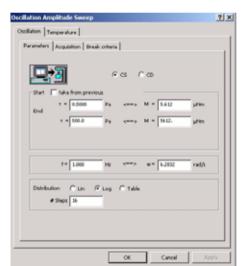
The linear visco-elastic range is frequency dependent! Therefore you will need to perform the stress sweep at least for the highest and lowest frequency of the frequency range you are interested in.

Some materials will show overlapping linear visco—elastic ranges within the interesting frequency range. That is to say you may find a stress value which is good for all frequencies. You may pick this stress value to run a frequency sweep.

A lot of materials won't allow the use of a single stress value. Instead you may notice that the critical point of the stress sweep is reached at the same maximum deformation.

For that occasion you can use the CD mode, which performs any oscillation experiment the way that a set deformation value is achieved.





Dependent on the frequencies this deformation requires different stresses, which are automatically adjusted.

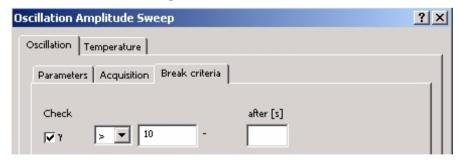
Instead of using the CD mode you also can split the whole frequency range in sections, each section using a stress value tested to be good for that particular range.

When examining a material for the first time it is a good start to define a stress sweep as shown in the screen print as follows.

Notice that the torque range has been set – not the stress! It covers three decades, starting with a value about five to ten times higher than the minimum specified torque.

As you don't know the material in the early phase of characterization a too high stress value may result in a shear like motion. To avoid this you can set Break criteria.

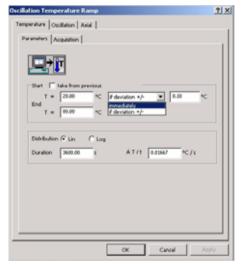
Which will limit e.g. the achieved deformation.



Occasionally the stress sweep is also used to determine a yield stress. It is simply the limiting stress from the linear visco—elastic range. If you recall the frequency dependency this looks quiet critical. You may do so, but you need to give notice about the test conditions at which this yield point was measured.

However, when comparing two materials, the unequal limits for the linear visco-elastic region may allow a differentiation between these materials, which couldn't be found in a shear experiment.





8.2.12 Oscillation Temperature Sweep

The Oscillation Temperature Sweep is very similar to the Oscillation Time Sweep, with the only difference that the temperature is changing in accordance to the settings made.

The temperature may change continuously in a ramp:

Start

In case you have other measurement elements defined ahead of the Temperature Sweep you can make use of the last set temperature from these elements by check marking the "take from previous" box.

When doing a temperature ramp you also have the choice to have the software to check for the Start temperature being achieved, by setting if deviation...

For a temperature step program this is done for each tempe-

rature under the Acquisition tab.

End

As you need it or your thermal controller is suitable for.

Duration

You can either fill in the total ramping time or the heating rate, respectively. Please consider the capabilities of your heating device. E.g. circulators probably allow a maximum heating rate of approx. 1°/min.

Also please keep in mind that the thermal controller may heats up very fast, but that doesn't mean that the sample has caught up with temperature. In most cases you probably need a time element, to observe ongoing material change after end temperature has been reached by the device.

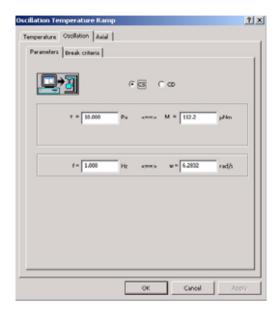
Acquisition

Doing a temperature ramp it is recommended to have only one repetition and zero periods of waiting time, in order to gain data points as fast as possible, because temperature and supposedly material properties are changing fast.

So far we basically discussed the parameter for changing the temperature. As in the time sweep you need to make settings for the oscillatory operation of the rheometer, defined under

the Oscillation tab.





The Stress (CS mode): The stress value you pick needs to be in the linear visco-elastic range, which is determined by the Stress Sweep. It is recommended that this value correlates with a torque, that is at least 5times higher than the specified minimum torque. It also needs to create a reasonable deformation. Check the angular displacement $\varphi,$ which should be approx. 10 to 100 times the angular resolution specified.

To be in accordance with the linear visco—elastic range requirement becomes quiet tricky, due to the fact that from juvenile to final stage the material changes can be very drastic. It is possible to split the whole test procedure in several segments with varying conditions. You will need to run several pre—tests to work out a suitable JOB procedure.

E.g., it can be anticipated that higher stress values will be necessary for an ongoing curing reaction, as the deformation becomes smaller and smaller with the material getting harder and harder. Therefore you may use the break criteria tool. If deformation falls below a certain value the measurement element will be stopped and the test proceeds with the next element, which is defined the same way as the previous but with a 10 times higher stress value. The same thing can be accomplished with the CD mode, but the usage of the break criteria is much faster!

Note: It might be not worth putting to much effort into the subject of linear visco-elasticity, as the comparison of results measured under the same conditions probably contains the necessary information.

Deformation (CD mode): The instrument will detect the necessary stress value to accomplish the requested deformation automatically.

You may need to check that a reasonable stress value occurred. Do so by looking for the torque M, which needs to be at

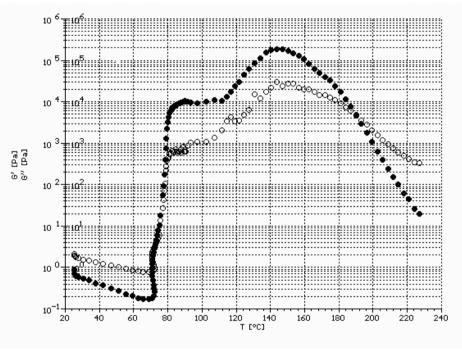
least 5 times the minimum torque specified.

Note, this mode is not recommended for very fast changing materials, as the control loop can not follow up adjusting the test parameter rapidly enough!

Frequency: Values of 1 to 10Hz are common. The frequency should be high enough, so that the measurement of a single data point is fast compared to the change of the material.

What do we get?

Commonly G' and G" are presented logarithmically as function of a linear temperature.



The given example is the fusion and degradation curve of a PVC plastisol. At the beginning the material is paste like, it has higher viscous than elastic properties, indicated by a higher loss modulus G" versus storage modulus G' value. With increasing temperature both material functions decrease. It might be even seen that the material becomes more viscous. This can be expected, as the higher temperature increases the mobility of molecules. At a certain temperature, the onset of fusion, G' and G" increase rapidly. It is noticeable that the material now has become dominantly elastic. As temperature increases the material undergoes its final curing till a maximum is reached, the onset of degradation. Finally the level of degradation has turned the material into a viscous paste again.

Note: The very same data can be presented as function of time showing the temperature as one more curve within the diagram.

Points to evaluate will be onsets, plateaus, crossovers and the related temperatures or times, respectively.







8.2.13 Oscillation Temperature Sweep

The Oscillation Temperature Sweep is very similar to the Oscillation Time Sweep, with the only difference that the temperature is changing in accordance to the settings made.

The temperature may change continuously in a ramp:

Start

In case you have other measurement elements defined ahead of the Temperature Sweep you can make use of the last set temperature from these elements by check marking the "take from previous" box.

When doing a temperature ramp you also have the choice to have the software to check for the Start temperature being achieved, by setting if deviation...

For a temperature step program this is done for each tempe-

rature under the Acquisition tab.

End

As you need it or your thermal controller is suitable for.

Steps

When doing a temperature step curve it is necessary to set the step width, which is simply done by the number of steps. Remember for a range of 60°, getting a data point every five degree you need 60/5+1=13 data points!

Acquisition

Doing a temperature ramp it is recommended to have only one repetition and zero periods of waiting time, in order to gain data points as fast as possible, because temperature and supposedly material properties are changing fast.

Running a temperature step procedure you may improve the data point accuracy by averaging several repetitions.

This becomes possible, as equilibrium shall be reached anyway.

The measurement at each temperature step is done when the temperature has been stabilized for the set maximum waiting time or the temperature falls within the given tolerance of deviation °C after a certain minimum time.

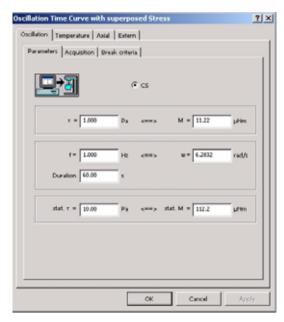
So far we basically discussed the parameter for changing the temperature. As in the time sweep you need to make settings for the oscillatory operation of the rheometer, defined under

the Oscillation tab

With respect to the rheological values, the issues discussed in the previous section apply.

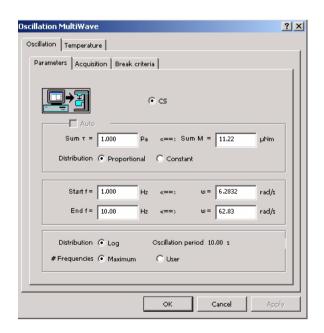
8.2.14 Oscillation: Time Curve with superposed Stress





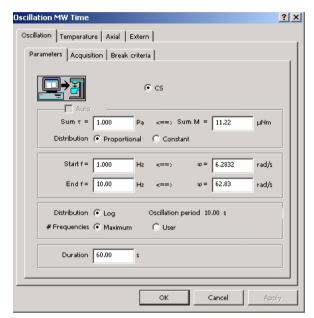
8.2.15 Oscillation: Multiwave





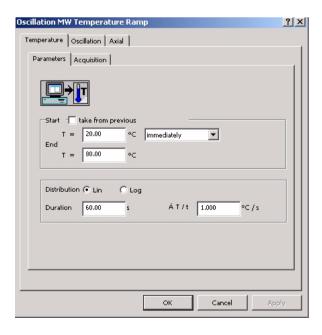


8.2.16 Oscillation: Multiwave-Time Curve



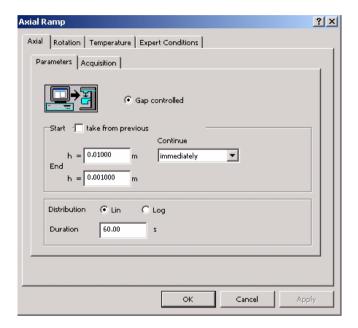
^{0sc} ╱<mark>╱</mark>

8.2.17 Oszillation: Multiwave-Temperature Ramp



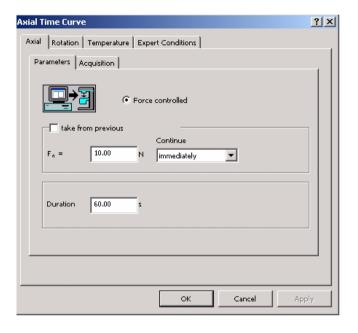


8.2.18 Axial-Ramp





8.2.19 Axial-Time Curve



8.3 Analysis elements



8.3.1 Regression

A regression analysis can be carried out with selected data. An extrapolation of the regression data beyond the calculated range is also possible.



8.3.2 Interpolation

An interpolation is carried out with selected data. Various methods are available for this purpose.



8.3.3 Area

The area underneath the selected graph line is calculated.



8.3.4 Thixotropy

The thixotropy test, also known as the hysteresis curve, is a procedure for determining time—dependent flow behaviour. It is very similar to a procedure for recording a flow curve, in its graphic presentation as well: shearing stress as a function of shear speed.



8.3.5 Curve discussion

This element of data analysis serves the purpose of calculating the smallest and/or greatest value (minima and maxima) in one reading. Several methods (minima, maxima, and others) can be selected simultaneously for one calculation.



8.3.6 Creep analysis

The creep analysis is an automatic analysis containing the three following steps: determining the zero viscosity, the elastic deformation proportion, and the elastic yielding directly from creep and recovery curves. Calculation of the first normal stress co–efficients, the characteristic relaxation time, and the plateau module.



8.3.7 Crossover

Calculates the point of intersection between the storage module G' and loss module G' from one oscillatory reading.



8.3.8 Flow limit

The flow limit is defined as the smallest necessary force needed to set the flow in motion. The existence of a flow limit is still a matter for discussion because rheology theoreticians maintain that every material flows; the only factor that makes any difference is the period of observation.

Nevertheless, it still makes sense to talk about the flow limit because in practice, after all, it obviously does exist. Even theoreticians cannot deny that there are plenty of substances that demonstrate a flow limit within a reasonable period of observation. For instance, it is possible still to see the impression of a finger in many crèmes even if one only opens the tin again on the next day or even several days later.



8.3.9 SHRP

This element serves the purpose of analysing the readings using SHRP (Strategic Highway Research Programme)



8.3.10 Reference

Compares the latest reading with preceding readings.



8.3.11 Time Temperature Superposition

Time Temperature Superposition calculation.

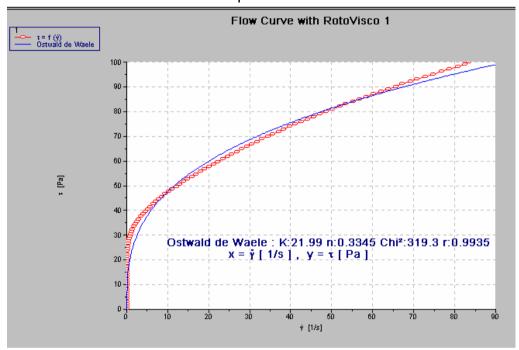
An extra installation key is needed to be able to install and use this feature.

8.3.13 Evaluation

8.3.13.1. Regressions

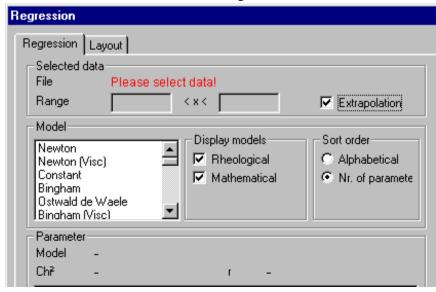
For the appreciation of the quality of the curve fit stands to her the correlation coefficient R to the disposalis.

If the regression delivers a correlation of R=1 or a very small value for chi² the fit was perfect.



Other new features are the possibility to extrapolate regressed data over the calculated range. This has to be treated carefully, because data are only calculated.

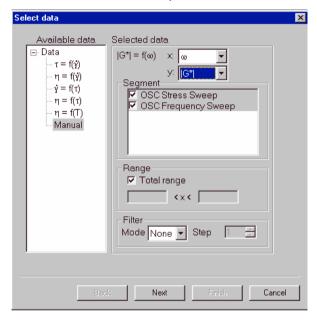
Additionally a warning (in red) is shown if no data are selected which is a must to do a regression.



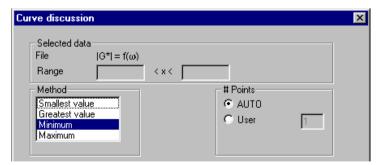
8.3.13.2. Curve Discussion

The RheoWin software has a data evaluation element in the JOB MANAGER section as well as in the DATA MANAGER (Analysis) to calculate the smallest and/or the biggest values (mimima and maxima) in a measurement.

To activate this new element, just drag it on the JOB list and double click it to edit the parameters.



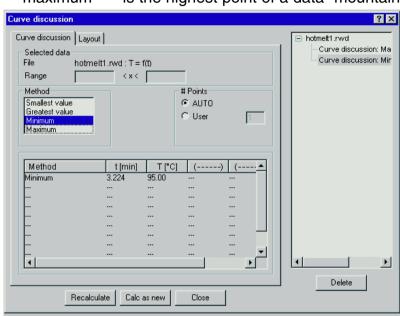
Select the value to be evaluated from the left list box (Available Data) or use manual and enter the desired quantities from the pull down menu. Choose the x-range where the data set should be evaluated (or leave it as it is) and press NEXT to continue.



Continue with NEXT to select the layout (color, lines) and complete it with FINISH

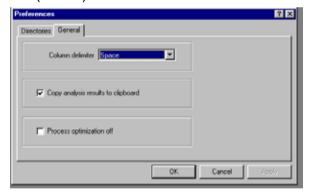
The same procedure is available in the DATA MANAGER using /ANALYSIS/CURVE DISCUSSION/. As usual a set of data (curve) and then the procedure has to be selected:

- smallest value looks for the smallest numerical value
- biggest value looks for the biggest numerical value
- minimum is the deepest point in a data "valley"



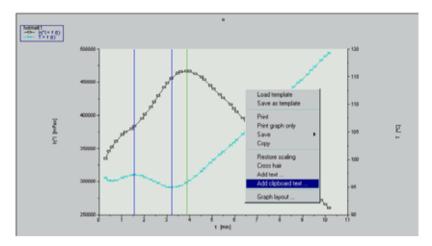
- maximum is the highest point of a data "mountain"

The result of such a curve discussion is graphically shown with colored lines and the result is transferred to the clipboard (labels).



Get DATA from the clipboard

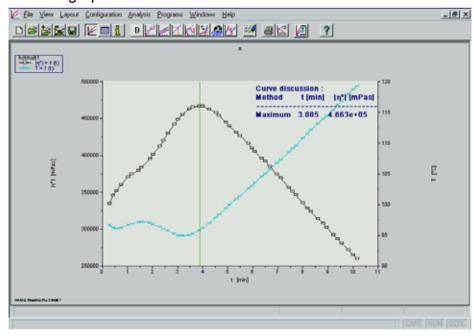
Activate the switch COPY ANALYSIS RESULTS TO CLIP-BOARD and then insert the results in a graph using the right mouse button:

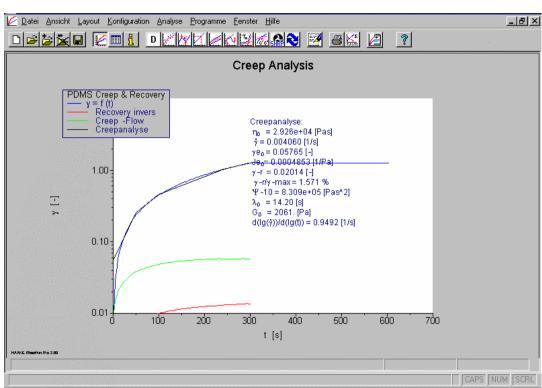


The test result is then available as label which can be edited and placed where needed.



The graph shows now the test result and the calculated data in a graph.





8.3.13.3. Creep/Recovery analysis

The Creep analysis is an automatic evaluation whose mathematics are explained in the following chapter:

1. Determination of relevant quantities based on creep and recovery curve:

Zero shear viscosity (or Newtonian viscosity): η_o Elastic deformation: γ_{eo}

Equilibrium shear compliance: $J_{eo} = \gamma_{eo}/\tau_o$

2. Calculation of derived quantities:

First normal stress coefficient: $\Psi_{10} = 2\eta_0^2 J_{eo}$ Characteristic relaxations time: $\lambda_0 = \eta_0 J_{eo}$

Plateau modulus (for polymers):

$$G_N = \frac{1}{J_{eo}}$$

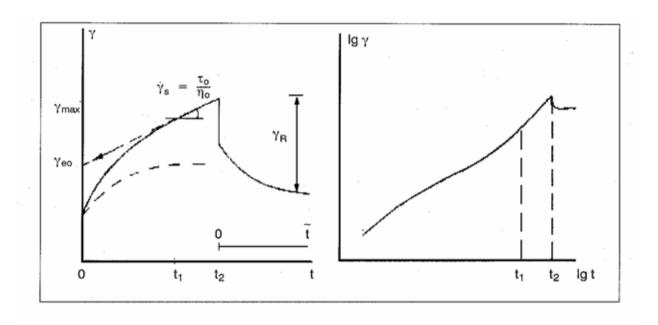
3. Special plots:

Transient elastic behavior: 'Creep minus Flow'

$$\gamma_e(t) \ = \ \left(\gamma(t) \ - \ \frac{\tau_o}{\eta_o}\right) \ bzw. \ J_e(t) \ = \ \left(J(t) \ - \ \frac{l}{\eta_o}\right)$$

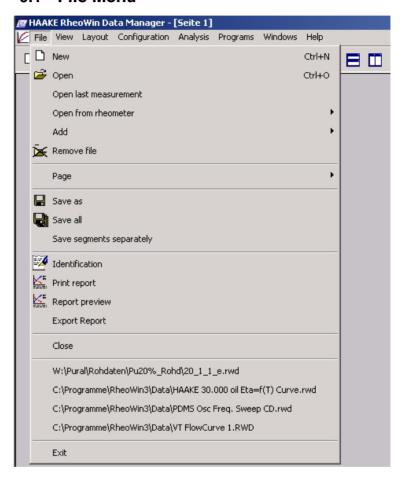
Modified recoverable strain curve:

$$\gamma_y(\tilde{t}) = (\gamma_{max} - \gamma(t))_{t = \tilde{t} = t_o} \text{ mit } t \leq t_o$$



9. Data Manager

9.1 File Menu



New

An empty standard page layout is opened with the "New" function. Files can now be added here and the layout adapted.

Open

With the "Open" function an existing data file (*.rwd) or a RheoWinPage file (*.rwp) can be loaded.

Open last measurement

The "Open last mesurement" function creates a new standard page layout and adds the data to the most recently completed job.

Open from rheometer

Add

The "Add" function adds a data file (*.rwd) to the currently active window or else adds the last reading.

Remove file

The "Remove file" function enables data from a reading to the removed from the currently active window. A selection dialogue appears in which the files to be deleted are selected and the action confirmed with "OK". Holding down the shift or "Ctrl" key during the selection enables several data to be highlighted at once.

Page

The "Open page" function opens and existing RheoWinPage file (*.rwp).

The "Save page" function saves the currently active window as a RheoWinPage file (*.rwp) with all its layout settings under the existing file name.

The "Save page as" function enables an edited page to be stored under a new file name. A selection dialogue appears with the file names that already exist.

Save as

The "Save as" function enables a page to be stored within a data file. The file first has to be selected from the following selection dialogue.

Mit der Funktion "Speichern als" kann eine Data-Datei innerhalb einer Seite unter einem neuen Dateinamen gespeichert werden. Es erscheint ein Auswahldialog mit den aktuell vorhandenen Dateien.

Save all

Save segments separately

The "Save segments separately" function enables segments to be stored within a data file. The file first has to be selected from the following selection dialogue the segments of which are to be saved, and then the segments that are to be saved can be selected.

Identification

Print report

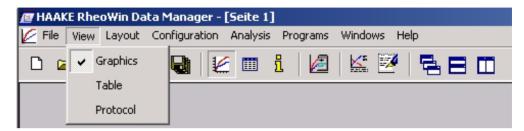
Rrint review

Export Report

Clost

Exit

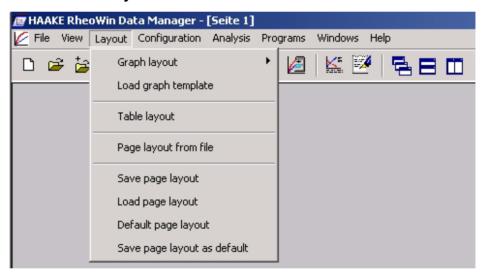
9.2 View Menu



This determines which information in the file is opened.

A detailed description of all sub-points in the "View" menu can be found in Section 10.

9.3 Layout Menu

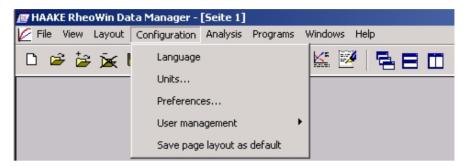


A detailed description of all sub-points in the "Layout" menu can be found in Section 10.

Page layout from file

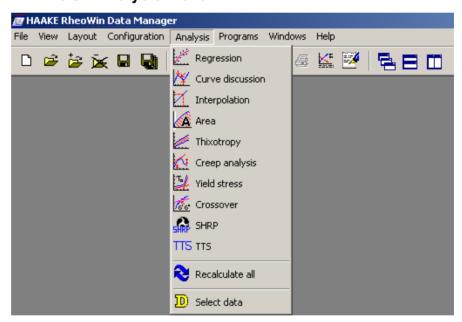
Here the layout defined in the job is defined.

9.4 Configuration Menu



A detailed description of all sub-points in the "Configuration" menu can be found in chapter 7.2.

9.5 Analysis Menu

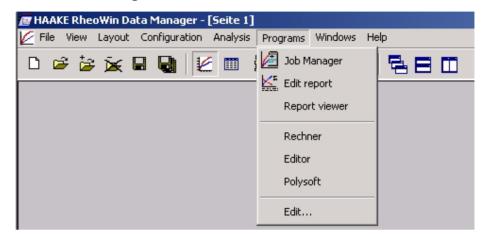


9.5.1 Analysis elements

The analyses elements are accessible via the "Analysis" main menu and also directly via the "Analysis elements" icon bar.

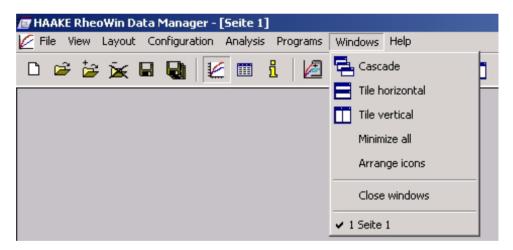
A detailed description of all sub-points in the "Analysis" menu can be found in chapter 8.3.

9.6 Programs Menu



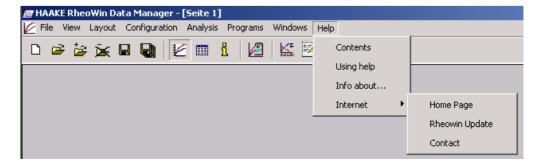
A detailed description of all sub-points in the "Programs" menu can be found in chapter 7.1.4.

9.7 Windows Menu



A detailed description of all sub-points in the "Windows" menu can be found in chapter 7.1.6.

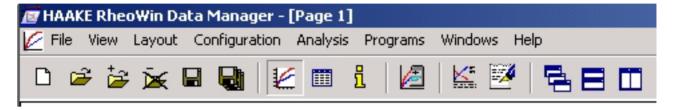
9.8 Help Menu



A detailed description of all sub-points in the "Help" menu can be found in chapter 7.1.7.

9.9 Tool Bar

9.9.1 General



The most important menu points of the main menu are directly accessible via the general Tool bar:

A detailed description of all sub-points in the "Tool Bar" menu can be found in chapter 7.1.8.

Display and output

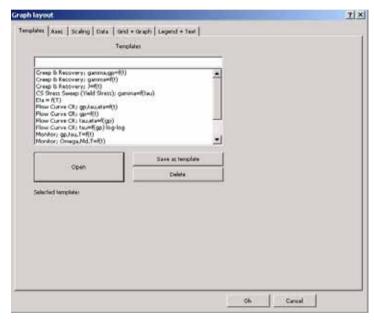
10. Display and output

10.1 Pages

10.2 Graph dialogue

The functions described below can be called up in the Data Manager via the "Layout" menu or by double-clicking in the "Graphic" view.





Predefined layouts for the "Graphics" output are loaded. The templates are supplied with the programme and can be found in the RheoWin folder "...\RheoWin3\Templates" and have the file extension *.qds

Only those templates are shown that are kept in this templates folder, Which can be altered under the "Settings" configuration.

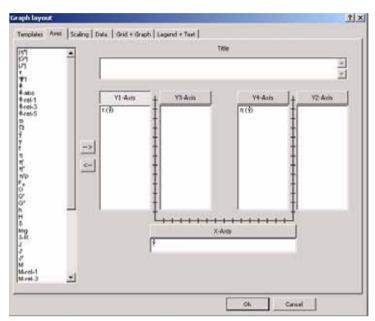
Save as template

When changes have been made the current layout is saved as a future template.

Deleted

The marked template is deleted after a safety check message has been accepted.

Display and output



10.2.2 Axes

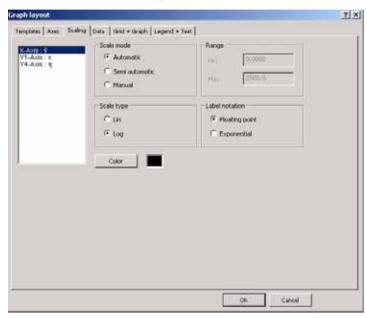
The following is the procedure for selecting the dimensions on the individual axes.

In order to add a dimension to an axis, the relevant axis is selected with a click of the left mouse button. The required dimension is then selected from the left—hand list and marked with one more left—click. The dimension is then transferred to the list of axes by clicking on the "Add" button (with the arrow pointing to the right).

In order to remove a dimension from an axis, one left-click selects the relevant dimension and a second on the "Remove" button (with the arrow pointing to the left) removes the dimension from the list of axes.

After a click on "OK" the graphic element is shown with the new or altered dimensions.

A diagram title can be entered in the "Title" field. The title can be further formatted with a right—click on the title in the graphics display.



10.2.3 Scaling

The scaling of each individual axis can be set independently of the other axes.

Three kinds of scaling are available:

Automatic

The limits of the axes are prescribed by the readings.

Semi automatic

Manuel

The upper and lower limits can be stated in the range–fields "Min" and "Max".

The sub-divisions of the axes can be either linear or logarithmic.

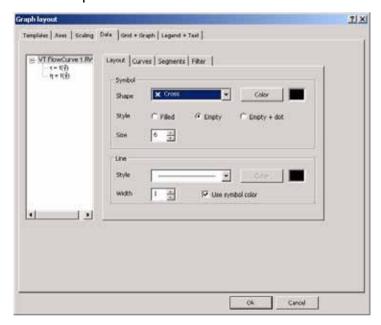
The numbers along the axes can be issued as a flowing decimal or in an exponential format.

The individual axis labels can also have various colours allocated to them. This makes the display easier to understand if several axes are being used.

10.2.4 Data

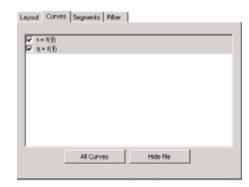
Here the presentation of the graph lines of readings is defined.

Layout: symbols and lines are allocated to the lines in the individual files. The symbols can be varied in colour, size, and filling, and the lines can be in the same colour as the symbols or can be defined independently of them. The thickness and pattern of the lines can also be varied.



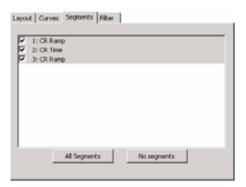
Curves

The presentation of the graph lines of the individual files can be switched on or off. The "All graph lines" button results in the presentation of all the lines, the "Hide lines" button results in the presentation of none of the lines of this file.



Segments

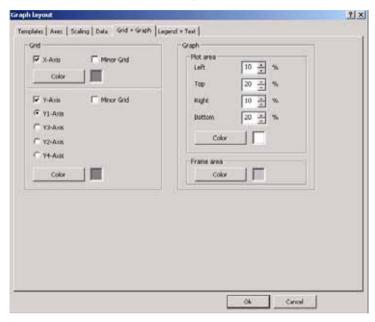
If a number of segments are defined in the Job Editor it will be possible here also to switch the presentation of the individual segments on or off.



Filter

If not all data points of a file are to be presented, a filter can be used to hide data points.





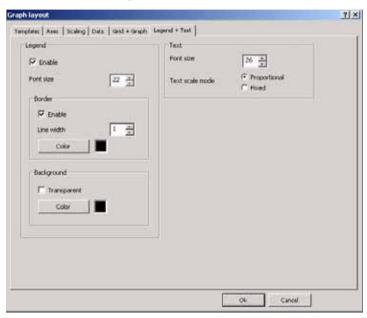
10.2.5 **Gird + Graph**

Gird

The main and auxiliary grilles can each be defined for the x-axis and the y-axis. Different colours can be allocated to each axis. It is possible to define which y-axis is to be used for generating the grid.

Graph

The size of the margin around the diagram is defined under "Graph line area". The sum of the values for Left and Right and for Top and Bottom must not exceed 100 %. The background colours for the margin and the diagram area can likewise be selected.



10.2.6 Legend + Text

Legend

The font size, frame colour and thickness, and the background colour (including transparency) can be defined for the activated legend.

Text

The font size and scale of the axis labelling is defined here.

10.2.6.1. Context-dependent Popup Menu

A right—click on the diagram surface produces a **context menu** containing the following options:



Load template

Predefined layouts can be loaded in the same way as under "Graphic layout – templates". In addition to this the folder can be selected in which the template file (file extension *.gds) can be found.

Save as template

After any changes have been made the current layout is stored as a future template.

Print

Print garph only

Save

The current graph can be saved under one of the following formats: "Meta File" (*.wmf), "Enhanced Meta File" (*.emf), or "Device Independent Bitmap" (*.dib).

Copy

Copies the graph onto the Notepad, from where it can be inserted into other programmes.

Restore scaling

Within the drawing area the presentation can be enlarged by generating an area with the left mouse button onto this area. The original scale can be presented by selecting the menu point "Restore scaling".

Cross hair

When the mouse pointer is moved across the drawing area, an aiming cross appears at the tip of the point. The co-ordinates of the cross appear at bottom right in the status bar.

Add text....

A text box is generated that can be positioned anywhere required and then edited or deleted with a right-click.

Add clipboard text...

A text box is generated that contains text from the Windows Notepad. It can be edited or deleted with a right—click.

Graph layout...

Opens the dialogue window for the graphic layout.

Axes

A right-click on the labelling of an axis enables this axis to be switched from a linear to a logarithmic display and back.

Text



In the case of text boxes a right-click produces a menu that makes it possible to edit or delete the box.

Positioning

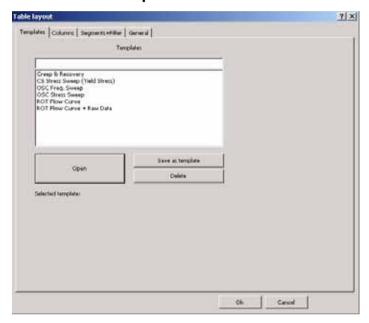
Text boxes and the legend can be positioned anywhere by Drag&Drop with the left mouse button.

10.3 Table

Table layout menu

The functions described below can be called up in the Data Manager via the "Layout" menu or with a right-click on the "Table" view via the context menu.

10.3.1 Template



Predefined layouts can be loaded for the "Table" issue. The templates are supplied with the programme and can be found in the RheoWin folder "...\RheoWin3\Templates" and have the file extension *.tds

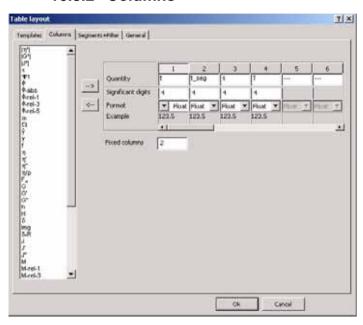
Only those templates are shown that are kept in this templates folder, Which can be altered under the "Settings" configuration.

Save as tempplate

When changes have been made the current layout is saved as a future template.

Deleted

The marked template is deleted after a safety check message has been accepted.



10.3.2 Columns

The following is the procedure for selecting the dimensions on the individual axes.

Quantity

In order to add a dimension to an axis, the relevant axis is selected with a click of the left mouse button. The required dimension is then selected from the left—hand list and marked with one more left—click. The dimension is then transferred to the list of axes by clicking on the "Add" button (with the arrow pointing to the right).

In order to remove a dimension or a column from an axis, one left–click selects the relevant dimension and a second on the "Remove" button (with the arrow pointing to the left) removes the dimension from the list of axes. The columns on the right move one place to the left.

Significant figures

The number of significant figures can be defined as required.

Die Anzahl der signifikanten Stellen kann festgelegt werden.

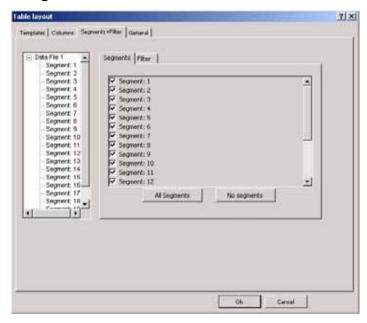
Formats

The formats available are: floating decimal, exponential, and integers (Int.).

Fixed columns

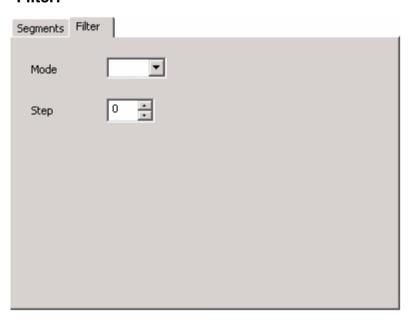
10.3.3 Segments + Filter

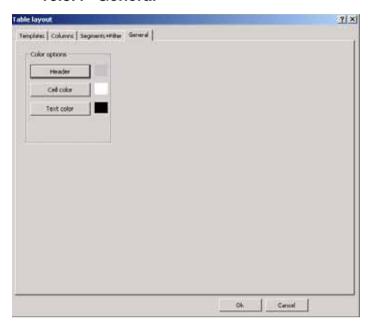
Segments:



If several different segments are defined in the Job Editor the presentation of the individual readings of the segments in the column can be switched on or off.

Filter:



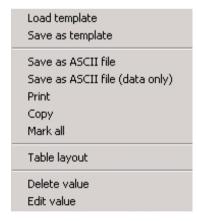


10.3.4 General

The colours for the background of the headers and the tables and of the lettering can be defined here.

10.3.4.1. Context-dependent Popup Menu

A right—click on the diagram surface produces a **context menu** containing the following options:



Load template

As under "Table layout – templates", predefined layouts can be loaded, but her the folder can be selected in which the template file can be found (file extension *.gds).

Save as template

When changes have been made the current layout is saved as a future template.

Save as ASC II file

Saves the table including headers and all columns in a text file (*.txt). The file name and the folder can be stated. The divisor between neighbouring columns is shown as a semicolon.

Save as ASC II file (data only)

Only saves the readings columns, without the headers, in a text file (*.txt). The "Numbers / Segment numbers" column is not issued. The file name and the folder can be stated. The divisor between neighbouring columns is shown as a semicolon.

Print

Prints the table out in the stated format.

Copy

Copies the highlighted fields onto the Notepad, from where they can be inserted into other programmes. The divisor between neighbouring columns is shown as a TAB mark.

Mark all

Highlights the complete table including any empty columns.

Delelte value

Deletes the currently highlighted values without any safety check question.

Edit value

Makes it possible to alter the currently highlighted values. Only one value can be highlighted for this function.

Info Q-Channels

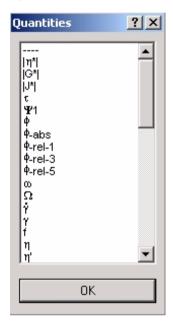
Shows information from the Q-Channel.

Table layout

For this purpose the dialogue window for the "Table" layout is opened.

10.3.5 Columns

One right—click on the header of a column makes it possible to select a dimension directly that is to be shown in this column.



10.4 Protocol

The protocol that is to be displayed via the "View" menu contains all the information on a reading including data that have been entered via the "Identification" element or the "File – Identification" menu point, data on the measuring device including its serial number and driver version, and data on the measuring system, as well as a brief summary of the element definitions.

10.5 Report

Windows - vertical

Rheometrie / Rheologie

11. Rheometrie / Rheologie

Rheometrical measuring modes:

Categorized into preset values:

CD: Controlled Deformation

Measuring mode for the determination of relaxation modulus.

(HAAKE RotoVisco / RheoStress)

CR: Controlled Rate

Measuring mode, e.g. for the recording of flow curves and the analysis of thixotropy; here the shear stress reaction of the substance on a preset shear rate ramp is evaluated.

(HAAKE ViscoTester / RotoVisco / RheoStress)

CS: Controlled Stress

Measuring mode e.g. for the examination of a sample's structure or for the recording of flow curves in the very low shear rate range; here the deformation reaction of the substance on a preset shear stress ramp is evaluated.

(HAAKE RheoStress)

Categorized into signal forms:

Steady Rotation:

Creep/Recovery

CS measuring mode to determine the viscous and elastic properties of a material, e.g. for the determination of the zero-viscosity or as a criterion of shelf life.

(HAAKE RheoStress)

Stress Growth/Decay

CR measuring mode to determine the time behavior and steady state flow curves.

(HAAKE ViscoTester / RotoVisco / RheoStress)

Steady Rotation with ramps:

Measuring mode where the stress changes over the time e.g. to determine a yield point or a dynamic flow curve (thixotropy loop).

(HAAKE ViscoTester / RotoVisco / RheoStress)

Rheometrie / Rheologie

Oscillating movement:

OSC: Oscillation

Measuring mode for the non-destructive determination of elastic and viscous material properties.

Here e.g. the influence of the frequency by forced oscillating stress on the storage and loss modules (G' and G") can be investigated.

The measuring data gained in the linear visco-elastic range allow conclusions on other physical quantities (e.g. molecular quantities for polymers) (HAAKE RheoStress)

Flow properties regarding viscosity behavior:

Newtonian: Property of substances where the viscosity will not change under shear

rate and shear stress.

(HAAKE Falling Ball Viscometer, System Höppler)

Pseudoplastic: Property of substances where the viscosity will decrease under shear

rate and shear stress.

(Most common material behavior)

Dilatant: Property of substances where the viscosity will increase under shear rate

and shear stress.

Thixotropic: Non-Newtonian substances where the viscosity decreases under shear

(structure break-down). The substances will eventually regain their vis-

cosity after the shearing has stopped.

Rheopectic: Non-Newtonian substances where the viscosity increases under shear

(structure build-up). The substances will eventually regain their viscosity

after the shearing has stopped.

(Rare phenomenon)

Plastic: Property of non-Newtonian substances which only start flowing after being

subject to a certain force (shear stress), i.e. after a certain yield point. The yield point strongly depends on external parameters like temperature and change rate of the acting force. Therefore, a "practical" yield point is determined taking in account the environmental conditions specific for the

application.

(Measuring modes: CD, CS)

(HAAKE ViscoTester 550 / RotoVisco1 / RheoStress)

Rheometrie / Rheologie

Typical quantities of rheometry and rheology:

Instrument quantities:

torque M_d

Ω - angular velocity rotation angle

– angular velocity ($2 \cdot \pi \cdot f$) ω

 F_N - normal force

R, h, ... - dimensions of sensor

etc.

Measuring parameter:

Т - temperature

- pressure р t - time

- etc.

Rheometrical quantities:

τ shear stress

shear rate deformation

 normal stress differences N_1, N_2

etc.

Rheological quantities:

Material functions:

 η , η^* , η^- – viscosities

 $\Psi_1,\,\Psi_2$ – normal stress functions

G - shearing modulus

G*, G' G" – dynamic shear moduli

J*, J', J" - compliance

dynamic compliances

etc.

Material parameters:

- zero viscosity η_0

- yield point $\tau_{\sf V}$

- 1st normal stress coefficient Ψ_{10}

etc.

Tips & Tricks

12. Tips & Tricks

Q-channel

In the Data Manager inside a table press the right mouse button to open a window for the value Q.

The Q-channel gives information as to what extend the limits of the instrument are reached:

Value		Description		
1	Bad G'	G' is smaller than 20% of the inertia correction		
2	Min Angle	The amplitude of the angle is too small		
4	Max Angle	The amplitude of the angle is too large		
8	Max Omega	Max. speed exceeded		
16	Compliance	G-modul of the is larger than 10% of G* of the sample		

Other problems

If you encounter problems with the software we need the following information about your computer equipment in order to be able to help you:

- Thermo Haake Software version, e.g. "1.00".
- Computer
- Printer
- nterface

This information can also be obtained with the help of the Microsoft program MSINFO.EXE.

This file can be found under the following path and is directly accessible:

C:\WINDOWS\MSAPPS\MSINFO\MSINFO.EXE

This file can also be accessed from all Microsoft Office programs (MS Word etc.):

In the main menu click on "?" (for help) and in the opening menu on "Info". In the following window click on "Systeminfo".

Installation of RheoWin files

All of the files that comprise RheoWin are installed in the RheoWin directory (e.g. C:\Program Files\Rheowin) and the different RheoWin subdirectories (e.g. C:\Program Files\Rheowin\Drivers etc.).

No RheoWin files are installed in the windows or in the windows system (and similar) directories.

Microsoft DLL's installed during RheoWin installation

Tips & Tricks

The following DLL files are only installed if they are not yet present on the system or if there are older versions of those files on the system. This is mainly dependend on the type and version of the operating system. All of these files are orginally supplied by Microsoft.

Name	Directory	Version	Description	Remarks
Asycfilt.dll	System 1)	2.40.4275	Microsoft OLE 2.40 for Win NTand Win 95	
Ctl3d32.dll	System 1)	2.31.000	Ctl3D 3D Windows Controls	
Comctl32.dll	System 1)	4.72.3110.1	Custom Controls Library	
Comctl32.dll	System 1)	4.72.3612.1702	Custom Controls Library (for Win NT)	
Expsrv.dll	System 1)	6.0.8540	Visual Basic for Applications Runtime – Expression Service	
Mfc42.dll	System 1)	6.00.8447.0	MFCDLL Shared Library – Retail Version	
Msjint35.dll	System 1)	3.51.0623.0	Microsoft Jet Database Engine International DLL	
Msjter35.dll	System 1)	3.51.0623.0	Microsoft Jet Database Engine Error DLL	
Msvcrt.dll	System 1)	6.00.8397.0	Microsoft (R) C Runtime Library	
Msvcrt40.dll	System 1)	4.10.6038	Microsoft® C Runtime Library	
Stdole2.tlb	System 1)	2.40.4275	Microsoft OLE 2.40 for Win NTand Win 95	
Vbajet32.dll	System 1)	6.1.8268	Visual Basic for Applications Develop- ment Environment – Expression Ser- vice Loader	
Vbar332.dll	System 1)	3.0.6908	Visual Basic for Applications Runtime – Expression Service	
Msjet35.dll	System 1)	3.51.2723.0	Microsoft Jet Engine Library	Entry in Registry
Msrd2x35.dll	System 1)	3.51.0623.0	Microsoft (R) Red ISAM	Entry in Registry
Oleaut32.dll	System 1)	2.40.4275	Microsoft OLE 2.40 for Win NTand Win 95	Entry in Registry
Dao350.dll	MS Shared DAO 2)	3.51.1608.0	Microsoft DAO 3.51 Object Library	Entry in Registry
Dao2535.tlb	2)	NA	NA	

- 1) Windows system directory, for example: C:\windows\sy-
- 2) Microsoft shared files directory, for example: C:\Program Files\Common Files\Microsoft Shared\DAO

RheoWin entries in the registry

Like most windows software RheoWin uses the windows registry to save and retrieve certain setting and variables. All RheoWin information is stored under the key HKEY_LO-CAL_MACHINE\Software\HAAKE\RheoWin.

Order no. 006-0564

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Bitte wenden Sie sich bei Servicefragen an uns, unsere Partnerfirmen oder an die für Sie zuständige Generalvertretung, die Ihnen das Gerät geliefert hat.

Please get in contact with us or the authorized agent who supplied you with the unit if you have any services questions.

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