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Rheology - Part 2: General principles of rotational and oscillatory rheometry (ISO/DIS 3219-2:2020)

Rheologie - Teil 2: Allgemeine Grundlagen der Rotations- und Oszillationsrheometrie (ISO/DIS 3219-2:2020)

Rhéologie - Partie 2: Principes généraux de la rhéométrie rotative et oscillatoire(ISO/DIS 3219-2:2020)

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Rheology —

Part 2:
General principles of rotational and oscillatory rheometry

Rhéologie —

Partie 2: Principes généraux du rhéométrie rotational et oscillatoire

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Member bodies are requested to consult relevant national interests in ISO/TC 61/SC 5 before casting their ballot to the e-Balloting application.

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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.

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 cooperation with ISO/TC 61, *Plastics*, SC 5, *Physical-chemical properties*.

This document cancels and replaces ISO 3219:1993, ISO 2884-1:1999 und ISO 2884-2:2003, 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;
- The information on cone-and-plate viscometer operated at a high rate of shear (ISO 2884-1:1999) and on disc or ball viscometer operated at a specified speed (ISO 2884-2:2003) has been integrated in the ISO 3219 standards series.

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.

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 http://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 defined by the relevant measuring geometry (cc – coaxial cylinders, cp – cone-plate).

Note 2 to entry: The distance between the boundary surfaces is given by the difference in the radii (cc), the cone angle (cp) 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 k_τ and the strain factor 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 equation:

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

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.

3.4

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

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

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

4 Measuring principles

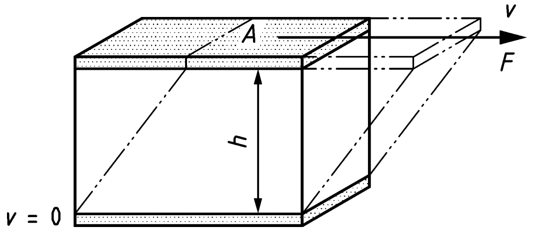
4.1 General

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). The material behaviour is described by several measurement points. The prerequisites for recording the individual measuring points are described in detail in ISO 3219-3 [2]. Error analysis is described in ISO 3219-4 [3] and ISO 3219-5 [4].

4.2 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 here (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

v velocity
A shear plane
h gap width
F shear force

Figure 1 — Two-plates 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:

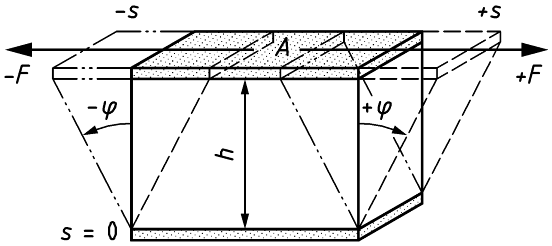
$$\eta = \frac{\tau}{\dot{\gamma}} \tag{3}$$

where

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

4.3 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 (Figure 2).



Key

s	deflection path
φ	deflection angle
h	gap width
F	shear force

Figure 2 — Two-plates 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:

$$\gamma = \frac{s}{h} \tag{4}$$

where

γ	is the shear strain, dimensionless;
s	is the deflection path, in metres;
h	is the gap width, in metres.

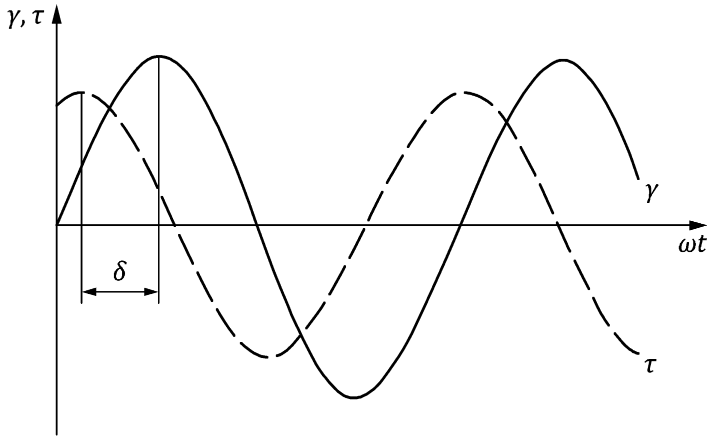
In the oscillatory test, the shear strain γ varies sinusoidally as a function of time t, see Figure 3. The associated shear stress τ is shifted within the viscoelastic range by the loss angle δ at the same angular frequency ω. Formulae 5 and 6 apply:

$$\gamma(t) = \gamma_0 \sin(\omega t) \tag{5}$$

$$\tau(t) = \tau_0 \sin(\omega t + \delta) \tag{6}$$

where

γ_0	is the amplitude of the shear strain, dimensionless;
τ_0	is the amplitude of the shear stress, in pascals;
δ	is the loss angle, in radians;
ω	is the angular frequency, in radians per second;
t	is the time, in seconds.



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 $\delta = 0 \equiv 0°$, 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 $\delta = \pi/2 \equiv 90°$, 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 loss factor tan δ, see Formula 11:

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

where

tan δ is the loss factor, dimensionless.

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;

ω is the angular frequency, in radians per second.

5 Measuring assembly

5.1 Measuring device

The rheological properties are investigated using a measuring device (viscometer or rheometer).

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.

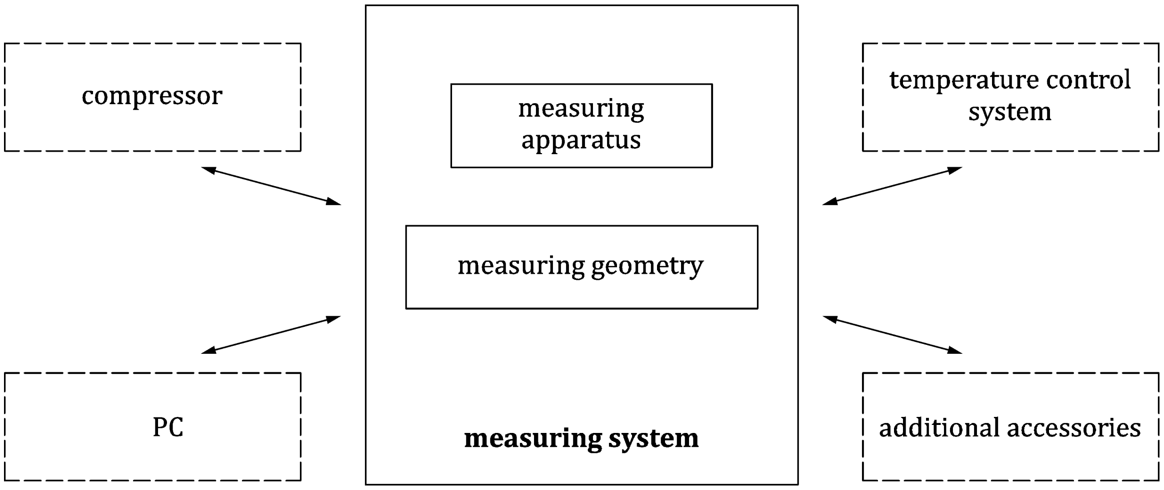


Figure 4 — Example of a measuring assembly

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 (see also ISO 3219-4). 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; see ISO 3219-3 for further information.

5.2 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).

NOTE There are different temperature control systems available, e.g. Peltier elements, liquid thermostats, cryostats, convection or radiation temperature chambers; see ISO 3219-3 for further information.

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; see ISO 3219-4 and ISO 3219-5 for further information.

5.3 Measuring geometries

5.3.1 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; see ISO 3219-4. 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 application range, advantages and disadvantages of each measuring geometry are described in more detail in ISO 3219-3.

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 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. The influence of these corrections on the measured values is negligible relative to the total measurement error, see ISO 3219-4 and ISO 3219-5.

Derivations of the basic flows for the absolute measuring geometries are presented in A.2.

5.3.2 Absolute measuring geometries

5.3.2.1 Coaxial cylinders measuring geometry

5.3.2.1.1 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.