Sampling Protocol (Guidelines) for Feeds and Ingredients

Compiled by P H Henning for AFMA
April 2006

Introduction

The animal feed industry and its customers deal mostly in lots or batches of feeds or ingredients. Information is often required about a batch for various important reasons. These include quality control, intelligence on opposition products and settling of disputes. It would usually be impractical to handle or keep a batch of feed or ingredient in the way required to gain and manage the necessary information about it. The solution is a sample. "Sample" is defined as follows:

> (Oxford): a relatively small quantity of material, or an individual object, from which the quality of the mass, group, species etc. which it represents may be inferred
>
> (Websters): a representative portion of a whole; a small segment or quantity taken as evidence of the quality or character of the entire group or lot

The above refers to a so-called representative sample. The objective of a sample is to reflect the truth about the "whole", be it a batch or a bag or any other container of material. As such there are important characteristics of any sample, including the need to know:

- What is the "whole" which the sample is supposed to describe or represent
- Does it accurately reflect the "whole"
- Is the sample still representative of the original material

Sample size is also an important issue. The quantity of sample must be sufficient for its purpose but for various reasons an unnecessary large sample quantity is undesirable. Sampling is clearly not a simple matter and there are a number of aspects to consider in the quest for reliable samples.

The purpose of this protocol is to supply a set of guidelines to AFMA members on how to obtain samples of an acceptable standard and how to handle those samples once they are obtained.

The nature of "a sample"

A sample is taken from a batch or lot to learn specific things about that batch as a whole. If the entire batch was to be 100% homogeneous throughout, at least in terms of the characteristic(s) of interest, and of a suitable physical dimension, then sampling would have been as simple as taking one handful of material from the most convenient point, stuffing it in a container and "there you have a representative sample". In practice batches of feeds or ingredients are seldom 100% homogeneous, and it consists of materials of all shapes and sizes. In a real-life situation the need may often be to obtain a representative sample of ca 500 grams from a batch of 10 tons.

Principles and guidelines rather than rules

The most important requirement of a sample is that it must accurately reflect the "whole". The truth is that there is great variation in the nature and homogeneity of the various "wholes" of interest, even just to AFMA members and their clients. There are also substantial differences between companies in infrastructure, logistics and operation. This makes it impractical to lay down a fixed

set of rules for sampling. It is suggested that AFMA accepts a set of sampling principles or guidelines and that each member then draw up their own Standing Operational Procedure (SOP) for Sampling, which should adhere to the accepted principles but make allowance for their unique setup and circumstances. The SOP's should cover all situations, from sampling large batches down to taking samples of in-house or competitor products in the field. AFMA may keep a file of such SOP's from participating members as a reference and possibly for a sub-committee to use to improve and refine AFMA's guidelines. These SOP's should cover the following:

- Collection,
- Identification,
- Handling, and
- Storage of samples.

Guidelines

The following guidelines should be used to draw up a suitable SOP for sampling. These guidelines should be revised regularly and improved where applicable:

1. Define the "whole"

Define clearly the "whole" or entity which the sample has to represent. Without this it is difficult to judge sample adequacy. A clear definition will enable selection of the most suitable sampling protocol. The business of a typical company may include a number of such "wholes" and it may be necessary to consider them separately in as far as they may require different sampling protocols.

2. Define the purpose of the sample

For each type of sample to be obtained the purpose of the sample must be clear. This will determine both the specific sampling method to be used as well as the quantity of sample required. The latter includes aspects such as mass and the number and size of containers of the sample required.

3. Choose an appropriate sampling method

The most appropriate method will depend, amongst others, on dimension of the shipment (e.g. size and format of containers), conveying equipment used and sampling equipment available. Prior knowledge and experience of the specific ingredients in question should also be used to select the appropriate sampling method. Points to consider include:

- Degree of homogeneity in whole
- What must the sample show about the "whole"
- Is the "whole" a batch or is it free-flowing?

A specific material to be sampled may be in the form of a batch at some stage, e.g. a truck full of grain, and free-flowing at another, e.g. when that truckload is off-loaded using a conveyor belt or shute. It then has to be decided what point is the most convenient, feasible and desirable for taking the necessary sample(s). It is generally more likely to obtain a good representative sample where the material to be sampled is in motion.

4. Homogenization

The more homogeneous the "whole" that has to be sampled the more accurate any samples taken from it will be. Ensure that the "whole" is as homogeneous as practically possible before taking a sample. Where practical it may involve mixing the whole lot through thoroughly with a shovel, making sure any lumps present are broken up.

5. Sample hierarchy

The following hierarchy can be described in the sampling process:

- Incremental samples: Samples of approximately equal size, taken throughout the "whole" according to a predetermined sampling plan (note that the incremental sample is not the same as a "random spot sample", which usually refers to a single sample taken at random from the "whole").

- Composite sample: All the incremental samples are thoroughly mixed to form a single homogeneous composite sample

- Reduced sample: If the composite sample is too large it may be reduced by quartering, riffler or a Boerner Divider to arrive at a reduced sample

- Final sample: The reduced sample, or the composite sample, is again thoroughly mixed and divided into the final samples that are placed into appropriate containers and retained for analyses and reference.

6. Accuracy and precision

A sample will seldom if ever reflect the original "whole" that it was taken from with 100% certainty. There is always some level of uncertainty. Accuracy and precision are two types of uncertainties associated with sampling. Accuracy is defined as the closeness of measured values to the true value. Another term associated with accuracy is bias, i.e. something which causes the average of measured values to deviate from the true value in a consistent manner. Precision is defined as the closeness of measured values to each other. Another term for precision is variability. The three well-known statistical measures of variability, namely variance, standard deviation and coefficient of variation (CV) can be used as measures of precision in sampling.

It is extremely important for reliable sampling that any procedure allows every individual item in a lot an equal chance of being chosen. This is called random sampling. Sampling procedures which prohibit or reduce the chances of any item in the lot from being chosen, e.g. sampling probe that does not allow larger particles into the probe, results in bias.

Two common sampling schemes used in the feed industry include simple random sampling and systematic sampling. The first applies mostly to material in lots or batches whilst the second applies to free-flowing material.

7. Sampling a lot or batch

Method:
When drawing a sample from a bulk container, a sampling or probing pattern (existing or to be designed) should be used. This normally allows for an aliquot of the material to be taken from each of a predetermined number of spots or positions distributed more or less evenly throughout the whole of the material to be sampled. Each aliquot represents an incremental sample. When sampling a lot of material contained in a number of individual containers (e.g.

bags) then a predetermined number of containers should be selected at random from the whole and these containers sampled. For example if the lot consists of 10 bags or less then all bags should be sampled. If the lot consists of 11 bags or more then sample 10 bags selected at random. Part of the contents of each selected container is then taken as incremental sample. Where practical each selected container may for example be emptied and worked up with a shovel and then one shovelful of material taken as the incremental sample.

Apparatus:
Slotted grain probes, also called shuttered sampling spears, may be used for collection of samples from ingredients and feeds. It basically consists of two tubes, one inside the other, with a tapered point to allow easy penetration of a mass of material. The inner tube is divided into compartments and slots on both tubes allow the probe/spear to be pushed into a mass of material without collecting any material while being pushed in. Once the probe is in the desired position rotation of the inner tube within the outer tube allows slots to align in such a way that material can flow into the compartments. Opposite rotation then closes the compartments before the probe is withdrawn. These probes come in various standard lengths. The probe should be inserted at a 10-degree angle from the vertical to obtain a cross section of material. It should be inserted as close to the bottom of the container as possible. Tapered bag tiers are used to sample closed bags of powdered or granular commodities.

8. Sampling of a stream or "free-flowing" material

This method of sampling involves taking an initial random sample from a stream of the material to be sampled, followed by repeated collection of similar sampling units at equal intervals thereafter. The material may be flowing through a spout or on a conveyor belt. Sampling can be done by hand, using a pelican sampler, or by an automatic mechanical sampler. In order to obtain the most representative sample of the material flow, care should be taken to cut an increment across the entire cross section of the material stream, perpendicular to the flow, at a location where the meal is flowing freely and at a uniform rate. Furthermore small increments of material should be taken along the entire length of the moving stream.

9. Number and size of samples

The number and size of incremental samples will basically depend on the homogeneity of the lot and the practical effectiveness and ease of sampling. Generally the smaller the concentration and/or the less homogeneous the distribution of a characteristic of interest in the "whole", the greater the number of incremental samples required.

As a general rule each incremental sample should be 200 grams and one incremental sample should be taken per 200 kg of material. The resulting composite sample may be reduced when it is too large. It is also generally recommended to take at least six, but preferably 10, incremental samples from any lot of material which is too large to homogenise properly before sampling.

Final sample size should be in the order of 500 grams. This is considered a good compromise between sufficient material for various analyses and back-up on the one hand and ease of handling and storage on the other hand.

10. Sampling policy

Every company should draw up a clear, unambiguous sampling policy that (1) is in line with the above guidelines, (2) is suitable for their specific setup, (3) covers all aspects of interest, and (4) is acceptable to their stake-holders.

11. Responsible person and trained personnel

A person (or specific persons) should be made responsible for sampling. Such person(s) should be properly trained in respect of sampling and should be held accountable for application of correct sampling procedures.

12. Identification and labelling of samples

A clear, informative and patently unambiguous sample identification and labeling system should be designed and used. Any markings on sample containers should be on the container and not on the lid and should be done with ink which cannot come off under any circumstances. Information on the label should include the following:

- Sample identification number
- Name of material
- Date of sampling
- Name of responsible person and company

This is the basic information that should be on any sample container. Other information may be added but there may not be enough space and it may be cumbersome to write on containers when stick-on labels are not used. If the sample is adequately identified other information can be easily stored and accessed in a separate record system.

13. Sample record system

Design and implement a sample record system whereby the following information is maintained for each sample:

- Sample identification number
- Name of material
- Date of sampling
- Name of responsible person and company
- Description of material from which it originated
- Details of sampling method used to derive from the whole in question to this sample representing the whole
- Name and contact information of the person who took the sample, as well as any person referred to in Article 15 (3) (c) of Act 36 of 1947, where applicable
- Name and contact information of the person to whom the sample belongs or who have the primary interest in the sample
- Place of sampling
- Purpose(s) of the sample
- Suggested date up to which the sample should be kept (expiry date)

- Prescribed storage conditions
- Location for storage of the specific sample.

The above incorporates all information required by Act 36 of 1947, in respect of animal feed samples.

Since storage space is usually at a premium any good record system must have a facility that will inform when samples have no further function and should be removed from storage and destroyed. In the animal feed industry samples of ingredients and feeds should normally be kept until the time when the animals that consumed the feed have in turn been consumed by the human food chain.

14. Containers and apparatus

Ensure that all apparatus and containers used for collection, handling and storage of samples are clean, free of any contaminants and made of suitable material that will not interact with the sample in any way that may compromise the planned analyses. Sample containers should be of the correct size (i.e. not wasting space) and uniform of shape (for convenient handling); It should not be unduly heavy for ease of handling and shipping. It should not be fragile and should be able to handle the proposed conditions of storage. It should be transparent when possible since it always helps to see what is inside without having to open the container.

15. Sealing of samples

Where applicable it may be prudent to secure the top of the sampling container with a seal, to prevent tampering with samples and to forestall any accusations of foul play where the sample will be used in settling of disputes. This seal is in addition to the mere proper closing of the container and must be such that any disturbance of its integrity will be obvious. There appears to be no standard method of sealing. One method is the use of an authentic printed sticker that can be firmly affixed to both the container and its lid in such a way that the lid cannot be removed without breaking the seal.

16. Storage facilities and system

All samples should always be stored under appropriate storage conditions, to allow the sample to remain representative of the whole from which it was taken. i.e. basically to minimise the risk of chemical changes. This normally means a cool, dry and dark place for dry samples and frozen (minus 20 degrees Celsius) for wet samples. Sample storage facilities should also be secure to prevent any unauthorised access to samples.
Concluding remarks

Correct sampling is important because a lot of money is going to be spent on analyses of these samples, and potentially expensive decisions regarding the "whole(s)" on which the samples supposedly reflect and are going to be made based on these samples. Furthermore companies make economically important decisions based on comparisons and judgements of samples. They must then have the peace of mind that the samples are a truthful reflection of the whole.

END

Revised 29/5/06

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