

BROOKFIELD KF10 and KF20

Falling Ball Viscometer

Operating Instructions

Manual No. **M09-352-C0512**



with offices in: Boston • Chicago • London • Stuttgart • Guangzhou

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Table of Contents

I. INTRODUCTION	3
I.1 Components	3
I.2 Specifications	4
I.3 Details on Viscosity Measurement Range.....	5
I.4 Description of the Equipment	5
I.5 Safety Symbols and Precautions	8
I.6 Cleaning	8
II. GETTING STARTED.....	9
II.1 Choice of Balls	9
II.2 Filling the Sample Tube.....	9
II.3 Temperature Control of the Sample	10
II.4 Measuring the Falling Time	10
III. CALCULATIONS	12
III.1 Dynamic Viscosity	12
III.2 Kinetmatic Viscosity	13
IV. DETERMINATION OF THE NON-NEWTONIAN BEHAVIOR	14
IV.1 Thixotrophy/Rheopexy	14
IV.2 Structural Viscosity (Pseudoplasticity and Dilatancy).....	14
Appendix A - Maintenance	15
Appendix B - Calibration of the Ball Constants.....	16
Appendix C - Warranty Repair and Service	17

I. INTRODUCTION

Falling Ball Viscometer, Models KF10 and KF20, comply with the German industry standard DIN 53015. The measuring principle, according to Höppler, is to determine the falling time of a ball in a cylindrical glass tube filled with liquid. The working angle of the falling tube in the KF10 is fixed in the DIN 53015 position of 80° relative to horizontal. The water jacket, surrounding the falling tube, when connected to a Brookfield circulating temperature bath provides for precise temperature control of the sample.

The user calculates the dynamic viscosity of the sample by determining the falling time of the ball between the upper and lower ring marks displayed on the falling tube. Using data on the ball constants, the density difference between the liquid sample and the ball, and the working angle constant, a mathematical equation is used to convert the time measurement to a viscosity value in centipoise.

Note: The ball constants (forwards and backwards) and ball densities are listed on the test certificate accompanying the instrument. You must provide the density value for the liquid that you are testing.

The six (6) balls with different diameters and densities enable the KF10 to measure a wide range of viscosities. The ability to adjust the angle of inclination of the KF20 extends the measuring range for low viscosity liquids.

For non-Newtonian liquids, by subjecting the same sample to repetitive measurements with the KF20 at different angles, pseudoplastic or dilatant behavior may be determined as explained in Section 7. Time dependent behavior (thixotropy and rheopexy) may also be noted.

The ease of operation and precise temperature control, using a Brookfield circulating temperature bath, allows for very reproducible measuring results.

I.1 Components

Component	Part No.	Quantity
Falling Ball Viscometer:		1
KF10 at fixed DIN position of 80°	KF10	-
OR KF20 variable angle with position 80°, 70°, 60° 50°	KF20	-
Set of (6) balls with gauge (FB68) in a carrying case (FB22) Supplied w/certificate stating diameter and mass of each ball	FB21C	1
-Ball 1 (glass)	FB1	-
-Ball 2 (glass)	FB2	-
-Ball 3 (nickel and iron)	FB3	-
-Ball 4 (nickel and iron)	FB4	-
-Ball 5 (steel)	FB5	-
-Ball 6 (steel)	FB6	-
Ball Tweezers	FB51	1

Wire Cleaning Brush for Sample Tube	FB53	1
Brush to clean loose debris from Falling Balls	FB52	1
Leather cloth for polishing Falling Balls	FB70	1
Viscometer Seal A, 16mm x 20mm (Perbunane)	FB31	4
Thermometer Seal, 10mm x 14mm x 2mm (Silicone)	FB32	1
Thermometer, 0°C to +100°C*	TM1	1
Operator Manual	M09-352	1
Instrument Case	FB71	1
Latex rubber tubing, 5/16 I.D. x 1/16 wall	FB69	1
Certificate of Calibration	_____	1

*Other temperature measurement options are available. Contact your Brookfield dealer for information.

1.2 Specifications

Viscosity Range:	0.5 - 7×10^4 mPa•s (cP)
Falling Time-Lower Limit:	60 s for Ball No.1 30 s for Ball Nos. 2,3,4,5 and 6
Falling Time-Upper limit:	300 s <i>Materials with viscosity $> 7 \times 10^4$ mPa•s require running times of over 300 s.</i>
Measuring Distance:	100 mm (50 mm between adjacent ring marks) in both directions
Fall Tube Inner Diameter:	15.94mm +/- 0.01mm
Set of Balls:	6 balls
Working Angle:	KF10: 80° (DIN position) KF20: 80°, 70°, 60°, 50°
Temperature Range:	-60°C - +150°C
Sample Volume:	40 mL
Dimensions:	180 mm x 220 mm x 330 mm
Weight:	6.4 lbs, 2.9 kg (empty sample tube and empty water jacket)

I.3 Details on Viscosity Measurement Range

Per DIN 53015, the Falling Ball method is suitable for measuring dynamic viscosities ranging from 0.6 mPa·s to 250,000 mPa·s at temperatures from -60°C to 150°C. Use is made of six balls having different diameters, each ball covering part of the range. All guideline values and referenced parameters in the following table are per DIN 53015.

Ball No.	Viscosity measurement range (guideline value) (mPa·s)	Material (Recommendation)	Density (guideline value) (g/cm ³)	Ball diameter (mm)	Deviation from circularity (mm)	Calibration constant (guideline value) (mPa·s·cm ³ /g·s)
1	0.5 to 10	Borosilicate glass	2.4	15.81 ± .01	± 0.0005	0.007
2	9 to 140	Borosilicate glass	2.4	15.6 ± 0.05	± 0.0005	0.09
3	40 to 700	Ni/iron	8.1	15.6 ± 0.05	± 0.001	0.09
4	150 to 5,000	Ni/iron	8.1	15.2 ± 0.1	± 0.001	0.7
5	1,500 to 50,000	Ni/iron	8.1	14.0 ± 0.5	± 0.001	7
6	Above 7,500	Ni/iron	8.1	11.0 ± 1	± 0.002	35

I.4 Description of the Equipment

Refer to Figures 1 through 4. Specific items identified on the Falling Ball Viscometer are identified by parentheses () in the following steps:

1. The Falling Ball Viscometer must be level. The level is adjusted using the two Leveling Screws (4) on the base. Adjust so that the bubble level on top of the Falling Ball Viscometer is centered within the circle. Check level periodically during use.
2. a. The working angles of the KF20 are 80°, 70°, 60° and 50° relative to horizontal. The DIN 53015 working position of 80° is the preferred position. The different working angles are secured by a locking adjustment screw (5). To select a working angle, the adjustment screw (5) should be loosened by turning it counter-clockwise approximately one rotation. After the working angle is selected, the adjustment screw should then be tightened again.
b. The working angle of the KF10 Viscometer is fixed at the DIN 53015 position of 80° relative to horizontal.
3. The two running directions of the balls can be chosen by swivelling the viscometer, which is mounted in the stand, and is secured by a locking mechanism (14).
4. The sample tube (6) is surrounded by a water jacket which is fixed between the upper plate (7) and lower plate (8). The upper locking plug (16) with lid (20), the lower locking plug (17) and accompanying seals (gaskets and washers) (19), and the caps (18) are designed to perform the following functions within the sample tube:
 - a. keep the liquid sample tightly sealed

- b. eliminate the formation of air bubbles
 - c. avoid a build up of pressure (see Fig. 3 and 4)
5. Mounted on the lower plate are tubes (10) to which the circulating temperature bath tubing is attached.

NOTE: Any alteration, modification or replacement of the sample tube, water jacket, falling tube screw fittings, tension rods or balls renders the ball constants invalid and requires the re-calibration of the viscometer. See Appendix B.

6. The thermometer fastening screw (11) and sealing washer (13) with inserted thermometer is screwed into the screw neck (12) in the upper plate. The thermometer fastening screw should be tightened securely to prevent fluid leakage.
7. The primary function of the ball gauge is to distinguish the two glass balls (Ball No. 1 and Ball No. 2) from each other. Ball No. 1 will not pass through the ball gauge, whereas Ball No. 2 will pass through. The ball gauge may also be used to help identify Ball No. 2 through 4.

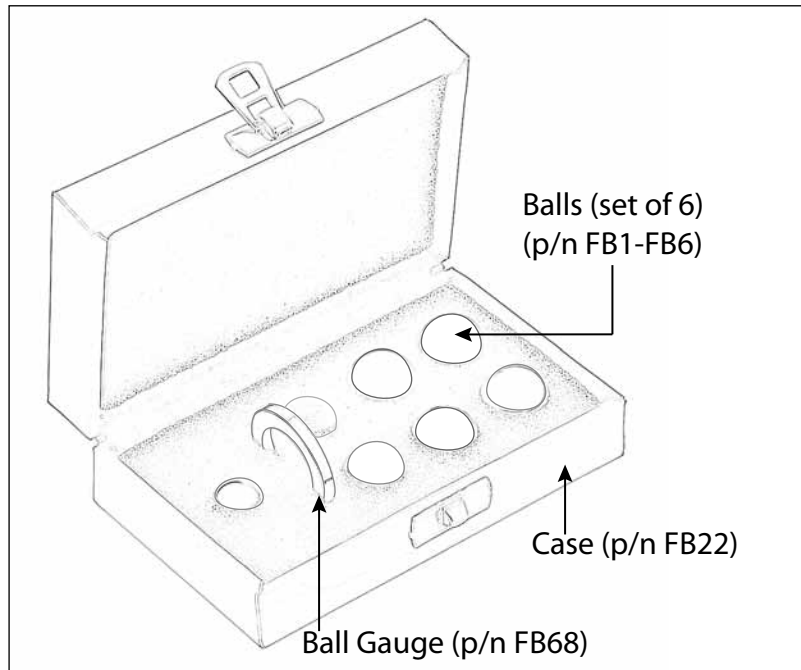


Figure 1

NOTES:

Ball diameters, weights, densities and ball constants (forwards and backwards) are listed in the test certificate accompanying the viscometer.

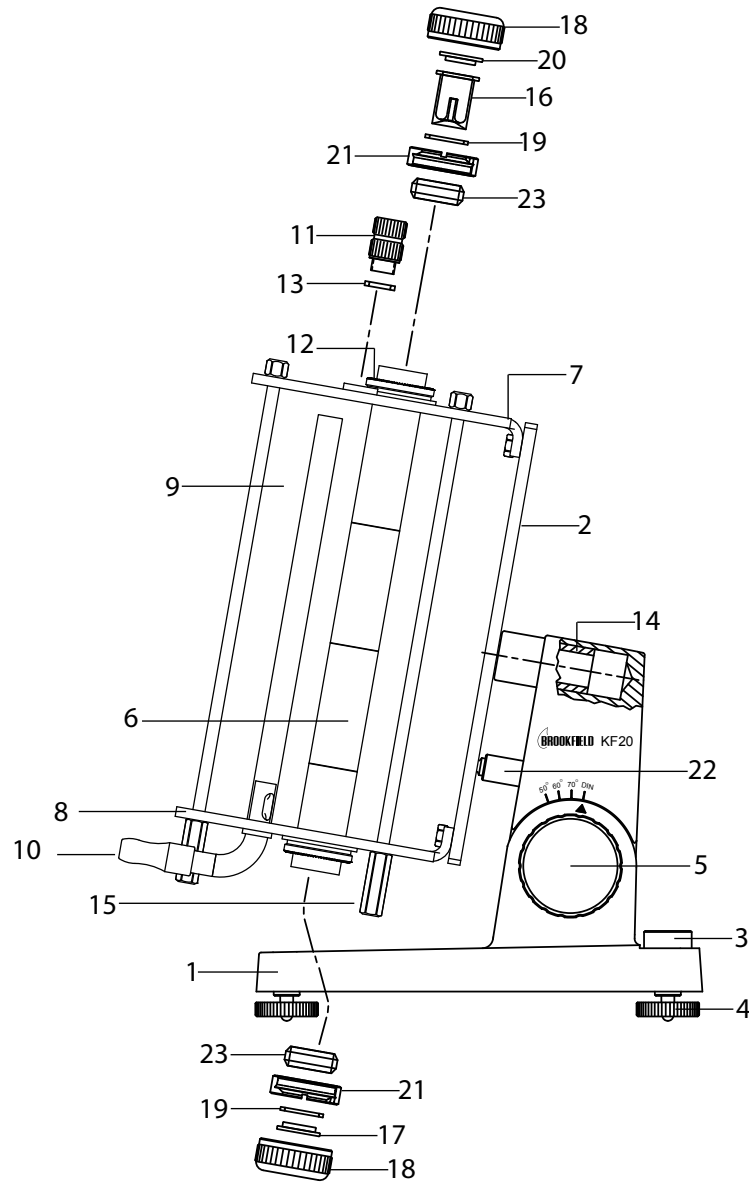


Figure 2: Falling Ball Viscometer KF20

1. Stand - KF10 (Part No. FB73) KF20 (Part No. FB74)	13. Thermometer seal, 10mm x 14mm x 2mm (silicone) (Part No. FB32)
2. Viscometer	14. Bearing for viscometer rotation
3. Bubble Level	15. Nuts
4. Leveling Feet (Part No. FB75)	16. Upper Locking Plug (Part No. FB64)
5. Adjustment screw for angle (KF20 Only)	17. Lower Locking Plug (Part No. FB66)
6. Glass Tube (15.94mm dia.) (Part No. FB61)	18. Cap (Part No. FB63)
7. Upper Plate	19. Viscometer seal A 16mm x 20mm (perbunane) (Part No. FB31)
8. Lower Plate	20. Lid (for upper locking plug) (Part No. FB65)
9. Waterbath Jacket (glass) (Part No. FB41)	21. Falling tube screw fitting (Part No. FB67)
10. Inlet port for connection to water bath	22. Stop to hold viscometer in position
11. Thermometer screw (Part No. FB42)	23. Seal for falling tube (Part No. FB62)
12. M10x1 Thermometer Attachment Thread	

I.5 Safety Symbols and Precautions

Safety Symbols

The following explains safety symbols which may be found in this operating manual.



Refer to the manual for specific warning or caution information to avoid personal injury or damage to the instrument.

Precautions



If this instrument is used in a manner not specified by the manufacturer, the protection provided by the instrument may be impaired.



This instrument is not intended for use in a potentially hazardous environment.



The user should ensure that the substances placed under test do not release poisonous, toxic or flammable gases at the temperatures to which they are subjected to during the testing.

I.6 Cleaning

Great care should be given to cleaning of the sample tube, the locking plugs and the balls. These components (material: glass, Ni-iron, steel, perbunan, silicone, chromium-plated surfaces) must not be damaged or subject to chemical action by the cleaning fluid.

The cleaning procedure is to be carried out in the following sequence:

- The viscometer is pulled out of the bearing guide (14) in the stand by turning it 90° and placed in a suitable collecting basin.
- Unscrew the caps and remove the locking plugs in such a way that the ball does not fall into the collecting basin (possibly damaging the ball). The ball collector (Part No. FB23) is an optional item that can be purchased on request.
- Clean the sample tube using a suitable cleaning agent for the material being measured with the cleaning brush (Part No. FB53).
- There must not be any residue remaining in the sample tube or on the balls after they have been cleaned and are dry. Wipe with a Kim wipe or cloth that will not leave fibers, if necessary.
- When cleaning, be careful that the cleaning agent does not come into contact with the equipment outside of the measuring tube (potential for varnish damage).

II. GETTING STARTED

II.1 Choice of Balls

The balls are chosen in such a way that the minimum falling time is not less than what is shown in the table and the maximum falling time is not greater than 300 s. The DIN 53015 indicates that a falling time greater than 300 s is allowed, but for practical reasons, a shorter test time makes more sense.

Ball No.	Diameter [mm]	Minimum falling time [s]	Lower measuring range limit [mPa·s]	Upper measuring range limit [mPa·s]
1	15.81	60	0.5	10
2	15.60	30	2.5	130
3	15.60	30	20	700
4	15.20	30	200	7800
5	14.00	30	1000	45000
6	11.00	30	5500	70000

The specifications for the ball constant and the ball density are taken from the test certificate which came with the equipment.

The exchange of balls or equipment components between different viscometers is not permitted. Otherwise, the ball constants lose their validity.

When the falling time for the ball is less than the minimum time, turbulence may occur.

II.2 Filling the Sample Tube

To fill the sample tube:

- The sample tube is locked on the lower plate with the lower locking plug, seal, and cap.
- The liquid is filled up to approximately 25 mm beneath the top of the sample tube without air bubbles. Use a glass filter to remove any impurities when introducing a liquid into the tube.
- The ball is polished with the leather cloth and fibres are removed with the small cleaning brush, before being inserted into the tube with the ball tweezers. The ball must not be touched after polishing. The ball will travel to the bottom of the tube. Possible air bubbles in the sample, or trapped below the ball, are removed with a suitable rod, by rotating the ball.
- Insert the upper locking plug with seal into the sample tube. In so doing, the sample must enter the inside of the upper locking plug through the opening. The upper locking plug must not be filled more than half way with the sample in order to minimize pressure build-up due

to the air bubbles. Due to the design of the upper locking plug, air bubbles cannot get into the sample tube.

- The sample must be free of air bubbles between the two locking plugs. The lid of the upper locking plug is attached and the temperature control (desired test temperature) must be achieved. **After proper temperature control is achieved, the upper cap is screwed on.**

 **Do not heat the sample with the upper lid attached as pressure may build in the tube.**

NOTE: Gas bubbles can be removed by warming up the sample for a short time (approximately 20 degrees above the measuring temperature with the upper locking plug removed) or by lightly tapping on the lid of the upper locking plug.

II.3 Temperature Control of the Sample

The following are suggested working fluids for the circulating temperature bath:

Temperature Range	Bath Working Fluids	Tubing
-60 to +20°C	Water (deionized) - glycol-mixture; mixed in accordance with the manufacturer's instructions for the temperature range	Insulated Latex tubing, secured with tube band clips
+1°C to +80°C	Distilled water	Latex tube
+80°C to +150°C	Transparent thermostatic oil	Insulated Fluran tube, secured with tube band clips

The tubing from the circulating bath should be pushed tightly onto the viscometer tubes. By pulling gently, check whether the tubing is firmly attached. Tubing and circulating bath are available on request from your Brookfield dealer. If the water bath jacket has condensation on the glass, rub with alcohol.

The sample tube is sealed with the upper cap after temperature set point has been achieved. Allow 30 minutes for thermal equilibrium.

Falling ball viscometers provide precise temperature control for the sample. By measuring your sample at multiple temperatures, you can determine the temperature viscosity curve.

II.4 Measuring the Falling Time

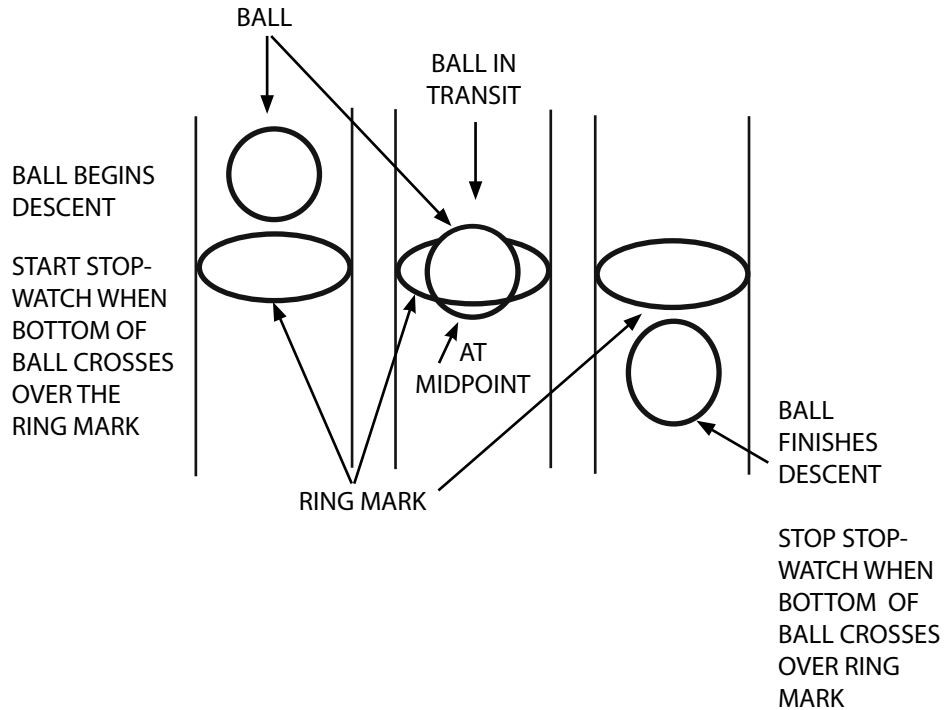
Before beginning the measurement, the upper cap is loosened (unscrewed) again to let off possible pressure.

The time which the balls take to run between the top and bottom ring marks in the sample tube is determined with a stop watch (resolution 0.01 s).

It is recommended that you record the passage of the lower ball point using the ring marks as follows. Position your eyes at the same height as the ring marks so that these appear as a line. A dark paper, placed behind the viscometer with its edge at the same height as the ring mark, shows the ball periphery more distinctly.

With dark liquids, you can better observe the ball in the sample tube by looking from behind the instrument.

Possible variations in the measuring times may be due to impurities in the sample, air bubbles or the fact that it has not been brought to the right temperature (insufficient temperature control). Even 0.1°C change in temperature is clearly measurable. The first forward and return passage of the ball can be used to achieve a thorough mixing (temperature equalization) of the sample before running the viscosity test.



III. CALCULATIONS

III.1 Dynamic Viscosity

With Newtonian liquids absolute values of the dynamic viscosity are calculated, whereas, for non-Newtonian liquids, relative values of the dynamic viscosity (apparent viscosity) are calculated.

The dynamic viscosity is calculated according to the following equation:

Equation 1:

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

where:

- η dynamic viscosity [mPa·s]
- t travelling time of the ball [s]
- ρ_1 density of the ball according to the test certificate [g/cm³]
- ρ_2 density of the sample [g/cm³]
- K ball constant according to test certificate [mPa·cm³/g]
- F working angle constant

Angle of inclination α (applied to the level)	Working angle constant F
80° (DIN)	1.0
70°	0.952
60°	0.879
50°	0.778

The density and ball constant are each stated in the test certificate.

Consideration for buoyancy of the ball in the sample is accounted for by means of $(\rho_1 - \rho_2)$ in equation (1).

The density of the sample can be determined by:

- referring to the material specifications from the manufacturer of the fluid
- measuring with a densitometer

Note: Be sure to measure the sample density at the same temperature at which the viscosity will be measured.

The density of the sample must be determined exactly when the amount $(\rho_1 - \rho_2)$ becomes small. The use of the glass ball requires the determination of the density of the sample ρ_2 to the 3rd decimal position in g/cm³. For metal balls, the 2nd decimal position is sufficient. For glass balls, the density of the measuring substance is determined to 0.001 g/cm³, for metal balls to 0.01 g/cm³.

III.2 Kinematic Viscosity

The conversion of the dynamic viscosity into the kinematic viscosity is accomplished using the following equation:

Equation 2:
$$v = \frac{\eta}{\rho_2}$$

v Kinematic viscosity [mm²/s]
 η Dynamic viscosity [mPa•s]
 ρ_2 Density of the sample [g/cm³]

IV. DETERMINATION OF THE NON-NEWTONIAN BEHAVIOR

Non-Newtonian behavior can be determined when different measurement times are recorded with repeated tests.

IV.1 Thixotropy/Rheopexy

Thixotropy (rheopexy) is indicated if the travelling times for a ball decreases (increases) when repeated measurements are made on the same volume of sample.

NOTE: If temperature control is not done correctly, thixotropy or rheopexy can be inferred by mistake.

Rotational or Capillary Viscometers should be used for better determination of flow behavior.

IV.2 Structural Viscosity (Pseudoplasticity and Dilatancy)

Using the KF20 Falling Ball Viscometer at different angles may serve to determine pseudoplastic or dilatant behavior for non-Newtonian liquids. If the sample is non-Newtonian, the travel time of the ball multiplied by the sine of the working angle for the KF20 will not remain constant. This infers non-Newtonian flow behavior. For pseudoplasticity, the calculated value decreases; for dilatancy, it increases.

NOTE: If temperature control is not done correctly, pseudoplasticity or dilatancy can be inferred by mistake.

Details about the relative values for pseudoplasticity and dilatancy are related to the diameter of the ball and the working angle.

More sophisticated equipment, like rotational viscometers/rheometers, should be used for detailed examination of non-Newtonian materials.

Appendix A - Maintenance

A.1 Exchanging the Sample Tube



Caution Glass Components. Excessive force may result in broken glass.

1. Loosen the two setscrews of the sample tube screw fittings (above and below).
 - a. Empty the water jacket and sample tube. Make sure the water jacket is clean on the inside surface before reassembling.
2. Unscrew both of the sample tube screw fittings using a ring nut key.
3. Pull off the rubber seal (washer) on one end of the sample tube.
4. Pull the sample tube out at the other end.
5. Insert the new sample tube and also wet the sample tube gaskets and washers.
6. Assemble the sample tube in the reverse order. Observe that the ends of the sample tube project evenly from the upper and lower plates.
7. Re-calibrate all the ball constants according to Appendix B.

A.2 Exchanging the Water Bath Jacket



Caution Glass Components. Excessive force may result in broken glass.

1. Remove the sample tube.
 - a. Empty the water jacket and sample tube. Make sure the water jacket is clean on the inside surface before reassembling.
2. Unscrew the upper and lower plates on the connecting bar.
3. Unscrew the three lower nuts on the viscometer.
4. Replace rubber washers and insert the new water bath jacket.
5. Put on the upper plate and screw down the nuts evenly.
6. Fix the lower plate an the lower connecting bar.
7. Assemble the sample tube.
8. Calibrate all the ball constants according to Appendix B.

A.3 Exchanging the Ball or the Viscometer

1. Exchange the balls or viscometer.
2. Re-calibrate the ball constants according to Appendix B.

Appendix B - Calibration of the Ball Constants

Brookfield's certification of the instrument is performed with Ball #2 and Ball #3 as well as using the Cannon N44 viscosity standard with a nominal value of 92mPa·s at 20°C.

Re-calibration of the ball constants is required if:

1. changes in the sample tube or water jacket were made
2. one or more balls were replaced

The calibration requires the use of a Newtonian viscosity standard.

Choice of the viscosity standard is according to the ball; the standards are provided in 500 mL bottles and can be obtained from your Brookfield dealer.

BALL NO.	1	2	3	4	5	6
Recommended Viscosity Standards (cP, mPa·s)	Cannon	Cannon	Cannon	Cannon	Cannon	N/A
	S3	N10	N100	N350	N1000	N/A
	3.9 cP @ 20°C	21 cP @ 20°C	283 cP @ 20°C	830 cP @ 20°C	2900 cP @ 20°C	N/A

The calibration is made according to the method in DIN 53015 at 20°C ± .05 C. A suitably calibrated thermometer can be obtained from your Brookfield dealer, on request.

Ball #6 is not recommended for use in performing a calibration check.

The ball constants are determined from (5) running times, in both forward and reverse direction.

The ball constant is calculated according to the following equation:

$$\text{Equation 3: } K = \frac{\eta}{(\rho_1 - \rho_2) \cdot t}$$

- t Mean value from 5 running times [s]
- η Dynamic viscosity of the calibrating fluid [mPa·s] at 20°C ± .05 C
- ρ₁ Density of ball [g/cm³]
- ρ₂ Density of calibrating fluid [g/cm³]

Note: Be sure to measure the sample density at the same temperature at which the viscosity will be measured.

The expected value of the constant should be similar to the constant stated in the test certificate.

For Ball No. 6, the ball constant changes significantly as a function of the falling tube diameter and diameter of the ball, so that the ball constant is calculated according to the following equation:

Equation 4:

$$K_6 = 1.4057(D - d_6) \left(0.75042 + 1.82637 \frac{d_6}{D} \right)$$

- D Falling tube diameter (see calibration certificate)
- d₆ Diameter of Ball No. 6 (see calibration certificate)
- K₆ Ball constant of Ball No. 6

Appendix C - Warranty Repair and Service

Brookfield Viscometers are guaranteed for one year from date of purchase against defects in materials and workmanship. They are certified using primary viscosity standards traceable to the National Institute of Standards and Technology (NIST). The Viscometer must be returned to **Brookfield Engineering Laboratories, Inc.** or the Brookfield dealer from whom it was purchased for no charge warranty service. Transportation is at the purchaser's expense. The Viscometer should be shipped in its carrying case together with all spindles and the guardleg if originally provided with the instrument.

For repair or service in the **United States** call Brookfield to obtain a Return Authorization Number. Record this number on the Repair Return Form which you can download from the Brookfield website. A tear cut copy is at the end of the manual. Return your instrument to:

Brookfield Engineering Laboratories, Inc.
11 Commerce Boulevard
Middleboro, MA 02346 U.S.A.

Telephone: (508) 946-6200 FAX: (508) 923-5009
www.brookfieldengineering.com

For repair or service outside the United States consult Brookfield Engineering Laboratories, Inc. or the dealer from whom you purchased the instrument.

For repair or service in the **United Kingdom** return to:

Brookfield Viscometers Limited
1 Whitehall Estate
Flex Meadow, Pinnacles West
Harlow, Essex CM19 5TJ, United Kingdom

Telephone: (44) 27/945 1774 FAX: (44) 27/945 1775
www.brookfield.co.uk

For repair or service in **Germany** return to:

RheoTec Messtechnik GmbH
(A Division of Brookfield)
Schutterwaelder Strasse 23
D-01458 Ottendorf-Okrilla, Germany

Telephone: (49) (035205) 5967-0 FAX: (49) (035205) 5967-30
www.rheotec.de

For repair or service in **China** return to:

Guangzhou Brookfield Viscometers and Texture Instruments Service Company Ltd.
Room C1, 5/F, Tianxing Building East Tower, No. 21, Zhongshan Yi Road, Yuexiu District
Guangzhou, 510600, P. R. China

Telephone: (86) 20/3760-0548 FAX: (86) 20/3760-0548
www.brookfield.com.cn

On-site service at your facility is also available from Brookfield. Please contact our Service Department in the United States, United Kingdom, Germany or China for details.

