Determination of viscosity of polymers and resins in the liquid state or as emulsions or dispersions using a rotational viscometer with defined shear rate (ISO 3219:1993)

English version of DIN EN ISO 3219

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This standard incorporates the English version of ISO 3219.

ICS 83.080.00

Descriptors: Plastics, polymer, resin, viscosity, testing.

Kunststoffe; Polymere/Harze in flüssigem, emulgiertem oder dispergiertem Zustand; Bestimmung der Viskosität mit einem Rotationsviskosimeter bei definiertem Geschwindigkeitsgefälle (ISO 3219:1993)

European Standard EN ISO 3219:1994 has the status of a DIN Standard.

A comma is used as the decimal marker.

National foreword

This standard has been published in accordance with a decision taken by CEN/TC 139 to adopt, without alteration, International Standard ISO 3219 as a European Standard.

The responsible German body involved in its preparation was the *Normenausschuß Anstrichstoffe und ähnliche Beschichtungsstoffe* (Paints and Varnished Standards Committee).

DIN 50014 ist the standard corresponding to International Standard ISO 291 referred to in clause 2.

Standard referred to

(and not included in **Normative reference)**

DIN 50014 Artificial climates in technical applications; standard atmospheres

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Descriptors: Plastics, polymer, resin, viscosity, testing.

English version

Plastics

Polymers/resins in the liquid state or as emulsions or dispersions

Determination of viscosity using a rotational viscometer with defined shear rate
(ISO 3219:1993)

Plastiques; polymères/résines à l'état liquide, en émulsion ou en dispersion; détermination de la viscosité au moyen d'un viscomètre rotatif à gradient de vitesse de cisaillement défini
(ISO 3219:1993)

Kunststoffe; Polymere/Harze in flüssigem, emulgiertem oder dispergiertem Zustand; Bestimmung der Viskosität mit einem Rotationsviskosimeter bei definiertem Geschwindigkeitsgefälle
(ISO 3219:1993)

This European Standard was approved by CEN on 1994-08-22 and is identical to the ISO Standard as referred to.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat: rue de Stassart 36, B-1050 Brussels

Foreword

International Standard

ISO 3219:1993 Plastics; polymers/resins in the liquid state or as emulsions or dispersions; determination of viscosity using a rotational viscometer with defined shear rate

has been taken over as a European Standard by CEN/TC 139 'Paints and varnishes' from the work of ISO/TC 35 'Paints and varnishes' of the International Organization for Standardization (ISO).

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, and conflicting national standards withdrawn, by February 1995 at the latest.

In accordance with the CEN/CENELEC Internal Regulations, the following countries are bound to implement this European Standard:

Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

Endorsement notice

The text of the International Standard ISO 3219:1993 was approved by CEN as a European Standard without any modification.

1 Scope

This International Standard specifies the general principles of a method for determining the viscosity of polymers and resins in the liquid, emulsified or dispersed state, including polymer dispersions, at a defined shear rate by means of rotational viscometers with standard geometry.

Viscosity determinations made in accordance with this standard consist of establishing the relationship between the shear stress and the shear rate. The results obtained with different instruments in accordance with this standard are comparable and apply to controlled shear as well as controlled stress instruments.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 291:1977, *Plastics — Standard atmospheres for conditioning and testing.*

3 Principle

The viscosity of a fluid sample is measured using a rotational viscometer with defined characteristics, which permits the simultaneous measurement of the shear rate used and the shear stress applied.

The viscosity η is determined using the following equation:

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

where

τ is the shear stress;

$\dot{\gamma}$ is the shear rate.

According to the International System of Units (SI), the unit of dynamic viscosity is the pascal second (Pa·s):

$$1\ \text{Pa·s} = 1\ \text{N·s/m}^2$$

NOTES

1 Symbols are in accordance with ISO 31-3:1992, *Quantities and units — Part 3: Mechanics.*

2 If the viscosity depends on the shear rate at which the measurement is made, i.e. $\eta = f(\dot{\gamma})$, the fluid is said to exhibit non-Newtonian behaviour. Fluids with a viscosity independent of the shear rate are stated to exhibit Newtonian behaviour.

4 Apparatus

4.1 Rotational viscometer

4.1.1 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 at a constant angular velocity 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 coaxial-cylinder systems and cone-and-plate systems, among others.

The dimensions of the measuring system shall be so specified as to satisfy the conditions specified in annexes A and B, which are designed to ensure a geometrically similar flow field for all types of measurement and all common types of basic instrument.

4.1.2 Basic 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. generation of a defined torque and measurement of the resulting rotational frequency).

The apparatus shall have a torque-measurement accuracy within 2 % of the full-scale reading. Within the regular working range of the instrument, the accuracy of rotational-frequency measurement shall be within 2 % of the measured value. The repeatability of viscosity measurement shall be ± 2 %.

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

The range of shear rates varies greatly with different equipment. The choice of a particular basic instrument and appropriate measuring system shall be made by considering the range of viscosities and shear rates to be measured.

4.2 Temperature-control device

The temperature of the circulating bath liquid or the temperature of the electrically heated walls shall be maintained constant to within ± 0,2 °C over the temperature range 0 °C to 50 °C and to within ± 0,5 °C at temperatures beyond these limits.

Closer tolerances (e.g. ± 0,1 °C) may be necessary for more precise measurements.

4.3 Thermometer

The accuracy of the thermometer shall be ± 0,05 °C.

5 Sampling

The sampling method, including any special methods of sample preparation and introduction into the viscometer, shall be as specified in the test standard for the product in question.

The samples shall not contain any visible impurities or air bubbles.

It samples are hygroscopic or contain any volatile ingredients, the sample containers shall be tightly closed to minimize any effects on the viscosity.

6 Test conditions

6.1 Calibration

Viscometers shall be calibrated periodically, e.g. by measuring the torque characteristics or using reference liquids of known viscosity (Newtonian fluids). If the best-fit straight line drawn through the measured points for the reference fluid 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 reference liquids used for calibration shall lie in the same range as that of the sample(s) to be measured.

6.2 Test temperature

Generally, because of the temperature dependence of the 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.

Further details shall be as specified in the test standard for the product in question.

NOTE 4 Heat is dissipated in the sample during the measurement. In the case of Newtonian liquids under adiabatic test conditions, the rate of heat dissipation is given by $\eta \cdot \dot{\gamma}^2$ (units W/m^3) and may cause an increase in the temperature of the sample.

6.3 Selection of shear rate

The shear rate shall be as specified in the test standard for the product in question.

It is advantageous in the case of all Newtonian products, and specially recommended in the case of non-Newtonian products, that measurements be made for as many shear rates (at least four) as possible, depending on the settings or programmes for rotational frequency (or torque in the case of fixed-shear-stress instruments) allowed by the basic instrument, and at widely differing shear rates so that a comprehensive graph of viscosity vs. shear rate may be drawn.

In order to compare viscosities measured on different instruments, it is recommended that the shear rate be selected from a series consisting of the following values:

$1{,}00\ s^{-1}$, $2{,}50\ s^{-1}$, $6{,}30\ s^{-1}$, $16{,}0\ s^{-1}$, $40{,}0\ s^{-1}$, $100\ s^{-1}$, $250\ s^{-1}$;

or

$1{,}00\ s^{-1}$, $2{,}50\ s^{-1}$, $5{,}00\ s^{-1}$, $10{,}0\ s^{-1}$, $25{,}0\ s^{-1}$, $50{,}0\ s^{-1}$, $100\ s^{-1}$;

and these values multipled or divided by 100.

If a given basic instrument does not permit these values to be selected, shear-rate values shall be selected from the viscosity curve.

In the case of non-Newtonian fluids, the measurements shall be started with increasing shear rates, i.e. increasing speed until the maximum speed is reached, and then decreasing the speed, making further measurements at decreasing shear rates.

NOTE 5 In this way, thixotropy and rheopexy can be assessed, although only qualitatively.

In the case of thixotropic and rheopexic liquids, the test conditions shall be as specified in the test standard for the product in question.

Prior to measurement, the sample in the viscometer shall have sufficient time to recover any thixotropic structure. This time will depend on the nature of the particular sample.

If the readings at increasing and decreasing shear rate show only random differences, the two readings may be averaged. If a consistent difference is observed, as in the case of thixotropic systems, both values shall be recorded.

6.4 Procedure

Unless otherwise specified by the test standard for the product in question, make three determinations in accordance with annex A or B, as applicable, each with a new portion of the sample.

For the evaluation of the viscosity measurements, see annexes A and B.

If the viscosity of a particular product is required to be measured at different temperatures, determine the viscosity curve at each temperature with the same sample portion, provided the measuring system of the size chosen remains suitable (the fact that the viscosity varies with temperature means that it may be necessary to change the measuring system).

For each repeat determination, use a new sample if possible, and determine the viscosity by commencing with increasing temperatures and sub-sequently using decreasing temperatures.

Prior to measurement, the sample in the viscometer should have sufficient time to attain the required temperature.

7 Expression of results

Calculate the viscosity η in pascal seconds, using the relationships given in the instruction manual or the tables or nomograms attached to the apparatus. Calculate the arithmetic mean of the three determinations.

When stating viscosity values, give, between parentheses, the temperature and shear rate at which the viscosity was measured, e.g.

$$\eta\left(23\ °C,\ 1\ 600\ s^{-1}\right) = 4{,}25\ Pa{\cdot}s$$

Where viscosity measurements are made at different temperatures and shear rates, plot curves to demonstrate these relations.

8 Test report

The test report shall include the following information:

a) the number and year of publication of this International Standard;

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

c) the date of sampling;

d) the test temperature in degrees Celsius;

e) details of the preparation of the sample;

f) a description of the viscometer measuring system used;

g) a viscosity curve plotted from all the corresponding values of the shear stress τ, in pascals, and the shear rate $\dot{\gamma}$, in reciprocal seconds, obtained;

h) in the case of single-point measurements, the viscosity, including the temperature and shear rate at which the determination was carried out (see clause 7);

i) in the case of thixotropic and rheopexic liquids, the conditions, e.g. ramp times and total shear, used;

j) the measurement times (i.e. the periods of time which elapsed, after the required shear rate had been reached, before even reading was made);

k) the individual results of the viscosity determinations, in pascal seconds or millipascal seconds, and the arithmetic mean of these results;

l) any test conditions that have been agreed upon but which deviate from this International Standard, e.g. the use of measuring systems of different dimensions;

m) the date of the test.

Annex A
(normative)

Coaxial-cylinder viscometers

A.1 System characteristics

The measuring system comprises a cup (i.e. the outer cylinder with a closed base) 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*.

A.2 Methods of calculation

The shear stress τ and the shear rate $\dot{\gamma}$ are not constant over the annular cross-section of rotational viscometers with coaxial cylinders, but decrease from the inside to the outside (Searle type) or *vice versa* (Conette type). Moreover, the variation in $\dot{\gamma}$ also depends on the rheological properties of the material under test.

It is convenient to calculate τ and $\dot{\gamma}$ as "representative" values[1] which do not occur at the surface of the measuring system itself (i.e. at the external radius r_e or the internal radius r_i), but at a certain distance inside the annulus. It has been shown (both by theory and experiment) that the representative values τ_{rep} and $\dot{\gamma}_{rep}$, as calculated from equations (A.2) and (A.3), describe, to a very good approximation, the flow behaviour of fluids with a local power law index in the range 0,3 to 2.

The shear stress, expressed in pascals, is calculated, using equations (A.1) and (A.2), from the torque M measured at the inner cylinder (i.e. at radius r_i) or at the outer cylinder (i.e. at radius r_e), these two radii being expressed in metres.

$$\tau_i = \frac{M}{2\pi L r_i^2 C_L}; \quad \tau_e = \frac{M}{2\pi L r_e^2 C_L} \qquad \ldots (A.1)$$

$$\tau_{rep} = \frac{\tau_i + \tau_e}{2} = \frac{1+\delta^2}{2\delta^2} \times \tau_i = \frac{1+\delta^2}{2} \times \tau_e$$

$$= \frac{1+\delta^2}{2\delta^2} \times \frac{M}{2\pi L r_i^2 C_L} \qquad \ldots (A.2)$$

where, in addition to the above-mentioned quantities

M is the torque, expressed in newton metres;

δ is the ratio of the radius of the outer cylinder to that of the inner cylinder;

L is the length, in metres, of the inner cylinder;

C_L is an end-effect correction factor that accounts for the torque acting at the end faces of the measuring system (this correction factor depends on the geometry of the measuring system and on the rheological properties of the liquid, and must be determined experimentally for each type of measuring-system geometry).

The representative shear rate, expressed in radians per second, is obtained from

$$\dot{\gamma}_{rep} = \omega \times \frac{1+\delta^2}{\delta^2 - 1} \qquad \ldots (A.3)$$

where ω is the rotational velocity, in radians per second.

If the rotational frequency n is expressed in revolutions per minute, then

$$\omega = \frac{2\pi n}{60} = 0{,}104\ 7\ n$$

A.3 Standard geometry (see figure A.1)

The dimensions of this type of measuring system matched to a given viscometer are based on the following ratios ensuring a geometrically similar flow field for all tasks and basic instruments:

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1) See Giesekus H. and Langer G.: Determination of the true flow curves of non-Newtonian liquids and plastics using the representative viscosity method, *Rheologica Acta*, **16**, 1977, No. 1, pp. 1-22.

$\delta = \frac{r_e}{r_i} = 1{,}084\ 7$

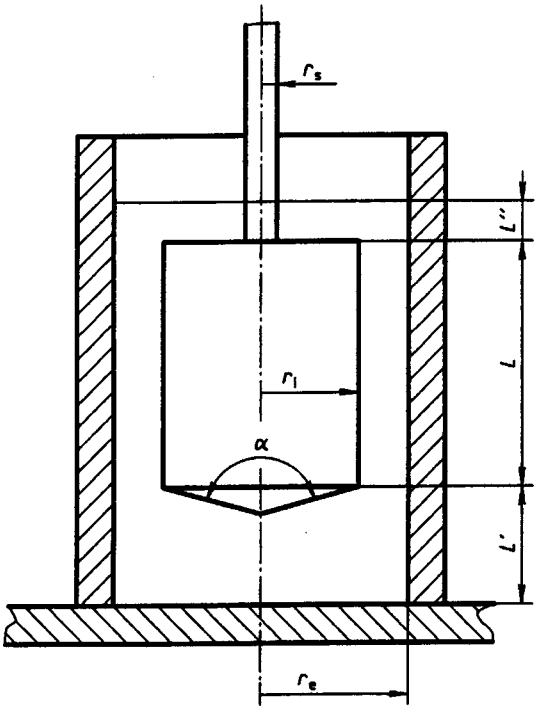
$\frac{L}{r_i} = 3$

$\frac{L'}{r_i} = 1$

$\frac{L''}{r_i} = 1$

$\frac{r_s}{r_i} = 0{,}3$

$\alpha = 120°$



δ is the ratio of the radius of the outer cylinder to that of the inner cylinder;

L is the length of the inner cylinder;

L' is the distance between the bottom edge of the inner cylinder and the bottom of the outer cylinder;

L'' is the length of the immersed part of the shaft;

r_i is the radius of the inner cylinder;

r_e is the radius of the outer cylinder;

r_s is the radius of the shaft;

α is the apex angle of the cone at the bottom of the inner cylinder.

NOTES

1 The cone at the lower end of the inner cylinder facilitates insertion of the cylinder into the cup filled with the liquid under test without causing the formation of air bubbles.

2 Coaxial-cylinder systems require precise alignment of the axes of the inner and outer cylinders.

Figure A.1 — Standard-geometry coaxial-cylinder system

The sample volume depends only on the radius r_i and is given by the equation:

$$V = 8{,}17 r_i^3 \quad \ldots (A.4)$$

For measuring systems with this standard geometry, the end-effect correction factor C_L is independent of the radius r_i. For Newtonian liquids,

$$C_L = 1{,}10$$

has been found as an empirical value. For non-Newtonian liquids, C_L is not constant, but depends on the shear rate $\dot{\gamma}$ and on the rheological properties of the liquid.

NOTE 6 For shear-thinning liquids, C_L may reach values of up to 1,2 at certain shear rates. For visco-plastic liquids exhibiting a yield value, C_L values of up to 1,28 have been observed at low shear rates.

Using $C_L = 1{,}10$ (Newtonian liquids), $\delta^2 = 1{,}176\ 57$ and $\tau_{rep} = 0{,}925\tau_i = 1{,}088\tau_e$, the following numerical relationships are obtained if the representative shear stress τ_{rep} is expressed in pascals, the torque M in newton metres, the representative shear rate $\dot{\gamma}_{rep}$ and the rotational velocity ω in radians per second, the internal radius r_i in metres and the rotational frequency n in reciprocal minutes:

$$\tau_{rep} = 0{,}044\ 6 \times \frac{M}{r_i^3} \quad \ldots (A.5)$$

$$\dot{\gamma}_{rep} = 12{,}33\ \omega = 1{,}291 n \quad \ldots (A.6)$$

A.4 Other geometries

If for any reason the standard geometry cannot be used, measuring systems of other dimensions may be chosen. In order to use the methods of calculation given in A.2, the following requirements shall be met:

$$\delta = \frac{r_e}{r_i} \leqslant 1{,}2$$

$$\frac{L}{r_i} \geqslant 3$$

$$\frac{L'}{r_i} \geqslant 1$$

$$90° \leqslant \alpha \leqslant 150°$$

The end-effect correction C_L has different (usually higher) values from those with the standard geometry.

NOTE 7 The choice of a narrow annulus, e.g. $\delta \leqslant 1{,}2$, ensures that the simple and easily quantifiable concept of representative viscosity is a good approximation. It can be shown that the representative viscosity at the corresponding shear rate differs only slightly from the true value (by $\leqslant 3{,}5$ %). For the standard geometry, the error is in general much lower.

A.5 Treatments of results

Using a rectangular coordinate system with linear scales, plot the torque readings from the instrument and the corresponding values of rotational frequency n. Draw a smooth curve to fit the points. Read off this curve pairs of values for torque and rotational frequency and convert them to the corresponding values for shear stress and shear rate using the following equations:

equation (A.2) or (A.5) for shear stress τ;

equation (A.3) or (A.6) for shear rate $\dot{\gamma}$.

If possible, select those values of τ or $\dot{\gamma}$ that form a geometrical progression. The plot of these pairs of quantities is the curve $\tau = f(\dot{\gamma})$.

If this flow curve is a straight line passing through the origin, the viscosity can be expressed as a single value given by the slope, i.e. the ratio $\tau/\dot{\gamma}$ for any pair of values $(\tau, \dot{\gamma})$.

If the curve is non-linear, corresponding values of τ and $\dot{\gamma}$ can be read off and the ratio $\tau/\dot{\gamma}$ plotted against τ or $\dot{\gamma}$ as a shear-stress- or shear-rate-dependent viscosity [viscosity function $\eta(\tau)$ or $\eta(\dot{\gamma})$].

Round all measured and calculated values to three significant figures, e.g.

$\dot{\gamma} = 42{,}8\ s^{-1}$; $\eta = 0{,}318$ Pa·s

$\tau = 13{,}6$ Pa; $\theta = 23{,}0$ °C

Annex B
(normative)

Cone-and-plate system

B.1 System characteristics

The measuring system consists of a rotating cone and shaft and a stationary plate (see 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.

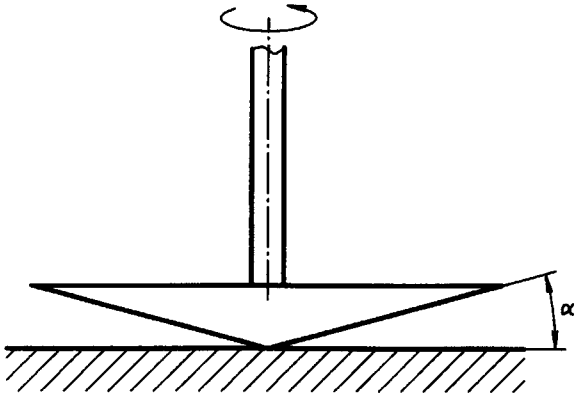


Figure B.1 — Cone-and-plate system geometry

B.2 Method of calculation

If $\alpha \leqslant 0{,}05$ rad (i.e. $\alpha \leqslant 3°$), the following equations are applicable for the calculation of the shear stress τ and shear rate $\dot{\gamma}$.

$$\tau = \frac{3M}{2\pi r^3} \quad \ldots (B.1)$$

$$\dot{\gamma} = \frac{\omega}{\alpha} \quad \ldots (B.2)$$

where

M is the torque, in newton metres;

r is the radius, in metres, of the cone;

α is the angle, in radians, between the cone and the plate (1 rad = 180°/π);

ω is the angular velocity, in radians per second.

In order to avoid friction caused by contact between the cone and the plate, truncated cones can be used. This configuration can also be used if the liquid under test contains solid particles.

Cone-and-plate systems require precise alignment of the cone axis perpendicular to the plate and also exact setting of the point of contact between the apex of the cone and the plate (or exact setting of the gap in the case of truncated cones).

Precise filling of the gap between cone and plate is also important (do not overfill or underfill).

NOTE 8 The fact that the gap width changes with temperature will also have to be taken into account.