INTERNATIONAL STANDARD

ISO 1157

Second edition
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Plastics — Cellulose acetate in dilute solution — Determination of viscosity number and viscosity ratio

Plastiques — Acétate de cellulose en solution diluée — Détermination de l'indice de viscosité et du rapport de viscosité

Reference number
ISO 1157:1990(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 1157 was prepared by Technical Committee ISO/TC 61, *Plastics*.

This second edition cancels and replaces the first edition (ISO 1157:1975), of which it constitutes a minor revision.

International Organization for Standardization
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Plastics — Cellulose acetate in dilute solution — Determination of viscosity number and viscosity ratio

WARNING — The use of this International Standard may involve hazardous materials, operations and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a method for determining the viscosity number and viscosity ratio of cellulose acetate in dilute solution in a mixture of dichloromethane and methanol.

The method applies to cellulose acetate with not less than 50 % acetic acid yield.

2 Normative reference

The following standard contains provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the edition indicated was valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the standard 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.*

3 Definitions

For the definition of viscosity number, and for other terms, definitions and formulae, see ISO 1628-1.

4 Principle

The times of flow of a solvent and of a solution of cellulose acetate at a concentration of 5 mg/ml in that solvent are measured at 25 °C by conventional methods. The viscosity number and ratio are calculated from these measurements and from the known concentration of the solution. Density difference and kinetic energy corrections are small in this method and are not applied.

5 Solvents

During the determination, use only solvents of recognized analytical grade.

5.1 Dichloromethane, d_{20}^{20} 1,321 to 1,331, not less than 95 % by volume distilling between 39 °C and 40,5 °C at a pressure of 1 013 mbar (760 mmHg).

SAFETY PRECAUTIONS — Dichloromethane is harmful by inhalation. Avoid contact with the skin.

5.2 Methanol, d_{20}^{20} 0,792 to 0,795, distillation range 64,5 °C to 65,5 °C at a pressure of 1 013 mbar (760 mmHg).

SAFETY PRECAUTIONS — Methanol is highly flammable and toxic by inhalation or if swallowed. Keep the container tightly closed and away from sources of ignition. Do not smoke. Avoid contact with the skin.

6 Apparatus

Ordinary laboratory apparatus, plus the following:

6.1 Volumetric flask, capacity 100 ml, with ground-glass stopper.

6.2 Thermostatic bath, capable of being maintained at 25 °C ± 0,05 °C.

6.3 Viscometer, suspended-level Ubbelohde type, with essential dimensions as shown in figure 1, or any other viscometer which can be shown to give the same results.

6.4 Desiccator, with a suitable drying agent such as anhydrous calcium chloride.

6.5 Analytical balance, accurate to 0,1 mg.

6.6 Stopwatch, reading to the nearest 0,1 s.

7 Procedure

7.1 Clean the viscometer before it is used, after discordant readings, and at intervals during regular use. Use a mixture of equal volumes of concentrated sulfuric acid and a saturated solution of potassium dichromate in water, subsequently rinsing it with water followed by acetone, and drying 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.

7.2 Dry an adequate quantity of cellulose acetate sample in a thermostatic oven at 105 °C ± 2 °C for 3 h and cool it in the desiccator (6.4).

7.3 Prepare the solvent mixture by adding 90 parts by volume of dichloromethane (5.1) to 10 parts by volume of methanol (5.2), both solvents being maintained at 25 °C ± 0,05 °C.

7.4 Weigh out 0,5 g ± 0,5 mg of dry cellulose acetate to the nearest 0,1 mg and transfer it quantitatively to the volumetric flask (6.1). Add approximately 60 ml of solvent mixture, taking care to avoid the formation of lumps, and insert the stopper. Shake the mixture gently for 5 min and place the flask for 55 min in the thermostatic bath (6.2) at 25 °C ± 0,05 °C. Repeat this cycle of agitation and standing until the cellulose acetate has dissolved completely. Maintain the solution at 25 °C ± 0,05 °C and add solvent mixture at the same temperature until the volume of solution is exactly 100 ml. Stopper the volumetric flask, mix by inverting it several times and allow to stand for 2 h.

7.5 The time of flow of the solution and of the solvent mixture is determined in the same viscometer.

7.5.1 If an Ubbelohde viscometer is used, proceed as follows:

Pipette solution from the volumetric flask (6.1) into tube L of the viscometer (see figure 1), immersed in the thermostatic bath (6.2) to a depth of approximately 20 mm above the upper graduation mark and supported so that tube N is vertical.

The volume of liquid in the viscometer shall be such that the surface after draining lies between the two filling marks.

After not less than 10 min, blow the liquid with dust-free air, or draw it by suction, into the upper bulb until it reaches approximately the centre of that bulb. Place a finger over tube N until the liquid flows away from the lower end of the capillary. Then remove the finger and measure the time interval for passage of the meniscus between the two graduation marks (E and F).

Blow or draw the liquid into the upper bulb and again measure the time of flow.

7.5.2 The above procedure shall be suitably modified if a viscometer other than the Ubbelohde type is used.

7.5.3 The time of flow of the solution shall be taken as the mean of two determinations which shall not differ by more than 0,4 % of the smaller result. The mean time of flow of the solvent mixture shall be determined in the same way.

The test shall be repeated with a fresh solution if two determinations, out of not more than four, differing by less than 0,4 % of the smaller result are not obtained.

If the viscosity number and ratio are to be related to the molecular chain length, valid comparisons can be made only between samples of similar acetic acid yield.

When the sample contains plasticizers, additives, fillers, etc., which influence the viscosity, they shall be eliminated by mutually agreed procedures.

8 Expression of results

8.1 The result should preferably be expressed as a viscosity number, which is given in millilitres per gram by the formula

$$\frac{t_s - t_0}{t_0 c}$$

where

t_s is the time of flow of the solution, in seconds;

t_0 is the time of flow of the solvent mixture, in seconds;

c is the concentration, in grams of cellulose acetate per millilitre, of the solution.

8.2 The viscosity ratio is given by the formula

$$\frac{t_s}{t_0}$$

where t_s and t_0 are as defined above.

9 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 disputed results as long as these data are not available.

10 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

b) all details necessary for the complete identification of the sample tested;

c) treatment of the sample before the test, if any;

d) the viscosity number, reported to the nearest 0,1 ml/g, and/or the viscosity ratio;

e) the test temperature;

f) any departure from the procedure specified in this International Standard, and any incidents which may have affected the results.

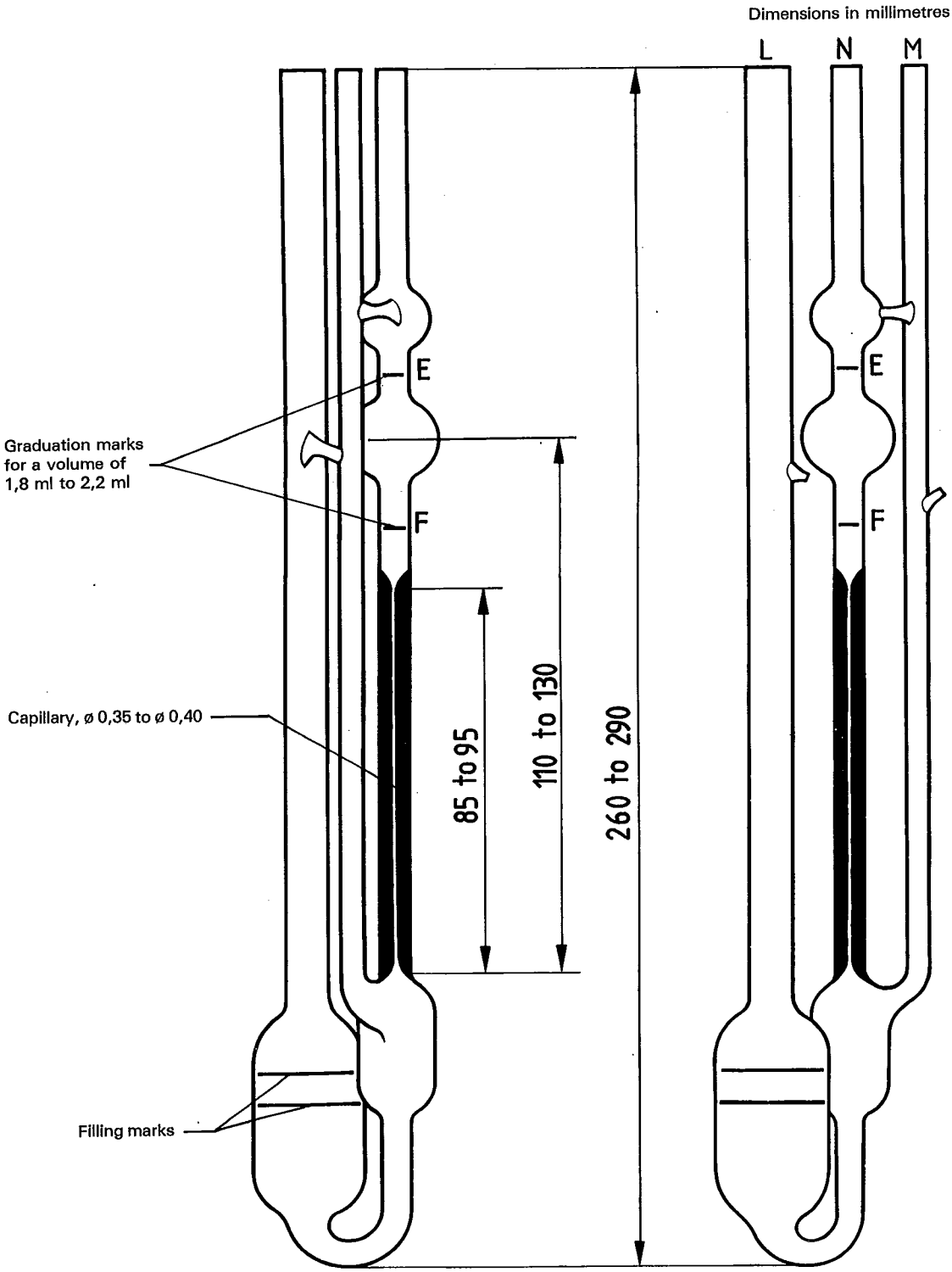

Figure 1 — Ubbelohde viscometer

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