

**GUIDANCE DOCUMENT FOR THE IMPLEMENTATION OF COMMISSION
REGULATION (EU) No 691/2013 OF 19 JULY 2013 AMENDING REGULATION
(EC) No 152/2009 AS REGARDS METHODS OF SAMPLING AND ANALYSIS**

**ENDORSED BY THE STANDING COMMITTEE ON THE FOOD CHAIN AND
ANIMAL HEALTH SECTION ANIMAL NUTRITION AT ITS MEETING ON 16-17
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IMPORTANT DISCLAIMER

**This document has no formal legal status and, in the event of a dispute,
ultimate responsibility for the interpretation of the law lies with the Court of
Justice**

NOTE

This document is an evolving document and might be updated to take account
of the experience gained by the competent authorities in the implementation
of Regulation (EU) No 691/2013 or this guidance document

SCOPE

The sampling provisions provided for in Commission Regulation (EU) 691/2013
do not cover the sampling of feed to control the presence of micro-organisms
and consequently this guidance document do also not cover the sampling of
feed to control the presence of micro-organisms.

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PART A. GUIDANCE ON SPECIFIC ASPECTS OF THE SAMPLING PROCEDURE

I Sampling and sample preparation:

The lot (or sampled portion) shall be sampled by repeatedly taking incremental samples at various single positions in the lot (or sampled portion). These **incremental samples shall be combined by mixing to form an aggregate sample from** which representative final samples shall be prepared by representative dividing:

- one aggregate sample per sampled portion (or lot)
- from each aggregate sample at least two final samples are taken – in case also a third final sample is taken for reference, it has to be prepared from the same aggregate sample. The final samples (for control, defence and reference) shall be prepared of approximately the same amount of weight.

a. Control of constituents or substances uniformly distributed

The aggregate sample can be reduced to at least 2 kg (3kg for the control of the presence of pesticide residues in pulses, cereal grains and tree nuts) **preferably** using a mechanical or automatic divider (insofar the nature of the feed enables the use of an divider). From the reduced sample the final samples shall be taken. The final sample is completely homogenised (grinding) and after homogenisation of the final samples, the test aliquot is taken for analysis. Completely homogenised by grinding means that the homogenised sample passes a sieve with mesh size of 1mm.

b. Control of substances or constituents likely to be distributed non-uniformly

- the aggregate sample shall be homogenised, by grinding **or** by mixing, and from the homogenised aggregate sample, the final samples are taken

or

- the aggregate sample is reduced to at least 2 kg making use of a mechanical or automatic divider (only exception in case the nature of the feed does not enable the use of a divider). The reduced sample is to be homogenised by mixing or by grinding.

Grinding cannot be applied in case of examination by visual inspection or by microscopy. After homogenisation, the final samples are taken from the homogenised reduced sample. The final samples are completely homogenised by grinding and from the homogenised final samples, the test aliquot is taken.

II Guidance on sampled portion (not applicable to roughages/forage)

Disclaimer: The sampling inspector can deviate from this guidance on sampled portion if appropriate as the determination of the size of the sampled portion is the responsibility of the inspector.

- In the case of relative small lots (<50 tons) and all parts of the lots are accessible (static/dynamic), it is advised to sample the whole lot (sampled portion = lot)
- In the case of larger lots (50 – 500 tons)
 - For the control of substances homogeneously distributed throughout the lot, a portion of at least 50 tons can be sampled
 - For the control of substances heterogeneously distributed throughout the lot, it is advised to sample the whole lot insofar all parts are accessible (sampled portion = lot)
- In the case of large lots (> 500 tons)
 - For the control of substances homogeneously distributed throughout the lot, a portion can be sampled (at least 100 tons insofar accessible)
 - For the control of substances heterogeneously distributed throughout the lot, it is advised to sample at least 10 % of the lot with a minimum portion of 500 tons, insofar accessible
 - If only a portion of a lot of feed is sampled, it shall be presumed that all of the feed in the lot is so affected, unless following a detailed assessment there is no evidence that the rest of the lot fails to satisfy the EU requirements. The way how the detailed assessment has to be performed is to be agreed in advance with the competent authorities. An example of detailed assessment is to subdivide the lot into physically separated sublots of e.g. 500 tonnes and to sample and analyse the sublots separately to separate out the compliant from the non-compliant sublots.

III. Guidance on assistance/co-operation from feed business operator to enable the inspector to perform the sampling correctly in acceptable conditions

Feed business operators have to provide assistance to the inspector to enable the inspector to perform the sampling correctly and under acceptable conditions.

Exact guidance is difficult to provide as it will largely depend on the ad hoc situation. The assistance can consist of:

- Unloading of the lot
- Placing the pallets with bags as such that they are accessible from all sides
- Providing a sheltered place for sampling
- Separate the portion to be sampled (selected by the official inspector) from the rest of the lot
- ...

PART B. GUIDANCE ON SAMPLING OF LARGE BATCHES /LOTS – SILO'S

1. Sampling equipment

There are many different types of sampling equipment or devices. The most suitable equipment should be chosen taken into account the product to be sampled, the quantity required and the containers to be used.

Examples of equipment (non-exhaustive) to sample flowing feed and static lots are provided for in Annex to the EN-ISO 24333-2009 standard.

2. Sampling equipment and incremental samples

When sampling with a spear (sampling probe/sampling device) with several apertures/openings, the feed collected by a single aperture/opening can be considered as one incremental sample on the condition that in each aperture/opening the minimum quantity of incremental sample size is collected. Also a maximum of 1 incremental sample per 0.5 m length of spear can be accepted.

Examples:

Spear of 1 m with 2 apertures/openings, collected quantity 100-150 g per aperture/opening = 2 incremental samples

Spear of 2 m with 1 aperture/opening, collected quantity 200 g = 1 incremental sample.

Spear of 2 m with 4 apertures/openings, collected quantity 100-150 g per aperture/opening = 4 incremental samples

Spear of 6 m with 4 apertures/openings, collected quantity 250 g per aperture/opening = 4 incremental samples

Spear of 3 m with 8 apertures/openings, collected quantity 100-150 g per aperture/opening = 8 incremental samples

Vacuum spear of 9 meters length, sample taken over the complete length: 18 incremental samples.

3. Sizes of bulk shipments and large batches in storage

Bulk shipments are transported in vessels – the size of the vessels can vary from 500 t to cape size vessels of more than 90.000 t. The holds also vary depending on the constitution of the vessel itself.

Some examples of **transport by ship**:

- Panamax for about 60.000 metric tonnes in 7 up to 9 holds
- Handy-Max for about 35/45.000 metric tonnes in 5 up to 7 holds
- Handy for about 15/25.000 metric tonnes in 3 to 5 holds
- Coaster for about 2/5.000 metric tonnes in 2 to 3 holds
- River barges for 500 up to about 2.500 metric tonnes in 1 to 2 holds.

Consequently one hold can be from 2 up to 10/12 meters deep.

Storage:

Storage facilities (warehouses and silos) vary also, as they depend on the material they are destined to contain – there are differences of storage facilities between e.g. grains and flour because it depends on the flowing of the material stored.

- Flat-(horizontal) warehouses have a size of 15/20 meters x 40 or 60 or 80 meters length or even more and cereals are stored 4 meters (but very often more) high.
- Vertical silos (metallic or concrete) can have storage capacity between 500 to 20.000 tonnes per cell with 4 to 50 cells in the same building and the height can go from 10 to 50 meters.

4. General principles when sampling large batches

In case the mode of transport or storage of a batch does not enable to take incremental samples across the whole batch, sampling of such batches should preferably be done when the product/batch is in flow or in cases where it is feasible, the lot to be sampled should be moved to another silo, ... to enable sampling across the batch.

In the case of large warehouses destined to store feed, operators should be encouraged to install equipment in the warehouse enabling (automatic) sampling across the whole stored batch.

5. Sampling of batches transported by ship**5.1. Sampling of batches transported by ship by dynamic sampling**

The sampling of large batches in ships is carried out while the feed is in flow (dynamic sampling).

The sampling has to be done per hold (entity that can physically be separated). However to keep the balance of the ship, holds are emptied partly one after the other so that the initial physical separation does no longer exist after transfer into silos. So sampling can be performed in function of the initial physical separation or in function of the separation after transfer into storage.

The unloading of a ship of feed can take several days: 50.000 tonnes with an unload capacity of 100 to 750 tonnes an hour can take 65 hours (3 days) to 500 hours (20 days).

Even if the sample is taken automatically, the presence of an inspector is necessary. Therefore it is not (always) feasible or appropriate (from resources and cost point of view) for an inspector to have to be present during the whole operation of unloading. Therefore sampling is allowed to be undertaken on a portion of the lot to be sampled and the result is considered representative for the sampled portion of the lot. It is presumed that all of the feed in that lot is so affected, unless following a detailed assessment there is no evidence that the rest of the lot fails to satisfy the EU requirements.

Example

Batch of 10.000 tonnes

Unloading speed is 500 tonnes an hour: total unloading time = 20 hours

Inspector decides to sample only part of the batch. He decides to sample 1000 tonnes which means that the sampling time is 2 hours.

The number of incremental samples is determined taking into account the size of the sampled portion of the whole batch and the substance to be controlled.

Control for non-uniformly distributed substances/products:

The sample must consist of 132 ($100 + \sqrt{1000} = 132$) incremental samples of 100 grams, resulting in an aggregate sample of 13.2 kg.

This means that an incremental sample must be taken every 54 sec. or every 7.5 tonnes

Control for uniformly distributed substances/products

The sample must consist of 72 ($40 + \sqrt{1000} = 72$) incremental samples of 100 grams, resulting in an aggregate sample of 7.2 kg.

This means that an incremental sample must be taken every 100 sec. or every 13.8 tonnes

5.2. Sampling of batches transported by ship by static sampling

In situations where the sampling is done in a static way, the same procedure as foreseen for silos accessible from above has to be applied, this means the **length** of the spear (sampling probe) to be used for the sampling of static batch/consignment stored in hold should be sufficient to sample at least a significant portion of the lot.

There has to be a representative sampling of the accessible part (from above) of the consignment/hold performed.

Example

Hold of 20 m length and 20 m width and 10 m depth = $4000 \text{ m}^3 = 3000 \text{ tonnes}$
Insofar accessible from above with a spear of 1.5 m, the accessible part is $600 \text{ m}^3 = 450 \text{ tonnes}$.

The number of incremental samples is determined taking into account the size of the sampled portion of the whole batch and the substance to be controlled.

Control for non-uniformly distributed substances/products:

The sample must consist of 100 incremental samples of 100 grams, resulting in an aggregate sample of 10 kg.

If the spear has 3 openings/apertures, the sample must be taken on at least 34 sampling points.

Control for uniformly distributed substances/products

The sample must consist of 40 incremental samples of 100 grams, resulting in an aggregate sample of 4 kg.

If the spear has 3 openings/apertures, the sample must be taken on at least 14 sampling points.

6. Sampling of large batches stored in warehouses

See point 4

The **length** of the spear (sampling probe) to be used for the sampling of static batch/consignment stored in warehouse should be sufficient to sample at least a significant portion of the lot.

A representative sampling of the accessible part of the consignment has to be performed.

The analytical result of this sample is decisive to determine the compliance/non-compliance of the whole batch. It is presumed that the rest of the batch is equally affected unless, following a detailed assessment (in particular based on results provided by the feed business operator), there is no evidence that the rest of the lot fails to satisfy the EU requirements.

Example 1:

Cereals stored at warehouse 30 m large – 50 m deep – 4 m high = 6000 m³ = about 4500 tonnes accessible from 1 side (30 meter side)

- **Possibility to sample with spear of 2m:** 30 m x 2 m x 4m = 240 m³ = about 180 tonnes As the sampled is not 10 % of the total lot size, the possibility of the use of a longer spear (of about 5 metres) must be considered.

For the control of non-uniformly distributed substances/products

100 incremental samples of 100 grams – resulting in 10 kg sample

(if the spear has 4 apertures/openings: the batch has to be sampled at 25 sampling points representatively located along the accessible side)

For the control of uniformly distributed substances/products

40 incremental samples of 100 grams – resulting in 4 kg sample

(if the spear has 4 apertures/openings: the batch has to be sampled at 10 sampling points representatively located along the accessible side)

- **Possibility to sample with spear (with engine) of 5 meter long:** 30 m x 5 m x 4 m = 600 m³ = about 450 tonnes

For the control of non-uniformly distributed substances/products

100 incremental samples of 100 grams – resulting in 10 kg sample

(if the spear has 10 apertures/openings: the batch has to be sampled at 10 sampling points representatively located along the accessible side)

For the control of uniformly distributed substances/products

40 incremental samples of 100 grams – resulting in 4 kg sample

(if the spear has 10 apertures/openings: the batch has to be sampled at 4 sampling points representatively located along the accessible side)

Example 2:

Cereals stored at warehouse 30 m long – 30 m deep – 4 m high = 3600 m^3 = about 2700 tonnes accessible from 4 sides (120 meter side)

- **Possibility to sample with spear of 2m:** $120 \text{ m} \times 2 \text{ m} \times 2 \text{ m} = 480 \text{ m}^3$ = about 360 tonnes

For the control of non-uniformly distributed substances/products

100 incremental samples of 100 grams – resulting in 10 kg sample

(if the spear has 4 apertures/openings: the batch has to be sampled at 25 sampling points representatively located along the accessible side)

For the control of uniformly distributed substances/products

40 incremental samples of 100 grams – resulting in 4 kg sample

(if the spear has 4 apertures/openings: the batch has to be sampled at 10 sampling points representatively located along the accessible side)

- **Possibility to sample with spear (with engine) of 5 meter long:** $120 \text{ m} \times 5 \text{ m} \times 2 \text{ m} = 1200 \text{ m}^3$ = about 800 tonnes

For the control of non-uniformly distributed substances/products

$100 + \sqrt{800} = 128$ incremental samples of 100 grams – resulting in 12.8 kg sample

(if the spear has 4 apertures/openings: the batch has to be sampled at 32 sampling points representatively located along the accessible side)

For the control of uniformly distributed substances/products

$40 + \sqrt{800} = 68$ incremental samples of 100 grams – resulting in 6.8 kg sample

(if the spear has 4 apertures/openings: the batch has to be sampled at 17 sampling points representatively located along the accessible side)

7. Sampling of silos

7.1. Sampling of silos (easily) accessible from above

See point 4

The **length** of the spear (sampling probe) to be used for the sampling of static batch/consignment stored in silo should be sufficient to sample at least a significant portion of the lot.

There has to be a representative sampling of the accessible part of the consignment performed.

The analytical result of this sample is decisive to determine the compliance/non compliance of the whole batch. It is presumed that the rest of the batch is equally affected unless, following a detailed assessment (in particular based on results provided by the feed business operator), there is no evidence that the rest of the lot fails to satisfy the EU requirements.

For examples see point 6.

7.2. Sampling of silo's not accessible from above (closed cylindrical silos)

See point 4

7.2.1. Sampling of silo's not accessible from above (closed cylindrical silo's) with size >100 tonnes

Feed stored in such silo's cannot be sampled in a static way and therefore in case the feed in the silo has to be sampled and there is no possibility to move the consignment, agreement has to be made with the operator that he has to inform the inspector when the silo will be unloaded in order to enable sampling when feed is in flow.

7.2.2. Sampling of silo's not accessible from above (closed cylindrical silos) with reasonable size (< 100 tonnes)

Sampling procedure involves the release into a recipient of a quantity of 50 to 100 kg and to take the sample in a representative way from this 50 -100 kg.

Example for a silo of 25 tonnes:

Silo of 25 tonnes / 50 – 100 kg to be released in recipient / sample taken from this 50 – 100 kg // size of aggregate sample relates to whole consignment (25 tonnes) → means 4 kg sample – number of incremental samples relate to quantity released:

For control of **uniformly distributed substances** means 7 incremental samples of about 600 grammes.

For control of **non-uniformly distributed substances** means 18 incremental samples of about 220 grammes.

The analytical result of this sample is decisive to determine the compliance/non compliance of the whole batch. It is presumed that the rest of the batch is equally affected unless, following a detailed assessment, there is no evidence that the rest of the lot fails to satisfy the EU requirements.

8. Sampling of bulk consignments in closed containers

Can only be sampled when unloaded. In many cases it is not possible to sample at point of import and therefore in such cases where containers are to be sampled the sampling must take place during unloading at the point of destination.

PART C. EXAMPLES OF SAMPLING OF PACKAGED FEED

a) Lot of 400 bags of 25 kg

- Control of homogeneously distributed constituent
5 bags to be sampled – aggregate sample must be 4 kg – this can be obtained by taking 1 incremental sample per bag of each 800 grammes or better to take several (x) incremental samples per bag of each 800/x grammes.
- Control of constituent likely to be distributed non-uniformly
13 bags to be sampled – aggregate sample must be 4 kg – this can be obtained by taking 1 incremental sample per bag of each 300 grammes or better to take 2 or 3 (x) incremental samples per bag of each 300/x grammes.

b) Lot of 20 000 packages of 750 grammes

Control of homogeneously distributed constituent: $\frac{1}{4}$ of $\sqrt{20000} = 141/4 = 36$ (rounded to the next whole number) packages to be sampled. From each package at least 110/115 grammes is taken to obtain an aggregate sample of 4 kg.

c) Lot of 50 big bags of 2 tonnes

Lot size is 100 tonnes. Sampling to be performed according to loose feed.

- Control of homogeneously distributed constituent:

$\sqrt{20} \times 100 = 44.7$ with a maximum of 40 incremental samples.

40 incremental samples to be taken from the big bags. Depending on the accessibility of the big bags, there are different ways to take the 40 incremental samples e.g.

- * 4 incremental samples from 10 bags each
- * 8 incremental samples from 5 bags each.

- Control of constituent likely to be distributed non-uniformly:

≥ 80 tonnes = 100 incremental samples

100 incremental samples to be taken from the big bags. Depending on the accessibility of the big bags, there are different ways to take the 100 incremental samples e.g.

- * 10 incremental samples from 10 bags each
- * 20 incremental samples from 5 bags each.

PART D. SAMPLING OF ROUGHAGE AND SUCCULENT FEED MATERIALS

1. Purpose and scope

These guidance notes provide instructions for the collection, packaging and transport of samples from roughage and succulent feed materials until the arrival at the laboratory (testing facility). Guidance is provided on best practice for sampling roughage/forage.

2. General information

2.1. Sampling time

Especially for silage is the timing of sampling important. The ensiling process should be completed. This is according to the material after 6 - 8 weeks the case. Hay should also only be sampled after a certain storage time (8-10 weeks). Top and edge layers or adulterated parts, which are not suitable for feeding, are to be excluded from sampling or samples from these parts should be collected separately for specific purposes.

2.2. Delimitation of the lot

Criteria for defining a lot may include the uniform botanic composition, vegetation stage and the cutting time. But also the weed infestation, the soil conditions of harvested surfaces, the period on the field, etc., can be criteria for the determination of a lot.

In the silo, a lot may be a silo section with observable uniform appearance. Additional criteria for the delimitation of a lot can be applied e.g. in case of "sandwich silage" fodder where different subspecies have been ensiled. A feed stack can therefore consist of several lots

3. Apparatus and tools

The tools for sampling and transport means must be such that they do not affect or change the feed samples (e.g. contamination with substances or microbial agents or thermally induced changes). It is particularly important to ensure that e.g. no rusted devices and or instruments made of zinc are used. Plastic devices are to be preferred where possible.

For silage and hay, sampling devices are commercially available which are operated by hand or driven by an electric drive in the feed stack. There is also special drilling equipment available for the collection of samples of silage with high density.

In many cases however, the incremental samples are to be taken by hand.

To sample a loose heap, like hay or dried green pellets, a shovel can be used.

Note: The devices and tools have to be used in clean condition and must be thoroughly cleaned after each use.

3.1 Basic tools for taking samples such as hackers, knife, cutter, shovel, fork, hay forceps, ...

3.2 Tools for drill samples.

Depending on the type, degree and depth of the silo, various devices are recommended, such as a short drill, longer drill (depending on the depth of the silo). Drilling with motor allow the removal of material to a greater depth.

3.3 Sufficiently large container for mixing the incremental samples (bucket or tub).

3.4 New plastic film as a substrate.

3.5 New thick-walled plastic bag, suitable for at least 1 kg sample material.

3.6 Paper bags for dry sample material.

3.7 Sealer with suction device or, alternatively, appropriate tools such as clips for airtight sealing of the plastic bags.

3.8 Waterproof pen to label the packed samples.

3.9 Cool bag/box with cooling batteries, or other suitable cooling devices.

3.10 Insulating material

3.11 Tape

3.12 Disinfectants: commercially available ready to use spray disinfectant (for example ethanol/propanol mixture)

3.13 Disposable gloves

3.14 Refrigerator, adjustable from 2 °C to 8 °C.

4. Sampling procedure – roughages – succulent feed materials

In certain cases it is necessary to divide a batch into a number of batches if necessary. The aggregate sample should not weigh less than 4 kg in case of humid roughages or silages and not weigh less than 1 kg with straw, hay and dried forage. The incremental samples should be approximately equal.

4.1. Forage

The samples may be obtained in different ways. The size of incremental samples is based on the size of the aggregate sample. The number of samples depends on the feed type and the heterogeneity of the lot, but should in grassland not be less than 50 incremental samples.

4.1.1 Sampling in the field (pasture, permanent grassland)

For sampling in the field, a careful delimitation of the lot is required, especially for very heterogeneous parcels from which, if necessary, the areas that are not eaten by the animals must be excluded. If necessary, a determination of the plants present in the parcel is required in view of the delimitation of the lot. This includes the determination of the presence of valuable forage plants or the presence of toxic plant species.

Incremental samples are preferably taken by diagonal crossing of the parcel with a fixed frame (taking into account the composition of vegetation and topography). Plants are then harvested at a suitable cutting height (average height at harvest, bite height of grazing species, usually 3-5 cm above the ground). Suitable for this are grass cutting devices or other suitable cutting device. The incremental samples should, especially when taken under strong drying weather conditions, be taken within a time period kept as short as possible.

Since the concentration of some constituents (sugar, nitrate and others) may have a day variation, the time of sampling shall be mentioned in the sampling record.

The incremental samples are combined in a large container or on a plastic film and mixed. The laboratory sample is formed by taking the parts of the sample by hand. Please note that fine parts (fine plant parts, earth impurities) should not be shaken off selectively, but have to be taken into account when dividing samples. For old and mature parts (dead plants) the state of the material is to be mentioned in the sampling record.

4.1.2. Sampling at harvest (swath, chopping stream)

If the parcel for the production of hay, silage or other preserved fodder is already cut, the sampling can be done from the swath. Incremental samples are removed at regular intervals from the swath and combined to form the aggregate samples. Irregular swath sizes are proportionally taken into account (influence on dry matter content). Polluted/damaged sites (e.g. mole-hill, compressed by tire) should be omitted, insofar it can be assumed that they do not appear in a large proportion in the harvested material.

4.1.3. Flood sites, areas with tire tracks or damage by trampling

The share of these damaged areas of the total area to be sampled should be estimated/calculated. Samples from these severely damaged zones are taken as sub-samples and then, if necessary, such samples are analysed separately as part of a risk assessment, to the extent it is suspected that such heavily damaged/polluted sublots can be present in the entire harvested material or can be taken up by the grazing animals.

4.1.4. Sampling at ensiling

Another type of sampling, which is often practiced for a prediction/estimate of the feed value, is taking the samples just before or during the ensilage, i.e. taking the samples when put into the silo (3-4 incremental samples per harvest wagon). Such aggregate sample is able to give an average value for the harvested crop, which is, however, subsequently modified by the silage process. It must be ensured that the incremental samples are not exposed for a long time to direct sunlight (causing change of moisture content, degradation easily fermentable ingredients). A cool-box is recommended for interim storage for a limited period of time.

4.1.5. Sampling at feeding

In case the forage is fed in the stable, the incremental samples are taken at the feeding box. It is important to observe that the feeding box was pre-cleaned before from the remainders of previous feed. The samples shall be taken from the total depth of the material.

4.2. Silage

For the preservation, horizontal silos are preferred in different constructions. Other forms are tower silos, bag silos or bale silage in various designs (round and square bales/bags).

Depending on the type of silage (design of silos), the quantitative requirements of the number of incremental samples cannot be met. In view of avoiding possible spoilage by oxygen penetration, a limitation of the incremental samples is in certain situations advisable.

Sampling should only take place at the end of the fermentation i.e. 6-8 weeks after ensiling.

It is preferable to sample an open silo.

4.2.1. Horizontal silo - closed

For the sampling of the closed horizontal silo, the cover must be cut and incremental samples are taken with a suitable sampling device (drill) from that spot (where the cover has been cut). Samples can be removed from the lower layers with the same sample device (drill), even in case of relative high stack heights. At very high stack heights however, it is usually not possible, to take samples from the bottom layers of the silo.

To minimize the risk of spoilage due to air infiltration, the number of incremental samples should be limited. Important is to close the sampling holes made by the sampling device with suitable material (e.g. silage) and to seal the cover at the puncture points airtight with tape to prevent spoilage by exposure to air/air infiltration.

The number of incremental samples should be at least 3 taken at different locations according to good professional practice.

4.2.2. Horizontal silo – open - sampling from the cutting surface

Sampling at the cutting surface of an open horizontal silo is preferable. When sampling, it is important to observe that the sampling takes place at a fresh cutting surface. In case the sampling cannot take place immediately after feed withdrawal, it is appropriate before sampling to remove a layer/slice of about 30 cm of the cutting surface. Incremental samples have to be taken spread over the entire cutting surface.

In case of different silage materials in one silo (sandwich silage) or several layers can be distinguished (as regards colour, density, chop size, etc.), it is advisable in such cases to define and delimitate different lots.

The covering layers and the edges are to be excluded from the sampling or should be sampled separately for specific purposes.

The number of incremental samples to be taken depends on the size of the sampled cutting surface. 1 incremental sample / 4 m² should not be exceeded (with a maximum of 20 incremental samples). It is important that no loosening of silage layers behind the cutting surface occurs, which would result in spoilage.

4.2.3. Tower silos

In general, no or not representative samples can be taken from closed tower silos. The sampling is usually only possible from the discharge opening at the top or bottom. An orientation could be to take incremental samples at feeding. With a top sampling, additional samples could theoretically be taken with a drill/sampling device up to a limited depth. A sampling at the top layer of a tower silo should be avoided with respect to occupational safety.

4.2.4. Bag silos/ bale silage

The sampling of bale silage should be preferable done on an open bale/bag, since there is a risk of spoilage as a result of the oxygen exposure/air infiltration when the sampled silage bales/bags are not directly used for feeding. The number of bales/bags that can be sampled in this way without losses is limited depending on the feeding speed.

The total number of bales/bags to be sampled depends on the lot (number of bales/bags in the lot, separate storage after cutting, the homogeneity of the harvested area or other criteria). For a representative sampling it is recommended to sample at least 5 bales/bags per hectare of harvested area.

4.2.4.1. Sampling of open bales/bags

If there is an immediate feeding, then it is relatively easy to collect samples from the open rolled round bales or open spread of square bales/bags. 10 incremental samples should be taken by hand from round bales evenly across all layers or 20 incremental samples from square bales (big bag) after removal of the covering layer. The remaining amount of the silage must then be fed directly or discarded.

4.2.4.2. Sampling of unopened/unwrapped bales/bags

Only freely accessible bales/bags should be sampled and the sampling sites/holes are immediately very carefully sealed.

The sampling of the unwrapped round bales has to be made with a suitable sampling device through a puncture of the radial centre of the bale through all the layers to the core of the bale. This approach enables a representative sampling of the sampled bales.

In closed square bales/bags it is recommended to perform the sample with a suitable drill by taking the sample at two sites at opposite ends of the bale from the long side.

It is important to fill the sampling site immediately after sampling with suitable material (silage/ straw/hay) and close the hole airtight with tape (see silo). Since sampled bales/bags have limited stability, they should preferably be used by priority for feeding.

4.2.5. Silage tube

Sampling of the cutting surface at the open silage tube

Sampling of the closed silage tube is in principle possible. However as it is difficult to close the silage tube properly after having taken the sample (air exposure occurs), sampling of closed silage tubes should be avoided.

4.3 Hay

4.3.1. Swath

The sampling of the swath of dried hay occurs before the pressing operation or prior to loading in the case of loose hay. At least 20 incremental samples are taken by hand from the swath at regular distances evenly distributed across the entire pasture area.

When taking incremental samples, care has to be taken that the sample reflects the natural ratio of the leaves and stems. The incremental samples are combined into a single coarse-mesh bag (such as onion bags) for aggregate sample. The bag is stacked along with the rest of the pressed bales or loose hay and, by the end of the ripening process (after 8-12 weeks) has to be sent unchanged to the testing facility. Advantage of this sampling method is the possibility to exactly relate the sample to a certain pasture/forage area.

4.3.2. Haystack

The sampling of incremental samples of loose hay from a haystack is usually limited to the accessible boundary layers and in particular when it concerns long hay. In case a chopping of the hay has taken place, it is possible to sample at a greater depth. The sampling should be done with mechanical aids (fork, hay forceps etc.) because sampling by hand entails the risk that long stem material is preferred and leaf material is lost. It is recommended to take 20 to 30 incremental samples evenly distributed on the haystack.

4.3.3. Hay bales

The sampling of hay bales can be performed both on unopened and on opened bales, the latter being more accessible and the sampling is similar to open silage bales and requires less work and effort. In contrast to the open silage bales, there is no risk of spoilage for hay bales and the hay can be continued to be fed.

4.3.3.1. Sampling of open bales

The bleached outer layers of the hay bale are not used for sampling, since it has by transporting, stacking and the storage process less leaf material compared to the rest of the bale material. From the other parts/layers of the hay bale, at least ten incremental samples are taken by hand evenly distributed from the outside to the inside (core) in the case of round bales. In the case of square bales (big bales), 20 incremental samples are taken by hand evenly distributed from one side to the other side of the bale. If it concerns smaller bales (20-25 kg) then it is recommended that at least 5 incremental samples are taken distributed evenly over the entire cross-section. The incremental samples are combined into a clean container for the aggregate sample.

4.3.3.2. Sampling of unopened bales

The sampling of intact square or round bales is carried out with appropriate tools analogous to the procedure for silage bales/bags.

4.4. Straw

Same sampling procedure as for hay.

Particular to straw is the very large portion of stem material with small portion of leaf and ear components.

4.5. Dried green forage

Dried green forage for ruminant diets is generally in the form of pellets or briquettes. The sampling can be performed using shovels or other suitable tools to take the incremental samples from the heap.

4.6. Succulent feed (feed with high moisture content)

Succulent feeds are commonly known as parts of processed products with a dry matter content of more than 55 % as beetroots, roots, tubers, maize by-products, brewer's grains, beet pulp, citrus and apple pomace and others.

Beets, roots and tubers are usually collected as single entities at random as an incremental sample, then ground in a suitable mill (roughly ground as done for the feeding of the animals) and from the well-mixed aggregate sample the laboratory sample is taken. To avoid biological reactions (metabolisation of sugars), a quick (cooled) transport to the laboratory or quick preservation of the sample has to be ensured.

4.6.1. Brewers' grains

Brewers' grains are usually fed fresh or ensiled. From a heap of fresh brewers' grains, the incremental samples are taken with suitable shovel and combined into an aggregate sample.

When brewer's grains are preserved in horizontal silos, the stack height is less than what is usual for roughage. Therefore, the sample can be taken in a similar manner as roughage silage whereby the silage-cover/film is pricked and the incremental samples are taken with a suitable sampling device. Since the material is much more homogeneous than roughage, it is possibly not necessary to go with the sampling device up to the bottom of the stack/heap.

4.6.2. Pressed pulp

Pressed pulp can be fed fresh only for a very limited period of time. They are usually ensiled in the horizontal silo or silo tube.

When the sampling is to be carried out on a closed silo, attention has to be paid to a careful closure of the puncture holes.

More preferable, if feasible, is the sampling on the cutting surface of the silo. It has to be taken into account that often stratification to a certain extent with respect to density and moisture content is present. On the other hand, it is usually a homogeneous material, so that the delimitation of the lot is easier.

5. Sampling in case of suspicion or already occurred damage

On the basis of a suspicion - or already occurred damage - a possible delimitation of the affected part of the feed is required on the basis of observed adverse health effects. In the sample record, the relation of the affected part of the feed with the rest of the feed stock is needed. Careful visual inspection appropriate to possibly identify the suspected toxicant (such as mould hot spots, increased incidence of weeds/poisonous plants or insects). If appropriate, attention is to be paid to (environmental) factors that could have led to the observed adverse health effects (paint residues, fertilizers, lubricants and fuel residues, etc ...)

Following these visual observations, a further delimitation of different parts of the affected feed might be necessary and all the different parts need to be sampled separately.

6. Sample division / homogenization

In general, a sample division/reduction of the aggregate sample is not possible with commercial sample dividers. It is possible to spread hay or silage on a handmade frame that enables a two- or four division. Another possibility is to spread the aggregate sample on a solid substrate (foil) in a thin layer (possibly after a size reduction of the large stem material) and to take by hand sub-samples for the representative sample (random selection of sectors where subsamples have to be taken)

The bags for the control samples and reference samples should be filled alternately and strict attention has to be paid to these subsamples as regards e.g. fine (leaf) plant parts, earth impurities.

7. Packaging, transport and delivery

7.1 Sending of samples

In general, the final samples are be put in clean sealable plastic bag (minimal air inclusion or vacuuming). For hay and straw, the samples can be put in paper bags.

7.2 Sample Preservation

Samples should be rapidly delivered to the testing facility. Fresh and silage samples should be kept as cool as possible, if necessary, deep frozen. If possible, no additional effects/influence from sunlight, high temperatures or contamination. For frozen samples, change can occur during thawing.

7.3 Sampling record, identification of the sample

The sampling record should contain:

- Type of feed or composition (vegetation).
- Number of areas in the silo with tonnage
- Harvest time, harvest number and time of mowing
- Date and place of sampling
- Intended use
- Information on the sampling procedure and on the lot
- Storage time of the lot
- Deviations from specified sampling procedures
- Any other relevant information

The final sample is placed in a labelled plastic bag. The bag is, after a vacuum is created, closed with a specific device. Alternatively, a nearly anaerobic condition can be achieved by manually removing the air (pressure on the sample) and directly closing the bag.

The final sample is to be stored cool and as soon as possible - preferably on the day of sampling, but no later than the following day – sent to the testing facility. An uninterrupted cold chain at a temperature of $\leq 8^{\circ}\text{C}$ is required. If these temperatures cannot be complied with, this should be noted in the sampling record. Specific chemical analysis (pH, determination of fermentation acids) cannot provide representative results for the lot if certain conditions of storage and transport are not complied with.

Note:

When using cooling batteries it should always be insulated with a suitable material as local freezing of the sample in plastic bags is possible.

The arrival of moist feed in the testing laboratory should be announced so that an immediate processing of the sample can be ensured.

8. Some final remarks

Sampling drill: For the sampling of forage samples different drills/sampling devices are commercially available. It includes both devices for manual operation as well devices with electrical supply. Certain equipment is supplied with additional equipment to facilitate the ejection of the core sample taken from bores/drills or to facilitate the withdrawal of the drill from the feed stock. Frequently, the drill is provided with a serrated edge/blade to facilitate the penetration into fibre-rich material.

Feeds with high moisture content are microbiologically perishable feed with a water content of more than 16 % which cannot be homogenised.

Quick fermenting silage is a feed with a dry matter content of 28-35 % (whole-plant corn silage, weak wilted grass silage or intermediate crop silage).

Slowly fermenting silages have a higher dry matter content of 45% and more (corn products or hay).