The viscosity measurement on chocolate should be made at exactly 40 °C (104 °F). The viscosity changes by between 1 and 4% per °C, depending upon the recipe and the shear rate with the bigger effect being at the higher shear rates. The reason for choosing this temperature is that, at temperatures below about 35 °C (94 °F), crystallisation begins to occur, which makes the viscosity unstable. Above about 45 °C (125 °F) protein reactions within the milk can cause thickening to occur, especially at low shear rates. Dark chocolate is also affected, especially if there is no lecithin present (Chevalley, 1999).

When a sample is taken directly from a conche and provided the temperature has not fallen below 35 °C (94 °F), the measurement may be taken as soon as the sample temperature has reached 40 °C (104 °F). All other samples, whether they are liquid or solid chocolate, must be heated to remove all the fat crystals. Any moisture will thicken the chocolate, so this should be done in dry conditions and never over a water bath. If the chocolate is solid it should be divided into lumps rather than grated and melted in a sealed container in an incubator or oven. The liquid chocolate should be brought to 50 °C (122 °F) and then cooled again to 40 °C (104 °F). A 100 g (3.5 oz) sample of chocolate was found to take 75 min in an oven at 52 °C (126 °F) to reach this temperature (Aeschlimann and Beckett, 2000). Prolonged storage of milk chocolates should be avoided as thickening occurs with time. This is even more likely to happen for white chocolates, which should be stored at a lower temperature. Ideally milk chocolate should not be kept in the liquid state for more than 2-3 h before the measurement is taken. Where the measurement is not going to take place for an extended time, then it is preferable to store a sample in solid form and re-liquefy it, rather than keep it in the liquid state.

### 11.8.2 Checking the viscometer

If the bob has become scratched or bent it should be replaced. The calibration should also be checked at regular intervals with calibration oil, whose apparent viscosity in the mid-shear rate range is similar to that of the chocolates being tested.

The viscometer cup and bob should be washed and thoroughly dried between samples, as any residual moisture will affect the results.

As noted earlier, temperature has a big effect on the viscosity. The temperature of the fluid being used to heat the cup is often measured elsewhere in the circuit. The actual temperature should be determined by placing chocolate in the cup and measuring its temperature with a calibrated thermometer.

# 11.8.3 Preconditioning

When the sample is placed in the cup it is unlikely to be at the correct temperature throughout. Chocolate is a poor conductor of heat, so some mixing by stirring with the bob is required. If this is done too vigorously the chocolate will become thinner (chocolate is shear-thinning or thixotropic when it is stirred – see Section 11.2). The chocolate should therefore be stirred at a low shear rate until the shear stress reading becomes stable; and it has also been proposed that the

chocolate is being kept at rest for about 30 s before the actual measurement is taken. The method presently recommenced by the International Confectionary Association (ICA, 2000) uses a pre-shear period at 5 s<sup>-1</sup> to be applied until the viscosity reading is stable at  $\leq 2\%$ .

### 11.8.4 Shear rate range

Although in theory the lower the shear rate the better the determination of the yield value, viscosity readings of chocolate at low shear rates tend to be highly irreproducible due to the complex structure of this system. Inter-laboratory trials have shown that 5 s<sup>-1</sup> can produce repeatable measurements on most viscometers. The highest shear rate will depend upon the thickness of the chocolate and the scale range. It is not advisable to exceed 80% of the full scale reading. Viscometers should be able to provide readings up to about 50 s<sup>-1</sup> on a standard enrobing or moulding chocolate. This shear rate corresponds to the maximum value recommended in the ICA method (ICA, 2000).

## 11.8.5 Holding time at the maximum shear rate

Measurements of shear stress can then be taken over a range of shear rates. The increase in rate can be continuous or carried out as a series of steps. After a period at the maximum shear, the measurements are repeated as the shear rate is reduced again. At the maximum shear rate, the shear stress will slowly decrease due to the thixotropic nature of chocolate. It is impractical to wait for a long time in the middle of the measurement until this decrease has stopped. The final measurement therefore depends upon the time the viscometer remains at this shear rate. It is therefore necessary to standardise this when carrying out comparative measurements. Normally this should be of the order of 1 min (ICA, 2000).

### 11.8.6 Hysteresis

If the shear stress is plotted against the shear rate for both ascending and descending curves, it is often found that the descending values are lower at a particular shear rate for most of the curve. The difference between the two is known as hysteresis and is a measure of how well the chocolate has been processed. A poorly mixed or conched chocolate will have a big difference between the two curves. In this case the chocolate is being further processed by the shear within the viscometer itself.

### 11.8.7 Overall measurement time

The overall measurement time will also affect the final result (Aeschlimann and Beckett, 2000) and so needs to be standardised. The ICA standard method (ICA, 2000) recommends an overall time of 7 min, comprising of 3 min of ascending shear rate, 1 min at maximum shear rate, followed by 3 min of descending shear rate.