

The measurement of Solid Fat Content (SFC) in edible oils and fats is an essential measurement in the bakery, confectionery and margarine industries. It is important that raw materials are characterised according to their melting profile, and rapid and convenient methods are required for doing this.

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

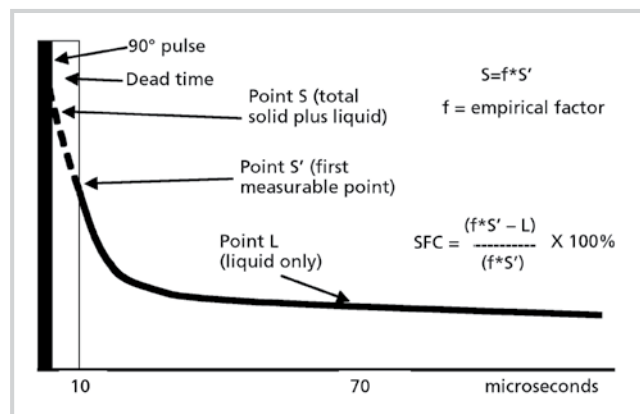
Advantages of benchtop NMR

- Complies with all internationally-accepted official direct methods for SFC measurement: AOCS Cd 16b-93; ISO 8292-1; and IUPAC 2.150
- NMR measurement time is short (typically 6 seconds) but sample tempering is required (see below)
- The NMR technique is non-destructive, so repeatability measurements can be made conveniently
- Standards are available for calculation of f-factor (see below) and thereafter for routine quality control checks
- NMR is very stable over the long-term, therefore requires little re-calibration
- Application software available in a range of languages

Method

There are two NMR methods for determination of SFC, known as the Indirect and Direct Methods.

The Indirect Method relies on measuring only the liquid part of the sample, and the instrument is calibrated by referencing to the signal from the sample when fully melted. It is necessary to weigh the samples and a correction needs to be made since the



NMR measurements are temperature sensitive. Care is required to ensure that the entire sample is within the measurement volume of the tube as any sample smeared on the walls of the tube contributes to the sample weight, but not to the signal, leading to erroneous results. The Indirect Method therefore has a number of extra complexities.

In contrast, the Direct Method takes measurements of both the solid and liquid components in the samples, and calculates the ratio between them. Measurement of NMR signals from solids can generally only be carried out on pulsed NMR instruments operating at 20MHz or above, therefore the Direct Method is limited to such instruments.

As the Direct Method is a ratio method, it is not necessary to weigh the samples, and it is not sensitive to sample temperature or loading, all reasons why the Direct Method is the most common NMR method of SFC determination.

The Direct Method

The Direct Method works by measuring both the solid and liquid signals from the Free Induction Decay (FID) of the sample.

This is possible because the 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 (see Figure 1 opposite), at one point S, corresponding to the total solid plus liquid signal, and another at point L which corresponds to the liquid only signal, after the solid signal has died away. Simple arithmetic then yields the ratio between the solid and liquid signals, which is the SFC ($SFC = (S-L) \cdot 100/S$).

In practice, it is not possible to take a measurement at point S, immediately after the 90° pulse, because the 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 a point S'. Given S' does not represent the total signal from the solid and liquid, a correction needs to be applied. It assumes a fixed ratio (known as the f-factor) between the points S' and S, thus S is determined by measuring S' and multiplying by f before completing the rest of the calculation.

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



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The correction is determined by measuring samples of known SFC then calculating the f-factor required to give the correct results. Instruments are supplied with a set of artificial standards designed to represent approximately 0, 30 and 70% SFC (exact values are assigned by the manufacturer) and the operator uses them in an automatic routine to set the f-factor for the instrument.

Tempering

Before measuring the SFC, edible oil and fat samples need to be tempered to stabilise their crystal structures. The tempering process involves placing the samples in temperature controlled blocks for set periods of time. The number and value of the temperature steps, and the time at each temperature are set by the official method being followed, either International Union of Pure and Applied Chemistry (IUPAC) or American Oil Chemists' Society (AOCS).

Samples must also be measured at a range of temperatures, also determined by the official method. Further details concerning the official methods are available from Oxford Instruments.

Official Methods

The **MQC-23** complies with all official direct methods for SFC measurements (AOCS Cd 16b-93, ISO 8292-1, and IUPAC 2.150).

Recommended Instrument Configuration

The **MQC-23** with 0.55 Tesla magnet (fitted with a 10mm diameter (2ml) probe) is ideal for this application. The 'Solid Fat Content' package comprises:

- The **MQC-23** which can be controlled using its own built-in computer using Microsoft® Windows® or via a stand-alone PC
- Dedicated software suite for Solid Fat Content measurements, providing simple routines for calibration, measurement and results reporting and management
- A set of 3 standards at 0, 30 and 70% Solid Fat Content for determining the f-factor and thereafter routine quality control checks
- Sample tubes
- Installation manual
- Software manual

In addition to this package you will also require:

- Water baths and aluminium blocks for tempering and stabilising samples at each temperature that SFC will be measured (N.B. at least one will need to be refrigerated)

The instrument offers multiple advantages over other instruments on the market:

- Compliance with all official SFC direct methods
- Applications software available in several languages including English, Chinese, German, French, Japanese and Spanish
- Small benchtop footprint
- Set of standards for instrument calibration
- Low maintenance
- Specific "SFC" applications software
- The sample tubes are recyclable, lowering consumable costs
- Minimal sample preparation

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