

### Summary

- Up to 250 times faster than wet chemistry methods\*
- No hazardous solvents required; no hazardous waste produced
- Easiest, most reliable technique available; suitable for unskilled operators – simple, intuitive visual software
- Simple linear calibration; no chemometrics
- Insensitive to sample form and additives

\* Not including sample conditioning time



### Application

Measuring the oil content of dried snack foods is essential for quality control to ensure that the products meet their nutritional values specifications. 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.

### Advantages of NMR

Solvent extraction techniques are commonly used for determination of fat content. However, they tend to be slow, laborious, 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 calibration against the reference technique used. Although they are fast and easy to maintain, many secondary techniques need to be calibrated and maintained regularly.

Maintenance and consumables also 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<sub>2</sub> 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.

In contrast, Nuclear Magnetic Resonance (NMR) provides a simple, accurate and inexpensive measurement of oil content in snack foods which is routinely used in factories around the world.

### Method

Benchtop NMR detects the signal from oil and distinguishes it from signals from solids and bound water (<10% moisture).

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**Calibration**

Although a primary calibration can be produced using a single sample of oil (100%), Setting Up Samples (SUSs) that are stable long term can be allocated to reference concentrations (nominally 10, 25 and 40%). Subsequently the mid-range SUS is used for routine checks to ensure that an instrument provides results within acceptance limits. Should the result fall outside these limits, the low and high SUSs are used to recalibrate the instrument.

The results are comparable to those of acid hydrolysis followed by soxhlet extraction (e.g. Weibull-Stoldt) which provides a measurement of the total amount of oil. Should the user require the results to match those from Soxhlet, they will need to measure a representative set of samples using both NMR and Soxhlet to determine the skew and bias from the 100% oil calibration thus in the long term only one calibration needs to be maintained.

**Measurement**

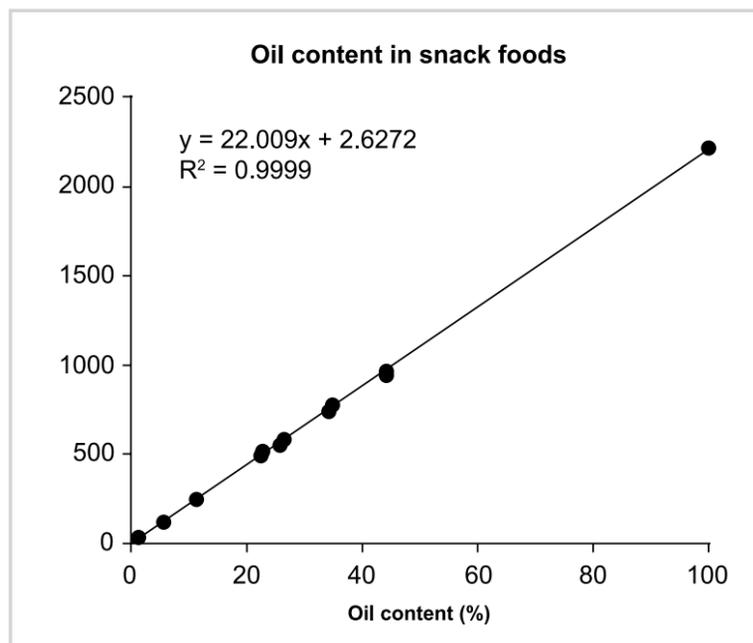
A sample vial is tared then filled to a given height with the sample using a dedicated packing tool. Each sample is then weighed before being placed in a conditioning block for 20 minutes. The conditioning temperature is usually 40°C, the same as the magnet, for optimum precision. For some oils, particularly palm oil, the conditioning temperature needs to be higher to ensure the oil has totally melted.

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
<b>Standard error of estimate (%)</b>			<b>0.23</b>

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

**Results**

Table 1 summarises the results of several samples that were selected according to their characteristic compositions and manufacturing process. The oil contents determined by NMR are compared against those determined by the reference technique, in this case solvent extraction. The NMR data (signal/mass) plotted against those reference values in Figure 1 demonstrates the accuracy of the NMR technique as all the data points align on a straight line and the results are relative to the NMR signal of 100% corn oil from which the samples were produced.



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

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 between fresh or processed corn oil. Table 2 shows that the repeatability (or precision) of the measurement on the same sample is very good. Table 3 shows there is only a small difference in the apparent oil content between corn, sunflower, cottonseed and even palm oil owing to differences in their hydrogen density.

**Conclusion**

- A primary calibration can be produced using a single sample of oil which in practice can cover concentrations ranging from 0 to 100%. A separate calibration may be required for low concentrations
- NMR is very stable over the long term and rarely needs calibration adjustment. If required, this can be done simply using stable Setting Up Samples which recreates the original calibration carried out during installation
- NMR is virtually insensitive to sample granularity and additives such as spices, flavours, colours and salt
- NMR penetrates through the whole sample and is insensitive to air voids, which means it provides the most accurate measurement of the total amount of oil in a given volume of sample
- The measurement precision is typically better than 0.1% oil
- The NMR technique is non-destructive so the same sample may be measured several times before being analysed by other techniques
- Sample measurement time is rapid (32 seconds)\*

\* For optimal precision samples should be conditioned at 40°C for 20 minutes in a dry block heater prior to analysis.

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
<b>Standard deviation (%)</b>	<b>0.04</b>
<b>Precision (2σ, %)</b>	<b>0.09</b>

**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



## Oxford Instruments Ready-to-Run Application Package

The **MQC-23** with a 0.55 Tesla (23 MHz) magnet, fitted with a 26 mm diameter (10 ml sample) probe is ideal for this application. The Oil in Snack Food package comprises:

- **MQC-23** which can be controlled using its own built-in computer operating Microsoft Windows or via a stand-alone PC
- **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
- Test/tuning sample

- 23 mm diameter sample vials with caps
- PTFE sample vial holders
- PTFE sample packing tool
- Installation manual
- Method sheet

### In addition you may require:

- A dry block heater and aluminium block with holes for sample conditioning at 40°C
- A precision balance



## Oxford Instruments Magnetic Resonance

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