

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION R 1599

PLASTICS

DETERMINATION OF VISCOSITY LOSS ON MOULDING
OF CELLULOSE ACETATE

1st EDITION

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BRIEF HISTORY

The ISO Recommendation R 1599, *Plastics – Determination of viscosity loss on moulding of cellulose acetate*, 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 Draft ISO Recommendation No. 1599 which was circulated to all the ISO Member Bodies for enquiry in May 1968. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Austria	Israel	South Africa, Rep. of
Belgium	Italy	Spain
Brazil	Japan	Sweden
Czechoslovakia	Korea, Rep. of	Switzerland
Germany	Netherlands	Turkey
Hungary	Poland	U.A.R.
India	Portugal	United Kingdom
Iran	Romania	U.S.A.

The following Member Body opposed the approval of the Draft :

France

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

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PLASTICS

DETERMINATION OF VISCOSITY LOSS ON MOULDING
OF CELLULOSE ACETATE

1. SCOPE

This ISO Recommendation describes 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 depolymerisation of cellulose acetate, which generally increases brittleness in the moulded product.

2. FIELD OF APPLICATION

- 2.1 This method is suitable for cellulose acetate which does not contain additives, fillers, etc. which may interfere with the determination of viscosity.
- 2.2 It is suitable for cellulose acetate having an acetic acid yield above 50 %.

3. PRINCIPLE OF METHOD

Cellulose acetate plasticized with dimethylphthalate is moulded under specified conditions of temperature, pressure and time. After cooling, the moulding is ground up. The viscosity ratio of the moulding and also that of the original cellulose acetate are determined according to ISO Recommendation R 1157, *Plastics – Determination of viscosity number and viscosity ratio of cellulose acetate in dilute solution*. The percentage viscosity loss is calculated from the values of viscosity before and after moulding.

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

4. REAGENTS

- 4.1 *Dimethylphthalate*, analytical grade, having at 20 °C/20 °C a relative density within the range 1.191 to 1.195 and purity more than 99 % (m/m).
- 4.2 *Reagents* for viscosity ratio determination in accordance with ISO Recommendation R 1157.

5. APPARATUS

- 5.1 *Compression press*, capable of a pressure of at least 8 MN/m² (80 kgf/cm²) on the moulding surface area with means of heating to 200 °C and water cooling.
- 5.2 *Mould* (see Figure, page 8, for a suitable type) to produce mouldings of thickness within the range 1.5 to 5 mm. A particular thickness within this range may be specified.

5.3 *Apparatus for viscosity ratio determination*, in accordance with ISO Recommendation R 1157.

5.4 *Stainless steel grinder*, electrically operated.

5.5 *Oven*, thermostatically controlled at $70 \pm 2^\circ\text{C}$.

6. TEST SAMPLE

6.1 The sample of cellulose acetate must be in the form of powder passing entirely through a sieve of mesh $710\ \mu\text{m}$; it should be ground if necessary.

6.2 The moisture content of the sample is determined according to ISO Recommendation R 585, *Plastics – Determination of the moisture content of non-plasticized cellulose acetate*.

7. PROCEDURE

7.1 Weigh, in a glass bottle, with a precision of $\pm 0.5\ \text{g}$, the quantity of the test sample (6.1) corresponding to $100\ \text{g}$ of dry cellulose acetate. In another glass bottle, weigh $45\ \text{g}$ of dimethylphthalate with a precision of $\pm 0.5\ \text{g}$. Slowly add the dimethylphthalate (4.1) to the cellulose acetate with constant stirring and continue to stir for at least 5 minutes after all the dimethylphthalate has been added.

7.2 Heat the mixed material for 2 hours at $70 \pm 2^\circ\text{C}$ to remove moisture and complete the absorption of plasticizer.

7.3 Place a suitable quantity of the heated mixture in the mould, which is at a temperature of $200 \pm 2^\circ\text{C}$. Apply contact pressure for 2 minutes then full pressure (at least $8\ \text{MN/m}^2$ ($80\ \text{kgf/cm}^2$) of moulding area) for a further 10 ± 0.5 minutes for a moulding $1.5\ \text{mm}$ thick. This time is increased by 0.5 minutes for each 0.5 mm above 1.5 mm thickness.

7.4 Apply cooling immediately until the moulding is rigid enough for ejection. The rate of cooling is such that the mould temperature two minutes after onset of cooling is at least 30°C below the moulding temperature.

7.5 Grind the moulded cellulose acetate to a size less than $0.9\ \text{mm}$, avoiding excessive heating, and dry it at $70 \pm 2^\circ\text{C}$ for 2 hours.

7.6 Prepare a dichloromethane-methanol solution in accordance with ISO Recommendation R 1157, but containing $0.725\ \text{g}$ of material (i.e. $0.500\ \text{g}$ of cellulose acetate) to $100\ \text{ml}$ of solution, and determine the viscosity ratio.

7.7 Determine the viscosity ratio of a $5\ \text{g/l}$ solution of the original cellulose acetate according to ISO Recommendation R 1157.

7.8 Make two complete determinations according to clauses 7.1 to 7.7. If the difference between determinations is more than 10 % of their mean value, two further determinations should be made.