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

Application Note 3

Accurate and fast determination of oil and water 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 calibrations are much easier to maintain than for Near Infra-Red (NIR). NMR is also used for measuring oil yield in development of improved crop varieties and by seed crushers to measure oil content of the press cake and residues to determine extraction efficiency (ISO 10632:2000).

Method

The oil and water in seeds method involves differentiating the two analytes on the basis of their NMR relaxation times. The solid signal decays rapidly leaving a signal dependant on the oil and bound water only. Oil is then individually determined by using an NMR refocussing technique to measure the signal at a convenient time after the bound water has decayed. The water is determined by taking the difference between these two signals. This methodology is described in ISO 10565:1998.

Advantages of benchtop NMR

- NMR is a very stable technique over the long-term and therefore requires little re-calibration
- NMR is a bulk technique, and is not sensitive to surface variations
- 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

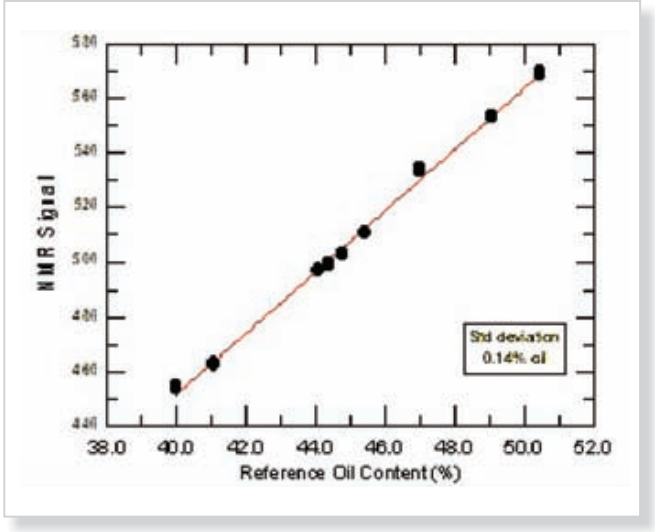

Figure 1: *Calibration for oil content of rape seeds in presence of water*

Calibration and Results

As NMR is a comparative technique, a set of calibration standards consisting of real seeds with known oil and water contents must be obtained before measurements can take place. The quality of the calibration will always be dependent on the accuracy of this reference data. Although only three well characterised seed standards are required, it is recommended that at least six should be used, with oil and water contents that span the concentrations of interest. The seed samples are weighed before and after drying to obtain reference values for water content. Oil reference values are normally determined using solvent extraction. Alternatively, a primary oil calibration can be produced by NMR using a single sample of the pure oil to be analysed.

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Since different types of seed (and oil) result in slightly different NMR signals, best accuracy is achieved when the standards are of the same species as the measurement samples. 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 measurement of up to four sample constituents. Measurement time was 16 seconds per sample.

The resulting calibrations are shown in figures 1 and 2.

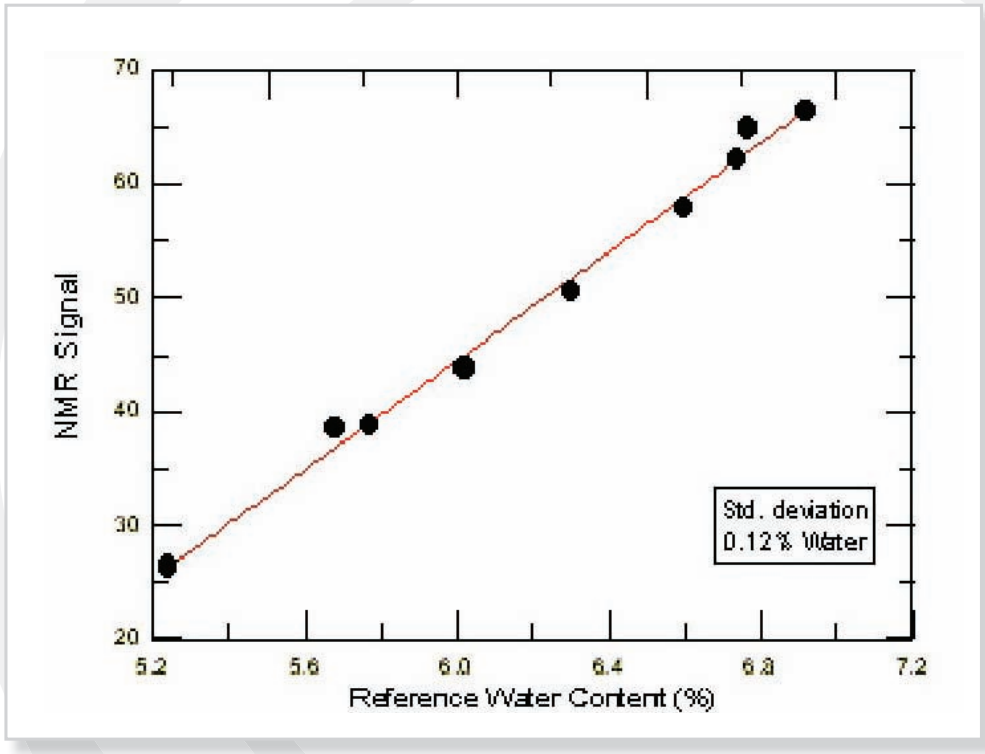

Figure 2: *Calibration for water content of rape seeds in presence of oil*

Table 1: 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 0.24%. The results from both sets of experiments are shown in Table 1.

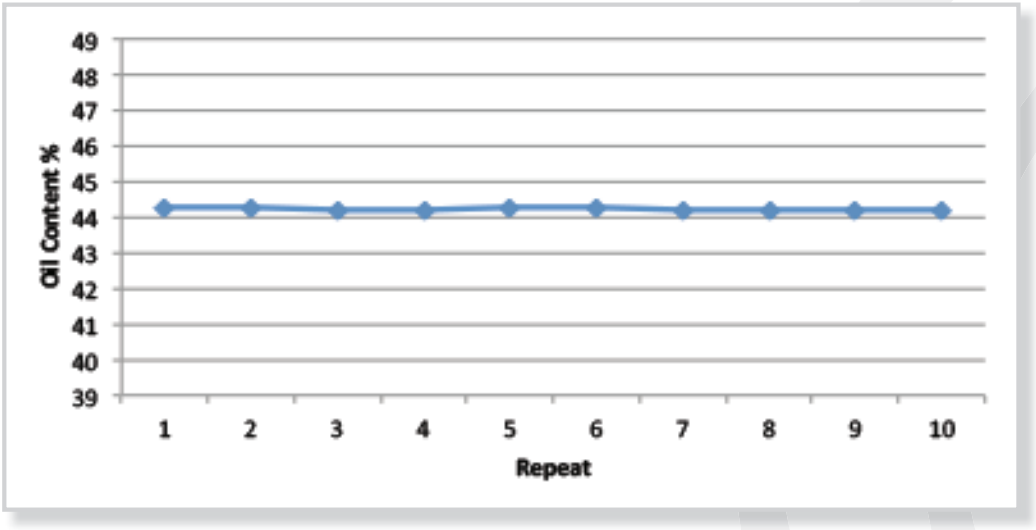

Figure 3: *Instrument repeatability*

Comprehensive Oil and Water in Seeds packages are available, which include:

- The **MQC**5 (or **MQC**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 Water in Seeds' application
- Glass tubes
- Oil and water "calibration maintenance standards" (for 51 mm, 40 mm and 26 mm probes only)
- Installation manual
- Method sheet

In addition you may also wish to purchase:

- Required: A precision balance (2 decimal places for 80 mL and 40 mL, and 3 decimal places for 14 mL, 7 mL and 1 mL samples)
- 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 specifies 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.

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**5 with 51 mm (80 mL), or 40 mm (40 mL) diameter probes.

For small, low quantity or single seed analysis

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

The instrument offers multiple advantages:

- High signal sensitivity
- Reproducible results without frequent recalibration
- Minimal sample preparation
- Low maintenance
- Auto-weighing facility
- Small benchtop footprint
- Automatic post-measurement calculation of oil content (with respect to dry weight and water)
- "Calibration maintenance standards" (26 mm, 40 mm, 51 mm probes)

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