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Oilseeds — Manual or automatic discontinuous sampling

Graines oléagineuses — Échantillonnage discontinu manuel ou automatique

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Foreword

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

This first edition of ISO 21294, together with ISO 21293, cancels and replaces ISO 542:1990, which has been technically revised.

Introduction

Most oilseeds are marketed on the basis of the result of analysis of the samples representing lots, and disputes are invariably settled by reference to these samples. Careless or inaccurate sampling practices could lead to misunderstandings, delays and unwarranted financial adjustments.

Correct sampling is a difficult process and one that requires the most careful attention. Emphasis cannot therefore be too strongly laid on the necessity of obtaining a representative sample of oilseeds for analysis.

Oilseeds — Manual or automatic discontinuous sampling

1 Scope

This document specifies the requirements for discontinuous sampling of oilseeds, using the manual or automatic method, for the purpose of assessing their quality and condition.

NOTE An example of "condition" is an odour due to a treatment product.

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

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:

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

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

3.1
discontinuous sampling
sampling by automatic or manual means of at least one position within a *lot* (3.3) of both static and moving oilseeds

Note 1 to entry: Manual sampling of moving oilseeds is considered discontinuous sampling.

Note 2 to entry: For comparison, continuous sampling is the automatic uninterrupted sampling of moving oilseeds within a lot across the entire flow (for example, a permanent sampling system on a conveyor belt or any circulation flow that enables continuous sample taking throughout the loading or discharge of the consignment; there is no break in the sampling procedure).

EXAMPLE Hand-scoops, manual and/or automatic samplers (sequenced), shovels, suitable sampling buckets, etc. are means of sequenced sampling and are part of discontinuous sampling.

3.2
consignment
quantity of oilseeds dispatched or received at one time and covered by a particular contract or shipping document

Note 1 to entry: The consignment can be composed of one or more lots or part of a lot.

3.3
lot
stated quantity of the *consignment* (3.2) presumed to be of uniform characteristics, which can be sampled in order to determine its quality and condition

Note 1 to entry: The quantity of the lot can be of a mass up to 5 000 t.

3.4
increment sample
amount of material taken at one time at each individual sampling point (at each individual sampling time for moving lot) throughout a *lot* (3.3)

3.5
bulk sample
quantity of oilseeds obtained by combining and blending the increments taken from any one particular *lot* (3.3)

3.6
homogenization
thorough blending by mechanical or manual means so that any contaminants or foreign materials are thoroughly distributed throughout the bulk or *laboratory sample* (3.7)

3.7
laboratory sample
representative quantity of oilseeds obtained by *homogenization* (3.6) and division of the *bulk sample* (3.5) and intended for analysis or other examination in a laboratory

4 Principles

4.1 Representative samples

The laboratory samples shall be as representative as possible of the lots from which they are taken. Each consignment shall be divided, actually or notionally, into lots of 500 t to 5 000 t according to its size. At least a minimum number of increments shall be taken from each lot and homogenized carefully in order to obtain a bulk sample from which laboratory samples may be taken by successive division. A laboratory sample shall result from the sampling of each lot (see Table 1).

A diagram showing the different steps for laboratory samples is given in Annex A.

4.2 Oilseeds sampling

This document covers both the discontinuous sampling of oilseeds in motion while being transferred and the sampling of static lots. Samples shall be taken using manual or automatic methods. Where possible, sampling should be performed when the oilseeds are flowing (e.g. during loading or unloading) so that the constituent parts of the lot have the same probability of being sampled and comply with the minimum number and mass of increments prescribed.

Sampling shall be carried out in such a manner as to protect the samples, the sampling instruments and the container in which the samples are placed to prevent unintentional adventitious contamination such as rain, dust, introduction of foreign material, etc.

All sampling operations shall be carried out over a sufficiently short period of time so as to avoid any alteration in the composition of the samples. If one of the sampling stages requires too long a period of time, the samples or intermediate samples shall be preserved in airtight containers.

It is extremely important that the preparation/reduction of a bulk sample be done using the appropriate equipment and method. Failure to complete this process accurately will impact any analysis conducted on a sample that might or might not be representative of the lot. It is important to maintain the representativeness of the lot being sampled, specifically by avoiding segregation of materials in order to achieve random sampling, which shall be carried out using unbiased methods.

5 Equipment and devices

5.1 Special care shall be taken to ensure that all sampling apparatus is clean, dry, free from foreign odours and made from material which will not contaminate or alter the quality and the condition of the oilseeds.

The devices used for sampling and division fall under the categories described in 5.2 to 5.4; examples are given in each case.

NOTE Examples of a device used for sampling and division are given in Annex B. There are a number of different types of sampling equipment or devices. It is advisable to choose the most appropriate equipment according to the type of oilseed to be sampled and the containers (e.g. bottles, jars or tins) to be used.

5.2 Devices for sampling from bags: sack-type tapered sampling probes, cylindrical samplers, conical samplers and hand-scoops.

5.3 Devices for sampling bulk products: large shovels, hand-scoops, cylindrical samplers, conical samplers, automatic samplers and other devices for taking small periodical increments discontinuously from a flow of oilseeds

NOTE This list is not exhaustive.

5.4 Devices for mixing and reduction: dividing instruments, shovels and quartering irons.

6 Time and place of sampling and limitation of the size of lots

6.1 General information

Whether the consignment is in bulk or in bags, sampling shall be normally carried out during, and at the place of, loading into or discharge from the ship, barge, wagon or lorry or at the time of entry into or exit from the silo or warehouse, as agreed between the parties concerned. According to the consignment mass of the sampled product, each laboratory sample should be identified with the bulk tonnage of the sampled product and should represent a lot up to 5 000 t plus a remainder (see Table 1). Special requirements for bulk transfer are given in 6.2.

6.2 Bulk transfer

6.2.1 General

It is generally advisable to use the procedures described in 6.2.2 to 6.2.4 for the bulk transfer of oilseeds.

6.2.2 Transfer to lorries and wagons

The increments should be taken from the flow of product (preferred method) during the entire loading or entire discharge (particularly for tanker-wagons where internal sampling is not possible), or in the lorry or wagon, as soon as possible after loading. At least five different positions should be sampled according to the size of the lorry or wagon (see 7.2.3.3) for the purpose of providing one bulk sample (see Table 1).

6.2.3 Transfer to barges

The increments should be taken systematically and randomly from the flow of product (preferred method) during the entire loading or entire discharge. Each hold should be sampled throughout the entire duration of loading for the purpose of providing one bulk sample.

6.2.4 Transfer to silos or warehouses

The increments should be taken from conveyor belts taking into account the rate of movement of these belts for the purpose of providing one bulk sample.

6.3 Lot size: number of laboratory samples

Table 1 gives the following information.

— The lot size depending on the total consignment mass.

— The minimum number of increment samples per lot.

— The number of laboratory samples to prepare is determined according to the number of lots. There shall be one laboratory sample for each lot and one for the remainder, if present.

The minimum increment mass, depending on the size of the oilseed and the lot size shall be determined by dividing the "bulk sample mass" (presented in Table 2) by the "number of increments" (presented in Table 1).

Table 1 — Lot size, number of increment samples and laboratory samples according to the mass of the consignment

Total consignment mass tonnes t	**Lot size** t	**Minimum number of increment samples per lot** min. number	**Number of laboratory samples per consignment** total mass/lot size
< 500	0 to 500	20	1
500 to 5 000	500	20	1 to 10
> 5 000 to 10 000	1 000	30	5 to 10
> 10 000 to 25 000	2 500	40	4 to 10
> 25 000	5 000	50	5 to 15

Sampling shall be made by full lots plus a remainder. If the remainder is less than 500 t, it shall be added to the previous lot.

The following is a list of examples.

— A consignment of a total mass of 500 t implies a lot of 500 t and will be represented by one laboratory sample.

— A consignment of a total mass of 2 000 t implies four lots of 500 t and will be represented by four laboratory samples.

— A consignment of a total mass of 6 000 t implies six lots of 1 000 t and will be represented by six laboratory samples.

— A consignment of a total mass of 20 000 t implies eight lots of 2 500 t and will be represented by eight laboratory samples.

— A consignment of a total mass of 20 490 t implies seven lots of 2 500 t, and one lot of 2 990 t and will be represented by eight laboratory samples.

— A consignment of a total mass of 20 500 t implies eight lots of 2 500 t, and one lot of 500 t and will be represented by nine laboratory samples.

— A consignment of a total mass of 67 000 t implies 13 lots of 5 000 t and a remaining lot of 2 000 t and will be represented by 14 laboratory samples.

7 Method for taking samples

7.1 General information

As the composition of a lot is seldom, if ever, homogeneous, even in the case of undamaged lots, it is necessary to take a sufficient number of increments to provide a representative bulk sample. Parts of lots which are damaged by sea water or otherwise damaged in transit or out of condition, as well as loose material and sweepings that have been recovered, shall be sampled separately from the sound material. Each type of damaged material shall be assessed by mass, sampled and separated from the sound material.

NOTE The term "loose material" is used to designate material that has leaked from its original container but is not unduly contaminated.

7.2 Number of increments

7.2.1 General requirements on taking increments

A series of increments is taken from different parts of the lot, evenly distributed, so that when they are composited they are representative of the entire lot.

The use of devices to sample bags should maximize sampling a cross-section of the bag to account for possible segregation of product and admixtures, depending on composition of particle sizes and their relative densities within the bag.

According to circumstances (see Table 1), the increments shall be taken from products in bulk or in bags by means of the sampling devices mentioned in Clause 5 and used in accordance with 7.2.2 and 7.2.3.

7.2.2 Products in bags

Increments shall be randomly taken from 2 % of the bags forming the lot, with a minimum of five bags. If the bags are open, the increments may be taken using cylindrical samplers, conical samplers or other appropriate instruments, preferably after the bags have been emptied.

If the bags (for example, jute bags) are closed, the increments may be taken using sack-type tapered sampling probes.

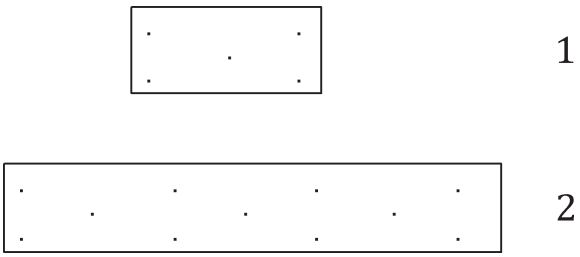
7.2.3 Products in bulk

7.2.3.1 When sampling takes place while the product is in motion, which is the preferred method, increments shall be taken across the whole section of the flow, perpendicular to the direction of flow, and at time intervals that span the duration of product flow, and the sampling frequency adjusted according to the rate of loading or discharge.

7.2.3.2 When bulk material is sampled in holds during discharge, the increments should be taken at regular intervals determined by the rate of discharge and throughout the entire unloading process.

7.2.3.3 When sampling takes place from laden wagons or lorries, the increments shall be taken at three levels at least (owing to the fact that layering may occur, particularly in vehicles in motion) with a

cylindrical sampler or conical sampler, depending on the product, and at the following points shown in Figure 1.



Key

1 wagons up to 30 t

2 wagons more than 30 t

Figure 1 — Sampling points (example)

To comply with the minimal number of 20 increment samples to be taken (see Table 1), an alveolate sampler (see Annex B) or a sampler designed to allow capture of material along the length of the probe shall be used. The sampler shall ensure the probe is pushed to the bottom of the hold, if possible, to make sure that samples are representative of the product. In this case, each compartment is considered as an increment sample.

When sampling takes place from a dumper truck where an automatic core sampler goes totally through the oilseeds layer and which enables sampling without segregation of materials, three sampling points are sufficient (at the front, in the middle and at the back).

If using a manual "probe sampler" to sample prior to unloading (i.e. static sampling), the probe shall be compartmented (see the example in Annex B) to allow capture of material along the length of the probe, and the sampler shall ensure the probe is pushed to the bottom of the hold, if possible, to make sure that samples are representative of the product.

If the type of wagon or lorry does not allow samples to be taken in this manner, the method of sampling shall be as described for products in motion (7.2.3.1), which remains in any case the recommended method.

7.2.3.4 If sampling takes place from weigh hoppers, the increments shall be taken by means of cylindrical samplers or shovels, in accordance with sampling capabilities and sampling opportunities at the location of loading or unloading.

7.2.4 Aggregation of the bulk sample

The mass of individual incremental samples being aggregated should have approximately the same size, and all increments shall be combined to make the bulk sample. Proper blending of the aggregate sample should be performed before the sample is reduced or sub-sampled. Mechanical methods for homogenization of materials are preferred, but where manual blending is performed, attention shall be given to segregation effects as it pertains to the product.

7.2.5 Division of the bulk sample

7.2.5.1 General

The bulk sample shall be reduced to obtain the required number of laboratory samples for each lot of specified mass (see Table 2) by using a method and equipment that will give representative laboratory samples. Specific equipment may be used when handling very large bulk samples, but it shall be capable of producing representative laboratory samples.

The equipment shall be thoroughly cleaned between each sample to avoid cross-contamination.

7.2.5.2 Coning and quartering method

Thoroughly mix the bulk sample by repeating operations at least twice before dividing. Work on a clean, non-absorbent surface.

Gather the oilseeds together into a cone-shaped pile. Flatten out the surface of the pile and then divide the pile into quarters: A, B, C, and D. Discard two diagonally opposed quarters (B and C) and mix the two remaining quarters (A and D) (see Figure 2). Repeat the whole process until the laboratory sample of the required size is obtained.

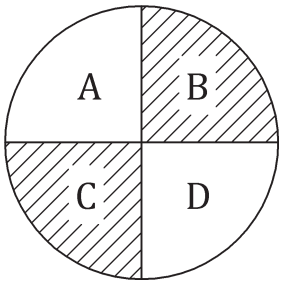


Figure 2 — Coning and quartering method

7.2.6 Sample dividers

Use the divider on a flat surface.

Dividers shall have a channel width adapted to the size of the largest particulate being sampled in order to not introduce a bias.

7.2.6.1 Cone-shaped divider

7.2.6.1.1 To reduce a bulk sample, use an apparatus with a stand, if necessary, and use suitable collection boxes or buckets.

7.2.6.1.2 Homogenize the sample by repeating operations at least three times and by remixing the subsamples in the hopper.

7.2.6.1.3 Pour the bulk sample into the closed hopper.

7.2.6.1.4 Collect two sub-samples in the two collection boxes (or buckets).

7.2.6.1.5 Keep the contents of one of the collection boxes and return the other.

7.2.6.1.6 Put the two empty collection boxes back in position.

7.2.6.1.7 Repeat operations as many times as necessary, alternating the collection boxes to be kept until the laboratory sample of the required size is obtained.

7.2.6.2 Rotary mechanical divider

Switch on the centrifugal divider. Pour the bulk sample into the upper hopper.

Proceed as in 7.2.6.1.1 to 7.2.6.1.7.

7.2.6.3 Riffle divider

Riffle dividers shall only be used for small samples (less than 2 kg).

Proceed as in 7.2.6.1.2 to 7.2.6.1.7.

Care should be taken to ensure the riffle divider is used correctly, as the accuracy of this equipment is heavily influenced by operator error.

It is important that the mass of the seed be uniformly distributed along the entire width of the divider before being poured onto the rifles. The width of the flow of grain on the rifles shall be perfectly perpendicular to the rifles.

8 Sample size per lot and for dispatch to the laboratory

The sizes of samples given in Table 2 are usually suitable. Larger or smaller samples may be required in some cases, according to the tests to be carried out. The minimum increment sample mass is included in Table 2.

Whatever the size of the bulk sample, it shall be representative of the lot.

Table 2 — Bulk sample size and laboratory sample size according to the lot size and nature of the product

Lot size	≤ 500 t	1 000 t	2 500 t and above	All lots size
Nature of product	bulk sample minimum mass per lot kg			Laboratory sample minimum mass kg
Copra	40	60	80	5
Medium-size and large seeds (as defined in ISO 664)	30	40	60	2 to 5
Small seeds (as defined in ISO 664)	8	12	16	1 to 2

9 Packing and labelling of samples

9.1 Packing of samples

The laboratory samples shall be packed in containers that preserve the integrity of the sample (e.g. jars). The containers shall be completely filled. If the moisture has to be preserved, the closures shall be sealed to avoid any change in the original moisture content of the sample.

For aflatoxin analysis only, the sample shall be protected from light.

9.2 Labelling of samples

9.2.1 If paper labels are used, their quality and size shall be suitable for the purpose. The eyelet hole in the label shall be reinforced.

9.2.2 Each label shall bear at least the following information:

a) ship or road vehicle;

b) from;

