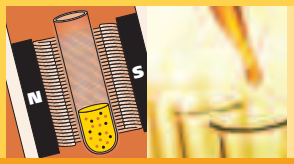
Application note

Measurement of Oil Content in Dried Snack Foods



NMR

Measuring the oil content of dried snack foods is essential for quality control to ensure that the products meet their nutritional values. The amount of oil used is also important in terms of the significant cost of the raw material and the effect it may have on the texture and perceived quality of the product.

Method

Solvent extraction techniques are commonly used for determination of fat content. However, they tend to be slow, cumbersome, inaccurate and require skilled personnel. In addition, many of the often hazardous chemicals used are becoming increasingly unacceptable according to international environmental standards. Despite these issues solvent extraction continues to be used as a reference measurement for quality control.

Instrumental methods are often referred to as secondary techniques since they are usually set up to match the results produced by solvent extraction. To provide a result equivalent to the traditional extraction techniques, secondary techniques require a correlation or a correction against the reference technique used. Although they are fast and easy to maintain, many secondary techniques need to be calibrated and maintained regularly. Also, maintenance and consumables add significantly to the cost of ownership. For example, although Supercritical Fluid Extraction (SFE) is reasonably fast, it requires high maintenance and the cost of compressed CO_2, used to extract oil is also significant. Near Infra-Red (NIR) is commonly used for on-line monitoring but is difficult to apply on opaque samples as it can only scan the surface. It is also complex to calibrate as measurements are sensitive to product granularity and spices, and therefore it is difficult to maintain accurate results on a large variety of product types. For that primary reason, NIR has limited applicability for the quality control of oil content in snack foods.

Although Nuclear Magnetic Resonance (NMR) has been used for decades for measuring oil content, it is only now that it is being used for measurement of oil content in snack foods. NMR has great advantages over other secondary techniques:

- It can be calibrated to cover a concentration range from 0 to 100% oil.
- NMR is very stable over the long term and rarely needs calibration adjustment..
- It is virtually insensitive to sample granularity and additives such as spices.
- Because NMR penetrates through the whole sample and is insensitive to air voids, it provides the most accurate measurement of the total amount of oil in a given volume of sample.
- A primary calibration can be produced using a single sample of oil. The measurement precision is typically better than 0.1% oil. The results are then comparable to those of acid hydrolysis followed by soxhlet extraction (e.g. Weibull-Stoldt) which provides a measurement of the total amount of oil.

OXFORD INSTRUMENTS

Benchtop NMR for Food & Agriculture

- The measurement is rapid (less than a minute), although it requires the sample to be left to condition at 40°C for 15 minutes prior to analysis.
- The NMR technique is non-destructive so the same sample may be measured several times before being analysed by other techniques.

Calibration and Results

Table 1 summarises the results of several samples that were selected according to their characteristic compositions and manufacturing process. The samples were analysed and their NMR values (signal/mass) compared against their respective reference values.

The data points plotted in Figure 1, demonstrate the accuracy of the NMR technique as the NMR signal data aligns on a straight line and the results are relative to the NMR signal of 100% corn oil. Although the instrument can be calibrated using a 100% corn oil sample, Setting-Up-Samples (SUSs) that are stable long term can be allocated to reference concentrations for routine checks. Regular checks are used to ensure that an instrument provides results within acceptance limits.

Table 1 also shows that there is no significant effect whether the samples have been crushed or thoroughly ground.

Also, when different oil types are compared, there is no significant difference whether it is fresh or processed corn oil. Table 2 shows that the repeatability (or precision) of the measurement on the same sample is very good. There is only a small difference between corn oil, sunflower oil, cottonseed oil and even palm oil, as shown in Table 3.

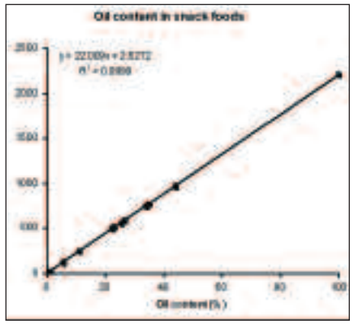


Figure 1: Correlation between NMR signal/mass and reference values

Snack product	Ref (%)	NMR (%)	Diff (%)
A crushed	0.83	0.91	0.08
A ground	0.83	1.00	0.17
B crushed	5.59	5.46	-0.13
B ground	5.59	5.64	0.05
C crushed	11.07	11.00	-0.07
C ground	11.07	11.04	-0.03
E ground	22.18	22.44	0.26
F ground	22.98	23.29	0.31
G crushed	25.58	25.02	-0.56
G ground	25.58	25.11	-0.47
H crushed	26.51	26.60	0.09
H ground	26.51	26.60	0.09
I crushed	33.96	34.06	0.10
I ground	33.96	33.68	-0.28
J ground	34.84	35.01	0.17
H crushed	43.90	43.66	-0.24
H crushed	43.90	44.34	0.44
Corn oil product line 1	100.00	99.80	-0.20
Corn oil product line 2	100.00	100.37	0.37
Standard error of estimate (%)			**0.23**

Table 1: Comparison between NMR results and reference values obtained from solvent extraction

Measurement number	Oil content by NMR (%)
1	34.74
2	34.69
3	34.80
4	34.70
5	34.77
6	34.69
7	34.72
8	34.79
9	34.74
10	34.71
Standard deviation (%)	**0.04**
Precision (2σ, %)	**0.09**

Table 2: Repeatability measurements by NMR

Oil type	Oil content by NMR (%)
Corn	100.00
Cotton seed	99.12
Sunflower	99.59
Palm	99.95

Table 3: Comparison of NMR measurements on different oils

Recommended Instrument

The **MQC**-23 with a 0.55 Tesla (23 MHz) magnet, fitted with a 26mm diameter (21ml) probe is a suitable instrument for this application. The Oil in Snack Food package comprises:

- **MQC**-23 with a built-in computer, operating the latest version of Microsoft® Windows® (no separate PC is required)
- **MultiQuant** software including **RI Calibration**, **RI Analysis**, and the **EasyCal** 'Oil in Snack Food' application
- Three setting up standards (SUSs) at 10, 25 and 40% oil content for calibration, maintenance and quality control
- 26mm diameter sample vials
- PTFE sample holder
- PTFE sample packing tool
- Installation manual
- Method sheet

In addition to this package you will also require:

- A dry heater and aluminium block with holes for sample conditioning
- A precision balance

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

- High signal sensitivity. This is particularly important for maintaining a good measurement precision on snack foods where the oil content and packing density can be low
- Small benchtop footprint
- Low maintenance
- The sample tubes are recyclable, lowering consumable costs
- Minimal sample preparation

Application note

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