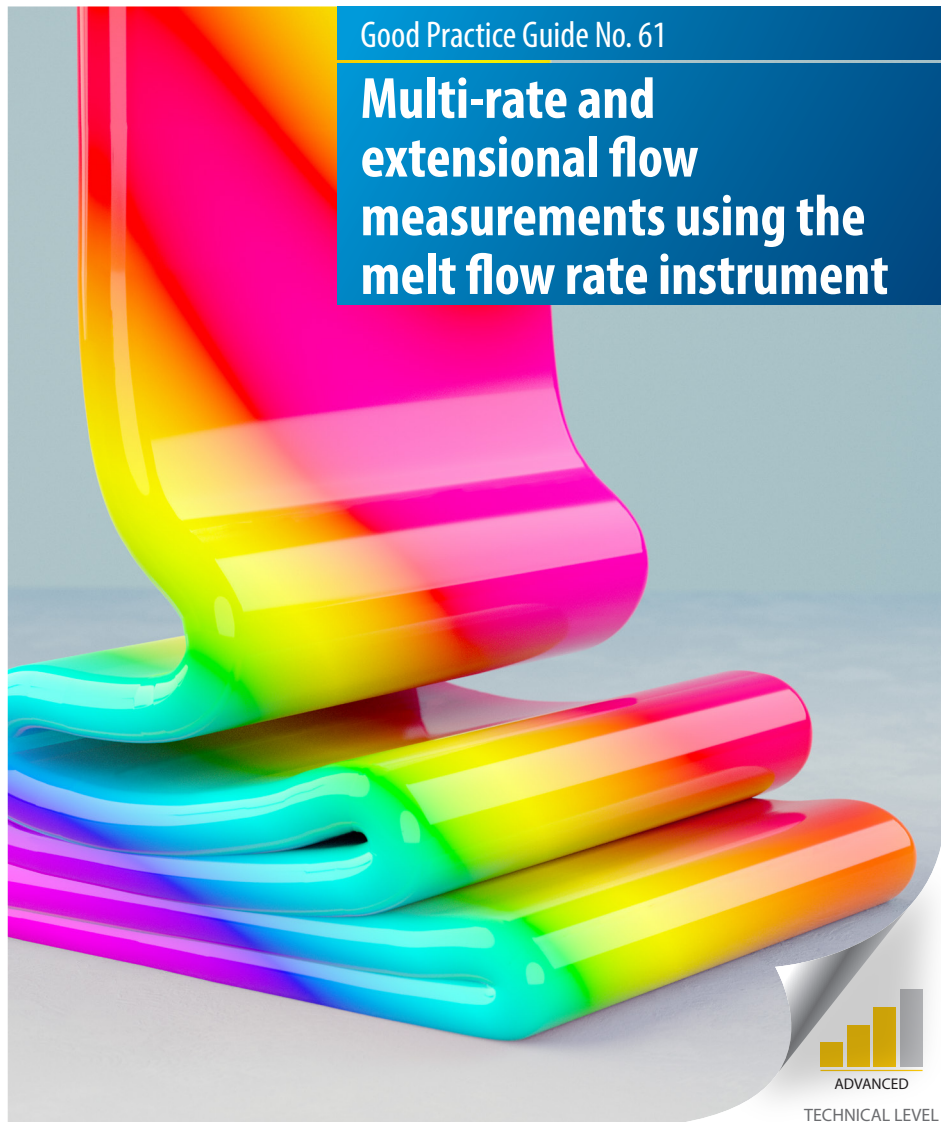




Good Practice Guide No. 61

Multi-rate and extensional flow measurements using the melt flow rate instrument



ADVANCED

TECHNICAL LEVEL

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Abstract

This guide presents the procedures and analyses necessary to carry out testing using new methods based on the use of the melt flow rate equipment for characterising the shear and extensional flow behaviours of polymer melts. These methods cover:

- multiple flow rate testing, obtained through the use of various loads,
- the use of an additional short-die geometry to separately quantify shear and extensional flow effects, and
- extrudate swell and draw-down measurements, to assess the materials extensional flow behaviour.

The multi-load test method will enable melt flow rate data to be extrapolated more reliably to higher shear rates. The use of the short die geometry and the measurement of extrudate draw-down will address the characterisation of the extensional flow behaviour of polymers that is often critical to their processability.

These methods provide improved, yet affordable, materials quality control testing for polymers. Thus it is expected that the guide will be of particular value to those involved in quality control testing of materials, in particular where the production processes are dominated by extensional or stretching flows.

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Chapter 1: Scope

1. Scope

This guide presents the procedures and analyses for new methods based on the use of the melt flow rate equipment for characterising the shear and extensional flow behaviours of polymer melts. These methods cover:

- multiple flow rate testing, obtained through the use of various loads,
- the use of an additional short-die geometry to separately quantify shear and extensional flow effects, and
- extrudate swell and draw-down measurements, to assess the materials extensional flow behaviour.

The multi-load test method will enable melt flow rate data to be extrapolated more reliably to higher shear rates. The use of the short die geometry and the measurement of extrudate draw-down will address the characterisation of the extensional flow behaviour of polymers that is often critical to their processability. These methods provide improved, yet affordable, materials quality control testing for polymers.

Chapter 2: Introduction

2. Introduction

The melt flow rate method, or melt flow index as it was historically known, has been in existence for several decades. It is globally used for materials specification and quality control in the plastics industry. It fulfils a requirement for rapid materials characterisation, specifically for checking the quality of materials and for assessing their processability, both of these in terms of the materials' ease of flow. The method is tightly specified by standards, e.g. ISO 1133 [1]. This, combined with the method's simplicity and relatively low cost are its strengths for quality control and materials specification purposes. However, it is widely accepted within the industry that the method has limitations, which have been reviewed elsewhere [2-5]. Two of the main limitations are addressed here. Firstly, the method is typically used as a single point method, i.e. a single value is obtained to describe the complex viscoelastic flow behaviour of the polymer melt. Thus by nature of its simplicity the melt flow rate is obviously a very crude descriptor. Secondly, the output of the existing method is dominated by the shear flow behaviour of the material. As many production processes involve considerable extensional flow deformation, e.g. film blowing and blow moulding, a method that characterises the material in a predominantly shear flow deformation is not necessarily appropriate, and misleading information on the processability of the material may result.

In progressing these developments an important objective was to maximise the potential uptake by industry of such measurement technology. The melt flow rate technique is too ingrained in the global polymer industry for the successful introduction of a replacement or substantially modified method. Therefore the adopted approach has been to consider the development of methods that can be carried out using the melt flow rate instrument. These additional measurements should be viewed initially as being complimentary to the existing melt flow rate measurements. Once their value to industry has been demonstrated then there is potential scope for considering their use instead of the standard method. Furthermore, through these additional methods the capability and value of the melt flow rate instrument is enhanced as it can then be used to obtain additional, appropriate information on the flow behaviour of polymer melts.

This guide presents some of the recent work carried out to address the limitations identified in the melt flow rate method by providing affordable developments to the method. These developments entail:

- the measurement of melt flow rate at higher shear rates, thus making the method more reliable in predicting the performance of materials in processing, and
- the characterisation of the extensional flow behaviour of polymer melts: a property that is important in both processing and quality control yet is normally overlooked by traditional characterisation methods. This is achieved through two basic methods:
 - i) the use of a short die to determine entrance pressure drop data, and
 - ii) by the measurement of the draw-down behaviour of the extrudate.

The basis for the selection of these methods is detailed in a report by Rides [6].

In summary, the guide:

- describes the existing melt flow rate method (Chapter 3)
- describes the multi-rate method (Chapter 4)
- describes the use of short dies for determination of shear viscosity and entrance pressure drop data, the latter being related to extensional viscosity behaviour (Chapter 5)
- describes the use of extrudate swell and draw-down measurements to characterise the extensional flow behaviour (Chapter 6)
- summarises the methods and presents practical advice and defines good practice on their use (Chapter 4 - 7)
- illustrates the new measurement methods by presenting example results obtained on a range of materials by NPL (Chapter 4 - 6)
- presents detailed analyses of the existing and new melt flow rate methods (Appendices A1 – A6)

For the extensional flow measurements an obvious question is “Which method should I use?”. The answer depends largely on the materials being tested and the industrial process by which they are to be processed. A good, simple guide is to use the method that mimics, most closely, the process. Thus for extrusion, the short die technique would be appropriate. Provided the material can be measured using the standard melt flow rate die then there is unlikely to be a problem measuring it using a short length die (albeit of different diameter perhaps). For blow moulding, particularly the parison forming stage, then the extrudate draw-down method would be appropriate. However, there are obvious limitations to the extrudate draw-down technique such as the extrudate must be able to support itself. Furthermore, it must not draw down too rapidly nor too slowly as these will result in measurement problems and consequently unsatisfactory results. However, this can be controlled to some extent by selecting the load to yield a melt volume flow rate value that is up to ≈ 4 . Results are presented that illustrate these methods and thus provide further guidance on the selection of the appropriate technique.

Chapter 3: Existing melt flow rate method

3. Existing melt flow rate method

The melt flow rate method, put simply, is to measure the quantity of material (pre-heated in a barrel) that is extruded through a die in a given time when a specified weight is applied to the piston, Figure 1. The current standard ISO 1133 [1] covers two procedures, specifically the melt mass flow rate (MFR) and the melt volume flow rate (MVR). The difference between these two measures is that in the former the mass of material extruded in a given time is measured, and in the latter the volume of material extruded is measured. Thus a single figure is obtained for either MFR or MVR that characterises the flow behaviour of the material. MFR is expressed in g/10 minutes and MVR in cm³/10 minutes. The MFR and MVR are thus measures of the ease of flow: the higher the melt flow rate number the easier the material flows. The term “melt flow rate” is used herein to indicate both MFR and MVR methods. An analysis of the melt flow rate method in its normal use is presented in detail in Appendix A2.

The MVR is preferred as a measure of the flow behaviour of a material as it is independent of its density and is thus a true measure of its flow properties. To illustrate this point, when comparing materials of different densities, e.g. two materials having different filler levels, the filler content will affect the MFR value through its contribution to the density (the density of the filler usually being significantly greater than that of the polymer). Furthermore, to illustrate its importance, MVR is specified by the CAMPUS database [7] rather than MFR.

All melt flow rate testing conditions are tightly specified by the ISO standard ISO 1133. The only parameters that are allowed to vary are the test load and the temperature. However, for any given type of material, e.g. polypropylene, there is normally only one set of test conditions permitted: that set having been selected or optimised for that class of material. As an exception, and indicative of the wider range of grades available, for polyethylene there are four permitted loads although the temperature is the same in each case. However, the criteria for selection of the loads are also tightly specified leaving the operator no choice.

The ISO standard for the melt flow index testing is ISO 1133: Plastics - Determination of the melt mass flow rate (MFR) and melt volume flow rate (MVR) of thermoplastics. The other significant standard that specifies the procedure for melt flow rate testing is ASTM D1238 ‘Standard test method for flow rates of thermoplastics by extrusion plastometer’.

Chapter 4: Higher flow rate test

- Background
- Method
- Procedure
- Example results

4. Higher flow rate testing using the melt flow rate instrument

4.1 Background

A frequent shortcoming of the melt flow rate method is the inability to use the data generated on a material to predict with any degree of confidence or accuracy its processing behaviour. Frequently the melt flow rate values are obtained at very low shear rates (see Table 1) and the use of such data to predict processing behaviour at higher shear rates can be problematic. However, the development of the method to higher flow rates, which requires greater loads, is limited by the desire to use existing melt flow rate instruments and their current load capacities. Also, in extending testing to higher rates the divide between melt flow rate testing and capillary extrusion rheometry testing will be reduced. This divide is not sufficient to warrant the introduction of a further instrument. To improve on this position it is therefore considered appropriate that the extrapolation of data to higher rates requires improvement, rather than developing the method to test at significantly higher rates. The developments reported describe a method for generating data that can then be extrapolated to higher rates thus providing an ability to compare more reliably the processability of different materials.

4.2 Method

All melt flow rate measurements were made using a standard Ray-Ran Test Equipment Limited 5MPCA Advanced Melt Flow System instrument. Although application of the new methods described here are not restricted to this instrument there may be a need to modify the procedures described to take into account variations between different instruments. The 5MPCA Advanced Melt Flow System instrument has a linear displacement transducer that allows the region over which the measurements are taken to be split into many segments. In the measurements reported herein, 20 separate determinations of melt flow rate were made per barrel charge, Figure 2. Multi-load tests were carried out using standard melt flow rate loads. Typically, for a single barrel charge, three or four different loads could be applied. Thus for each load in a multi-load test approximately 5 separate determinations of melt flow rate were obtained. It was found preferable to apply the loads in increasing value as this allowed the operator, who manually applied the load, to exert better control over the progress of the test. For automated loading systems this requirement may not be necessary, Figure 3. A further advantage of using the multi-segment capability of the instrument is that it is easier to assess the stability (degradation or cross-linking) of the polymer during a test at constant conditions by monitoring any changes in melt flow rate values as the test progresses.

In all cases, except where specified, the procedures adopted for using the melt flow rate instrument will be as specified in the standard ISO 1133.

The multi-rate test method applies primarily to instruments capable of performing automated measurement of the melt volume flow rate (method B of ISO 1133), although it is expected that it can equally be applied to the manual melt mass flow rate method (method A of ISO 1133) provided the melt flow rate values are not too high resulting in a very short test duration. The MVR method is preferred for reasons of accuracy and reduced effort on behalf of the operator in carrying out the testing.

4.3 Procedure

1. The procedure, except where specified below, is the same as that specified in the standard ISO 1133.
2. Select the range of loads to be used for the multi-load test.

At least three loads should be used to enable reliable extrapolation of data to higher shear rates to be made.

Use a wide range of loads, where possible, to maximise the range of melt flow rate and hence shear rate values obtained from the testing. Again this improves the extrapolation to higher shear rates.

One of the loads should be that specified for the material by ISO 1133. Other loads should preferably straddle that value. However, more emphasis should be on using higher rather than lower loads if the data are principally to be used to predict how the material will behave in processing at higher shear rates.

WARNING: *The maximum applied load using the instrument at NPL was limited to 25 kg for reasons of safety as the loads were manually handled. However, for other instruments assessment of the safety of such operations must be performed and appropriate limits set.*

3. Apply the lowest load first and then subsequently the higher loads in increasing weight order.

Increasing the applied load, rather than decreasing it is preferable as it allows greater control over the progress of the test. For automatically loaded instruments this may not be the case and a decreasing loading regime may be preferred.

4. Record the melt flow rate values for each of the loads applied. To achieve this, sampling may be done at a different interval to that specified in the standard ISO 1133. This may be necessary due to the rate at which the test proceeds.

It is important to ensure that any transients in behaviour due to adding or removing the loads do not significantly affect the measured melt flow rate values. Where multi-load tests on a single barrel charge are performed ensure that sampling is

carried out such that any transients are allowed to decay before measurements are made. Where measurements are made over a number of intervals, as is the case for the multi-segment approach of the instrument used herein, discard those intervals over which a change in load was made. Also discard any subsequent intervals for which transients are evident (e.g Figure 2, test reference rr010, Measurement segment 15).

5. Present the results in the form:

MVR / applied load / temperature

e.g. 5 cm³/10mins / 2.16 kg / 190 °C.

or in such a manner that the load and temperature for each MVR value can be identified (as described above MVR are preferred, but this does not exclude the presentation of MFR values).

6. To extrapolate the data to the processing conditions of interest plot the logarithm of melt flow rate values as a function of the logarithm of load, Figure 4. Such a plot will tend to be reasonably linear and enable more reliable extrapolation to be made to higher loads, and therefore to higher shear rates. To evaluate at what shear rates the various melt flow rate values corresponded to see Table 1 (Appendix A2, equations A2.19 or A2.10).

MVR cm ³ / 10minutes	Apparent shear rate 1/s
0.1	0.185
0.3	0.554
1	1.85
3	5.54
10	18.5
30	55.4
100	185
300	554
1000	1850

Table 1: Equivalence of MVR and apparent shear rate values

A plot of the logarithm of apparent shear rate versus the logarithm of load may also be helpful for comparing the behaviour of materials, given the range of shear rates that occur in the production process for which the materials are being considered.

4.4 Example results

The effect of the applied load on the melt flow rate can be clearly seen for a linear low density polyethylene (LLDPE) at 190 °C, Figure 2. The test progresses from left to right, the x-axis indicating the test segment from 1 to 20. At the change-over from one load to another, spurious results may occur for that test segment: these have been included here but should be discarded, as is the case for results presented subsequently, as appropriate (e.g Figure 2, test reference rr010, Measurement segment 15). As expected, a larger applied load results in a higher measured melt volume flow rate. Thus it is demonstrated that the flow behaviour over a wide range of rates can be determined by applying loads that are specified in standards, i.e. 2.16 kg, 3.8 kg, 5 kg and 21.6 kg in this case. The lower load of 0.325 kg specified in ISO 1133 has not been used for this material as the MVR value would have been too small for reliable determination. Similarly the additional loads of 1.2 kg and 10 kg have also not been used as it was preferable to obtain data over a wider range of loads as possible rather than to use all the loads available.

The results of 12 tests performed on a high density polyethylene (HDPE), each comprising of one or more loads from the range 2.16 kg, 5 kg, 12.5 kg and 21.6 kg, are presented in Figure 3. These results clearly show the effect of load on the measured MVR and that, within scatter, the results are independent of applying either an increasing or decreasing load. Previous oscillatory rheometry measurements [8] indicated that this material was stable for 2 hours at 150 °C. It is also noted that the first result per test, i.e. test segment 1, is often spurious due to transients at the start of the test and that it is important to disregard such results if possible.

To extrapolate the data to higher loads, and thus effectively higher shear rates, the data for a HDPE have been plotted in Figures 4 and 5 (these data are indicated by the legend caption incorporating “standard die” as data obtained using short dies are also presented in these plots and are discussed in the next chapter). Extrapolation to higher melt flow rate values and thus higher shear rates is obviously reasonably straight-forward given the relative linearity of the data when plotted on logarithmic scales.

Chapter 5: Shear viscosity

- Background
- Method
- Procedure
- Example results

5. Shear viscosity and entrance pressure drop data determination using standard and short dies

5.1 Background

The use of a single long die in an extrusion experiment can only provide, at best, an approximation to shear viscosity data. To obtain accurate shear viscosity values at least one extra die has to be used. If a very short length die is used then a measure of the extensional flow behaviour can also be determined. The principle behind the use of a short length die is to obtain a measure of the resistance to flow in a converging region, characterised by the “entrance pressure drop”. This can then be related to the extensional flow behaviour of the material [8 - 13]. This method is simple, yields quantitative data and is potentially cheap. The basis for the method, when used in a controlled flow rate mode, is presented in detail by Rides and Allen [10]. The approach is now applied to the controlled stress mode (i.e. applied load) as used for MFR and MVR measurements.

5.2 Method

To provide the simplest experimental case to determine shear viscosity and entrance pressure drop data a die of very short length was used in addition to the standard length MFR die (shear viscosity corrected for entrance effects). The short dies used were either 1 mm or 2.095 mm in diameter and had a nominal length of 0.25 mm.

5.3 Procedure

1. The procedure, except where specified below, is the same as specified in the standard ISO 1133.
2. Testing may be carried out using only a single load or with multiple loads as specified in Chapter 4.
3. The standard die is replaced by the short length die. Insert a spacer below the short die such that the top of the die is in the same position as is the case for the 8 mm length standard die. All dies are to be manufactured to the tolerances, surface finish and hardness and using materials as specified in ISO 1133.
4. Present the results in the form:

MVR / applied load / temperature / die length / die diameter

e.g. 5 cm³/10mins / 2.16 kg / 190 °C / L = 4 mm / D = 2.095 mm

using units, L and D to avoid potential confusion. Alternatively, the results can be expressed in the form:

MFR / applied load / temperature / die length / die diameter

e.g. 5 g/10mins / 2.16 kg / 190 °C / L = 4 mm / D = 2.095 mm

or such that all test conditions corresponding to the measured melt flow rate data are preserved.

The theory that describes the operation of the melt flow rate instrument in its normal use is presented in detail in Appendix A2. Extensions of that theory to the use of the instrument for determining entrance pressure drop data using a short die, and the subsequent correction of standard melt flow rate data to determine reliable shear viscosity data are presented in Appendices A3 and A4 respectively.

5.4 Example results

Multi-load results obtained using a standard die and a short die are presented for a high density polyethylene tested at 190 °C, Figure 4. The use of a standard die of length 8 mm or a short die of length 0.26 mm (same diameter as the standard die, i.e. 2.095 mm) results in different MVR values being obtained. Obviously the short die presents a lower resistance to flow and thus the MVR values are correspondingly higher, in this case by a factor of approximately x6, Figure 4.

However, by using a smaller diameter, short die (diameter 1 mm and length 0.24 mm) then MVR values of similar magnitude to those obtained using a standard die can be achieved, Figure 5. This can be of particular benefit when measuring materials that have a high MVR value using the standard die. For such materials, testing using a short die of 2.095 mm diameter would result in very high MVR values that may cause measurement problems and consequently large errors.

MFR values obtained using both the standard and short dies for a HDPE are presented in Figure 6. These and additional data have been interpreted as shear viscosity and entrance pressure drop data, as indicated in Appendices A3 and A4, and presented in Figures 7 to 9. The repeatability of measurements of entrance pressure drop is good, Figure 8. The effect of the die diameter on the entrance pressure drop as a function of apparent shear rate is also shown to be negligible, Figure 9, thus indicating that a smaller diameter die may be used with confidence to determine entrance pressure drop data for use in correcting shear viscosity data. Capillary extrusion rheometry measurements were made on the same materials for comparative purposes using a Rosand twin-bore rheometer, and the results are also presented, Figures 7 to 9. Good agreement between melt flow rate and capillary extrusion results was obtained. Generally, the level of agreement of results was within approximately

10%, thus providing confidence in the use of a melt flow rate instrument for generating quantitatively accurate rheological data.

It is noted that the breadth of the shear rate range that can easily be achieved by both capillary extrusion rheometers and melt flow rate instruments is similar. For the melt flow rate instrument the range of loads over which measurements can easily be made is one decade, i.e. 2.16 kg to 21.6 kg (not including the use of the lower load of 0.325 kg). Similarly, for capillary extrusion rheometry the standard ISO 11443 [14] specifies that measurements should not be made using the pressure transducer outside the range of 10% to 90% of their normal capacity. Thus the breadth of the load ranges, and hence shear rate ranges, of the two instruments are very similar. Obviously the pressure transducer in the extrusion rheometer can be changed for one of a different range thereby extending the capability of the instrument further, though this obviously results in an increase in the time and hence cost of testing.

Chapter 6: Extrudate swell measurement

- Background
- Method and theory
- Procedure
- Example results

6. Extrudate swell and draw-down measurements

6.1 Background

Both the extrudate swelling and the draw-down behaviours are characteristic of the flow behaviour of the polymer, in particular its viscoelastic extensional behaviour. However there are many factors, both experimental and material dependant, that contribute to the extrudate swell and therefore complicate both its measurement and application to industrial practice. Nevertheless, the measurement of extrudate swell and extrudate draw-down will correlate directly with the behaviour of melts in extrusion and blow moulding (parison sag) respectively. Whereas parison sag is predominantly governed by the extensional flow behaviour of the material, extrudate swell is a more complex phenomenon that is governed by the full rheological behaviour of the material. Nevertheless, for polymer melts the extensional flow behaviour can have a significant affect on the extrudate swell behaviour and, more importantly, extrudate swell is a measure that is of direct relevance to converters. It is thus considered a very valid measurement to pursue as a quality control procedure.

The reproducibility of extrudate swell results can be very poor due to the large number of variables within the test method that affect the swelling behaviour [15]. The use of the melt flow rate instrument significantly reduces these variables to a minimum. A significant feature of the method is the non-isothermal behaviour of the measurement that cools the extrudate and controls [limits] the amount that the sample draws down. Draw-down will only occur to any significant extent in the melt phase. The behaviour of the material in such a test is complex but it closely resembles many polymer processes which is its strong point as a quality control method.

6.2 Method and theory

In summary, measurements of extrudate diameter and hence draw-down were taken during a standard, single-load melt flow rate test. Extrudate swell measurements were made on the extrudate, cooled to room temperature after completion of the test, using a hand-held digital micrometer. Approximate extensional viscosity values are deduced from the change in diameter of the extrudate as the test progresses.

Automated, non-contact extrudate swell measurements were also made using a Mitutoyo Laser Scanning Micrometer (LSM-3000) mounted on the melt flow rate instrument. Data thus obtained have been used for comparative purposes but not for determining approximate extensional viscosity data. The measurement of extrudate diameter was at approximately 45 mm beneath the exit of the die. The load was selected to result in an MVR value of not more than approximately 4. As a consequence of this, when the extrudate reached the point at

which the laser micrometer was scanning, its outer surface had frozen and therefore changed relatively little subsequent to that point.

Theory to interpret the extrudate draw-down behaviour in terms of extensional viscosity is presented in Appendix A5. Although the measurement is not simple, in that it is under non-isothermal and variable suspended-load conditions, a more complex, rigorous analysis was considered unnecessary for the purposes of quality control. Briefly, the strain and time to strain, estimated from the extrudate diameter data, can be used to calculate approximate transient extensional viscosity values. The strain is determined from the reduction in cross sectional area of the extrudate, and the time taken to strain is obtained from the MVR and the extrudate dimensions. The stress can be determined from the suspended mass of extrudate and the extrudate dimensions. Thus an approximate extensional viscosity can be determined, as detailed as in Appendix A5.

The procedure below is for measurements made using the hand-held micrometer on the cooled extrudate. Laser micrometer measurements were not used for determining approximate extensional viscosity values.

6.3 Procedure

1. The procedure, except where specified below, is the same as specified in the standard ISO 1133.
2. Commence the melt flow rate test using a single load value that yields a MVR of not more than approximately 4.
3. Immediately cut off any extrudate suspended from the die exit.
4. Allow new extrudate to form beneath the die as the test progresses. During this phase the extrudate should be freely hanging from the die exit. Allow the test to progress until the extrudate almost reaches the base of the instrument (a drop of approximately 260 mm from the die in the case of the instrument used here).
5. When the extrudate has reached half way to the base of the instrument from the die, measure the distance from the die exit to the point at which the material crystallizes (crystallization can be observed visually in semi-crystalline materials by a change of appearance, e.g. clouding for transparent materials, and can also be observed for filled materials although slightly less obviously).
6. When the extrudate has almost reached the base of the instrument, remove the extrudate carefully, holding the top portion of the extrudate and allow it to cool to room temperature.

7. If possible, allow further draw-down tests to continue using the same barrel charge, and also allow the melt flow rate determination to finish.
8. Measure the diameter of the extrudate using a micrometer at 10 mm intervals¹ from the end first extruded, in two orthogonal (0° and 90°) orientations to obtain an average extrudate diameter as a function of position along the extrudate. Ignore the section of extrudate last extruded as this is most likely to be affected by the removal/handling process (e.g. for values obtained at positions greater than 220 mm in Figure 11).
9. Plot the results of average diameter versus position from the end first extruded. If necessary extrapolate the data back to the first point(s) which often exhibits an irregular trend, possible due to distortion due to the free end. If appropriate use the extrapolated data for the first point(s) rather than the experimental data.
10. From the melt flow rate test determine the melt volume flow rate for the material.
11. Using the analysis presented in Appendix A5 calculate the various parameters to yield strains, strain rates and tensile stresses, from which extensional viscosities can be calculated.

6.4 Example results

Hand-held micrometer and laser scanning micrometer results are presented for a linear low density polyethylene (HGF000 – Figures 10 and 11). Further results on other materials are presented by Rides et al [16]. These results demonstrate quite good repeatability of both methods. Furthermore, a comparison of the hand-held and laser micrometer results demonstrate a good level of agreement between methods, Figure 12.

Comparison of the behaviour of various grades - two high density polyethylenes (HGH000 and HFU000), a low density polyethylene (HGE000) and the linear low density polyethylene (HGF000) - which exhibited quite different extensional flow behaviours [10, 17], indicates significant differences in the extrudate swell and draw-down behaviours, Figures 13 and 14. These differences were exhibited in both the laser scanning micrometer and hand-held micrometer results. However, the loads used for these tests differed in order to obtain similar melt volume flow rate values of not more than approximately 4. The loads used were: HFU000 and HGE000 - 5 kg, HGH000 - 12.5 kg and HGF000 - 2.16 kg. Thus the interpretation of these curves is not straightforward. To interpret these data an analysis, which determines

¹ An interval of 10 mm has been used in the measurements reported here. Alternative values could be used.

approximate extensional viscosity values from this draw-down behaviour, is presented in Appendix A5.

A comparison of the derived approximate extensional viscosity values for these four polyethylenes is presented in Figure 15 along with tensile stress growth coefficient² data obtained from an extensional rheometer for the same materials [17]. The comparison of draw-down data with the maximum tensile stress growth coefficient data³ is not particularly valid as in the draw-down method the extrudate is stretched to a small degree (i.e. low strain) whereas in determining the maximum tensile stress growth coefficient data, the melt undergoes significantly higher strains. However, from those extensional tests data have been obtained at equivalent strains as those occurring in the draw-down tests and comparison is more appropriate. For all but the LLDPE the draw-down tests underpredict the values of extensional viscosity by a factor of approximately x2 to x3. However the trend is the same which, incidentally, is different to the trend exhibited by the maximum tensile stress growth coefficient values. The reason for the better agreement⁴ of the LLDPE data is considered to be due to the fact that the material is neither particularly strain hardening nor strain rate dependent and is thus affected less by the assumptions made in analysing the draw-down data.

Analysis of the sensitivity of derived approximate extensional viscosity data to input values, for example for a high density polyethylenes (HGH000) suggests that the method is not particularly sensitive to any of the input values, Figure 16.

Thus these draw-down measurements indicate the valuable qualitative assessment of the extensional flow behaviour of polymers melts that can be made simply using the melt flow rate instrument and a hand-held micrometer. It is perhaps important to stress that the full analysis of the draw-down in terms of an approximate extensional viscosity is not necessary for quality control purposes, but that the absolute values of extrudate swell at two positions along the extrudate and their ratio may well suffice.

² The tensile stress growth coefficient is the ratio of the tensile stress to strain rate. It is effectively a transient extensional viscosity.

³ Effectively the extensional viscosity at failure.

⁴ The agreement may be fortuitous to some degree due to the many assumptions made in analysing the raw data.

Chapter 7: Discussion

7. Discussion

Three methods have been presented that address the measurement of melt flow rate at higher shear rates (multi-load testing) and the characterisation of the extensional flow behaviour of polymer melts (through entrance pressure drop and extrudate swell and draw-down measurements). The latter, extrudate swell and draw-down, may be considered to be two methods but have been presented as one here, with most emphasis being placed on the draw-down behaviour.

The multi-load testing can be applied in theory to any material, although problems may be experienced for materials of very high or low viscosity for which the basic melt flow rate test is problematic anyway.

As discussed earlier, for the extensional flow measurements an obvious question is “Which method should I use?”. The answer depends largely on the materials being tested and the industrial process by which they are to be processed. A good, simple guide is to use the method that mimics, most closely, the process. Thus for extrusion, the short die technique would be appropriate. Provided the material can be measured using the standard melt flow rate die then there is unlikely to be a problem measuring it using a short length die (albeit of different diameter perhaps). For blow moulding, particularly the parison forming stage, then the extrudate draw-down method would be appropriate. However, there are obvious limitations to the extrudate draw-down technique such as the extrudate must be able to support itself. Furthermore, it must not draw down too rapidly nor too slowly as these will result in measurement problems and consequently unsatisfactory results. However, this can be controlled to some extent by selecting the load to yield a melt volume flow rate value that is up to ≈ 4 .

The benefits of these methods have been assessed through industrial case studies. Results are reported elsewhere [18].

Chapter 8: Summary

8. Summary

The melt flow rate method is widely used in the polymer industry and it is likely to remain as a dominant tool for quality control and assurance for some time. However, it is generally accepted within the industry that the melt flow rate method has limitations. To address these limitations various developments have been undertaken and presented. These developments address:

- the measurement of melt flow rate at higher shear rates (multi-load testing) - thus making the method more appropriate for predicting the performance of materials in processing, and
- the characterisation of the extensional flow behaviour of polymer melts (through entrance pressure drop and extrudate swell measurements) - a property that is important in processing yet is normally overlooked by traditional characterisation methods.

It is noted that these developments are not intended to replace the melt flow rate method but to supplement it to enhance the capability and value of the method to provide additional appropriate information on the flow behaviour of polymer melts thereby addressing some of the limitations identified.

In conclusion:

- this work has demonstrated that significantly more information can be obtained from the melt flow rate instrument, with only minimal modification, than is currently the practice,
- multi-rate data can be obtained with no additional equipment beyond the standard range of melt flow rate weights,
- entrance pressure drop and shear viscosity data corrected for entrance effects can be obtained using minimal additional equipment, i.e. a short length die,
- good agreement can be obtained between melt flow rate and capillary extrusion rheometry instruments for shear viscosity and entrance pressure drop,
- entrance pressure drop values as a function of shear rate are not significantly influenced by the die diameter in the melt flow rate test, and
- valuable qualitative measures of the extensional flow behaviour of the polymer can be obtained by measurement of the extrudate draw-down behaviour during melt flow rate tests.

Chapter 9: References and Figures

- References
- Figures

9. References and Figures

9.1 References

1. ISO 1133: Plastics - Determination of the melt mass flow rate (MFR) and melt volume flow rate (MVR) of thermoplastics.
2. Rides, M., Review of melt mass flow rate and melt volume flow rate measurement for polymer melts, NPL Report CMMT(A)231, October 1999.
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9.2 Figures

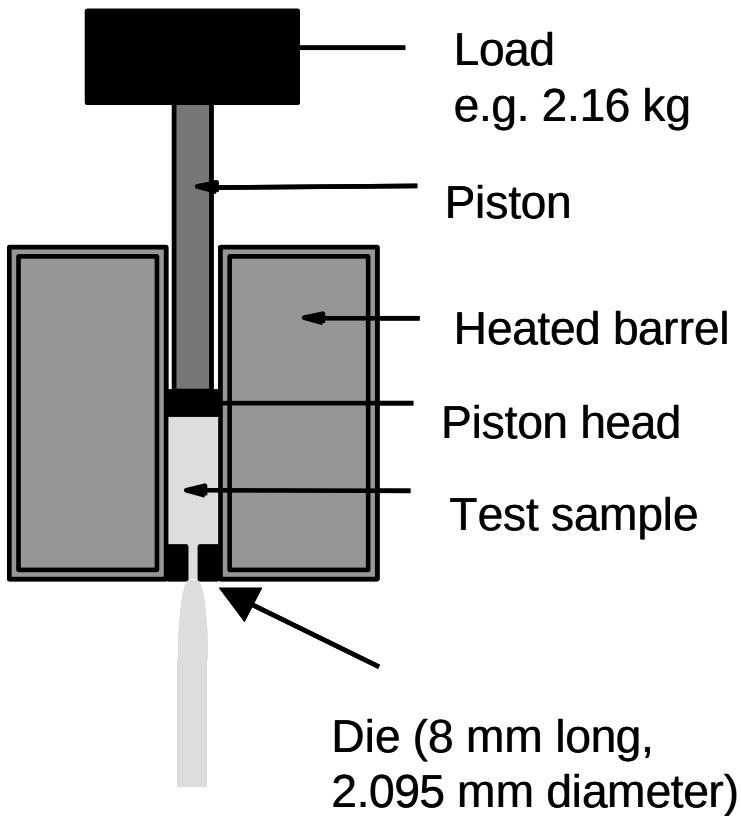


Figure 1 Schematic diagram of the melt flow rate apparatus.

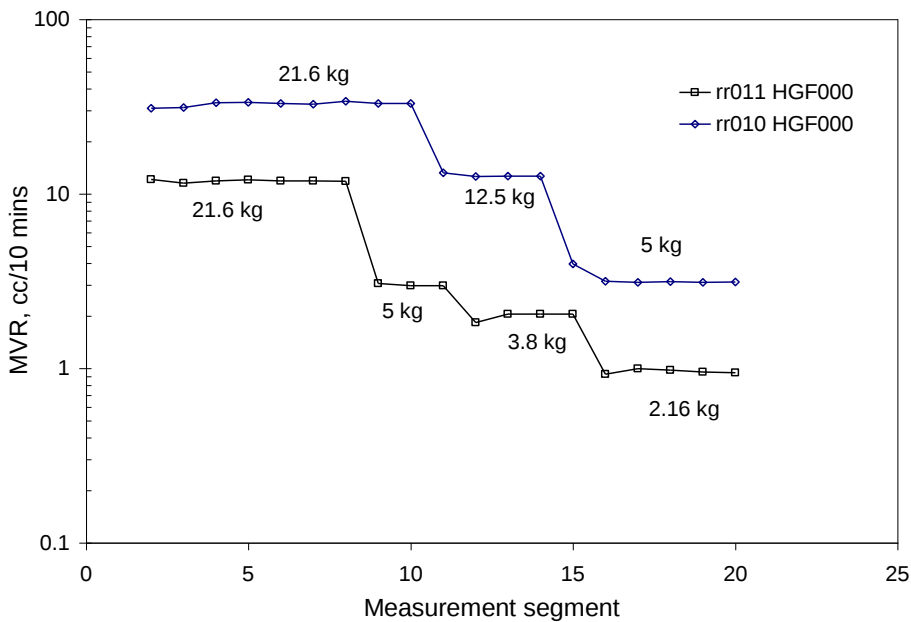


Figure 2 The effect of test load on melt volume flow rate values for a LLDPE at 190 °C Note: The “rr” value gives the melt flow rate test reference.

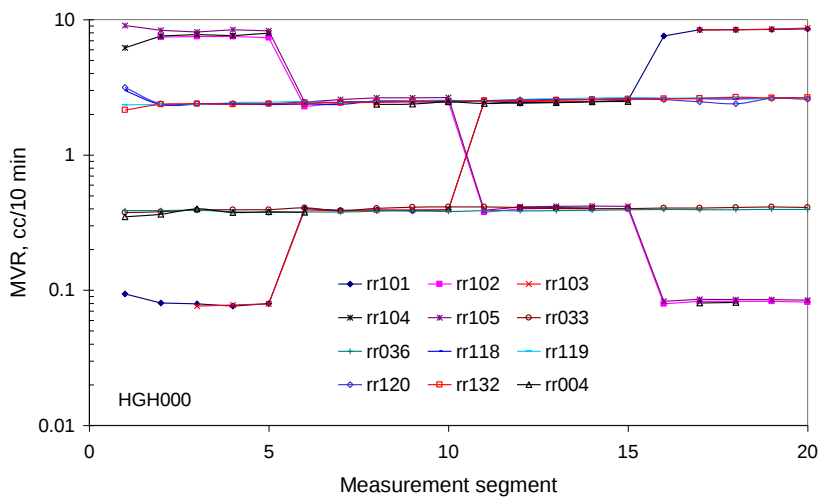


Figure 3 The effect of multi-load test order on melt volume flow rate for a HDPE (HGH000) at 190 °C.

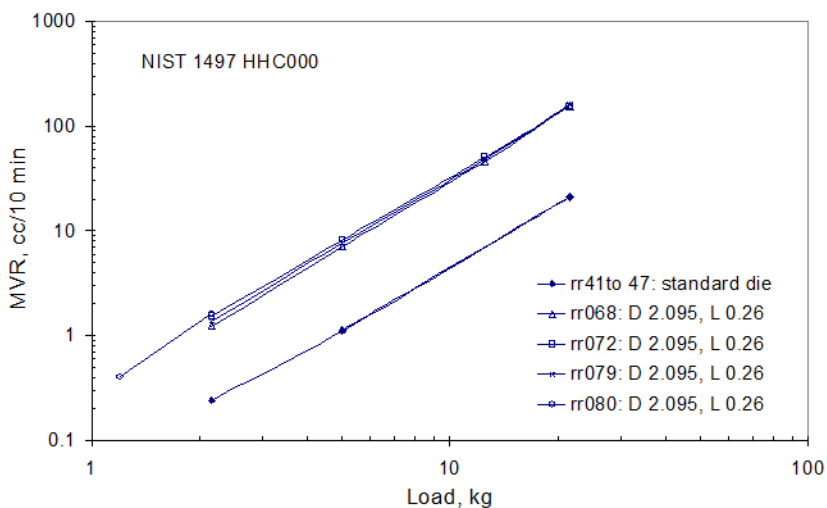


Figure 4 Comparison of melt mass flow rate values measured using a standard die with those measured using a short length die (0.26 mm) of standard diameter for a HDPE (HHC000) at 190 °C.

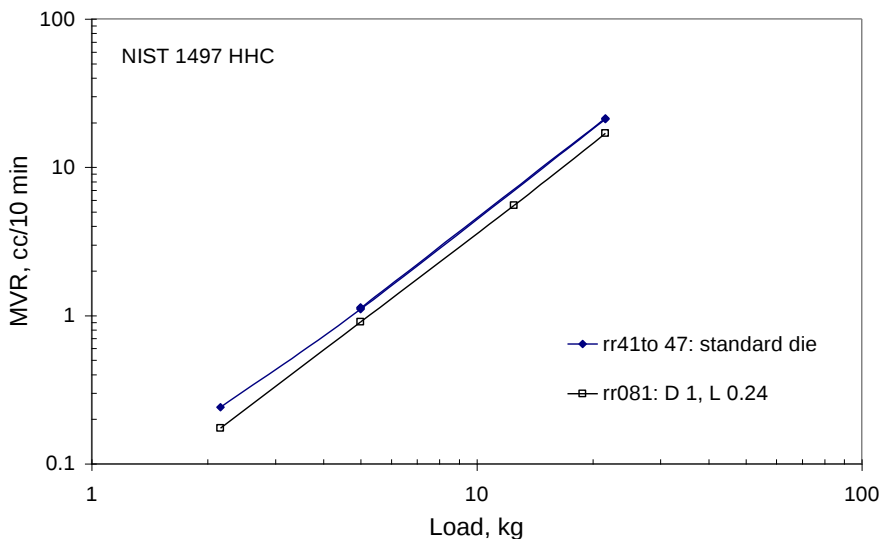


Figure 5 Comparison of melt mass flow rate values measured using a standard die with those measured using a short length die (0.24 mm) of smaller diameter (1 mm) (rr081) for a HDPE at 190 °C.

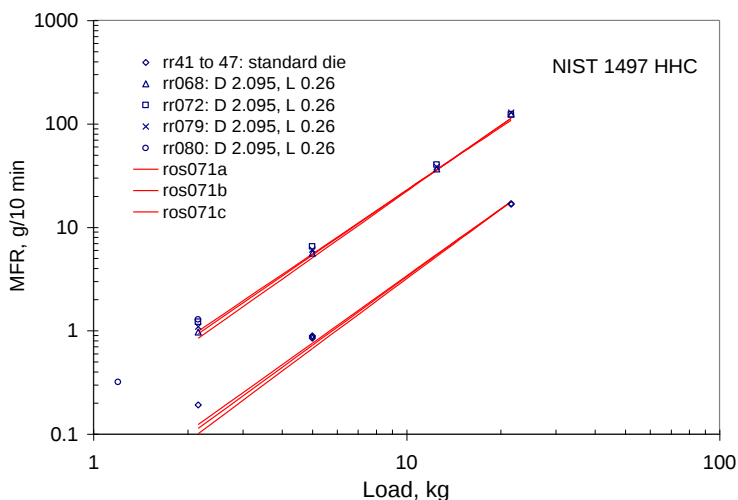


Figure 6 Comparison of predictions, using capillary rheometry data, of melt mass flow rate values measured using a standard die and using a short length die (0.26 mm) of standard diameter for a HDPE at 190 °C (lines are predicted values). Note: The “ros” value gives the capillary extrusion test reference.

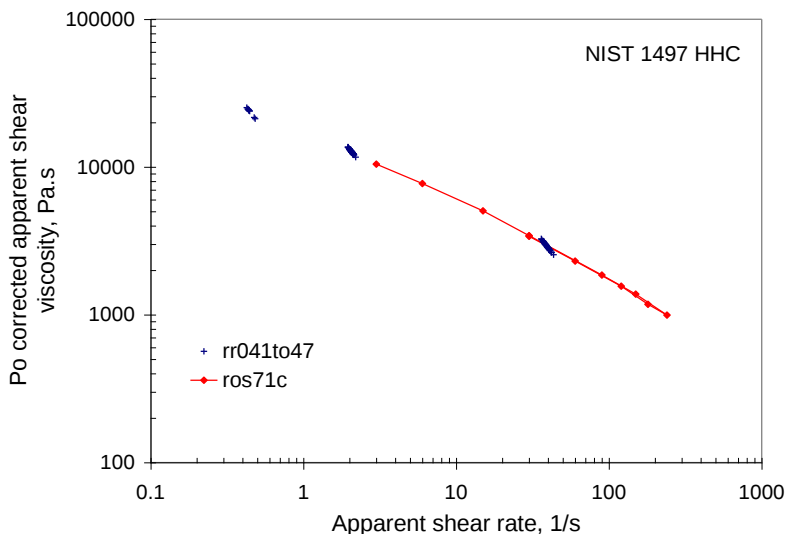


Figure 7 Comparison of shear viscosity values, corrected for entrance effects, measured using an extrusion rheometer (ros071c) with values measured using a melt flow rate instrument with a normal and a short die (0.26 mm length, 2.095 mm diameter) (rr041 to 47) for a HDPE at 190 °C.

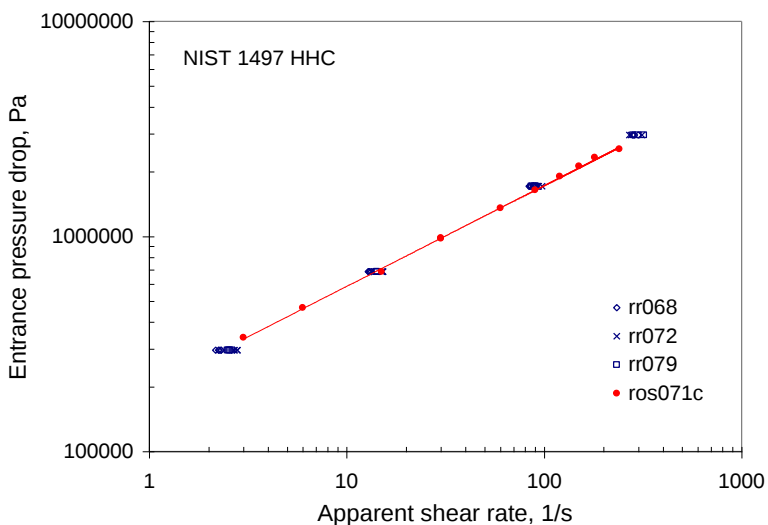


Figure 8 Repeatability of entrance pressure drop measurements using a melt flow rate instrument with a short die (0.26 mm length, diameter 2.095 mm) compared with extrusion rheometry data (ros071c) for a HDPE at 190 °C.

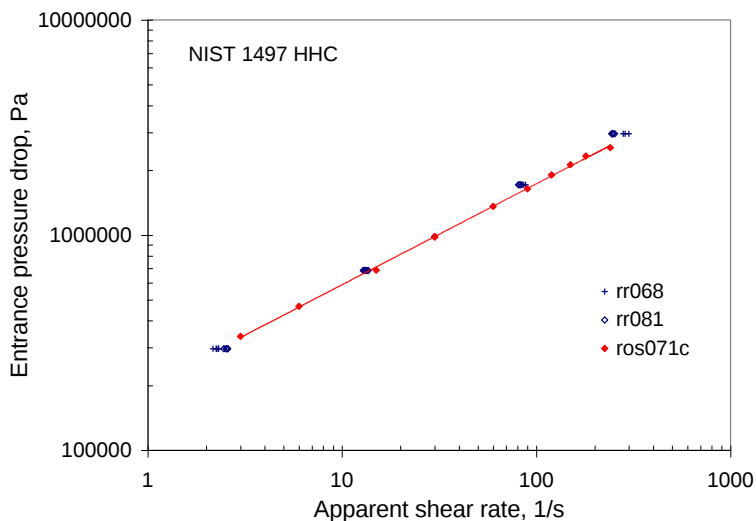


Figure 9 Comparison of entrance pressure drop values measured using an extrusion rheometer (ros071c) with values measured using a melt flow rate instrument with short dies (rr068 - 0.26 mm length, diameter 2.095 mm and rr081 - 0.24 mm length, diameter 1 mm) for a HDPE at 190 °C.

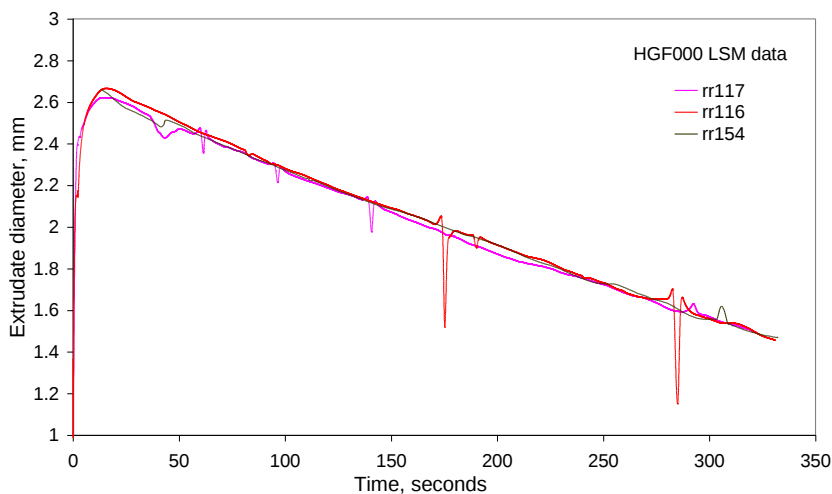


Figure 10 Repeatability of extrudate swell measurements using a laser scanning micrometer for a LLDPE (HGF000) at 190 °C and a load of 2.16 kg.

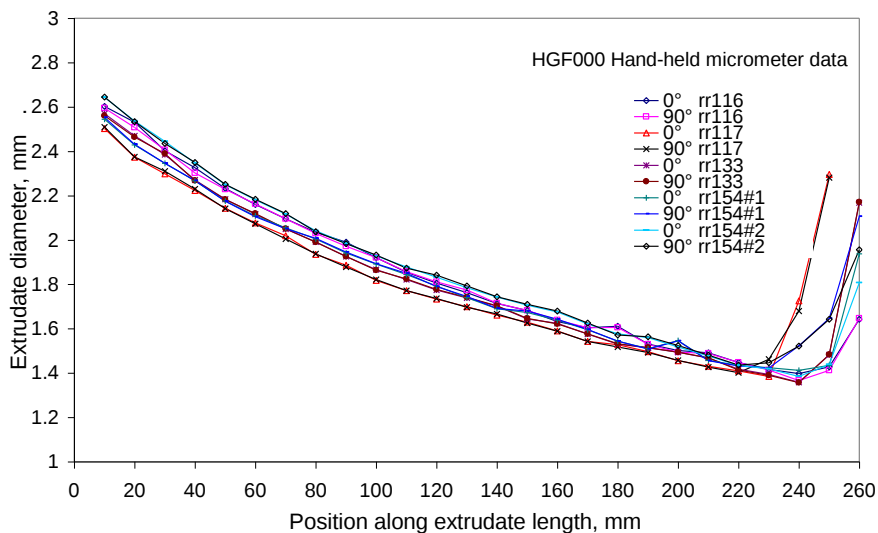


Figure 11 Repeatability of extrudate swell measurements using a hand-held micrometer for a LLDPE (HGF000) at 190 °C and a load of 2.16 kg.

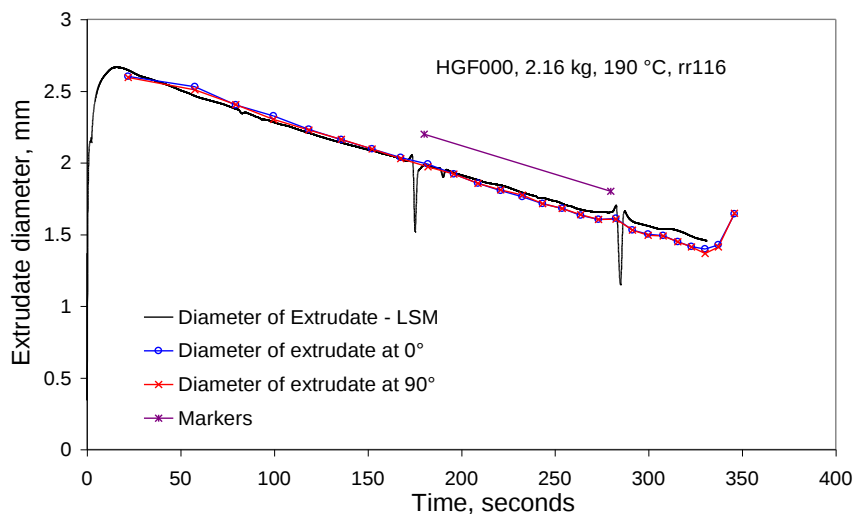


Figure 12 Comparison of laser-scanning and hand-held micrometer measurements of extrudate diameter for a LLDPE (HGF000) at 190 °C and 2.16 kg.

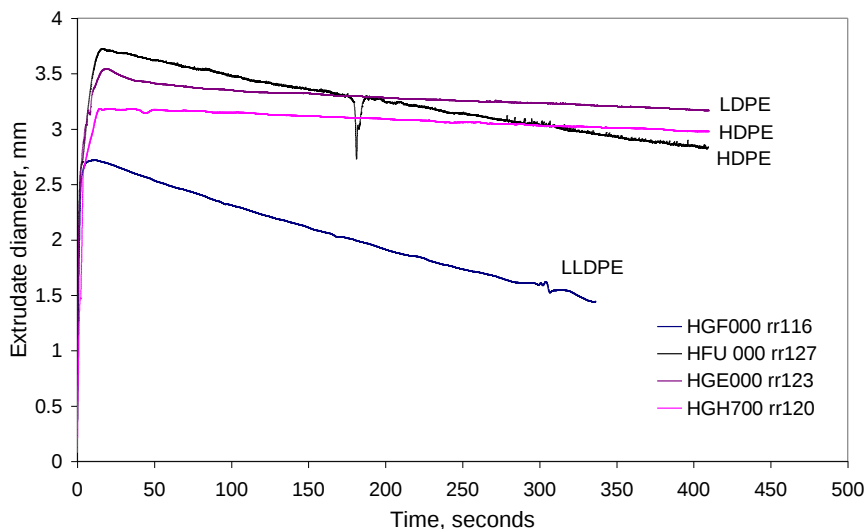


Figure 13 Comparison of laser-scanning micrometer measurements of extrudate diameter for a range of polyethylenes.

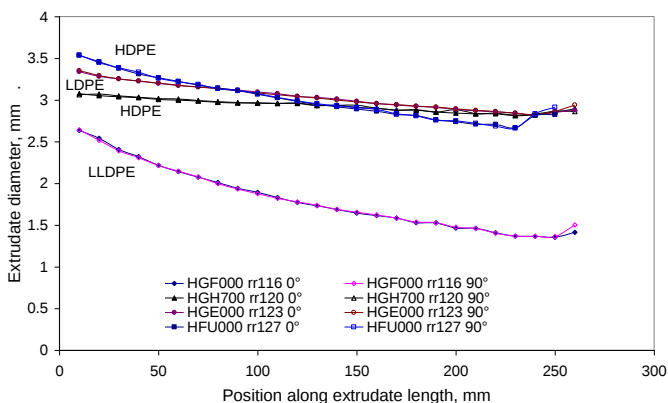


Figure 14 Comparison of hand-held micrometer measurements of extrudate diameter for a range of polyethylenes.

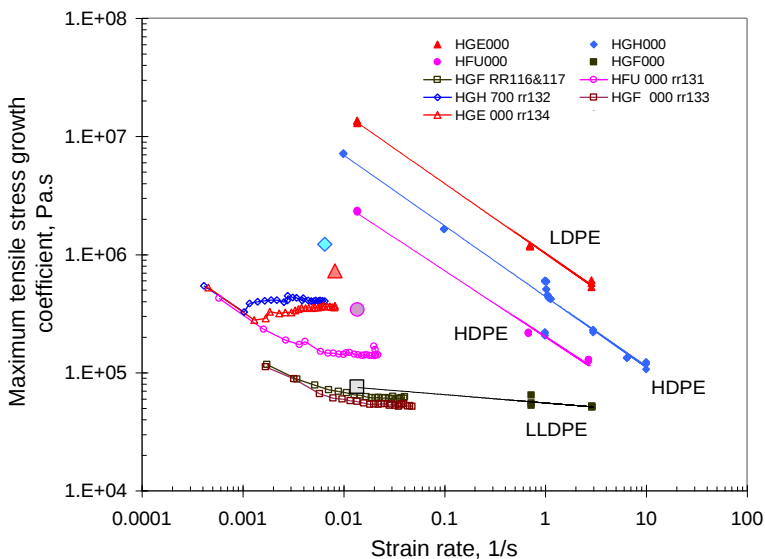


Figure 15 Interpretation of hand-held micrometer measurements of extrudate diameter as approximate extensional viscosities for a range of polyethylenes. [Solid points with lines indicate maximum tensile stress growth coefficient values. Open points with lines indicate draw-down values. Large single point symbols indicate values determined from tensile extensional testing at strains equivalent to that for the highest strain rate data points from draw-down measurements for each material. Symbols indicate materials as for other tests. Tensile extensional data obtained at 150 °C.]

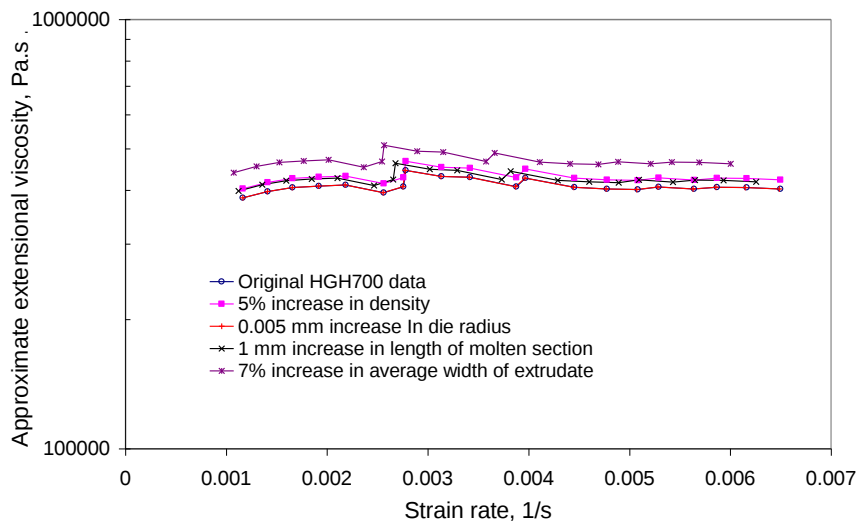


Figure 16 Sensitivity analysis of derived extensional viscosity values from draw-down data for a HDPE (HGH000).

Chapter 10: Appendices

- Appendix A1: Nomenclature
- Appendix A2: Analysis of melt flow rate testing
- Appendix A3: Entrance pressure drop method
- Appendix A4: Correction for entrance effects
- Appendix A5: Extrudate draw down analysis
- Appendix A6: Analysis of viscous heating in the flow

Appendix A1: Nomenclature

For the purpose of this document the following terminology is used, except where otherwise specified:

MFR melt mass flow rate has units of grams although it is expressed as g/10 minutes indicating it is the mass in grams extruded in 10 minutes

MVR melt volume flow rate has units of cm³ although it is expressed as cm³/10 minutes indicating it is the volume extruded in cm³ in 10 minutes

m is the mass extruded in time t, grams

t_{ref} is a reference time equal to 600 s (≡ 10 minutes)

ρ is the density of the polymer melt at the test temperature, g/cm³

ℓ is the distance moved by the piston in time t, m

t time, s

Q volume flow rate, m³/s

$\dot{\gamma}_a$ apparent shear rate, s⁻¹

τ_a apparent shear stress in the die (not Bagley end-corrected), Pa

τ shear stress in the die (Bagley end-corrected), Pa

η_a apparent shear viscosity (not Bagley end-corrected), Pa.s. It is the ratio of apparent shear stress to apparent shear rate.

η_a apparent shear viscosity (Bagley end-corrected), Pa.s. It is the ratio of shear stress to apparent shear rate.

P pressure drop, Pa

P_e entrance pressure drop, Pa

P_s shear flow pressure drop along the capillary die, Pa

D barrel diameter, m

R die radius, m

L die length, m

P₀, x constants in the entrance pressure drop power law model

Nomenclature specific to the draw-down analysis

R_0	radius of the extrudate at the die exit (= die radius), m
R_c	radius of the extrudate at the crystallisation point, m
R_1	radius of the extrudate first extruded, m
L_1	length of extrudate from the crystallisation point to the lower end, m
L_0	length of extrudate from the die exit to the crystallisation point, m
M	mass of extrudate below the crystallisation point, kg
σ	tensile stress acting on the mid-point of the molten section, Pa
V_{r1}	volume of extrudate below the crystallisation point, m ³
A_1	cross-sectional area of the extrudate first extruded, m ²
A_c	cross-sectional area of the extrudate at the crystallisation point, m ²
V_{av}	average velocity of the extrudate in the molten section, m.s ⁻¹
t_0	time for a particle to traverse the molten section, s
$\dot{\epsilon}_{av}$	average strain rate in the molten section, s ⁻¹
ϵ	Hencky strain, dimensionless

Appendix A2: Analysis of melt flow rate testing

General

The melt mass flow rate (MFR) is given, according to ISO 1133 [1], by the expression

$$MFR = \frac{m t_{ref}}{t} \quad (A2.1)$$

and is determined experimentally by measuring the mass of material m extruded in a given time t and scaled appropriately using a reference time t_{ref} of 600 seconds (10 minutes) thereby determining the amount of material that would have been extruded in 10 minutes. The relationship

$$MVR = \frac{MFR}{\rho} \quad (A2.2)$$

is also given in ISO 1133 relating the melt mass flow rate (MFR) to the melt volume flow rate (MVR) where ρ is the density of the polymer melt at the test temperature. MFR has units of grams although it is expressed as g/10 minutes indicating it is the mass extruded in 10 minutes. MVR has units of cm^3 , although similarly it is expressed as $\text{cm}^3/10$ minutes indicating it is the volume extruded in 10 minutes.

Thus the melt volume flow rate is given by the expression

$$MVR = \left[\frac{m t_{ref}}{t} \right] \times \frac{1}{\rho} \quad (A2.3)$$

Alternatively, as the quotient of the mass extruded to its density is equivalent to the volume extruded then this may be re-written as

$$MVR = \left[\frac{t_{ref}}{t} \right] \ell \frac{\pi D^2}{4} \times 10^6 \quad (A2.4)$$

Where ℓ is the distance moved by the piston in time t and D is the diameter of the barrel. The factor of 10^6 is introduced here and elsewhere below to account for the differences in units used for the various parameters (for units see Nomenclature Appendix A1). MVR is determined experimentally by measuring the time taken for the piston to move a given distance or vice-versa.

Apparent shear rate

The volume flow rate Q can be determined from the MVR and is given by

$$Q = \frac{MVR \times 10^{-6}}{600} \quad (\text{A2.5})$$

or, substituting for MVR using equation A2.4, by

$$Q = \frac{\ell \pi D^2}{4t} \quad (\text{A2.6})$$

The apparent shear rate $\dot{\gamma}_a$ in the melt at the wall of the die is given [14, 18] by

$$\dot{\gamma}_a = \frac{4Q}{\pi R^3} \quad (\text{A2.7})$$

and thus in the melt flow rate die, substituting for Q using equation A2.6, by

$$\dot{\gamma}_a = \frac{\ell D^2}{R^3 t} \quad (\text{A2.8})$$

Alternatively, the apparent shear rate is given, using equations A2.5 and A2.7, by

$$\dot{\gamma}_a = \frac{4(MVR / 600) \times 10^{-6}}{\pi R^3} \quad (\text{A2.9})$$

or in terms of MFR by

$$\dot{\gamma}_a = \frac{4(MFR / 600 \rho) \times 10^{-6}}{\pi R^3} \quad (\text{A2.10})$$

The apparent shear rate is the shear rate that would have occurred if the material exhibited a Newtonian behaviour, i.e. the viscosity is independent of shear rate, and is a convenient measure for describing the flow. In practice, polymer melts are non-Newtonian, i.e. they are shear rate thinning. The true shear rate $\dot{\gamma}$, Rabinowitsch corrected for the non-Newtonian velocity profile resulting from the shear thinning behaviour, is given by

$$\dot{\gamma} = \left(\frac{3n+1}{4n} \right) \dot{\gamma}_a \quad (\text{A2.11})$$

or

$$\dot{\gamma} = \left(\frac{3n+1}{4n} \right) \frac{4Q}{\pi R^3} \quad (\text{A2.12})$$

where n is a power law index. The value of $n-1$ is equivalent to the gradient of the plot of $\log(\text{apparent shear viscosity})$ versus $\log(\text{apparent shear rate})$.

Apparent shear stress and apparent shear viscosity

The pressure P above the die, assuming zero pressure loss due to fluid flow in the barrel and no frictional forces between the piston and the barrel, is determined from a balance of forces acting on the piston. Thus

$$Wg = \frac{P\pi D^2}{4} \quad (\text{A2.13})$$

where W is the applied load and g is the acceleration due to gravity. Thus the pressure P above the die is given by

$$P = \frac{4Wg}{\pi D^2} \quad (\text{A2.14})$$

This pressure is “lost” through viscous resistance by material flowing into and along the length of the die. Assuming no entrance pressure drop, a balance of forces along a die of length L and radius R yields

$$P\pi R^2 = 2\pi RL\tau_a \quad (\text{A2.15})$$

Where τ_a is the apparent shear stress in the die at the wall.

Thus

$$\tau_a = \frac{PR}{2L} \quad (\text{A2.16})$$

or, substituting for P using equation A2.14, by

$$\tau_a = \frac{4Wg}{\pi D^2} \frac{R}{2L} \quad (\text{A2.17})$$

An apparent shear viscosity η_a (not Bagley end-corrected) can be simply defined for the MFR and MVR test as the ratio of the apparent shear stress to apparent shear rate

$$\eta_a = \frac{\tau_a}{\dot{\gamma}_a} \quad (\text{A2.18})$$

Thus the apparent shear viscosity is given, using equations A2.10 and A2.17, by

$$\eta_a = \frac{300Wg\rho R^4}{D^2 L \times MFR \times 10^{-6}} \quad (\text{A2.19})$$

or in terms of MVR by

$$\eta_a = \frac{300WgR^4}{D^2 L \times MVR \times 10^{-6}} \quad (A2.20)$$

Thus for each load an apparent shear viscosity (not corrected for entrance effects) and the corresponding apparent shear rate can be determined. In using several loads a flow curve can thus be determined.

An analysis of the effect of viscous heating in the melt flow rate test is presented in Appendix A6.

Appendix A3: Determination of the extensional flow behaviour using the entrance pressure drop method

The extensional flow characterisation method is based on the measurement of the material's resistance to flow in a converging region. The degree of resistance to flow, characterised by the entrance pressure drop, is used as a measure of the extensional flow behaviour of the polymer. The basis for the method, when used in a controlled flow rate mode, is presented in detail by Rides and Allen [10, 19]. Briefly, by using either a short die or dies of different length but of the same diameter the magnitude of the entrance pressure drop can be determined. The approach is now applied to the controlled stress mode as used for melt flow rate measurements.

To provide the simplest experimental case a die of short length (typically 0.25 mm) is used. In using a short die it is assumed that the total pressure drop is due solely to the entrance pressure drop, i.e. there is no shear flow pressure drop along the length of the short die, and thus the die is effectively of zero length. Then the entrance pressure drop P_e is given by

$$P_e = \frac{4Wg}{\pi D^2} \quad (\text{A3.1})$$

where W is the applied load, g is the acceleration due to gravity and D is the barrel diameter.

For these tests the apparent shear rate is given by

$$\dot{\gamma}_a = \frac{4(MVR_s / 600) \times 10^{-6}}{\pi R^3} \quad (\text{A3.2})$$

or in terms of MFR by

$$\dot{\gamma}_a = \frac{4(MFR_s / 600\rho) \times 10^{-6}}{\pi R^3} \quad (\text{A3.3})$$

where the subscript s indicates the MVR or MFR values determined using the short die.

Thus in using a short die for a given load a single datum point for entrance pressure drop and the corresponding apparent shear rate can be determined. By performing tests using different loads with the short die, the apparent shear rate dependence of the entrance pressure drop P_e can be determined. A power-law model has been found useful for fitting such data [10]:

$$P_e = P_o (\dot{\gamma}_a^x) \quad (\text{A3.4})$$

where P_o and x are constants.

Appendix A4: Correction of apparent shear viscosity values for entrance effects

The entrance pressure drop is not normally negligible for polymer melts and consequently there is an error in the apparent shear viscosity values determined when using only one long die, as presented in Appendix 2. For this reason those values are referred to as apparent shear viscosity values that are not corrected for the Bagley effect: the Bagley effect referring to the entrance pressure drop correction. For the standard MFR die this can result in an error of the order of approximately 30%. To correct for this the following approach is used.

Assuming a non-zero entrance pressure drop P_e , the pressure drop along the die length P_s is given by

$$P_s = P - P_e \quad (\text{A4.1})$$

where P is given by equation A2.14. Re-writing equations A2.14 and A2.20 in terms of pressure yield

$$\eta_a = P \left[\frac{75\pi R^4}{L \times MVR \times 10^{-6}} \right] \quad (\text{A4.2})$$

or in terms of MFR by

$$\eta_a = P \left[\frac{75\rho\pi R^4}{L \times MFR \times 10^{-6}} \right] \quad (\text{A4.3})$$

Thus to determine shear viscosity values corrected for entrance effects only the pressure drop due to resistance to shear flow must be used in the above two equations. Thus substituting P by P_s (using equations A2.14 and A4.1) yields

$$\eta_a = \left(\frac{4Wg}{\pi D^2} - P_e \right) \frac{75\pi R^4}{L \times MVR \times 10^{-6}} \quad (\text{A4.4})$$

or in terms of MFR by

$$\eta_a = \left(\frac{4Wg}{\pi D^2} - P_e \right) \frac{75\rho\pi R^4}{L \times MFR \times 10^{-6}} \quad (\text{A4.5})$$

where P_e is the entrance pressure drop calculated at the apparent shear rate occurring for the standard die test, and MFR and MVR refer to the values also obtained using the standard 8 mm length die. P_e is calculated using equation A3.4. The apparent shear rates used for the determination of P_e are given by

$$\dot{\gamma}_a = \frac{4(MVR / 600) \times 10^{-6}}{\pi R^3} \quad (\text{A4.6})$$

or in terms of MFR by

$$\dot{\gamma}_a = \frac{4(MFR / 600\rho) \times 10^{-6}}{\pi R^3} \quad (\text{A4.7})$$

Thus from the apparent shear rate versus entrance pressure drop data determined using the short die and various loads the magnitude of the entrance pressure drop at any apparent shear rate can be derived by interpolation or extrapolation using, for example, a power-law fit, equation A3.4.

It is noted that it may be necessary to extrapolate short die MFR data to carry out the correction to some of the standard MFR data. Such extrapolations should be carried out with care as the accuracy of extrapolated values can be poor, and is dependant on the quality and quantity of the data used to extrapolate. Statistical and uncertainty analyses can provide further information on the confidence that one can place on such extrapolated values.

Thus shear viscosity data, obtained using the standard die, can be corrected for entrance pressure drop effects using short die data.

Appendix A5: Extrudate draw down analysis

This analysis is for interpreting the straining behaviour of molten extrudate immediately beneath the die exit. The straining, or draw-down, occurs due to the weight of extrudate hanging on the molten section. It is, in effect, parison sag. The assumptions are made that the geometry of the extrudate is defined by two truncated cones joined end-to-end, by their smaller ends that are of the same diameter to maintain continuity. The point at which the two cones meet is taken to be the point at which the surface of the extrudate crystallises. The upper cone is therefore of molten material whereas the lower cone is effectively of solidified material. All deformation therefore occurs in the upper cone. The distance from the die exit to the point of crystallisation is also assumed to be constant throughout the test.

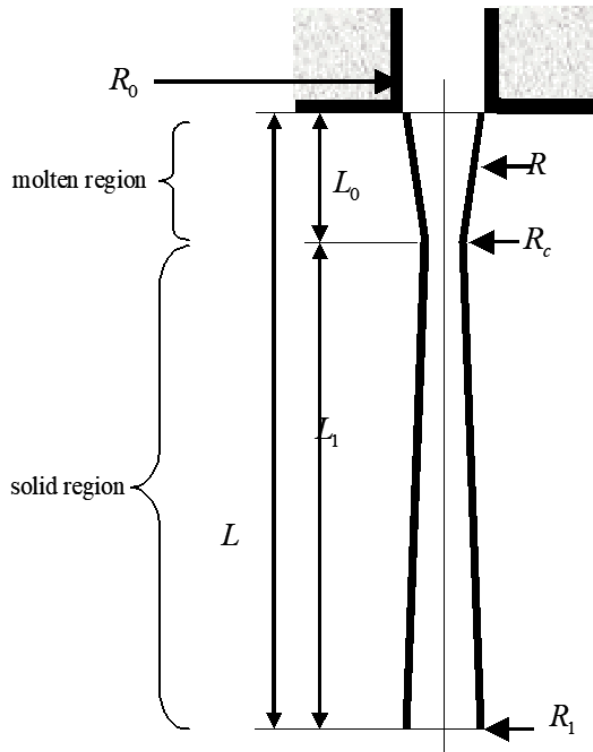


Figure A1: Schematic diagram of draw-down in melt flow rate testing (R_c is the point at which the surface crystallizes)

The volume of the lower truncated conical section below the crystallisation point ($r = R_c$) is given by

$$V_{R1} = \frac{1}{3} \pi L_1 (R_c^2 + R_1 R_c + R_1^2) \quad (\text{A5.1})$$

Thus the mass M of extrudate suspended beneath the crystallisation point ($r = R_c$) is given by

$$M = V_{R1} r \quad (\text{A5.2})$$

where r is the density which is assumed to be constant.

The stress s at the mid point along the length of the upper conical section

(where $r = (R_o + R_c)/2$) is estimated by

$$\sigma = \frac{Mg}{\pi \left(\frac{R_c + R_o}{2} \right)^2} \quad (\text{A5.3})$$

[Note: As the length of the upper truncated conical section is small compared with that of the lower section then the contribution of its lower half, via its mass, to the stress at the mid-point of the upper section is assumed to be negligible.]

The strain e that the material undergoes due to draw down is related to the reduction in cross-sectional area of the extrudate. Assuming that the material first extruded undergoes no strain due to draw-down, as no load is suspended from it, then the strain of material at the crystallisation point is given by

$$\varepsilon = \ell n \left(\frac{A_1}{A_c} \right) \quad (\text{A5.4})$$

where A denotes the cross-sectional area at the positions indicated by the suffices. Thus

$$\varepsilon = \ell n \left(\frac{R_1^2}{R_c^2} \right) \quad (\text{A5.5})$$

The flow rate is determined from the measured melt volume flow rate by

$$Q = \frac{MVR \times 10^{-6}}{600} \quad (\text{A5.6})$$

Thus the average velocity V_{av} of a particle traversing from the die exit to the crystallisation point (i.e. L_0) is estimated by

$$V_{av} = \frac{Q}{\pi \left(\frac{R_0 + R_c}{2} \right)^2} \quad (A5.7)$$

The time taken t_0 for a particle to traverse from R_0 to R_c is given by the ratio of the length traversed to the average velocity thus

$$t_0 = \frac{L_0}{V_{av}} \quad (A5.8)$$

The average strain rate for a particle traversing from R_0 to R_c is given by the ratio of strain to time

$$\dot{\epsilon}_{av} = \frac{\epsilon}{t_0} \quad (A5.9)$$

Finally an approximate extensional viscosity η_e is given by

$$\eta_e = \frac{\sigma}{\dot{\epsilon}_{av}} \quad (A5.10)$$

Thus by appropriate substitution of these equations an approximate extensional viscosity value can be derived from the draw-down data where the extrudate radius R_c (i.e. the radius frozen at a particular moment) is measured as a function of the length L_1 from the end that was first extruded.

Thus, by substitution

$$\eta_e = \frac{Mg}{\pi \left(\frac{R_c + R_0}{2} \right)^2} \frac{t_0}{\ell n \left(\frac{R_1^2}{R_c^2} \right)} \quad (A5.11)$$

It is considered, given the assumptions made above, that the most reliable values for extensional viscosity will be obtained when the mass of suspended extrudate is largest and thus the stresses, strains and strain rates that the material in the molten zone is experiencing are also largest. This corresponds to data obtained towards the end of the test.

Hencky strain

The *Hencky strain* ϵ (also referred to as the natural or true strain) is given by the natural logarithm of the elongation ratio

$$\epsilon = \ln(\ell / \ell_o) \quad (\text{A5.12})$$

where ℓ and ℓ_o are the current and original specimen lengths. Assuming conservation of volume of the specimen then

$$\ell_o A_o = \ell A \quad (\text{A5.13})$$

where A and A_o are the current and original specimen cross sectional areas. Substitution using areas for lengths in equation A5.12 yields

$$\epsilon = \ln(A_o/A) \quad (\text{A5.14})$$

Appendix A6: Analysis of viscous heating in the flow

This analysis is done on the basis of an energy balance and assumes that all the work done by the piston is converted to heat in the sample. The work done by the piston per unit time w_F is given by

$$w_F = \frac{Wg\ell}{t} \quad (\text{A6.1})$$

The energy in heating the flowing material per unit time is given by

$$w_\theta = \rho C_p \Delta\theta \frac{\ell}{t} \frac{\pi D^2}{4} \quad (\text{A6.2})$$

where ρ is the melt density, C_p is the specific heat capacity and $\Delta\theta$ is the temperature rise in the sample. Thus from the energy balance

$$w_F = w_\theta \quad (\text{A6.3})$$

then

$$\rho C_p \Delta\theta \frac{\ell}{t} \frac{\pi D^2}{4} = \frac{Wg\ell}{t} \quad (\text{A6.4})$$

or, using equation A2.14

$$\Delta\theta = \frac{Wg}{\rho C_p \left(\frac{\pi D^2}{4} \right)} = \frac{P}{\rho C_p} \quad (\text{A6.5})$$

Thus using typical values for a polymer

$$W = 2.16 \text{ kg}$$

$$g = 9.81 \text{ m.s}^{-2}$$

$$D = 9.55 \times 10^{-3} \text{ m}$$

$$\rho = 1000 \text{ kg/m}^3$$

$$C_p = 2000 \text{ J/(kg.K)}$$

the maximum average temperature rise $\Delta\theta$ is estimated to be $\approx 0.15^\circ\text{C}$ for a 2.16 kg load test. For a 21.6 kg load test the effect is estimated to be $\approx 1.5^\circ\text{C}$ and for a 50 kg load it is $\approx 3.4^\circ\text{C}$.

These estimates assume that all the energy was dissipated as heat evenly through the specimen and that no heat was dissipated from the melt to the surrounding environment. These factors would result in a reduction of the temperature increase. For a HDPE (HGH000)

the shear flow temperature dependence was estimated to be less than 1 %/°C. The effect on the flow behaviour can thus be estimated. Uncertainty analysis has shown that a 1.5 °C error in set temperature would result in a 3% - 4% error in MVR for such a material. The effect on MFR and MVR testing would be expected to be less as these temperatures would be the maximum reached, i.e. that at the exit of the die: the average temperature of the material in the die would be less. The effect of the lower load of 2.16 kg is therefore considered negligible, but at higher loads the effect becomes potentially significant. Although the effect is perhaps not relevant when using the MFR test for comparative purpose it may be significant when using the data to extrapolate to higher shear rates.