

INTERNATIONAL STANDARD

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Second edition
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Plastics — Cellulose acetate — Determination of viscosity loss on moulding

*Plastiques — Acétate de cellulose — Détermination de la perte de
viscosité au moulage*



Reference number
ISO 1599:1990(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 1599 was prepared by Technical Committee ISO/TC 61, *Plastics*.

This second edition cancels and replaces the first edition (ISO 1599:1975), of which it constitutes a minor revision.

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Plastics — Cellulose acetate — Determination of viscosity loss on moulding

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 the determination of the reduction in viscosity which occurs when cellulose acetate is moulded. The viscosity loss on moulding is related to the depolymerization of cellulose acetate, which generally increases brittleness in the moulded product.

This method is suitable for cellulose acetate which does not contain additives, fillers, etc., which may interfere with the determination of viscosity.

It is suitable for cellulose acetate having an acetic acid yield above 50 %.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were 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 editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 565:1990, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*.

ISO 585:1990¹⁾, *Plastics — Unplasticized cellulose acetate — Determination of moisture content*.

ISO 1157:1990, *Plastics — Cellulose acetate in dilute solution — Determination of viscosity number and viscosity ratio*.

3 Principle

Cellulose acetate plasticized with dimethyl phthalate is moulded under specified conditions of temperature, pressure and time. After cooling, the moulding is ground up. The viscosity ratio of the ground material from the moulding and also that of the original cellulose acetate are determined in accordance with ISO 1157. The percentage viscosity loss is calculated from the values of viscosity before and after moulding.

NOTE 1 Since the moulded cellulose acetate contains dimethyl phthalate, this is also present in the solution for viscosity measurement; the solution of original cellulose acetate does not contain dimethyl phthalate. However, the concentration of dimethyl phthalate in the solution for viscosity measurement is too small to have any significant effect on viscosity.

4 Reagents

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

4.1 Dimethyl phthalate, analytical grade, d_{20}^{20} 1,191 to 1,195, purity more than 99 % (m/m).

4.2 Solvents for viscosity ratio determination, as specified in ISO 1157.

1) To be published.

4.3 Propan-2-ol.

SAFETY PRECAUTIONS — Propan-2-ol is highly flammable. Keep the container tightly closed and away from sources of ignition. Do not smoke.

5 Apparatus

Ordinary laboratory apparatus, plus the following:

5.1 Hydraulic press, capable of exerting a pressure of at least 8 MN/m² on the moulding surface, with means of heating to 200 °C and with water cooling.

5.2 Mould (see figure 1 for a suitable type), with polished surfaces which are substantially flat and parallel, to produce mouldings of thickness within the range 1,5 mm to 5 mm. A particular thickness within this range may be specified.

5.3 Apparatus for viscosity ratio determination, as specified in ISO 1157.

5.4 Stainless-steel grinder, electrically operated.

5.5 Oven, capable of being thermostatically maintained at 60 °C ± 2 °C or 70 °C ± 2 °C.

6 Test sample

6.1 If proceeding in accordance with 7.1.1 (first method of incorporating the plasticizer), the sample of cellulose acetate shall be in the form of powder passing entirely through a sieve of 710 µm mesh size (as defined in ISO 565); it shall be ground if necessary, avoiding excessive heating of the sample.

If proceeding in accordance with 7.1.2 (second method of incorporating the plasticizer), the cellulose acetate need not be ground.

6.2 Determine the moisture content of the sample in accordance with ISO 585.

7 Procedure

7.1 Either of the methods described below may be used for the incorporation of plasticizer.

7.1.1 First method

Weigh into a glass bottle, to within ± 0,5 g, the quantity of the sample corresponding to 100 g of dry cellulose acetate. Into another glass bottle, weigh 45 g of dimethyl phthalate (4.1), to within ± 0,5 g. Slowly add the dimethyl phthalate to the cellulose acetate with constant stirring and continue to stir for

at least 5 min after all the dimethyl phthalate has been added. Proceed in accordance with 7.2 to 7.8.

7.1.2 Second method

Place 200 g, weighed to the nearest 1 g, of the cellulose acetate in a 2 litre vessel. The moisture content of the sample shall be less than 0,5 %; if not, there is a risk of bubbles forming in the moulding. Add about one-half of the following mixture:

— dimethyl phthalate (4.1):
75 ml ± 0,5 ml (90 g ± 0,6 g);

— propan-2-ol (4.3): 400 ml ± 0,5 ml.

Homogenize by mixing briskly with a glass stirrer. Pour in the rest of the solvent/plasticizer mixture, and, after having mixed again and stoppered the vessel, place it immediately on a roller mixer operating between 50 rpm and 70 rpm. After 2 h of mixing, tap the vessel with the palm of the hand in order to dislodge any powder which may have become stuck to the sides. Place on the roller mixer again and mix for a further 4 h.

Pour the product into a porcelain dish, cover with a sheet of filter paper and let it stand in the open, at room temperature, overnight. Then place in an oven, thermostatically maintained at 60 °C ± 2 °C, and leave for 3 h to eliminate part of the solvent. At the end of this time, place the product in the original clean vessel again. Homogenize it for one or two minutes, by rapid mixing with a mixing rod equipped with a blade turning at 10 000 rpm. (This operation is to destroy any agglomerates that may have been produced during the stoving.) Stopper the vessel, then let it stand again at room temperature for about 20 h. Proceed in accordance with 7.2 to 7.8.

7.2 Heat the mixed material for 2 h at 70 °C ± 2 °C to remove moisture and complete the absorption of plasticizer.

7.3 Place a suitable quantity of the heated mixture in the mould (5.2), pre-heated to a temperature of 200 °C ± 2 °C. Apply contact pressure for 2 min, then full pressure (at least 8 MN/m² at the moulding surface) for a further 10 min ± 0,5 min for a moulding 1,5 mm thick. This time shall be increased by 0,5 min for each 0,5 mm above 1,5 mm thickness.

7.4 Release the pressure and start cooling immediately, continuing the cooling until the moulding is rigid enough to be ejected without being deformed. The rate of cooling shall be such that the mould temperature 2 min after the start of cooling is at least 30 °C below the moulding temperature.

7.5 Grind the moulded cellulose acetate to a size less than 0,9 mm, avoiding excessive heating, and dry it at 70 °C ± 2 °C for 2 h.

7.6 Prepare a solution of the ground material in dichloromethane/methanol mixture as specified in ISO 1157, but containing 0,725 g of material (i.e. 0,500 g of cellulose acetate) in 100 ml of solution, and determine the viscosity ratio in accordance with ISO 1157.

7.7 Determine the viscosity ratio of a 5 g/l solution of the original cellulose acetate in accordance with ISO 1157.

7.8 Make two complete determinations in accordance with 7.1 to 7.7. If the difference between the determinations is more than 10 % of their mean value, two further determinations shall be made.

8 Expression of results

8.1 The viscosity loss on moulding, expressed as a percentage of the viscosity ratio of the original cellulose acetate, is calculated from the formula

$$100 \times \frac{\eta_{r,a} - \eta_{r,b}}{\eta_{r,a}}$$

where

$\eta_{r,a}$ is the viscosity ratio of the original cellulose acetate;

$\eta_{r,b}$ is the viscosity ratio of the cellulose acetate after moulding.

NOTE 2 If the same viscometer is used in the two determinations, it is sufficient to determine the flow time of each solution. In that case, the viscosity loss on moulding is calculated from the formula

$$100 \times \frac{t_a - t_b}{t_a}$$

where

t_a is the flow time of the original cellulose acetate solution, in seconds;

t_b is the flow time of the solution of moulded cellulose acetate, in seconds.

8.2 The result is the mean of two valid determinations (see 7.8).

9 Precision

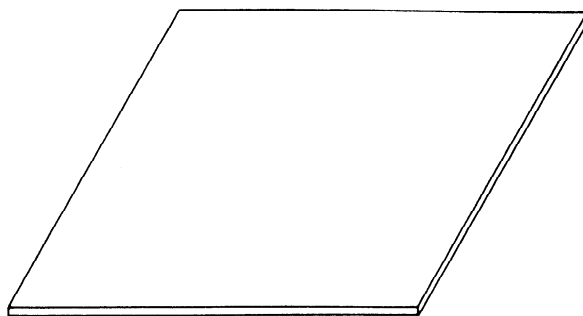
The precision of this test method is not known because inter-laboratory data are not available. This method may not be suitable for use in specifications or in case of disputed results as long as these data are not available.

10 Test report

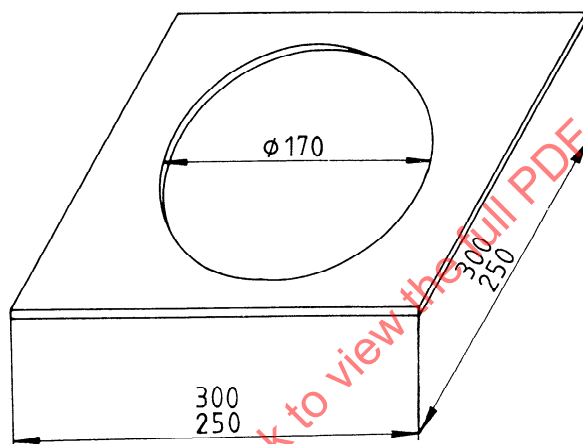
The test report shall include the following particulars:

- a reference to this International Standard;
- all details necessary for the complete identification of the product tested, including type, manufacturer's code number, source, trade name, etc.;
- the viscosity loss on moulding (see 8.2);
- the date of the test.

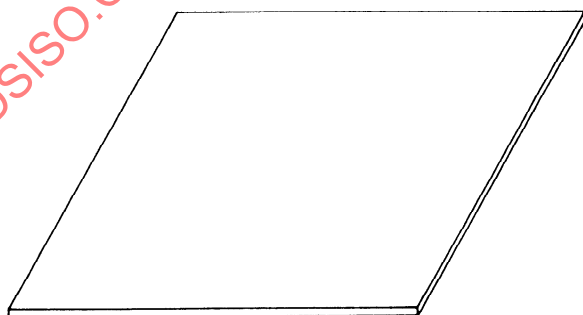
Dimensions in millimetres



1a Flat plate, 1,5 mm thick, of stainless steel or nickel-plated steel polished on the lower surface



1b Stainless-steel moulding template, 1,5 mm to 5 mm thick



1c Flat plate, 1,5 mm thick, of stainless steel or nickel-plated steel polished on the upper surface

Figure 1 — Suitable mould (see 5.2)