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R 1228

PLASTICS

DETERMINATION OF VISCOSITY NUMBER OF POLY(ETHYLENE TEREPHTHALATE) IN DILUTE SOLUTION

1st EDITION

July 1970

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BRIEF HISTORY

The ISO Recommendation R 1228, *Plastics – Determination of viscosity number of poly(ethylene terephthalate) in dilute solution*, 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 a Draft ISO Recommendation No. 1002, which was circulated to all the ISO Member Bodies for enquiry in July 1966 and approved by the requisite majority. In view of the comments that were submitted by some Member Bodies, however, the text of the Draft ISO Recommendation was amended from the technical point of view. A second Draft ISO Recommendation No. 1002 was then circulated to all the ISO Member Bodies for enquiry in July 1968. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

No Member Body opposed the approval of the second Draft.

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

ISO Recommendation | R 1228 | July 1970

PLASTICS

DETERMINATION OF VISCOSITY NUMBER OF POLY(ETHYLENE TEREPHTHALATE) IN DILUTE SOLUTION

1. SCOPE

This ISO Recommendation describes 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 Recommendation R 1628, *Plastics – Directives for the standardisation of methods for the determination of the dilute solution viscosity of polymers.*

2. PRINCIPLE OF THE METHOD

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 %, freezing point higher than 80 °C.

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

One such solvent could be a mixture of phenol/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 on page 8, or any other viscometer meeting the requirements of ISO Recommendation R 1628, particularly Table 1.

4.5 *Vacuum oven,* pressure less than 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 0.0001 g.

4.9 *Stop-watch,* reading to 0.1 second.

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 three hours, at a pressure less than 1 mmHg, and then cool in a desiccator. Weigh 1.0 ± 0.002 g of sample to the nearest 0.0002 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 and 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 should be determined in advance; to achieve the desired 0.01 g/ml solution, the 1.0 ± 0.002 g of resin should be reduced proportionately to the reduced volume of solvent, or the displaced volume of solvent at 20 °C should 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, page 8) which should 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 should be such that, after draining, the level lies between the two filling marks. After not less than 15 minutes 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 should be taken as the mean of the two determinations which should not differ by more than 0.4 %. The mean time of flow of the solvent is determined in the same manner.