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Plastics — Determination of the transient extensional viscosity of polymer melts

Plastiques — Détermination de la viscosité élongationelle transitoire des polymères à l'état fondu

Reference number
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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.

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The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 20965 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

Plastics — Determination of the transient extensional viscosity of polymer melts

1 Scope

This International Standard specifies the general principles of a method for determining the transient extensional viscosity of polymer melts. The procedure details the measurement of polymer melt specimens stretched uniaxially under conditions of constant strain rate and constant temperature.

The method is capable of measuring the transient extensional viscosity of polymer melts at Hencky strain rates typically in the range 0,01 s^{-1} to 1 s^{-1}, at Hencky strains up to approximately 4 and at temperatures up to approximately 250 °C (see Notes 1 and 2). It is suitable for measuring transient extensional viscosity values typically in the range from approximately 10^4 Pa·s to 10^7 Pa·s (see Note 3).

NOTE 1 Hencky strains and strain rates are used (see Clause 3).

NOTE 2 Values of strain, strain rate and temperature outside these limiting values may be attained.

NOTE 3 The operating limit of an instrument, in terms of the lowest transient extensional viscosity values that can be measured, is due to a combination of factors, including the ability of the specimen to maintain its shape during testing and the resolution of the instrument.

A list of documents related to this International Standard is given in the Bibliography.

2 Normative references

The following referenced documents are indispensable for the application 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 472, *Plastics — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472, together with the following, apply.

Definitions 3.1 to 3.5 are given by Whorlow [1] for strains and strain rates, and by the Nomenclature Committee of the Society of Rheology for start-up flow in tensile uniaxial extension at constant Hencky strain rate [2].

3.1
Hencky strain

ε

strain given by the natural logarithm of the elongation ratio

$$\varepsilon = \ln(l/l_0) \qquad (1)$$

where l is the specimen length and l_0 is the original specimen length

NOTE 1 It is also referred to as the natural or true strain.

NOTE 2 It is dimensionless.

3.2
Hencky strain rate

$\dot{\varepsilon}$

rate of change of Hencky strain with time, given by

$$\dot{\varepsilon} = 1/l \times \partial l / \partial t \tag{2}$$

where t is time

NOTE 1 It is independent of the original specimen length l_0.

NOTE 2 It is expressed in reciprocal seconds.

3.3
net tensile stress

σ_E

for tensile uniaxial extension, stress given by

$$\sigma_\mathrm{E} = \sigma_{11} - \sigma_{22} = \sigma_{11} - \sigma_{33} = \sigma_{\mathrm{zz}} - \sigma_{\mathrm{rr}} \tag{3}$$

where σ_{ii} is a stress tensor in either rectangular or axisymmetric co-ordinates

NOTE 1 The tensile stress growth function is indicated by $\sigma_\mathrm{E}{}^+$ where the $^+$ indicates start-up of flow.

NOTE 2 Net tensile stress is expressed in pascals.

3.4
tensile stress growth coefficient

$\eta_\mathrm{E}{}^+$

ratio of the net tensile stress to the Hencky strain rate

$$\eta_\mathrm{E}{}^+(t,\dot{\varepsilon}) = \sigma_\mathrm{E} / \dot{\varepsilon} \tag{4}$$

for tensile uniaxial extension, where t is time and $^+$ indicates start-up of flow

NOTE 1 It is also known for the purposes of this International Standard as “transient extensional viscosity”.

NOTE 2 It is a transient term.

NOTE 3 It is expressed in pascal seconds.

3.5
tensile viscosity

η_E

term given by

$$\eta_\mathrm{E}(t,\dot{\varepsilon}) = \lim_{t\to\infty}[\eta_\mathrm{E}^{+}(t,\dot{\varepsilon})] \tag{5}$$

NOTE 1 It is the limiting tensile stress growth coefficient value and represents an equilibrium extensional viscosity if a steady value is achieved. However, for materials that do not exhibit a steady-state behaviour, the use of an “equilibrium extensional viscosity” such as this is not appropriate.

NOTE 2 It is expressed in pascal seconds.

4 General principles

In contrast to shear flow where reference is normally made only to steady shear flow behaviour, extensional flow behaviour is best described as being transient. In describing the transient behaviour of materials in extension at constant strain rate, they may exhibit either an unbounded stress growth behaviour in which the stress continually increases with increasing strain until the material fails, or the stress reaches a steady value with increasing strain thus yielding a tensile or equilibrium extensional viscosity. The latter occurs typically at large strains. An equilibrium extensional viscosity is thus dependent on strain rate but not on strain or time. Normally, the extensional viscosity will vary as a function of both strain and strain rate as well as temperature.

In describing and modelling plastics processing, the use of Hencky strain is preferred. The rate of Hencky strain of an element of fluid within a flow is independent of its original length and is determined only from the velocity field of that element. It is thus a more suitable characteristic of the flow. Strain and strain rate are taken by default herein to be Hencky values.

Stretching flow methods can be used to generate quantitatively accurate data on the extensional viscoelasticity of polymer melts. In carrying out extensional flow measurements, there are four types of measurement that are normally made: constant strain rate, constant stress, constant force and constant speed. This International Standard describes the first of these: constant strain rate. In this method, the strain rate is uniform throughout the specimen and is held constant with time.

The basic principle behind stretching flow measurements is to subject a specimen to a tensile stretching deformation. By measurement of the force and deformation of the specimen, the stresses and strains and hence strain rate can be determined.

5 Apparatus

5.1 General description

The measuring apparatus shall consist of one of the following types, shown in Figures 1 to 4. These types define the various instrument configurations. The notation used in these figures is defined in 8.1.

Type A: Two rotating clamps. Each clamp shall consist of either a single rotating element or a pair of rotating elements — only the pair arrangement is shown. The force exerted on the specimen can be measured at the fixed or rotating end.

NOTE It is likely that the force will be easier to measure, and will be measured with greater accuracy, on a fixed clamp rather than on a moving clamp as there will be fewer complications due, for example, to vibration and the inertia of the clamp which may introduce noise and errors into the force signal.

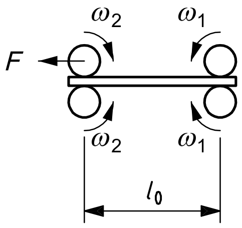

Figure 1 — Schematic diagram of type A test instrument

Type B: A single rotating clamp and a fixed clamp. The rotating clamp shall consist of either a single rotating element or a pair of rotating elements. The force exerted on the specimen is normally measured at the fixed end.

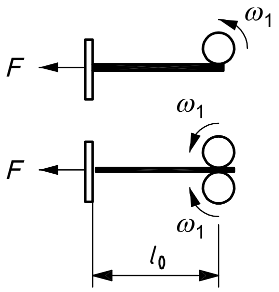

Figure 2 — Schematic diagram of type B test instrument

Type C: Two translating (non-rotating) clamps.

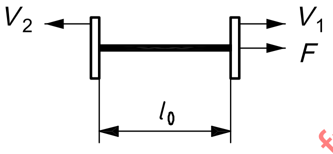

Figure 3 — Schematic diagram of type C test instrument

Type D: Single translating (non-rotating) clamp.

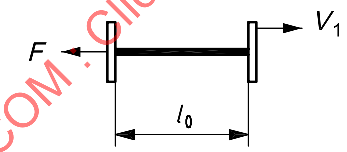

Figure 4 — Schematic diagram of type D test instrument

In each of these configurations, the specimen is mounted between the clamps and is stretched uniaxially. The requirements for the apparatus are that it shall permit the measurement or determination of the force acting on the specimen, and the strain and strain rate of the specimen subjected to a constant strain rate under isothermal conditions. The strain and strain rate of the specimen shall either be derived from the displacements and/or speeds of the clamp or clamps, or be measured directly from the dimensions and/or local velocities of the specimen.

5.2 Silicon bath/temperature-controlled chamber

Heating may be provided by placing the specimen in a silicone oil bath or in a temperature-controlled chamber with a forced gas flow through it.

NOTE 1 When heating using forced gas, a gas may be used in the chamber surrounding the test specimen to provide the required test environment, for example nitrogen to provide an inert atmosphere.

NOTE 2 The use of a silicone oil bath may permit more rapid heating of the specimen.

For low-viscosity materials, it is essential to support the specimen during heat-up and testing (to avoid it sagging under the influence of gravity).

NOTE 3 The use of a silicone oil bath results in the specimen being supported by the silicone oil due to its buoyancy, particularly if the densities of the silicone oil and specimen are matched at the test temperature. If a forced-gas oven is used, then support of the specimen can be obtained by the cushioning effect provided by the gas.

Silicone oil may be absorbed by some polymers. A check should preferably be made to see if the immersion time affects the measured properties of the polymer by varying the length of the immersion time (see Annex A and also Note 1 in 7.1). When quantitative results are required, then this check shall be made.

NOTE 4 Even if the silicone oil does not affect the shape of the tensile stress growth coefficient versus strain (or time) plot, it may affect the point of failure of the specimen. Thus assessment of the effect of the silicone oil on the measured properties should consider both of these aspects.

NOTE 5 Alternative methods for checking the effect of immersion in silicone oil on the specimen may also be used. Such methods include the measurement of the mass or dimensions of the specimen before and after immersing in silicone oil and identifying whether a change has occurred due to that immersion — see Annex A.

5.3 Temperature measurement and control

The test temperature should preferably be measured using a device that is mounted close to the specimen. Contact of the device with the specimen is not permitted. It is essential to mount temperature sensors in at least two positions to monitor temperature uniformity along the length of the specimen.

NOTE The uniformity of the temperature along the specimen length is critical to the measurement of the transient extensional flow properties of polymer melts. Localized hot spots will result in excessive strain in those regions. This may lead to premature failure, particularly for materials that do not exhibit a high degree of strain hardening.

The spatial temperature variation shall be within ± 0,75 °C.

The temporal temperature variation shall be within ± 1,0 °C of the set temperature.

The temperature-measuring device shall have a resolution of 0,1 °C and shall be calibrated using a device accurate to within ± 0,1 °C.

5.4 Strain and strain rate measurement

The strain and strain rate of the specimen shall be determined either from measurement of the displacements and/or speeds of the clamp or clamps, or measured directly from the dimensions and/or local velocities of the specimen.

NOTE The diameter of the specimen may be measured during the test by use of optical or cutting methods to derive strains and strain rates and to assess the uniformity of deformation. The cutting method results in the test being terminated once the cuts have been made and thus prevents data to failure from being obtained. Local velocities may be measured using optical methods.

Corrections for slippage of the specimen at the clamp or clamps may be applied, obtained through independent measurement of the strain of the specimen during testing through measurement of its diameter or local velocities by other methods.

The apparatus shall have an accuracy of strain determination or measurement to within ± 3 % of the absolute value.

The apparatus shall have an accuracy of strain rate determination or measurement to within ± 3 % of the absolute value.

5.5 Force measurement

The force on the specimen shall be measured during the test by an appropriate means, for example a leaf spring arrangement (see Note 1).

The resolution of the force-measuring device should preferably be no greater than 0,1 % of the full-scale value.

The apparatus shall have an accuracy of force measurement to within ± 2 % of the full-scale value (see Note 2).

NOTE 1 Typical peak forces measured in testing of polyethylenes are estimated to be up to ≈ 1 N for specimens approximately 3 mm in diameter.

NOTE 2 It is desirable, in particular for accurate measurements at low forces, that the accuracy of the force measurement device be within ± 2 % of absolute, but this may be difficult to achieve in the lower part of the force transducer's range.

5.6 Calibration

The force, displacement, rate of displacement and temperature functions of the rheometer shall be calibrated periodically.

It is preferable that calibration be carried out at the test temperature as measurement of these functions, in particular that of force, may be temperature sensitive.

NOTE No traceable standard reference materials are known to exist for checking the calibration of such instruments. Where a reference material is used for checking the instrument, it is preferable that the transient extensional viscosity of the reference material, and the dimensions of the specimen produced using it, have values that are similar to those encountered or used during normal operation of the instrument.

6 Sampling and specimen preparation

6.1 Sampling

The sampling method, including any special methods of specimen preparation and introduction into the rheometer, shall be as specified in the relevant materials standard or otherwise by agreement.

If samples or specimens are hygroscopic or contain volatile ingredients, then they shall be stored to prevent or minimize any effects on the measurements. Drying of samples may be required prior to preparing test specimens.

As the test specimens are typically small, being of the order of a few grams, it is essential that they be representative of the material being sampled. Repeat testing may be used to identify batch-to-batch or within-batch variation.

6.2 Specimen preparation

The specimen shall be either cylindrical or rectangular in cross-section.

Test specimens in the form of a cylinder may be produced by extrusion or by injection, transfer or compression moulding.

Test specimens in the form of a strip may be produced by extrusion or by injection or compression moulding or by cutting from sheet.

The length-to-diameter ratio of cylindrical specimens should preferably be at least 10.

NOTE A length-to-diameter ratio of at least 10 is required to minimize end-effects. However, a longer specimen will result in a reduction in the maximum strain rate that can be achieved. The magnitude of the end-errors can be assessed by using specimens of different length or diameter to produce different length-to-diameter aspect ratios. The effect on measured values can then be determined.

The specimen shall not contain any visible impurities, voids or air bubbles. The specimen shall not show any obvious discoloration prior to or after testing.

For cylindrical specimens, measure the diameter D of the specimen at at least three positions along its length. Repeat these measurements after rotating the specimen by 90°. Calculate an average value for the diameter from these measurements.

For rectangular specimens, measure the width and thickness at at least three positions along its length. Calculate average values for the width b and thickness h from these measurements.

Calculate the cross-sectional area of the specimen from the measurements.

The diameter, or width and thickness, of the specimen, as appropriate, shall be determined to and be uniform to within ± 2 % of their average value.

6.3 Specimen mounting

Specimens may be either gripped by the clamps or attached using adhesive to studs that are then clamped into the instrument.

Attachment by a suitable high-temperature epoxy adhesive has been found suitable. Treat the ends of the specimen by passing them through a butane flame and then dipping them into concentrated sulfuric acid for 30 s. Prevent any other part of the specimen, except that to be bonded, from being exposed to either the flame or the acid. Dip the ends of the specimen into the adhesive and then attach them to the studs. Place the specimen with its studs into an oven and cure the adhesive using a suitable time-temperature cycle (100 °C for 1 h has been found suitable). Allow the specimen to cool before handling.

7 Procedure

7.1 Specimen loading

Mount a specimen in place in the rheometer.

Measure the length of the specimen between the clamps to within 1 % of its absolute value.

After mounting the specimen in the instrument, immerse it in the silicone oil bath or place it in the temperature-controlled chamber (see 5.2). Where possible, bring the bath or chamber to the test temperature before inserting the specimen to reduce the time spent by the specimen reaching and equilibrating at the test temperature. Allow the specimen and apparatus to reach thermal equilibrium at the test temperature. This period of time is referred to as the equilibration time.

NOTE 1 The adequacy of the time allowed for the specimen to reach thermal equilibrium and the effects of the silicone oil, degradation, crosslinking and other time-dependent phenomena on the specimen can be checked by varying the time for which the specimen is in the oil bath or environmental chamber before testing. The effect on the measured values can then be assessed. For measurements in silicone oil of specimens approximately 3 mm in diameter, an equilibration time of approximately 5 min has been found to be sufficient for testing at a temperature of 150 °C.

A correction for thermal expansion of the specimen during heating may be necessary. A correction for effects arising from stress relaxation of the specimen, if unclamped during the temperature equilibration period, may also be required. Both of these effects may result in a change in the critical dimensions of the specimen (i.e. thickness and width, or diameter) that may need to be taken into account.

NOTE 2 As an example, measurements of a PE-HD indicate a 20 % decrease in density on heating from 25 °C to 150 °C which, if accommodated solely by a change in the diameter of a cylindrical specimen, would result in an ≈ 10 % increase in diameter of the specimen. Furthermore, stress relaxation of extruded specimens may result in a recovery (shrinkage) in length with a corresponding increase in the specimen's critical dimensions (e.g. diameter) if unclamped during the temperature equilibration period.

7.2 Pre-conditioning of the specimen

The specimen may be pre-conditioned by applying a known strain prior to testing and allowing the induced stresses to relax to zero before commencing the test.

7.3 Testing

Subject the specimen to a constant strain rate deformation either until the specimen fails or up to a set strain value.

Record the force, clamp speeds, and strain and strain rate data as functions of time, as appropriate.

Analyse the data using the appropriate equations presented in Clause 8. Corrections for effects such as machine compliance, end-effects, errors in strain and strain rate determination and thermal expansion or stress relaxation of the specimen may be applied, as necessary.

It may be necessary to check for degradation or crosslinking, particularly when testing at low strain rates when the test duration is long (see Note). Also check for premature failure of the specimen or breakage or pull-out at the clamps.

NOTE The effects of degradation, crosslinking and silicone oil on the specimen can be checked by varying the time for which the specimen is immersed in the oil bath or temperature-controlled chamber before and during testing. The effect of the immersion on measured values can then be assessed.

8 Analysis of extensional flow measurements

8.1 Symbols used

v	instantaneous speed of separation of the ends of the specimen, m·s^{-1}
V	speed of the ends of the specimen (types C and D only), m·s^{-1}
ω	angular speed of rotating clamps, rad·s^{-1}
r	rotating clamp radius, m
t	time, s
l_0	original specimen length, m
l	specimen length at time t, m
A_0	original specimen cross-sectional area, m^2
A	specimen cross-sectional area at time t, m^2
F	force, N
ε	Hencky strain (dimensionless)
$\dot{\varepsilon}$	Hencky strain rate, s^{-1}
σ_E	net tensile stress, Pa
η_E^+	tensile stress growth coefficient, Pa·s

8.2 Analysis of extensional flow

8.2.1 General

Rearranging the Hencky strain equation, Equation (1), yields

$$l = l_0 \mathrm{e}^{\varepsilon} \tag{6}$$

where l_0 is the original specimen length, l is the specimen length at time t and ε is the Hencky strain.

At constant Hencky strain rate $\dot{\varepsilon}$

$$\varepsilon = \dot{\varepsilon} t \tag{7}$$

where t is time.

Thus

$$l = l_0 \mathrm{e}^{\dot{\varepsilon} t} \tag{8}$$

Assuming conservation of volume of the specimen, then

$$l_0 A_0 = lA \tag{9}$$

where A_0 is the original specimen cross-sectional area and A is the specimen cross-sectional area at time t.

The net tensile stress σ_E, expressed as the ratio of the force F applied to the specimen to the cross-sectional area of the specimen A, is given by

$$\sigma_\mathrm{E} = \frac{F}{A} \tag{10}$$

which can be rewritten using Equations (8) and (9) as

$$\sigma_\mathrm{E} = \frac{F \mathrm{e}^{\dot{\varepsilon} t}}{A_0} \tag{11}$$

Thus the tensile stress growth coefficient, the ratio of net tensile stress to Hencky strain rate, is given by

$$\eta_\mathrm{E}^{+} = \frac{F \mathrm{e}^{\dot{\varepsilon} t}}{A_0 \dot{\varepsilon}} \tag{12}$$

8.2.2 Analysis for type A and B instruments (rotating clamps)

For type A instruments, the effective length of the specimen is equal to the distance between the points at which the specimen touches the rotating clamps at a tangent. For type B instruments, it is equal to the distance between the fixed clamp and the point at which the specimen touches the rotating clamp at a tangent. It is indicated by l_o in Figures 1 and 2. The effective specimen length l_0 remains unchanged during the test as the specimen is wound onto the rotating clamp or clamps. The instantaneous speed of separation of the effective ends of the specimen, v, is given by

$$v = (\varpi_1 + \varpi_2) r \tag{13}$$

where ω_1 and ω_2 are the angular speeds of the rotating clamps and r is the radius of the rotating clamps, assuming no slippage of the specimen with the rotating clamps.

In the case of type B instruments, $\omega_2 = 0$.

NOTE For type A instruments, the angular speeds ω_1 and ω_2 need not be the same.

The speed of separation can also be written as

$$v = \partial l / \partial t \tag{14}$$

Thus, by using the definition of Hencky strain rate given in Equation (2), the strain rate in the specimen of effective length l_0 is given by

$$\dot{\varepsilon} = \frac{(\omega_1 + \omega_2) r}{l_0} \tag{15}$$

Thus, for an instrument with a clamp that is rotating at constant angular speed (or clamps rotating at constant angular speeds), the strain rate is constant.

NOTE The specimen length may change with force due to the compliance of the force transducer, thereby affecting the uniformity of the strain rate.

The strain ε is determined as the integral of the strain rate with respect to time and, as the strain rate is constant, is given, using Equation (7), by

$$\varepsilon = \frac{(\omega_1 + \omega_2) r t}{l_0} \tag{16}$$

The net tensile stress is given by Equation (11) and the tensile stress growth coefficient by Equation (12), substituting for strain rate as appropriate.

8.2.3 Analysis for type C and D instruments (translating clamps)

In the case of translating clamps (type C and D instruments), the speed of separation of the ends of the specimen, v (i.e. of the clamps) is given by

$$v = V_1 + V_2 \tag{17}$$

where V_1 and V_2 are the speeds of the individual clamps.

Using Equations (8) and (14),

$$V_1 + V_2 = l_0 \dot{\varepsilon} \mathrm{e}^{\dot{\varepsilon} t} \tag{18}$$

where l_0 is the original specimen length.

Thus, for a specified constant strain rate, the sum of the velocity profiles of the clamps as a function of time is defined. For a specified constant strain rate, the strain is given by Equation (7), the net tensile stress by Equation (11) and the tensile stress growth coefficient by Equation (12).

In the case of a single translating clamp (type D instrument), then $V_2 = 0$.

9 Precision

The results of an intercomparison of stretching methods made in 1998 [3] comprising six laboratories indicated that the variation in tensile stress growth coefficient values was up to approximately ± 60 %. Measurements were performed mainly over a strain rate range of 0,01 s^{-1} to 1 s^{-1}, and measured tensile stress growth coefficient values were predominantly in the range 5×10^4 Pa·s to 3×10^6 Pa·s. The variation in peak tensile stress growth coefficient values was up to approximately ± 100 %. A high-density polyethylene that was stable at 190 °C for in excess of 2 h was used as the intercomparison material. Samples of the material were provided to each of the participants, who produced their own specimens. Measurements were made at 150 °C and 190 °C.

NOTE The quoted variation in tensile stress growth coefficient values excludes data at short times/low strains for which the variation is greater due to the resolution of the measuring equipment being insufficient for reliable measurement of the low forces generated. Measurement errors will be strongly dependent on the magnitude of the force to be measured which, in turn, will be strongly dependent on the strain. The values quoted are indicative of the various instruments that were involved in the intercomparison.

An analysis of the uncertainties of transient extensional viscosity measurements is presented in Annex B.

10 Test report

The test report shall include the following information, as applicable:

a) a reference to this International Standard;

b) the reference number of the test;

c) the date of the test;

d) operator identification;

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

f) the test specimen dimensions;

g) details of any pretreatment of the material and of the method of preparation and loading of the specimen;

h) details of the instrument used;

i) the set value for the test temperature, in degrees Celsius;

j) details of the equilibration time and any pre-conditioning of the specimen;

k) the duration of thermal stability, in seconds;

l) the test duration, in seconds;

m) the clamp speeds;

n) any of the following, presented as functions of strain, strain rate, temperature and time, as required:

the net tensile stress, in pascals,

the tensile stress growth coefficient, in pascal seconds;

o) details of any corrections applied to the data;

p) any visual observations of premature failure or degradation of the specimen;

q) any test conditions agreed on that deviate from this International Standard.

Annex A
(informative)

Checking for swelling of specimens due to immersion in silicone oil or other fluids

Assessment of the effect of the immersion time on the measured properties of a polymer can be carried out by varying the length of the immersion time. A change in the measured properties with immersion time may be an indication that the time allowed for the specimen to reach thermal equilibrium was insufficient, that the specimen absorbed the immersion fluid (e.g. silicone oil) which affected its properties, or the specimen degraded, crosslinked or underwent other time-dependent changes.

Methods for assessing the effect of the immersion fluid on the specimen also include the measurement of its mass and dimensions. However, these methods do not identify the effect of the immersion on the extensional properties of the specimen. A measured change in the dimensions or mass may be an indication that the properties of the specimen may be affected.

The mass, or dimensions, of a specimen at room temperature are measured, as appropriate, depending on the method to be used. The specimen is then subjected to immersion in silicone oil (or another fluid) at the test temperature for a period equivalent to the full duration of the test, including the time taken for the specimen to equilibrate at the test temperature. The specimen is not subjected to extensional testing, nor is there any need to clamp it in place. The specimen, after immersion for the set period, is then removed from the silicone oil and dried. Solvents should not be used to remove any traces of silicone oil from the surface of the specimen as these themselves might affect the specimen. The specimen mass, or dimensions, is then remeasured.

However, the mass of the specimen may increase solely due to the residual silicone oil on its surface, rather than to oil being absorbed. To check and correct for this, if necessary, a similar specimen is weighed, placed in cold silicone oil and removed immediately after wetting. It is then dried, using the same method as for the specimen immersed for the full test duration, and reweighed. Any difference in mass is due to traces of silicone oil on the specimen that cannot be removed by the drying procedure used. This difference should be used to correct any increase in mass of the specimen immersed in oil at the test temperature.

The mass, or dimensions, prior to immersion and after immersion are compared and can be used to assess whether immersion affected the specimen. An increase in specimen mass due to immersion in oil at the test temperature is an indication of absorption of oil by the specimen that may affect its measured performance.

Changes in the dimensions of the specimen resulting in an increase in volume is also an indication that the specimen has absorbed oil. However, changes are likely to occur in the dimensions of the specimen, for example due to relaxation of any stresses frozen into the specimen during production of the material. Thus only a change in volume of the specimen should be taken as an indication of absorption of oil. The uncertainties in the dimensional measurements, and consequently in the determination of the specimen volume, should be determined to assess whether any measured changes in volume are significant.

Annex B
(informative)

Uncertainties in transient extensional viscosity testing

B.1 Introduction

Due to the considerable stretching that a specimen normally undergoes during testing, particularly on reaching high strain values, there is a very significant decrease in the cross-sectional area of the specimen, accompanied by a similarly large decrease in the measured force. As a consequence, there is a significant increase in the uncertainty of the measurements. To understand and quantify the level of confidence that one can put in the accuracy of the results obtained, it is important that the uncertainty in the measurements, in particular that due to the accuracy and resolution of the force measurement, are known.

An example of an analysis of the uncertainties in transient extensional viscosity measurements is presented for an extensional rheometer having a single rotating clamp and a fixed clamp, i.e. a type B instrument. The equations can be used to calculate the uncertainties in the derived tensile stress growth coefficient values, given the uncertainties in each of the components of the measurement. Similar analyses can be carried out for other instrument configurations by following the principles presented. For further details, see Reference [5].

B.2 Uncertainty analysis — General

The combined uncertainty $u_\mathrm{c}(y)$ of the measurand y (the quantity to be measured) can be determined from the partial derivatives of the function and the uncertainties in the parameters. Assuming that individual uncertainty sources are uncorrelated, the combined uncertainty $u_\mathrm{c}(y)$ can be computed using the root sum squares:

$$u_\mathrm{c}(y) = \sqrt{\sum_{i=1}^{m} [c_i u(x_i)]^2} \qquad \text{(B.1)}$$

where c_i is the sensitivity coefficient (partial derivative) associated with an input quantity x_i, and $u(x_i)$ is the uncertainty in that quantity.

The combined uncertainty $u_\mathrm{c}(y)$ corresponds to one standard deviation and therefore has an associated confidence level of approximately 68 %. Assuming a normal distribution, then it can be assumed that an expanded uncertainty U for a 95 % confidence level can be determined using a coverage factor of 2 (i.e. equivalent to 2 standard deviations). The relative uncertainty is the ratio of the uncertainty in the parameter to the value of the parameter.

B.3 Analysis of the uncertainties in the measurement of transient extensional viscosity

Given the expression for transient extensional viscosity η_E [Equations (12) and (16) with $\omega_2 = 0$; the subscript “1” of ω_1 has been dropped for clarity]:

$$\eta_\mathrm{E} = \frac{F l_0 \mathrm{e}^{\left(\frac{\omega r t}{l_0}\right)}}{A_0 \omega r} \qquad \text{(B.2)}$$

then the sensitivity coefficients c_i are given by

$$\frac{\partial \eta_\mathrm{E}}{\mathrm{d}F} = \frac{l_0 \mathrm{e}^{\left(\frac{\omega r t}{l_0}\right)}}{A_0 \omega r} \qquad \text{(B.3)}$$

$$\frac{\partial \eta_\mathrm{E}}{\partial A_0} = \frac{-F l_0 \mathrm{e}^{\left(\frac{\omega r t}{l_0}\right)}}{{A_0}^2 \omega r} \qquad \text{(B.4)}$$

$$\frac{\partial \eta_\mathrm{E}}{\partial r} = \frac{F l_0}{A_0 \omega} \left(\frac{-\mathrm{e}^{\left(\frac{\omega r t}{l_0}\right)}}{r^2} + \frac{\omega t \mathrm{e}^{\left(\frac{\omega r t}{l_0}\right)}}{l_0 r} \right) \qquad \text{(B.5)}$$

$$\frac{\partial \eta_\mathrm{E}}{\partial \omega} = \frac{F l_0}{A_0 r} \left(\frac{-\mathrm{e}^{\left(\frac{\omega r t}{l_0}\right)}}{\omega^2} + \frac{r t \mathrm{e}^{\left(\frac{\omega r t}{l_0}\right)}}{l_0 \omega} \right) \qquad \text{(B.6)}$$

$$\frac{\partial \eta_\mathrm{E}}{\partial l_0} = \frac{F}{A_0 \omega r} \left(\mathrm{e}^{\left(\frac{\omega r t}{l_0}\right)} - \frac{\omega r t \mathrm{e}^{\left(\frac{\omega r t}{l_0}\right)}}{l_0} \right) \qquad \text{(B.7)}$$

$$\frac{\partial \eta_\mathrm{E}}{\partial t} = \frac{F \mathrm{e}^{\left(\frac{\omega r t}{l_0}\right)}}{A_0} \qquad \text{(B.8)}$$

The combined relative uncertainty in the tensile stress growth coefficient values is evaluated as

$$\left(\frac{\mathrm{u}_{\eta_\mathrm{E}}}{\eta_\mathrm{E}}\right)^2 = \left(\frac{\partial \eta_\mathrm{E}}{\partial F}\,\frac{u_F}{\eta_\mathrm{E}}\right)^2 + \left(\frac{\partial \eta_\mathrm{E}}{\partial A_0}\,\frac{u_{A_0}}{\eta_\mathrm{E}}\right)^2 + \left(\frac{\partial \eta_\mathrm{E}}{\partial r}\,\frac{u_r}{\eta_\mathrm{E}}\right)^2 + \left(\frac{\partial \eta_\mathrm{E}}{\partial \omega}\,\frac{u_\omega}{\eta_\mathrm{E}}\right)^2 + \left(\frac{\partial \eta_\mathrm{E}}{\partial l_0}\,\frac{u_{l_0}}{\eta_\mathrm{E}}\right)^2 + \left(\frac{\partial \eta_\mathrm{E}}{\partial t}\,\frac{u_t}{\eta_\mathrm{E}}\right)^2 \qquad \text{(B.9)}$$

where the partial derivatives are given by Equations (B.3) to (B.8).

In a similar fashion, the combined relative uncertainty in strain rate is given by

$$\left(\frac{u_{\dot{\varepsilon}}}{\dot{\varepsilon}}\right)^2 = \left(\frac{u_{\omega}}{\omega}\right)^2 + \left(\frac{u_r}{r}\right)^2 + \left(\frac{u_{l_0}}{l_0}\right)^2 \tag{B.10}$$

and that for strain by

$$\left(\frac{u_{\varepsilon}}{\varepsilon}\right)^2 = \left(\frac{u_{\omega}}{\omega}\right)^2 + \left(\frac{u_r}{r}\right)^2 + \left(\frac{u_t}{t}\right)^2 + \left(\frac{u_{l_0}}{l_0}\right)^2 \tag{B.11}$$

The values of the terms in the above uncertainty equations, except for the force terms, are relatively easy to determine. The difficulty with the force term is that the force decreases considerably at high strain values and thus the relative uncertainty in the force will increase significantly as the test progresses. To determine the effect of its contribution to the combined uncertainty in the determination of the transient extensional viscosity values, further analysis, presented below, is required.

The force F supported by the specimen during testing is given by

$$F = \sigma_{\mathrm{E}} A \tag{B.12}$$

where A is the cross-sectional area of the specimen and σ_{E} is the net tensile stress. Using the definition of the tensile stress growth coefficient $\eta_{\mathrm{E}}{}^{+}$ (see 3.4), then Equation (B.12) becomes

$$F = \eta_{\mathrm{E}}{}^{+} \dot{\varepsilon} A \tag{B.13}$$

or, using Equations (6), (9) and (16),

$$F = \eta_{\mathrm{E}}{}^{+} \dot{\varepsilon} A_0 \mathrm{e}^{\left(\frac{-\omega r t}{l_0}\right)} \tag{B.14}$$

Assuming that the transient extensional viscosity as a function of time can be fitted reasonably well using a power law model (see Figure B.1), then the equation of a straight-line fit to the data is of the form

$$\log(\eta_{\mathrm{E}}{}^{+}) = m \log t + \log(\eta_{\mathrm{E,\,0}}{}^{+}) \tag{B.15}$$

where $\eta_{\mathrm{E}}{}^{+} = \eta_{\mathrm{E,\,0}}{}^{+}$ at t = 1 s and $\eta_{\mathrm{E,\,0}}{}^{+}$ and m are constants.

Equation (B.15) can be rewritten as

$$\eta_{\mathrm{E}}{}^{+} = \eta_{\mathrm{E,\,0}}{}^{+} t^{m} \tag{B.16}$$

The force over the test duration is thus approximated by

$$F = \eta_{\mathrm{E,\,0}}{}^{+} t^{m} \dot{\varepsilon} A_0 \mathrm{e}^{\left(\frac{-\omega r t}{l_0}\right)} \tag{B.17}$$

Given the values of $\eta_{\mathrm{E,\,0}}{}^{+}$ and m, the force F can be determined as a function of time and, assuming that all strain rate data fall on the same master curve (see Figure B.1), also as a function of strain if one ignores any additional strain-hardening phenomenon. Thus the relative uncertainty in the force F, given the decreasing magnitude of the force at high strains, can be reasonably accurately determined.