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**Plastics — Determination of the  
transient extensional viscosity of  
polymer melts**

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

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# Contents

	Page
Foreword.....	iv
<b>1 Scope</b> .....	<b>1</b>
<b>2 Normative references</b> .....	<b>1</b>
<b>3 Terms and definitions</b> .....	<b>1</b>
<b>4 General principles</b> .....	<b>3</b>
<b>5 Apparatus</b> .....	<b>3</b>
5.1 General description.....	3
5.2 Silicon oil bath/temperature-controlled chamber.....	5
5.3 Temperature measurement and control.....	6
5.4 Strain and strain rate measurement.....	6
5.5 Force measurement.....	6
5.6 Calibration.....	6
<b>6 Sampling and specimen preparation</b> .....	<b>7</b>
6.1 Sampling.....	7
6.2 Specimen preparation.....	7
6.3 Specimen mounting.....	7
<b>7 Procedure</b> .....	<b>8</b>
7.1 Specimen loading.....	8
7.2 Pre-conditioning of the specimen.....	8
7.3 Testing.....	8
<b>8 Analysis of extensional flow measurements</b> .....	<b>9</b>
8.1 Symbols used.....	9
8.2 Analysis of extensional flow.....	9
8.2.1 General.....	9
8.2.2 Analysis for type A and B instruments (rotating clamps).....	10
8.2.3 Analysis for type C and D instruments (translating clamps).....	11
<b>9 Precision</b> .....	<b>11</b>
<b>10 Test report</b> .....	<b>12</b>
<b>Annex A (informative) Checking for swelling of specimens due to immersion in silicone oil or other fluids</b> .....	<b>13</b>
<b>Annex B (informative) Uncertainties in transient extensional viscosity testing</b> .....	<b>14</b>
<b>Bibliography</b> .....	<b>19</b>

## 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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see [www.iso.org/directives](http://www.iso.org/directives)).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see [www.iso.org/iso/foreword.html](http://www.iso.org/iso/foreword.html).

This document 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 20965:2005), which has been technically revised.

The main changes compared to the previous edition are as follows:

- figures have been updated and figure keys have been introduced;
- calibration period in 5.6 has been clarified.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at [www.iso.org/members.html](http://www.iso.org/members.html).

# Plastics — Determination of the transient extensional viscosity of polymer melts

## 1 Scope

This document 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  $0,01 \text{ s}^{-1}$  to  $1 \text{ s}^{-1}$ , at Hencky strains up to approximately 4 and at temperatures up to approximately  $250 \text{ °C}$  (see NOTES 1 and 2). It is suitable for measuring transient extensional viscosity values typically in the range from approximately  $10^4 \text{ Pa}\cdot\text{s}$  to  $10^7 \text{ Pa}\cdot\text{s}$  (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 can 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.

## 2 Normative references (standards.iteh.ai)

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 472, *Plastics — Vocabulary*

## 3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

### 3.1

#### Hencky strain

$\varepsilon$

strain given by the natural logarithm of the elongation ratio given by [Formula \(1\)](#)

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

where

$l$  is the specimen length and

$l_0$  is the original specimen length

Note 1 to entry: It is also referred to as the natural or true strain.

## ISO 20965:2021(E)

Note 2 to entry: It is dimensionless.

### 3.2 Hencky strain rate

$\varepsilon$   
rate of change of Hencky strain with time, given by [Formula \(2\)](#)

$$\varepsilon = 1/l \times \partial l / \partial t \quad (2)$$

where  $t$  is the time

Note 1 to entry: It is independent of the original specimen length  $l_0$ .

Note 2 to entry: It is expressed in reciprocal seconds.

### 3.3 net tensile stress

$\sigma_E$   
for tensile uniaxial extension, stress given by [Formula \(3\)](#)

$$\sigma_E = \sigma_{11} - \sigma_{22} = \sigma_{11} - \sigma_{33} = \sigma_{zz} - \sigma_{rr} \quad (3)$$

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

Note 1 to entry: The tensile stress growth function is indicated by  $\sigma_E^+$  where the + indicates start-up of flow.

Note 2 to entry: Net tensile stress is expressed in pascals.

### 3.4 tensile stress growth coefficient

$\eta_E^+$   
ratio of the net tensile stress to the Hencky strain rate, given by [Formula \(4\)](#)

$$\eta_E^+(t, \varepsilon) = \sigma_E / \varepsilon \quad (4)$$

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

Note 1 to entry: It is also known for the purposes of this document as “transient extensional viscosity”.

Note 2 to entry: It is a transient term.

Note 3 to entry: It is expressed in pascal seconds.

### 3.5 tensile viscosity

$\eta_E$   
term given by [Formula \(5\)](#)

$$\eta_E(t, \varepsilon) = \lim_{t \rightarrow \infty} [\eta_E^+(t, \varepsilon)] \quad (5)$$

Note 1 to entry: 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 to entry: 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 varies 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 document 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.

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## 5 Apparatus

### 5.1 General description

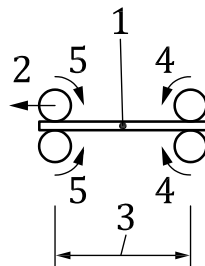
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The measuring apparatus shall consist of one of the following types, shown in [Figure 1](#) to [Figure 4](#). These types define the various instrument configurations. The notation used in these figures is defined in [8.1](#).

**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 can introduce noise and errors into the force signal.

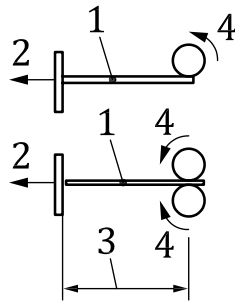


#### Key

- 1 specimen
- 2 force,  $F$ , expressed in N
- 3 original specimen length,  $l_0$ , expressed in m
- 4 angular speed of rotating clamps of side 1,  $\omega_1$ , expressed in  $\text{rad}\cdot\text{s}^{-1}$
- 5 angular speed of rotating clamps of side 2,  $\omega_2$ , expressed in  $\text{rad}\cdot\text{s}^{-1}$

**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.

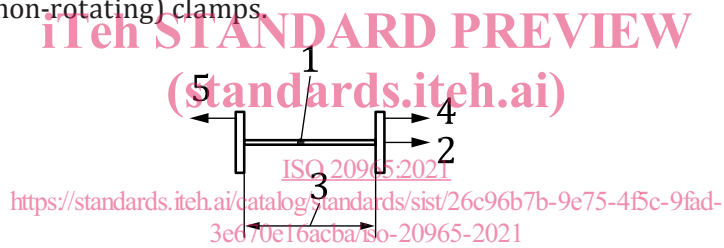


**Key**

- 1 specimen
- 2 force,  $F$ , expressed in N
- 3 original specimen length,  $l_0$ , expressed in m
- 4 angular speed of rotating clamps,  $\omega_1$ , expressed in  $\text{rad}\cdot\text{s}^{-1}$

**Figure 2 — Schematic diagram of type B test instrument**

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



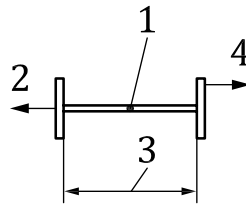
**Key**

- 1 specimen
- 2 force,  $F$ , expressed in N
- 3 original specimen length,  $l_0$ , expressed in m
- 4 speed of the ends of the specimen of side 1,  $V_1$ , expressed in  $\text{m}\cdot\text{s}^{-1}$
- 5 speed of the ends of the specimen of side 2,  $V_2$ , expressed in  $\text{m}\cdot\text{s}^{-1}$

**Figure 3 — Schematic diagram of type C test instrument**

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



**Key**

- 1 specimen
- 2 force,  $F$ , expressed in N
- 3 original specimen length,  $l_0$ , expressed in m
- 4 speed of the ends of the specimen,  $m \cdot s^{-1}$ ,  $V_1$ , expressed in  $m \cdot s^{-1}$

**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 oil 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 can 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 can 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 can 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 NOTE 1 in [7.1](#)). When quantitative results are required, then this check shall be made.

Even if the silicone oil does not affect the shape of the tensile stress growth coefficient versus strain (or time) plot, it can 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 4 Alternative methods for checking the effect of immersion in silicone oil on the specimen can 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 result in excessive strain in those regions. This can lead to premature failure, particularly for materials that do not exhibit a high degree of strain hardening.

The spatial temperature variation shall be within  $\pm 0,75$  K.

The temporal temperature variation shall be within  $\pm 1,0$  K of the set temperature.

The temperature-measuring device shall have a resolution of 0,1 K and shall be calibrated using a device accurate to within  $\pm 0,1$  K.

### 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 can 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 can 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  $\pm 3$  % of the absolute value.

The apparatus shall have an accuracy of strain rate determination or measurement to within  $\pm 3$  % 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).

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  $\pm 2$  % of the full-scale value.

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

### 5.6 Calibration

The force, displacement, rate of displacement and temperature functions of the rheometer shall be calibrated at least once in 6 months.

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

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 can 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 results 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 in at least three positions along its length. Repeat these measurements after rotating the specimen by  $90^\circ$ . Calculate an average value for the diameter from these measurements.

For rectangular specimens, measure the width and thickness in 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  $\pm 2\%$  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