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Oilseeds — Determination of oil content — Method using continuous-wave low-resolution nuclear magnetic resonance spectrometry (Rapid method)

Graines oléagineuses — Détermination de la teneur en huile — Méthode par spectrométrie de résonance magnétique nucléaire à basse résolution et à onde continue (Méthode rapide)

Reference number
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Foreword

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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.

International Standard ISO 5511 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Sub-Committee SC 2, *Oleaginous seeds and fruits*.

This second edition cancels and replaces the first edition (ISO 5511:1984), of which it constitutes a technical revision.

Annex A of this International Standard is for information only.

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INTERNATIONAL STANDARD ISO 5511:1992(E)

Oilseeds — Determination of oil content — Method using continuous-wave low-resolution nuclear magnetic resonance spectrometry (Rapid method)

1 Scope

This International Standard specifies a rapid method for the determination of the oil content of oilseeds using *continuous-wave low-resolution* nuclear magnetic resonance spectrometry.

Under normal conditions of use, it does not apply to oilseeds which do not yield oil which is completely liquid at 20 °C (e.g. shea, palm, illipe, cocoa, etc.).

This method has been successfully tested on the following oilseeds: rapeseed, soya, sunflower seed and groundnuts.

NOTE 1 The reference method for the determination of the oil content of oilseeds is specified in ISO 659.

2 Normative references

The *following standards contain* provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 659:1988, *Oilseeds — Determination of hexane extract (or light petroleum extract), called "oil content".*

ISO 664:1990, *Oilseeds — Reduction of laboratory sample to test sample.*

ISO 665:1977, *Oilseeds — Determination of moisture and volatile matter content.*

ISO 771:1977, *Oilseed residues — Determination of moisture and volatile matter content.*

3 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1 oil content: The mass fraction of *organic* substances, which are liquid at the temperature of measurement (in principle 20 °C) of the oilseeds, determined using the method specified in this International Standard.

The oil content is expressed as a percentage by mass.

3.2 single test result: Result obtained by carrying out a specified test method one time according to the prescribed procedure.

3.3 repeatability conditions: Conditions where mutally independent test results are obtained with the same method on identical test material in the *same laboratory* by the same operator using the same equipment within short intervals of time. [ISO 5725:1986, 3.1.7]

4 Principle

Determination using continuous-wave low-resolution nuclear magnetic resonance (NMR) spectrometry of the content of liquid components containing hydrogen which are present in oilseeds which have been previously dried at 103 °C ± 2 °C, and taking into account the effect of solids (oilseed residue).

5 Materials

5.1 Calibration oil, crude oil from seeds of the same botanical species and of similar geographical origin and chemical composition to those of the seeds for analysis, extracted in the laboratory carrying out the analysis, in accordance with the

method specified in ISO 659, less than 1 month previously.

Store the oil under dry conditions preventing oxidation.

5.2 Oilseed residue, from seeds of the same botanical species, and of similar geographical origin and chemical composition, to those of the seeds for analysis, from which the oil has been extracted in the laboratory carrying out the analysis, in accordance with the method specified in ISO 659, less than 1 month previously.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

6.1 Apparatus required for the drying method (see ISO 665).

6.2 Continuous-wave low-resolution NMR spectrometer

6.3 Measuring tubes, preferably with closures, suitable for use with the NMR spectrometer (6.2) and of the largest possible capacity, made from a non-conducting non-magnetic material which does not contain hydrogen [e.g. glass or polytetrafluoroethylene (PTFE)].

6.4 Desiccator, containing an efficient desiccant.

7 Sampling

It is important that the sample received by the laboratory be truly representative and not damaged or changed during transport and storage. Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 542.

8 Preparation of the test sample

Prepare the test sample in accordance with ISO 664. Then remove any particles of ferrous metals using a magnet.

NOTE 2 Particles of ferrous metals will falsify the results.

9 Procedure

IMPORTANT — Since the effect of temperature variation on the results obtained is very large (the oil response decreases by approximately 0,3 % per degree Celsius), all operations for calibrating the instrument and all measuring operations shall be carried out strictly at the same temperature. It is recommended, if the necessary apparatus is available, to control thermostatically the "effective" part of the instrument and to place the seeds, before measurement, in insulated metal blocks at the measuring temperature. Otherwise, carry out a simplified calibration of the apparatus every 30 min (see 9.2.5).

9.1 Check on repeatability (if required)

If it is required to establish whether the repeatability requirement (see clause 11) is met, carry out two single determinations, each including the two intermediate determinations (9.6), under repeatability conditions (see 3.3).

9.2 Calibration of the instrument

9.2.1 Adjust the NMR spectrometer (6.2) in accordance with the manufacturer's instructions, then adjust to zero after placing an empty measuring tube (6.3) in the magnetic field.

9.2.2 Into four measuring tubes identical to that used for the adjustment (i.e. also giving a reading of zero when they are placed empty in the magnetic field), weigh, to the nearest 0,01 g, 5 g, 10 g, 20 g, and, if the apparatus so permits, 30 g of the calibration oil (5.1), taking care that no oil adheres to the sides of the tubes above the effective part.

9.2.3 Measure the response of the instrument for each of the four tubes, taking $\overline{R}_5$, $\overline{R}_{10}$, $\overline{R}_{20}$ and $\overline{R}_{30}$ as the mean values of five readings of the integrator on each of the four tubes, during the integration time giving the best repeatability of measurement, using a level of radiofrequency energy sufficient to obtain a satisfactory signal in relation to the background noise, but not producing more than approximately 1 % saturation. The correct selection of the level of saturation may be carried out by the operator if the relation between the radiofrequency energy and the saturation is known. If not, the correct adjustment of the level of saturation shall be obtained from the manufacturer of the instrument.

9.2.4 Draw a straight line representing the values of $\overline{R}_x$ as a function of the mass of oil placed in the tubes. This line shall pass through the origin. If it does not, contact the manufacturer to adjust the instrument or make the necessary adjustment if the instrument so permits.

9.2.5 Check the calibration of the instrument regularly (if possible, once each day). If the instrument and the samples are not thermostatically controlled, check the variation in the gradient of the calibration line by measuring the response of a 20 g or 30 g sample every 30 min.

9.3 Test portion

Weigh, to the nearest 0,01 g, a sufficient quantity of the prepared test sample (clause 8), but at least 20 g, to fill the effective part of a measuring tube (6.3).

NOTE 3 The effective part corresponds to the tube height included in the magnet airgap (approximately 20 g of seeds) but some instruments cannot accommodate 20 g of some species of seed. If less than 20 g of seed is used, the test portion is less representative and, in this case, several successive measurements should be taken.

9.4 Drying

Dry the test portion and determine its moisture content in accordance with the method specified in ISO 665.

Place the desiccator (6.4) containing the dried seeds in immediate proximity to the NMR spectrometer (6.2) in a room in which the temperature (generally about 20 °C) does not show sudden variations and leave for at least 3 h before carrying out the determination so that the seed and the apparatus are at the same temperature.

NOTE 4 If possible, use a temperature-controlled room.

9.5 Measurement

Quantitatively and rapidly transfer the dry seeds into a measuring tube (6.3) identical to that used for the calibration. If possible, close the tube to prevent absorption of moisture.

Without altering the adjustment of the instrument, measure the response for the tube. Let $\overline{R}$ be the mean value of this response for five readings of the integrator using the integration time chosen previously.

9.6 Correction for oilseed residue

9.6.1 Into a measuring tube (6.3) identical to that used for the calibration, weigh, to the nearest 0,01 g, a sufficient quantity (mass m_r) of dried oilseed residue from which the oil has been extracted (5.2) to fill the effective part of the tube. Keep it at room temperature.

NOTE 5 Just before use, dry the residue at 103 °C ± 2 °C in accordance with the method specified in ISO 771, and cool in the desiccator (6.4).

9.6.2 Without altering the adjustment of the instrument, measure the response for the tube and take as the result the mean value, $\overline{R}_r$, of this response for five readings of the integrator using the integration time chosen previously.

9.6.3 Repeat successively the operations described in 9.6.1 and 9.6.2 a further five to ten times, using the same mass m_r of the dried oilseed residue (9.6.1). Take $\overline{T}$ as the mean value of the mean responses obtained. This value needs to be determined only about once per month.

NOTE 6 As a guide, rapeseed data have shown that, without correction, this method gives higher results than the reference method specified in ISO 659 by approximately, on average, 0,30 % (*m/m*).

9.7 Number of determinations

Carry out a minimum of two determinations on test portions taken from the same test sample, particularly if the sample is heterogeneous.

10 Expression of results

10.1 Calculate the apparent oil content of the seeds using the formula

$$w_a = \frac{\overline{R} \times m_x \times 100\ \%}{\overline{R}_x \times m_0}$$

where

w_a is the apparent oil content, expressed as a percentage by mass, of the seeds;

m_0 is the mass, in grams, of the test portion before drying (9.3);

m_x is the mass, in grams, of crude oil corresponding to the response $\overline{R}_x$, read from the calibration line of the instrument (9.2);

$\overline{R}$ is the mean value of the responses of the instrument for the test portion, determined in 9.5;

$\overline{R}_x$ is the response of the instrument corresponding to the mass m_x of crude oil, read from the calibration line of the instrument (9.2).

10.2 Calculate the response factor of the residue using the formula

$$w_r = \frac{\overline{T} \times m_x \times 100\ \%}{\overline{R}_x \times m_r}$$

where

w_r is the response factor, expressed as a percentage by mass, of the residue;

m_r is the mass, in grams, of the dried oilseed residue from which the oil has been extracted, used in 9.6;

$\overline{T}$ is the mean value of the responses of the instrument for the mass m_t of dried oilseed residue from which the oil has been extracted, determined in 9.6;

m_x and $\overline{R}_x$ have the same meanings as in 10.1.

10.3 Calculate the oil content of the seeds using the formula

$$w = \frac{(w_a - w_r)[1 - (w_H/100)]\ \%}{1 - (w_r/100)}$$

where

w is the oil content of the seeds, expressed as a percentage by mass;

w_H is the moisture content of the seeds, expressed as a percentage by mass, determined in accordance with ISO 665;

w_a and w_r have the same meanings as in 10.1 and 10.2 respectively.

Take as the result the arithmetic mean of the results of the two determinations (9.7), provided that their difference does not exceed 0,4 % (m/m). If their difference does exceed 0,4 % (m/m), discard the results of the two determinations and repeat the procedure (clause 9).

Express the result to the nearest 0,1 % (m/m).

NOTE 7 For a given type of seed, the response of the residue is generally independent of the samples. For example, in the case of rapeseeds (see note 6 in 9.6), the oil content of the seeds is approximately equal to

$$w = w_a - 0{,}30\ \%\ (m/m)$$

11 Repeatability

The difference between two single test results (see 3.2) obtained under repeatability conditions (see 3.3) shall not exceed 0,6 % (m/m).

Reject both results if their absolute difference exceeds 0,6 % (m/m) and carry out two new single determinations (each time according to clauses 9 and 10).

12 Test report

The test report shall specify the method used, the results obtained, the name and type of NMR spectrometer used and, if applicable, all adjustments made. It shall also mention all operating details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the results.

The test report shall include all information necessary for the complete identification of the sample.

Annex A
(informative)

Bibliography

[1] ISO 542:1990, *Oilseeds — Sampling.*

[2] ISO 5725:1986, *Precision of test methods — Determination of repeatability and reproducibility for a standard test method by inter-laboratory tests.*

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