



BSI Standards Publication

Rheology

Part 2: General principles of rotational and oscillatory rheometry

National foreword

This British Standard is the UK implementation of EN ISO 3219-2:2021. It is identical to ISO 3219-2:2021. Together with BS EN ISO 3219-1:2021, ISO 3219-3, ISO 3219-4 and ISO 3219-5, it supersedes BS EN ISO 3219:1995, which will be withdrawn on publication of the rest of the 3219 series.

ISO 3219-2:2021 is one part of a series of five standards covering rotational and oscillatory rheometry. The new series technically revises and expands the scope of BS EN ISO 3219:1995 from the measurement of viscosity at defined shear rates to a consideration of the principles and practise of rotational and oscillatory rheometry. ISO 3219-2:2021 covers the general principles of rotational and oscillatory rheometry, replacing and enlarging the following sections of BS EN ISO 3219:1995: 3 (Principle), 4 (Apparatus) and Annexes A and B (Measurement geometries including methods of calculation).

BS EN ISO 3219:1995 will remain current until all 5 parts of the new standard are published.

The UK participation in its preparation was entrusted to Technical Committee STI/10, Test methods for paints.

A list of organizations represented on this committee can be obtained on request to its committee manager.

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Rhéologie - Partie 2: Principes généraux de la rhéométrie rotative et oscillatoire (ISO 3219-2:2021)

Rheologie - Teil 2: Allgemeine Grundlagen der Rotations- und Oszillationsrheometrie (ISO 3219-2:2021)

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CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

European foreword

This document (EN ISO 3219-2:2021) has been prepared by Technical Committee ISO/TC 35 "Paints and varnishes" in collaboration with Technical Committee CEN/TC 139 "Paints and varnishes" the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by November 2021, and conflicting national standards shall be withdrawn at the latest by November 2021.

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

This document supersedes EN ISO 3219:1994.

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Endorsement notice

The text of ISO 3219-2:2021 has been approved by CEN as EN ISO 3219-2:2021 without any modification.

Contents

Page

Foreword	iv
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Symbols	3
5 Measuring principles	4
5.1 General.....	4
5.2 Rotational rheometry.....	5
5.3 Oscillatory rheometry.....	6
6 Measuring assembly	8
6.1 General.....	8
6.2 Temperature control systems.....	9
6.3 Measuring geometries.....	9
6.3.1 General.....	9
6.3.2 Absolute measuring geometries.....	10
6.3.3 Relative measuring geometries.....	20
6.4 Selected optional accessories.....	24
6.4.1 Cover with or without solvent trap.....	24
6.4.2 Passive and active thermal covers.....	25
6.4.3 Stepped plates.....	26
Annex A (informative) Information on rheometry and flow field patterns	27
Bibliography	45

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.

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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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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 have 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 ($\text{rad} \cdot \text{m}^{-3}$). 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 6.3.3.1.2 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·s ⁻²
Angular acceleration of torque transducer	$\ddot{\varphi}_D^*$	rad·s ⁻²
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)	$\Omega, (\dot{\varphi})$	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	N·m·s
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	$\text{rad} \cdot (\text{N} \cdot \text{m})^{-1}$
Imaginary part of the complex viscosity	η''	$\text{Pa} \cdot \text{s}$
Imaginary unit	i	1
Loss angle, phase angle	δ	rad
Loss factor	$\tan \delta$	1
Moment of inertia	I	$\text{N} \cdot \text{m} \cdot \text{s}^2$
Real part of the complex viscosity	η'	$\text{Pa} \cdot \text{s}$
Rotational speed	n	s^{-1} or min^{-1}
Sample torque	M_p^*	$\text{N} \cdot \text{m}$
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_{τ}	m^{-3}
Shear viscosity	η	$\text{Pa} \cdot \text{s}$
Strain factor	k_{γ}	rad^{-1}
Temperature	T	$^{\circ}\text{C}$ or K
Time	t	s
Torque	M	$\text{N} \cdot \text{m}$
Torque applied by motor	M_M^*	$\text{N} \cdot \text{m}$
Torque caused by bearing friction	M_L^*	$\text{N} \cdot \text{m}$
Torque caused by transducer inertia	M_I^*	$\text{N} \cdot \text{m}$
Torque measured by transducer	M_m^*	$\text{N} \cdot \text{m}$
Torsional compliance of the measurement system	C	$\text{rad} \cdot (\text{N} \cdot \text{m})^{-1}$
Velocity	v	$\text{m} \cdot \text{s}^{-1}$
NOTE The parameters marked with an * refer to complex-valued parameters whose real part is denoted by ' and imaginary part by ''.		

5 Measuring principles

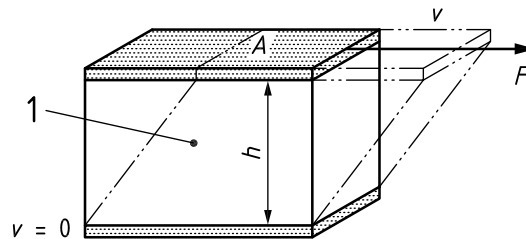
5.1 General

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 [\(3\)](#):

$$\tau = \frac{F}{A} \quad (1)$$

where

- τ is the shear stress, in pascals;
- F is the shear force, in newtons;
- A is the shear plane, in square metres.

$$\dot{\gamma} = \frac{v}{h} \quad (2)$$

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.

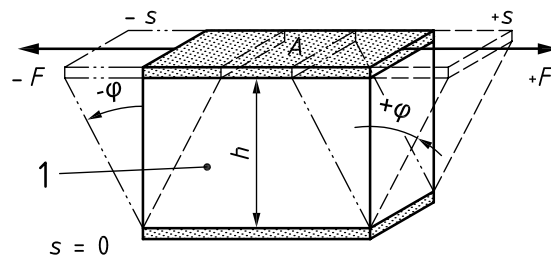
Based on the Newtonian law of viscosity, the shear viscosity can be calculated using [Formula \(3\)](#):

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

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 $\tan \delta$ 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;

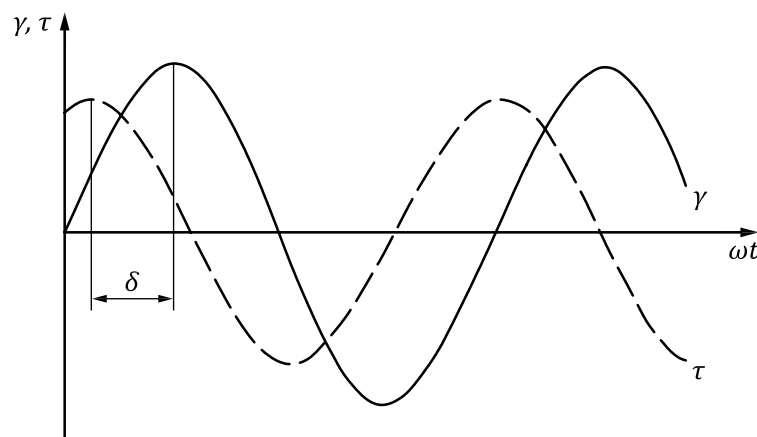
t is the time, in seconds.

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

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 ($^{\circ}$) are commonly used in practice as the unit for the loss angle δ . The following conversion applies: $2\pi \text{ rad} = 360^{\circ}$.

In the case of ideal elastic behaviour (in accordance with Hooke's law), the loss angle has a value of $\delta = 0^{\circ}$, 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 $\delta = \pi/2 = 90^{\circ}$, 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 [\(8\)](#):

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

$$|G^*| = \sqrt{G'^2 + G''^2} \quad (8)$$

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 [\(10\)](#).

$$G' = \frac{\tau_0}{\gamma_0} \cos \delta \quad (9)$$

$$G'' = \frac{\tau_0}{\gamma_0} \sin \delta \quad (10)$$

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

$$\tan \delta = \frac{G''}{G'} \quad (11)$$

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 \(12\)](#):

$$|\eta^*| = \frac{|G^*|}{\omega} \quad (12)$$

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.

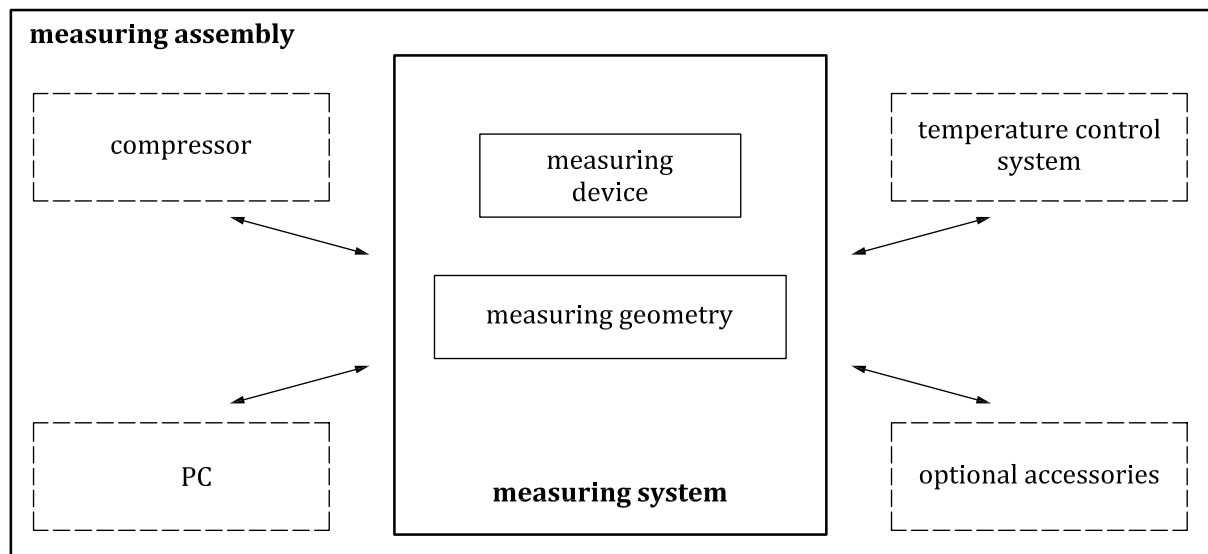


Figure 4 — Example of a measuring assembly

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.