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Oilseed meals — Determination of moisture and volatile matter content

Tourteaux de graines oléagineuses — Détermination de la teneur en eau et en matières volatiles

Reference number
ISO/FDIS 771:2021(E)

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

This second edition cancels and replaces the first edition (ISO 771:1977), which has been technically revised. The main changes compared with the previous edition are as follows:

— organization of a new collaborative trial in order to add repeatability and reproducibility data.

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.

Oilseed meals — Determination of moisture and volatile matter content

1 Scope

This document specifies a method for the determination of the moisture and volatile matter content of oilseed meals obtained by the extraction of oil from oilseeds by pressure and/or solvent.

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 5502, *Oilseed residues — Preparation of test samples*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

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

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

3.1
moisture and volatile matter content
loss in weight measured under the operating conditions specified in this document

Note 1 to entry: The moisture and volatile matter content is expressed as a mass fraction in grams per 100 g.

4 Principle

The sample is ground to a particle size of 1 mm, followed by drying of a test portion at (103 ± 2) °C in an oven at atmospheric pressure, until practically constant mass is reached.

5 Apparatus

5.1 Analytical balance, readability 0,000 1 g, weighing precision 0,001 g.

5.2 Mechanical mill, easy to clean and allowing the meals to be ground, without heating and without appreciable change in the moisture, volatile matter and oil content, to particles passing completely through the sieve (5.3).

5.3 Sieve, with apertures of diameter 1 mm.

5.4 Flat-bottomed vessel, of metal, resistant to attack under the test conditions, provided with a well-fitting lid and allowing the test portion to be spread to about 0,2 g/cm^2 (e.g. diameter of vessel 50 mm to 70 mm, height about 30 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 and provided with a metal plate that allows vessels (5.4) to cool rapidly.

6 Sample

6.1 Sampling

Sampling is not part of the method specified in this document. A recommended sampling method is given in ISO 5500. A representative sample should have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

6.2 Preparation of test sample

Prepare the test sample in accordance with ISO 5502. Grind the contract sample in the previously well-cleaned mechanical mill (5.2), collect the grindings, mix carefully and carry out the analysis without delay.

7 Procedure

7.1 Carry out two determinations on each samples.

7.2 Weigh the vessel to the nearest 0,001 g with the lid (5.4), after leaving it open for at least 30 min in the desiccator (5.6) at laboratory temperature. Note mass m_0.

7.3 Weigh, to the nearest 0,001 g, about 5 g of the test sample into the vessel (5.4), spread the sample uniformly over the whole base of the vessel, and re-weigh with its lid. Carry out these operations as quickly as possible, in order to avoid any appreciable change in moisture content. Note mass m_1.

7.4 Place the vessel with the test portion in the oven (5.5), which has previously been set to a temperature of (103 ± 2) °C, and take off the lid.

If several vessels are in the oven together, arrange them in such a way that air can circulate freely between them.

7.5 Close the oven. After 2 h, reckoned from the time when the temperature in the oven has returned to (103 ± 2) °C, open the oven, replace the lid on the vessel before removal from the oven and transfer to the desiccator. As soon as the vessel has cooled to laboratory temperature, weigh it to the nearest 0,001 g.

During the drying, do not open the oven to add other test portions.

7.6 Return the vessel, with the lid removed, to the oven. After 1 h, repeat the operations of closing the vessel, allowing to cool, and weighing. Note Mass m_2.

7.7 If the difference between the two weighings is equal to or less than 0,005 g, regard the determination as finished. If not, repeat the procedure and dry again for 1 h in the oven, until the difference between two successive weighings is equal to or less than 0,005 g.

NOTE For most oilseed meals, a single 4 h period in the oven at (103 ± 2) °C gives equivalent results, but it is the responsibility of the analyst to confirm this in each particular case.

8 Expression of results

8.1 Method of calculation and formula

The moisture and volatile matter content w, as a mass fraction in grams per 100 g, is given by Formula (1):

$$w = \frac{m_1 - m_2}{m_1 - m_0} \times 100 \quad (1)$$

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.

Use the mean value from the 2 reps if repeatability conditions are satisfied (9.2) and express the result to one decimal place.

9 Precision

9.1 Results of interlaboratory test

Details of an interlaboratory test on the precision of the method are summarized in Annex A. The values derived from this interlaboratory test might not be applicable to concentration ranges and matrices other than those given.

9.2 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 r given as followed:

— for mean value less than 4,0 % (in mass fraction) : r = 0,2 %;

— for mean value more than 4,0 % (in mass fraction): $r = 0{,}018x + 0{,}13$ (see Figure A.1).

With x corresponding to the mean value of two reps. Some examples of r values are given in Annex B.

9.3 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 R given as followed:

— for mean value less than 4,0 % (in mass fraction): R = 0,4 %;

— for mean value more than 4,0 % (in mass fraction): $R = 0{,}033x + 0{,}27$ (see Figure A.1).

With x corresponding to the mean value of two reps. Some examples of R values are given in Annex B.

10 Test report

The test report shall specify:

a) all information necessary for the complete identification of the sample;

b) the test method used with reference to this document, i.e. ISO 771:—;

c) all operating details not specified in this document, or regarded as optional, together with details of any incidents that could have influenced the result;

d) the test result(s) obtained;

e) the date of the test.

Annex A
(informative)

Results of an international collaborative trial

The precision of the method was established by interlaboratory tests carried out in accordance with ISO 5725-1 and ISO 5725-2. Eleven laboratories participated in the tests. The following nine samples were used in the test.

A: Expeller rapeseed meal

B: Rapeseed meal

C: Dried rapeseed meal

D: Expeller sunflower meal

E: Post-extraction sunflower meal

F: Sunflower meal

G: Dried sunflower meal

H: Ground expeller soya meal

I: Soya meal

The results of the interlaboratory tests are given in Table A.1.

Table A.1 — Summary of statistical results (results in g/100 g)

Sample	**A**	**B**	**C**	**D**	**E**	**F**	**G**	**H**	**I**
Number of participating laboratories (N)	11	11	11	11	11	11	11	11	11
Number of laboratories retained after eliminating outliers (n)	11	11	11	11	11	11	11	11	11
Number of individual test results of all laboratories on each sample (z)	2	2	2	2	2	2	2	2	2
Mean value (m), g/100g	**4,57**	**11,82**	**8,15**	**6,04**	**7,81**	**12,16**	**8,09**	**6,17**	**10,94**
Repeatability standard deviation (s_r)	0,07	0,06	0,04	0,08	0,06	0,12	0,08	0,05	0,06
Relative repeatability ($C_{V,r}$), %	1,54	0,53	0,53	1,33	0,73	0,99	0,99	0,86	0,54
Repeatability limit r (s_r × 2,8)	**0,20**	**0,18**	**0,12**	**0,22**	**0,16**	**0,34**	**0,22**	**0,15**	**0,16**
Reproducibility standard deviation (s_R)	0,11	0,20	0,15	0,16	0,16	0,24	0,15	0,11	0,14
Relative reproducibility ($C_{V,R}$), %	2,34	1,73	1,80	2,67	2,03	1,95	1,87	1,85	1,24
Reproducibility limit R (s_R × 2,8)	**0,30**	**0,57**	**0,41**	**0,45**	**0,44**	**0,66**	**0,42**	**0,32**	**0,38**