SFI or SFC? Why the difference?

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Over 30 years ago, while working at Unilever Research, I used nuclear magnetic resonance spectroscopy (NMR) to determine solid fat content (SFC). It was so obviously superior to dilimetry that I used it from then on for my research work. Towards the end of the 1970s the commercial side of Unilever had come to the same conclusion; NMR was rapidly introduced into all the factories and dilatometers were consigned to company museums. The rest of the European industry and then the rest of the world soon followed suit, with the exception of the United States.

Yet now at the start of the 21st century the fats and oils industry in the United States is still filling dilatometer tubes and manually reading capillary columns while everyone else is filling a simple cylindrical tube and popping it into an instrument which gives a direct solid fat reading within a few seconds. How did the United States fall so far behind and how can it catch up?

SFI and SFC by NMR (SFCN) differ. There are three reasons for this:

- the methods are based on different principles;
- the methods have different inherent assumptions and approximations;
- different tempering regimes are usually used.

The dilatation is the volume change that would result in complete melting at the specified temperature. Following the AOCS Official Method Cd 10-57, the dilatation is reported in mL/kg which gives results on a scale from 0 to about 100, denoted as SFI and superficially similar to 0 to 100% solid fat. The AOCS method does not specifically say that results are expressed as percent solid fat, but this is very commonly assumed. For example, the text Fats and Oils — Formulating and Processing for Applications by R.D. O’Brien, published in 2004, presents tables that say ‘Solid Fat Index (%) at:’ and figures which have ‘percent’ on the y axis.

In estimating SFC, Bailey assumed that fats have an approximate Total Melting Dilatation (TMD) of 100 mL/kg. In another paper, which was a foundation for the AOCS method, a TMD of 100mL/kg was also assumed and the intent of the “Solid Content Index” (sic) was to give “the approximate amount of solids expressed as percentage.” However, the TMD actually varies from about 65 to about 115 mL/kg depending on the molecular weight, the fatty acids and the polymorphic form of the fat. The result is that the SFI is usually significantly different from the true or absolute level of solid fat.

The NMR technique measures the number of hydrogen nuclei in the solid and liquid phases of the fat. Following a short electromagnetic pulse the magnetic signal in the detector declines or decays. The decay signal from the solid state is much faster than from the liquid state. The NMR method is closer to measuring the true or absolute SFC than the SFI method, but it does nevertheless involve assumptions and approximations.

It is assumed that the hydrogen density of a fat or triglyceride is identical to its mass. This is approximately true, but because it ignores the oxygen atoms in the triglycerides (C atoms can also be ignored as C and H are in approximately constant proportions), errors are introduced where there is a wide range of chain length in the fatty acids of the fat or where the solid and liquid phases contain very different chain lengths. This error can be corrected using the determined fatty acid composition, but for the vast majority of fats it is relatively small and can sensibly be ignored.

For technical reasons, it is not possible to measure the signal at zero time. It is therefore measured after a delay of about 10 μs. An extrapolation factor—the f factor—is then applied in the Direct Method to convert the signal to its value at zero time. The f factor varies with temperature, polymorph, crystal size and fatty acid chain length. The error due to its use may be several percentage values. However, for the standard margarine and shortening formulations prepared every day in a refinery, this absolute error is perhaps no more than 2%. It is certainly much less than the error in the SFI determination and, because it is constant and consistent for a given measurement method, it is ignored in practice.

Before the solid fat content can be determined, the fat must be exposed to a defined temperature history: first, to melt it completely to destroy all traces of solid fat; then to cool it to crystallize it substantially to completion; then to hold it at the measuring temperature to come to equilibrium at that temperature. An additional step is often introduced: holding the fat at a particular temperature which is not the measuring temperature. This step is referred to as a tempering step.

The SFI method includes a 15 minute tempering step at 26.7°C/80°F and this is an important difference between the European SFCN method and the SFI method used in the United States. Tempering affects the measured SFCs, even if no polymorphic change occurs. In Figure 1 a simple phase diagram of two triglycerides, A and B, is shown. The fat is assumed to be a mixture of A and B represented by the dashed vertical line. If the fat is held at temperature T1 then the solid:liquid ratio is given by \( ab:bc \). The liquid phase has a composition, \( a \), and the solid phase composition, \( c \). After tempering at \( T_1 \), the fat is transferred to 0°C quickly, thus effectively ‘freezing’ it in the solid and liquid phases established at \( T_1 \).
Thus the fat originally melted over the range \( T_4 \) to \( T_2 \), but after tempering at \( T_1 \) it melts over the range \( T_5 \) to \( T_3 \). If we were to raise the tempering temperature, it can be inferred that \( T_5 \) and \( T_3 \) would also rise. Thus we can conclude that the higher the temperature at which a fat is tempered the higher its final melting point. The SFC will be raised above the tempering temperature and lowered below it as shown in Figure 2.

Having explained the background of the SFI and SFC\(_N\) techniques and the clear advantages of NMR, why has the industry in the United States not abandoned SFI? One reason is that each company, whether producer or consumer, has built up databases of SFI values for the various fats of interest to them. There is no simple equation to convert SFC\(_N\) to SFI or vice versa.

In 1999, AOCS revised its NMR methods to bring them more into line with other international standards, by removing the tempering step at 26.7°C/80°F. However, commendable as it is to seek to harmonize methods, we have seen that this actually means that the NMR and SFI results are even more divergent than they need be.

In Figure 3 solid fat data for palm oil are shown. Applying the NMR method to samples tempered as for SFI (SFI\(_N\)) gives results much closer to SFI than when this tempering is omitted. Useful relationships between SFI\(_N\) and SFI can be developed, making conversion from one to the other relatively straightforward.

Thus a suitable way forward for the industry in the United States might be to change to an NMR method incorporating the SFI tempering step. There is no reason to abandon it; it serves the practical purpose of getting the sample closer to the state that a shortening is in after tempering in the plant warehouse. Indeed it could usefully be used by European industry for this purpose.

Contrary to the impression I may have given in the introduction, scientists in the United States were actively involved in studying the NMR method in the 1970s. P&G’s Bryan Madison and R.C. Hill published conclusions in 1978 similar to mine:

“The tempering period at 26.7°C is perhaps the principal difference from present NMR methods. This step was introduced for several reasons. First, the official AOCS dilatometry method includes the same tempering temperature. Second, a major field of interest is the phase behavior of shortenings which are usually tempered for some hours at a temperature near 26.7°C after being packed. Third, it was demonstrated experimentally that sample equilibrium was reached more rapidly and reproducibly when the 26.7°C tempering step was included in the method. Fourth, in order to compare solid fat results between pulsed NMR and dilatometry or between labs, it is essential that test samples have the same temperature history. This is a characteristic of the sample not of the measuring technique and cannot be avoided or overlooked if precise, high quality results are to be obtained.” (JAOCS 55:328-331, 1978).

Finally, it is hoped that a large oils and fats producer in the United States, or a group of producers, agree to make the change to NMR and assist their customers in understanding the benefits, just as happened in Europe all those years ago.

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**Background reading**
