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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 307

PLASTICS

DETERMINATION OF THE VISCOSITY NUMBER OF POLYAMIDES IN DILUTE SOLUTION

1st EDITION
May 1963

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 307, *Determination of the Viscosity Number of Polyamides in Dilute Solution*, was drawn up by Technical Committee ISO/TC 61, *Plastics*, the Secretariat of which is held by the American Standards Association, Inc. (ASA).

Work on this question by the Technical Committee began in 1956 and led, in 1959, to the adoption of a Draft ISO Recommendation.

In October 1960, this Draft ISO Recommendation (No. 381) 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:

Australia
Austria
Belgium
Chile
Czechoslovakia
Germany
Hungary
India
Israel
Italy
Japan
Netherlands
New Zealand
Poland
Romania
Spain
Sweden
Switzerland
Turkey
United Kingdom
U.S.A.
U.S.S.R.

One Member Body opposed the approval of the Draft:

France.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in May 1963, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 307 May 1963

PLASTICS

DETERMINATION OF THE VISCOSITY NUMBER OF POLYAMIDES IN DILUTE SOLUTION

1. SCOPE

1.1 This method of test is designed to determine the viscosity number (see Appendix A, page 8) of a solution in formic acid or metacresol of polyamides. The method applies only to those resins that give stable solutions and reproducible results under the conditions of test described below. The times of flow of the solvent and of a solution containing 0.005 g/ml of polyamide are measured at 25 °C by conventional methods, and the viscosity number is calculated from these measurements and from the known concentration of the solution. Density and kinetic energy corrections are small in this method and are not applied.

1.2 If monomer * is known to be present in the sample, the amount should be determined by the relevant ISO method ** and reported. The presence of monomer affects considerably the determination of viscosity number of certain types of polyamides. If the sample contains an additive or a filler, which would affect the viscosity measurement, it should be removed by a procedure agreed between the contracting parties.

1.3 This method is meant to give an insight into the molecular structure of polyamides and for the purpose of comparing different types. For control purposes in the manufacture of polyamides or for specifications, viscosity is normally determined in concentrated solution in accordance with the relevant ISO Recommendation.***

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* In this text, monomer is understood as " monomer and low molecular mass polymer " of the relevant ISO Recommendation (see below **).

** ISO Recommendation under preparation, *Plastics—Determination of the Percentage of Monomer and Low Molecular Weight Polymer in Polyamide Resins*, at present Draft ISO Recommendation No. 512.

*** ISO Recommendation under preparation, *Plastics—Determination of the "Relative Viscosity" of Polyamide Resins in Concentrated Solution*, at present Draft ISO Recommendation No. 513.

2. APPARATUS

The apparatus consists of the following:

(*a*) *Automatic pipette*, 50 ml and

(*b*) *Flat-bottomed flask*, 150 ml, with ground-glass stopper, or

Volumetric flask, 50 ml, with ground-glass stopper.

(*c*) *Filter funnel* of sintered glass with a porosity of 40 to 90 μm,* or

Conical sieve of stainless steel with apertures about 0.075 mm^2.

(*d*) *Bath*, maintained at 25 ± 0.05 °C.

(*e*) *Viscometer* of suspended level Ubbelohde type; its essential dimensions are shown in the figure (page 6),
alternatively, any other viscometer that can be shown to give the same results.

(*f*) *Balance*, to weigh to 0.000 1 g.

(*g*) *Stop-watch* reading to 0.1 second.

(*h*) *Burette*.

(*i*) *Bottle* of brown glass connected to an automatic burette and fitted with a calcium chloride tube on the air inlet.

Possibly means for drying (see clause 4.1).

3. SOLVENTS AND CHEMICALS

The following solvents and chemicals are required:

(1) *Formic acid*, containing 90 ± 0.2 per cent by mass of chemically pure formic acid, the remainder being water (see Appendix B, page 8),** or

Metacresol, freshly distilled, melting point 11 to 12 °C.

(2) *Cleaning liquids* (distilled water, concentrated sulphuric acid, saturated aqueous solution of potassium dichromate, ethanol, acetone).

4. PREPARATION OF SAMPLE

4.1 The sample should be in the form of small pieces or powder. The water content, determined in accordance with the relevant ISO Recommendation,*** should not exceed 0.4 per cent.

4.2 Alternatively, if the water content exceeds 0.4 per cent and is known, the mass of resin taken should be adjusted to give the specified weight of dry material.

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* M. Bechtold, Zeitschrift für Physikalische Chemie, 60 (1907), 257 and 64 (1908), 328.

** Formic acid should be used whenever possible, i.e. when the resin is soluble in this solvent.

*** See ISO Recommendation R 62, *Plastics—Determination of Water Absorption.*

5. PROCEDURE

5.1 Before it is first used, after discordant readings and at regular intervals during use, the viscometer (*e*) is cleaned first with the solvent (1) or (2) and successively with water and with a mixture of equal volumes of concentrated sulphuric acid and a saturated solution of potassium dichromate (3) in water. After rinsing with distilled water, the viscometer should be washed with 95 to 96 per cent ethanol, followed by acetone, and dried by drawing through it a stream of air free from dust. Between successive satisfactory determinations, the viscometer is washed with several portions of solvent, followed by acetone and dried as described.

A quantity of 0.25 ± 0.005 g of resin is weighed on the balance (*f*) to the nearest 0.0002 g and transferred quantitatively to the 150 ml glass-stoppered flask (*b*), then 50 ml of solvent is pipetted into the flask, care being taken to avoid the formation of lumps, and the stopper is inserted.

5.2 When metacresol (2) is used, the mixture is heated at approximately 95 to 100 °C for 2 hours. If a longer period of time is required to dissolve the resin, this should be reported.
When formic acid (1) is used as the solvent, the resin is dissolved by warming the mixture and shaking, care being taken that the temperature does not exceed 30 °C.

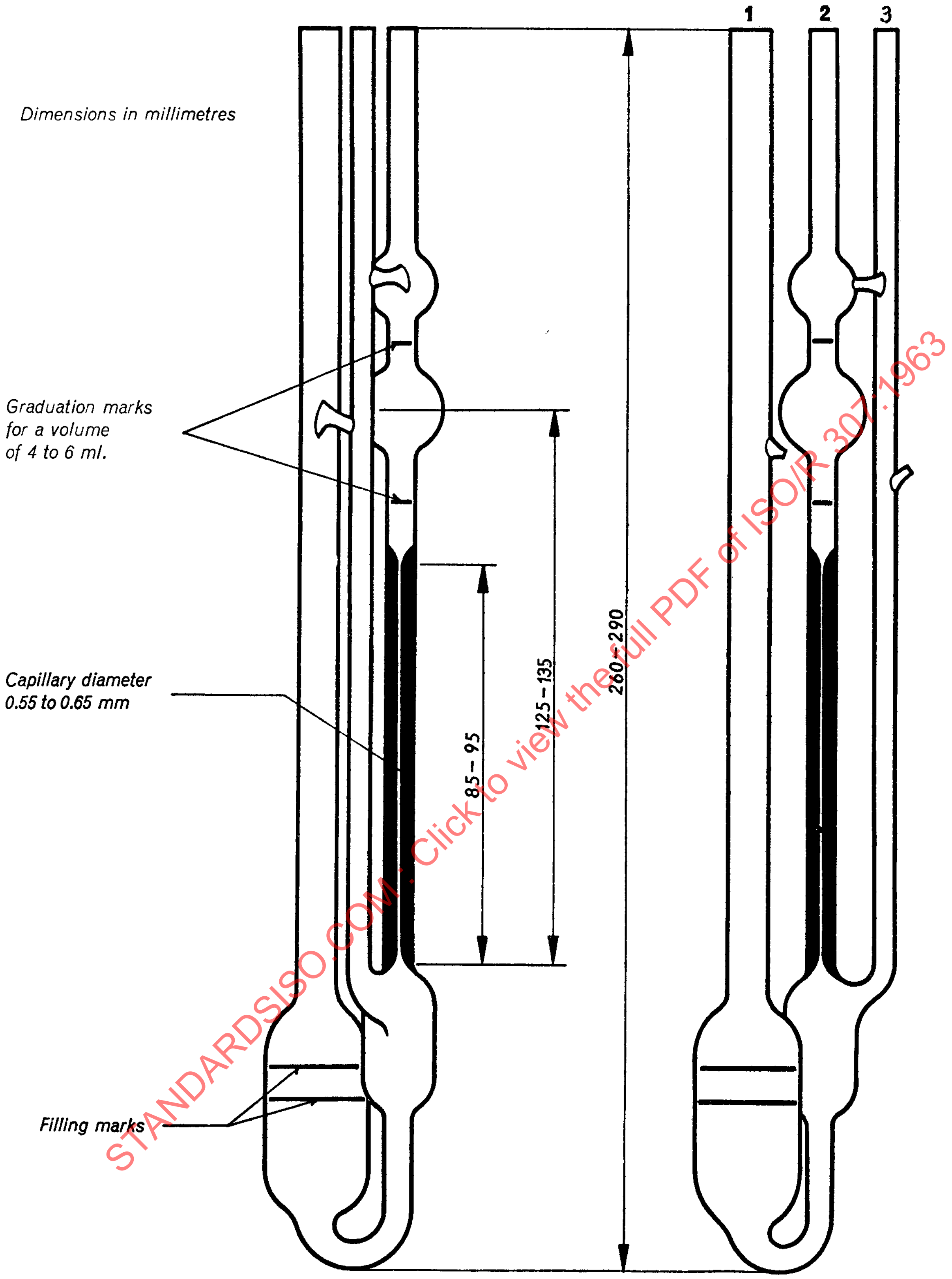
The solution is then cooled to about 25 °C.

Alternatively, when formic acid is used as the solvent, the weighed sample of resin is transferred quantitatively to the 50 ml glass-stoppered volumetric flask (*b*), 40 ml of solvent added and the stopper inserted. The resin is dissolved as described in the previous paragraph, the same precautions being taken. The solution is cooled at 25 °C and the volume made up to the 50 ml mark. Care should be taken that solution and solvent are well mixed.

5.3 The times of flow of the solution and of the solvent are determined in the same viscometer; in the case of metacresol, the portion of the solvent used as reference is heated under the same conditions as the solution. If an Ubbelohde viscometer is used, these measurements are made as follows:

The liquid is filtered through the sintered glass filter funnel (*c*) directly into the tube (No. 1) (see figure) of the viscometer, which is immersed in the bath maintained at 25 ± 0.05 °C, to a depth of approximately 20 mm above the upper graduation mark, and supported so that the tube (No. 1) is vertical. The volume of liquid in the viscometer should be such that, after draining, the level lies between the two filling marks. After not less than 10 minutes, the liquid is blown with dust-free air, or drawn, into the upper bulb until it reaches approximately the centre of the bulb. A finger is then placed over the tube (No. 2) until the liquid drops away from the lower end of the capillary. After removal of the finger, the time interval, which should not be shorter than 100 seconds, for the passage of the meniscus between the two graduation marks is measured to the nearest 0.4 second. The liquid is then blown or drawn into the upper bulb and the time of flow again measured. If a viscometer other than the Ubbelohde is used, the above procedure should be suitably modified.

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 second. The mean time of flow of the solvent should be determined in the same manner.



FIGURE—Ubbelohde Viscometer

NOTE.—The capillary diameter should be chosen so that the time of flow will be not less than 100 seconds; in the figure, the given diameter is suitable for formic acid; for the same type of viscometer, a larger diameter is required for metacresol.