INTERNATIONAL STANDARD

ISO 658

Third edition
2002-04-01

Oilseeds — Determination of content of impurities

Graines oléagineuses — Détermination de la teneur en impuretés

Reference number
ISO 658:2002(E)

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Printed in Switzerland

Contents

Page

Foreword iv
1 Scope 1
2 Normative references 1
3 Terms and definitions 1
4 Principle 2
5 Apparatus 2
6 Sampling 2
7 Preparation of test sample 2
8 Procedure 2
8.1 Test portion 3
8.2 Determination 3
9 Expression of results 4
9.1 Method of calculation 4
10 Precision 7
10.1 Interlaboratory test 7
10.2 Repeatability 7
10.3 Reproducibility 7
11 Test report 8
Annex A (informative) **Results of interlaboratory trial** 9
Bibliography 10

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

The main task of technical committees is to prepare International Standards. 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.

ISO 658 was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 2, *Oleaginous seeds and fruits*.

This third edition cancels and replaces the second edition (ISO 658:1988), which has been technically revised.

Annex A of this International Standard is for information only.

Oilseeds — Determination of content of impurities

1 Scope

This International Standard specifies a method for the determination of the impurities content in oilseeds used as primary industrial materials. It also defines the various categories of what are usually understood to be impurities.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents 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 659, *Oilseeds — Determination of oil content (Reference method)*

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

3 Terms and definitions

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

3.1
impurities in oilseeds
all foreign matter, organic and inorganic, other than seeds of the species under consideration

3.2
fines in oilseeds
particles passing through the sieves of aperture sizes given in Table 1, according to the species being analysed

NOTE In the case of groundnuts, meal from the seeds contained in the fines is not regarded as an impurity.

3.3
non-oleaginous impurities
non-oleaginous foreign bodies, fragments of stalks, leaves and all other non-oleaginous parts belonging to the oleaginous seed analysed, retained by the sieves of aperture sizes given in Table 1

EXAMPLES Bits of wood, pieces of metal, stones, seeds of non-oleaginous plants, and bits of shell, loose or adhering to palm kernels.

NOTE In the case of seeds sold in their shells, for example sunflower seeds (*Helianthus annuus* L.) or pumpkin seeds (*Cucurbita pepo* L.), the loose shells are regarded as impurities only if their proportion is larger than that of the corresponding kernels present in the same sample.

3.4
oleaginous impurities
oilseeds other than those of the species under consideration

4 Principle

The impurities are separated, by sieving and sorting, into three categories as follows:

— fines;

— non-oleaginous impurities;

— oleaginous impurities.

The mass of total impurities is determined or, on request, the mass of each category of impurity.

5 Apparatus

5.1 Sieves, having round holes with diameters as given in Table 1.

5.2 Tweezers, or other suitable instruments.

5.3 Analytical balance, capable of being read to the nearest 0,005 g.

5.4 Sample dividers, capable of taking 10 g sample aliquots of small seeds and 100 g sample aliquots of sunflower seeds and soyabeans.

Table 1 — Aperture sizes of sieves

Nature of product	Aperture diameter mm
Copra	2,0
Medium and larger sized seeds (see ISO 664)	1,0
Small seeds (see ISO 664)	0,5

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 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.

8 Procedure

NOTE If it is required to check whether the repeatability limits (10.2) are met, carry out two single determinations in accordance with 8.2.2 to 8.2.3.

8.1 Test portion

Take as the test portion a complete test sample (see clause 7). For a complete analysis, two or four test samples are necessary (see 10.2).

Weigh the test portion to the nearest 0,1 g.

8.2 Determination

8.2.1 General

The determination of impurities content shall be carried out sufficiently quickly to avoid any appreciable change in the moisture content of the seed.

8.2.2 Separation of fines

Separate the fines quantitatively by sieving the test portion on the sieve (see 5.1) and collecting them.

In the case of groundnuts, collect the total fines thus obtained, which include non-oleaginous fines and fines from the seed. Weigh them to the nearest 0,01 g and determine their oil content by the method specified in ISO 659. Determine also the oil content of the pure seeds by the method specified in ISO 659 in order to calculate the content of non-oleaginous fines.

8.2.3 Separation of oleaginous and non-oleaginous impurities

8.2.3.1 Copra and medium and larger sized seeds, apart from sunflower seeds and soya beans

In the material retained by the sieve (see 5.1), separate, by means of tweezers or any other suitable instrument (see 5.2), the non-oleaginous impurities (see 3.3), if necessary detaching bits of shell adhering to the seeds (as is the case with palm kernels) from the oleaginous impurities (see 3.4).

Weigh together, to the nearest 0,01 g, the non-oleaginous and oleaginous impurities and the fines (see 8.2.2), except in the case of groundnuts.

On request, weigh separately, to the nearest 0,01 g, each category of impurity.

If stipulated in the contract, note the nature of the oleaginous impurities in order that this can be recorded in the test report.

8.2.3.2 Sunflower seeds and soya beans

In the material retained by the sieve (see 5.1), separate, by means of tweezers or any other appropriate instrument (see 5.2), the impurities whose dimensions differ clearly from those of the sunflower seeds or soya beans being examined (large impurities). Class these impurities into two categories (oleaginous and non-oleaginous) and weigh each of them to within 0,01 g.

Using an aliquot portion (minimum 100 g, weighed to the nearest 0,1 g) of partially sorted sunflower seeds or soya beans, separate, by manual sorting, the oleaginous impurities and the non-oleaginous impurities (small impurities). Weigh each of these two fractions to the nearest 0,01 g.

8.2.3.3 Small seeds

Transfer the residue from the sieve (see 5.1) to a second sieve so as to retain impurities larger than the seeds, or separate these impurities by means of tweezers or any other suitable instrument (see 5.2). To assist the removal of large impurities, a 3,15 mm mesh may be used.

On request, sort this fraction into non-oleaginous impurities (see 3.3) and oleaginous impurities (see 3.4).

Weigh separately, to the nearest 0,01 g, the fines (see 8.2.2) and the impurities (non-oleaginous and oleaginous) larger than the seeds, and also the partially sorted seeds.

Using an aliquot portion of the latter fraction of seeds (at least 10 g, weighed to the nearest 0,01 g), separate, by sorting, the small non-oleaginous impurities from the small foreign oleaginous seeds. Weigh these two fractions of impurities, to the nearest 0,005 g, together or, on request, separately.

NOTE Specific methods for the determination of the content of *Sinapis arvensis* (wild mustard) seeds in rapeseeds (*Brassica napus*) and of turnip rape (*Brassica rapa*) have been published (see reference [3]).

8.2.4 Grouping of foreign oilseeds

If required, the foreign oilseeds may be grouped and weighed according to species, in order to show in the test report the mass fraction, in percent, of each species.

9 Expression of results

9.1 Method of calculation

9.1.1 Express the results as a mass fraction, in percent, of total impurities. On request, the percentage of each category of impurity may be indicated.

9.1.2 When the determination of impurities content has been carried out on the whole test portion (see 8.2.3.1), the calculation shall be as follows:

a) total impurities, I_t, expressed as a mass fraction in percent

$$I_t = \frac{m_4}{m_0} \times 100\ \% \qquad (1)$$

or

$$I_t = P + I_n + I_0 \qquad (1) = (2) + (3) + (4)$$

b) fines, P, expressed as a mass fraction in percent

$$P = \frac{m_1}{m_0} \times 100\ \% \qquad (2)$$

c) non-oleaginous impurities, I_n, expressed as a mass fraction in percent

$$I_n = \frac{m_2}{m_0} \times 100\ \% \qquad (3)$$

d) oleaginous impurities, I_0, expressed as a mass fraction in percent

$$I_0 = \frac{m_3}{m_0} \times 100\ \% \qquad (4)$$

where

m_0 is the mass, in grams, of the test portion;

m_1, m_2, m_3 are the respective masses, in grams, of each category of impurity;

m_4 is the mass, in grams, of all the impurities, including the fines.

9.1.3 When only a part of the impurities has been separated from the whole of the test portion and the other from an aliquot portion of the remainder (see 8.2.3.2 and 8.2.3.3), the calculation shall be as follows:

a) total impurities, I_t, expressed as a mass fraction in percent

$$I_t = \left[\frac{m_4}{m_0} + \frac{(m_0 - m_4)m_5}{m_0 \times m_b} \right] \times 100\ \% \qquad (5)$$

or

$$I_t = P + I_n + I_0 \qquad (5) = (6) + (7) + (8)$$

b) fines, P, expressed as a mass fraction in percent

$$P = \frac{m_1}{m_0} \times 100\ \% \qquad (6)$$

c) non-oleaginous impurities, I_n, expressed as a mass fraction in percent

$$I_n = \left[m_{2a} + \left(m_{2b} \times \frac{m_a}{m_b} \right) \right] \times \frac{100\ \%}{m_0} \qquad (7)$$

d) oleaginous impurities, I_0, expressed as a mass fraction in percent

$$I_0 = \left[m_{3a} + \left(m_{3b} \times \frac{m_a}{m_b} \right) \right] \times \frac{100\ \%}{m_0} \qquad (8)$$

where

m_0 is the mass, in grams, of the initial test portion;

m_1 is the mass, in grams, of the fines;

m_{2a} is the mass, in grams, of the fraction of non-oleaginous impurities larger than seeds of the basic species and separated from the whole test portion;

m_{2b} is the mass, in grams, of the fraction of small non-oleaginous impurities separated from the aliquot portion of the residue obtained after elimination, from the test portion, of fines and impurities larger than seeds of the basic species;

m_{3a} is the mass, in grams, of the fraction of oleaginous impurities larger than seeds of the basic species and separated from the whole test portion;

m_{3b} is the mass, in grams, of the fraction of small oleaginous impurities separated from the aliquot portion of the residue obtained after elimination, from the test portion, of fines and impurities larger than seeds of the basic species;

m_a is the mass, in grams, of the residue obtained after elimination, from the initial test portion, of fines and impurities larger than seeds of the basic species:

$$m_a = m_0 - m_1 - m_{2a} - m_{3a}$$

m_b is the mass, in grams, of the aliquot portion of the residue of mass m_a before the small impurities have been separated;

m_4 is the mass, in grams, of the fines and of the fraction of impurities larger than seeds of the basic species and separated from the whole test portion;

m_5 is the mass, in grams, of the fraction of impurities separated from the aliquot portion of the residue obtained after elimination, from the test portion, of the fines and the impurities larger than seeds of the basic species.

9.1.4 In the case of groundnuts, the calculation shall be as follows:

a) total impurities, I_t, expressed as a mass fraction in percent

$$I_t = \left[\frac{m_1}{m_0} \left(1 - \frac{H_2}{H_1} \right) + \frac{m_4}{m_0} \right] \times 100\ \% \qquad (9)$$

or

$$I_t = P_s + I_n + I_0 \qquad (9) = (11) + (12) + (13)$$

b) total fines, P, expressed as a mass fraction in percent

$$P = \frac{m_1}{m_0} \times 100\ \% \qquad (10)$$

c) foreign fines, P_s, expressed as a mass fraction in percent

$$P_s = \frac{m_1}{m_0} \times \left(1 - \frac{H_2}{H_1} \right) \times 100\ \% \qquad (11)$$

d) non-oleaginous impurities, I_n, expressed as a mass fraction in percent

$$I_n = \frac{m_2}{m_0} \times 100\ \% \qquad (12)$$

e) oleaginous impurities, I_0, expressed as a mass fraction in percent

$$I_0 = \frac{m_3}{m_0} \times 100\ \% \qquad (13)$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the fines;

m_2 and m_3 are the respective masses, in grams, of the non-oleaginous impurities and the oleaginous impurities;

m_4 is the mass, in grams, of the impurities other than the fines;

H_1 is the oil content, expressed as a mass fraction in percent, of the pure seed;

H_2 is the oil content, expressed as a mass fraction in percent, of the fines.

9.1.5 Take as the result the arithmetic mean of the two determinations, if the conditions of repeatability (see 10.2) are satisfied.

9.1.6 Report the results to two decimal places for mass fractions of impurities not exceeding 0,5 % and to one decimal place for impurities contents above this limit.

10 Precision

10.1 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 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 values given in Table 2.

If the difference is greater than the limit indicated in Table 2, obtain two other test portions. Analyse one as before and keep the other for a fourth determination if necessary. In this case, take as the result the arithmetic mean of the result obtained from the third analysis and the nearest result obtained from the previous analyses, provided that the difference does not exceed the allowed limit.

Failing this, analyse also the fourth test portion and take as the result the mean of the four determinations.

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 values given in Table 2.

Table 2 — Repeatability and reproducibility limits

Values expressed as mass fraction in percent

Impurities content	Repeatability limit, r	Reproducibility limit, R
Up to and including 0,5	0,2	0,4
Over 0,5 to 1,0 inclusive	0,4	0,8
Over 1,0 to 2,0 inclusive	0,6	1,8
Over 2,0 to 3,0 inclusive	0,8	2,4
Over 3,0 to 4,0 inclusive	1,0	3,0
Over 4,0 to 5,0 inclusive	1,2	3,6
Over 5,0 to 6,0 inclusive	1,4	4,2
Over 6,0	1,6	4,8

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 details not specified in this International Standard, or regarded as optional, together with details of any incidents which may have influenced the test results;

— the test results obtained (total impurities and, on request, each category of impurity); if the product contains foreign oleaginous seeds and, if stipulated by the contract, indicate not only their total mass fraction but also their nature; if required, the mass fraction of each species of foreign oleaginous seeds may also be indicated;

— if the repeatability has been checked, the final quoted result obtained.

Annex A
(informative)

Results of interlaboratory trial

An international collaborative test involving 13 laboratories in 6 countries was carried out on 5 samples:

— groundnut kernels;

— linseed;

— rapeseed;

— soya beans;

— sunflower seeds.

The test was organized by the Leatherhead Food Research Association in 1998 and the results obtained were subjected to statistical analysis in accordance with ISO 5725-1 [4] and ISO 5725-2 [5] to give the precision data shown in Table A.1.

Table A.1 — Precision data

	Groundnut kernels	Linseed	Rapeseed	Soya beans	Sunflower seeds
Number of laboratories after eliminating outliers	8	13	13	12	12
Number of accepted results	8	13	13	12	12
Mean value, % (mass fraction)	0,00	3,72	1,31	0,77	1,10
Repeatability standard deviation (s_r)	0,004	0,203	0,077	0,065	0,112
Repeatability limit (r)	0,01	0,57	0,21	0,18	0,31
Reproducibility standard deviation (s_R)	0,005	0,923	0,687	0,174	0,348
Reproducibility limit (R)	0,014	2,58	1,92	0,49	0,97

Bibliography

[1] ISO 542, *Oilseeds — Sampling*

[2] Determination of the content of *Sinapis arvensis* (wild mustard) seeds in rape seeds. *Official Journal of the European Communities*, No. L 300 – 20.11.1975

[3] ANDREW M., HAMILTON R.J. and ROSSEL J.B. The chemical differentiation between *Sinapis arvensis and Brassica napus* seeds by surface wax analysis. *Fat Science Technology*, **89**, 1987, pp. 7-15

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

[5] ISO 5725-2:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

ICS 67.200.20

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