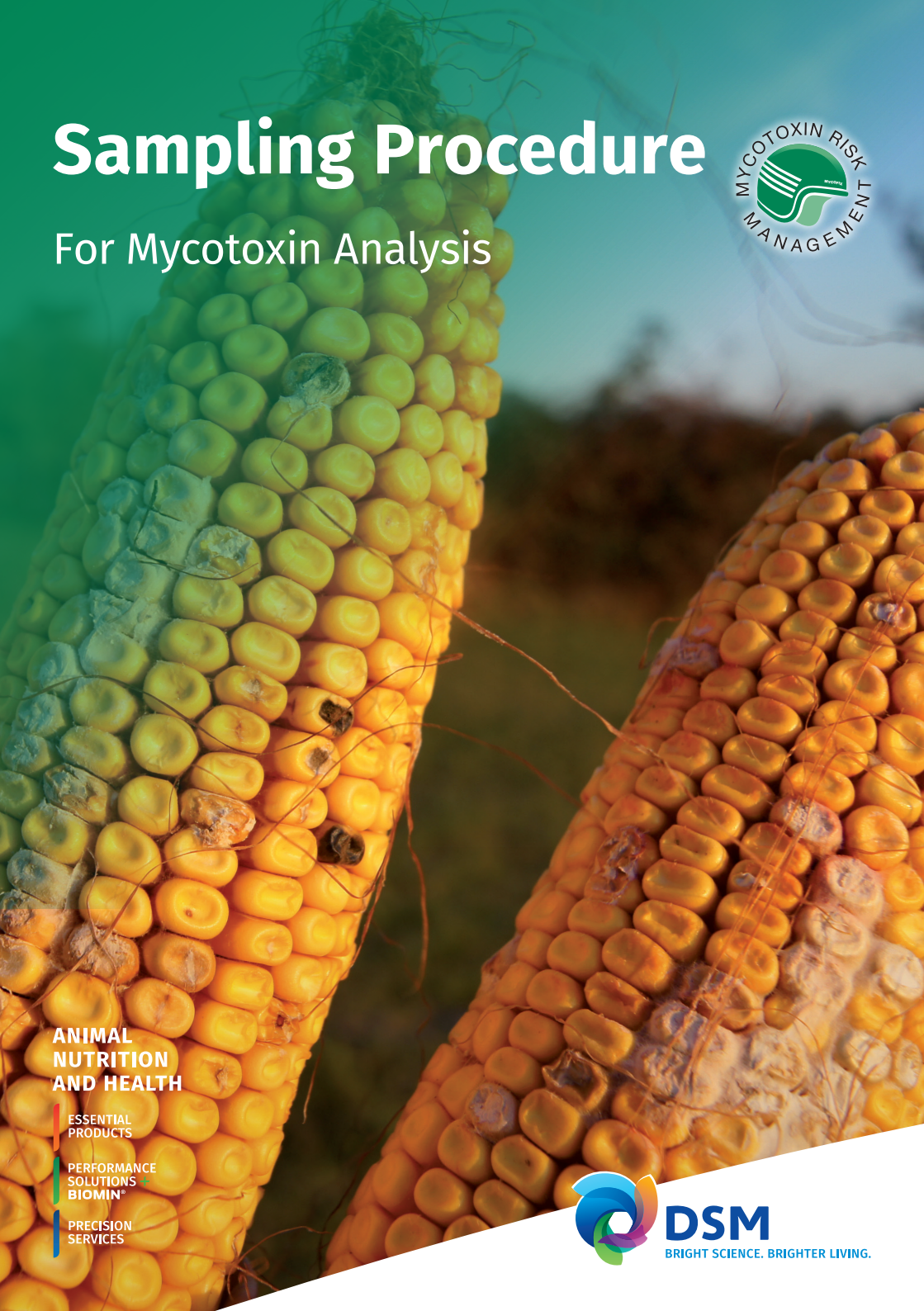
Sampling Procedure

For Mycotoxin Analysis



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Contents

01 Introduction

Proper sampling and sample preparation are the foundations of quality mycotoxin testing. Assessing mycotoxins can be difficult due to the uneven distribution of mycotoxins in the commodity, especially in whole kernels. Different parts of the lot may contain different concentrations of mycotoxins (*Figure 1*).

Figure 1. *Inhomogeneous distribution of mycotoxins (dark orange) in grains.*

Aim

02

The main objective of an effective sampling procedure is to obtain a representative sample of whole grain, finished feed or meal by collecting sufficient sub-samples to reduce random sampling errors. Sampling is usually the largest source of error in mycotoxin analysis and can result in around 75 % of the total uncertainty (*Figure 2*). Despite the use of appropriate equipment, sampling error is large because of the inhomogeneous distribution among contaminated particles within a lot. A suitable sampling procedure should minimize both the buyer's and the seller's risk to the lowest possible level.

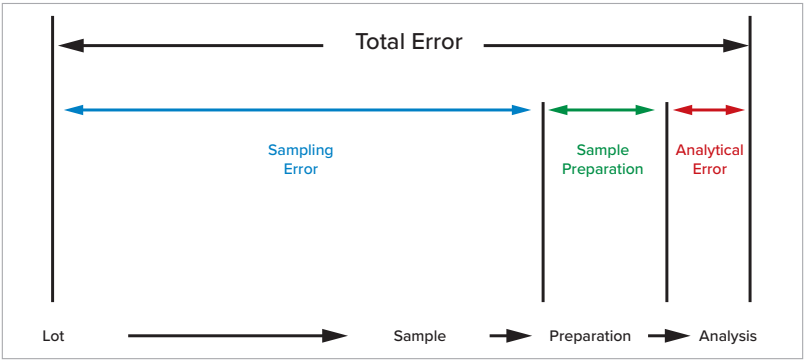

Figure 2. *Total error of the mycotoxin analysis procedure as a sum of sampling, sample preparation and analytical errors.*

- It is easier to obtain a representative sample from a moving stream of product than from a static lot.
- Increasing the number of samples can reduce sampling error.

Definitions

03

The following terms are described in the Commission Regulation (EC) No 401/2006 of 23 February 2006 “Laying down the Methods of Sampling and Analysis for the Official Control of the Levels of Mycotoxins in Foodstuffs” and as in the EN ISO 6497:2005 No 76/371/EC “Animal Feeding Stuffs – Sampling”.

Consignment: a specified quantity of feeding stuff on offer, dispatched or received at one time.

Lot: an identified quantity of a consignment having characteristics presumed to be uniform.

Incremental sample: a quantity of material taken at one time from a single point in a lot.

Aggregate sample: the combined total of all the incremental samples taken from the lot.

Laboratory sample: a sample intended for laboratory analysis.

KEEP IN MIND

- Sampling should be conducted on a regular basis.
- Every new batch should be sampled.
- Improper product storage may affect the quality of grain.
- Improper storage conditions may lead to variations in silage quality.

Sample Selection

04

Every individual item in the lot should have an equal chance of being selected: this is a method called random sampling. It is essential to select equipment that is adequate for sampling. For example, probes should be able to sample large particles and reach every location in the lot. If the lot has been blended thoroughly during handling then it is assumed that all particles are distributed uniformly and representative samples may be collected. However, when particles are not distributed uniformly, the aggregate sample should be an accumulation of several small incremental samples taken from many different locations throughout the lot (*Figure 3*). In general, sampling is best achieved when the lot is in motion.

BASIC PRINCIPLES TO OBTAIN A REPRESENTATIVE SAMPLE

- Consider the number of samples required for a proper analysis.
- Collect small incremental samples from several areas of the lot.
- Combine these samples to form a single aggregate sample.
- Mix well and take a subsample to provide a final sample for analysis.

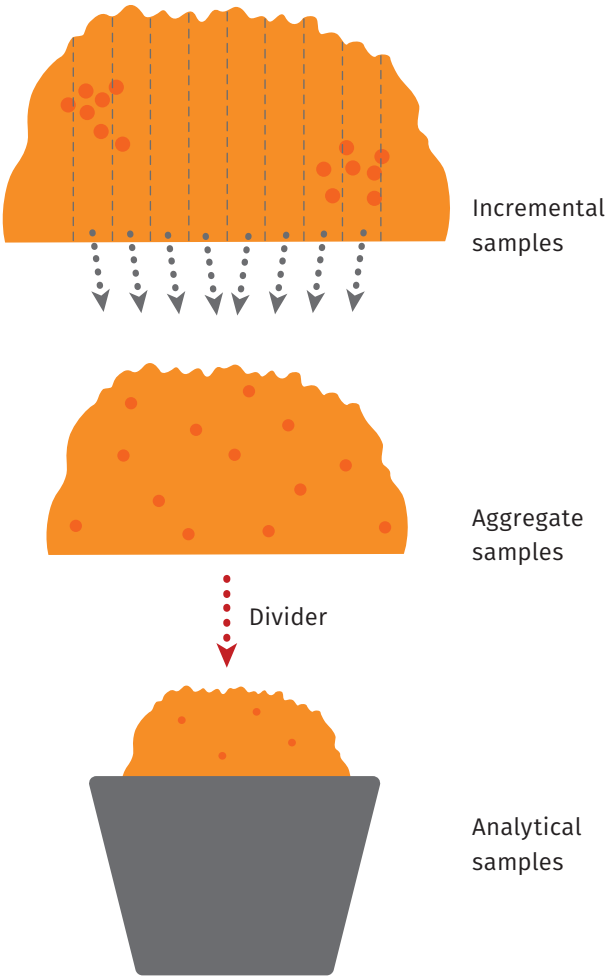

Figure 3. *Basic principles of proper sampling: incremental samples are collected from the lot and mixed to create an aggregate sample. An analytical sample is then taken from the homogeneous aggregate sample.*

Sampling Equipment

05

Select a sampling device that is appropriate for the particle size of the commodity, the size of the sample to be taken and the size of the container.

5.1. Manual sampling

a) Grain probe or trier: for samples originating from cars, trucks, wagons or barge loads, or for sampling products in motion at low flow rates (*Figure 4*).

Probe/trier should be long enough to reach the bottom of the container or the whole depth of the grain!

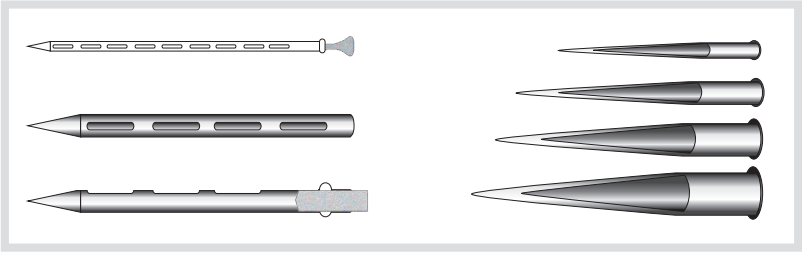

Figure 4. *Examples of grain probes and triers used for manual sampling.*

b) Pelican grain sampler: for samples originating from a falling stream of bulk grain (*Figure 5*).

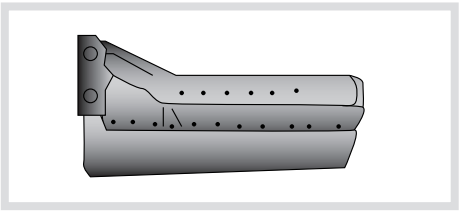

Figure 5. *Pelican grain sampler.*

5.2. Automatic sampling

For sampling of commodities in motion at **high flow rates** (*Figure 6*).

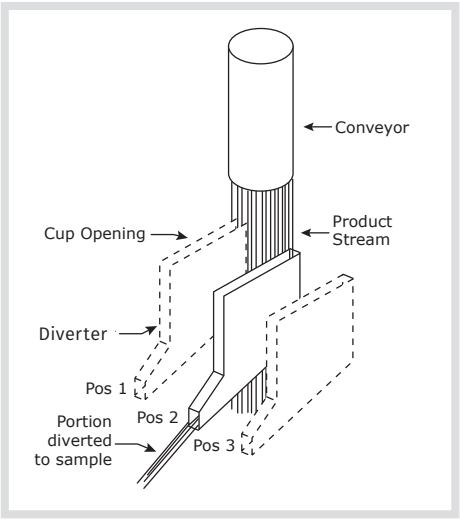

Figure 6. *Cross cut sampler moves at a constant velocity to cut through the entire stream of product.*

Cleanliness

06

To ensure that the properties of the samples and the sampled lot are not affected, use only clean and dry equipment. Clean the apparatus thoroughly after use. Personnel should wear disposable gloves.

Sample Containers

07

The containers should not change any of the characteristics of the sample to be tested. Use a paper bag when collecting laboratory samples of dry components to avoid humidity and mold growth. Use a plastic bag that can be refrigerated when sampling silage. For moist samples use airtight bags that can be vacuum-packed. Containers and bags should be capable of being sealed so that it is not possible to open and reseal them without being detected.

SAFETY

- Anyone sampling from bulk grain should be accompanied.
- Attention should be paid to the presence of loading equipment, lorries and/or trailers.
- No one should enter a closed silo.

08 Sampling Procedure

Depending on the type of product that will be sampled, a minimum number of incremental samples should be collected in order to obtain a representative sample for analysis. *Figure 7* illustrates the basic steps for a successful sampling procedure.

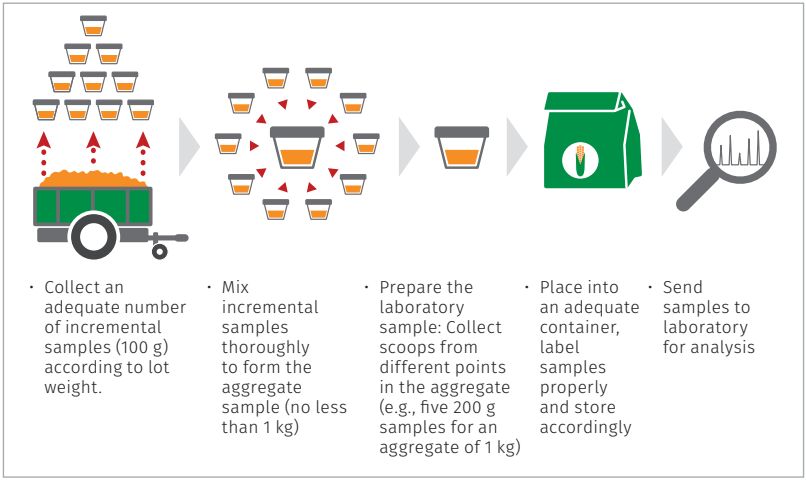

Figure 7. *Steps for a successful sampling procedure.*

The following sampling procedures are divided into two groups: solid feed and roughages. Solid feed samples can be collected either during transfer or at the place of storage. Roughages are divided into fresh and ensiled material, and straw (*Figure 8*).

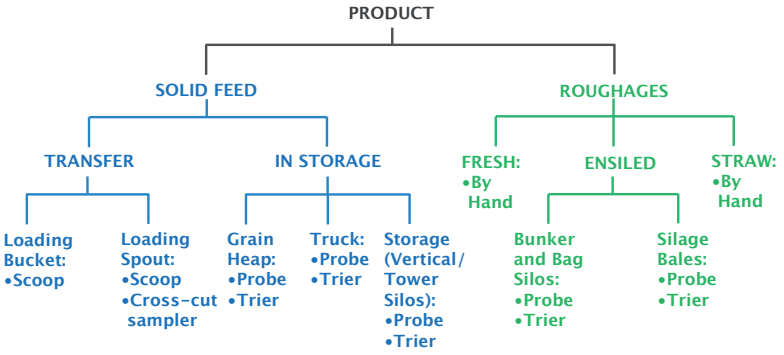

Figure 8. *Categorization of feed products and conventional sampling equipment used.*

8.1. Solid feeding stuffs - grains, seeds, beans, pellets, meals and powders

When sampling from bulk, make sure that all parts of the lot have an equal chance of being selected. Consider the number of incremental samples (100 g) that will be required to create an aggregate sample that is suitable for analysis (Table 1). The aggregate sample should always be at least 1 kg.

Table 1 – *Number of samples required for analysis depending on lot size (EC No 401/2006)*

Lot weight (metric tons)	Number of Samples	Aggregate Sample Weight (kg)
Up to 1	10	1
Up to 10	40	4
Up to 20	60	6
More than 50**	100	10

* For lots weighing more than 50 tons, calculate the number of samples using the following formula: √ (20 x lot weight in tons) = number of incremental samples.

Take precautions as dry meals may explode because of their dusty consistency. Due to their processed nature, meals are more susceptible to microbial damage. In such cases, isolate any evidently spoiled part of the lot and sample separately.

Accessing the entire lot is sometimes difficult. In such cases, the best samples are obtained from product that is being transferred from one container to another, or from the truck to the place of storage.

8.1.1. Sampling during transfer (i.e. loading bucket, loading spout)

Collect incremental samples of products (100 g) in periodic intervals while they are being transferred from one place to another (*Figure 9*).

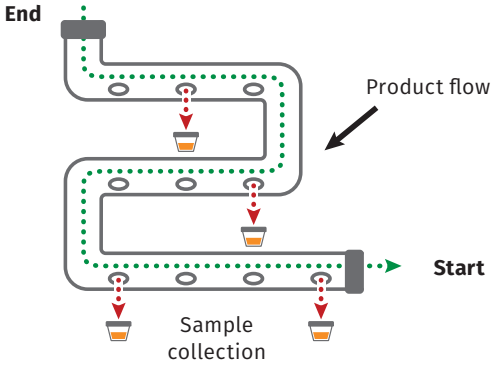

Figure 9. *Sampling from a moving stream of product. Collect incremental samples of products (100 g) at periodic intervals during transfer.*

When transferring with a loading bucket, take scoop samples from the grain in each bucket being loaded (*Figure 10*).

Figure 10. *Sampling of grains during transfer with loading bucket. The* ***red x*** *represents the spot where the sample should be collected.*

When transferring sampling material with a loading spout, collect the sample automatically from the moving stream using a cross-cut sampler (see Sampling Equipment section) or manually by cutting through the stream with a cup or scoop (*Figure 11*).

Figure 11. *Sampling of grains from the moving stream of a loading spout. The* ***red x*** *represents the spot where the sample should be collected. Collect as many samples as necessary, changing the position of the cup or scoop each time.*

8.1.2. Sampling in place of storage (grain heap, storage silos) and bulk samples in trucks

Collect incremental samples (100 g) from various places distributed throughout the lot where the grain is accessible. The minimum length of the sampling probe should be two meters.

Grain heap: Collect incremental samples from across the entire surface area so that all parts of the lot have an equal chance of selection (*Figure 12*).

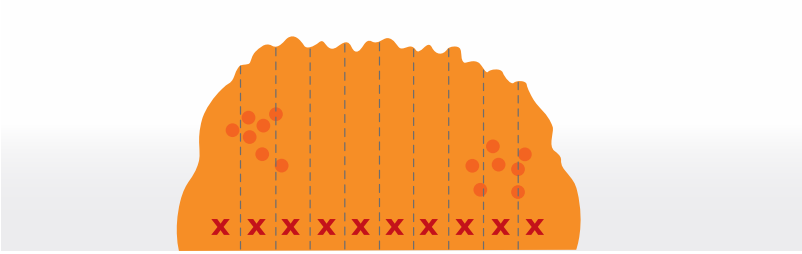

Figure 12. *Sampling of grain heaps. Each* ***red x*** *represents a spot where a sample should be collected.*

Vertical/tower silos* with accessible grains from above: Collect samples from the entire surface as illustrated in *Figure 13*.

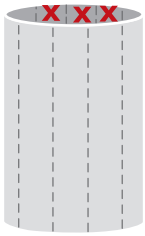

Figure 13. *Sampling of grains in a tower silo. Each* ***red x*** *represents a spot where a sample should be collected.*

*For closed silos containing lots greater than 100 tons, sampling cannot be conducted statistically. The grain must be transferred to a new silo in order to collect proper samples

Truck: Collect incremental samples using a spear sampler (*Figure 14*).

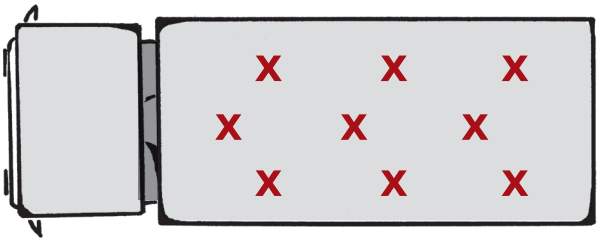

Figure 14. *Sampling of grains in a truck. Each* ***red x*** *represents a spot where a sample should be collected.*

8.2. Roughages – fresh and ensiled roughages or straw

For roughages, the most practical way to take a sample is by hand. If possible, collect samples while the material is in motion. Be sure that all layers are represented equally. For sampling after ensiling, wait until the fermentation is complete. The minimum number of incremental samples for roughages is illustrated in Table 2.

Table 2 – *The minimum number of increments required when sampling roughages*

Lot weight [metric tons]	Minimum number of increments
≤ 5	10
≥ 5	√(40 x metric tons) (max 50 metric tons)

Fresh roughage: Leaves and stems are distributed unevenly in a truckload with more leaf material along the edges. Collect incremental samples by hand as the truck is unloading.

Bunker and bag silos: Collect incremental samples by puncturing the plastic cover using a sharp, cone-shaped sampling device. Holes should be evenly distributed over the entire surface of the silo (*Figure 15*). Refill each hole cautiously immediately afterwards and cover using a strong tape to prevent possible contamination.

Figure 15. *Spots where an incremental sample should be collected when sampling bunker silos or bag silos.*

Silage/Straw bales: Due to variations in the distribution of leaves, stems and other materials it is recommended to sample 20 small rectangular bales or 15 large bales. Combine 15 – 20 incremental samples from each bale to form an aggregate sample (*Figure 16*).

Figure 16. *Each **red x** indicates a spot where an incremental sample should be collected when sampling (a) square and (b) round bales.*

- Refrigerate or freeze silage samples until they are ready for analysis.
- Do not ship samples late in the week to avoid having them stay at the post office over the weekend.
- Use express mail to minimize shipping time.

Storage 09

Samples should be stored in a cool, dry place, safe from rodent attack. Use a paper bag when collecting laboratory samples of dry components to avoid humidity and hence mold growth. Use a plastic bag that can be refrigerated when sampling silage.

Labelling 10

Write a detailed description of the sampled product on the label including:

- Date sampled
- Origin of the lot (country, region, farm name, address or postcode of origin if possible)
- Year of production
- Size of the lot sampled
- Short description of sampling procedure
- Stage of sampling (storage, feed mill, ...)

References:
Commission Regulation (EC) No 401/2006 of 23 February 2006:
Laying down the Methods of Sampling and Analysis for the Official Control of the Levels of Mycotoxins in Foodstuffs
EN ISO 6497:2005 No 76/371/EC: Animal Feeding Stuffs – Sampling
HGCA Grain Sampling Guide:
http://www.hgca.com/content.output/5148/5148/Crop%20Management/Crop%20Management/Grain%20storage%20and%20sampling.mspx
Sampling Hay, Silage and Total Mixed Rations for Analysis (Undersander):
http://learningstore.uwex.edu/assets/pdfs/A2309.pdf
Sampling Feeds for Mycotoxin Aanalyis (Whitacker, 2003)

8 steps for taking the perfect sample for mycotoxin analysis

Mycotoxins are naturally inhomogenous in their distribution. There will be hot-spots of mycotoxins in an otherwise 'clean' batch. To get a true analysis result, sampling is really important. Follow these steps to get your sampling right.

1 Assess the size of your batch and work out how many sub samples you need to take.

For example, 1 ton of raw material = 1 kg of aggregate sample (10 x 100 g samples). The exact amount of sample depends on lot type and size according to EC Regulation No 401/2006.

2 Check the sampling method according to the material being sampled.

Your DSM representative can provide you with the best practices for each material, making sure you collect and store your sample correctly.

3 Use the right equipment for the job.

There are lots of tools out there so do your research and make sure you have the right tool for the job.

4 Take incremental samples and mix them together thoroughly to form an aggregate sample.

Make sure incremental samples are thoroughly mixed together so that all components are homogeneously distributed.

5 Take the samples you will send off for analysis from your aggregate sample.

The laboratory carrying out the analysis will tell you how much sample they need. The general recommendation is at least 1 kg. Make sure you send them at least this amount, if not more. And consider storing some material for future reference too.

6 Make sure the bag or container is well sealed, and labelled with all the necessary information.

Your DSM representative can provide sampling bags or advise on other sample containers that avoid altering the quality of the sampled material.

7 Post your sample off to the laboratory the same day.

Try not to send samples late in the week as they might be stuck in the post office over the weekend. Instead, store samples in a fridge or freezer and post them the following week.

8 Enjoy the benefits of receiving accurate and reliable mycotoxin analysis results.

But your sampling journey doesn't end there. Take regular samples to stay well informed about the mycotoxins in your raw materials or finished feeds.

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