Customer focus is a matter of survival nowadays. Quality, assurance is the biggest contributing factor towards this. In the plastics industry there has been an increased demand for standard methods to verify raw material. For the receiving inspection for raw materials, there are a couple of rheological methods that are in use for thermoplastics. Melt Flow Rate (MFR) gives information about rheological properties of a material with user friendly equipment and at a relatively low cost. One MFR-value provides only one measurement on a viscosity curve. An extension to two MFR-values at different loads gives another measurement on the viscosity curve and a better picture of the material.

1 SCOPE

The Flow Rate Ratio (FRR) is based on two Melt Flow Rate (MFR) values instead of only one MFR as generally measured at present. MFR is a determination of the amount of melted material which, in ten minutes, is pressed through a die by a piston using a constant load. The material used consists of polyethylene grades. This method can of course be used for other thermoplastics which could be tested under the same conditions as the ones listed in the Annex, Table 1.

Through FRR, the previous MFR is now extended to two measurements at different loads. This gives a higher accuracy in quality control for plastics. An improvement of accuracy is achieved since the ratio between two MFR-values (FRR) indicates shear-thinning properties. The MFR-value obtained by the lowest weight indicates the zero shear viscosity which correlates with the weight average molecular weight ($M_w$). By quoting MFR (for the lowest weight) and FRR more information is supplied about the material.

Note: The low rates of shear obtained during a MFR test are not comparable to the shear rates under normal processing conditions.
2 FIELD OF APPLICATION

This evaluation is intended for polyethylene materials although it should be possible to use the method for other thermoplastics.

3 REFERENCES

Documents which are necessary for the application


4 DEFINITIONS

Melt flow rate (MFR)
The quantity of thermoplastic material extruded in a given time under specified test conditions

Flow Rate Ratio (FRR)
The ratio between two Melt Flow Rate values

Shear rate
The rate of change of shear strain

Viscosity
The property of resistance to steady flow exhibited within the body of a material

5 SAMPLES

The test specimen can be in the form of granules, powder, strips of films or material cut into small pieces, as long as it can fit into the top of the cylinder.

Three measurements per MFR/MVR are required.

6 METHOD OF TEST

6.1 Principle

The material is extruded through a die under pressure for ten minutes, while the amount of material in grams is registered.

6.2 Apparatus


6.2.1 Temperature calibration, cleaning and maintenance of the apparatus


6.3 Preparation of test samples

The specimen may be in the form of powder, granules, strips of film or material cut into pieces. It is very important that materials to be tested are similar in size to one another.

Moisture in the material affects reproducibility and might accelerate degradation. Moist material should therefore be dried before testing.

Generally there is no need for test specimens to be conditioned. If necessary eliminate volatile oxidation products, since this can affect the measurement. This applies especially for strips of films that have already gone through a manufacturing process or are being recycled and have therefore been susceptible to oxidative degradation. Strips that contain air bubbles are not accepted. If bubbles are obtained it is recommended to preform the powder.

6.4 Procedure

On some equipment only melt volume-flow rate (MVR) can be obtained. In order to obtain melt mass-flow rate (MFR) use the following equation

\[ MFR = MVR \times \delta \]

where \( \delta \) (for melted polyethylene at 190 °C) = 0.765 g/cm³.

Both expressions are described under this section.

6.4.1 Test conditions

For test conditions, temperature and load see Annex (Table 1).

For polyethylene 4 minutes preheating time is recommended.

6.4.2 Manual procedure

With this technique an appropriate amount of the extruded material is cut off. This is performed during a specific time and the extrudate is weighed.

6.4.2.1 Preparation

Clean the apparatus according to 6.2 and make sure that the temperature is right.

Charge the cylinder with a predetermined amount of material with respect to the anticipated melt flow in Annex, Table 2.

Compress the material gradually with a packing rod mentioned in 6.2. Use only hand pressure. For material that has been susceptible to oxidative degradation this process should be completed within 1 minute so that as little air as possible is trapped in the material. Put the unloaded piston in the cylinder.

After preheating time according to 6.4.1, put the desired weight on the piston. The temperature should by this time have returned to the selected value.

With a slight hand pressure push the piston downwards until a bubble-free filament is extruded. Stop when the lower mark on the piston is 5-10 mm above the top edge of the cylinder. This operation should not take longer than 1 minute. Cut off the extrudate with a cutting tool and discard.

6.4.2.2 Testing

Let the loaded piston descend under gravity. When the lower mark on the piston reaches the top edge of the cylinder start the stopwatch and at the same time discard the extrudate.
Select cut-offs at identical time intervals. Register the exact time for every single cut-off. The single cut-offs should not be less than 10 mm and preferably in between 10-20 mm long.

The time intervals depend upon the melt flow of the material and should be chosen according to Annex, Table 2.

Stop cutting when the upper mark on the piston has reached the top edge of the cylinder. Discard cut-offs containing air bubbles. The cut-offs should be weighed to the nearest 1 mg after cooling. Calculate the average mass of three accepted cut-offs. If the difference between the maximum or minimum value of the separate measurements exceeds 15 % of the average, discard and repeat the test on new material.

The time between charging the cylinder until the last measurement shall not exceed 25 minutes.

6.4.3 Automatic procedure

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

1. Measurement of the distance the piston moves in a specified time.
2. Measurement of the time in which the piston moves a specified distance.

In order to receive repeatable determinations for MFR between 0.1 g/10 min and 50 g/10 min an accuracy to the nearest 0.1 mm is needed. For MVR between 0.1 cm³/10 min and 50 cm³/min an accuracy to the nearest 0.1 seconds is needed.

For preparation, charging and preheating see 6.4.2.1.

6.4.4 Testing

When the lower mark on the piston reaches the top edge of the cylinder start the automatic measurements.

For principle 1. The distance moved by the piston at predetermined times (three or more) is measured.

For principle 2. The time it takes for the upper mark on the piston to move certain distances (three or more) is measured.

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

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

6.5 Expression of result

6.5.1 Manual procedure

The MFR (g/10 min), is given by the following equation

\[
MFR(T,M) = \frac{t_{ref} \cdot m}{t}
\]

where

\( T \) = test temperature (°C)
\( M \) = nominal load (kg)
\( t_{ref} \) = reference time (10 min), (s)
\( m \) = average mass of cut-offs (g)
\( t \) = time-interval for cut-offs (s)

Express the result in two significant figures.

6.5.2 Automatic Procedure

6.5.2.1 Melt volume-flow rate (MVR)

The MVR (cm³/10 min) is given by the following equation

\[
MVR(T,M) = \frac{A \cdot t_{ref} \cdot I}{t}
\]

where

\( T \) = test temperature (°C)
\( M \) = nominal load (kg)
\( t_{ref} \) = reference time (10 min), (s)
\( A \) = cross sectional area of the piston and the cylinder (= 0.711 cm²), (cm²)
\( I \) = predetermined distance moved by the piston or the mean value of individual distance measurements, (cm)
\( t \) = time interval for cut-offs (s)

Express the result in two significant figures.

6.5.2.2 Melt mass-flow rate (MFR)

MFR (g/10 min) is given by the following equation

\[
MFR(T,M) = \frac{A \cdot t_{ref} \cdot I \cdot \delta}{t}
\]

where

\( T \) = test temperature (°C)
\( M \) = nominal load (kg)
\( t_{ref} \) = reference time (10 min), (s)
\( A \) = cross sectional area of the piston and the cylinder (= 0.711 cm²), (cm²)
\( I \) = predetermined distance moved by the piston or the mean value of individual distance measurements, (cm)
\( t \) = time interval for cut-offs (s)
\( d \) = density (for melted polyethylene 190 °C) = 0.765 g/cm³

Express the result in two significant figures.

6.6 Flow Rate Ratio (FRR)

The FRR is given by the following equation

\[
FRR = \frac{MFR(T,M_1)}{MFR(T,M_2)} \quad M_1 > M_2
\]

where

\( M \) = nominal load (kg)
\( T \) = test temperature (°C)
The ratio between two MFR values is designated FRR. Each MFR value is based on the average of three measurements. The MFR values are determined at different loads. The FRR is used as a rheological method for verifying raw material. Instead of just one measurement on the viscosity curve an extension to two MFR measurements gives a better picture of the material. By quoting the MFR at minimum load, an indication of zero shear viscosity is obtained and by quoting the FRR the shear-thinning properties are revealed.

It is desirable to obtain a ratio as large as possible so that enhanced relevance is reached. If possible conditions 4 and 7 in Annex, Table 1, should be used. It is recommended in situations where MFR (190/5) > 2 g/10 min to change the load to 2.16 kg.

Express the result in one decimal.

6.7 Accuracy

Bear in mind that the repeatability of crosslinked polyethylene may decrease. This is possible because thermal degradation during the preheating stage might cause changes in melt flow rate.

An inter-laboratory test has been performed in the Scandinavian countries and the reproducibility (R) of the method was estimated according to the following results. In these results repeatability was not taken into consideration since that has been done in other tests and good repeatability has been achieved.

<table>
<thead>
<tr>
<th>FRR</th>
<th>23.4</th>
<th>84.3</th>
<th>33.6</th>
<th>23.8</th>
</tr>
</thead>
<tbody>
<tr>
<td>(load)</td>
<td>(21.65)</td>
<td>(21.62/1.6)</td>
<td>(21.6/5.0)</td>
<td>(21.6/2.16)</td>
</tr>
</tbody>
</table>

| R    | 0.73 | 2.71 | 1.39 | 0.46 |

6.8 Test report

The test report shall contain the following information:

a) Information as to the test laboratory
b) Purpose of the test
c) A reference to this test method
d) Identification and description of the physical form of the material
e) Details of conditioning
f) Temperature and load used during the test
c) Date of test
h) Identification of the test equipment used
i) Procedure used (manual/automatic)
j) Test result in MFR (g/10 min) or MVR (cm²/10 min) for the minimum load
k) Test result in FRR
l) Average mass of the cut-offs and the cut-off time intervals for manual procedure
m) Any deviations from this method
n) Date of test and signature
o) Information about behaviour during test e.g. uneven flow rate, smell and discoloration.
ANNEX

Table 1.

<table>
<thead>
<tr>
<th>Conditions, No</th>
<th>Test temperature, T [°C]</th>
<th>Nominal load, M [kg]</th>
</tr>
</thead>
<tbody>
<tr>
<td>4</td>
<td>190</td>
<td>2.160</td>
</tr>
<tr>
<td>18</td>
<td>190</td>
<td>5.000</td>
</tr>
<tr>
<td>7</td>
<td>190</td>
<td>21.600</td>
</tr>
</tbody>
</table>

The condition numbers are identical to the ones in ISO 1133: 1991.

Table 2.

<table>
<thead>
<tr>
<th>Expected Melt Flow Rate [g/10 min]</th>
<th>Mass of test portion into cylinder [g]</th>
<th>Extrudate cut-off interval [s]</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1-0.5</td>
<td>3-5</td>
<td>240</td>
</tr>
<tr>
<td>&gt; 0.5-1</td>
<td>4-5</td>
<td>120</td>
</tr>
<tr>
<td>&gt; 1.3-5</td>
<td>4-5</td>
<td>60</td>
</tr>
<tr>
<td>&gt; 3.5-10.0</td>
<td>6-8</td>
<td>30</td>
</tr>
<tr>
<td>&gt; 10.0</td>
<td>6-8</td>
<td>5-15</td>
</tr>
</tbody>
</table>

When the density of the material is greater than 1.0 g/cm³, a greater portion of material might be necessary. It is recommended that material with a MFR less than 0.1 g/10 min and greater than 100 g/10 min should not be measured.

For material having a MFR greater than 25 g/10 min it may be necessary to measure the cut-off intervals to the nearest 0.1 s or to use automatic procedure, in order to achieve good repeatability.