melting enthalpy measurements by DSC, (iii) solid fat content (SFC) measurements by NMR or (iv) temperature-dependent flow curve measurements by thermorheometry (see also Chapter 24).

13.4.1 Tempermeters

Figure 13.2 illustrates schematic details of a *tempermeter*, which consists of a cooling block (Peltier element) to hold a constant temperature (isothermal cooling) of 0–10 °C (32–50 °F), the sample tube made from highly conducting aluminium or copper, a probe shroud and insulator and a thermistor/thermocouple probe, plus a recorder or computer to store and plot the temperature versus cooling time data. An optimum cooling temperature depends on the geometry, heat capacity and heat conductivity of sample and sample cup. Using 25 ml aluminium tubes/cups an optimum of 8 °C (46 °F) was found for a large number of chocolates and fillings. Different types of cooling curves are illustrated in Figure 13.3.

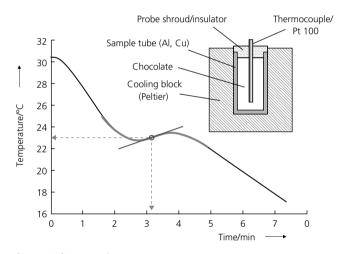


Figure 13.2 Schematic diagram of tempermeter.

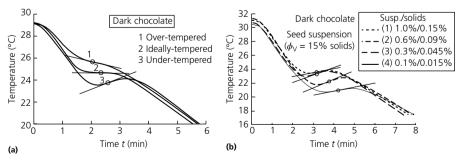


Figure 13.3 Selection of typical temper curves. (a) Conventionally tempered; (b) seed tempered.

In Figure 13.3a it can be seen that the chocolate sample cools with about a constant gradient for a period of time, then levels off producing different tangent slopes at the inflection point, depending on the degree of temper. This is due to crystal growth, with the latent heat being released and thus generating sufficient heat to retard cooling. The sample is in a semi-solid state at this time. Once most of the latent heat has been liberated, cooling continues until complete solidification occurs. Graph 1 in Figure 13.3a shows an over-tempered sample which cools slowly with a negative tangent slope in the inflection point. Graph 2 in Figure 13.3a shows a well tempered sample according to conventional tempering, which cools faster than the previous sample 1 and shows a horizontal slope of the inflection point tangent indicating a slightly delayed, but more pronounced increase in crystallisation heat release. Graph 3 represents an under-tempered chocolate sample, which first cools quickly due to the lack of crystals generating crystallisation heat, but followed by a further delayed and more intensive release of crystallisation heat leading to some reheating and a related positive slope of the inflection point tangent. Setting for sample 3 takes place over a longer period of time.

Beside fat composition the rate of crystallisation and setting are also influenced by a number of additional factors such as the type and amount of emulsifier used. This can be investigated using the tempermeter cooling curve.

The tempermeter cooling curve serves only as a guide, because it varies according to the type of tempering process, that is depending on residence time, temperature and mechanical treatment. Figure 13.3b demonstrates temper curves for β_{v_I} -seed tempered dark chocolates, which indicate that good temper curve shapes are process-specific. According to the conventional scale for the degree of temper, all seed tempered samples from lowest to highest crystal seed suspension fractions of 0.1–1.0%, (equivalent to about 0.02 to 0.2% of solid crystal mass in the chocolate) would be classified as under-tempered if originating from a conventional tempering process. However product quality was demonstrated to be optimum if $\geq 0.3\%$ wt of seeding crystal suspension (equals $\geq 0.06\%$ crystal mass, related to total masse) were added.

Figure 13.4 shows the inflection point tangent slope values ($m_{_{\rm IP}}$) as a function of the inflection point temperature ($T_{_{\rm IP}}$). This relationship can be interpreted as crystallisation intensity ($m_{_{\rm IP}}$) as a function of supercooling ($T_{_{\rm IP}}$) of the fat melt. The fewer crystal nuclei present in the tempered masse, the greater the supercooling before massive crystallisation will start and lead to an increased $m_{_{\rm IP}}$. Below a certain critical supercooling temperature, T^* , crystallisation kinetics is expected to slow down due to a dominating increase of viscosity, which greatly reduces the diffusion controlled mechanisms. From Figure 13.4 one such T^* value is shown in the range of about $22\,^{\circ}$ C ($72\,^{\circ}$ F). Furthermore it has to be expected that an increased fraction of less stable polymorph crystal nuclei forming during supercooling within the tempermeter will also accelerate the release of heat of crystallisation and thus increase $m_{_{\rm IP}}$. This effect is also seen for the conventionally crystallised samples. It is expected that the major reason why