Determination of Oil and Moisture Content in Fish Feed

Application Note 4

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 because 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, 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 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 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.

NMR has been used for decades to measure oil and moisture contents (for instance, the ISO10565 standard method for seeds) which has also been found to be applicable to fish feed. NMR has great advantages over other secondary techniques.

Advantages of benchtop NMR

- A primary calibration can be produced using an oil sample which covers a concentration range from 0 to 100%. The results are comparable to those of acid hydrolysis followed by soxhlet extraction for the measurement of the total amount of oil
- NMR is stable over the long term, therefore requires little recalibration
- 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
- The sample measurement time is short (less than one minute)*
- Calibration maintenance samples samples with a range of oil and water contents, are used to check and maintain the original calibration
- The NMR technique is non-destructive so repeatability measurements can be made conveniently

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.

*It is recommended to pre-condition the sample for 15 minutes at 40ºC for better precision.

OXFORD INSTRUMENTS

The Business of Science®

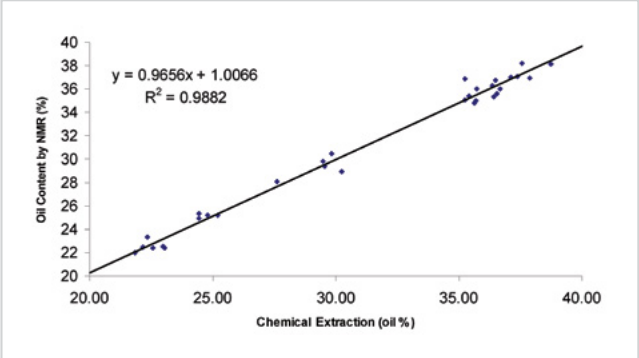

Figure 1: *Comparative results between predicted NMR results and reference oil contents (%)*

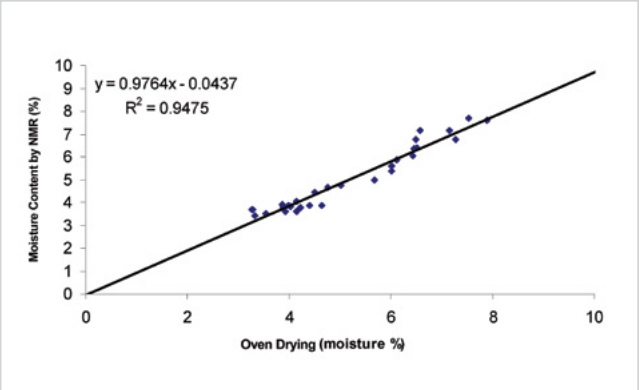

Figure 2: *Comparative results between predicted NMR results and reference moisture contents (%)*

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. Several samples of fish feed were analysed by NMR and their oil and moisture values predicted by the calibration 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 Configuration

The **MQC+** with a 0.55 Tesla magnet fitted with a 26 mm diameter (10ml) probe is ideal for this application. The "Oil and Moisture in Fish Feed" package comprises:

- The **MQC+** with a built-in computer operating Microsoft® Windows® 10 (no separate PC is required).
- **MultiQuant** software including **RI Calibration**, **RI Analysis**, and the **EasyCal** 'Oil and Moisture in Fish Feed' application
- 26 mm diameter vials
- Oil and moisture calibration maintenance samples
- PTFE sample holders
- PTFE sample packing tool
- User manuals
- Method sheet

In addition to this package you will also require:

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

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

- High signal sensitivity
- Small benchtop footprint
- Specific "Oil and Moisture in Fish Feed" applications software
- Low maintenance
- Minimal sample preparation

visit **www.oxford-instruments.com/mqc** for more information or email: **magres@oxinst.com**

OXFORD INSTRUMENTS

The Business of Science®

CE