RHEOTEST Messgeräte Medingen GmbH



HÖPPLER® KF 3.2 Operation manual

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1. Applications

Viscometer type HÖPPLER® KF 3.2 is a falling ball viscometer to DIN 53 015. The measured parameter is the time of fall of the ball in a cylindrical tube inclined by 10 deg with respect to the vertical plane and filled with the liquid subject to investigation. Viscometer type HÖPPLER® KF 3.2 chiefly is used to measure the viscosity of Newtonian liquids.

Measured quantity: Dynamic viscosity in Pascal seconds (Pa·s) or milli-Pascal seconds (mPa·s). For non-Newtonian liquids, viscometer HÖPPLER® KF 3.2 provides under identical conditions of measurement reproducible values that in many instances will meet the requirements of industrial process measurement (c.f. para 7.). Viscometer HÖPPLER® KF 3.2 offers advantages particularly because of its narrow accuracy tolerances, accurate temperature control of the medium subject to investigation, and due to air-tight sealing that prevents volatilization and film formation during measurement. No temperature correction is necessary. Determination of the ball fall times in two measuring positions essentially diminishes the time required to determine several points of measurement. This viscometer suits stationary mounting for frequent use.

2. Specification

Viscometer, precision make, type HÖPPLER® KF 3.2, set of six balls, measuring distance 100 mm, tube inclined by 10 deg with respect to the vertical plane, swinging viscometer section, two measuring positions for to and fro travel of ball.

Measuring range 0,6 to 70 000 mPas

above for measuring times exceeding 300 sec

Times of fall 30 to 300 sec and above

Accuracy tolerances 0,5 to 2 % of measured value, depending on ball diameter

Temperature range -60 to +150 °C

Filling volume 40 ml

Dimensions (width x depth x height) 205 mm x185 mm x 315 mm (excl. packing)

Weight 2,9 kg (excl. packing)

5,6 kg (incl. packing)

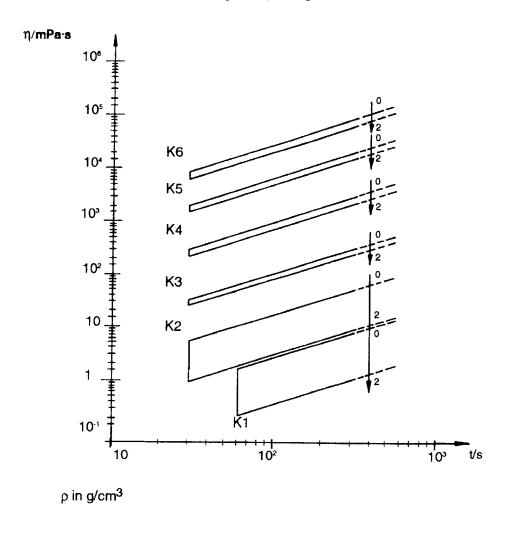


Figure 1 Viscosity measuring ranges in response to time of fall of balls versus density of substance subject to measurement.

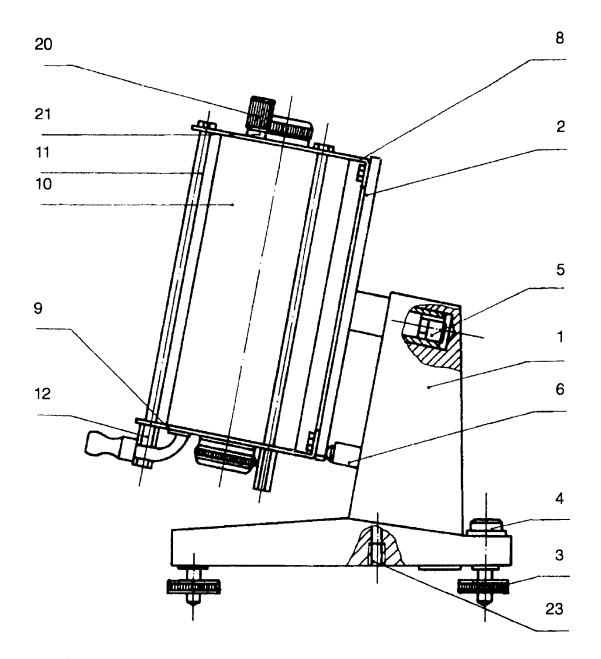


Figure 2 HÖPPLER® KF 3.2

- 1 base
- 2 Viscometer
- 3 adjusting screw
- 4 water level
- 5 supporting pin
- 6 socket
- 7 fall tube
- 8 top plate, complete
- 9 bottom plate, complete
- 10 water bath jacket
- 11 connecting rod
- 12 nut

- 13 rubber packing
- 14 fall tube screw union
- 15 stopper II
- 16 cover
- 17 stopper l
- 18 sealing cap
- 19 gasket
- 20 thermometer screw
- 21 gasket
- 22 rubber ring
- 23 securing screw for transportation

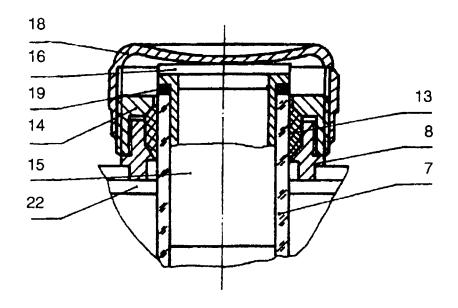


Figure 3 Fall tube screw union to top plate, complete

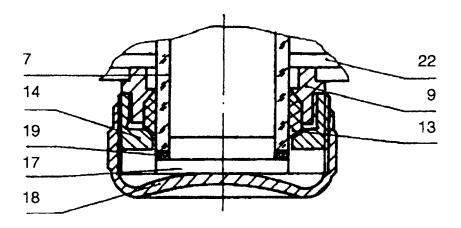


Figure 4 Fall tube screw union to bottom plate, complete

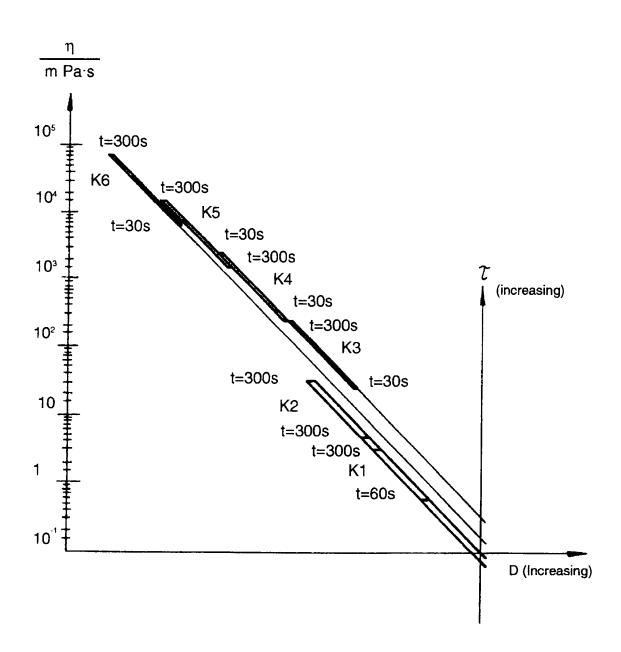


Figure 5 K1 through K2 - glass ball K3 through K6 - metal ball

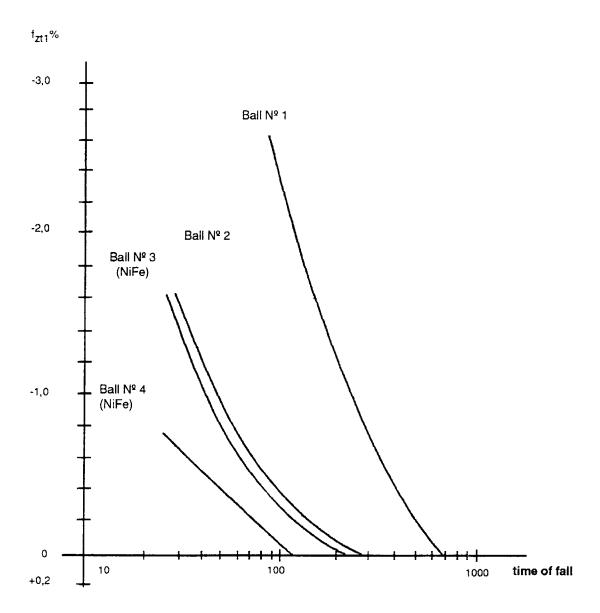


Figure 6 Systematic additional error for short fall times

3. Description of Viscometer

Viscometer HÖPPLER KF® 3.2 (c.f. Figure 2) comprises two main sub units, viz base (1) and viscometer (2). The base features three adjusting screws (3) for horizontal alignment of viscometer to complete water level (4). The viscometer is inclined by 10 deg with respect to the vertical plane. Supporting pin (5) in the base permits to swing the viscometer optionally into either of both measuring positions. A locking device engaging automatically in either measuring position secures the viscometer. A fitting key at the height of the supporting pin prevents slipping of the viscometer from the base. Water bath jacket (10) fitted between complete top plate (8) and complete bottom plate (9) encloses fall tube (7) in the viscometer. Rubber rings (22) prevent leakage of the temperature control liquid. A detachable joint comprising connecting rod (11) and nut (12) outside the water bath warrants interchangeability of the water bath jacket. The fall tube is fastened in the top plate (c.f. Figure 3) and bottom plate (of Figure 4) by means of rubber packings (13) and fall tube screw unions (14). Two threaded pins M 2 x 4 prevent acidental loosening of the fall tube screw union.

The scope of delivery of viscometer HÖPPLER® KF 3.2 includes a set of chromium-plated brass stoppers comprising stopper II (15) and cover (16) for the fall tube top end, and stopper I (17) for the fall tube bottom end. Stopper II is a capillary stopper intended to keep during measurement the fall tube free from air bubbles. Screw down under slight pressure sealing cape (18). Gaskets (19) seal the fall tube liquid-tight.

The bottom plate is fitted with a short inlet tube and a long outlet tube to feed and drain the temperature control liquid.

At special order, a set of eleven thermometers will be supplied alongside with the viscometer. The thermometer is fastened in thermometer screw (20) by a frictional holder and can be easily interchanged by another one having a different measuring range. A screw union in the top plate of the viscometer holds the thermometer screw with thermometer. Gasket (21) prevents leakage of the temperature control liquid. If the graduation of the thermometer should not be in a suitable position to take readings, rotate the thermometer to requirement in the thermometer screw.

The scope of delivery of the viscometer includes a case with a set of six balls of different diameters and made from different materials. A ball gage provides identification of the balls the diameters of which differ but insignificantly. Ball 1 is bigger than the bore of the ball gage, and all the other balls will pass the gage with more or less clearance depending on their sizes.

According to the ball diameter, the measuring accuracy is within the range \le 0,5 to \ge 2 %. The measuring distance of the ball between annular marks is 100 mm and 50 mm, respectively.

The accessories to the viscometer include a set of stoppers made from Perbunan that is resistant to gasoline. These stoppers seal the fall tube during measurements of aqueous solutions and similar fluids.

To loosen and tighten the fall tube screw unions and threaded pins M 2×4 securing the latter, the standard accessories include a flat wrench and a screw driver 0.18 \times 1.5. A ball catch available at special order will be useful to empty the fall tube.

A case houses for transportation the viscometer together with the sets of balls and thermometers, operating instructions manual, test certificate, and accessories.

For frequent stationary use of the viscometer, unscrew screw (23) at bottom of base to dismantle the base and fasten viscometer keeping in mind its alignment to water level.

4. To Prepare Measurement

4.1 To Choose Balls

Table 1 - Set of Balls for Viscometer Type HÖPPLER® KF 3.2

Ball No	Dia	Constant	Minimum time of fall	Measuring (basing on min time of fall) bottom limit	g range (basing on 300 sec time of fall) top limit	Accuracy abs value Newtonian liquids at +20 °C	Reproducibility (depending on substance)
	mm	mPa · cm³ · g°	¹ s	mPa	as	%	%
1	15,805	0,009	60	0,6	. 5	≤2,0	±1,00
2	15,630	0,07	30	3	30	≤0,5	±0,25
3	15,560	0,13	30	25	250	≤0,5	±0,25
4	15,600	0,7	30	200 4	4 800	≤1,0	±0,50
5	14,000	6,4	30	1 250	12 500	≤1,0	±0,50
6	11,000	34,0	30	7 000	70 000	≤1,5	±0,75

All rights to a different assortment of the ball set are reserved.

We recommend to not shorten the minimum times of fall specified in each case because otherwise a turbulent flow would occur. Under such conditions, the measured viscosities would be excessively high. With times of fall exceeding 300 sec, liquids having higher viscosities than those specified in the Table above may be investigated.

Glass balls and fall tube are made from glass, metal balls consist of a special nickel-steel alloy having a precisely defined coefficient of linear expansion. The fall tube diameter accuracy is $\pm 1~\mu m$, and the balls except both the smallest ones are manufactured with a diameter accuracy of $\pm 0.2~\mu m$. With respect to both smallest balls $\pm 0.5~m$ m tolerance is sufficient. The fall tube and the balls are finished with high precision and therefore have to be handled workmanlike. The ball weights are vacuum-corrected and the specified weights suit identification by the user to avoid confusion. All viscometers are tested in our works before dispatch. The results of such precision tests are recorded in the test certificate concerned.

4.2 To Prepare the Measurement

Clean the fall tube, plugs, and balls workmanlike before start of measurement. Maximum cleanliness is very important. To clean the fall tube, first withdraw viscometer from the supporting pin guide in the base and put it into a tray to collect the liquid subject to measurement. Having loosened the sealing caps and removed the stoppers, collect balls using the ball catch when they leave the fall tube (never permit them to drop into the collecting tray). After the liquid subject to measurement has been drained from the fall tube, use cleaning rod included into the accessories for preliminary cleaning of fall tube. Remove the remaining thin film by suitable solvents using the cleaning brush. Dry fall tube rinsing it with sulfuric ether methylene chloride or with other appropriate substances. Consider duly any regulations concerning protection of laboratory. Be careful that no damp deposits by subcooling during rinsing must be present on the inner tube wall when filling in the substance to be investigated.

Hot soda lye containing 5 % concentrated ammonia yielded good results to degrease the fall tube when aqueous solutions are involved. Rinse tube with distilled water. Water must run down smoothly the inner wall of the fall tube.

Choose balls for measurement, clean them using the deerskin rag included into the accessories and remove any dust and fibre particles by means of the brush and ball pincette.

4.3 To Fill Fall Tube

Close fall tube at bottom plate by means of stopper I and sealing cap (c.f. Figure 4) pressing against top plate of fall tube to prevent its axial displacement. Fill low viscosity liquids through a glass filter tube G 3 into the fall tube. Keep duly in mind that the bigger balls will respond strongly to impurities. Fill in liquid up to about 25 mm below fall tube top rim and introduce the required ball using the ball pincette included into the scope of delivery. Push ball downwards using the glass rod if air bubbles are found below the ball. Close fall tube by means of stopper II and cover (c.f. Figure 3). Tighten uniformly both sealing caps under slight pressure. Use set of metal stoppers with any liquids to which rubber is not resistant. Otherwise, it would be advantageous to use the rubber stoppers consisting of Perbunan resistant to temperature and to gasoline or benzene. Liquids being filled in must be free from combined gases that will produce troublesome gas bubbles with increasing measuring temperatures. In most instances, it will suffice to heat the liquid to be investigated briefly up to about 10 K above the maximum expected measuring temperature and to fill it into the fall tube while it is hot. If notwithstanding such procedure air bubbles are found below the ball after the liquid has been filled in, expel them striking gently against the top sealing cap and discharge them with stopper II through the capillary. Begin investigation only after such precautionary measures. Duly observe and follow any regulations concerning protection of laboratory relating to the substance subject to measurement.

4.4 Temperature Control

Recommended temperature control liquids:

Temperature Range	Temperature Control Liquid	Flexible Tubes to Thermostat
-60 to 10 °C	spirit	Perbunan rubber hose, glass wool insulated
+ 1 to 95 °C +20 to 150 °C	distilled water fluid transparent methyl phenyl silicone oils	Perbunan rubber hose, without insulation Perbunan rubber hose, without insulation

With high or low working temperatures secure flexible connecting tubes to thermostat by means of hose clips.

Prevent damp deposits on the water bath jacket rubbing it with alcohol.

With respect to temperature control of the liquid subject to investigation, viscometer type HÖPPLER® KF 3.2 belongs to the most accurate ones. No correction of temperature is necessary. The viscosity of many liquids depends heavily on temperature. Errors due to poor temperature control of the liquid being investigated could exceed the guaranteed accuracy tolerances. Find out the temperature response of the viscosity of the substance you are investigating.

Seal fall tube by means of top sealing cap only after the liquid being investigated has reached the final temperature. Otherwise, the pressure being built up in the measuring tube would falsify the measured values or destroy the fall tube.

4.5 To Illuminate and Align Viscometer

Place viscometer in front of a white illuminated background. Chiefly with dark liquids, the absorption of infrared radiation could considerably falsify results of measurement. We recommend to use an adiathermic lamp emitting light that is almost free from heat radiation.

Align viscometer to water level by means of adjusting screws.

5. To Determine Time of Fall

Take time of fall between both annular marks using an accurate stopwatch providing reading down to 1/1.000 sec. Errors due to inaccurate reading of time could exceed the guaranteed accuracy tolerances. Divergences of the times of measurement essentially will depend on the constancy of temperature. The accuracy of the viscometer provides that even temperature changes of some hundredths of one Kelvin would suffice to vary the times of fall. Errors could be due to pressure produced by mounting the top sealing cap before reaching the rated temperature in the measuring chamber.

To better mark the lower periphery of the ball, hold a black strip of cardboard behind the viscometer, with glass balls at short distance above the annular marks, with metal balls somewhat below the annular marks. The outline of the ball will appear clearly visible, especially when an adiathermic lamp is used for illumination.

Opaque liquids can be examined safely observing the bearing point of the ball on the fall tube wall in an oblique angle from below.

6. To Determine Dynamic Viscosity from Time of Fall

Calculate dynamic viscosity to the formula::

$$\eta = t \cdot (\varrho_1 - \varrho_2) \cdot K$$

η - dynamic viscosity, milli Pascal second (mPa s)

t - time of fall (sec)

K - ball constant [(mPa s)/g]

 ϱ_1 and K are given for each ball in the test certificate. The expression in brackets($\varrho_1-\varrho_2$) comprises the correction for buoyancy. To calculate the density ϱ_2 from the weight, for high accuracy viscosity measurement consider duly the correction for air buoyancy of the liquid with respect to the weights:

$$\rho_2 = \frac{m_2}{v_2} \cdot (1 - \frac{\rho_3}{\rho_4}) + \rho_3$$

where

m₂ - uncorrected weight of liquid (g)

v₂ - volume of liquid (cm³)

Q3 - density of air (g/cm³)

Q4 - density of weights (g/cm³)

Accuracy in determination of the liquid density will be the better the more the latter is approaching the density of the ball and thus the amount $(\varrho_1 - \varrho_1)$ is decreasing.

Using glass balls (density $\varrho_1 \approx 2,22$ g/cm³), determination of the liquid density ϱ_2 with an accuracy down to the third decimal in g/cm³ is recommended. With respect to metal balls (density $\varrho_1 \approx 8,1$ g/cm³), an accuracy down to the second decimal in g/cm³ will suffice.

For liquids having a known coefficient of expansion γ , density ϱ_{2T} inherent to measuring temperature T can be calculated from the known density ϱ_{2To} at temperature T_o adopting the formula:

$$\varrho_{2T} = \varrho_{2To} [1 - \gamma (T - T_0)]$$

where

T - measuring temperature (K)

To - temperature (K) for which density ϱ_{2To} is known.

Considering the small coefficient of expansion of the balls, the change of its densities will be negligibly small.

7. Flow Anomalies

Non-Newtonian liquids, e.g. colloidal substances, show so-called flow anomalies which manifest themselves in repeated determinations of the time of fall by a specific trend or quite different viscosity values resulting with balls having different diameters. Two phenomena prevail generally. Keep duly in mind that flow anomalies must not be simulated neither by insufficient temperature control (c.f. para 4.4) nor by pressure changes (c.f. para 5.). An essential advantage of the falling ball viscometer is the fact that hermetic sealing of the substance subject to investigation in the fall tube excludes simulation of flow anomalies due to volatilization or film formation.

7.1 Structural Viscosity, Dilatation

Liquids featuring structural viscosity or dilatation do not follow the law of proportionality between shear stress and changing speed. This phenomenon manifests itself by quite different viscosity values obtained with different balls while the times of fall T_1 of a ball feature no tendency. In case of dilatation, viscosity will increase overproportionally with increasing shear stress and underproportionally with structural viscosity. Any viscometers operating on but a single shear stress do not suit accurate measurement of this phenomenon. Balls of different sizes will produce different shear stresses τ (c.f. Figure 5). These are not constant over the crescent shaped gap between the ball and the fall tube. Thus, no accurate figure about the actual shear stress can be given. However, the shear stress differences among different balls suffice to identify structural viscosity or dilatation. Because most measurements are industrial comparative measurements, in such instances the statement of the measured viscosity and of the fall ball concerned could suffice.

The HÖPPLER® KD pressure-ball suits approximate determination of structural viscosity and dilatation. For accurate determination, rotary viscometer RHEOTEST® should be used.

7.2 Thixotropy, Rheopexy

Thixotropy is understood as isothermal, reversible, purely mechanical gel-sol conversion of a substance. This phenomenon occurs much more frequently than generally supposed. Carrying out measurements using a Höppler viscometer, thixotropy in parallel with progressive destruction of structures manifests itself by continuous reduction of the times of fall down to a minimum value. Because an exact measurement of this phenomenon is often desirable, proceed as follows:

Fill in liquid and wait some time for structural regeneration of the substance being measured. The first and longest time of fall that is measured is the gel viscosity. Subsequently, have the ball fall repeatedly in either direction until there is no more decrease of the time of fall. This shortest time of fall is the sol viscosity. The relative decrease of viscosity is a direct measure of thixotropy and thus a useful characteristic of the substance being tested. Substances featuring rheopexy (antithixotropy) will solidify under the effect of shear stress, i. e. the times of fall of the ball will increase. Rheopexy is the opposite to thixotropy and occurs relatively seldom. Both thixotropy and rheopexy suit well determination by means of viscometer HÖPPLER KF® 3.2 because two measuring positions provide measuring values in brief intervals.

8. Conversion of Dynamic Viscosity

Convert the dynamic viscosity η into kinematic viscosity ν with the equation:

$$\frac{\eta (mPa \cdot s)}{p_2 \cdot (g / cm^3)} = v \cdot (mm^2 \cdot s^{-1})$$

9. Repairs

Repairs involving substitution, dismantling, rotation or displacement of the fall tube invalidate the original calibration and requires repetition of the procedure to para 10.

To replace fall tube

- 1. Turn loose both threaded pins of fall tube screw unions.
- 2. Dismantle fall tube screw unions using ring nut wrench.
- Strip rubber packing from one end of fall tube, remove fall tube.
 Fasten fall tube packings with some glycerine, mount new fall tube.
- 5. Fasten fall tube in reversed order of disassembly. The tube mark (etched strip) shall point exactly at the arrow on the top plate

To replace water bath jacket

- 1. Dismantle fall tube to paras 1. through 3. above.
- 2. Unscrew three bottom nuts
- 3. Unscrew two screws from top and bottom plate, remove top plate from bar.
- 4. Replace rubber rings and mount new water bath jacket.
- 5. Fit top plate and tighten nuts uniformly until plate is fastened tightly. Tighten both screws of bottom plate in bar (oblong holes).
- 6. Mount fall tube to description above, paras 4. and 5.

10. To Calibrate Viscometer

To repeat calibration after any modifications on the fall tube (substitution, dismantling e.g. to clean water bath) or on balls (substitution), you will require calibration liquids supplied by our company - please order referring table 2. In our company, the calibration liquids are subject to continuous inspection according to DIN ISO 9001 and they are always available in original bottles of 50 cm³ capacity each and under accurate specification of their actual viscosity and density, c. f. List for Orders.

NF	2	≈	2	mPa·s	NF	700	~	700	mPa · s
NF	4	≈	4	mPa·s	NF	1 000	≈	1 000	mPa·s
NF	10	≈	10	mPa · s	NF	3 000	≈	3 000	mPa·s
NF	20	*	20	mPa·s	NF	5 000	~	5 000	mPa·s
NF	30	~	30	mPa·s	NF	10 000	~	10 000	mPa·s
NF	60	≈	60	mPa·s	NF	15 000	≈	15 000	mPa·s
NF	115	≈	115	mPa · s	NF	50 000	~	50 000	mPa · s
NF	250	≈	250	mPa · s					

Order separately test certificates of calibration liquids. The testing temperature is ± 20 °C $\pm 0,005$ K. and shall be strictly observed. Perform testing very carefully to avoid any errors that otherwise would occur permanently with the results of measurement.

Table 2 informs about the most suitable calibration liquid for each ball size

Table 2

Ball No.	1	2	3	4	5
Calibration Liquid (NF)	4	20	115	1 000	3 000

Table 1 (c.f. para 4.1) states the constant to be expected. As a general rule applies that the response of balls to changing diameters will be the higher the larger they are. Wider fall tubes or smaller balls, respectively, involve larger constants. The narrow serial tolerance of max 0.015 mm would suffice to produce considerable changes of the ball constant with larger balls.

Calculate constants from the times of fall as measured to the equation:

$$K = \frac{\eta}{(\rho \rho - \rho 2) \cdot t}$$

(for symbols, refer to para (6)

Accurate temperature determination is of utmost importance to be able to introduce the correct viscosity into the formula.

Changes of the ball diameter in the range from 8 to 14 mm will involve but insignificant changes of the ball constants. Find out ball constant to ball No 6 adopting the following equation:

$$K_6 = 1,4057 (D-d_6) (0,750412 + 1,82637 d_6/D)$$

D - fall tube diameter

d₆ - diameter of ball No 6

K₆ - constant of ball No 6

For D, d₆, c.f. calibration Certificate

11. References

The literature on falling ball viscometers has become extremely voluminous. Therefore, please forward your inquiries to our address, or consult your closest technical library.

Our testing laboratory shall be glad to consult you on specific problems and to suggest methods of measurement.

12. List for Ordering

bottle NF

bottle NF

bottle NF

bottle NF

bottle NF

4 (50 cm³)

(50 cm³)

(50 cm³)

 (50 cm^3)

3 000 (50 cm³)

20

115

1 000

Order No Viscometer HÖPPLER® KF 3.2 2082.1.00001 scope of delivery: 1 set of balls (6 balls and one ball gage, in case) 1 supervisory thermometer -1 ... +21 °C, graduation 0.1 K, in case 1 ball pincette 1 glass filter 1 glass rod 1 brush 2 cleaning brush 1 flat wrench 1 cleaning rod 1 deerskin rag 1 screw driver 0.18 x 1.5 4 gasket A 16 x 20 (Perbunan) 2 packing 1 calibration certificate 1 operating instructions manual 1 guaranty certificate Accessories at special request: Supervisory thermometers 1 thermometer - 60 to - 19 °C, scale interval 0.5 K 8700.3.36020 1 thermometer - 21 to + 1 °C, scale interval 0.1 K 8700.3.36021 1 thermometer - 1 to + 21 °C, scale interval 0.1 K 8700.3.36022 + 19 to + 41 °C, scale interval 0.1 K 1 thermometer 8700.3.36023 1 thermometer + 39 to + 61 °C, scale interval 0.1 K 8700.3.36024 1 thermometer + 59 to + 81 °C, scale interval 0.1 K 8700.3.36025 1 thermometer + 79 to +101 °C, scale interval 0.2 K 8700.3.36026 1 thermometer + 99 to +126 °C, scale interval 0.2 K 8700.3.36027 1 thermometer +124 to +151 °C, scale interval 0.2 K 8700.3.36028 1 thermometer 0 to +100 °C, scale interval 0.5 K 8700.3.36030 1 thermometer + 19 to + 21 °C, scale interval 0.02 K 8700.3.36029 (calibrated at request) 1 case for 11 supervisory thermometers 8700.3.01901 1 set of 11 supervisory thermometers (mentioned above) incl. case 8700.2.35200 Calibration liquids

2081.2.07200

2081.2.07400

2081.2.07700

2081.2.08000

2081.2.08100

Charles	Order No
Spares	
Fall tube* with gasket	2082.2.14800
Expedient to empty fall tube, ball catch, complete	2082.2.09100
Ball 1, complete* (ball in case)	2082.2.08100
Ball 2, complete* (ball in case)	2082.2.08300
Ball 3, complete* (ball in case)	2082.2.08400
Ball 4, complete* (ball in case)	2082.2.08500
Ball 5, complete* (ball in case)	2082.2.08600
Ball 6, complete* (ball in case)	2082.2.08700
Ball gage	2081.3.06301
Stopper I	2081.3.06311
Cover	2081.3.06312
Stopper II	2081.3.06313
Ball case	2082.2.06400
Gasket A 16 x 20 (Perbunan)	LP - 000001
Gasket	
	2082.3.06201
Packing	7530.3.01902
Rubber ring	2081.3.02001
Rubber packing	2081.3.02005
Water bath Jacket* with gasket	2082.2.14700
Thermometer screw	2081.2.02400
Ball pincette	2081.3.06204
Glass filter	2081.3.06205
Glass rod	2081.3.06206
Brush	2081.3.06207
Cleaning brush	2082.6.06203
Flat wrench	2081.3.06209
Cleaning rod	2081.2.06500

^{*} Using spares, determine again ball constant(s) to para 10. Therefore, also order calibration liquid concerned.

RHEOTEST Messgeräte Medingen GmbH



Measuring equipment

Viscometers for laboratory and industrial use

Rotary viscometer

Viscometer for laboratory RHEOTEST® RN
Viscometer for industrial use RHEOTEST® PR

Capillary viscometer

Viscometer for laboratory RHEOTEST® LK Viscometer for industrial use RHEOTEST® PK

•Ball pressure viscometer

Viscometer for laboratory HÖPPLER® KD 3.1

•Falling ball viscometer

Viscometer for laboratory HÖPPLER® KF 3.2

RHEOTEST Messgeräte Medingen GmbH

Medingen

Rödertalstrasse 1

D-01458 Ottendorf-Okrilla (GERMANY)

Phone: + 49 - (0) 35205 - 580 Fax: + 49 - (0) 35205 - 58297

e-Mail: Rheotest-Medingen@t-online.de

Internet: www.rheotest.de