International Standard 1628/5

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Plastics — Determination of viscosity number and limiting viscosity number — Part 5 : Poly(alkylene terephthalates)

Plastiques — Détermination de l'indice de viscosité et de l'indice limite de viscosité — Partie 5 : Poly(alkylène téréphtalates)

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Foreword

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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. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 1628/5 was prepared by Technical Committee ISO/TC 61, *Plastics*.

It cancels and replaces ISO 1228-1975, of which it constitutes a technical revision.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Plastics — Determination of viscosity number and limiting viscosity number — Part 5 : Poly(alkylene terephthalates)

1 Scope and field of application

This part of ISO 1628 specifies a method for determining the viscosity number of dilute solutions of poly(ethylene terephthalate), poly(butylene terephthalate) and copolymers of the two. With poly(ethylene terephthalate) a mixture of 1 part by mass of phenol and 1 part by mass of 1,2-dichlorobenzene is used as solvent. With the other polymers *m*-cresol is used as solvent.

NOTE — For the definition of viscosity number and for other terms, definitions and formulae, see ISO 1628/1.

2 References

ISO 1628/1, *Plastics — Guidelines for the standardization of methods for the determination of viscosity number and limiting viscosity number of polymers in dilute solution — Part 1 : General conditions.*

ISO 3105, *Glass capillary kinematic viscometers — Specification and operating instructions.*

ISO 3451/2, *Plastics — Determination of ash — Part 2 : Poly(alkylene terephthalates).*

3 Principle

Measurement of the times of flow of the solvent and a solution of resin at a concentration of 0,005 g/ml at 25 °C by conventional methods; calculation of the viscosity number 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.

4 Solvents

WARNING — The solvents are hazardous. Avoid contact with the skin and inhalation of the vapours.

4.1 Phenol/1,2-dichlorobenzene

Weigh out 1 part by mass phenol (analytical grade) and dissolve in 1 part by mass 1,2-dichlorobenzene (analytical grade). Work to an accuracy of 1 % or better in the weighings.

4.2 *m*-Cresol

This compound shall meet the following specification :

appearance : clear and colourless;

content of *m*-cresol : 99 % (m/m) min.;

content of *o*-cresol : 0,3 % (m/m) max.;

content of water : 0,13 % (m/m) max.

NOTE — A solvent of the required purity can be obtained by distillation of chemically pure *m*-cresol, preferably *in vacuo*, pressure compensation being effected with nitrogen in order to avoid oxidation. The purity of the solvent may be checked by gas chromatography.

4.3 Storage of the solvents

Protect the solvents from light by storing them, for example, in stoppered brown glass bottles.

Determine the flow time of the solvents at least once each day that they are used (see 6.6). If the flow time of a solvent differs by more than 1 % from the initial value, reject the solvent.

5 Apparatus

5.1 Mill, to reduce the sample to a grain size of about 0,5 mm.

5.2 Volumetric flask, of capacity 100 ml, fitted with a ground glass stopper.

5.3 Erlenmeyer flask, of capacity 150 ml, fitted with a ground glass stopper.

5.4 Burette, graduated in divisions of 0,1 ml.

5.5 Steam bath, or other means to heat the contents of a flask to 90-100 °C.

5.6 Oil bath, capable of being controlled at 135-140 °C, or other means to heat the contents of a flask to that temperature range.

5.7 Magnetic stirrer, or other equipment to continuously agitate the contents of a stoppered flask.

5.8 Thermostatic bath, capable of being controlled at 25 ± 0,05 °C.

5.9 Viscometers, suspended-level Ubbelohde type, sizes Nos. 1C and 2, respectively, according to ISO 3105. The essential dimensions of the viscometers are shown in the figure.

Other types of viscometer listed in ISO 3105 may be used, provided that the results are equivalent to those of the Ubbelohde viscometers specified above. However, in the case of dispute, Ubbelohde viscometers shall be used.

5.10 Stainless steel sieve, with a nominal aperture size of 63 to 90 µm, or **sintered glass filter funnel** with 40 to 100 µm pore diameter.

5.11 Analytical balance, accurate to 0,1 mg.

5.12 Stop-watch, accurate to 0,1 s.

6 Procedure

6.1 Cleaning the viscometer

WARNING — Chromic acid solutions and other similar cleaning reagents are hazardous chemicals. Avoid contact with the skin and inhalation of any vapours.

Clean the viscometer (5.9) before use, after discordant readings and at intervals during regular use. To do this, let it stand for at least 12 h filled with a cleaning reagent, such as a chromic acid solution prepared by mixing 1 part by volume of concentrated sulphuric acid with 1 part by volume of a saturated solution of potassium dichromate. Remove the cleaning reagent, rinse the viscometer with distilled water followed by a suitable volatile solvent, such as freshly distilled acetone, and dry, for example with a slow stream of filtered air.

After the determination, drain the viscometer, rinse with the solvent, then rinse with freshly distilled acetone or a similar volatile solvent, and dry as described.

If the next solution to be measured is of a polymer of the same type and of a similar viscosity, it is permissible to drain the viscometer, wash it with the solution to be measured, then fill it with this solution.

6.2 Selection of the solvent

With poly(ethylene terephthalate), use phenol/1,2-dichlorobenzene (4.1) as the solvent. With poly(butylene terephthalate) and copolymers of ethylene terephthalate and butylene terephthalate, use *m*-cresol (4.2) as the solvent.

6.3 Determination of inorganic materials or other additives in the sample

Contents of inorganic materials or other additives exceeding 0,5 % (m/m) each shall be taken into account in the preparation of the test solution (6.4).

6.3.1 Determination of the content of inorganic materials

If the sample contains inorganic materials, such as fillers or glass fibres, determine the content according to ISO 3451/2.

6.3.2 Determination of the content of other additives

If the sample contains other additives, such as polyalkenes or flame retardants, determine the content in an appropriate way. Report the procedure(s) applied.

6.4 Preparation of the test solution

Use one of the following procedures :

6.4.1 Procedure A

Weigh, to the nearest 0,2 mg, a test portion, m_1, in grams, in the range

$$\frac{0,5}{1 - [(i + o)/100]} \pm 0,01$$

where

i is the content of inorganic materials, expressed as a percentage by mass, in the sample, determined in accordance with 6.3.1;

o is the content of other additives, expressed as a percentage by mass, in the sample, determined in accordance with 6.3.2.

The corrections for i and o need only be applied if they exceed 0,5 % (m/m) each.

Transfer the test portion to the volumetric flask (5.2), add 60 ml of solvent, stopper the flask and warm on the steam bath (5.5), with occasional stirring, until the polymer has dissolved completely. On the steam bath, no degradation of the polymer occurs. If, however, long waiting times are undesirable for other reasons, the dissolution may be accelerated by applying continuous agitation, for example by the magnetic stirrer (5.7).

Samples of highly crystalline poly(ethylene terephthalate), such as postcondensed moulding chips, which may have crystallinities around 65 %, will not dissolve on the steam bath, not even with continuous stirring. To dissolve such samples, the material shall be ground in the mill (5.1) and dissolved at a temperature of 135-140 °C (5.6) applying continous stirring. At 135-140 °C, some degradation of the polymer occurs. Therefore, avoid heating times over 30 min.

After dissolution, cool the flask and its contents to 25 ± 2 °C, make up to 100 ml with solvent held at this temperature and mix well. If in dissolving a magnetic stirrer has been used, first remove it from the solution and rinse it with the make-up solvent, ensuring that all the washings enter the flask. The concentration c of the polymer in the solution, expressed in grams per millilitre, used in calculating the viscosity number (clause 7), is computed as

$$0{,}01 \left(1 - \frac{i + o}{100}\right) m_1$$

NOTE — Usually, the concentration will differ slightly from 0,005 g/ml. The effect on the viscosity number can be neglected, however, since, over the range considered, the viscosity relative increment may be considered to be a linear function of the concentration.

6.4.2 Procedure B

Weigh, to the nearest 0,2 mg, a test portion, m_2, in the range 0,4 to 0,6 g.

Transfer the test portion to the Erlenmeyer flask (5.3). Add by burette (5.4), to the nearest 0,1 ml, a volume of solvent, in millilitres, equal to

$$200 \left(1 - \frac{i + o}{100}\right) m_2$$

where i and o have the same meaning as in 6.4.1.

The corrections for i and o need only be applied if they exceed 0,5 % (m/m) each. The solvent added shall be at 25 ± 2 °C.

Stopper the flask. Dissolve the polymer by heating the flask on the steam bath, or, if required, in the oil bath, following the procedures and observing the precautions mentioned in 6.4.1. After dissolution, cool the flask to room temperature.

6.5 Preparatory operations

With phenol/1,2-dichlorobenzene as the solvent, use a viscometer size No. 1C. With *m*-cresol, use a viscometer No. 2.

Filter the test solution through the screen or sintered glass filter (5.10) into tube L of the viscometer (see the figure). The volume should be such that, after draining, the level lies between the filling marks. Carry out this filling operation away from the thermostatic bath (5.8) to prevent contamination of the bath in case of accidental spills.

Mount the viscometer in the thermostatic bath, ensuring that tube N is vertical and that the upper graduation mark, E, is at least 30 mm below the surface of the bath liquid. Allow at least 15 min for the charged viscometer to attain the temperature of the bath, (25 ± 0,05 °C).

6.6 Measurement of flow times

Close tube M and apply suction to tube N or pressure to tube L until the liquid reaches a level about 5 mm above mark E. Hold the liquid at this level by closing tube N. Open tube M so that the liquid drains away from the lower end of the capillary tube.

When the liquid is clear of the capillary end and of the lower end of tube M, open tube N. Measure the flow time to an accuracy of 0,2 s as the time taken for the lowest point of the meniscus to pass from the top edge of mark E, to the top edge of mark F. For polymer solutions containing small amounts of finely divided pigments, for example carbon black, it may be necessary to view the top of the meniscus.

NOTE — The opening and closing of the tubes may conveniently be carried out by means of taps or clips on clean flexible tubing attached to the end of the tubes.

Repeat the measurement of flow time immediately, without emptying and recharging until two successive flow times agree within 0,25 %. Take the mean of these two flow times as the flow time of the test solution.

Determine the mean flow time of the solvent in the same viscometer and in the same manner as that of the test solution. It suffices to measure this time once for each series of determinations. However, measure the time at least once each day the solvent is used. If two successive determinations of the mean flow time differ by more than 0,4 s, clean the viscometer.

7 Expression of results

Calculate the viscosity number, expressed in millitres per gram, according to the formula

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

where

t is the flow time, in seconds, of the test solution;

t_0 is the flow time, in seconds, of the solvent;

c is the concentration of the polymer in the test solution in grams per millilitre.

When procedure A has been applied in preparing the test solution, calculate c as described in 6.4.1. When procedure B has been applied, c is equal to 0,005 g/ml.

With each polymer sample, carry out at least two determinations of the viscosity number, each time using a fresh solution, until two successive values differ by no more than 3 %. Report the mean of these two values, rounded off to the nearest unit, as the viscosity number of the sample.

8 Test report

The test report shall include the following information :

a) a reference to this International Standard;

b) complete identification of the sample;

c) if the sample contains more than 0,5 % (m/m) of other additives according to 6.3.2, a description of the procedure(s) used for their determination;

d) details of the viscometer used if it differs from the Ubbelohde type, sizes No. 1C and 2 according to ISO 3105;

e) the viscosity number obtained.

Dimensions in millimetres

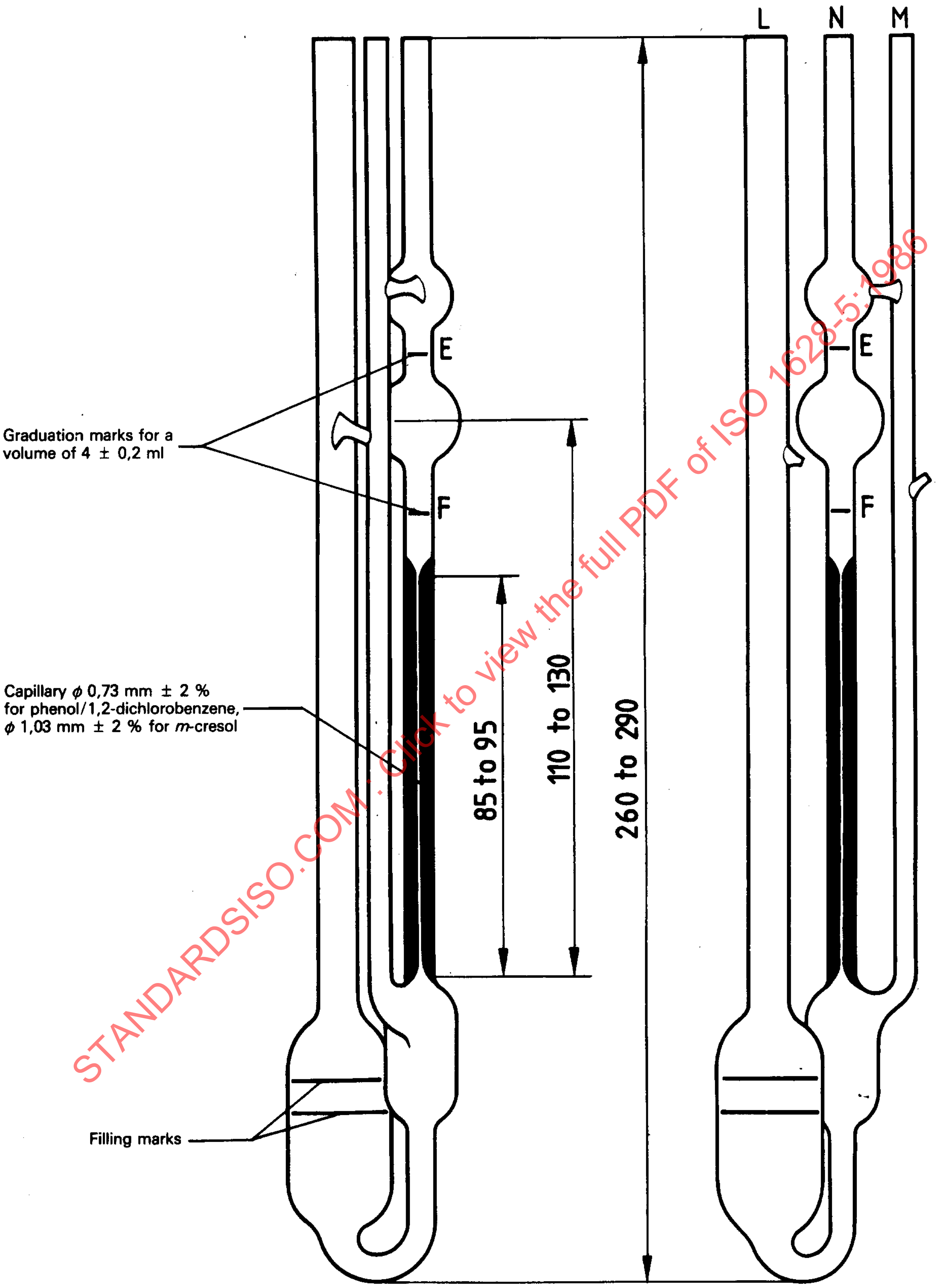

Figure — Ubbelohde viscometer