Instruction Manual

HAAKE Viscotester 550

version 1.2

Part of Thermo Fisher Scientific

Thermo SCIENTIFIC

Konformitätserklärung /
Declaration of Conformity

006-0229

Produktbezeichnung / Product name	**Identifikation / Identification**
HAAKE Viscotester VT 550	362-0001 (002-7026) 362-0002 (002-7026)

Hersteller / Manufacturer

Thermo Electron (Karlsruhe) GmbH
Dieselstraße 4
D – 76227 Karlsruhe
Germany

Richtlinie / Norm
Directive / Standard

2004/108/EG	**Richtlinie für elektromagnetische Verträglichkeit** **Electromagnetic Compatibility Directive**
EN 61326-1: 2006	Elektrische Mess-, Steuer-, Regel- und Laborgeräte - EMV-Anforderungen, Teil 1 Allgemeine Anforderungen Electrical equipment for measurement, control and laboratory use EMC-requirements, Part 1 general requirements
2006/95/EG	**Niederspannungsrichtlinie** **Low voltage directive**
EN 61010-1: 2001	Sicherheitsbestimmungen für elektrische Mess-, Steuer-, Regel-, und Laborgeräte - allgemeine Anforderungen Safety requirements for electrical equipment for measurement, control and laboratory use - general requirements

Wir erklären in unserer ausschließlichen Verantwortung, dass das Produkt, auf das sich diese Erklärung bezieht, mit den oben genannten Normen, normativen Dokumenten und den Bestimmungen der genannten Richtlinien übereinstimmt.
Die Prüfprotokolle werden bei Thermo Electron (Karlsruhe) 10 Jahre aufbewahrt.
We declare under our sole responsibility, that this product to which this declaration relates is in conformity with the a.m. standards or other normative documents and is following the provisions of the a.m. directives.
All test certificates will be kept by Thermo Electron (Karlsruhe) for 10 years.

	12.10.2007	Thermo Electron (Karlsruhe) GmbH Dieselstr. 4 * 76227 Karlsruhe Tel. + 49-721-4094-444, Fax + 49-721-4094-418
Unterschrift / Signature Geschäftsleitung/Business Management	Datum/Date	Hersteller/Manufacturer

This instruction manual is part of several information brochures and instruction manuals which together form the documentation for the HAAKE Viscotester® 550.

1. **Book "A Practical Approach to Rheology and Rheometry"**
   Measuring technique and data interpretation
   (Available from Thermo Fisher Scientific under Order No. 222-1347)

2. **Instruction Manual for the HAAKE Viscotester® 550**
   Description of the function elements and
   measuring ranges of the measuring instrument

3. **Instruction Manual for the Operating Software OS550**
   Description of the functions and menus

4. **Instruction Manual for the Application Software HAAKE Viscotester® 550**
   Description of the functions and menus

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The symbols used in this manual and their meaning:

!	Warns that damages to the device and injuries to the user are possible.
☞	Denotes an important remark.
1	Indicates the next operating step to be carried out and…
⇒	…what happens as a result thereof.

1. Quality Assurance

Dear customer,
Thermo Fisher Scientific implements a **Q**uality **M**anagement **S**ystem certified according to DIN/EN/ISO 9001:2000.
This guarantees the presence of organizational structures which are necessary to ensure that our products are developed, manufactured and managed according to our customers expectations. Internal and external audits are carried out on a regular basis to ensure that our **QMS** is fully functional. We also check our products during the manufacturing process to certify that they are produced according to the specifications as well as to monitor correct functioning and to confirm that they are safe. This is why we initiate this monitoring process of important characteristics already during manufacturing and record the results for future reference.

The “Final Test” label on the product is a sign that this unit has fulfilled all requirements at the time of final manufacturing.

Please inform us if, despite our precautionary measures, you should find any product defects. You can thus help us to avoid such faults in future.

2. Your Contact at Thermo Fisher Scientific

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

International / Germany
Thermo Fisher Scientific
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Tel. +49(0)721 4094–444
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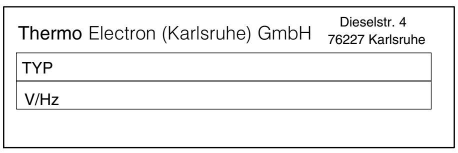
support.mc.de@thermofisher.com
www.thermo.com/mc

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



Unit name printed on the front of the unit and specified on the name plate.

3. Safety Notes and Warnings

The rheometer/viscometer corresponds to the relevant safety regulations. However you are solely responsible for the correct handling and proper usage of the instrument.

This instrument exclusively determines the rheological behavior of fluid and half-solid materials. These materials may not be tested if people can be hurt or devices be damaged.

- ! **The device may not be operated if there are any doubts regarding a safe operation due to the outer appearance (e.g. damages).**
- ! **A safe operation of the instrument cannot be guaranteed if the user does not comply with this instruction manual.**
- ! **Ensure that this instruction manual is made readily available to every operator.**
- ! **This unit should only be used for the applications it was designed for.**
- ! **Make sure that the unit has been switched off before you connect or disconnect the cables. This is to avoid electrostatic charging resulting in a defect of the electronic circuit boards.**
- ! **Do not operate the unit with wet or oily hands.**
- ! **Do not immerse the unit in water or expose it to spray water.**
- ! **Do not clean the unit using solvents (fire danger!) – a damp cloth applied with a household cleaning substance is often sufficient.**
- ! **Repairs, alterations or any work involving opening up the unit should only be carried out by specialist personnel. Considerable damage can be caused by incorrect repair work. The Thermo Fisher Scientific service department is at your disposal for any repairs you may require.**
- ! **Have the unit serviced by specialists at regular intervals.**

! We do not know which substances you intend to test using this unit. Many substances are…

- inflammable, easily ignited, explosive
- hazardous to health
- environmentally unsafe

i.e.: **dangerous**

You alone are responsible for your handling of these substances!

Our advice:

- If in doubt, consult a safety specialist
- Read the product manufacturer’s or supplier’s “EU SAFETY DATA SHEET”
- Read the REGULATIONS CONCERNING DANGEROUS MATERIALS
- Observe the “Guidelines for Laboratories”

4. Unpacking

4.1 Damaged in transit?

- Inform delivery agent (freight agent, railway)
- Prepare damage report

Before returning instrument:

- Inform manufacturer
  (minor damage can often be rectified in site)

4.2 Contents of delivery

The HAAKE Viscotester® 550 is supplied in a transport case together with a power supply unit.

4.3 Accessories

Approximately thirty different sensor systems which are described in detail in the section “**Sensor Systems**” are available for various applications and measuring ranges. Furthermore, Thermo Fisher Scientific supplies the following optional accessories:

Order No.:	Description:
002-6697	Power unit
222-1143	Pt100 temperature sensor (rigid)
333-0472	Pt100 temperature sensor for temperature control vessel
333-0269	Pt100 temperature sensor for measuring cup MV/SV
808-0579	Universal joint for E, FL rotors
222-1139	Adapter for ISO-rotors
091-5035	Software HAAKE RheoWin for HAAKE Viscotester® 550
092-1005	Operating software OS550
222-1323	HAAKE Viscotester ® 550 computer connection package consisting of: Operating software OS550 (092-1005) Cable PC – VT550 (222-1320) RS232 adapter 9 to 25 Pin (222-1322) RS232 printer adapter (222-1321)

4.4 Information concerning the CE sign

Thermo Scientific electrical equipment for measurement, control and laboratory use bears the CE marking.

The CE marking attests the compliance of the product with the EC-Directives which are necessary to apply and confirms that the apparatus meets all relevant essential requirements of the directive, the defined relevant protection requirements.

The conformity assessment procedures were performed following a defined methodology according to each applicable directive.

The council decision 93/465/EEC shall be authoritative concerning the modules of the various phases of the conformity assessment procedures and the rules for the affixing and use of the CE marking, which are intended to be used in the technical harmonization directives.

To confirm compliance with the EC-Directive 2004/108/EC Electromagnetic Compatibility (EMC) our product was tested according to the EMC requirements for emission and immunity for electrical equipment for measurement, control and laboratory use.

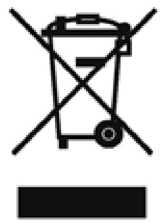
Compliance with the protection requirements areas (domestic establishments and establishments directly connected to a low voltage power supply network which supplies buildings used for domestic purposes) and industrial areas is ensured.

Our strict standards regarding operating quality and resulting considerable amount of time and money spent on development and testing reflect our commitment to guarantee the high level of quality of our products even under extreme electromagnetic conditions.

Practice however also shows that even electrical equipment which bears the CE marking such as monitors or analytical instruments can be affected if their manufactures accept an interference (e.g. the flickering of a monitor) as the minimum operating quality under electromagnetic compatibility phenomena. For this reason we recommend you to observe a minimum distance of approx. 1 m from such equipment.

4.5 WEEE Compliance

This product is required to comply with the European Union’s Waste Electrical & Electronic Equipment (WEEE) Directive 2002/96/EC. It is marked with the following symbol:



Thermo Fisher Scientific has contracted with one or more recycling/disposal companies in each EU Member State, and this product should be disposed of or recycled through them. Further information on Thermo Fisher Scientific compliance with these Directives, the recyclers in your country, and information on Thermo Fisher Scientific products which may assist the detection of substances subject to the RoHS Directive are available at www.thermo.com/WEEERoHS

5. Unit Description

The HAAKE Viscotester® VT550 is...

a laboratory instrument, i.e. it is designed for usage by skilled and trained specialist personnel.

a Viscotester, i.e. it is generally used to examine the rheological (i.e. physical) material parameters of liquid or semi-solid substances. The chemical, ecological and physiological characteristics of the substance to be tested should be taken into account of by the user.

a mobile, electric instrument, i.e. the particular conditions prevalent to the operation location should be taken into consideration. This means in particular: The unit must not be used in a potentially explosive environment.

A completely functional unit consists of the following sub-components:

a) HAAKEViscotester® 550 and power supply unit, temperature control vessel with sensor system, Pt100 temperature sensor and stand

Pic.1:HAAKE Viscotester® 550 wiht cilynder geometry

b) (not illustrated) HAAKE Viscotester® 550 and power supply unit, Pt100 temperature sensor immersion rotor, stand.

c) HAAKE Viscotester® 550 and power supply unit, HAAKE PK100 / 200 -sensor system with cone.

Pic. 2:HAAKE Viscotester® 550 with PK100

Pic. 3:HAAKE Viscotester® 550 with PK200

5.1 Operating possibilities

The HAAKE Viscotester® 550 can be used as a stationary laboratory viscometer.

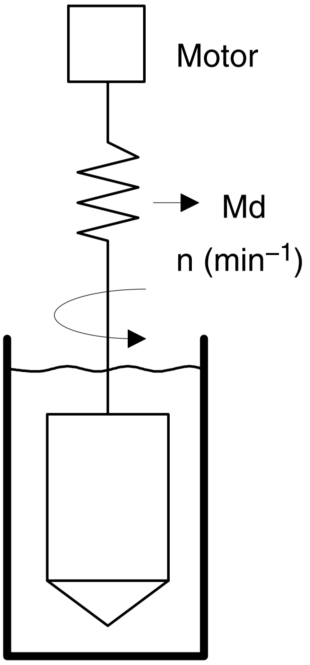
- in this case it is fixed to the stand or installed on the HAAKE PK100 / 200.

or as a portable unit, to be used “in-the-field”.

- The HAAKE Viscotester® 550 has been designed with an optimum weight distribution so that it can be comfortably hand-held.

Pic. 4: HAAKE Viscotester® 550 wit Immersion tube

☞ The measuring shaft is extremely sensitive to knocks and bumps. Please bear this in mind when operating this instrument! (i.e. when putting it down, packing it away, unpacking it etc.).



5.2 Functional principle

The substance to be measured is located in the measuring gap of the sensor system. The rotor is rotated at a preset speed (n). The substance to be measured exerts a resistance to this rotational movement (due to it's viscosity) which becomes apparent as a (braking) torque value (Md) applied on the measuring shaft of the HAAKE Viscotester® 550.

The built-in computer calculates the relevant measuring values for the following factors from the measured variables of speed, torque and sensor geometry (system factor)

Viscosity η	in mPas
Shear rate $\dot{\gamma}$	in s^{-1}
Shear stress τ	in Pa

The temperature T is also calculated in °C if a temperature sensor is attached.

The results are shown on the display of the HAAKE Viscotester® 550 and passed on to a computer or printer via the RS232 interface if connected.

6. Setting Up

6.1 Stand

The usage of the stand enables the HAAKE Viscotester® 550 to be used in a position suitable for most applications.

It consists of the following sub-components:

- Stand base
- Stand post
- Clamping bracket

Assembly:

1. Fix the clamping bracket to the HAAKE Viscotester® 550 using the three countersunk screws (M4 x 8 DIN 966).

2. Attach the stand post to the stand base. Insert the stand post far enough into the stand base so that both screw recesses are visible from the rear of the stand base.

   Two locking screws (hex-socket set screw 3 mm M6 x 1) are located at these recesses and which should now be screwed tight flush to the base.

3. Now attach the HAAKE Viscotester® 550 to the stand post and secure at the desired height.

4. Adjust the stand:

   The stand has four adjustment screws; these can be used to compensate for uneven surfaces. They should always be set to ensure that measuring shaft of the HAAKE Viscotester® 550 is as perpendicular as possible.

Notes on handling:

- locate the stand on a fixed, vibration-free surface.
- Ensure that the complete test assembly is stable and cannot tip over.

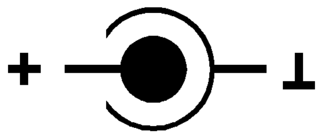
6.2 Power supply unit

The power supply unit converts the mains voltage to the correct operating voltage of the HAAKE Viscotesters® 550. It can be connected to all standard mains voltage ratings without separate alteration or switching. It has no mains switch and can be left connected permanently to the mains; this connection is therefore is only broken when the mains plug is removed from the socket.

The operating voltage (“output voltage”) generated is a so-called “protective low voltage with safe separation” (VDE 0100T4100) and is thus “contact insensitive” (EN 61010T1). The casing is made according to protective class II and is therefore not connected to the protective conductor of the mains supply (protective insulation acc. to VDE 0100T410)

The complete technical specifications are detailed in the chapter **Technical Specifications**.

The connecting plug is wired as follows:

inner contact  outer contact

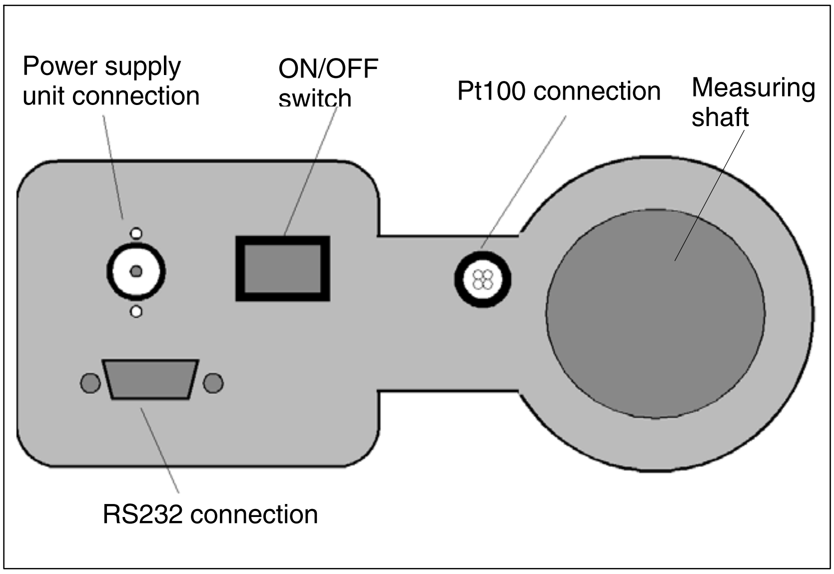
Mains voltage range . . . . 100–240 V ± 10%

Mains frequency . . . . 47–63 Hz

Output voltage . . . . 18 V ± 2%, D.C.

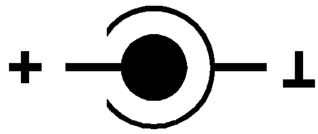
7. Functional Elements

Unit bottom



Power supply unit connection socket

! **Only the correct HAAKE power supply unit should be connected at this socket. The usage of other power supply units would probably seriously damage the electronics of the HAAKE Viscotester® 550,**



permissible voltage: 18 V ± 2%, D.C.

ON/OFF switch

The switch serves to the HAAKE Viscotester® 550 on and off. The decimal point lights up on the HAAKE Viscotester® 550 display to indicate operational status.

Measuring shaft

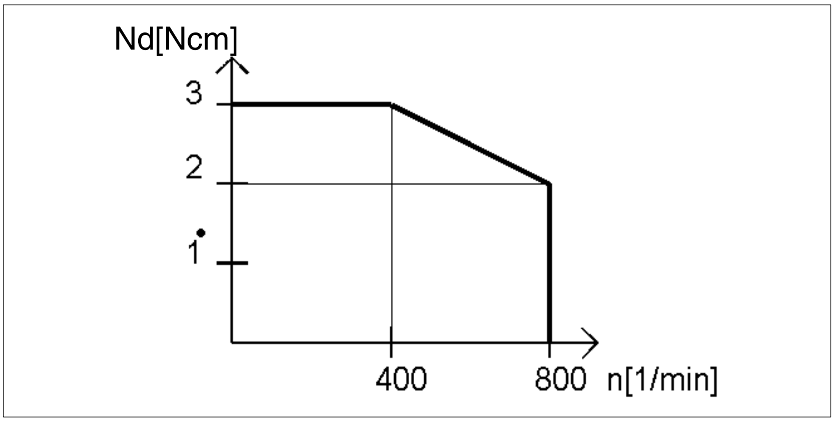
The sensor system rotor is connected to the measuring shaft via the screw coupling: the measuring shaft should be held by the knurled nut, the rotor then carefully inserted and rotated tight without using excessive force. The measuring shaft and coupling must remain free of dirt or contamination. The coupling serves two main purposes:

1. the secure connection of the (internal) drive and measuring system with the rotor,

2. the centering of the rotor, i.e. the concentric rotation of the rotor **without** “impact” or “balance error”.

“Impact” or “balance error” lead to measurement errors and can irreparably damage the measuring system of the HAAKE Viscotester® 550. The presettable speed and measurable torque can vary within a certain range.

Torque-Speed Characteristic Curve HAAKE Viscotester® 550

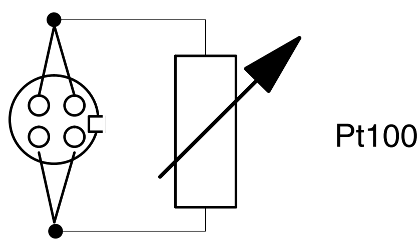


The significant points are:

Speed:	0,5 – 800 min^{-1}
in the CD mode:	0,0125 – 5 min^{-1}
Torque:	0,01 – 3 Ncm

PT100 sensor connection

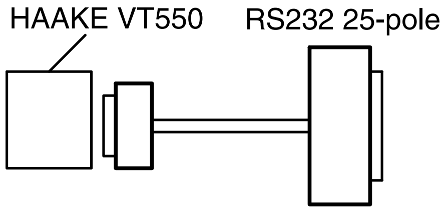
Two-wire Pt100 temperature sensors can be connected to this socket. The temperature display usually must be calibrated before usage.



Serial interface RS232

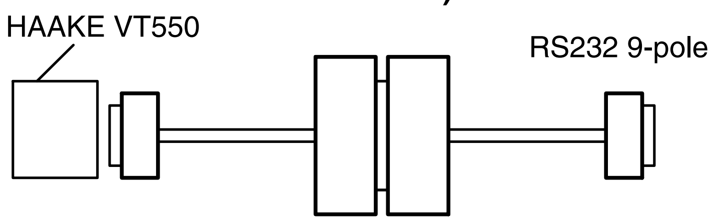
The HAAKE Viscotester® 550 can exchange data with other connected units via this interface. The type of connection cable required depends on the hardware configuration of the connected device in question:

a) Connection VT – 25-pole RS232



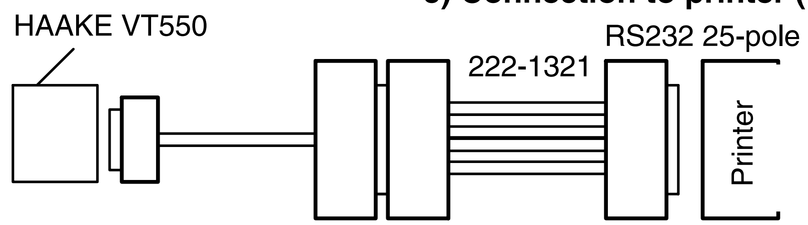
Cable, Order No.: 222-1320

b) Connection VT – 9-pole RS232



Cable, Order No.: 222-1320
and Order No.: 222-1322

c) Connection to printer (cable + printer adaptor)



Cable, Order No.: 222-1320
and Order No.: 222-1321

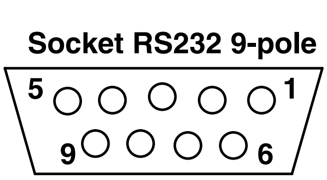
Free printer storage must amount to at least 8 KByte.

The operating mode is determined as follows:

- 9600 Baud
- 8 data bits, 1 stop bit
- without parity
- hardware handshake RTS/CTS

Pin configuration:

Socket RS232 9-pole



Pin 2	TxD	Transmit Data	Output VT550
Pin 3	RxD	Receive Data	Input VT550
Pin 5	GND	Signal Ground	
Pin 7	CTS	Clear to Send	Input VT550
Pin 8	RTS	Request to Send	Output VT550

8. Connecting the Unit to the Mains

The power supply unit for the HAAKE Viscotester® 550 (Order No. 002–6967) can be used for all usual input voltages. The power supply unit can remain permanently connected to the mains (with or without the HAAKE Viscotester® 550 connected).

The unit must only be connected to a socket with a protective earth. Compare your local voltage with the rating specified on the name plate before connecting the unit to the mains.

9. Operating Modes

Preliminary note

The HAAKE Viscotester® 550 has a wide variety of measuring possibilities. The operation of the HAAKE Viscotester® 550 was split as outlined below to keep operating steps and multiple key assignments on the HAAKE Viscotester® 550 keypad as simple as possible:

- Calling up all stored data via the keypad and display
- Entering variable parameters (speed, procedures, etc.) via a PC and the operating software OS550. (Only the temperature offset and sensor adaptation can be entered via the keyboard).

We can thus differentiate between two main operating modes:

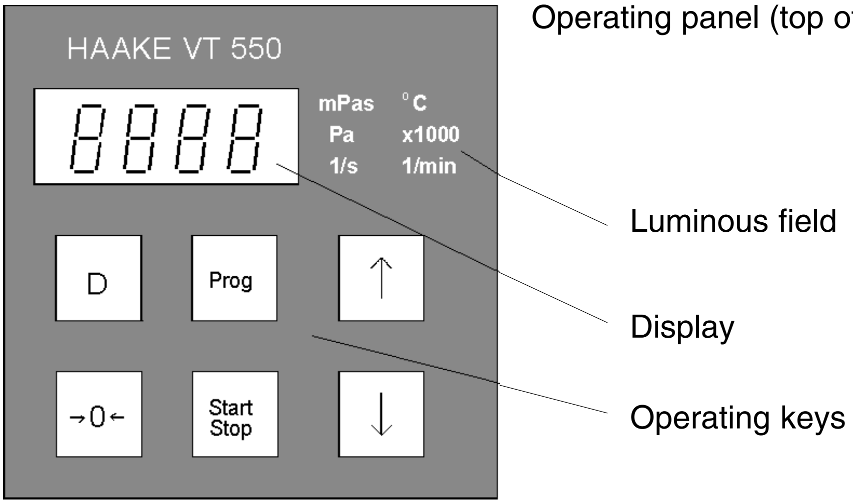
Normal / Manual Operation (see also chapter 9.2)

The entire operation is carried out via the keypad. So-called procedures can also be run in this mode: the HAAKE Viscotester® 550 works through an entered and stored procedure; data is collated and automatically sent to a connected printer.

PC Operation (see also chapter 9.3)

The HAAKE Viscotester® 550 is controlled directly from a PC via the so-called applications software: measurement and evaluation are carried out automatically. Manual operation is locked but the HAAKE Viscotester® 550 can be stopped via the keypad.

9.1 Manual operation



Operating panel (top of unit)

Element:	Function:	Description:
D	Display key	Used for selecting the desired variable: Viscosity, shear stress, temperature, speed and shear rate. The luminous field indicates the respective measuring unit:
mPas		Luminous field for viscosity display in mPas
Pa		Luminous field for shear stress display in Pa Special case: If the sensor system = 0, the displayed value corresponds to torque in Ncm (as the f and M factors = 1, compare with chapter 9.4). This field flashes if the torque signal is either less than 5% or more than 100% of the maximum torque. This signifies: – the measuring fault has increased – torque is too high. If the torque value exceeds 115% of the maximum torque (see torque-speed characteristic curve), the motor blocks (due to overload protection) and has to be restarted (at a lower speed if necessary).
x1000		Luminous field for measuring range expansion for viscosity and shear stress display, i.e. the displayed value should be multiplied by one thousand when this field is illuminated.
°C		Luminous field for temperature display in °C – the display shows +350°C when no sensor is connected – the display shows –50°C when the connection is shorted

Element:	Function:	Description:
1/min		Luminous field for speed display in 1/min
1/s		Luminous field for shear rate display in 1/s
Prog	Program key	Key for selecting sensor systems, speed programs, procedures and temperature compensation (offset). Depressing this key once displays the last selected mode (**S**ystem, Spee**d** program, **P**rocedure) for a few seconds and repeatedly pressing this key allows the user to switch to the next mode. Examples: Display "S. 33" **s**ensor system no. 33 Display "d. 5" spee**d** program no. 5 Display "P. 10" **P**rocedure no. 10 Display "o. 1.0" **O**ffset 1.0 °C; luminous field °C lights up The display shows "d. E" or "P. E" for speed program and procedure respectively in the external mode (computer mode). These values cannot be altered as long as the application software has access to the HAAKE Viscotester® 550.
↑ ↓	Selection keys	This key allows the user to switch upwards or downwards between the various parameters (sensor system, speed program, procedure number) after first pressing the program key. Pressing these keys just once displays the last selected speed level for a few seconds. Repeated pressing of these keys switches upwards or downwards between the various speeds in the speed program. The current speed values can be displayed via the D key display "1/min".
→0←	Zero point key	Key for torque value zero point correction. Pressing this key once sets the current torque value to zero. A zero point correction is carried out automatically when the unit is witched on. Zero point calibration is not possible if the fault message "E. I" (unit is faulty) is shown on the display.
Start Stop	Start/stop key	Pressing this key starts the motor at the speed selected. This start is also indicated by the flashing decimal point in the display. Pressing this key again stops the motor and the decimal point stops flashing.

9.2 PC operation using the "OS550" software

The PC operating mode for the HAAKE Viscotester® 550 has two main fields of application:

1. The speed programs, speed and procedures which can be loaded via the respective keys (in the manual mode) should at all times (although permanently stored) be accessible for alteration by the user depending on the requirements of the specific application.
2. Measuring procedures and their evaluation, representation and documentation which are highly application-specific should be defined once and from then on be automatically available as a standard routine to be run fully automatically as often as desired.

Both above mentioned examples require the following:

- a PC
- a connection cable HAAKE Viscotester® 550 – PC
- the operating software "OS550"
- the application software

the second example additionally requires:

- an RS232 printer
- a connection cable PC – printer and adaptor

Certain minimum requirements are placed on the computer and peripheral equipment (if any) to be used by the hard- and software:

PC:

- 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
- Graphics adapter, resolution 1024 x 768
- Serial interface (RS232C) for measuring instrument
- Microsoft® or PS/2® mouse

Peripheral equipment:

Plotter with an RS232 interface

9.3 Display Modus Display HAAKE Viscotester® 550 with Procedures and Operating Software 2.1

Existing HAAKE Viscotester® 550 can be converted by exchanging the EPROM.

At the end of the procedure the CD- and CR-values displayed will remain "frozen" in both measuring modi. The display is blinking and the values can be called off with the D-key. Pressing the STOP-key will finally stop the procedure and the unit will show the actual values again. The printer output of the measuring values remains unchanged.

Display	**CD-Modus** (procedure 1+2) **during/after** procedure	**CR-Modus** (procedure 3-10) **during/after** procedure	**Normal Operation** (procedure 0)
Tau [Pa] actual Tau_{max} [Pa] Tau [Pa] of the last measuring point	 x x	x x	x
n [min^{-1}] actual n [min^{-1}] at Tau_{max} n [min^{-1}] of the last measuring point	 x x	x x	x
D [s^{-1}] actual D [s^{-1}] at Tau_{max} D [s^{-1}] of the last measuring point	 x x	x x	x
η [mPas] actual η [mPas] at Tau_{max} η [mPas]	 x x	x x	x
T [°C] actual T [°C] at Tau_{max} T [°C] of the last measuring point	 x x	x x	x

The change is available as of version 2.1 of the operating software. The version number is shortly displayed when switching on the HAAKE Viscotester® 550.

9.4 Operating notes

System factors

The geometric characteristics of a sensor system are described by the so-called SYSTEM FACTORS (“f” and “M”). They are required for calculating the variables shear stress, shear rate and viscosity from the torque and speed values (see chapter 8). Every sensor system has a “description” and a “system number”:

e.g. Description: MV 1

System no.: 3

The f and M factors are saved under the system no. i.e. in this case system no. 3 → f = 65.7 / M = 2.340.

The HAAKE Viscotester® 550 can save 45 pairs of system factors f and M under the system numbers 0 – 44.

The system numbers 0 – 29 are already defined by default and the system numbers 30 – 44 are available for user-definition. The system numbers 30 – 39 have however been reserved in advance (PK and PQ).

The system numbers are selected as follows:

		The display shows the currently selected variable.	
Prog	press several times	The display shows	S. 1
↑ or ↓		The display shows e.g. as selected	S. 3
	wait approx. 5 secs.	The display returns to the original mode	

Operating Modes

System No.	Description	f Factor	M Factor
0	–	1.0	1.000
1	MV-DIN	61.4	1.290
2	SV-DIN	369.4	1.290
3	MV 1	65.7	2.340
4	MV 2	76.8	0.900
5	MV 3	111.0	0.440
6	SV 1	253.0	0.890
7	SV 2	768.0	0.890
8	NV	36.3	5.410
9	MV – E	55.8	1.290
10	SV – E	322.1	1.290
11	MV 1 P	65.7	2.000
12	MV 2 P	76.8	0.880
13	SV 2 P	768.0	0.780
14	HV 1 – DIN	2527.5	1.290
15	E 3	8.5	0.100
16	E 30	85.4	0.100
17	E 100	285.0	0.100
18	E 500	1424.5	0.100
19	E 1000	2850.0	0.100
20	FL 10	54.0	0.209
21	FL 100	564.0	0.209
22	FL 1000	5246.8	0.209
23	B 1	14.3	0.100
24	B 2	57.0	0.100
25	B 3	143.0	0.100
26	B 4	285.5	0.100
27	B 5	570.0	0.100
28	B 6	1430.0	0.100
29	B 7	5715.0	0.100
30	PK1, 2°	1740.0	3.000
31	PK1, 1°	1740.0	6.000
32	PK1, 0.5°	1740.0	12.000
33	PK2, 1°	4775.0	6.000
34	PK2, 0.5°	4775.0	12.000
35	PK5, 2°	306.0	3.000
36	PK5, 1°	306.0	6.000
37	PQ 1	2320.0	1.470
38	PQ 2	6366.0	1.050
39	PQ 5	408.0	2.620
40 – 44	USER	––	––
–	HS 1	1173.0	40.0
–	HS 2	1142.4	10.0

System factors – Calculation procedure:

Viscosity describes by definition the relationship between shear stress τ and shear rate $\dot{\gamma}$

$$\eta = \frac{\tau}{\dot{\gamma}} \qquad (1)$$

Starting with the measured values for “torque Md” and “speed n”, the geometric characteristics of the various sensor systems are accounted for in the calculation by the system factors; the equation (1) can now be supplemented as follows:

$$\eta = \frac{f \cdot Md}{M \cdot n} \qquad (2)$$

The further steps in the calculation process are not illustrated here, but they result in the following equations:

Cylinder systems:

$$f = \frac{0.01}{2\pi L R_i^{\,2}} \quad \text{and} \quad M = \frac{\pi}{15} \frac{R_a^{\,2}}{R_a^{\,2} - R_i^{\,2}}$$

Plate-cone systems:

$$f = \frac{0.03}{2\pi R_i^{\,3}} \quad \text{and} \quad M = \frac{\pi}{30 \cdot \alpha}$$

where:

R:	radius of the rotor, (inner cylinder or cone)	measured in	[m]
Ra	radius of the cup, (outer cylinder)	”	[m]
L	rotor length, (inner cylinder)	”	[m]
α	angle of rotor, (cone)	”	[rad]
Md	torque,	”	[Ncm]
n	rotor speed,	”	[1/min]

Example: Sensor system MV1 (see table in chapter 8.2)

R_i = 20.04 mm = 0.02004 m; R_i^2 = 0.0004016 m^2

R_a = 21.00 mm = 0.0210 m; R_a^2 = 0.000441 m^2

L = 60.00 mm = 0.0600 m; L = 0.0600 m

the following calculation can thus be made:

f = 66.05 [Pa/Ncm] and

M = 2.344 [min/s]

and thus the measured (maximum) value for

torque Md = 3 Ncm = 0.03 Nm and

speed n = 800 min^{-1}

the highest values for:

shear stress $\tau = f \cdot Md = 66.05 \cdot 3Ncm = 198.15$ [N/m^2]=[Pa]

shear rate $\dot{\gamma} = M \cdot n = 2.344 \cdot 800 = 1875$ [1/s]

☞ The values shown in the sensor system tables vary slightly in the figures specified for f. This takes into account any possible end effects in the measuring gap.

The table on page 21 shows the system factors of all sensor systems which are used as standard with the HAAKE Viscotester® 550. Most of these values are already stored in the HAAKE Viscotester® 550.

Speed programs

The HAAKE Viscotester® 550 has ten speed programs with ten levels each. The rows one to six are programmed as default and cannot be altered and the rows 7 to 10 can be programmed as desired using the OS550 software.
The speeds can be entered when the HAAKE Viscotester® 550 is running.

The following programs are preset:

Rows	1	2	3		4		5	6	7	8	9	10
#	Std. HAAKE [l/min.]	LOG HAAKE [l/min]	A $\dot{\gamma}$ ISO 3219 [1/s]	A ISO 3219 [1/min]	B Gpkt ISO 3219 [1/s]	B $\dot{\gamma}$ ISO 3219 [1/min]	Fann [1/min]	ISO 2555 [1/min]	free	free	free	free
1	5.0	0.5	1.0	0.8	0.6	0.5	3.0	2.0				
2	8.3	1.1	2.5	1.9	1.0	0.8	6.0	2.5				
3	13.9	2.6	5.0	3.9	2.5	1.9	30.0	4.0				
4	23.2	5.9	10.0	7.8	6.3	4.9	59.9	5.0				
5	45.3	13.3	25.0	19.4	16.0	12.4	90.1	10.0				
6	64.6	30.1	50.0	38.8	40.0	31.0	100.2	20.0				
7	107.8	68.3	100.0	77.6	100.0	77.6	179.9	50.0				
8	179.6	155.3	250.0	193.9	250.0	193.9	200.1	59.9				
9	297.6	352.4	500.0	387.6	630.0	488.3	300.0	100.2				
10	500.0	800.4	1000.0	775.6	1000.0	775.6	600.0	200.1				

They are selected as follows:

	The display shows the currently selected variable.	
[Prog] press	The display shows	d. 1
[↑] or [↓]	The display shows	d. 3
wait approx. 5 s	The display switches back to the variable shown to start with.	
[↑] or [↓]	Increasing or decreasing speed values from the selected speed program are shown in the display.	

List of Procedures

The procedures are pre-assigned as follows, but can be changed.

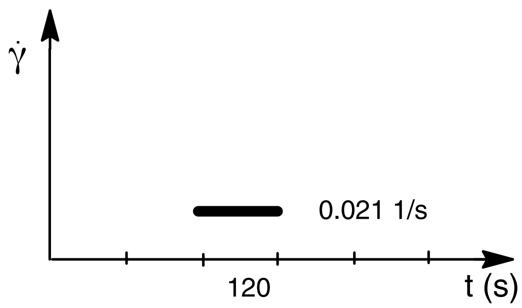
Proc. no.	Function	Sensor system
1	Determination of the yield point	FL10
2	Determination of the yield point	FL10
3	Flow curve up	PK 5 1°
4	Flow curve up	PK 5 1°
5	Flow curve up/down	MV-DIN
6	Thixotropy	MV-DIN
7	Thixotropy/flow curve	MV-DIN
8	Hardening	PQ2
9	1 point measurement	MV-DIN
10	3 point measurement	MV-DIN

The complete parameters can be outputted under the menu '**Print**'.

The procedures no. 5 and 6 are special procedures, which always display the average value of the viscosity over all measuring points of the segment instead of the viscosity.

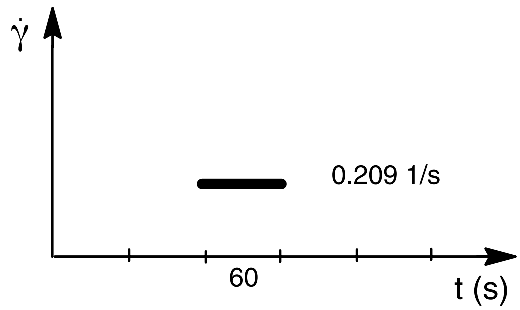
Procedures No. 1 Determination of the yield point

System 20 FL 10
D3 = 0.021 1/s
t3 = 120 s
Mp3 = 100



Procedures No. 2 Determination of the yield point

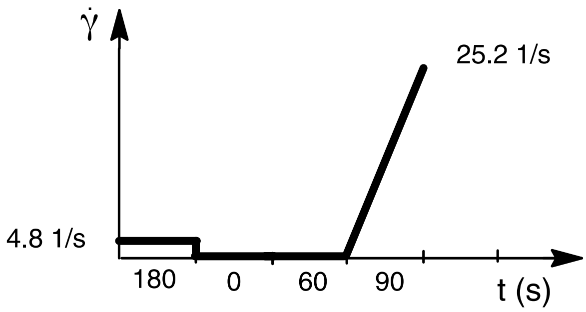
System 20   FL 10
D3   = 0.209 1/s
t3   = 60 s
Mp3 = 20



Procedures No. 3 Flow curve up

System 36   PK 5 1°

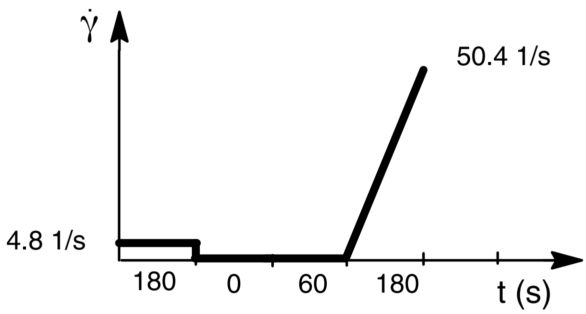
D1 = 4.8 1/s	D2 = 0	by	D3 = 0	D4 =25.2 1/s
t1 = 180 s	t2 = 0		t3 = 60 s	t4 =90 s
Mp1 = 7	Mp2 = 0		Mp3 = 1	Mp4 =35



Procedures No. 4 Flow curve up

System 36   PK 5 1°

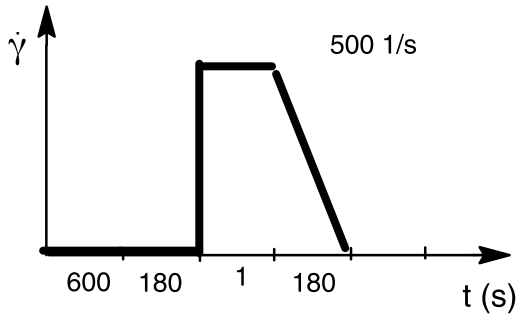
D1 = 4.8 1/s	D2 = 0 1/s	by	D3 = 0 1/s	D4 =50.4 1/s
t1 = 180 s	t2 = 0		t3 = 60 s	t4 =180 s
Mp1 = 7	Mp2 = 0		Mp3 = 1	Mp4 =35



Procedures No. 5 Flow curve up/down

System 1 MV–DIN

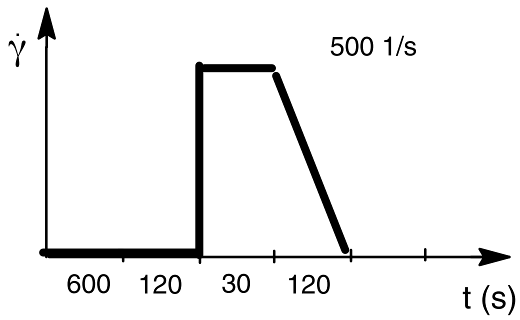
D1 = 0 1/s by	D2 = 0 1/s	D3 = 500 1/s	D4 = 0 1/s
t1 = 600 s	t2 = 180 s	t3 = 1 s	t4 = 180 s
Mp1 = 1	Mp2 = 20	Mp3 = 1	Mp4 = 20



Procedures No. 6 Thixotropy

System 1 MV–DIN

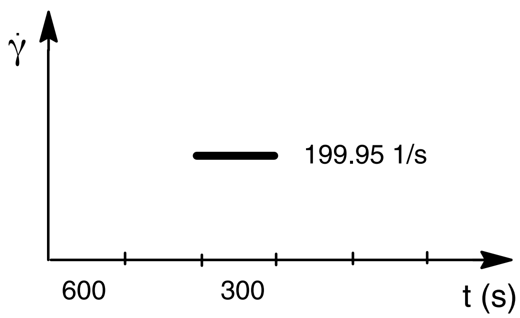
D1 = 0 1/s by	D2 = 0 1/s	D3 = 500 1/s	D4 = 0 1/s
t1 = 600 s	t2 = 120 s	t3 = 30 s	t4 = 120 s
Mp1 = 1	Mp2 = 45	Mp3 = 9	Mp4 = 45



Procedures No. 7 Thixotropy/flow curve

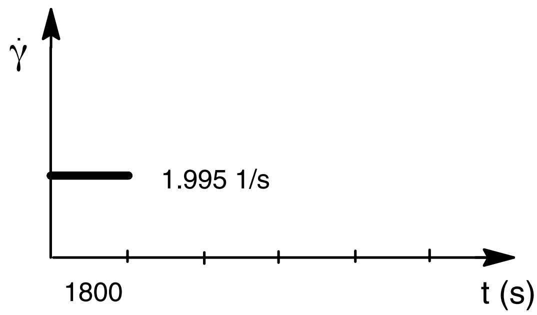
System 1 MV–DIN

D1 = 0 1/s	D2 = 0 1/s	D3 = 199.95 1/s
t1 = 600 s	t2 = 0 s	t3 = 300 s
Mp1 = 1	Mp2 = 0	Mp3 = 99



Procedures No. 8 Hardening

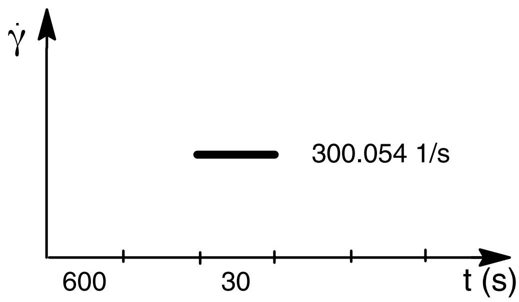
System 38 PQ 2
D1 = 1.995 1/s
t1 = 1800 s
Mp1 = 100



Procedures No. 9 1 point measurement

System 1 MV−DIN

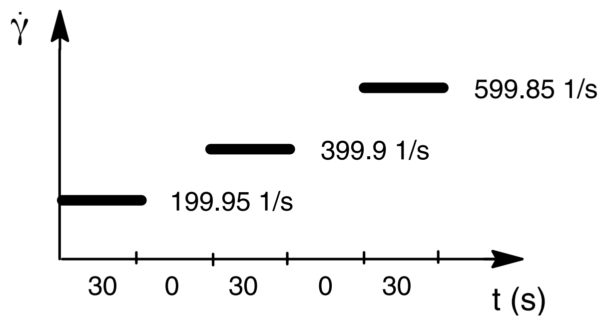
D1 = 0 1/s	D2 = 0 1/s	D3 = 300.054 1/s
t1 = 600 s	t2 = 0 s	t3 = 30 s
Mp1 = 1	Mp2 = 0	Mp3 = 30



Procedures No. 10 3 point measurement

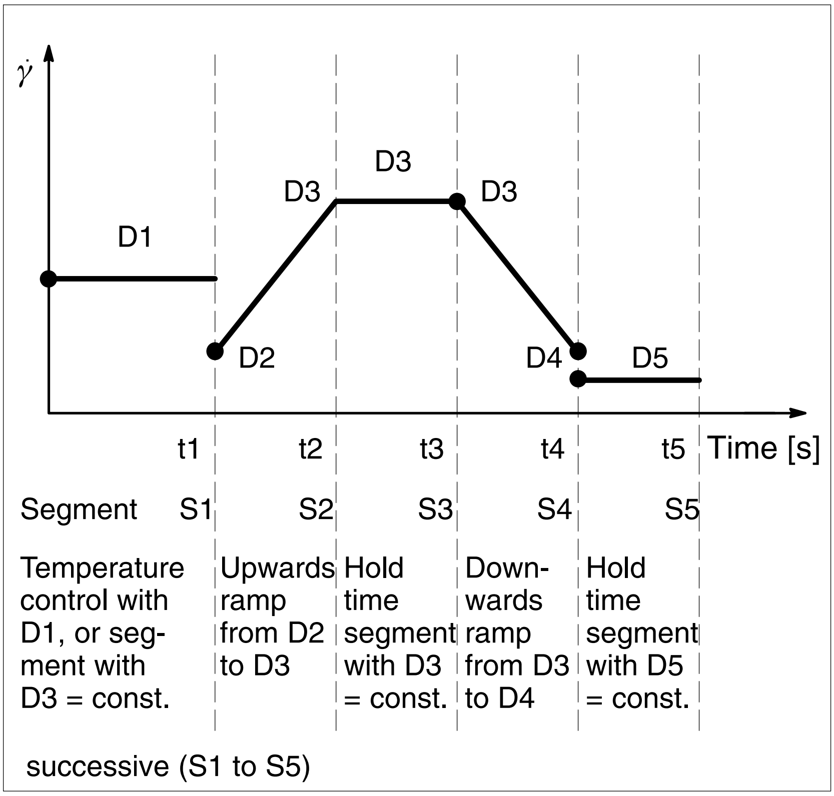
System 1 MV−DIN

D1 = 199.95 1/s	D2 = 0 1/s	D3 = 399.0 1/s	D4 = 0 1/s	D5 = 599.85 1/s
t1 = 30 s	t 2 = 0 s	t3 = 30 s	t4 = 0 s	t5 = 30 s
Mp1 = 13	Mp2=0	Mp3= 13	Mp4 = 0	Mp5 = 13



HAAKE HAAKE Viscotester® 550 procedures

A sequence program called a procedure is incorporated within the HAAKE Viscotester® 550. This procedure is illustrated below:



A procedure consists of 5 successive freely selectable values (S1 to S5):

- system number for the procedure
- shear rate D1 to D5
  in the range $0 \leq n_i \leq 800$ [1/min]
- measuring time per segment t1 to t5
  in the range $0 \leq t_i \leq 36000$ [s]
- number of measuring points per segment Mp1 to Mp5
  in the range $0 \leq Mp_i \leq 100$

A segment will be carried out if t > 0; it will not be carried out if t = 0 is set. At least one measuring point must be defined per activated segment. All segments **together** must not contain more than one hundred measuring points.

Ten different procedures can be stored for usage with the HAAKE Viscotester® 550 under the numbers 1 to 10. All the parameters described above are freely programmable using the OS550 software and remain permanently stored in the HAAKE Viscotester® 550.

The procedures are selected as follows:

	The display shows the currently selected variable.	
Prog press	The display shows	P. 1
↑ or ↓	The display shows the desired procedure number	P. 5
	Important! The VT550 is in normal mode	P. 0
	P. 1 and	P. 2
	Special procedure for yield point determination "CD mode"	
wait approx. 5 s	The display switches back to the variable shown to start with.	

After the procedure has terminated all measuring data is passed on to a connected printer for data output via the serial interface. The measuring data is deleted if there is no printer connected at the serial interface.

The data can also be passed on to a suitable terminal program if a printer is not available.

The protocol can written in one of three languages. Language definition is carried out in the OS550 software.

The shear stress is displayed with the yield point procedures 1 and 2. The maximum value encountered during the procedure is saved and flashes in the display at the end of the procedure. This value can be deleted by pressing the key Start/Stop. The HAAKE Viscotester® 550 can now be restarted.

The procedure protocol contains the following information:

Protocol heading:

HAAKE Viscotester VT550
Substance:________Sample No.:_______Date:________Page 1/1
Company:_________Operator: _________________Analysis No. 23
System #: 1 f: 61.4 M: 1.290

Protocol page no.

System factors of the selected procedure

Successive analysis no.
This number is counted upwards at the start of every procedure and is saved permanently.

1st data block:

Prog.	Seg.	$\dot{\gamma}$[1/s]	t[s]	#	Eta[mPas]	Tau_max[Pa]
7	1	10.0	20	10	3.036E+02	4.391E+00
.	.	.	.	.	.	.
.	.	.	.	.	.	.

Set value for segment $\dot{\gamma}$, measuring time, number of measuring points

Shear stress for segment

Segment No.

Viscosity mean value for segment

Procedure

2nd data block:

#	Seg.	t[s]	Temp. [C]	D[1/s]	Tau[Pa]	Eta[mPas]
1	1	2.0	20.1	1.00	4.023E+00	3.140E+02
.	.	.	.	.	.	.
.	.	.	.	.	.	.

Time until measuring point

Segment No.

Current measuring point value (a measuring value (Tau) outside the HAAKE Viscotester® 550 limit value is marked with a *)

Measuring point

Temperature display

Select temperature display using the D key.

A temperature offset for sensor adaptation can be entered via the keyboard (Prog key). This adjustment should be carried out for every sensor to be attached or when exchanging sensors.

Immerse the Pt100 temperature in a temperature controlled bath. The bath must have reached a constant temperature; this can be checked using a calibrated thermometer with a resolution of at least 1/10 °C. The Pt100 sensor should now be situated as close to the thermometer as possible and left to settle for at least one minute (temperature display).

e.g.	HAAKE Viscotester® 550V display	37.1 °C
	Reference thermometer	37.9 °C
	Temperature deviation:	0.8 °C

This deviation (“Off-set”) is entered via the HAAKE Viscotester® 550 keypad as the correction value.

Prog press	The display shows the stored correction value	0. x
↑ or ↓	Enter calculated offset value, the display shows (for example above)	0.0.8
wait approx. 5 secs.	The display returns to the original mode	

This calibration should be carried out everay time a sensor is exchanged.

Note:

Sensor not connected:	display +350 °C
Sensor shorted:	display –50 °C

10. Sensor Systems

! **Make sure that the unit has been switched off before you connect or disconnect the cables. This is to avoid electrostatic charging resulting in a defect of the electronic circuit boards.**

The HAAKE Viscotester® 550 can be operated with most of the sensor systems available in the Thermo Fisher Scientific program. Apart from this compatibility, an adapter allows operation with sensors of other types.

The sensor system consists of…
a rotor and stator.

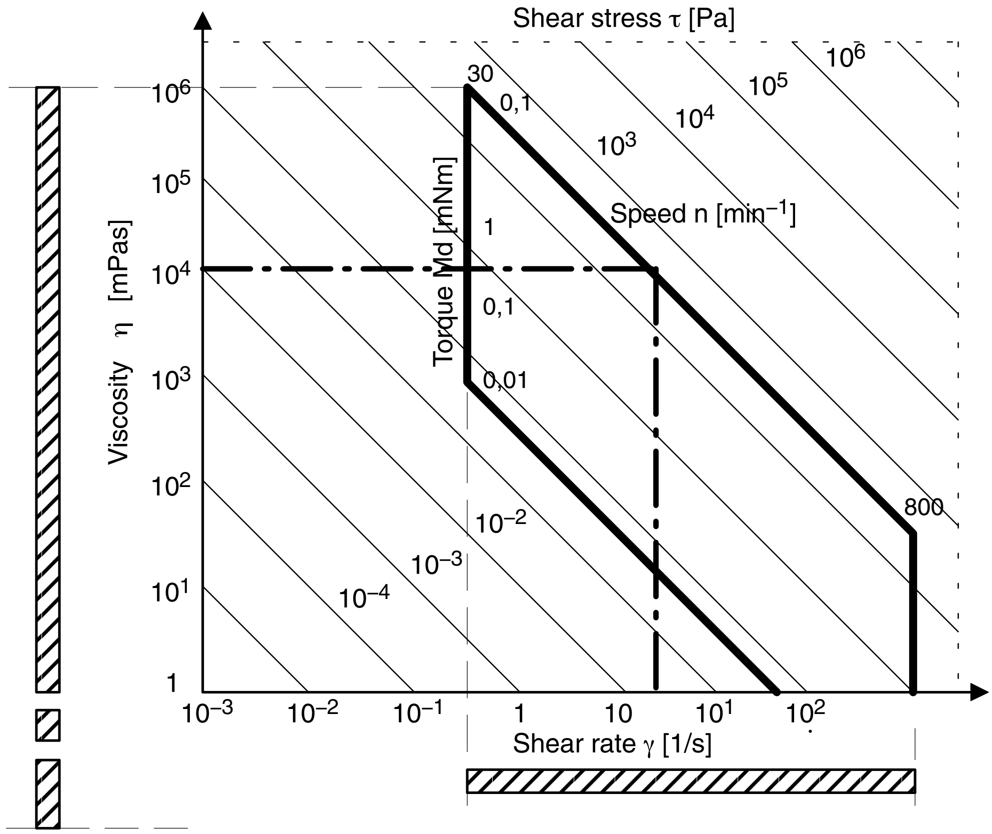
With the exception of the cone and plate and immersion sensor systems, all systems have to be used with the temperature control vessel.

Measuring ranges – Basic measuring ranges

A certain viscosity and shear rate range can be achieved with each sensor system. Thereby a certain viscosity measuring range is set. This relationship can be illustrated in a viscosity-shear rate diagram. Both coordinate axes are scaled logarithmically (in order to be able to cover the larger ranges). In this diagram, the straight lines drawn at angle of 45° represent lines of constant shear stress.

Example: Measuring range of the MV system

The measuring range of this sensor system is shown in the diagram. It is reached if the HAAKE Viscotester® 550HAAKE VT550 speed range (i.e. the range from 0.5 to 800 min^{-1}) and the full measuring range for torque (0.01 to 30 mNm) are used.



The application limits of the HAAKE Viscotester® 550 with this sensor system are marked with "///". The example shown above shows in this case that:
the substance with $\eta = 10^4$ mPas can be measured in the shear rate range of ~0.2 to 20 s^{-1}, which corresponds to the test speeds of 0.5 to 10 min^{-1}; this generates shear stress values of approx. 0.07 to 2 Pa.

The actual effective range for a measuring evaluation is however normally smaller in practice: It is determined by the characteristics of the substance as well as sample preparation as e.g.

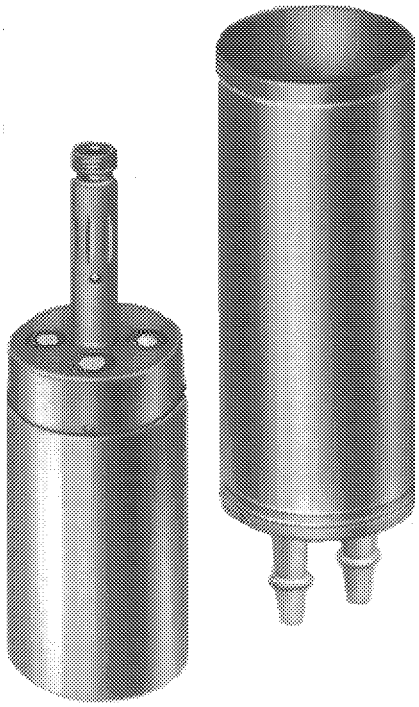
a) samples of high viscosity cannot be filled without air bubbles.

b) instable flow conditions with samples of low viscosity distort the measuring results (vortices).

c) phase separation, sample rupture, slippage of liquids give faulty values.

d) normal stress of viscoelastic samples empties the measuring gap.

The sensor systems stored in the HAAKE Viscotester® 550 are described and their measuring ranges illustrated on the next few pages.



10.1 Sensor System NV

Application:

The NV is primarily used for viscosity measurements of low viscosity liquids such as oils, diluted solutions, fruit juices etc. working in the medium shear rate range.

The sensor system NV consists of the cup and a bell-shaped rotor. It is classified as a coaxial cylinder sensor system with two gaps for shearing the samples on the inside and on the outside of the rotor.

This sensor system is used with the temperature vessel which is connected to an accurate thermal liquid circulator.

The amount of sample should be adjusted so that during operation the top surface of the inner cylinder is just covered.

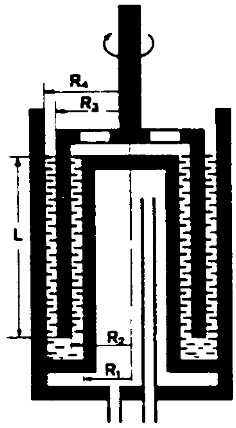
The bell-shaped rotor must not be contacted by the liquid sample on its top end-face. Excess sample must be removed by a pipette or syringe through any of the four holes in the top face of the rotor.

Samples of low viscosity require the sealing rings to prevent the liquid sample to leak through the bottom of the cup.

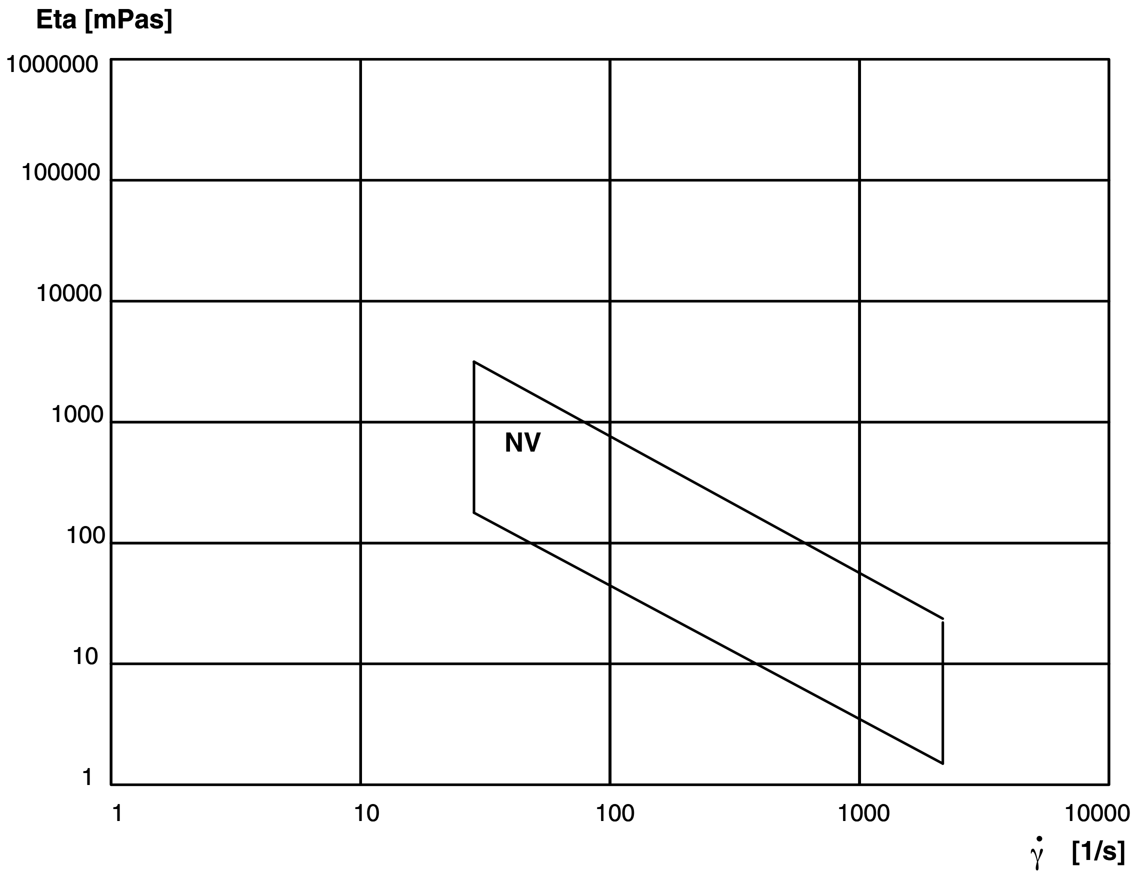
Always keep a sufficient supply of sealing rings in stock as they may deteriorate mechanically or chemically and need to be replaced. (Order No. 807-0701).

Cleaning:

Remove the cross bar at the bottom of the cup and slide the inner cylindrical insert downward to simplify cleaning of the cup.



Sensor System	NV
System No.:	*8*
Inner Cylinder (Rotor) Radius R_2; R_3 (mm) Height L (mm)	 17.85; 20.1 60
Outer Cylinder (Cup) Radius R_1; R_4 (mm)	 17.5; 20.5
Radii Ratio R_a/R_i Gap Width (mm) Sample Volume V (cm^3) Temperature (°C)	1.02 0.35 9.0 –30/100
System Factors f (Pa/Ncm) M (min/s)	 36.3 5.41



10.2 Sensor System MV

Application:

The MV is primarily used for viscosity measurements of medium viscosity liquids such as heavy oils, paints, varnishes, resins, emulsions etc. working in the medium shear rate range.

The coaxial cylinder sensor system consists of a MV cup used with three different rotors to provide different viscosity measuring ranges. MV1 and MV2 are both available in plastic (Phenolic thermoset) and in stainless steel 18/8. The plastic rotors are of low weight and allow higher rates of rotor acceleration. They are mechanically and chemically safe for temperatures up to 100°C.

This sensor system requires the temperature vessel connected to an accurate thermal liquid circulator.

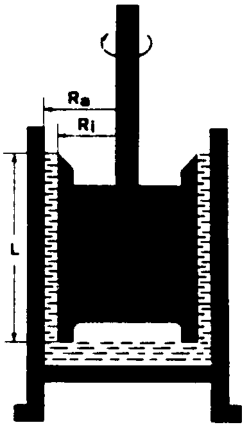
The top and the bottom surfaces of the rotors are recessed to minimize ”end effects”, i.e. their influence on torque. An air bubble is retained in the bottom recess, while the upper recess accommodates any excess sample.

The required amount of sample depends on the type of rotor used. For reference three ring marks are provided on the inside wall of the cup. The lower mark indicates the approximate sample volume required when the MV1 rotor is used in the MV cup. The middle mark applies to the MV2 and MV-DIN rotors while the upper mark applies to the MV3 rotor. During a test the liquid level of the sample should just overflow into the upper recess of the rotor. The liquid level must not rise above the upper rotor rim for more than 1 to 2 mm. Excess sample may be removed by sucking it back with a suitable syringe.

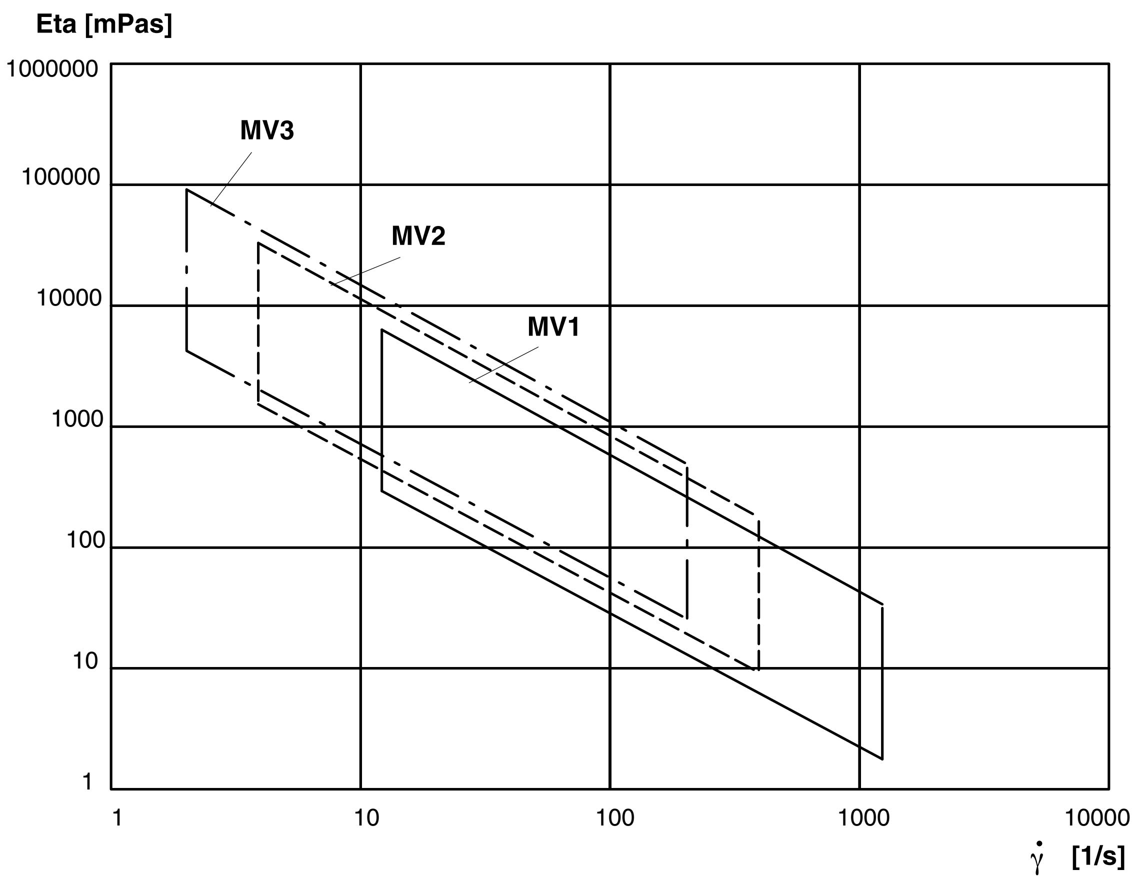
Decreasing the temperature of a sample which just fills the annular gap between cup and rotor to its upper rim will cause the sample volume to shrink. This gives erroneous viscosity values below the true viscosity level. When a sample has to be measured at various temperatures the sensor system should be overfilled. Alternative rotor: MV DIN

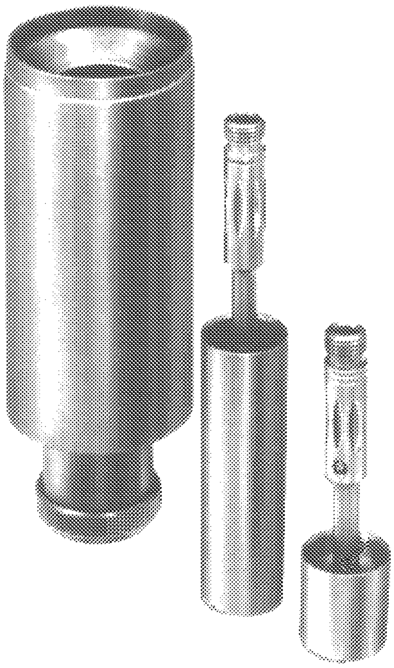
Cleaning:

To remove the bottom of the cup, first loosen the knurled screw and take out the cross-bar. (Order No. for the sealing ring: 807-0458)



Sensor System		MV1	MV2	MV3
System No.:		*3*	*4*	*5*
Inner Cylinder (Rotor)				
Radius R_i	(mm)	20.04	18.4	15.2
Height L	(mm)	60.0	60.0	60.0
Outer Cylinder (Cup)				
Radius R_a	(mm)	21.0	21.0	21.0
Radii Ratio	R_a/R_i	1.05	1.14	1.38
Gap Width	(mm)	0.96	2.6	5.8
Sample Volume V	(cm^3)	34.0	46.0	66.0
Temperature	(°C)		-30 / 100	
System Factors				
f (Pa/Ncm)		65.7	76.8	111.0
M (min/s)		2.340	0.900	0.440





10.3 Sensor System SV

Application:

The SV is primarily used for viscosity measurements of high viscosity liquids and pastes such as greases, cremes, ointments, plastisols etc. working in the low to medium shear rate range.

This coaxial cylinder sensor system consists of a SV cup with two rotors. It provides two different measuring ranges and requires a temperature vessel connected to an accurate thermal liquid circulator.

The top and the bottom surfaces of the rotors are recessed to minimize ”end effects”, i.e. their influence on torque. An air bubble is retained in the bottom recess, while the upper recess accommodates any excess sample.

The required amount of sample depends on the type of rotor used. For reference two ring marks are provided on the inside wall of the cup. The lower mark is a guide for the SV1 rotor, the upper for the SV2 rotor. The SV2 rotor requires less sample when the solid spacer is placed into the cup.

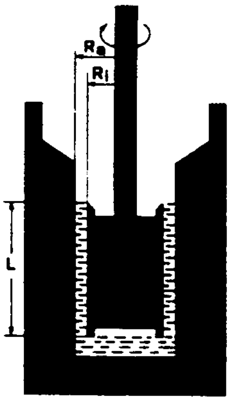
☞ **Note:** Decreasing the temperature of a sample which just fills the annular gap between cup and rotor to its upper rim will cause the sample volume to shrink. This gives erroneous viscosity values below the true viscosity level.

When a sample has to be measured at various temperatures, the sensor system should be overfilled to such an extent that the sample will be slightly above the rim of the rotor even at the lowest temperature. Alternative rotor: SV DIN

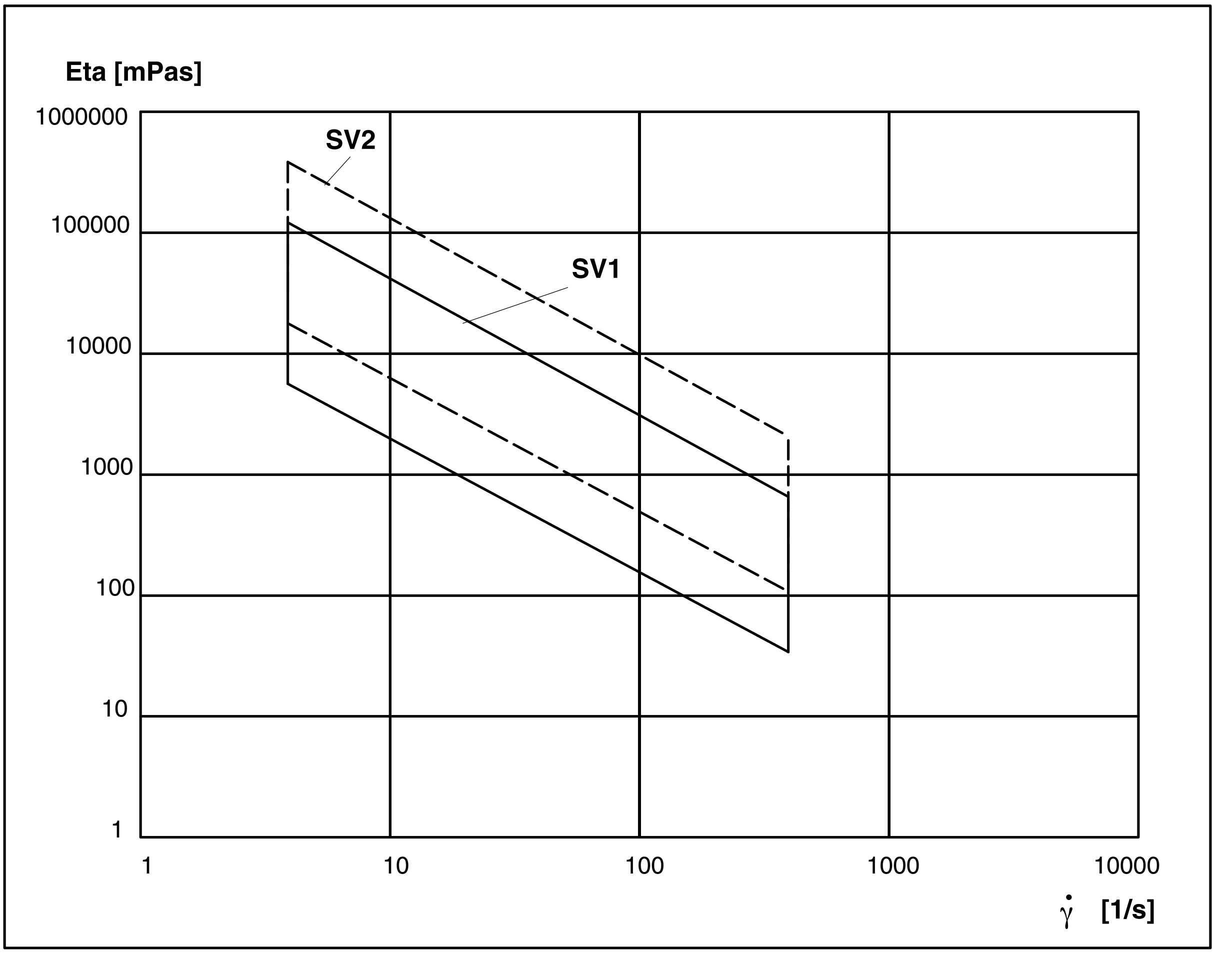
The special rotor SV2FL is designed as a star-shaped rotor and is used with the standard SV cup for the measurement of thixotropic samples.

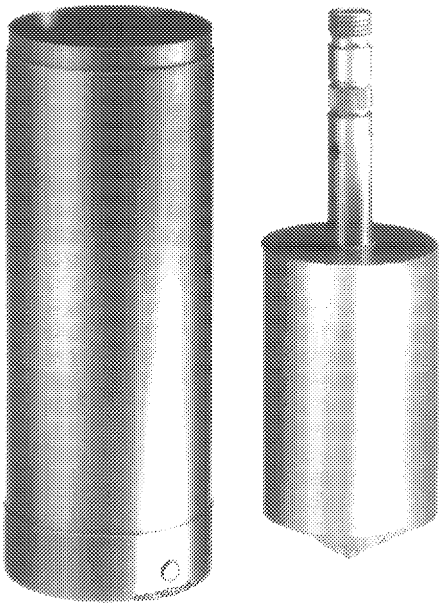
Cleaning:

The bottom of the cup can be unscrewed and removed.



Sensor System	**SV1**	**SV2**	**SV2FL**
System No.:	*6*	*7*	*–*
Inner Cylinder (Rotor) Radius R_i (mm) Height L (mm)	 10.1 61.4	 10.1 19.6	 10.1 19.6
Outer Cylinder (Cup) Radius R_a (mm)	 11.55	 11.55	 11.55
Radii Ratio R_a/R_i Gap Width (mm) Sample Volume V (cm^3) Temperature (°C)	1.14 1.45 12.0 	1.14 1.45 6.0 -30 / 100	– 1.45 10.0
System Factors f (Pa/Ncm) M (min/s)	 253.0 0.890	 768.0 0.890	 - -





10.4 Sensor System DIN 53019

Application:

The sensor system has been standardized for the viscosity measurements of water-based plastic dispersions (DIN 53 788), and of paints (DIN 53 214).

This coaxial cylinder sensor system consists of the cup MV and the rotor MV DIN or the cup SV and the rotor SV DIN.

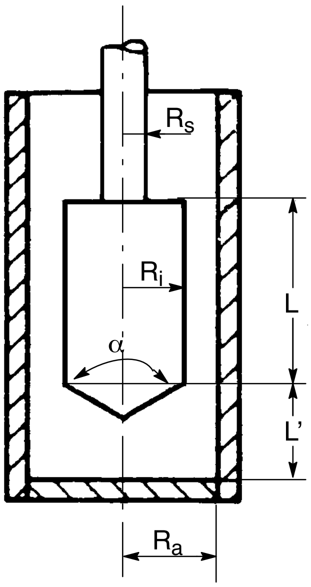
This sensor system meets the requirements of the German Standard DIN 53 019 for rotational viscometers.

DIN 53019 defines the characteristic geometrical ratios as follows:

$$\frac{R_a}{R_i} = 1,0847 \qquad \frac{R_s}{R_i} = \leq 0,3$$

$$\frac{L}{R_i} = 3 \qquad \frac{L'}{R_i} = 1$$

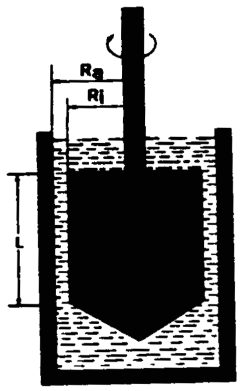
α = 120° (2,094 rad) ± 1°



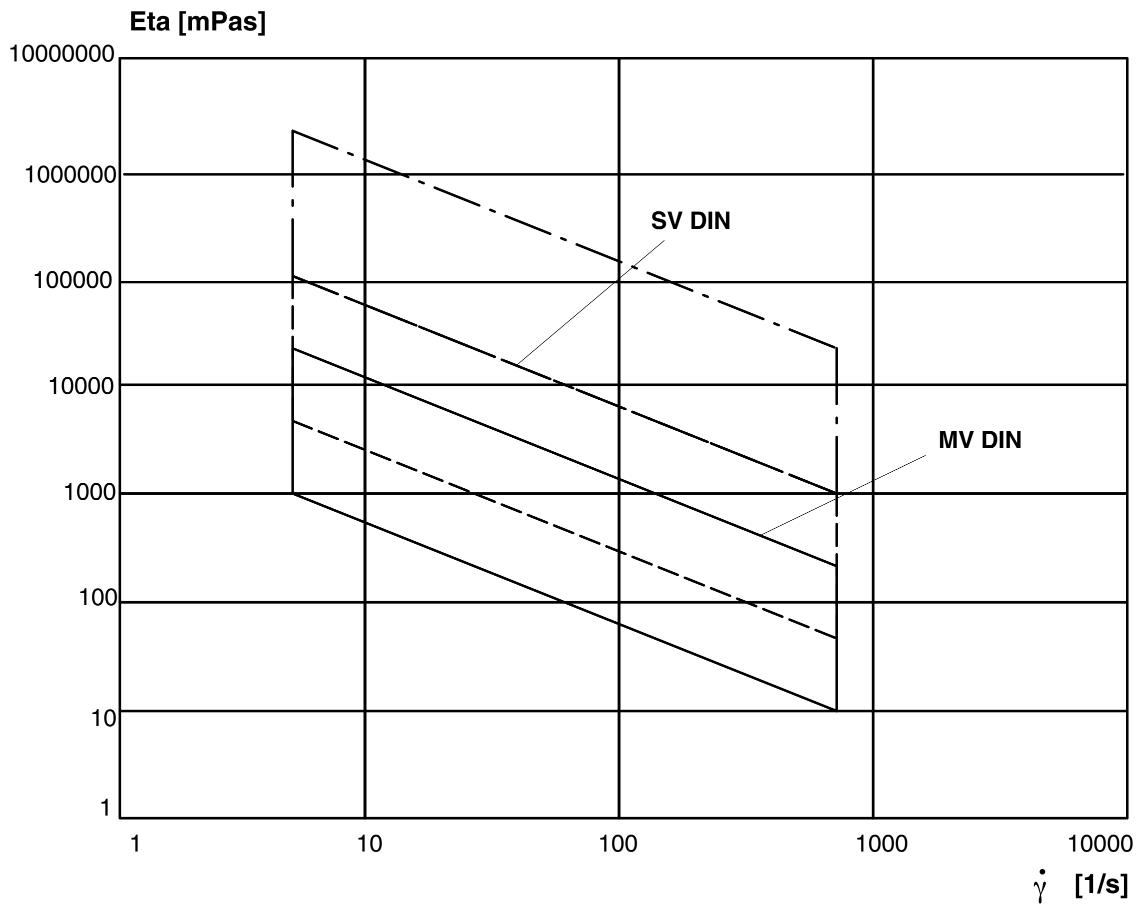
The sensor system should also be used when samples are subjected to programmed temperature increases and decreases. These temperature changes will only lead to the temperature related change of the sample volume in this sensor system. Its influence on the viscosity measurement will be small unless an insufficiently overfilled cup leads to a just partly filled annular gap when the temperature drop is very low.

Cleaning:

The bottom of the cup can be removed. (Order No. for the sealing ring of the MV Beaker: 807-0458)



Sensor System (DIN 53019)	MV–DIN	SV–DIN
System No.:	*1*	*2*
Inner Cylinder (Rotor) Radius R_i (mm) Height L (mm)	 19.36 58.08	 10.65 31.45
Outer Cylinder (Cup) Radius R_a (mm)	 21.0	 11.55
Radii Ratio R_a/R_i Gap Width (mm) Sample Volume V (cm^3) Temperature (°C)	1.0847 1.64 46.0 -30 / 100	1.0847 0.9 14.0
System Factors f (Pa/Ncm) M (min/s)	 61.4 1.290	 369.4 1.290

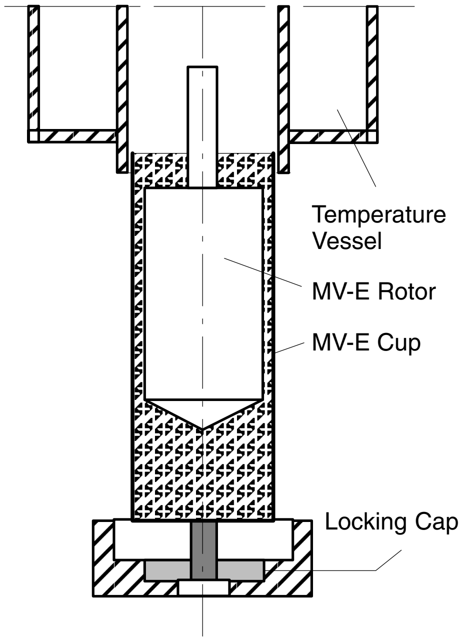


10.5 Disposable Sensor System DIN-E

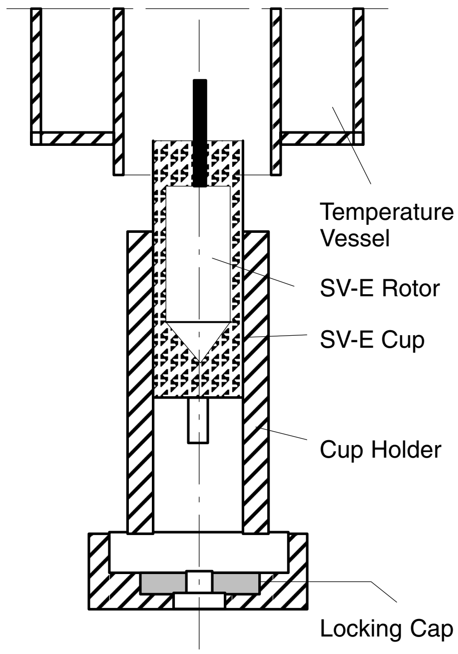
Application:

The disposable sensors are recommended when cleaning is a problem or when many tests with various samples have to be run. The disposable part is the measuring cup made of aluminum whereas the rotor remains the same; its shape makes cleaning easy.

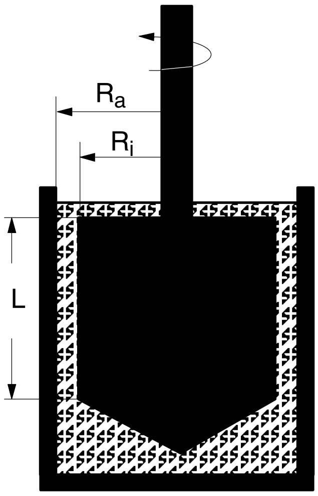
There are two disposable sensors available for use with the regular temperature vessel. One is the system MV-E DIN consisting of aluminum cups (disposable) and a stainless steel rotor MV-E. While the cups with the sample are discarded at the end of the test, the rotors are cleaned and used anew. The other system is the SV-E DIN system consisting of a stainless steel holder, aluminum cup (disposable) and a stainless steel rotor SV-E. Both systems have similar measuring ranges as the system MV/SV DIN. A special locking cap is required to hold the cups in the temperature vessel.



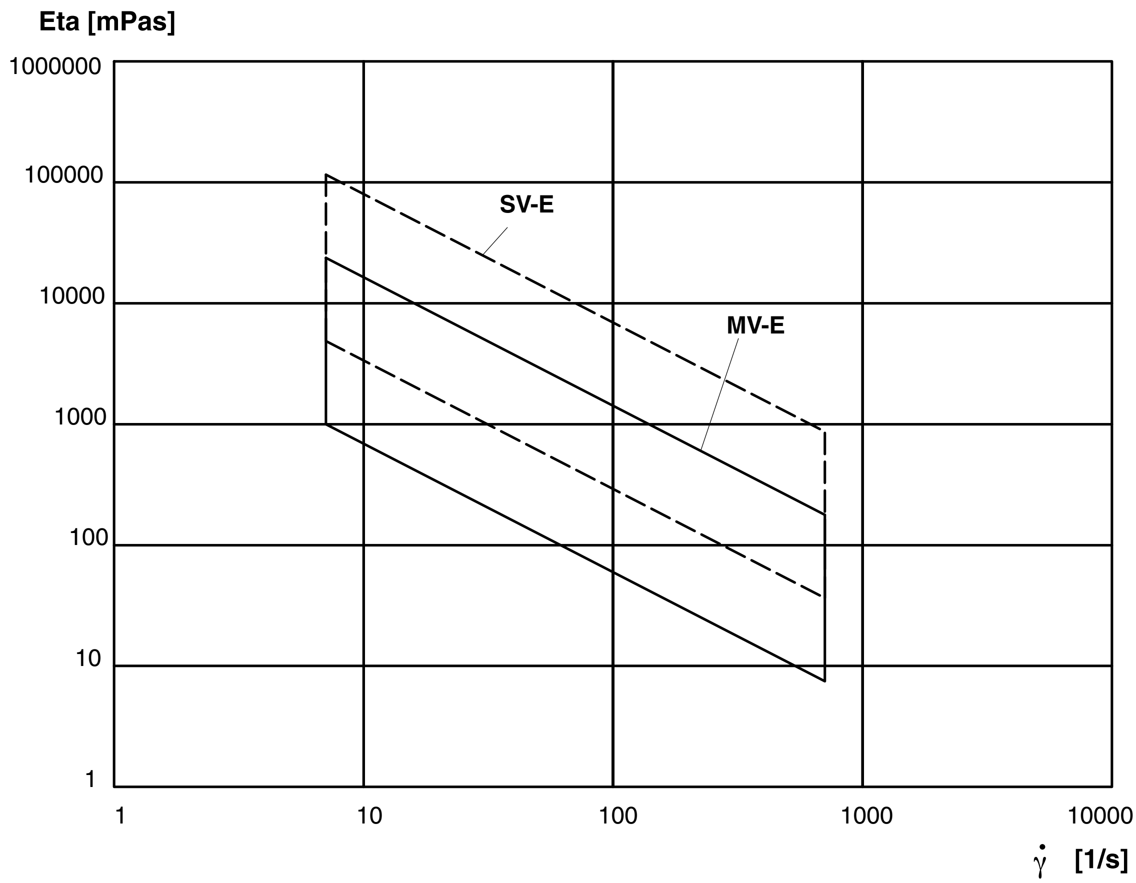
System MV-E



System SV-E



Sensor System	MV–E	SV–E
System No.:	*9*	*10*
Inner Cylinder (Rotor)		
Radius R_i (mm)	20.005	11.15
Height L (mm)	60.0	33.4
Outer Cylinder (Cup)		
Radius R_a (mm)	21.7	12.1
Radii Ratio R_a/R_i	1.0847	1.0847
Gap Width (mm)	1.7	0.95
Sample Volume V (cm^3)	65.0	17,0
Temperature (°C)	0/60	0/60
System Factors		
f (Pa/Ncm)	55.8	322.1
M (min/s)	1.290	1.290



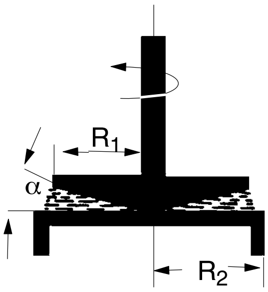
10.6 Sensor System PK ”Cone and Plate”

Application:

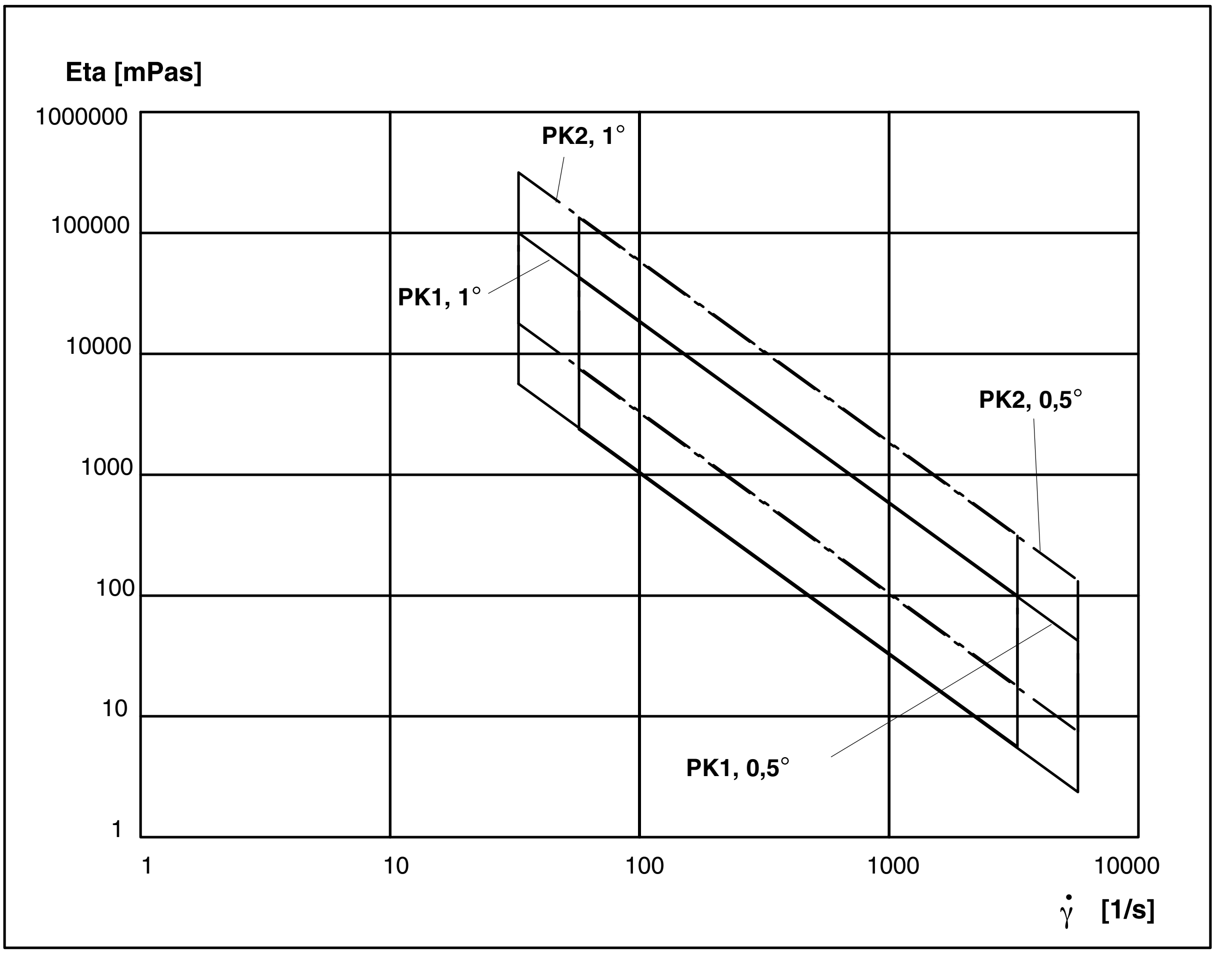
These sensor systems are primarily used when only small sample volumes are available, when testing at high shear rates is required and when cleaning after a test would pose tremendous problems in coaxial cylinder systems. Typical samples tested with ‘cone and plate’ are printing inks, resistor pastes, lipstick base materials, thermosetting polymers etc.

Samples are sheared in the angular gap between the cone and the plate. The design ensures that the shear rate is constant from the tip to the outer rim of the cone. Cones with cone angles of 0.5° and 1.0° are listed. Cones with angles above 4° are not recommendable from a rheological point of view.

The width of the cone radius determines the viscosity range which can be measured. A large radius allows lower viscosities to be measured. Small sample sizes and the effect of high shear causes the temperature of a sample to rise above the required test temperature due to frictional heat. In cases where this phenomenon becomes dominant, it is advisable to check some points on the flow curve at high shear rates. Tests are then run at fixed rotor speeds for short periods of time only. Time intervals are allowed between different speed values to allow the temperature of the sample to settle on the required level.



Sensor System PK	PK1 0.5°	PK1 1.0°	PK2 0.5°	PK2 1.0°
System No.:	*32*	*31*	*34*	*33*
Cone				
Radius R_1 (mm)	14	14	10	10
Angle α (10^2 rad)	0.873	1.74	0.873	1.74
Plate				
Radius R_2 (mm)	15	15	15	15
Sample Volume V (cm^3)	0,05	0,1	0,01	0,04
Temperatur (°C)	0/80	0/80	0/80	0/80
System Factors				
f (Pa/Ncm)	1740.0	1740.0	4775.0	4775.0
M (min/s)	12.000	6.000	12.000	6.000



10.7 Excerpt from the HAAKE PK100 D sensor system instruction manual

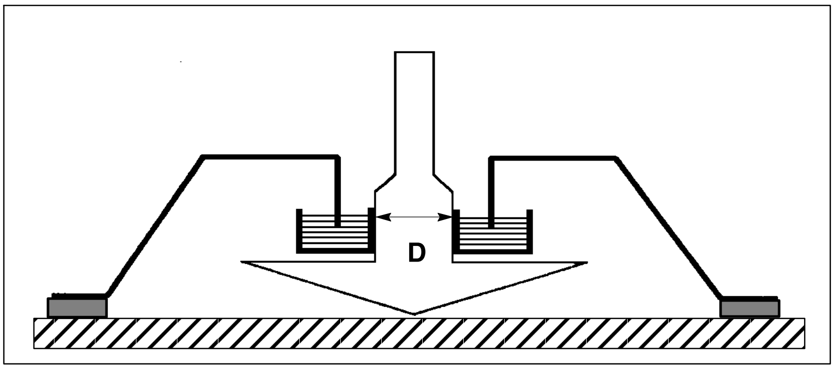
The cone-and-plate sensor system requires careful adjustment of the distance between cone-and-plate. Cones which are not truncated should only be employed for substances without solid particles. Substances containing particles and abrasives require truncated cones for measurement otherwise the cone and the plate might become damaged. In adjusting the distance Δh it is assumed that the theoretical tip of the cone is just touching the plate.

☞ **Note!** The size of the particles contained in the sample substance should not exceed 1/3 of the set distance Δh between cone-and-plate.

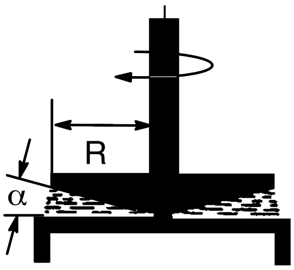
As the sample and the cone are heated via the lower plate, sufficient heating time (approx. 5 to 10 min.) should be allowed. It is advisable to determine the proper heating time by preliminary tests. While the substance is sheared one should wait until the viscosity reaches a constant value.

Solvent Trap

The use of a solvent trap for minimizing the effects of evaporation is advantageous. However, this feature can only be employed in combination with cones having a shaft diameter at the lower end of D = 12 mm.

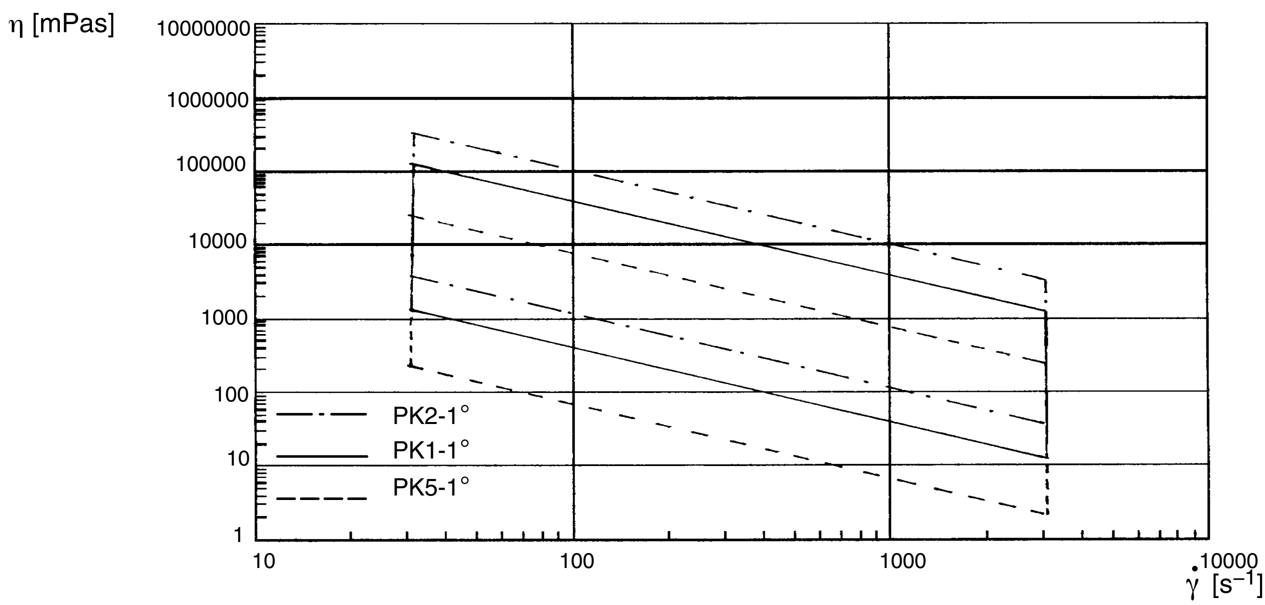


Calculation Factors HAAKE Viscotester® 550:



Sensor System PK	PK1 0.5°	PK1 1.0°	PK2 0.5°	PK2 1.0°	PK5 0.5°	PK5 1.0°	PK1* 2.0°	PK5* 2.0°
Cone								
Radius R [mm]	14	14	10	10	25	25	14	25
Cone Angle (10^{-2} rad)	0.873	1.74	0.873	1.74	0.873	1.74	3.49	3.49
Sample Volume [cm^3]	0.05	0.1	0.01	0.04	0.3	0.6	0.2	1.2
Calculation Factors								
f (Pa/Ncm)	1740.0	1740.0	4775.0	4775.0	306.0	306.0	1740.0	306.0
M (min/s) (approx.)	12	6	12	6	12	6	3	3
Gap Setting [µm]	0	0	0	0	0	0	70	100

* = truncated cone



10.8 Sensor System Parallel-Plates

The parallel-plate sensor system should be employed for substances containing particles exceeding 30μm in diameter.

☞ **Note!** The size of the particles contained in the sample substance should not exceed 1/3 of the set distance Δh between plate and plate.

The parallel-plate sensor system requires careful adjustment of the plate as the shear rate is dependent on the set distance Δh between the two plates.

The shear rate factor at the edge of the plate is calculated for the VT550 as follows:

$$M = \frac{\pi \cdot R}{3 \cdot \Delta h}$$

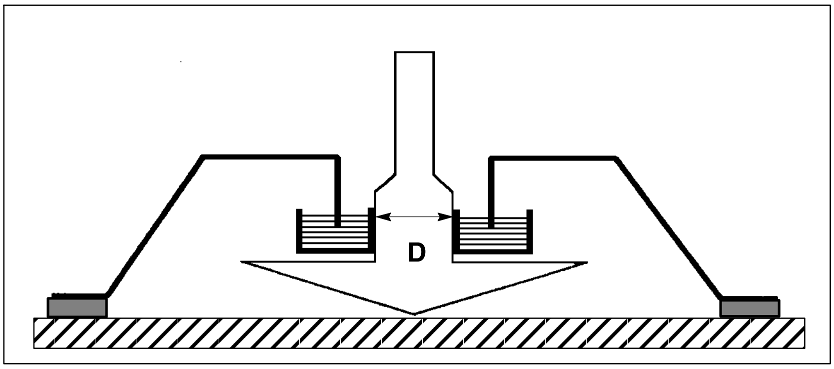
Example PQ2:

$R = 10\ mm;\ \Delta h = 1\ mm$

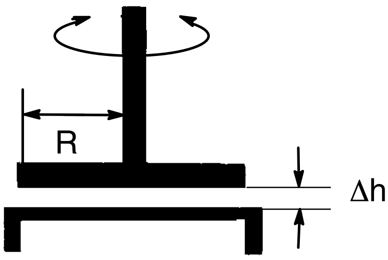
$$M = \frac{\pi \cdot 10}{3 \cdot 1} = 1.05\ (min/s)$$

Solvent Trap

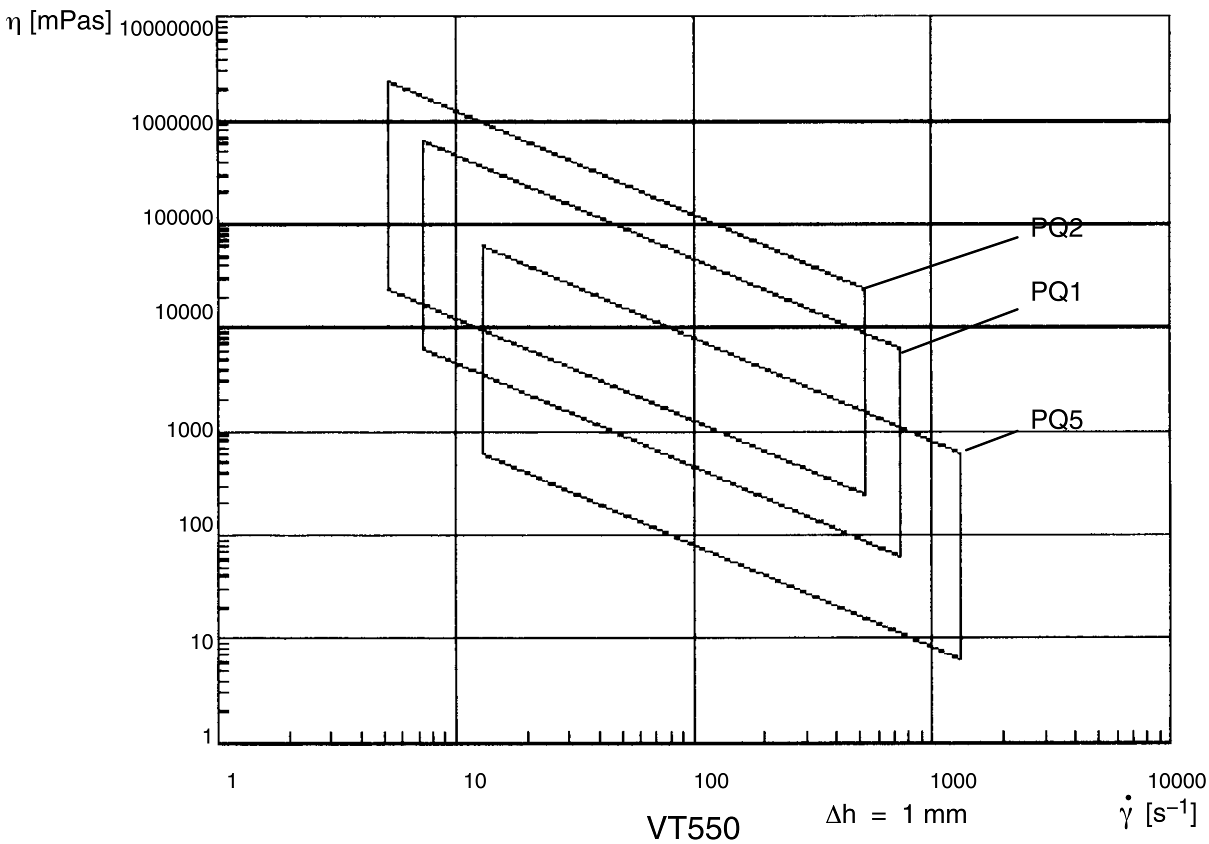
The use of a solvent trap for minimizing the effects of evaporation is advantageous. However, this feature can only be employed in combination with plates having a shaft diameter at the lower end of D = 12 mm.

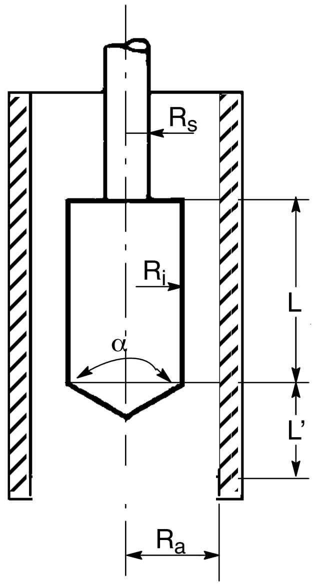


Calculation Factors HAAKE Viscotester® 550:



Sensor System PQ	PQ1	PQ2	PQ5
Cone Radius R [mm]	14	10	25
Sample Volume V · Δh* [cm^3]	0.7	0.4	2
Calculation Factors VT500/501: f (Pa/Ncm) M/ Δh*(min/s)	2320.0 1.470	6366.0 1.050	408.0 2.620





10.9 Immersion sensor system

Application:

The sensor system has been standardized for the viscosity measurements of water-based plastic dispersions (DIN 53 019), paints (DIN 53 214) and tensides (DIN 53 921).

It consists of an immersion tube and a rotor, i.e. the cup and the DIN rotor.

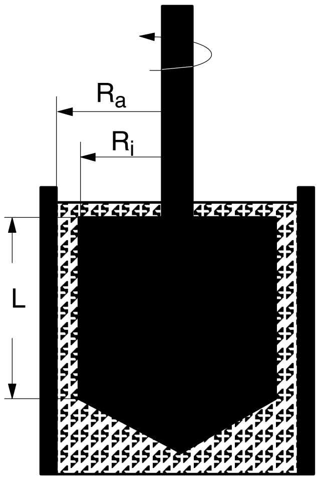
This sensor system meets the requirements of the German Standard DIN 53 019 for rotational viscometers which defines the characteristic geometrical ratios as follows:

$$\frac{R_a}{R_i} = 1,0847 \qquad \frac{R_s}{R_i} = \leq 0,3$$

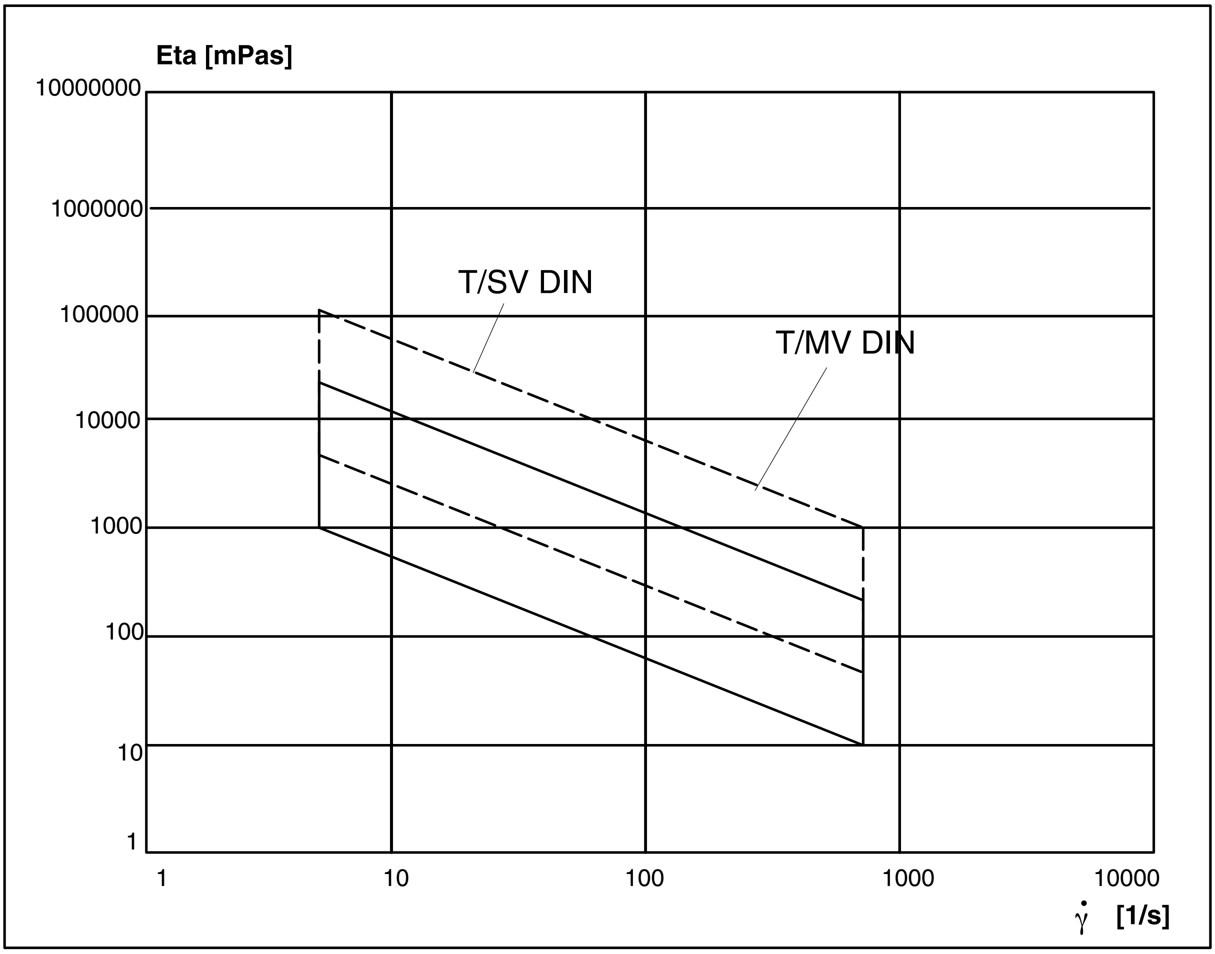
$$\frac{L}{R_i} = 3 \qquad \frac{L'}{R_i} = 1$$

α = 120° (2,094 rad) ± 1°

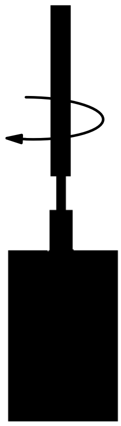
This sensor system was especially standardized in order to provide measuring values which are independent of handling- and instrument-specific variables.



Sensor system (DIN 53019)	**T/MV DIN**	**T/SV DIN**
System No.:	*1*	*2*
Inner cylinder Radius R_i (mm) Height L (mm)	 19.36 58.08	 10.65 31.45
Outer cylinder Radius R_a (mm)	 21.0	 11.55
Radii ratio Gap width (mm) Sample volume (cm^3) Temperature (°C)	1.0847 1.64 – 100	1.0847 0.9 – 100
System factors f (Pa/Ncm) M (min/s)	 61.4 1.29	 369.4 1.29



10.10 Immersion Sensor System E



Application:

The immersion sensor systems are cylindrical rotors connected to the HAAKE Viscotester® 550 by means of an adapter.

They are used for viscosity measurements in open vessels like beakers, buckets, tins etc. to do simple and quick measurements of non-homogenous or particle containing samples.

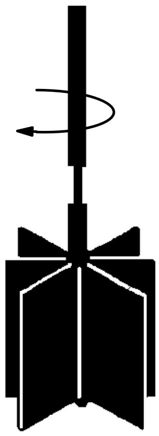
In order to avoid wall influences, the interior diameter of the vessel should be at least four times larger than the rotor diameter. If the measurements have to be made with small vessels, the wall influence can be compensated by calibration of the rotor/vessel combination with Newtonian oils. High temperature measurements require an increased distance between the rotor and the VT in order to prevent damage to the VT. Approximately 20 cm is sufficient to carry out tests at high temperature. The temperature range is from -50 to 200°C.

The immersion sensor systems are unsuitable for the determination of the absolute viscosity of non-Newtonian samples!

Sensor System	E3	E30	E100	E500	E1000
System No.:	*15*	*16*	*17*	*18*	*19*
Rotor Radius (mm) Height (mm)	 25 116	 12 50.5	 8 34.5	 5 18	 3.5 17.5
Temperature (°C)	-30/200				
System Factors f (Pa/Ncm) M (min/s)	 8.5 0.100	 85.4 0.100	 285.0 0.100	 1424.5 0.100	 2850.0 0.100

10.11 Immersion Sensor System FL

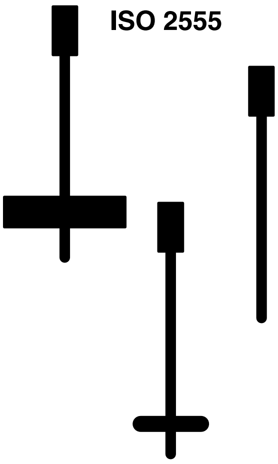
Application:



Star shaped rotors (3 types) - they can be successfully employed to measure cremes, gels etc. which are often highly thixotropic losing their structure even before the test just by forcing them into small annular gaps. Cutting into the sample with the star-shaped rotor will leave the structure of the sample undamaged.

Yield points are determined from the peaks of a τ/time-curve: When the yield point is surpassed, the rotor ”cuts a hole” into the sample and the test is finished.

Sensor System	**FL10**	**FL100**	**FL1000**
System No.:	*20*	*21*	*22*
Rotor Radius (mm) Height (mm)	 20 60	 11 16	 5 8.8
Temperature (°C)	–30 / 200		
System Factors f (Pa/Ncm) M (min/s)	 54.0 0.209	 564.0 0.209	 5246.8 0.209



10.12 Sensor System IS0 2555

ISO 2555 and many ASTM-standards define disk-shaped rotors for viscosity measurements. The resulting measurement values are relative data, but comparable on identical measuring conditions, i.e results which will be obtained with the same speed and the same ISO rotor.

With the IS0-Adapter (Order No.: 222-1140) these rotors may be connected to the HAAKE Viscotester. It is recommended to use the ISO 2555 speed range. (Speed row 6) page 24.

System	**B1**	**B2**	**B3**	**B4**	**B5**	**B6**	**B7**
System No.:	*23*	*24*	*25*	*26*	*27*	*28*	*29*
Rotor							
Radius (mm)	28.13	23.5	17.35	13.65	10.55	7.3	1.6
Length (mm)	22.5	1.65	1.65	1.65	1.65	1.65	50.4
Temp. (°C)	-30 /200						
System Factors							
f (Pa/Ncm)	14.3	57.0	143.0	285.5	570.0	1430.0	5715.0
M (min/s)	0.100	0.100	0.100	0.100	0.100	0.100	0.100

10.13 Temperature control vessel

The temperature control vessel has three functions:

a) It connects the sensor system to the measuring drive
b) It centers the cup and rotor
c) It temperature controls the test substance.

The temperature control vessel is attached to the HAAKE Viscotester® 550 via two knurled-headed screws. It can be rotated around the guide rod so that the hose connections are in the correct direction.

The temperature control vessel has two hose connection nozzles (thread M10 x 1.5) for connecting a Thermo Fisher Scientific heating circulator. The flow direction is indicated by the arrows.

A small protective cover is supplied with the temperature control vessel. It can be used to cover up the sensor system (e.g. for substances which evaporate easily or at high temperatures).

The protective cover should be attached to the rotor before it is fixed to the measuring shaft. Moisten the outside of the cup (e.g. with glycerine) to optimize the heat transfer from the temperature control vessel to the cup. The temperature can be measured using a Pt100 sensor (Order no. 333–0427).

The following hoses can be used according to the temperature range and heat transfer liquid used:

PVC hose, water	up to 60 °C
Perbunan hose,	–40 °C to 100 °C
Silicone hose (not with silicone oil)	–30 °C to 100 °C
Stainless steel hose (all liquids)	–30 °C to 100 °C

! **All hose connections must be secured using hose clamps!**
The metal hose requires no extra securing devices.

! **The temperature control vessel should only be heated up to 100°C – higher temperatures will damage the HAAKE Viscotester® 550!**

DANGER: There is a risk of burns!

11. Measuring

11.1 Fundamental principles

This manual is not intended and should not be used as a substitute for specialized literature on viscometry. A few basic phenomena have been summarized below in order to enable a better understanding of the measuring process:

11.1.1 Newtonian substances

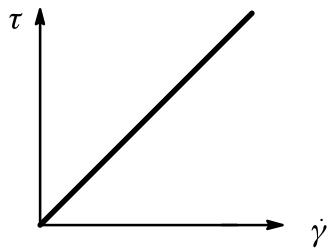
Behavior: Their viscosity does not change when the HAAKE Viscotester® 550 speed is altered.

Explanation: Viscosity is a genuine material constant.
The substance is fully characterized by one single numerical figure.

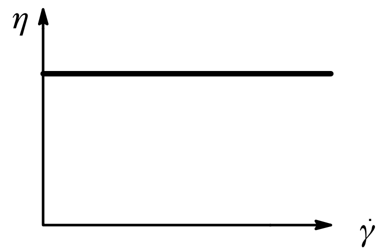
! This value changes however with temperature!

Mathematically: $\eta = \text{const.}$

Graphic Representation:



the “flow curve” is a straight line from the origin



the viscosity curve is a parallel line to the $\dot{\gamma}$ axis

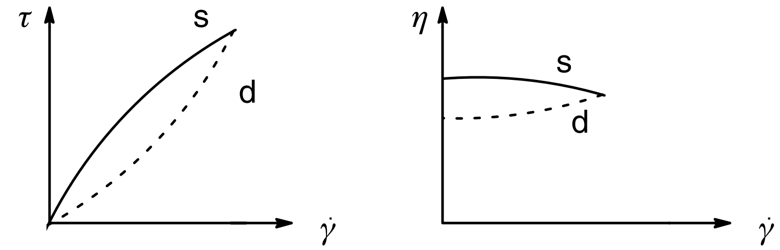
11.1.2 Non-Newtonian substances

Behavior: Their viscosity changes when the HAAKE Viscotester® 550 speed is altered.

Explanation: Viscosity is *not* a genuine material constant.
The substance is fully characterized by one single numerical figure, but rather by a number of value pairs of $\tau / \dot{\gamma}$.

Mathematically: $\eta = \eta\ (\dot{\gamma})$

Graphic Representation:



These substances are also called structurally viscose, whereby...

curve s is pseudoplastic,	η decreases as n increases (common),
and curve is d dilatant,	η increases as n increases (rare).

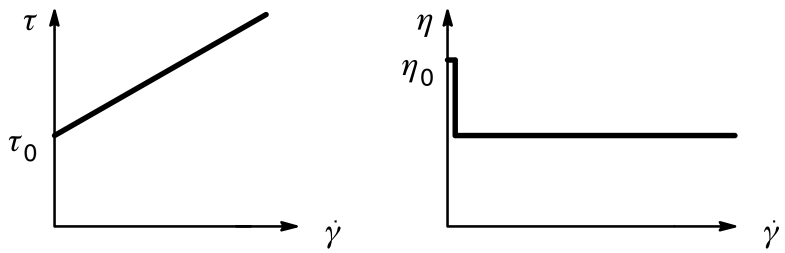
11.1.3 Substances with yield values

Behavior: Their viscosity is (virtually) infinite at speeds close to zero.

Explanation: The substance has a yield value: Flow does not start until the shear stress has exceeded a minimum value (τ_0); it behaves like a solid body below τ_0.

Mathematically: $\eta = \eta_0 + \eta\,(\dot{\gamma})$

Graphic Representation:



Substance with yield value and Newtonian flow behavior = Bingham flow behavior.

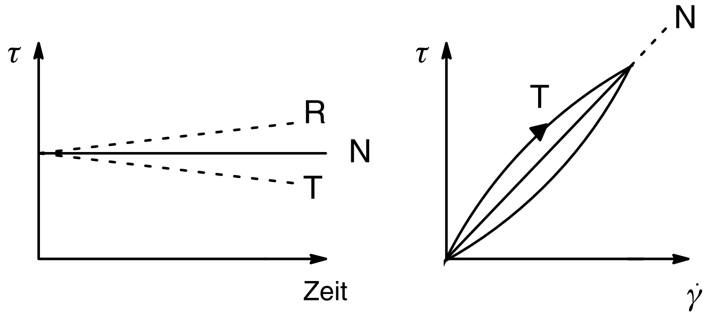
11.1.4 Substances with “time-dependent” behavior

Behavior: Their viscosity changes at a constant speed as time elapses at this speed.

Cause: The inner structures are destroyed due to the influence of speed (“thixotropic” behavior T: common) or reinforced (“rheopectic” behavior R: rare).

Mathematically: $\eta = \eta\ (\dot{\gamma}, t)$

Graphic Representation:



N is a “Newtonian substance”, T exhibits thixotropic behavior and R rheopectic behavior. The degree of alteration depends on the speed. This relationship is seen at it’s clearest in the “Flow curve (τ-$\dot{\gamma}$ diagram) at increasing/decreasing speed”: contrary to the Newtonian substances (N), the upwards and downwards curves with time-dependent viscosity are not identical: they show an hysteresis area between the two curves. The area is a measure of this effect. Thixotropic behavior is signified by the curve running in the direction of the arrow (larger η outwards than on the return) and rheopectic behavior is characterized by a curve running in the opposite direction.

Conclusion:

1. The test procedure described in 9.1.4 (diagram on right) “Increasing /decreasing speed program” is the most common method. All characteristics described under 9.1.1 to 9.1.3 are thus automatically recognized.
2. The characteristic described in 9.1.3 requires a more exact test procedure: Especially low speeds are required in order to accurately establish the point of the onset of flow.

SPECIAL ROUTINES HAVE BEEN CREATED FOR THIS PURPOSE IN THE HAAKE VT550. THESE ARE PROCEDURES (1) AND (2) AND ARE DESIGNATED ”CD-MODE”
(“Controlled Deformation”).

11.2 Normal or manual mode

In this mode…

- the test parameters are entered via the keypad of the HAAKE Viscotester® 550
- the test results are shown on the display and/or sent to the the printer.

This differs from the computer mode.

The measuring procedure can be described by the following steps:

1. Fix the sensor system with the rotor to the HAAKE Viscotester® 550
2. Zero point correction
3. Fill the test substance into the cup
4. **Temperature control vessel:** Insert cup into the temperature control vessel and secure **or**
   Plate/Cone: attach plate to cone.
5. **Immersion tube:** Lower HAAKE Viscotester® 550 and immerse the immersion tube into the measuring vessel
   or
   immersion sensor system E: Lower HAAKE Viscotester® 550 and immerse the rotor into the measuring vessel
6. Temperature control the substance in the sensor system
7. Enter the sensor system number via the HAAKE Viscotester® 550 keypad
8. Select procedure
9. Select speed program
10. Read off temperature and compare with set value
11. Select speed level (e.g. 1)
12. Start motor [Stop Start]
13. Switch display to viscosity (η) [D] > Pas
14. Read off and note displayed value

15 Set display to shear rate ($\dot{\gamma}$)
D > 1/s

16 Read off and note displayed value

17 Select speed level (e.g. 2)

18 Set display to viscosity (η) D > Pas

19 Read off and note displayed value

20 Set display to shear rate ($\dot{\gamma}$)
D > 1/s

21 Read off and note displayed value

22 etc.

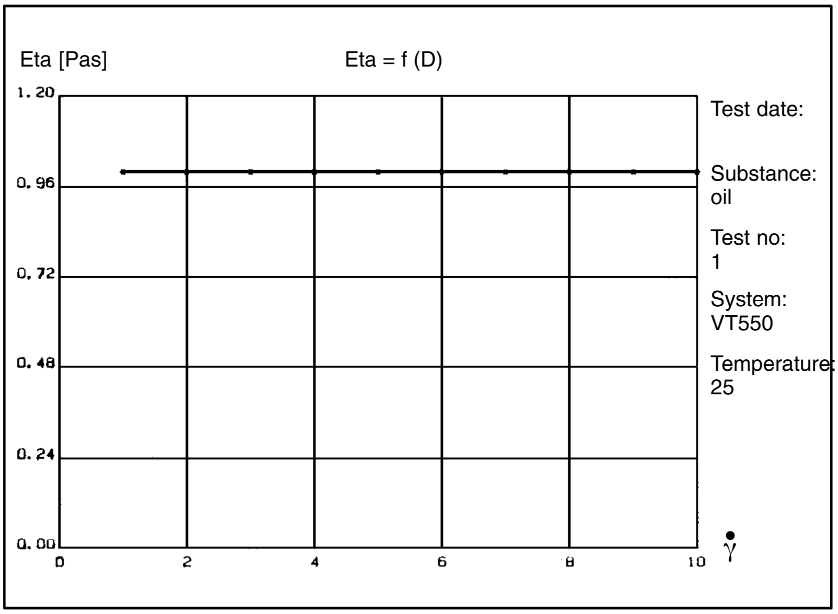
23 Stop motor Stop Start

24 Repeat measurement if necessary or continue with next test

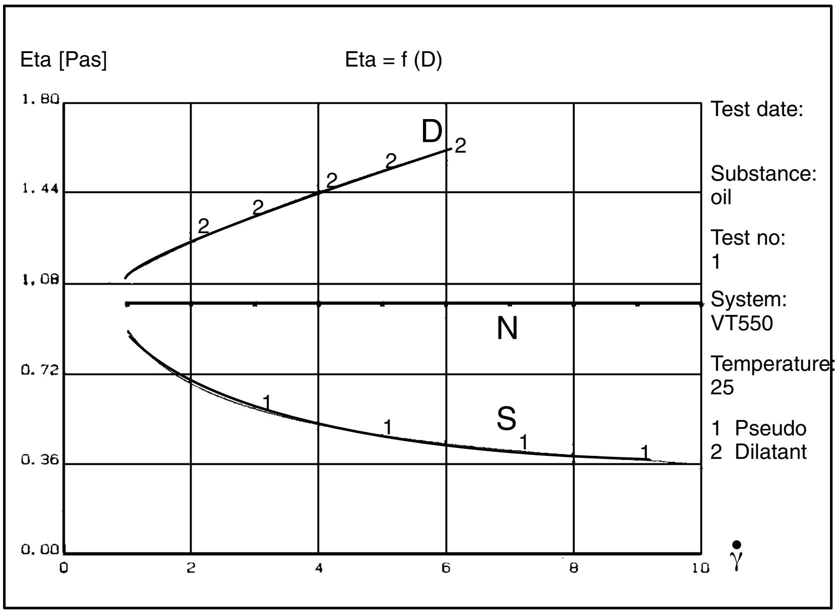
Example of results in table-form

Level	η(mPas)	$\dot{\gamma}$ (s^{-1})	T (°C)
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			

After all measuring points have been evaluated in table-form, the measuring results can be display in a graph.



This viscosity curve gives an indication of the flow behavior of the test substance. The viscosity curve should normally look something like the one of the curves shown below.



(N) Newtonian flow behavior
(S) Structurally viscose flow behavior
(D) Dilatant flow behavior

11.2.1 Measuring with system no. 0: The system factors f and M are known but not stored

In this case the built-in microprocessor is not able to carry out any calculations and thus cannot display:

- shear stress,
- shear rate,
- viscosity

- Torque Md (Ncm) is shown instead of shear stress τ (Pa) in the display
- Speed n (min^{-1}) is shown instead of shear rate $\dot{\gamma}$ (s^{-1}) in the display.

The measuring procedure itself follows the sequence outlined under 9.1.1 and the measuring values are entered in the table manually (as shown in the following example).

Example: Sensor system MV1P with f = 65.7 and M = 2

Speed level	Measuring value		Calculation value		
	n(min^{-1})	Md(Ncm)	$\dot{\gamma}$ (1/s)	τ(Pa)	η(Pa·s)
1	5.0	0.56	10.0	33	3.3
2	8.3	0.7	16.6	46	2.77
3	13.9	1.0	27.8	66	2.37
4	23.2	1.2	46.4	79	1.7
5	45.3	1.4	90.6	92	1.01
6	64.5	1.5	129	99	0.77
7	107.8	1.6	215.6	105	0.49

The measuring value pairs Md (torque) and n (speed) in the table are then manually converted, i.e.:

shear stress $\tau = Md \cdot f$ [Pa]

shear rate $\dot{\gamma} = n \cdot M$ [s^{-1}]

viscosity $\eta = \tau / \dot{\gamma}$ [Pa · s]

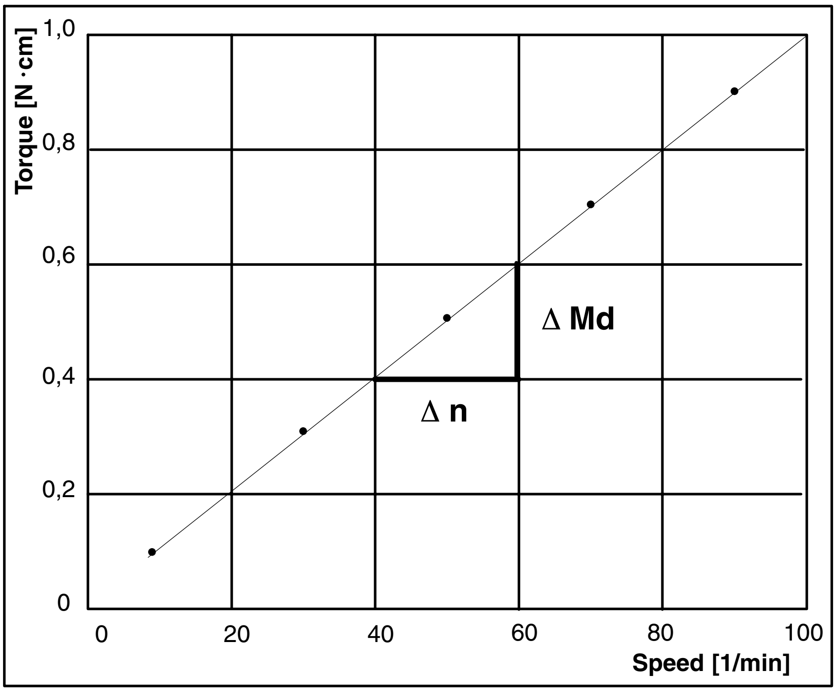
The results of these calculations are also incorporated within the table. The results can be represented graphically for renewed evaluation.

11.2.2 Measuring with system no. 0: The system factors f and M are not (or no longer) known but the substance viscosity is known

This measurement serves the purpose of determining the system factors. This process is more commonly known as "calibration".

The measuring process exactly follows the procedure outlined above and a substance with a known viscosity is used (e.g. normal oil).

The measuring results gained according to chapter 9.1.1 are graphically displayed as torque Md = function (speed):



The connection of the measuring points must result in a straight line which should intersect the zero point.

In the example:

The following values can be gathered from the "calibration line":

for n = 20 : Md = 0.25
for n = 60 : Md = 0.75

so $\Delta n = 60 - 20 = 40$ so $\Delta Md = 0.75 - 0.25 = 0.5$

Thus $\Delta n / \Delta Md = 40/0.5 = 80$

The equations from chapter 9.1 are summarized and suitably adapted for calculating f and M.

$$\eta = \frac{\tau}{\dot{\gamma}} = \frac{f \cdot Md}{M \cdot n} = \left(\frac{f}{M}\right) \cdot \left(\frac{Md}{n}\right)$$

or $$\frac{f}{M} = \eta \cdot \frac{n}{Md} \mathrel{\hat{=}} \eta \cdot \left(\frac{\Delta n}{\Delta Md}\right)$$

η is known, $\left(\frac{\Delta n}{\Delta Md}\right)$ is taken from the graphic representation – only the relationship of “f to M” is available as the result.

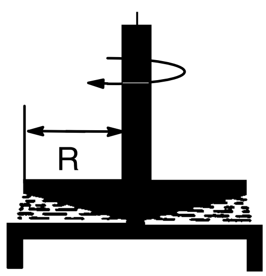
f or M can be calculated from the geometrical data depending on the type of sensor system.

The equations are now as follows:

$$f = M_{ber.} \cdot \eta \cdot \left(\frac{\Delta n}{\Delta Md}\right)$$

or $$M = \frac{f_{ber.}}{\eta \cdot \left(\frac{\Delta n}{\Delta Md}\right)}$$

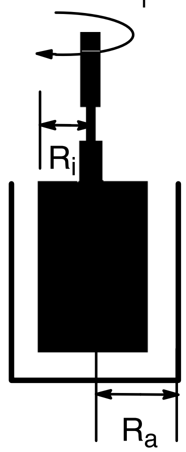
a) Plate-cone



$$f_{cor} = \frac{0.03}{2 \cdot \pi \cdot R^3}$$

$$M_{cor} = \frac{6}{\alpha};\ \alpha\ (^\circ)$$

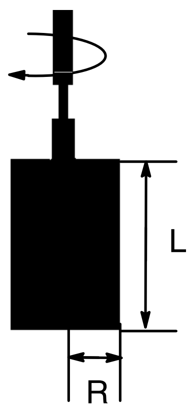
b) Cylinder sensor system ($\frac{R_a}{R_i} \leq 1,2$)



$$f_{cor} = \frac{0.01}{2 \cdot \pi \cdot L \cdot R_i^{\,2}}$$

$$M_{cor} = \frac{\pi}{15} \cdot \frac{R_a^{\,2}}{R_a^{\,2} - R_i^{\,2}}$$

c) Immersion sensor system E



$$f_{cor} = \frac{0.01}{2 \cdot \pi \cdot L \cdot R^2} \quad \text{(E)}$$

$$M_{cor} = \frac{0,01}{4\,\pi\,R^3\left(\frac{L}{2R} + \frac{1}{3}\right)} \quad \text{(FL)}$$

11.3 Computer mode

The HAAKE Viscotester® 550 is connected to the PC via the RS232 serial interface (see chapter 5, page 14)

The application software runs on the PC; it takes over all control, measuring and evaluation functions. Manual operation is locked. Only the STOP function is still functional.

11.3.1 PC requirements

- IBM or IBM–compatible computer with processor Pentium IV or later
- minimum 256 MB RAM
- CD−ROM drive
- Graphics adapter, resolution 1024 x 768
- Serial interface (RS232C) for measuring instrument
- Microsoft® or PS/2®mouse

These minimum requirements should be exceeded for optimum performance.

11.3.2 Communication HAAKE Viscotester® 550 – PC

All commands sent to the HAAKE Viscotester® 550 which require a return answer begin with **R** (Read). Simple control commands without a return answer begin with a **W** (Write) and, if executed without a fault, are confirmed with an end sign (‘$’).

Faulty commands are answered with the fault code “**Fxxx**” (see chapter 10.6).

The execution time of all commands (apart from measuring time commands) which are executed correctly is saved. This saved time (time marker) can be read directly after execution by entering the command “**R ZZ**”.

11.4 Software HAAKE RheoWin

The HAAKE application software for the HAAKE Viscotester® 550 is supplied with a separate instruction manual.

11.5 Operating software OS550

The HAAKE operating software OS550 for the HAAKE Viscotester® 550 is supplied with a separate instruction manual.

12. RS232C Interface

Communication via the interface:

Abbreviations in the following description:
x = number, v = plus/minus, z = letter.

12.1 Control commands with the application software

Command	Description	Reply
R V1	Read version of operating software	'V1 1.2–5/94'
W ZI	Set measuring time counter to zero and re-start	'$'
R MZ	Read current measuring time	'MZxxxxxx.xxx' Unit s
R ZZ	Read time marker (time since last command)	'ZZxxxxxx.xxx' Unit s
R DM	Read torque	'DMvx.xxxxxxEvxx' Unit Nm
R WG	Read angular velocity	'WGvxxx.xxx' Unit rad/s
R ST	Read temperature	'STvxxx.xx' Unit °C
W ME	Set VT550 to external operation (PC). The VT550 is run at the speed specified in the CR command.	'$'
W CRvxxx.xxx	Set speed in the external mode. (In units of rad/s). The VT550 must have previously been set to external mode. The motor is activated. The measuring time is given as the reply.	'MZxxxxxx.xxx' Unit s
W MS	Stop motor in external mode. The motor is switched off.	'$'
W MI	Switch VT550 to internal mode. Speed setting is carried out at the VT550.	'$'

12.2 Commands: System factors

The desired system number (see chapter 7.3) is first specified then the f and M factors are sent/read for this system number

The numerical values are limited: f factor = 6553.5 max.,
M factor = 65.535 max.

☞ Only the factors 30 to 44 can be user-assigned; all factors can however be read.

Command	Description	Reply
W FN xx	Specify system number	'$'
W FF xxxx.x	Specify f factor for system number	'$'
W MF xx.xxx	Specify M factor for system number	'$'
R FF	Read f factor for system number	'FFxxxx.x'
R MF	Read M factor for system number	'MFxx.xxx'

12.3 Commands: Speeds/speed programs

The numbers of the desired speed program (see chapter 7.3) are first specified and subsequently all ten speed values for this speed program are sent/read. The set value is speed n in units of min^{-1}.

The numerical values are limited: $0.1 \leq n \leq 800.0\ min^{-1}$

☞ Only the speed programs 7 to 10 can be altered; all speed programs can however be read.

Command	Description	Reply
W DR xx	Specify speed program	'$'
W DZ xxx.x	Specify speed for speed program. This command must be entered ten times; once for each of the speed levels 1 to 10.	'$'
R DZ	Read speed for speed program. This command must be entered ten times; once for each of the speed levels 1 to 10.	'DZvxxx.x' Unit l/min

12.4 Commands: Procedure parameters

The number of the procedure (1 to 10) to be programmed is first entered. Subsequently a whole data block (16 commands) is sent to the HAAKE Viscotester® 550. The desired protocol language is sent separately and is valid for all procedures.

The data block consists of:

- shear rate $\dot{\gamma}_1$ to $\dot{\gamma}_5$ for speeds
  in the range of $0 \leq n \leq 800$ [min^{-1}]
- measuring time per segment t_1 to t_5
  in the range of $0 \leq t \leq 36000$ [s]
- number of measuring points per segment Mp_1 to Mp_5
  in the range of $0 \leq Mp \leq 100$
- System number

Please note:

1. The command sequence must be observed. Only complete data blocks can be sent.
2. The total of all measuring points in all segments must be ≤ 100.
3. The set value “shear rate” is converted to “speed” and then sent to the HAAKE Viscotester® 550.

Command	Description	Reply
W PN xx	Set procedure number	’$’
W PD xxxxx.x	Set the procedure data for the procedure number. This command must be repeated sixteen times (for order see above).	’$’
R PD	Read the procedure data for the procedure number. This command must be repeated sixteen times (for order see above).	’PDxxxxx.x’
W SP xx	Set the protocol language xx = 1 German xx = 2 English xx = 3 French	’$’
R SP	Read the protocol language	’SPxx’ For code, see above

12.5 Commands: Calibrating the HAAKE Viscotester$^{®}$ 550

The entire calibration of the HAAKE Viscotester$^{®}$ 550 is carried out using software commands via the interface. Calibration parameters are evaluated during calibration which are then permanently stored in the EPROM of the HAAKE Viscotester$^{®}$ 550. These values can be read via the OS550 operating software.

1. Temperature calibration

A known and exact reference resistor which is attached to the Pt100 socket of the HAAKE Viscotester$^{®}$ 550 is used for calibration. The calibration resistance is entered. The correction factor is calculated from the difference between the resistance set value and the measured actual value. An offset value can be entered via the keypad or programmed and read off using the OS550 operating software for adapting a temperature sensor.

2. Torque calibration

The HAAKE Viscotester$^{®}$ 550 torque signal should be calibrated at at least two points (zero point and the maximum value for 100% torque) using a suitable weight calibration device. The correction factor for calibration is calculated from the difference between the manually calculated torque set value and the measured actual value.

See table overleaf.

Command	Description	Reply
W C1	Calibrate temperature the reference value must have been previously entered	’$’
W C2 vxxx.x	Set temperature offset	’$’
W C3 xxx.x	Set resistance reference value in units of Ohm	’$’
R CT	Read temperature calibration value	’CTx.xxxx’
R CO	Read temperature offset value	’COvxxx.x’
W C4	Initialize torque calibration Current calibration value is reset. Motor is activated i.e. started at speed zero (for stopping). Display switches to torque.	’$’
W C5	Torque zero point calibration Current torque (with and without attached weight) is stored as the zero point for calibration.	’$’
W C6	Calibrate maximum torque value The calibration value is calculated from the current torque value (with weight for 100%) and the zero point and then stored.	’$’
W C7	Finish calibration Motor is switched off.	’$’
W C8	Test calibration Motor is activated. The calibration value remains saved. Calibration can be checked by attaching weights.	’$’
R CM	Read torque calibration value.	’CM+xx.xxx’

12.6 Error messages during communication HAAKE Viscotester® 550 – PC

Error message	Description
F001	Unknown input command
F011	Transmission error SIO0 (PC interface) Parity error
F012	Receive buffer overload SIO0 (PC interface)
F020	Motor was manually stopped during external mode
F021	Motor was stopped in external mode due to torque overloading
F022	Set value outside permitted range
F090	Command not allowed (CR set value without external switching)
F099	Internal coordination error (command interpreter cannot be activated)

13. Maintenance

The HAAKE Viscotester® 550 is a robust but also sensitive measuring instrument. It does not require any special care if the following points are observed:

- avoid bumps or knocks (especially for the measuring shaft!)
- clean all test material residue off the measuring shaft and screw coupling after usage
- when dirty: do not use cleaning solvents!
  unit components can be damaged and/or combustible fumes can result
- take care not to kink mains or other connection cables. Do not expose cables to tensile load or temperatures above 70 °C
- Always transport or store unit in its case.

13.1 Servicing

Every unit will show some signs of wear after a certain period of time. Check the unit at regular intervals for the following points:

- signs of corrosion on the measuring shaft or screw coupling of the HAAKE Viscotester® 550
- the incorrect alignment of the measuring shaft
- cracks and splits in the power supply unit casing
- damage to the mains cable, plug and/or the connection cable from the HAAKE Viscotester® 550 to the power supply unit.

13.2 Repairs

Due to the modular design of the HAAKE Viscotester® 550 system, damaged or faulty components can be easily exchanged for replacement parts. Repairs should only be carried out by specially equipped and trained personnel.

! ON NO ACCOUNT should you attempt to open up the unit. This warning applies especially to the powers supply unit:
Tampering can have FATAL consequences!

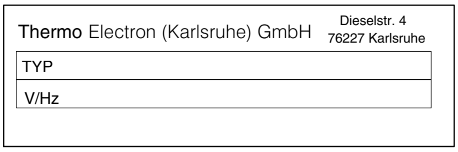
Please contact the Thermo Fisher Scientific-SERVICE department in case of repairs.

You can help us and yourself if you specify the complete type no. printed on the name plate when reporting the damage.

! PULL OUT THE PLUG BEFORE you attempt to examine the defective unit!

A list of some spare parts

Power supply unit
Attachment screws for the temperature control vessel
Knurled nut for attachment bracket
Insulated jacket for temperature control vessel



13.3 Thermo Fisher Scientific-Service department address

Thermo Fisher Scientific SERVICE
Dieselstraße 4
76227 Karlsruhe

Tel.: (0721) 4094-444
Fax: (0721) 4094-360
E-Mail: Support.mc.de@Thermofisher.com

14. Maintenance Instructions

The system check on the HAAKE Viscotester® VT550 must be carried out once yearly.

1. Check the metering shaft for permissible clearance and smooth running.*
2. Check the speed step with digital speed measuring device.*
3. Test and, if necessary, replace damping oil.*
4. Calibrate torque measurement with thread measuring instrument and box of weights, check linearity of torque indication.*
5. Check functioning of the devices with the measuring instrument e.g. MV−DIN with Thermo Fisher Scientific test iquid (Newtonian).
6. Check and adjust the temperature measurement input with the PT−100 simulator.*
7. Check EPROM version and system factors, update EPROMs to current version with Thermo Fisher Scientific test programme.*
8. Check serial interface and data transmission with diagnosis programme and interface tester.
9. Check all solid of revolution for smooth running and any damage; minor irregularities can be eliminated immediately while servicing.*
10. Check housing ventilation incl. internal cleaning *

*** Check by official Thermo Fisher Scientific service.**

Point 5 is recommended every 3 months.

15. Technical Specifications

15.1 General details

Electrical and non-electrical safety according to EN 61010-1 (acc. to IEC 1010-1)

Test environment	indoors
Ambient temperature [° C]	5 to 40
Humidity, relative [%] 5 to 30 °C: 31 to 40 °C: linearly decreasing	80 % 50 %
Air pressure [mbar]	790 (corresponds to approx. 2000 mNN)
Excess voltage category (acc. IEC 664)	II
Contamination level (acc. IEC 664)	2

15.2 Power supply unit

Short description	Autoswitch power supply unit
Input voltage range [$V_{eff.}$]	100... 240 ± 10 %
Input current [$A_{eff.}$]	max. 0.5
Mains frequency range [Hz]	47 – 63
Total wattage [W]	max. 30
Protective class	2
Mains connection	EN 60950
Output voltage [V]	18 ±2 % D.C., protective voltage
Output current [A]	max. 1.1
Unit connection	fixed connection cable, length approx. 2 m with plug acc. to DIN 45323
Casing: Material Dimensions [mm] Type of protection (gem. EN 60529)	PPE 100 x 36 x 63 IP 20

15.3 HAAKE Viscotester VT550

Drive:	servo-motor
Power output [W]:	4
Torque [Ncm]:	max. 3, from 400 min^{-1} linearly decreasing to 2 at 800 min^{-1}
Speed [min^{-1}]:	max. 800, min 0.1; in the CD modus max. 5.0, min. 0.0125
Step angle [deg.]:	1.8; in the CD modus 0.056
Torque measurement	
Measuring principle:	reaction torque, torsion spring, frictionless
Measuring angle at max. torque [deg.]:	approx. 0.4
Measuring value recording:	inductive, non-contact
Resolution [deg.]:	approx. 3 x 10^{-4}
Uncertainty [% fsd]:	+ 0.5
Temperature measurement	
Sensor:	Pt100 acc. to DIN IEC 751
Resolution [deg.]:	0.1
Absolute uncertainty [deg.]:	+ 0.1
Interface:	RS232 fully duplex
Baud rate:	9600, fixed
Plug:	Sub-D: 9-pole
Electronics:	microprocessor, 16 Bit
Casing:	
Material:	aluminium/stainless steel
Color:	RAL 9002
Weight [kg]:	approx. 1.9

16. Terms of Rheological Measurements

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)

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 viscosity 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)

Typical quantities of rheometry and rheology:

Instrument quantities:

M_d	– torque
Ω	– angular velocity
ϕ	– rotation angle
ω	– angular velocity ($2 \cdot \pi \cdot f$)
F_N	– normal force
R, h, ...	– dimensions of sensor
	– etc.

Measuring parameter:

T	– temperature
p	– pressure
t	– time
	– etc.

Rheometrical quantities:

τ	– shear stress
$\dot{\gamma}$	– shear rate
γ	– deformation
N_1, N_2	– normal stress differences
	– etc.

Rheological quantities:

Material functions:

η, $\eta*$, η^+, η^-	– viscosities
Ψ_1, Ψ_2	– normal stress functions
G	– shearing modulus
G*, G' G"	– dynamic shear moduli
J	– compliance
J*, J', J"	– dynamic compliances
	– etc.

Material parameters:

η_0	– zero viscosity
τ_y	– yield point
Ψ_{10}	– 1st normal stress coefficient
	– etc.

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