
**Plastics — Determination of the viscosity
of polymers in dilute solution using
capillary viscometers —**

**Part 3:
Polyethylenes and polypropylenes**

*Plastiques — Détermination de la viscosité des polymères en solution
diluée à l'aide de viscosimètres à capillaires —*

Partie 3: Polyéthylènes et polypropylènes



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Contents

Page

Foreword	iv
1 Scope	1
2 Normative references	1
3 Principle	1
4 Solvent	2
5 Apparatus	2
6 Test sample	4
7 Procedure	4
7.1 Cleaning the viscometer	4
7.2 Measurement of efflux time of solvent	4
7.3 Choice of polymer solution concentration	4
7.4 Preparation of the solution	5
7.5 Measurement of efflux time of solution	5
8 Expression of results	6
8.1 Reduced viscosity, I	6
8.2 Intrinsic viscosity, $[\eta]$	6
8.3 Precision	7
9 Test report	7

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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 1628-3 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

This third edition cancels and replaces the second edition (ISO 1628-3:2001), which has been technically revised. The main changes relate to the way in which the solution of the polymer under test is prepared and introduced into the viscometer.

ISO 1628 consists of the following parts, under the general title *Plastics — Determination of the viscosity of polymers in dilute solution using capillary viscometers*:

- *Part 1: General principles*
- *Part 2: Poly(vinyl chloride) resins*
- *Part 3: Polyethylenes and polypropylenes*
- *Part 4: Polycarbonate (PC) moulding and extrusion materials*
- *Part 5: Thermoplastic polyester (TP) homopolymers and copolymers*
- *Part 6: Methyl methacrylate polymers*

Plastics — Determination of the viscosity of polymers in dilute solution using capillary viscometers —

Part 3: Polyethylenes and polypropylenes

1 Scope

This part of ISO 1628 defines particular conditions for determining the reduced viscosity (also known as viscosity number) and intrinsic viscosity of polyethylenes and polypropylenes at 135 °C in dilute solution.

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

NOTE Reduced viscosity is also known as the Staudinger function (J_v) and intrinsic viscosity as the Staudinger index (J_G).

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 1628-1:2009, *Plastics — Determination of the viscosity of polymers in dilute solution using capillary viscometers — Part 1: General principles*

ISO 3105:1994, *Glass capillary kinematic viscometers — Specifications 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 reduced viscosity and intrinsic viscosity are calculated from these measurements and from the known concentration of the solution.

Ethylene polymers and isotactic polypropylenes 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 shall be prevented by suitable means, for example distilling in the presence of hydroquinone.

Immediately after redistillation of the decahydronaphthalene, 0,2 % by mass 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;
- tetrakis[methylene 3-(3',5'-di-*tert*-butyl-4'-hydroxyphenyl)-*n*-propionate]methane.

Filter the solvent through a P100 sintered-glass funnel. 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, preferably either type 0B as specified in Table B.4 of ISO 3105:1994 or type 0a as specified in Table B.9 of ISO 3105:1994, having a nominal viscometer constant of 0,005 (mm²/s)/s. These viscometers are shown in Figure 1 and Figure 2 of ISO 1628-1:2009 and the principal parameters are given in Table 1. Other types of viscometer described in ISO 3105:1994 may be used provided they give equivalent results. However, in cases of dispute, an Ubbelohde viscometer shall be used.

Table 1 — Ubbelohde viscometers recommended for the determination of the dilute-solution viscosity of polymers

Type	Nominal viscometer constant (mm ² /s)/s	Inside diameter of tube R mm (±2 %)	Volume of bulb C ml (±5 %)	Inside diameter of tube P mm (±5 %)
0B	0,005	0,46	3,0	6,0
0a	0,005	0,53	5,7	6,0

If the polymer solution needs to be filtered, the modified Ubbelohde viscometer design shown in Figure 1 may be used (the essential dimensions of the modified design are the same as those of the type 0B and type 0a viscometers, but tube L has been modified to include a filter).

- 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, accurate to 0,1 mg.

5.8 Shaker, fitted in a heating bath capable of maintaining a temperature of $150\text{ °C} \pm 2\text{ °C}$, for dissolving the polymer in the solvent. The shaker may be of the rotating, the reciprocating or another suitable type.

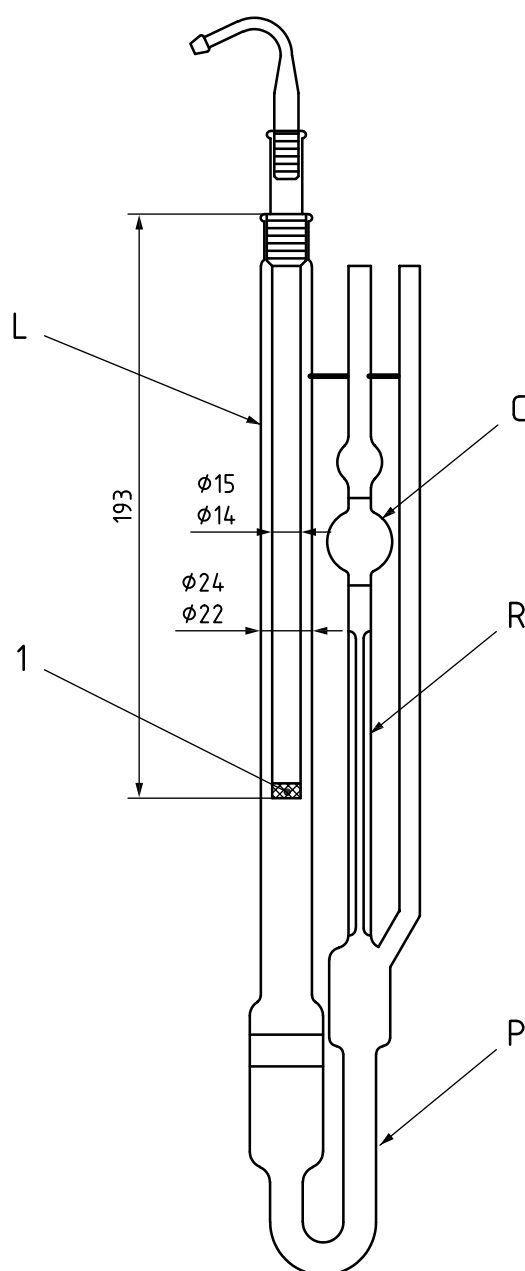
NOTE Certain types of shaker are not suitable for preparation of PE-UHMW solutions.

5.9 Glass-stoppered flask, capacity 100 ml.

5.10 Graduated pipette, capacity 50 ml.

5.11 Filter funnel, with a P100 sintered-glass disc.

Dimensions in millimetres



Key

1 P100 sintered-glass disc

Figure 1 — Ubbelohde viscometer with filter stick

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). If the sample is in the form of granules, reduce the particle size by grinding in the same way.

Since the reduced viscosity of material from the surface of a manufactured article could differ from that from the interior, one can

- either measure the reduced viscosity for the whole article by grinding it, homogenizing the ground material, dissolving a portion and taking a test sample from the solution;
- or measure the reduced viscosities for the surface and the interior separately by cutting small pieces from the surface and from the interior, grinding and homogenizing 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 1628-1:2009.

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. Remove the solvent by suction and dry the viscometer with dust-free and oil-free air or nitrogen.

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

7.3.1 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 2,0.

7.3.2 If the approximate value of the reduced viscosity of the polymer is known, choose the concentration from Table 2.

Table 2 — Concentration c to be used as a function of the reduced viscosity to be measured

Reduced viscosity ml/g	Concentration, c g/ml
≥ 40 but < 200	0,005
200 to 1 000	0,001
$> 1\ 000$ but $\leq 5\ 000$	0,000 2

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

If the reduced viscosity so obtained is not in the range prescribed in Table 2 for that concentration, repeat the test with the concentration in Table 2 corresponding to the value of the viscosity 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:

$$m = cV\gamma$$

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 sample, with an accuracy of 0,2 mg, into a glass-stoppered flask (5.9) (for example m will equal 0,055 3 g for a concentration of 0,001 g/ml). When doing this, be careful to avoid polymer particles spreading over the wall of the flask.

Add the volume V of solvent, transferring it with a graduated pipette (5.10) from another glass-stoppered flask (5.9) maintained at 20 °C \pm 0,1 °C in thermostatic bath 5.4. Place the flask on the shaker (5.8) and shake it in the heating bath at 150 °C for 1 h. If, after 1 h, visual examination reveals gels or particles in suspension, discard the solution and prepare a fresh solution. If no gels or particles in suspension are observed, continue to shake the flask at 150 °C at a slow speed for another 1 h.

If complete dissolution has not been achieved in 2 h, shaking at 150 °C may be continued for another 2 h, but this shall be mentioned in the test report.

7.5 Measurement of efflux time of solution

Immerse the whole viscometer in a vertical position in the 135 °C thermostatic bath (5.3) and fix it in place. If the polymer solution is going to need to be filtered before the determination, insert the filter stick in the viscometer (see Figure 1). Allow the viscometer (and filter stick, if used) to reach the temperature of the bath, and keep the viscometer at this temperature during the whole determination. Quickly (within a period of 20 s), introduce the required volume of polymer solution (at 150 °C) into the viscometer. If a filter stick has been used, remove it. Stopper tube L of the viscometer. Keep the viscometer in the thermostatic bath at 135 °C for 15 min.

Whether the solution was filtered or not shall be mentioned in the test report.

NOTE 1 Filtration will reduce the viscosity measured for PE-UHMW samples.

NOTE 2 Filling the viscometer is made easier if a glass funnel warmed with a heating mantle is used. This helps to prevent polymer from precipitating out of the solution.

Using suction, bring the liquid level up to approximately 10 mm above the upper graduation mark (between the two bulbs above the capillary of the viscometer).

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

Calculate the reduced viscosity, I , expressed in millilitres per gram, using the equation:

$$I = \frac{t - t_0}{t_0 c}$$

where

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

t_0 is the average efflux time, in seconds, of the solvent used (including the antioxidant added);

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

$$c = \frac{m}{V\gamma}$$

m , V and γ having the same meaning as in 7.4.1.

Round the calculated value to the nearest whole number.

8.2 Intrinsic viscosity, $[\eta]$

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

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

where

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

c and I 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.

Round the calculated value to the nearest whole number.

8.3 Precision

The precision of this test method is not known because interlaboratory data are not available. This method may not be suitable for use in specifications or in the case of 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 exceeded 2 h;
- f) whether the solution was filtered or not;
- g) the reduced viscosity, giving in parentheses the concentration used, for example: $\eta (0,001) = 380 \text{ ml/g}$;
- h) the intrinsic viscosity, giving in parentheses the constant k calculated for the same concentration;
- i) the date of the test.

