i'Tec_T9
May 2005

Keywords: Carry-over, Plant, Tracer

Dual-tracer analysis of animal feed carry-over patterns during plant manufacture

This test was designed to study changes in carry-over patterns at four sampling stations at a given industrial site, both before and after pelleting, using two tracers: Chlortetracycline (CTC) and the micro-tracer RF-Blue Lake (MT). This site was the same one used to study trends in compliance and homogeneity (i'Tec_H12).

1. Principle

The two tracers were incorporated together through a single premix that was injected into two tracer batches via the standard additives incorporation circuit. Next, two collector batches initially free of tracer were manufactured. These 4 batches were sampled:

- at the mixer output (SM)
- at the entry to the silo bin upstream of the press (EB)
- at the press conditioner output (SB)
- at the entrance to the finished product bins (EC)

These samples were used to assess changes in carry-over patterns throughout the plant.

2. Equipment and apparatus

2.1. Tracers

The two tracers (Table 1) were incorporated using a 0.8% premix (8 kg/ton) in the final feed product. The microtracer and the chlortetracycline were incorporated at dosages of 250 ppm and 800 ppm respectively.

	MT	CTC premix
Bulk density (g/cm^3)	2.976	493.7
D50 (Sieving) (µm)	65.1	217.2
Standard geometric deviation	*1.47*	*1.59*
Angle of repose (flow) (°)	29.6	70.6

Table 1: Tracers - physical characteristics

2.2. Feed

Four 2.5-ton batches of medicated pig feed with a liquid content of approx. 1% were used. The physical characterisations of the two tracer batches are shown below (Table 2 - Figure 1).

	L1	L2
Bulk density (g/l)	630.6	641.9
D50 (Sieving) (µm)	436.0	410.3
Standard geometric deviation	*2.16*	*2.15*
Angle of repose (flow) (°)	64.5	62.6

Table 2: Physical characteristics of the animal meal

L1 – SM **L2 – SM**

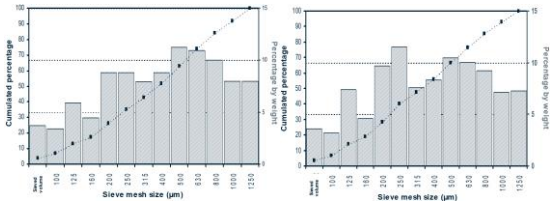

Figure 1: Animal feed particle size spectra

2.3. Circuit

The following equipment was set up between the various sampling stations:

Premix incorporation point
Weighing bin
2 Hatches
Hopper
Lock
Pneumatic conveyor belt
Cyclone
Lock
2 Hatches
Hopper above the mixer
Mixer
Hopper below the mixer
Chain conveyor

Mixer output (SM)
Elevator
3 distribution boxes
Chain conveyor
Bin
Screw
Molasser
Elevator

Entry to the silo bin upstream of the press (EB)
Distribution boxes
Silo bin upstream of the press
Hopper
Feed screw
Conditioner

Press input (SB)
Press
Cooler
Elevator
Chain conveyor

Entry to the loading bin (EC)

3. Method

3.1. Testing

The premix containing the tracers was added to the two tracer batches. Data on the weighings taken during manufacture of the 4 batches and the pelleting conditions was collated.
Sampling points were chosen so as to ensure that they were located in passing flows. The aim was to collect around 40 samples taken from every batch and at all 4 sampling points.
Sampling times ranged from 5.5 seconds to 37 seconds depending on the sampling station. At mixer output, samples were taken using an on-site cross-sampler. At the entrances to the silo and the bin the samples were taken directly in sampling pots. At the entry to the press (silo bin output), the samples were taken using a plastic hand and transferred to the pots.

3.2. Processing the samples

The sample processing procedure conforms to the recommendations set out in the technical rules (i'Tec_T2), and consists in grouping aliquots of each sample taken. Given the large number of samples (40), these groupings were made based on the model: 3/28/9
For pellet analyses (EC), each primary sample was ground separately using a roll mill prior to grouping.
The microtracers were extracted magnetically based on the protocol described in i'Tec_H17; optical density was determined following solubilization. Tracer quantity was calculated via its linear relationship with an optical density of 629 nm. Chlortetracycline underwent HPLC-analysis based on a protocol that enables detection up to a threshold of 0.2 ppm.

3.3. Data processing

The data were processed according to the recommendations set out in the technical rules in i'Tec_T2.

4. Results

4.1. Conditions governing batch manufacture

The die was less than one month old, and therefore in the first quarter of its lifetime. The die had a thickness of 58 mm and was pierced with 6,720 4-mm diameter holes, giving a compression index of 14.5. The two rollers had a honeycomb structure. Pelleting conditions were recorded (Table 3). The first tracer batch had a difficult start due to jamming. The other batch pelleting operations proceeded smoothly.
Moisture content analyses were carried out on aggregate samples made up from each batch at all 4 sampling stations. The results demonstrated that there were only minor fluctuations in moisture content. These fluctuations were corrected in order to be able to track changes in tracer concentrations at a constant moisture content value.

	L1	L2	L3	L4
Flow (t/h)	5.2	6.6	6.5	6.5
Temp. at conditioner output (°)	58	64	64	64

Table 3: Pelleting conditions

Batch durability and the percentage of fines were determined (Eurotest) post-test using aggregate samples made up from the two tracer batches. While a slight variance in the percentage of fines was recorded, batch durability was comparable.

	% of fines	Durability
L1	0.9	90.4
L2	1.6	90.3

Table 4: Percentage of fines and durability for the first two batches

4.2. Changes in carry-over patterns

Table 5 gives the results of carry-over assessments for both tracers according to sampling location.
The collector and tracer batch analysis results clearly demonstrated that, irrespective of the sampling station, product losses recorded in both tracer batches were never equal to product gains measured in the collector batches. Expressed as the cumulated masses for both tracer batches, the missing quantity of tracer increased overall according to distance from the sampling stations (Figure 2) until reaching nearly 900 g for the chlortetracycline base and 600 g for the microtracer.

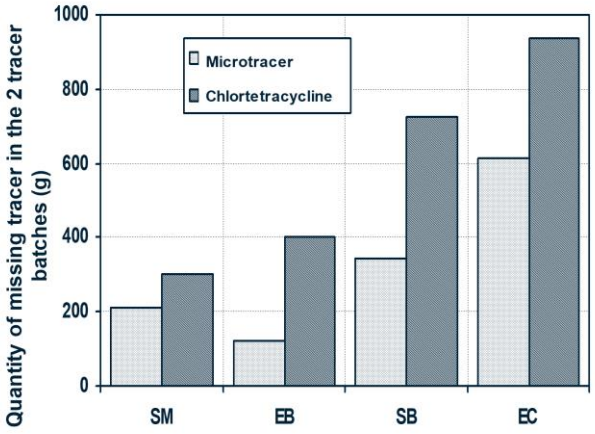

Figure 2: Quantities of missing tracer in the 2 tracer batches for both tracers according to sampling point

Cumulating the quantities in both collector batches (Figure 3: Quantities of tracer recovered in the 2 collector batches for both tracers according to sampling point) gave a very different result:

- Tracer recovery did not increase in relation to sampling point
- Tracer recovery appeared only partial. After converting to a percentage, recovery was approx. 30% at most for both tracers at the entrance to the silo bin upstream of the press, and approx. 10% at the other sampling points (Figure 4).

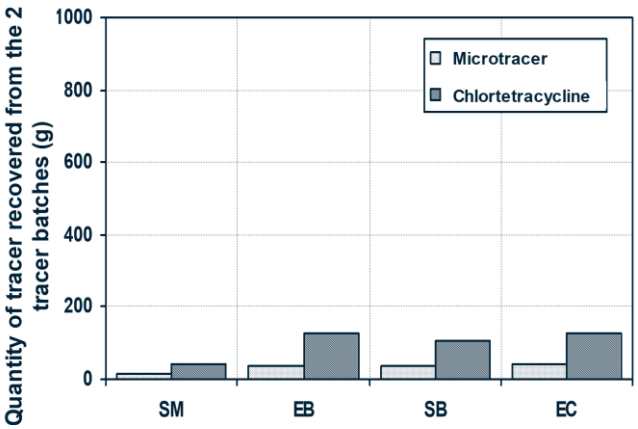

Figure 3: Quantities of tracer recovered in the 2 collector batches for both tracers according to sampling point

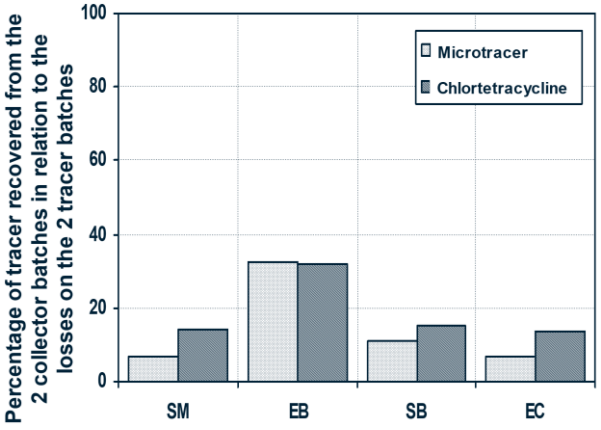

Figure 4: Percentages of tracer recovered from the 2 collector batches for both tracers according to sampling point

		Microtracer		Chlortetracycline	
		Conc. (ppm)	% conc. L2	Conc. (ppm)	% conc. L2
Mixer output	Tracer batch 2	182.2	-	701.0	-
	Collector batch 1	5.2	**2.3**	16.2	**2.3**
	Collector batch 2	0.7	**0.3**	0.9	**0.1**
Silo bin input	Tracer batch 2	222.5	-	675.9	-
	Collector batch 1	12.4	**5.5**	44.7	**6.6**
	Collector batch 2	2.9	**1.3**	6.0	**0.9**
Silo bin output	Tracer batch 2	187.1	-	621.4	-
	Collector batch 1	9.2	**5.1**	37.2	**6.0**
	Collector batch 2	3.3	**1.4**	5.9	**0.9**
Finished prod. bin input	Tracer batch 2	122.6	-	587.5	-
	Collector batch 1	14.0	**6.2**	44.4	**7.6**
	Collector batch 2	2.9	**1.3**	6.5	**1.1**

Table 5: Concentrations for the last three batches and percentage carry-over in the two collector batches for both tracers and at each sampling station

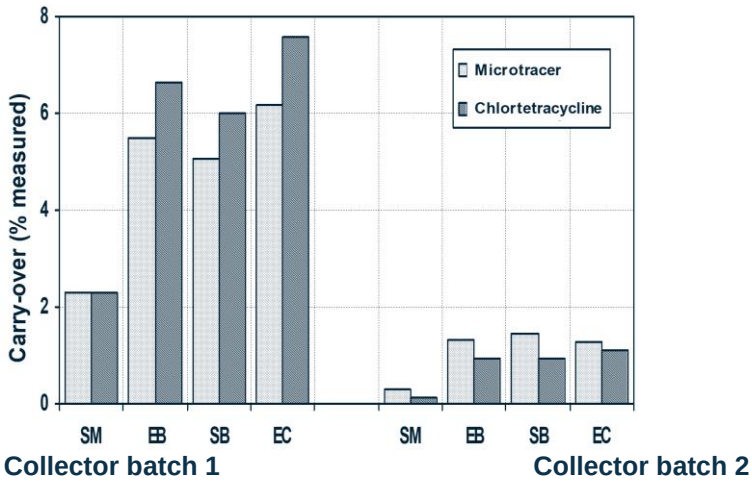

Figure 5: Change in mean carry-over levels in the two collector batches for the 2 tracers expressed as a percentage of the concentration measured in the last tracer batch (L2)

This observation, clearly illustrated here by this comprehensive test, can be generalised to all tests. However, while it demonstrates that carry-over can never be accurately estimated by a simple mass balance calculation, it does not explain either what became of the 70 - 90% of the mass of tracer that seemingly disappeared, or why it went missing. It is also surprising that tracer recovery did not increase in the order of the sampling stations, as is apparently the case for tracer loss. Its oscillation is also unex-

pected as, by this reasoning, recovered tracer is then being lost. These findings would suggest that most carry-over is barely noticeable. All that remains is to speculate on how to explain this phenomenon and to establish whether it corresponds to fact or artefact.

Carry-over analysed as a percentage of the concentrations measured in the last tracer batch (Figure 5) revealed that most carry-over at this plant (about 2/3, i.e. approx. 4%) occurs during transfers between the mixer and the entrance to the silo bin upstream of the press. As the other third (approx. 2%) is already detectable at mixer output, it would appear to be generated by the upstream additive incorporation circuit.

After entering the silo bin upstream of the press, the two tracers gave fairly similar results for carry-over stagnation. The tracers remained in agreement even after pelleting, despite partial destruction of the microtracer due to the heat treatment (see l'Tec_H12). All these observations can be directly reproduced on the two collecting batches.

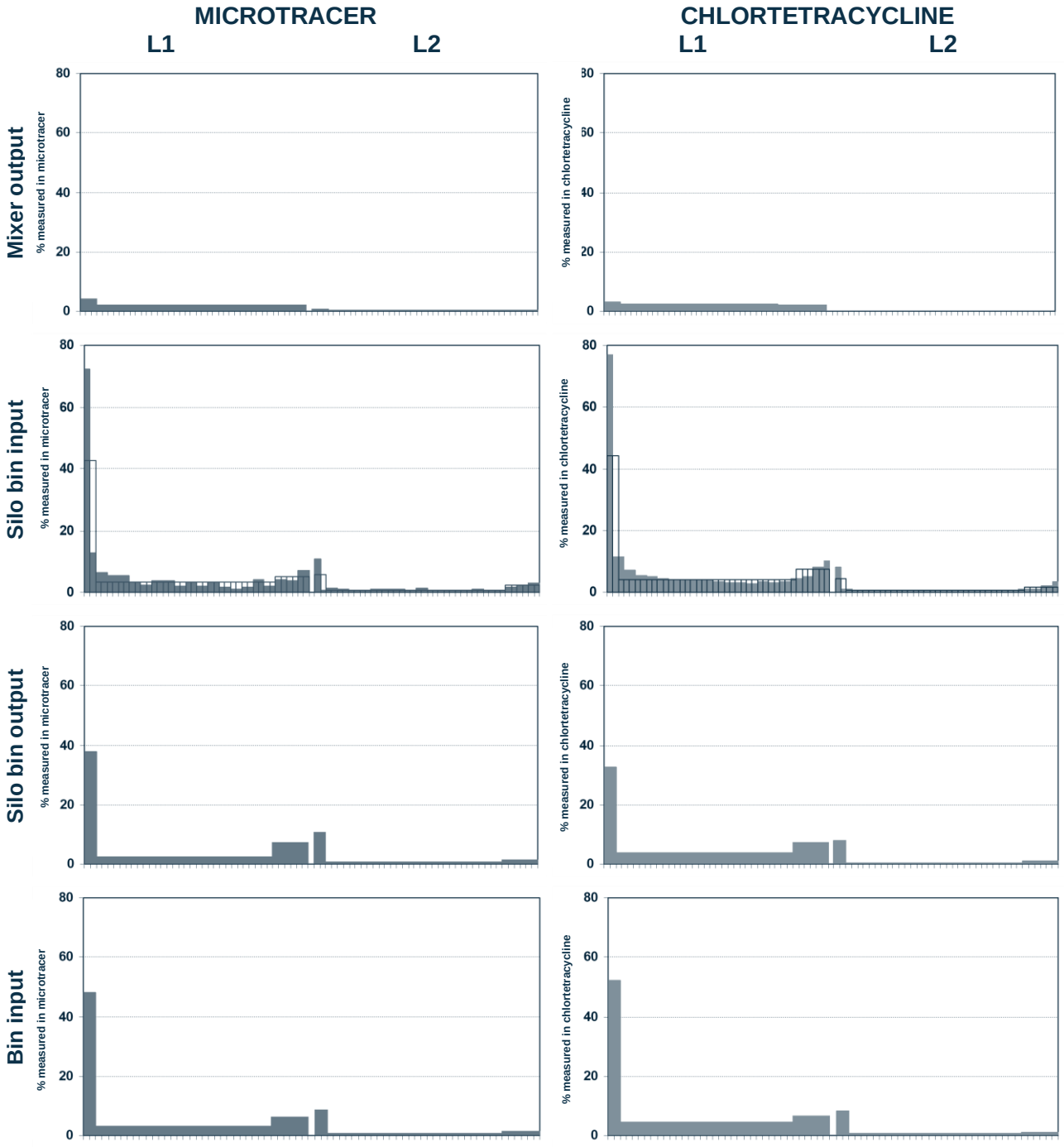

Figure 6: Changes in concentration of the two tracers according to throughput of the two collector batches at each sampling point

A study of the histograms showing carry-over profiles (Figure 6) clearly illustrates the behavioural similarity of the two tracers. The uniform, low-level carry-over apparent at mixer output changes, as demonstrated by the notable 40% peak occurring at the beginning of the batch at the entrance to the silo bin upstream of the press. Surprisingly, despite the complexity and length of the circuit, this peak remains constant during batch transfer through the circuit and up to the entrance of the finished product bin. The carry-over profiles observed at the silo bin output and following pelleting are even surprisingly similar to those recorded at the entrance to the silo bin. Even the batch-end increase in concentration persists after its appearance at the entrance to the silo bin upstream of the press. This result would confirm batch carry-over according to a "plug flow" model. If the carry-over profile at each sampling point does in fact derive from the same product masses circulating in the plant with no remixing, this type of result could influence how trade professionals deal with traceability or how they manage the effects of carry-over on animals. Some animals may receive higher doses of additives if this lack of remixing persists up to the feeding trough.

At the entrance to the silo bin upstream of the press and further down the line, carry-over in the batch core increases by approx. 2% with respect to the level observed at mixer output. This demonstrates that this core carry-over observed at the entrance to the silo bin upstream of the press cannot be explained solely by the circuit upstream of the mixer, but appears also to be generated to a large extent by the circuit between mixer output and the entrance to the silo bin upstream of the press.

A reservation could also be stated about the degree to which the first sample is representative of feed carry-over between this and the following sample. Tests carried out on delivery trucks have revealed that assessment errors could be made at this level.

However, the detailed measurement taken with the two tracers at the entrance to the silo bin upstream of the press validates the aggregation procedure adopted in the technical rules as the dataset from the procedure enables an effective carry-over diagnostic of the manufacturing process. Also, this physical grouping of samples with mean concentrations is also equivalent to the mathematically suggested procedure for delivery trucks (See i'Doc_T8).

5. Conclusion

Despite the moderate pelleting conditions applied at this plant, some damage to the microtracer and/or the colorant it contains has been observed after pelleting. Regardless of this deterioration though, and the recovery rate that is under the recommended level, a comparison of carry-over profiles against those obtained for chlortetracycline post-pelleting gave comparable mean carry-over levels and similar profiles. If the industrial used only microtracer to ascertain the carry-over diagnostic for its manufacturing process, this would be strictly identical to the diagnostic made using chlortetracycline.

To explain the absence of a relationship between the loss of tracer observed in tracer batches and the gains obtained in collector batches, it could be suggested that the highest concentrations were incorrectly analysed; this could also explain the absence of an increase in carry-over between the two tracer batches (see I'Tec_H12). When analysing the concentration of active ingredients such as chlortetracycline, the concentration is calculated in relation to a chlortetracycline solution rather than to an extract derived from a powdered product used during the test under the feed's extraction conditions. This practice could be partly responsible for the erroneous concentration value in the tracer batch feed. The use of microtracers, however, does not involve either this practice or the mass balance calculation, suggesting that this theory does not provide a complete explanation. Another theory, which does not in any way conflict with the previous one, suggests that the tracers might pass into the circuit outside of the sampling periods and would therefore not be recorded.

A field trial would provide further examination of and insight into this issue. This would consist in comparing a carry-over assessment performed according to the rules (sample taken at the entry to the silo bin upstream of the press or at the entry to the finished products bin) with an assessment made following thorough remixing of the collector and tracer batches following an in-plant transfer. This test currently possible at sites equipped with mixers upstream of the loading location.

6. Bibliography

i'Tec_T2. Règles techniques pour l'évaluation du niveau de contaminations croisées entre aliments. Fév 2000.

i'Tec_H12. Evolution de la conformité et de la distribution de deux traceurs dans des aliments au cours de leur fabrication dans une usine. May 2005

i'Tec_H17.Méthode d'analyse du microtraceur RF bleu lake après extraction en milieu aqueux. Oct. 2003.

i'Doc_T8. Elaboration d'une méthodologie pour l'évaluation des contaminations croisées par des additifs dans un camion de livraison d'aliment du bétail. Nov. 2003.

i'Doc_T9. Nouvelle technique d'extraction du microtraceur RF Bleu lake et son évaluation. Jul. 2004.