SLOVENSKI STANDARD
SIST ISO 1628-3:1996

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Polimerni materiali - Določanje viskoznostnega števila in mejnega viskoznostnega števila - 3. del: Polietileni in polipropileni

Plastics -- Determination of viscosity number and limiting viscosity number -- Part 3: Polyethylenes and polypropylenes

Plastiques -- Détermination de l'indice de viscosité et de l'indice limite de viscosité -- Partie 3: Polyéthylènes et polypropylènes

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83.080.20	Plastomeri	Thermoplastic materials

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INTERNATIONAL STANDARD

ISO 1628-3

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Plastics — Determination of viscosity number and limiting viscosity number —

Part 3: Polyethylenes and polypropylenes

Plastiques — Détermination de l'indice de viscosité et de l'indice limite de viscosité —
Partie 3: Polyéthylènes et polypropylènes

Reference number
ISO 1628-3:1991(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 1628-3 was prepared by Technical Committee ISO/TC 61, *Plastics*.

It cancels and replaces International Standard ISO 1191:1975, of which it constitutes a technical revision.

ISO 1628 consists of the following parts, under the general title *Plastics — Determination of viscosity number and limiting viscosity number*:

— *Part 1: General conditions*

— *Part 2: Poly(vinyl chloride) resins*

— *Part 3: Polyethylenes and polypropylenes*

— *Part 4: Polycarbonate (PC) moulding and extrusion materials*

— *Part 5: Poly(alkylene terephthalates)*

— *Part 6: Methyl methacrylate polymers*

(The general title of ISO 1628-1 is *Guidelines for the standardization of methods for the determination of viscosity number and limiting viscosity number of polymers in dilute solution*)

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Plastics — Determination of viscosity number and limiting viscosity number —

Part 3:
Polyethylenes and polypropylenes

1 Scope

This part of ISO 1628 defines particular conditions for determining the viscosity number and limiting viscosity number of polyethylenes and polypropylenes at 135 °C in dilute solution. It is applicable to low, medium and high-density polyethylenes and to a wide range of polypropylenes, including pure isotactic, stereoblock and atactic polymers.

The viscosity of polymer solutions may be affected by additives present in the sample. The value of a viscosity number determined by this method may therefore be unreliable if the sample contains fillers or other additives.

NOTE 1 Viscosity number is also known as the Staudinger function (J_v) and limiting viscosity number as the Staudinger index (J_g).

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 1628. 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 1628 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 1628-1:1984, *Guidelines for the standardization of methods for the determination of viscosity number and limiting viscosity number of polymers in dilute solution — Part 1: General conditions.*

ISO 3105:1976, *Glass capillary kinematic viscometers — Specification and operating instructions.*

3 Principle

The times of flow of a solvent and a solution of polymer at a specified concentration in that solvent are measured at 135 °C. The viscosity number and limiting viscosity number are calculated from these measurements and from the known concentration of the solution.

Ethylene and isotactic polypropylene polymers are not soluble at room temperature in any known solvents. Precautions must therefore be taken during the test to avoid any precipitation of polymer, which would give an incorrect solution concentration.

4 Solvent

4.1 Decahydronaphthalene, analytical reagent grade, redistilled at a temperature not higher than 65 °C and a pressure of approximately 500 Pa; its peroxidation is prevented by suitable means, for example distilling in the presence of hydroquinone.

Immediately after redistillation, 0,2 % (m/m) of antioxidant shall be added to inhibit oxidation during the viscosity determination. Antioxidants which have been found satisfactory include:

— 4,4′-thio-bis-(6-*tert*-butyl-3-methyl)phenol;

— bis-(2-hydroxy-3-*tert*-butyl-5-methyl)phenylmethane;

— octadecyl-3-(3′,5′di-*tert*-butyl-4′-hydroxyphenyl) *n*-propionate;

— and tetrakis[methylene 3-(3′,5′di-*tert*-butyl-4′-hydroxyphenyl) *n*-propionate]methane.

Solvent stabilized in this way gives polymer solutions that are resistant to oxidation at 135 °C for several hours.

5 Apparatus

5.1 Ubbelohde viscometer, of which the essential dimensions are shown in figure 1, or another type of viscometer listed in ISO 3105, provided it gives equivalent results to an Ubbelohde viscometer. However, in cases of dispute, an Ubbelohde viscometer shall be used.

The preferred Ubbelohde viscometer design is shown in figure 1, but other Ubbelohde viscometers can be used, provided that their capillary diameter and length and upper-bulb volume are the same as those indicated in figure 1. If a viscometer of a type different from that shown in figure 1 is used, the polymer solution is prepared in a 100 ml flask and the solution is filtered into the viscometer through a dry P 100 sintered-glass funnel. Before the filtration, the funnel is maintained at 150 °C in the oven (5.8).

5.2 Viscometer holder, to hold the viscometer firmly in the correct position in the thermostatic bath.

5.3 Thermostatic bath, maintained at 135 °C ± 0,2 °C, in accordance with ISO 1628-1.

5.4 Thermostatic bath, maintained at 20 °C ± 0,1 °C.

5.5 Temperature-measuring device, in accordance with ISO 1628-1 (scale division 0,1 °C).

5.6 Timer, of any kind provided that it can be read to the nearest 0,1 s or better, and that it operates at a rate which is constant to ± 0,1 % over 15 min.

5.7 Analytical balance, to weigh to an accuracy of 0,1 mg.

5.8 Drying oven, maintained at 150 °C ± 2 °C.

5.9 Glass-stoppered flask, 100 ml.

5.10 Graduated pipette, 50 ml.

6 Test sample

6.1 The test sample shall be in the form of dry powder or very small pieces to facilitate dissolution.

6.2 If the sample is a manufactured article, cut it into very small pieces (for example by cutting and grinding in the presence of solid carbon dioxide).

Since the viscosity number of material from the sample surface could differ from that from the interior, one can

— either measure the viscosity number for the whole article by grinding it, homogenising the ground material, dissolving a portion and taking a test sample from the solution;

— or measure the viscosity numbers for the surface and the interior separately by cutting small pieces from the surface and from the interior, grinding and homogenising them separately and testing a sample taken from each.

7 Procedure

7.1 Cleaning the viscometer

7.1.1 Before its utilization, or when inconsistent results are obtained, and in any case at regular intervals, clean the viscometer as specified in annex A of ISO 1628-1:1984.

7.1.2 Between two successive determinations, clean the viscometer with the solvent being used for the determinations. During the cleaning procedure, maintain the viscometer, as well as the solvent, at 135 °C in thermostatic bath 5.3. After cleaning, remove the viscometer from the bath, cool to room temperature, wash with previously distilled and filtered acetone and dry in a dust-free air stream or under vacuum.

7.2 Measurement of efflux time of solvent

Determine the efflux time of the solvent by the procedure given in 7.5 for the solution. Use the average value of three consecutive readings agreeing to within 0,2 s in the calculation of the viscosity number.

7.3 Choice of polymer solution concentration

The polymer concentration shall be such that the ratio between the solution efflux time t and the solvent efflux time t_0 lies between 1,2 and 2.

Dimensions in millimetres

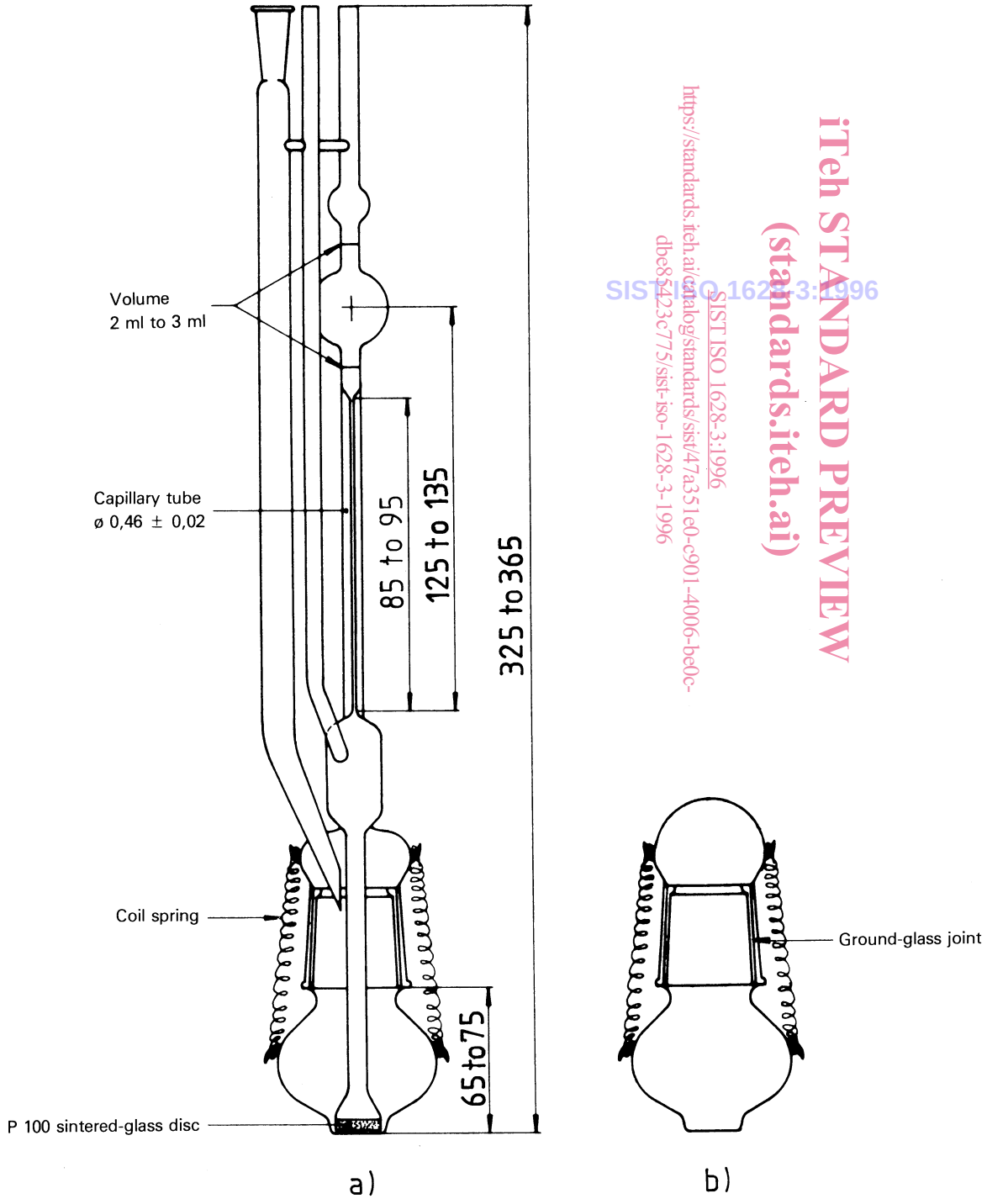

Figure 1 — Ubbelohde viscometer modified for high-temperature tests

7.3.1 If the approximate value of the viscosity number of the polymer is known, choose the concentration from table 1.

Table 1 — Concentration c to be used as a function of the viscosity number to be measured

Viscosity number ml/g	Concentration, c g/ml
40 to < 200	0,005
200 to 1 000	0,001
> 1 000 to 5 000	0,000 2

7.3.2 If the approximate value of the viscosity number of the polymer is not known, test a solution of concentration 0,001 g/ml.

If the viscosity number so obtained is not in the range prescribed in table 1 for that concentration, repeat the test with the concentration in table 1 corresponding to the value of the number obtained.

7.4 Preparation of the solution

7.4.1 Calculate, to the nearest 1 mg, the mass m, in grams, of sample to be dissolved, using the equation:

$$m = c \cdot V \cdot \gamma$$

where

c is the concentration, in grams per millilitre, of the solution at 135 °C;

V is the volume, in millilitres, of solvent used, measured at 20 °C (usually the solution is prepared using 50 ml of solvent);

γ is the coefficient of expansion of the solvent between 20 °C and 135 °C, and is equal to the ratio of the densities at these temperatures, i.e.

$$\gamma = \frac{\rho_{20}}{\rho_{135}} = \frac{0{,}888}{0{,}802} = 1{,}107$$

Experimental evidence has shown that this value is constant even if the density at 20 °C (ρ_{20}) is not exactly 0,888 g/ml.

7.4.2 Weigh the calculated amount of the sample, with an accuracy of 0,2 mg, into the viscometer vessel (for example m will equal 0,055 3 g for a concentration of 0,001 g/ml).

Add the volume V of solvent, transferring it with a graduated pipette (5.10) from a glass-stoppered flask (5.9) maintained at 20 °C ± 0,1 °C in the thermostatic bath (5.4). Close the viscometer vessel [see figure 1 b)] and place it and the viscometer in the oven (5.8) at 150 °C. Keep the vessel in the oven until visual examination shows no undissolved particles, gels or particles of foreign matter, removing the vessel from the oven and examining the solution every 10 min, and stirring the solution carefully so that the polymer particles are not spread over the wall of the vessel.

If complete dissolution has not been achieved in 2 h, the heating may be continued for another 2 h, stirring and examining the vessel every 10 min, but this shall be mentioned in the test report.

7.5 Measurement of efflux of solution

Fit the viscometer vessel on the viscometer with a small amount of grease and hold it firmly in place with the springs as shown in figure 1 a).

Quickly immerse the whole viscometer in the 135 °C thermostatic bath (5.3), fix in a vertical position and allow to stand for 15 min.

By aspiration, bring the liquid level up to approximately 10 mm above the upper graduation mark on the upper bulb of the viscometer.

Allow the solution to drain through the capillary. When the meniscus is at the upper mark on the upper bulb, start the timer (5.6) and determine the time for the solution to drop to the level of the lower mark on the bulb.

Measure the efflux time for the solution several times until three consecutive readings do not differ from their average value by more than 0,2 s.

Note the average of these three values.

8 Expression of results

8.1 Viscosity number, VN

Calculate the viscosity number VN, expressed in millilitres per gram, using the equation

$$\mathrm{VN} = \frac{t - t_0}{t_0 \cdot c}$$

where

t is the average efflux time, in seconds, of the solution;

t_0 is the average efflux time, in seconds, of the pure solvent;

c is the concentration, in grams per millilitre, of the solution at 135 °C, given by

$$c = \frac{m}{V \cdot \gamma + (1{,}107 m / \rho_{20})}$$

m, V, γ and ρ_{20} having the same meaning as in 7.4.1.

The calculated value shall be rounded off to the nearest whole number.

8.2 Limiting viscosity number, $[\eta]$

Calculate the limiting viscosity number $[\eta]$, expressed in millilitres per gram, using the equation

$$[\eta] = \frac{VN}{1 + k \cdot c \cdot VN}$$

where

k is a coefficient depending on polymer concentration and polymer structure;

c and VN have the same meaning as in 8.1.

An approximate value of $[\eta]$ can be calculated using $k = 0{,}27$ but, in the case of precise determinations, k has to be determined for each range of concentrations and for each series of samples.

The calculated value shall be rounded off to the nearest whole number.

8.3 Precision

The precision of this test method is not known because inter-laboratory data are not available. This method may not be suitable for use in specifications or in case of any disputed results as long as these data are not available.

9 Test report

The test report shall include the following particulars:

a) a reference to this part of ISO 1628;

b) all details necessary for the complete identification of the material tested (type, origin, manufacturer's trade mark);

c) any treatment given to the sample before testing (cutting, grinding, etc.);

d) if applicable, the part of a manufactured article tested (whole article, surface or interior);

e) the dissolution time, if this exceeds 2 h;

f) the viscosity number, giving in parentheses the concentration used, for example: VN(0,001) = 380 ml/g;

g) the limiting viscosity number, giving in parentheses the constant k calculated for the same concentration.