

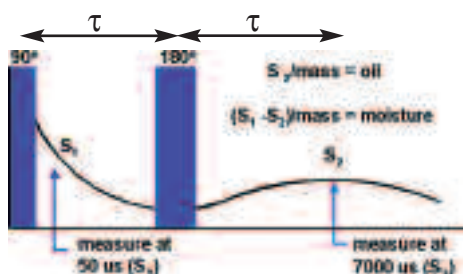
Measurement of Oil and Moisture in Seeds according to ISO 10565:1998



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 in seed method is based on the Free Induction Decay (FID)/spin-echo pulse sequence which is used to detect both NMR signals with short T2 relaxation times, such as those from solids or tightly bound water and long T2 signals such as those from free liquids (e.g. oil).



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 fat. 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.

Larger moisture levels can be measured by altering the NMR acquisition parameters, however the method will no longer be ISO compliant.

The oil and 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.

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 calibration will always be dependent on the quality 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. Since different types of seed 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 samples of rape seed were received, with oil content varying from 39% to 51%, and water content varying from 5.2% to 7.1%. Calibrations for oil and water were then 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.



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	0.214					

The resulting calibrations are shown in figures 1 and 2.

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.

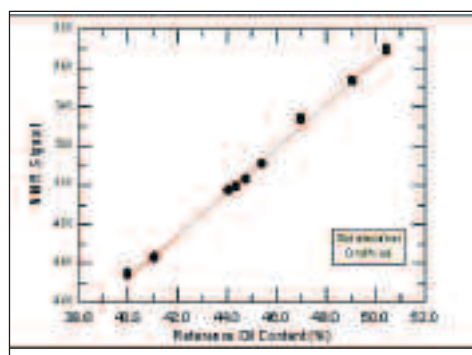


Figure 1: Calibration for oil content of rape seeds in presence of water

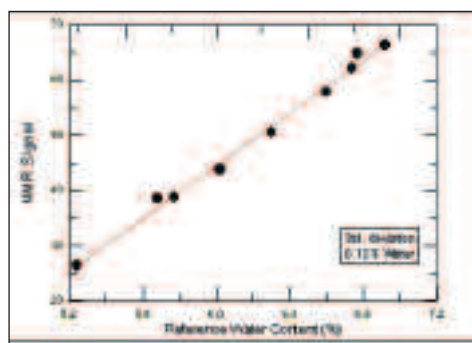


Figure 2: Calibration for water content of rape seeds in presence of oil

Recommended Instrument

The **MQC-23** fitted with a 26mm diameter (21ml) probe is a suitable instrument for this application and conforms to the industry standard ISO 10565:1998. The Oil and Moisture in Seeds 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 Seeds' application
- Glass tubes
- Installation manual
- Method sheet

In addition to this package you will also require:

- A dry heater and aluminium block with holes for sample conditioning at a fixed temperature (between 19 and 28°C)
- 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

Note: Other instruments/packages are available for larger seeds, or single seed analysis. Please contact Oxford Instruments for further details.

Oxford Instruments Molecular Biotoools Ltd

UK

Tubney Woods
Abingdon
Oxfordshire
OX13 5QX
Tel: +44 (0)1865 393 200
Fax: +44 (0)1865 393 333

USA

8403 Cross Park Drive
Suite 3F
Austin
Texas 78754
USA
Tel: +1 (512) 339-0640
Fax: +1 (512) 339-0620

China

Unit 1609, Liu Lin Mansion
No. 1 Huai Hai Road (Middle)
Shanghai 200021
China
Tel: +86 (0) 21 6387 6749
Fax: +86 (0) 21 6373 7749

Email:
benchtopNMR@oxinst.co.uk

Visit our website at
www.oxford-instruments.com