

Recommended Practice for the Measurement of Viscous Properties of Completion Fluids

**ANSI/API Recommended Practice 13M
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**ISO 13503-1 Petroleum and natural gas industries—
Completion fluids and materials—
Part 1: Measurement of viscous properties of
completion fluids**



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API Foreword

This standard replaces API Recommended Practice RP 39 Recommended Practices on Measuring the Viscous Properties of Cross-linked Water-based Fracturing Fluids, 3rd Edition, May 1998.

This standard shall become effective on the date printed on the cover but may be used voluntarily from the date of distribution.

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Standards referenced herein may be replaced by other international or national standards that can be shown to meet or exceed the requirements of the referenced standard.

In this American National standard, editorial changes have been made and are listed in Annex A.

The modifications have not been changed in the body of this standard, but are noted by an arrow (➔) in the margin for reference to Annex A.

Suggested revisions are invited and should be submitted to the API, Standards Department, 1220 L Street, NW, Washington, DC 20005, or by email to standards@api.org.

This American National Standard is under the jurisdiction of the API Subcommittee 13, Drilling and Completion Fluids. This standard is considered identical to the English version of ISO 13503-1. ISO 13503-1 was prepared by Technical Committee ISO/TC 67, Materials, equipment and offshore structures for petroleum and natural gas industries, Subcommittee SC3, Drilling and completion fluids, and well cement.

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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*.

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*

The following part is under preparation:

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

Introduction

For the purpose 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 39, third edition, May 1998 [1].

In this part of ISO 13503, where practical, U.S. Customary units 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

fixed inner cylinder of a concentric-cylinder viscometer

2.2

completion fluid

any fluid used during the completion phase of a 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**shear history**

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

2.9**shear-history simulator**

apparatus used to simulate shear history in a fluid

2.10**shear rate**

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

2.11**shear stress**

force required to sustain fluid flow

2.12**viscoelastic fluid**

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

2.13**viscosity**

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

3 Abbreviated terms

r/min revolutions per minute

pH negative logarithm (to the base 10) of hydrogen ion concentration

ASTM American Society for Testing Materials

DIN Deutsches Institut für Normung

4 Measurement and precision

Temperatures shall be measured to an accuracy of $\pm 1\text{ }^{\circ}\text{C}$ ($\pm 2\text{ }^{\circ}\text{F}$); pH shall be measured to an accuracy of $\pm 0,1$ units. All other quantitative measurements shall be made to an accuracy of $\pm 2\%$, unless specified otherwise.

5 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. Following preparation, all fluids, except those intended to be used as fracturing fluids, shall be filtered through a filter of pore diameter $2\text{ }\mu\text{m}$. Minimize the entrainment of air during the filtration process.

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, seawater (location), 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 (required only for fracturing fluids);
- h) pH (for aqueous fluids, where applicable);
- i) all other aspects of the fluid preparation which are known to affect the outcome of the viscosity measurement should be reported.

6 Fluid preparation using shear-history simulation (optional)

6.1 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.

6.2 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.

6.3 Conditions for sample delivery

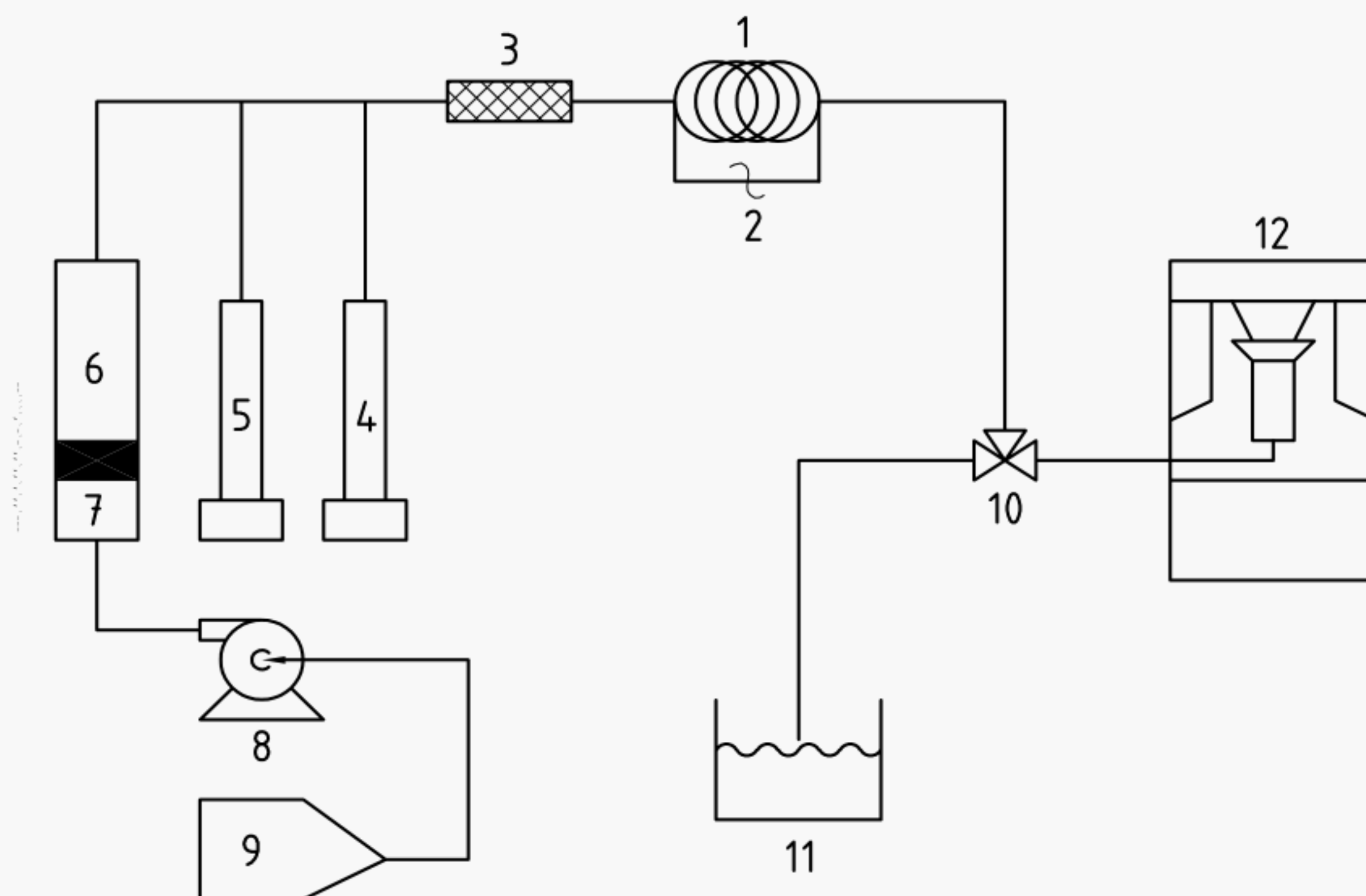
The following conditions shall be fulfilled:

- a) continuous delivery of base fluid while additives are added and cup is being filled;
- b) constant shear rate within the shear-history tubing;
- c) while fluid is being injected into the viscometer, shear rate within gap of the viscometer is a nominal 100 s^{-1} .

6.4 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 $1\,350 \text{ s}^{-1}$ for 5 min.

**Key**

- 1 tubing coil
- 2 differential pressure measurement device (optional)
- 3 static mixing device
- 4 high-pressure syringe pump for final additive e.g. crosslinker or activator
- 5 high-pressure syringe pump for second additive, if needed
- 6 base (e.g. uncrosslinked) fluid in floating-piston accumulator
- 7 oil from pump moving floating piston, which in turn moves base fluid
- 8 positive displacement pump
- 9 reservoir for pump oil
- 10 flow diversion valve
- 11 container for fluid
- 12 pressurized concentric-cylinder viscometer

Figure 1 — Shear-history diagram**6.5 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 9.6.2).

7 Instrument calibration

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

8 Measurement procedures

8.1 General

The procedures given in this clause are organized based on 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.

8.2 Non-crosslinked fluids (see 2.6)

8.2.1 Introduction

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.

8.2.2 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.

8.2.2.1 Non-pressurized concentric-cylinder viscometer¹⁾, to measure viscous and rheological properties at ambient pressure and at temperatures below the boiling point of the fluid.

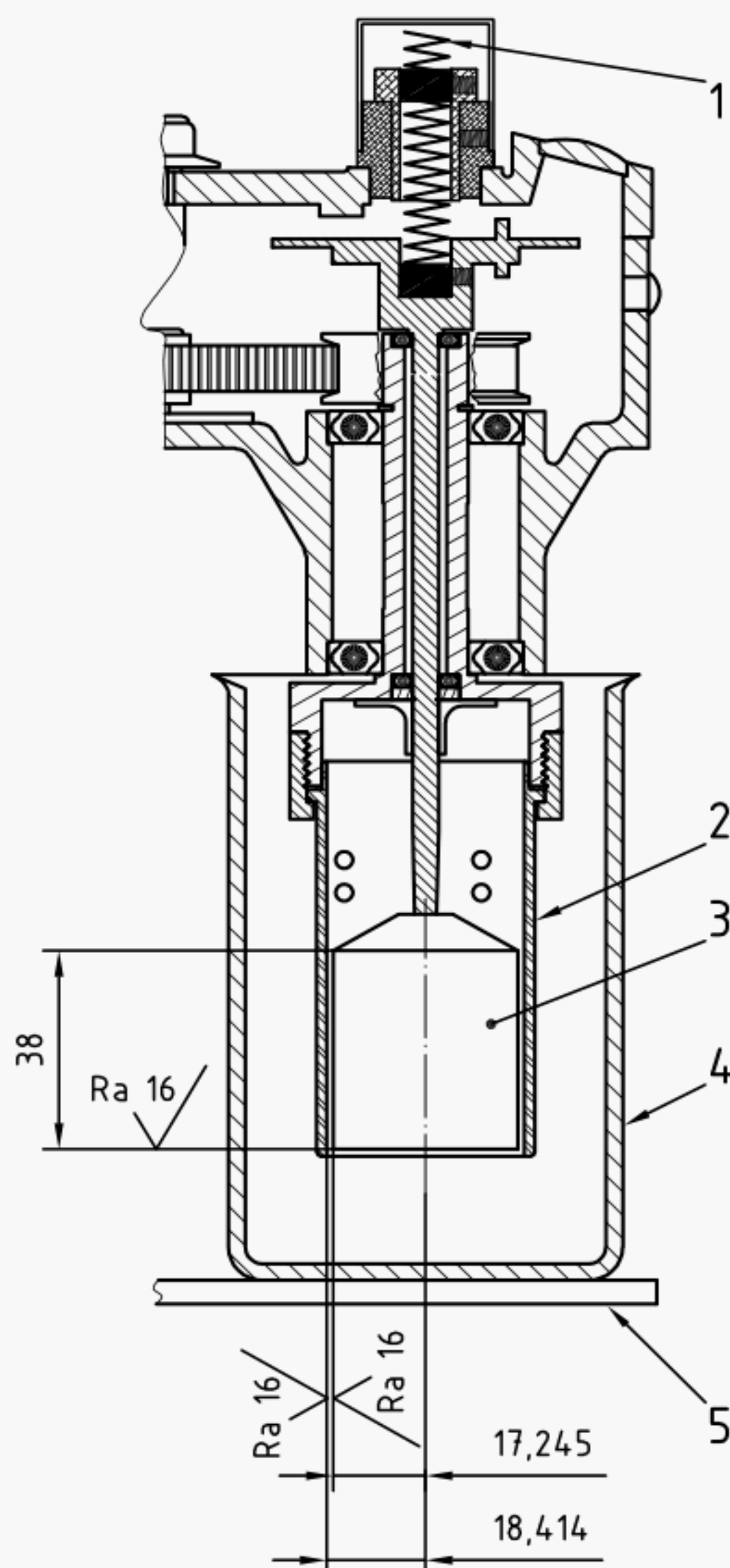
Multiple-point measurements may be suitable for the calculation of rheological parameters.

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

- a) rotor (or sleeve)
 - 1) inside diameter: 36,83 mm (1,450 in),
 - 2) should be concentric with bob and extend the full length of bob;
- b) bob
 - 1) diameter: 34,49 mm (1,358 in),
 - 2) cylinder length: 38 mm (1,496 in),
 - 3) cylindrical body with a flat, closed bottom and a tapered top with a truncated cone angle of 60° degrees.

1) 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 part of ISO 13503 and does not constitute an endorsement by ISO of these products.

Dimensions in millimetres

**Key**

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

Figure 2 — Geometry of a non-pressurized concentric-cylinder viscometer**8.2.2.1.1 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.

8.2.2.1.2 Operation

8.2.2.1.2.1 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).

8.2.2.1.2.2 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 the highest shear rate. Record the average reading 20 s after reading is stabilized at each shear rate.

8.2.2.1.3 Calculations

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

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

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

For rheological calculations, see Clause 9.

8.2.2.2 Pressurized concentric-cylinder viscometer²⁾, to measure the viscous and rheological properties of completion 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 with the dimensions shown in Figure 3 may be used.

8.2.2.2.1 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.

Use one of the following calibration methods:

a) preferred method

Verify 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 are not significantly affected by the pressure, some calibration oils, in particular silicone oils, are affected by pressure.

2) Examples of pressurized concentric-cylinder viscometers are the Fann Model 50 viscometer equipped with rotor 1, bob 5 (R1B5); Nordman Model 5001 equipped with rotor 1, bob 5 (R1B5); or viscometers with equivalent geometry. This information is given for the convenience of users of this part of ISO 13503 and does not constitute an endorsement by ISO of these products.

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.

8.2.2.2.2 Operation**8.2.2.2.2.1 Instrument preparation**

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

8.2.2.2.2.2 Procedure

The following procedures shall be followed.

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 mixing procedure. Place 52 cm³ of fluid in the viscometer. This volume is sufficient 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 8.2.2.2.2 b)], elapsed time is defined as zero ($t = 0$) immediately after completing this ramp, and fluid heating is 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 after completing 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

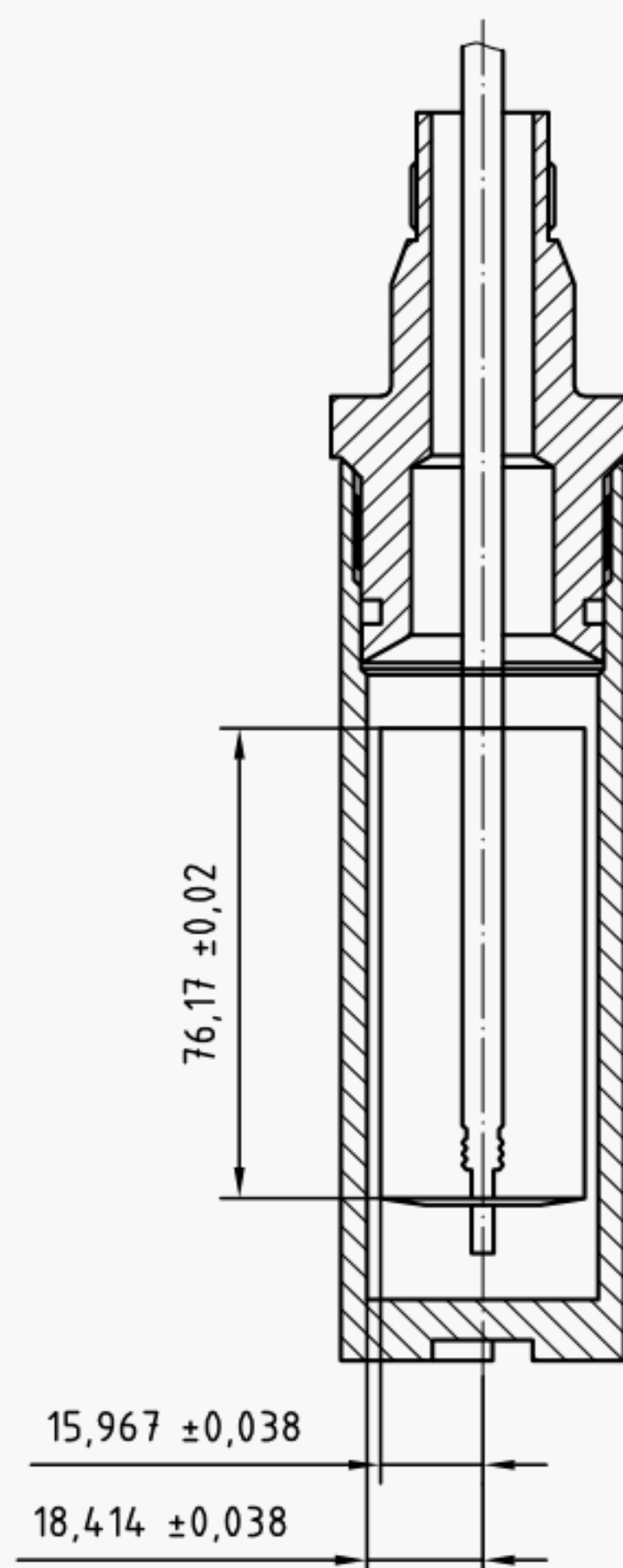
Viscometer speed r/min	Shear rate s^{-1}
29,5	25
59,0	50
88,5	75
118	100

8.2.2.2.3 Calculations

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

For rheological calculations, see Clause 9.

Dimensions in millimetres

**Figure 3 — Geometry of a pressurized concentric-cylinder viscometer**

8.3 Viscoelastic fluids

8.3.1 Introduction

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.

8.3.2 Apparatus

The properties of viscoelastic 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 may be suitable for determining the rheological behaviour of fluids.

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

- a) 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.

8.3.2.1 Pressurized concentric-cylinder viscometer, having the dimensions shown in Figure 3.

8.3.2.1.1 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 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.

8.3.2.1.2 Operation

8.3.2.1.2.1 Instrument preparation

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

8.3.2.1.2.2 Procedure

The following procedures shall be followed.

- 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. Place 52 cm³ of fluid into the viscometer. This volume is sufficient 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.

Optional: ambient temperature shear ramp [described in 8.3.2.1.2.2 b)]. elapsed time is defined as zero ($t = 0$) immediately after completing this ramp, and fluid heating is 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 $\pm 3 \text{ °C}$ ($\pm 5 \text{ °F}$) 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 elapsed time before the ramp begins. Starting at $t = 20 \text{ min}$, shear rate ramps shall begin every 15 min up to $t = 2 \text{ h } 5 \text{ min}$. Beginning at $t = 2 \text{ h } 35 \text{ min}$ 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 ramp 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 after completing 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.

8.3.2.1.3 Calculations

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

For rheological calculations, see Clause 9.

9 Calculation procedures

9.1 General concepts

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

$$\tau = K \dot{\gamma}^n \quad (1)$$

where

τ is the shear stress;

K is the fluid consistency index;

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

n is the flow behaviour index.

9.2 Brief review of geometry-independent rheology vs. nominal rheology

9.2.1 For a power-law fluid, the shear rate at the measurement surface depends on the geometry of the viscometer and 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 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.

9.2.2 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 geometry-independent consistency index will provide the proper shear stress value and hence pressure loss in the selected geometry.

9.2.3 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 K . Equations 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:

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

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

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

where

τ is the shear stress, in mPa (lbf/ft²);

K_v is the geometry-dependent consistency index, in mPa·s^{*n*} (lbf·s^{*n*}/ft²);

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

K is the geometry-independent consistency index, in mPa·s^{*n*} (lbf·s^{*n*}/ft²);

$\dot{\gamma}$ is the shear rate at the measurement surface, 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 at the measurement surface, in mPa·s (cP).

- b) Calculation of viscosity at the measurement surface, using SI units for K :

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

where

μ is the viscosity at the measurement surface, in mPa·s (cP);

K is the geometry-independent consistency index, in mPa·s^{*n*} (lbf·s^{*n*}/ft²);

$\dot{\gamma}$ is the shear rate at the measurement surface, in s⁻¹;

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

9.3 Limitations/problems that may 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 vs. shear rate;
- b) slip (non-homogeneous) flow, due to:
 - 1) fluids with high normal forces (e.g. highly elastic fluids) may climb out of the gap in a concentric-cylinder viscometer,
 - 2) 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) rheopectic material will build up structure with time while being sheared at a constant rate.

9.4 Calculation method for concentric-cylinder viscometers

9.4.1 General

Many concentric-cylinder viscometers available to the industry have computerized data acquisition, data reduction and data analysis capabilities. When using these instruments, the conversion of torque and rotation rate to shear stress and shear rate, respectively, is done automatically and therefore is transparent to the user. In some instances, these instruments also have the capability of providing an automated power-law data analysis. Procedures provided in this subclause can be used to verify the proper functioning of software found on these instruments. These procedures are also to be used for data reduction and analysis when working with viscometers that are not automated.

9.4.2 Calculation of shear stress from torque values

Helical torsion springs are typically attached to the stationary cylinder 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 from the following equation:

$$M = c \cdot \theta \quad (6)$$

Torque is also a force acting through a distance:

$$M = F \cdot R_i \quad (7)$$

where

M is the torque, in newton metres (lbf·ft);

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

θ is the spring deflection, in radians (degrees);

F is the shear force tangential to the cylinder surface, in newtons (pound-force);

R_i is the radius of the stationary cylinder, in metres (feet).

A force balance allows the shear stress to be calculated from torque measurements:

$$\tau A = M/R_i \quad (8)$$

$$\tau = M/2\pi R_i^2 l \quad (9)$$

where

τ is the shear stress acting on the stationary inner cylinder, in millipascals (lbf/ft²);

A is the surface area of the stationary inner cylinder, in square metres (square feet);

l is the stationary inner cylinder length, in metres (feet).

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 some method other than a torsion spring to sense torque.

9.4.3 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 by the following equation:

$$\dot{\gamma}_n = 2\omega / [1 - (R_i/R_o)^2] \quad (10)$$

where

$\dot{\gamma}_n$ is the nominal shear rate at the surface of the stationary inner cylinder, in reciprocal seconds;

ω is the angular velocity of the rotating outer cylinder, in radians per second;

R_i is the radius of the inner stationary cylinder, in metres (feet);

R_o is the inner radius of the outer rotating cylinder, in metres (feet).

9.4.4 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:

$$\log_{10} \tau = \log_{10} K_v + n \log_{10} \dot{\gamma}_n \quad (11)$$

which is of the form:

$$y = ax + b \quad (12)$$

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 millipascals (lbf/ft²);

$\dot{\gamma}_n$ is the nominal shear rate, in reciprocal seconds.

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

Using base-10 logarithms,

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

A goodness-of-fit coefficient, the coefficient of determination, R^2 , shall be reported for each calculation of n and K_v . K_v may be converted to $\text{lbf}\cdot\text{s}^n/\text{ft}^2$ by dividing by 478,8.

9.4.5 Geometry-independent fluid consistency index calculation

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

$$K = K_v \{ [1 - (R_i/R_o)^2] / n [1 - (R_i/R_o)^{2/n}] \}^{-n} \quad (14)$$

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

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

and that for a pipe, K_p , is calculated as:

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

9.5 Bingham plastic parameters for completion fluids

A Bingham plastic material possesses a yield point, τ_y , which is the shear stress that shall be exceeded before the material exhibits fluid behaviour. At shear stress values greater than the yield point, the material exhibits a linear shear stress versus shear rate response. The proportionality constant is defined as the slope of the linear shear stress versus shear rate response and is called the plastic viscosity μ_p . The empirical significance of the model parameters has become extremely important in describing the behaviour of certain completion fluids. The ease with which these model parameters can be derived from the 600 r/min and 300 r/min dial readings of the non-pressurized viscometer has also contributed to their widespread use. The instrument provides the Bingham plastic model parameters in USC units as follows:

$$\mu_p \text{ (cP)} = \theta_{600} - \theta_{300} \quad (17)$$

$$\tau_y \text{ (lbf/100 ft}^2\text{)} = \theta_{300} - \mu_p \quad (18)$$

where θ_{600} and θ_{300} are the dial readings at 600 r/min and 300 r/min, respectively, from a non-pressurized viscometer with rotor 1, bob 1 (R1B1) combination and a No.1 spring (i.e. spring factor = 1).

The model parameters are expressed in SI units, determined from the USC units, as follows:

$$\mu_p \text{ (mPa}\cdot\text{s)} = \mu_p \text{ (cP)} \quad (19)$$

$$\tau_y \text{ (Pa)} = 0,4788 \tau_y \text{ (lbf/100 ft}^2\text{)} \quad (20)$$

9.6 Calculations for optional shear-history simulation

9.6.1 Flowrate 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 flow rate and tubing length needed to provide the desired preconditioning can be calculated. For example, to maintain a shear rate for a given time:

$$v = \frac{\dot{\gamma}_n \cdot d}{8} \quad (21)$$

$$q_V = 0,785 \, 4 \, d^2 v \quad (22)$$

$$l = v \cdot t \quad (23)$$

where

v is the bulk average fluid velocity, in metres per second (feet per second);

$\dot{\gamma}_n$ is the Newtonian (nominal) shear rate, in reciprocal seconds;

d is the tubing ID, in metres (feet);

q_V is the volume flowrate, in cubic metres per second (cubic feet per second);

l is the tubing length, in metres (feet);

t is the time, in seconds.

9.6.2 Minimum radius of curvature

The tubing length required may be very long and in most cases it will be desirable to coil the tubing to confine it to a small space. Coil diameters should be made as large as practical to minimize any additional energy dissipation caused by the curvature. A large increase in the resistance to flow occurs when the Dean Number is > 10 .

The Dean number is defined as follows:

$$De = Re \sqrt{\frac{R}{r}} \quad (24)$$

where

De is the Dean number;

Re is the Reynolds number;

R is the tube cross-sectional radius, in metres (feet);

r is the radius of curvature, in metres (feet).

To minimize the increase in flow resistance, the radius of curvature should be greater than the minimum expressed by the following inequality:

$$r \succ \frac{Re^2 R}{100} \quad (25)$$

10 Test report

The test report should include at least the following information:

a) General data

- 1) date;
- 2) name of person(s) performing test;
- 3) name of organization/laboratory.

b) Fluid data

- 1) fluid type
 - i) non-crosslinked;
 - ii) viscoelastic.
- 1) fluid application
 - i) completion, non-fracturing;
 - ii) fracturing.

- c) Base fluid data
 - 1) source;
 - 2) aqueous:
 - i) deionized water;
 - ii) field water;
 - iii) seawater.
 - 3) water pH;
 - 4) hydrocarbon;
 - 5) acid type and concentration.
- d) Mixing apparatus data
 - 1) manufacturer;
 - 2) model number;
 - 3) container volume.
- e) Fluid composition and preparation
 - 1) volume mixed;
 - 2) filtration criteria (non-crosslinked);
 - 3) mixing procedure (per supplier specifications);
 - 4) components;
 - 5) amount of each component;
 - 6) ageing or holding time prior to measurements (if required);
 - 7) fluid temperature (crosslinked);
 - 8) fluid pH.
- f) Fluid mixing speed data
 - 1) mixing speeds with time at each speed.
- g) Shear history data
 - 1) low or high shear rate simulation;
 - 2) fluid temperature.
- h) Viscometer
 - 1) apparatus type;
 - 2) rotor/bob;

- 3) spring factor;
- 4) spring constant;
- 5) date last calibrated.
- i) Results
 - 1) all measurements;
 - 2) all calculations.
- j) ISO procedure
 - 1) for single-point measurement:
 - i) shear rate;
 - ii) viscosity at time and temperature.
 - 2) multipoint measurement at each shear rate ramp:
 - i) elapsed time at the beginning of the ramp;
 - ii) increasing or decreasing of shear rates;
 - iii) shear rate between ramps;
 - iv) apparent viscosity at 40 s^{-1} and 100 s^{-1} ;
 - v) power-law parameters K_v , n , R^2 ;
 - vi) viscosity and fluid temperature vs. elapsed time.
- k) Modifications of this procedure (if this procedure is modified it cannot claim compliance with this part of ISO 13503-1)
 - 1) description of the modifications in sufficient detail to allow others to reproduce the method.

Annex A (Informative)

National Adoption editorial Changes

The following modifications have not been changed in the body of this standard, but are noted by an arrow (➔) in the margin for reference to this Annex A.

Clause	Editorial Change
2.5	Add to the end of the definition the phrase "except by diffusion".
5	Append the 2 nd sentence with "only if filtering will not remove products added to the fluids during preparation."
6.5 d	Remove "essentially" from "...fluid flowing through them is essentially the same..."
8.2.2.1.2.1	Correct grammar to "Other vessels may be used. However, ..."
8.2.2.2.2.2 b)	Correct grammar to "After 4 h 5 min, the time elapsed when ramps begin is at the discretion of the operator; however, these shall be reported."
8.3.2.1.2.2 b	Correct grammar to "... at the discretion of the operator; however, these shall be reported."

Bibliography

- [1] API RP 39, *Recommended Practice on Measuring the Viscous Properties of a Cross-linked Water-based Fracturing Fluid*, third edition, May 1998



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