International Standard 5500

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Oilseed residues — Sampling

Tourteaux de graines oléagineuses — Échantillonnage

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 5500 was prepared by Technical Committee ISO/TC 34, *Agricultural food products.*

This second edition cancels and replaces the first edition (ISO 5500-1984), to which a third annex (annex C) has been added.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Oilseed residues — Sampling

0 Introduction

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

Practically all oilseed residues are sold on the basis of the result of analysis of the samples, and disputes are invariably settled by reference to the sample, so that careless or inaccurate sampling could lead to misunderstanding, delay and unwarranted financial adjustments.

The procedures given in this International Standard are recognised as good practice and it is strongly recommended that they be followed whenever practicable.

1 Scope and field of application

This International Standard specifies methods of sampling oilseed residues.

It is applicable to all oilseed residues, regardless of their presentation, i.e. whether in the form of meals, agglomerates or slab cakes.

Annex C gives a method, reflecting the present state of knowledge, of sampling oilseed residues containing undesirable substances which are likely to be non-uniformly distributed, such as mycotoxins, castor-oil seed husks and poisonous seeds.

2 Definitions

For the purpose of this International Standard, the following definitions apply.

2.1 consignment : The quantity of oilseed residues dispatched or received at one time and covered by a particular contract or shipping document. It may be composed of one or more lots or parts of a lot.

2.2 lot : A stated quantity of the consignment, of mass not exceeding 500 t, presumed to be of uniform characteristics, and which will allow the quality to be assessed.

2.3 increment : A small quantity of oilseed residues taken at one time from a single position in the lot. A series of increments is taken from different parts of the lot, so that, when they are bulked, they are representative of the lot.

2.4 bulk sample : The quantity of oilseed residues formed by combining and blending the increments taken from any one particular lot.

2.5 reduced sample : The quantity of oilseed residues obtained by successive divisions of the bulk sample and which will allow identical laboratory samples representative of the lot to be prepared.

2.6 laboratory sample : A sample representing the quality of the lot, obtained from the reduced sample and intended for analysis or other examination.

3 General

3.1 Samples shall be fully representative of the lots from which they are taken. For this purpose, each consignment shall be divided, actually or notionally, into lots of mass not exceeding 500 t and a number of increments shall be taken from each lot and carefully mixed to give a bulk sample from which laboratory samples are obtained by successive division.

3.2 Special care is necessary to ensure that all sampling apparatus is clean, dry, free from foreign odours and made from material which will not contaminate the oilseed residue.

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, from adventitious contamination such as rain, dust, etc.

Material adhering to the outside of the sampling instrument shall be removed before the contents are discharged.

3.3 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 will require too long a period of time, the samples or intermediate samples shall be preserved in airtight containers.

If samples are required for the determination of volatile hydrocarbons, it is particularly important that loss by evaporation be avoided. Plastics containers are not suitable.

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4 Apparatus

NOTE — Examples of sampling apparatus are illustrated in annex A. Many different types and variations of apparatus are available, and the dimensions and designs given in the figures are included solely as a guide.

The apparatus required for sampling products other than slab cake falls under the following headings, examples being given in each case.

4.1 Apparatus for sampling from bags : sack-type spears or triers, cylindrical samplers, conical samplers and hand-scoops.

4.2 Apparatus for sampling products in bulk : hand-scoops, cylindrical samplers, conical samplers, mechanical samplers and other apparatus for taking small, periodical increments from a flow of oilseed residues.

4.3 Apparatus for mixing and dividing : dividing instruments, shovels and quartering irons.

5 Time and place of sampling and limitation of the size of lot

5.1 General

Sampling is normally carried out, whether the consignment is in bulk or in sacks, 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 depot, as agreed between the parties concerned. Each lot shall be of mass 500 t or part thereof. Special requirements for bulk transfer are given in 5.2.

5.2 Bulk transfer

It is generally advisable to use the following procedures in the case of the bulk transfer of residues from a ship or barge.

5.2.1 Transfer to lorries and wagons

The increment should be taken either from the flow of product (preferred method) during loading or discharge (particularly for tanker-wagons where internal sampling is not possible), or in the lorry or wagon, as soon as possible after loading, by sampling at at least three or five different positions according to the size of the lorry or wagon (see 6.2.1.2.3), for the purpose of providing one bulk sample per 500 t lot or part thereof.

5.2.2 Transfer to barges

The increments should be selected during loading by sampling from each hold, throughout the duration of loading, for the purpose of providing one bulk sample per 500 t lot or part thereof.

5.2.3 Transfer to silos or depots

The increments should be taken from conveyor belts taking into account the rate of movement of these belts, or preferably by means of an automatic sampler on the transfer circuit, again taking into account the rate of movement, for the purpose of providing one bulk sample per 500 t lot or part thereof.

6 Method of taking samples

6.1 General

Sampling shall be carried out by sampling superintendents appointed by the parties concerned.

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 sea-damaged or otherwise damaged in transit or out of condition, as well as loose[1] material and sweepings which 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 sound material.

6.2 Oilseed residues in the form of meals or agglomerates

6.2.1 Taking of increments

According to the circumstances, the increments shall be taken from products in sacks or in bulk by means of the sampling apparatus mentioned in 4.1 and 4.2, used as described in 6.2.1.1 and 6.2.1.2.

If lumps (agglomerates) have formed, these shall be incorporated in the increments, in approximately the proportions present in the lot.

6.2.1.1 Products in sacks

Increments shall be taken from the number of sacks specified in table 1.

Table 1 — Number of sacks to be sampled

Number of sacks in the lot	Number of sacks to be sampled
up to 10	each sack
11 to 100	10, sampled at random
more than 100	square root (approximately) of the total number, sampled at random according to a suitable sampling plan*

* See, for example, annex B.

1) This term is used to designate material which has leaked from its original container, but is not unduly contaminated.

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If the sacks are open, the increments may be taken using cylindrical samplers, conical samplers or other appropriate instruments.

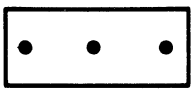
If the sacks (for example jute sacks) are closed, the increments may be taken using sack-type spears or triers.

6.2.1.2 Products in bulk

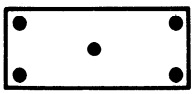
6.2.1.2.1 When sampling takes place while the product is in motion, increments shall be taken across the whole section of the flow, perpendicular to the direction of flow, and at time intervals depending on the rate of flow. If automatic instruments are used, they shall have a slot opening which is at least three times the size of the largest particles.

6.2.1.2.2 When bulk material is sampled in holds during discharge, the increments shall be taken from as many places as possible, excluding the run, and at intervals determined by the rate of discharge.

6.2.1.2.3 If sampling takes place from laden wagons or lorries, the increments shall be taken at three levels at least, with a cylindrical sampler or conical sampler, depending on the product, and at the following points :



lorries up to 15 t: at 3 sampling points (in the centre and at approximately 50 cm from the walls)



lorries or wagons of 15 t and over : at 5 sampling points

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, which, generally, shall be preferred.

6.2.1.2.4 The method to be applied for silos and depots depends necessarily on local conditions.

6.2.2 Preparation of reduced sample

The bulk sample shall be mixed and reduced using the apparatus mentioned in 4.3, until a reduced sample, the size of which depends on the required number of laboratory samples, is obtained. Any lumps shall be separately crushed and remixed with the bulk sample before reduction.

Quartering is carried out as follows. After mixing, the product is divided into four parts, for example using a quartering iron, and two diagonally opposite quarters are discarded and the remainder is mixed again. The dividing and discarding operations are repeated until the sample is reduced to the required size.

6.2.3 Preparation of laboratory samples

The number of laboratory samples to be prepared, by division of the reduced sample, for analysis and arbitration, shall be agreed between the parties concerned. In the absence of such an agreement, the number of laboratory samples should be at least five for each 500 t lot or part thereof.

6.3 Slab cakes

6.3.1 Taking of increments

6.3.1.1 Slab cakes in containers

One cake shall be taken from each of a number of containers (sacks) selected at random, this number not being less than 2 % of the total number of containers in the lot. Care should be taken to avoid taking increment primary samples from the same part of each container.

6.3.1.2 Slab cakes in bulk

Five slab cakes shall be selected at random from each 500 t lot.

6.3.2 Preparation of laboratory samples

Each slab cake taken as specified in 6.3.1 shall be broken into eight pieces of approximately equal size. Each laboratory sample shall be formed by taking one piece of cake, in such a way that corner pieces and centre pieces are represented, and combining the required number of pieces from different cakes to give a laboratory sample of the required size. The number of laboratory samples to be prepared for analysis and arbitration shall be agreed between the parties concerned. In the absence of such an agreement, the number of laboratory samples shall be at least five per 500 t or part thereof.

7 Sizes of samples

7.1 Sound material

The sizes of samples given in tables 2 and 3 are usually suitable. Larger or smaller samples may be required in some cases, according to the tests to be carried out.

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

7.1.1 Residue meals or agglomerates

See table 2.

Table 2 — Sizes of samples of meals or agglomerates

Lot	Increment	Bulk sample	Laboratory sample
t	kg	kg	kg
up to 500	0,1 (minimum)	10 to 50	2 (in bags) or 1 (in tins)

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

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7.1.2 Residue slab cakes

See table 3.

Table 3 — Sizes of samples of slab cakes

Lot t	Increment	Bulk sample	Laboratory sample
up to 500	1 cake	5 cakes	5 × 1/8 cake (approximately 6 kg)

7.2 Damaged material

If the material has been damaged by water, two samples, each of 1 kg, shall be taken and stored in sealed bottles or jars. For material damaged by other means, samples of 2 kg should be taken.

8 Packing and labelling of samples

8.1 Packing of samples

Laboratory samples shall be packed in bags of closely woven cloth, or in polyethylene bags or metal boxes.

Samples for the determination of moisture or other volatile matter, or for any analysis that may be influenced by a change of moisture content, shall be packed in airtight and watertight containers fitted with airtight and watertight closures. The containers shall be completely filled and the closures shall be sealed to prevent loosening or tampering. Plastics containers are not suitable if the sample is intended for the determination of volatile hydrocarbons.

8.2 Labelling of samples

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

8.2.2 Each label shall bear at least the following information :

1) Ship or road vehicle
2) From
3) To
4) Date arrived
5) Quantity
6) Bulk/sacks
7) Goods
8) Identification mark or lot number
9) Number and date of bill of lading or contract
10) Date of sampling
11) Place and point of sampling
12) Sampled by
13) Name of organization responsible for terms of contract

The information recorded on the label shall be permanent.

8.2.3 Labels for samples of damaged material shall also indicate the nature of the damage and the proportion or tonnage so affected.

9 Dispatch of samples

Laboratory samples shall be dispatched as soon as possible, and only in exceptional circumstances more than 48 h after sampling has been completed, non-business days excluded.

10 Sampling report

The sampling report shall make reference to this International Standard and shall indicate the condition of the product sampled, any modifications to the technique described in this International Standard, and all the circumstances that may have influenced sampling.

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Annex A

Examples of sampling apparatus

Dimensions in millimetres

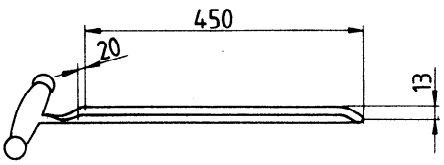

Figure 1 — Sampling spear (open trier)

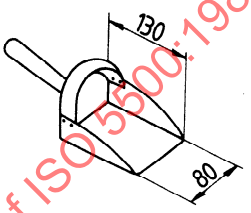

Figure 2 — Hand-scoop

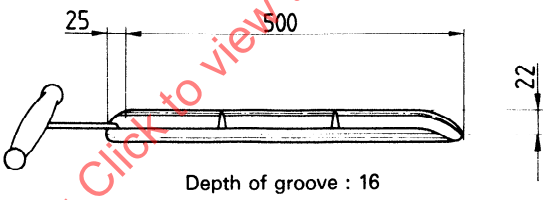

Figure 3 — Divided sampling spear (open trier)

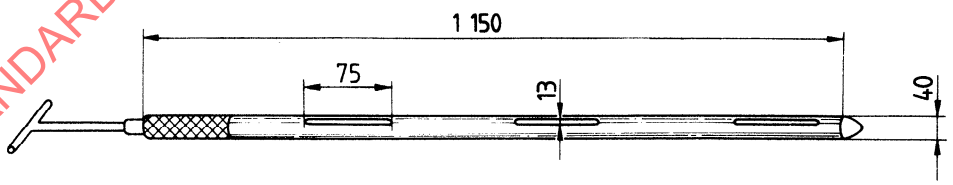

Figure 4 — Cylindrical sampler (divided bulk probe)

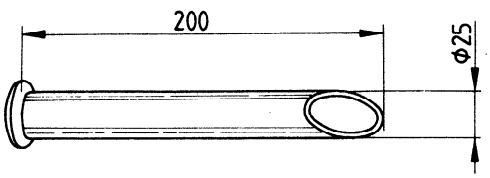

Figure 5 — Running iron (sack-type trier)

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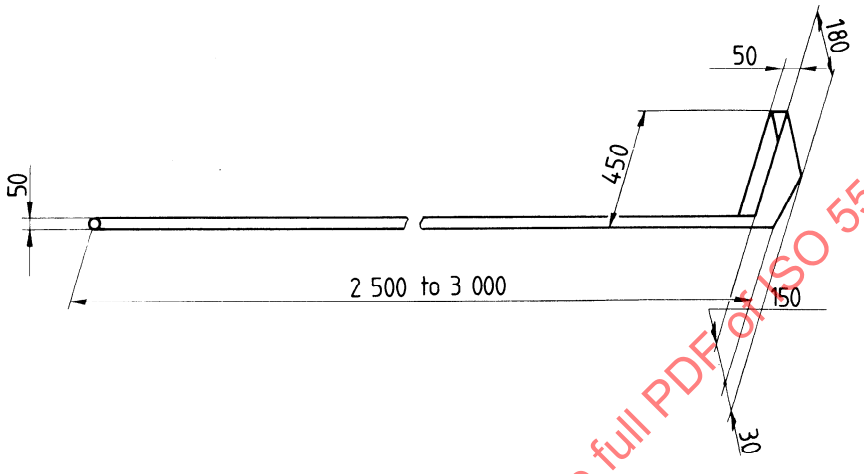

Figure 6 — Falling stream sampler (Pelican type)

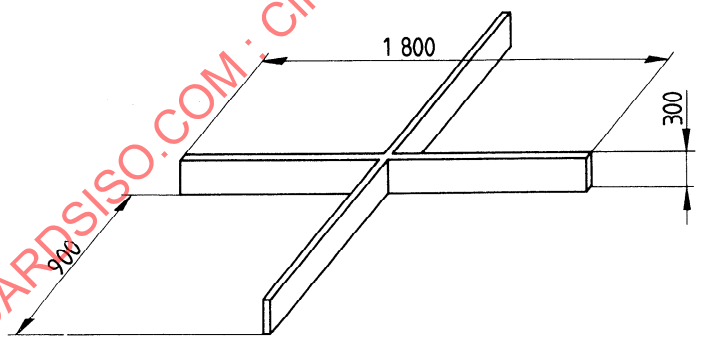

Figure 7 — Quartering irons

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Annex B

Sampling plan for lots comprising more than 100 sacks

For lots comprising more than 100 sacks, the number of sacks to be sampled is approximately the square root of the number of sacks in the lot. The lot shall be divided notionally into a number of groups, each comprising a number n of sacks corresponding to the square root of the number N of sacks in the lot (rounded up to the nearest whole number). For values of N from 101 to 10 000, the number n of sacks making up a group shall be as indicated in table 4. In each of these groups, one sack shall be chosen at random for sampling.

If there are sacks remaining after the lot has been divided into a number n of sacks, one sack shall also be taken for sampling from among those remaining.

In order to verify that the selection is random, it is recommended that the sampler write down the numbers 1, . . . , n, and, during sampling, crosses off each number before selecting for sampling, in the group of n sacks, the sack corresponding to this number.

Example :

The lot comprises 200 sacks (N). For N from 197 to 225 sacks, the number n of each group is equal to 15 sacks. Write down the numbers 1, 2, 3, . . ., 14, 15. Cross out a number, for example 7. Take the seventh sack of the first group of 15 sacks and take a sample from it. Then cross out another number, for example 3, take the third sack of the second group and take a sample from it. Continue in this way until 13 groups of 15 sacks (a total of 195 sacks) have been sampled. The remaining group is less than 15 sacks; take another sack at random. In total, there are therefore 14 sacks ($= n - 1$) sampled from a lot of 200 sacks.

Table 4 — Sampling plan for lots comprising more than 100 sacks

N = Number of sacks in lot; n = number of sacks in group

N	n	N	n	N	n
101 ... 121	11	1 601 ... 1 681	41	4 901 ... 5 041	71
122 ... 144	12	1 682 ... 1 764	42	5 042 ... 5 184	72
145 ... 169	13	1 765 ... 1 849	43	5 185 ... 5 329	73
170 ... 196	14	1 850 ... 1 936	44	5 330 ... 5 476	74
197 ... 225	15	1 937 ... 2 025	45	5 477 ... 5 625	75
226 ... 256	16	2 026 ... 2 116	46	5 626 ... 5 776	76
257 ... 289	17	2 117 ... 2 209	47	5 777 ... 5 929	77
290 ... 324	18	2 210 ... 2 304	48	5 930 ... 6 084	78
325 ... 361	19	2 305 ... 2 401	49	6 085 ... 6 241	79
362 ... 400	20	2 402 ... 2 500	50	6 242 ... 6 400	80
401 ... 441	21	2 501 ... 2 601	51	6 401 ... 6 561	81
442 ... 484	22	2 602 ... 2 704	52	6 562 ... 6 724	82
485 ... 529	23	2 705 ... 2 809	53	6 725 ... 6 889	83
530 ... 576	24	2 810 ... 2 916	54	6 890 ... 7 056	84
577 ... 625	25	2 917 ... 3 025	55	7 057 ... 7 225	85
626 ... 676	26	3 026 ... 3 136	56	7 226 ... 7 396	86
677 ... 729	27	3 137 ... 3 249	57	7 397 ... 7 569	87
730 ... 784	28	3 250 ... 3 364	58	7 570 ... 7 744	88
785 ... 841	29	3 365 ... 3 481	59	7 745 ... 7 921	89
842 ... 900	30	3 482 ... 3 600	60	7 922 ... 8 100	90
901 ... 961	31	3 601 ... 3 721	61	8 101 ... 8 281	91
962 ... 1 024	32	3 722 ... 3 844	62	8 282 ... 8 464	92
1 025 ... 1 089	33	3 845 ... 3 969	63	8 465 ... 8 649	93
1 090 ... 1 156	34	3 970 ... 4 096	64	8 650 ... 8 836	94
1 157 ... 1 225	35	4 097 ... 4 225	65	8 837 ... 9 025	95
1 226 ... 1 296	36	4 226 ... 4 356	66	9 026 ... 9 216	96
1 297 ... 1 369	37	4 357 ... 4 489	67	9 217 ... 9 409	97
1 370 ... 1 444	38	4 490 ... 4 624	68	9 410 ... 9 604	98
1 445 ... 1 521	39	4 625 ... 4 761	69	9 605 ... 9 801	99
1 522 ... 1 600	40	4 762 ... 4 900	70	9 802 ... 10 000	100

For lots comprising more than 10 000 sacks, n corresponds to the square root of N, rounded up to the nearest whole number.

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