

# New Method for Determination of Solid Fat Content in Cosmetic Products by SFC NMR

Jampala Balaji<sup>1</sup>, Amit Kumar .T<sup>2</sup>, Dr. Krishan Manrai<sup>3</sup>, Sarath Chandra Prakash N.K<sup>4</sup>

<sup>1,3,4</sup>R&D Department, the Himalaya Drug Company, Bangalore, India-562162

<sup>2</sup> Oxford Application Specialist Laboratory, Mumbai, Maharashtra, India

**Abstract:** A new analytical NMR method for the determination of Solid Fat Content (SFC) in cosmetic formulations e.g. Kajal and lip balms, which are based on natural waxes and vegetable oils composition. By using NMR technique solid fat content in the formulations quantified easily. The traditional method for measuring SFC has been dilatometry but this is regarded as slow, inaccurate and cumbersome. However, low resolution Nuclear Magnetic Resonance (NMR) has been the method of choice for the determination of SFC. The method based on direct measurement of the NMR signal from the solid and liquid fat. The method performed in the temperature between 0 °C to 60 °C, the results are good reproducible and accurate. The method is more consistent and reduced the cost of analysis.

**Keywords:** SFC, NMR, Cosmetic products, Lip-Balms, Kajal

## 1. Introduction

The fats which are solid at room temperature are called solid fats, like beef fat, butter. Most solid fats are high in saturated fats or trans fats contain cholesterol. These fats tend to raise bad cholesterol in blood and if these fats containing food products are taken, it gives heart disease. Solid Fat Content is one of the major characteristics of fat that it will change consistency with higher and lower temperatures, from more or less solid to liquid. The SFC defines the percentage of the solid parts of fats at certain temperatures. The determination of SFC in food products and cosmetics products is most important, because it affects the quality of the product [1][2].

Lip-Balms are wax type substances, which are used for chapped or dry lips. Lip-balms are containing bees wax or carnauba wax or paraffin wax [3]. These all waxes are containing fatty acids with solid fat components. These are especially during the heating phases, have a major impact on the final product. Signs of instability such as aeration, deformation and cracking may result from inadequate preparation [4].

Kajal is an ancient eye cosmetic, traditionally made by grinding galena and other ingredients. It used as eyeliner to contour and/ or darken the eyelids and as mascara for the eyelashes. These are also contains fatty acid components. SFC in these also most important for maintaining stable product.

Pulsed NMR technique is well established for the determination of SFC [5]. The SFC value is determined by detecting the NMR signal from both liquid and solid components in the fat sample, or by detecting the change in the liquid signal as it is displaced by solid [6]. In the "pulse" method a short intense burst of radio frequency (RF) energy is applied to the sample in the static magnetic field to cause excitation of the Hydrogen in the fat. The SFC value is determined in the NMR by taking two measurement points on the FID (Free Induction Decay). FID amplitude at point S (corresponding to total solids plus liquids), and at point L

(corresponding liquids only). Based on the equation, which is shown in the figure-1, calculated the SFC. In the equation "f" is empirical factor.

The determination of solid fat content by pulsed NMR is having two types of methods [7], the direct method and indirect method. The indirect method measures only the liquid signal and compares it to the signal from a fully melted sample and calculates the SFC. In the direct method by measuring both the signals of solid and liquid components then calculates the SFC.

In the indirect method the sample is heated to a stable state at a specific temperature and then stabilizes at measurement temperature, otherwise specified, measurement temperatures can be 0 °C, 5 °C, 10 °C, 15 °C, 20 °C, 25 °C, 30 °C, 40 °C, 50 °C and 60 °C. After electromagnetic equilibration in the static magnetic field of the NMR spectrometer and application of a 90° radiofrequency pulse, the magnetization decay signal from the protons in the liquid phase is measured and the solid fat calculate by compare with reference standard consisting of liquid fat. Based on the liquid fat measuring, the SFC calculated by the direct method.

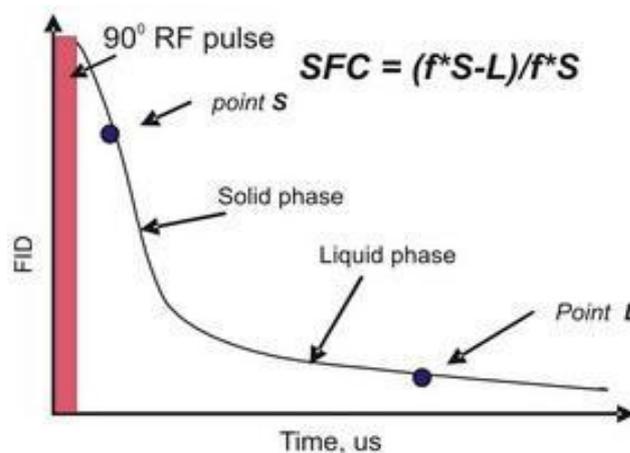


Figure 1: SFC calculation by NMR

The aim of the method was to measure the solid fat content in cosmetic products like lip-balms and kajal with simple and good reproducible results. In this method performed based on IUPAC method in the temperature levels 0 °C, 5 °C, 10 °C, 15 °C, 20 °C, 25 °C, 30 °C, 40 °C, 50 °C and 60 °C. The results are good reproducible.

## 2. Experimental

The sample, which was required to determine solid fat content at different temperatures, the sample was heated to 100°C causing the sample to separate into its different components. The top melted wax layer was pipetted into two NMR tubes (up to a height of 4-5cm) as shown in Figure 2. Each sample was then heated in the oven at 100°C further to ensure that their temperature history were similar (and to remove moisture).



Figure 2: Samples prepared in the NMR tubes

The samples were then placed in a dry block at 0°C for at least an hour followed by conditioning at 5°C, 10°C, 15°C, 20°C, 25°C, 30°C, 40°C, 50°C and then finally at 60°C for at least 30 minutes prior to each measurement of solid fat content. Please note that the each sample was measured at each temperature in series, which should give equivalent results to the parallel method.

Measurements were made using the Oxford Instruments MQC-23 low-field NMR analyser with 0.55 Tesla magnet and 10mm diameter probe operating at a hydrogen resonance frequency of 23.4 MHz's. The ordering was controlled by, and the NMR measurements made, using Oxford Instruments' widely acclaimed SF Direct software.

## 3. Results and Discussion

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<sub>0</sub>', corresponding to the total solid plus liquid signal, and another at point S<sub>70</sub> which corresponds to the liquid only signal, after the solid signal has died away. Therefore in theory, 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<sub>0</sub>, 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<sub>11</sub>. Given that S<sub>11</sub> 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 S<sub>0</sub> and S<sub>11</sub> due to loss of the solid signal during the first 11 μs of the decay. The melting profile of the sample was showed in figure-3 for analysis.

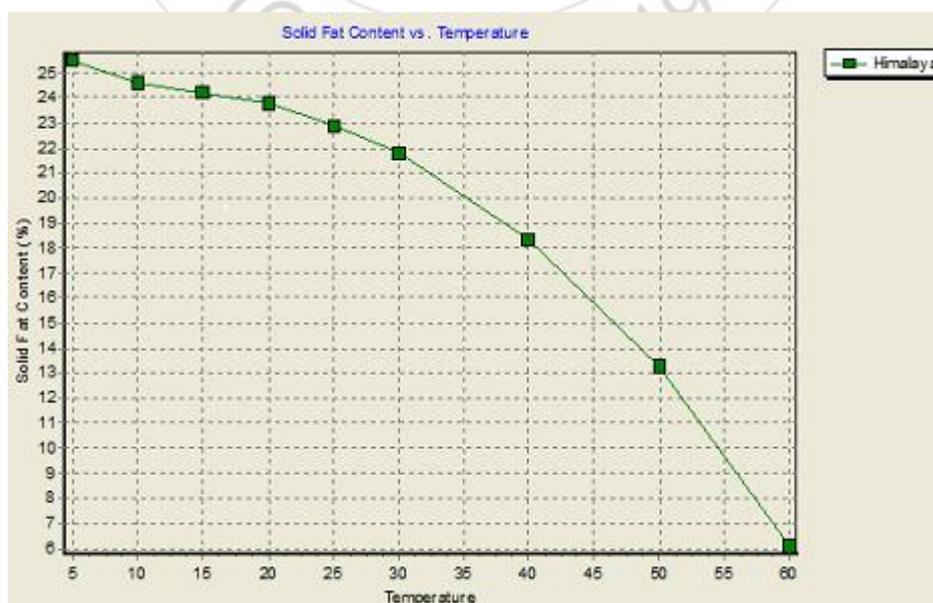


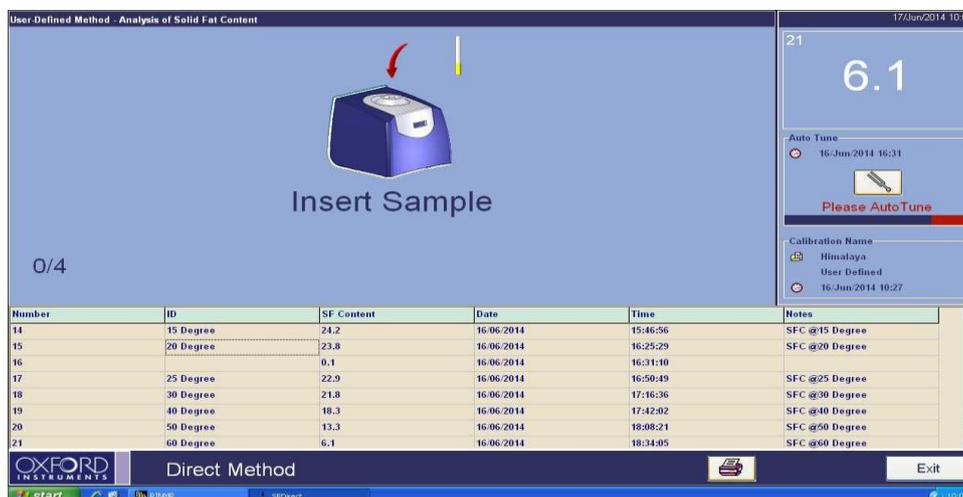
Figure 3: Solid Fat Content Melting profile of the wax from the sample

The results of the solid fat content for the sample with sub samples and average in each different temperature levels are showed in Table-1.

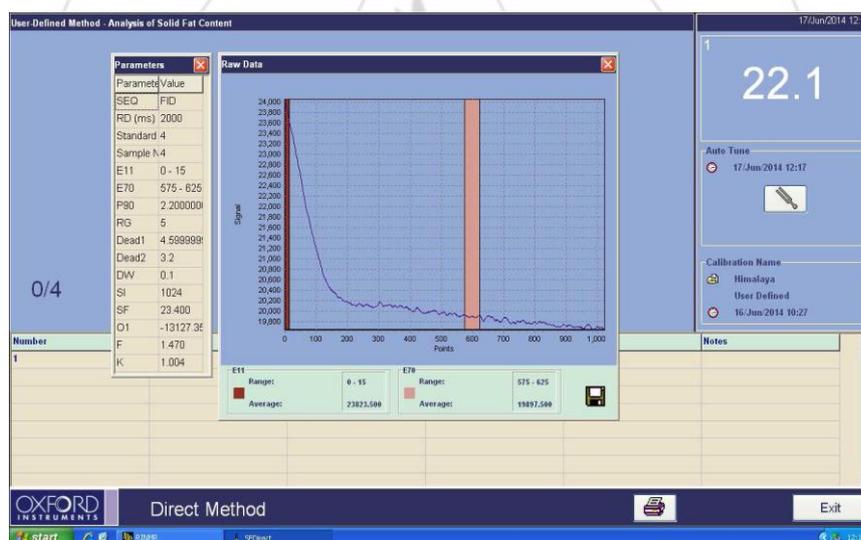
**Table1: SFC of samples at various temperatures**

Sl.No	Temperature (°C)	SFC (%) of Subsample-1	SFC (%) of Subsample-2	Average SFC (%)
1	5	25.50	26.10	25.80

2	10	24.60	24.90	24.75
3	15	24.00	24.20	24.10
4	20	23.80	23.60	23.70
5	25	22.90	22.70	22.80
6	30	21.80	21.50	21.65
7	40	18.30	17.90	18.10
8	50	13.00	13.30	13.15
9	60	6.10	4.30	5.20



**Figure 4: Software for SFC analysis by NMR.**



**Figure 5: Raw data of sample for SFC analysis by NMR**

#### 4. Conclusion

The results show that the MQC can be used to provide reproducible measurements of solid fat content. By using this method we directly calculate the solid fat content and the results are reproducible and the analysis time will be very less. As per best of my knowledge, this method is very good suitable for analysis of solid fat content in cosmetic products.

#### References

[1] Karlshamns Oils and Fat Academy, "Cocoa butter alternatives", Karlshamns Oils and Fats AB, Karlshamn, Sweden, 1991.

- [2] Aleksandra Torbica · Olga Jovanovic · Biljana Pajin, "The advantages of solid fat content determination in cocoa butter and cocoa butter equivalents by the Karlshamns method"
- [3] Alessandra Ribeiro Fernandes, Michelli Ferrera Dario, Claudinéia Aparecida Sales de Oliveira Pinto, Telma Mary Kaneko, André Rolim Baby, Maria Valéria Robles Velasco, "Stability evaluation of organic Lip Balm", Brazilian Journal of Pharmaceutical Sciences, vol. 49, n. 2, 2013.
- [4] Denavarre, M.G, "The chemistry and manufacture of cosmetics", 2.ed. Orlando, v.3, p.699, 1975.
- [5] TAN, Y.A. "26th Palm Oil Familiarisation Programme", Kuala Lumpur, Quality monitoring (chemical and instrumental technique), 2006.

- [6] Nazaruddin Ramli, "Determination of Solid Fat Content (SFC) in Oils and Fats by pulsed- NMR Analyzer", Advanced Chemical Analysis of Food Laboratory, STKM 2622, 2013.
- [7] Ethiopian Standard, "Animal and vegetable fats and oils- Determination of solid fat content by pulsed NMR", ES ISO 8292-2, 2012.

