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Oilseed residues — Simultaneous determination of oil and water contents — Method using pulsed nuclear magnetic resonance spectroscopy

Tourteaux de graines oléagineuses — Détermination simultanée de la teneur en huile et en eau — Méthode par résonance magnétique nucléaire pulsée

Reference number
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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 10632 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Subcommittee SC 2, *Oleaginous seeds and fruits*.

Annex A of this International Standard is for information only.

Introduction

The method specified in this International Standard has been subjected to an international ring test. However, no precision data have been calculated because of an insufficient number of participating laboratories. See clause 11 and annex A.

Oilseed residues — Simultaneous determination of oil and water contents — Method using pulsed nuclear magnetic resonance spectroscopy

1 Scope

This International Standard specifies a rapid method using nuclear magnetic resonance (NMR) for the determination of the oil and water contents of oilseed residues obtained after oil extraction by pressure or solvents (excluding mixed products).

It is applicable to oilseed residues as flour, plates or agglomerates, provided that the particles are smaller than 2 mm and that the water content is not higher than the conversion threshold.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 734-1, *Oilseed residues — Determination of oil content — Part 1: Extraction method with hexane (or light petroleum).*

ISO 734-2, *Oilseed residues — Determination of oil content — Part 2: Rapid extraction method*.

ISO 771, *Oilseed residues — Determination of moisture and volatile matter content*.

ISO 5502, *Oilseed residues — Preparation of test samples*.

3 Principle

The test sample is placed in the magnetic field of a pulsed NMR spectrometer. An alternating electromagnetic field is applied in the form of an intense 90° radiofrequency (RF) pulse which excites all the hydrogen nuclei. The free induction decay (FID) is recorded following the 90° pulse. The maximum amplitude of this signal is proportional to the total number of protons from the water and oil phases of the sample.

A second RF pulse, a so-called 180° pulse, is then applied to produce a spin-echo signal when only the signal from the oil phase contributes to the FID.

NOTE 1 The maximum amplitude of this echo signal is proportional to the oil content. It varies with the sample temperature following a complex law. An increase in temperature decreases the measured value of the echo.

The difference between the two amplitudes, which is proportional to the water content, is calculated.

After suitable calibration of the apparatus, the measured signals are automatically converted into percentages of oil or water.

NOTE 2 Simultaneous indications of the oil and water contents can be given by some spectrometers equipped with a minicomputer and specific program.

4 Calibration samples

4.1 General

Calibration samples shall be homogeneous and free from impurities.

4.2 Samples for moisture-content calibration

The water content of oilseed residues can vary depending on storage conditions. It is therefore necessary to determine this just prior to calibration in accordance with ISO 771.

4.3 Samples for oil-content calibration

Samples from oilseed residues shall be of the same species as the test samples and of similar fatty acid compositions (especially for the analysis of rapeseeds which are rich in erucic acid, and for sunflower seeds which are rich in oleic acid). Oil content shall be determined using the reference method described in ISO 734-1.

5 Apparatus

Usual laboratory apparatus and, in particular, the following.

5.1 Pulsed low-resolution NMR spectrometer, suitable for measurement of the oil content and water content of oilseeds.

The instrument's parameters shall follow the manufacturer's instructions or specifications.

CAUTION: Remove metallic objects from the proximity of the NMR spectrometer.

5.2 Sample tubes, made of glass, suitable for use with the NMR spectrometer.

5.3 Analytical balance, electronic, capable of weighing to an accuracy of 0,01 g, by preference linked to the NMR spectrometer so that the sample mass is recorded directly.

5.4 Drying oven, capable of being maintained at 103 °C ± 2 °C.

5.5 Dishes, made of glass or metal, of diameter 7 cm, and provided with lids.

5.6 Desiccator, equipped with an efficient desiccant.

5.7 Mechanical grinding mill or **mortar,** easy to clean, and capable of grinding the plates or agglomerates of oilseed residues to particles smaller than 2 mm, without heating and without perceptible modifications in water, volatile matter and oil contents.

6 Sampling

It is important that the laboratory receive a sample which is truly representative and has not been damaged or changed during transport or storage.

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 5500.

7 Preparation of test sample

Prepare the test sample in accordance with ISO 5502.

Remove from the prepared test sample all metallic objects (e.g. staples, needles, etc.).

8 Calibration procedure

8.1 General procedure

8.1.1 In many oilseed residues, the oil content is low (1 % to 3 %) compared to the water content (8 % to 12 %). The available range of products has a very small oil or water content lower, usually lower than 2 %. In this case, procedure B is to be preferred, carried out on two samples to give six points on the calibration curve. When the water content is higher than the oil content, the NMR signal S_1, measured after 50 µs, is very strong compared to the spin-echo signal S_2 of the oil. So, for the calibration and measurement of the oil content, it is recommended to use the value (in decibels) of the NMR signal amplification of the spectrometer optimized for the highest signal, which will be consequently less effective for the lowest signal.

8.1.2 Use the set-up parameters of the NMR spectrometer (5.1) recommended by the manufacturer and optimize them by preliminary tests. For all calibration and measurement operations, follow the user's manual. Pay strict attention that all operations during calibration and measurement are carried out under the same conditions and, in particular, at the same temperature (± 2 °C).

A minimum of three calibration samples is necessary, although more than three samples may be used.

8.1.3 Enter the parameters for the measurements (pulse sequence, attenuation, etc.) of the oil or water content (as applicable) into the NMR spectrometer following the manufacturer's recommendations, and specify a code number under which the calibration curve is to be stored. The total measurement time shall be a minimum of 25 s.

8.1.4 Set the apparatus to the calibration mode.

8.1.5 Introduce a portion of the first calibration sample into a tared sample tube (5.2), up to the optimum height specified by the manufacturer, and transfer the value of the sample mass from the balance to the NMR spectrometer.

NOTE Manual transfer of the mass of the calibration sample to the NMR spectrometer is also possible.

8.1.6 Enter, as applicable, the value of either the water content (as a percentage by mass) or the oil content into the spectrometer.

8.1.7 Introduce the sample tube containing the first calibration sample into the measuring head and determine the water or oil content.

8.1.8 Repeat steps 8.1.5 to 8.1.7 for the two (or more) other calibration samples.

8.1.9 Calculate automatically or manually the calibration parameters of the calibration curve.

The correlation coefficient shall normally be greater than 0,95. If this in not the case, check the values obtained using the reference methods specified in ISO 734 and ISO 771, respectively, or repeat the calibration procedure using three (or more) other calibration samples.

Store the calibration curve under the code number chosen in step 8.1.3.

8.1.10 Complete the oil-content calibration curve by using an empty tube and by repeating steps 8.1.5 to 8.1.7. This part of the method is not applicable to the water content calibration curve.

8.2 Calibration procedure for the determination of water content

8.2.1 Procedure A: Three calibration samples with known water contents

To obtain a reliable calibration graph, it is necessary that the water contents of the calibration samples span the largest possible range. However, a zero water content is not permissible.

Follow the general procedure specified in 8.1.2 to 8.1.9.

8.2.2 Procedure B: One calibration sample with known water content

NOTE In this calibration procedure, the quantity of water detected by the NMR spectrometer is varied by varying the height to which the sample tubes are filled with the calibration sample.

8.2.2.1 Follow the general procedure specified in 8.1.2 to 8.1.4.

8.2.2.2 Introduce into three tarred sample tubes (5.2) portions of the calibration sample with known water content to three different heights, in accordance with the manufacturer's instructions, and weigh them. Note the corresponding masses in grams as m_1, m_2 and m_3.

The minimum number of calibration points for establishing the calibration curve is three.

8.2.2.3 Calculate the relative water content, w, as a percentage by mass, of each of the three portions of the calibration sample as follows:

$$w_1 = \frac{m_1 \times W}{m_1}$$

$$w_2 = \frac{m_2 \times W}{m_1}$$

$$w_3 = \frac{m_3 \times W}{m_1}$$

where

m_1, m_2, m_3	are the masses, in grams, of the three portions of the calibration sample in each of the three tubes, where m_1 is the largest mass;
W	is the water content, as a percentage by mass, of the calibration sample, determined in accordance with ISO 771.

8.2.2.4 Enter into the NMR spectrometer the value, m_1, of the mass of the first portion.

8.2.2.5 Enter into the NMR spectrometer the value, w_1, of the relative water content of the first portion.

8.2.2.6 Insert the tube containing the first portion of the calibration sample into the measuring head and carry out the determination.

8.2.2.7 Repeat the operations described in 8.2.2.4 to 8.2.2.6 with the two other portions of the calibration sample, always entering m_1 in step 8.2.2.4 and the respective relative water contents w_2 and w_3 in step 8.2.2.5.

8.2.2.8 Calculate the calibration parameters in accordance with step 8.1.9.

8.3 Calibration procedure for the determination of oil content

8.3.1 Procedure A: Three calibration samples with known oil contents

Use the samples specified in 4.3.

Follow the general procedure specified in 8.1.2 to 8.1.10.

8.3.2 Procedure B: One calibration sample with known oil content

NOTE In this calibration procedure, the quantity of oil detected by the NMR spectrometer is varied by varying the height to which the sample tubes are filled with the calibration sample.

8.3.2.1 Follow the general procedure specified in 8.1.2.

8.3.2.2 Introduce into three tarred sample tubes (5.2) the calibration sample with known oil content to three different heights in accordance with the manufacturer's instructions and weigh. Note the corresponding masses in grams as m_1, m_2 and m_3.

8.3.2.3 Calculate the relative oil content, h, as a percentage by mass, in the three portions of the calibration sample as follows:

$$h_1 = \frac{m_1 \times H}{m_1}$$

$$h_2 = \frac{m_2 \times H}{m_1}$$

$$h_3 = \frac{m_3 \times H}{m_1}$$

where

m_1, m_2, m_3 are the masses, in grams, of the three portions of the calibration sample ;

H is the oil content, as a percentage by mass, of the calibration sample, determined in accordance with ISO 734, part 1 or 2.

8.3.2.4 Follow the general procedure specified in 8.1.3 to 8.1.9, entering the value of the oil content, in grams, in step 8.1.6.

8.3.2.5 Enter into the NMR spectrometer the value, m_1, of the mass of the first portion.

8.3.2.6 Enter into the NMR spectrometer the value, h_1, of the relative oil content of the first portion.

8.3.2.7 Insert the tube containing the first portion of the calibration sample into the measuring head and carry out the determination.

8.3.2.8 Repeat the operations described in 8.3.2.5 to 8.3.2.7 with the two other portions of the calibration sample, always entering m_1 in step 8.3.2.5, then the respective relative oil contents h_2 and h_3 in step 8.3.2.6.

8.3.2.9 To obtain the zero-point on the calibration curve, enter into the NMR spectrometer the value, m_1, of the mass, and a relative oil content of 0,01 %. Insert an empty tube into the measuring head and carry out the determination.

8.3.2.10 Calculate the calibration parameters in accordance with 8.1.9.

9 Procedure

9.1 General

Carry out at least two determinations on test portions taken from the same sample.

9.2 Requirements for the test room and conditioning of the test sample

The temperature of the test room shall be maintained constant between 17 °C and 28 °C. Therefore, it is recommended that the test room be temperature controlled.

Transfer the test samples (clause 7) to the test room at least 60 min before the determination to allow them to reach equilibrium temperature.

9.3 Test portions

NOTE The precision of the method is improved if the NMR measurements are carried out on test portions of constant volume.

Prepare test portions of constant volume using one of the following methods:

a) measure a chosen volume of the test sample (9.2) and then transfer quantitatively this volume to a tared sample tube (5.2) (e.g. use 40 ml of test sample for sample tubes 40 mm in diameter);

b) transfer sufficient quantity of the conditioned test sample (9.2) to the sample tube to fill it to an optimum height of 30 mm ± 5 mm; it is recommended that the sample for sample tubes be marked at a height of 30 mm using a felt-tipped pen.

9.4 Determination

IMPORTANT — Check the calibration prior to each series of measurements (or at least once a day), using a calibration sample of known oil and water contents.

9.4.1 Select the programme and the instrument parameters for the water or the oil content, as applicable, and the number on the calibration curve corresponding to the intended test and to the species oilseed residues

9.4.2 Set the apparatus to the measurement mode.

9.4.3 Weigh the test portion (9.3) and transfer the value of the mass from the balance to the NMR spectrometer.

9.4.4 Insert the sample tube containing the test portion into the measuring head and determine the water and oil contents.

10 Expression of results

Take as the result the arithmetic mean of the results of the two or more determinations.

11 Precision

Details of an interlaboratory test on the method are summarized in annex A.

In accordance with ISO 5725-1 and ISO 5725-2, repeatability and reproducibility values have not been calculated, because the number of participating laboratories in the ring test was insufficient.

12 Test report

The test report shall specify:

- all information necessary for the complete identification of the sample;
- the sampling method used, if known;
- the NMR spectrometer used;
- the test method used, with reference to this International Standard;
- all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s);
- the test result(s) obtained, or if the repeatability has been checked, the final quoted result obtained.

Annex A
(informative)

Results of interlaboratory test

An interlaboratory test was carried out at the international level in 1996 and gave the results shown in Table A.1.

Table A.1 — Determination of water and oil contents

Sample	Laboratory No.	Water content, %		Oil content, %	
			Mean value		Mean value
Rapeseed	1				
	2	6,0 6,2	6,1	19,3 19,7	19,5
	3	5,4 5,5	5,5	19,8 20,3	20,1
	4				19,3
Sunflower (extraction)	1				
	2	8,2 8,3	8,3	0,9 0,9	0,9
	3	8,0 8,2	8,1	1,6 2,0	1,8
	4				1,0
Sunflower (pressure)	1				
	2	6,7 6,9	6,8	19,4 19,5	19,4
	3	6,4 6,5	6,5	18,4 18,7	18,6
	4				18,7
Soya	1	6,03 5,80	5,92	1,05 1,03	1,04
	2	7,9 8	8	2,1 2,1	2,1
	3	8,3 8,1	8,2	0,14 0,22	0,18
	4				1,9

Bibliography

[1] ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions.*

[2] ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.*

[3] ISO 5500*, Oilseed residues — Sampling.*

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