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***English translation of article in German language***

# "Simultaneous Carry-Over Analysis of Various Locations before the Mixer with Microtracer"

Determining the mixing accuracy and degree of carry-over using Microtracers is a widely accepted standard for quality control in the manufacture of premixes and animal feed.

This article describes the option for checking the homogeneity of a test mix in a batch, differentiated according to the various locations before the mixer where the feed is added for production. In addition to identify the degree of mixing accuracy, it also determines the actual percentage of carry-over for each location.

## Introduction

Premix and animal feed manufacturers produce their products consecutively one after the other. Good mixing quality and very little carry-over from one production batch to another are therefore essential requirements for maintaining good quality standards.

Animal feed ingredients can be added to a mixer from various locations, either manually through a handtipping system or through microdosing silos, for example.

It is essential to determine the mixing accuracy in animal feeds for economic as well as ethical reasons so that the feed being produced does not contain too much or too little of the formulated components. Pursuant to EU Regulation (EC) No. 183/2005 "Feed Hygiene Regulation", authorities require that the carry-over of individual components from one batch to the next must be kept low in order to protect animals against health complications.

A reliable and accurate, but also fast and practical method, is therefore essential in order to determine the homogeneity and degree of carry-over.

Microtracer analyses are used in over 100 countries, including Brazil, EU, Japan, Mexico, and the United States. It has been integrated into the Dutch GMP+ and Belgium Ovocom quality assurance systems since 2006 and is recognised by over 50 national and international authorities. The analysis performed by MTSE (Micro Tracer Services Europe GmbH) provides reliable, accurate results with the Microtracer Analysis System that can be performed on-site or in the MTSE laboratory. The Microtracer particles consist of iron particles of the same size coated with internationally approved food-grade colours. The Microtracer FSS-lake used in this study is a

tracer with a mean particle size of 100 µm, coated with water insoluble so-called “lake” dyes. This method is suitable for regular use, because it doesn’t interfere in the production process. Since the particles and their food-grade paint are not toxic, all of the test batches remain usable afterwards.

Simultaneous use of Microtracers in different colours make it possible to optimize different mixing times within one production batch. In this report the advantage of the use of three different Microtracers which were added to three different locations before the mixer in production batch is described.

**Microtracer Method of Analysing the Mixing Precision and Carry-Over**

The Microtracers are added during production as a premix at doses of 10 g per ton of feed (mixing accuracy 1:100,000) in 4 kg of limestone. Two batches are analysed: The first is analysed for homogeneity, the second is analysed without the addition of Microtracers to check for carry-over level.

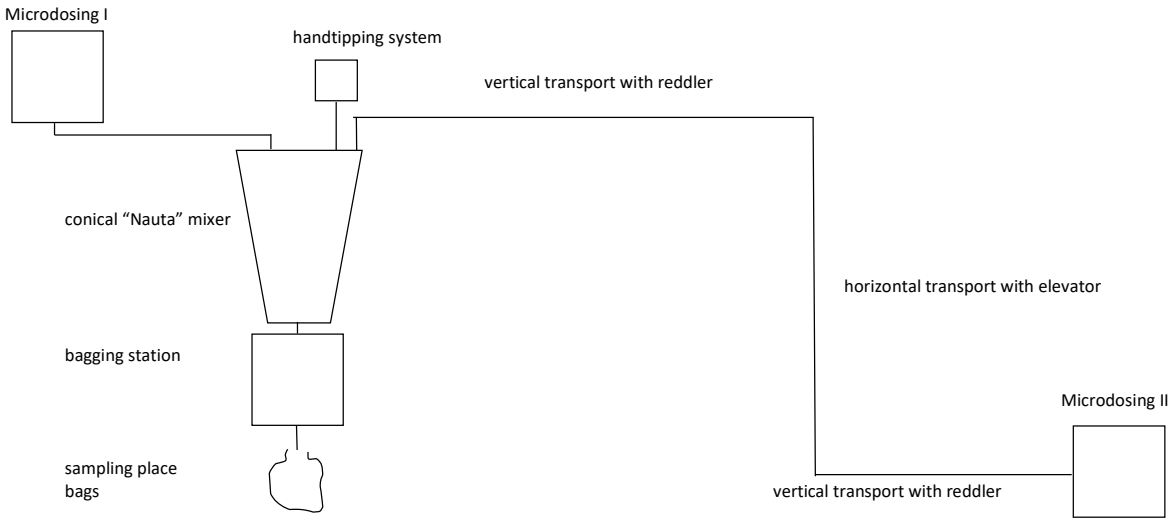
In this report, a production line was inspected with a conical “Nauta” model mixer. Figure 1 shows an overview of the mixer and the various transport systems. The locations for the three Microtracers added are marked:

a) Microdosing I – FSS-red lake
b) Microdosing II – FSS-green lake
c) Handtipping system FSS-blue lake

Time of addition into the mixer:
1.) Microdosing I

2.) Microdosing II and handtipping system one right after the other.

**Figure 1: Schematic Overview of the Production Line**



In the example being analysed here, 14 samples were taken from bags of batch 1 for the determination of mixing accuracy, and 19 samples were taken from bags of batch 2 for carry-over analysis.

The so-called rotary detector® was used to separate the Microtracer particles from the feed sample for analytical purposes. A development solution was used to make the food-grade colour chromatographically visible on filter paper and the particles were then counted using computer-controlled TraCo® software. The analysis is done in accordance with the principles of Poisson distribution for counting individual occasional events happening separately from each other (here: particles). The following criteria of probabilities p for determination of the homogeneity are applicable:

> 25% – excellent mix; 5% < p < 25% – good mix; 1% < p < 5% – acceptable mix; p < 1% – inhomogeneous mix.

**Results of the Analysis of Microtracer Particles**

Under the test conditions described, batch 1 achieved a good or excellent mixture for the addition places microdosing I and manual handtipping system. A mixing accuracy of 1:100,000 in the mixer is not achieved for the addition place microdosing II (probability 0%, corresponding to an incomplete mixture).

The carry-over rates from manual handtipping system to the bagged product was calculated to 5.4 %. The calculated value for carry-over of microdosing I into the bagged product amounted to 5.9 %, while the carry-over from microdosing II to the bagged product showed a carry-over of 13 %. Based on these values, subtraction yielded a carry-over from microdosing I to the mixer of 0.5 % and from microdosing II to the mixer of 7.6 %.

It must be noted that the determined degree of carry-over for microdosing II in batch 1 is not significant, since the incomplete mixture may cause the calculated value to differ considerably from the actual value here.

Tables 1 and 2 summarise the results of the measurements.

**Table 1: Mixing accuracy**

| Addition place of Microtracer | Microdosing I | Handtipping system directly into mixer | Microdosing II |
|---|---|---|---|
| Mixing time, s: | 95 s | 95 s | 95 s |
| Sampling place | bags | bags | bags |
| Number of samples | 14 | 14 | 14 |
| Sample assayed, g: | 44,1 | 44,1 | 44,1 |
| Tracer colour: | red | blue | green |
| Tracer used per metric ton, g: | 10 | 10 | 10 |
| Mean particle count, particles: | 159 | 58 | 116 |
| $Chi^2$-square value | 21,1 | 8,78 | 54,42 |
| **Probability, %:** | **7,1** | **79** | **0** |
| **Recovery rate, %:** | **93** | **98** | **125** |
| Mixing is | good | excellent | incomplete |

**Table 2: Degree of carry-over**

| Addition place of Microtracer in homogeneity batch | Microdosing I | Handtipping system directly into mixer | Microdosing II |
|---|---|---|---|
| Sampling place | bags | bags | bags |
| Number of samples | 19 | 19 | 19 |
| Sample assayed, g: | 1167 | 1167 | 1167 |
| Tracer colour in homogeneity batch | rot | blau | grün |
| Particle count for 100 % carry-over | 4199 | 1543 | 3082 |
| Mean particle count, particles: | 248 | 83 | 416 |
| **Mean carry-over level, %:** | **5,9** | **5,4** | **13** |

**Discussion of the Determined Mixture Accuracy and Carry-Over Levels**

The measuring results give information about the mixture accuracy and carry-over rates of components from various locations before the mixer used in the production line.

In order to achieve a complete mix for the microdosing II location, the mixing parameters of the mixer must be optimised. This particular case required monitoring of the transport from a to b. The long path from microdosing II to the mixer and the time at which they were introduced to the mixer resulted in an incomplete mixture in the bags. The components of microdosing II were the last components added to the mixer. As a result, the subsequent mixing time of 95 seconds was not enough to achieve a complete mixture. It was therefore necessary to increase the mixing time.

Using the calculated degree of carry-over, the production line should be carefully inspected in order to identify problematic pathways responsible for no complete mixing and for holding back of individual components.

The producer can make improvements based on these identified problem areas and measures to be taken could include:

- Regular identification of CCPs (Critical Control Points) using set up corresponding monitoring processes at these critical control points
- Introduction of flushing batches (with possible sacrifices in efficiency, since batches will have to be prepared and stored separately)
- Construction-related changes (e.g. replacing parts of the installation that have been worn out by use; shortening the feed paths or installing new machinery)

**Outlook**

Reliable analytical methods such as the Microtracer analysis are increasingly being used to determine the mixing accuracy and degree of carry-over, because they are simple, inexpensive, and fast to execute. As was shown in this example, Microtracers in various colours can be used to identify carry-over risks in a production system.

All companies involved in the production and processing of feed and food now and in the future follow the HACCP concept for quality assurance, which calls for regular monitoring of so-called critical control points in a production system. These critical control points can be fast and accurately identified with the Microtracer Analysis System.