DATASHEET No. 29
July 2012

Keywords: Carry-over, Measurement

Technical rules for assessing the level of carry-over between feedstuff batches

Industrial obligations allied with French and European regulations are driving feedstuff manufacturers to assess the level of carry-over on their production lines. These technical rules, which are recognised by several certification reference systems, can also be applied to manufacturers of mineral and additive premixes.

These carry-overs consist of technically unavoidable traces of residual product in the production chain that may be transferred to follow-up batches during manufacture. They may also be referred to as "cross-contaminations" when this involves the transfer of contaminants. These technical rules are intended to provide a measurement tool that will facilitate technical mastery over such carry-overs and the presence of such traces.

These recommendations have been established on the basis of:

- settings known to impact on the results or their interpretation,
- the results of a bibliographical study on carry-overs between compound feeds,
- the results and observations obtained from industrial assessment campaigns carried out since 1999 jointly with the DGAL (French Directorate General on Food Safety).

Technical comments and rationales are provided in the form of cross-references at the end of this document, identified by numbered superscripts. Other comments and rationales are given in other technical datasheets (Nos 42, 45, 58, 78). Important concepts are underlined.

Compliance with these rules is a condition both for building a database on industrial performance in this area, and for enabling a comparison either between different sites or of a given site over a certain period of time.

1. Objective

These rules form a basic method for assessing carry-over levels (TIL). This assessment must be performed in tandem with a TIL-related hazard analysis. This analysis is used to specify the performance conditions according to the intended purpose. Note that this concerns the measurement of how carry-overs impact on follow-up batches; this type of measurement cannot be used to assess one-off, accidental or random carry-overs. This assessment forms part of an on-site carry-over monitoring system.

2. Principle

A batch corresponds to a mixer load.

The method consists in:

- Choosing a tracer and its related use conditions,
- Choosing a circuit that runs between the tracer's point of incorporation and the sampling point,
- Choosing and producing batches that contain the evenly distributed tracer (tracer batches),
- Choosing and then directly producing tracer-free batches in the same circuit[1] (collector batches),
- Taking samples from these batches,
- Processing the samples,
- Determining tracer concentration in the samples

3. Apparatus

3.1. Tracer characteristics

The tracer is selected based on the following criteria:

- Where possible, it should be a tracer used by the plant and selected with respect to the hazard analysis
- It should be derived from a single source. This means that it must not be present in the other ingredients found in tracer and collector batches.
- It should be incorporated at a rate sufficient to ensure that, given its detection limit, it provides for detecting a minimum carry-over of 0.5% [2].
- It should be analysed using a method that is accurate[3], repeatable[4], sensitive[5], has a low quantification threshold[6], and is simple and cost-effective[7].
- It should be stable in relation to the manufacturing process between its point of incorporation and the sampling point.

It must be possible to incorporate it directly into or scatter it over a media.

3.2. Batch characteristics

Two types of product can be used depending on the test focus:

- when assessing the production tool, a compound feed that is representative of the manufacture at the plant being tested may be used,
- when investigating the process in order to

develop corrective measures, a crushed raw material or a mix of crushed raw materials, with a defined set of physical characterizations, may be used.

It is recommended to use tracer and collector batches of a similar type in order to facilitate interpretation.

3.3. Circuit

The circuit runs between the tracer's point of incorporation and the sampling point. It is chosen according to the hazard analysis. The same circuit must be used for all the batches; there must be no modifications made between batches. Where necessary, the guarantee of the products having been incorporated at the tracer introduction station will have to be validated when producing the collector batches.

4. Methods

4.1. Batch size

Batch size must be representative of the plant's manufacturing practices. It is recommended that tracer and collector batches should be the same size[8].

4.2. Batch number

At least two "tracer" batches (T_1, T_2, ...T_n) have to be programmed to pass through the manufacturing circuit one after the other up to the sampling point[9].

At least two[10] "collector" batches (C_1, C_2, ...C_n) have to be programmed to pass through the same circuit one after the other and just after the tracer batches up to the sampling point.

A rinse can be programmed between the tracer and collector batches (this is not considered as a collector batch if it is common practice in the plant's operating procedures). Where appropriate, the rinse's presence and routing will have to be specified.

4.3. Sampling

4.3.1. Sampling point

This should enable operators to take samples reliably, rapidly and in complete safety. It is preferable to take the samples from a moderate product flow (< 70 t/h) without modifying the normal operating conditions. It is recommended to take the samples at the press gate inlet or at the loading station unit inlet[11].

4.3.2. Sampling procedure

The sampling procedure must provide for:

- taking samples safely,
- obtaining samples that are representative of the product flow (Gy, 1996),
- obtaining samples of the desired size,
- and be fit for use at the sampling point.

It is recommended to take the following precautions[12]:

- attach the sampling equipment to a fixed point outside the circuit.
- make sure there are no moving parts in the sampling zone (dual-direction boxes, pneumatic hatches, elevator buckets, etc.)
- wear a mask and goggles if dust is generated.

It is recommended to modify the flow penetration mode with each sample.

4.3.3. Sample number and size

The aim is to collect 30 samples from the final tracer batch (T_n) and from all the collector batches. Any reduction in the number of samples taken from collector batches and, therefore, any decrease in sampling frequency will have an adverse effect on the representativeness of the test and the measured TIL level[13].

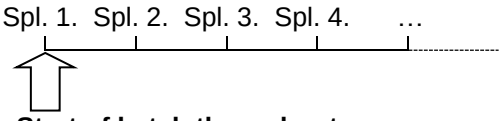
Sample size may vary between 100 and 1000 g, with a minimum variation between the samples for a given test[14].

4.3.4. Frequency

This is determined as follows:

- Measure the throughput time for the initial tracer batch at the sampling station.
- Calculate the time between two samples by dividing the throughput time by the number of samples to be taken plus 1, i.e. 31 for 30 samples.

To take a sample, at the start of batch throughput, the stopwatch is activated simultaneously with sample 1:



Start of batch throughput

Batch start is considered effective when the sampling tool fills completely during a clean, direct penetration of the flow for a short period of time. Batch end is considered effective when the sampling tool no longer fills completely during the same period of time.

The samples are taken at each time period and packaged in chronological order. Samples have to be taken up to the end of the batch throughput, which is marked by a clear and significant reduction in flow rate.

4.4. Testing

4.4.1. Batch follow through in the manufacturing circuit

Each batch has to be managed as a different feed[15]. During manufacture, each time that an intermediary storage phase is performed (hopper under the mixer, hopper on the molasser, silo bin upper the press, silo bin of dispatch, etc.), it is vital to wait until emptying is complete before sending in the next batch in compliance with common industrial practices. The initial tracer batch is used to run in all the transfer manoeuvres.

4.4.2. Additional data collection

This data is used to interpret the results:

- physical characterizations of the tracer and product,

- method used to incorporate the tracer: incorporation point, expected concentration in the feed, incorporation rate and the concentration of any possible premix, incorporation timeline, etc.
- quantities of dosed raw materials (including molasses, fats, and other liquids) upstream of the sampling point,
- line characteristics (brand, type, status, equipment size where appropriate, etc.),
- all the information on all the operations performed during the test between the tracer's point of incorporation and the sampling point (molassing, pelleting, coating, etc.),
- any changes that have occurred since the previous test (equipment and apparatus, method, etc.),
- formula(s) and weighings (Dosing log)
- any deviation from this method.

4.4.3. Sample processing and analysis

Each primary sample is homogenized and an aliquot portion representative of a constant, equivalent weight is sampled. The aliquot portions of the final tracer batch are pooled in order to make up a single, overall sample unless this batch has been set aside for an homogeneity test used to determine its mean concentration.

The aliquot portions from the primary samples of a given collector batch are pooled and mixed by group in order to make up three pooled samples:

- Group A: aliquot portions of the first 2 primary samples.
- Group B: aliquot portions of the intermediate primary samples (approx. 22 samples).
- Group C: aliquot portions of the last 6 primary samples.

If the number of collected samples is greater than 30, it may be decided to distribute them on a pro rata basis (6/74/20%).
The laboratory analyses each of the pooled samples in order to establish their tracer concentration.
The laboratory processes each sample to make it possible to take a representative test portion from each one. Therefore, if an analysis is carried out on a test portion that is smaller than the samples, it is recommended to finely grinding the whole mass of the sample (without destroying the tracer), then re-homogenise it and finally divide it to produce a sub-sample of a size that is as close as possible to that of the test portion.
It may be necessary to repeat the analysis for low concentrations.
The bulk density and grain size of the feeds may also be analysed in order to characterise the test conditions.

4.5. Expression and interpretation of the results

The mean concentration of a collector batch (CMC) is calculated using the following equation:

$$CMC = \frac{C_a \times 2 + C_b \times (n-8) + C_c \times 6}{n}$$

n: Total number of samples taken from the corresponding collector batch
Ca: Concentration of tracer in pooled sample A
Cb: Concentration of tracer in pooled sample B
Cc: Concentration of tracer in pooled sample C

The carry-over levels for each pooled sample from the collector batches are expressed as a carry-over rate: Percentage calculated according to the respective concentrations of tracer in the collector batches readjusted to the measured concentration of the final tracer batch.
The mean carry-over rate for a collector batch (TMTC) is calculated using the following equation:

$$TMTC = 100 \times \frac{CMC}{CMT}$$

CMT: Tracer concentration in the final tracer batch obtained by analysing the pooled sample from this batch or by averaging the analysis results of a homogeneity test performed on this batch.

In any case, the concentration of the final tracer batch must lie within 70% and 110% of the expected concentration (or 80% to 110% for manufacture of medicated feeds according to the BPFDAM [good manufacturing and wholesale distribution practices for medicated feedstuffs]). The distribution of tracer in the tracer batch is assumed to be homogeneous; this homogeneity will have been validated prior to testing.
The results obtained using this method must be assessed with respect to the:

- trend profile for the carry-over rate within each collector batch and throughout all the collector batches (degrowth curve),
- manufacturing practices,
- test procedures (sampling point, selected tracer, batch size, etc.),
- tested manufacturing circuit,
- obligations of the industrial site,
- analysis of the hazards involved.

The composition of the three pooled samples from each collector batch is useful in determining the carry-over profile.
Carry-over levels or concentrations should logically decrease in the order of the batches.

5. Bibliography

- Gy P. 1996. L'échantillonnage des lots de matières en vue de leur analyse (Sampling material batches for analysis). Ed. Masson, ISBN 2-225-85366-5, 148 p.
- Special Tecaliman report N° 40, 2000. Synthèse des travaux en vue de l'élaboration de règles techniques pour l'évaluation du niveau

d'homogénéisation d'un mélange et du niveau de contaminations croisées entre aliments en alimentation animale. (Summary of the findings aimed at establishing technical rules for assessing the level of homogenisation of a mix and the level of cross-contamination between animal feeds).

- Special Tecaliman report N° 41, 2000. Enquête auprès des fabricants d'aliments composés sur leurs essais d'évaluation de l'homogénéité des mélanges et des taux de contaminations croisées. (Survey of compound feed manufacturers concerning their mix homogeneity assessment testing and cross-contamination rates). Tecaliman - 2000.
- Special Tecaliman report N° 49, 2002. Synthèse bibliographique sur les contaminations croisées en alimentation animale. (Bibliographic summary of cross-contaminations in animal feed).
- Special Tecaliman report N° 52, 2003. Résultats d'essais industriels sur l'homogénéité et les contaminations croisées de 2000 à 2002. (Results of industrial tests on homogeneity and cross-contaminations from 2000 to 2002)
- Special Tecaliman report N° 65, 2008. Recherche de traceurs de mélange et de contaminations croisées. (Search for mix and cross-contamination tracers).
- Special Tecaliman report N° 68, 2010. Amélioration de la technique d'analyse et des modalités d'usage du microtraceur. (Improvements in microtracer analysis technique and operating procedures).
- Report 4: Essais industriels d'évaluation des niveaux de contaminations croisées. (Industrial tests designed to assess cross-contamination levels). Tecaliman - 2000.
- Technical Datasheet N° 42, 2002 Evaluation des contaminations croisées, de l'incorporation des additifs au chargement des produits finis (Assessment of cross-contaminations, from the incorporation of additives up to loading of the finished products).
- Technical Datasheet N° 45, 2003 Comparaison traceurs internes/traceurs externes (Comparison of internal/external tracers).
- Technical Datasheet N° 58, 2005 Evolution des contaminations croisées d'aliments au cours de leur fabrication dans une usine à l'aide de deux traceurs (Dual-tracer analysis of patterns of animal feed cross-contamination during factory manufacture).
- Technical Datasheet N° 78, 2011 Effet de la taille des lots sur les niveaux de transfert inter-lots (Impact of batch size on carry-over levels).

Cross-references

[1] Without taking account of gravity fall

[2] For instance, incorporation at 200 ppm and detection of 1 ppm

[3] Measurement accuracy: closeness of the agreement between a measurand and an actual value for the specific quantity being measured. (Standard NF X 07-001).

[4] Repeatability of the results: closeness of the agreement between the results of successive measurements of the quantity being measured, where all the measurements are taken under the same measurement conditions. (Standard NF X 07-001).

[5] Sensitivity: ability to detect small variations in analyte (the analysed substance). (Standard V 03-110).

[6] Quantification limit: the smallest quantity of analyte to be examined in a sample, that can be dosed under the experimental conditions described in the method with a defined variability. (Standard V 03-110).

[7] Due to the number of analyses required.

[8] In order to make it easier to determine the conditions for testing and sampling frequency. Tecaliman has demonstrated that size variations between collector and tracer batches only have a minor impact on TIL levels (Technical datasheet N°78).

[9] In order to obtain a concentration of the second tracer batch that is as close as possible to the expected concentration.

[10] In order to test the line for possible perpetuation of the carry-over created by the selected tracer.

[11] Tecaliman's findings, and the results illustrated in technical datasheets N°42 and 58 in particular, demonstrate that there is only minor fluctuation of carry-over levels downstream of the silo bin upper the press inlet and that this sampling point is the easiest to use at a plant while minimizing disruptions to manufacturing practices.

[12] Sampling issues may arise in the presence of high flow rates, powerful suction, the generation of dust clouds, or at the time of detecting batch ends.

[13] Reducing the number of samples strengthens the impact of the samples with the highest concentration on the mean, thus maximizing the carry-over level.

[14] This involves a compromise between sampling constraints, laboratory processing and shipping costs, and the average size of feed rations for factory-fed animals.

[15] Take account of the management of batch follow-throughs and automations planned during follow-throughs between batches regarded as incompatible.