thermoscientific

Thermo Scientific

HAAKE FallingBallViscometer Type C

Instruction Manual

002-7585 Version 2.4 March 2020

ThermoFisher SCIENTIFIC

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1. Quality Assurance

Dear customer,
Thermo Fisher Scientific implements a **Q**uality **M**anagement **S**ystem certified according to ISO 9001:2008.
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. The results are recorded 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 Contacts 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.

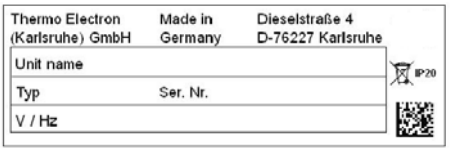
If you have any questions regarding installation or technical issues, please contact the helpdesk. You will find the contact form at:

https://tfs-3.secure.force.com/materialcharacterization/

Thermo Electron (Karlsruhe) GmbH
Part of Thermo Fisher Scientific
Dieselstraße 4
D-76227 Karlsruhe, Germany
Tel. +49(0)721 4094-444
Fax +49(0)721 4094-300

support.mc.de@thermofisher.com
www.thermofisher.com

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.
Type: Order No. 356-0001
Serial number:

_ _ _ _ _ _ _ _ _ _ _ _	12 digit number (e.g. 201625085062)
_ _ _ _	Manufacturing year 4 digit number (e.g. 2016)
_ _ _ _ _	Production order no.. 5 digit number (e.g. 25085)
_ _ _	Serial no. 3 digit number (e.g. 062)

3. Key to Symbols

! Warns the user of possible damage to the unit, draws attention to the risk of injury or contains safety notes and warnings.

☞ Denotes an important remark.

1 Indicates the next operating step to be carried out and…

⇒ …what happens as a result thereof.


Warns the user of possible damage to the unit.

4. Safety Notes and Warnings

These notes are intended to draw your attention to risks which only **YOU** can recognize and avoid or overcome. They are intended to enhance your own safety consciousness.

We have set the highest quality standards for ourselves and this unit during development and production. Every unit meets relevant safety regulations. **The correct unit usage and proper handling is however solely your responsibility.** The following notes must be observed:

! This instruction manual must be carefully studied! It contains important information on the connection to the local mains supply, correct unit usage and safe handling.

! Check for transportation damage during unpacking. Get in contact with supplier and/or carrier for settlement of damage claims. Do not try to start up a damaged unit before the damage has been repaired or you have ascertained the effect of the damage.

! Ensure that this manual is always at hand for every unit operator.

! Only use this unit solely for the intended application.

! Repairs, alterations or modifications must only be carried out only by a professional trained by Thermo Fisher Scientific to handle this equipment. Considerable damage can be caused by improper repairs. The Thermo Fisher Scientific-service department is at your disposal for repair work.

! Thermo Fisher Scientific does not assume responsibility for independent repairs and consequences of improper use.

! Do not clean the unit with solvents (fire risk!) – a wet cloth soaked in household detergent is normally sufficient.

! Only use the heat transfer liquids recommended by Thermo Fisher Scientific. Please refer to the respective EC – Safety Data Sheet.

Severe skin burns can be caused by contact with hot unit parts!

The Falling Ball Viscometer can be operated using a suitable circulator and appropriate bath liquid in the temperature range up to +150 °C which even considering cooling and insulation may heat up parts of the Falling Ball Viscometer enough to cause serious skin burns if contact is made.

Thermo Fisher Scientific recommends the use of personal protective equipment (gloves, safety glasses, laboratory coat, and safety shoes) when operating with the Falling Ball Viscometer. When working at high temperatures wear use temperature–resistant gloves.

! We do not know what substance you will be examining with this device. Many substances are:

- flammable or explosive
- unhealthy
- dangerous for the environnent

You alone are responsible for the handling of these substances!

Our advice:

- If in doubt, consult a safety specialist.
- Read the safety data sheet valid for your country the product manufacturer’s or supplier’s
- Read relevant regulations concerning dangerous materials
- Observe relevant guidelines for laboratories in your country

5. Unpacking

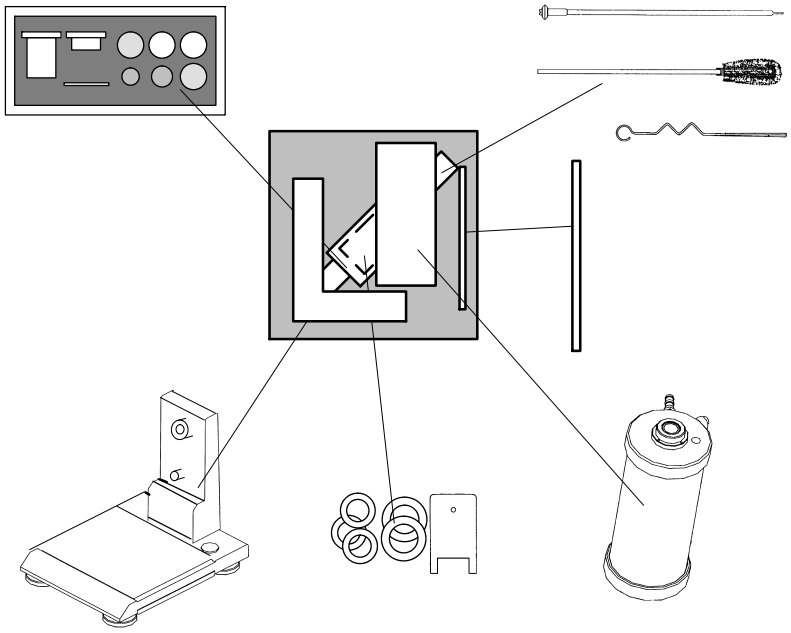
5.1 Transportation damage?

- Notify carrier (forwarding merchant, railroad, post office etc.),
- Compile a damage report.

5.2 Before return delivery:

- Inform dealer or manufacturer
  (Small problems can often be dealt with on the spot).

5.3 Contents of Delivery



The following standard accessory is delivered together with the falling ball viscometer:

Order-No.	Description	
002-7580	Falling ball viscometer, ready for testing	
800-0182	Set of balls 1–6 (see chapter 10.3)	
800-0012	Hollow stopper	
800-0013	Stopper	
800-0119	Ball tweezers	
800-0125	Cleaning piston	
800-0131	Cleaning brush	
800-0014	Cover plate	
800-0027	Stopper gasket	∅ = 15.6 mm
002-5176	Thermometer gasket	∅ = 10 mm
003-2110	Socket wrench (see chapter 10.2)	
LEERER MERKER	Instruction manual	
089–2094	Calibration certifikate	

5.4 Material for use

No other material except for cleaning material (800-0125 cleaning piston, 800-0131 cleaning brush) is necessary at normal use.

5.5 Spare parts

See chapter 13.

5.6 Waste disposal

The transportation packing is made out of paper and can be recycled.

5.7 WEEE Compliance

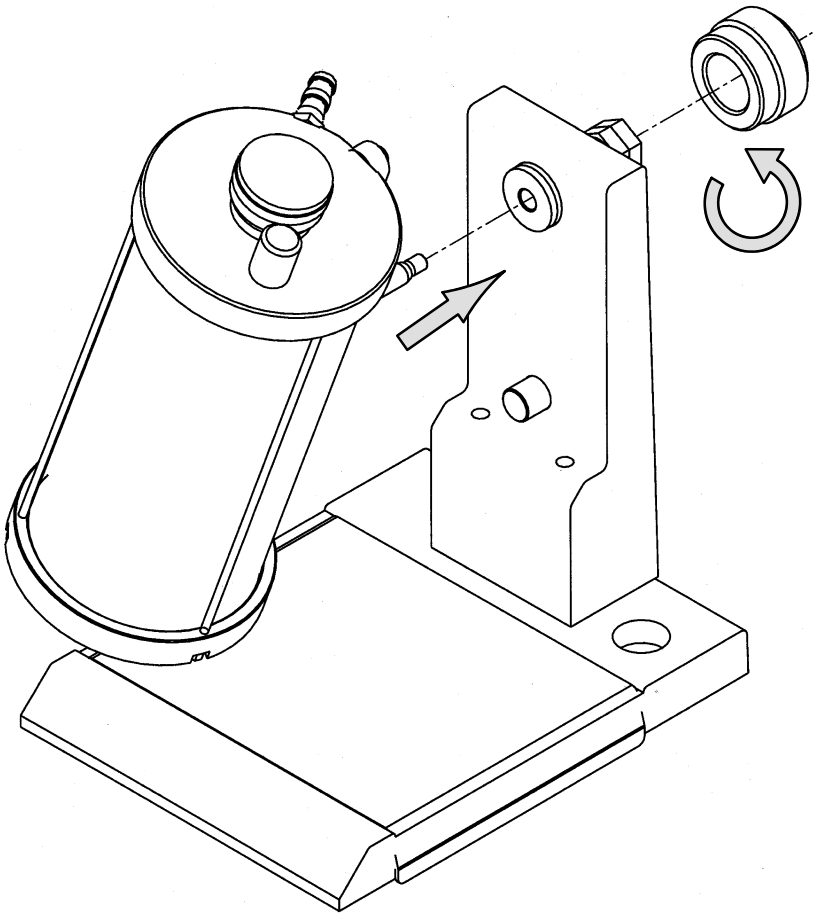
This product is required to comply with the European Union's Waste Electrical & Electronic Equipment (WEEE) Directive 2012/19/EU. It is marked with the following symbol:

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.

6. Setting Up

6.1 Assembly

Insert the measuring tube into the stand and fasten it with the knurled nut.



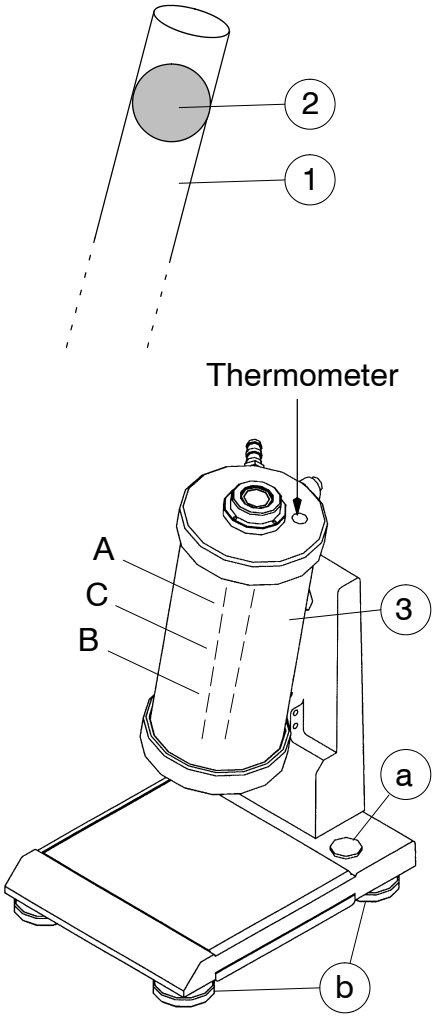
7. Unit Description

7.1 Principle of the measurement

The HAAKE Falling Ball Viscometer measures the viscosity of transparent newtonian liquids. This viscosity is correlated to the time a ball requires to fall a defined distance. The rolling and sliding movement of the ball through the sample filled into a slightly inclined cylindrical measuring tube is described by means of the fall time. The test results are given as the dynamic viscosity using the internationally standardized absolute unit of ”milli Pascal seconds” (mPa·s).

Note: 1 mPa·s = 1 cP (centi Poise)

The Falling Ball Viscometer corresponds to the requirements of many international standards, i.e. ISO 12058 and the german standard DIN 53 015.

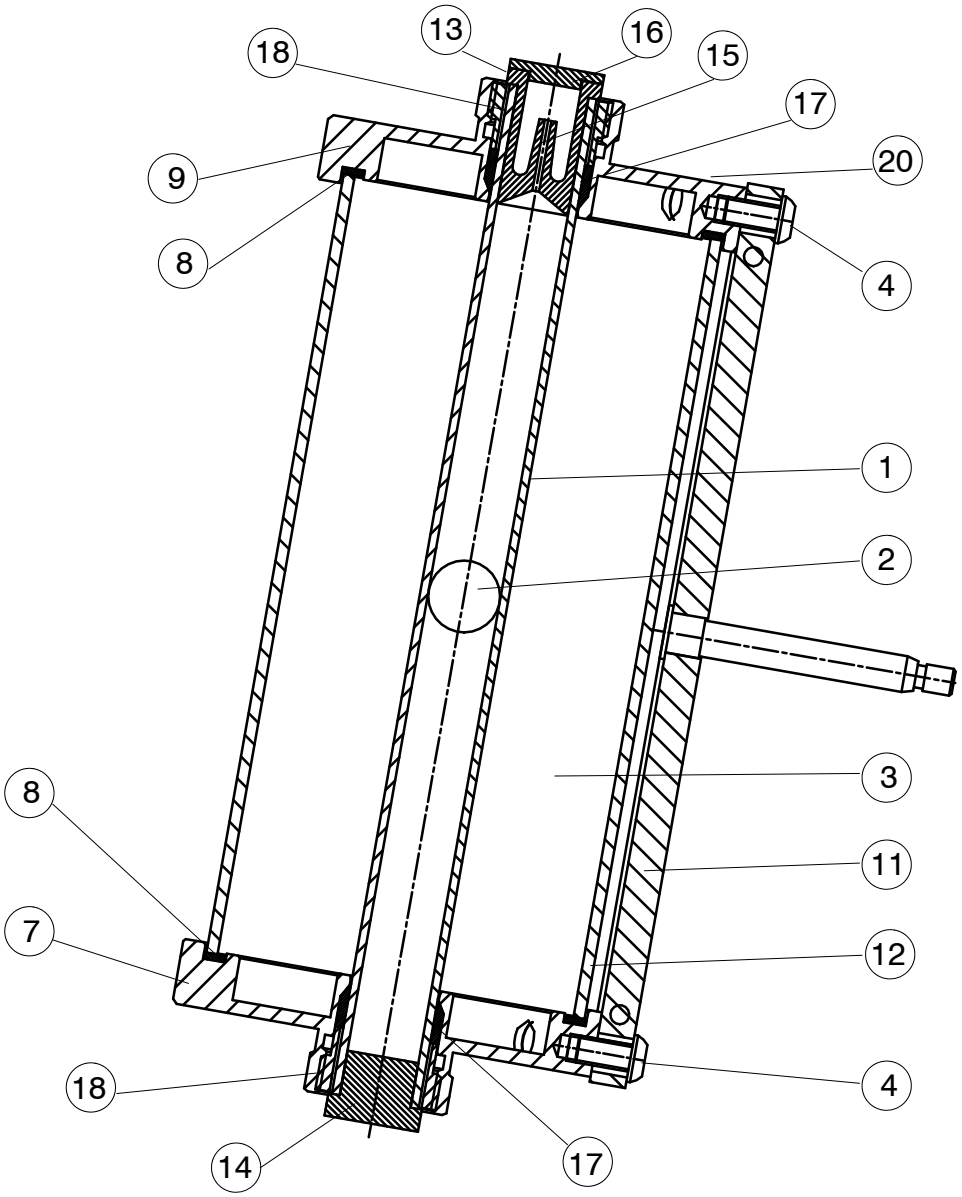


7.2 Description of the instrument

The heart of the instrument is the measuring tube made of glass **1** and a ball **2**. This tube carries two ring marks A and B, which are spaced 100 mm apart and which limit the measuring distance. The ring mark C is in the middle between A and B. The measuring tube is jacketed by means of an outer glass tube, which encloses a room **3** to be filled with a temperature controlled liquid. The measuring tube is fastened to the stand in such a way that its axis is inclined with respect to the vertical by 10° during the measurement.

The measuring tube together with the jacket may be pivoted in order to turn the tube upside down again to let the ball return to the initial position before a measurement. The measuring tube is closed on both sides by two stoppers, one of which **13** contains a capillary and a small reservoir. This stopper prevents undesirable changes of pressure in the liquid sample and has a passage for air bubbles when the temperature is being changed. The viscometer incloses all samples completely to prevent volatization and film forming. The stand may be levelled by means of its water level **a** and the levelling screws **b**. Ein Thermometer, welches auswechselbar ist, ermöglicht das genaue Ablesen in dem Temperaturbereich der geplanten Messung.
As standard, the Falling Ball Viscometer is supplied with a thermometer in the range of –1 °C to + 26 °C. Optionally other thermometers are available, which are listed in section 13.2.

8. Functional Elements



1 Falling tube
2 Ball
3 Tempering room
4 Screw
7 Set screw
8 Gasket
9 Cover
11 Brace
12 Jacket tube
13 Hollow stopper
14 Stopper
15 Capillary
16 Closing plate
17 Gasket for falling tube
18 Threaded bush
20 Connecting rod

9. Measuring

9.1 Preparation for a test

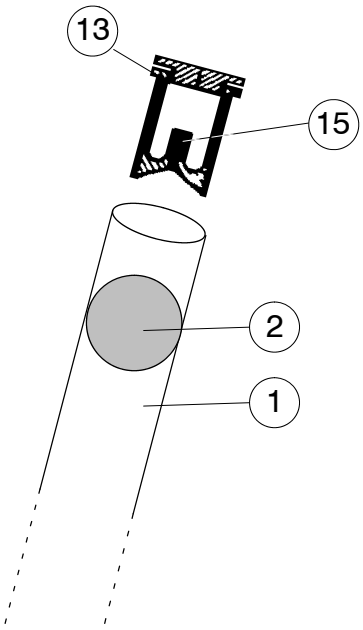
9.1.1 Temperature control

The HAAKE Falling Ball Viscometer may be temperature controlled in a temperature range from –20 °C up to +150°C using liquid circulators.

Ensure that the sample to be measured is homogeneously tempered. For this purpose a sufficient tempering time must be provided, at least 30 minutes. Ideally, the sample was pre-heated to the measurement temperature.

The temperature in the jacket around the measuring tube must be maintained within a temperature tolerance of ± 0.03°C for test temperatures between 10 up to 80°C.
For test temperatures beyond these limits the tolerances may be increased to ± 0.05°C.

The tempering room **3** must be free of air bubbles.



Severe skin burns can be caused by contact with hot unit parts!

9.1.2 Loading the sample

All parts of the viscometer being in direct contact with the sample must be kept clean and dry.

A sample volume of approximately 45 cm^3 is poured into the measuring tube **1** up to 20 mm below the rim of the tube. Then the ball **2** is placed into the tube and the hollow stopper **13** is introduced. The liquid should reach a level just beyond the capillary **15**. The sample in the tube must be free of air bubbles.

Only then does the measurement begin.

9.1.3 Selection of the balls

The standard ball set contains 6 balls, which pass through the measuring tube of an inner diameter of approximately 15.94 ±0.01 mm.

Order-No.	Ball No.	Made of	Density (standard values) ρ g/cm³	Diameter of the ball mm	Constant K (approx.) mPa·s·cm³/g·s	Recomm. measuring range mPa·s
800-0002	1	boron silica glass	2.2	15.81 ± 0.01	0.007	0.6 – 10
800-0003	2	boron silica glass	2.2	15.6 ± 0.05	0.09	9 – 140
800-0004	3	nickel iron alloy [1]	8.1	15.6 ± 0.05	0.09	40 – 700
800-0005	4	nickel iron alloy [1]	8.1	15.2 ±1	0.7	150 – 5000
800-0006	5	nickel iron alloy [1]	8.1	14.0 ± 0.5	7	1500 – 50 000
800-0007	6	nickel iron alloy [1]	8.1	11.0 ± 1	35	> 7500

[1] W.-No. 1.3912

Additionally the following balls are deliverable:

Order-No.	Ball	Made of	Density (standard values) ρ g/cm³	Diameter of the ball mm	Constant K (approx.) mPa·s·cm³/g·s	Recomm. measuring range mPa·s
800-0009	G	boron silica glass	2.2	15.91 ± 0.02	–	gases
800-0010	G3	boron silica glass	2.2	15.30	0.4	20 to 200
800-0011	G4	boron silica glass	2.2	14.40	3.5	150 to 1500

The measuring ranges for viscosity indicated are related to DIN 53015 (as of February 2001).

9.2 Measurement of the falling times

The jacket tube snaps into a defined 10°-position at the bottom of the instrument.

By turning over the jacket tube, the ball is set to the measuring position.

The falling time of the ball moving from the ring mark A to ring mark B is determined by using a stop watch. The time period starts when the lower periphery of the ball touches the ring mark A, which must appear as a straight line. The falling time ends when the lower periphery of the ball touches the ring mark B, which again must appear as a straight line. If one uses the distance AC or CB to reduce very long falling times for high viscous liquids the double of the measuring time period must be taken into account.

Turning the jacket tube 180° again the ball returns to its start position. It is good practice to take the mean value out of several falling time values (3 to 5).

When testing dark liquids it is usually very difficult to see the lower part of the ball. In this case we advise to take the ball equator when it passes through the ring marks.

9.3 Evaluation of the test results

The dynamic viscosity η (in mPa·s) is calculated using the following equation:

$$\eta = K (\rho_1 - \rho_2) \cdot t$$

where:

K = ball constant in mPa·s·cm^3/g·s (see chapter 11)

ρ_1 = density of the ball in g/cm^3 (see chapter 11)

ρ_2 = density of the liquid to be measured at the measuring temperature in g/cm^3

t = falling time of the ball in seconds.

Test results:

The dynamic viscosity η is given in units of mPa·s (cP) and must be completed by stating the sample temperature.

The dynamic viscosity η may be converted to the kinematic viscosity ν by using the following equation:

$$\nu = \frac{\eta}{\rho}$$

ν = kinematic viscosity [mm^2/s] [1 mm^2/s = 1 cSt]

η = dynamic viscosity [mPa·s]

ρ = density of the liquid sample [g/cm^3]

To evaluate the reliability of the results the following criteria may be used:

9.3.1 Reproducibility (one person, one instrument)

If one person determines two test results under identical test conditions, these results are supposed to be acceptable if they do not vary more than the figures stated in the table below from the average value.

9.3.2 Comparability (several persons, several different instruments)

If two sets of test results are reached in two different laboratories under comparable conditions, these results are supposed to be acceptable if they do not vary more than the figures stated in the table below from the average value.

Ball-No.	Reproducibility %	Comparability %	
		DIN 53015	ISO 12058
1	1.0	2	2
2, 3, 4	0.5	1	2
5	0.7	1.5	1
6	1.5	3	3

☞ The falling ball viscometer is suitable only for measurements on Newtonian fluids and is calibrated.

9.4 Viscosity determination of gases

The viscosity determination of gases has to be done with ball G which is made out of glass.

1 The measuring tube must be closed with rubber stoppers fitted with glass stopcocks.

☞ Measuring tube, glass ball and gas must be clean and dry.

2 The tube is flushed several times with the gas to be tested to push out any remains of air. Then the tube filled with the gas sample is closed with the stopcocks and raised to the test temperature.

The calculation of the gas viscosity is based on the comparison with the viscosity of air at 20 ° C (η = 1815·10^{-5} mPa·s):

$$\eta_{gas} = \frac{F_G}{F_A} \cdot 1815 \cdot 10^{-5} \text{ (mPa} \cdot \text{s)}$$

where:

η_{gas}: viscosity of the gas at the temperature T

F_G : falling time of the ball in the gas at the temperature T

F_A : falling time of the ball in air at a temperature of +20°C

Viscosity of the air at a temperature of +20°C:
$1815 \cdot 10^{-5}$ (mPa · s)

10. Cleaning the Measuring Tube

1 Usually the tube is cleaned by rinsing it with a suitable solvent.

2 High viscous liquids (glue and heavy oils, etc.) have to be removed with the cleaning piston which is supplied with the instrument. Push this piston slowly through the tube.

⇒ After this, there will be only a thin film of the liquid left on the walls of the tube which then can be removed with a solvent.

☞ If the plug is removed during cleaning, it can subsequently be inserted into the downpipe more easily if it is moistened.

11. Calibration of the Falling Ball Viscometer

The falling ball viscometer is checked according to the HAAKE regulations before delivery. The geometry relates strictly on the DIN 53015/ISO 12058 and therefore the listed ball constants can be used.
The calibration of the instrument can be done using nationally recyclable calibration fluids. This procedure is also recommended for new balls or after repairs and for falling ball viscometers integrated into a measuring procedure according to ISO 9000.

Testing ...

periodically: to be defined by the user or to be read in the QC-hand book.

with nationally recyclable standards: Calibration fluids acknowledged in the corresponding user country, e.g. Europe: DKD/PTB calibration fluids, in Japan: JIS fluids or in the USA: Cannon calibration fluids.

The calibration excludes the influence of allowed tolerances when measuring balls and falling tubes.

The following Thermo Fisher Scientific products can be used for testing the instrument:

Order-No.	Type	η (mPas) at 20°C	for balls
082-5042	E7	5	1
082-5043	E200	120	2, 3
082-5044	E2000	1900	4
082-5046	E6000	6000	5
082-5336	E15000	15000	6

For calibration of the falling ball viscometer the following calibration fluids (recyclable in Europe) are recommended:

Order-No.	Type	η (mPas) at 20°C	for balls
082-5303	100 BW	100	2, 3
082-5304	2000 AW	2000	4, 5
082-5305	10000 B	10000	5, 6

Calibration

The calibration fluids should be stored in dark rooms and be used within 3 months after their date of expiry.

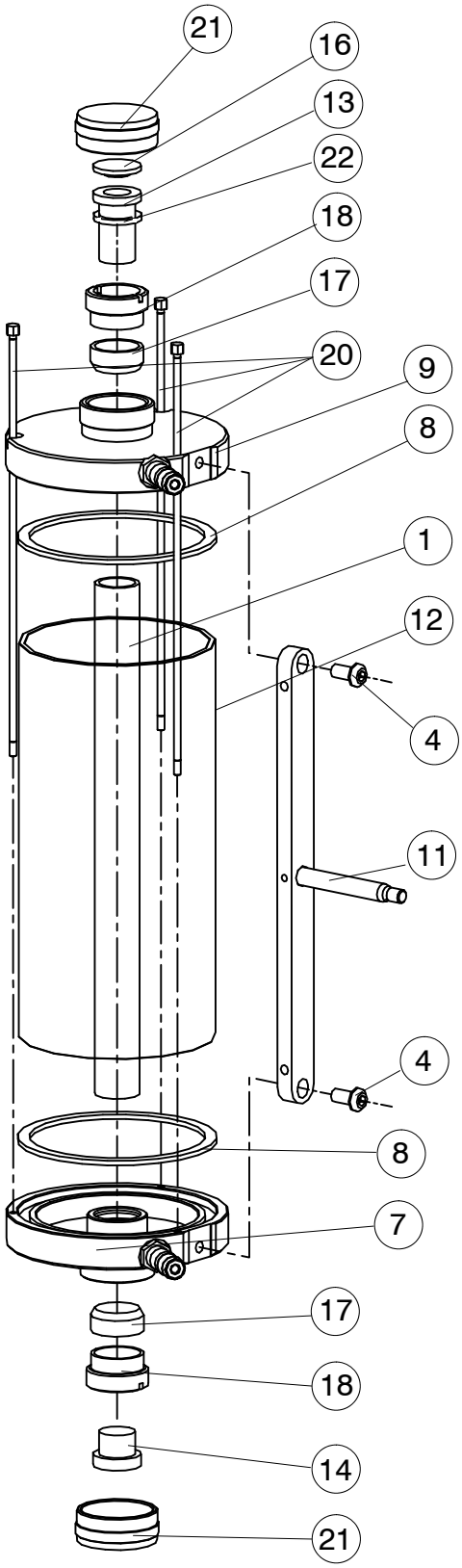
The disposal depends of the composition of the substance which is listed in the "*EC - SAFETY DATA SHEET*".

It is absolutely essential that for calibration the instructions for filling the tube and cleaning are closely adhered to.
It is very important to observe the test temperature of the calibration liquids as indicated on the bottles.

☞ **To avoid contamination the test fluid should never be poured back into the original bottle.**

An officially calibrated thermometer is required which allows reading the temperature with an accuracy of ± 0.02°C.

12. Maintenance by the User



12.1 Exchange of the measuring tube and the jacket tube

1 Unscrew the screws **4** and remove brace **11**.

2 Loosen threaded bush **18** with socket spanner no. 003-2110.

3 Unscrew connecting rods **20**.

4 Now remove both cover lids **7** and **9**.

5 Exchange jacket tube **12**.

6 In order to exchange the measuring tube remove bush **19** and gasket **17**.

Assembly

7 Screw jacket tube **12**, flat gasket **8**, cover **7** and **9** together with the connecting rods **20**.

! Don't tighten the connecting rods initially.

8 Insert measuring tube **1** with gasket **17** into the covers **7** and **9**.

9 Mount brace **11**, don't tighten the screws **4** initially.

10 Please tighten the connecting rods **20** evenly and carefully.

! Recommended tightening moment 0,8-1,0 Nm. Please control, whether the jacket tube is moveable axially.

11 Fix the screws **4**.
Please control, whether the jacket tube is moveable axially.

12 Fix threaded bush **18** with the socket spanner 003-2110 slightly.

12.2 Order numbers

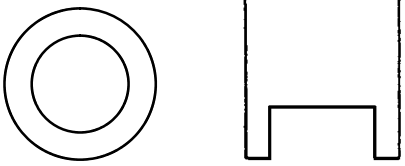
002-7575 Stand (cast iron)
002-2917 Levelling screws of the stand
002-6968 Falling tube (**1**)
800-0190 Balls (**2**)
002-8746 Screws M 6x12 (**4**)
003-2108 Cover (**7**)
003-2109 Cover (**9**)
002-9391 Brace, mounted (**11**)
800-0030 Jacket tube(**12**)
800-0012 Hollow plug (**13**) (**15**)
800-0013 Plug, brass goldplated (**14**)
800–0016 Plug, viton (**14**)
800-0014 Closing plate (**16**)
003-2079 Threaded bush (**18**)
003-2106 Connecting rod (**20**)
800-0052 Closing Gap (**21**)
799-3001 Set of gasket (**8**),(**17**),(**22**)

13. Spare Parts

13.1 Recommended spare parts for a period of 3 years

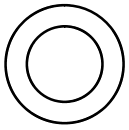
800-0119	Ball tweezers
800-0131	Cleaning brush (3 x)
800-0125	Cleaning piston (3 x)
800-0182	Set of balls 1 – 6
002-6968	Falling tube
800-0030	Jacket tube
800-0015	Hollow plug, Viton
800-0090	Hollow plug, Brass
800-0017	Closing plate, Viton
800-0014	Closing plate, Brass
800-0016	Plug, Viton
800-0093	Plug, Brass
800-0052	Closing Gap
801-0190	Spirit level
222–2323	Checking thermometer –1 to 26° C, scaling 0.1° C
800-0176	Stop watch
799-3001	Set of gasket

13.2 Single parts



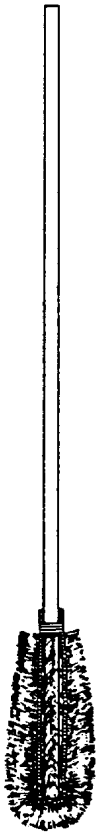
800-0027
gasket ∅ 15.6
(teflon)

003-2110
socket spanner



002-5176
gasket ∅ 10.0
(viton)

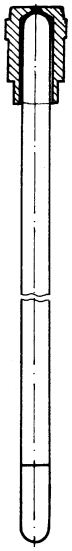
800-0119
ball tweezers



800-0131
cleaning brush

800-0125
cleaning piston

13.3 High-resolution thermometer



Control thermometers:

222-2322	-35	to 1 °C,	scaling 0.2 °C,	accuracy 0.2 °C
222-2323	-1	to 26 °C,	scaling 0.1 °C,	accuracy 0.1 °C
222-2324	24	to 51 °C,	scaling 0.1 °C,	accuracy 0.1 °C
222-2325	49	to 76 °C,	scaling 0.1 °C,	accuracy 0.1 °C
222-2326	74	to 101 °C,	scaling 0.1 °C,	accuracy 0.1 °C
222-2327	50	to 150 °C,	scaling 0.5 °C,	accuracy 0.5 °C

*Delivery with control thermometer from June 2017;
Delivery with mercury thermometer up to the end of May 2017,
please refer to the instruction manual with version 2.2.

13.3.1 The use of a thermometer with wetting expansion liquid

This thermometer, which you purchased, is a measuring device of high quality. Since it works with an expansion liquid which wets glass, there have to be observed the following operating instructions in order to avoid wrong measurement results.

1. a. A thermometer with a range not exceeding 115 °C should be warmed carefully till the thermometer filling enters into the safety chamber at the upper end of the capillary tube and fills it to about a third. During this procedure also possible separated liquid parts are rejoined with the liquid column.
1. b. An instrument with a range above 115 °C is warmed carefully till the highest temperature of the range is achieved (possibly separated liquid parts are rejoined before by strong swinging).
2. Afterwards let the thermometer cool down very slowly to achieve the wetting liquid flows down as completely as possible. For most exact temperature determinations the speed of the cooling should be about 1 °C per minute down to the temperature at which shall be measured.
3. After the cooling process measurement can be carried out. Should be worked afterwards at higher tem-

peratures, then no further pretreatment is needed.

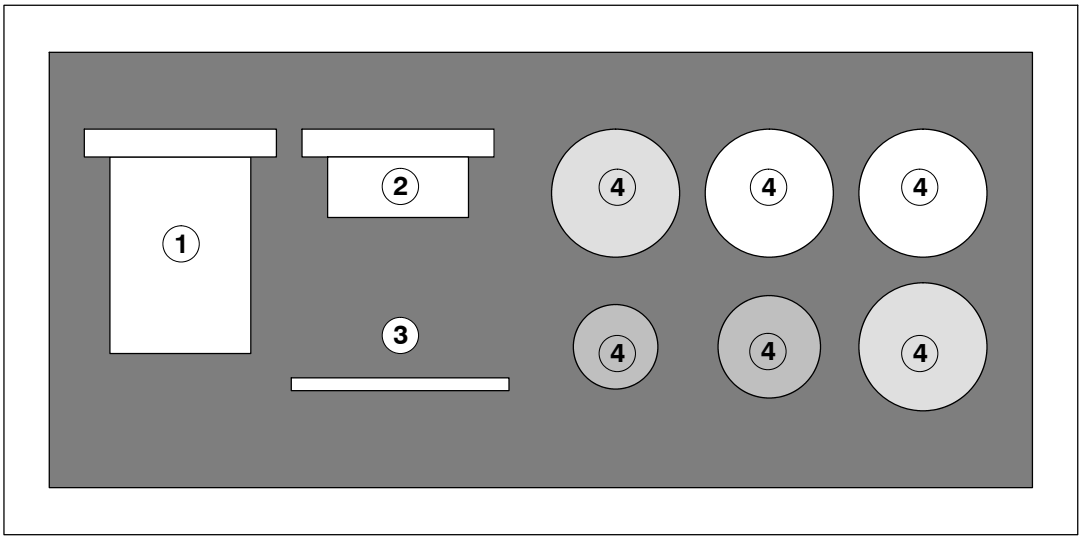
4 Before every new measurement at a lower temperature than just indicated by the thermometer, there has to be proceeded as described at point 2. In case that parts of separated filling are noticed, follow once more point 1 and 2.

5 Normally the thermometer is calibrated for total immersion, i. e. it shows exactly, if it will be immersed up to the reading point into the medium to be measured. At temperatures from about +50 °C and higher, the reading point should be above the medium a little bit to avoid that liquid distils colourless in the safety chamber (source of errors). The somewhat emerged part of the liquid results a small error, which can be determined by a correction of the emergent column.

6 A partial immersion instrument should be handled according to above points 1 to 4 as well. The destillate as mentioned in point 5 however must not be feared. The stipulated Average Temperature of Emergent Column has yet to be kept exactly.

7 The thermometer should be kept upright in order to avoid a separation of the liquid column. A vertical position of the instruments is also recommended, if it is in longer use.

13.4 Contents of the ball box



(1) 800-0012 Hollow plug (brass)

(2) 800-0014 Cover plate

(3) 800-0118 Ball gauge to differ between the balls G, 1 and 2

(4) Balls:

800-0002 Ball 1; ∅ 15.81; boron silica glass
800-0003 Ball 2; ∅ 15.6; boron silica glass
800-0004 Ball 3; ∅ 15.6; nickel iron alloy[1]
800-0005 Ball 4; ∅ 15.2; nickel iron alloy[1]
800-0006 Ball 5; ∅ 14.0; nickel iron alloy[1]
800-0007 Ball 6; ∅ 11.0; nickel iron alloy[1]

[1]material number 1.3912

14. Appendix

14.1 Example: Evaluation
(sugar solution of 40 %)

Density of ball 2:	2.2 (g/cm^3)
Density of the solution:	1.18 (g/cm^3)
Ball constant K :	0.09 (mPa·s·cm^3/g·s)
Falling time:	61 s
Measuring temperature:	20.0°C

The absolute viscosity is ...

$\eta_{20°C} = 0{,}09 \cdot (2{,}2 - 1{,}18) \cdot 61 = 5{,}60$ (mPa · s)

In most cases the densities of the test liquids are known. The evaluation may be simplified by introducing a factor which includes the densities. In our example of the sugar solution the exact factor is ...

$(\rho_1 - \rho_2) \cdot K = 0{,}1098$ (mPa · s / s)

14.2 Example: Calibration of ball 1

K = ball constant to be found

η_E = 4.63 [mPa·s] viscosity of the standard liquid

ρ_1 = 2.217 [g/cm^3] density of the ball

ρ_2 = 0.81 [g/cm^3] density of the standard liquid

t = 417.7 [s] average of the falling time

For the calculation of the ball constant K the following formula applies:

$$K = \frac{\eta_E}{(\rho_1 - \rho_2) \cdot t} = \frac{4.63}{(2.217 - 0.81) \cdot 417.7} = 0.00788$$

The determination of the constants of the other balls should be done in the same way.

Contacting us

If you have any questions regarding installation or technical issues, please contact the helpdesk. You will find the contact form at:

https://tfs-3.secure.force.com/materialcharacterization/

There are several ways to contact Thermo Fisher Scientific for the information you need.

❖ **To contact Technical Support or Sales, Germany and International**

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