INTERNATIONAL STANDARD 1228

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION •МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Plastics — Poly(ethylene terephthalate) in dilute solution — Determination of viscosity number

Matières plastiques — Poly(éthylène téréphtalate) en solution diluée — Détermination de l'indice de viscosité

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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.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 61 has reviewed ISO Recommendation R 1228 and found it technically suitable for transformation. International Standard ISO 1228 therefore replaces ISO Recommendation R 1228-1970 to which it is technically identical.

ISO Recommendation R 1228 was approved by the Member Bodies of the following countries :

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

No Member Body expressed disapproval of the Recommendation.

The Member Bodies of the following countries disapproved the transformation of ISO/R 1228 into an International Standard :

Canada
United Kingdom

Printed in Switzerland

Plastics — Poly(ethylene terephthalate) in dilute solution — Determination of viscosity number

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for determining the viscosity number of a solution of poly(ethylene terephthalate) in *o*-chlorophenol.

NOTE — For the definition of viscosity number, and for other terms, definitions and formulae, see ISO/R 1628, *Plastics — Directives for the standardization of methods for the determination of the dilute solution viscosity of polymers.*

2 PRINCIPLE

The times of flow of the solvent and a solution of resin at a concentration level of 0,01 g/ml 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 difference and kinetic energy corrections are small in this method and are not applied.

3 SOLVENT

***o*-chlorophenol** (analytical grade), water content less than 0,15 % and freezing point higher than 8 °C.

Alternatively, any other solvent may be used which gives the same results and is consistent with the requirements of table 2 in ISO/R 1628.

One such solvent could be a mixture of phenol and tetrachloroethane in the ratio of 1 : 1 to 2 : 1.

NOTE — Since *o*-chlorophenol is somewhat toxic, care must be taken to prevent contact with the skin and eyes or breathing the vapour.

4 APPARATUS

4.1 Volumetric flask, 100 ml, with ground glass stopper.

4.2 Steam bath, with electrical heater and provision for magnetic stirring or mixing, capable of maintaining a temperature of 90 to 100 °C.

4.3 Thermostatic bath, maintained at 25 ± 0,05 °C.

4.4 Viscometer, suspended-level Ubbelohde type, of which the essential dimensions are shown in the figure, or any other viscometer meeting the requirements of ISO/R 1628, particularly table 1.

4.5 Vacuum oven, pressure less than 1,333 mbar (1 mmHg), temperature 100 ± 2 °C.

4.6 Desiccator.

4.7 Stainless steel screens, with 63 to 90 µm nominal size of aperture, or sintered glass filter funnel with 50 to 100 µm pore diameter.

4.8 Analytical balance, to weigh to 0,000 1 g.

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

5 PROCEDURE

5.1 Clean the viscometer (4.4) before it is used, after discordant readings and at intervals during regular use.

After discordant readings, drain any remaining solution from the viscometer, wash well with chloroform followed by acetone, then dry by drawing through it a stream of air free from dust. Then, as prior to use, wash with a mixture of equal volumes of concentrated sulphuric acid and a saturated solution of potassium dichromate in water. Rinse it with water followed by acetone and dry it again by drawing through it a stream of air free from dust.

Between successive satisfactory determinations, wash the viscometer with the same solvent which has been used for the determination, then with acetone and dry as described.

If the next solution to be measured is of similar viscosity, it is permissible to drain the viscometer, wash it with the solution to be measured, then fill with the solution to be measured.

5.2 If necessary to reduce water content of the polymer below 0,5 %, dry the polymer in the vacuum oven (4.5) at 100 °C for 3 h, at a pressure less than 1,333 mbar (1 mmHg), and then cool in a desiccator. Weigh 1,0 ± 0,002 g of sample to the nearest 0,000 2 g, transfer it quantitatively to a conical glass-stoppered volumetric flask (4.1), add 60 ml of solvent and warm on the steam bath (4.2), stirring until the sample has dissolved.

Avoid prolonged warming which may cause thermal degradation of the polymer. Cool to 20 °C, make up the volume to the 100 ml mark with solvent at 20 °C and mix well. (If a magnetic stirrer is used in the volumetric flask, its displacement of solvent at 20 °C shall be determined in advance; to achieve the desired 0,01 g/ml solution, the 1,0 ± 0,002 g of resin shall be reduced proportionately to the reduced volume of solvent, or the displaced volume of solvent at 20 °C shall be added above the 100 ml mark.)

5.3 Filter the liquid through the sintered glass filter funnel or the screen (4.7) directly into tube 1 of the viscometer (see figure), which shall be immersed in the bath (4.3) maintained at 25 ± 0,05 °C to a depth of approximately 20 mm above the upper graduation mark and supported so that tube 2 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 15 min blow or draw the liquid, with dust-free air, into the upper bulb until it reaches approximately the centre of the bulb. Then place a finger over tube 2 until the liquid drops away from the lower end of the capillary.

Remove the finger and measure the time interval for the passage of the meniscus between the two graduation marks. Then blow or draw the liquid into the upper bulb and measure again the time of flow of the solution.

The time of flow of the solution shall be taken as the mean of the two determinations which shall not differ by more than 0,4 %. The mean time of flow of the solvent is determined in the same manner.

6 EXPRESSION OF RESULTS

The viscosity number, expressed in millilitres per gram, is calculated as follows :

$$\text{Viscosity number} = \frac{t_s - t_0}{t_0\, c}$$

where

t_s is the time of flow of the solution, in seconds;

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

c is the concentration in grams of resin per millilitre of solution.

The calculated value is rounded off to the nearest unit.

7 TEST REPORT

The test report shall include the following particulars :

a) complete identification of the sample;

b) viscosity number reported to the nearest whole number;

c) whether alternative viscometers or solvents were used;

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

e) test date.

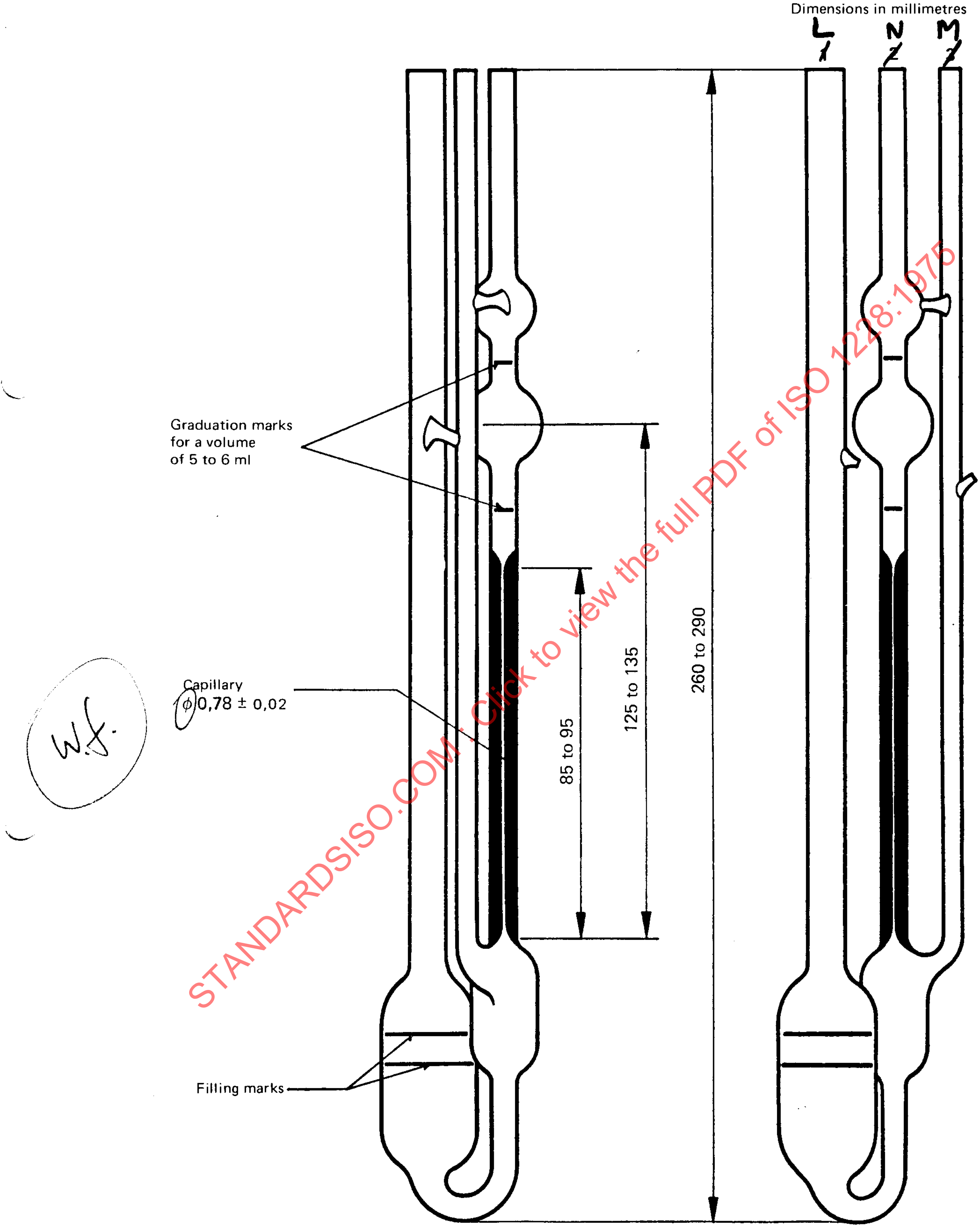

FIGURE – **Ubbelohde viscometer**