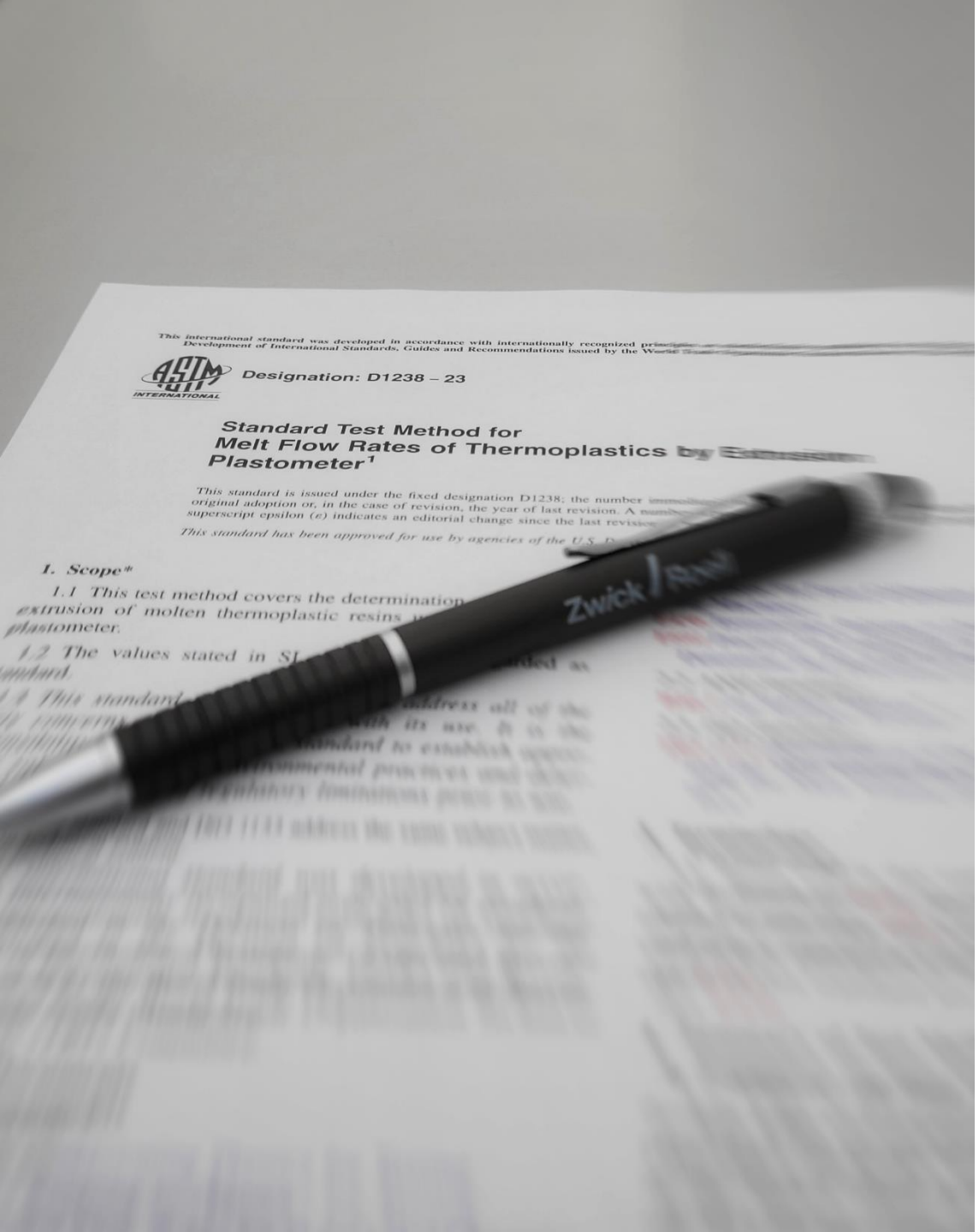


Measurement of Melt Flow Rates in ISO and ASTM

Simplified test methods explained along the example of the new ASTM D1238-2023

Agenda

1. The recent changes in ASTM D 1238
2. Advantages offered by the new ASTM
3. Principles of measuring melt flow rates
4. Today‘s challenge to organize a melt-flow lab
5. Simplified testing using the Aflow plastometer

Zwick Roell

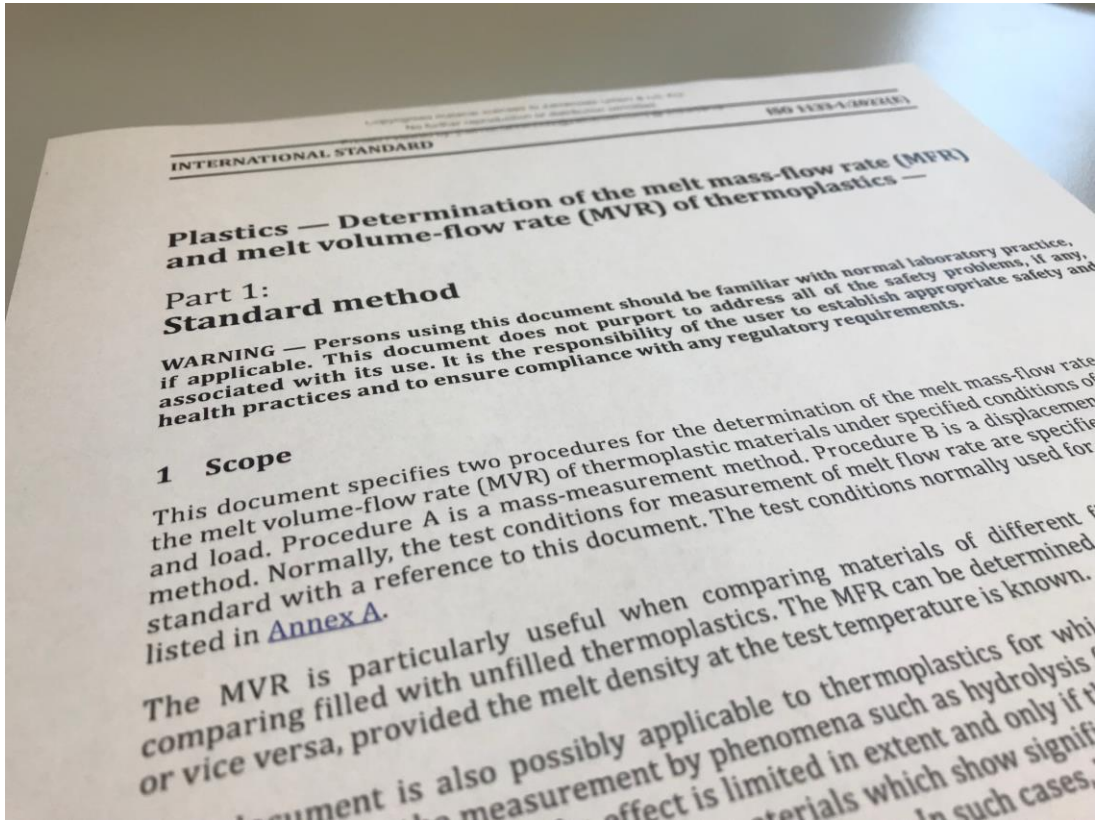
ASTM D1238 allows to use plastometers which apply the test weight by a drive system in load control mode.

Important new definitions are:

- ✓ As an acceptable alternative, the test force can be applied via a drive system working with a load cell … (§6.1.1)
- ✓ The load cell in combination with the piston shall be calibrated to show that the weight of piston and load is within a tolerance of ± 0.5% of the selected load (§6.4.3 and §6.4.4)
- ✓ … shall ensure that no effect of temperature is present which is bigger than the given tolerances for the weights.
- ✓ .. the applied force shall comply with ASTM E4 and with the requirements in section 6.4.3 (± 0.5% accuracy and repeatability (§6.14)
- ✓ Precision and Bias statement for Load Controlled Tests (§18) was added

The force-controlled load apply system in an Aflow Melt Indexer is well protected against overload and temperature.

ISO 1133-1/-2 allows the use of force-controlled instruments already since more than 20 years.



- Experience with load-controlled instruments available
- Participation in round-robin tests (proficiency tests) shows that there is no significant difference in results
- Calibration of force measurement channels can be performed using the same equipment as for static and dynamic testing machines.
- Alternatively, a scale can be used for calibration and checks

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The new ASTM version offers valuable advantages

Lab organization and testing becomes significantly easier and more reliable

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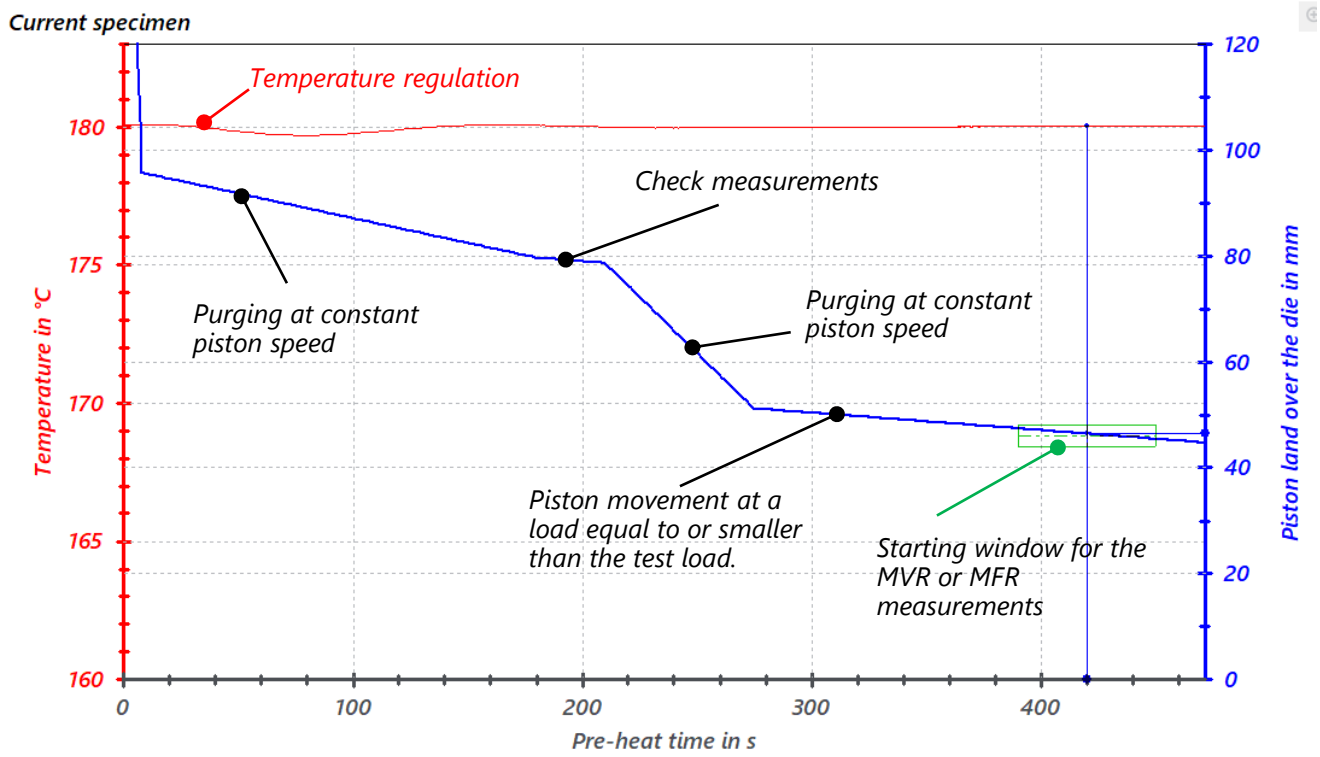
A load controlled plastometer allows fully automatic and flexible reactions during the preheat phase of the test

- The instrument reacts according to the present situation of filling quantity and material flow.
- Both load-controlled movements and simple speed-controlled piston movements are used to control the piston movement for different filling quantities and for a very large range of MFR values.
- The aim is to securely achieve the start window for the measurements as defined by the standard.



The diagram shows how the Melt Indexer reacts during the pre-heat phase in case of a MVR of 1.4 cm^3/10min and a filling quantity of 5 g.

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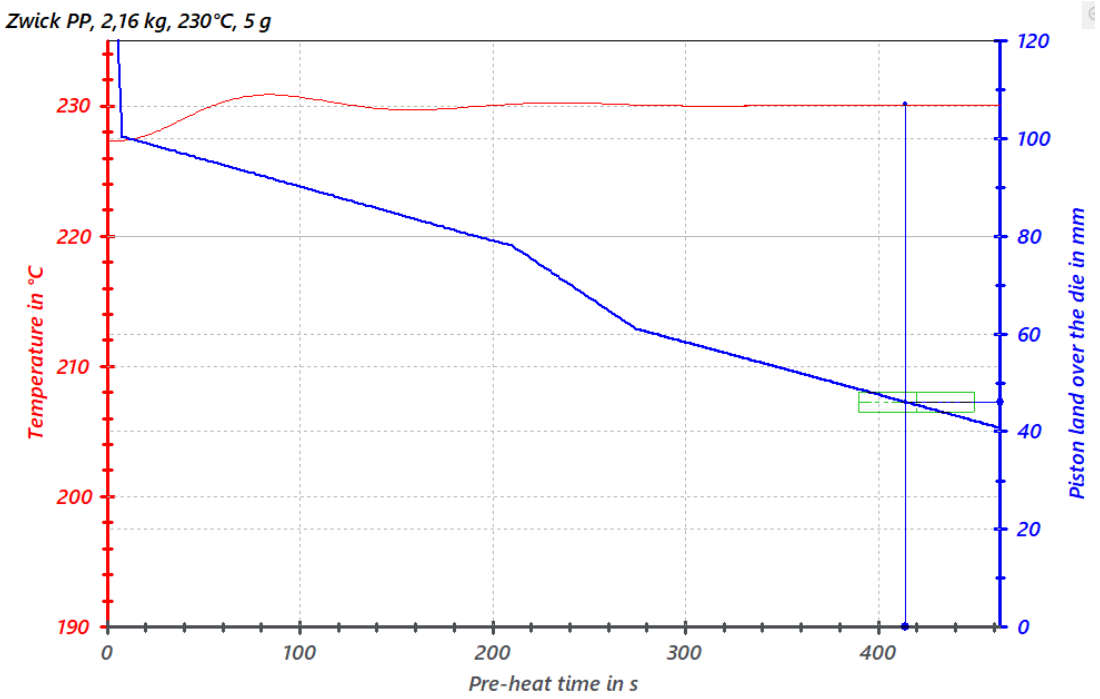
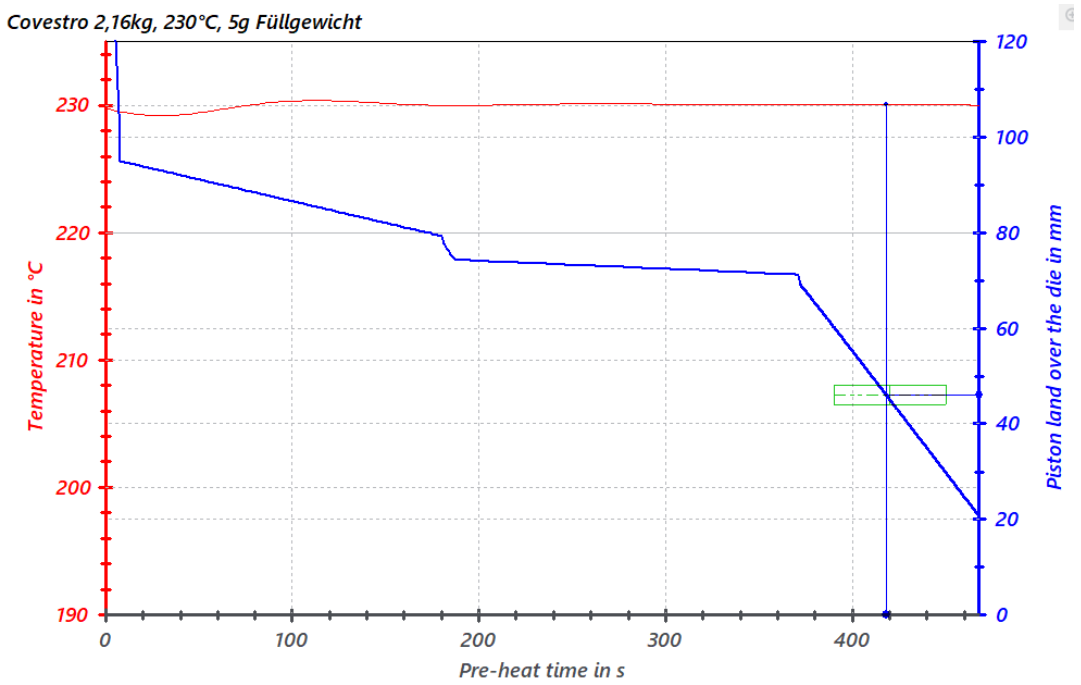
The start position for MVR measurement can safely be met – without exact estimation and pre-programming.



PP measured at 230°C, 2.16 kg and a filling quantity of 5 g.
The MVR was measured at **4.61 cm³/10min**.



PP measured at 230°C, 2.16 kg and a filling quantity of 5 g.
The MVR was measured at **21.7 cm³/10min**.

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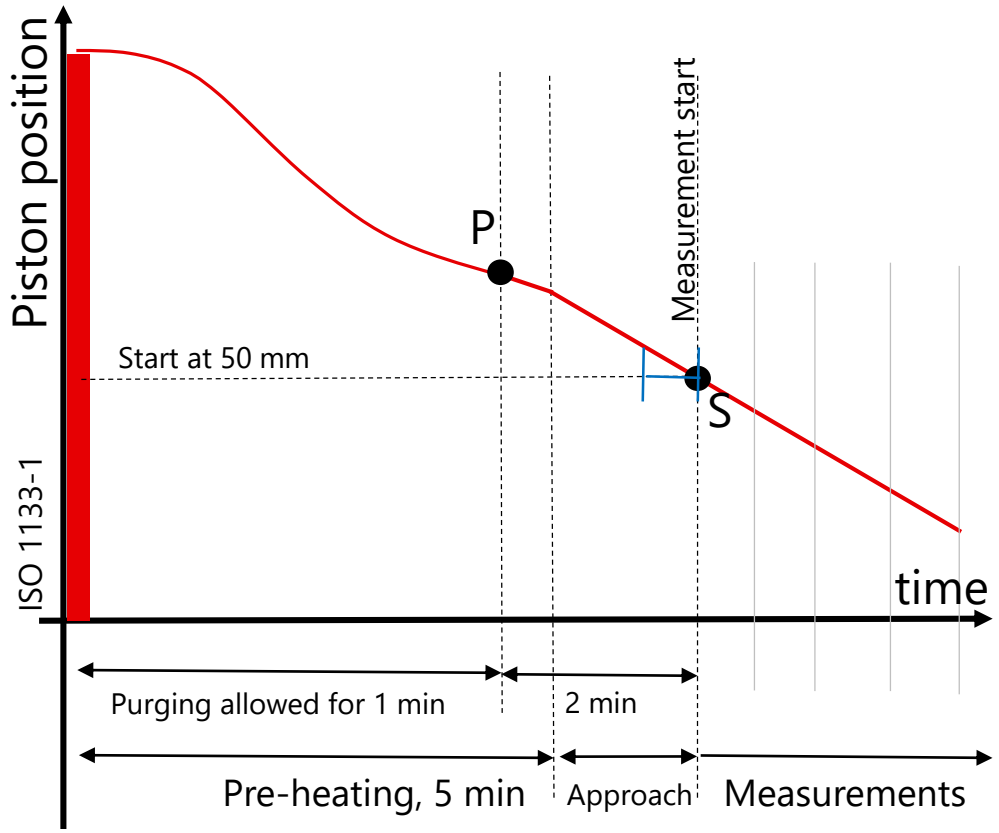
ISO 1133-1 is less strict regarding the test procedure. Part 2 defines narrow timing tolerances, similar to ASTM



ISO 1133-1 – Standard method

- The starting point of the measurement is defined in form of a piston position (50 mm above die)
- There is no strict tolerance for the approach-time between pre-heating and measurements

ISO 1133-2 – Moisture sensitive and time depending materials

- Strict definitions for the timing of the sequence
- 1 min for sample handling and charging
- The starting point of the measurement is defined in form of a piston position (50 mm above die)
- Allowed approach time between pre-heat and measurements is 30s to 60s
- The measurement shall start between 5,5 and 6 min after completion of charging

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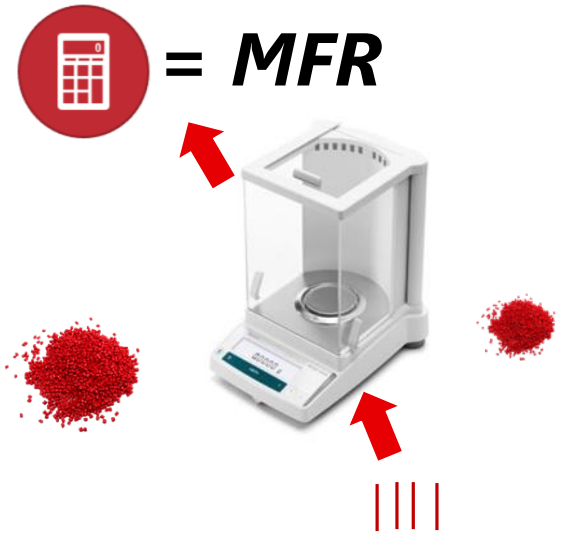
Method A represents a very manual test sequence for measuring the melt flow rate, MFR in g/10 min.

Test sequence to ASTM method A

- ❑ place the standard die (2.095 / 8 mm) into the well cleaned barrel
- ❑ Heat the instrument to the test temperature
- ❑ Determine the polymer amount for the test
  by estimation of the expected melt flow rate MFR
- ❑ Fill the material into the heated barrel
- ❑ Place the piston and the mass piece to be used for the test
- ❑ Purge within the first 5 min, or stop the piston movement at a piston land of 71 mm depending on the estimated melt flow rate MFR
- ❑ Start measurements after 7 ± 0.5 min of preheat time and at a piston land of 46 ± 2 mm
- ❑ Cut the extruded filaments in time intervals between 15 seconds and 6 minutes, depending on the estimated melt flow rate MFR
- ❑ Weight the cut extrudates individually to the nearest 0.1 mg
- ❑ Calculate the MFR value in g/10 min
- ❑ Clean the die, piston and barrel carefully for the next test.

➢ **According to ASTM D1238, reasonable measurements range from about 0.15 g/10 min to round about 50 g/10 min.**



Method A requires several manual operational steps and a good estimation of the expected MFR to fulfil all requirements set by the standard.

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Method B allows a fully automatic test sequence. The result is the melt volume rate, MVR, in cm^3/ 10 min.

Test sequence to ASTM method B

- Use a plastometer equipped with a transducer to measure piston position and travel
- Prepare the test as described for method A
- Purge within the first 5 min, or stop the piston movement at a piston land of 71 mm, depending on the present melt flow rate MFR
- Start measurements after 7 ± 0.5 min of preheat time and at a piston land of 46 ± 2 mm
- Determine the time needed for a piston travel of 6.35 mm (MVR ≤ 10) or 25.4 mm (MVR > 10), depending on the present melt flow rate MFR
- Calculate the MVR value in cm^3/10 min
- Calculate the MFR value in g/10 min using the melt density of the polymer
- Clean the die, piston and barrel carefully for the next test.

➢ **According to ASTM D1238, reasonable measurements range from about 0.5 g/10 min to round about 1500 g/10 min when using a die plug. High quality plastometers such as the Mflow can measure an even larger MVR range with good accuracy and precision.**

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Method C is used for polymers of high flow rate. The “half size“ die reduces the MVR by about a factor of 4.

Test sequence to ASTM method C

- Method C is a variant of method B, used to measure polymers of low viscosity, means high melt volume rate.
- The “half size” die being used has a bore of 1.048 mm and is 4 mm high
- Start measurements after 7 ± 0.5 min of preheat time and at a piston land of **50 ± 2 mm** (slightly higher compared to method B)
- Determine the time needed for a piston travel of 25.4 mm
- Calculate the MVR value in cm^3/10 min
- Calculate the MFR value in g/10 min using the melt density of the polymer
- Clean the die, piston and barrel carefully for the next test.

➢ **The melt volume rate appears to be about 4 times lower compared to method B. This makes it much easier to measure polymers of very low viscosity with good precision.**

Method D is used to perform MVR measurements under different loads from a single charge of polymer.

Multi-weight sequence to ASTM method D

- The method is used to determine the melt flow rate MFR under different loads.
- Weights may be changed in increasing or decreasing direction
- Measurements under two or more weight levels are possible
- Start the first measurement after 7 ± 0.5 min of preheat time and at a piston land of 46 ± 2 mm
- Determine the time needed for a predefined piston travel
- Change the weight and wait until the flow is stabilized
- Determine the time needed for a predefined piston travel for the next load
- Calculate the MFR value in g/10 min for each load step used
- Calculate the Flow Rate Ratio, FRR, as the quotient between the MFR measured at different loads
- Clean the die, piston, and barrel carefully for the next test.

➢ **The Flow Rate Ratio indicates the behavior of a polymer melt at different loads and therefore at different shear rates. This gives an information about the shear-thinning property of a polymer melt which is itself related to the molecular mass distribution.**

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Organizing melt index measurements in a lab can be a quite demanding task

There are lots of things to organize

- Actual and traceable calibration of all instruments
- Lab routines on how to handle specimen material
- Pre-conditioning of the polymer
- Polymer-related test parameters, such as
  - The standard to be used
  - The method to be used (A, B, C, or D)
  - Test temperature
  - Test load

Furthermore, for each single polymer grade:

Melt Flow testing requires an estimation of the result for correct parameter setting

- Amount of material to be tested (2.5g to 8 g)
- Use of a piston stop or die plug for high flow-rate materials
- Loading and purging sequences during pre-heat phase
- Different procedures to correctly meet the starting point (i.e. 46 ± 2 mm, 7 ± 0.5 min) for the measurement
- Use of correct travel or time intervals for the measurement according to the selected method and the Melt Flow Rate present.

(Method A: Time intervals ranging from 15 s to 6 min, Method B: travel intervals either 25.4 mm or 6.35 mm)

Simplified testing using the Aflow plastometer

19.09.2023 ASTM D 1238:2023 Helmut Fahrenholz

Zwick/Roell


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The Aflow offers the most simple programming.

All you need to do …

- ❑ Select ASTM D1238, method B
- ❑ Select temperature and test load
- ❑ Fill 5 g of polymer into the plastometers barrel
- ❑ Start the test

✓The Aflow knows how to perform the test !

No need for any further program settings or administration of test procedures

Cflow Mflow Aflow

MFR and MVR measurement

- Standard-compliant to ISO and ASTM
- Efficient and reliable
- Unmatched automatic functions

Cflow
manual, for occasional measurements

Mflow
modular system that grows along with your requirements

Aflow
reliable test results even in harsh production environments Optionally with accessories for controlled pre-compaction, cleaning and intelligent recognition of the optimum test parameters

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