BRITISH STANDARD

BS EN ISO
10565:1998
BS 4289-11:1998

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

The European Standard EN ISO 10565:1998 has the status of a British Standard

ICS 67.200.20

National foreword

This British Standard is the English language version of EN ISO 10565:1998. It is identical with ISO 10565:1998. It supersedes BS EN ISO 10565:1997 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee AW/2, Oilseeds and residues, which has the responsibility to:

- — aid enquirers to understand the text;
- — present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;
- — monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this committee can be obtained on request to its secretary.

Cross-references

Attention is drawn to the fact that CEN and CENELEC Standards normally include an annex which lists normative references to international publications with their corresponding European publications. The British Standards which implement these international or European publications may be found in the BSI Standards Catalogue under the section entitled "International Standards Correspondence Index", or by using the "Find" facility of the BSI Standards Electronic Catalogue.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

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Summary of pages

This document comprises a front cover, an inside front cover, the EN ISO title page, the EN ISO foreword page, the ISO title page, page ii, pages 1 to 12, the annex ZA page and a back cover.

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

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.

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Ref. No. EN ISO 10565:1998 E

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).

EN ISO 10565:1998

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

S

Reference number
ISO 10565:1998(E)

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 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.

Descriptors: agricultural products, plant products, oilseeds, tests, determination of content, oils, water, nuclear magnetic resonance method.

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.

Store the calibration curve under the code number chosen in step 8.1.2.

8.1.9 Complete the oil-content calibration curve by using an empty tube and by repeating steps 8.1.4 to 8.1.6. 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 water content of 0 is not permissible.

Follow the general procedure specified in 8.1.1 to 8.1.8.

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 8.1.1 to 8.1.3.

8.2.2.2 Introduce into three tared sample tubes (5.2) portions of the calibration sample with known water content to approximate heights of 35 mm, 30 mm and 25 mm (20 mm minimum) and weigh them. Note the corresponding masses, in grams, as m_1, m_2 and m_3.

NOTE Three calibration points is the minimum for establishing the calibration curve.

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 and 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 665.

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, then 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 8.1.8.

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 specifies in 4.1.

Follow the general procedure specified in 8.1.1 to 8.1.9.

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 8.1.1.

8.3.2.2 Introduce into three tared sample tubes (5.2) portions of the calibration sample with known oil content to approximate heights of 35 mm, 30 mm and 25 mm (20 mm minimum) 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 and 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 659.

8.3.2.4 Follow the general procedure specified in 8.1.2 to 8.1.8, entering the value of the oil content, in grams, in step 8.1.5.

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 8.1.8.

9 Procedure

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

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

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

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

9.2 Test portion

NOTE 1 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 (see 9.1) and then transfer quantitatively this volume to a tared sample tube (see 5.2) (e.g. use 40 ml of test sample for sample tubes 40 mm in diameter).

b) Transfer a sufficient quantity of the conditioned test sample (see 9.1) to the sample tube to fill it to an optimum height ± 5 mm. The optimum height is indicated on tubes by a scored line or it can be manually marked using a felt-tipped pen.

If carrying out measurements on dehulled sunflower seeds, ensure that they are well distributed in the sample tubes.

NOTE 2 Follow the manufacturers' instructions for suitable sample tube filling heights and the ranges of mass variation recommended for different seeds.

NOTE 3 For a filling height of 30 mm, the following ranges of masses are recommended:

whole rapeseeds	22 g to 25 g
whole sunflower seeds	14 g to 17 g
whole soya beans	21 g to 24 g

9.3 Determination

WARNING — 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.

9.3.1 General case

9.3.1.1 Select the program and the instrument parameters for the water or oil content, as applicable, and the number of the calibration curve corresponding to the intended test and to the species of seeds.

9.3.1.2 Set the apparatus to the measurement mode.

9.3.1.3 Weigh the test portion (see 9.2) and transfer the value of the mass from the balance to the NMR spectrometer.

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

9.3.2 Rapeseeds and sunflower seeds having a water content greater than 10 % (*m/m*) and soya beans having a water content greater than 14 % (*m/m*)

In this case, the pulsed NMR and the spin-echo method do not give a correct value because the excess water has a different behaviour and contributes, together with the oil, to the spin-echo signal. This excess water shall therefore be removed by drying to below 10 %.

9.3.2.1 Transfer quantitatively the previously weighed test portion of mass m into dishes (5.5), with the lids removed, and dry for 1 h in the oven (5.4) set at 103 °C.

9.3.2.2 Remove the dishes from the oven, cover them with a lid and allow them to cool in the desiccator (5.6). Check that they have reached the measuring temperature (after approximately 1 h).

9.3.2.3 Transfer quantitatively the dried test portion to a previously weighed sample tube. Weigh and deduce the mass, m_0, of the dried test portion.

9.3.2.4 Follow the procedure specified in 9.3.1.1 and 9.3.1.2.

9.3.2.5 Enter the value of the original mass, m, of the test portion into the NMR spectrometer.

9.3.2.6 Insert the sample tube containing the test portion into the measuring head and determine the water content, W_b.

9.3.2.7 Correct the reading obtained for the water content, W_b, to give the total water content W, as a percentage by mass, using the following formula:

$$W = W_b + \frac{m - m_0}{m} \times 100$$

where

m is the original mass, in grams, of the test portion, determined in 9.3.2.1;

m_0 is the mass, in grams, of the dried test portion, determinded in 9.3.2.3;

W_b is the water content, expressed as a percentage by mass, measured in 9.3.2.6.

10 Expression of results

Check whether the repeatability requirement (see 11.2) is satisfied.

If so, take as the result the arithmetic mean of the results of the two or more determinations. If not, reject the results and carry out two further determinations.

11 Precision

11.1 Interlaboratory tests

Details of interlaboratory tests on the precision of the method are summarized in annex A. The values derived from these interlaboratory tests may not be applicable to concentation ranges and matrices other than those given.

11.2 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in not more than 5 % of cases be greater than the following values:

Oil content	**Water content**
0,6 % (*m*/*m*) for rapeseed	0,2 % (*m*/*m*) for rapeseed
0,8 % (*m*/*m*) for sunflower seed	0,2 % (*m*/*m*) for sunflower seed
0,6 % (*m*/*m*) for soya bean	0,3 % (*m*/*m*) for soya bean
0,4 % (*m*/*m*) for linseed	0,2 % (*m*/*m*) for linseed

11.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, will in not more than 5 % of cases be greater than the following values:

Oil content	**Water content**
1,5 % (*m*/*m*) for rapeseed	0,6 % (*m*/*m*) for rapeseed
1,5 % (*m*/*m*) for sunflower seed	0,5 % (*m*/*m*) for sunflower seed
0,8 % (*m*/*m*) for soya bean	1,8 % (*m*/*m*) for soya bean
0,8 % (*m*/*m*) for linseed	0,4 % (*m*/*m*) for linseed

12 Test report

The test report shall specify:

- all information necessary for the complete identification of the sample;
- the NMR spectrometer used;
- the sampling method used, if known;
- 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;
- if the repeatability has been checked, the final quoted result obtained.

Annex A
(informative)

Results of interlaboratory tests

Two interlaboratory tests were carried out at the international level, the first test on oil content (see table A.1) and the second on water content (see table A.2).

For the interlaboratory tests, the NMR signal on the FID was recorded 50 µs after the 90° pulse. The 180° pulse was set 3 500 µs after the 90° pulse, and the oil signal was recorded 7 000 µs after the 90° pulse. The time between two 90° pulses was 1 s and the total measuring time was 16 s.

The results of the tests were obtained by the exclusive use of BRUKER "Minispec — 10 MHz" apparatus.

The statistical results were determined in accordance with ISO 5725 [3]1).

Table A.1 — Interlaboratory test on oil content

Parameters	**Sample**						
	Rapeseed 1	**Rapeseed 2**	**Sunflower seed**	**Soya bean 1**	**Soya bean 2**	**Linseed 1**	**Linseed 2**
Number of laboratories participating	13	13	12	12	12	12	12
Number of laboratories retained after eliminating outliers	13	13	11	12	12	10	9
Mean value, % (*m/m*)	40,83	41,38	47,89	20,00	19,23	38,03	42,00
Repeatability standard deviation, s_r, % (*m/m*)	0,25	0,15	0,33	0,09	0,21	0,15	0,09
Coefficient of variation of repeatability, %	0,63	0,38	0,70	0,42	1,06	0,38	0,22
Repeatability limit, r (2,83 s_r), % (*m/m*)	0,72	0,45	0,94	0,24	0,58	0,41	0,27
Reproducibility standard deviation, s_R, % (*m/m*)	0,55	0,55	0,40	0,29	0,27	0,20	0,26
Coefficient of variation of reproducibility, %	1,35	1,33	0,84	1,45	1,42	0,53	0,63
Reproducibility limit, R (2,83 s_R),% (*m/m*)	1,56	1,56	1,13	0,82	0,77	0,57	0,74

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1) ISO 5725:1986 (now withdrawn) was used to obtain the precision data.

Table A.2 — Interlaboratory test on water content

Parameters	Sample						
	Rapeseed 1	Rapeseed 2	Sunflower seed	Soya bean 1	Soya bean 2	Linseed 1	Linseed 2
Number of laboratories participating	13	13	12	12	12	12	12
Number of laboratories retained after eliminating outliers	12	12	12	12	12	12	10
Mean value, % (*m/m*)	7,01	6,01	4,92	9,90	8,96	5,68	5,68
Repeatability standard deviation, s_r % (*m/m*)	0,04	0,03	0,08	0,09	0,14	0,04	0,06
Coefficient of variation of repeatability, %	0,60	0,58	1,54	0,94	1,55	0,74	1,11
Repeatability limit, r (2,83 s_r), % (*m/m*)	0,11	0,09	0,21	0,26	0,39	0,12	0,18
Reproducibility standard deviation, s_R, % (*m/m*)	0,24	0,36	0,16	0,58	0,79	0,17	0,09
Coefficient of variation of reproducibility, %	3,41	6,01	3,31	5,83	8,79	2,98	1,56
Reproducibility limit, R (2,83 s_R), % (*m/m*)	0,67	1,02	0,46	1,63	2,23	0,48	0,25

Annex B
(informative)

Bibliography

[1] ISO 658:1988, *Oilseeds — Determination of impurities content (under revision).*

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

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

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

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

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