i'Tec_H18
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Key-words: Mixing, Carry-over, General

Database on homogenisation performance and carry-over test in French feed factories

Analysis of 2000-2018 results

In 2019, like every three years or so, Tecaliman carried out a survey following previous surveys.

1. Area of Investigation

1.1. Factories

Participating companies were asked about their evaluation results from **2016** to the end of **2018**. These data therefore supplemented the previous data acquired for 2000 to 2015.
As usual, all results were collected, even those which have led to unsatisfactory results for industrialists and which have subsequently been the subject of corrective measures and new tests in so far as these results have been presented to certifying or accreditations agencies.
In order to validate the minimum compliance of the tests, the survey required a certain number of data in a mandatory manner. If these data were not transmitted, the results were removed from treatment. Thus, only tests carried out in France, under conditions which comply with or are compatible with the established rules (Tec Tecaliman T2 and H1) in feed plants, were treated.
The main reasons for this selection were:

- failure to comply with the tracer recovery rate rule limiting its acceptability to 70-110%.
- For carry-over test, the results were accepted globally, as presented to the DDCSPP or auditors of the animal nutrition certification compendium, but tests that have been done without following the recommendation to take samples at least at the entrance of the silo before pellet mills.

An evaluation of the representativeness of the tested plant fleet is carried out. The participating companies reported mill production on a 10,000 tons fractional scale. Treatment shows that the number of panel plants increases from year to year until 2003, then remains relatively stable (Figure 1) before increasing over the last three years.

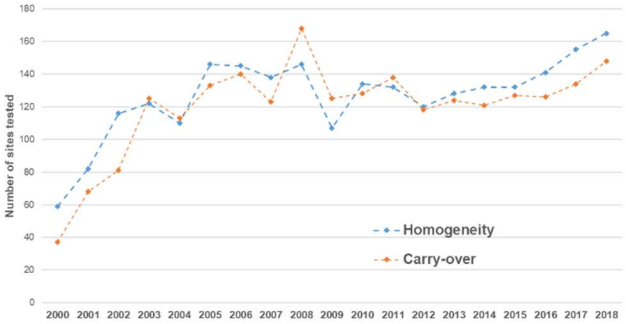

Figure 1 : Evolution of the total number of feed manufacturing plant that transmitted by year, all sampling points combined

In terms of production level, the representativeness has increased from year to year to be finally very significant of the total production in France and corresponds to about 80% of the French production over the last years (Figure 2).

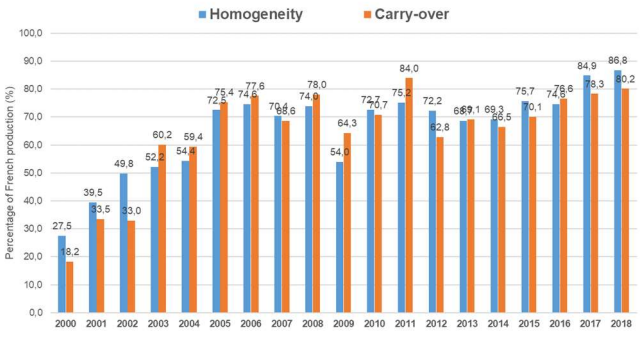

Figure 2 : Percentage of French production by year, all sampling points combined

1.2. Tracers

The results treated relate only to tracers which are mainly supplied by a single source and incorporated in powder form. The tracers selected are grouped into 3

groups:

- **Internal**: Amoxicillin, Chlortetracycline, Decoquinate, Dimetridazole, Flubendazole, Lasalocid, Monensin, Narasin, Oxytetracycline, Paracetamol, Salinomycin, Sulfadiazin, sulfadimethoxin, Tilmicosin, Tiamulin, Tylosin
- **External**: Microtracer F, Microtracer RF blue or red, Microtracer RF blue lake super fine
- **Trace elements**: Chloride, Copper, Magnesium, Manganese, Ytterbium, Zinc, …

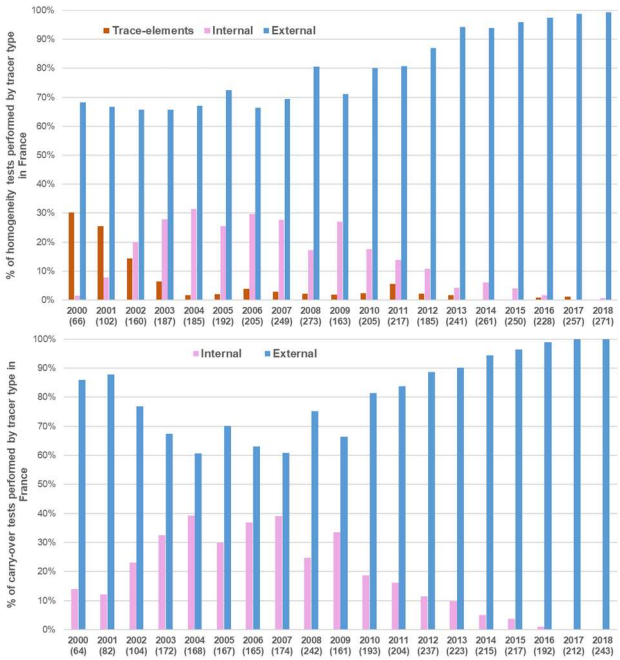

Figure 3 : Percentages of homogeneity assessments (top) and carry-over test (bottom) by years and groups of tracers

Trace elements are only accepted as part of the homogenization capability assessments. The tracers used in carry-over test are those highlighted, although there is some doubt as to the capacity of laboratories to have the levels of trace analysis necessary for a good evaluation of carry-over: detection limit of at least 0.5% of the tracer batch dose. This is the case for all drug molecules other than OTC and CTC.

The distribution of tracer types, based on assessments, shows that a majority is now being performed using external tracers (Figure 3): 99% homogeneity and 100% carry-over for 2018. In the field of homogeneity, the use of trace elements has disappeared since 2014.

2. Homogeneity

A total of 3897 evaluations were selected. **All conditions and all years combined, the median coefficient of variation (CV) is 4.1%**, below the ANCG (Animal Nutrition Certification Guide OQUALIM) compliance target of 5.0% (Table 1).
About 65% of the results are below this target of 5% on all data and 70% over the last 3 years. 95% of values are less than 10%, another ANCG limit. **Thus, during the capability tests of their mixer, the plants have a 95% chance of being in the compliance zone and 30% will have to be monitored.** Over time, the annual distribution clearly shows a gradual decrease in the median of about 0.15%/year until 2013, demonstrating the improvement in the performance of the profession (Figure 4) over 12 years. This progress seems to have stopped since. However, the database seems more complete over the last few years, it is possible that plants that have not yet achieved the performance of others have entered the panel.

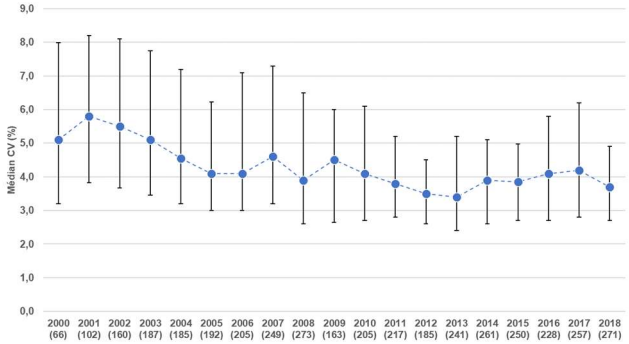

Figure 4 : Change in median population of coefficients of variation over years (number of individuals per year in parentheses)

A refocusing of the population around the median with disappearance of the high extremes is visible on the evolution of interquartile intervals (Figure 5) even if, at the same time as the stagnation of the median since 2013, this interval is no longer reduced.
A description of CV populations is given by groups of years (Table 1).

From a statistical point of view, it is possible to say that there are 3 groups of years that overlap more or less, in the descending order of the CVs: from 2000/2003, then from 2004/2008 and finally 2009/2018.
The observation of populations by year in the form of frequency curves (Figure 6) every 3 years shows that it is the entire population of coefficients of variation that migrates to lower values.

A progressive disappearance of CVs between 5 and 10% is observed mainly for the benefit of group 2.5/5.0%. The existence of extreme values remains constant, although their frequency and levels have declined significantly in recent years.

The beginning of population stagnation that has been observed since 2011 and probably corresponds to the trend that was expected; **This is a progressive shift from total CVs to minimum CVs driven by unexplained variations in analytical errors and fundamental sampling error**.
Since the ANCG sets two compliance and acceptability limits of 5% and 10%, it is interesting to look at the evolution of the percentages of tests having obtained results in the three classes established by these two thresholds (Table 2 - Figure 7). It is then easy to see that the class below 5% has increased to represent up

to 75% in the years 2013-2015 to the detriment essentially of the class 5/10%, but a decline of this progression is observed over the last three years. The last class > 10% only decreased slightly to slightly below 10% over the last three years.
Several hypotheses can be formulated to try to understand this evolution:

- The arrival of “new” plants in the panel (visible by the increase in the number of tests covered by the panel) for which the optimisation work has not been fully carried out
- Slight loss of breath in commitment to quality homogenization
- Aging of a portion of the mixer fleet or mobiles requiring change.

In these assumptions, the first will be of interest to us because it is also possible to see on Figure 6, the percentage of results above 20% increase significantly between 2015 and 2018.

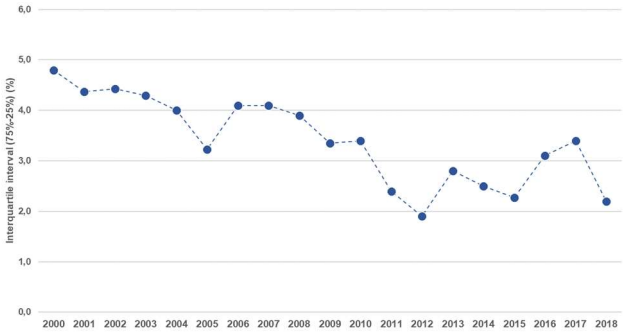

Figure 5 : Evolution of the interquartile population interval of coefficients of variation as a function of years (number of individuals per year in parentheses)

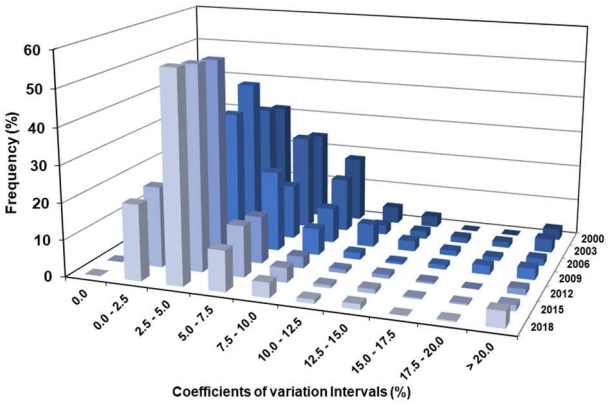

Figure 6 : Coefficient of variation frequency curves across all conditions every 3 years from 2000 to 2018

Groups of years	Nb..	Mini.	⇨10%	⇨20%	⇨30%	⇨40%	⇨50%	Med.	⇨60%	⇨70%	⇨80%	⇨90%	⇨100%	Maxi.
		Percentage of total population												
2000-2003	515	1.2	2.4	3.2	3.9	4.7	**5.4**	6.4	7.2	8.9	11.3	35.4		
2004-2006	582	0.7	2.2	2.8	3.3	3.8	**4.2**	4.9	6.1	7.7	12.0	130.0		
2007-2009	685	0.9	2.0	2.5	3.1	3.7	**4.3**	4.9	5.7	7.6	11.4	73.7		
2010-2012	607	0.8	1.9	2.4	2.9	3.3	**3.7**	4.3	4.9	6.0	9.4	59.2		
2013-2015	752	0.7	1.8	2.3	2.8	3.3	**3.8**	4.3	4.8	5.8	8.1	81.5		
2016-2018	756	0.7	2.0	2.5	3.0	3.5	**4.0**	4.5	5.0	6.5	9.6	63.1		

Table 1 : Population distributions of total coefficients of variation by groups of years

Groups of years	% of tests with CV		
	< 5 %	5 % à 10 %	> 10 %
2000-2003	44.9	41.1	14.0
2004-2006	61.6	25.3	13.0
2007-2009	62.0	24.8	13.1
2010-2012	72.4	18.4	9.2
2013-2015	75.4	17.0	7.6
2016-2018	71.4	19.1	9.5

Table 2 : Evolution of average CV percentages by “compliance/acceptability” class over time

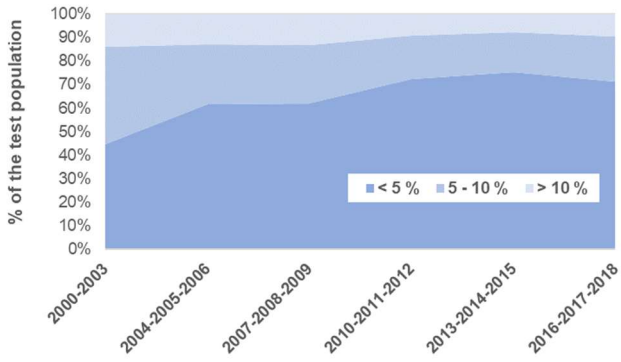

Figure 7 : Changes in cumulative average percentages by “compliance/acceptability” class as a function of years

3. Carry-over

3.1. Overall analysis

A total of 3435 results were received, but only 2244 are actionable against the technical rules or are finalized data for presentation to the auditors. However, a large part of these results (27.5%) are tests performed at the mixer outlet, which is not the recommendation made in the rules. The communication that has been made on this point has borne fruit from 2013 to 2014, with a decrease of more than 20% of the percentage of tests done at this point but this percentage has tended to stagnate since around 23% without progression over the last years. Due to the significant share of this population, the overall results are given without these values obtained on a short circuit (Table 3).

For all years and all sampling points, the median for the first lot is 2.7% and 0.6% for the second. If only the results after the entry of the pellet mill silo are taken into account, the medians move respectively to 2.9% and 0.6%. However, for the last 6 years alone, they are, under the same conditions, 2.7% and 0.5%. The medians of these populations are therefore still below the 5% target for feed. In the last 6 years, about 86% of the population is below 5% in the first collecting batch, 60% below 3% and 11.5% below 1%. Populations are fairly close to the median (Figure 8) and, this is even more true for the second collecting batch with, in this case, the problem of the approach of detection limits of the tracers used. Thus, results at zero can be interpreted as lower than the detection thresholds.

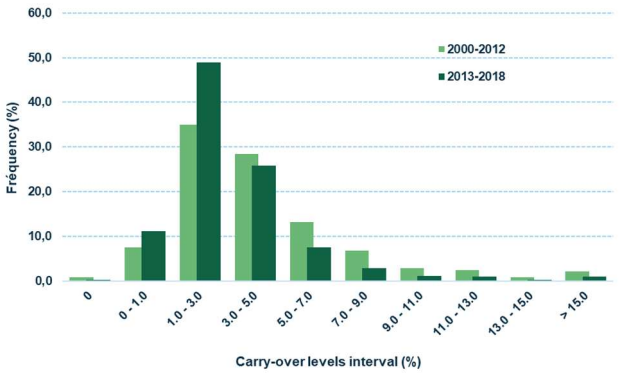

Figure 8 : Frequency histogram of carry-over levels to the first collecting batch

The median for collecting batch 2 is 0.5% for the last 6 years, which implies that in about 29% of cases, industrialists are required to put in place corrective measures in order to be able to manufacture finished or intended feed “animal product exporters”, since the limit target was set exactly at 1% in this case.

		Nb..	Percentage of total population										
			Mini.					Med.					Maxi.
			⇨10%	⇨20%	⇨30%	⇨40%	⇨50%	⇨60%	⇨70%	⇨80%	⇨90%	⇨100%	
All data	**C1**	3435	0.0	0.8	1.3	1.8	2.2	**2.7**	3.2	3.8	4.8	6.8	94.0
	C2	3435	0.0	0.2	0.3	0.4	0.5	**0.6**	0.7	0.9	1.2	1.7	39.0
Only after entry of pellet mill silos	**C1**	2244	0.0	1.1	1.6	2.1	2.5	**2.9**	3.5	4.2	5.1	7.0	52.3
	C2	2244	0.0	0.2	0.3	0.4	0.5	**0.6**	0.8	1.0	1.3	1.8	39.0

Table 3 : Distribution of carry-over levels populations of the first 2 collecting batches (C1 and C2) obtained from 2000 to 2018 under all conditions and only on the population of the results obtained at least after the entry of pellet mills silos over all years

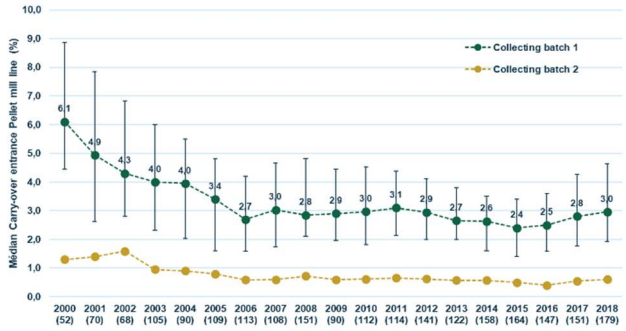

Figure 9 : Evolution of the median of carry-over levels populations after entry of pellet mills silos from the 2 collecting batches

The analysis by year (Figure 9) shows, for the first collecting batch, a gradual decrease in the median until 2006 and a relative stabilization slightly below 3.0% since that year (Table 4). It should be noted that if this median is stabilized, however, the overall population tends to shrink as shown by the steady decrease in interquartile intervals (Figure 10).

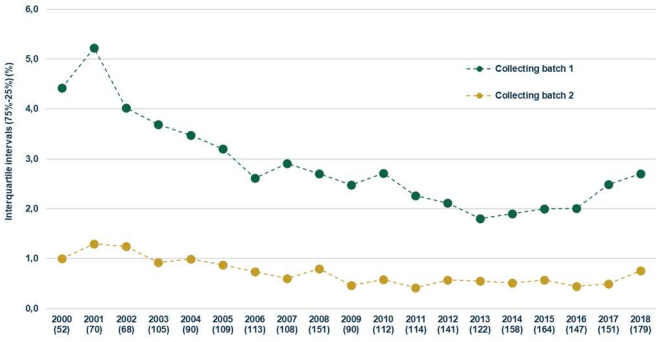

Figure 10 : Evolution of the interquartile interval (difference 75%-25%) of the carry-over level populations after entry of pellet mills silos of the 2 collecting batches

Changes in population distributions every 3 years (Figure 11) show that the population in the 7.5-10% range decreases regularly, as do the upper ranges.

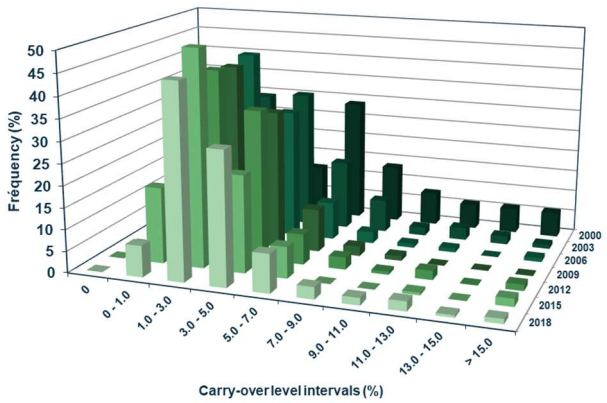

Figure 11 : Frequency curves of contaminant populations of the first collecting batch based on years every 3 years from 2000 to 2018

As a result, factories are being driven to move towards a narrow area of better overall quality, driven by increasing demands from public authorities and certification requirements, which leads to a decrease in the prevalence of extreme points.

However, the slight shift from the median to lower values is a sign of a difficulty for the profession to move clearly towards improving its performance in the current state of technology and without a radical change in practices. Evidence of this is the tightening of the population around the visible median until 2013, which has tended to disappear over the past 2 years.

4. Conclusion

Overall, the French animal nutrition profession is making good progress in mastering these phenomena, even if signs of exhaustion appear, which seems logical because of the available techniques. In the case of carry-over levels, a significant effort has been made to carry out these tests correctly at the pellet mills line entrance, and it is appropriate to continue in this direction.

All this data can be used as a reference to evaluate the progress made by each industrial site in controlling its performance.

	Years	Nb	Percentage of total population										
			Mini.					Med.					Maxi.
				⇨10%	⇨20%	⇨30%	⇨40%	⇨50%	⇨60%	⇨70%	⇨80%	⇨90%	⇨100%
Collecting batch 1	2000	52	0.4	1.6	3.2	4.9	5.4	**6.1**	6.8	8.2	10.4	13.5	28.2
	2001	70	0.2	1.7	2.4	3.0	4.1	**4.9**	6.6	7.5	8.8	11.7	38.2
	2002	68	1.0	1.9	2.4	3.1	3.8	**4.3**	5.2	6.4	8.0	9.8	23.2
	2003	105	0.0	1.6	2.0	2.5	3.4	**4.0**	4.5	5.3	6.2	8.3	16.3
	2004	90	0.0	1.2	1.8	2.3	3.2	**4.0**	4.5	5.2	6.7	9.6	21.7
	2005	109	0.0	0.7	1.3	2.0	2.6	**3.4**	4.1	4.6	6.0	8.7	17.5
	2006	113	0.2	0.7	1.3	1.7	2.2	**2.7**	3.2	3.8	4.5	5.5	23.9
	2007	108	0.0	1.2	1.7	2.0	2.4	**3.0**	3.6	4.3	5.1	6.6	52.3
	2008	151	0.4	1.4	1.8	2.3	2.6	**2.8**	3.7	4.6	5.4	7.4	18.9
	2009	90	0.0	1.0	1.7	2.1	2.5	**2.9**	3.4	4.2	4.6	5.5	9.1
	2010	112	0.3	1.1	1.6	2.1	2.5	**3.0**	3.4	3.9	4.8	6.1	37.0
	2011	114	0.0	1.1	2.0	2.4	2.7	**3.1**	3.7	4.0	4.8	6.2	12.0
	2012	141	0.1	1.1	1.8	2.2	2.5	**2.9**	3.4	3.8	4.3	6.3	17.0
	2013	122	0.1	1.1	1.8	2.1	2.3	**2.7**	3.1	3.5	4.5	5.5	18.7
	2014	158	0.2	0.8	1.5	1.9	2.3	**2.6**	2.9	3.3	3.9	5.7	22.1
	2015	164	0.2	0.8	1.2	1.6	2.0	**2.4**	2.9	3.1	3.7	4.8	22.8
	2016	147	0.0	0.9	1.5	1.7	2.0	**2.5**	2.8	3.2	4.4	5.9	12.5
	2017	151	0.0	1.0	1.6	2.0	2.4	**2.8**	3.1	3.8	4.8	6.7	18.4
	2018	179	0.2	1.3	1.7	2.1	2.5	**3.0**	3.6	4.2	4.9	6.5	24.5
Collecting batch 2	2000	52	0.1	0.3	0.6	0.8	1.2	**1.3**	1.6	1.7	1.9	2.2	10.6
	2001	70	0.0	0.4	0.7	0.9	1.1	**1.4**	1.6	2.0	2.7	5.3	10.6
	2002	68	0.2	0.6	0.8	1.0	1.3	**1.6**	1.7	2.0	2.4	3.4	8.3
	2003	105	0.0	0.4	0.5	0.6	0.8	**1.0**	1.1	1.4	1.6	2.4	9.1
	2004	90	0.0	0.2	0.4	0.6	0.8	**0.9**	1.1	1.3	1.6	2.3	4.0
	2005	109	0.0	0.2	0.4	0.5	0.7	**0.8**	1.0	1.2	1.6	2.3	5.7
	2006	113	0.0	0.2	0.3	0.3	0.5	**0.6**	0.8	0.9	1.2	1.6	8.7
	2007	108	0.0	0.2	0.3	0.4	0.5	**0.6**	0.8	0.9	1.2	1.6	4.0
	2008	151	0.1	0.2	0.4	0.4	0.5	**0.7**	0.8	1.1	1.4	2.1	6.1
	2009	90	0.0	0.3	0.4	0.5	0.5	**0.6**	0.7	0.8	1.1	1.3	2.1
	2010	112	0.0	0.2	0.3	0.4	0.5	**0.6**	0.7	0.9	1.0	1.4	12.0
	2011	114	0.0	0.3	0.4	0.5	0.5	**0.7**	0.7	0.9	1.0	1.3	39.0
	2012	141	0.0	0.3	0.3	0.5	0.5	**0.6**	0.7	0.9	1.1	1.5	3.8
	2013	122	0.0	0.3	0.3	0.4	0.5	**0.6**	0.7	0.8	0.9	1.3	3.8
	2014	158	0.0	0.3	0.3	0.4	0.4	**0.6**	0.6	0.8	0.9	1.4	4.1
	2015	164	0.0	0.2	0.3	0.3	0.4	**0.5**	0.6	0.8	0.9	1.4	15.9
	2016	147	0.0	0.2	0.3	0.3	0.4	**0.4**	0.5	0.6	0.8	1.0	4.3
	2017	151	0.0	0.2	0.3	0.4	0.4	**0.6**	0.7	0.8	1.0	1.5	5.2
	2018	179	0.0	0.3	0.3	0.4	0.5	**0.6**	0.8	0.9	1.3	1.7	4.6

Table 4 : Répartitions des populations de transferts inter-lots après entrée boisseaux de presse des lots collecteurs en fonction des années de 2000 à 2018

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