INTERNATIONAL STANDARD ISO 307

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Plastics — Determination of viscosity number of polyamides in dilute solution

Matières plastiques — Détermination de l'indice de viscosité des résines polyamides en solution diluée

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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.

International Standard ISO 307 was drawn up by Technical Committee ISO/TC 61, *Plastics.*

It was submitted directly to the ISO Council, in accordance with clause 6.12.1 of the Directives for the technical work of ISO. It cancels and replaces ISO Recommendation R 307-1963, which had been approved by the member bodies of the following countries :

Australia
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The member body of the following country had expressed disapproval of the document on technical grounds :

France

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Plastics – Determination of viscosity number of polyamides in dilute solution

1 SCOPE AND FIELD OF APPLICATION

1.1 This International Standard specifies a method for the determination of the viscosity number (see annex A) of a dilute solution of polyamide in formic acid or metacresol. This method is applicable only to those resins that give stable solutions and reproducible results under the specified conditions of test.

1.2 If a monomer[1] is known to be present in the sample, the amount should be determined by the procedure described in ISO/R 599 and reported. The presence of monomer affects considerably the determination of viscosity number of certain types of polyamide. 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 interested parties.

1.3 This method is meant to give an insight into the molecular structure of polyamides and to allow the comparison of different types.

2 REFERENCES

ISO/R 599, *Plastics – Determination of the percentage of extractable material in polyamides.*

ISO/R 960, *Plastics – Determination of the water content in polyamides.*

3 PRINCIPLE

Measurement at 25 °C, by conventional methods, of the times of flow of the solvent and of a solution containing 0,005 g/ml of polyamide.

Calculation of the viscosity number 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.

4 REAGENTS

Use only reagents of recognized analytical reagent grade and only distilled water or water of equivalent purity.

4.1 Formic acid, containing 90 ± 0,2 % (*m/m*) of chemically pure formic acid, the remainder being water (see annex B),[2] or

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

4.3 Cleaning liquids : distilled water, concentrated sulphuric acid, saturated aqueous solution of potassium dichromate, 95 to 96 % (*V/V*) ethanol, and acetone.

5 APPARATUS

5.1 Automatic pipette, capacity 50 ml.

5.2 Flat-bottomed flask, capacity 150 ml, with ground glass stopper, or

5.3 Volumetric flask, capacity 50 ml, with ground glass stopper.

5.4 Filter funnel, or sintered glass, with a pore size index greater than 40 μm but not greater than 100 μm,[3] or

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[1] In this International Standard, monomer is understood as "monomer and low molecular mass polymer" as determined according to ISO/R 599.

[2] Formic acid shall be used whenever possible, i.e. when the resin is soluble in this solvent.

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

5.5 Conical sieve, of stainless steel, with apertures about 0,075 mm².

5.6 Water bath, capable of being maintained at 25 ± 0,05 °C.

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

5.8 Balance, accurate to 0,000 1 g.

5.9 Stop-watch, reading to 0,1 s.

5.10 Burette.

5.11 Bottle, of brown glass, connected to an automatic burette and fitted with a calcium chloride tube on the air inlet.

6 PREPARATION OF THE SAMPLE

The sample shall be in the form of small pieces or powder. The water content, determined in accordance with ISO/R 960, should not exceed 0,4 % (*m*/*m*).

Alternatively, however, if the water content does exceed 0,4 % (*m*/*m*) and is known, the mass of the test portion (7.2) may be adjusted to give the specified mass of dry material.

7 PROCEDURE

7.1 Cleaning of viscometer

Before it is first used, after discordant readings and at regular intervals during use, clean the viscometer (5.7) first with the solvent (4.1 or 4.2) and successively with water and with a mixture of equal volumes of concentrated sulphuric acid and a saturated solution of potassium dichromate in water. After rinsing with distilled water, wash the viscometer with 95 to 96 % (*V*/*V*) ethanol, followed by acetone, and dry by drawing through it a stream of air free from dust. Between successive satisfactory determinations, wash the viscometer with several portions of solvent, followed by acetone, and dry as described.

7.2 Test portion

Weigh, to the nearest 0,000 2 g, 0,25 ± 0,005 g of resin.

7.3 Preparation of the test solution

Proceed as in 7.3.1 or 7.3.2.

7.3.1 Transfer the test portion (7.2) quantitatively to the 150 ml flask (5.2). Then pipette 50 ml of solvent (4.1 or 4.2) into the flask, taking care to avoid the formation of lumps, and insert the stopper.

a) When formic acid (4.1) is used as the solvent, dissolve the resin by warming the mixture and shaking, taking care that the temperature does not exceed 30 °C.

b) When metacresol (4.2) is used as the solvent, heat the mixture at approximately 95 to 100 °C for 2 h. If a longer period of time is required to dissolve the resin, this shall be reported.

Cool the solution to about 25 °C.

7.3.2 Alternatively, when formic acid (4.1) is used as the solvent, transfer the test portion (7.2) quantitatively to the 50 ml volumetric flask (5.3), add 40 ml of the formic acid and insert the stopper. Dissolve the resin by warming the mixture and shaking, taking care that the temperature does not exceed 30 °C.

Cool the solution to 25 °C, dilute to the mark with the formic acid and mix thoroughly.

7.4 Viscometer measurements

7.4.1 Determine the times of flow of the test solution (7.3) and of the solvent in the same viscometer; in the case of metacresol, heat the portion of the solvent used as reference under the same conditions as used for the preparation of the test solution [7.3.1 b)].

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

Filter the liquid through the sintered glass filter funnel (5.4) or the conical sieve (5.5) directly into tube No. 1 (see figure) of the viscometer, which is immersed in the bath (5.6) maintained at 25 ± 0,05 °C, to a depth of approximately 20 mm above the upper graduation mark, and supported so that tube No. 1 is vertical. The volume of liquid in the viscometer shall be such that, after draining, the level lies between the two filling marks. After not less than 10 min, blow the liquid with dust-free air, or draw it, into the upper bulb until it reaches approximately the centre of the bulb. Then place a finger over tube No. 2 until the liquid drops away from the lower end of the capillary. After removal of the finger, measure, to the nearest 0,4 s, the time-interval (which should be not less than 100 s) for the passage of meniscus between the two graduation marks. Then blow or draw the liquid into the upper bulb and again measure the time of the flow.

7.4.3 If a viscometer other than the Ubbelohde is used, modify the above procedure accordingly.

7.4.4 Take the time of flow of the solution as the mean of the results of two determinations which do not differ by more than 0,4 s. Determine the mean time of flow of the solvent in the same manner.

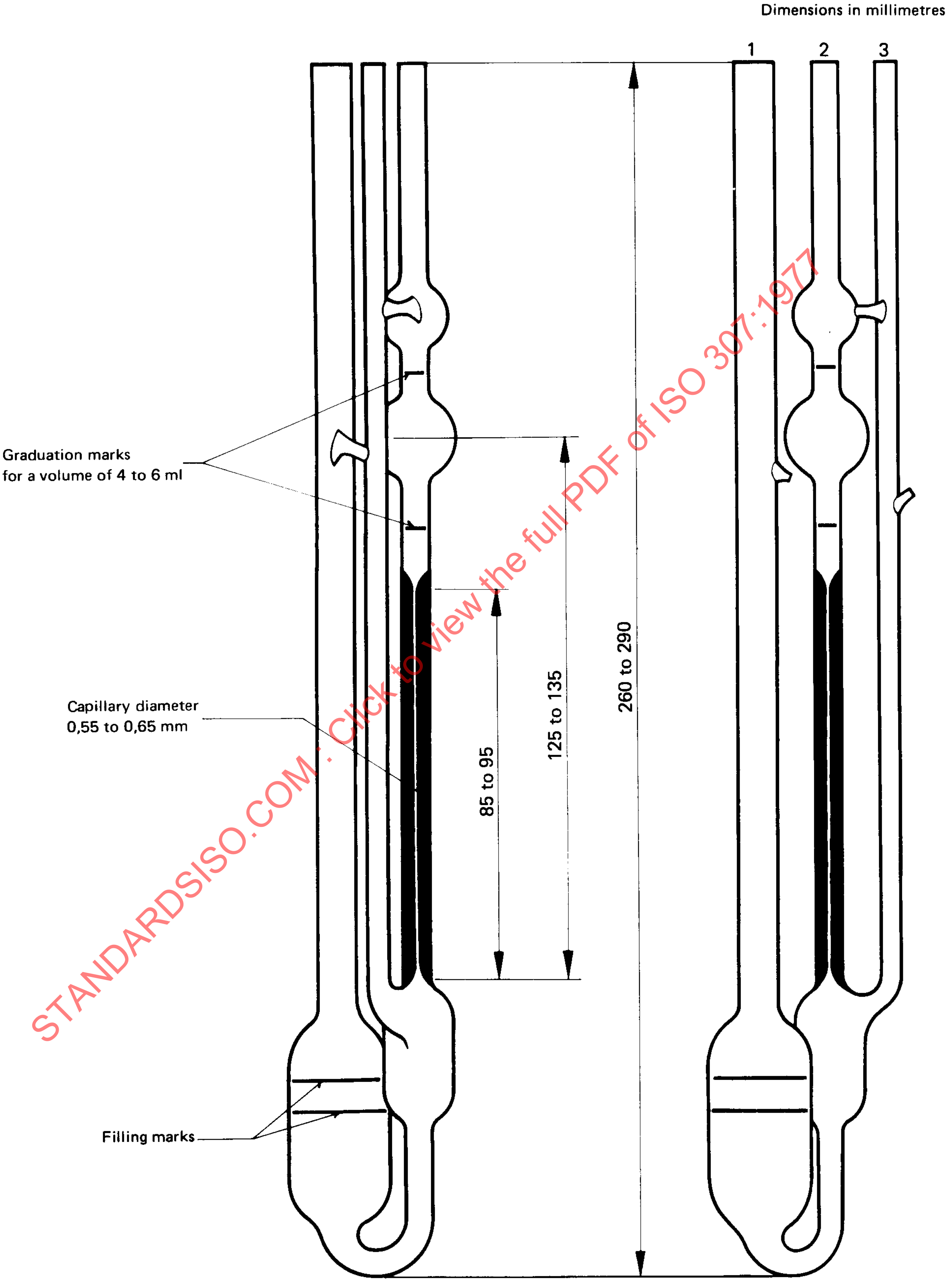

FIGURE — **Ubbelohde viscometer**

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

8 EXPRESSION OF RESULTS

The viscosity number is given by the formula :

$$\frac{t - t_0}{t_0 \, c}$$

where

t is the time of flow of the test solution;

t_0 is the time of flow of the solvent;

c is the concentration, in grams of solute per millilitre of test solution or, with sufficient accuracy for this method, in grams per millilitre of solvent.

Report the result to the nearest whole number.

9 TEST REPORT

The test report shall include the following particulars :

a) reference to this International Standard;

b) complete identification of the material tested, including type, source, manufacturer's code numbers and trade name, etc.;

c) monomer content, if any;

d) details of treatment of the sample, if any, before the test;

e) water content or conditions of drying;

f) type of viscometer used;

g) solvent used for viscosity measurement;

h) viscosity number;

i) test conditions, if any, deviating from those of this International Standard and likely to have affected the results.

ANNEX A

USE OF THE TERM "VISCOSITY NUMBER"

The term "viscosity number", formerly "reduced viscosity", has been recommended for adoption by the International Union of Pure and Applied Chemistry (IUPAC). Details are given in the *Journal of Polymer Science, VIII* (1952), No. 3, 269-270, where the viscosity number is defined as

$$\frac{\eta - \eta_0}{\eta_0 \, c}$$

where

η is the absolute viscosity of the solution;

η_0 is the absolute viscosity of the solvent;

c is the concentration, in grams of solute per millilitre of solution.

It has been argued that the term "viscosity number" is not wholly satisfactory, because the quantity that is described is not without dimensions. The term is used in this method for lack of one more suitable, but it is intended to replace it when a more acceptable term is recommended by IUPAC.

ANNEX B

PREPARATION OF FORMIC ACID

The formic acid solution to be used shall contain 90 ± 0,2 % (*m/m*) of chemically pure formic acid. To prepare this solution, dilute commercially concentrated formic acid, of analytical grade, with distilled water to 90 % or more. Determine the exact acid content of this solution by titration with sodium hydroxide, using phenolphthalein as indicator. Calculate the amount of distilled water to be added, if the solution does not contain between 89,8 and 90,2 % formic acid, from the result of the titration. After adding this further quantity of distilled water, determine the acid content again by titration as indicated above. Store the formic acid solution in a brown-glass bottle closed with a ground glass stopper. In operation, the stopper can be connected to an automatic burette, provided with a calcium chloride tube on the air inlet. Check the acid content of the formic acid every 2 weeks.