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

Part 1: **Standard method**

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

Partie 1: Méthode normale

ISO/CEN PARALLEL PROCESSING

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 249, *Plastics,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 1133-1:2011), of which it constitutes a minor revision. The changes are as follows:

— references to withdrawn standards in Annex B (informative), Annex D (informative) and Bibliography have been updated;

— editorial corrections.

A list of all parts in the ISO 1133 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

For stable materials that are not rheologically sensitive to the time-temperature history experienced during melt flow rate testing, this document is recommended.

For materials whose rheological behaviour is sensitive to the test's time-temperature history, e.g. materials which degrade during the test, ISO 1133-2 is recommended. Also, ISO 1133-2 is considered to be particularly relevant for moisture-sensitive plastics.

NOTE At the time of publication, there is no evidence to suggest that the use of ISO 1133-2 for stable materials results in better precision in comparison with the use of this document.

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

Part 1:
Standard method

WARNING — Persons using this document should be familiar with normal laboratory practice, if applicable. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any regulatory requirements.

1 Scope

This document specifies two procedures 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. Procedure A is a mass-measurement method. Procedure B is a displacement-measurement method. Normally, the test conditions for measurement of melt flow rate are specified in the material standard with a reference to this document. The test conditions normally used for thermoplastics are listed in Annex A.

The MVR is particularly useful when comparing materials of different filler content and when comparing filled with unfilled thermoplastics. The MFR can be determined from MVR measurements, or vice versa, provided the melt density at the test temperature is known.

This document is also possibly applicable to thermoplastics for which the rheological behaviour is affected during the measurement by phenomena such as hydrolysis (chain scission), condensation and cross-linking, but only if the effect is limited in extent and only if the repeatability and reproducibility are within an acceptable range. For materials which show significantly affected rheological behaviour during testing, this document is not appropriate. In such cases, ISO 1133-2 applies.

NOTE The rates of shear in these methods are much smaller than those used under normal conditions of processing, and therefore it is possible that data obtained by these methods for various thermoplastics will not always correlate with their behaviour during processing. Both methods are used primarily in quality control.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
melt mass-flow rate
MFR
rate of extrusion of a molten resin through a die of specified length and diameter under prescribed conditions of temperature, load and piston position in the cylinder of an extrusion plastometer, the rate being determined as the mass extruded over a specified time

Note 1 to entry: MFR is expressed in units of grams per 10 min. Alternative units accepted by SI are decigrams per minute, where 1 g/10 min is equivalent to 1 dg/min.

3.2
melt volume-flow rate
MVR
rate of extrusion of a molten resin through a die of specified length and diameter under prescribed conditions of temperature, load and piston position in the cylinder of an extrusion plastometer, the rate being determined as the volume extruded over a specified time

Note 1 to entry: MVR is expressed in units of cubic centimetres per 10 min.

3.3
load
combined force exerted by the mass of the piston and the added weight, or weights, as specified by the conditions of the test

Note 1 to entry: Load is expressed as the mass, in kilograms, exerting it.

3.4
preformed compacted charge
test sample prepared as a compressed charge of polymer sample

Note 1 to entry: In order to introduce samples quickly into the bore of the cylinder and to ensure void-free extrudate, it may be necessary to preform samples originally in the form of, for example, powders or flakes into a compacted charge.

3.5
time-temperature history
history of the temperature and time to which the sample is exposed during testing including sample preparation

3.6
standard die
die having a nominal length of 8,000 mm and a nominal bore diameter of 2,095 mm

3.7
half size die
die having a nominal length of 4,000 mm and a nominal bore diameter of 1,050 mm

3.8
moisture-sensitive plastics
plastics having rheological properties that are sensitive to their moisture content

Note 1 to entry: Plastics which, when containing absorbed water and heated above their glass transition temperatures (for amorphous plastics) or melting point (for semi-crystalline plastics), undergo hydrolysis resulting in a reduction in molar mass and consequently a reduction in melt viscosity and an increase in MFR and MVR.

4 Principle

The melt mass-flow rate (MFR) and the melt volume-flow rate (MVR) are determined by extruding molten material from the cylinder of a plastometer through a die of specified length and diameter under preset conditions of temperature and load.

For measurement of MFR (procedure A), timed segments of the extrudate are weighed and used to calculate the extrusion rate, in grams per 10 min.

For measurement of MVR (procedure B), the distance that the piston moves in a specified time or the time required for the piston to move a specified distance is recorded and used to calculate the extrusion rate in cubic centimetres per 10 min.

MVR can be converted to MFR, or vice versa, if the melt density of the material at the test temperature is known.

NOTE The density of the melt is required at the test temperature and pressure. In practice, the pressure is low and values obtained at the test temperature and ambient pressure suffice.

5 Apparatus

5.1 Extrusion plastometer

5.1.1 General. The basic apparatus comprises an extrusion plastometer operating at a fixed temperature. The general design is as shown in Figure 1. The thermoplastic material, which is contained in a vertical cylinder, is extruded through a die by a piston loaded with a known weight. The apparatus consists of the following essential parts.

5.1.2 Cylinder. The cylinder shall have a length between 115 mm and 180 mm and an internal diameter of (9,550 ± 0,007) mm and shall be fixed in a vertical position (see 5.1.6).

The cylinder shall be manufactured from a material resistant to wear and corrosion up to the maximum temperature of the heating system. The bore shall be manufactured using techniques and materials that produce a Vickers hardness of no less than 500 (HV 5 to HV 100) (see ISO 6507-1) and shall be manufactured by a technique that produces a surface roughness of less than *Ra* (arithmetical mean deviation) equal to 0,25 µm (see ISO 4287). The finish, properties and dimensions of its surface shall not be affected by the material being tested.

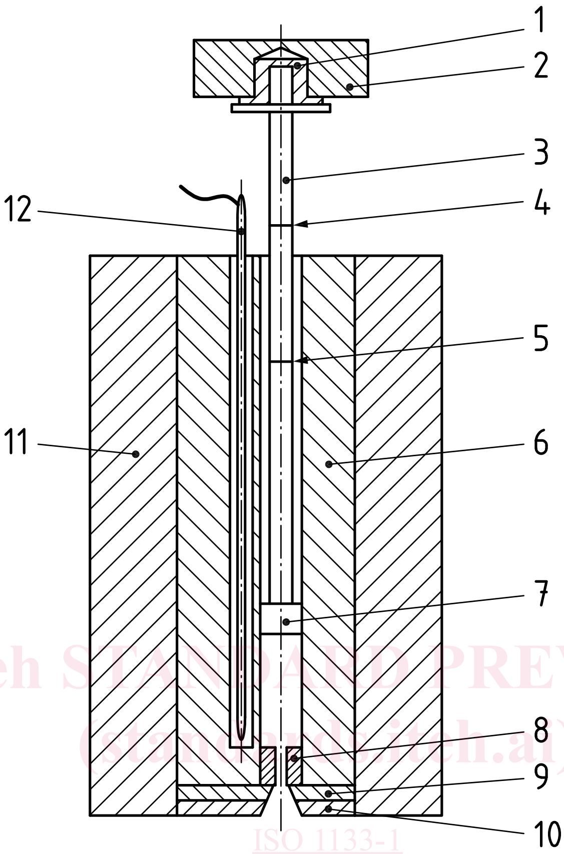
NOTE 1 For particular materials, it is possible that measurements will be required at temperatures up to 450 °C.

The base of the cylinder shall be thermally insulated in such a way that the area of 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.

A piston guide or other suitable means of minimizing friction due to misalignment of the piston shall be provided.

NOTE 2 Excessive wear of the piston head, piston and cylinder and erratic results can be indications of misalignment of the piston. Regular visual checking for wear and change to the surface appearance of the piston head, piston and cylinder is recommended.

5.1.3 Piston. The piston shall have a working length 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 (9,474 ± 0,007) mm. The lower edge of the piston head shall have a radius of $(0{,}4^{\ 0{,}0}_{-0{,}1})$ mm and the upper edge shall have its sharp edge removed. Above the head, the piston shall be relieved to ≤ 9,0 mm diameter (see Figure 2).



Key

1 insulation
2 removable weight
3 piston
4 upper reference mark
5 lower reference mark
6 cylinder
7 piston head
8 die
9 die retaining plate
10 insulating plate
11 insulation
12 temperature sensor

Figure 1 — Typical apparatus for determining melt flow rate, showing one possible configuration

The piston shall be manufactured from a material resistant to wear and corrosion up to the maximum temperature of the heating system, and its properties and dimensions shall not be affected by the material being tested. To ensure satisfactory operation of the apparatus, the cylinder and the piston head shall be made of materials of different hardness. It is convenient for ease of maintenance and renewal to make the cylinder of the harder material.

Along the piston stem, two thin annular reference marks shall be scribed (30 ± 0,2) mm apart and so positioned that the upper mark is aligned with the top of the cylinder when the distance between the lower edge of the piston head and the top of the standard die is 20 mm. These annular marks on the piston are used as reference points during the measurements (see 8.4 and 9.5).

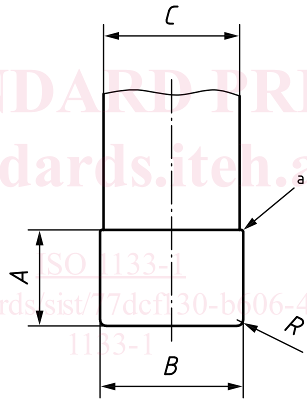
A stud may be added at the top of the piston to position and support the removable weights, but the piston shall be thermally insulated from the weights.

The piston may be either hollow or solid. In tests with very low loads the piston may need to be hollow, otherwise it may not be possible to obtain the lowest prescribed load.

Table 1 — Dimensions of piston head

Dimensions in millimetres

Length of head, *A*	6,35 ± 0,10
Diameter of head, *B*	9,474 ± 0,007
Diameter of stem, *C*	≤ 9,0
Radius of lower edge, *R*	$0,4^{\,0,0}_{-0,1}$



Key

A length of head

B diameter of head

C diameter of stem

R radius of lower edge

[a] Sharp edge removed.

Figure 2 — Schematic of piston head

5.1.4 Temperature-control system. For all cylinder temperatures that can be set, the temperature control shall be such that between (10 ± 1) mm and (70 ± 1) mm above the top of the standard die, the temperature differences measured do not exceed those given in Table 2 throughout the duration of the test.

NOTE The temperature can be measured and controlled with, for example, thermocouples or platinum-resistance sensors embedded in the wall of the cylinder. If the apparatus is equipped in this way, it is possible that the temperature is not exactly the same as that in the melt, but the temperature-control system can be calibrated (see 7.1) to read the in-melt temperature.

The temperature-control system shall allow the test temperature to be set in steps of 0,1 °C or less.

Table 2 — Maximum allowable deviation from required test temperature with distance and with time over the duration of the test

Temperatures in degrees Celsius

Test temperature *T*	**Maximum permitted deviation from the required test temperature:**[a]	
	at (10 ± 1) mm above the top surface of the standard die[b]	**from (10 ± 1) mm to (70 ± 1) mm above the top surface of the standard die**[b]
125 ≤ *T* < 250	±1,0[c]	±2,0
250 ≤ *T* < 300	±1,0[c]	±2,5
300 ≤ *T*	±1,0	±3,0

[a] The maximum permitted deviation from the required test temperature is the difference between the true value of temperature and the required test temperature. It shall be assessed over the normal duration of a test, typically less than 25 min.

[b] When using a 4 mm length half size die (see 5.1.5), the readings shall be made an additional 4 mm above the top surface of the die.

[c] For test temperatures < 300 °C, the temperature at 10 mm above the top surface of the die shall not vary with time by greater than 1 °C in range.

5.1.5 Die. The die shall be made of tungsten carbide or hardened steel. For testing potentially corrosive materials, dies made of cobalt-chromium-tungsten alloy, chromalloy, synthetic sapphire or other suitable materials may be used.

The die shall be (8,000 ± 0,025) mm in length. The interior of the bore shall be manufactured circular, straight and uniform in diameter such that in all positions it is within ± 0,005 mm of a true cylinder of diameter 2,095 mm.

The bore shall be hardened by a technique that produces a Vickers hardness of no less than 500 (HV 5 to HV 100) (see ISO 6507-1) and shall be manufactured by a technique that produces a surface roughness of less than *Ra* (arithmetical mean deviation) = 0,25 µm (see ISO 4287).

The bore diameter shall be checked regularly with a go/no-go gauge. If outside the tolerance limits, the die shall be discarded. If the no-go gauge enters the bore to any extent the die shall be discarded.

The die shall have ends that are flat, perpendicular to the axis of the bore and free from visible machining marks. The flat surfaces of the die shall be checked to ensure that the area around the bore is not chipped. Any chipping causes errors and chipped dies shall be discarded.

The die shall have an outside diameter such that it moves freely within the cylinder, but that there is no flow of material along its outside, i.e. between the die and the cylinder, during the test.

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.

If testing materials with an MFR > 75 g/10 min or an MVR > 75 cm^3/10 min, a half size die of length (4,000 ± 0,025) mm and bore diameter (1,050 ± 0,005) mm may be used. No spacer shall be used in the cylinder below this die to increase the apparent length to 8,000 mm.

The die of nominal length 8,000 mm and bore of nominal internal diameter 2,095 mm is taken to be the standard die for use in testing. When reporting MFR and MVR values obtained using a half size die, it shall be stated that a half size die was used.

5.1.6 Means of setting and maintaining the cylinder 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 leaning to one side or bending under heavy loads. A dummy piston with a spirit level on its upper end is also a suitable means of checking conformity with this requirement.

