INTERNATIONAL STANDARD ISO 1233

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

Plastics – Determination of viscosity number of methyl methacrylate polymers and copolymers in dilute solution

Matières plastiques – Détermination de l'indice de viscosité des polymères et copolymères du méthacrylate de méthyle en solution diluée

First edition – 1975-09-15

UDC 678.744.3 : 532.13

Ref. No. ISO 1233-1975 (E)

Descriptors : plastics, viscosity index, polymers, copolymers, acrylic copolymers, methyl methacrylate, viscosimetric analysis.

Price based on 3 pages

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.

International Standard ISO 1233 (originally draft International Standard ISO/DIS 824) was drawn up by Technical Committee ISO/TC 61, *Plastics*, and circulated to the Member Bodies in August 1969.

It has been approved by the Member Bodies of the following countries :

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No Member Body expressed disapproval of the document.

Printed in Switzerland

Plastics — Determination of viscosity number of methyl methacrylate polymers and copolymers in dilute solution

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for determining the viscosity number of poly(methyl methacrylate), or copolymers in which the principal ingredient is methyl methacrylate, in dilute solution in chloroform.

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 are measured at 25 °C by conventional methods. The concentration of resin has to be chosen such that the ratio of the time of flow of the solution to the time of flow of the solvent is at least 1,2 and less than 1,4. The viscosity number is calculated from these measurements and from the known concentration of the solution. Density difference and kinetic energy errors are small in this method and corrections are not applied.

3 SOLVENT

Redistilled chloroform, the fraction distilling between 60,5 and 61,5 °C at a pressure of 1 013 mbar (760 mmHg) collected in a brown bottle and stored in the dark.

4 APPARATUS

4.1 Volumetric flask, 100 ml with ground glass stopper.

4.2 Sintered glass filter funnel, with 40 µm maximum pore diameter.

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

4.4 Viscometer, suspended level Ubbelohde type, of which the essential dimensions are as shown in the figure.[1]

4.5 Analytical balance, to weigh to an accuracy of 0,000 1 g.

4.6 Stop-watch, reading to the nearest 0,1 s.

5 PROCEDURE

5.1 Clean the viscometer (4.4) before it is used, and after discordant readings and at intervals during regular use. Use 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 by drawing through it a stream of air free from dust. Between successive satisfactory determinations, wash the viscometer with acetone and dry as described.

5.2 Weigh a quantity of resin between 0,2 and 0,3 g to the nearest 0,000 1 g and transfer it quantitatively to the volumetric flask (4.1). Add approximately 60 ml of solvent, taking care to avoid the formation of lumps, and insert the stopper.

For routine determinations and for the estimation of the proper concentration, shake the mixture gently until the resin has dissolved completely. The mixture may be warmed slightly to hasten solution. For reference determinations, shake the mixture gently until the resin has dissolved completely, then allow the solution to stand 16 ± 0,5 h. By means of thermostatic control, maintain the temperature of the solution at 25 ± 0,05 °C and add solvent at 25 ± 0,05 °C until the volume of the solution is exactly 100 ml.

The viscometric measurement should yield a flow time of the solution 20 to 40 % higher than the flow time of the solvent. If this requirement is not met at the initially chosen concentration of resin, the concentration has to be changed accordingly.

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1) In some cases, viscometer dimensions other than those given in the figure would be required to avoid shear rate effects. These dimensions shall then be stated explicitly in the test report. These dimensions shall permit the operating conditions prescribed in ISO/R 1628 to be respected.

5.3 The time of flow of the solution and of the solvent shall be determined in the same viscometer, suitably immersed in the same or other thermostatic bath, maintained at 25 ± 0,05 °C.

5.3.1 If an Ubbelohde viscometer is used, proceed as follows : filter the liquid through the sintered glass filter funnel (4.2) directly into tube (1) (see figure) of the viscometer immersed in the thermostatic bath (4.3) to a depth of approximately 20 mm above the upper graduation mark, supported so that the tube (2) is vertical.

The volume of liquid in the viscometer shall be such that the surface after draining lies between the two filling marks.

After not less than 10 min, blow the liquid with dust-free air, or draw it with a suction bulb, into the upper bulb until it reaches approximately the centre of that bulb. Place a finger over the tube (2) until the liquid flows away from the lower end of the capillary. Then remove the finger and measure the time-interval for the passage of the meniscus between the two graduation marks.

Blow or draw the liquid into the upper bulb and again measure the time of flow of the liquid.

5.3.2 The above procedure shall be suitably modified if a viscometer other than the Ubbelohde is used.

5.3.3 Take the time of flow of the solution as the mean of two determinations which do not differ by more than 0,4 % of the smaller result.

Repeat the test with a fresh solution if two (of four or fewer) results are obtained which differ by more than 0,4 % of the smaller result.

Determine the mean time of flow of the solvent in the same way.

NOTES

1 If the sample contains fillers, determine the quantity therein (for example by ashing of a sample) and use the corrected value for the concentration of the polymer in the calculation. The resulting solution may be clarified in a high-speed centrifuge.

2 Samples containing more than 1 % soluble additives should be extracted with a suitable solvent such as ethyl ether, petroleum ether or cyclohexane, dried free of the extracting solvent and then treated as described in clause 5. Such extraction may remove oligomers and complete removal of solvent is difficult. The results obtained in this way should be treated with caution. Calculate the corrected mass of polymer in the solution as in note 1.

6 EXPRESSION OF RESULTS

6.1 The result should preferably be expressed as viscosity number, calculated in millilitres per gram from the formula

$$\frac{t_s - t_o}{t_o C}$$

where

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

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

C is the concentration, in grams of resin per millilitre of solution.

6.2 The result can be expressed also as limiting viscosity number in the same units : millilitres per gram. This expression is useful for polymers whose different commercial grades require more than one concentration to be used (see clause 2).

The limiting viscosity number is calculated from the viscosity number by means of the approximate formula

$$\frac{\text{V.N.}}{1 + k\,(\text{V.N.} \times C)}$$

where

k is a constant conventionally taken as equal to 0,3;

V.N. is the viscosity number, in millilitres per gram;

C is the concentration, in grams of resin per millilitre of solution.

7 TEST REPORT

The test report shall include the following particulars :

a) complete identification of the sample tested;

b) treatments applied to the sample before testing, if any;

c) viscosity number, reported to the nearest whole number, and the concentration used;

d) all test conditions not specified in this International Standard and which may have affected the results;

e) porosity of the glass filter used;

f) the identification of the method of test, by reference to this International Standard or an equivalent national standard.

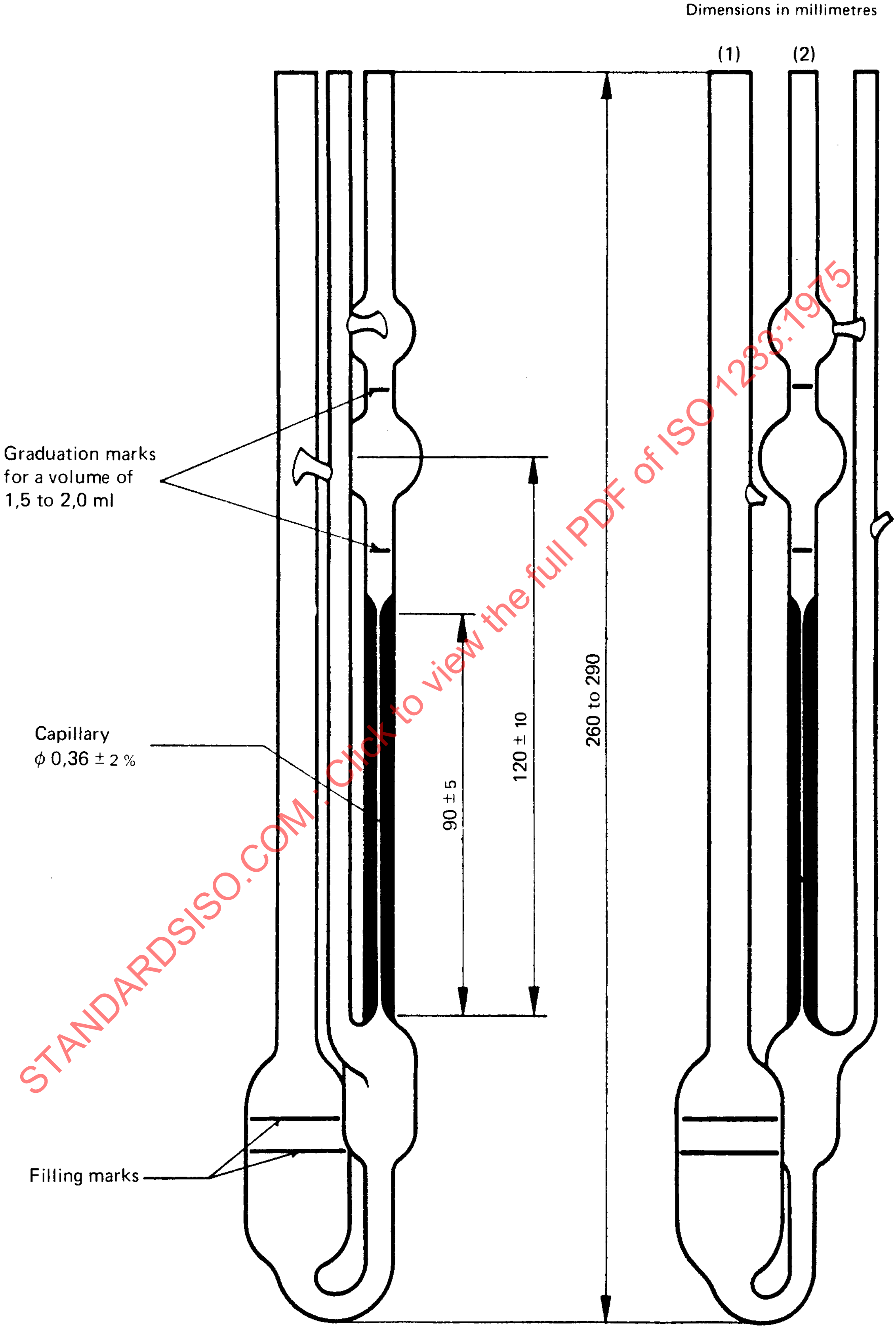

FIGURE – **Ubbelohde viscometer**