
INTERNATIONAL STANDARD



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

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

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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 carried 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 1157 and found it technically suitable for transformation. International Standard ISO 1157 therefore replaces ISO Recommendation R 1157-1970 to which it is technically identical.

ISO Recommendation R 1157 was approved by the Member Bodies of the following countries :

Australia	Germany	Poland
Austria	Greece	Romania
Belgium	Hungary	Spain
Canada	India	Sweden
Chile	Ireland	Switzerland
Colombia	Israel	Turkey
Czechoslovakia	Italy	United Kingdom
Egypt, Arab Rep. of	Japan	U.S.A.
Finland	Netherlands	U.S.S.R.
France	New Zealand	

No Member Body expressed disapproval of the Recommendation.

The Member Body of the following country disapproved the transformation of ISO/R 1157 into an International Standard :

Canada

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

1 SCOPE AND FIELD OF APPLICATION

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

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

The times of flow of the solvent and of a solution of cellulose acetate at a concentration of 0,005 g/ml 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.

3 SOLVENTS

3.1 Dichloromethane, analytical reagent grade, with relative density (20 °C/20 °C) of 1,321 to 1,331. Not less than 95 % by volume is distilled between 39 and 40,5 °C at a pressure of 1 013 mbar (760 mmHg).

3.2 Methanol, analytical reagent grade, with relative density (20 °C/20 °C) of 0,792 to 0,795 and distillation range of 64,5 to 65,5 °C at a pressure of 1 013 mbar (760 mmHg).

4 APPARATUS

4.1 Volumetric flask, 100 ml, with ground glass stopper.

4.2 Thermostatic bath, maintained at $25 \pm 0,05$ °C.

4.3 Viscometer, suspended level Ubbelohde type, of which the essential dimensions are shown in the figure, or any other viscometer which can be shown to give the same results.

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

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

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

5 PROCEDURE

5.1 Clean the viscometer before it is used, after discordant readings, and at intervals during regular use. Use a mixture of equal volumes of concentrated 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.

5.2 Dry an adequate quantity of cellulose acetate sample in a thermostatic oven at 105 ± 2 °C for 3 h and cool it in the desiccator (4.4).

5.3 Prepare the solvent by adding 90 parts by volume of dichloromethane (3.1) to 10 parts by volume of methanol (3.2), both solvents being maintained at $25 \pm 0,05$ °C.

5.4 Weigh $0,5 \pm 0,000 5$ g of dry cellulose acetate to the nearest 0,000 1 g and transfer quantitatively to the volumetric flask (4.1). Add approximately 60 ml of solvent, 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 (4.2) at $25 \pm 0,05$ °C. Repeat this cycle of agitation and standing until the cellulose acetate has dissolved completely. Maintain the solution at $25 \pm 0,05$ °C and add solvent at the same temperature until the volume of solution is exactly 100 ml. Stopper the volumetric flask, stir by inverting it several times and allow to stand for 2 h.

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

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

Pipette the liquid from the volumetric flask (4.1) into tube 1 of the viscometer (see figure), immersed in the thermostatic bath (4.2) to a depth of approximately 20 mm above the upper graduation mark and supported so that tube 2 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 2 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.

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

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

5.5.3 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 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.

NOTES

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

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

6 EXPRESSION OF RESULTS

6.1 The result should preferably be expressed as 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, in seconds;

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

6.2 The viscosity ratio is given by the formula

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

7 TEST REPORT

The report shall include the following particulars :

- a) complete identification of the sample tested;
- b) treatments applied to the sample before testing, if any;
- c) viscosity number, reported to the nearest 0,1 and/or viscosity ratio;
- d) all test conditions not specified in this International Standard and which may have affected the results;
- e) the test temperature.

Dimensions in millimetres

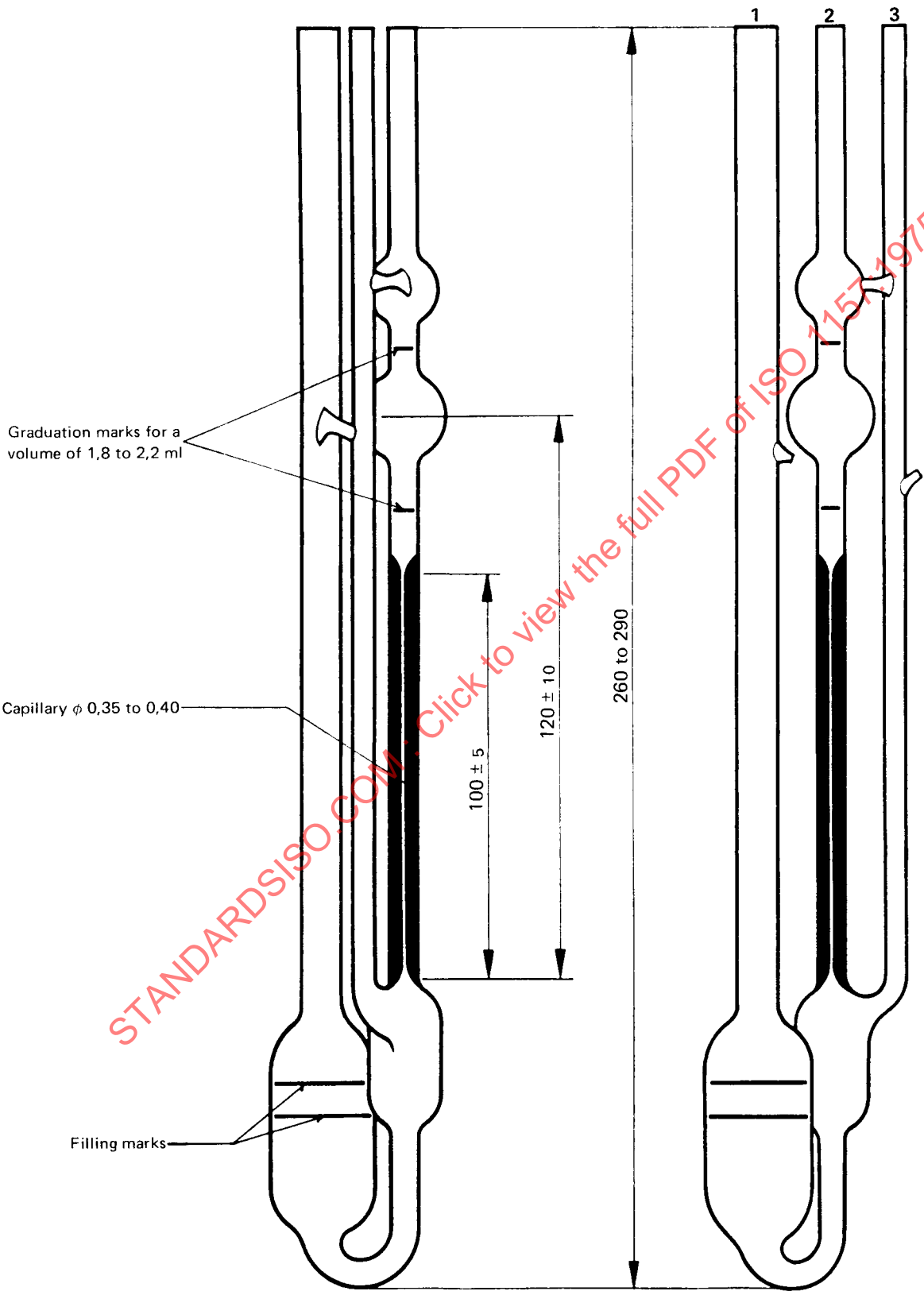


FIGURE — Ubbelohde viscometer

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