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Oilseeds — Simultaneous determination of oil and water contents — Method using pulsed nuclear magnetic resonance spectrometry

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

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Foreword

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This document was prepared by Technical Committee ISO/TC 34, *Food products,* Subcommittee SC 2, *Oleaginous seeds and fruits and oilseed meals*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 307, *Oilseeds, vegetable and animal fats and oils and their by-products - Methods of sampling and analysis*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This third edition cancels and replaces the second edition (ISO 10565:1998), which has been technically revised.

The main changes are as follows:

— the scope has been clarified;

— additional information has been added to the protocol;

— the results of a test to compare different equipment have been added.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Oilseeds — Simultaneous determination of oil and water contents — Method using pulsed nuclear magnetic resonance spectrometry

1 Scope

This document specifies a rapid method for the determination of the oil and water contents in oilseeds using pulsed nuclear magnetic resonance (NMR).

It is applicable to rapeseeds, linseeds and sunflower seeds with a water content less than 10 % and soya beans with a water content less than 14 %. For seeds with higher water contents, drying is necessary before the water and oil content can be determined by pulsed NMR.

NOTE 1 This method has been tested with rapeseeds, soya beans, linseeds and sunflower seeds. This does not, however, preclude its applicability to other commercial seeds whose oil is liquid at the temperature of measurement.

NOTE 2 The reproducibility values obtained are generally higher than those obtained by the reference methods (see ISO 659 and ISO 665) because they depend on the variability between the instruments and on that existing between the calibrations, which depends on the measurement accuracy of the reference methods.

NOTE 3 The results of the comparison test between different magnets and different volumes of seed samples (see Annex B) are in coherence with the results of the interlaboratory test (see Annex A).

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 659, *Oilseeds — Determination of oil content (Reference method)*

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

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

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Principle

Insertion of the test sample into the magnetic field of a pulsed NMR spectrometer.

Application of an alternating electromagnetic field in the form of an intense 90° radiofrequency (RF) pulse which excites all the hydrogen nuclei. Recording of the free induction decay (FID) 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.

Application of the second RF pulse, a so-called “180° pulse”, 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.

Calculation of the difference between the two amplitudes, which is proportional to the water content.

Automatic conversion of the measured signals, after suitable calibration of the apparatus, 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 a specific program.

5 Samples

5.1 General

Transfer the calibration and test samples to the test room at least 60 min before the determination to allow them to reach equilibrium temperature.

For rapeseeds, linseeds and sunflower seeds with a water content greater than a mass fraction of 10 % and soybeans with a water content greater than a mass fraction of 14 %, refer to 10.4.2. In this case, the NMR spin-echo method does not give a correct value because the excess water (free water) contributes, together with the oil, to the spin-echo signal. This excess water shall therefore be removed by drying to a moisture level of below a mass fraction of 10 % for all seeds.

5.2 Calibration samples

5.2.1 General

Samples of oilseeds shall be of the same species as the test samples and of similar fatty acid compositions (e.g. for the analysis of rapeseeds which are rich in erucic acid or sunflower seeds which are rich in oleic acid).

Calibration samples shall be homogeneous and free from impurities. A definition of impurities is given in ISO 658[1].

5.2.2 Samples for moisture-content calibration

The water content of seeds can vary depending on storage conditions. Water content shall therefore be determined in accordance with ISO 665 just prior to calibration.

5.2.3 Samples for oil-content calibration

Oil content shall be determined using the reference method specified in ISO 659.

6 Apparatus

Usual laboratory apparatus and, in particular, the following shall be used.

6.1 Pulsed low-resolution NMR spectrometer, suitable for measurement of the oil content and water content of oilseeds, and meeting the precision requirements of 12.2 and 12.3.

The instrument’s parameters shall be in accordance with the instructions/specifications from the manufacturer.

For all calibration and measurement operations, follow the user’s manual.

The temperature of the test room shall be maintained in accordance with the instructions of the manufacturer. Ensure that all operations during sample preconditioning before measurement (see 5.1), calibration and measurement are carried out under the same conditions and, in particular, at the same temperature (±2 °C). Therefore, the test room should be temperature controlled.

NOTE Usual range for the test room temperature is between 17 °C and 28 °C.

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

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

Follow the manufacturers' instructions for suitable sample tube filling heights and the ranges of mass variation recommended for different seeds.

For a filling height of 30 mm with tubes of diameter 40 mm (which is equivalent to a volume of 40 ml), the following ranges of masses are recommended:

— whole rapeseeds and linseeds: 22 g to 25 g;

— whole sunflower seeds: 14 g to 17 g;

— whole soya beans: 21 g to 24 g.

For medium seeds (sunflower, soya and safflower), the tubes should be filled to at least 40 ml in order to guarantee the representativeness of the sample to be analysed.

6.3 Analytical balance, electronic, capable of weighing to an accuracy of ± 0,01 g.

This equipment may be linked to the NMR spectrometer so that the sample mass is recorded directly by the NMR or linked to a minicomputer (see NOTE 2 in Clause 4).

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

6.5 Dishes, made of glass or metal, of diameter 7 cm to 10 cm, and provided with lids.

7 Sampling

Sampling is not part of the method specified in this document. A recommended sampling method is given in ISO 21294[3].

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

8 Preparation of test sample

Prepare the test sample in accordance with ISO 664.

Remove from the prepared test sample all metallic objects (e.g. staples, needles). Whole seeds shall be homogeneous and, as far as possible, free from impurities.

9 Calibration procedure

9.1 General

9.1.1 To develop calibration, a minimum of three calibration samples shall be used, although more than three samples should be used. These samples can either be from three or more independent calibration samples, each with a different and known water and oil content (procedure A) or from a single calibration