INTERNATIONAL STANDARD

ISO 665

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Oilseeds — Determination of moisture and volatile matter content

Graines oléagineuses — Détermination de la teneur en eau et en matières volatiles

Reference number
ISO 665:2000(E)

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

This second edition cancels and replaces the first edition (ISO 665:1977), which has been technically revised.

Annex A of this International Standard is for information only.

This corrected version of ISO 665 :2000 incorporates a correction in the Foreword of the English version. The year of publication of the first edition was given as 1997 instead of 1977.

Oilseeds — Determination of moisture and volatile matter content

1 Scope

This International Standard specifies a method for the determination of the moisture and volatile matter content of oilseeds.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of this publication do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent edition of the normative document 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 664:1990, *Oilseeds — Reduction of laboratory sample to test sample.*

3 Term and definition

For the purposes of this International Standard, the following term and definition applies.

3.1
moisture and volatile matter content
loss in mass measured under the operating conditions specified in this International Standard

NOTE It is expressed as a mass fraction, in percent [formerly given as % (m/m)] of the mass of the initial sample.

4 Principle

The moisture and volatile matter content of a test portion is determined, either on the material as received (pure seed and impurities) or, if required, on the pure seed alone, by drying at 103 °C ± 2 °C in an oven at atmospheric pressure, until practically constant mass is reached.

5 Apparatus

Usual laboratory apparatus and, in particular, the following.

5.1 Analytical balance, capable of weighing to the nearest 0,001 g.

5.2 Mechanical mill, easy to clean, suitable for the kind of seed and allowing the latter to be ground without heating and without appreciable change in moisture, volatile matter and oil content.

5.3 Mechanical grater or, if not available, a hand-operated grater.

5.4 Flat-bottomed vessel, either of metal or glass, at the discretion of the analyst.

If metal is used, it shall be resistant to attack under the test conditions. The vessel shall be provided with a well-fitting lid and shall allow the test portion to be spread to about 0,2 g/cm^2 (e.g. a vessel of diameter 70 mm and height 30 mm to 40 mm). Glass vessels with ground closures may also be used.

5.5 Electric oven, with thermostatic control and good natural ventilation, capable of being regulated so that the temperature of the air and of the shelves in the neighbourhood of the test portions lies between 101 °C and 105 °C in normal operation.

5.6 Desiccator, containing an efficient desiccant such as phosphorus(V) oxide, silica gel, activated alumina, etc., and provided with a ceramic plate which allows vessels (5.4) to cool rapidly.

6 Sampling

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 542 [1].

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

7 Preparation of test sample

7.1 Prepare the test sample by reducing the laboratory sample in accordance with ISO 664. If large non-oleaginous foreign bodies have been separated before reduction of the laboratory sample, make allowance for this in the calculation (see 9.2). According to the requirements of the contract, take a sample as received or after separation of the impurities.

7.2 In the case of copra, grate the product by hand or, preferably, use a mechanical grater (5.3) which allows the whole sample to be treated. When grating by hand, which does not allow all the analysis sample to be grated, endeavour to obtain a test sample which is as representative as possible and, to this end, take account of the size and colour of different fragments.

The length of the particles after grating may exceed 2 mm, but shall not be greater than 5 mm. Mix the particles carefully and carry out the determination without delay.

7.3 In the case of seeds of medium size (groundnut, etc.), except for safflower seed, sunflower seed, soya beans and cottonseed with adherent linters, grind the test sample in the mechanical mill (5.2), which has previously been well cleaned, until the major dimension of the particles obtained is not greater than 2 mm. Reject the first particles (about one-twentieth of the sample). Collect the rest, mix carefully and carry out the determination without delay.

7.4 Small seeds (linseed, colza, hemp, etc.) as well as safflower seed, sunflower seed, soya beans and cottonseed with adherent linters, are analysed without previous grinding.

8 Procedure

8.1 Test portion

8.1.1 Dry the vessel with its lid for 1 h at 103 °C prior to being placed in the desiccator before weighing. Weigh the vessel (see 5.4) with its lid to the nearest 0,001 g, after leaving it open for at least 30 min in the desiccator (see 5.6) at laboratory temperature.

8.1.2 Then weigh into the vessel, to the nearest 0,001 g,

— either 5 g ± 0,5 g of the grated product (see 7.2) in the case of copra, or meal (see 7.3), or medium-sized seeds other than safflower seed, sunflower seed, soya beans or cottonseed with adherent linters,

— or 5 g to 10 g of whole seed in the case of safflower seed, sunflower seed, soya beans, cottonseed with adherent linters and small seeds.

Spread the material evenly over the whole base of the vessel and close the vessel with its lid. Weigh the whole to the nearest 0,001 g.

8.1.3 Carry out these operations as quickly as possible, to avoid any appreciable change in moisture content.

8.2 Determination

Place the vessel containing the test portion, with the lid removed, in the oven (see 5.5) set at 103 °C ± 2 °C. Close the oven. After 3 h (12 h to 16 h in the case of cottonseed with adherent linters), reckoned from the time when the temperature returns to 103 °C, open the oven. Immediately close the vessel with its lid and place it in the desiccator. As soon as the vessel has cooled to laboratory temperature, weigh it to the nearest 0,001 g.

Return the vessel, with the lid removed, to the oven. After 1 h, repeat the operations of closing the vessel, allowing it to cool, and weighing it.

If the difference between the two weighings is equal to or less than 0,005 g (for a 5 g test portion), regard the determination as finished. If not, subject the test portion to successive 1 h periods in the oven, until the difference between two successive weighings is equal to or less than 0,005 g.

Never put moist products in the oven together with products that are nearly dry, as this will result in the latter being partially rehydrated.

Carry out two determinations on the same test sample.

9 Expression of results

9.1 The moisture and volatile matter content, w, as a percentage by mass of the sample as received, is equal to:

$$w = \frac{m_1 - m_2}{m_1 - m_0} \times 100\ \%$$

where

m_0 is the mass, in grams, of the vessel;

m_1 is the mass, in grams, of the vessel and test portion before drying;

m_2 is the mass, in grams, of the vessel and test portion after drying.

Take as the result the arithmetic mean of the results of the two determinations (8.2) if the difference between the results is smaller than 0,2 % (mass fraction). Otherwise, repeat the determination on two other test portions. If this time the difference again exceeds 0,2 g per 100 g of sample, take as the result the arithmetic mean of the four determinations carried out, provided that the maximum difference between the individual results does not exceed 0,5 g per 100 g of sample.

Report result to one decimal place.

9.2 If, before the analysis, large non-oleaginous foreign bodies have been separated from the sample (see 7.1), multiply the result obtained in accordance with 9.1 by:

$$\frac{100\ \% - X}{100\ \%}$$

where X is the percentage by mass of large impurities, previously separated, in the initial material as received.

9.3 If the determination of moisture and volatile matter has been carried out on pure seed, calculate the moisture and volatile matter content by means of the formula given in 9.1.

10 Precision

10.1 Interlaboratory tests

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

10.2 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 (absolute values):

— for rapeseed 0,2 %;

— for soya beans 0,4 %;

— for sunflower seeds 0,2 %.

10.3 Reproducibility

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

— for rapeseed 0,4 %;

— for soya beans 2,0 %;

— for sunflower seeds 0,4 %.

11 Test report

The test report shall specify:

— all information necessary for the complete identification of the sample;

— the sampling method used, if known;

— the test method used, with reference to this International Standard;

— all operating conditions not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the result;

— the result obtained (arithmetic mean of the two determinations if the repeatability conditions have been checked) indicating clearly whether the result represents the "moisture content" of the product as received or the "moisture content" of the pure seeds.

Annex A
(informative)

Results of interlaboratory tests on the determination of moisture and volatile matter content of rapeseeds, sunflower seeds and soya seeds

A.1 Rapeseeds

An interlaboratory test was conducted in France, in 1986/1987. The number of laboratories participating was 15, and the number of repetitions was 2 (see Table A.1).

Table A.1 — Results of interlaboratory test on rapeseeds

	Ring 1	Ring 2	Ring 3
Number of laboratories retained after eliminating outliers	14	12	15
Mean value of the entire product, %	7,83	8,27	9,06
Repeatability standard deviation, s_r, of the entire product, %	0,04	0,10	0,05
Coefficient of variation of repeatability, %	0,5	1,1	0,5
Repeatability limit, r [2,8 × s_r], of the entire product, %	0,12	0,27	0,13
Reproducibility standard deviation, s_R, of the entire product, %	0,16	0 ,20	0,13
Coefficient of variation of reproducibility, %	2,0	2,5	1,5
Reproducibility limit, R [2,8 × s_R], of the entire product, %	0,44	0,58	0,38

A.2 Sunflower seeds

An interlaboratory test was conducted in France in 1986/1987. The number of laboratories participating was 15 and the number of repetitions was 2 (see Table A.2).

Table A.2 — Results of interlaboratory test on sunflower seeds

	Ring 1	Ring 2	Ring 3
Number of laboratories retained after eliminating outliers	10	14	13
Mean value of the entire product, %	7,32	7,79	8,29
Repeatability standard deviation, s_r, of the entire product, %	0,06	0,08	0,09
Coefficient of variation of repeatability, %	0,8	1,1	1,1
Repeatability limit, r [2,8 × s_r], of the entire product, %	0,16	0,24	0,25
Reproducibility standard deviation, s_R, of the entire product, %	0,07	0,13	0,18
Coefficient of variation of reproducibility, %	0,9	1,6	2,2
Reproducibility limit, R [2,8 × s_R], of the entire product, %	0,19	0,36	0,51

A.3 Soya seeds

Two interlaboratory tests were organized in France at the international level by the Centre technique interprofessionnel des oléagineux métropolitains (CETIOM).

The results obtained were subjected to statistical analysis in accordance with ISO 5725-1 [2] and ISO 5725-2 [3] to give the precision data shown in Tables A.3 and A.4.

The first test was carried out in 1996. The number of laboratories participating was 11, with 3 samples of soya beans and 2 repetitions (see Table 3).

The second test was carried out in 1997. The number of laboratories participating was 13, with 3 samples of soya beans and 2 repetitions (see Table A.4).

Table A.3 — Results of the first test

Sample	**1**	**2**	**3**
Number of participating laboratories	11	11	11
Number of participating laboratories after eliminating outliers	9	10	8
Mean value, %	5,18	3,86	16,16
Repeatability standard deviation, s_r, %	0,084	0,080	0,065
Coefficient of variation of repeatability, %	1,62	2,069	0,402
Repeatability limit, r [$2{,}8 \times s_r$], %	0,238	0,226	0,184
Reproducibility standard deviation, s_R, %	0,262	0,466	0,399
Coefficient of variation of reproducibility, %	5,059	12,072	2,469
Reproducibility limit, R [$2{,}8 \times s_R$], %	0,742	1,319	1,129

Table A.4 — Results of the second test

Sample	**1**	**2**	**3**
Number of participating laboratories	13	13	13
Number of participating laboratories after eliminating outliers	12	13	13
Mean value, %	12,60	18,01	13,20
Repeatability standard deviation, s_r, %	0,061	0,147	0,092
Coefficient of variation of repeatability, %	0,486	0,815	0,697
Repeatability limit, r [$2{,}8 \times s_r$], %	0,173	0,415	0,260
Reproducibility standard deviation, s_R, %	0,312	1,252	0,544
Coefficient of variation of reproducibility, %	2,478	6,95	4,118
Reproducibility limit, R [$2{,}8 \times s_R$], %	0,884	3,542	1,538