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Homogeneity of mixes
Summary of experimental data obtained by Tecaliman

In addition to the data published in Special Report no. 41, the base data compiled by Tecaliman between 1993 and 2001 may form the basis for comparing future results. Over the course of the period up to 2001, Tecaliman conducted 364 assessments of tracer dispersion, under all conditions. These assessments were carried out as pilot studies or on industrial sites, on feeds or premixes, in the mixer or following transfer, for various types of mixers and with various types of tracers. The sample sizes were between 100 and 1000 g and there were 10 to 40 of them, depending on the test. The results obtained were analysed as a whole, and for all conditions, or divided on the basis of three criteria[1]:

- The type of matrix: feed or premix
- the type of tracer: internal tracer, trace element or external tracer
- the type of test: pilot or industrial

1. Comparison of variances

254 assessments have enabled inter and intra sample variances to be compared, by means of variance analyses based on a random model, thanks to the repetition of analyses on each of the samples.
In **44.1 %** of cases (i.e. 112 tests), no significant difference was detected (Table 1) between the samples. Consequently, an "interpretable" $CV_{homogeneity}$ value was only obtained for 142 assessments.

There may be two possible explanations for this lack of significance:

- The mix is heterogeneous, but the sampling and analysis process, combined with the tracer used, produces even greater differences and, therefore, is not effective enough to detect it.
- The mix is homogeneous and the sampling and analysis process is not yet effective enough to fully detect this.

	No. of tests	Number of insignificant tests	% of insignificant tests
All tests	254	112	44.1
Feed	151	44	29.1
Premix	103	68	66.0
Pilot study	110	63	57.3
Industrial study	144	49	34.0
Internal tracer	62	33	53.2
Trace elements	62	21	33.9
External tracer	130	58	44.6

Table 1: Distribution of the significance of variance analysis tests using the random model obtained during the course of tracer distribution assessments conducted by Tecaliman since 1993 where the analyses were carried out at least in duplicate

In the case of feed/premix comparisons, obtaining a larger number of insignificant tests with premixes must be linked to that of the CV_{total} value, which is broadly lower (Table 2). Therefore, it appears that, very often, during tests conducted on premixes, the tracers used do not enable the homogeneity to be properly characterised. The same kind of finding can be obtained by comparing pilot/industrial studies with the best homogeneity and a higher percentage of insignificant tests in pilot studies.

A comparison of the types of tracers appears more complex, but a combination of a higher percentage of insignificant tests and a greater CV_{total} value leads, in their case, to a focus on the first explanation, i.e. analytical processes obtaining variations that are greater than those originating from the mixing process, which are already significant.

[1] The description of populations is conventional, providing (in brackets) successively: the mean, standard deviation and median.

2. Study of populations

For CV_{total} values, the frequency distribution of the 364 results (5.96, 4.44, 4.90) is closer to that of a Poisson distribution than of a Normal distribution (Figure 1) like that obtained by the IFF (1982). Consequently, a marked difference is noted between the mean and the median, with the mean being shifted upwards, by the inclusion of a number of high results. With this kind of distribution, it is preferable to view the median and a distribution of individuals on the basis of the frequency of occurrence, even if population comparisons are made on the basis of means and standard deviations. These distributions of CV_{total} values and $CV_{homogeneity}$ values in the case of significant tests only, based on the different divisions made, appear in Table 2 and Table 3.

For the CV_{total} value, the population of 206 tests obtained for feeds (6.7, 4.9, 5.4) appears different from that of 158 tests conducted on premixes (158, 5.0, 3.6, 4.0). There is a 1.4% difference between the medians and 1.7 % between the means. A statistical comparison of the two populations produces a significant difference (with a risk of 5 %). The same applies for these kinds of populations, but by only studying the $CV_{homogeneity}$ values the tests resulted in significant differences being obtained: 105 tests on feeds (7.2, 5.5, 5.4) and 35 tests on premixes (4.7, 4.5, 3.1).

Therefore, the mixes tested by Tecaliman on a premix matrix appear more homogeneous overall. This observation bears out those made by other authors (Heping and Chuanping, 1988 - Van Den Bosch, 1992 - Heidenreich, 1998).
A comparison of test populations undertaken during pilot studies and within plants could lead it to be thought that the coefficients of variation for industrial tests are higher than those determined by the pilot study. Nevertheless, a statistical comparison only reveals significant differences (with a risk of 5 %) in the case of $CV_{homogeneity}$ values:

- CV_{total} value: 110 Pilot tests (5.0, 4.6, 3.7) and 254 Industrial tests (6.4, 4.3, 5.4)
- $CV_{homogeneity}$ value (significant tests): 47 Pilot tests (4.4, 4.4, 2.8) and 95 Industrial tests (7.6, 5.5, 5.9)

It does not seem wise to interpret these results as a systematic distance between these two assessment areas, which would result in a possible problem of scale transfer, as the tracers, matrices and devices for these two populations are all different. However, it appears that the mixes produced in a plant are, after removing the variations of analytical processes, a little more heterogeneous than those from the pilot study.
Consequently, it appears necessary to compare a result obtained on an industrial site with a similar population.

Having noted a difference between feeds and premixes, the creation of populations divided on the basis of combinations of these four factors seems advisable (Table 4). This kind of division could be continued by also taking account of the sampling location. In this case, a population that appears interesting is that of CV_{total} values for feeds produced in an industrial setting and sampled at the mixer outlet (121 tests), the CV_{total} value mean obtained, for all other test conditions, is 6.7 % and the median is 5.8% (Table 4).

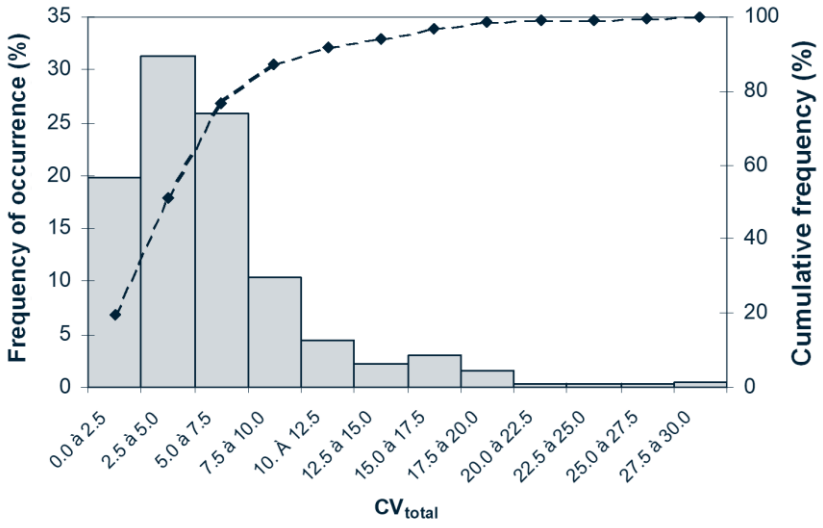

Figure 1: Frequency of CV_{total} values in a set of 364 results of distribution assessments carried out by Tecaliman

	Indiv. No.	Percentage of the total population										
		Min.					Med.					Max.
			⇨10%	⇨20%	⇨30%	⇨40%	⇨50%	⇨60%	⇨70%	⇨80%	⇨90%	⇨100%
Entire population	364	1.0	2.0	2.6	3.5	4.0	**4.9**	5.6	6.4	8.0	11.4	30.0
Feed	206	1.2	2.2	3.2	3.8	4.7	**5.4**	6.1	7.4	9.4	13.2	30.0
Premix	158	1.0	1.9	2.4	2.9	3.6	**4.0**	4.7	5.6	6.6	8.2	23.8
Pilot study	110	1.2	1.8	2.2	2.5	3.2	**3.7**	4.3	5.2	6.2	8.9	29.0
Industrial study	254	1.0	2.2	3.1	3.9	4.6	**5.4**	6.0	7.0	8.5	11.8	30.0
Internal tracer	77	1.8	2.5	3.5	4.2	5.3	**5.6**	6.3	7.0	8.7	12.2	29.0
Trace elements	138	1.0	2.1	2.6	3.3	4.0	**4.6**	5.9	6.6	7.9	10.4	18.3
External tracer	149	1.2	1.9	2.4	3.1	3.7	**4.4**	5.1	5.7	7.8	12.1	30.0

Table 2: CV_{total} values obtained during the course of tracer distribution assessments conducted by Tecaliman since 1993

	Indiv. No.	Percentage of the total population										
		Min.					Med.					Max.
			⇨10%	⇨20%	⇨30%	⇨40%	⇨50%	⇨60%	⇨70%	⇨80%	⇨90%	⇨100%
Entire population	140	0.7	1.7	2.4	3.2	3.8	**4.8**	5.9	7.7	10.2	15.0	29.0
Feed	105	0.7	1.7	2.4	3.5	4.6	**5.4**	7.1	9.2	10.7	15.1	29.0
Premix	35	1.0	1.7	2.3	2.5	2.8	**3.1**	3.6	4.5	5.5	8.5	20.3
Pilot study	45	0.7	1.1	1.7	2.1	2.5	**2.8**	3.2	3.9	5.5	9.4	20.3
Industrial study	95	1.0	2.2	3.3	4.1	4.9	**5.9**	7.6	9.4	10.9	15.3	29.0
Internal tracer	29	1.5	1.7	2.4	3.0	3.7	**4.7**	5.9	6.7	7.9	11.1	20.3
Trace elements	41	1.0	1.8	2.6	4.0	4.9	**5.4**	6.0	7.3	10.3	11.8	16.8
External tracer	70	0.7	1.6	2.2	3.1	3.5	**4.1**	5.3	8.8	11.1	15.4	29.0

Table 3: $CV_{homogeneity}$ values obtained during tracer distribution assessments conducted by Tecaliman since 1993 where the variance test is significant

	Indiv. No.	Percentage of the total population										
		Min.					Med.					Max.
			⇨10%	⇨20%	⇨30%	⇨40%	⇨50%	⇨60%	⇨70%	⇨80%	⇨90%	⇨100%
Industrial/Feed	165	1.3	2.8	3.7	4.5	5.3	**5.8**	6.7	7.9	9.8	14.0	30.0
Industrial/Premix	89	1.0	1.8	2.2	2.7	3.6	**4.0**	5.1	5.9	6.6	8.2	17.5
Pilot/Feed	41	1.2	1.5	1.9	2.1	2.4	**2.9**	3.3	4.7	5.6	8.8	29.0
Pilot/Premix	69	1.2	2.1	2.4	3.0	3.6	**4.0**	4.6	5.2	6.6	9.2	23.8
Industrial/Feed/Mixer outlet	121	1.3	3.0	4.0	4.7	5.3	**5.8**	6.3	7.3	9.0	11.3	18.9

Table 4: CV_{total} values obtained during the course of tracer distribution assessments conducted by Tecaliman since 1993 distributed on the basis of the type of test and the type of matrix

The division of results by type of tracer used has also been studied. The curves plotted in Figure 2 and Figure 3 take the form of cumulative frequencies, the data in Table 2 and Table 3 relates to the types of tracer.

The statistical tests for side by side comparison of populations only detect a significant difference between the population of CV_{total} values for

internal tracers and that for trace elements.
No significant difference is detected between the population of coefficients of variation established for external tracers and those for the two other types of tracer. Consequently even though, seemingly, the population of CV_{total} values for internal tracers appears to have values that are a little higher (Figure 2) with a very small difference of 1.2 % from the median, this is offset by the capacity and reliability of external tracers in detecting a drift towards heterogeneity (Figure 3) with higher $CV_{homogeneity}$ values when the values rise above the medians for populations.

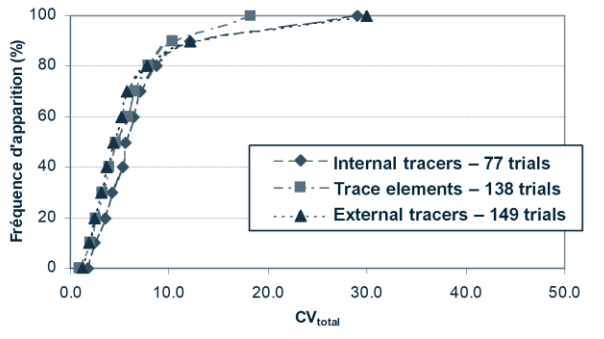

Figure 2: CV_{total} value frequency curve for distribution assessments conducted by Tecaliman based on the types of tracers used

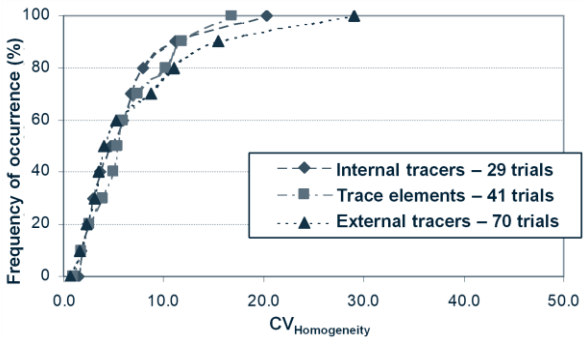

Figure 3: $CV_{homogeneity}$ value frequency curve for distribution assessments conducted by Tecaliman based on the types of tracers used for significant variance tests

3. Conclusion

A difference in the population of coefficients of variation was detected between feeds and premixes. A comparison between pilot and industrial tests only reveals a significant difference between $CV_{homogeneity}$ values. Therefore, it seems advisable to make a distinction between the four populations segmented by these two pairs.
However, no significant difference was detected between the populations formed with internal tracers or trace elements, on the one hand, and external tracers, on the other hand. This observation enables us to regard the latter as forming a valid basis for assessing the homogeneity of mixes, or even providing access to improved food safety, as they appear to magnify the occurrence of possible dispersion problems.

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