

Determination of Solid Fat Content (SFC) in Oils and Fats by SPIN Track NMR Analyzer (revised in 2017)

1. Introduction

Quality of food products containing fats and oils strongly depends on solid fat content (SFC) that characterizes the crystallization behavior at different temperatures. SFC analysis is an essential measurement in the bakery, confectionery and fat industries and based on melting curve determination. The traditional methods for SFC determination are slow, irreproducible and require additional chemicals (e. g. dilatometry). Last years NMR has been established as the method for determination of SFC. Measurements of SFC by NMR can be performed quickly and accurately.

2. Method

There exist two approaches to measure SFC by NMR: Indirect and Direct. Indirect method based on liquid part measurement that requires weighting of samples and keeping constant volume of it. So, it can be characterized as complicated. Direct method is based on direct calculation of the ratio between solid and liquid parts of the sample, but requires high precision pulse NMR devices with operating frequency of about 20MHz or above. On the other hand, there is nothing to be done more than loading of the sample to sensor.

So, the basic method bases on direct measurement of solid and liquid parts ratio of the sample. For this two measuring points on each Free Induction Decay (FID) are used, because of signal from solids decays much faster than signals from liquids. FID is the signal after the power radio-frequency excitation of the sample; this is the signal caused by relaxation of hydrogen proton magnetic spins back to equilibrium state after disturbing after disturbing. FID amplitude at the point S corresponding to total solids plus liquids and at the point L to liquids only. The specific ratio can be found using equation shown on Fig.1. This ratio is considered as the SFC value.

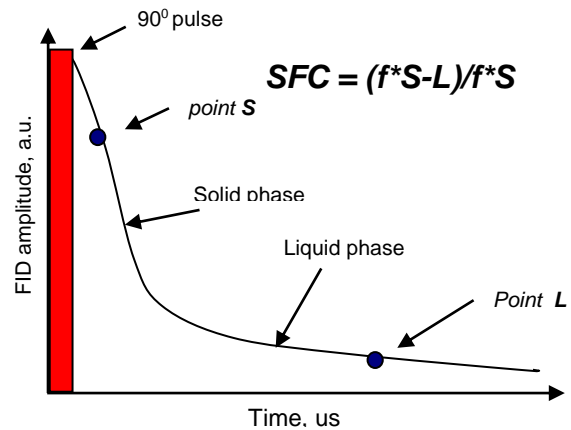


Fig. 1. SFC calculation based on FID

The measurements are very exact if S point corresponds to maximal FID amplitude. It is not possible to acquire first point S immediately after power pulse due to so-called “ring time” or “dead time” – few microseconds while resonance processes damp in disturbed sensor circuit. The duration of dead time has to be as short as possible to make measurements more exact. F-factor (f) allows to predict FID amplitude value (S) immediately after RF pulse and can be determined by measuring samples with known SFC content.

3. NMR equipment



Fig. 2. NMR analyzer **Spin Track**

NMR analyzer **Spin Track** (Fig. 2) from Resonance System Ltd. is ideal for SFC measurements because of short dead time (<10 microseconds), high acquisition rate and high signal to noise ratio which make measurements very reproducible and accurate.

Small weight and mobility allow its usage in production routine measurements and in advanced laboratories for complicated analysis. **Spin Track** fulfills the requirements of international standards like **AOCS Cd 16b-93**, **AOCS Cd 16-81 revised in 2000**, **ISO 8292**, **IUPAC 2.150**. **Spin Track** is built utilizing up-to-date electronics, very simple to use because all measurements are made automatically pressing one button.

4. Measurements and calibration

The volume of the sample is 1-3 ml. SFC measurements cycle for 5 point melting curve using special container for samples and thermostat:

- melting at 80..100°C and holding for 15 min
- maintaining sample temperature at 60°C for 5-15 min
- maintaining sample temperature at 0°C for 60 min
- holding sample at required temperature for measurement (typically 10°C / 15°C / 20°C / 25°C / 30°C / 35°C) for 30 -35 min

Every sample is placed into sensor for 6 s and measurement process is started. Full cycle requires ≈110 min. It is possible to use only one sample for whole cycle with consequently holding at all temperatures but this method requires approximately ≈230 min.

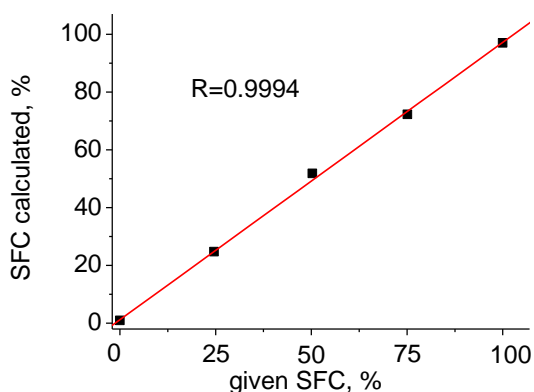


Fig. 3. Typical calibration curve of **Spin Track** analyzer

The procedure of calibration can be performed using the set of original calibration samples developed by Resonance Systems Ltd. This set contains samples with 25, 50 and 75% of solid phase. Daily calibration of the analyzer is very important to obtain correct results.

Calibration curve of the **Spin Track** analyzer is given above (Fig. 3). Sample with 0% of SFC was distilled water, 100% of Solid content – PMMA.

Analyzer that calibrated on original calibration samples set from Resonance Systems Ltd. has excellent reproducibility. Results of test performed by the center of certification and metrology are presented in table 1.

Table 1

Sfc, %	1 st day	2 nd day	7 th day
23.6	23.3	24.0	23.8
35.4	35.4	35.5	35.2
47.6	47.5	47.5	47.3
56.3	56.5	56.5	56.4
76.8	76.5	76.9	76.6

Every sample was measured with **Spin Track** and measurements were repeated after 1, 2 and 7 days. Samples were held at stable temperature and moisture conditions during this period.

Calibration samples should be stored in vertical position at 20-23°C and 25-30% of air moisture being kept out from direct light and overheating.

5. Software

NMR spectrometer is controlled by original software **Relax** developed by Resonance Systems Ltd.

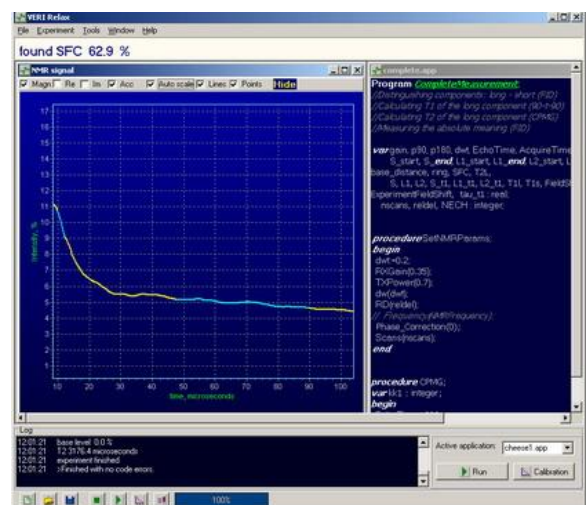


Fig. 4. **Relax** software screenshot

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