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International Standard 1133

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Plastics — Determination of the melt flow rate of thermoplastics

Plastiques — Détermination de l'indice de fluidité à chaud des thermoplastiques

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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.

International Standard ISO 1133 was developed by Technical Committee ISO/TC 61, *Plastics*, and was circulated to the member bodies in December 1979.

It has been approved by the member bodies of the following countries:

Australia	France	Romania
Austria	Germany, F. R.	South Africa, Rep. of
Belgium	Hungary	Spain
Brazil	India	Sweden
Canada	Italy	Switzerland
China	Japan	United Kingdom
Czechoslovakia	Korea, Rep. of	USA
Egypt, Arab Rep. of	Netherlands	USSR
Finland	Poland	

No member body expressed disapproval of the document.

This International Standard cancels and replaces ISO Recommendations R 292-1967 and R 1133-1969, of which it constitutes a technical revision.

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Plastics — Determination of the melt flow rate of thermoplastics

1 Scope and field of application

1.1 This International Standard specifies a method for the determination of the melt flow rate of molten thermoplastic materials under specified conditions of temperature and pressure. When the test conditions are specified in a material specification, this method may be used to characterize the melt flow rate of the material.

NOTE — Test conditions commonly used are listed in the annex.

1.2 The melt flow rate of high polymers is dependent on the rate of shear. The rates of shear in this test are much smaller than those used under normal conditions of fabrication, and therefore data obtained by this method for various thermoplastics may not always correlate with their behaviour in actual use.

The method is useful in quality control.

1.3 When the method is used with certain materials, consideration should be given to the factors leading to the loss of reproducibility. Such factors include

a) thermal degradation or crosslinking of the material, causing the melt flow rate to change during preheating or the period of test; powdered materials requiring long preheating times are sensitive to this effect. In certain cases the inclusion of stabilizers is necessary to reduce the variability;

b) filled or reinforced materials, where the distribution or orientation of the filler may affect the melt flow rate.

2 References

ISO/R 81, *Vickers hardness test for steel (load 5 to 100 kgf).*

ISO 468, *Surface roughness — Parameters, their values and general rules for specifying requirements.*

ISO 1622/1, *Plastics — Polystyrene moulding and extrusion materials — Part 1: Designation*[1]

ISO 1872/1, *Plastics — Polyethylene and ethylene-copolymer thermoplastics.*[2]

ISO 1873/1, *Plastics — Polypropylene and propylene-copolymer thermoplastics — Part 1 : Designation.*

ISO 2580/1, *Plastics — Acrylonitrile butadiene styrene (ABS) moulding and extrusion materials — Part 1 : Designation.*

ISO 2897/1, *Plastics — Impact-resistant polystyrenes — Part 1: Designation.*[3]

ISO 4613/1, *Plastics — Ethylene/vinyl acetate (E/VAC) copolymer material — Part 1: Designation.*[4]

ISO 4894/1, *Plastics — Styrene/acrylonitrile (SAN) copolymer moulding and extrusion materials — Part 1 : Designation.*

1) At present ISO 1622-1975, *Plastics — Polystyrene moulding and extrusion materials — Designation.*

2) At present at the stage of draft. (Revision of ISO 1872-1972.)

3) At present ISO 2897-1976, *Plastics — Designation of impact-resistant polystyrenes.*

4) At present at the stage of draft.

3 Apparatus

3.1 Basic apparatus

The apparatus is basically an extrusion plastometer operating at a fixed temperature (capillary rheometer). The general design is as shown in the figure. The thermoplastic material, which is contained in a vertical metal cylinder, is extruded through a die by a loaded piston. The apparatus consists of the following essential parts.

3.1.1 Steel cylinder fixed in a vertical position and suitably insulated for operation up to 300 °C. The cylinder length shall be between 115 and 180 mm and the internal diameter between 9,500 and 10,000 mm. The bore of the cylinder shall be uniform to within ± 0,025 mm. The base of the cylinder shall be thermally insulated in such a way that the area of the exposed metal is less than 4 cm^2 and it is recommended that an insulating material such as polytetrafluoroethylene (thickness about 3 mm) be used in order to avoid sticking of the extrudate.

The bore shall be suitably hardened to no less than 500 Vickers hardness (HV) (see ISO/R 81) and shall have a surface finish better than R_a 0,25 µm (arithmetical mean deviation, see ISO 468). When loads greater than 12,500 kg are used, an additional piston guide should be provided.

3.1.2 Steel piston (see notes 1 and 2), whose working length is at least as long as the cylinder. The piston shall have a head 6,35 ± 0,10 mm in length. The diameter of the head shall be less than the internal diameter of the cylinder by 0,075 ± 0,015 mm. Furthermore, it is necessary to know the diameter of the head with an accuracy of ± 0,025 mm in order to make the calculation required by 3.1.6. The lower edge of the head shall have a radius of 0,4 mm and the upper edge shall have its sharp edge removed. Above the head, the piston shall be relieved to about 9 mm diameter. A stud may be added at the top of the piston to support the removable load, but the piston shall be thermally insulated from the load. Along the piston stem, two thin annular reference marks shall be scribed 30 mm apart and so positioned that the upper one is aligned with the top of the cylinder when the distance between the lower edge of the piston head and the top of the die is 20 mm (see note 3).

NOTES

1 To ensure satisfactory operation of the apparatus, the cylinder and the piston should be made of steel of different hardnesses. It is convenient for ease of maintenance and renewal to make the cylinder of the harder steel.

2 The piston may be either hollow or solid. In tests with lower loads the piston should be hollow, otherwise it may not be possible to obtain the lowest prescribed load. When the test is performed with the higher loads, the hollow piston is not desirable, as the higher load may distort such a piston. In such tests a solid piston or a hollow piston with suitable guides should be used. When using this latter modification, it is essential that the heat loss along the piston, which is generally longer than usual, does not alter the test temperature of the material.

3 The annular reference marks show the length of cylinder within which all useful cut-offs should be taken.

3.1.3 Heating and **thermostatic devices**, such that the selected temperature of the material in the cylinder can be maintained within ± 0,5 K. Automatic temperature control is strongly recommended.

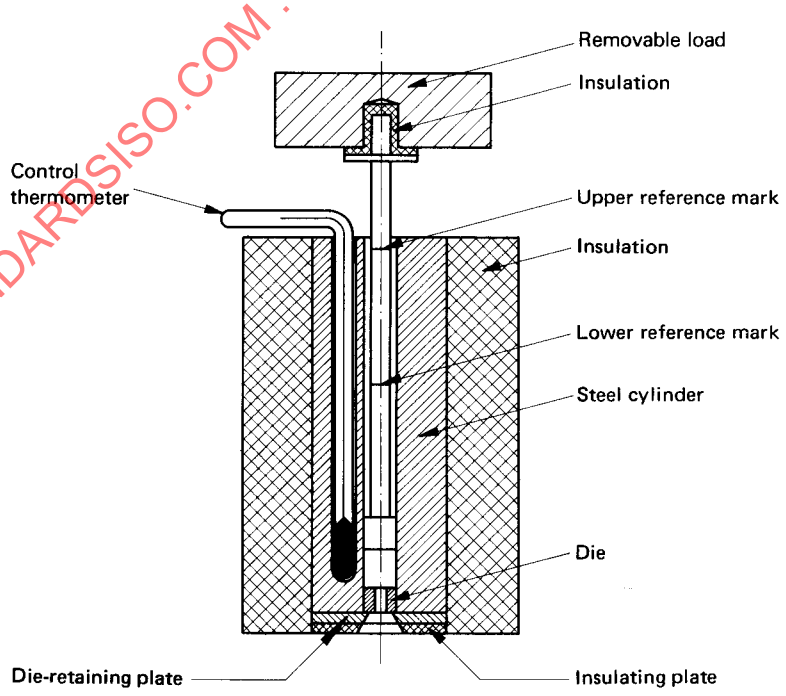

Figure — Typical apparatus for determining melt flow rate (showing one of the possible methods of retaining the die and one type of piston)

3.1.4 Mercury-in-glass thermometer (control thermometer) or another temperature-measuring device, located as close as possible to the hole of the cylinder and 15 mm from the base. This measuring device shall be calibrated to permit temperature measurement to ± 0,1 K. A heat-transfer medium, such as a suitable low-melting alloy or silicone oil, may be used between the thermometer and the cylinder.

3.1.5 Dies, made of tungsten carbide or hardened steel, 8,000 ± 0,025 mm in length. The internal diameter shall be chosen according to the relevant specifications (see the annex) for each material but it shall be circular, straight and uniform in diameter such that in all positions it is within ± 0,005 mm of a true cylinder of the declared nominal diameter.

The bore shall be suitably hardened to no less than 500 Vickers hardness (HV) (see ISO/R 81) and shall have a surface finish better than R_a 0,25 µm (arithmetical mean deviation, see ISO 468). The die shall not project beyond the base of the cylinder (see the figure) and shall be mounted so that the bore is co-axial with that of the cylinder bore.

3.1.6 Removable load, on the top of the piston, which consists of a set of weights which may be adjusted so that the combined mass, M, of the load and the piston, in grams, with a tolerance of ± 0,5 % is given by the equation

$$M = K\frac{D^2}{d^4}$$

where

K is a die factor. The value of K depends on the die dimensions and corresponds to a range of shear rates (see the annex);

D is the measured diameter of the piston head, in millimetres, to ± 0,025 mm;

d is the measured diameter of the die, in millimetres, to ± 0,005 mm.

3.1.7 Means of setting and maintaining the cylinder truly vertical. A two-directional bubble level, set normal to the cylinder axis and adjustable supports for the apparatus are suitable for the purpose.

NOTE — This is to avoid excessive friction caused by the piston or bending under heavy loads.

3.2 Accessory equipment

3.2.1 Equipment for introducing samples into the cylinder : packing rod made of nonabrasive material.

3.2.2 Tool for cutting off the extruded sample. A sharp-edged spatula has been found suitable.

3.2.3 Cleaning equipment.

3.2.4 Stop-watch accurate to ± 0,1 s.

3.2.5 Balance, accurate to ± 0,000 5 g.

3.2.6 Mercury-in-glass thermometer (calibration thermometer) or another temperature measuring device. This measuring device shall be calibrated to permit temperature measurement to ± 0,1 K at the temperature and immersion conditions to be used when calibrating the control thermometer (3.1.4) in accordance with 5.1.

3.2.7 An alternative **mechanical loading device** may be used for the higher loads.

4 Test specimen

4.1 The test specimen may be in any form that can be introduced into the bore of the cylinder, for example powder, granules, strips of films.

NOTE — Some materials in powder form do not give a bubble-free filament if they are not previously pressed.

4.2 The test specimen shall be conditioned and, if necessary, stabilized prior to the test, in accordance with the material specifications.

5 Temperature calibration, cleaning and maintenance of the apparatus

5.1 Verify the accuracy of the control thermometer at least once each day that the apparatus is used or whenever the temperature of test is changed, whichever is the more frequent. For this purpose, adjust the cylinder heating and control systems until the cylinder will remain at the required temperature as indicated by the control thermometer. Preheat a calibration thermometer to the same temperature. Then charge the cylinder with a sample of the material to be tested, or a material representative thereof (see the note), using the same technique as for a test (see 6.2). Four minutes after completing the charging of the material, the calibration thermometer shall be then introduced into the cylinder hold and immersed in the material therein until the tip of the bulb is 10 mm from the upper face of the die. After a further interval of at least 4 min, the temperature indicated by the control thermometer shall then be corrected by algebraic addition of the difference between the temperatures read on the two thermometers.

NOTE — It is essential that the material used during calibration be sufficiently fluid to permit, for instance, a mercury-filled thermometer bulb, to be introduced without excessive force and risk of damage. A material with an MFR of greater than 45 (2,095 mm bore die; 21,2 N; 600 s) at the temperature of calibration has been found suitable.

If such a material is used for calibration purpose in place of a more viscous material which is to be tested, the dummy material should have a thermal diffusivity similar to that of the material to be tested, so that warm-up behaviour is similar. It is necessary that the quantity charged for calibration be such that when the calibration thermometer is subsequently introduced, an appropriate portion of the thermometer is immersed for accurate temperature measurement. This can be checked by inspecting the level of material coating the end of the calibration thermometer after eventual removal of that thermometer from the cylinder.

5.2 The apparatus shall be cleaned thoroughly after each determination. The cylinder may be cleaned with cloth patches. The piston shall be cleaned while hot with a cloth and a suitable solvent. The die may be cleaned with a closely fitting brass reamer or wooden peg, followed by immersion in boiling solvent. Pyrolytic cleaning in a nitrogen atmosphere at about 550 °C may also be used. On no account shall abrasives or materials likely to damage the surface of the piston, cylinder, or die be used. Special precautions shall be taken to avoid exposure to toxic fumes when using solvents at high temperatures.

5.3 It is recommended that, at fairly frequent intervals, for example once a week for instruments in constant use, the insulating plate and the die-retaining plate, if fitted as in the figure, should be removed, and the cylinder cleaned throughout.

6 Procedure

6.1 Clean the apparatus (see clause 5). Before beginning a series of tests, ensure that the cylinder and piston have been at the selected temperature for not less than 15 min.

6.2 Then charge the cylinder with 4 to 8 g of the sample according to the anticipated melt flow rate (see, for example, table 1). During the charging, compress the material with the packing rod (3.2.1) by using hand pressure. To ensure a charge as free from air as possible for material susceptible to oxidative degradation, complete the charging process in 1 min. Put the piston, loaded or unloaded according to the flow rate of the material, in the cylinder.

NOTE — If the melt flow rate of the material is high, that is, more than 10 g/600 s, the loss of sample while pre-heating will be appreciable. In this case, an unloaded piston or one carrying a smaller weight may be used during the pre-heating period, and then changed to the desired weight at the end of the 4 min pre-heating time.

Table 1

Melt flow rate	Mass of the sample in the cylinder*	Time-interval
g/600 s	g	s
0,1 to 0,5	4 to 5	240**
> 0,5 to 1	4 to 5	120
> 1 to 3,5	4 to 5	60
> 3,5 to 10	6 to 8	30
> 10	6 to 8	5 to 15***

* When the density of the material is greater than 1,0 g/cm^3, it may be necessary to increase the mass of the sample.

** It is recommended that melt flow rate should not be measured if the value obtained in this test is less than 0,1 g/600 s.

*** To achieve adequate reproducibility when testing materials having an MFR greater than 25 g/600 s, it may be necessary either to control and measure cut off intervals automatically to less than 0,1 s or to derive melt flow data from measurements of piston displacement rates and knowledge of the density of the material at the temperature of test.

6.3 Four minutes after completing the introduction of the sample, during which time the temperature shall have returned to that selected, place the selected load on the piston, if it was unloaded or under-loaded. Depending on the actual viscosity of the material, allow the piston to descend under gravity or push it down faster using hand pressure, until a bubble-free filament is extruded and the lower mark is 5 to 10 mm above the top edge of the cylinder. The time for this operation shall not exceed 1 min. Cut off and discard the extrudate. Then allow the loaded piston to descend under gravity. When the lower reference mark has reached the top edge of the cylinder, record the time, cut off simultaneously the extruded portion with the cutting tool and again discard.

Then collect successive cut-offs in order to measure the extrusion rate, at time-intervals depending on the melt flow rate so chosen that the length of a single cut-off is not less than 10 mm and preferably between 10 and 20 mm (see time-intervals in table 1 as a guide).

Stop cutting when the upper mark of the piston stem reaches the top edge of the cylinder. Discard any cut-off containing visible air bubbles. After cooling, weigh individually, to the nearest 0,001 g, the remaining cut-offs, which shall number at least three, and calculate their average mass. If the difference between the maximum and the minimum value of the individual weighings exceeds 15 % of the average, discard the result and repeat the test on a fresh portion of the sample.

7 Expression of results

The melt flow rate (MFR) expressed in grams per the reference time, is given by the equation

$$\mathrm{MFR}\ (T, M) = \frac{S \times m}{t}$$

where

T is the test temperature, in degrees Celsius;

M is the nominal load, in kilograms (the actual load is calculated according to 3.1.6);

S is the reference time, in seconds; this is the chosen time (see the annex) to which the cut-off time intervals are related;

m is the average mass of the cut-offs, in grams;

t is the cut-off time-interval, in seconds.

NOTES

1 The diameter of the die used shall be stated if it is other than 2,090 to 2,100 mm.

2 The reference time chosen shall be stated if it is other than 600 s.

Express the result to two significant figures.

8 Test report

The test report shall include the following particulars :

a) reference to this International Standard;