INTERNATIONAL STANDARD

ISO 5502

Second edition
1992-10-15

Oilseed residues — Preparation of test samples

Tourteaux de graines oléagineuses — Préparation des échantillons pour essai

Reference number
ISO 5502:1992(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 5502 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Sub-Committee SC 2, *Oleaginous seeds and fruits*.

This second edition cancels and replaces the first edition (ISO 5502:1983), clause 2, subclause 5.4 and figure 2 of which have been technically revised.

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Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Oilseed residues — Preparation of test samples

1 Scope

This International Standard specifies methods for the preparation of test samples of oilseed residues by the reduction of laboratory samples.

For the purposes of this International Standard, the term **oilseed residues** includes meals, extractions, expeller cakes or slab cakes[1] resulting from the production of crude vegetable oils from oilseeds by pressure or solvent extraction. It does not include compounded products.

NOTE 1 The sampling of oilseed residues for the preparation of laboratory samples is described in ISO 5500:1986, *Oilseed residues — Sampling*.

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 565:1990, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*.

ISO 771:1977, *Oilseed residues — Determination of moisture and volatile matter content*.

3 Principle

Grinding of the laboratory sample, with or without preliminary breaking, crushing, grinding or drying. Division of the sample thus obtained by suitable means, taking care that the test sample, from which the test portion(s) will be taken, truly represents the totality of the laboratory sample.

4 Apparatus

4.1 Mechanical mill, easy to clean and allowing the oilseed residue to be ground, without heating and without appreciable change in its oil and moisture and volatile matter contents, until it passes completely through a sieve of aperture size 1,00 mm [or 2,80 mm (see 5.1.3.1)].

4.2 Crushing apparatus, if required, for example an iron pestle and mortar, or other means for breaking or crushing pieces of oilseed residues to a size suitable for introduction into the mechanical mill (4.1).

4.3 Sieves, of aperture sizes 1,00 mm and 2,80 mm, made from metal wire cloth, and complying with the requirements of ISO 565.

4.4 Dividing apparatus, quartering apparatus, conical divider (see figure 1), multiple-slot divider (see figure 2) or other dividing apparatus, which will ensure uniform distribution of the components of the laboratory sample in the test sample.

4.5 Sample container, suitable for protecting the test sample from change in composition, and of such a size that it will be almost completely filled by the test sample.

1) In this context, **slab cakes** are cakes of oilseed residues produced by hydraulic presses, and have a typical mass of about 10 kg.

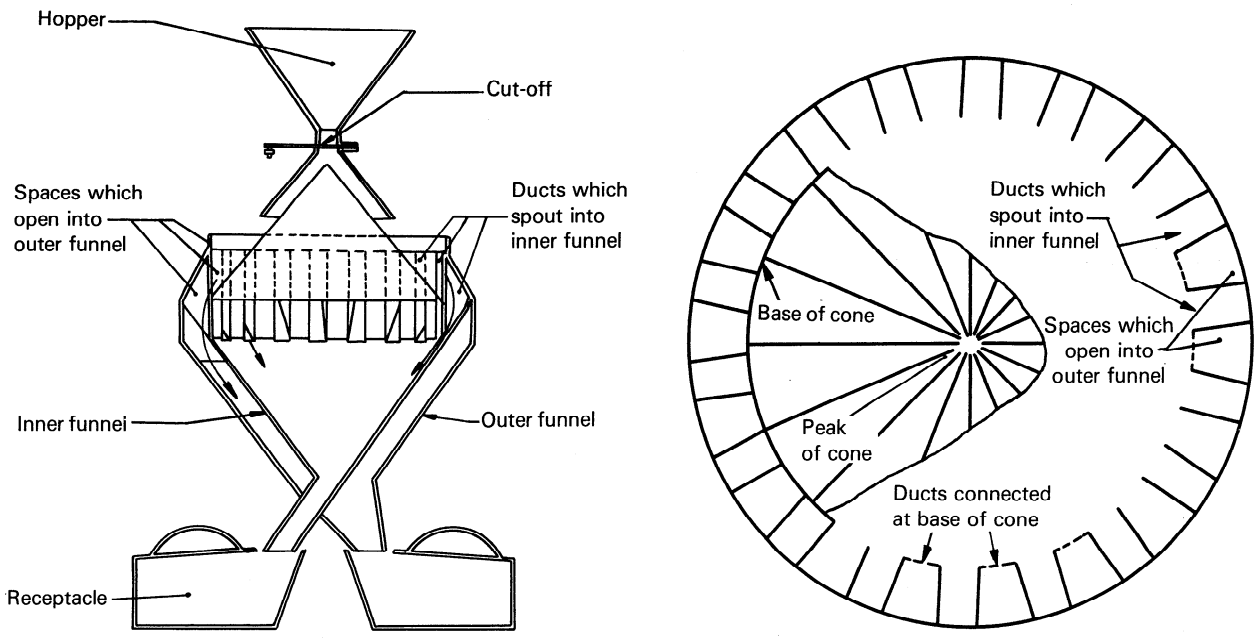

Figure 1 — Conical divider

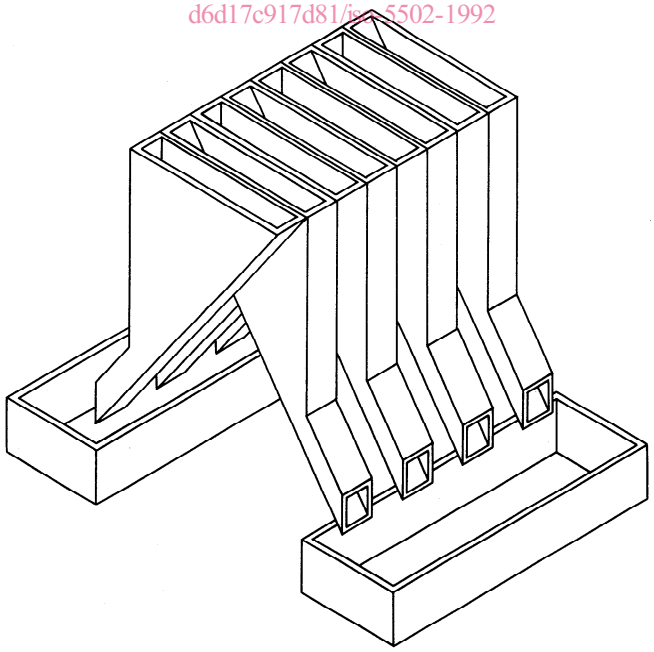

Figure 2 — Multiple-slot divider

5 Procedure

Using the laboratory sample as received, proceed as follows.

5.1 Grinding (general case)

With some mechanical mills, fine grinding may lead to a loss or gain of moisture and volatile matter, and allowance for this shall be made as indicated in clause 6.

Carry out grinding as rapidly as possible, avoiding unnecessary exposure to the atmosphere. If necessary, first break or crush the pieces to a suitable size for grinding. Use the first twentieth of the laboratory sample to complete the cleaning of the mechanical mill (4.1) and to establish the fineness of grinding, and then discard it.

It is essential that the samples are well mixed before each operation.

5.1.1 Fine samples

5.1.1.1 If the laboratory sample passes the 1,00 mm sieve (4.3) completely, mix it thoroughly.

5.1.1.2 Divide the mixture successively using appropriate dividing apparatus (4.4), or by quartering with the aid of a spatula 25 cm in length, until a sample of at least 100 g, and of suitable mass for all the determinations required, is obtained.

5.1.2 Coarse samples

5.1.2.1 If the laboratory sample does not pass the 1,00 mm sieve completely but passes the 2,80 mm sieve completely, mix it thoroughly.

5.1.2.2 Carefully grind a portion of at least 100 g, and of suitable mass for all the determinations required, in the previously well-cleaned mechanical mill (4.1) until it passes the 1,00 mm sieve completely.

5.1.3 Very coarse samples

5.1.3.1 If the laboratory sample is very coarse, carefully grind it in the previously well-cleaned mechanical mill (4.1) until it passes the 2,80 mm sieve completely. Mix it thoroughly.

5.1.3.2 Divide the ground laboratory sample successively by means of appropriate dividing apparatus (4.4) until a sample of not less than 100 g, and of suitable mass for all the determinations required, is obtained. Grind this sample in the previously well-cleaned mechanical mill (4.1) until it passes the 1,00 mm sieve completely.

5.2 Grinding (special cases)

5.2.1 Moist samples

Except for fine samples (5.1.1), if the laboratory sample is appreciably moist, or if, for any reason, the mixing and grinding operations are likely to result in a loss or gain of moisture and volatile matter, take a sample for the determination of the moisture and volatile matter contents immediately after the preliminary mixing procedure described in 5.1.2.1, or after the preliminary grinding procedure described in 5.1.3.1.

Determine the moisture and volatile matter content by the method described in ISO 771. Also determine the moisture and volatile matter content of the prepared test sample by the same method, so that the results of analyses may be corrected to relate to the sample in its original condition as regards moisture and volatile matter content (see clause 6).

5.2.2 Samples difficult to grind

If the physical condition of the sample makes grinding difficult, take a sample for the determination of the moisture and volatile matter contents immediately after the preliminary mixing procedure described in 5.1.2.1, or after the preliminary grinding procedure described in 5.1.3.1.

Determine the moisture and volatile matter content by the method described in ISO 771. Dry the sample until it can be ground with the iron pestle and mortar (4.2), or by other means, so that it passes the 1,00 mm sieve completely. Then determine the moisture and volatile matter content of the prepared test sample by the same method, so that the results of analyses may be corrected to relate to the sample in its original condition as regards moisture and volatile matter content (see clause 6).

5.2.3 Samples for which there are special requirements

5.2.3.1 For determinations requiring special degrees of fineness of grinding (for example, determination of urease activity), further grinding may be necessary. In such cases, prepare a test sample as described in 5.1, 5.2.1 or 5.2.2, but having the required degree of fineness.

5.2.3.2 For the preparation of test samples for the determination of residual extraction solvent content

(volatile hydrocarbons), see ISO 8892.[2)]

6 Correction factor

6.1 General

If the grinding or mixing operations are likely to result in a loss or gain of moisture and volatile matter, it is necessary to use a correction factor to relate the results of analyses to the sample in its original condition as regards moisture and volatile matter content.

6.2 Calculation

The correction factor C, expressed as a percentage by mass, is given by the equation

$$C = \frac{100\ \% - U_0}{100\ \% - U_1}$$

where

U_0 is the moisture and volatile matter content, expressed as a percentage by mass, of the sample after the preliminary treatment described in 5.1.2.1 or 5.1.3.1;

U_1 is the moisture and volatile matter content, expressed as a percentage by mass, of the prepared test sample.

6.3 Use of the correction factor

Multiply the results of analyses, expressed as percentages by mass, by the correction factor C.

7 Storage of the test sample

Transfer the prepared test sample without delay to the sample container (4.5) and close it.

2) ISO 8892:1987, *Oilseed residues — Determination of total residual hexane.*

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UDC 633.85:620.11

Descriptors: agricultural products, plant products, oilseeds, oilseed residues, sampling, specimen preparation, sampling equipment.

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