INTERNATIONAL STANDARD 1191

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Plastics — Polyethylenes and polypropylenes in dilute solution — Determination of viscosity number and of limiting viscosity number

Matières plastiques — Polyéthylènes et polypropylènes en solution diluée — Détermination de l'indice de viscosité et de l'indice limite de viscosité

First edition — 1975-06-15

UDC 678.742 : 532.13

Ref. No. ISO 1191-1975 (E)

Descriptors : plastics, thermoplastic resins, polyethylene, polypropylene, solutions, tests, physical tests, viscosity index, viscometers.

Price based on 4 pages

FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is caried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 61 has reviewed ISO Recommendation R 1191 and found it technically suitable for transformation. International Standard ISO 1191 therefore replaces ISO Recommendation R 1191-1970 to which it is technically identical.

ISO Recommendation R 1191 was approved by the Member Bodies of the following countries :

Australia	Germany	Romania
Austria	Hungary	South Africa, Rep. of
Belgium	India	Spain
Brazil	Iran	Sweden
Canada	Israel	Switzerland
Chile	Italy	United Kingdom
Colombia	Japan	U.S.A.
Czechoslovakia	Korea, Rep. of	U.S.S.R.
Egypt, Arab Rep. of	Netherlands	Yugoslavia
Finland	New Zealand	
France	Poland	

No Member Body expressed disapproval of the Recommendation.

The Member Bodies of the following countries disapproved the transformation of ISO/R 1191 into an International Standard :

Canada
United Kingdom

Printed in Switzerland

Plastics — Polyethylenes and polypropylenes in dilute solution — Determination of viscosity number and of limiting viscosity number

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method 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 viscosity number determined by this method may therefore be unreliable if the sample contains fillers or other additives.

NOTE — For the definition of viscosity number, and for other terms, definitions and formulae, see ISO/R 1628, *Plastics — Directives for the standardization of methods for the determination of the dilute solution viscosity of polymers.*

2 PRINCIPLE

Measurement of the times of flow of the solvent and of a solution of polymer at a specified concentration at 135 °C. Calculation of the viscosity number and limiting viscosity number from these measurements and from the known concentration of the solution.

NOTE — Ethylene and propylene isotactic polymers are not soluble at room temperature in any known solvents. Precautions are taken during the test to avoid any precipitation of polymer which would lead to incorrect solution concentration.

3 SOLVENT

Decahydronaphthalene, analytical reagent grade, redistilled at a temperature not higher than 65 °C and a pressure of approximately 5 mbar, its peroxidation being prevented by suitable means, for example distilling in presence of hydroquinone.

Immediately after redistillation, 0,2 % (*m/m*) of antioxidant may be added to inhibit oxidation during the viscosity determination. Phenyl-β-naphthylamine and tertiary-thio-bismethyl-butyl-phenol have been found satisfactory. Solvent stabilized in this way gives solutions of polymers which are resistant to oxidation at 135 °C for several hours.

4 APPARATUS

4.1 Volumetric flask, 100 ml.

4.2 Pipettes or burettes, 50 ml.

4.3 Thermostatic bath maintained at 135 ± 0,2 °C.

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

4.5 Pycnometer.

4.6 Ubbelohde viscometer, of which the essential dimensions are as shown in the figure. The viscometer design shown in this figure has been found satisfactory, but other Ubbelohde viscometers can be used, subject to the condition that their capillary diameter and length and upper bulb volume are those indicated in the figure.

4.7 Oven, maintained at 150 °C.

4.8 Analytical balance, to weigh to an accuracy of 0,000 1 g.

4.9 Stop-watch, reading to the nearest 0,1 s.

5 TEST SAMPLE

5.1 The sample must be in the form of dry powder or very small chips to facilitate solution.

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

NOTE — In this case, since the viscosity number of the sample surface could differ from that of the inner part, one can

— either measure *the global viscosity number*, by grinding the *whole* of the manufactured article, mixing the chips, dissolving them and taking a test sample from the solution;

— or measure *both the surface and the inner part viscosity numbers*, by cutting small pieces from the surface and from the inner part and testing a homogeneous sample taken from each series of chips.

6 PROCEDURE

6.1 Cleaning of viscometer

Clean the viscometer (4.6) before it is used, after discordant readings and at intervals during regular use. Use a mixture of equal volumes of concentrated sulphuric acid and a saturated solution of potassium dichromate in water.

Rinse it with water followed by acetone and dry it by drawing through it a stream of air free from dust. Between successive satisfactory determinations, wash the viscometer with acetone and dry as described.

6.2 Measurement of efflux time of solvent

Determine the efflux time of the solvent according to the procedure given in 6.5 for the solution.

Use the average value of three consecutive readings agreeing to within 0,2 s in the calculation of the viscosity number.

6.3 Choice of concentration for polymer solution

The solution concentration shall be such that the viscosity increment,

$$\frac{\eta}{\eta_0} - 1 = \frac{t}{t_0} - 1$$

is larger than 0,2 and smaller than 1.

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

TABLE – **Concentrations c to be used, as a function of the viscosity number that will be measured**

Viscosity number ml/g	Concentration c g/ml
40 to 200 (exclusive)	0,005
200 to (and including) 1 000	0,001
1 000 (exclusive) to 5 000	0,000 2

6.3.2 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 by the above table for that concentration, carry out the definitive test by choosing the right concentration according to the value of the viscosity number so obtained.

6.4 Preparation of the solution

6.4.1 Calculate, to the nearest 1 mg, the mass m of the sample to be dissolved by the equation :

$$m = cVe$$

where

m is the mass, in grams, of the sample;

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

V is the solvent volume, in millilitres, at 20 °C (usually the solution is prepared by using 50 ml of solvent and $V = 50$);

e is the expansion coefficient of the solvent from 20 to 135 °C, equal to the ratio of the densities at these temperatures :

$$e = \frac{\rho_{20}}{\rho_{135}} = \frac{0{,}888}{0{,}802} = 1{,}107^*$$

6.4.2 Weigh the calculated mass m of the sample, with an accuracy of 0,2 mg, into the viscometer vessel (for example $m = 0{,}055\ 3$ g for the concentration 0,001 g/ml).

Add the volume V of solvent, taking it with the pipette from the volumetric flask (4.1) maintained at 20 ± 0,1 °C in the thermostatic bath (4.4). Close the viscometer vessel (see figure, diagram *b*)) and place it in the oven (4.7) at 150 °C.

Keep it under continuous shaking for 2 h, then examine the solution. If undissolved particles or gels are present, continue the shaking for 2 h more (and mention this in the test report).

6.5 Measurement of efflux time of solution

Attach the viscometer vessel to the viscometer with a little grease and hold it firmly in place with the springs as shown in the figure, diagram *a*). Quickly immerse the whole viscometer in the constant temperature bath at 135 ± 0,2 °C (4.3), 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. When the meniscus is at the upper mark on the upper bulb, start the timer and determine the interval for the solution to drain to 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.

7 EXPRESSION OF RESULTS

7.1 Viscosity number (V.N.)

The viscosity number, in millilitres per gram, is calculated as follows :

$$\text{V.N.} = \frac{t - t_0}{t_0\, c}$$

where

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

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

* According to experimental evidence this value is constant even if the density at 20 °C (ρ_{20}) is not exactly 0,888 g/ml.

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

The calculated value shall be rounded off to the nearest unit.

7.2 Limiting viscosity number (L.V.N. or $[\eta]$)

The limiting viscosity number (see ISO/R 1628), in millilitres per gram, is calculated as follows :

$$\text{L.V.N.} = [\eta] = \frac{\text{V.N.}}{1 + 0{,}27\left(\frac{t - t_0}{t_0}\right)}$$

where t and t_0 have the same meaning as in 7.1.

The calculated value shall be rounded off to the nearest unit.

8 TEST REPORT

The test report shall include the following particulars :

a) complete identification of the material tested (type, source, trade-name, etc.);

b) any treatment given to the sample before testing (cutting, grinding and, for a finished product, sampling from the surface, or from the inner part, etc.);

c) if necessary, which part of the manufactured article has been tested (surface, inner part, etc.) and which viscosity number has been calculated (surface number, inner number, global number, etc.);

d) the heating time of the solution, if this was 4 h;

e) the number itself, i.e. :

the viscosity number, giving between parentheses the concentration used, for example :

V.N. (0,001) = 380 ml/g

and/or

the limiting viscosity number, for example :

L.V.N. = $[\eta]$ = 345 ml/g

Dimensions in millimetres

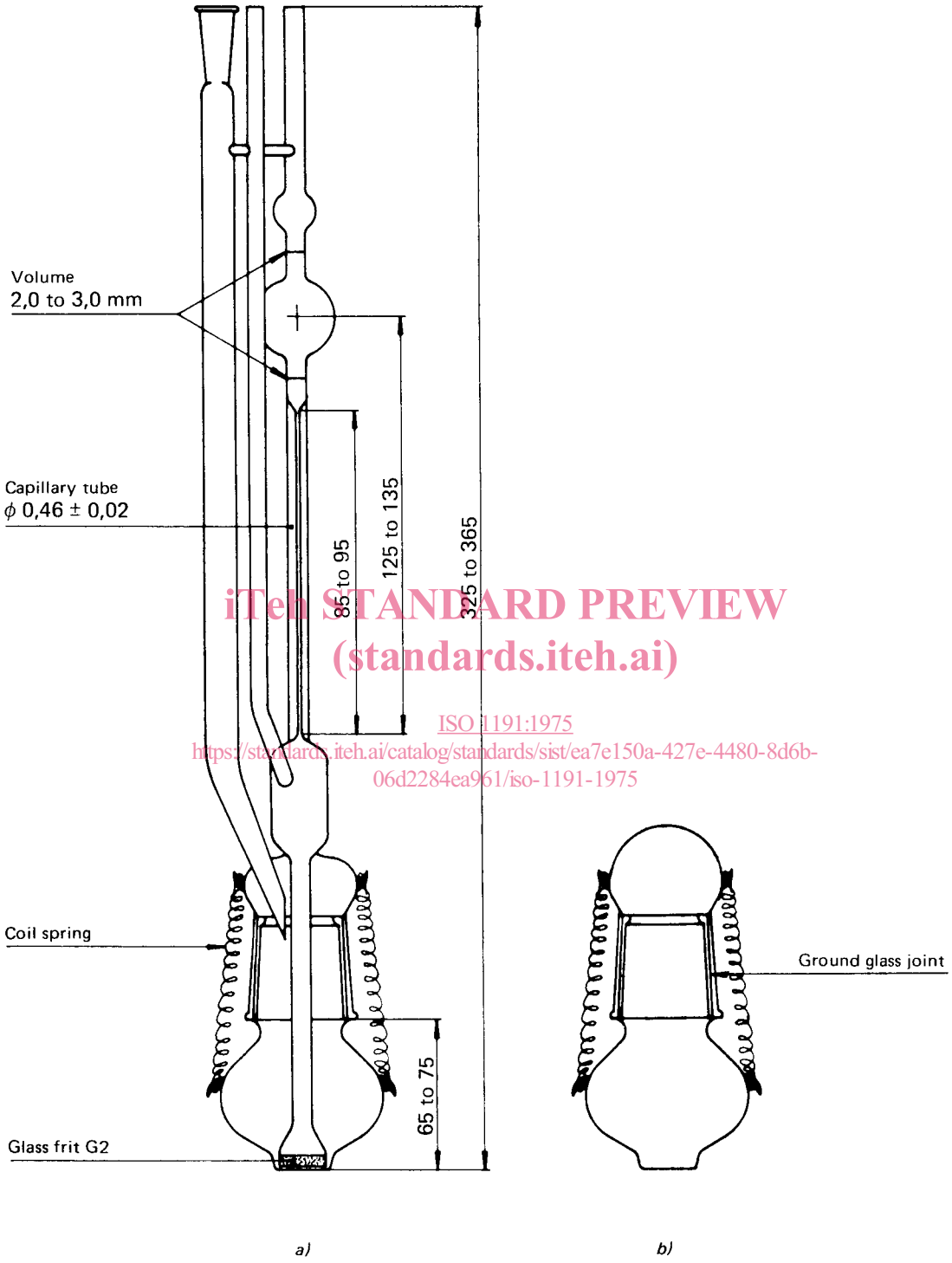

FIGURE – **Ubbelohde viscometer modified for high temperature tests**