INTERNATIONAL STANDARD

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Plastics — Polymers/resins in the liquid state or as emulsions or dispersions — Determination of viscosity using a rotational viscometer with defined shear rate

Plastiques — Polymères/résines à l'état liquide, en émulsion ou en dispersion — Détermination de la viscosité au moyen d'un viscosimètre rotatif à gradient de vitesse de cisaillement défini

Reference number
ISO 3219:1993(E)

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 3219 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

This second edition cancels and replaces the first edition (ISO 3219:1977), of which it constitutes a technical revision.

It was prepared in liaison with ISO/TC 45, *Rubber and rubber products*, and ISO/TC 35, *Paints and varnishes*.

Annexes A and B form an integral part of this International Standard.

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Plastics — Polymers/resins in the liquid state or as emulsions or dispersions — Determination of viscosity using a rotational viscometer with defined shear rate

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.

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

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.