UDC 678.073 : 539.389 : 620.1

Ref. No. : ISO/R 1133-1969 (E)

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 1133

PLASTICS

DETERMINATION OF THE MELT FLOW RATE OF THERMOPLASTICS

1st EDITION

November 1969

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BRIEF HISTORY

The ISO Recommendation R 1133, *Plastics – Determination of the melt flow rate of thermoplastics*, was drawn up by Technical Committee ISO/TC 61, *Plastics*, the Secretariat of which is held by the American National Standards Institute (ANSI).

Work on this question led to the adoption of a Draft ISO Recommendation.

In July 1966, this Draft ISO Recommendation (No. 1001) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Argentina	Germany	South Africa, Rep. of
Australia	Hungary	Spain
Austria	India	Sweden
Belgium	Iran	Switzerland
Brazil	Italy	Turkey
Canada	Japan	U.A.R.
Chile	Korea, Rep. of	U.S.A.
Czechoslovakia	Netherlands	U.S.S.R.
Finland	Poland	Yugoslavia
France	Romania	

One Member Body opposed the approval of the Draft :

United Kingdom

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in November 1969, to accept it as an ISO RECOMMENDATION.

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1. SCOPE

1.1 The apparatus and test procedure described in this ISO Recommendation are intended for measuring the fluidity, in arbitrary units, of molten thermoplastic materials under specified conditions of temperature and pressure. The test conditions for each material are indicated in the material specifications.

NOTE. – *Test conditions commonly used are listed in the Appendix.*

1.2 The melt fluidity 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.

2. APPARATUS

2.1 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, page 9. 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.

2.1.1 *Steel cylinder*, fixed in a vertical position and suitably insulated for operation up to 300 °C. The cylinder length should be between 115 mm (4.5 in) and 180 mm (7.3 in), and the internal diameter between 9.500 mm (0.374 in) and 10.000 mm (0.394 in). It should be uniform along its length to within ± 0.025 mm (± 0.001 in). The base of the cylinder should be thermally insulated in such a way that the area of the exposed metal is less than 4 cm^2 (0.6 in^2) and it is recommended that the insulating material used be polytetrafluoroethylene (thickness about 3 mm (1/8 in)), in order to avoid sticking of the extrudate.

2.1.2 *Steel piston* (see Notes 1 and 2 below), whose working length is at least as long as the cylinder. The piston should have a head 6.35 ± 0.10 mm (0.250 ± 0.005 in) in length. The diameter of the head should be less than the internal diameter of the cylinder by 0.075 ± 0.015 mm (0.0030 ± 0.0006 in). Furthermore, it is necessary to know the diameter of the head with an accuracy of ± 0.025 mm (± 0.001 in) in order to make the calculation required by clause 2.1.6. The lower edge of the head should have a radius of 0.4 mm (1/64 in) and the upper edge should have its sharp edge removed. Above the head, the piston should be relieved to about 9 mm (0.35 in) diameter. A stud may be added at the top of the piston to support the removable load, but the piston should be thermally insulated from the load. Along the piston stem, two thin annular reference marks should be scribed 30 mm (1.20 in) 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 (0.8 in) (see Note 3 below).

NOTES

1. To ensure satisfactory operation of the apparatus, the cylinder and the piston should be made of steel of different hardness. 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 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 these cases a solid piston should be used or hollow piston with suitable guides. When using this modification it should be certain 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.

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2.1.3 *Heating and thermostating devices*, such that the selected temperature of the material in the cylinder can be maintained within ± 0.5 °C. Automatic temperature control is strongly recommended.

2.1.4 *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 (0.6 in) from the base. This measuring device should be calibrated to permit temperature measurement to ± 0.1 °C. A heat transfer medium, such as a suitable low-melting alloy or silicone oil, may be used between the thermometer and the cylinder.

2.1.5 *Dies*, made of hardened steel, 8.000 ± 0.025 mm (0.315 ± 0.001 in) in length. The internal diameter should be chosen according to the relevant specifications (see Appendix) for each material, but it should be uniform along its length to within ± 0.005 mm (± 0.0002 in). The die should not project beyond the base of the cylinder (see Figure).

NOTE. – All surfaces of the apparatus in contact with the material under test should have a high polish.

2.1.6 *Removable load*, on the top of the piston, consisting of a set of weights which may be adjusted so that the combined mass of the load and the piston applies a force P on the material, with a tolerance of ± 0.5 %, calculated in grammes-force according to the following formula :

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

where

K is a factor depending on the die and on the approximate selected force (see Appendix);

D is the measured diameter of the piston head, in millimetres, to ± 0.025 mm (± 0.001 in);

d is the measured diameter of the die, in millimetres, to ± 0.005 mm (± 0.0002 in).

2.2 Accessory equipment

2.2.1 *Equipment* for introducing samples into the cylinder.

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

2.2.3 *Cleaning equipment.*

2.2.4 *Stop watch*, accurate to ± 0.1 second.

2.2.5 *Balance*, accurate to ± 0.0005 g.

3. TEST SPECIMEN

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

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

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

4. TEMPERATURE CALIBRATION, CLEANING AND MAINTENANCE OF THE APPARATUS

4.1 First check that the specified test temperature has been reached, as indicated by the thermometer in the block. Then introduce into the cylinder hole a second thermometer, as described in clause 2.1.4. This thermometer should be immersed in the thermoplastic material and the bulb should be distant 10 mm (0.4 in) from the upper face of the die. The temperature indicated by the control thermometer should be corrected by algebraic addition of the difference between the temperatures read on the two thermometers.

4.2 Clean the apparatus thoroughly after each determination. The cylinder may be cleaned with cloth patches. The piston should 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 should abrasives or materials likely to damage the surface of the piston, cylinder, or die be used. Special precautions should be taken to avoid exposure to toxic fumes when using solvents at high temperatures.

4.3 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.

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5. PROCEDURE

5.1 Clean the apparatus (see section 4). Before beginning a series of tests, the temperature of the cylinder and piston should have been at the selected temperature for not less than 15 minutes.

5.2 Then charge the cylinder with 4 to 8 g of the sample according to the anticipated flow rate (see, for example, Table 1). During the charging, the material is compressed by using hand pressure on the piston. To ensure a charge as free from air as possible for material susceptible to oxidative degradation, the charging process must be completed in 1 minute. The piston, loaded or unloaded according to the flow rate of the material (see Note below), is left in the cylinder.

NOTE. – If the flow rate of the material is high, that is, 10 to 25 g per 10 minutes, 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-heat period, which can be changed to the desired weight at the end of the 4 minute pre-heating time.

TABLE 1

Flow rate	Mass of the sample in the cylinder	Time interval
g/10 min	g	seconds
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 to 25	6 to 8	10 to 15

5.3 Four minutes after completing the introduction of the sample, during which time *the temperature should have returned to that selected*, the selected load is placed on the piston, if it was unloaded or under-loaded. The piston is allowed to descend under gravity or pushed down faster using hand pressure, until a bubble-free filament is extruded and the lower mark is 5 to 10 mm (0.2 to 0.4 in) above the top edge of the cylinder. The time for this operation should not exceed 1 minute. The extrudate is cut off and discarded. The loaded piston is then allowed to descend under gravity. Only when the lower reference mark has reached the top edge of the cylinder, the time is recorded and the extruded portion cut off simultaneously with the cutting tool and again discarded.

Successive cut-offs are then collected in order to measure the extrusion rate, at time intervals depending on the flow rate so chosen that the length of a single cut-off is not less than 10 mm (0.4 in) and preferably between 10 and 20 mm (0.4 and 0.8 in) (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. Any cut-off containing visible air bubbles is discarded. The remaining cut-offs, which number at least three, are weighed individually, after cooling, to the nearest milligramme and their average mass calculated. If the difference between the maximum and the minimum value of the individual weighings exceeds 15 % of the average, the result is discarded and the test repeated on a fresh portion of the sample.

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6. EXPRESSION OF RESULTS

The melt flow rate should be expressed to two significant figures, using the following formula :

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

where

- T is the test temperature;
- M is the nominal load in kilogrammes (the actual load is calculated according to clause 2.1.6);
- S is the reference time in seconds; this is the chosen time (see Appendix) to which the cut-off time intervals are related;
- m is the average mass of the cut-offs in grammes;
- t is the cut-off time interval in seconds.

NOTES

1. The diameter of the die used should be stated if it is other than 2.090 to 2.100 mm.
2. The reference time chosen should be stated if it is other than 600 seconds.

7. TEST REPORT

The test report should include the following particulars :

(*a*) nature and physical form of the material with which the cylinder is charged;

(*b*) details of conditioning;

(*c*) details of any stabilization (see clause 3.2);

(*d*) diameter of die, temperature and load used to carry out the test;

(*e*) flow rate in grammes per the reference time;

(*f*) report of any unusual behaviour of the test specimen such as discoloration, sticking, extrudate distortion, or unexpected variation in flow rate.

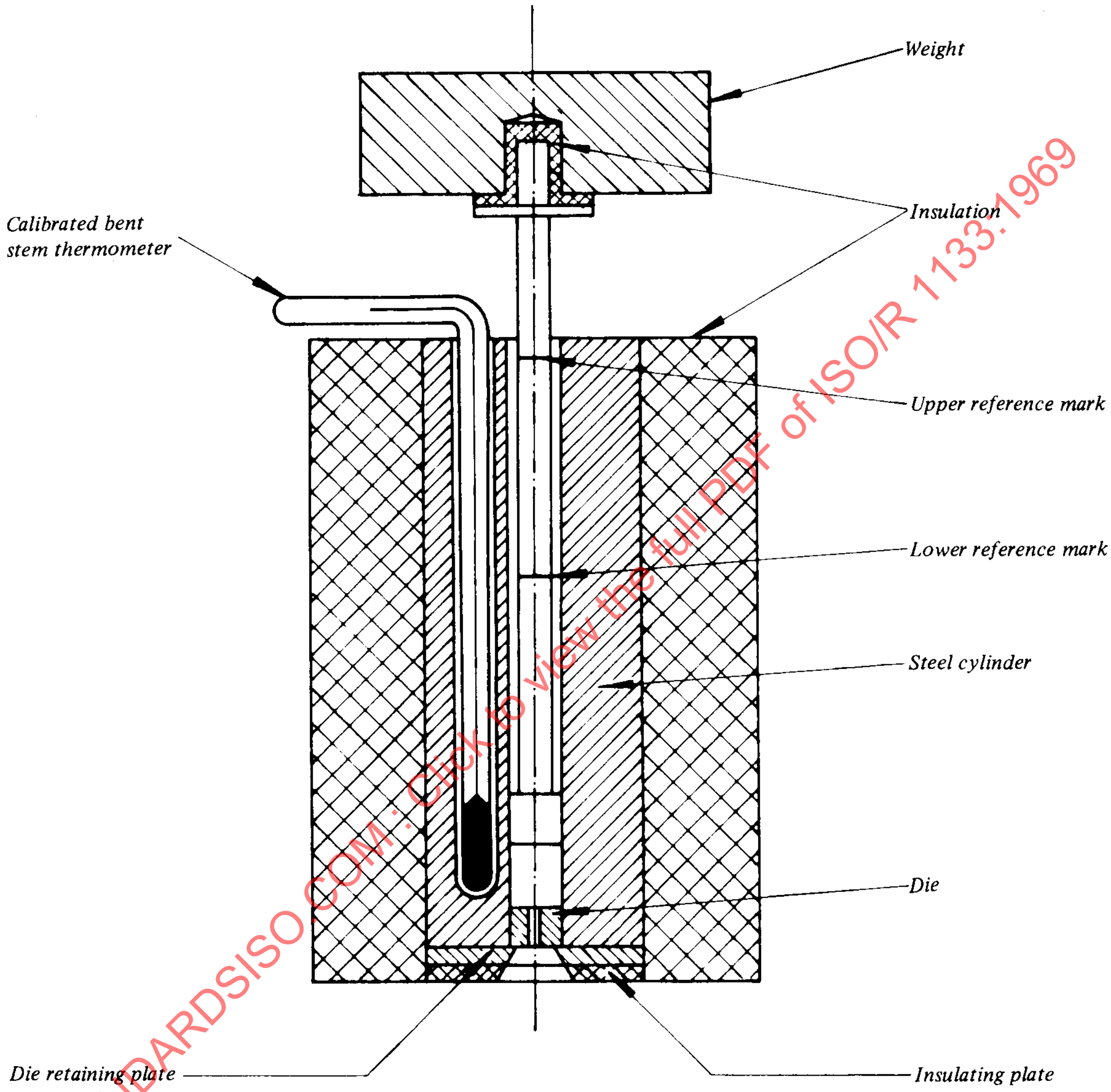

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FIGURE – **Typical apparatus for determining melt flow rate**
(showing one of the possible methods of retaining the die
and one type of piston)