i'Tec_T5
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Assessment of carry-over, from the incorporation of additives to the loading of finished products

Within the framework of developing the methodology for assessing carry-over on industrial sites, in 1999 Tecaliman carried out two tests during the course of which carry-over was assessed using a number of additives, as far as the pellet loading station (Tecaliman, 2000a, 2000b). The aim of these two assessments, which are summarised here, was to determine the best location in the plant for valid and effective assessment of carry-over. After several years implementing approval procedures and the "desire" of certain industrial operators to carry out these kinds of tests, urged on by DSVs, it appeared useful to note that these tests were carried out, how they were carried out and what their conclusions were.

1. Apparatus

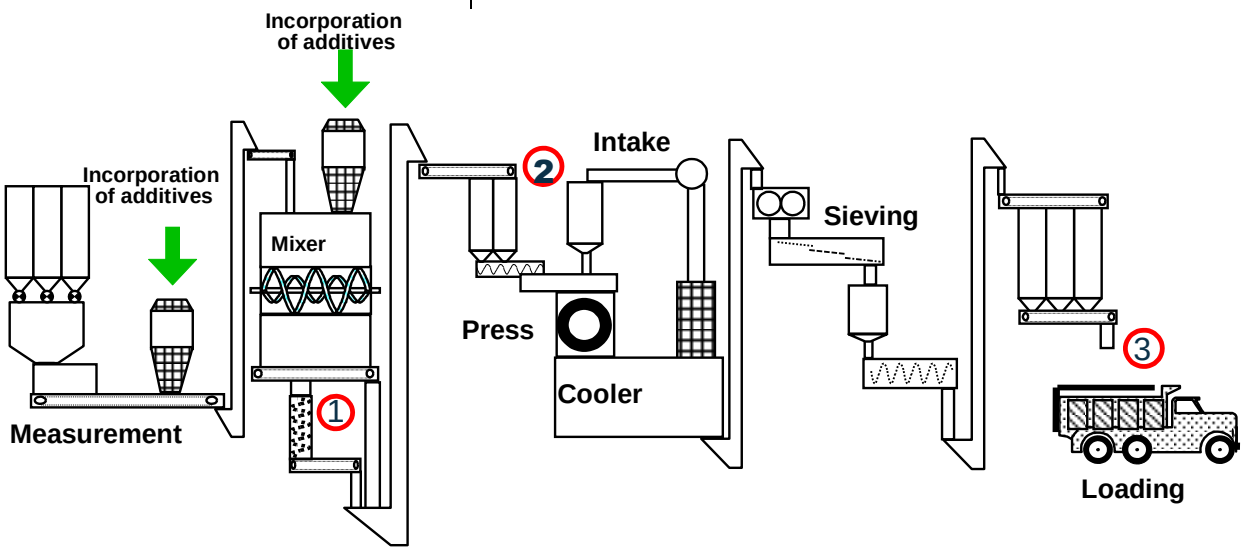
1.1. Plants

These tests were carried out in two plants: A and B, with different diagrams. Samples were taken at three points marked with an arrow in the table and outlined in the figure below:

1. At the mixer outlet
2. At the press valve intake
3. At the loading station

Units	A	B
Bag emptying station	✓	✓
Raw material flow in the elevator		✓
Hopper above the mixer		✓
Mixer	✓	✓
Hopper under the mixer	✓	✓
Screw or Conveyor	✓	✓
Elevator	✓	✓
Revolver	✓	✓
Cell		✓
Silo bin upper the press	✓	✓
Press	✓	✓

Units (Continued)	A	B	
Vertical cooler	✓	✓	
Elevator	✓	✓	
Conveyor		✓	
Siever	✓	✓	
Conveyor	✓		
Revolver	✓	✓	←1
Cell	✓	✓	
Extractor	✓		←2
Weighing bin	✓		
Cell	✓		3→
Truck	✓	✓	



Standard layout of plant diagrams highlighting the sampling points

1.2. The tracers

Five internal tracers were used. Taking account of the weighing actually carried out in the 2 plants, the anticipated concentrations for each of these tracers are:

	A	B
Meticlorpindol	198	199
Dimetridazole	198	199
Nicarbazin	124	125
Monensin	124	-
Lasalocid	124	-

Anticipated concentrations in the tracer batches

The key physical characteristics of these products appear in the following table.

Tracers	Median diameter	Apparent (bulk) density	Hausner ratio	Angle of repose	Dust level
	µm	g/cm³		°	mg/ 25g
Meticlorpindol	290.6	0.412	1.170	72.1	5.8
Dimetridazole	122.8	0.704	1.124	53.1	4.6
Nicarbazin	435.2	0.559	1.047	42.0	15.3
Monensin	527.2	0.614	1.047	45.7	18.1
Lasalocid	574.8	0.591	1.042	44.8	3.3

Tracers - physical characteristics

It is possible to see that:

- The products characterised by a large grain size are Lasalocid, Monensin and Nicarbazin.
- Dimetridazole is the densest product
- Meticlorpindol is distinguished by its flow difficulties.
- Monensin is the dustiest product.

1.3. The feeds

Two tracer batches are produced in succession. In theory, their size equates to 2/3rds of the volume of the cylinder delineated by the mixer's outer flight. The feed is developed on the basis of a feed formula used frequently by the plant, but the incorporation of all these additives in tracer batches makes it an artificial feed that needs to be recycled.
Two collection batches are then produced. Their size is identical to that of the tracer batches.
It is the same formula, but without the incorporation of tracers. The 0.5% of premix is replaced by 0.5 % of premix carrier.

The physical characteristics of the feeds appear in the following table.

	A	B
Median diameter by sieving (µm)	**604**	**651**
Mean apparent (bulk) density (g/cm³)	**608**	**651**
Angle of repose (°)	**65.0**	**60.2**

Feeds - physical characteristics

2. Method

Overall, the protocol followed complies with the technical rules produced subsequently (Tecaliman 2000c). Only departures from this protocol are detailed here.

2.1. Sequence of batches

Each batch is viewed as a different feed. During manufacture, each time there is an intermediate storage phase (hopper under the mixer, hopper above the molasser, silo bin upper the press, cooler, etc.), the process waits until it is completely empty before sending in the next batch. The initial tracer batch is used to bed in all the transfer operations and to time the transit times. This procedure may result in differences from normal operation of the system and draw the attention of personnel to these instructions.

2.2. Sampling conditions

The flows are different, as are the periods between sampling, for each sampling point:

Plants	A			B		
Points	**1**	**2**	**3**	**1**	**2**	**3**
Average flow (kg/s)	25	20	111	25	21	148
Period (s)	5.75	8.25	1.50	6.00	7.75	1.00

Sampling conditions depending on the sampling point

For samples, at the loading point, the team is doubled, which enables the sampling frequency to be increased, as each team samples half the time.

2.3. Sample processing

For the second tracer batch and the two collection batches, an overall sample is created by taking an aliquot from all the primary samples. This procedure enables the mean contamination to be determined for collection batches.

2.4. Analysis of tracers and processing the results

All the tracers are analysed in a single laboratory.
Contamination is expressed as a raw concentration,

in relation to the moisture content of the feed at the mixer outlet or as a percentage of the concentration measured in the second tracer batch.

3. Results

3.1. Tracer batches

The greatest difference between the concentration measured and that expected is that detected for Lasalocid in plant A, which is approximately 15 %.

Plants	A			B		
Points	1	2	3	1	2	3
Moisture content (%)	12.6	12.6	12.6	11.5	11.4	13.1
Meticlorpindol	213	213	213	206	206	210
Dimetridazole	196	188	190	194	188	185
Nicarbazin	127	112	119	120	118	127
Monensin	120	123	124			
Lasalocid	107	105	108			

Concentrations measured in the second tracer batch

3.2. Changes between points

Concentrations of internal tracers are always lower at the mixer outlet and there is always an increase in concentrations between this point and the following one.
However, changes are variable between the silo bin upper the press and the loading station:

- Relative stagnation for Meticlorpindol
- An increase for Dimetridazole of 2.1 ppm for plant A and 0.5 ppm for plant B
- A reduction of approximately 1 ppm for Monensin and Lasalocid in plant A
- A reduction of more than one ppm for Nicarbazin in plant A and an increase of 1.6 ppm in plant B

However, overall, in 5 of the 8 measurements taken for the first collection batch, contaminations at the intake of silo bin upper the press are equal to or higher than those measured on loading. Observations of the second collection batches confirm this finding, although the results are more modest because of the large number of measurements below the detection level.
This result could be explained either by certain difficulties analysing the pellets, which could result in lower apparent contamination, or by a spread of contamination within the system, which renders it less clear for the batch itself.

Plants	A			B		
Points	1	2	3	1	2	3
Meticlorpindol	2.1	4.9	4.9	3.8	8.6	8.2
Dimetridazole	2.1	6	8.1	3.8	10.9	11.4
Nicarbazin	<	2.3	<	1.5	1.4	3.0
Monensin	<	3.5	2.6			
Lasalocid	<	3.5	2.4			

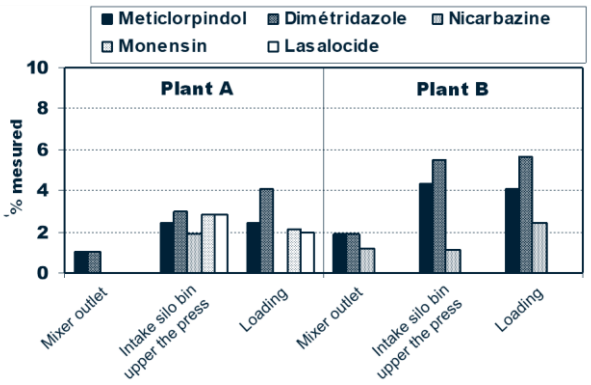
Concentration of tracers (in ppm) in the first collection batch

<: below the detection threshold

Plants	A			B		
Points	1	2	3	1	2	3
Meticlorpindol	1.0	2.3	2.3	1.8	4.2	3.9
Dimetridazole	1.1	3.2	4.3	2.0	5.8	6.1
Nicarbazin	<	2.1	<	1.3	1.2	2.4
Monensin	<	2.8	2.1			
Lasalocid	<	3.3	2.2			

Contamination of the first collection batch as a percentage of the concentration measured in the second tracer batch

<: below the detection threshold



Bar chart of the % of contamination expressed as a % of the concentration measured in the second tracer batch depending on the sampling point and tracer

Plants	A			B		
Points	1	2	3	1	2	3
Meticlorpindol	<	2.0	<	<	1.5	1.7
Dimetridazole	<	<	<	<	1.5	1.4
Nicarbazin	<	<	<	<	<	<
Monensin	<	<	<			
Lasalocid	<	<	<			

Concentration of tracers in the second collection batch

<: below the detection threshold

Plants	A			B		
Points	1	2	3	1	2	3
Meticlorpindol	<	0.9	<	<	0.7	0.8
Dimetridazole	<	<	<	<	0.8	0.8
Nicarbazin	<	<	<	<	<	<
Monensin	<	<	<			
Lasalocid	<	<	<			

Contamination of the second collection batch as a percentage of the concentration measured in the second tracer batch

<: *below the detection threshold*

3.3. Comparison between additives

In the first collection batch, products added together but amounting to less than 200 ppm (Monensin, Lasalocid, Nicarbazin) do not enable results to be obtained at all the sampling points.
Consequently, the concentrations are below the detection threshold:

- at the mixer outlet for Monensin, Lasalocid and Nicarbazin
- at the loading station in plant A, for Nicarbazin.

In the second collection batch, none of these three additives incorporated at 125 ppm enable possible contamination to be deduced or measured.
Expressed as a percentage of the concentration measured in the tracer batch, contamination for the first collection batch in plant A is relatively similar from one additive to another at the intake of the silo bin upper the press and is between 2.1 and 3.3 %. At the loading station, this contamination is close to 2 % for three of the five tracers, 4.3 % for Dimetridazole and below the detection threshold for Nicarbazin.
In plant B, only three additives are tested. More significant differences are detected with contamination between 1.2 and 5.8 % at the intake of the silo bin upper the press and 2.4 and 6.1 % at the loading station.
However, because of the relative similarity of the physical characteristics of these additives, it appears difficult to determine the origins of these differences.
Consequently, a comparison of the behaviour of tracers reveals that there are differences within the same plant and between plants. This tends to show that interaction between the additives and the plant results in different kinds of carry-over depending on the configuration.

3.4. Comparison between plants

The two plants tested have relatively uncontaminated diagrams when compared to the results of the investigation conducted at the same time in French plants (Tecaliman, 2000d).
However, it is possible to note that contamination is more significant in plant B by approximately 1 %, in particular at the mixer outlet. This finding could lead to the conclusion that the longer route taken by additives in the case of plant B results in slightly more contamination.

4. Conclusions

The most significant contamination is often that measured at the intake of the silo bin upper the press. It is likely that this is linked to the “free” behaviour of additives while they are not fixed in the feed’s matrix by means of pelleting. With this in mind, sampling at the loading station is not justifiable as it does not provide any additional information, while resulting in more onerous and expensive tests, because the entire industrial site is stopped for the duration of tests. This period is longer than that usually generated by the production of a batch, as it is necessary to wait for each piece of equipment to be emptied at each stage, in particular, at the pelleting and cooling units.
Sampling at the mixer outlet is beneficial, in order to distinguish between the contribution of the additive circuit, located upstream, which is potentially one of the most significant sources of contamination, compared to the contribution of the downstream circuit. However, it may be that contamination at this point is extremely low. This sampling point is a beneficial one to use if the upstream circuit is suspected of being a source of contamination. The introduction of two sampling points (mixer outlet and intake of the silo bin upper the press) results in an obligation to have sampling teams on the circuit at the same time and will double the cost of processing samples and analysis.
Finally, the incorporation of additives at concentrations of 125 ppm does not provide any guarantee of quantitative results for the first collection batch and makes the lack of a quantitative result for the second collection batch a certainty. This results in an additive offering the possibility of detecting contamination of 0.5 % at least being used as a tracer. This equates to the detection of 1 ppm in the collection batches, if the concentration of tracer batches was 200 ppm.

5. Bibliographic references

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