

# Determination of Oil and Moisture Content in Fish Feed



Fish feed manufacturers produce pellets that vary in size, oil content (from 5 to 40%) and moisture (usually less than 10%). Measuring the oil and moisture content is essential for quality control to ensure that the products meet their nutritional values. The amount of oil used is particularly important in terms of the significant cost of the raw material and the affect it may have on the texture and perceived quality of the product. Low resolution Nuclear Magnetic Resonance (NMR) provides a method which is quick and easy to perform, simple to calibrate, and not dependent on the sample matrix.

## 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<sub>2</sub> used to extract oil is also significant. Near Infra-Red (NIR) is usually used for trend analysis and is often applied to on-line monitoring. However it is difficult to apply on opaque samples as it can only scan the surface. It is also complex to calibrate and 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 fish feed of various types.

Although NMR has been used for decades to measure oil and moisture contents (for instance, the ISO10565 standard method for seeds), it has only recently been applied to fish feed. 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 a calibration adjustment.
- It is virtually insensitive to additives and sample granularity has no significant effect.
- Because NMR penetrates through the whole sample and is insensitive to the presence of air voids, it provides the most accurate measurement of the total amount of oil in a given volume of sample.



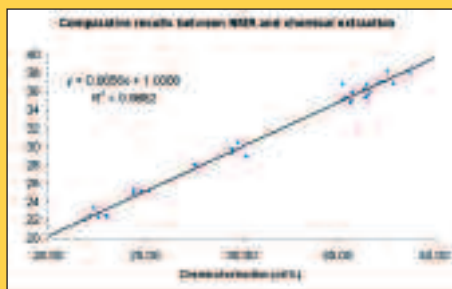


Figure 1: Comparative results between predicted NMR results and reference oil contents (%)

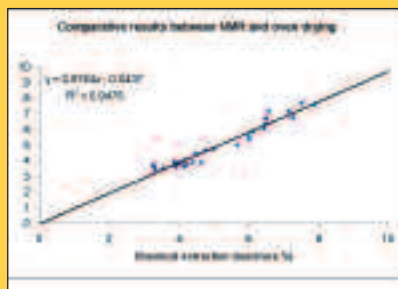


Figure 2: Comparative results between predicted NMR results and reference moisture contents (%)

- A primary calibration is produced using a single sample of oil. The measurement precision is typically less than 0.1% oil. The results are then comparable to those of acid hydrolysis followed by soxhlet extraction for the measurement of the total amount of oil.
- The measurement is rapid (less than a minute), although it is recommended to pre-condition the sample for 15 minutes at 40°C.
- The NMR technique is non-destructive so the same sample may be measured several times before being analysed by other techniques.

Samples are simply loaded into pre-tared glass vials, weighed, conditioned, then inserted into the instrument which detects the sample, automatically starting the NMR analysis. The instrument returns the oil and moisture content values in less than one minute.

## Calibration and Results

The NMR instrument is initially calibrated using a 100% oil sample or a set of reference standards with known oil and moisture contents. Setting-Up-Samples (SUSs) that are stable over the 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. Several samples of fish feed were analysed by NMR and their oil and moisture signal/mass values were compared against the contents determined by solvent extraction and oven drying respectively.

Figures 1 and 2 show those results align on a straight line demonstrating the accuracy of the NMR technique. The measurement precision is typically less than 0.2%.

## 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 and Moisture in Fish 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 and Moisture in Fish Feed' application
- 26mm diameter 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
- Small benchtop footprint
- Low maintenance
- The sample tubes are recyclable, lowering consumable costs
- Minimal sample preparation

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