INTERNATIONAL STANDARD

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Plastics — Determination of creep behaviour —

Part 1:
Tensile creep

Plastiques — Détermination du comportement au fluage —

Partie 1: Fluage en traction

Reference number
ISO 899-1:1993(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 899-1 was prepared by Technical Committee ISO/TC 61, *Plastics*, Sub-Committee SC 2, *Mechanical properties*.

Together with ISO 899-2, it cancels and replaces ISO 899:1981 and ISO 6602:1985, which have been technically revised.

ISO 899 consists of the following parts, under the general title *Plastics — Determination of creep behaviour*:

— *Part 1: Tensile creep*

— *Part 2: Flexural creep by three-point loading*

Annexes A and B of this part of ISO 899 are for information only.

International Organization for Standardization
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Plastics — Determination of creep behaviour —

Part 1:
Tensile creep

1 Scope

1.1 This part of ISO 899 specifies a method for determining the tensile creep of plastics in the form of standard test specimens under specified conditions such as those of pretreatment, temperature and humidity.

1.2 The method is suitable for use with rigid and semi-rigid non-reinforced, filled and fibre-reinforced plastics materials (see ISO 472 for definitions) in the form of dumb-bell-shaped test specimens moulded directly or machined from sheets or moulded articles.

1.3 The method is intended to provide data for engineering-design and research and development purposes.

1.4 Tensile creep may vary significantly with differences in specimen preparation and dimensions and in the test environment. The thermal history of the test specimen can also have profound effects on its creep behaviour (see annex A). Consequently, when precise comparative results are required, these factors must be carefully controlled.

1.5 If tensile-creep properties are to be used for engineering-design purposes, the plastics materials should be tested over a broad range of stresses, times and environmental conditions.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 899. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 899 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

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

ISO 472:1988, *Plastics — Vocabulary.*

ISO 527-1:1993, *Plastics — Determination of tensile properties — Part 1: General principles.*

ISO 527-2:1993, *Plastics — Determination of tensile properties — Part 2: Test conditions for moulding and extrusion plastics.*

3 Definitions

For the purposes of this part of ISO 899, the definitions given in ISO 472 and the following definitions apply.

3.1 creep: The increase in strain with time when a constant force is applied.

3.2 initial stress, σ**:** The tensile force per unit area of the initial cross-section within the gauge length.

It is given by the equation

$$\sigma = \frac{F}{A}$$

where

F is the force, in newtons;

A is the initial cross-sectional area of the specimen, in square millimetres.

The stress is expressed in megapascals.

3.3 extension, $(\Delta L)_t$**:** The increase in the distance between the gauge marks, expressed in millimetres, at time t.

It is given by the equation

$$(\Delta L)_t = L_t - L_0$$

where

L_t is the gauge length, in millimetres, at any given time t during the test;

L_0 is the original gauge length, in millimetres, of the specimen after application of a preload but prior to application of the test load.

3.4 tensile-creep strain, ε_t**:** The change in length per unit original length of the gauge length produced by the applied load at any given time during a creep test. It is expressed as a dimensionless ratio or as a percentage.

It is given by the equation

$$\varepsilon_t = \frac{(\Delta L)_t}{L_0}$$

or

$$\varepsilon_t = \frac{(\Delta L)_t}{L_0} \times 100\ (\%)$$

3.5 tensile-creep modulus, E_t**:** The ratio of initial stress to creep strain, calculated as in 7.1.

3.6 isochronous stress-strain curve: A Cartesian plot of stress versus creep strain, at a specific time after application of the test load.

3.7 time to rupture: The period of time which elapses between the point in time at which the specimen is fully loaded and the rupture point.

3.8 creep-strength limit: That initial stress which will just cause rupture ($\sigma_{B,t}$) or will produce a specified strain ($\sigma_{\varepsilon,t}$) at a specified time t, at a given temperature and relative humidity.

3.9 recovery from creep: The decrease in strain at any given time after completely unloading the specimen, expressed as a percentage of the strain just prior to the removal of the load.

4 Apparatus

4.1 Gripping device, capable of ensuring that the direction of the load applied to the test specimen coincides as closely as possible with the longitudinal axis of the specimen. This ensures that the test specimen is subjected to simple stress and that the stresses in the loaded section of the specimen may be assumed to be uniformly distributed over cross-sections perpendicular to the direction of the applied load.

NOTE 1 It is recommended that grips be used that will allow the specimen to be fixed in place, correctly aligned, prior to applying the load. Self-locking grips which allow the specimen to move as the load increases are not suitable for this test.

4.2 Loading system, capable of ensuring that the load is applied smoothly, without causing transient overloading, and that the load is maintained to within ± 1 % of the desired load. In creep-to-rupture tests, provision shall be made to prevent any shocks which occur at the moment of rupture being transmitted to adjacent loading systems. The loading mechanism shall allow rapid, smooth and reproducible loading.

4.3 Extension-measuring device, comprising any contactless or contact device capable of measuring the extension of the specimen gauge length under load without influencing the specimen behaviour by mechanical effects (e.g. undesirable deformations, notches), other physical effects (e.g. heating of the specimen) or chemical effects. In the case of contactless (optical) measurement of the strain, the longitudinal axis of the specimen shall be perpendicular to the optical axis of the measuring device. The accuracy of the extension-measuring device shall be within ± 0,01 mm.

For creep-to-rupture tests, it is recommended that the extension be measured by means of a contactless optical system operating on the cathetometer principle. Automatic indication of time to rupture is highly desirable. The gauge length shall be marked on the specimen, either by attaching (metal) clips with scratched-on gauge marks, or by gauge marks ruled with an inert, thermally stable paint.

Electrical-resistance strain gauges are suitable only if the material tested is of such a nature as to permit such strain gauges to be attached to the specimen by means of adhesive and only if the adhesion quality is constant during the duration of the test.

4.4 Time-measurement device, accurate to 0,1 %.

4.5 Micrometer, reading to 0,01 mm or closer, for measuring the thickness and width of the test specimen.

5 Test specimens

Use test specimens of the same shape and dimensions as specified for the determination of tensile properties (see ISO 527-2).

6 Procedure

6.1 Conditioning and test atmosphere

Condition the test specimens as specified in the International Standard for the material under test. In the absence of any information on conditioning, use the most appropriate set of conditions specified in ISO 291, unless otherwise agreed upon by the interested parties.

NOTE 2 The creep behaviour will be affected not only by the thermal history of the specimen under test, but also by the temperature and (where applicable) humidity used in conditioning.

Conduct the test in the same atmosphere as used for conditioning, unless otherwise agreed upon by the interested parties, e.g. for testing at elevated or low temperatures. Ensure that the variation in temperature during the duration of the test remains within ± 2 °C.

6.2 Measurement of test-specimen dimensions

Measure the dimensions of the conditioned test specimens in accordance with ISO 527-1:1993, subclause 9.2.

6.3 Mounting the test specimens

Mount a conditioned and measured specimen in the grips and set up the extension-measuring device as required.

6.4 Selection of stress value

Select a stress value appropriate to the application envisaged for the material under test, and calculate, using the equation given in 3.2, the test load to be applied to the test specimen.

6.5 Loading procedure

6.5.1 Preloading

When it is necessary to preload the test specimen prior to increasing the load to the test load, for example in order to eliminate backlash by the test gear, take care to ensure that the preload does not influence the test results. Do not apply the preload until the temperature and humidity of the test specimen (gripped in the test apparatus) correspond to the test conditions. Measure the gauge length after application of the preload. Maintain the preload during the whole duration of the test.

6.5.2 Loading

Load the test specimen progressively so that full loading of the test specimen is reached between 1 s and 5 s after the beginning of the application of the load. Use the same rate of loading for each of a series of tests on one material.

Take the total load (including the preload) to be the test load.

6.6 Extension-measurement schedule

Record the point in time at which the specimen is fully loaded as $t = 0$. Unless the extension is automatically and/or continuously recorded, choose the times for making individual measurements as a function of the creep curve obtained from the particular material under test. It is preferable to use the following measurement schedule:

1 min, 3 min, 6 min, 12 min and 30 min;

1 h, 2 h, 5 h, 10 h, 20 h, 50 h, 100 h, 200 h, 500 h, 1 000 h, etc.

If discontinuities are suspected or encountered in the creep-strain versus time plot, take readings more frequently than recommended above.

6.7 Time measurement

Measure, to within ± 0,1 % or ± 2 s (whichever is the less severe tolerance), the total time which has elapsed up to each creep measurement.

6.8 Temperature and humidity control

Unless temperature and relative humidity (where applicable) are recorded automatically, record them at the beginning of the test and then at least three times a day initially. When it has become evident that the conditions are stable within the specified limits, they may be checked less frequently.

6.9 Measurement of recovery rate (optional)

Upon completion of non-rupture tests, remove the load rapidly and smoothly and measure the recovery rate using, for instance, the same schedule as was used for creep measurement.

7 Expression of results

7.1 Method of calculation

Calculate the tensile-creep modulus E_t by dividing the initial stress σ by the strain ε_t at each of the selected measurement times.

It is given, in megapascals, by the equation

$$E_t = \frac{\sigma}{\varepsilon_t} = \frac{F \cdot L_0}{A \cdot (\Delta L)_t}$$

where

F is the applied force, in newtons;

L_0 is the initial gauge length, in millimetres;

A is the initial cross-sectional area, in square millimetres, of the specimen;

$(\Delta L)_t$ is the extension, in millimetres, at time t.

7.2 Presentation of results

7.2.1 Creep curves

If testing is carried out at different temperatures, the raw data should preferably be presented, for each temperature, as a series of creep curves showing the tensile strain plotted against the logarithm of time, one curve being plotted for each initial stress used (see figure 1).

The data may also be presented in other ways, e.g. as described in 7.2.2 and 7.2.3, to provide information required for particular applications.

7.2.2 Creep-modulus/time curves

For each initial stress used, the tensile-creep modulus, calculated in accordance with 7.1, may be plotted against the logarithm of the time under load (see figure 2).

If testing is carried out at different temperatures, plot a series of curves for each temperature.

7.2.3 Isochronous stress-strain curves

An isochronous stress-strain curve is a Cartesian plot showing how the strain depends on the applied load, at a specific point in time after application of the load. Several curves are normally plotted, corresponding to times under load of 1 h, 10 h, 100 h, 1 000 h, and 10 000 h. Since each creep test gives only one point on each curve, it is necessary to carry out the test at at least three different stresses, and preferably more, to obtain an isochronous curve.

To obtain an isochronous stress-strain curve for a particular time under load (say 10 h) from a series of creep curves as shown in figure 1, read off, from each creep curve, the strain at 10 h, and plot these strain values (x-axis) against the corresponding stress values (y-axis). Repeat the process for other times to obtain a series of isochronous curves (see figure 3).

If testing is carried out at different temperatures, plot a series of curves for each temperature.

7.2.4 Three-dimensional representation

A relationship of the form $\varepsilon = f(t, \sigma)$ exists between the different types of curve (see figures 1 to 3) that can be derived from the raw creep-test data. This relationship can be represented as a surface in a three-dimensional space (see reference [1], annex B).

All the curves that can be derived from the raw creep-test data form part of this surface. Because of the experimental error inherent in each measurement, the points corresponding to the actual measurements normally do not lie on the curves but just off them.

The surface $\varepsilon = f(t, \sigma)$ can therefore be generated by deriving a number of the curves which form it, but a number of sophisticated smoothing operations are usually necessary. Computer techniques permit this to be done rapidly and reliably.

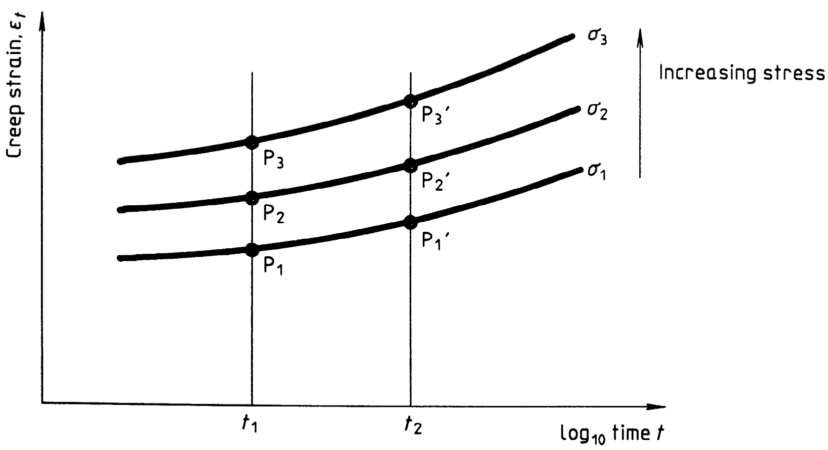


Figure 1 — Creep curves

7.2.5 Creep-to-rupture curves

Creep-to-rupture curves allow the prediction of the time to failure at any stress. They may be plotted as stress against log time (see figure 4) or log stress against log time.

7.3 Precision

The precision of this test method is not known because interlaboratory data are not available. When interlaboratory data are obtained, a precision statement will be added at the next revision.

8 Test report

The test report shall include the following particulars:

a) a reference to this part of ISO 899;

b) a complete description of the material tested, including all pertinent information on composition, preparation, manufacturer, tradename, code number, date of manufacture, type of moulding and any annealing;

c) the dimensions of each test specimen;

d) the method of preparation of the test specimens;

e) the directions of the principal axes of the test specimens with respect to the dimensions of the product or some known or inferred orientation in the material;

f) details of the atmosphere used for conditioning and testing;

g) the creep-test data for each temperature at which testing was carried out, presented in one or more of the graphical forms described in 7.2, or in tabular form;

h) if recovery-rate measurements are made, the time-dependent strain after unloading the test specimen (see 6.9).

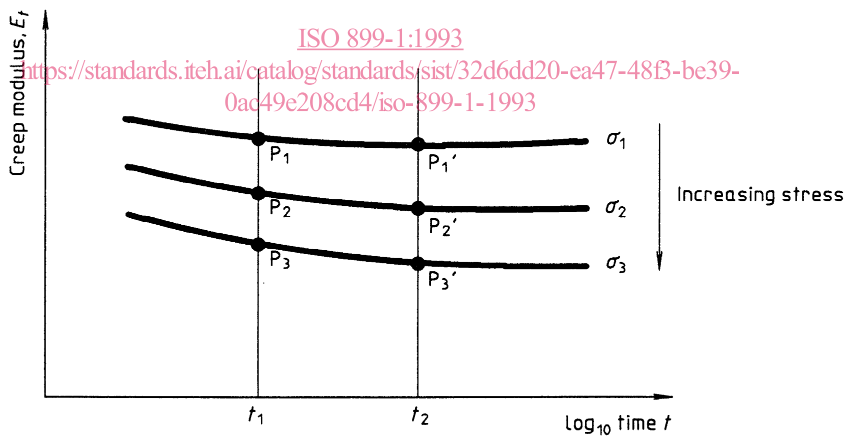


Figure 2 — Creep-modulus/time curves

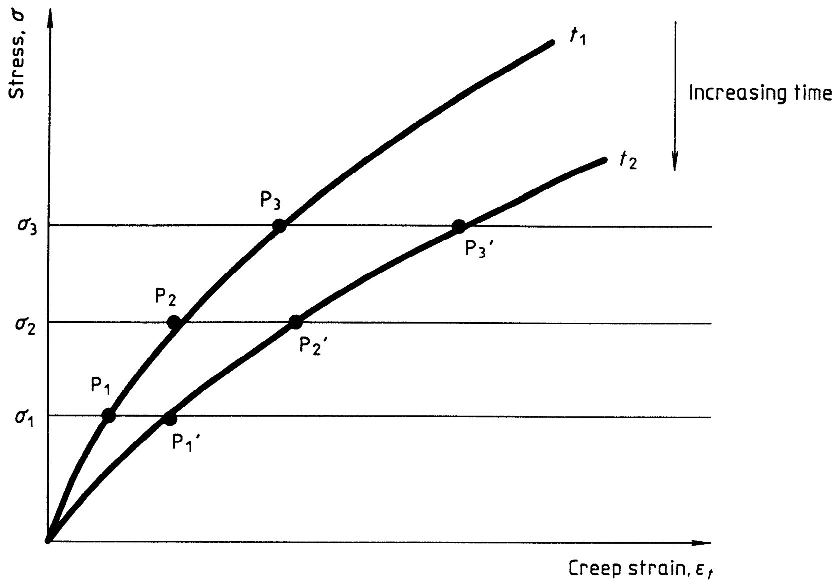
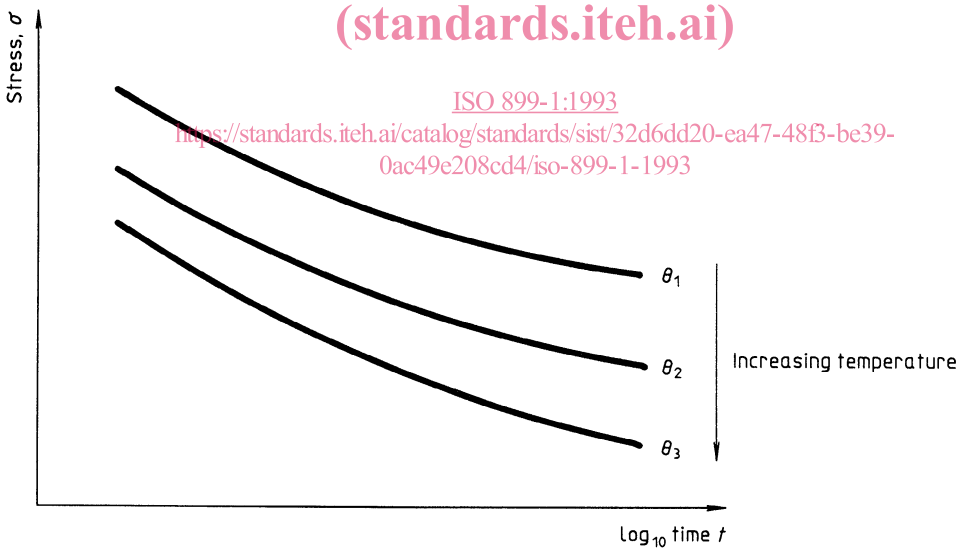


Figure 3 — Isochronous stress-strain curves



NOTE — The stress σ may also be plotted on a logarithmic scale.

Figure 4 — Creep-to-rupture curves

Annex A
(informative)

Physical-ageing effects on the creep of polymers

A.1 General

Physical ageing takes place when a polymer is cooled from an elevated temperature at which the molecular mobility is high to a lower temperature at which relaxation times for molecular motions are long in comparison with the storage time at that temperature. Under these circumstances, changes in the structure will take place over a long period of time, involving rearrangement in the shape and packing of molecules as the polymer approaches the equilibrium structural state for the lower temperature. Associated with this ageing process, there is a progressive decrease in the molecular mobility of the polymer, even when the temperature remains constant. As a direct consequence of this, the creep deformation produced by an applied stress will depend upon the age of the polymer, creep rates being lower in more highly aged material.

This is illustrated in figure A.1 which shows creep compliance curves for PVC specimens of different ages. Each of these specimens has been rapidly cooled from a temperature of 85 °C (close to T_g) and stored at the test temperature of 23 °C for different times t_e prior to load application. The physical age of a specimen is then defined by the time t_e and it can be seen that the older the specimen the further its creep curve is shifted on the time axis.

A.2 Creep at elevated temperatures

The influence of physical ageing on creep behaviour is more complicated when measurements are made at elevated temperatures following a storage period at a lower (ambient) temperature. Under these circumstances, the physical ageing that takes place during storage at the lower temperature is temporarily reversed when the specimen is heated to the test temperature. The rate at which this takes place depends on the size of the temperature change and the age of the specimen when the temperature is raised. Following the reduction in the apparent (or effective) age of the specimen, physical ageing is reactivated at the higher temperature. Again, the timescale over which this happens depends on the test conditions. One consequence of these changes in age state caused by the temperature increase is thus a dependence of the creep behaviour at the elevated temperature on the dwell time at this temperature prior to load application.

Typical ways in which this type of thermal history influences creep compliance are illustrated in figures A.2 and A.3. In figure A.2, specimens were stored for a period t_{e1} of 200 h at a temperature of 23 °C prior to heating to the test temperature of 44 °C. Creep curves were then obtained after different periods t_{e2} at 44 °C prior to load application. Despite the relatively long storage period t_{e1} at ambient temperature, the creep behaviour shows a strong dependence on the dwell time t_{e2}.

In figure A.3, creep tests were carried out under the same conditions but following a storage period t_{e1} of greater than 1 year at 23 °C prior to heating to the test temperature. The progressive reduction in the effective age of the specimens is actually observed here as a shift in the curves to shorter creep times, and results from the more extensive structural changes that have taken place in the specimens through physical ageing before heating.