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

GENERAL NOTES

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, based on melting curve determination, is an essential measurement in the bakery, confectionery and fat industries. 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.

BASICS OF THE METHOD

There exist two approaches to measure SFC by NMR: Indirect and Direct. Indirect method is based on comparison of the sample with triolein sample and requires the measurement of 2 similar samples to improve the accuracy. So, this method is relatively 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 20 MHz or above. On the other hand, there is nothing to be done more than loading of the sample in to the sensor.

So, the basic method is involving the direct measurement of solid and liquid parts ratio of a sample. Generally, fat samples are characterized by a two-trend of the Free Induction Decay (FID): since the signal from solids decays much faster than signals from liquids, they show a rapid decrease at early time (solid phase) and a slow decrease at long time (liquid phase). For this purpose, two measured points on the FID are selected, one located at early times (point S) and one located at long times (point L).

FID is the signal after the power radio-frequency (RF) excitation of the sample; this is the signal caused by relaxation of hydrogen proton magnetic spins back to equilibrium state after the RF perturbation. FID amplitude at the point S corresponds to solids and liquids amount while the point L corresponds to liquid amount only. The specific ratio can be found using equation shown on Fig.1, where the f factor is evaluated by a previous calibration. This ratio is considered as the SFC value.

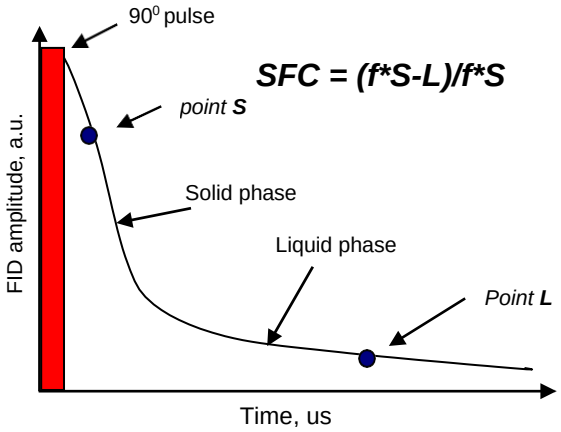

Fig. 1. SFC calculation based on FID

The measurements are very exact if S point corresponds to maximal FID amplitude (ideally at t=0.0 us), but It is not possible to acquire the 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 predicting the real FID magnitude right after RF pulse and can be determined by measuring samples with known SFC content.

INSTRUMENTATION

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

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

Spin Track fulfills all requirements of international standards like **AOCS Cd 16b-93, AOCS Cd 16-81 revised in 2000, ISO 8292, IUPAC 2.150.**

The **Solid Fat Content Analyzer** package comprises:

- Spin Track NMR Analyzer with thermally stabilized at 40 °C magnet system;
- PC with pre-installed Microsoft OS © Windows 7, 8, 10 or 11* and Relax 8 software;
- Thermostats "ST-80"**;
- Calibration samples (values within the range 0…100%);
- Test tubes with outer diameter 10;
- Plastic caps for test tubes;
- Installation Manual;
- Method Sheet;
- Autosampler (optionally).

* Determined by the PC manufacturer
** Depends on desired number of temperature points in melting curve

CALIBRATION AND MEASUREMENT

The workflow consists of the following steps (simplified procedure, see ISO 8292 recommendation for more detailed description):

1. Melting at 70-80 °C;
2. Filling sample tube (sample height has to be from 3 to 5 cm);
3. Tempering at 60 °C within 10 minutes;
4. Crystallizing at 0 °C within 60 minutes;
5. Tempering at desired temperature within 30 minutes;
6. Inserting sample tube in a detector manually or by the autosampler;
7. Running a measurement which is taking few seconds;
8. All measurement results are recorded in a spreadsheet, saved and can be accessed both on a computer and on-line.

The procedure of calibration is performed using the set of original calibration samples developed by Resonance System. This set contains samples with 0,~30,~70 and 100 % of solid phase. Daily calibration of the analyzer is very important to obtain correct results.

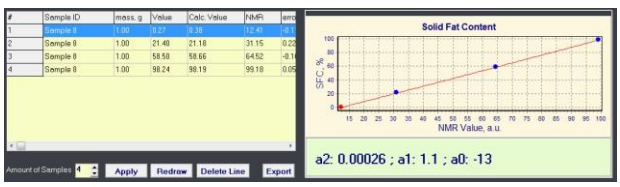

Fig. 3. Typical calibration curve of **Spin Track** analyzer

Calibration curve of the **Spin Track** analyzer is given above (Fig. 3). Samples of melting curves are shown on Fig. 4.

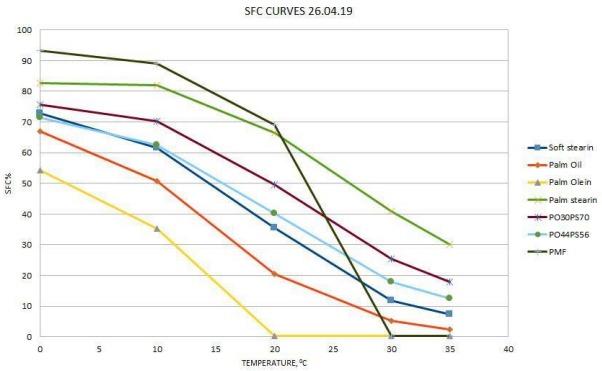

Fig. 4. Samples of melting curves

CONTACTS

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www.nmr-design.com

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