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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

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

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

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

ISO 1133-2 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

ISO 1133 consists of the following parts, under the general title *Plastics — Determination of the melt mass-flow rate (MFR) and melt volume-flow rate (MVR) of thermoplastics*:

— *Part 1: Standard method*

— *Part 2: Method for materials sensitive to time-temperature history and/or moisture*

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.

5.1.4 Temperature-control system. For all cylinder temperatures used, the absolute temperature shall be such that between 0 mm and 70 mm above the die surface the maximum deviation from the required test temperature does not exceed ±1 °C.

For all cylinder temperatures used, the relative distribution of the temperature shall be such that between 0 mm and 70 mm above the die surface the maximum deviation does not exceed ±0,3 °C with distance and with time throughout the test.

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

NOTE 1 Tight tolerances on temperature are considered necessary as the time-temperature history of the material during the test can have a significant effect on measured rheological behaviour. It is therefore necessary to specify these conditions more tightly than in ISO 1133-1 in order to achieve measurement precision comparable with that obtained using ISO 1133-1 with stable materials.

NOTE 2 The temperature can be measured and controlled with temperature measuring devices 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 to read the in melt temperature.

5.1.5 Die. See ISO 1133-1.

The standard die of (8,000 ± 0,025) mm in length, (2,095 ± 0,005) mm in diameter, as specified in ISO 1133-1, shall be used unless stated otherwise in the relevant materials standard or agreed by the interested parties.

When using a standard die for testing low melt-viscosity materials such as bottle-grade PET, the material may extrude through the die during charging, and it might also be difficult to ensure void-free extrudate resulting in reduced precision and accuracy. In these cases the use of the half size die, specified in ISO 1133-1, is recommended (8.1).

5.1.6 Means of setting and maintaining the cylinder truly vertical. See ISO 1133-1.

5.1.7 Load. See ISO 1133-1.

5.2 Accessory equipment

5.2.1 General. For the purposes of this part of ISO 1133, the accessory equipment given in ISO 1133-1 and the following equipment apply.

5.2.2 Packing rod. See ISO 1133-1.

5.2.3 Cleaning equipment. See ISO 1133-1.

5.2.3.1 Go/no-go gauge. See ISO 1133-1.

5.2.3.2 Temperature verification device. See ISO 1133-1.

The temperature verification device shall have sufficient accuracy and precision to enable verification of the MVR/MFR instrument to the temperature tolerances specified in 5.1.4.

The equipment used to calibrate the temperature without material in the cylinder differs from that specified in ISO 1133-1, since verifying the temperature at 10 mm intervals from 0 mm to 70 mm above the standard die is required for this test method (Annex A; Note 5).

5.2.3.3 Die plug. See ISO 1133-1.

5.2.3.4 Piston/weight support. See ISO 1133-1.

5.2.3.5 Preforming device. See ISO 1133-1.

5.2.3.6 Drying equipment. Vacuum ovens or hot air dryers used to remove moisture from the samples (6.2) where required by the material standard.

The use of vacuum ovens is preferred as this should result in exposing the materials to be dried to shorter times at lower temperatures, thereby reducing the magnitude of any changes occurring to the rheological properties of the materials due, for example, to hydrolysis.

5.2.3.7 Moisture content determination. The moisture content shall be determined according to ISO 15512, unless otherwise specified by the material standard.

5.2.4 Equipment for melt mass flow rate determination (Procedure A). See ISO 1133-1.

If the instrument is not equipped with an automatic cut-off device, manual cutting may be used if the accuracy of the measurement is at least the same as the MVR measurement.

5.2.5 Equipment for melt volume flow rate determination (Procedure B). See ISO 1133-1.

6 Test sample

6.1 Sample form

See ISO 1133-1.

6.2 Sample pre-treatment and storage

The test sample shall be treated, e.g. dried, prior to testing in accordance with the appropriate material specification standard. When a preformed solid charge of powder or flakes (ISO 1133-1) is used as the test sample, the material shall be dried prior to preforming. If the sample pre-treatment is not specified in the relevant material standard, it shall be agreed between the interested parties.

For materials that are sensitive to moisture, the moisture content shall be such that its effect on the MVR or MFR of the material under the test conditions used is minimized as far as possible. The material shall be handled both before and after drying in such a way that absorption of moisture is prevented or minimized, e.g. by preventing moisture transfer caused by skin-sample contact or uptake from the atmosphere.

Immediately after drying, the material shall be transferred into a dry, preferably hot, moisture-proof container preventing moisture uptake. Then the material shall be allowed to cool down to ambient temperature and the test shall be carried out within 4 h after transfer to the dry container or within 2 d when stored in a desiccator containing a drying agent, unless otherwise specified in the relevant material standard or agreed between the interested parties.

All preformed charges, after removing from the charge forming instrument (ISO 1133-1) and before testing, shall be handled and stored in the same manner to improve repeatability of measurements, except that preformed charges may need to be tested without cooling to avoid distortion of the charge which would prohibit its insertion into the MVR/MFR cylinder.

For comparison of results, e.g. with other laboratories, the material should be cooled down in order to prevent differences in temperature history, or the procedure should be agreed between the interested parties. However, for production control or practical purposes it might be preferable to charge the MVR equipment with the material directly from the oven.

The material should not be allowed to cool down to ambient temperature inside a (vacuum) oven, as due to the very long cooling down time the time-temperature history of the sample is quite different compared with cooling down in a container and may have a significant influence on the results.

NOTE The effect of moisture content on the rheological behaviour of the material can be determined by carrying out repeat experiments on samples with different moisture contents.

7 Temperature verification, cleaning and maintenance of the apparatus

7.1 Verification of the temperature-control system

7.1.1 Verification procedure

When verifying the temperature variation with position and time, set the temperature-control system of the MVR/MFR equipment to the required temperature and wait at least the prescribed time mentioned in the equipment manual until the cylinder remains at the set temperature as indicated by the instrument control thermometer.

Verify the temperature variation in the cylinder at (10 ± 1) mm intervals from 0 mm above the top of the standard die up to and including (70 ± 1) mm above the top of the standard die using a calibrated temperature-measuring device. The temperature variation shall be measured at each of the 10 mm intervals by recording, at each position, the temperature at 1 min intervals until 10 min after the first stable temperature reading.

A method for performing the verification is presented in Annex A.

NOTE The time until the temperature reading becomes stable after immersing or repositioning the temperature measuring device in the cylinder depends on the equipment used. Information on the response time of the temperature-measuring device may be available from the device supplier.

7.1.2 Material used during temperature verification

See ISO 1133-1 and Annex A.

7.2 Cleaning the apparatus

See ISO 1133-1.

7.3 Vertical alignment of the instrument

See ISO 1133-1.

8 Procedural set-up

8.1 General

The automatic measurement of distance and time for the piston movement (ISO 1133-1, procedure B) is preferred as it can result in better repeatability than the MFR method (ISO 1133-1, procedure A) for testing materials that exhibit a high rheological sensitivity to the time-temperature history and/or moisture.

If the MVR value is higher than 40 cm^3/10 min, use of the half size die may be considered (5.1.5).

MFR values can be determined from MVR values, or vice versa, given the density of the polymer at the test temperature (see ISO 1133-1).

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.