61

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Plastics — Determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastics

Plastiques — Détermination de l'indice de fluidité à chaud des thermoplastiques, en masse (MFR) et en volume (MVR)

Reference number
ISO 1133:1991(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 1133 was prepared by Technical Committee ISO/TC 61, *Plastics*.

This second edition cancels and replaces the first edition (ISO 1133:1981). This new edition includes, in addition to the previously described method for the determination of the melt mass-flow rate, a new procedure for the automatic measurement of both melt mass-flow rate and melt volume-flow rate.

Annex A forms an integral part of this International Standard.

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Plastics — Determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastics

1 Scope

1.1 This International Standard specifies a method for the determination of the melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) of thermoplastic materials under specified conditions of temperature and load. Normally, the test conditions for measurement of melt flow rate are specified in the material standard with a reference to this International Standard. The test conditions normally used for thermoplastics are listed in annex A. The melt volume-flow rate will normally be found useful when comparing filled and unfilled thermoplastics. The melt mass-flow rate can now be determined by automatic measurement provided the melt density at the test temperature is known.

1.2 The melt mass-flow rate and melt volume-flow rate of thermoplastics are 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.

Both methods are useful in quality control.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 1622-1:1985, *Plastics — Polystyrene (PS) moulding and extrusion materials — Part 1: Designation.*

ISO 1872-1:1986, *Plastics — Polyethylene (PE) and ethylene copolymer thermoplastics — Part 1: Designation.*

ISO 1873-1:1986, *Plastics — Polypropylene (PP) and propylene-copolymer thermoplastics — Part 1: Designation.*

ISO 2580-1:1990, *Plastics — Acrylonitrile/butadiene/styrene (ABS) moulding and extrusion materials — Part 1: Designation.*

ISO 2897-1:1990, *Plastics — Impact-resistant polystyrene (SB) moulding and extrusion materials — Part 1: Designation.*

ISO 4613-1:1985, *Plastics — Ethylene/vinyl acetate copolymer thermoplastics (E/VAC) — Part 1: Designation.*

ISO 4894-1:1990, *Plastics — Styrene/acrylonitrile (SAN) copolymer moulding and extrusion materials — Part 1: Designation.*

ISO 6402-1:1990, *Plastics — Impact-resistant acrylonitrile/styrene moulding and extrusion materials (ASA, AES, ACS), excluding butadiene-modified materials — Part 1: Designation.*

ISO 6507-1:1982, *Metallic materials — Hardness test — Vickers test — Part 1: HV 5 to HV 100.*

ISO 7391-1:1987, *Plastics — Polycarbonate moulding and extrusion materials — Part 1: Designation.*

ISO 7792-2:1988, *Plastics — Polyalkylene terephthalates — Part 2: Preparation of test specimens and determination of properties.*

ISO 8257-1:1987, *Plastics — Poly(methyl methacrylate) (PMMA) moulding and extrusion materials — Part 1: Designation.*

ISO 9988-1:1991, *Plastics — Polyoxymethylene (POM) moulding and extrusion materials — Part 1: Designation.*

3 Apparatus

3.1 Basic apparatus

The apparatus is basically an extrusion plastometer (capillary rheometer) operating at a fixed temperature. The general design is as shown in figure 1. 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 400 °C. The cylinder length shall be between 115 mm and 180 mm and the internal diameter 9,55 mm ± 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 Al_2O_3 ceramic fibre or another suitable material be used in order to avoid sticking of the extrudate.

The bore shall be suitably hardened to a Vickers hardness of no less than 500 (HV 5 to HV 100) (see ISO 6507-1). A piston guide shall be provided to prevent additional friction caused by misalignment of the piston.

3.1.2 Steel piston, having a working length at least as long as the cylinder. The piston shall have a head 6,35 mm ± 0,1 mm in length. The diameter of the head shall be less than the internal diameter of the cylinder by 0,075 mm ± 0,01 mm. 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. These annular marks on the piston are used as reference points during the determination (see 6.3 and 7.4.3).

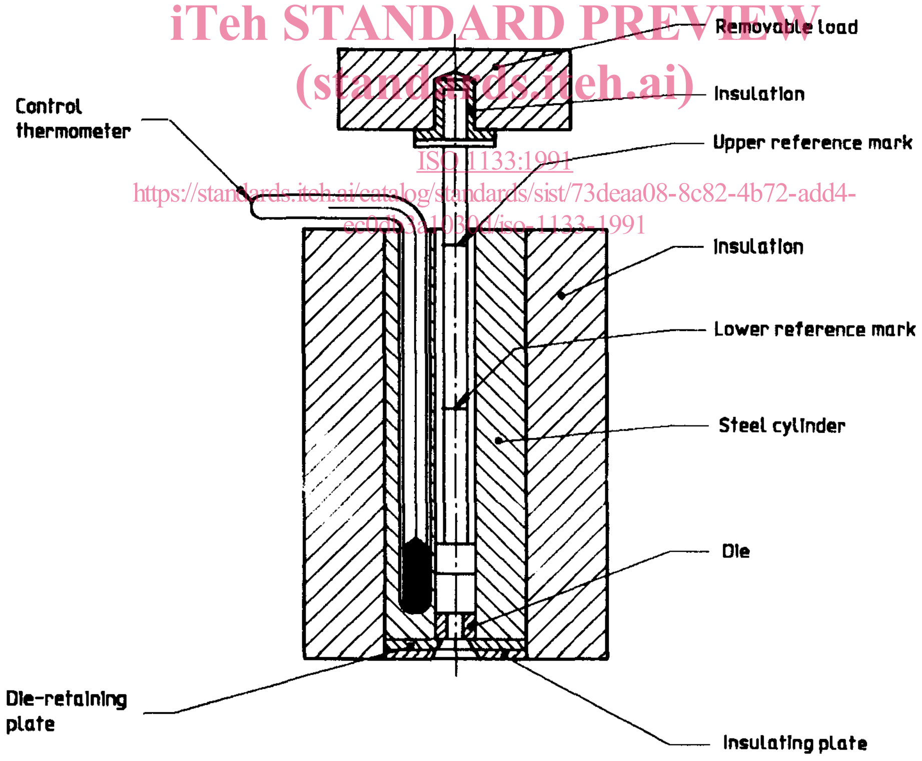

Figure 1 — Typical apparatus for determining melt flow rate (showing one of the possible methods of retaining the die and one type of piston)

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

The piston may be either hollow or solid. In tests with lower loads, the piston shall 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 shall 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.

3.1.3 Temperature-control system, such that the selected temperature of the material in the cylinder can be maintained to within ± 0,5 °C. Automatic temperature control is strongly recommended.

3.1.4 Dies, made of tungsten carbide or hardened steel, 8,000 mm ± 0,025 mm in length. The interior shall be circular, straight and uniform in diameter such that in all positions it is within ± 0,005 mm of a true cylinder of nominal diameter 2,095 mm.

The bore shall be suitably hardened to a Vickers hardness of no less than 500 (HV 5 to HV 100) (see ISO 6507-1). The die shall not project beyond the base of the cylinder (see figure 1) and shall be mounted so that its bore is co-axial with the cylinder bore.

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

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 of the load and the piston gives the selected nominal load to an accuracy of ± 0,5 %. An alternative mechanical loading device may be used for the higher loads.

3.2 Accessory equipment

3.2.1 General

3.2.1.1 Equipment for introducing samples into the cylinder, consisting of a packing rod made of non-abrasive material.

3.2.1.2 Cleaning equipment.

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

3.2.2 For procedure A

3.2.2.1 Cutting tool, for cutting off the extruded sample. A sharp-edged spatula has been found suitable.

3.2.2.2 Stop-watch, accurate to ± 0,1 s.

3.2.2.3 Balance, accurate to ± 0,5 mg.

3.2.3 For procedure B

Measurement equipment, for the automatic measurement of distance and time for the piston movement.

The equipment shall have the capacity to obtain three measurements for each sample in the cylinder.

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 or strips of films.

NOTE 1 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 Calibration of the temperature-control system

5.1.1 Verify the accuracy of the temperature-control system (3.1.3) 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 temperature-control system until the cylinder will remain at the required temperature as indicated by the control thermometer. Preheat a calibration thermometer (3.2.1.3) to the same temperature. Then charge the cylinder with a small quantity (3 to 4 pellets) of the material to be tested, or a material representative thereof (see 5.1.2), using the same technique as for a test (see 6.2). Four minutes after completing the

charging of the material, introduce the calibration thermometer into the sample chamber and immerse it 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, correct the temperature indicated by the control thermometer by algebraic addition of the difference between the temperatures read on the two thermometers.

5.1.2 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 g/10 min (2,16 kg charge) at the temperature of calibration has been found suitable.

If such a material is used for calibration purposes in place of a more viscous material which is to be tested, the dummy material shall 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, removing the thermometer from the cylinder if necessary.

5.2 Cleaning the apparatus

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. The die may be cleaned with a closely fitting brass reamer or wooden peg. 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.

5.3 Maintenance of apparatus

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 figure 1, be removed, and the cylinder cleaned throughout.

6 Procedure A

6.1 Clean the apparatus (see 5.2). Before beginning a series of tests, ensure that the cylinder (3.1.1) and piston (3.1.2) have been at the selected temperature for not less than 15 min.

6.2 Then charge the cylinder with 3 g 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 (3.2.1.1), 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.

If the melt flow rate of the material is high, that is, more than 10 g/10 min, the loss of sample during preheating will be appreciable. In this case, use an unloaded piston or one carrying a smaller weight during the preheating period, and then change to the desired weight at the end of the 4 min preheating time.

Table 1

Melt flow rate g/10 min	Mass of test portion in cylinder[1] g	Extrudate cut-off time-interval s
0,1 to 0,5	3 to 5	240[2]
> 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[3]

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

2) It is recommended that melt flow rate should not be measured if the value obtained in this test is less than 0,1 g/10 min or greater then 100 g/10 min.

3) To achieve adequate repeatability when testing materials having an MFR greater than 25 g/10 min, it may be necessary either to control and measure cut-off intervals automatically to less than 0,1 s or to use procedure B.

6.3 Four minutes after completing the introduction of the test portion, 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. The time for this operation shall not exceed 1 min. Cut off the extrudate with the cutting tool (3.2.2.1), and discard. Then allow the loaded piston to descend under gravity. When the lower reference mark has reached the top edge of the cylinder, start the stopwatch (3.2.2.2), and simultaneously cut off 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 mm and 20 mm (see cut-off time-intervals in table 1 as a guide).

For low values of MFR (and MVR), it may not be possible to take a cut-off with a length of 10 mm or more within the maximum time-interval of 240 s. In this case, procedure B shall be used.

Stop cutting when the upper mark on 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 1 mg, 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.

The time between charging the cylinder and the last measurement shall not exceed 25 min.

6.4 The melt mass-flow rate (MFR), expressed in grams per 10 min, is given by the equation

$$\mathrm{MFR}(\theta, m_{\mathrm{nom}}) = \frac{t_{\mathrm{ref}} \cdot m}{t}$$

where

θ is the test temperature, in degrees Celsius;

m_{nom} is the nominal load, in kilograms;

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

t_{ref} is the reference time (10 min), in seconds (600 s);

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

Express the result to two significant figures.

7 Procedure B

7.1 Principle

The melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) are determined by using either of the following two principles:

a) measurement of the distance the piston moves in a specified time;

or

b) measurement of the time in which the piston moves a specified distance.

7.2 Optimum measurement accuracy

For repeatable determination of MFR between 0,1 g/10 min and 50 g/10 min or MVR between 0,1 cm^3/10 min and 50 cm^3/10 min, the movement of the piston has to be measured to the nearest $\pm$ 0,1 mm and the time to an accuracy of 0,1 s.

7.3 Pretreatment

Follow procedure A specified in 6.1 to 6.3 (to end of first paragraph).

7.4 Determination

7.4.1 When the lower reference mark has reached the top edge of the cylinder, start the automatic measurement. Proceed as specified in 7.4.2 a) if using the principle given in 7.1 a) or as specified in 7.4.2 b) if using the principle given in 7.1 b).

7.4.2 Measure

a) the distance moved by the piston at predetermined times (three or more)

or

b) the times taken by the reference mark to cover a specified distance (three or more).

Stop the measurement when the upper mark on the piston stem reaches the top edge of the cylinder.

7.4.3 The time between charging the cylinder and the last measurement shall not exceed 25 min.

7.5 Expression of results

7.5.1 The melt volume-flow rate (MVR), expressed in cubic centimetres per 10 min, is given by the equation

$$\mathrm{MVR}(\theta, m_{\mathrm{nom}}) = \frac{A \cdot t_{\mathrm{ref}} \cdot l}{t} = \frac{427 l}{t}$$

where

θ is the test temperature, in degrees Celsius;

m_{nom} is the nominal load, in kilograms;

A is the mean cross-sectional area, in square centimetres, of the piston and the cylinder (= 0,711 cm^2);

t_{ref} is the reference time (10 min), in seconds (600 s);

t is the predetermined time of measurement [see 7.4.2 a)] or the mean value of individual time measurements [see 7.4.2 b)], in seconds;

l is the predetermined distance moved by the piston [see 7.4.2 b)] or the mean value of individual distance measurements [see 7.4.2 a)], in centimetres.

7.5.2 The melt mass-flow rate (MFR), expressed in grams per 10 min, is given by the equation

$$\mathrm{MFR}(\theta, m_{\mathrm{nom}}) = \frac{A \cdot l \cdot \rho \cdot t_{\mathrm{ref}}}{t} = \frac{427 l \cdot \rho}{t}$$

where

θ, m_{nom}, A, l, t_{ref} and t are as defined in 7.5.1;

ρ is the density, in grams per cubic centimetre, of the melt at the test temperature and is given by the equation

$$\rho = \frac{m}{0{,}711 l}$$

m being the mass, determined by weighing, of a known extruded volume of length l.

7.5.3 Express the result to two significant figures.

8 Precision

When the method is used with certain materials, consideration shall be given to the factors leading to a decrease in repeatability. Such factors include

a) thermal degradation or crosslinking of the material, causing the melt flow rate to change during the preheating or test period; powdered materials requiring long preheating times are sensitive to this effect and, 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.

The precision of the method is not known because inter-laboratory data are not available. A single precision statement would not be suitable because of the number of materials covered. However, a coefficient of variation of about ± 10 % could be expected.

9 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

b) all details necessary for the complete identification of the test sample, including the physical form of the material with which the cylinder was charged;

c) the details of conditioning;

d) the details of any stabilization (see 4.2);

e) the temperature and load used in the test;

f) the melt mass-flow rate, in grams per 10 min, or the melt volume-flow rate, in cubic centimetres per 10 min, expressed to two significant figures;

g) a report of any unusual behaviour of the test portion, such as discoloration, sticking, extrudate distortion or unexpected variation in melt flow rate.

Annex A
(normative)

Test conditions for melt flow rate determination

The conditions used shall be as indicated in the appropriate material designation or specification. Table A.1 indicates test conditions that have been found useful. Table A.2 indicates test conditions that are presently specified in relevant International Standards. Other test conditions not listed here may be used, if necessary, for a particular material.

Table A.1

Conditions		Test temperature, θ	Nominal load (combined), m_{nom}
No.	Code-letter	°C	kg
1	A	250	2,160
2	B	150	2,160
4	D	190	2,160
6	F	190	10,000
7	G	190	21,600
8	H	200	5,000
12	M	230	2,160
13	N	230	3,800
17	S	280	2,160
18	T	190	5,000
19	U	220	10,000
21	W	300	1,200
22	Z	125	0,325

NOTE — If, in the future, conditions other than those listed in this table are necessary, e.g. for new thermoplastics, only the loads already in use shall be chosen. Temperatures shall also be selected from those already in the table. If absolutely necessary, new temperatures might have to be taken because of the nature of the new thermoplastic. In this case, application to ISO/TC 61/SC 5 shall be made to include the new conditions. If approved, a suitable code-letter will provisionally be issued and the standard amended at the 5-year revision.