i'Tec_L6
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Proposal for technical rules for assessing the degree of homogenisation of liquid additives

These rules take the form of a proposal aimed at allowing test results to be compared. These rules have not yet been validated in an industrial context, and may therefore be subject to future modification. They have been drafted as part of the implementation of technical rules designed to assess the degree of homogenisation of all additives in a mix, followed by additives in powdered form (i'Tec_H1). It has been decided to propose rules suited to additives incorporated as solutions. These recommendations have been established on the basis of:

- previous studies and research on powdered additives.
- pilot and bibliographical studies carried out at Tecaliman on this topic.
- industrial observations.

These rules are only guidelines; there is no obligation to apply them. Each user is free to use their preferred method provided that they can demonstrate that their result, and its calculation method, lie within a field of reliability.
Note, however, that the procedure used must be strictly identical in each test in order to minimise bias when comparing results.

1. Focus

Study the distribution pattern of a substance incorporated as a solution into a mix produced at an animal feed plant. This substance is referred to as a tracer. A direct consequence of this technical requirement is that the measured homogeneity applies solely to the tracer; its full extrapolation would therefore raise the potential for errors concerning other mix components incorporated into the mix in a similar manner (i.e. other liquids, liquids incorporated via other means or circuits, etc.).

2. Principle

The method consists in:

- Selecting a tracer.
- Preparing a mix containing the tracer.
- Taking the samples.
- Dosing the tracer in the samples
- Obtaining a result in the form of a coefficient of variation.

The assessment results provide a snapshot of how well the tracer has been incorporated under the test conditions.

3. Equipment and apparatus

3.1. Choice of tracer

The tracer is the analysed molecule. It is incorporated as a solution and may correspond to a variable percentage of this solution.
While the tracer used may be a product that presents a particular interest or advantage it may not, however, possess the necessary qualities for being a useable tracer. In other words, it may prove impossible to evaluate the homogeneity of certain tracers without risking significant errors.
It is recommended that homogeneity tracer selection(s) be based upon the following criteria:

- It must be possible to incorporate the tracers at low concentrations of between 100 and 500 ppm.
- It must be possible to dose them using an analysis method that is accurate, repeatable, sensitive, simple and cost-effective.
- They must be mainly, or even exclusively, vehicled by a single source (absence of endogenous tracers or option for a differential analysis comparing endogenous and exogenous tracers).
- It must be possible to incorporate them upstream of the sampling location.
- They must not be destroyed or modified by the operations performed between the time of their incorporation and the sampling point.
- They may be internal or external.

3.2. Mix base

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

- when assessing the homogeneity of animal feeds produced by the industrial tool, a compound feed that is representative of the manufacture at the plant being tested may be used,
- when investigating the incorporation process a ground raw material or a mix of ground raw materials, possibly with a defined set of physical characterizations, may be used.

4. Method

4.1. Method of incorporation

Both before and at the time of taking the samples, care must be taken to ensure that the products are incorporated according to the plant's standard manufacturing practice.

4.2. Sampling location

It is recommended to take the samples as close as possible to the point of incorporation. The best-case scenario is in a passing flow. Carry-over risks would have to be taken into account to ensure this does not affect the assessment.

4.3. Sampling procedure

The sampling procedure should be chosen so as to ensure that it:

- gives each component a statistically equivalent chance of being sampled
- ensures that samples can be taken safely,
- ensures that the samples taken are representative,
- provides samples of the desired size,
- is suitable for use at the sampling point,
- causes minimum alteration or disturbance of the samples.

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

- to cut the flow in differing directions from one sample to the next,
- to choose sampling points with low flow rates.

It is strongly advised not to take samples in static product batches (in mixers, hoppers, bins, boxes, etc.).

4.4. Number of samples

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

4.5. Sample size

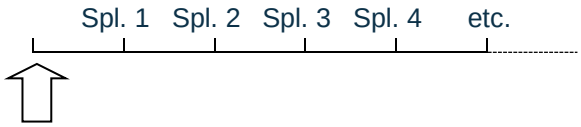
Sample size may range between 100g and 1000g. It is recommended to minimize variations in sample size for a given test.

4.6. Sampling frequency

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 that lapsed between the two samples by dividing the throughput time by the number of samples to be taken plus 1, i.e. 21 for 20 samples.

When getting ready to begin sampling, the stopwatch is activated at the start of batch throughput in order to allow a suitable time to pass before taking the first sample:



Start of batch throughput

The samples are taken at each time period and packed in numerical order. Sampling should continue until batch throughput is complete.
Should batch throughput halt at the sampling point due to a time delay, the stopwatch stops and then starts again once batch throughput restarts. This injection system time delay function has to be initially taken into account when calculating sampling frequency.

5. Testing

5.1. Preliminary jobs and checks

The following has to be checked at the sampling point:

- presence of the staff and equipment required to carry out the tests.
- product flow rate.
- throughput time for a similar batch, used to calculate sampling frequency.

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 properties of the solution (tracer content, viscosity, density, etc.),
- characteristics of the point of incorporation,
- quantities of raw materials dosed, including other liquids,
- sequence in which the products are incorporated into the mixer,
- list of and respective times for all mixing phases (premix, incorporation sequence for liquids and solids, homogenisation time, etc.),
- mix conditions (fill rate, blade speed, etc.).

A check-list can be used to facilitate this data collection.

6. Sample processing and analysis

The packing, shipping and laboratory processing of the samples must be carried out under conditions that preserve their representativeness of the manufactured product.
One tracer dosing is performed for each sample selected for analysis. These analyses must be performed according to current standardised procedures.
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), 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.

Should there be doubts over whether the accuracy of the analysis method is having a significant effect or should the effect be unknown, it is advised to duplicate the analyses on each sample. This will modify the statistical processing of the results.
The bulk density and particle size of the feeds may also be analysed in order to characterise the test conditions.

7. Processing the results

The results undergo statistical processing. For single analyses (one analysis per sample) on each sample, the calculation of the variance (Vtot) and mean (m) for all the analyses will be used to calculate an overall coefficient of variation, based on the equation:

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

If the sample analyses are duplicated, the overall variance value may be deleted, as the residual variance includes the analytical variance (see i'Tec_H3). To do this, a randomised model variance analysis is performed. This makes it possible to compute the homogeneity variance (Vhom) by determining "inter-sample" variance. This gives CVhom (see Tecaliman's i'Tec_H3) as:

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

8. Interpreting the results

This interpretation is based primarily on the decision tree shown on the following page.
Mean concentration is deemed acceptable if it lies between 70 and 110% of the expected concentration. Any deviations from these values may be accepted if they can be explained by the industrial context. CV conformity (homogeneity or overall) is evaluated based on three benchmark criteria:

- The industrial's chosen quality objective.
- How the results change over time (from one test to another).
- the data collected over the industry as a whole and possible variations recorded from one test to another.

In the event of a nonconformity, the cause should be researched based on the various factors likely to be involved:

- Equipment (point of incorporation, condition of the nozzles, etc.)
- Spraying procedures (flow rates, start and end of spraying operations, etc.)
- Products (shape of the matrix, data on the tracer, etc.)
- etc.

9. Conclusion

The homogeneity assessment method can be summarised as follows:

1) Define the objective

- Test plant performance for every feed it manufactures.
- Study the process in order to identify variance factors or performance improvement procedures.
- Study a product and/or a line
- etc.

2) Select the equipment and apparatus

- Tracer and its solution
- Media
- Circuit

3) Establish the method

- Sampling location
- Sampling procedure
- Number of samples
- Sampling frequency
- Number and method of the analyses on each sample

4) Choose controlled factors

- Fill rate
- Incorporation time
- Incorporated quantity
- Flow rate at the sampling point,

5) Measure the factors

- Tracer properties
- Media properties
- Circuit properties
- Concentration of tracer in the samples

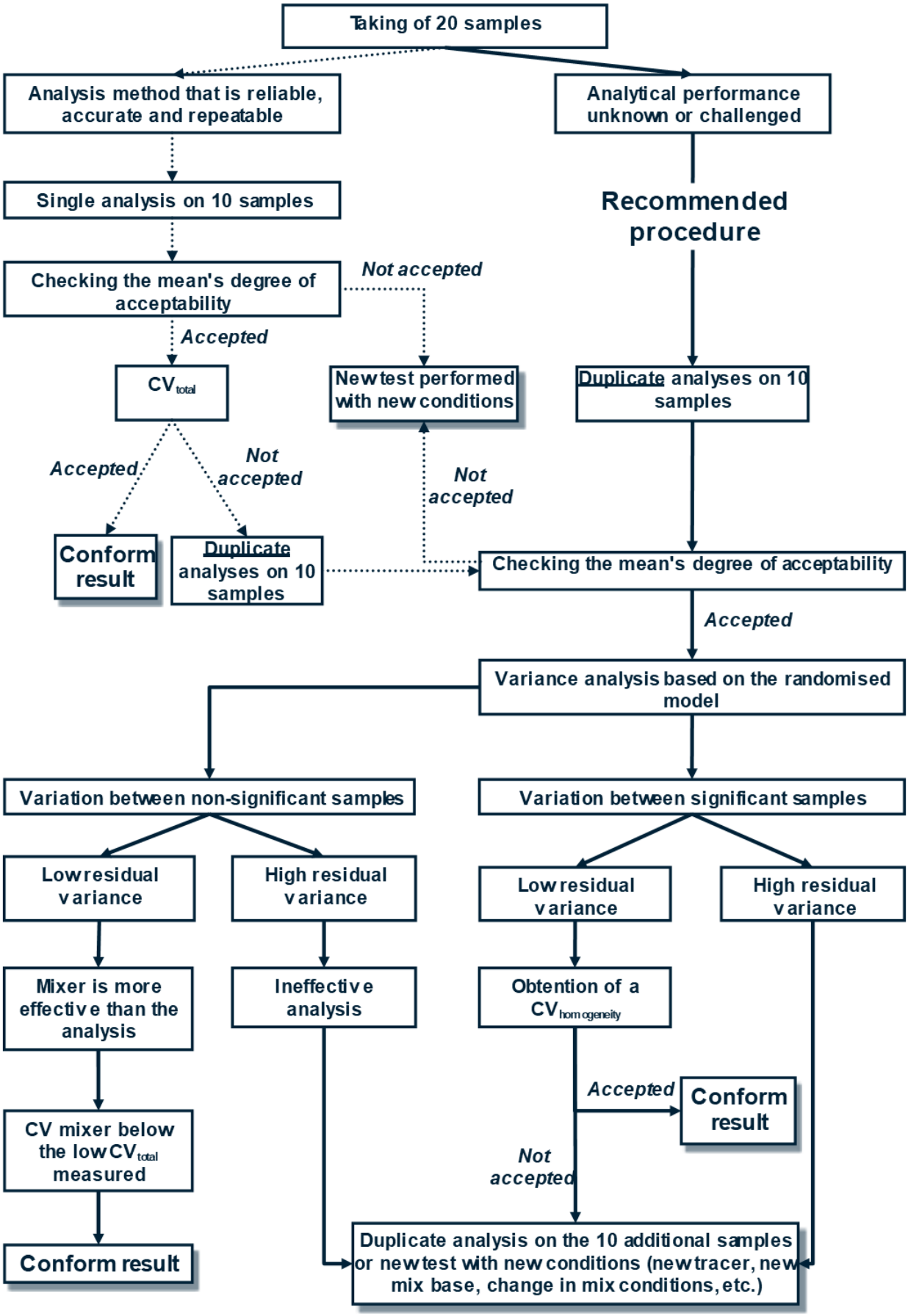
6) Record the results

- Mean concentration
- Coefficient(s) of variation

7) Interpret the results

- According to the decision tree
- According to all the factors involved in the result
- According to the plant's objective and industry-wide datasets

The result cannot be extrapolated to other conditions without risking errors.


Taking of 20 samples
Analysis method that is reliable, accurate and repeatable
Analytical performance unknown or challenged
Single analysis on 10 samples
Recommended procedure
Checking the mean's degree of acceptability
Not accepted
Accepted
CV total
New test performed with new conditions
Duplicate analyses on 10 samples
Accepted
Not accepted
Not accepted
Conform result
Duplicate analyses on 10 samples
Checking the mean's degree of acceptability
Accepted
Variance analysis based on the randomised model
Variation between non-significant samples
Variation between significant samples
Low residual variance
High residual variance
Low residual variance
High residual variance
Mixer is more effective than the analysis
Ineffective analysis
Obtention of a CV homogeneity
Accepted
Conform result
Not accepted
CV mixer below the low CV total measured
Conform result
Duplicate analysis on the 10 additional samples or new test with new conditions (new tracer, new mix base, change in mix conditions, etc.)