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1998-11-01

Animal feeding stuffs — Preparation of test samples

Aliments des animaux — Préparation des échantillons pour essai

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Reference number
ISO 6498:1998(E)

Foreword

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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 6498 was prepared by Technical Committee ISO/TC 34, *Agricultural food products*, Subcommittee SC 10, *Animal feeding stuffs*.

This second edition cancels and replaces the first edition (ISO 6498:1983), which has been technically revised.

Annex A of this International Standard is for information only.

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Animal feeding stuffs — Preparation of test samples

1 Scope

This International Standard specifies methods for the preparation of test samples from laboratory samples of animal feeding stuffs including pet foods.

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 6492, *Animal feeding stuffs — Determination of fat content*.

ISO 6496, *Animal feeding stuffs — Determination of moisture content*.

3 Terms and definitions

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

3.1

laboratory sample

sample representative of the quality and condition of the lot, obtained by reduction of the bulk sample and intended for analysis or other examination

3.2

test sample

representative portion of the laboratory sample, obtained by dividing by means of a sample divider or by hand, if necessary after reduction of the particle size

3.3

test portion

representative portion of the test sample or laboratory sample

4 Principle

For solids, the laboratory sample is thoroughly mixed and divided successively using a specified procedure until a test sample of a suitable size is obtained. A process of crushing, grinding, mincing or homogenizing is used, where appropriate, to ensure that the test sample, from which the test portion(s) will be taken, truly represents the laboratory sample. In the case of a fluid feeding stuff, the laboratory sample is mixed mechanically and a representative test sample is obtained while the fluid is agitated.

5 Apparatus

5.1 Mechanical mill, easy to clean and capable of grinding feeding stuffs without generation of excessive heat and without causing appreciable change in moisture, until the sample passes completely through a sieve of appropriate aperture size (5.5).

A few feeding stuffs are likely to lose or gain moisture. In these cases, it is necessary to apply a correction factor to the results (see 7.2 and clause 8).

NOTE The screen size in the mill is not necessarily the same as the sieve size for checking the extent of grinding.

5.2 Mechanical stirrer or homogenizer

5.3 Mincer, fitted with a 4 mm plate.

5.4 Crushing apparatus, for example, a pestle and mortar.

5.5 Sieves, of aperture sizes 1,00 mm, 2,80 mm and 4,00 mm, made from woven metal wire cloth.

5.6 Dividing or quartering apparatus, such as a conical divider (see Figure A.1), multiple-slot divider with a sorting system (see Figure A.2), or other dividing apparatus that will ensure uniform distribution of the components of the laboratory sample in the test sample.

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

It is essential that the container can be securely closed.

6 Sampling

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 6497 [1].

It is important that the laboratory receive a sample which is truly representative and has not been damaged or changed during transport or storage.

Store the sample in such a way that deterioration and change in composition are minimized.

7 Procedure

WARNING: It is emphasized that care is to be taken to ensure that the apparatus is not a source of contamination.

7.1 Grinding

7.1.1 General

Grinding of some samples may lead to a loss or gain of moisture and, if appropriate, allowance for this should be made (see 7.2 and clause 8). Grinding should be as rapid as possible and exposure to the atmosphere should be minimized. If necessary, first break or crush the pieces to a suitable size for grinding. It is essential that the sample be thoroughly mixed before each stage of the procedure.

7.1.2 Fine samples

If the laboratory sample passes the 1,00 mm sieve (5.5) completely, mix it thoroughly. Divide the mixture successively using the dividing or quartering apparatus (5.6) until a test sample of suitable size is obtained (see 7.9).

7.1.3 Coarse samples

7.1.3.1 If the laboratory sample does not pass the 1,00 mm sieve (5.5) completely, but passes the 2,80 mm sieve completely, mix it thoroughly and prepare a sample of suitable size (see 7.9) by successive divisions as in 7.1.2.

7.1.3.2 Carefully grind this sample in the well-cleaned mill (5.1) as in 7.1.1, until it passes through the 1,00 mm sieve completely.

7.1.4 Coarser samples

7.1.4.1 If the laboratory sample does not pass completely through the 2,80 mm sieve (5.5), carefully grind it in the well-cleaned mill (5.1) until it passes the 2,80 mm sieve completely. Mix it thoroughly.

7.1.4.2 Divide the ground laboratory sample successively by means of the dividing apparatus (5.6) until a test sample of suitable size (see 7.9) for all the determinations required is obtained. Grind this sample in the well-cleaned mill (5.1) until it passes the 1,00 mm sieve (5.5) completely.

7.2 Samples likely to lose or gain moisture

If grinding operations are likely to result in loss or gain of moisture, determine the moisture content by the method described in ISO 6496. Apply the method to the well-mixed laboratory sample as received and to the prepared test sample, so that the results of analyses may be corrected to relate to the sample in its original condition as regards moisture content (see clause 8).

7.3 Samples difficult to grind

If the condition of a laboratory sample not passing through the 1,00 mm sieve (5.5) makes grinding difficult, take a portion immediately after the preliminary mixing described in 7.1.3.1, or after the preliminary grinding procedure described in 7.1.4.1.

Determine the moisture content by the method described in ISO 6496. Dry the sample until, after crushing with the pestle and mortar (5.4) or by other means, it passes the 1,00 mm sieve completely. Again determine the moisture content of the prepared test sample so that the results of analyses may be corrected to relate to the sample in its original condition as regards moisture content (see clause 8).

7.4 Moist feeding stuffs such as canned or chilled pet foods

Homogenize the laboratory sample (which may be the entire contents of a can or other package) using the mechanical stirrer or homogenizer (5.2). Thoroughly mix the homogenized sample. Fill a clean, dry sample container (5.7) and close tightly. Take test portions as soon as possible, preferably immediately. Otherwise store the test sample at between 0 °C and 4 °C.

7.5 Frozen feeding stuffs

Cut or break the laboratory sample into small pieces with a suitable implement. Immediately pass the pieces through the mincer (5.3). Mix the minced sample until any liquid that has separated out is thoroughly re-dispersed throughout the sample. Fill a clean, dry sample container (5.7) and close tightly. Take test portions as soon as possible, preferably immediately, storing the test sample at between 0 °C and 4 °C.

7.6 Feeding stuffs of intermediate moisture content

Slowly pass the laboratory sample through the mincer (5.3). Thoroughly mix the minced sample and immediately pass it through a 4,00 mm sieve (5.5). Fill a clean, dry sample container (5.7) and close tightly.

If the nature of the laboratory sample is such that it cannot be minced, mix and grind it as well as possible by hand.

7.7 Silage and liquid samples

7.7.1 Grass or cereal silage

Pass the entire laboratory sample through the mechanical mill (5.1) if possible, or otherwise chop it as finely as possible. Mix thoroughly and transfer a test sample of at least 100 g to a sample container (5.7).

If the laboratory sample cannot be passed through the mill or cannot be finely chopped, mix it as thoroughly as possible and determine the moisture content by the method described in ISO 6496. Dry the laboratory sample (for example, overnight at between 60 °C and 70 °C in an electrically heated oven, well ventilated) and then pass it through the mechanical mill (5.1). Mix thoroughly and transfer a test sample of at least 100 g to a sample container (5.7). Determine the moisture content of the prepared test sample by the method described in ISO 6496 and apply a correction to all results (see clause 8).

7.7.2 Liquid samples including fish silage

Mix the laboratory sample using a mechanical stirrer or homogenizer (5.2), so that any separated material (ground bone, oil, etc.) is completely dispersed. During agitation, transfer 50 ml to 100 ml to a sample container (5.7) using a ladle, beaker or wide-bore pipette.

7.8 Samples for which there are special requirements

NOTE 1 Some determinations may require special preparation of the test samples. The specific procedures required are described in the relevant section of the test method.

For determinations requiring special degrees of fineness, further grinding may be necessary. In such cases, prepare another test sample as described in 7.1, 7.2 or 7.3, but having the required degree of fineness.

In some cases it may be necessary to avoid breaking or damaging the laboratory sample, for example, for the determination of pellet hardness.

NOTE 2 If the laboratory sample is thought to be non-homogeneous, for example for analytes such as mycotoxins or medicinal additives, it may be necessary to grind the whole of the sample and then reduce the sample size to produce a suitable test portion.

If the sample is fatty, the test sample may be prepared by warming and mixing. In some cases preliminary extraction of fat may be necessary. This shall be carried out in accordance with ISO 6492.

If microbiological examination is required, the sample shall be handled under sterile conditions and in such a way that the microbial condition does not change.

7.9 Size and storage of test samples

Prepare a sufficient test sample for all the determinations likely to be required and not less than 100 g. Completely fill the chosen container (5.7) without delay and close it securely.

Store the test sample under such conditions that changes are reduced to a minimum, paying particular attention to the avoidance of exposure to light and to the effect of temperature.

8 Correction factor

8.1 General

If there is likely to be a loss or gain of moisture during the grinding or mixing operations, it is necessary to use a correction factor to relate the results of analyses to the sample in its original condition as regards moisture content. A similar concept applies if a preliminary extraction of fat is carried out.

8.2 Calculation

Calculate the correction factor f by the equation:

$$f = \frac{100\% - w_0}{100\% - w_1}$$

where

f is the correction factor;

w_0 is the mass fraction of moisture, expressed in percent, of the laboratory sample, determined by the method described in ISO 6496;

w_1 is the mass fraction of moisture, expressed in percent, of the prepared test sample, determined by the method described in ISO 6496.

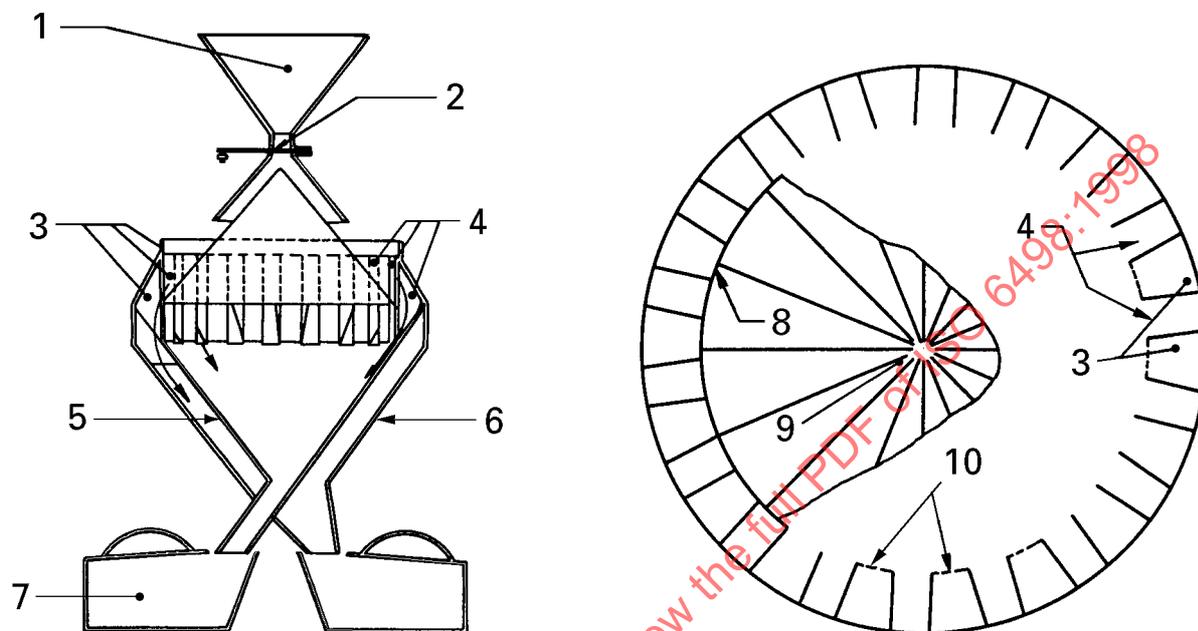
8.3 Correction of results

Multiply the results of analyses by the correction factor f .

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Annex A
(informative)

Examples of dividing apparatus



Key

- | | |
|---------------------------------------|---------------------------------------|
| 1 Hopper | 6 Outer funnel |
| 2 Cut-off | 7 Receptacle |
| 3 Spaces which open into outer funnel | 8 Base of cone |
| 4 Ducts which spout into inner funnel | 9 Peak of cone |
| 5 Inner funnel | 10 Ducts connected below base of cone |

Figure A.1 — Conical divider

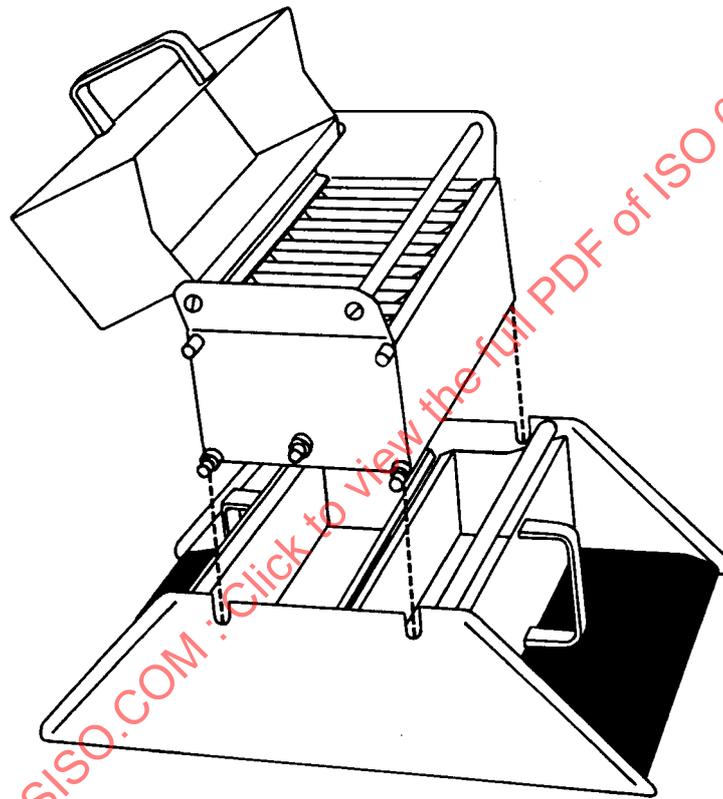


Figure A.2 — Multiple-slot divider with sorting system