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Animal feeding stuffs — Guidelines for sample preparation

Aliments des animaux — Lignes directrices pour la préparation des échantillons

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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 2.

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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 6498 was prepared by the European Committee for Standardization (CEN) Technical Committee TC 327, *Animal feeding stuffs — Methods of sampling and analysis*, in collaboration with ISO Technical Committee TC 34, *Food products*, Subcommittee SC 10, *Animal feeding stuffs*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This third edition cancels and replaces the second edition (ISO 6498:1998), which has been technically revised.

This corrected version of ISO 6498:2012 incorporates the following correction: in 7.1, paragraph 4, the phrase “particle sizes below 4 mm ± 2 mm (4 mm to 6 mm) can be” has been substituted by “particle sizes of 4 mm to 6 mm can be”.

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Animal feeding stuffs — Guidelines for sample preparation

1 Scope

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

NOTE 1 The guidelines mostly derive from those developed by AAFCO (see Reference [7]).

The guidelines are overruled by special instructions and regulations for sample preparation demanded by specific analysis methods.

NOTE 2 Such analysis methods are developed by ISO and CEN.

NOTE 3 This International Standard does not include special guidelines for sample preparation for microbiological analysis of microorganisms like yeasts, bacteria and moulds. Nonetheless, for microorganisms which are used as feed additives (probiotics), some important aspects of sample preparation are addressed.

2 Terms and definitions

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

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2.1 Definitions concerning “sample”

2.1.1
lot
quantity of material that is assumed to be of the same production process and represented by specified sampling rules

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NOTE For the purposes of this International Standard, the rules are those of Commission Regulation (EC) No. 152/2009.[3]

2.1.2
laboratory sample
sample as prepared (from the lot) for sending to the laboratory and intended for inspection or testing

2.1.3
test sample
subsample or sample prepared from the laboratory sample and from which test portions will be taken

2.1.4
test portion
quantity of material drawn from the test sample (or from the laboratory sample if both are the same)

2.1.5
reserve sample
material left over from the laboratory sample when divided or subsampled test samples have been taken and on which no further particle size reduction is done

NOTE If, for example, mycotoxin or genetically modified organism analyses are done on the whole laboratory sample, then the reserve sample is also reduced to the corresponding particle sizes. The reserve sample should be stored under conditions maintaining integrity.

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2.2 Definitions concerning “parameters”

2.2.1
parameter
analyte or constituent or microorganism for which the feeding stuff is to be analysed by microscopic, microbiological, biological or chemical procedures

2.2.1.1
stable parameter
analyte or constituent or microorganism which does not degrade during sample preparation on common handling or storage at room temperatures of 20 °C to 25 °C

2.2.1.2
unstable parameter
analyte or constituent or microorganism which degrades during sample preparation on common handling or storage at room temperatures of 20 °C to 25 °C because they are volatile, degradable, or sensitive to temperature, light, enzymatic degradation or chemical oxidation

NOTE Stability of parameters in this context refers only to the influence of sample preparation, such as intensive grinding, and not to a minimum shelf-life specified by producers or on the label, e.g. for a feed (additive).

Table 1 — Classification (in general) of stable or unstable parameters and reasons for degradation with a view to sample preparation

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Origin	Stable parameters	Unstable parameters	Reason(s) for degradation/change
Nutrients	(Crude) protein, fat, ash, fibre	Moisture	Temperature (volatile)
	Starch, sugar, lactose	Ammonia	Temperature (volatile)
	Gas production and enzyme-soluble organic substance production in *in vitro* tests	Organic acids (e.g. lactic acid, acetic acid, butyric acid, fumaric acid, formic acid)	Temperature (volatile)
	Minerals (e.g. Ca, P, Mg, Na, K, Cl)	Unsaturated fatty acids	Air oxidation (can result in production of short-chain fatty acids)
Feed additives	Trace elements (e.g. Cu, Zn, Mn, Fe, Se, Co)	Vitamins (e.g. vitamin A, C, D, E)	Temperature, ultraviolet (UV) light, air oxidation (sensitive)
	Amino acids (e.g. lysine, methionine, tryptophan)	1,2-Propanediol, ethylene glycol	Temperature (volatile)
	Enzymes (e.g. phytases, non-starch polysaccharide enyzmes)	Microorganisms like probiotics (e.g. *Saccharomyces cerevisiae*, *Enterococcus faecium*)	Temperature (freezing), pressure (sensitive to grinding); moisture/dryness (influences growth of microorganisms)
Undesirable substances	Heavy metals (e.g. As, Pb, Cd, Hg)	Mycotoxins (e.g. aflatoxin B_1, deoxynivalenol, fumonisins, ochratoxin A, T-2 toxin, HT-2 toxin, zearalenone, ergot alkaloids)	Mould growth and change of mycotoxins possible at room temperature; UV light (sensitive – aflatoxin B_1)
	Dioxins and polychlorinated biphenyls (PCBs) with similar effects to dioxins	Drugs, antibiotics, pesticides	Temperature (sensitive)
		Hydrocyanic acid	Temperature (volatile)
Banned substances	Proteins of animal origin	Banned drugs, banned antibiotics	Temperature (sensitive)
(Other) Microorganisms		Yeasts, bacteria, moulds	Temperature (sensitive), dryness, influx of oxygen (anaerobiosis)

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2.3 Examples of animal feeding stuffs characteristics

Some examples of animal feeding stuffs characteristics are given here to assist with the identification and grouping of a laboratory sample based on the terms and annexes used in these guidelines.

NOTE Definitions of animal feeding stuffs are given in legislation worldwide. Sample definitions from European directives and, for straight feeds, in an alphabetical list from a German committee are given in References [4][5][6][8].

2.3.1
birdseed
seeds that are intended to feed birds

EXAMPLES Grains and oilseeds.

2.3.2
whole cottonseed
unprocessed cottonseed product, including the hulls, lint, and meat

2.3.3
mineral mix
supplementary feed that mainly consists of mineral ingredients in either granular, bead or small pellet form and which is free flowing as an entire mix

NOTE Mineral pellets are an agglomerated mineral mix formed by a mechanical process (in general).

2.3.4
dry feeds
feed ingredient or complete animal feed which typically contains a moisture mass fraction of not more than 15 %

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NOTE Dry feed pellets are an agglomerated dry feed produced by a mechanical process (in general).

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2.3.5
green fodder
edible parts of plants, other than separated grain, that can provide feed for grazing animals or that can be harvested for feeding, including browse, herbage, and mast

NOTE Generally, the term refers to more digestible material in contrast to less-digestible plant material, known as roughage.

2.3.6
silage
forage preserved in a succulent condition by organic acids produced by anaerobic fermentation of sugars in the forage

2.3.7
roughage
fibrous, coarsely textured parts of plants

EXAMPLES Stovers, straws, hulls, cobs, and stalks.

2.3.8
hay
aerial portion of grass especially cut and dried for animal feeding

2.3.9
haylage
forage preserved in a succulent condition by organic acids produced by anaerobic fermentation of sugars in the forage with a moisture mass fraction of about 45 %

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2.3.10
total mixed ration
TMR
single mixture of all feed ingredients (forages, grains, and supplements) that is supplied to an animal for a 24 h period

NOTE In practice, the 24 h allotment of the mixture may be offered in one or more feedings.

2.3.11
byproduct
product which remains after processes for the production of ingredients from plant material

EXAMPLE Dried distillers grains with solubles (DDGSs) from fermentation.

2.3.12
oilseed
any seed from which oil is extracted

EXAMPLE Sunflower seeds.

2.3.13
large block feed
molasses block feed
agglomerated feed compressed into a solid mass that is cohesive enough to hold its form

NOTE Large block feed weighs over 1 kg, generally about 20 kg. It may be marketed as a mineral block or a “caramelized” molasses drum, containing various minerals and nutrients. Samples may be received by the laboratory as large chunks, cores or “sticky clumps”.

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2.3.14
liquid feed
feed product not solid and not aeriform

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NOTE A liquid feed contains sufficient moisture to flow readily and may contain molasses.

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2.3.15
canned pet food
feed product for pets which has been processed, packaged, sealed and sterilized for preservation in cans or similar containers

2.3.16
semi-moist feed
meat-based feed product for pets or aquatic animals that has been partially dried to prevent microbial decomposition

NOTE The moisture mass fraction may range from 15 % to 40 %. The product is generally in the form of strips or cubes and is designed to be stored at room temperature.

2.3.17
dog chew
rawhide bone
meat and skin or peel strip that has been nearly completely dried to a leather-like consistency

2.3.18
premixture
mixture of one or more micro-ingredients with diluent or carrier

NOTE Premixtures are used to facilitate uniform dispersion of the micro-ingredients (e.g. vitamins, probiotics, drugs or antibiotics) into a final feed.

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2.3.19
range and alfalfa hay pellet
agglomerated feed formed by compacting and forcing the mix through, for example, square openings by a mechanical process

NOTE The pellets are mostly about 2 cm in diameter and 5 cm in length (volume about 16 cm^3) and may contain molasses; this definition also applies to alfalfa cubes (chopped alfalfa hay) of larger dimensions.

2.3.20
texturized feed
sticky feed
mix of assorted grains and commercial feed (generally pelleted), all of which has been treated with a coating of, for example, molasses

NOTE Some of the grains may have been steam heated or rolled prior to incorporation into the texturized feed.

2.3.21
aquatic feed
feed which is fed to aquatic animals and which has been mechanically processed into encapsulated pellets, flakes, crumble, and as packaged sealed powder

2.4 Definitions concerning “sample preparation procedure”

2.4.1
homogeneity
degree to which a property or a constituent is uniformly distributed throughout a quantity of material

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NOTE Homogeneity may be considered to have been achieved in a practical sense when the sampling error of the processed portion is negligible compared to the total error of the measurement system. Since homogeneity depends on the size of the units under consideration, a mixture of two materials may be inhomogeneous at the molecular or atomic level, but sufficiently homogeneous at the particulate level. However, uniform visual appearance does not ensure compositional homogeneity.

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2.4.2
partial drying
part of the sample preparation procedure for feedstuff samples with a high moisture content (dry mass fraction <85 %), in which the sample is carefully dried to allow subsequent sample preparation procedures to be applied, such as particle size reduction by grinding with a mill

NOTE 1 The partial drying procedure depends on the feeding stuff [e.g. at temperatures below 55 °C to 60 °C for silages], and on the heat stability of the parameters (e.g. 70 °C ± 10 °C for drugs and antibiotics).

NOTE 2 Samples for microbiological analysis should not be dried (at temperatures above 40 °C).

NOTE 3 Partial drying can also be achieved by a freeze-drying procedure, which is a careful drying process using a vacuum to allow moisture to evaporate.

2.4.3
coarse grinding
first grinding step of the whole sample when the laboratory sample contains large lumps or when its particle size is above about 6 mm before mass reduction

NOTE Coarse grinding is a special kind of particle size reduction that ensures homogeneity of the laboratory sample for subsampling purposes.

2.4.4
mass reduction
part of the sample preparation procedure to reduce the mass of a laboratory sample by dividing or subsampling it using (stationary or rotary) dividers or fractional (alternate) shovelling, without changing the consistency of the sample

NOTE After mass reduction, all subsamples should have the same properties as the original laboratory sample.

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2.4.5
particle size reduction
part of the sample preparation procedure achieved by chopping, crushing, cutting, blending (homogenizing), macerating, milling (grinding), pressing, pulverizing to obtain a homogeneous test sample for further analysis

NOTE In general, particle size reduction follows the mass reduction step of the sample preparation procedure with different sieve size options to ensure integrity of the test sample(s).

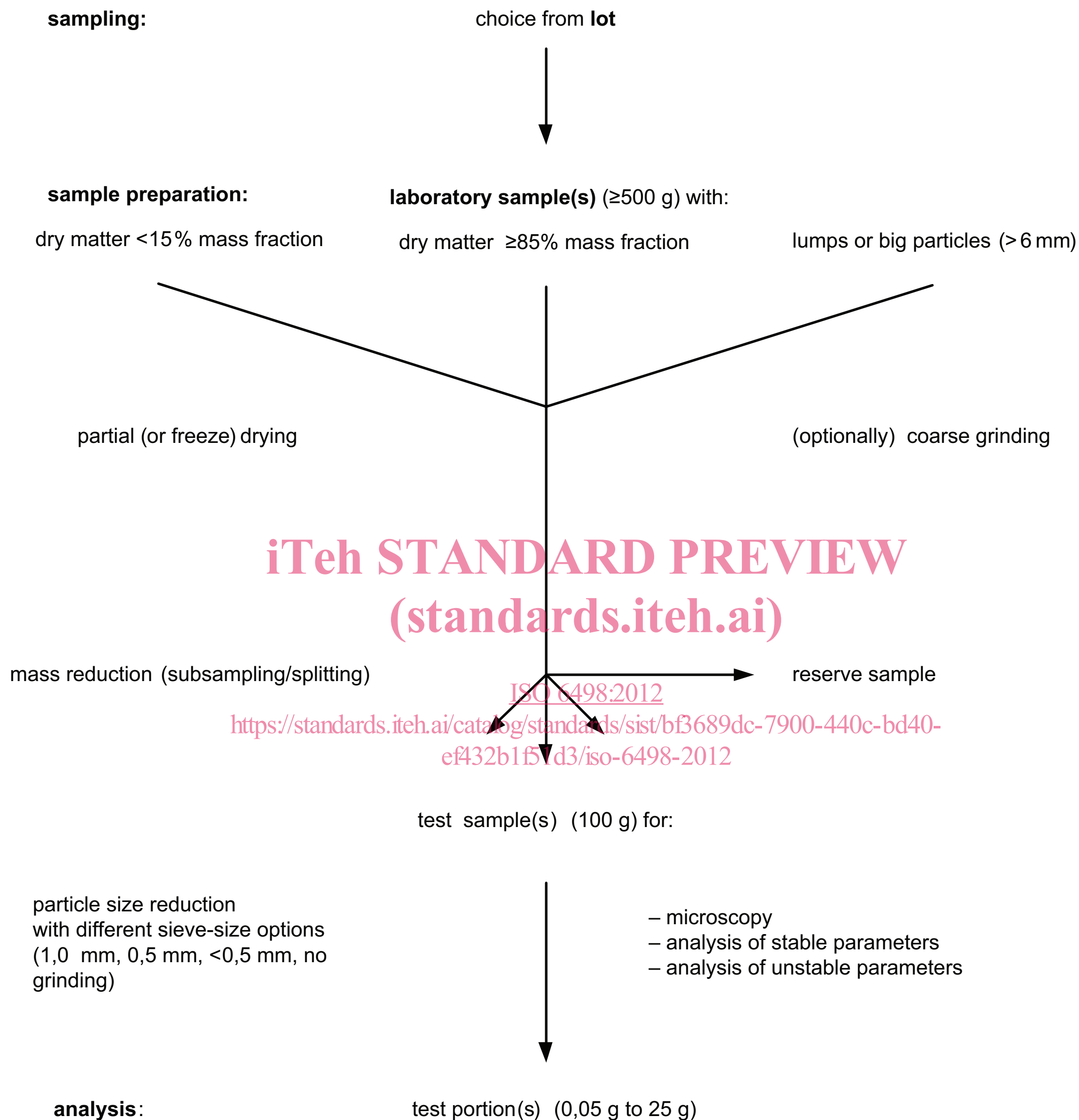

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Figure 1 — Illustration of definitions concerning "sample", "substances" and "sample preparation procedure"

3 Principle

All sample preparation steps depend on the different properties of the feedstuffs and on the parameters to be analysed. In each case, any special instructions concerning sample preparation in the analysis methods require consideration.

The guidelines describe the procedure for preparing — from a sample arriving at a laboratory (in general with a minimum mass of 0,5 kg) — a homogeneous test sample (having a minimum mass of 100 g) with the same constitution and composition and free from contamination.

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In some cases, the laboratory sample size can be less than 500 g (i.e. in standards for feed additives), but it is necessary to follow statutory regulations and, in every case, the sample size should be large enough to be representative.

In general, the whole laboratory sample is reduced in mass and in particle size to obtain one or more test samples for the analysis of stable and unstable parameters, for microscopy analysis and for reserve. If the analysis protocol and the intended proceeding of the reserve sample permit it, the laboratory sample should preferably be pre-ground completely to an adequate coarse particle size before being reduced further, in order to ensure homogeneity of the test samples.

From a test portion (0,05 g to 25 g and above) prepared for weighing in the feedstuff analysis, representative results should be achieved on the laboratory sample and finally on the whole lot from which the sample was drawn.

Consequently, all steps for sample preparation should be performed quickly, under convenient and very clean conditions, so that there can be no degradation of sensitive analytes, no contamination and no oxidation due to the influence of excessive temperature, daylight, air or residues on the apparatus used or from the samples prepared previously or simultaneously. In particular, contamination from sample to sample should be prevented.

A loss or a change of moisture mass fraction ("content") during sample preparation should be avoided. In any case, it is necessary to take into account that, in order to be suitable for official control, results require correction (to origin moisture content, dry mass fraction 88 % or 100 %).

For feedstuffs with a higher moisture content (dry matter <85 % mass fraction), partial drying or freeze-drying before mass reduction can be necessary.

For feedstuffs with lumps or particle sizes >6 mm, coarser grinding of the whole laboratory sample to a particle size of <6 mm before mass reduction or subsampling is absolutely necessary.

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The samples have to be stored at every stage of the sample preparation under adequate conditions (e.g. at room temperature, refrigerated, frozen, in an airtight container, protected from light or in the dark) to maintain their integrity.

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For microbiological analyses, all sample preparation steps need to be done under aseptic conditions. Laboratory samples should be neither frozen nor heated (>40 °C), nor subjected to vacuum or oxygen levels higher than those present in atmospheric air.

4 Consideration of sample preparation errors

Sample preparation steps have been shown to be some of the largest sources of laboratory error, a fact which is generally overlooked. This type of error can prove much larger than that arising from subsequent analytical procedures.

4.1 Subsampling and other errors

4.1.1 General

Errors deriving from sample heterogeneity may add to the total subsampling error (TSE) on two levels (Reference [12]).

4.1.2 Constitutional heterogeneity

On a first level, constitutional heterogeneity is a measure of the fact that not all the particles of the laboratory sample have the same composition (shape, size, density, etc.). If a large overall difference between the individual fragments exists, the constitutional heterogeneity is large, but if the fragments are more homogeneous, constitutional heterogeneity is lower. The total contribution to heterogeneity is never zero, however, as that would imply that all fragments are strictly identical. Mixing and blending does not change constitutional heterogeneity. The only ways to alter the constitutional heterogeneity of any given material are by comminution (crushing or cutting) or other methods which alter the physical properties of a sample. The reduction of the average grain size is the dominant factor in reducing constitutional heterogeneity by such means.

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Therefore, an initial coarse grinding (pre-grinding) of the whole laboratory sample is necessary before subsampling or division to reduce constitutional heterogeneity.

This fundamental subsampling error (FSE) can be controlled by selecting the test sample mass (see 4.2) appropriately. Therefore, collect enough mass to ensure that particles of all different compositions are contained in the subsample or division. The larger the particle size of a material, the larger the subsample mass has to be to minimize error.

4.1.3 Distributional heterogeneity

On a second level, distributional heterogeneity is a measure of the non-random distribution of particles in the sample, as a result mainly of the action of gravitational force on particles of different densities, sizes and shapes, which leads to a grouping and segregation of all particles. Particles with large differences in size or density tend to segregate or stratify heavily, with the smallest or densest particles sinking to the bottom of the sample. For the sake of illustration, imagine a laboratory sample consisting of black and white spheres and with significantly different grain size distributions. If all the black spheres are found at the bottom of the sample and the white spheres are more to the top, the system displays a very high distributional heterogeneity. If, on the other hand, the spheres were well mixed (homogenized), the distributional heterogeneity of the system would be significantly reduced.

To reduce this grouping and segregation error (GSE), mix or blend the sample before subsampling and collect many increments at random from the laboratory sample (see 4.3).

Mixing is not adequate for many materials. For some materials and circumstances, mixing may actually increase segregation instead of reducing the grouping and segregation error. As long as gravity exists, there will be segregation. Many materials always display an innate propensity for segregation, even immediately after mixing suspensions, e.g. highly density-fractionated materials. Such systems require constant monitoring and treatment but, once this feature has been duly recognized, it can always be dealt with satisfactory.

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Incrementing (i.e. the collection of many random increments from the laboratory sample to make up the subsample or division) always works by reducing error from distributional heterogeneity and takes less time and equipment to implement. Thirty increments are generally adequate. More increments are required for very heterogeneous materials and, if little segregation is known to exist, fewer increments can be used, but in no case can fewer than 10 be recommended.

4.1.4 Other errors

Other errors that arise from sample preparation include the loss or gain in analyte content arising from such mechanisms as grinding, excessive heat, loss of fines, contamination, and electrostatic separation. These errors can be large and are usually a result of carelessness or lack of knowledge.

4.2 Minimum mass

To be properly representative of a laboratory sample, the subsample or division shall have adequate mass with a view to fundamental subsampling error (FSE) and maximum particle size (“minimum mass”) (see Table 2).

The mass required depends on the acceptable error in the subsample or division, on the density, heterogeneity, and content of the analyte particles in the matrix, and on the largest particle size (see calculations in Annex A, Examples 1 to 3 and Tables A.1 to A.3).

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Table 2 — Minimum mass: expected coefficient of variation (CV) from laboratory subsampling; assumed density, 1 g/cm^3

Maximum particle size mm d	**FSE (expected CV)** %				
	15	10	5	2	1
	Minimum mass g				
0,5	0,06	0,13	0,5	3	12,5
0,75	0,2	0,4	2	10,5	42
1	0,4	1	4	25	100
2	4	8	32	200	400
5	56	125	500	3 130	12 500

NOTE For materials with densities other than 1 g/cm^3, the entries can be multiplied by the density of the material of interest; for example, the subsampling of a material with a largest particle size of 2 mm, a tolerable subsampling CV of 5 % and a density of 0,5 g/cm^3 would require 16 g.

4.3 Errors associated with division techniques

The data in Table 3 demonstrate the error associated with various division techniques for a model mixture of sand particles. Figure 2 demonstrates the representativity (i.e sum of the sampling error related to precision and accuracy) of 17 different mass reduction devices for a model mixture containing mass fractions of 89,9 % wheat, 10,0 % rapeseed, and 0,10 % glass (see References [11][12]). The primary difference in the mass reduction methods is the number of increments selected. For this to be true, structurally correct use of the mass reduction devices is required (e.g. equal probability for the selection of all particles, no loss of particles, centre of gravity rule obeyed, parallel cuts) which is difficult or impossible to obtain with shovelling and grab sampling methods. Therefore mass reduction methods based on grab sampling or shovelling methods can have substantial problems with precision and accuracy on trace components present as separate particles, which may be due to selective loss or poor sampling of smaller particles (see References [11][12]). It can be concluded from Table 6 and Figure 2 that more increments lead to improvements for the mass reduction in the laboratory by reducing the sampling error. In general, a rotational divider reaches several hundred increments, a stationary riffle divider about 10 to 34 increments, and coning and quartering only two increments. Therefore, coning and quartering are not recommended in the critical mass reduction step in the laboratory, i.e. the mass reduction step with the largest contribution to the total error. The preparation of the final test portion, where the ratio between the mass of the laboratory sample and the mass of the final test portion is 100 to 10 000, can usually be considered the critical step of the mass reduction of the laboratory sample. Grab sampling is to be totally avoided for the critical mass reduction step unless it has been established that the sampling error is insignificant compared to the total analytical error.

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Table 3 — Test results from division of a mixture containing mass fractions of 60 % coarse sand with 40 % fine sand, P = 0,6 (ISO 664[1])

Method[a]	**Number of increments**	**Standard deviation of samples** % s_r	**Variance** %^{2} s_r^2	**Estimated maximum sample error** %
Coning and quartering	2	6,81	46,4	22,7
Stationary riffling	10 to 12	1,01	1,02	3,4
Rotary riffling	>100	0,125	0,016	0,42
Random variation		0,076	0,005 8	0,25

[a] Stationary rifflers with a higher number of increments and less subsampling error are available (see Reference [11]).

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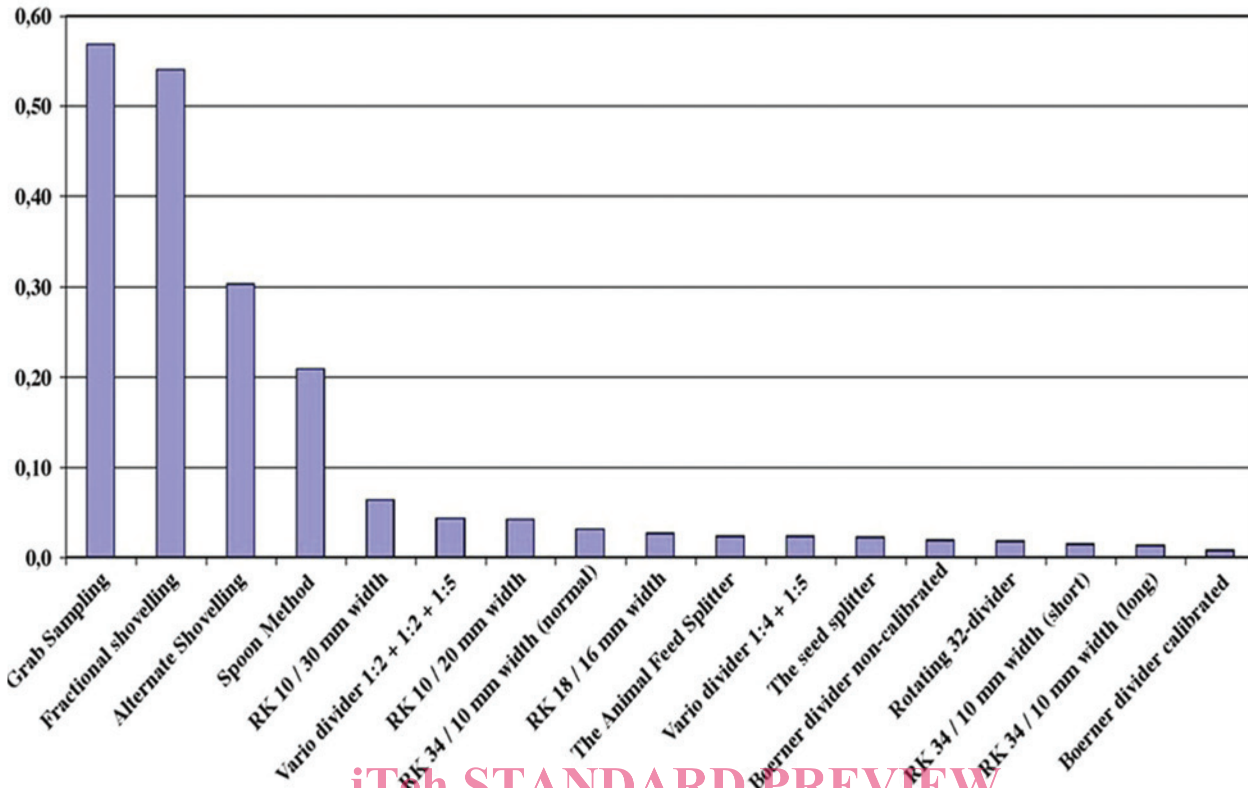

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NOTE Representativity should be as low as possible. Higher sums thus mean lower reliability. RK n indicates a riffle divider with n chutes (see Reference [11]).

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Figure 2 — Pooled representativity, r^2, equal to the square of the bias plus the square of the precision, for a model mixture of wheat, rapeseed, and glass

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5 Safety precautions

The mills for crushing, cutting and grinding have sharp moving blades. Never put hands or fingers past the edges of the introduction chamber. Never open the mills until they have completely stopped. Check to see that safety interlocks on all equipment are operating properly.

Wear appropriate personnel protective equipment as required in the laboratory. Safety is of great importance during the sample preparation phase of the analysis.

Operate the dust ventilation system during dust generation procedures. To minimize dust, use a vacuum cleaner to clean the hood area, mills, and work area.

Check that all electrical equipment is properly earthed and maintained. Do not place metal items or aluminium foil in the microwave oven when using it for drying samples.

6 Apparatus

Usual laboratory equipment and in particular the following. All equipment used should be appropriate to the risk of contamination and oxidation during sample preparation.

6.1 Equipment for sample preparation in general.

6.1.1 Brushes for cleaning grinders, etc.

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6.1.2 Compressed air blower for cleaning.

6.1.3 Vacuum cleaner.

6.1.4 Systems for microbe reduction of mills, **equipment for disinfection** and **flame treatment** for microbiological analysis.

6.2 Drying systems.

6.2.1 Lyophilization system, forced-air drying oven capable of being maintained at 55 °C ± 5 °C or **microwave oven**, household type, or **vacuum oven**.

6.2.2 Moisture dish (pan) made of plastics, aluminium or glass, e.g. with ≥50 mm diameter, ≤40 mm deep.

6.3 Equipment for mass and particle size reduction of "wet" feeds (e.g. forages, silages).

6.3.1 Garden pruning clippers for cutting forages or a **paper cutter** for small sample volumes or a **laboratory forage chopper** for large volumes and a **ceramic cutter**, especially when trace elements are of interest.

6.3.2 Cutting mill with 6 mm and 1 mm screens.

6.3.3 Shearing-type mill with forage head and 1 mm screen.

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6.3.4 Riffle sample divider, the minimum chute width shall be at least $2d + 5$ mm, where d is the diameter of the largest particle.

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https://standards.iteh.ai/catalog/standards/sist/bb5689de-7900-4406-bd46-ef432b1f51d3/iso-6498-2012

6.3.5 Sterile cutter or **disinfected mill** when microbiological analysis (e.g. of probiotics) is of interest.

6.4 Equipment for mass and particle size reduction of "dry" feeds (e.g. cereals, mineral mixtures, pelleted feeding stuffs).

6.4.1 Riffle divider.

6.4.2 Rotary divider with vibratory feeder.

6.4.3 Shearing grinding mill equipped with 1,0 mm, 0,5 mm and <0,5 mm sieves.

6.4.4 Cutting mill with 4 mm to 6 mm screens.

6.4.5 Shearing blending mill (e.g. household coffee mill).

6.5 Equipment for the storage of samples.

6.5.1 Sterile bottles with airtight lids (e.g. brown glass bottles for unstable parameters like vitamins) and especially for microbiological purposes.

6.5.2 Wide-mouth bottles with screw cap, plastic.

6.5.3 Plastic bags with low microbe content, with an airtight closure or for setting to vacuum for microbiological purposes.

6.5.4 Refrigerator.

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