mobility, the relaxation time for molecules in the liquid stage is much shorter than for those in the solid state. It is therefore possible to determine the relative proportions of these two states in the sample.

The solid fat content is expressed as the percentage of the total fat that is measured as being solid. The value depends on which of the two standard methods has been used, and the method should be quoted along with the result. The direct method Cd 16b-93 (AOCS, 1993), ISO 8292 requires calibration with a control containing paraffin/acrylic glass, whereas the indirect method Cd 16-81 (AOCS, 1981) compares measured signals with those coming from a de-crystallised sample. According to Padar (2006), for cocoa butter samples the accuracy of indirect method is higher but more time-consuming than the direct method. The direct method results are available within seconds, but need a correction factor which depends on crystal type and temperature. This causes a systematic bias, which has to be taken into account.

It should be noted that even at room temperature a substantial proportion of the fat phase is still liquid in fat samples, as well as in a normal milk chocolates. The samples must be carefully prepared beforehand, with the fat taken through prescribed temperature cycles to ensure correct crystalisation.

When vegetable fats are used, it is important to know how the chocolate will behave at higher temperatures to be sure of good eating properties. This can be determined by carrying out NMR measurements at a series of temperatures.

24.3.3.2 Differential scanning calorimetry

Not only can differential scanning calorimetry (DSC) measure the amount of solid fat present in a sample, but it can also determine its crystalline state, which is vital to obtain good quality product (see Chapters 7 and 13). DSC enables the relative amounts of each of the crystalline states to be determined. This relies upon the fact that when a crystal melts, large amount of latent heat are taken in (the reverse to solidification). In DSC measuring cell the sample (*note*: the sample weight, approx. 10–20 mg, must be measured with accuracy of ±0.1 mg) is taken relatively quickly through a predetermined temperature range (in the order of 0.5 °C/min; 1 °F/min), and the energy change monitored. Peaks (see Figure 22.32) occur in the temperature ranges corresponding to the crystalline state present. The onset of a peak corresponds to the temperature at which a particular crystal form starts to melt, while the peak maximum corresponds to the temperature at which the rate of melting is greatest. This information can be used to indicate the crystal type. The peak height, position and resolution are strongly dependent upon the scanning speed (rate of temperature change); see Cebula *et al.* (1992).

A common technique used with chocolate is to plunge the sample into liquid nitrogen. Any liquid fat remaining then takes up the very unstable crystal form. When the sample is heated, peaks in the range 28–32 °C (85–90 °F) enable the proportion of the more stable forms to be determined.

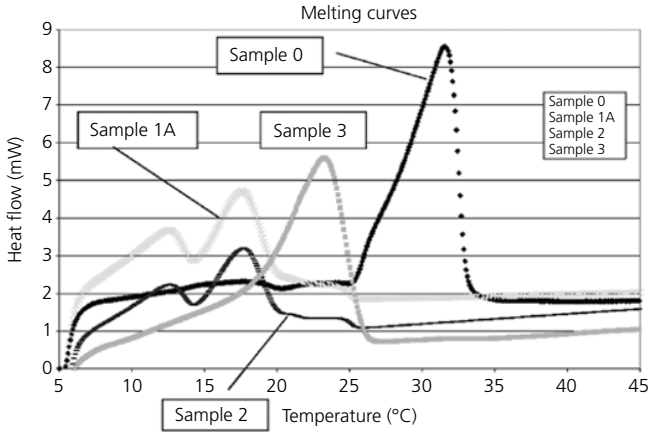

Figure 24.32 DCS plots of milk chocolate samples obtained using different cooling rates (sample 0: control; sample 1A: cooling rate 10 °C/min; sample 2: cooling rate 1 °C/min; sample 3: cooling rate 0.1 °C/min).

24.3.4 Particle size measurement

The original methods employed for determining particle size in milled material, chocolate masses and filling masses, such as the micrometer screw and wet sieving have been largely replaced in the laboratory and/or production areas by laser diffractometry (see also Section 24.2.12). For approximate, fast, on-line measurements however, micrometers are still commonly used, although laser diffractometry does provide more useful and accurate information. Measurements of the particle distribution are more useful than those of the largest particle, which is all that a micrometer can indicate. Traditionally, only the laborious wet sieving approach was available for determining particle size distributions and then only at the larger end of the size spectra. A laser diffractometer can produce the results in about 10 min and is largely independent of the operator. This has helped the manufacturer to optimise both processes and recipes. If fats/oil are used as dispersants, it is possible to run laser diffractometers in laboratory areas located close to the production lines.

Laser diffractometry: The optical components of a laser diffraction particle size instrument are shown in Figure 24.33. Prior to each measurement, the particles from the sample to be measured are dispersed in a liquid for example a special oil or, in certain cases, an organic solvent. This dispersion is aided by ultrasound treatment, which also breaks up agglomerates of particles. The sample is then recirculated through a cell located in the optical path of the laser. This movement prevents particles from sedimenting to the bottom of the cell. Although the dispersed particles are in motion, the laser diffraction pattern of the beam of coherent monochromatic light (laser illumination) is stationery, and contains information on the particle distribution. The laser beam is between 5 and 20 mm diameter, and the image of the diffraction pattern is formed in the focal plane of