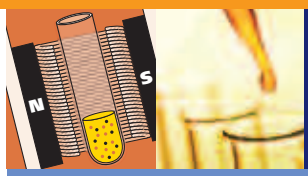
Measurement of Oil and Moisture in Seeds according to ISO 10565:1998



NMR

Benchtop NMR for Food & Agriculture

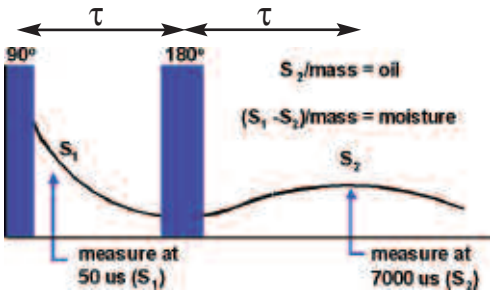
Accurate and fast determination of oil content is important to breeders, growers and buyers for determining the commercial value of oil-bearing crops such as rape (canola), sunflower, linseed, soya bean and groundnut. Nuclear Magnetic Resonance (NMR) offers a clean, rapid and accurate alternative to 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. Alternatively it is possible to produce a primary oil calibration using a single sample of the oil to be analysed. Real samples can be dried to provide reference values which after NMR analysis can be entered later to produce the moisture calibration. 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 samples of rape seed were analysed, with oil content varying from 39% to 51%, and water content varying 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.

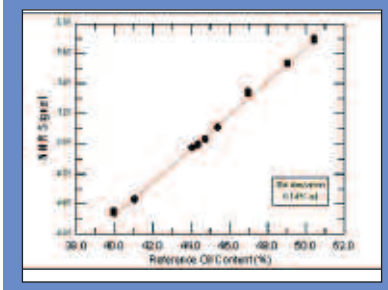


Figure 1: Calibration for oil content of rape seeds in presence of water

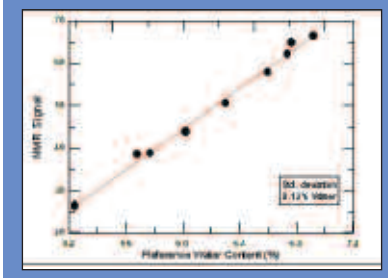
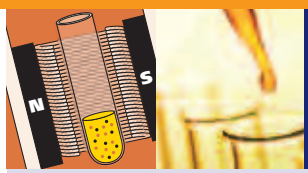


Figure 2: Calibration for water content of rape seeds in presence of oil

The Business of Science®

OXFORD INSTRUMENTS



NMR

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

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 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:

- **MQC**-5 (or -23) with a computer incorporated, 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 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
- The sample tubes are recyclable, so there are no consumables
- Minimal sample preparation

Distributed by:

Oxford Instruments Molecular Biotools 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 (21) 6387 6749
Fax: +86 (21) 6373 7749

E-mail: benchtopNMR@oxinst.co.uk

Visit our web site at
www.oxford-instruments.com

OXFORD INSTRUMENTS