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Fine ceramics (advanced ceramics, advanced technical ceramics) — Measurement of viscosity of ceramic slurry by use of a rotational viscometer

Céramiques techniques — Mesure de la viscosité des céramiques en suspension au moyen d'un viscosimètre rotatif

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Foreword

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This document was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

Fine ceramics (advanced ceramics, advanced technical ceramics) — Measurement of viscosity of ceramic slurry by use of a rotational viscometer

1 Scope

This document specifies a method for measurement of the viscosity of a ceramic slurry using a rotational viscometer.

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/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

ISO 20507, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 20507 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
shear stress
stress acting on planes parallel to the direction of flow when the fluid is subject to laminar flow

Note 1 to entry: The SI units are pascals (Pa).

3.2
shear rate
gradient of laminar flow rate perpendicular to the fluid flow

Note 1 to entry: The SI units are s^{-1}.

3.3
viscosity
ratio of shear stress to shear rate

Note 1 to entry: This ratio is representative of the internal resistance of the fluid to flow.

Note 2 to entry: The SI unit of viscosity is Pa × s.

Note 3 to entry: mPa × s = 1 cP in terms of c.g.s. units [where 1 *P* = 1 g/(cm × s)].

Note 4 to entry: Viscosity is sometimes called ‘dynamic viscosity’ or ‘shear viscosity’ for clarification. The measured viscosity is conventionally called ‘apparent viscosity’ since the gradient of shear rate is not identical for all parts of the spindle.

3.4
Newtonian fluid
fluid in which shear stress is proportional to shear rate

Note 1 to entry: The ratio of shear stress to shear rate is viscosity.

3.5
non-Newtonian fluid
fluid in which shear stress is not proportional to shear rate

Note 1 to entry: The non-Newtonian behaviour can be determined by measuring shear stress using varying shear rate (or angular velocity). Viscosity of the non-Newtonian fluid changes with the shear rate, in contrast with Newtonian fluid, which has constant viscosity with the shear rate.

3.6
thixotropy
flow behaviour of fluid that shows time dependence, such that the apparent viscosity decreases with time for a constant shear rate, and recovers slowly with withdrawal of the shear force

3.7
shear thickening
<dilatant> increase of shear viscosity of fluid when the shear rate (or stress) is increased

3.8
shear thinning
<pseudoplasticity> decrease of shear viscosity of fluid when the shear rate (or stress) is increased

3.9
Bingham plastic
linear shear stress/shear rate relationship starting from a finite yield stress value below which the fluid does not flow

3.10
flow curve
curve showing the relationship between shear rate and shear stress

Note 1 to entry: See Figure 1 a).

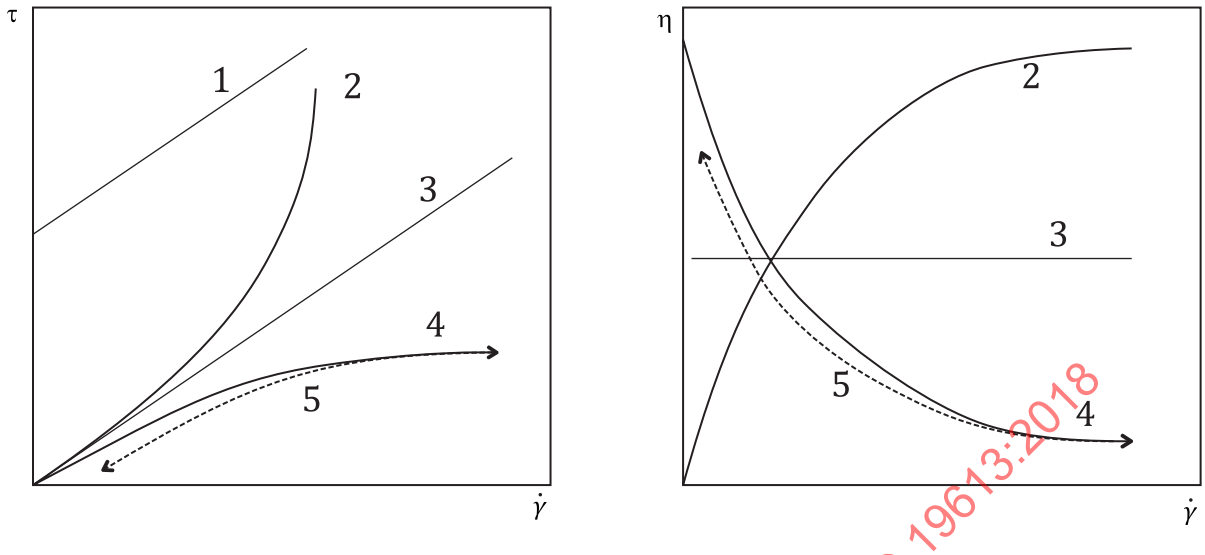
3.11
viscosity curve
curve showing the relationship between shear rate (or shear stress) and viscosity

Note 1 to entry: See Figure 1 b).

3.12
ceramic slurry
suspension of grinding frit, clay or ceramic powders mixed or dispersed in water or other liquid

Note 1 to entry: ceramic slurry is used in ceramic processes such as dip coating, spray coating, screen printing, slip casting, tape casting, spray drying and polishing.

Note 2 to entry: The category may include low-viscosity colloids with small particles with sizes in the nanometre or micrometre range, and high-viscosity pastes with inhomogeneous mixtures.



a) Flow curve **b) Viscosity curve**

Key

$\dot{\gamma}$	shear rate	2	shear thickening (dilatant)
τ	shear stress	3	Newtonian
η	viscosity	4	shear thinning (pseudoplastic)
1	Bingham plastic	5	thixotropy (time dependent)

Figure 1 — Summary of rheological behaviours

4 Principle

4.1 Rotational viscometer with defined shear rate

The viscosity, η, of a ceramic slurry measured using a rotational viscometer with a defined shear rate is determined using Formula (1):

$$\eta = \frac{\tau}{\dot{\gamma}} \tag{1}$$

where

η is the viscosity, in Pa × s;

τ is the shear stress, in Pa;

$\dot{\gamma}$ is the shear rate, in s^{-1}.

NOTE Symbols are in accordance with ISO 80000-4.

4.2 Single cylinder viscometer

A spindle of cylindrical or disk-like shape is driven in the ceramic slurry at a fixed angular velocity. Torque is developed from the fluid resistance, and it depends on the viscosity of the slurry. Theoretically,

shear rate (or shear stress) cannot be determined, but viscosity can be measured as a function of angular velocity.

NOTE The gradient of shear rate is not identical for all parts of the spindle. Therefore, the measurement result is conventionally called apparent viscosity because it is not the viscosity determined from the gradient of a known shear rate.

5 Apparatus

5.1 General

Rotational viscometers have various measuring geometries. Representative rotational viscometers having a defined shear rate are a coaxial cylinder system (Annex A), a cone and plate system (Annex B), and a parallel plates system (Annex C). In contrast, single cylinder viscometers without a defined shear rate (Annex D) are able to give an apparent viscosity relatively quickly and reproducibly, and to provide a comparison between samples.

5.2 Rotational viscometer with defined shear rate.

5.2.1 Measuring system

The measuring system shall consist of two rigid, symmetrical, coaxial surfaces between which the fluid whose viscosity is to be measured is placed. One of these surfaces shall rotate while the other remains at rest. The measuring system shall be such that the shear rate can be defined for each measurement. A torque-measuring device shall be connected to one of the surfaces, thus permitting determination of the torque required to overcome the viscous resistance of the fluid. Suitable measuring systems are the coaxial double cylinder system (Annex A), cone and plate system (Annex B), and parallel plate system (Annex C), as prescribed by JIS 8803 and ISO 20507. The dimensions of each measuring system are detailed in Annexes A, B, and C, which are designed to ensure a geometrically similar flow field for all types of measurement and all common types of basic instrument.

5.2.2 Basic capacity of the instrument

The basic instrument shall be designed to permit alternative rotors and stators to be fitted, for the generation of a range of defined rotational frequencies (stepwise or continuously variable), and for measuring the resulting torque, or vice versa (i.e. measurement of the necessary angular velocity to generate a defined torque). The apparatus shall have a torque-measurement accuracy of 2 % of the full-scale reading. Within the regular working range of the instrument, the accuracy of rotational-frequency measurement shall be 2 % of the measured value. The repeatability of viscosity measurement shall be ±2 %.

NOTE By using different measuring systems and rotational frequencies, most commercial instruments cover a viscosity range of at least 10^{-2} Pa × s to 10^{3} Pa × s.

5.2.3 Installation of rotational viscometer

5.2.3.1 Coaxial double cylinder viscometer

a) The axis of rotation of the viscometer is installed vertically.

b) The surface of the sample is maintained at a lower position than the surface of the thermostat or the top of the liquid jacket in the outer cylinder.

c) The inner cylinder is placed with its axis coinciding with the axis of the outer cylinder; the distance from the bottom of the inner cylinder to the bottom of the outer cylinder is more than 5 mm. The upper surface of the inner cylinder is more than 5 mm below the surface of the sample.

5.2.3.2 Cone and plate viscometer

a) The axis of rotation of the viscometer is installed vertically, and the plate horizontally.

b) The surface of the sample is maintained at a lower position than the surface of the thermostat or the top of the liquid jacket in cone and plate.

c) The cone is adjusted so its tip lies on the axis of rotation, and the centre of the parallel plate is also positioned on the axis.

NOTE The tip of the cone may cause friction if it is in contact with the plate. The tip means the imaginary tip of the cone obtained by extrapolation if the end is cut flat.

5.3 Single cylinder viscometer.

The single cylinder viscometer has two main components relevant to this standard.

5.3.1 Main body of single cylinder viscometer

It is necessary to select the proper model of viscometer for the viscosity to be measured, each model being suited to a specific range of viscosities. Brookfield provides single cylinder viscometers that are classified as LV (low viscosity), RV (medium viscosity) and HA/HB (high viscosity), as prescribed in ISO 2555. Other manufacturers also provide equivalent systems. The suitable uses of cylinder-type viscometers are also determined by special spindles such as Krebs or vane types. Some single cylinder viscometers do not have a demarcation point between the body/shaft and spindle to define the amount of rotating surface submersed, so the shear rate is not fully determined in that case.

5.3.2 Spindle

Do not use spindles that are corroded or show eccentric rotation. Choose the spindle taking into account the value of viscosity to be measured, based on the model of viscometer and angular velocity. It is preferable to use the same spindle to compare samples, even when the measured value is outside the spindle's optimum range, rather than change spindles and rely on instrument accuracy. For an unknown sample without any prior information, choose the proper exchangeable spindle that gives a proper magnitude of torque when tested through the full range of angular velocity.

5.4 Temperature-control device.

5.4.1 Thermostat

The circulation temperature of a constant temperature bath or electric heated wall temperature of a constant temperature bath shall be maintained within ± 0,2 °C for temperatures between 0 and 50 °C. Wider tolerances (e.g. ±0,4 °C) are often sufficient outside this temperature range. Closer tolerances (e.g. ±0,1 °C) may be necessary for precise measurements.

5.4.2 Thermometer

The accuracy of the thermometer shall be ± 0,2 °C or the apparatus shall contain a device to measure temperature with equivalent accuracy.

6 Calibration of the rotational viscometer

Viscometers shall be calibrated periodically, e.g. by measuring the torque characteristics or by using reference liquids of known viscosity (Newtonian fluids). Standard pre-mixed liquids are preferable to the use of distilled water and diluted standard solutions. If the best-fit straight line drawn through the measured points for the reference liquid does not pass through the origin of the coordinate system, within the limits of the accuracy of the method, the procedure and the apparatus shall be checked more

extensively in accordance with the manufacturer's instructions. The viscosity of a standard liquid used for calibration should be in the same range as the viscosity of samples to be measured.

7 Measurement condition

7.1 Temperature

Generally, because of the temperature dependence of viscosity, measurements for comparison purposes shall be carried out at the same temperature. If measurements are required to be made at ambient temperature, a measurement temperature of 23,0 °C ± 0,2 °C is preferred.

7.2 Selection of geometry and/or spindle (shear rate or angular velocity)

Choose the suitable viscometer, spindle and geometry to measure the viscosity of the sample. Measure the viscosity at defined shear rates (or rotational frequencies) through the full possible measurement range. Viscosity should be measured at more than four defined shear rates to determine a flow curve or viscosity curve, preferably covering a wide range of shear rates (or rotational speeds). In measuring a non-Newtonian sample, it is necessary to consider the possible range of viscosity when choosing shear rate or rotational speed.

NOTE The occurrence of Taylor flow poses a limit to the highest shear rate used for low-viscosity ceramic slurry, if using a rotational viscometer with inner cylinder rotation.

8 Pretreatment of the sample

8.1 General

Pretreatment of the sample shall include tempering of the sample to the desired measurement temperature using a constant temperature bath. Any additional pretreatment shall be performed under controlled temperature conditions.

If any settling or sedimentation of solid particles has occurred, it is necessary to disperse the sample constituents by stirring, mixing or shaking. For some samples a simple manual shaking could be sufficient to homogenize the slurry, but this procedure is hardly reproducible. Therefore, it is advisable to perform this step using equipment such as mechanical stirrers or ultrasonic baths. Ceramic slurry may generate bubbles during mixing, changing the viscosity, so care should be taken during this step.

8.2 Degassing

Although it is effective to degas by decompression if bubbling has occurred, it is not recommended because this may change the after-preparation properties of the sample. It has to be reported if performed.

8.3 Mixing and dispersion by ultrasonication

Dispersion by rapidly stirring the sample for 5 min before measurement or ultrasonication may increase the temperature of ceramic slurry. This is not a recommended treatment since it may change the slurry viscosity, a result of irreversible changes in the polymer dispersant or binder additives. Moreover, it has to be reported if it is unavoidable for dispersion.

9 Measurement procedure

9.1 Sampling

Take the sample carefully without pretreatment when there is no settling or sedimentation of solid particles is observed. Transfer the sample into the viscosity measurement container, taking care not to create bubbles. The sample in the container of the viscometer needs sufficient time to recover its structure prior to measurements; this varies with the slurry. However, it has to be reported if it is unavoidable for dispersion. Single cylinder viscometers such as the Brookfield generally cannot fully break down the thixotropic structure of many ceramic slips and would give anomalously high and unstable viscosity readings. Slips need to be in a stable agitated condition before testing. A recommended preparation procedure is to mechanically stir slip samples for 5 min before measurement unless the sample has been kept agitated.

9.2 Measurement at fixed shear rate or angular velocity

Allow enough time in the viscosity measurement for stabilization with the selected measurement system and the fixed shear rate (or angular velocity) to cover the various samples being compared. In the case of a flow curve, the measurement shall be started with increasing shear rate, followed by decreasing rate, until the maximum and minimum shear rates are reached, respectively. In this case, it should be measured in as short a period as possible.

NOTE If the stabilization time is short at the defined shear rate (or angular velocity), the viscosity is not the value in equilibrium at the shear rate (or angular velocity).

It is desirable to seal the container to prevent drying if there is concern about drying during measurement.

9.3 Repeat

Make at least three determinations each with a new portion of the sample.

10 Expression of the results

Record the final viscosity point after stabilization, corresponding to the fixed shear rate (or angular velocity) which was selected. In the case of fluctuations of viscosity during measurements, i.e. without stabilization, take several points to make an average. When there is a discrepancy between the viscosity obtained while increasing the shear rate and the viscosity obtained while decreasing it, make an average for that shear rate. In the case of a thixotropic slurry, record two values each of viscosity obtained while increasing and decreasing shear rate. Record two or more determinations of viscosity, and their average, along with measurement temperature and shear rate (or angular velocity if the shear rate is not determined). The results are rounded off with three effective significant figures.

When the shear rate and shear stress are measured, the flow curve is plotted. If the flow curve passes through the origin and is linear, viscosity is given by the slope (Newtonian). If the flow curve is not proportional, viscosity depends on the shear rate or shear stress (non-Newtonian). In this case, a viscosity curve, of viscosity (η) against shear rate, is plotted.

11 Test report

The test report shall be in accordance with the provisions of ISO/IEC 17025 and shall include the following information.

a) the name of the testing establishment, the date of the test, report identification, number, operator and signatory;

b) a reference to this document, i.e. ISO 19613;

c) all details necessary for identification of the material tested;

d) the date of sampling;

e) the temperature in degrees Celsius and humidity;

f) the type of viscometer, geometry and spindle;

g) the shear rate (or an angular velocity if shear rate is not defined), the measured viscosity and the average of viscosity (P × s) when the viscosity is measured at a fixed shear rate (or angular velocity in the case of undefined shear rate);

h) the flow curve (shear stress against shear rate) or the viscosity curve (shear rate against viscosity) when viscosity has been at measured values of shear rate;

i) details of sample preparation, any other unusual points, the stabilization time and torque if necessary;

j) the date of measurement (YYYY-MM-DD).

Annex A
(informative)

Coaxial double cylinder system

A.1 General

This annex provides supplementary information related to the measurement equipment, and is not a requirement of this document.

A.2 System characteristics

The measuring system comprises a cup (i.e. the outer cylinder with a closed bottom) and a bob (i.e. the inner cylinder with the shaft as shown in Figure A.1). The bob may act as the rotor and the cup as the stator, or vice versa.

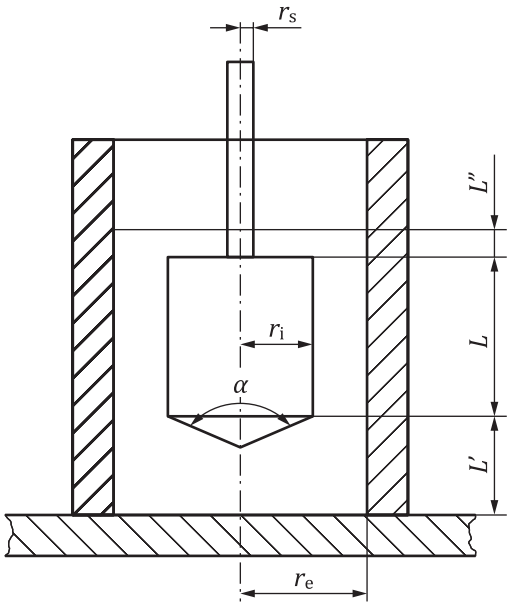
Viscosity measurement by a coaxial double cylinder viscometer shall include the following aspects:

a) Besides viscosity measurements, the flow curves of non-Newtonian liquids can be obtained by a series of measurements with varied angular velocity (or torque).

b) A slip velocity (or slip stress) relatively close to the theoretical value can be observed.

A.3 Standard geometry

The physical dimensions for a given viscometer of this type shall be based on the following ratios (Table A.1), ensuring a geometrically similar flow field for all tasks employing the basic instrument design:



Key

L length of the inner cylinder, in m
L' distance between the bottom edge of the inner cylinder and the bottom of the outer cylinder, in m
L'' length of the immersed part of the shaft, in m
r_i radius of the inner cylinder, in m
r_e radius of the outer cylinder, in m
r_s radius of the shaft, in m
α apex angle of the cone at the bottom of the inner cylinder, in degrees (°)
δ ratio of the radius of the outer cylinder to that of the inner cylinder

Figure A.1 — Standard geometry coaxial double cylinder system

Table A.1 — Example of dimensional ratios of coaxial double cylinder viscometer

Features	Recommended dimensions	Dimensional tolerance
$\delta = r_e/r_i$	1,08	≤1,2
L/r_i	3	≥3
L'/r_i	1	≥1
L''/r_i	1	—
r_s/r_i	0,3	—
α	120°	$90° \leq \alpha \leq 150°$

A.4 Types of coaxial double cylinder viscometer

A.4.1 Constant velocity outer cylinder system

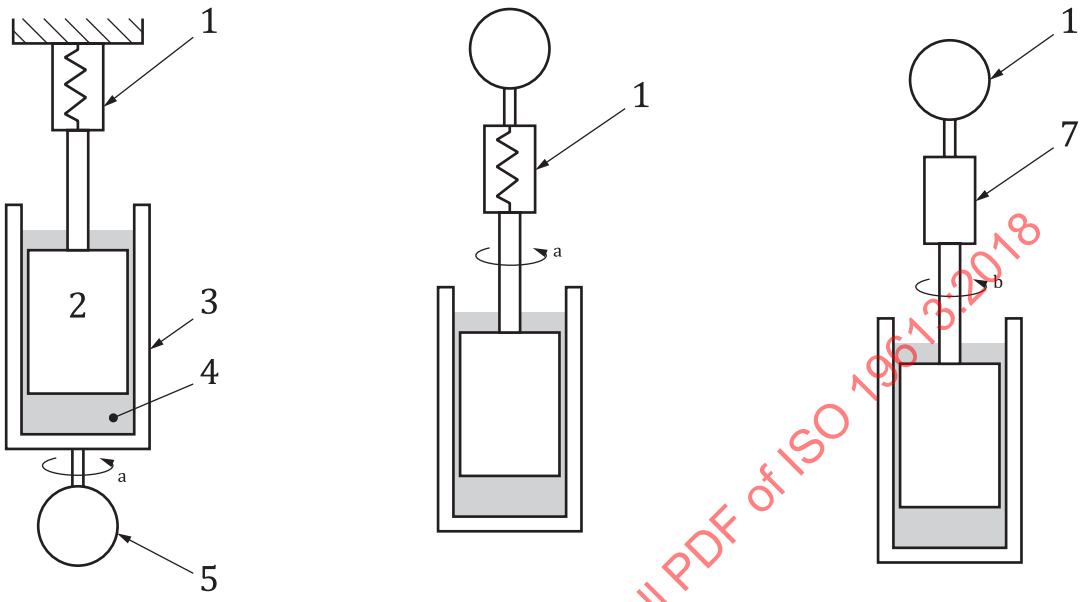
This type of viscometer rotates the outer cylinder freely at a constant angular velocity; Figure A.2 a) shows the design indicating its principle and Table A.1 specifies the ratios of its dimensions.

A.4.2 Constant velocity inner cylinder system

This type of viscometer rotates the inner cylinder freely at a constant angular velocity; Figure A.2 b) shows the design indicating its principle and Table A.1 specifies the ratios of its dimensions.

A.4.3 Constant torque system

This type of viscometer rotates the inner cylinder freely at a constant torque; Figure A.2 c) shows the design indicating its principle and Table A.1 specifies the ratios of its dimensions.



a) Constant velocity outer cylinder system **b) Constant velocity inner cylinder system** **c) Constant torque system**

Key

1	torque detector	5	constant angular velocity
2	inner cylinder	6	motor
3	outer cylinder	7	rotary-detection of movement sensor
4	specimen	8	constant torque

Figure A.2 — Coaxial double cylinder viscometer

Annex B
(informative)

Cone and plate system

B.1 General

This annex provides supplementary information related to the measurement equipment, and is not a requirement of this document.

B.2 System characteristics: features and geometry

The measuring system consists of a rotating cone and shaft mounted above a stationary plate (Figure B.1).

The angle, α, between the cone and the plate shall be as small as possible, preferably not greater than 1° and in no case greater than 4°. When the angle is greater than 1°, this shall be stated in the test report. The advantage of the cone and plate system is that, at such small angles, the shear rate across the conical gap may be considered constant.

Measurement of viscosity using the cone and plate viscometer has the following aspects:

a) In addition to measurements of viscosity, it is possible to directly obtain flow curves of non-Newtonian liquids using varied angular velocity (or torque). This is due to an almost uniform shear rate field in all parts of the sample.

b) A relatively small amount of sample is sufficient for this measurement. As a result, the temperature of the sample can be charged in a short time.

c) However, because the gap between the cone and plate is small, the particle size of the dispersed substance in liquid may, in some cases, prevent the apparatus being used as a viscometer.

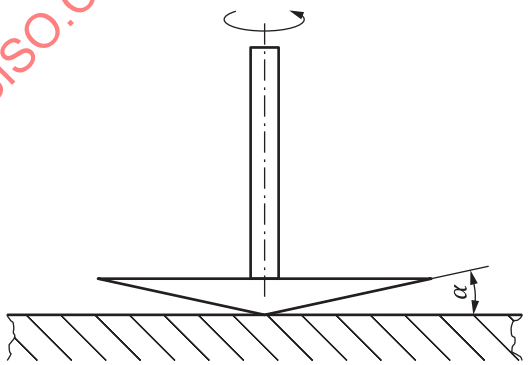


Figure B.1 — Standard geometry cone and plate system

Annex C
(informative)

Parallel plate system

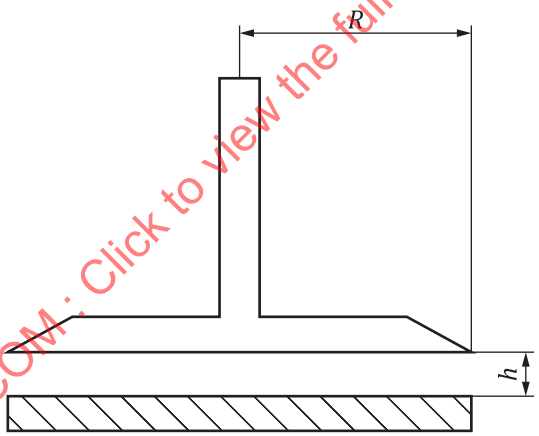
C.1 General

This annex provides supplementary information related to the measurement equipment, and is not a requirement of this document.

C.2 System characteristics: features and geometry

The measurement system consists of a rotational plate and a fixed plate (Figure C.1). The distance between the two plates should preferably be in the range 0,3 mm to 10 mm. It is recommended that the plate separation should exceed 10 times the maximum particle size in the slurry.

Shear rate is not constant with position, as it is zero at the centre of rotation, increasing toward the edge of the plates.



Key

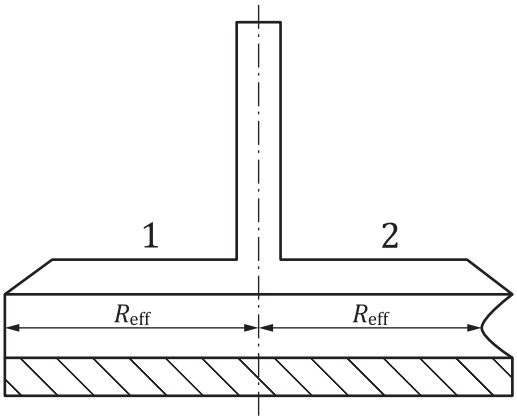
R radius of the plate

h distance between the plates

Figure C.1 — Standard geometry parallel plate system

C.3 Notes

In this method, a flow curve can be measured using a small amount of sample. However, the measurement may be influenced by the small cross section between the plates, sample charging and sedimentation of solid particles in comparison with other methods. In addition, a slurry that is easy to dry and precipitate needs rapid measurement to prevent it drying and forming a seal. Drying at the edge of the plate is observed with this method, and care is required to avoid influencing the measurement results (Figure C.2).



Key

1 normal sample filling

2 underestimated sample filling

R_{eff} effective radius

Figure C.2 — State of charging of slurry in a parallel plate system

When the viscosity of a high-density suspension is measured, there is particle slippage on the plate surface. This phenomenon is called wall slip; the measured viscosity is lower than the real viscosity of the sample resulting from the thin layer formed adjacent to the surface. Sandpaper attached to the plate or a surface with a cross-hatched grooved pattern machined into the face will provide a rough surface, minimizing wall slip. In some cases, the measured viscosity has lower reliability if the sample between the plates begins to escape above a certain shear rate. This phenomenon is called edge fracture. A sample exhibiting this phenomenon should be carefully loaded to pre-shear during setup (Figure C.3).

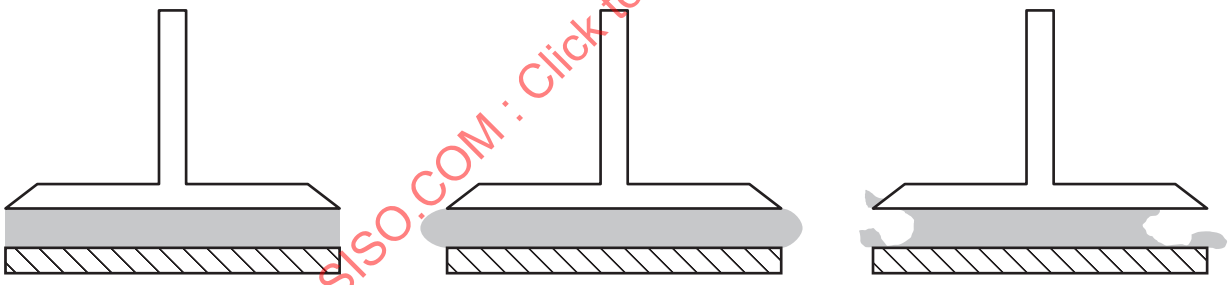


Figure C.3 — Phenomenon of edge fracture in a parallel plate system

Annex D
(informative)

Single cylinder viscometer

D.1 General

This annex provides supplementary information related to the measurement equipment, and is not a requirement of this document.

D.2 System characteristics: features

Figure D.1 shows an example diagram of single cylinder viscometer. Use of a single cylinder type viscometer has the following advantages:

a) Handling is simple because of simplicity of construction compared with other types of rotational viscometers.

b) Though the slip velocity (or slip stress) cannot be theoretically determined, it is possible to ascertain the viscosity-related behaviour by varying the angular velocity (rotational velocity).

c) Because a sample container that is relatively large compared with the rotating cylinder can be used, a measurement of a disperse liquid system can be made without disturbing its properties.