i'Tec_T11
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**Keywords:** Carry-over, Homogeneity

# Effect of tracer concentration on homogeneity and carry-over

These 2 industrial trials, designed to identify whether tracer concentration has any effect on carry-over, compared the same product at two very different concentrations (200 and 1000 ppm) in order to determine the rate of carry-over for a given quantity of premix fed into the circuit. Two tracer batches and two collector batches were used at each concentration, and sampled at two sampling locations: at the mixer output and the elevator output.

These concentrations were chosen to span the conventional test concentration (250 ppm) and to correspond to a range of uses for drug molecules.

## 1. Apparatus and methods

This section only details the methodology, as the rest of the method conforms to the technical rules.

The 2 tests at each plant were carried out sequentially and in increasing order of concentration of the tracer batch: test at 200 ppm, then at 1000 ppm.

### 1.1. Premixes

A single tracer was used for premixes: RF-blue lake microtracer. Each premix was made to be incorporated into two tracer batches at a rate of 5 kg per ton. The only parameter change concerned microtracer concentration, which was 4% for the lowest concentration and 20% for the highest concentration. Equivalent weights of premix media were injected into the same station (bag emptier) in each of the 2 collector batches.

### 1.2. Feedstuffs

| Feedstuff | $D_{50}$ (µm) | Bulk density (g/l) | Angle of repose (°) |
|---|---|---|---|
| Ruminant | 533.0 | 555.6 | 64.4 |
| Duck | 483.8 | 618.8 | 60.7 |

**Table 1: Physical characteristics of the feedstuffs tested at the 2 plants**

The feedstuffs tested at both sites were fairly standard (Table 1). The duck feed used in the 5-t batch at plant 2 was denser and more fine-grained than the ruminant feed produced for the 4-t batch at plant 1. Despite this, the duck feed still flowed slightly more smoothly than the ruminant feed. Particle size distributions were relatively well centred, but showed a moderate spread.

### 1.3. Homogenisation

Table 2 lists the technical characteristics of the 2 mixers tested.

| Plants | 1 | 2 |
|---|---|---|
| Type of impeller | Double-ribbon | Double-ribbon |
| Vessel shape | Trough | Trough |
| Design capacity | 8 $m^3$ | 12 $m^3$ |
| Incorp. of liquids | yes | yes |

**Table 2: Technical characteristics of the mixers at the 2 plants**

At plant 1, the mixer fill rate was approx. 90% of the manufacturer's reference fill rate, with a homogenisation time of 3.0 min.

At plant 2, the mixer fill rate was approx. 66% of the manufacturer's reference fill rate, with a homogenisation time of 4.5 min.

Homogenisation was tested on the second tracer batches, while the first batches were sampled to determine the recovery rate for these batches.

### 1.4. Test procedures

The two plants that conducted the tests were chosen for their high levels of recorded carry-over. It was important to choose sites that would provide high carry-over levels in order to ensure that the factor under study had a visible effect. Neither of these sites, therefore, could be said to represent median trade levels.

Table 3 lists the technical characteristics of the 2 tested circuits. Both these circuits are fairly similar, particularly the sections upstream of the mixer, as premixes undergo significant handling before arriving at the mixer. Plant 1 handling includes a screw conveyor that is absent in the other cases. Downstream of the mixer, the plant 1 circuit also includes a molasser that is absent at plant 2.

| Plant 1 | Plant 2 |
|---|---|
| **Incorporation of the premix** | |
| Bag emptying station - Weighing bin | Bag emptying hopper |
| Hopper | |
| Screw - Elevator- Chain conveyor | 2 chain conveyors - Elevator |
| Hopper on the mixer | Hopper on the mixer |
| Mixer | Mixer |
| Hopper under the mixer | Hopper under the mixer |
| Chain conveyor | Chain conveyor |
| **Sampling at mixer output** | |
| Molasser | Distribution box |
| Elevator | Elevator |
| Revolver distributor | Revolver distributor |
| **Sample taking at the entrance to the silo bin upstream of the press** | |

**Table 3: Technical characteristics of the circuits at the 2 plants**

# 2. Results

## 2.1. Recovery rates and homogenisation

At both plants, all recovery rates lie within the acceptability limits of the technical rules.

At plant 1 (Table 4), during the 200-ppm test, the first tracer batch was found to be significantly less concentrated than expected (94.1%). Passing through the circuit downstream of the mixer has little impact on the rates (from 94.1% to 92.7% and from 101.3% to 101.6%). Conversely, the concentration of the second tracer rose as soon as it exited the mixer output (101.3%), as if the second batch picked up the amount of tracer lost by the first batch. During the 1000-ppm test, the first tracer batch did not appear to lose any tracer upstream of the mixer (98.4%) while, this time, it was the second tracer batch that was found to be less concentrated than expected (91.6%). On the other hand, both tracer batches recorded significantly lower concentrations after having passed through the circuit downstream of the mixer (from 98.4% to 90.5%, i.e. -68.6 ppm and from 91.6% to 86.5%, i.e. -51.7 ppm).

This would suggest that during both tests at plant 1, tracer loss and recovery are recorded both upstream and downstream of the mixer.

| **Tests** | **200 ppm** | | | | | | **1000 ppm** | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Tracer batches** | **[ ] Expected** | **Mixer output** | | **Silo bin input** | | **Variation [ ] (EB-SM)** | **[ ] Expected** | **Mixer output** | | **Silo bin input** | | **Variation [ ] (EB-SM)** |
| **Indicator** | **[ppm]** | **[ppm]** | **%** | **[ppm]** | **%** | **[ppm]** | **[ppm]** | **[ppm]** | **%** | **[ppm]** | **%** | **[ppm]** |
| **T1** | 200.5 | 188.1 | **94.1** | 185.3 | **92.7** | **- 2.8** | 992.2 | 967.0 | **98.4** | 898.4 | **90.5** | **-68.6** |
| **T2** | 199.8 | 202.5 | **101.3** | 203.7 | **101.6** | **+ 1.2** | 1011.2 | 926.2 | **91.6** | 874.5 | **86.5** | **-51.7** |
| **Variation [ppm] (T2-T1)** | | **+14.4** | | **+18.4** | | | | **-40.8** | | **-23.9** | | |

**Table 4: Recovery rate in tracer batches for both tests at plant 1**

| **Tests** | **200 ppm** | | | | | | **1000 ppm** | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Tracer batches** | **[ ] Expected** | **Mixer output** | | **Silo bin input** | | **Variation [ ] (EB-SM)** | **[ ] Expected** | **Mixer output** | | **Silo bin input** | | **Variation [ ] (EB-SM)** |
| **Indicator** | **[ppm]** | **[ppm]** | **%** | **[ppm]** | **%** | **[ppm]** | **[ppm]** | **[ppm]** | **%** | **[ppm]** | **%** | **[ppm]** |
| **T1** | 203.1 | 184.6 | **90.6** | 154.7 | **76.2** | **-29.9** | 1017.0 | 858.1 | **89.4** | 959.5 | **94.2** | **+101.4** |
| **T2** | 203.5 | 199.9 | **98.3** | 207.9 | **102.2** | **+8.0** | 1018.3 | 1067.9 | **104.9** | 1076.2 | **105.7** | **+8.3** |
| **Variation [ppm] (T2-T1)** | | **+15.3** | | **+53.2** | | | | **+209.8** | | **+116.7** | | |

**Table 5: Recovery rate in tracer batches for both tests at plant 2**

At plant 2 (Table 5), the first two tracer batches lost large quantities of tracer upstream of the mixer, whatever the concentration (approx. 10% of the expected quantity). As it passed through the circuit downstream of the mixer, tracer batch 1 for the 200-ppm test lost a large quantity of tracer (29.9 ppm), but this loss was not repeated during the 1000-ppm test. The recovery rate for tracer batch 2 for the 200-ppm test was close to 100%, indicating that it recovered some of the tracer lost by the first batch. The same observation can be made for the 1000-ppm test, which highlights the effect of these deposition and recovery phenomena in the circuit upstream of the mixer in this plant. Downstream of the mixer, the second tracer batches showed no loss of tracer and recorded recovery rates close to, and even greater than, 100%.

The plant 1 homogenisation process gave excellent

mixer performance results for both concentrations, within the trade-specified zone of compliance (Table 6). The passage through the downstream circuit resulted in slight demixing, particularly during the1000-ppm test.

Mixer performance at plant 2 was inferior, with coefficients of variation at mixer output of 5.5%, although still within the trade-specified zone of acceptability. However, the coefficients of variation improved at the entrance to the silo bin, as if some homogenisation had already occurred downstream of the mixer. Despite this, there was a fairly minor oscillation around the 5% reference value.

At both these plants, mixer performance was either conform or within the limits of acceptability meaning that the resulting profiles cannot be used to define special recommendations that would help to improve performance.

| Plants | Stations | 200 ppm | 1000 ppm |
|---|---|---|---|
| 1 | Mixer output | 3.3 | 3.3 |
| | Silo bin input | 3.7 | 4.8 |
| 2 | Mixer output | 5.5 | 5.5 |
| | Silo bin input | 4.0 | 4.2 |

**Table 6: Microtracer $CV_{total}$ following in-plant homogenisation**

## 3. Carry-over

At plant 1 (Table 7), as expected, carry-over rates in both tests were significantly higher than trade-specified conformity levels (3 or 5%).

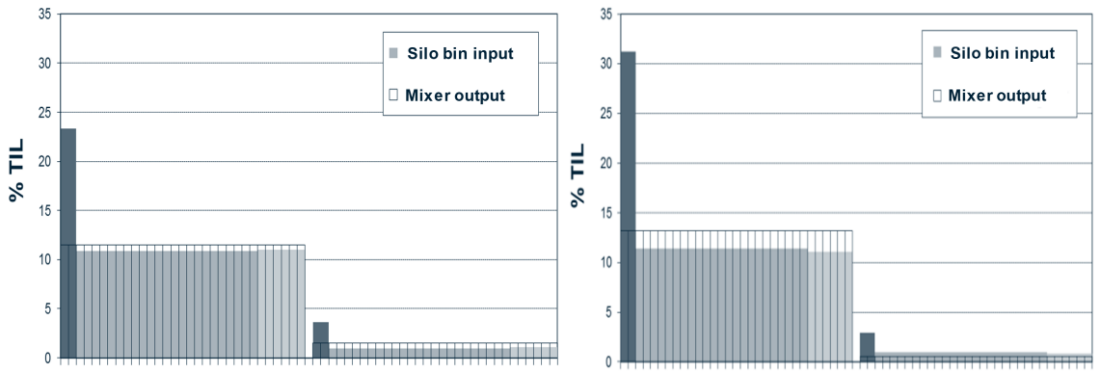
Whatever the concentration of the tracer batches, the percentage of carry-over gave similar results, i.e. 12 to 13%. The concentration of the tracer batches did not appear to have any effect on this percentage. Conversely, the concentration of the collector batches produced after a more concentrated tracer batch was higher.

At this plant, the circuit upstream of the mixer, which includes a screw conveyor, had a major impact on the first collector batch. However, this circuit is cleaned rapidly, as the level of carry-over dropped off significantly between the two collector batches. This is clearly illustrated in the carry-over profiles (Figure 1). The downstream circuit appears to have very little effect, despite the presence of a molasser.

The change in concentration of the tracer batches had no effect on the carry-over profile.

| Tests | 200 ppm | | | | 1000 ppm | | | |
|---|---|---|---|---|---|---|---|---|
| Stations | Mixer output | | Silo bin input | | Mixer output | | Silo bin input | |
| Indicator | [] (ppm) | Carry-over (%) | [] (ppm) | Carry-over (%) | [] (ppm) | Carry-over (%) | [] (ppm) | Carry-over (%) |
| **Overall T1** | **188.1** | | **185.3** | | **967.0** | | **898.4** | |
| **Overall T2** | **202.5** | | **203.7** | | **926.2** | | **874.5** | |
| $C1_a$ | | | 47.6 | 23.4 | | | 272.9 | 31.2 |
| $C1_b$ | | | 22.1 | 10.9 | | | 99.7 | 11.4 |
| $C1_c$ | | | 22.4 | 11.0 | | | 97.0 | 11.1 |
| **Overall C1** | **23.8** | **11.8** | **23.8** | **11.7** | **122.4** | **13.2** | **110.4** | **12.6** |
| $C2_a$ | | | 7.3 | 3.6 | | | 25.6 | 2.9 |
| $C2_b$ | | | 1.9 | 0.9 | | | 8.4 | 1.0 |
| $C2_c$ | | | 2.2 | 1.1 | | | 7.3 | 0.8 |
| **Overall C2** | **2.3** | **1.1** | **2.3** | **1.1** | **5.5** | **0.5** | **9.3** | **1.1** |

**Table 7: Concentrations and carry-over levels for the 2 tests at plant 1 designed to study the effect of tracer concentration**



**Test at 200 ppm** **Test at 1000 ppm**

**Figure 1: Carry-over profiles at plant 1 designed to study the effect of tracer concentration**

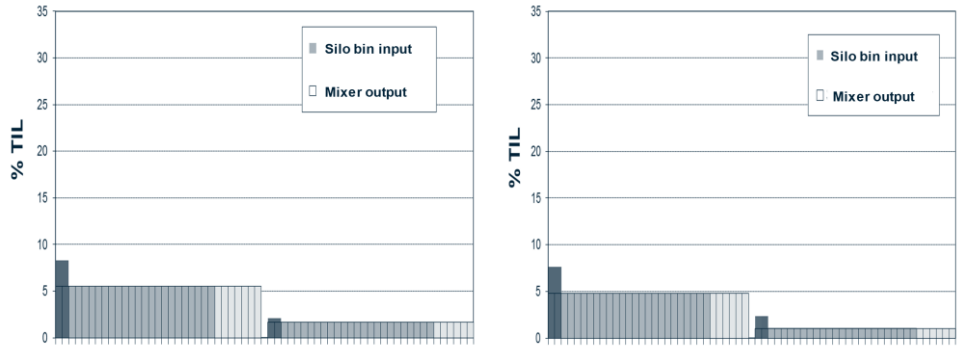
At plant 2 (Table 4) also, most of the carry-over observed at the silo bin input was generated by the passage through the circuit upstream of the mixer (for the first collector batches: 5.5% and 4.8%). At this industrial site, these levels also exceeded the trade-specified conformity threshold.

As at plant 1, the change in concentration of the tracer batches had no major impact on the measured carry-over levels. At plant 2, carry-over levels were actually higher at the lowest concentration, while the opposite observation was made at the plant 1.

Neither was there any visible effect on the carry-over profiles at the 2 sampling points (Figure 2). Lastly, at plant 2, carry-over levels took longer to decrease between the two collector batches than at plant 1.

| **Tests** | **200 ppm** | | | | **1000 ppm** | | | |
|---|---|---|---|---|---|---|---|---|
| **Stations** | **Mixer output** | | **Silo bin input** | | **Mixer output** | | **Silo bin input** | |
| **Indicator** | **[] (ppm)** | **Carry-over (%)** | **[] (ppm)** | **Carry-over (%)** | **[] (ppm)** | **Carry-over (%)** | **[] (ppm)** | **Carry-over (%)** |
| **Overall T1** | **184.6** | | **154.7** | | **858.1** | | **959.5** | |
| **Overall T2** | **199.9** | | **207.9** | | **1067.9** | | **1076.3** | |
| **$C1_a$** | | | 17.2 | 8.3 | | | 82.1 | 7.6 |
| **$C1_b$** | | | 11.5 | 5.6 | | | 51.7 | 4.8 |
| **$C1_c$** | | | 10.4 | 5.0 | | | 48.4 | 4.5 |
| **Overall C1** | **11.0** | **5.5** | **1.7** | **5.6** | **50.9** | **4.8** | **53.0** | **4.9** |
| **$C2_a$** | | | 4.4 | 2.1 | | | 25.3 | 2.3 |
| **$C2_b$** | | | 3.5 | 1.7 | | | 11.7 | 1.1 |
| **$C2_c$** | | | 2.7 | 1.3 | | | 11.5 | 1.1 |
| **Overall C2** | **3.3** | **1.7** | **3.4** | **1.6** | **10.8** | **1.0** | **12.5** | **1.2** |

**Table 8: Concentrations and carry-over levels for the 2 tests at plant 2 designed to study the effect of tracer concentration**




**Test at 200 ppm** **Test at 1000 ppm**

**Figure 2: Carry-over profiles at plant 2 designed to study the effect of tracer concentration**

## 4. Conclusion

On the whole, similar carry-over levels were recorded at both industrial sites at the two tested concentrations. While an apparent increase in carry-over was detected at plant 1 along with the increase in concentration, the opposite trend was observed at plant 2.

At both plants, a 5-fold increase in concentration had no effect on either the levels of measured carry-over, or on carry-over profiles. While there is no identifiable ratio between this increase and the levels of carry-over, collector batches did indeed experience a 5-fold increase in concentration.