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BSI Group Headquarters

389 Chiswick High Road London W4 4AL

Tel: +44 (0)20 8996 9000

Fax: +44 (0)20 8996 7400

www.bsigroup.com

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Responsible Committee Secretary: Ms Katherine Imbert (BSI)
Direct tel: 0208 996 6450
E-mail: katherine.imbert@bsigroup.com

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Introduction

This draft standard is based on national and international discussions. Your comments on this draft are invited and will assist in the preparation of the consequent standard.

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Date: xx/xx/20xx	Document: ISO/DIS xxxx
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1	2	(3)	4	5	(6)	7
MB	Clause No./ Subclause No./Annex (e.g. 3.1)	Paragraph/ Figure/Table/Note	Type of comment	Comment (justification for change) by the MB	Proposed change by the MB	Secretariat observations on each comment submitted
	3.1	Definition 1	ed	Definition is ambiguous and needs clarifying.	Amend to read '...so that the mains connector to which no connection...'	
	6.4	Paragraph 2	te	The use of the UV photometer as an alternative cannot be supported as serious problems have been encountered in its use in the UK.	Delete reference to UV photometer.	



DRAFT International Standard

ISO/DIS 3219-3

Rheology —

Part 3: Test procedure and examples for the evaluation of results when using rotational and oscillatory rheometry

Rhéologie —

*Partie 3: Mode opératoire d'essai et exemples d'évaluation
des résultats en cas d'utilisation de la rhéométrie rotative et
oscillatoire*

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CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
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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 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*.

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 3:

Test procedure and examples for the evaluation of results when using rotational and oscillatory rheometry

1 Scope

This document provides guidelines for selecting the measuring device, measuring geometry and temperature control unit. The general principles of test performance are described, and example evaluations of rotational and oscillatory rheometry are provided.

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 3219-1:2021, *Rheology — Part 1: Vocabulary and symbols for rotational and oscillatory rheometry*

IEC 61010 (all parts), *Safety requirements for electrical equipment for measurement, control, and laboratory use*

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

crystallization temperature

T_c

temperature at which crystallization starts and partial order is established when the melt solidifies

Note 1 to entry: The unit of the crystallization temperature T_c is degrees Celsius (°C).

3.2

gel point

transition point from sol to gel in oscillation in the linear-viscoelastic range, where the loss angle δ is independent of frequency

Note 1 to entry: The gel point can be in oscillation in the linear-viscoelastic range, where the loss angle δ is independent of frequency.

Note 2 to entry: In practice, the gel point is usually measured at one frequency and determined on the basis of the loss angle $\delta = 45^\circ$ or on the basis of the loss factor $\tan \delta = 1$.

3.3 gel time pot life

period of time for which a crosslinking material remains processable while a user-defined criterion is satisfied

Note 1 to entry: In rotation, the gel time can be determined with controlled shear rate or shear stress, or it can be determined in oscillation with controlled values for the amplitude of the shear strain or shear stress and for the frequency.

Note 2 to entry: In practice, relative measuring geometries or alternative methods are also used, such as flow cups.

3.4 glass transition temperature

T_g
approximate midpoint of the temperature range in which the glass transformation takes place

Note 1 to entry: The glass transition temperature differs depending on the test procedure and the measuring conditions.

3.5 sample preparation

all loads acting on the material to be measured for the preceding time up to the time of the measurement

3.6 shear stress relaxation modulus shear relaxation modulus

G
ratio of shear stress τ to shear strain γ during the relaxation test

Note 1 to entry: The unit for the shear stress relaxation modulus G is Pascal (Pa).

3.7 sol/gel transition

transition range from viscoelastic liquid (sol) to viscoelastic solid (gel)

3.8 trimming

removal of excess sample volume

Note 1 to entry: With plate-plate and cone-plate measuring geometries, a trimming position is often selected being larger as the gap width and at which the excess portion of the sample is removed flush to the edge of the measuring geometry. When the measuring position is subsequently approached, this ensures optimum filling of the gap.

4 Guidelines for selection

4.1 General

The choice of measuring device, measuring geometry, temperature control unit, measuring method and further accessories depends on the sample and its rheological requirements.

4.2 Selection of the measuring device

In ISO 3219-2 the differences between the different measuring devices for the determination of rheological properties and their setups are described. A rotational viscometer can only be used to measure the shear viscosity (viscometry). With a rotational rheometer, it is possible to carry out basic rotational and oscillatory tests (see [Clause 7](#)) (rheometry). For this reason, in addition to the viscosity function, this can be used to determine the viscoelastic properties, e.g. shear storage modulus and shear loss modulus.

For viscometric measurements, all viscometers are principally suitable, regardless of the type of bearing used in the drive and/or detection unit. For oscillatory measurements, creep tests and relaxation tests,

devices with low internal friction should be used, e.g. devices with air bearings or magnetic bearings. Some devices with ball bearings also offer the option of performing oscillatory measurements, but only within a restricted measuring range. During performance of the measurement, it shall be ensured that the work is carried out within the specifications of the measuring device, e.g. taking into account the torque limits and data acquisition rate.

To cover the broadest possible range of applications, the viscometer or rheometer shall be capable of working with different measuring geometries. The ranges of torque and angular velocity, and therefore the accessible measuring range, depend on the measuring device.

4.3 Selection of the measuring geometry

The measuring geometry shall be selected taking into account the available measuring device, the sample properties and the measuring profile. [Table 1](#) contains recommendations on usage of the different measuring geometries. Here, CC stands for coaxial cylinder, CP for cone-plate, PP for plate-plate, DG for double gap and RMG for relative measuring geometries (e.g. vane sensor, details see ISO 3219-2). In general, when selecting an absolute measuring geometry, the maximum size of heterogeneous components, e.g. the particle size in the sample, shall not exceed 10 % of the gap width. If, as a result, it is not possible to use absolute measuring geometries, then it is necessary to select relative measuring geometries. Optional accessories, such as a hood with a solvent trap to avoid solvent loss are described in ISO 3219-2.

Table 1 — Selection of the measuring geometry

Criteria	Recommended measuring geometry				
	Coaxial cylinders CC	Cone-plate CP	Plate-plate PP	Double gap DG	Relative RMG
Sample properties that determine the selection of the measuring geometry					
Low viscosity	x	x ^a		x	
High viscosity		x	x		
Pasty, gel-like			x		
Low surface tension	x			x	
Inhomogeneity, e.g. air bubbles, foaming					x
Further criteria for the selection of a measuring geometry					
Availability of a small sample volume		x	x		
Cleaning of the measuring geometry difficult	x ^b	x	x		
Cleaning of the measuring geometry not possible			x ^b		
Prevention of sedimentation/separation/phase separation	x ^c			x ^c	x ^c
Avoidance of wall slip	x ^d	x ^d	x ^d	x ^d	
^a for homogeneous samples using small cone angles ^b use of disposable measuring geometries ^c surface-treated (e.g. helical spiral groove) ^d surface-treated (e.g. sandblasting, profiling). The surface roughness R_a of the sandblasted geometry shall be at least ten times smaller than the measuring gap.					

4.4 Selection of the temperature control system

In the following guidelines, the best-suited temperature control system for each measurement is presented. [Figure 1](#) relates to the recommendation for selection of the temperature control system. It shall be taken into account that the temperature display can deviate from the true sample temperature. The temperature

gradient within the sample can be minimized by using an active or passive hood. Any chemical reactions that take place with air (e.g. oxygen, humidity) can be avoided under an inert gas atmosphere.

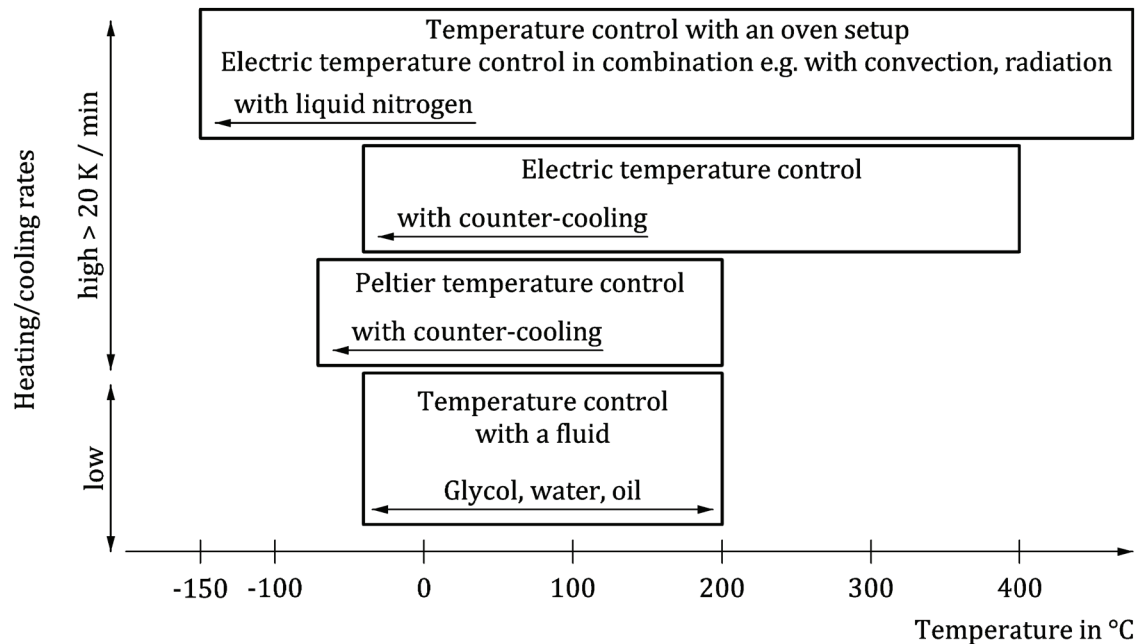


Figure 1 — Selection of the temperature control system

4.5 Selection of the measuring method

In Table 2, typical rheological requirements are listed with recommendations for meaningful measuring methods. Here, CR stands for controlled rate, CS for controlled stress and CD for controlled deformation (also called controlled strain, see ISO 3219-1:2021, 3.47 and Table 1). During performance of the measurement, it shall be ensured that the work is carried out within the specifications of the measuring device used.

Table 2 — Selection of measuring methods for viscometers and rheometers based on the example of coating materials

Application	Flow curve and viscosity curve CR	Flow curve and viscosity curve CS	Amplitude sweep CD or CS	Frequency sweep CD or CS	Creep test	Step test Oscillation/rotation/oscillation or rotation/rotation	Temperature sweep/time-dependent test in rotation or oscillation
Shelf life/sedimentation/phase separation	x ^a	x ^a	x ^b	x	x		x
Starting pumping (yield point)	x	x			x		
Sagging/levelling					x	x	
Pumping/mixing/stirring	x	x					
Painting/brushing/blade application (manual)	x	x				x	
Spraying	x	x				x	
High-speed coating	x						
Gel time/pot life/open time/processing time							x
^a evaluation via zero shear viscosity or yield point (if applicable, adaptation via rheological model function)							
^b for determination of the linear-viscoelastic range (LVR)							
^c only in oscillation							

Table 2 (continued)

Application	Flow curve and viscosity curve CR	Flow curve and viscosity curve CS	Amplitude sweep CD or CS	Frequency sweep CD or CS	Creep test	Step test Oscillation/rotation/oscillation or rotation/rotation/rotation	Temperature sweep/time-dependent test in rotation or oscillation
Gel point							x ^c
Temperature behaviour (glass transition temperature, crystallization temperature, melting temperature, crosslinking temperature)							x ^c
Hardening/crosslinking							x
^a evaluation via zero shear viscosity or yield point (if applicable, adaptation via rheological model function) ^b for determination of the linear-viscoelastic range (LVR) ^c only in oscillation							

The shear rate range plays an important role in rotational measurements. Representative shear rate ranges are shown in [Figure 2](#) for selected applications from [Table 2](#). The shear rates can be calculated as described in [Annex A](#).

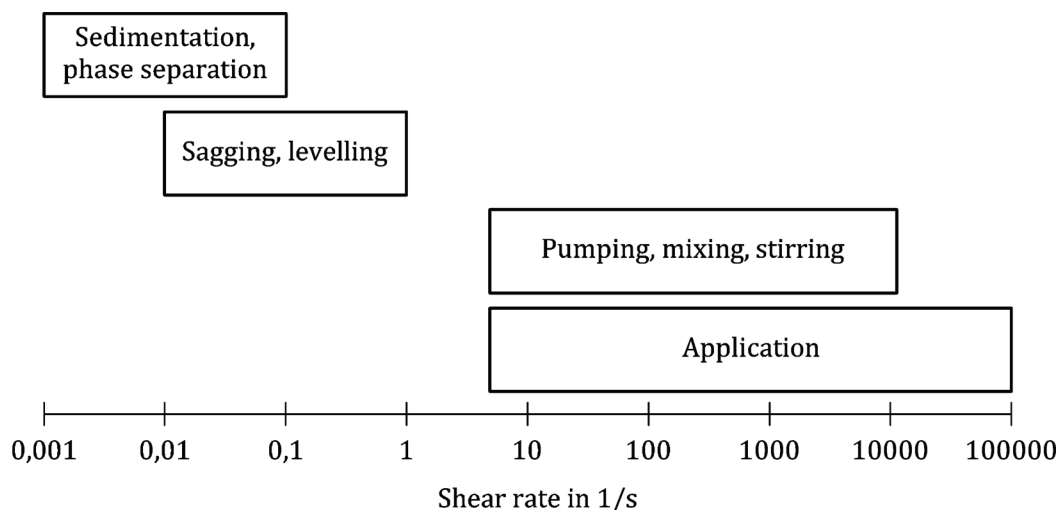


Figure 2 — Typical shear rate ranges for different applications based on the example of coating materials

Typical rheological parameters are shown in [Table 3](#) with reference to the corresponding sub-clause in this standard.

Table 3 — Rheological parameters and references

Rheological parameter	Description in sub-clauses
Flow behaviour	7.2.3 Flow curves and viscosity curves
Yield point	7.2.1.2 Specification of a constant shear stress (evaluation via the creep curve) 7.2.3.1 Specification of a variable shear rate (evaluation via curve fitting; or via the viscosity maximum method) 7.2.3.2 Specification of a variable shear stress (analogous to 7.2.3.1 ; as a plateau value or by means of regression lines)
Gel point	7.3.1 Time-dependent tests in oscillation 7.3.2 Temperature-dependent tests in oscillation
Gel time/pot life Processing time Processing temperature Crosslinking temperature	7.2.1.1 Specification of a constant shear rate 7.2.1.2 Specification of a constant shear stress 7.2.2 Temperature-dependent tests in rotation 7.3.1 Time-dependent tests in oscillation 7.3.2 Temperature-dependent tests in oscillation
Glass transition temperature	7.3.2 Temperature-dependent tests in oscillation
Hysteresis area	8.2 . Flow curves and hysteresis area
Crystallization temperature	7.3.2 Temperature-dependent tests in oscillation
Linearity limit	7.3.3 Amplitude sweeps (end of the LVR)
LVR (linear-viscoelastic range)	7.3.3 Amplitude sweeps
Master curve	8.4 . Master curve by means of time/temperature shift of frequency sweeps
Average molar mass	7.2.1.3 Specification of a constant shear strain (value of the initial shear stress relaxation modulus) 7.3.4 Frequency sweeps (position of the crossover point of the shear storage modulus and shear loss modulus) 8.4 Master curve by means of time/temperature shift of frequency sweeps (position of the crossover point of the shear storage modulus and shear loss modulus)
Molar mass distribution	7.2.1.3 Specification of a constant shear strain (via the time-based curve of the shear stress relaxation modulus) 7.3.4 Frequency sweeps (position of the crossover point of the shear storage modulus and shear loss modulus) 8.4 . Master curve by means of time/temperature shift of frequency sweeps (position of the crossover point of the shear storage modulus and shear loss modulus)
Zero shear viscosity	7.2.1.2 Specification of a constant shear stress (via the creep curve) 7.2.3.1 Specification of a variable shear rate (as a plateau value at low shear rates) 7.2.3.2 Specification of a variable shear stress (analogous to 7.2.3.1) 7.3.4 Frequency sweeps (plateau of the viscosity at low frequencies)
Recovery	8.6 Creep and recovery test
Shear compliance	7.2.1.2 Specification of a constant shear stress (via creep test) 8.6 Creep and recovery test (analogous to 7.2.1.2)
Melting temperature	7.3.2 Temperature-dependent tests in oscillation
Sol/gel transition	7.3.1 Time-dependent tests in oscillation 7.3.2 Temperature-dependent tests in oscillation
Structure recovery	8.5 Step tests for determination of the time-dependent structural change
Thixotropy	8.5 Step tests for determination of the time-dependent structural change

5 Preconditions for a measurement

5.1 Ambient conditions

The ambient conditions under which the rheometer is operated shall meet the requirements of IEC 61010.

It is recommended that the measuring device is placed in a climate-controlled room [temperature: $(23 \pm 2) ^\circ\text{C}$, relative humidity: $(50 \pm 5) \%$].

5.2 Sample preparation, filling and cleaning

The sample preparation can play a decisive role for the reproducibility of the measurement. In order to obtain comparable results, it is necessary to coordinate all aspects of the sample preparation between the interested parties, to adhere to these and to log any deviations ([Clause 9](#)).

In the ideal scenario, the sample should be filled in such a way that the smallest possible shear load is applied so that any damage to the structure is kept as minimal as possible. After the approach to the measuring gap, the sample shall fill the measuring gap completely and free of air bubbles.

With plate-plate and cone-plate measuring geometries, the sample is trimmed, which means that any excess leaked from the measuring gap is removed using a suitable tool. For specific sample types, such as asphalt binders or hotmelt adhesives, it might be needed to adjust the temperature of the trimming tool, but to use a certain temperature range for the trimming tool. For the removal of the excess sample, first a trimming position above the measuring position is approached, the sample is trimmed, and then the measuring position is approached, which produces a slightly curved sample edge. Experience shows that a gap is chosen for the trimming position that is around 1 % to 5 % larger than the gap width. During trimming, it is recommended to block the upper measuring geometry so that any structure present in the sample cannot be damaged due to twisting of the geometry. The excess sample can be scraped off e.g. using a spatula (see [Figure 3](#)). Use of a lower recess plate makes the trimming process easier.

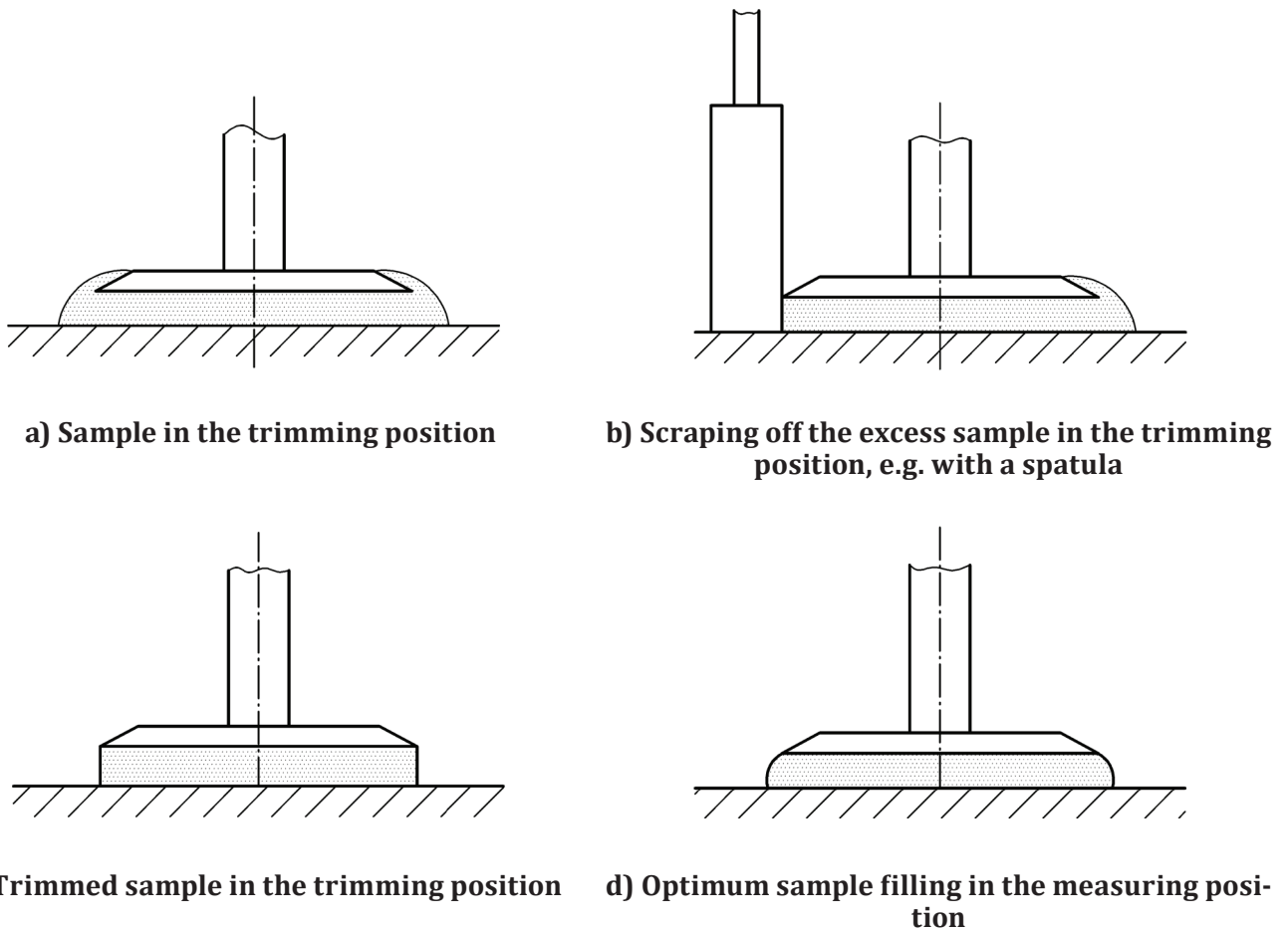


Figure 3 — Filling a plate-plate measuring geometry

After the measurement, the measuring geometry shall be completely cleaned, with no residue left behind. The measuring geometry shall be checked for cleanliness prior to the next measurement.

6 Performance of the measurement

6.1 General

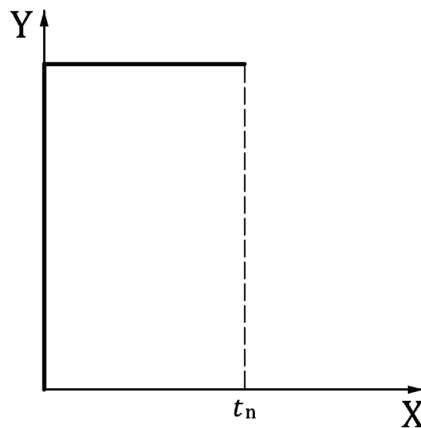
Depending on the sample and the rheological issue being investigated, a suitable measuring system and a meaningful measuring method shall be selected from the guidelines (Clause 4) and, where applicable, with the aid of preliminary tests. The measuring system used shall be adjusted, calibrated and verified. Independently of the temperature control of the sample, prior to the start of the measurement it may be necessary to allow a longer waiting time for recovery of the sample structure or to carry out a defined pre shear.

All measuring conditions and boundary conditions shall be documented in a report (see Clause 9).

6.2 Measuring profile

Once a measuring method has been chosen, the measuring profile is set. It may contain various measuring segments (basic tests – see Clause 7), each of which is defined by the specified parameter(s) and by the duration and number of measuring points.

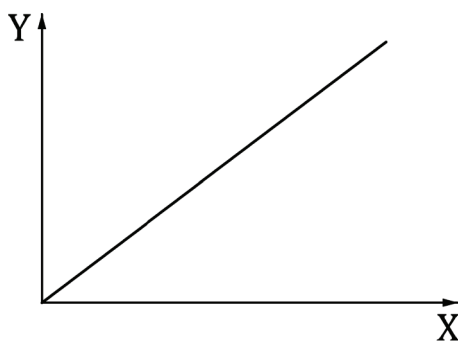
The specified parameters of a measuring segment can be constant (see Figure 4) or can be varied over time. The variation can be continuous (continuous ramp, see Figure 5 a) or discrete (stepped ramp, see Fig. 5 b).



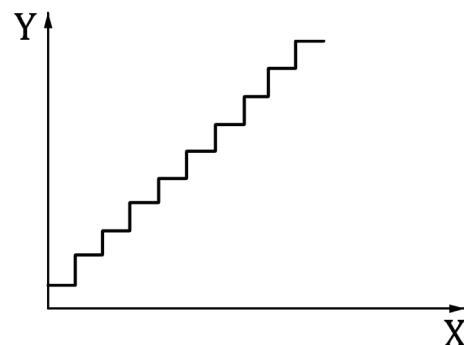
Key

X	time t
Y	specified parameter: e.g. shear rate $\dot{\gamma}$, shear stress τ , shear strain γ , temperature T
t_n	end time to switch off the specified parameter

Figure 4 — Illustration of a constant specified parameter



a) Continuous ramp



b) Stepped ramp