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Technical rules for assessing the homogenisation performance of a batch mixer

Industrial obligations allied with French and European regulations are driving feedstuff manufacturers to assess the homogenisation performance of their mixers. These technical rules, which are recognised by several certification reference systems, can also be adapted for use by manufacturers of mashes, additive premixes, and powdered dietetic and mineral feeds (adjusting the choice of tracer, sample sizes, etc. as necessary).
Technical mastery over homogeneity is based on an assessment of how uniformly additives and premixes are distributed in feedstuffs. As such, technical rules for assessing a mixer's homogenisation performance have been defined, and established, based on:

- settings with a known impact on the results or their interpretation,
- the results of a bibliographical review on the homogenisation of 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).

These rules provide businesses with a tool for technical mastery of the homogeneity of feedstuffs and take the form of recommendations; each user is free to use their preferred method provided that they can demonstrate that the result and its determination lie within a certain dependability range.
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.
Technical comments and rationales may be provided in the form of cross-references at the end of this document, identified by numbered superscripts. Important concepts are underlined.

1. Objective

- To test the homogenisation performance of a batch mixer under a given set of conditions. This is done by studying the distribution of a tracer in a mix of solids. The test conditions must be representative of the plant's chosen manufacturing practices.

2. Principle

The method consists in:

- stating the objective (to test a given practice, test a product, etc.),
- defining the test conditions (choice of tracer, mixing conditions, sampling conditions, etc.),
- preparing a mix containing the tracer,
- taking the samples,
- quantifying the amount of tracer in the samples,
- interpreting the results.

The assessment results provide a snapshot of the tracer's behaviour under the test conditions.

3. Apparatus

3.1. Choice of tracer

The following criteria are recommended for selecting the best tracer to use for testing the homogenisation capability of a mixer for powdered ingredients:

- It must be capable of being quantified using a method that is accurate[1] [2], repeatable[3], sensitive[4], simple and cost-effective[5].
- It should derive mainly, or even better, exclusively, from a single source[6].
- It is recommended that its main source comprises at least 1 000 000 part./g.[7].
- It must be stable with respect to the manufacturing process between the point of its incorporation and the point of sampling.
- It must be possible to incorporate it directly into a media or by scattering it over a media.

Other, less effective, tracers may be used; any deviation from the recommendations may provide grounds for interpreting a nonconform result.
If selecting a tracer with doubts over the analytical performance, the recommended procedure is to carry out duplicate analyses on each sample (see § 5.5).
Other objectives may lead to the adoption of different tracer selection criteria (mash feeds, liquids incorporated into the batches, mineral or dietetic feeds, etc.).

3.2. Mix base

It is preferable to use a product that is representative of the plant's production output[8].

4. Production

4.1. Mix conditions

These must conform to the plant's standard practices (mixer's fill rate, mixing time, point at which the tracer is incorporated, etc.). Different sets of conditions can be tested for the purposes of experimentation.

4.2. Sampling method and location

It is recommended to take the samples at a point that is as close as possible to the mixer output. The sampling method and location should make it possible to:

- take samples safely,
- obtain samples that are representative of the product flow (Gy, 1996),
- obtain samples of the desired size (Gy, 1996),
- arrive at a result that is close to the result that would be obtained ex-plant[9],
- test a performance that covers the full range of the plant's product presentations.

To ensure the collection of a representative sample, it is strongly recommended[10]:

- to cut the flow in differing directions from one sample to the next,
- to choose sampling points with moderate flow rates[11].

It is recommended to take the following precautions:

- 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.

4.3. Number of samples

It is recommended to take at least 20 samples[12]. It is advised to provide extra sample containers to ensure that samples can be collected right up to the end of the batch.

4.4. Sample size

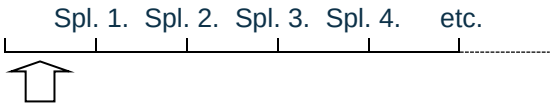
This can vary between 100 and 1000 g[13]. It is recommended to minimize variations in sample size for a given test.

4.5. Sampling frequency

This frequency is designed to distribute sampling points over the whole batch.
This is determined as follows:

- measure the throughput time for a batch that is similar to the batch produced during the test at the sampling point,
- calculate the time between two samples by dividing the throughput time by the number of samples to be taken plus 1, i.e. 21 for 20 samples.

At the beginning of the batch throughput, the stopwatch is activated in order to allow a suitable time to pass before taking the first sample[14]:



Beginning 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.

5. Testing

5.1. Preliminary tasks and checks

The following has to be checked at the sampling point:

- Safety of the sampling conditions.
- Availability of the persons and equipments required to carry out the tests.
- Throughput time for a similar batch, in order to determine the sampling frequency.
- Absence of any factors that could disturb the test (strong or irregular flow, generation of dust, etc.).

5.2. Additional data collection

It is recommended to collect the following data in order to facilitate both subsequent interpretation of the test results, and the comparison of how the results change over time:

- physical characterizations of the tracer and the 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,
- mixer characteristics (brand, type, status, equipment size, etc.),
- all the information on all the operations performed during the test between the tracer incorporation station and the sampling point (molassing, pelleting, coating, etc.),
- any changes that have occurred since the previous test (equipment and apparatus, practices, etc.),
- formula(s) and weighings (Dosing log),
- any deviation from this method,
- list of and respective times for all mixing phases (pre-homogenisation, incorporation sequence for liquids and solids, homogenisation time, etc.),

- mixing conditions.

5.3. Sample processing and analysis

The packaging, shipping and laboratory processing of the samples must be carried out under conditions that preserve their representativeness of the manufactured mix.
One tracer dosing is made in each sample selected for analysis. These analyses must be performed according to validated procedures, particularly in terms of repeatability.
If an analysis is carried out on a test portion that is smaller than the samples, it is recommended to finely grind 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.
The bulk density and grain size of the feeds may also be analysed in order to characterise the test conditions.

5.4. Processing the results

The results undergo statistical processing. Firstly, a calculation of the mean (m) of all the analyses and the identification of the expected concentration (C) according to the weighings is used to determine the tracer's recovery rate (% TR):

$$TR = 100 . \frac{m}{C}$$

For single analyses (one analysis per sample) on each sample, the calculations of the variance (V_{tot}) and mean (m) for all the analyses are used to calculate an overall coefficient of variation (as a %), based on the equation:

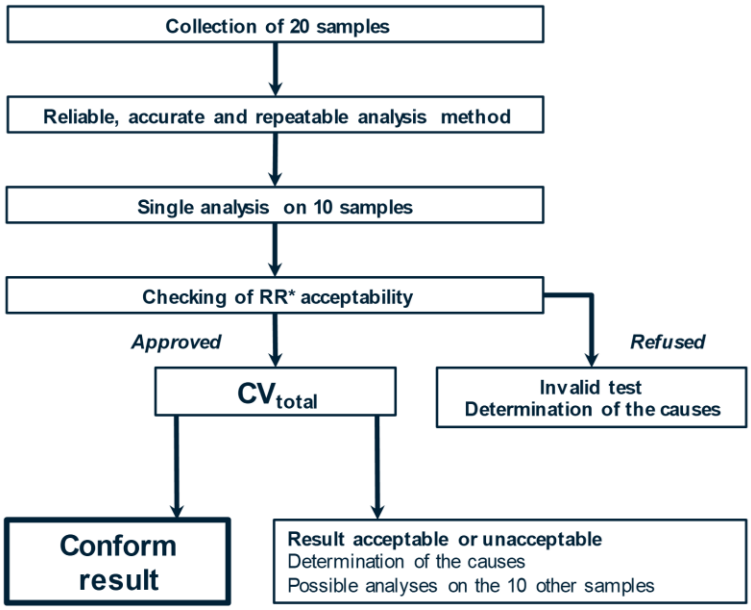
$$CV_{tot} = 100 . \frac{\sqrt{V_{tot}}}{m}$$

In cases where the overall variance could be explained largely by variability in the analysis method, it is recommended to duplicate the analyses on each sample. This will modify the statistical processing of the results and make it possible to derive from the overall variance, the residual variance including the analytical variance.
To do this, a variance analysis is performed based on the randomised model (see Technical Datasheet N°35). This makes it possible to compute the homogeneity variance (V_{hom}) by determining the "inter-samples" variance. This gives a CV_{hom} of:

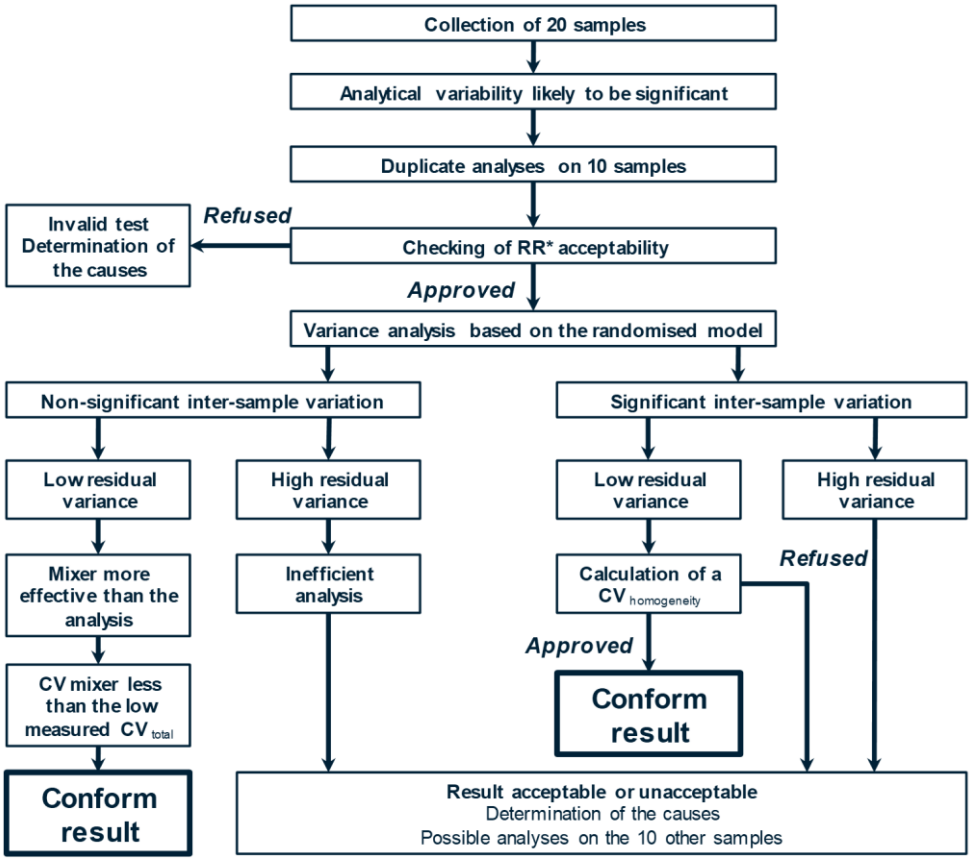
$$CV_{hom} = 100 . \frac{\sqrt{V_{hom}}}{m}$$

5.5. Interpretation of the results

This interpretation is based primarily on the decision trees shown on the following page. The first tree corresponds to the conventional case, while the second is used if there are doubts over the analytical performance of the selected tracer.



* Permissible recovery rate (RR) ranging between 70% and 110%[15] (80% to 110% for the manufacture of medicated feeds).



CV conformity (homogeneity or overall) is evaluated based on:

- the obligations of the industrial site,
- the quality objective selected by the industrial in relation to the threshold values indicated in the corresponding guides.

Interpretation of the results is based on:

- the change in inter-sample concentrations,
- the change in CVs over time (from one test to another). It is strongly recommended to track this change given the comparable nature of the results (closeness of assessment conditions).
- the data collected over the industry as a whole and the variation recorded from one test to another concerning:
  - manufacturing practices,
  - test procedures (sampling point, selected tracer, batch size, etc.),
  - the tested manufacturing circuit.

6. Bibliography

- **i'Doc_H2** : 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 carry-over between animal feeds). Tecaliman - 2000.
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- Report 3: Essais industriels d'évaluation de l'homogénéité d'aliments pondeuses en farine à la sortie de 16 sites industriels. (Factory assessment tests on the homogeneity of feedstuffs in the form of meal for laying hens at the output of 16 industrial sites) Tecaliman - 2000.
- Le point sur l'homogénéité des aliments composés. (Review of the homogeneity of compound feeds) Tecaliman - 1998.
- **i'Tec_H3**

Cross-references

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

[2] Analysis quality depends primarily on the choice of sample processing protocol, and also on the method used to interpret the results.

[3] 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).

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

[5] Due to the large number of analyses required.

[6] This is to ensure that it is solely their behaviour in the mix that is represented. Due to the diversity of their sources, formula ingredients, such as proteins, fats or ash, cannot be used as a basis for testing the homogenisation performance of powdered ingredient mixers as they are usually already distributed fairly uniformly throughout the feed components.

[7] This figure ensures a presence of at least 10,000 particles in each sample when the product is introduced at a concentration of 100 ppm and the sample size equals 100g. It is possible to choose a tracer that contains a lower number of particles per gram, but it should be noted that any decrease in this particle number may result in a higher coefficient of variation.

[8] Technical mastery over the product's composition and physical characterization will provide scope for some test standardisation, thus facilitating the collection of historical data on comparisons.

[9] Various data support the existence of a small variation between the homogeneity achieved at the mixer output and that recorded in the loaded batch, i.e. the bibliography and, in particular, the study carried out by the IFF in 1982 (Einfluss der physikalischen stoffeigenschaften am beispiel spurenelemente auf die mischgüte und anforderungen an verarbeitungsanlagen beim einsatz von mikrokomponenten im mischftutter. Bull. Inf. N° 183, 10-26.), survey data, and one of the tests carried out on feeds for laying hens.

[10] It has been demonstrated that the tracer may follow preferential flows in the feed flow.

[11] If possible less than 70 t/h.

[12] This involves a compromise between the statistical validity of the results and the analytical cost of a given test. This number is important, as it is necessary to determine as accurately as possible a standard deviation rather than a mean. A study on how variation coefficients change as a function of sample number demonstrated that both this choice and that of the number of samples analysed were the most pragmatic solutions while still ensuring the statistical validity. This is a target objective, the achievement of which is facilitated by the method used to determine sampling frequency. However, this number can withstand variations of a few samples.

[13] This corresponds to a compromise between sampling constraints, representativeness, and the laboratory's sample processing options.

[14] The initial sample is collected after a certain time, so as to limit the potential impact of cross-contaminations on the homogeneity measurement and to obtain a balanced distribution of samples over the whole batch throughput.

[15] The aim here is to check the homogeneity of the feedstuff rather than its labelling compliance. Attention should therefore be focused on the distribution pattern. However, note than any reduction in the mean, at a constant standard deviation, will increase the coefficient of variation. This means that a loss of tracer may generate an apparent increase in heterogeneity.