

Plastics - Determination of the melt mass-flow rate (MFR) and melt volume-flow rate (MVR) of thermoplastics - Part 2: Method for materials sensitive to time-temperature history and/or moisture (ISO 1133-2:2011)

EESTI STANDARDI EESSÕNA

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English Version

Plastics - Determination of the melt mass-flow rate (MFR) and
melt volume-flow rate (MVR) of thermoplastics - Part 2: Method
for materials sensitive to time-temperature history and/or
moisture (ISO 1133-2:2011)

Plastiques - Détermination de l'indice de fluidité à chaud
des thermoplastiques, en masse (MFR) et en volume
(MVR) - Partie 2: Méthode pour les matériaux sensibles à
l'historique temps-température et/ou à l'humidité (ISO
1133-2:2011)

Kunststoffe - Bestimmung der Schmelze-Massefließrate
(MFR) und der Schmelze-Volumenfließrate (MVR) von
Thermoplasten - Teil 2: Verfahren für Materialien, die
empfindlich gegen eine zeit- bzw. temperaturabhängige
Vorgeschichte und/oder Feuchte sind (ISO 1133-2:2011)

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Foreword

This document (EN ISO 1133-2:2011) has been prepared by Technical Committee ISO/TC 61 "Plastics" in collaboration with Technical Committee CEN/TC 249 "Plastics" the secretariat of which is held by NBN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by June 2012, and conflicting national standards shall be withdrawn at the latest by June 2012.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

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Endorsement notice

The text of ISO 1133-2:2011 has been approved by CEN as a EN ISO 1133-2:2011 without any modification.

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Introduction

This part of ISO 1133 provides a method that is appropriate to those materials that exhibit a high rheological sensitivity to the time-temperature history experienced by the sample during the test and/or moisture. For such materials, ISO 1133-1, which has less-tightly specified testing conditions than this part of ISO 1133, is considered to be unsuitable for obtaining data of an acceptable level of precision (i.e. at least equivalent to that obtained by ISO 1133-1 for stable materials). This part of ISO 1133 is considered to be particularly relevant for moisture sensitive materials.

The primary difference between this part of ISO 1133 and ISO 1133-1 is that this part of ISO 1133 specifies tighter tolerances on the temperature, time line, sample amount and pre-treatment, resulting in more reproducible and accurate measurements.

The accuracy of MVR determination of thermoplastic materials whose rheological behaviour is affected by phenomena such as hydrolysis and condensation is often significantly influenced by:

- moisture content and sample conditioning;
- sample handling;
- a small difference in temperature, i.e. the temperature variation in the cylinder with position and/or time;
- the total time that the material is exposed to the test temperature;
- the sample volume;
- sample form (shape and size — pellets, powder, flake, etc.);
- cleaning of the apparatus.

In order to obtain accurate repeatable and reproducible results, not only does the equipment need to meet the requirements specified in this part of ISO 1133, but also the material handling and test procedure need to be followed precisely and consistently, particularly with respect to those details mentioned above to which the results are sensitive. Minor deviations from the equipment requirements, procedure and/or sample handling can result in considerable loss of repeatability, reproducibility and accuracy of the measurement.

In general, the test conditions for determination of MVR and MFR values are specified in the material standard and shall be referred to prior to conducting tests. Test conditions for the determination of MVR and MFR of materials whose rheological behaviour is affected by hydrolysis, condensation or cross-linking during the measurement are in many cases not yet mentioned in the materials standards. Standards for these materials are likely to be revised or developed in the future. Where no relevant material standard exists or where no test conditions are specified, then the drying and test conditions should be agreed between the interested parties.

NOTE At the time of publication, there is no evidence to suggest that the use of this part of ISO 1133 for stable materials results in better precision in comparison with the use of ISO 1133-1.

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

Part 2:

Method for materials sensitive to time-temperature history and/or moisture

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.

IMPORTANT — The equipment shall meet the requirements specified in this document and the measurements shall be carried out under specified conditions of temperature and load with special attention being paid to sample pre-treatment, strictly following the procedure prescribed in this document and in any applicable material standard.

1 Scope

This part of ISO 1133 specifies a procedure for the determination of the melt volume-flow rate (MVR) and melt mass-flow rate (MFR) of thermoplastic materials that exhibit a high rheological sensitivity to the time-temperature history experienced by the sample during the test and/or to moisture.

NOTE 1 Some grades of materials affected by hydrolysis are of, for example, poly(ethylene terephthalate) (PET), poly(butylene terephthalate) (PBT), poly(ethylene naphthalate) (PEN), other polyester types and polyamides; and by cross-linking are of, for example, thermoplastic elastomers (TPE) and thermoplastic vulcanizates (TPV). It is possible that this method will also be suitable for use with other materials.

It is possible that this method will not be appropriate for materials whose rheological behaviour is extremely affected during testing (see Note 2).

NOTE 2 For materials where the coefficient of variation of the MFR or MVR results is found to be higher than the precision mentioned in ISO 1133-1, the viscosity number in dilute solution (ISO 307, ISO 1628) can be more appropriate for characterization purposes.

NOTE 3 Minor deviations from the equipment requirements, procedure and/or sample handling can result in considerable loss of reproducibility, repeatability and accuracy of the measurement. MVR results determined on different materials, indicating the repeatability of the test method of this part of ISO 1133 when measured under ideal measurement conditions, are reported in Annex B.

MFR values can be determined by calculation from MVR measurements provided the melt density at the test temperature and pressure is known, or by measurement using a cutting device provided that the accuracy of the measurement is at least the same as that of the MVR measurement.

NOTE 4 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.

The primary difference between this part of ISO 1133 and ISO 1133-1 is that this part of ISO 1133 specifies tighter tolerances on the temperature in the cylinder and on the time duration over which the material is subjected to that temperature. Thus the time-temperature history of the material is more tightly controlled and consequently, for materials that are likely to be affected by exposure to elevated temperatures, the variability of test results is reduced compared with whether the specifications of ISO 1133-1 were used.

This part of ISO 1133 also provides information for preparation and handling of moisture sensitive materials that again are critical to obtaining repeatable, reproducible and accurate data.

The test conditions for measurement of the MVR and MFR are often specified in the material standard. However, for those materials where there are no test conditions specified in the material standard, it is necessary for the test conditions to be agreed between the interested parties.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 472, *Plastics — Vocabulary*

ISO 1133-1, *Plastics — Determination of the melt mass-flow rate (MFR) and melt volume-flow rate (MVR) of thermoplastics — Part 1: Standard method*

ISO 15512, *Plastics — Determination of water content*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 and ISO 1133-1 apply.

4 Principle

The melt volume-flow rate (MVR) and melt mass-flow rate (MFR) 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, timed segments of the extrudate are weighed and the extrusion rate is calculated in grams per 10 min and recorded.

For measurement of MVR, the distance that the piston moves in a specified time or the time required for the piston to move a specified distance is determined to generate extrusion rate data in cubic centimetres per 10 min.

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

In comparison with ISO 1133-1, the permitted tolerances on the temperature, time line, sample amount and pre-treatment are tighter, resulting in more accurate measurements for time-temperature and moisture sensitive materials.

5 Apparatus

5.1 Extrusion plastometer

5.1.1 General. For the purposes of this part of ISO 1133, the apparatus specified in ISO 1133-1 and the following specifications apply. Where the following specifications differ from those presented in ISO 1133-1, the following specifications shall be used.

5.1.2 Cylinder. See ISO 1133-1.

5.1.3 Piston. See ISO 1133-1.