
**Plastics — Heatshrinkable films of
polyethylene, ethylene copolymers and
their mixtures — Determination of
shrinkage stress and contraction stress**

*Plastiques — Films thermorétractables en polyéthylène, en copolymères
d'éthylène et leurs mélanges — Détermination des contraintes de réfraction
et de contraction*

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ISO 14616:1997

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Foreword

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Plastics — Heatshrinkable films of polyethylene, ethylene copolymers and their mixtures — Determination of shrinkage stress and contraction stress

1 Scope

The purpose of this standard is to describe a conventional method for measuring the shrinking and contracting forces of heatshrinkable films made from polyethylene, ethylene copolymers and their mixtures.

The method described in this standard can possibly be applied to other materials under proper operating conditions.

NOTE 1 This method also allows a shrinkage ratio to be assessed. However, the reference method for measuring the shrinkage ratio is ISO 11501, Plastics - Film and sheeting - Determination of dimensional change on heating.

2 Definitions

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For the purposes of this standard, the following definitions apply:

2.1 heatshrinkable film: Film which is extruded and stretched (hot drawn) during manufacture and which remains in this state after cooling down.

NOTE 2 When this film is again brought up to the temperature at which the stress was induced and then fixed during its manufacture, this stress is liberated and the film shrinks back.

Depending on the treatment applied, the film can be

- either heatshrinkable both lengthwise and crosswise: this film is called "biaxially oriented",
- or mainly heatshrinkable lengthwise: this film is called "uniaxially oriented".

2.2 shrinkage ratio: Decrease in the length of the specimen when it is brought up to the shrinkage temperature, expressed as a percentage of the initial specimen length.

2.3 shrinking force, F_r : Force developed by the film when it reaches the temperature corresponding to that at which the stress was induced at the time of manufacture.

A high film shrinkage is linked to this small force.

This small force and this high shrinkage permit the film to gently shrink down on the load.

2.4 contracting force, F_c : Force developed by the film during its cooling process.

This force, much greater than the shrinkage force, ensures the fastening of the load.

2.5 reference temperature, θ_r : Air temperature measured by the probe located 5 mm adjacent to the force measurement specimen 45 s after mounting in the heating hood. Under these conditions, it is considered that sufficient time is allowed for the thermocouple used for the measurement to reach the temperature.

3 Principle of the measurement

Specimens connected to a force meter or to a displacement transducer are rapidly brought up to the shrinkage temperature, then cooled in the open air to an ambient temperature of $23^\circ\text{C} \pm 2^\circ\text{C}$.

A device continuously records the reference temperature, the force and possibly the displacement. The recording of these measurements allows one to determine the optimal shrinking conditions.

4 Apparatus

4.1 Heating hood, vertically mobile, equipped with heating elements and with a regulating device capable of maintaining the air temperature at the specified temperature.

4.2 T-shaped bracket allowing the fixing of the specimen.

4.3 Force meter, allowing the measurement of the shrinkage and contraction forces within a force range of at least 20 N with an accuracy of $\pm 2\%$.

4.4 Displacement transducer, capable of measuring a displacement with an accuracy of ± 1 mm.

4.5 Multichannel acquisition device, allowing the continuous recording of the different variables: times, forces, displacement, ...

4.6 Thermocouple probe, having a diameter of 1,5 mm (max), capable of measuring the air temperature 5 mm off the force measurement specimen with an accuracy of $\pm 2^\circ\text{C}$.

The thermocouple tip shall be oriented towards the middle of the specimen.

This thermocouple is used for determining the reference temperature θ_r .

Depending on the heating resistors and on the location of the apparatus (influence of the environment), the performance characteristics of each individual testing apparatus may differ slightly.

It is therefore necessary to determine the relationship between the temperature θ_r (45 s after mounting the heating hood, see 2.5) and that displayed by the regulating device.

Plot the correlation curve between θ_r and the settings of the hood temperature regulating device.

This operation shall be repeated at regular intervals in order to detect any possible drift.

NOTE 3 In general, this correlation curve is determined for a range of temperatures θ_r between 100°C and 230°C.

4.7 Thickness gauge, capable of measuring to an accuracy of 1 µm.

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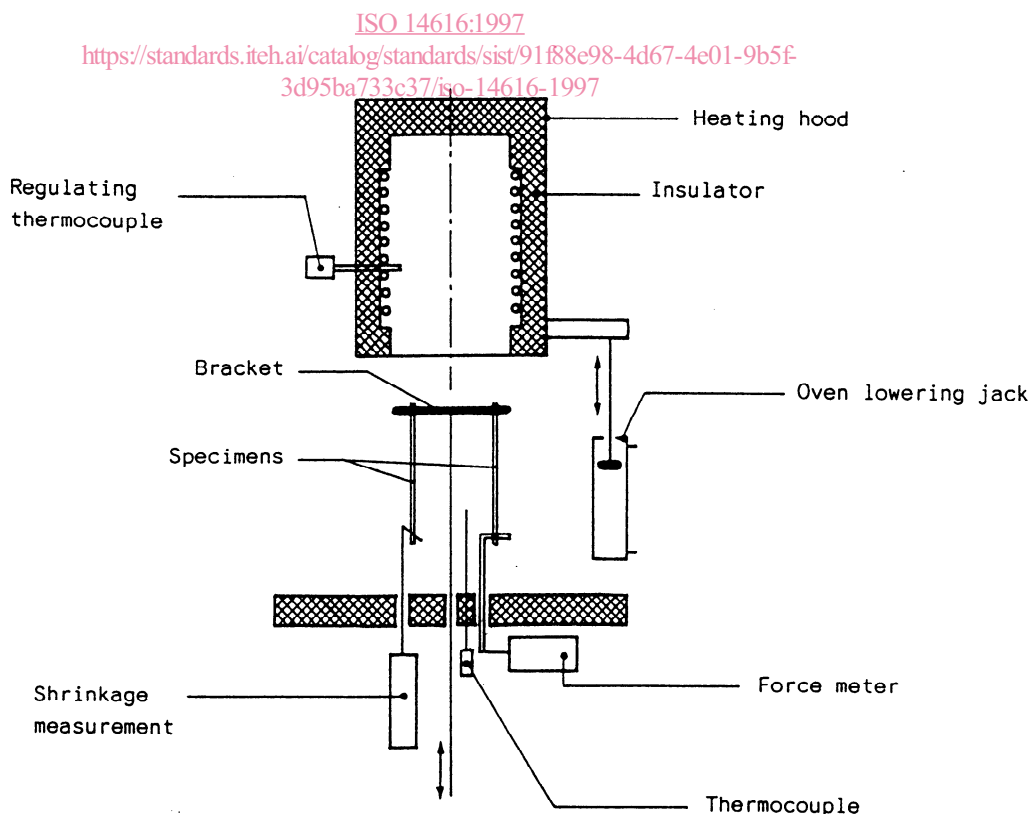


Figure 1 - Example of apparatus

5 Specimens

Prepare at least ten specimens in each direction (lengthwise and crosswise).

Their width shall be $15 \text{ mm} \pm 0,5 \text{ mm}$.

Their length shall be such that it is possible to have an effective length between jaws of 100 mm.

Their thickness, measured by the thickness gauge (4.7) to the nearest $1 \mu\text{m}$, is represented by the mean of three measurements repeated regularly on the specimen length.

6 Procedure

6.1 Measurement of the shrinking and contracting forces

The procedure is identical irrespective of the direction of the specimens, lengthwise or crosswise.

As the curves obtained with the lengthwise specimens are easier to develop, it is advised to begin testing in the lengthwise direction.

6.1.1 Install the specimens perfectly flat and straight on the force meter without applying any tensile force.

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6.1.2 In the absence of a reference temperature θ_r , known or specified by a product standard, set the temperature regulating device so as to obtain a reference temperature θ_r of $200^\circ\text{C} \pm 3^\circ\text{C}$ using the correlation curve defined in 4.6.

6.1.3 Lower the hood in order to bring the specimens to the shrinkage temperature.

Start the timer (or the recording of the time on the plotting table) and the recording of the force.

The film shrinkage temperature is reached when the maximum shrinking force is attained.

Record this force, F_r , in newtons.

When this force has decreased by around 15 % to 30 %, lift the hood and stop the timer (or the recording of the time). Record the time.

Continue to record the forces.

Record the maximum value of force attained by the contraction, F_c , in newtons.

6.1.4 If the maximum of the recorded shrinking force appears after less than 15 s exposure to the heat, repeat the test by lowering the reference temperature θ_r by successive steps of 10 °C until shrinkage is obtained between 15 s and 30 s.

If the maximum shrinking force does not appear after 30 s exposure to the heat, resume testing by raising the temperature by successive steps of 10 °C until shrinkage is obtained between 15 s and 30 s.

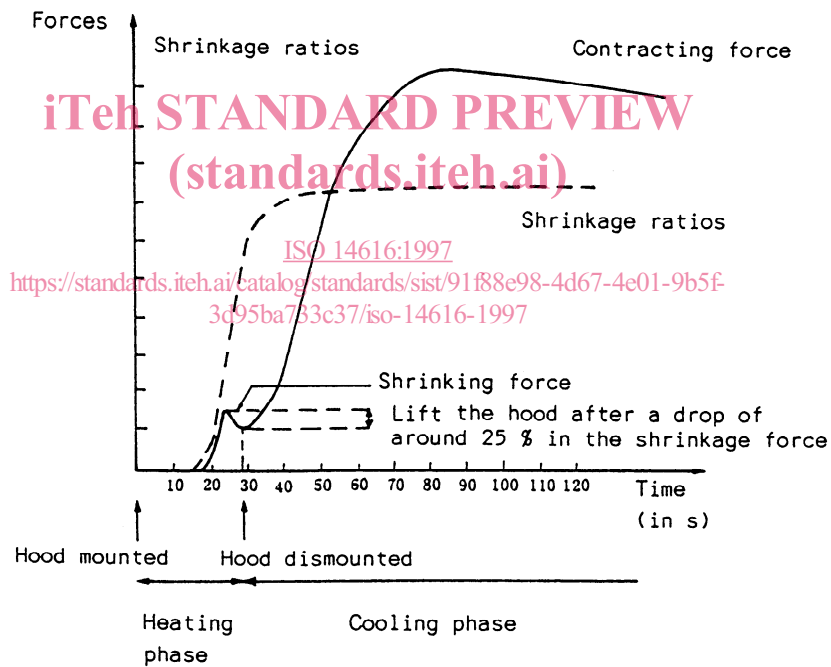


Figure 2 - Measurement of the forces and of the shrinkage ratio - Example of typical kinetics of the phenomena

6.2 Assessment of the shrinkage ratio

The assessment of the shrinkage ratio can be carried out either at the same time as that of the forces or consecutively, depending on the configuration of the apparatus.

It enables one to rapidly compare the properties of the shrinkable films in the production control procedure.

NOTE 4 This method does not replace the reference method for measuring the shrinkage ratio specified in ISO 11501.

6.2.1 Install the specimen perfectly flat and straight, between one of the arms of the bracket and the displacement transducer, the effective length of the specimen being 100 mm.

6.2.2 Conduct the measurement under the same conditions as those adopted for the measurement of the shrinking and contracting forces.

When they are conducted simultaneously, make additional provision for recording of the displacements.

Record the final value of the displacement after the cooling down of the specimen.

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7 Expression of results

Calculate:

- the shrinking stress in pascals, Pa

$$\sigma_r = \frac{F_r}{S} \times 10^{-6}$$

- the contracting stress in pascals, Pa

$$\sigma_c = \frac{F_c}{S} \times 10^{-6}$$

- the shrinkage ratio, R, in percentage

$$R = \frac{L_o - L}{L_o} \times 100$$

where:

F_r is the shrinking force, in newtons;

F_c is the contracting force in newtons;

S is the area of the initial cross section of the specimen, in square millimetres;

L_0 is the initial length of the specimen, in millimetres;

L is the length of the specimen after heat shrinking, in millimetres.

8 Precision

Since the precision of this test method is not known because inter-laboratory data are not available, this method is only applicable for comparison purposes within a laboratory.

9 Test report

The test report shall refer to this standard and shall mention the following information:

- a) type and origin of the film being tested;
- b) thickness of the film;
- c) degree of orientation;
- d) reference temperature θ_r ;
- e) value of the shrinking stress σ_r , in pascals, Pa, and value of contracting stress, σ_c ;
- f) shrinkage ratio, R , in percentage for information;
- g) mean and standard deviation;
- h) procedural details not provided for in the standard and incidents which may have influenced the results;
- i) date of test.