i'Tec_H10
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Determining the mean concentration of tracer in a batch of heterogeneous animal feed

During tests to assess the amount of carry-over in delivery trucks, it became apparent that the best approach would be to take a 200-kg sample with a heterogeneous distribution of tracer (Technical Datasheet No.49). One would expect that this heterogeneous sample would have to be homogenised with a mixer before testing it in order to determine the mean concentration. This would distribute the tracer evenly throughout the batch, meaning that the samples could be considered representative of the mean concentration of tracer in the original heterogeneous mix. The drawback of this protocol is that it requires both major investment in a mixer and a space-intensive installation.

1. Focus

This study has been designed to assess an alternative sampling protocol that uses a sample splitter system. These devices have to be able to produce a fraction of the heterogeneous batch that is representative of the concentration in tracer for this batch. This protocol would require equipment that is far less cost and space-intensive. The protocol is based on three methods, one that mixes and two that divide the samples, applied consecutively in order to ensure meaningful results.

2. Principle

These tests simulated the heterogeneous batch, and determined the mean concentration as follows:

- Either by computation
- Or after homogenising and/or dividing the batch
- Three heterogeneous batches of piglet feed were made up using fractions from homogenous batches with a known concentration of tracer:
- The first of these three batches was homogenised in a mixer, after which samples were taken from 8 fractions.
- This homogenised batch was then placed in a 1/16 sample splitter in order to obtain representative samples that could be compared against those produced by direct passage through this splitter.
- The second heterogeneous batch was divided in a 1/16 sample splitter and the results compared against those of the homogenised feedstuff that had also been divided using this splitter.
- The third heterogeneous batch was divided successively using a 1/2 divider, in order to obtain a sample set that was sized for analysis.

3. Apparatus and Equipment

3.1. Equipment

- 1/16 sample splitter
- ½ conical divider
- ½ riffle splitter
- Mixer with reverse pitch double screws - capacity 224 litres
- Scales
- Big bag (capacity 1000 litres)
- Small equipment (pots, scoops, etc.)

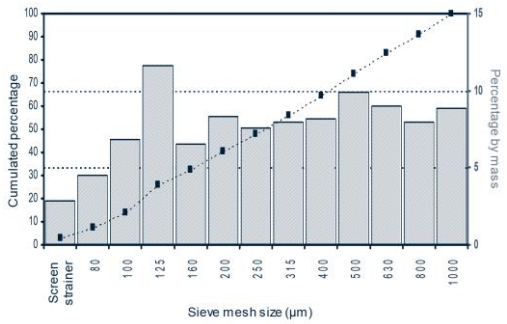
3.2. Feedstuff

Several quantities of homogenised feedstuff were prepared. These contained decreasing concentrations of microtracer:

- 3 x 20 kg feedstuff at 500 ppm
- 3 x 20 kg feedstuff at 250 ppm
- 3 x 20 kg feedstuff at 100 ppm
- 3 x 20 kg feedstuff at 50 ppm
- 3 x 20 kg feedstuff at 10 ppm
- 3 x 100 kg feedstuff at 0 ppm

The properties of the piglet feed used as a baseline feedstuff are given below.

Median diameter	323.3 µm
Standard geometric deviation	*2.23*
Bulk density	704.5 **g/cm^3**
Angle of repose	60.3°



Physical properties of piglet feed

4. Method

4.1. Analysing tracer concentration in feedstuff batches

This analysis uses a method designed by Tecaliman (Technical Datasheet No.46). The analysis uses RF-blue lake tracer, which is dosed using colorimetry.

4.2. Making up heterogeneous feedstuff batches

The feedstuff batches were poured into a 1000-litre big bag, in decreasing level of concentration. During this study, in order to replicate industrial processes as far as possible, the feedstuff was poured into the big bag through a mixer in drain mode.

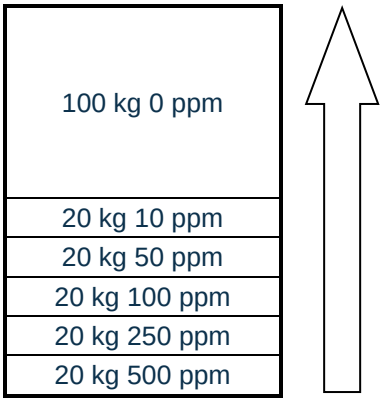


Figure 1: Make-up of the heterogeneous feedstuff batches

The heterogeneity of this mix was comparable to that of a feedstuff batch in which tracer concentration drops rapidly, similar to the effect obtained during the truck tests.
Based on these quantities and concentrations, the theoretical mean concentration of tracer in the heterogeneous batches was estimated at 91 ppm.

4.3. Sampling with a mixer

As the mixer used in this study only had a capacity of 224 litres, it was not possible to mix the 200 kg of feedstuff in one go. It was therefore necessary to cascade-mix 100-kg fractions, recombining 50-kg subfractions. After completing the mixing operation, the 200 kg of feedstuffs were collected in eight 25-kg bags. Two test portions were then taken from each bag (Figure 2), producing 16 samples.

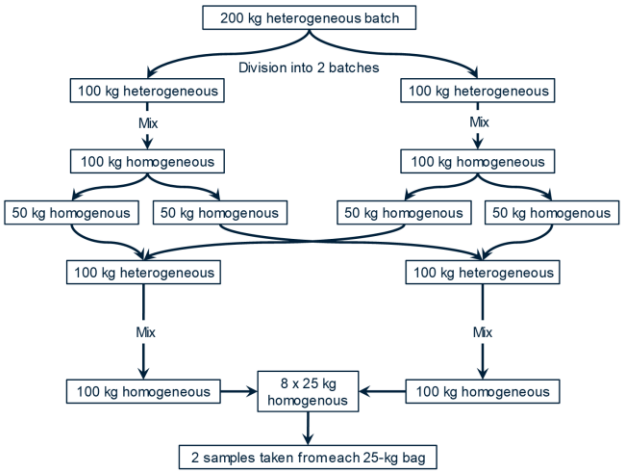


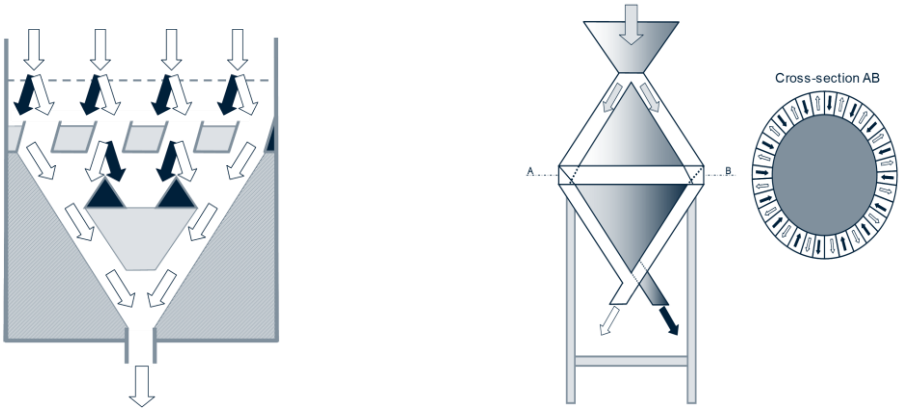
Figure 2: Sampling steps with the mixer

4.4. Sampling with a splitter

4.4.1. 1/16 sample splitter

The 1/16 sample splitter comprises a variable tilt plate with raised edges. The feedstuff is placed at the top of the plate. A feed system then distributes the feed over the whole width of the plate. During its descent, the feedstuff runs past four consecutive rows of splitters, each one of which divides the feed portion in half. Half of the feedstuff falls through the holes (Figure 3 - black arrows) while the other half continues to fall onto the row below (white arrows). Theoretically, by the time it has finished its descent one sixteenth of the feedstuff should have been collected.
Before starting the actual study, several test runs were made demonstrating that the coefficient of division was not in reality one sixteenth, and that the actual quantity obtained was approx. 1 to 2% rather than the expected 6.25%.
Having checked the apparatus, it appeared that these results were due partly to incorrect positioning of the holes and splitter elements, and partly to the flow rate being too low at the end of the drop. This drop in flow rate makes it easier for the feedstuff to exit.
After changing the plate's tilt angle and blocking certain holes (Figure 3), the final percentage equalled 6 to10%.

During the study, the feedstuff was sampled at the top of the big bag, and then poured through the sample splitter. The first of the resulting fractions was collected, and then poured through the splitter again.



1/16 sample splitter 1/2 conical divider

Figure 3: Diagram of the sample splitters in their operational state

4.4.2. ½ conical divider

The principle of the ½ divider is simpler. The feedstuff is poured through a funnel connected to a container that can hold a few litres. This container comprises a metal cone over which the feedstuff slides before arriving at a circular section that surrounds the cone (Figure 3). This circular section comprises many radial openings that are alternately connected to two separate chutes (see cross-section AB).
During the study, another sample of feedstuff was taken at the top of the big bag, and then poured into the divider. The resulting fraction was then poured through the divider again. This operation was repeated 7 times.
For each divider, the final fraction had to weigh between 700 g and 2.0 kg. This final fraction was weighed to establish how many test portions could be produced (test portion of between 90 and 250g). Depending on the mass obtained, this fraction was then passed through a riffle splitter in the lab 3 or 4 times (halving - ½ division) in order to obtain 8 or 16 samples.

4.5. Data processing

The assessment criteria for each protocol concern the mean, the coefficient of variation and the recovery rate. Student's t-test was used to compare the means, taking account of measurement variability.

5. Results

5.1. Expected concentrations

Analysing the homogenous batches earmarked for preparation of the heterogeneous batches made it possible to calculate the actual expected concentrations, i.e. 96.0 ppm for tests using the mixer and the 1/16 sample splitter, and 94.7 ppm for the test using the ½ divider.
The means shown correspond to the expected concentrations for each test, calculated based on the masses used and the concentrations of the homogenous batches.

5.2. Results

Table 1 shows the results of all the test analyses

	Mean (ppm)	CV (%)	Recovery rate (%)
Mixer	97.7	3.8	101.8
1/16 splitter (heterogeneous batch)	110.2	4.1	114.8
1/16 splitter (homogenous batch)	92.9	2.0	95.1
½ splitter (heterogeneous batch)	96.4	1.8	101.8

Table 1: Results of the analyses

5.3. Mixer

This test was designed to check the validity of the sampling protocol for a homogenised batch. The results demonstrated that this method is indeed valid. The expected mean was 96.0 ppm, while the actual mean was 97.7 ppm. This gives a recovery rate of 101.8%.
A Student t-test comparison of the measured mean against the expected mean (95% confidence interval) did not reveal any significant difference between the two means.
The samples can, therefore, be considered as representative of the batch. In addition, the results gave a satisfactory coefficient of variation, confirming the homogeneity of the batch when the samples were taken (3.8%). While the result of this mix-based sampling protocol can be considered valid, a major drawback remains in that it requires a mixer of this capacity.

5.4. 1/16 sample splitter

5.4.1. Heterogeneous batch

A mean concentration of 110.2 ppm was recorded. This was significantly higher than the expected

mean of 96.0 ppm, giving a recovery rate of 114.8%. A Student t-test was used to compare the measured mean against the expected mean.

These results would suggest that this splitter is not suitable for sampling a heterogeneous feedstuff batch.
However, the coefficient of variation for the measurements was correct (4.1%). The problem was not therefore the heterogeneity of the final samples, or inaccuracy of the analyses, but rather the representativeness of the final sample obtained after two runs through the splitter.
One possible assumption is that the splitter selects fine particles containing the tracer by a sieving effect. Another possible assumption would be that this splitter does not provide a representative fraction. In other words, perhaps the splitter is randomly allowing a larger quantity of a certain part of the heterogeneous batch to pass through, thereby producing an imbalance in the final fraction. For example, during the test, a greater quantity of feedstuff at 500 ppm may have passed into the extracted sample thus giving a final concentration that is too high.

5.4.2. Homogenous batch

The purpose of this test was to check post-test assumptions on the heterogeneous batch, and to find out whether the 1/16 sample splitter can actually produce a fraction that is representative of a homogenous batch.
The batch used for this test was that sampled during the mixer test.
As before, the expected mean was 96.0 ppm, and the actual mean 92.9 ppm. These concentrations only differed by 3.1 ppm (recovery rate: 95.1%) However, Student's t-test revealed that the measured concentration differed significantly from the expected concentration (confidence interval of 95%). Similarly, a comparison of the mean obtained after the mix against that obtained after splitting revealed a large difference.
While the recovery rate suggested that the resulting fraction was representative of the original batch and the 2.0% coefficient of variation indicated that the resulting fraction was homogeneous, the statistical results nevertheless demonstrated that this splitter does not provide samples that are sufficiently representative of a homogenous batch. The assumption that the splitter would cause a sieving effect was disproved, as the concentration obtained with this test was below the expected concentration. It is the second assumption therefore, that this splitter may randomly allow the passage of a larger quantity of a certain part of the batch, that should be chosen.

5.4.3. ½ divider

This last test was performed to determine whether the unsatisfactory results obtained with the 1/16 sample splitter derived from the splitting procedure or the splitter model used. The expected theoretical concentration was 94.7 ppm. The mean concentration was 96.4 ppm, i.e. a recovery rate of 101.8%, similar to that obtained with the mixer test. A Student t-test comparison of the measured mean against the expected mean (95% confidence interval) did not find any meaningful difference between the two means.
The coefficient of variation was very good, of the same order of size as the mixer test.
This demonstrated the effectiveness of forming samples by division using this type of sample divider.

6. Conclusion

The results obtained by putting the heterogeneous batch through a mixer were conform to expectations. Using a heterogeneous feedstuff batch, this methodology makes it possible to obtain test portions of a concentration that are equivalent to the mean concentration of the original batch.
The study of sample splitting procedures demonstrated the ineffectiveness of the 1/16 sample splitter, already set up at Tecaliman and used under a special set of conditions (blocking of a number of holes), in extracting a representative sample of a homogenous or heterogeneous batch of animal feed. It would appear that the splitter's design and geometry produce a feedstuff flow rate that varies in space and time, resulting in poor feedstuff distribution and a non-representative sample.
This does not mean though that the use of a splitter should be ruled out as the ½ divider gave satisfactory results, similar to those obtained with a mixer.
These observations suggest that a representative sample of a heterogeneous batch can be produced by dividing the sample, provided that a suitable sample splitting device is used.

7. Bibliography

i'Doc_T8, 2003 - Elaboration d'une méthodologie pour l'évaluation des contaminations croisées par des additifs dans un camion de livraison d'aliment du bétail
i'Tec_T8, 2003 - Règles techniques pour l'évaluation des contaminations croisées dans des camions de livraison.