

Simultaneous determination of water and oil content in seeds using NMR spectroscopy (revised in 2015)

Introduction

Seeds such as sunflower, soybean, groundnut, cotton seeds and rape are grown for their oil content; therefore an accurate and fast determination of oil content is important to breeders, growers and buyers. On the other hand excess of water or moisture content reduces cost and shelf life of agriculture products.

The traditional method of oil determination is based on solvent extraction, which is time consuming, uses harmful reactivities and solvents, moreover results depends on operator importance. Thus, such methods have low accuracy and reproducibility. IR spectroscopy is very suitable for this but is too difficult to calibrate devices. Moisture content can be evaluated by consequent drying and weighting but it requires a lot of time.

Method

Low-resolution NMR spectroscopy is a powerful technique for rapid simultaneous determination of both parameters. NMR provides the quantitative determination of water and oil fractions because water is bound to the cellulose matrix and NMR signal from water content decays quickly in comparison with relatively free oil signal.

In this application 2 pulses sequence FID-Spin echo is used (figure 1).

The NMR signal immediately after the 90° RF pulse is proportional to the total oil plus water content, and the echo signal after 180° RF pulse proportional to the oil content only. Pure water content can be determined by subtracting spin echo amplitude from initial FID amplitude.

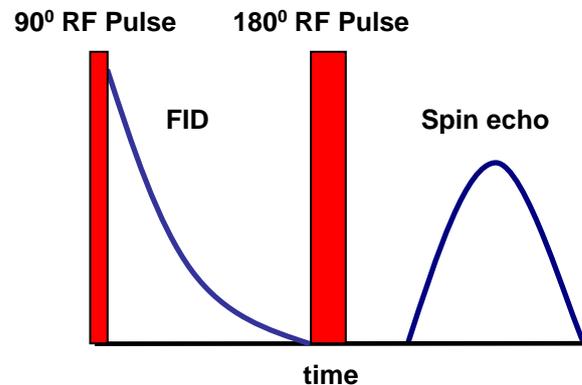


Fig 1. FID-Spin echo sequence

Equipment

NMR-analyzer **Spin Track** (figure 2) from Resonance System Ltd. is an ideal instrument for oil and moisture application because of short dead time, high acquisition rate and high signal to noise ratio that make measurements very reproducible and accurate. Small weight and mobility allow its using in industrial routine analysis as well as in specialized laboratories. **Spin Track** fulfills to requirements of standard **ISO 10565**.



Fig 2. NMR analyzer **Spin Track**

Spin Track based on modern electronics, very simple to use because of all measurements are made automatically pressing one button.

A majority of seeds are large in size. Usage of widely used NMR probes leads to inaccuracy in measurements due to large differences among individual seeds and requires a large number of seeds to be measured simultaneously. **Spin Track**

provides this using the sample of 45 ml volume. Calibration procedure for oil and moisture content meets **ISO 10565**. Calibration curves for oil and moisture content are shown at figures 4 and 5.

Measurement duration is not more than 2 min.

Measured parameter	Value range	Relative error	RMS error
Oil, %	5-60	0.5	0.2
Moisture, %	5-10	0.5	0.2

Using additional hardware increases measured moisture content up to 30%.

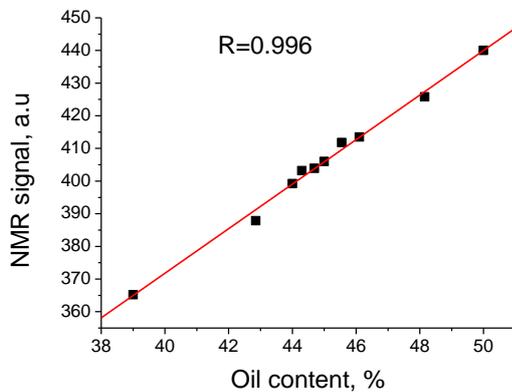


Fig. 4. Calibration for oil content

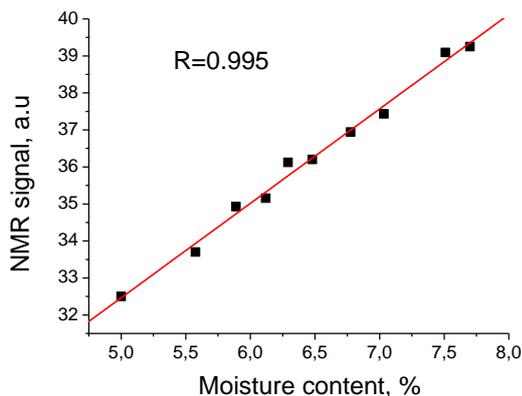


Fig. 5. Calibration for moisture content

Software

NMR spectrometer is controlled by original software **Relax** developed by Resonance Systems Ltd.

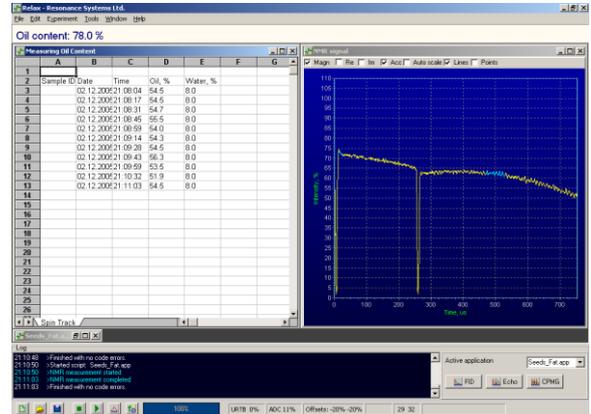


Fig. 3. Relax software interface

Relax allows:

- Automatic calibration procedure
- Automatic measurements
- Simultaneous determination of oil and moisture content
- Automatic report generation
- Data logging
- Daily check of the analyzer
- Test of all functional modules

Relax uses Pascal-oriented script language and experiment can be easily constructed by the operator. All basic measurement operations are embedded and included in standard offer.

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