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Second edition
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Petroleum and natural gas industries — Completion fluids and materials —

Part 1: Measurement of viscous properties of completion fluids

Industries du pétrole et du gaz naturel — Fluides de complétion et matériaux — Partie 1: Mesurage des propriétés visqueuses des fluides de complétion

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 13503-1 was prepared by Technical Committee ISO/TC 67, *Materials, equipment and offshore structures for petroleum, petrochemical and natural gas industries*, Subcommittee SC 3, *Drilling and completion fluids, and well cements*.

This second edition cancels and replaces the first edition (ISO 13503-1:2003), which has been technically revised. It also incorporates the Technical Corrigendum ISO 13503-1:2003/Cor.1:2005.

ISO 13503 consists of the following parts, under the general title *Petroleum and natural gas industries — Completion fluids and materials*:

— *Part 1: Measurement of viscous properties of completion fluids*

— *Part 2: Measurement of properties of proppants used in hydraulic fracturing and gravel-packing operations*

— *Part 3: Testing of heavy brines*

— *Part 4: Procedure for measuring stimulation and gravel-pack fluid leakoff under static conditions*

— *Part 5: Procedures for measuring the long-term conductivity of proppants*

— *Part 6: Procedure for measuring leakoff of completion fluids under dynamic conditions*

Introduction

For the purposes of this part of ISO 13503, completion fluids are defined as viscosified treating fluids used during the completion or workover of a petroleum- or natural-gas-producing well. The objective of this part of ISO 13503 is to provide a standard procedure for measuring the viscous properties of single-phase, non-particulate-laden completion fluids. These fluids are viscosified brines, gravel-pack carrier fluids, and fracturing fluids. These fluids can be either crosslinked or non-crosslinked (aqueous, hydrocarbon- or acid-based).

An optional shear-history simulation procedure is provided for fluids that are potentially shear-sensitive. This procedure is designed to simulate the shearing effects experienced by a fluid in surface apparatus and during the time it is being conveyed down the wellbore. Shear-history simulation is most often used during the development of new fracturing fluids to characterize their sensitivity to shear.

These standard procedures were compiled on the basis of several years of comparative testing, debate, discussion, and continued research by the industry.

This standard procedure is largely based on API RP 13M, first edition, July 2004.

In this part of ISO 13503, where practical, US Customary units (USC) are included in parentheses for convenience.

Petroleum and natural gas industries — Completion fluids and materials —

Part 1:
Measurement of viscous properties of completion fluids

1 Scope

This part of ISO 13503 provides consistent methodology for determining the viscosity of completion fluids used in the petroleum and natural gas industries. For certain cases, methods are also provided to determine the rheological properties of a fluid.

2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

2.1
bob
inner cylinder of a concentric-cylinder viscometer

2.2
completion fluid
viscosified treating fluid used during the completion or workover of a petroleum- or natural-gas-producing well

2.3
concentric-cylinder viscometer
rotational viscometer that consists of a concentric-cylindrical bob and a cylindrical rotor

2.4
elasticity
capability of a material to regain its original shape and condition upon removal of an acting stress

2.5
laminar flow
flow property of fluids in which all layers of the fluid move parallel to each other and no material is transferred between layers

2.6
non-crosslinked fluid
linear polymer-viscosified solution or any fluid that does not exhibit significant elasticity leading to the Weissenberg effect (bob climbing)

2.7
rheology
science of the deformation and flow of matter

2.8
rotor
outer rotating cylinder of a concentric-cylinder viscometer

2.9
shear history
sequence of shear rates and temperatures applied to fluids prior to and during measurements

2.10
shear-history simulator
apparatus used to simulate shear history in a fluid

2.11
shear rate
rate at which one particle of fluid is sliding by another particle divided by the distance between those particles

2.12
shear stress
force required to sustain fluid flow

2.13
viscoelastic fluid
crosslinked polymer solution or other fluid that exhibits significant elasticity, leading to the Weissenberg effect (bob climbing)

2.14
viscosity
measure of the internal friction of a fluid when caused to flow by an external force

3 Measurement and precision

Temperatures shall be measured to an accuracy of ±1 °C (±2 °F); pH shall be measured to an accuracy of ±0,1 units. All other quantitative measurements shall be made to an accuracy of ±2 %, unless specified otherwise.

4 Fluid preparation

Certain aspects of sample preparation and handling can affect the viscosity or rheological properties of a fluid. During all procedures, steps shall be taken to minimize entraining air into the fluid.

The procedure used to prepare the fluid sample shall be documented, including the following information:

a) description and/or composition of the base fluid; preparation of the fluid shall be described, starting with the fluid source, such as deionized water, tap water, completion brines, produced water, seawater or type of oil;

b) identification of mixing apparatus, container volume, and total volume of fluid prepared;

c) identification of each fluid component and amount added;

d) the order and method of addition of each component;

e) mixing speeds, with time at each speed;

f) ageing or holding time prior to measurements, if required;

g) temperature;

h) pH (for aqueous fluids);

i) all other aspects of the fluid preparation which are known to affect the outcome of the viscosity measurement, such as filtration of completion fluids.

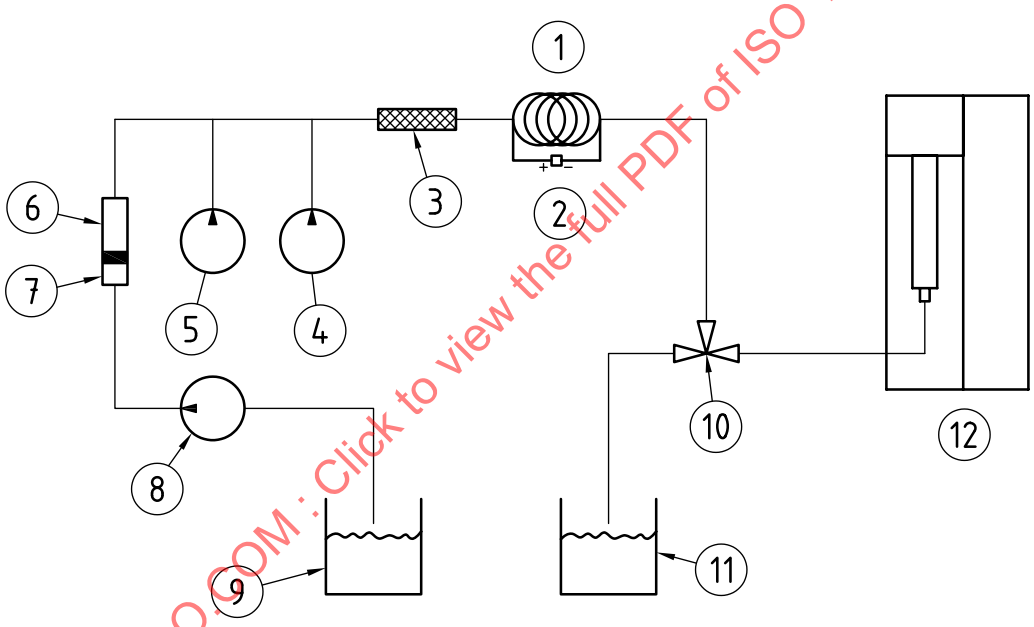
5 Fluid preparation using shear-history simulation (optional)

5.1 General

A shear-history simulation procedure is provided to simulate the effects of shear rate and time while a fluid is being conveyed down well tubulars. This procedure is intended to characterize the effect of shear history on fluid properties as part of the concept and development phase for a new fluid.

A shear-history apparatus is used to condition the fluid at specified shear rates, times and temperatures prior to injection into a viscometer. It consists of mixing apparatus, pumping apparatus and tubing to simulate significant aspects of the surface apparatus followed by shear conditions in the well tubulars. A shear-history apparatus that satisfies the requirements can be generically classified as a tube or pipe flow device that operates in the laminar flow regime. Flow shall occur in a single-pass mode.

A schematic diagram of a shear-history simulator connected to a pressurized concentric-cylinder viscometer is shown in Figure 1. In laminar flow, the energy dissipation rate is the same in any shear-history apparatus even if different tubing sizes are used. Thus, the design and functioning of the apparatus can vary and still meet the desired preconditioning criteria.



Key

1 tubing coil sized to provide shear rate and time
2 differential pressure measurement device (optional)
3 static mixing device
4 high-pressure injection for final additive, e.g. crosslinker or activator
5 high-pressure injection for second additive, if needed
6 base fluid (i.e. non-crosslinked) in piston accumulator
7 hydraulic oil from pump used to displace the base fluid
8 positive displacement pump
9 reservoir for hydraulic oil
10 flow diversion valve
11 collection container for fluid
12 pressurized concentric-cylinder viscometer

NOTE Based on the Chandler Model 5550 viscometer[1)].

Figure 1 — Shear-history simulation diagram

1) Chandler Model 5550 is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

5.2 Requirements for proper shear-history simulation

The following procedures shall be followed:

a) record and report the test temperature;

b) ensure thorough mixing of all fluid-activating additive(s) immediately before the fluid enters the shear-history tubing.

5.3 Conditions for sample delivery

The following conditions shall be fulfilled:

a) continuous delivery of base fluid while additives are added and the cup is being filled;

b) constant shear rate within the shear-history tubing;

c) while fluid is being injected into the viscometer, the shear rate within the gap of the viscometer is a nominal 100 s^{-1}.

5.4 Conditions for standard shear-history simulation

The following conditions shall be fulfilled:

a) for fluid temperatures less than or equal to 93 °C (200 °F), shear rate 675 s^{-1} for 2,5 min;

b) for fluid temperatures greater than 93 °C (200 °F), shear rate 1 350 s^{-1} for 5 min.

5.5 Operational considerations

The following conditions shall be fulfilled:

a) the pulsation caused by certain types of positive displacement pumps shall be minimized;

b) the base fluid shall be prepared, characterized and reported as described in Clause 5;

c) it is critical that a representative sample of the test fluid be injected into the viscometer; therefore, initially divert the fluid exiting the shear-history simulator away from the viscometer until stabilized flow and composition are established;

d) unions, valves and similar fittings shall have internal diameters such that the shear rate of the fluid flowing through them is essentially the same as within the tubing;

e) where the tubing is coiled, the diameter of the coil shall be larger than a critical value (see 8.5.2).

6 Instrument calibration

The instruments associated with these procedures shall be calibrated according to each manufacturer's recommended method.

7 Measurement procedures

7.1 General

The procedures given in 7.2 and 7.3 are organized according to the type of fluid on which the measurement is carried out. Where data are reported as being obtained using a particular procedure, the procedure given shall be followed exactly. The fluid shall not react with instrument surfaces to generate contaminants, change critical measurement dimensions, or impair proper mechanical operation.

7.2 Non-crosslinked fluids (see 2.6)

7.2.1 General

For proper rheological characterization of this type of fluid, the fluid shall wet the walls of the measuring chamber and remain within the annular gap.

7.2.2 Apparatus

For proper viscometric and rheological characterization, the apparatus used shall meet the following criteria:

a) the flow regime in the annular gap is laminar;

b) slippage of the fluid at the walls within the gap is negligible;

c) the fluid exhibits essentially time-independent behaviour during any given measurement.

7.2.2.1 Non-pressurized concentric-cylinder viscometer[2], to measure viscous and rheological properties at ambient pressure and at temperatures below the boiling point of the fluid.

Multiple-point measurements are required for the calculation of rheological parameters.

Any non-pressurized concentric-cylinder viscometer that is described by the following dimensions may be used (see Figure 2).

a) Rotor, R1:

   1) inside diameter equal to 36,83 mm (1,450 in);

   2) should be concentric with the bob and extend the full length of the bob;

   3) surfaces need to be smooth.

b) Bob, B1:

   1) diameter equal to 34,49 mm (1,358 in);

   2) cylinder length equal to 38 mm (1,496 in);

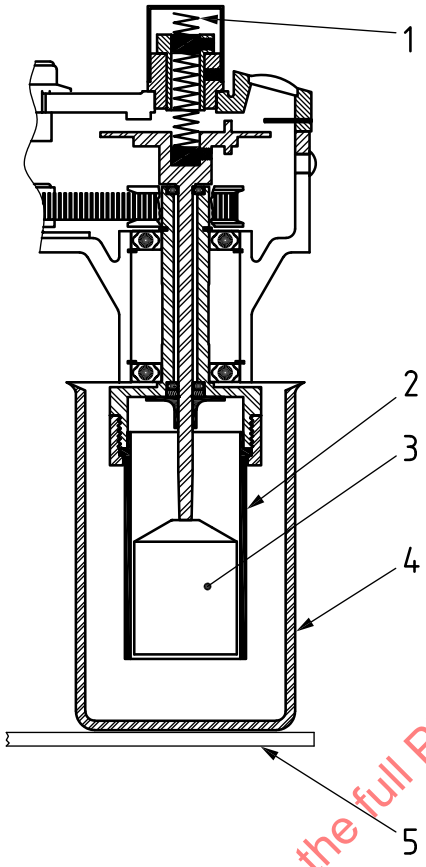
   3) cylindrical body with a flat, closed bottom and a tapered top with a truncated cone angle of 60°;

   4) surfaces need to be smooth.

c) Torsion spring:

   1) the equipment is usually supplied with a #1 spring; however, for less viscous fluids, a #0.2 spring may be appropriate.

2) Examples of non-pressurized concentric-cylinder viscometers are the Fann Model 35 viscometer equipped with rotor 1, bob 1 (R1B1) and appropriate spring; Chandler Model 3500 equipped with rotor 1, bob 1 (R1B1) and appropriate spring; OFI Model 800 equipped with rotor 1, bob 1 (R1B1) and appropriate spring; or viscometers with equivalent geometry. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.



Key

1 torsion spring
2 rotor R1
3 bob B1
4 sample cup
5 stage

Figure 2 — Geometry of a non-pressurized concentric-cylinder viscometer

7.2.2.1.1 Calibration

Calibration shall be carried out according to the manufacturer's recommended procedure, or using a standardized Newtonian calibration fluid traceable to an international/national standard such as ISO, ASTM, DIN, or equivalent.

Calibration oil viscosity shall be selected to encompass the shear rate and shear stress envelopes to be evaluated.

7.2.2.1.2 Operation

7.2.2.1.2.1 Preparation

Rotor and bob shall be properly aligned. All parts in contact with the fluid shall be at the same temperature as the fluid. Use of the standard cup provided by the manufacturer is recommended. Other vessels may be used; however, the vertical space between the bottom of the bob and bottom of the vessel shall be at least 13 mm (0,50 in).

7.2.2.1.2.2 Procedure

The non-crosslinked fluid sample to be tested shall be representative of the fluid as a whole, and air entrainment shall be minimal. After being placed in the viscometer, the fluid is stirred for 10 s to 15 s at the highest shear rate for which a measurement is to be made. Viscosity measurements should be made from lowest to highest shear rate. Record the average reading 20 s after the reading is stabilized at each shear rate.

7.2.2.1.3 Calculations

In order to convert a reading in revolutions per minute to the shear rate for the recommended R1B1 combination, use the following formula:

$$1 \text{ r/min} = 1{,}704 \text{ s}^{-1}$$

For shear stress at the bob, use 511 Pa (0,010 66 lb/100 ft^2) per degree of deflection.

Viscometric calculations shall be performed according to the manufacturer's specified procedure.

For rheological calculations, see Clause 8.

7.2.2.2 Pressurized concentric-cylinder viscometer[3)], to measure the viscous and rheological properties of fluids at elevated temperatures.

Pressurization minimizes the effect of entrained air on measured parameters and allows measurements to be made at temperatures above the atmospheric boiling point of the sample. Multiple-point measurements may be suitable for determining the rheological parameters of fluids.

Any pressurized concentric-cylinder viscometer that is described by the following dimensions may be used (see Figure 3).

a) Rotor, R1:

   1) inside diameter equal to 36,828 mm (1,450 in);

   2) should be concentric with the bob and extend the full length of the bob;

   3) surfaces need to be smooth.

b) Bob, B5:

   1) diameter equal to 31,934 mm (1,257 in);

   2) cylinder length equal to 76,17 mm (2,999 in);

   3) surfaces need to be smooth.

7.2.2.2.1 Calibration

Measure the temperature of the fluid being tested according to the manufacturer's specified procedure, which shall be traceable to a national/international standard such as ISO, ASTM, DIN, or equivalent.

Measure the rotor or sleeve speed according to the manufacturer's specified tachometer calibration procedure, which shall be traceable to a national/international standard such as ISO, ASTM, DIN, or equivalent.

3) Examples of pressurized concentric-cylinder viscometers are the Fann Model 50 viscometer equipped with rotor 1, bob 5 (R1B5); Chandler Model 5550 viscometer with rotor 1, bob 5 (R1B5), or viscometers with equivalent geometry. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

Use one of the following calibration methods.

a) Preferred method:

Verify the system using a standardized Newtonian calibration fluid traceable to a national/international standard such as ISO, ASTM, DIN, or equivalent. A calibration oil viscosity shall be selected to encompass the shear rate/shear stress envelope to be evaluated. The calibration shall be conducted at ambient pressure.

NOTE While the compressibility of aqueous fluids is not significantly affected by the pressure, some calibration oils, in particular silicone oils, are affected by pressure.

b) Alternative torque-only calibration:

Measure according to the manufacturer's specified calibration procedure (e.g. hanging weight), which shall be traceable to a national/international standard such as ISO, ASTM, DIN, or equivalent.

7.2.2.2.2 Operation

7.2.2.2.2.1 Instrument preparation

Pre-heat the thermal bath (if so equipped) to test temperature. All temperatures in this part of ISO 13503 refer to the actual temperature of the fluid.

7.2.2.2.2.2 Procedure

The following procedures shall be observed.

a) Loading, pressurizing and heating the fluid:

Load the fluid to be evaluated into the viscometer immediately after the last component is added according to the mixing procedure. Supply sufficient volume to fully cover the bob. Pressurize the system with nitrogen to a minimum of 2,75 MPa (400 psi) and immediately start shearing at 100 s^{-1}. When shearing of the fluid starts, define the elapsed time as zero ($t = 0$) and begin heating the fluid. All actions in this paragraph shall be completed within 45 s.

Optionally, for an ambient-temperature shear ramp [described in 7.2.2.2.2.2 b)], elapsed time is defined as zero ($t = 0$) immediately after completing this ramp, and fluid heating is then begun.

At 20 min elapsed time, the fluid temperature shall be no lower than 5 % below (base = 0 °C), and no higher than 3 °C (+5 °F) above, the desired test temperature. In addition, at 30 min elapsed time, and for the remainder of the test, the fluid temperature shall be within ±3 °C (±5 °F) of the test temperature.

b) Application of shear rate ramps:

The fluid shall be sheared at a constant 100 s^{-1} initially and between shear rate ramps.

The time reported for each shear rate ramp is the total time elapsed when the ramp begins. Starting at $t = 20$ min, shear rate ramps shall begin every 15 min up to $t = 2$ h 5 min. Beginning at $t = 2$ h 35 min and continuing up to 4 h 5 min, ramps shall begin every 30 min. After 4 h 5 min, the time elapsed when ramps begin is at the discretion of the operator; however, these shall be reported.

The specified shear rates for all shear rate ramps are 25 s^{-1}, 50 s^{-1}, 75 s^{-1} and 100 s^{-1}. The shear rates during a ramp shall occur in the sequence specified; however, the sequence of rates may be either monotonically increasing or decreasing. Following each change in shear rate, the fluid shall be allowed to equilibrate for 25 s. This is followed by 5 s of data collection. Each new shear rate shall be attained within the first 5 s following completion of data collection at the previous shear rate. When a sequence of increasing shear rates is used, a 40 s equilibration period shall be allowed before collecting data at 25 s^{-1}. Then, proceed as described above. Table 1 shows the viscometer speed, in revolutions per minute, corresponding to each shear rate based on the specified viscometer geometry.

c) Data reporting:

For each shear rate during a shear rate ramp, record viscosity data at least once per second and report the arithmetic average of the data obtained. At all other times, report viscosity data at least once per minute.

Table 1 — Viscometer speed and corresponding shear rate

Viscometer speed r/min	Shear rate s^{-1}
29,40	25
58,80	50
88,20	75
117,6	100

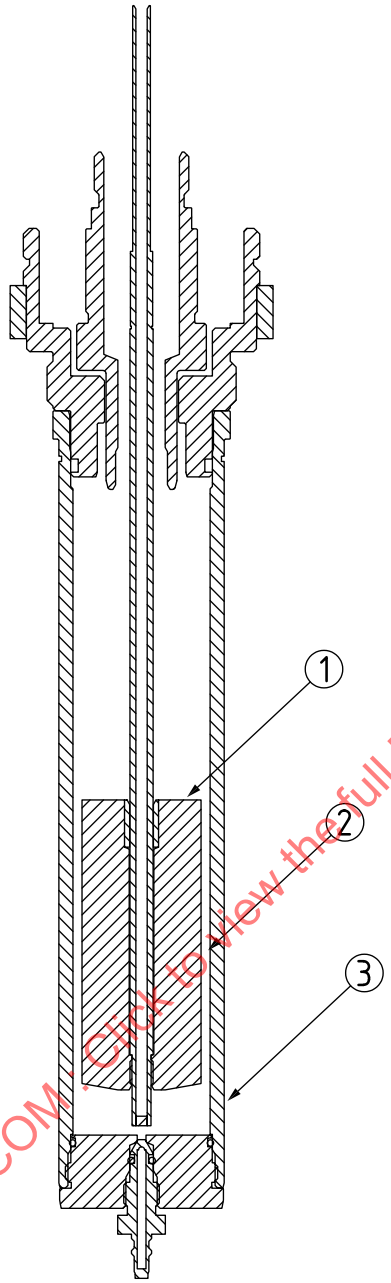
7.2.2.2.3 Calculations

In order to convert a reading in revolutions per minute to a shear rate, use the following formula:

$$1 \text{ r/min} = 0{,}850\ 3\ s^{-1}$$

Viscometric calculations shall be made according to the manufacturer's recommended calculation procedure.

For rheological calculations, see Clause 8.



Key

1 bob B5
2 measurement gap
3 rotor R1/sample cup

Figure 3 — Geometry of a pressurized concentric-cylinder viscometer

7.3 Crosslinked polymer and surfactant fluids

7.3.1 General

For proper rheological characterization of these types of fluids, the fluid shall wet the walls of the measuring chamber and remain within the annular gap.

7.3.2 Apparatus

The properties of viscoelastic surfactant fluids shall be measured in a pressurized viscometer with a relatively wide gap. Pressurization of the viscometer minimizes the effect of entrained air and reduces the Weissenberg effect (bob climb). Multiple-point measurements are suitable for determining the rheological behaviour of fluids.

For proper viscometric and rheological characterization, the following criteria shall be met:

a) the flow regime in the annular gap is laminar;

b) slippage of the fluid at the walls within the gap is negligible;

c) the fluid exhibits essentially time-independent behaviour during any given measurement.

7.3.2.1 Pressurized concentric-cylinder viscometer, with the dimensions specified in 7.2.2.2.

7.3.2.1.1 Calibration

Measure the temperature of the fluid being tested according to the manufacturer's specified calibration procedure, which shall be traceable to a national/international standard such as ISO, ASTM, DIN, or equivalent.

Measure the rotor speed according to the manufacturer's specified tachometer calibration procedure, which shall be traceable to a national/international standard such as ISO, ASTM, DIN, or equivalent.

Use one of the following calibration methods.

a) Preferred method:

   Verify the system using a standardized Newtonian calibration fluid traceable to a national/international standard such as ISO, ASTM, DIN, or equivalent. A calibration oil viscosity shall be selected to encompass the shear rate/shear stress envelope to be evaluated.

b) Alternative torque-only calibration:

   Measure according to the manufacturer's specified calibration procedure (e.g. hanging weight), which shall be traceable to an international/national standard such as ISO, ASTM, DIN, or equivalent.

7.3.2.1.2 Operation

7.3.2.1.2.1 Preparation

Pre-heat the thermal bath (if so equipped) to test temperature. All temperatures in this part of ISO 13503 refer to the actual temperature of the fluid.

7.3.2.1.2.2 Procedure

The following procedures shall be observed.

a) Loading, pressurizing and heating the fluid:

   Load the fluid to be evaluated into the viscometer immediately after the last component is added according to the mixing procedure. Supply sufficient volume to fully cover the bob. Pressurize the system with

nitrogen to a minimum of 2,76 MPa (400 psi) and immediately start shearing at 100 s^{-1}. When shearing of the fluid starts, the elapsed time is defined as zero ($t = 0$) and heating of the fluid is begun. All actions in this paragraph shall be completed within 45 s.

Optionally, for an ambient-temperature shear ramp [described in 7.3.2.1.2.2 b)], elapsed time is defined as zero ($t = 0$) immediately after completing this ramp, and fluid heating is then begun.

At 20 min elapsed time, the fluid temperature shall be no lower than 5 % below (base = 0 °C) and no higher than 3 °C (+5 °F) above the desired test temperature. In addition, at 30 min elapsed time, and for the remainder of the test, the fluid temperature shall be within ±3 °C (±5 °F) of the test temperature.

b) Application of shear rate ramps:

The fluid shall be sheared at a constant 100 s^{-1} initially and between shear rate ramps (see Table 1).

The time reported for each shear rate ramp is the elapsed time before the ramp begins. Starting at $t = 20$ min, shear rate ramps shall begin every 15 min up to $t = 2$ h 5 min. Beginning at $t = 2$ h 35 min and continuing up to 4 h 5 min, ramps shall begin every 30 min. After 4 h 5 min, the elapsed time before ramps begin is at the discretion of the operator; however, these shall be reported.

The specified shear rates for all shear rate ramps are 25 s^{-1}, 50 s^{-1}, 75 s^{-1} and 100 s^{-1}. The shear rates during a ramp shall occur in the specified sequence; however, the sequence of rates may be either monotonically increasing or decreasing. Following each change in shear rate, the fluid shall be allowed to equilibrate for 25 s. This is followed by 5 s of data collection. Each new shear rate shall be attained within the first 5 s following completion of data collection at the previous shear rate. When a sequence of increasing shear rates is used, a 40 s equilibration period shall be allowed before collecting data at a shear rate of 25 s^{-1}. Then, proceed as described above. Table 1 shows the viscometer speed, in revolutions per minute, corresponding to each shear rate based on the specified viscometer geometry.

c) Data reporting:

For each shear rate during a shear rate ramp, record viscosity data at least once per second and report the arithmetic average of the data obtained. At all other times, report viscosity data at least once per minute.

7.3.2.1.3 Calculations

Viscometric calculations shall be made according to the manufacturer's recommended calculation procedure.

For rheological calculations, see Clause 8.

8 Calculation procedures

8.1 General concepts

It is assumed that the fluid is homogeneous, with power-law behaviour as shown in Equation (1):

$$\tau = K\dot{\gamma}^{n} \tag{1}$$

where

τ is the shear stress;

K is the fluid consistency index;

$\dot{\gamma}$ is the shear rate;

n is the flow behaviour index.

8.2 Brief review of geometry-independent rheology versus nominal rheology

8.2.1 For a power-law fluid, the shear rate at the measurement surface depends on the geometry of the viscometer and on the flow behaviour index. The shear rate can be approximated using Newtonian behaviour, and this shear rate is known as the nominal Newtonian shear rate. The consistency index determined using the shear stress and the viscometer nominal shear rate is designated K_v. Similarly, the consistency indices are designated K_p and K_s, respectively, when the power-law model is expressed in terms of the nominal shear rate in a pipe and a slot (e.g. a fracture). The nominal shear rate and geometry-dependent consistency index may be converted, respectively, to the actual shear rate at the measurement surface and the geometry-independent consistency index, K, using the flow behaviour index.

8.2.2 Apparent (Newtonian) viscosity, μ_v, for the viscometer is calculated using a specific nominal shear rate expression with the corresponding geometry-dependent consistency index. Apparent viscosity values will differ between geometries. Although apparent viscosity differs between geometries, consistent shear stress values will be calculated using nominal shear rate with the appropriate geometry-dependent consistency index. Therefore, the power law expressed in terms of either a geometry-dependent or a geometry-independent consistency index will provide the proper shear stress value and, hence, pressure loss in the selected geometry.

8.2.3 The calculation approach used in this subclause is based on using nominal shear rate in the viscometer for data reduction, then converting the fluid consistency index, K_v, to the geometry-independent consistency index, K. Equations (2), (3) and (4) are provided for converting geometry-independent K to geometry-dependent consistency indices K_p and K_s for pipe and slot flows, respectively.

a) Basic equations:

$$\tau = K_v \left(\dot{\gamma}_n \right)^n = K \dot{\gamma}^n \tag{2}$$

$$\mu_v = \frac{\tau}{\dot{\gamma}_n} = K_v \dot{\gamma}_n^{(n-1)} \tag{3}$$

$$\mu = \frac{\tau}{\dot{\gamma}} = K \dot{\gamma}^{(n-1)} \tag{4}$$

where

τ is the shear stress, in mPa (lbf/ft^2);

K_v is the geometry-dependent consistency index, in mPa·s^n (lbf·s^n/ft^2);

$\dot{\gamma}_n$ is the nominal (Newtonian) shear rate, in s^{-1};

K is the geometry-independent consistency index, in mPa·s^n (lbf·s^n/ft^2);

$\dot{\gamma}$ is the shear rate, in s^{-1};

n is the power-law flow behaviour index, dimensionless;

μ_v is the nominal viscosity, in mPa·s (cP);

μ is the viscosity, in mPa·s (cP).

b) Calculation of viscosity, using SI units for K, with Equation (5):

$$\mu = K\dot{\gamma}^{(n-1)} \tag{5}$$

where

μ is the viscosity, in mPa·s (cP);

K is the geometry-independent consistency index, in mPa·s^n (lbf·s^n/ft^2);

$\dot{\gamma}$ is the shear rate, in s^{-1};

n is the power-law flow behaviour index, dimensionless.

8.3 Limitations/problems that can produce erroneous results

Non-power-law behaviour over the shear rate measurement range will be exhibited if the following occur:

a) change in power-law indices versus shear rate;

b) slip (non-homogeneous) flow, due to

1) fluids with high normal forces (e.g. highly elastic fluids), which may climb out of the gap in a concentric-cylinder viscometer,

2) an under- or over-filled viscometer cup,

3) thixotropic fluids, where the breakdown of internal structure is a function of time as well as shear rate,

4) the tendancy of rheopectic material to build up structure with time while being sheared at a constant rate.

8.4 Calculation method for concentric-cylinder viscometers

8.4.1 General

The following procedures may be used for data reduction and analysis when working with viscometers that are not automated.

8.4.2 Calculation of shear stress from torque values

Helical torsion springs are typically attached to the stationary cylinder (bob) in concentric-cylinder viscometers. Torque applied to the stationary cylinder by the fluid couple within the gap causes the torsion spring to deflect. The deflection is detected either electronically or visually through a dial reading.

Torque applied by the fluid couple within the gap can be determined using Equation (6):

$$M = c \cdot \theta \tag{6}$$

Torque is also force acting through a distance; see Equation (7):

$$M = F \cdot R_b \tag{7}$$

where

M is the torque, in N·m (lbf·ft);

c is the spring constant, in N·m/rad (lbf·ft/deg);

θ is the spring deflection, in rad (deg);

F is the shear force tangential to the cylinder surface, in N (lbf);

R_b is the outside radius of the bob, in m (ft).

A force balance allows the shear stress to be calculated from torque measurements; see Equations (8) and (9):

$$\tau A = M / R_b \tag{8}$$

$$\tau = M / 2\pi \cdot R_b^2 \cdot l \tag{9}$$

where

τ is the shear stress acting on the bob, in mPa (lbf/ft^2);

A is the surface area of the bob, in m^2 (ft^2);

l is the stationary inner cylinder length, in m (ft).

Many manufacturers supply interchangeable torsion springs of various strengths, which allow the instrument to be used over a broader range of viscosity. Factors for these springs are also available in their literature. Manufacturers' literature should also be consulted if the instrument uses any means other than a torsion spring to sense torque.

8.4.3 Nominal shear rate from angular velocity

The angular velocity, expressed in radians per second, is converted to nominal shear rate at the surface of the stationary inner cylinder (bob) using Equation (10):

$$\dot{\gamma}_n = 2\omega / [1 - (R_b / R_c)^2] \tag{10}$$

where

$\dot{\gamma}_n$ is the nominal shear rate at the surface of the bob, in s^{-1};

ω is the angular velocity of the rotor, in rad/s (deg/s);

R_b is the outside radius of the bob, in m (ft);

R_c is the inner radius of the rotor, in m (ft).

8.4.4 Consistency index calculation

For each shear rate ramp, perform a logarithmic linear regression of the power-law, expressed in terms of the viscometer consistency index, using Equation (11):

$$\log_{10} \tau = \log_{10} K_v + n \log_{10} \dot{\gamma}_n \tag{11}$$

which is of the form given in Equation (12):

$$y = ax + b \tag{12}$$

where

y is the $\log_{10}(\tau)$;

x is the $\log_{10}(\dot{\gamma}_n)$;

b is the $\log_{10}(K_v)$ at 1 s^{-1};

a is the slope of line, dimensionless;

τ is the shear stress, in mPa (lbf/ft^2);

$\dot{\gamma}_n$ is the nominal shear rate, in s^{-1}.

The intercept, b, resulting from the regression analysis can be converted to K_v using the following method.

Using base-10 logarithms,

$$K_v = 10^b \text{ mPa·s}^n \text{ (lbf·s}^n/\text{ft}^2) \tag{13}$$

The coefficient of determination, R^2, shall be reported for each calculation of n and K_v. K_v may be converted to $lbf·s^n/ft^2$ by dividing by 478,8.

8.4.5 Fluid consistency index calculation

Calculate the geometry-independent fluid consistency index, K, from the viscometer-specific K_v:

$$K = K_v\{[1 - (R_b / R_c)^2] / n[1 - (R_b / R_c)^{2/n}]\}^{-n} \tag{14}$$

The consistency index, K_s, for a slot (e.g. fracture) can be calculated using Equation (15):

$$K_s = K\left[\frac{2n+1}{3n}\right]^n \tag{15}$$

and that for a pipe, K_p, is calculated using Equation (16):

$$K_p = K\left[\frac{3n+1}{4n}\right]^n \tag{16}$$

8.5 Calculations for optional shear-history simulation

8.5.1 Flow rate and tubing length requirement

To keep a shear-history simulator within a reasonable size, a tubing internal diameter (ID) in the range of 0,002 m to 0,008 m [0,080 in to 0,305 in] is recommended. Once the ID of the tubing has been selected, the