SLOVENSKI STANDARD
SIST EN ISO 10565:1998

01-november-1998

Oljna semena - Simultano določevanje olja in vode - Spektrometrijska metoda z uporabo pulzne jedrske magnetne resonance (ISO 10565:1998)

Oilseeds - Simultaneous determination of oil and water contents - Method using pulsed nuclear magnetic resonance spectrometry (ISO 10565:1998)

Ölsamen - Gleichzeitige Bestimmung des Öl- und Wassergehaltes - Verfahren mit gepulster Kernresonanzspektroskopie (ISO 10565:1998)

Graines oléagineuses - Détermination simultanée de la teneur en huile et en eau - Méthode par spectrométrie par résonance magnétique nucléaire pulsée (ISO 10565:1998)

Ta slovenski standard je istoveten z: **EN ISO 10565:1998**

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67.200.20 Oljnice Oilseeds

SIST EN ISO 10565:1998 **en**

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English version

Oilseeds - Simultaneous determination of oil and water contents - Method using pulsed nuclear magnetic resonance spectrometry (ISO 10565:1998)

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Ölsamen - Gleichzeitige Bestimmung des Öl- und Wassergehaltes - Verfahren mit gepulster Kernresonanzspektroskopie (ISO 10565:1998)

This European Standard was approved by CEN on 15 August 1998.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

Foreword

The text of the International Standard ISO 10565:1998 has been prepared by Technical Committee ISO/TC 34 "Agricultural food products" in collaboration with Technical Committee CEN/TC 307 "Oilseeds, vegetable and animal fats and oils and their by-products - Methods of sampling and analysis", the secretariat of which is held by AFNOR.

This European Standard supersedes EN ISO 10565:1997.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by February 1999, and conflicting national standards shall be withdrawn at the latest by February 1999.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 10565:1998 was approved by CEN as a European Standard without any modification.

NOTE: Normative references to International Standards are listed in annex ZA (normative).

Annex ZA (normative)
Normative references to international publications with their relevant European publications

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

Publication	Year	Title	EN	Year
ISO 659	1998	Oilseeds - Determination of oil content (Reference method)	EN ISO 659	1998
ISO 664	1990	Oilseeds - Reduction of laboratory sample to test sample	EN ISO 664	1995
ISO 665	1977	Oilseeds – Determination of moisture and volatile matter content	EN ISO 665	1995

INTERNATIONAL STANDARD

ISO 10565

Second edition
1998-08-15

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 par résonance magnétique nucléaire pulsée

Reference number
ISO 10565:1998(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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 10565 was prepared by Technical Committee ISO/TC 34, *Agricultural food products,* Subcommittee SC 2, *Oleaginous seeds and fruits.*

This second edition cancels and replaces the first edition (ISO 10565:1993), which has been technically revised.

Annexes A and B of this International Standard are for information only.

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet iso@iso.ch

Printed in Switzerland

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

1 Scope

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

It is applicable to rapeseeds, soya beans, linseeds and sunflower seeds with a water content less than 10 %. For seeds with higher water contents, drying is necessary before the 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 are generally higher than those obtained by the drying method (ISO 665).

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.*

3 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.

4 Calibration samples

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

4.1 Samples for moisture-content calibration

In order to obtain a reliable calibration curve, it is recommended that the water contents of the calibration samples be less than 10 % for all seeds.

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.

4.2 Samples for oil-content calibration

Samples of oilseeds shall be of the same species as the test samples and of similar fatty acid compositions (for instance for the analysis of rapeseeds which are rich in erucic acid, or sunflower seeds which are rich in oleic acid). Oil content shall be determined using the reference method specified in ISO 659.

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, and meeting the precision requirements of 11.2 and 11.3.

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

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.

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 3).

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 to 10 cm, and provided with lids.

5.6 Desiccator, containing an efficient desiccant.

6 Sampling

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 542 [2].

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

7 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, etc.). Whole seeds shall be homogeneous and, as far as possible, free from impurities.

8 Calibration procedure

8.1 General

8.1.1 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. Ensure 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.

8.1.2 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 16 s.

8.1.3 Set the apparatus to the calibration mode.

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

NOTE A manual feed of the mass ot the calibration sample into the NMR spectrometer is also possible.

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

8.1.6 Introduce the sample tube containing the first calibration sample into the measuring head. Record automatically or manually the water or oil values thus obtained.

8.1.7 Repeat steps 8.1.4 to 8.1.6 for the two (or more) other calibration samples.

8.1.8 Calculate automatically or manually the calibration parameters of the calibration curve using the results obtained in 8.1.6 and 8.1.7.

The correlation coefficient shall normally be greater than 0,95. If this is not the case, check the values obtained using the reference methods specified in ISO 659 and ISO 665 respectively, or repeat the calibration procedure using three (or more) other calibration samples.