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# ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

# ISO RECOMMENDATION

# R 1157

PLASTICS

## DETERMINATION OF VISCOSITY NUMBER AND VISCOSITY RATIO OF CELLULOSE ACETATE IN DILUTE SOLUTION

1st EDITION

January 1970





For each individual country the only valid standard is the national standard of that country.

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## BRIEF HISTORY

The ISO Recommendation R 1157, *Plastics – Determination of viscosity number and viscosity ratio of cellulose acetate in dilute solution,* was drawn up by Technical Committee ISO/TC 61, *Plastics,* the Secretariat of which is held by the American National Standards Institute (ANSI).

Work on this question led to the adoption of a Draft ISO Recommendation.

In July 1965, this Draft ISO Recommendation (No. 825) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

No Member Body opposed the approval of the Draft.

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in January 1970, to accept it as an ISO RECOMMENDATION.

ISO Recommendation | R 1157 | January 1970

PLASTICS

# DETERMINATION OF VISCOSITY NUMBER AND VISCOSITY RATIO OF CELLULOSE ACETATE IN DILUTE SOLUTION



## 1. SCOPE AND FIELD OF APPLICATION

This ISO Recommendation describes 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 Recommendation R . . .*, *Plastics – Directives for the standardization of methods for the determination of the dilute solution viscosity of polymers.*

## 2. PRINCIPLE OF THE METHOD

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 1013 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 1013 mbar (760 mmHg).

## 4. APPARATUS

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

4.2 *Thermostatic bath,* maintained at 25 ± 0.05 °C.

4.3 *Viscometer,* suspended level Ubbelohde type, of which the essential dimensions are shown in the Figure on page 6, 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 0.0001 g.

4.6 *Stop-watch,* reading to the nearest 0.1 s.

* At present, Draft ISO Recommendation No. 1628.

## 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 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 hours 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 ± 0.05 °C.

5.4 Weigh 0.5 ± 0.0005 g of dry cellulose acetate to the nearest 0.0001 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 minutes and place the flask for 55 minutes in the thermostatic bath (4.2) at 25 ± 0.05 °C. Repeat this cycle of agitation and standing until the cellulose acetate has dissolved completely. Maintain the solution at 25 ± 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 hours.

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, page 6), 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 should be such that the surface after draining lies between the two filling marks.

After not less than 10 minutes, 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 should be suitably modified if a viscometer other than the Ubbelohde type is used.

5.5.3 The time of flow of the solution should be taken as the mean of two determinations which should not differ by more than 0.4 % of the smaller result. The mean time of flow of the solvent should be determined in the same way.

The test should 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 gramme by the following formula :

$$\frac{t_s - t_o}{t_o\, C}$$

where

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

$t_o$ is the time of flow of the solvent, in seconds;

$C$ is the concentration in grammes of cellulose acetate per millilitre of solution.

6.2 The viscosity ratio is given by the following formula :

$$\frac{t_s}{t_o}$$

where

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

$t_o$ is the time of flow of the solvent, in seconds.

## 7. TEST REPORT

The report should include the following information :

(*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 ISO Recommendation and which may have affected the results;

(*e*) the test temperature.

*Dimensions in millimetres*

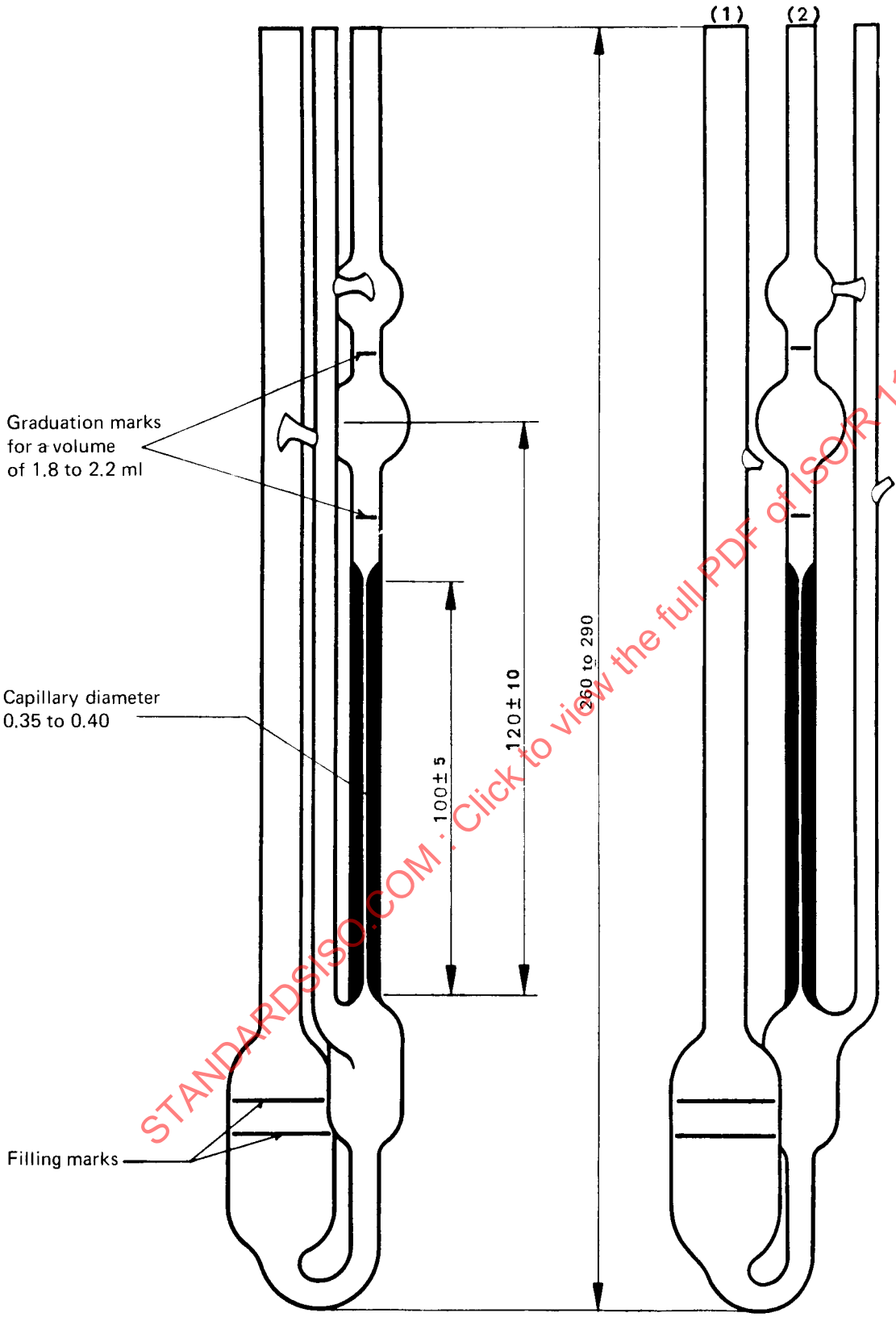


FIGURE – **Ubbelohde viscometer**