

## DEPARTMENT OF GRAIN SCIENCE

# Sampling: Procedures for Feed

**Tim Herrman**  
Extension State Leader  
Grain Science and Industry

Ingredient quality is the foundation on which an animal ration is built. Correct sampling and sample evaluation enables the processor to make inferences about the quality of incoming grain, protein sources, micro-nutrients, and finished feed.

Prior knowledge, based on ingredient data, allows assignment of the appropriate sampling pattern and sample size to different feed ingredients. This sampling pattern depends on the dimensions of shipment containers, conveying equipment, and sampling equipment.

Thus, a sampling program should be a dynamic process in which a company is always striving to achieve the greatest possible inference about different feed ingredients and finished feed at the lowest possible cost.

## Definitions

To facilitate a discussion of sampling, definitions of the different types of samples are presented below (Pierce 1994):

**Check Sample:** A sample that is carefully subdivided with portions sent to a number of laboratories for analysis and used as a check on laboratory assay procedures.

**Composite Sample:** A sample formed by compositing or accumulating and combining a number of discrete samples; useful in determining the average composition of a large amount, such as a shipload, carload, or truckload.

**Discrete Sample:** A sample representing a specific, usually small, amount of material. It also is known as an individual spot or grab sample and is useful in determining variations within a lot, adequacy of mixing, and other attributes that may vary throughout a larger amount of product or ingredient.

**Duplicate Sample:** A representative portion of an existing sample that is then provided to an additional laboratory and often used to resolve differences between laboratories.

**Official Sample:** One taken by a government official, either for regulatory purposes or to assign an official grade.

**Purchasing Sample:** A portion submitted by the supplier to a purchaser, purported to represent a lot offered for sale.

**Referee Sample:** A sample taken, often by an impartial sampler, and submitted to a referee laboratory for the purpose of arriving at a settlement between buyer and seller.

**Reference Sample:** A sample of known characteristics kept as a guide or comparison check for incoming ingredients and finished product. The reference sample may be used for visual comparison (e.g., color, texture).

**Retained Sample:** A duplicate portion of a lot retained in case an analysis is needed following use or distribution of the lot.

**Standard Sample:** One that has been carefully analyzed by experienced laboratories and provided to other laboratories as a means of standardizing or calibrating their methods or instruments.

**Working Sample:** The portion or portions of a sample used for analysis.

## Sampling Schemes

Common sampling schemes used in the feed and grain industry include simple random sampling, stratified random sampling, and systematic sampling.

A **simple random sampling** from a population of  $N$  sampling units gives equal probability to all units.

A **stratified random sample** is obtained by separating the population elements into non-overlapping groups, called strata. Then, a simple random sample is collected from each stratum. This typically is how shiploads are sampled — each hold represents a stratum and multiple samples are collected randomly within each hold.

A **systematic sample** involves random selection of one unit, and then repeated collection of sampling units at equal intervals thereafter. Systematic samples are easier to perform than a simple random sample and often provide greater information per unit cost than does simple random sampling. A diverter-type (D/T) mechanical sampler is an example of a systematic sampling system. The D/T is mounted in grain spouts, at the end of belts, or at the head of elevator legs, and the diverter moves through the grain (takes a cut) at timed intervals.

The feed industry uses a combination of these types of sampling schemes. Instructions for collecting an official sample of grain (GIPSA 1995) are listed in Table 1, and depicted in Figure 1. Bulk truck or rail shipments of grain or soybean meal are frequently sampled using a hand probe and employ a sampling pattern (Figure 1).

The bulk container may be stratified and sampled if there is an inferior portion of grain in the carrier (Table 2). Each sample is a minimum of 5 pounds (2 kilograms) in weight and is obtained in a balanced manner depicted below in Figure 2.

The prescribed procedure for sampling soybean meal at vessel loading facilities, defined by the National Oilseed Processors Association (NOPA), conforms to a systematic sampling design (Table 3).

**Table 1.** *The Grain Inspection, Processors, and Stockyard Administration sampling pattern that stratifies flat-bottom trucks or trailers for hand probe sample collection.*

Site A: Probe the grain approximately 2 feet (0.6 m) from the front and side.

Site B: Probe approximately halfway between the front and center, 2 feet (0.6 m) from the side.

Site C: Probe approximately  $\frac{3}{4}$  of the distance between the front and center of the truck, 2 feet (0.6 m) from the side.

Site D: Probe grain in the center of the carrier.

Sites E,F,G: Follow a similar pattern described above for Sites A,B,C for the back half of the carrier.

**Table 2.** *Instructions for sampling bulk carriers containing inferior portions of grain.*

Sample A. Probe the lot of grain as a whole (inferior and sound portions) as if both sections were in the same condition.

Sample B. Probe the portion containing the inferior grain thoroughly so a representative cross-section is obtained.

Sample C. Probe the sound portion to collect a representative sample.

**Bag** shipments of basemix, premix, and medicated feed articles should be sampled with a bag probe using procedures outlined in Table 4.

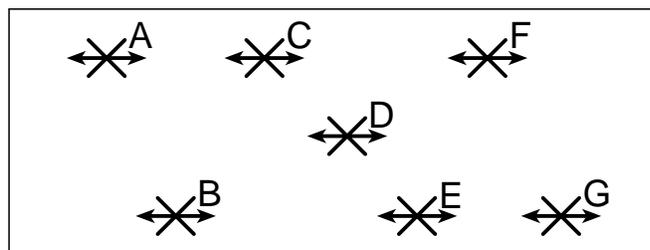
**Drums or barrels of liquid ingredients** such as fat or molasses can be sampled using a tube of glass or stainless steel,  $\frac{3}{8}$  to  $\frac{1}{2}$  inch in diameter and several feet long, referred to as a drum thief. Sample at least 10 percent of the containers and collect a minimum of 1 pint. Bulk shipments of liquid ingredients may be sampled using a bomb sampler or core sampler. In all cases, liquid ingredients should be subject to some stirring action (e.g., rolling drums) prior to sampling to ensure ingredient distribution.

**Forage** samples should contain several pounds of material. The sampling scheme, procedure, and sample preparation will vary depending on whether the material is a dry forage, silage, pasture, green chopped forage, or forage in the field.

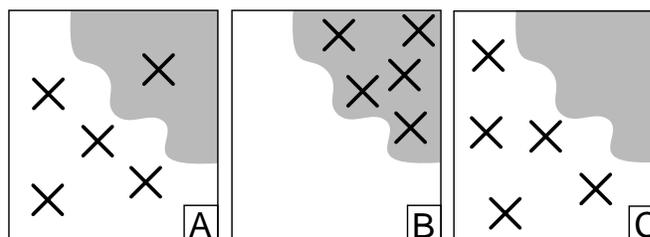
Collect **dry forage** from at least 20 different locations using a core sampler. If a core sampler is not available, hand (grab) sampling can be used. Try to avoid leaf loss when using this procedure.

Collecting **silage** samples from trench silos should be performed by removing a column 6 inches deep by 12 inches wide on the open face. Mix the silage and place a number of random handfuls in a plastic bag. Pack the sample tightly and seal the bag to exclude air.

**Figure 1.** *Sampling pattern for bulk carriers of grain*



**Figure 2.** *Sampling pattern for bulk carriers containing damaged grain*



**Table 3.** *NOPA procedures for sampling soybean meal at vessel loading facilities.*

- A. Sampling of soybean meal shall be done by an automatic mechanical sampler located in a spout or at the discharge of a belt conveyor, as appropriate. The sampler shall be designed to cut an increment from the entire cross section of the meal stream, perpendicular to the flow, at a location where the meal is flowing freely and at a uniform rate, in order to obtain the most representative sample of the meal flow. If the sampler is located in a spout, the spout slope must be 45 degrees or more from horizontal, and the flow must not be choked. When the diverter, or pelican, is stationary between cuts on either side of the meal stream, the opening shall be sealed to prevent dust from entering.
- B. The sampler system shall be located at a point beyond which no blending or addition to the product may be introduced prior to its being loaded onto the vessel.
- C. The activation of the sampler shall be regulated by an adjustable timer. When the average meal-flow rate through the sampler is less than 800 tons per hour, a sample, or cut, shall be taken for every 5 tons or less of meal flow. When the flow rate is between 800 and 1,200 tons per hour, a sample shall be taken for every 8 tons or less of meal flow. When the flow rate is 1,200 tons per hour or greater, a sample shall be taken for every 12 tons or less of meal flow. A minimum of 10 samples shall be taken during the loading of any one vessel.
- D. The diverter opening for cross-cut samplers and swing-type samples in which the diverter moves about a horizontal shaft (where the entire length of the diverter opening passes through the stream at the same speed) shall be of uniform width in the range of  $5/16$  inch to  $7/8$  inch. For rotary-type samples, in which the diverter moves about a vertical shaft and passes through the stream similar to a swinging door (with the outer end of the diverter moving at a higher speed than the inner end), the diverter opening width shall be a minimum of  $5/16$  inch at the end nearest the pivot, and shall increase in width in proportion to the distance from the pivot point. In all cases, the diverter shall cut the meal stream at an average speed of approximately 100 feet per minute.
- E. The sample taken by the automatic sampler may be reduced in size by one or more mechanical dividers, but the reduced sample must still be representative of the meal passing the sampler. The accuracy of the divider shall be equal in performance to a Jones or Boerner type divider. To comply with contract specifications, the entire sample may be further reduced through a Jones or Boerner divider or its equivalent. Then each portion of the sample must be placed in an official NOPA soybean meal sample bag.

**Table 4.** *Procedures for collecting bagged ingredients samples.*

Stand the sacks up on end and insert the probe into the top corner of the sack.  
Move the probe diagonally through the sack until the end of the probe touches the bottom corner opposite the top corner and withdraw sample.

Sample upright silos by taking random handfuls of silage from the unloader or wagon during the feeding period. Pack and seal as described above. Freeze samples that will not arrive at the laboratory within 12 hours.

**Pasture and field forage** sampling is difficult due to variations in soil fertility and moisture content. Randomly select eight to 10 locations for sampling, remove 1 square foot of forage at grazing height at each location, composite and mix subsamples, and reduce material to a 2-pound working sample. Immediately dry or freeze green pasture samples to prevent marked chemical changes.

For shipments of one to 10 bags, sample all bags; for shipments of 11 or more, sample 10 bags selected at random; for shipments less than five bags, collect at least five probes to gather enough material to perform an assay and retain a sample.

**Water** samples may be collected directly into a clean sample container from ponds, lakes, tanks, or other sources. Immerse the container, holding it neck down, to 1 foot below the surface, then turn it mouth up and allow the container to fill. Sample well water after extended pumping (2 to 4 minutes) to be sure the water being sampled has not been standing in pipes. Use a sterile container for samples from which a bacterial examination is performed.

**Finished feed** can be sampled as it is transferred to the delivery vehicle if feed is in bulk form. In the case of cattle feed that is mixed during transport, collecting the sample from the feed bunk is an acceptable practice.

Collecting feed samples from portable grinder-mixers during discharge into bulk feeders is a recommended practice, particularly when evaluating mixer uniformity (MF-1172).

### Sampling Equipment

Slotted grain probes may be used to collect a representative sample from grain, soybean meal, or finished feed. The grain probe should be long enough to penetrate at least  $\frac{3}{4}$  of the depth of the feedstuff. Official grain samples are collected using a  $1\frac{3}{8}$  inch diameter probe that consists of two tubes, one inside the other (Figure 3). The inner tube is divided into compartments that enable the individual collecting the sample to detect inconsistencies in grain quality across the profile (depth) of the carrier. This procedure is more labor-intensive since the contents of the probe must be emptied onto a tarp or trough and inspected before the grain is transferred into a container.

**Open-handed grain probes**, in which the inner tube is not divided into compartments, may be used for sampling feed ingredients including grain. The probe's contents are emptied from the handle end and mixing will occur, making it difficult to perform a visual inspection for load inconsistencies by depth.

An **open-handed spiral probe** is designed such that openings on the inside tube rotate around so it opens first at the bottom and then in gradual steps to the top. This assures a fair portion of the sample is collected across the profile (bottom to top) of the material.

Figure 3. Grain Probes

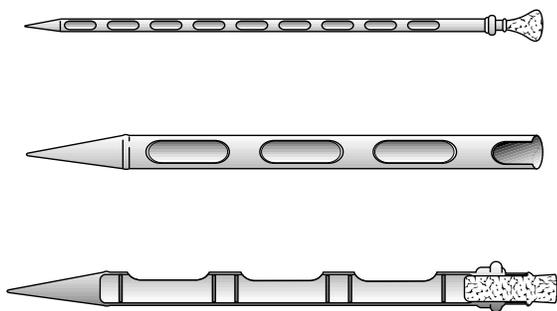
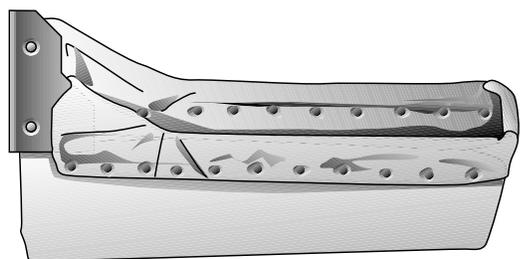


Figure 4. Pelican Grain Sampler



However, incorrect use of this probe can result in the opposite effect if the inside tube is rotated in the opposite direction, resulting in a disproportionate amount of sample collected from the top.

Probes come in standard lengths of 40, 51, 63, and 72 inches, 8, 10, and 12 feet. The probe should be inserted into the grain or feed ingredient at a 10-degree angle from the vertical, with the slots facing upward and completely closed. A 10-degree angle is used to obtain a cross section of material, while placing the end of the probe as close to the bottom of the carrier as possible. The slots must be kept closed until the probe is inserted as far as it will go. If the probe's slots are open as it enters the grain, a disproportionate amount of material from the top will fill the probe.

After the probe is fully inserted, open the slots and move the probe up and down quickly in two short motions. Close the slots completely, grasp the probe by the outer tube, and withdraw it from the grain.

The **Pelican grain sampler** (Figure 4) is used for on-line grain sampling by FGIS personnel. The Pelican is a leather pouch, approximately 7 inches deep and 18 inches long, with a band of iron inserted along the edge to hold the pouch open. The pouch is attached to a long pole. Pelicans are designed to catch grain as the pouch is swung or pulled through a falling stream of grain. The Pelican grain sampler would be useful for collecting grain, soybean meal, or complete feed samples while a truck is unloading.

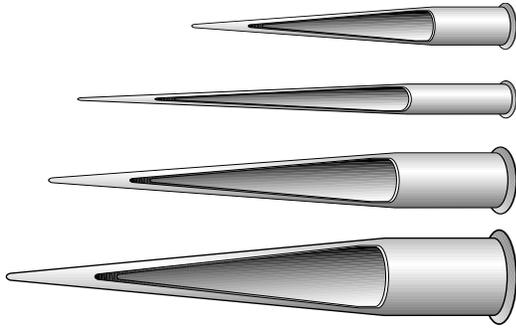
**Tapered bag triers** are constructed of stainless steel and are characterized by a sharp point, a tapered body, and an open throat. These triers are available in lengths from 6 to 12 inches (Figure 5.) Tapered bag triers are used to sample closed bags of powdered and granular commodities.

**Double-tube bag triers** are constructed of stainless steel or chrome-plated brass. These triers are available in various lengths and diameters, in both close-ended and open-ended models. These triers are used to sample closed and opened bags of powdered and granular ingredients.

**Single-tube, open-ended bag triers** are constructed of stainless steel tubing and are used to sample opened bags of dry, powdery commodities when removal of a core of material is desired.

**Bomb or zone samplers** are used to collect liquid ingredients from bulk carriers (Figure 6). These samplers consist of a closed cylinder ranging in size from 12 inches long by  $1\frac{3}{4}$  inch in diameter to 16 inches long by 3 inches in diameter; with capacities of 4 ounces and 32 ounces, respectively (Anonymous 2000).

**Figure 5. Tapered Bag Triers**



A valve lifts when the bottom of the tank is reached, or it can be manually lifted by a cord attached to the valve's plunger if intermediate depths are sampled.

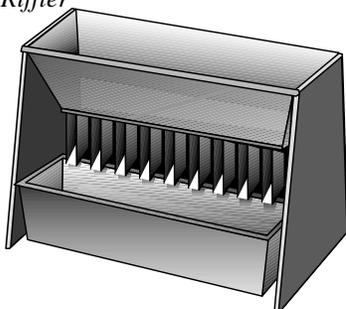
### Sample Reduction

The contents of each probe location should be mixed together prior to reducing the sample. Sample reduction may be performed using a riffler (Figure 7), Boerner Divider (Figure 8), or by quartering the sample (Figure 9). The end result of this process should produce a working sample of 1 to 2 pounds and a retained sample that should be kept for a predetermined time (usually until the meat animal is marketed and processed).

**Figure 6. Bomb Sampler**



**Figure 7. Riffler**



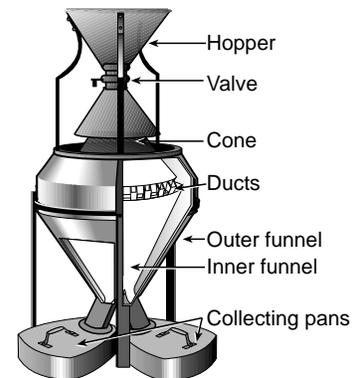
Complete feed and feed ingredients may be partitioned into uniform subsamples using a **riffler**. The sample is poured into the hopper, which is divided into equal portions by two series of chutes that discharge alternately in opposite directions into separate pans (Anonymous 2000).

The **Boerner Divider** is the grain industry's standard for splitting samples. A sample of grain is placed in the hopper and then released down a cone, where grain is cut into 38 separate streams, which rejoin into two streams and then empties into the pans (Anonymous 2000).

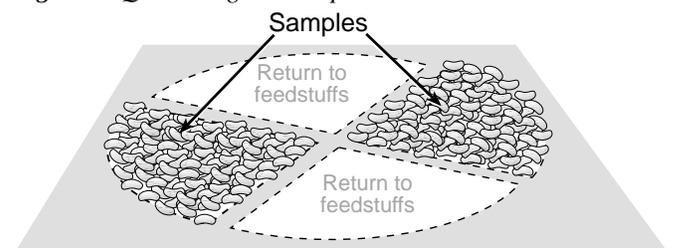
**Quartering** is a method for reducing the sample size of high-roughage feed (e.g., cattle feed) to a convenient amount for analysis. Spread the mixed composite sample on clean plastic or paper to form an even layer. Mark into quarters. Take two opposite quarters, mix, and repeat until the two quarters selected give the desired sample size.

Heavy plastic bags, zip-lock bags, or plastic containers with lids make excellent sample containers for dry ingredients or finished feed. Label samples as they are taken, identifying the date, sample number, and the contents (or ingredient to assay). Preservation of samples is highly important. Immediately freeze high-moisture feedstuffs, silage, or green forage. Store other materials in cool, dry locations.

**Figure 8. Boerner Divider**



**Figure 9. Quartering the Sample**



## Sampling Frequency and Retention

How often should **incoming ingredients** be sampled? With few exceptions, all ingredients should be sampled upon arrival and inspected for identity, physical purity, and compared with a reference sample. These samples, at a minimum, should be retained until the complete feed has been consumed by the animal and performed according to the label.

Commercial feed mills should collect and retain a sample of **complete feed** for each run of a given product. The sample should be retained as long as potential liability exists (e.g., until the meat animal is marketed and processed).

**Medicated feed** sampling and evaluation must conform to regulatory requirements. In the United States, the Food and Drug Administration (Hermann and Stokka 2000) requires feed mills licensed to utilize high-potency drugs that have a withdrawal period (Category II Type A) to perform routine assays. Three representative samples of medicated feed containing each drug or drug combination used shall be collected and assayed by approved official methods every year.

## Receiving Procedures

All feed processors should develop and follow a set of procedures for receiving feed ingredients. This should include inspection of the carrier's paperwork to ensure the correct material is on the carrier, a sensory inspection of ingredients collected from the sampling process, and documentation of receipt of those ingredients.

When receiving bulk material, inspect the shipping documents for ingredient identification, mill and supplier, and name of the individual hauling the cargo. Inspect the ingredient label and compare to previous labels. If no label accompanies the feed ingredients (with the exception of grain), do not unload the carrier until a label can be supplied. Check the label for the correct ingredient and analyses guarantees.

**Ingredients** should be examined for sensory characteristics (color, odor, texture, insect infestation, and moisture). This inspection procedure should be compared to reference samples or pre-established standards for comparison purposes. Do not unload ingredients that do not pass this initial inspection; particularly with bulk ingredients. Once the material is unloaded, you own it.

A receiving report that documents receipt of ingredients will augment a sampling program. This report should include the date, ingredient name, supplier, carrier name, license, bill of lading, purchase order or invoice number, time received, weight, bin number

where the ingredient was placed, sensory or physical qualities, and signature of the individual who unloads the material (Figure 10).

Procedures for evaluating physical (test weight, bulk density, foreign material) and nutritional properties of feed ingredients are described in Kansas State University Research and Extension publication MF-2037.

## Sampling for Livestock Health Problems

Proper treatment of a livestock problem depends on correct identification of the causative agent. A carefully compiled history will usually provide an adequate background for immediate and practical emergency treatment. A correct diagnosis of the causative agent usually rests upon sample assays of feed, water, surrounding environment, or animal tissue and body fluids.

A case report (Figure 11) form at the back of this bulletin lists important pieces of information to collect when livestock health problems occur (Wilcox 1972).

## Summary

Sampling is a critical part of any quality-assurance program. Steps involved with collecting a representative sample include following a sampling scheme; collecting enough sample to ensure it is representative; using the correct sampling equipment and procedure; inspecting the sample for its sensory characteristics; reducing the sample and preparing it for shipment, retention, or both; incorporating sampling into a structured method for receiving ingredients; sampling finished feed; and using sampling as a tool to help diagnose animal health problems.

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**Figure 10. Receiving Report**

RECEIVING REPORT		
Date Received		Date Unloaded
Commodity		
Shipper's Name		
Truck Name		
Trailer No.	PO No.	Bill of Lading
Weight Ticket No.		Net Weight
Bin No.		
Time In		
Time Out		
Unloaded By		
PHYSICAL QUALITIES		
Color _____		Odor _____
Texture _____		Moisture _____
Insects _____		
Sample No. Assigned		
Remarks		

Figure 11. Case Report Form

## Case Report Form

Date Report Prepared _____ Owner's Name _____ Address _____ City _____ State _____ Zip _____	Report Prepared By _____ Address _____ Telephone Number _____
If a company's product is in question, have they been notified? <input type="checkbox"/> Yes <input type="checkbox"/> No Date notified _____ Veterinarian's or fieldman's report is attached? <input type="checkbox"/> Yes <input type="checkbox"/> No Date called _____	
Sample Identification: (give each sample a number or letter) _____ _____ _____ _____	
Description of Case: (see suggested list of case information) Class of Livestock _____ Number _____ Age _____ Sex _____ Raised on Farm _____ If Purchased, When _____ Where _____ Describe symptoms in detail and date first noticed _____ _____ _____ Describe ration in detail and method of feeding. Include all changes made recently _____ _____ _____ Dates and details on every drug, vitamin, antibiotic, hormone, wormer, insecticide, vaccinations, disease-preventive shots or disease treatment used on livestock. Include castrations, dehorning, etc. _____ _____ _____ List any contact livestock could have had with poisons (refuse, trash pile, fertilizer sacks, paint cans, weed sprays, insect sprays, etc.) Any toxic weeds? _____ _____ _____ Weather conditions, especially sudden changes in preceding two weeks _____ Water supply checked for nitrates? _____ for salinity? _____ for potability? _____ Readily available to livestock? _____ Describe housing, lots and management problems _____ _____ _____ Other pertinent information _____ _____ _____ _____ I declare that to the best of my knowledge this is a true, complete and correct report.	
Signature of Livestock Owner _____	

Brand names appearing in this publication are for product identification purposes only. No endorsement is intended, nor is criticism implied of similar products not mentioned.

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