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Rheology —

Part 2:
General principles of rotational and oscillatory rheometry

—

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 9, *General test methods for paints and varnishes*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 139, *Paints and varnishes*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement), and in cooperation with ISO/TC 61, *Plastics*, SC 5, *Physical-chemical properties*.

This document cancels and replaces ISO 3219:1993, which has been technically revised. The main changes compared to the previous editions are as follows:

— plate-plate measuring geometry has been added;

— relative measuring geometries have been added;

— oscillatory rheometry has been added.

A list of all parts in the ISO 3219 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Rheology —

Part 2: General principles of rotational and oscillatory rheometry

1 Scope

This document specifies the general principles of rotational and oscillatory rheometry.

Detailed information is presented in Annex A. Further background information is covered in subsequent parts of the ISO 3219 series, which are currently in preparation.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3219-1, *Rheology — Part 1: General terms and definitions for rotational and oscillatory rheometry*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 3219-1 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
measuring gap
space between the boundary surfaces of the measuring geometry

3.2
gap width
h
H_{cc}
H_{cp}
distance between the boundary surfaces of the measuring geometry

Note 1 to entry: The symbol h refers to a gap width that can be varied (e.g. plate-plate measuring geometry); the symbol H refers to a gap width which is not variable and which is defined by the relevant measuring geometry. H_{cc} is the gap width of the coaxial-cylinders geometry. H_{cp} is the gap width of the cone-plate geometry.

Note 2 to entry: The distance between the boundary surfaces is given by the difference in the radii (coaxial cylinders), the cone angle (cone-plate) or the distance between the two plates.

Note 3 to entry: In cone-plate measuring geometries, the gap width varies as a function of the radius across the measuring geometry. The value H_{cp} is the distance between the flattened cone tip and the plate.

3.3
flow field coefficient
geometric factor
k
quotient of the shear stress factor (3.9) k_τ and the strain factor (3.8) k_γ

Note 1 to entry: The flow field coefficient *k* relates the angular velocity *Ω* and torque *M* to the shear viscosity *η* of the fluid as given by the following formula:

$$\eta = k \cdot \frac{M}{\Omega}$$

The flow field coefficient *k* is expressed in radians per cubic metre ($rad \cdot m^{-3}$). It can be calculated from the shape and dimensions of an *absolute measuring geometry* (3.7).

3.4
no-slip condition
presence of a relative velocity of zero between a boundary surface and the immediately adjacent fluid layer

3.5
wall slip
presence of a non-zero relative velocity between a boundary surface and the immediately adjacent fluid layer

3.6
relative measuring geometry

measuring geometry for which the flow profile and thus the rheological parameters cannot be calculated

Note 1 to entry: For relative measuring geometries, the viscosity shall not be given in pascal multiplied by seconds (Pa·s) except in the case of plate-plate measuring geometries if the correction referred to in 5.3.3.1.2 is used.

3.7
absolute measuring geometry
measuring geometry for which the flow profile and thus the rheological parameters can be calculated exactly for the entire sample, regardless of its flow properties

3.8
strain factor
k_γ
proportionality factor between the angular deflection *φ* and shear strain *γ* for *absolute measuring geometries* (3.7)

Note 1 to entry: The absolute value of the strain factor corresponds to the absolute value of the shear rate factor. The latter is the proportionality factor between the shear rate $\dot{\gamma}$ and the angular velocity *Ω*.

Note 2 to entry: This factor is called the shear rate factor in the rotation test and the strain factor in the oscillatory test.

Note 3 to entry: The strain factor k_γ has units of reciprocal radians (rad^{-1}).

3.9
shear stress factor
k_τ
proportionality factor between the torque *M* and the shear stress *τ* for *absolute measuring geometries* (3.7)

Note 1 to entry: The shear stress factor k_τ has units of reciprocal cubic metres (m^{-3}).

4 Symbols

Table 1 — Symbols and units

Meaning	Symbol	Unit
Absolute value of the complex shear modulus	$\|G^*\|$	Pa
Absolute value of the complex viscosity	$\|\eta^*\|$	Pa·s
Acceleration of the angular deflection	$\ddot{\varphi}$	rad·s^{-2}
Amplitude of the angular deflection of the motor	$\varphi^*_{M,0}$	rad
Amplitude of angular deflection of torque transducer	$\varphi^*_{D,0}$	rad
Amplitude of the angular deflection	φ_0	rad
Amplitude of the angular velocity	$\dot{\varphi}_0$	rad·s^{-1}
Amplitude of the shear rate	$\dot{\gamma}_0$	s^{-1}
Amplitude of the shear strain	γ_0	1
Amplitude of the shear stress	τ_0	Pa
Amplitude of the torque	M_0	N·m
Angular acceleration of motor	$\ddot{\varphi}^*_M$	rad
Angular acceleration of torque transducer	$\ddot{\varphi}^*_D$	rad
Angular deflection	φ	rad
Angular deflection of motor	φ^*_M	rad
Angular deflection of sample	φ^*_P	rad
Angular deflection of torque transducer	φ^*_D	rad
Angular frequency	ω	rad·s^{-1} or s^{-1}
Angular velocity across the measuring gap	$\omega(r)$	rad·s^{-1}
Angular velocity (presented in brackets: as the time derivative of the angular deflection)	Ω, $(\dot{\varphi})$	rad·s^{-1}
Angular velocity of motor	$\dot{\varphi}^*_M$	rad s^{-1}
Angular velocity of torque transducer	$\dot{\varphi}^*_D$	rad s^{-1}
Coefficient of bearing friction	D_L	
Coefficient of friction	D	N·m·s
Complex angular deflection	φ^*	rad
Complex shear modulus	G^*	Pa
Complex torque	M^*	N·m
Complex viscosity	η^*	Pa·s
Cone angle	α	° or rad
Deflection path	s	m
Drive loss factor	$\tan\zeta$	1
Drive phase angle	ζ	rad
Face factor	c_L	1
Flow field coefficient, geometric factor	k	rad·m^{-3}
Frequency	f	Hz
NOTE The parameters marked with an * refer to complex-valued parameters whose real part is denoted by ′ and imaginary part by ″.		

Table 1 *(continued)*

Meaning	Symbol	Unit
Gap width	H	m
Gap width defined by the coaxial cylinders geometry	H_{cc}	m
Gap width defined by the cone-plate geometry	H_{cp}	M
Geometry compliance	C_G	
Imaginary part of the complex viscosity	η''	Pa·s
Imaginary unit	i	1
Loss angle, phase angle	δ	rad
Loss factor	$\tan\delta$	1
Moment of inertia	I	N·m·s^2
Real part of the complex viscosity	η'	Pa·s
Rotational speed	n	s^{-1} or min^{-1}
Sample torque	M_P^*	N·m
Shear force	F	N
Shear loss modulus, viscous shear modulus	G''	Pa
Shear modulus	G	Pa
Shear plane	A	m^2
Shear rate factor	$k_{\dot{\gamma}}$	rad^{-1}
Shear rate, shear deformation rate	$\dot{\gamma}$	s^{-1}
Shear storage modulus, elastic shear modulus	G'	Pa
Shear strain, shear deformation	γ	1 or %
Shear stress	τ	Pa
Shear stress factor	$k_{\text{Ä}}$	m^{-3}
Shear viscosity	η	Pa·s
Strain factor	k_γ	rad^{-1}
Temperature	T	°C, K
Time	t	s
Torque	M	N·m
Torque applied by motor	M_M^*	N·m
Torque caused by bearing friction	M_L^*	N·m
Torque caused by transducer inertia	M_I^*	N·m
Torque measured by transducer	M_m^*	N·m
Torsional compliance of the measurement system	C	rad·(N·m)$^{-1}$
Velocity	v	m·s^{-1}
NOTE The parameters marked with an * refer to complex-valued parameters whose real part is denoted by ′ and imaginary part by ″.		

5 Measuring principles

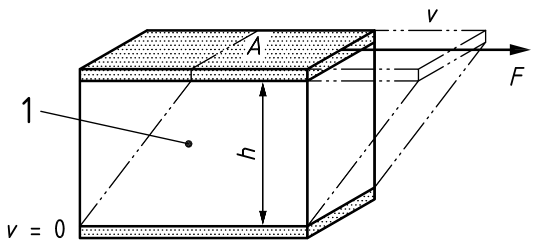
5.1 General

There are rotational tests, oscillatory tests and various step tests. The different tests can be combined with one another.

These can be carried out using various measuring types: controlled deformation (CD), controlled rate (CR) or controlled stress (CS).

5.2 Rotational rheometry

In the basic rotational test, the sample is subjected to constant or variable loading in one direction. The shear viscosity η is calculated from the measured data. The corresponding mechanical input and response parameters are listed in Tables A.1 and A.3. The basic parameters of the test can be represented schematically in terms of the two-plates model. An infinitesimal element of the measuring geometry is considered in this subclause (see Figure 1). The two-plates model consists of two parallel plates, each with a surface area A and with a gap width h, between which the sample is located. The velocity of the lower plate is zero (v = 0). The upper plate is moved by a defined shear force F, which results in a velocity v. It is assumed that the sample between the plates consists of layers that move at different velocities of between v = 0 and v.



Key

1 sample
v velocity
A shear plane
h gap width
F shear force

Figure 1 — Two-plate model with a simplified schematic representation of the basic parameters of a rotational test

With this model, the following parameters are calculated using Formulae (1) to (3):

$$\tau = \frac{F}{A} \tag{1}$$

where

τ is the shear stress, in pascals;

F is the shear force, in newtons;

A is the shear plane, in square metres.

$$\dot{\gamma} = \frac{v}{h} \tag{2}$$

where

$\dot{\gamma}$ is the shear rate, in reciprocal seconds;

v is the velocity, in metres per second;

h is the gap width, in metres.

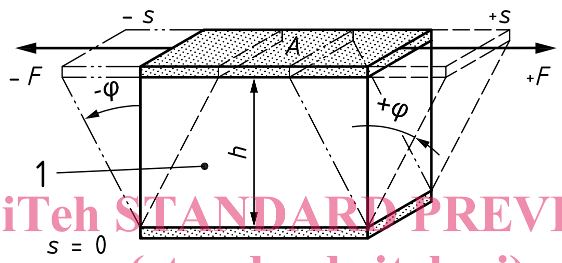
Based on the Newtonian law of viscosity, the shear viscosity can be calculated using Formula (3):

$$\eta = \frac{\tau}{\dot{\gamma}} \quad (3)$$

where η is the shear viscosity, in pascal multiplied by seconds.

5.3 Oscillatory rheometry

In the basic oscillatory test, the sample is stimulated with an angular deflection or torque amplitude at a given oscillation frequency. The resulting response oscillates with the same frequency and is characterized by an amplitude and phase shift. The corresponding mechanical input and response parameters are listed in Tables A.2 and A.3. Parameters such as the shear storage modulus G' (elastic shear modulus), the shear loss modulus G'' (viscous shear modulus), the absolute value of the complex viscosity $|\eta^*|$ and the loss factor tan δ can be calculated from the measured data in order to characterize the viscoelastic behaviour. The mathematical principles are presented in A.3. The basic parameter of the test can be represented schematically in terms of the two-plates model (see Figure 2).



Key

1 sample

s deflection path

φ deflection angle

A shear plane

h gap width

F shear force

Figure 2 — Two-plate model with a simplified schematic representation of the basic parameters of an oscillatory test

With this model, the following parameters can be calculated using Formula (4):

$$\gamma = \frac{s}{h} \quad (4)$$

where

γ is the shear strain, dimensionless;

s is the deflection path, in metres;

h is the gap width, in metres.

In the oscillatory test, the shear strain γ varies sinusoidally as a function of time t, see Figure 3. The associated shear stress τ is shifted within the viscoelastic range by the loss angle δ at the same angular frequency ω. Formulae (5) and (6) apply:

$$\gamma(t)=\gamma_0 \sin(\omega t) \quad (5)$$

where

γ_0 is the amplitude of the shear strain, dimensionless;

ω is the angular frequency, in radians per second;

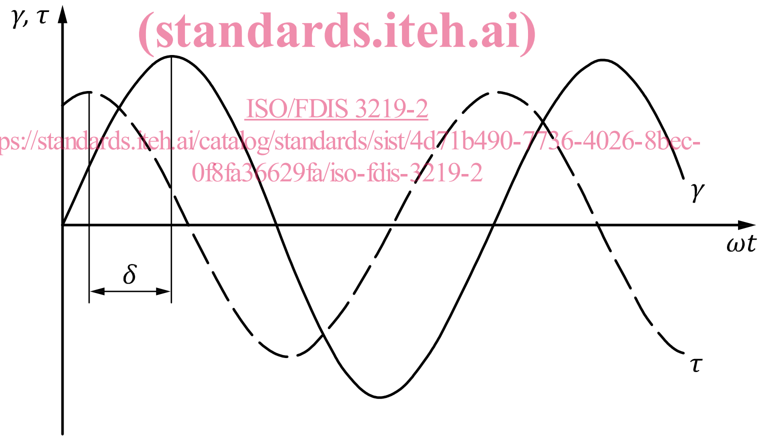
t is the time, in seconds.

$$\tau(t)=\tau_0 \sin(\omega t+\delta) \quad (6)$$

where

τ_0 is the amplitude of the shear stress, in pascals;

δ is the loss angle, in radians.



Key

γ shear strain

τ shear stress

ω angular frequency

t time

δ loss angle

Figure 3 — Schematic representation of the shear strain and shear stress functions for an oscillatory test

NOTE Degrees (°) are commonly used in practice as the unit for the loss angle δ. The following conversion applies: 2π rad = 360°.

In the case of ideal elastic behaviour (in accordance with Hooke's law), the loss angle has a value of δ = 0°, i.e. the shear strain and shear stress are always in phase. In the case of ideal viscous behaviour (in accordance with Newton's law), the loss angle has a value of $\delta = \pi/2$ = 90°, i.e. the shear stress curve is 90° ahead of the shear strain curve.

Using Hooke's elasticity law, the complex shear modulus G^* and its absolute value $|G^*|$ can be calculated using Formulae (7) and (8):

$$G^* = \frac{\tau(t)}{\gamma(t)} \tag{7}$$

$$|G^*| = \sqrt{G'^2 + G''^2} \tag{8}$$

where

G^* is the complex shear modulus, in pascals;

G' is the shear storage modulus, in pascals;

G'' is the shear loss modulus, in pascals;

G^* describes the overall viscoelastic behaviour.

This can be separated into an elastic component G' (shear storage modulus) and a viscous component G'' (shear loss modulus) using Formulae (9) and (10).

$$G' = \frac{\tau_0}{\gamma_0} \cos\delta \tag{9}$$

$$G'' = \frac{\tau_0}{\gamma_0} \sin\delta \tag{10}$$

The quotient of the shear loss modulus G'' and shear storage modulus G' is the dimensionless loss factor $\tan\delta$, see Formula (11):

$$\tan\delta = \frac{G''}{G'} \tag{11}$$

The ratio of the absolute value of the complex shear modulus G^* and the angular frequency ω is the absolute value of the complex viscosity η^*, see Formula (12):

$$|\eta^*| = \frac{|G^*|}{\omega} \tag{12}$$

where $|\eta^*|$ is the absolute value of the complex viscosity, in pascal multiplied by seconds.

6 Measuring assembly

6.1 General

The rheological properties are investigated using a measuring system consisting of a measuring device (viscometer or rheometer) and a measuring geometry (e.g. cone-plate).

The viscometer can only measure the viscosity in rotation (viscometry). This means that the viscosity function of the sample can be determined as a function of the parameters of time, temperature, shear rate, shear stress and others such as pressure.

With a rheometer, it is possible to carry out all basic tests in rotation and oscillation (rheometry). Alongside the viscosity function, the viscoelastic properties can be determined, e.g. shear storage modulus and shear loss modulus.

A measuring assembly, consisting of a measuring device, a measuring geometry and optional accessories, is shown in Figure 4. The measuring device and individual components, such as the temperature control system, can be computer-controlled.

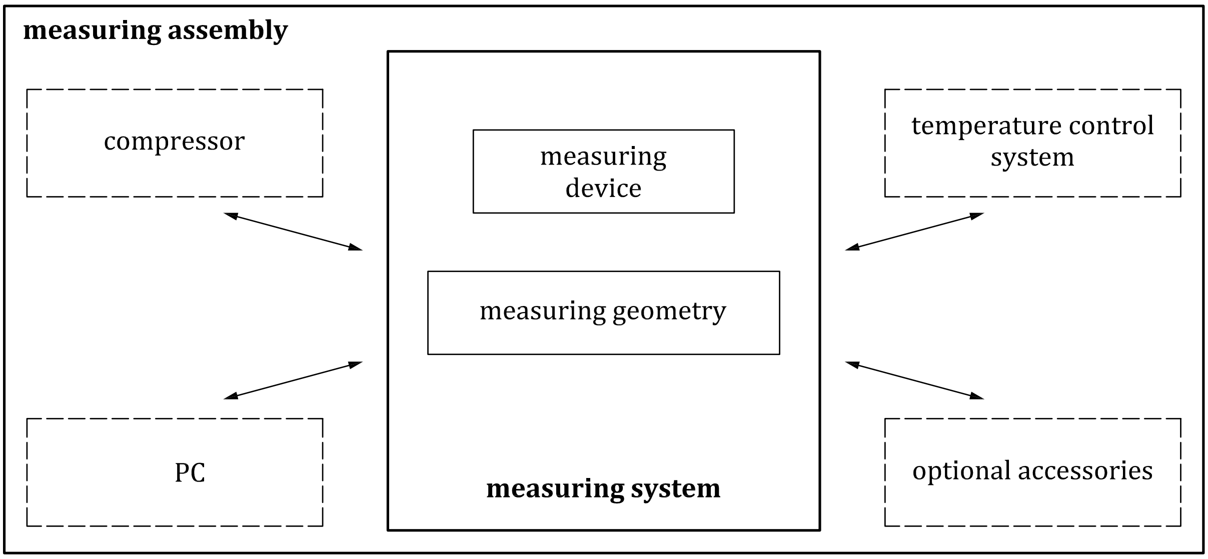


Figure 4 — Example of a measuring assembly

The sample to be investigated is located in a measuring gap where a defined flow profile is generated in the sample. A necessary prerequisite for this is a sufficiently small gap width. When viscometers or rheometers are used, they shall be able to impose or detect torque or rotational speed/angular deflection. The imposed parameter shall be adjustable both in time-dependent and time-independent manners.

For viscometric measurements, all viscometers are principally suitable, regardless of how the drive and/or detection unit are supported. For measurements in oscillation, rheometers shall be used that have the lowest possible internal friction in the drive or detection unit.

To cover the broadest possible range of applications, the viscometer or rheometer shall be able to work with different measuring geometries. The range of the torques or angular deflections, that result and the measuring range that can be achieved, depend on the measuring system. The type of measuring device and measuring geometry to be selected depends on the sample.

6.2 Temperature control systems

A temperature control system consists of one or more temperature control components for heating and/or cooling, including the required media (e.g. air, water, liquid nitrogen) and the necessary connections (e.g. hoses and insulation for these hoses).

The rheological properties of the sample are temperature-dependent. As a result, measures such as controlling of the sample temperature and its measurement with one or more temperature sensors in the immediate vicinity of the sample are required.

The temperature of the sample shall be kept constant as a function of time during the measurement period.

6.3 Measuring geometries

6.3.1 General

A measuring geometry consists of two parts that form a sample chamber where the sample is located. A measuring geometry consists of a rotor and a stator or of two rotors.

The measuring geometry shall be selected in such a way that its dimensions are suitable for the expected viscosity range and viscoelastic properties of the sample. With regard to its gap width, the measuring geometry shall also be selected in such a way that possible heterogeneities in the sample (e.g. particles, drops, air bubbles) are considered. The magnitude of these heterogeneities is to be determined in advance using suitable methods (e.g. microscopy, laser diffraction, sieving or determination of fineness of grind).

The absolute and relative measuring geometries of a rotational viscometer or rheometer are described below.

Coaxial cylinders, double-gap and cone-plate measuring geometries are absolute measuring geometries. All the others are relative measuring geometries.

In the case of an absolute measuring geometry, the flow profile within the complete sample can be calculated exactly, regardless of its flow properties. This applies under the condition of laminar flow, no-slip condition and without slip (wall slip or slip between flow layers).

In the case of relative measuring geometries apart from plate-plate measuring geometries, calculation of the flow profile is only possible if the flow properties of the sample are known.

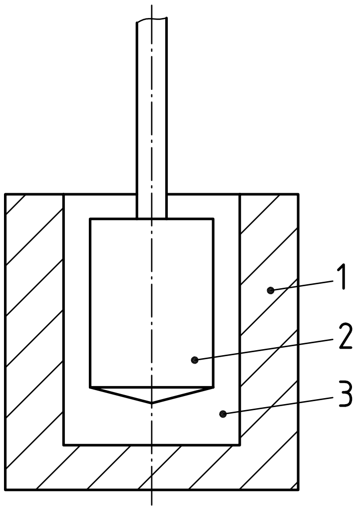
In practice, approximations are also used for absolute measuring geometries and thus corrections are carried out. Derivations of the basic flows for the absolute measuring geometries are presented in A.2.

6.3.2 Absolute measuring geometries

6.3.2.1 Coaxial cylinders measuring geometry

6.3.2.1.1 Description of the measuring geometry

The measuring geometry consists of a measuring cup (i.e. the outer cylinder) and a measuring bob (i.e. the inner cylinder with shaft, as shown in Figure 5). The measuring bob can serve as a rotor and the measuring cup as a stator (Searle principle), or vice versa (Couette principle); see Figure 6. If not indicated otherwise, the Searle principle is assumed below.



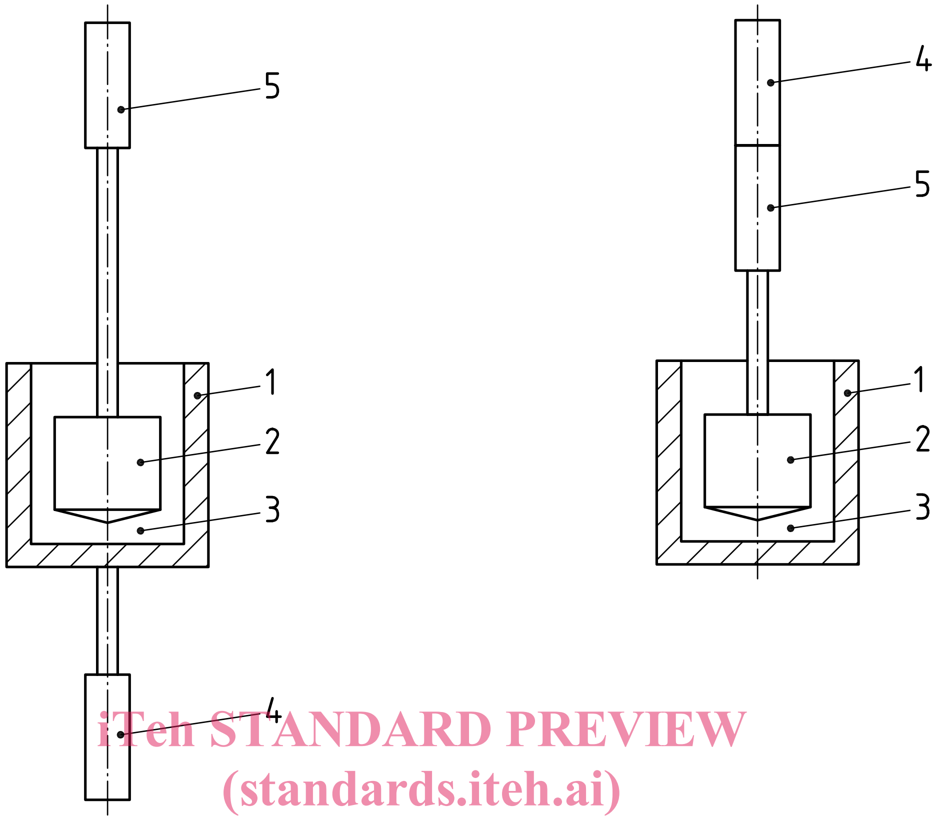
Key

1 measuring cup (outer cylinder)

2 measuring bob (inner cylinder)

3 sample chamber

Figure 5 — Schematic drawing of a coaxial cylinders measuring geometry



a) Couette principle b) Searle principle

Key

1 measuring cup (outer cylinder)

2 measuring bob (inner cylinder)

3 sample chamber

4 drive

5 measuring sensor

Figure 6 — Searle and Couette principles

The flow profile occurring in the measuring gap of the cylinder measuring geometry is calculated according to A.3.2. The measuring gap is the space between the shell surface of the measuring bob with a radius R_1 and the lateral surface of the measuring cup with a radius R_2 and the same length L; see Figure 7.

6.3.2.1.2 Calculation methods

Calculations of the shear stress τ and shear rate $\dot{\gamma}$ are ideally based on representative values that do not occur at the inner radius of the outer cylinder R_2 or outer radius of the inner cylinder R_1 of the measuring geometry but at a particular geometric position within the measuring gap. τ_{rep} is defined as the arithmetic mean of the shear stresses at the outer cylinder τ_1 and inner cylinder τ_2, which is a good approximation for the given ratio of radii ($\delta \leq 1,1$). For larger values and thus for relative measuring geometries see 6.3.3.2.

