INTERNATIONAL STANDARD 3219

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION •МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Plastics — Polymers in the liquid, emulsified or dispersed state — Determination of viscosity with a rotational viscometer working at defined shear rate

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

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 3219 was developed by Technical Committee ISO/TC 61, *Plastics,* and was circulated to the member bodies in November 1975.

It has been approved by the member bodies of the following countries :

Australia	Israel	Sweden
Austria	Italy	Switzerland
Belgium	Japan	Turkey
Brazil	Netherlands	United Kingdom
Czechoslovakia	New Zealand	U.S.A.
Finland	Poland	U.S.S.R.
France	Romania	
Hungary	Spain	

The member body of the following country expressed disapproval of the document on technical grounds :

Germany

Plastics — Polymers in the liquid, emulsified or dispersed state — Determination of viscosity with a rotational viscometer working at defined shear rate

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for determining the viscosity of polymers in the liquid or in the liquid-like state, with a rotational viscometer in which the theoretical shear rate employed in the test is defined (see note).

This International Standard is a general specification for rotational viscometers working at a defined shear rate. A separate document specific to each product (or group of products) for which the viscosity is required should define the particular conditions of test for the product : shear rate, temperature, etc. Such a document could be an individual standard for the product, a commercial contract, etc.

In principle, the results obtained with different instruments covered by the present International Standard are, if not identical, at least in close agreement.

NOTE — Equations given in this International Standard are theoretical but the shear rate calculated with them is very near the true shear rate of a Newtonian liquid provided that the apparatus characteristics agree with the present International Standard.

2 REFERENCES

ISO 3, *Preferred numbers — Series of preferred numbers.*

ISO 31/III, *Quantities and units of mechanics.*

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

3 PRINCIPLE

Measurement of viscosity using a rotational viscometer with defined characteristics, which permits the simultaneous measurement of the applied shear rate (see note to clause 1).

4 DEFINITION, SYMBOLS AND UNITS

viscosity (symbol η) (see ISO 31/III) : When a fluid is sheared between two parallel planes one of which is moving in its own plane linearly and uniformly with respect to the other, the viscosity of the fluid is defined by Newton's equation :

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

where

η is the viscosity;

τ is the shear stress;

$\dot{\gamma} = \frac{dv}{dz}$ is the shear rate;

v being the velocity of one plane with respect to the other, and

z the co-ordinate normal to the planes.

NOTE — Products with a viscosity independent of the shear rate at which the measurement is made are considered as having Newtonian behaviour and are called "Newtonian".

Others are considered as having non-Newtonian behaviour and are called "non-Newtonian".

The dimension of viscosity is $ML^{-1}T^{-1}$ and the SI unit is the newton second per square metre ($N{\cdot}s/m^2$) or pascal second (Pa·s).*

5 APPARATUS

5.1 Viscometers, coaxial cylinder, cone-and-plate or double-cone, or any combination of two of these, with the following characteristics :

5.1.1 For coaxial cylinder viscometers, where the diameters of the external and internal (stator or rotor) cylinders are respectively d_e and d_i, the value of d_e/d_i shall be as small as possible, preferably not greater than 1,1 and in no case greater than 1,5. Where the ratio is greater than 1,1, this shall be stated in the report and if possible a correction shall be made, this also being indicated in the report (see note).

* $1\ N{\cdot}s/m^2 = 1\ Pa{\cdot}s = 10^3$ cP (centipoise).

Moreover, if the apparatus is not of such a geometry (conical bottom with a guard ring at the top) as to correct for end effects, the following additional condition shall be satisfied :

$$\frac{h_i}{d_i} \geqslant 1{,}5$$

where h_i is the height of the internal cylinder.

For these viscometers, the shear rate to be recorded is the one that exists near the surface of the torque measuring cylinder, regardless of which is the moving member. The shear rate shall be calculated (as if the material were Newtonian), in seconds to the power minus one, from the relationship :

$$\dot{\gamma} = \frac{4 \pi n}{60} \times \frac{d^2}{{d_e}^2 - {d_i}^2}$$

where

n is the rotational frequency, in minutes to the power minus one;

$d = d_e$ if the internal cylinder is the torque measuring cylinder;

$d = d_i$ if the external cylinder is the torque measuring cylinder;

NOTE — Correction formulae should normally be given by the viscometer manufacturer.

5.1.2 For cone-and-plate and double-cone viscometers, the angle α between the generator of the cone and the plate or between the generators of the two cones shall be as small as possible, preferably not greater than 1° and in no case greater than 4°. Where the angle is greater than 1°, this shall be stated in the report and if possible a correction shall be made, this also being indicated in the report (see note in 5.1.1).

For these viscometers, the shear rate near the surface of the torque measuring member shall be calculated, in seconds to the power minus one, from the relationship :

$$\dot{\gamma} = \frac{2 \pi n}{60} \times \frac{1}{\tan\alpha}$$

$$\approx \frac{2 \pi n}{60} \times \frac{1}{\alpha}$$

where

n is the rotational frequency, in minutes to the power minus one;

α is the angle, in radians.

5.1.3 In the case of instruments combining two of the above principles, the individual requirements applicable to each shall be met unless the effect of one is minimal, when it may be dealt with by a correction.

5.1.4 The apparatus shall be such that the shear rate may be determined for each measurement.

The apparatus shall have an accuracy of viscosity measurement of better than 2 % of full scale reading for all combinations of rotor, stator and rotational frequency.

NOTE — By using different stators, rotors and rotational frequencies most commercial instruments cover a range of viscosities of at least 10^{-2} to 10^3 Pa·s (10 to 10^6 cP).

The range of shear rates varies greatly with different equipment. The choice of the particular viscometer to be used has to be made considering the viscosity range and the shear rate.

Adjustment and calibration of these instruments are usually carried out by the manufacturer.

Periodic verification of this adjustment and calibration is recommended and may be performed with liquids of known viscosity, either by the user or by an official standards laboratory.

5.2 Thermostat, permitting the product to be brought to, and be maintained at, the required test temperature (usually 23 °C, according to ISO 291), within ± 0,2 °C.

This tolerance of ± 0,2 °C is valid for temperatures between 0 °C and 50 °C. Outside these limits a larger tolerance (± 0,4 °C for example) is often sufficient. On the other hand for more precise measurements in the usual temperature range it may be necessary to use a tighter tolerance (± 0,1 °C for example).

Note that at high shear rates, the measurement itself causes heating of the material, which must be taken care of by the thermostat or, when suitable, a correction (often indicated by the manufacturer of the viscometer) may be made.

NOTE — Commercial viscometers usually incorporate a thermostat.

6 PROCEDURE

6.1 Selection of shear rate (recommended values)

The shear rate(s) shall be specified in the particular document for the product (see clause 1). However, if no such document exists, the following considerations shall guide the choice.

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

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

and these values multiplied or divided by 100.

NOTE — These values are preferred numbers of the R 5/2 series given in ISO 3.

6.1.2 When the product is Newtonian, the shear rate does not affect the result and in principle any shear rate could be chosen. However, as it is generally difficult to be certain that a product is Newtonian, the shear rate at which the measurement is made shall always be measured and noted.

6.1.3 If the viscosities of non-Newtonian products are to be compared, shear rates that are identical or at least very similar shall be used.

6.1.4 It is possible in the case of all products, and recommended in the case of non-Newtonian products, that measurements be made at widely differing shear rates so that the graph of viscosity/shear rate may be drawn.

6.2 Determination

Proceed according to the information supplied by the manufacturer of the viscometer, bearing in mind the following principal factors :

6.2.1 *Selection of rotor, stator and rotational frequency*

The shear rate(s), selected as in 6.1, is (are) obtained by a suitable choice of rotor, stator and rotational frequency.

If the instrument does not allow the direct measurement of viscosity at one of the selected shear rates, it is advisable to use the two closest values bracketing the selected value and to interpolate the result.

6.2.2 *Preparation and conditioning of the sample*

The sample shall be completely free of air bubbles and impurities before its introduction into the instrument.

For certain products, it may be necessary to condition the sample at specified temperatures for specified times.

6.2.3 *Products that are hygroscopic or contain volatile components*

If the product is hygroscopic or is composed of or contains volatile substances, a closed sample container shall be used.

6.2.4 *Temperature*

The test shall only be commenced when the temperature of the material under test has reached and is maintained at the required temperature within the prescribed tolerance (see 5.2).

6.2.5 *Test conditions*

The measurement can be made at one or more shear rates and at one or more temperatures. Further, it is possible to derive curves of viscosity as a function of shear rate or of temperature between limits which should be specified.

These conditions should be specified in the particular document (see clause 1) referring to the product or group of products.

6.2.6 *Precision of results*

In order to obtain the maximum precision, it is recommended that the instrumental conditions be chosen to give a deflection as near to the maximum as possible.

6.2.7 *Number of tests*

For a determination at a single shear rate and at a single temperature, at least three measurements shall be made on a single test portion. None of the results shall differ from their mean by more than 2 % of full scale. It is advisable to make measurements on several test portions.

For determinations at several shear rates or several temperatures, increase the number of tests accordingly.

6.2.8 *Drift during measurements*

If a drift occurs during the measurements, and this is not caused by an internal chemical reaction, heating caused by excessive speed of the rotor, evaporation of solvent, etc., it is possible that the product is thixotropic or rheopectic. The particular documents (see clause 1) concerning such products should state the time after which the reading shall be taken or stipulate the construction of the curve of viscosity as a function of time.

In this case the three measurements specified in 6.2.7 shall be made on three separate test portions and not on a single one.

7 EXPRESSION OF RESULTS

Calculate the average of the three measured viscosities, to three significant figures, for each test portion and each shear rate and/or temperature.

Express the results (corrected if necessary) in pascal seconds (Pa·s) (or centipoises, cP), to three significant figures, indicating in parentheses the temperature and the shear rate, as in the following example :

$$\eta\ (23\ ^\circ\text{C},\ 1\ 600\ \text{s}^{-1}) = 4{,}25\ \text{Pa·s (or 4 250 cP)}$$

Follow, if necessary, the instructions given in the particular documents (see clause 1) concerning the drawing of a curve for thixotropic or rheopectic materials (see 6.2.8).

8 TEST REPORT

The test report shall include a reference to this International Standard and the following particulars :

a) designation of the product under test;

b) temperature of the test;

c) trade name and model of the viscometer;

d) rotors, stators and rotational frequencies, including, if necessary, the value of d_e/d_i or the angle α of the apparatus used;

e) results, expressed in accordance with clause 7;

f) magnitude of any corrections which were necessary and the basis of their calculation;

g) date of the test.