

## Sampling of feeds after Post-Pelleting Liquid Application (PPLA) treatment

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### Introduction

As the trend in feedmilling moves toward PPLA treatment of feeds with heat sensitive micronutrients, the challenge for the feedmiller is how to obtain feed samples for lab analysis of the micronutrients, which can truly represent the whole bulk of feeds that he produced.

Sampling can be defined as taking a small portion from a whole mass of feed that can accurately represent the whole mass.

An analyzed result of a sample can never be better than the submitted sample from which it was derived from. So what are the chances that the sample truly represents the lot of feed treated with the micronutrients in the PPLA system?

Thus correct sampling method & procedures are important because time & money are invested in the production of feeds itself, beside the costs of micronutrients that are going to reduce the cost of feed and improve performance of the diet and the cost of installing the PPLA system that is treating the feeds.

On the other hand, an unplanned and badly taken sample can flaw a good micronutrient and its supplier, an effective PPLA system, a good process control and finished product specifications.

### The Approach

Prior to taking feed sample, plan, prepare & familiarize yourself or the person who is collecting the samples with the following knowledge:

1. Wash your sampling tools & hands – this is to prevent carryover from hands to samples, because those hands might be in contact with the micronutrient itself or some sources of endogenous enzyme that can cause assay results to be 'fictitious'.
2. Make sure that proper polyethylene bags are used for containing the samples. Prepare to double-bag the samples for extra security during delivery.
3. Mark out the bags for at least 6 number of sample and 1 for a 'blank' or

'untreated' sample. Marking should identify the feedmill, the person collecting the samples, the date of sampling, the pellet line and the sample number.

4. Identify the proper & safe location for collecting samples.
5. When splitting or reducing the size of samples, use proper equipment like RIFFLER or BOERNER DIVIDER or the QUARTERING Method.
6. Do not expose the feed samples to heat sources or other extremities. Always store samples in a cool shaded place.
7. In filling the Lab Analysis form, specific the type of assay required, the micronutrient dosage rate, and any other useful details.
8. Send out the feed samples with the filled Lab Analysis form as soon as possible.

### The method – Composite Sampling

Composite sampling can substantially reduce analytical costs because the number of required analysis is reduced by compositing several samples into one. By appropriate selection of the composite sample size and retesting of select individual samples, composite sampling may reveal the same information as would otherwise require more analyses

When the objective is to estimate the population mean and total, compositing will always reduce analytical cost; however, a sufficient number of composite samples must be obtained for estimating the Co-efficient of Variance (CV) or homogeneity of a particular lot of feeds.

Compositing simply refers to physically mixing individual samples to form a composite sample, as visualized in figure 1. A single analysis is then performed on the

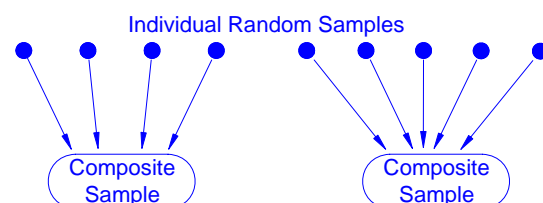


Figure 1: Forming Composite samples from individual random samples

composite, which is used to represent each of the original individual samples.

Generally, in the feedmill, the finished products are stored in their respective bins until they are discharged for bag packing or bulk loading onto trucks.

Referring to figure 2, just imagine you have a 20 ton finished product bin. And imagine you are able to 'cut' the population, literary, into 200 kg cubes; each cube represents 1% sample size of the population. And from each of the randomly selected 6 cubes, you grabbed 4 – 5 handfuls of feeds to form one composite sample of about 500 grams.

Analyzed results from of these 6 composite samples would tell you the mean recovery of the micronutrient, the CV or homogeneity of the micronutrient in that lot of feed, and the confidence level of that lot of feed being treated by the PPLA system.

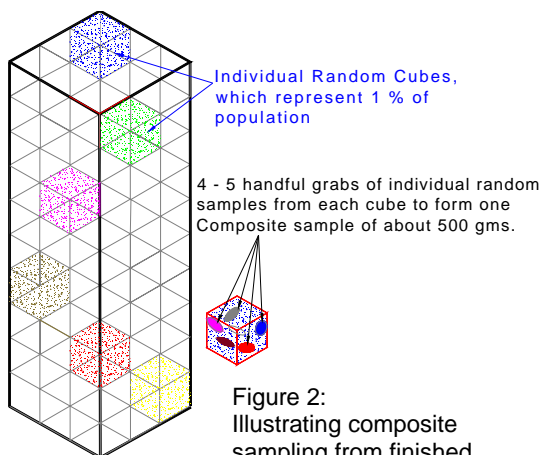


Figure 2:  
 Illustrating composite sampling from finished product storage bins, bulk trucks

However, if you need to send just ONE SAMPLE for micronutrient analysis to determine the mean recovery for the lot of feed, then from the 6 randomly selected cubes, take about 400 grams sample. Mixed them in a bucket or bag then use the Riffler or Boerner Divider to split and reduce the size to about 500 grams before sending it out to the laboratory.

If you do not have the abovementioned sample splitting equipment, then use the Quartering Method; This method require you to spread the feeds on a clean paper or plastic sheet to an even layer. The shape of the spread can be in a form of a circle or a square. Then divide the shape into 4 sections or quarters. Take opposite quarters to form a whole sample. You may have to

repeat the quartering method again to get to the right sample size of about 500 grams.

**The Procedure of Composite Sampling – In Actual Practice**

Drawing samples directly from a bulk container:

1. A 500 grams 'Blank' sample (without treatment of the micronutrient) should be collected prior to collecting treated samples.
2. Identify a safe location to collect the micronutrient-treated samples.
3. `Design & setup a sampling or probing pattern as illustrated in figure 2. By sizing up a bin, at least 6 numbers of predetermined 'cubes' can be located by its depth and position at the top.
4. With a slotted grain probes, take 4-5 samples from each predetermined spots.
5. Mixed the 4-5 samples collected from each spot together and reduce it to about 500 grams using proper Quartering method or splitting equipment.
6. Use pre-marked bag to contain each 500 grams sample and seal bag tightly. Double-bag the sample for extra protection during delivery.
7. When the 6 bags of micronutrient-treated samples are collected, send it out to the laboratory doing the analysis together with the Analysis Request Forms as soon as possible.

Sampling from a free-flowing stream of feeds:

The feed may be flowing through a spout or a conveyor belt. And sampling can be done by 'clean' hands or using a pelican sampler. Time intervals are used to 'cut' the population (the lot of feed) into segmented stream across the flow.

1. After taking a 500 grams 'Blank' sample before the PPLA system, proceed to identify the safe location to collect, at least 6 numbers of micronutrient-treated samples.
2. Set a time interval based on the feed flow rate to 'cut' the flow into segments of about 200kg (this is usually an estimate).
3. In a time interval segmented stream, grab 4-5 handfuls of feed in succession and placed the grabbed samples into the first marked bag.

4. Seal the bag tightly and allow for 3-4 numbers of 'time interval segmented stream' to lapse through before repeating step (3)
5. Repeat step (3) & (4) until you have at least 6 numbers of composite samples.
6. When the 6 bags of micronutrient-treated samples are collected, send it out to the laboratory doing the analysis together with the Analysis Request Forms as soon as possible.

Sampling from bags:

In some feedmills, bagging of the finished feeds into '50 kg bags' is not unusual. This bagging process actually provides the physical 'cutting' of the population or the lot of feed.

1. First, collect a 500 grams 'Blank' sample before the PPLA system.
2. After identifying the safe location to collect your samples. This is normally at the packaging line before the bags are sewn or sealed. Make sure that the treated finished feed is discharging to the correct bagging line.
3. Use 'clean' hands or a pelican sampler to grab an individual random sample of about 100 grams from the '50 kg bag' and put it into the marked sample bag.
4. Repeat this for the next 4~5 consecutive '50 kg bags', and placed the grabbed samples into the same marked sample bag until you have about 500 grams of composite feed sample.
5. You can mix the composite sample or leave it to the Laboratory to mix and grind them for assay.
6. Seal the sample bag and allow for a pre-determined interval (i.e. let 10-15 numbers of '50 kg bags' to lapse) before repeating step 3-5 until 6 nos. of composite samples are collected.
7. When the 6 bags of micronutrient-treated composite samples are collected, send it out to the laboratory that is doing the analysis together with the Analysis Request forms as soon as possible.

**Interpretation of Analysis Results**

It is a common practice to use results from feed samples to evaluate a micronutrient, a supplier, and also a PPLA system. Thus when sampling, consider the intent use of the results; the results must then be properly interpreted to be used effectively.

For micronutrient, the assay results must be compared to typical values for that micronutrient to determine if the micronutrient is acceptable for use in the feed diet.

The most common micronutrient used in today PPLA system is enzyme. And interpreting enzyme assay results could well be a challenge.

The example in figure 3 illustrates a Phytase (with a minimum guaranteed activity of 5000 U/gm) Assay on Broiler Grower feed samples; this had a mean Recovery of 652 U/kg (FTU). The Standard Deviation (SD) from the mean was 31 U/kg and the Co-efficient of Variance (CV% or homogeneity of PPLA treatment of Phytase) was 5%. Here all the results lie within 2 x SD from the mean, thus the level of confidence that the lot of feed was treated satisfactorily is 95%, where 2SD = 95%, and 1SD = 98%

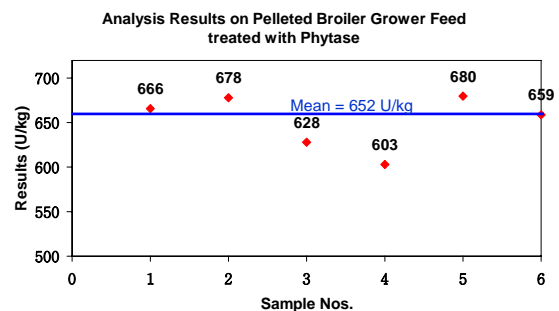


Figure 3: Phytase assay results on broiler grower feed

Typical value for Recovery of Phytase is 500 U/kg, at a dosage of 100 grams per ton of feed. However the above Mean Recovery was 652 U/kg, does that mean the system was over-dosing? Well, most enzymes are sold on a minimum guaranteed level of activity that is normally lower than the actual activity of the enzyme delivered. That is why, as a counter check it is important to tally the consumption of enzymes to the theoretical usage for that lot of treated feed where the samples came from.

Another example in figure 4 illustrate assay of Phytase (with a minimum guaranteed activity of 5000 U/gm) on Duck Grower feed samples; Mean Recovery was 434 U/kg, Standard Deviation from the mean was 34 U/kg with a Co-efficient of Variance of 8%. Confidence level was 95% that this lot of feed is treated satisfactory with the PPLA system.

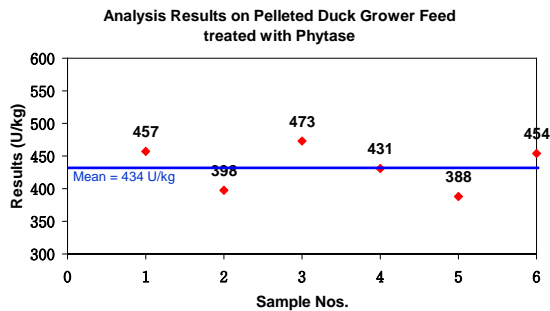


Figure 4: Phytase assay results on duck grower feed

Again, when comparing the mean recovery of 434 U/kg with typical value of 500 U/kg based on '100 grams per ton of feed' dosage. One may think that the PPLA system is under-dosing; however, if the actual usage of phytase tallies with the theoretical usage for that lot of feed where the samples are drawn from then consider the other probable causes:

- Analysis is often problematic due to the strong binding of the enzymes to some feed substrates that prevents complete enzyme extraction prior to assay.
- Enzymes, like all other proteins are susceptible to degradation by environmental factors like the extremes of pH, temperature, excessive friction, microbial growth and a range of others.

The importance of accuracy of dosing is obvious: too low overall addition level would decrease the effect of the additive and thereby damage the quality of feed, too high a dosage level would be unacceptable from an economic point of view as the micronutrients under consideration are usually of a high value.

McCoy et al. (1994) concluded that out of a mixing uniformity experiment with broiler chicks that depending on the uniformity test used, CV of up to 20 % may be adequate for maximum growth performance. According to Schwarz (1998b), a variation coefficient of less than 20% for phytase should be sufficient.

Based on these information and most importantly, the users' own experience with the animals' performance in the farm, a mean Recovery of no less than 80% and a CV of no more than 20% is an acceptable standard in the industry.

## Summary

Most feedmillers would want a peace of mind that the samples are a truthful reflection of the whole lot of feed from which the samples came from. They also want a peace of mind that the PPLA system is performing satisfactory to their expectation, and the micronutrients are homogenous in their feed in the right amount. Failing in any of the above, would be disastrous in the performance of animal in the farms especially so, when the diet is 'down-spec' and some critical ingredients are replaced in the diet formulation.

Thus, beside having a robust, fail-safe, efficient and accurate PPLA system, correct sampling method & procedure in getting representative samples are as important as producing the feeds itself.

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