BUREAU OF INDIAN STANDARDS
DRAFT FOR COMMENTS ONLY
(*Not to be reproduced without permission of BIS or*
used as an Indian Standard)

भारतीय मानक मसौदा

प्लास्टिक — परीक्षण पद्धतियाँ
भाग 1 पॉलिमर संरचना और आकार का लक्षण वर्णन
अनुभाग 1 श्यानता से आणविक द्रव्यमान का निर्धारण
[IS 8543 (Part 1/Sec 1) का पहला *पुनरीक्षण*]

Draft Indian Standards

PLASTICS — METHODS OF TESTING
PART 1 CHARACTERIZATION OF POLYMER STRUCTURE AND SIZE
SECTION 1 DETERMINATION OF MOLECULAR MASS FROM VISCOSITY

[*first revision of IS 8543 (Part 1/Sec 1)*]

ICS 83.080

Methods of Sampling and Test for Plastics Sectional Committee, PCD 27	Last date for Comments: **12 September 2025**

FOREWORD

(*Formal clauses to be added later*)

This standard was originally published in 1978. This revision (first) has been undertaken to:

— update the cross referred standards; and
— make the editorial corrections.

Methods of determination of molecular mass may be divided into two classifications, namely, primary and secondary. The former includes osmotic pressure and other colligative techniques, electron microscopy, gel permeation chromatography, ultra-centrifugal and end-group analysis. In the secondary methods solution viscosity is widely used because of the simplicity of technique and apparatus. It is also used as a process control method. It is applicable to all polymers that dissolve completely without chemical reaction or degradation to form solutions that are stable at temperature between room temperature and about 150°C.

The determination of dilute-solution viscosity provides one item of information towards the molecular characterization of polymers. When viscosity data are used in conjunction with other molecular parameters, the properties of polymers depending on their molecular structure may be better predicted.

With certain restrictions, satisfactory correlations can be obtained between dilute-solution viscosity and molecular parameters, such as molecular mass or chain length. The most limiting restrictions which should be observed are as follows.

> It must be known that the polymers used to establish the correlations and those to which they are applied do not consist of or contain branched species. Basically a measure of molecular size and not molecular mass, the dilute-solution viscosity may be correlated appropriately with the molecular mass or chain length only if there is a unique relationship between the mass and the size of the dissolved polymer molecules, This is the case for linear, but not for most branched polymers. Caution should be exercised in applying empirical viscosity molecular mass relationships to polymers of unknown or uncertain structure since unsuspected branching may exist.
>
> For reasons similar to those outlined above, it must be required that the polymers to which the correlations are applied have the same chemical composition as those used in establishing the relationships.

For polymers meeting the restrictions of para 4, empirical relationships can be developed between the dilute-solution viscosity of a polymer and its hydrodynamic volume or average chain dimension (radius of gyration or end-to-end distance). Such relationships depend upon any variables influencing the molecular size of the dissolved polymer. The most important of these variables are solvent type and temperature. Thus, the solution viscosity of a given polymer specimen depends on the choice of these variables, and they must always be specified with the viscosity for complete identification. The further empirical interpretation of viscosity data in terms of molecular mass rather than size requires control of the variables described in para 4**.**

The solution viscosity of a polymer of sufficiently high molecular mass may depend on rate of shear in the viscometer, and the viscosity of a polyelectrolyte (polymer containing ionisable chemical groupings) will depend on the composition and ionic strength of the solvent. Special precautions beyond the scope of this method are required when measuring such polymers.

Finally, the viscosity of polymer solutions may be affected drastically by the presence of recognized or unrecognized additives in the sample, including but not limited to colourants, fillers, or low-molecular mass species.

In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2: 2022 'Rules for rounding off numerical values (*second revision*)'.

1 SCOPE

This standard (Part 1/ Sec 1) prescribes method for determination of the dilute-solution viscosity of polymers and the calculation of molecular mass.

2 REFERENCES

The standards given below contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent edition of these standards.

IS No.	*Title*
IS 2828 : 2019/ ISO 472: 2013	Plastics - Vocabulary (*second revision*)
IS 1070 : 2023	Reagent Grade Water Specification (*fourth revision*)
IS 4825 : 1982	Specification for liquid - In - Glass solid - Stem reference thermometers (*first revision*)

3. TERMINOLOGY

For the purpose of this standard, definitions given in IS 2828 shall apply.

4 QUALITY OF REAGENTS

4.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals which do not contain impurities that affect the results of analysis.

5 APPARATUS

5.1 Volumetric Flasks, 100 ml or other size found convenient.

5.2 Transfer Pipettes, sizes between 1 and 25 ml, as required. Transfer pipettes for use with polymer solutions should have about 2 mm cut from their lower tips to permit more rapid transfer of the solution to the viscometer.

5.3 Constant Temperature Bath, capable of maintaining ± 0.1°C at the desired operating temperature (usually between 25 °C and 135 °C). Less stringent temperature control (± 0.2°C) is satisfactory upon demonstration that the precision of the results is not affected.

5.4 Viscometer, glass, capillary type, efflux time for the solvent and temperature used shall be greater than 200 s (except that efflux time for semi-micro viscometers shall be greater than 80 s), to eliminate the need for kinetic-energy corrections.

NOTE — Two types of viscometers, commonly used are as follows:

a) The first type is a constant volume device of simple construction, recommended for use where solution viscosity is to be measured at a single concentration, as for determination of the viscosity number or logarithmic viscosity number. It may also serve for the determination of the limiting viscosity number through measurement of several solutions having different concentrations.

b) The second type of viscometer, commonly called a dilution viscometer, is a time-saving device for the determination of limiting viscosity number since it does not require constant liquid volume for operation. Several concentrations of a polymer solution can be tested by adding a known quantity of the solvent at the test temperature directly to the viscometer, mixing, measuring the viscosity and then making the next dilution. The viscosity of the pure solvent shall be measured separately.

c) An alternative procedure is to start with the minimum volume of the pure solvent, then add aliquots of a concentrated stock solution to the viscometer to obtain values of the viscosity ratio at successively higher concentrations. The choice of procedures is dictated by the range of volumes with which the viscometer operates and the range of concentrations desired for test.

5.5 Timer, graduated in divisions of 0.1 s or less.

5.6 Thermometer, suitable for the specified test temperature and conforming to IS 4825.

5.7 Fritted Glass Filter Funnel, coarse grade.

6 MATERIAL

6.1 Solvents as required or as recommended in Annex A.

6.2 Heat Transfer Liquid, for constant temperature bath.

NOTES:

a) The following materials have been found suitable as heat transfer liquids:
  i) Silicone oil;
  ii) Mineral oil;
  iii) Groundnut oil;
  iv) Water; and
  v) Water-miscible liquid, such as glycerol or ethylene glycol.

b) The material selected shall not discolour or smoke on prolonged selected exposure at the test temperature, in some cases, discolouring may be inhibited by the use of an antioxidant. The use of water or water miscible liquid facilitates cleaning glassware used in the test.

6.3 Nitrogen, for purging.

7 PREPARATION OF SAMPLE

7.1 The sample need not be pre-dried or conditioned unless previous specific knowledge, for example that the material is known to be hygroscopic, makes such pre-treatment desirable (*see* Annex A).

7.2 If it is known that the sample dissolves only slowly in the selected solvent, pre-treatment to reduce the particle size of the sample is advisable.

NOTE — Some samples can be pulverized conveniently in a rotary cutting mill with a 20-mesh screen at the outlet of its pulverizing chamber. Care should be taken to avoid overheating the sample during pulverization, which might lead to thermal degradation. Low-melting polymers, or hard, tough samples, often may be satisfactorily pulverized only at very low temperature, for example, in the presence of dry ice or liquid nitrogen (*see* Annex A).

8 PROCEDURE

8.1 Weigh the sample (*see* Annex A for size and tolerance) in a tared 100 ml volumetric flask (or weigh and transfer quantitatively to the flask). If the sample is known to oxidize easily in the subsequent dissolving step, the flask may be purged with nitrogen.

NOTES:

1) Other sizes of volumetric flasks may be used, depending on viscometer size and amount of sample available. If another size flask is used, adjust the sample weights, solvent and solution volumes accordingly.

2) For greatest reliability of results and in the absence of specific information (*see* Annex A), select the sample size on the basis of experience to give a viscosity ratio near 1.5. If several concentrations of a solution of a single sample are to be used (*see* Note 3), select them so that the viscosity ratio falls in the range from l.2 to 2.0.

3) Preparation of a single solution may often suffice, either for determining the viscosity ratio or logarithmic viscosity number, or as a stock solution for use in a dilution viscometer to determine the limiting viscosity number. If more than one solution concentration is desired, weigh a series of specimens (often four) into separate flask, selecting the specimen weights to give the desired solution concentration. Duplicate specimens may be prepared, if desired.

8.2 Add approximately 50 ml of solvent to each solution flask, purge with nitrogen, if necessary, and shake the flask once every 10 min until solution is complete (alternatively, use a laboratory shaker). Elevated temperature may enhance solution rate (*see* Annex A) but should be used with caution since some polymers and solvents have limited high temperature stability. If solution is carried out at an elevated temperature, subject the flask of pure solvent to the same conditions as the polymer Solution.

NOTE — Complete solution of all the specimens is essential. When solution appears complete, examine the flask with care to be sure that no undissolved material, gel particles, or foreign matter is present.

8.3 Place the volumetric flask containing the solution and the pure solvent in the constant temperature bath maintained at the test temperature. Add solvent to the solution flask to a total volume of about 99 ml using solvent maintained at the bath temperature. Mix the solution thoroughly, and replace in the bath. After temperature equilibrium has been achieved (10 to 30 min), complete the dilution to the 100 ml mark by adding solvent maintained at the test temperature, using a transfer pipette. Mix the contents of the flask thoroughly.

NOTE — Be sure that the solution is uniformly mixed. If the test temperature is above ambient, avoid cooling the flask excessively while mixing.

8.4 Where necessary to prevent oxidation, purge the viscometer with a slow stream of nitrogen with the viscometer permanently positioned in the constant temperature bath at the required temperature, transfer a suitable amount of solution into the viscometer using a suitably modified transfer pipette. The amount varies with the type of viscometer used. If necessary, filter the solution through a fritted glass filter or equivalent into the viscometer taking care not to lose solvent in the process (pressure filtration). Filtration is usually desirable.

NOTE — If the solution is to be handled at elevated temperatures, the transfer pipette may be fitted with a suitable heating mantle to retard precipitation of polymer from the solution during the transfer.

8.5 After temperature equilibrium has been achieved (a minimum of 10 min), bring the liquid level in the viscometer above the upper graduation mark by means of gentle air (or preferably, nitrogen) pressure applied to the arm opposite to the capillary. Allow the solution to drain through the capillary. Start the timer exactly as the meniscus passes the upper graduation mark and stop it exactly as the meniscus passes the lower mark.

8.6 Determine the efflux time at least three times each for the solution and for the pure solvent. Three consecutive readings should agree to within 0.1 s or 0.1 percent of their mean, whichever is greater. Larger variations may result from foreign material in the viscometer or from inadequate temperature control and require repetition of the experiment after their cause is located and corrected.

9 CALCULATIONS

9.1 Viscosity Ratio

Calculate the viscosity ratio from the average efflux time for the solvent, t_o, and the average efflux time for the solution t, as follows:

$$\text{Viscosity ratio} = t/t_o$$

NOTE — Strictly, the viscosity ratio is defined as η / η_o where η, η_o are the viscosities of the solution and solvent respectively, and are related to the corresponding efflux time by:

$$\eta = Ct\sigma - E\sigma/t^2$$
$$\eta_o = Ct_o\sigma_o - E\sigma_o/t_o^2$$

where C and E are constants for the particular viscometer used. The equation in **9.1** follows if the second term in these relations, a kinetic-energy correction, is negligible, and the respective solvent and solution densities, σ_o and σ are substantially equal. The kinetic-energy correction is negligible for the recommended viscometers and efflux times.

9.2 Logarithmic Viscosity Number

Calculate the logarithmic viscosity ratio for each solution concentration as follows:

$$\text{Logarithmic Viscosity Number} = l_n\,(\eta/\,\eta_o)/C$$

Where

$l_n(\eta/\,\eta_o)$ is the natural logarithm of the viscosity ratio; and

C is the solution concentration in grams per millilitre (grams per millilitre of solution).

The units of logarithmic viscosity number are, therefore, ml/ g.

9.3 Limiting Viscosity Number

Plot the logarithmic viscosity number versus concentration, for several solution concentrations, on rectilinear graph paper. Draw the best straight line through the points and extrapolate it to zero concentration. The limiting viscosity number (η) is the intercept of the line at zero concentration.

NOTES:

1) The viscosity number $(\eta - \eta_o)/ \eta_o C$ may be calculated and plotted versus concentration on the same graph with the logarithmic viscosity number. The two lines should extrapolate to the same point (the limiting viscosity number) at C = 0; plotting both functions may serve to fix the limiting viscosity number with greater accuracy. If the limitation on viscosity ratio stated in Note 2 (**8.1**) is observed, the extrapolation lines should be accurately straight.

2**)** For some polymer-solvent systems, it may be demonstrated that the slopes of the lines of viscosity number and logarithmic viscosity number versus concentration are closely similar for all samples normally encountered.

10 PRECISION AND ACCURACY

For most polymer-solvent systems, the logarithmic and limiting viscosity numbers may be determined with a standard deviation for reproducibility of about 1.0 ml/ g. Since the viscosity numbers are available only from these measurements, accuracy of their values has no meaning.

11 REPORT

The report shall include the following.

a) Complete identification of the sample tested.
b) Conditioning procedure, if any.
c) One or more of the following:

  i) The viscosity ratio, given to one significant figure beyond the decimal point followed by the concentration of the test solution in g/ ml.
  ii) The logarithmic viscosity number in ml/ g carried to one significant figure beyond the decimal point followed by the concentration of the test solution in g/ ml.
  iii) The limiting viscosity number in ml/ g, carried to the decimal point.

d) The solvent employed and the test temperature.

ANNEX A

(*Clauses* 6.1 and 7)

DETAILS OF MATERIALS AND PROCEDURES FOR IMPORTANT POLYMER TYPES

A-1 POLYAMIDE

A-1.1 Recommended solvents are formic acid (90 ± 0.2 percent in water) or m-cresol, melting point 11 to 12°C.

A-1.2 Recommended dissolving conditions are for formic acid, 30°C; for *m*-cresol, 2 h, *Max* at 95 °C to 100 °C or 8 h at 50 °C.

A-1.3 The recommended test temperature is 27 ± 0.l °C.

A-1.4 The recommended solution concentration is 0.005 ± 0.00002 g/ ml.

A-2 POLYCARBONATE

A-2.1 Recommended solvents are methylene chloride (purified grade) and dry *p*-dioxane, 60 °C.

A-2.2 Recommended dissolving temperatures are; for methylene chloride, 30 °C; for p-dioxane, 60 °C.

A-2.3 If pigments or other fillers are present, the resin shall be dissolved in methylene chloride and the solution filtered. The methylene chloride shall be evaporated from the resulting clear solution to leave the resin in the form of a thin film. After this has been dried for several hours at 125 °C, samples shall be taken for viscosity measurement.

A-2.4 The recommended test temperature is 27 ± 0.l °C.

A-2.5 The recommended solution concentration is 0.0040 ± 0.00 02 g/ ml, or by convenient dilution from 0.010 & 0.000 02 g/ ml.

A-3 POLY(ETHYLENE) TEREPHTHALATE

A-3.1 Recommended solvents are *o*-chlorophenol (melting point 0 °C) or a mixture of 1 part trichloroacetic acid and 3 parts dichloromethane. Solvents should contain less than 0.15 percent water.

> CAUTION — Since *p*-chlorophenol is toxic, appropriate measures must be taken to avoid breathing its vapours or contact of the liquid with skin or eyes.

A-3.2 The samples should be dried to a water content of 0.03 percent or less. Drying may be carried out at 100°C for about 3 h under a pressure of 1 mmHg or less.

A-3.3 Recommended dissolving conditions are 2 to 3 h at 90 °C to 100 °C.

A-3.4 The recommended test temperature is 27 ± 0.l °C.

A-3.5 The recommended solution concentration is 0.0050 ± 0.000 2 g/ ml.

A-4 POLY METHYLMETHACRYLATE

A-4.1 Recommended solvent is 1, 2-dichloroethane (ethylene dichloride), reagent grade.

A-4.2 Pre-treatment of the sample should not include cutting or grinding in such a way as to cause shear degradation of the polymer.

A-4.3 Recommended dissolving conditions are 24 h at 30 °C.

A-4.4 The recommended solution concentration is 0.0020 ± 0.000 02 g/ml; but the recommendations of note under **8.2** should be construed to supersede this.

NOTE — The limiting viscosity number may be estimated from data obtained at a single concentration by use of the following equation:

$$(\eta) = (2^{1/2}/C)\,[\eta/\,\eta_o - 1 - l_a - (\eta - \eta_o)]^{1/2}$$ (*see* 9.1 and 9.2 for explanation)

A-5 POLY(VINYLCHLORIDE)

A-5.1 The recommended solvent is cyclohexanone, boiling point 155 °C to 156 °C (760 mmHg).

A-5.2 Samples should be dried to a moisture content below 0.1 percent.

A-5.3 The recommended dissolving temperature is 85 ± 10 °C.

A-5.4 The recommended test temperature is 27 ± 0.l °C.

A-5.5 The recommended solution concentration is 0.0020 ± 0.000 02 g/ ml.

NOTE — The limiting viscosity number may be estimated from data obtained at a single concentration by use of the following equation:

$$(\eta) = \tfrac{1}{4}\,[(\eta - \eta_o)/\,\eta_o C + \tfrac{3}{4}\,[l_n - (\eta/\eta_o)\,C]$$ (*see* **9.1** and **9.2** for explanation)

A-5.6 Precision

A-5.6.1 *Repeatability* — Duplicate results by the same analyst should not be considered suspect unless they differ by more than 1 percent.

A-5.6.2 *Reproducibility* — The average result of two determinations reported by one laboratory should not be considered suspect unless it differs from that of another laboratory by more than 2 percent.