Application Note 13

Determination of Solid Fat Content in Edible Oils and Fats

Application

Solid Fat Content (SFC) is an essential measurement for characterising edible oils/fats used in the bakery, confectionery and margarine industries for which a rapid and convenient method is required.

The melting profile (Solid Fat Content vs. Temperature) determines the specific application of the edible oil/fat thus is an important quality control parameter for both the suppliers and end users, as well as for the purpose of product development. Increasing legislation associated with reducing or eliminating trans fatty acids has caused renewed interest in the method since many conventional products will need to be reformulated.

Advantages of NMR

The traditional method for measuring SFC has been dilatometry but this is regarded as slow, inaccurate and cumbersome. However, for a number of years Nuclear Magnetic Resonance (NMR) has been the method of choice for the determination of SFC.

- NMR complies with all internationally-accepted official Direct Methods for SFC measurement: ISO 8292-1, AOCS Cd 16b-93; and IUPAC 2.150
- NMR measurement time is short (typically 6 seconds) although sample conditioning is required
- The NMR technique is non-destructive, so repeatability or other measurements can be made on the same sample
- NMR is very stable over the long-term and seldom requires re-calibration.

The Direct Method

The Direct Method works by measuring both the solid and liquid signals from the NMR Free Induction Decay (FID) of the sample. This is possible because signals from solids decay much faster than signals from liquids. It is therefore possible, in principle, to take measurements at two points on the FID (Figure 1), at point S_0, corresponding to the total solid plus liquid signal, and another at point S_{70} which corresponds to the liquid only signal, after the solid signal has died away. Simple arithmetic yields the percentage of Solid Fat which is given by $(S_0 - S_{70})/S_0 \times 100$. In practice, it is not possible to take a measurement at point S_0, immediately after the 90° radio frequency (RF) pulse. The short, high power RF pulse causes the sample probe to 'ring' for a few microseconds during which time measurements cannot be made. Instead, the first measurement is taken immediately after this ringing period (or dead time), at point S_{11}. Given that S_{11} does not represent the total signal from the solid and liquid, a correction needs to be applied. This correction assumes a fixed ratio (known as the f-factor) between S_0 and S_{11} due to loss of the solid signal during the first 11 µs of the decay.

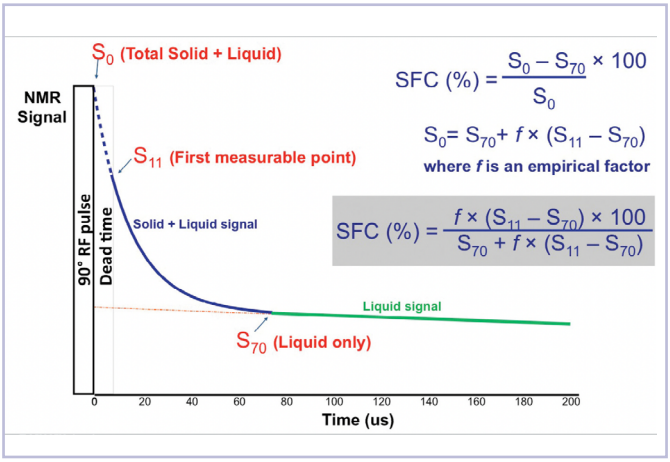

Figure 1: Schematic diagram showing how Solid Fat Content is derived from NMR measurements by the Direct Method.

Official methods also exist for the Indirect Method which measures the liquid signal only (an absolute measurement) and must therefore be referenced against the same sample when fully melted. As a consequence, the Indirect Method requires an additional temperature step as well as a temperature compensation sample, and care must be taken to keep all sample within the measurement volume of the probe.

In contrast, the Direct Method is a ratio measurement which is not sensitive to sample loading and does not require a compensation sample or an additional temperature step. The Direct Method is the preferred NMR method for determining SFC.

Tempering

Before measuring the SFC, edible oil and fat samples must be tempered in order to stabilise their crystal structures. The tempering process involves placing the samples in temperature controlled blocks for set periods of time. After melting the samples at 100°C, they are pre conditioned at 60°C and then 0°C to eliminate their thermal history. Stabilizing fats such as cocoa butter require a different temperature regime. Thereafter the samples are stabilised at a variety of temperatures of interest prior to NMR analysis. This may be carried out on the same sample in 'Series' or multiple aliquots of a sample in 'Parallel'. The Parallel method is usually preferred since it is faster, however it does require a separate conditioning block for each temperature, in addition to those for the preconditioning steps.

The number and value of the temperature steps, and the time that the samples are at each temperature are set by the official method being followed, such as the International Organisation for Standardization (ISO 8292-1), the American Oil Chemists' Society (AOCS Cd 16b-93), or the International Union of Pure and Applied Chemistry (IUPAC 2.150). Further details concerning the official methods are available from the respective organisations. Alternatively contact Oxford Instruments for advice.

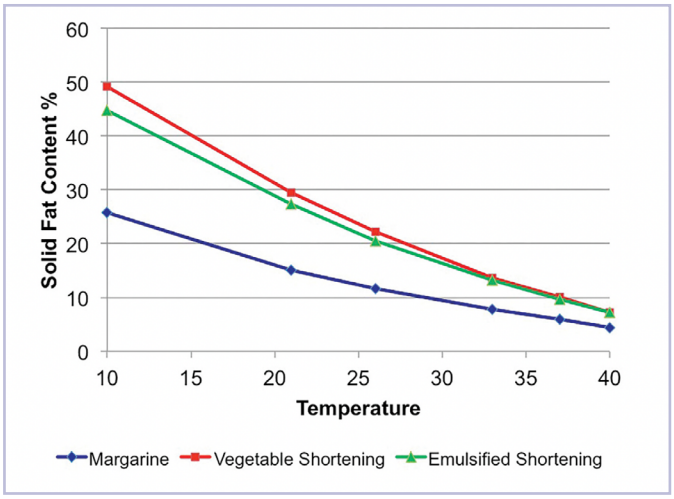

Figure 2: Graph showing melting profiles (Solid Fat Content vs. Temperature) of AOCS proficiency samples.

Calibration

An automatic software routine determines the f-factor using samples of known SFC. Artificial standards are supplied by the NMR manufacturer and are designed to represent approximately 0%, 30% and 70% SFC. Exact values are assigned and certified by the manufacturer.

Results

Figure 2 shows the results from three AOCS proficiency samples used to validate the instrument. The two most important factors in obtaining consistent results between laboratories, both of which are independent of the instrument, are:

(1) determination of the f-factor

(2) using the same tempering protocol as well as following the method in every detail.

Complete Package

Oxford Instruments offers a package especially tailored to the measurement of Solid Fat Content:

- Oxford Instruments **MQC+**23 NMR Analyser
  - 0.55 Tesla (23 MHz) high homogeneity magnet
  - Probe for 10 mm diameter sample tubes (2 ml sample volume)
  - Integrated system controller (no external PC required)
  - Integrated flat-screen display
- Dedicated software suite for Solid Fat Content measurements, providing simple routines for calibration, measurement and reporting of results
- A set of 3 artificial standards at 0, 30 and 70% Solid Fat Content for determining the f-factor and thereafter routine QC checks
- Test/tuning sample
- 10mm glass tubes
- User manuals

In addition to the above, the following are also required:

- Water baths or dry-blocks for preconditioning (at 60˚C and 0˚C)
- Water baths or dry-blocks for stabilising the samples at each SFC temperature. Typically 5-6 temperatures between 10˚C and 50˚C are chosen to clearly define the melting point profile.

The **MQC+** SFC analyser offers multiple advantages over other instruments on the market:

- Compliance with all official SFC Direct Methods
- Solid Fat Direct software guides the user through the measurement process, keeping a record of sample identification and measurement temperatures
- Instructions can be given in English, French, German, Spanish, Chinese and Japanese
- Solid Fat ProGen software gives the user flexibility to define key NMR parameters; measurement temperatures and method (Series, Parallel or Individual measurements)
- Solid Fat Reporter software allows data to be examined, plotted and exported to other programs; it also allows melting profile curves to be generated
- A set of standards for instrument calibration (f-factor determination)
- Small benchtop footprint
- Low maintenance costs

Summary

- Complies with official ISO, AOCS and IUPAC standard methods
- Simple calibration using stable standards
- Simple, intuitive visual software suitable for unskilled personnel

If you have any questions about this application note, please contact our experts:
magres@oxinst.com

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