Measurement of Oil and Moisture in Seeds according to ISO 10565:1998

Application Note 3

Accurate and fast determination of oil content is important to breeders, growers and buyers for determining the commercial value of oil-bearing crops such as rape (canola), sunflower, linseed, soya bean and groundnut. Nuclear Magnetic Resonance (NMR) offers a clean, rapid and accurate alternative to traditional wet chemical techniques and is easier to calibrate than Near Infra-Red (NIR).

Method

The oil and water in seeds method is based on a combination of the Free Induction Decay (FID) and spin echo pulse sequences. The combined pulse sequence can differentiate between signals from solids and bound water (short T_2 times) and oil (long T_2) and quantify both separately.

The most important parameter for this application is the time duration, τ, between the first (90°) and second (180°) radio frequency pulses; the value is 3.5 ms for the ISO standard method. This time is chosen in such a way that the signal from the tightly bound water will have decayed before acquisition of the echo giving signal solely from the oil. Therefore it is only applicable to samples that have less than 10% moisture. Samples with more than 10% moisture have to be oven-dried prior to NMR analysis.

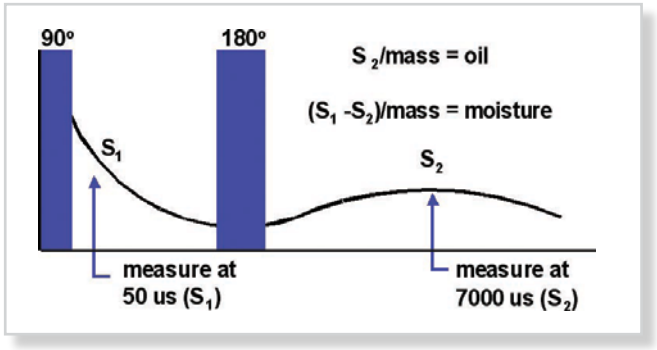
Higher moisture levels can be measured by altering the NMR acquisition parameters, however the method will no longer be ISO compliant. The total oil plus moisture signal is measured from the FID and the oil signal from the echo. The moisture signal is obtained by taking the difference between these two values.

Advantages of benchtop NMR

- NMR is a very stable technique over the long-term and therefore requires little re-calibration
- Minimal sample preparation is required
- No solvent extraction is required
- The NMR technique is non-destructive, so repeatability measurements can be made conveniently
- Sample measurement time is short (typically 16 seconds)
- "Setting-up" samples, with a range of oil and water contents are used to check and maintain the original calibration

Calibration and Results

As NMR is a comparative technique, a set of calibration standards of known oil and water contents must be obtained before measurements can take place. Thus, the quality of the calibration will always be dependent on the accuracy of the reference data. It is recommended that at least six calibration standards should be used with the oil and water contents spread evenly over the range of interest. Real seed samples can be used as calibration standards. They are weighed before and after drying to give their moisture content. The oil content is normally measured using a solvent extraction technique. Alternatively, a primary oil calibration can be produced using a single sample of the pure oil to be analysed. Since different types of seed (and oil) result in slightly different NMR signals, a better accuracy is achieved when all the standards are of the same species. If measurements on more than one species are required it is recommended that a separate calibration be created for each.



Nine independently-analysed rape seed samples were measured. Their oil content varied from 39% to 51% and their water content varied from 5.2% to 7.1%. Calibrations for oil and water were developed according to ISO 10565 using Oxford Instruments' **MultiQuant** software, which allows simultaneous calibration and measurement of up to four sample constituents. Measurement time was 16 seconds per sample.

The resulting calibrations are shown in figures 1 and 2.

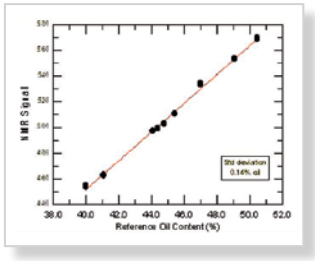


Figure 1: *Calibration for oil content of rape seeds in presence of water*

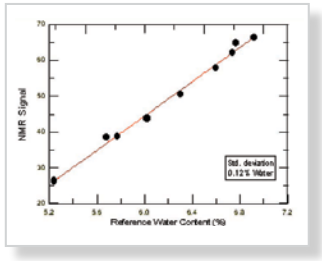


Figure 2: *Calibration for water content of rape seeds in presence of oil*

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Table 1: Results of instrument and sample repeatability

Value	Repeat Measurements										Mean	SD
44.25	44.29	44.25	44.22	44.23	44.26	44.27	44.22	44.23	44.22	44.18	44.24	0.03
Value	**Portion Measurements**										**Mean**	**SD**
39.5	39.7		39.2		39.4		39.5		39.8		39.5	0.24

Instrument repeatability for oil was then tested by measuring one sample ten times without removing it from the instrument. Sample repeatability was tested for oil content by measuring five different portions of the same sample. Instrument repeatability was shown to be 0.03% and sample repeatability 0.21%. The results from both sets of experiments are shown in Table 1 above.

Recommended Instrument Configuration

There are two instruments suitable for this application both of which conform to the industry standard ISO 10565:1998 for a range of sample volumes (given in brackets):

For large sample/seed analysis

- **MQC**-6 with 60mm (150ml) or 51mm (80ml) diameter probes.

For small, low quantity or single seed analysis

- **MQC**-23 with 26mm (14ml), 18mm (8ml) or 10mm (1ml) diameter probes.

All Oil and Moisture in Seeds packages comprise:

- The **MQC**-6 (or -23) which can be controlled using its own built-in computer using Microsoft Windows® or via a stand-alone PC
- **MultiQuant** software including **RI Calibration, RI Analysis**, and the **Easycal** 'Oil and Moisture in Seeds' application
- Glass tubes
- Installation manual
- Method sheet

In addition you may also wish to purchase:

- Optional: a dry heater and aluminium block with holes for sample conditioning at 40°C (26, 18 and 10mm probes only)

  N.B. The ISO method requires measurement at a nominal room temperature of 17-28°C. Conditioning at 40°C is preferable where precision measurements are required for oil content only.
- A precision balance
- Oil and moisture setting up standards (for 60, 51 and 26mm probes only)

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

- High signal sensitivity
- Small benchtop footprint
- Low maintenance
- Minimal sample preparation
- Auto-weighing facility
- Automatic post-measurement calculation of oil content (with respect to 9% moisture and dry weight)
- Optional "setting-up" standards for calibration maintenance

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