The reaction takes place with pyridine in another solvent, which may be methanol and is such that one molecule of water reacts with one molecule of iodine. Automatic systems exist that can give a measurement within a few minutes. In these systems a pre-weighed amount of chocolate is dispersed in a liquid (e.g. formamide, chloroform and methanol) in a reaction vessel. This is tightly sealed to stop moist air entering the container with its stirrer and two platinum electrodes. Any iodine present will depolarise the cathode and allow a current to flow between the electrodes. A Karl Fischer reagent containing pyridine is then added slowly in controlled amounts. Once the iodine is used up the current will stop. At this point the amount of added reagent corresponds to the amount of water present in the original chocolate. The result is normally higher than for oven drying, because it includes the bound water, but it is highly reproducible and the analysis time is shorter.

24.3.2 Determination of fat content (Soxhlet)

In-line fat measurement systems, such as those based on near-infrared reflectance, are used in the industry especially for ingredients such as milk powder. Often they are affected by other parameters such as particle size or colour and they need recalibrating for each product. In order to carry out this calibration, or to perform routine laboratory fat analysis on chocolate, the traditional Soxhlet method is often used (Beckett 2000).

The principle of the method is to use a solvent to dissolve the fat out of chocolate, before evaporating the solvent. This leaves the fat, which can be weighed directly. This is normally carried out in a glass system. The chocolate sample is weighed and cut into small pieces before being wrapped in a filter paper, which is in turn placed in a permeable thimble. It is now ready for placing the central part of the extraction system between the flask and the water-cooled condenser. The flask contains the solvent, normally petroleum ether, which is heated on an electric mantle. The boiling ether evaporates and passes through the side arm into the condenser. Here it condenses and runs back into the thimble dissolving the fat. The fat-containing liquid passes through the filter paper and collects in the middle container. Eventually the quantity of liquid builds up until it reaches level A in the siphon (please see Figure 24.31), when it empties back into the flask. In this way the solid particles are retained within the filter paper and the fat is collected in the flask, where the temperature is high enough to evaporate the ether, but not the fat itself.

After about 12 h the top two sections are removed and replaced by the petrol distillation unit. This collects all the petroleum ether and does not let it run back. The fat remains at the bottom of the flask and can be weighed.

This technique measures both free and bound (contained within cocoa or milk particles) fat. When investigating viscosity problems it is sometimes necessary to determine the bound proportion (Chapter 11). An estimate of this can be obtained by first treating the chocolate with a mild solvent for a short period to extract

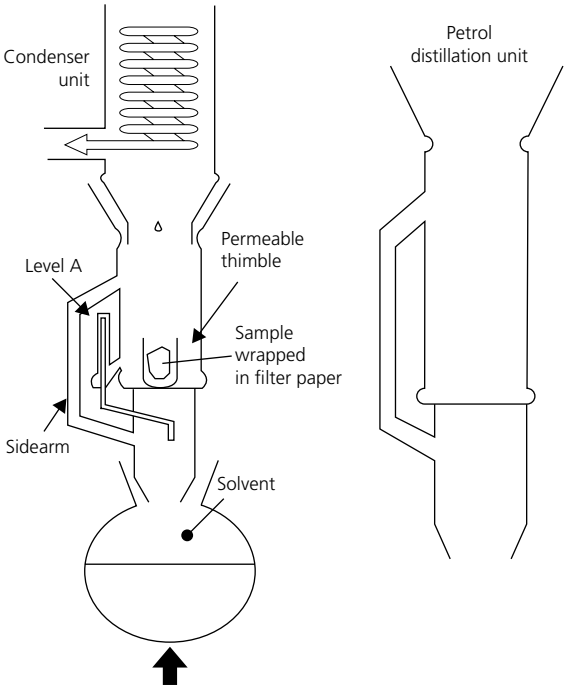

Figure 24.31 Schematic diagram of Soxhlet fat extraction apparatus. Reproduced from Becket (2000).

the easily soluble fat. The more tightly bound proportion is then determined by the standard procedure.

24.3.3 Solid fat content

24.3.3.1 Nuclear magnetic resonance

Two techniques are employed when using nuclear magnetic resonance (NMR) to measure the solid fat content (SFC) of cocoa butter and other fats: pulsed NMR and the continuous scanning method. Both are more reliable when carrying continuous measurements on pure fat systems. When chocolate is being analysed, account must be taken of the other solid components present, for example sugar, milk proteins and so on, which result in reduced sensitivity. When absolute values are required, very strict calibration techniques are needed, especially if a significant amount of water is present together with the fat.

Currently SFC measurements by pulsed NMR are often used. In pulsed NMR, the sample is held in a uniform magnetic field, which causes the protons in it to become polarised and magnetisation to occur. A short radio frequency pulse, at a pre-selected frequency, causes a rotation of this magnetisation vector around an axis normal to the original magnetic field. After the pulse, the longitudinal and transverse components of this rotating, magnetism return to equilibrium over different periods known as relaxation times. Owing to their greater