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014-PT

INTERLABORATORY COMPARISONS REPORT

Proficiency testing on physicochemical properties of pesticides formulations

01-2015-A – v2

Final Report

**This report replaces and cancels the previous final report 01-2015-A.
Error in the corrected pH dil. 1% result of Lab 15, pages 8 and 20.**

Validated on 2016/02/22 by Alain Dubois, Coordinator

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Our task is to preserve the
safety of the food chain and the
quality of food in order to
protect the health of humans,
animals and plants.



CONTENT

1. BASIC INFORMATION AND TIME SCHEDULE	3
2. STATISTICAL TREATMENT AND PERFORMANCE ASSESSMENT	4
3. REMARKS/COMMENTS OF THE PARTICIPANTS	5
4. TABLES AND CHARTS OF RESULTS	7
4.1. Legend for tables with data from laboratories	7
4.2. Results	8
4.2.1. Triadimenol content and diastereoisomers ratio (GLC-FID method CIPAC 398, CIPAC Handbook N, pp. 134 - 144)	9
4.2.2. Density (CIPAC MT 3.3.2, CIPAC Handbook F, pp. 19 – 21)	15
4.2.3. Acidity of formulation (CIPAC MT 191, CIPAC Handbook L, pp. 143 – 144)	17
4.2.4. pH of neat formulation (CIPAC MT 75.3, CIPAC Handbook J, pp. 131 – 132)	18
4.2.5. pH of 1% dilution (CIPAC MT 75.3, CIPAC Handbook J, pp. 131 – 132)	20
4.2.6. Wet sieve test (CIPAC MT 185, CIPAC Handbook K, pp. 149-150)	22
4.2.7. Spontaneity of dispersion (CIPAC MT 160, CIPAC Handbook F, pp. 391 - 394)	23
4.2.8. Suspensibility without creaming (chemical assay) (CIPAC MT 184, CIPAC Handbook K, pp. 142-148 and CIPAC 398 (CIPAC Handbook N, pp. 134 - 144) ..	25
4.2.9. Foaming properties (CIPAC MT 47.3, CIPAC republished method)	27
5. OTHER INFORMATIONS	28
5.1. Confidentiality	28
5.2. Comments of Technical Group.	28
5.3. Accreditation	29
6. LIST OF PARTICIPATING LABORATORIES (IN ALPHABETICAL ORDER)	29
7. LIST OF ABBREVIATIONS	29
8. ACKNOWLEDGEMENTS	30



1. BASIC INFORMATION AND TIME SCHEDULE

18 laboratories registered to participate in August - September 2015. 1 laboratory registered in October 2015. One HDPE bottle containing approximately 200 ml of the test material was sent to all laboratories on 1st of October 2015 (except for 1 participant which asked to delay the sending until 2015/10/15 and the participant registered later). The samples sent on 1st October were received between 2nd and 8th of October 2015 except for 1 sample received only on 26th of October (due to customs problems) and 1 parcel returned to sender. A new sample was sent on 12th of November and received the 13th of November. The sample for the participant who asked a delay of sending was never arrived. A new one was sent on 17th of December 2015 and was received on 5th of January 2016. 18 laboratories submitted their results to meet the deadline of the 7th of December 2015. The participant who received the sample in January could not submit the results before the deadline. His results were however included in the final report. So, there are some changes in the z-score values of the statistical evaluation compared to the preliminary report (see details further).

The test material is representative of a pesticide formulation. The product analyzed in this test is a commercial Aqueous Suspension Concentrate formulation (SC) containing 312 g/l of triadimenol (nominal content).

A particular FAO specification is available for this formulation: FAO Specification for Triadimenol Aqueous Suspension 398/SC (May 2011).

Test materials are prepared in a way to keep such sample variation to a minimum as described hereunder. All materials are also tested to ensure that the degree of variation within each batch is within defined criteria. Sample variation should therefore have a negligible effect on participant performance score.

The original sample (10 L) was packaged in 10 x 1L glass bottles, in plastic bag with vermiculite granular absorbents, placed in a cardboard box filled with loose-fill chips. The samples (6) were homogenised 10' on a rotary shaker at ± 45 rpm. They have been distributed, by portion of ± 33 ml, in 30 final subsamples randomly labelled. Samples 01-2015-A-001 to 021 were sent to participants. Samples 01-2015-A-022 to 01-2015-A-030 were stored for extra sending.





Homogeneity and stability

Given the results of previous PT, the nature of the formulation and legal requirements in terms of stability of the formulations on the market, the technical group had decided not to perform the homogeneity and stability testing.

The results of the active ingredient content (usually used to perform the homogeneity and stability testing) showed no problem of homogeneity or stability during this PT (see 4.2.1.).

Parameters analysed

- Active ingredient content and diastereoisomers ratio (GLC-FID method CIPAC 398, CIPAC Handbook N, pp. 134 - 144);
- Density (CIPAC MT 3.3.2, CIPAC Handbook F, pp. 19 – 21);
- Acidity or alkalinity of formulation (CIPAC MT 191, CIPAC Handbook L, pp. 143 – 14);
- pH of neat formulation and 1% dilution (CIPAC MT 75.3, CIPAC Handbook J, pp. 131 – 132);
- Wet sieve test (CIPAC MT 185, CIPAC Handbook K, pp. 149 – 150);
- Spontaneity of dispersion (CIPAC MT 160, CIPAC Handbook F, pp. 391 - 394);
- Suspensibility (CIPAC MT 184, CIPAC Handbook K, pp. 142 – 148);
- Foaming properties (CIPAC MT 47.3, CIPAC prepublished method).

2. STATISTICAL TREATMENT AND PERFORMANCE ASSESSMENT

The organization of the PT is carried out following the procedure LAB 21 P PT 01 – Organisation des comparaisons inter-laboratoires summarized in the Manual for the Participants (LAB-21-P-PT-01-D08).

Participants were invited to report the mean value of their results and the number of independent repetitions of the assays. The assigned values were determined as the consensus of participant's results. The major advantages of consensus values are the straightforward calculation and the fact that none of the participants is accorded higher status.

The determination of the assigned values and the statistical analysis of the PT results are carried out following the procedure LAB 21 P PT-106 - Statistiques according to the ISO 13528.

All participant results are reported in tables. Results are subject to statistical assessment to help detect trends and enable comparison, using a performance indicator known as z-score.

Z-score = $(x-X)/\sigma$ where

x	=	the result reported by the participating laboratory;
X	=	the robust mean;
σ	=	the standard deviation for proficiency assessment.

The z-score compares the participant's deviation from the reference value with the robust standard deviation accepted for the proficiency test, σ .

The z-score can be interpreted as:

$ z \leq 2$: satisfactory result;
$2 < z \leq 3$: questionable result;
$ z > 3$: unsatisfactory result.



3. REMARKS/COMMENTS OF THE PARTICIPANTS

Mentioned on the Analysis Request Form

- Lab 1: Diastereoisomer ratio: 84.44 : 15.58.
Spontaneity of dispersion: gravimetric 88.71% (n = 2).
Suspensibility: gravimetric 100.0% (n = 2).
Foaming properties: no result after 10" and 3' because no asked in MT 47.3.
- Lab 2: Density: OECD Guideline 109.
- Lab 3: Density: electronic densimetry.
Acidity: + 10 ml acetone.
Wet sieve test: 0.063 mm mesh size.
Suspensibility: determination by HPLC.
- Lab 4: Diastereoisomer A to B ratio: areaA/areaB = 5.9.
Wet sieve test: no residues on the sieve.
- Lab 5: Diastereoisomer ratio: 85 : 15.
Density: oscillation-type densimeter.
A.I. content, spontaneity of dispersion, suspensibility, foaming properties: double determination in 2 different days.
- Lab 6: A.I. content: in house GLC method.
Density: digitale measurement, U-tube principle (21.4°C).
Acidity: not established at the laboratory.
pH measurements: 21.2 – 21.3°C.
- Lab 8: A.I. content: method developed and validated by the test facility and based on CIPAC method 398. The content of triadimenol is determined after dissolution of the test item in toluene containing dioctylphtalate as internal standard. The separation is achieved by Gas chromatography with flame ionisation detection (GC-FID).
Diastereoisomers ratio: 83 : 17.
- Lab 9: Acidity: it is not a requirement in case that pH is between 4 to 10.
Foaming properties: MT 47.1, 1 ml/l, 21°C.
- Lab 10: Diastereoisomer ratio: 29 : 5.
Density: We used the principle of CIPAC MT 3.3.1 Hydrometer method. The density at 20°C of a 1:1 aqueous dilution (20 g formulation + 20 g distilled water) was determined on the apparatus Lauda Tensiometer TD-1 having glass body for immersing it into the liquid. The final density of SC formulation was recalculated as described in CIPAC MT 3.3.1.
- Lab 11: A.I. content: in house GLC method.
Foaming properties : MT 47.2, 25°C.
- Lab 13: Diastereoisomer ratio: 5.93 : 1.
A.I. content: adapted method. The temperature program of injector/oven has been modified because we haven't used the recommended column. Another IS has been used (dibutylphtalate) with a RT closed to the RT of the active ingredient. The concentration (split mode) recommended in the CIPAC method (10mg/ml) is too high. A concentration of 1mg/ml has been used.
- Lab 14: Diastereoisomer ratio: 85 : 15.
Acidity: not determined as pH >4 & < 10.



Lab 16: A.I. content: Modification of proposed temperatures.
pH formulation: 20°C.
pH dilution 1%: 20.5°C.
Foaming properties: MT 47.2.

Lab 17: Suspensibility: with acetone.

Lab 18: Foaming properties: MT 47.2.

Lab 19: A.I. content: GLC-MS.
Density : Densito 30PX, Mettler Toledo.

After preliminary report receipt

Lab 5: Acidity: regarding the Acidity for Lab 5 a value of 0,01% was noted in the table of results. We reported the result as <0,01%, the exact result is 0,001%.

Lab 15: pH 1%: we repeat and we got about 6.85*. It is higher too.
Wet sieve test: we checked our mesh. We repeat the sample and use a new one and we have 0.10.

Lab 17: pH: I sent you the pH values crossed; I mean that the ph of neat formulation must be 6.77 and the pH of 1% must be 6.30 but not vice versa, as I sent you.

Lab 19: We have not send any result to 4.2.6. Wet sieve test.
We have evaluated our result of the diastereoisomers ratio 4.2.1.3.
It was calculated by the area as usual done by GC-FID analyses, but the respons is not the same for the two isomers when using GC-MS.
So it should have been calculated by the found concentrations of each isomers.
We have therefore calculated the result again with the concentrations and found the ratio: 83:17.

(*) Note of the editors: 6.80 on the corrected Analysis request form.



4. TABLES AND CHARTS OF RESULTS

4.1. Legend for tables with data from laboratories

Short form	Description
Laboratory	Code of the laboratory for this PT.
Result	Reported result of the laboratory.
Z-score	Z-score according to the ISO 13528.
Number of results	Number of laboratories reporting a result for each individual analyte.
Number of results used for the statistical evaluation	Number of results used for the statistical evaluation after elimination of results responsible for the non normality of the distribution or sent after the deadline.
Minimum value	Minimum value from all reported quantitative results.
Maximum value	Maximum value from all reported quantitative results.
Stat. minimum value	Minimum value used for the statistical evaluation after elimination of the results responsible for the non normality of the distribution or sent after the deadline.
Stat. maximum value	Maximum value used for the statistical evaluation after elimination of the results responsible for the non normality of the distribution or sent after the deadline.
Median	Median of quantitative results after elimination of the results responsible for the non normality of the distribution or sent after the deadline.
Average / Mean value	Mean of all reported quantitative results after elimination of the results responsible of the non normality of the distribution or sent after the deadline.
Robust mean	Robust mean of quantitative results calculated using the algorithm A, ISO 13528 after elimination of the results responsible for the non normality of the distribution or sent after the deadline.
Expanded uncertainty of robust mean	$2 \times (1.25 \times \text{robust standard deviation} / \text{square root of number of results})$.
Robust standard deviation	Standard deviation of quantitative results calculated using the algorithm A, ISO 13528 after elimination of the results responsible for the non normality of the distribution or sent after the deadline.



4.2. Results

Labs Number	Parameters													
	Active ingredient (g/kg)	Active ingredient (g/l)	Isomers ratio	Density	Acidity (% H ₂ SO ₄)	pH formulation	pH dilution 1%	Wet sieve test (%)	Spontaneity of Dispersion (%)	Suspensibility (%)	Foaming properties at dilution 1 ml/l			
											10 sec	1 min	3 min	12 min
1	286.82	317.17	84.4 : 15.6	1.1082	0.1300	6.62	5.98	0.00	89.6	100.6		18.0		10.0
2				1.1000			6.1				21.0	19.0	16.0	14.0
3	280.97	312.92	85.7 : 14.3	1.1137	0.0025	6.76	6.37	0.001		99.9	28.0	21.0	17.0	15.0
4	282.58	315.08	85.5 : 14.5	1.1150	0.0020	6.87	6.23	0.00	93.7	99.6	25.0	18.0	14.0	10.0
5	278.60	310.50	85 : 15	1.1147	0.0100	6.80	6.28	0.02	100	100.1	10.0	8.0	6.0	5.0
6		319.00	85.4 : 14.6	1.1128		6.58	6.17	0.03	97.1	101.6	12.0	11.0	10.0	10.0
7				1.1130	0.002	6.49	6.11	0.002			20	18	10	10
8	273.90	307.10	83 : 17	1.1209	0.0010	6.90	6.34	0.1	96.2	101.4	16.0	12.0	10.0	8.0
9	272.73	305.46	85.7 : 14.3	1.1200		6.46	6.65	0.1	98.2	97.6	6.0	4.0	2.0	1.0
10	283.93	317.71	85.3 : 14.7	1.1190	0.0020	6.87	6.26	0.00	95.2	100.4	23.0	18.0	15.0	12.0
11	290.50	321.09	85.7 : 14.3	1.1053		7.02	6.55	0.00	99.8	99.7	26.0	22.0	16.0	12.0
12	281.27	308.36	84.6 : 15.4	1.0963		6.82	6.54	0.10	97.6	99.9	34.0	20.0	12.0	6.0
13	287.24	314.81	85.6 : 14.4	1.0960	0.0080	7.08	6.69	0.00	94.8	101.6	10.0	10.0	9.0	6.0
14	281.79	313.74	85 : 15	1.1134		6.79	6.4	0.00	96.5	91.2	30.0	26.0	20.0	14.0
15	276.53	302.80	85 : 15	1.0955		7.02	7.1 (6.80)	0.22 (0.1)		99.9	44.0 (34.0)	38.0 (26.0)	26.0 (16.0)	20.0 (14.0)
16	281.87	313.13	85 : 15	1.1109	0.0024	6.82	6.05	0.0139	83.9	86.6	16.0	13.0	12.0	10.0
17	288.19	318.45	85.7 : 14.3	1.1050	0.0010	6.30 (6.77)	6.77 (6.30)	0	105.8	100.5	20.0	14.0	10.0	8.0
18	271.00	301.00		1.1110			6.15	0.015		95.6	4.0	2.0	0.0	0.0
19	279.00	311.00	80 : 20 (83 : 17)	1.1147	0.0007	6.90	6.36							

Legend :

Result with questionable z-score	Result with unsatisfactory z-score	(Corrected result)
	Other than proposed method or conditions.	Outlier according to Grubbs test.
	Discarded from statistical evaluation due to non normality of the distribution.	Out of FAO specification (general or particular).
	Results not sent or sent after deadline by the participant (not taken into account for the statistical evaluation).	

The organizer asked to the participants to communicate the number of independent repetitions they made for each test. These data are asked for information only and are not mentioned in the tables to let them readable.

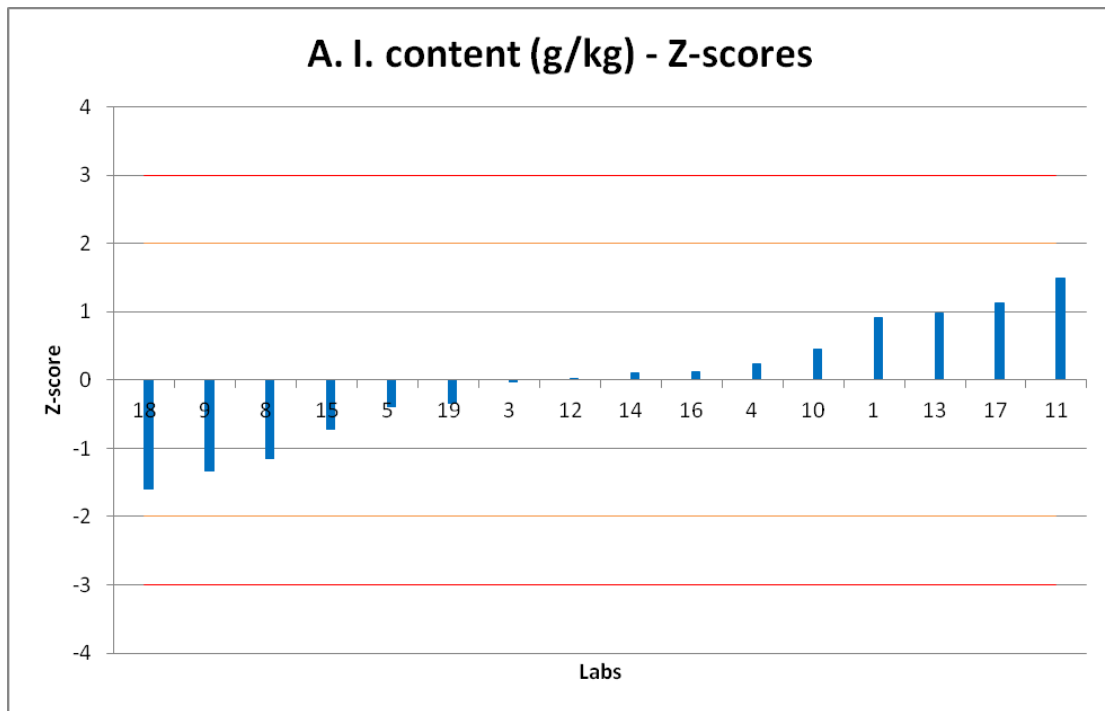


4.2.1. Triadimenol content and diastereoisomers ratio (GLC-FID method CIPAC 398, CIPAC Handbook N, pp. 134 - 144)

4.2.1.1 A.I. content in g/kg

Labs Number	Active ingredient (g/kg)	Z-score
1	286.82	0.91
2		
3	280.97	-0.02
4	282.58	0.24
5	278.60	-0.40
6		
7		
8	273.90	-1.14
9	272.73	-1.33
10	283.93	0.45
11	290.50	1.49
12	281.27	0.03
13	287.24	0.97
14	281.79	0.11
15	276.53	-0.72
16	281.87	0.12
17	288.19	1.13
18	271.00	-1.60
19	279.00	-0.33

Number of results	16
Number of results used for the statistical evaluation	16
Minimum value	271.00
maximum value	290.50
Stat. minimum value	271.00
Stat. maximum value	290.50
Median	281.53
Average	281.06
Robust mean (algorithm A, ISO 13528)	281.10
Expanded uncertainty of robust mean	3.94
Robust standard deviation (algorithm A, ISO 13528)	6.301



The Lab 19 used a MS detection. This change is considered as significant but, as his result is in the same range than those provided by the other participants, it was included in the global statistical evaluation.

All the results are statistically and analytically satisfactory.

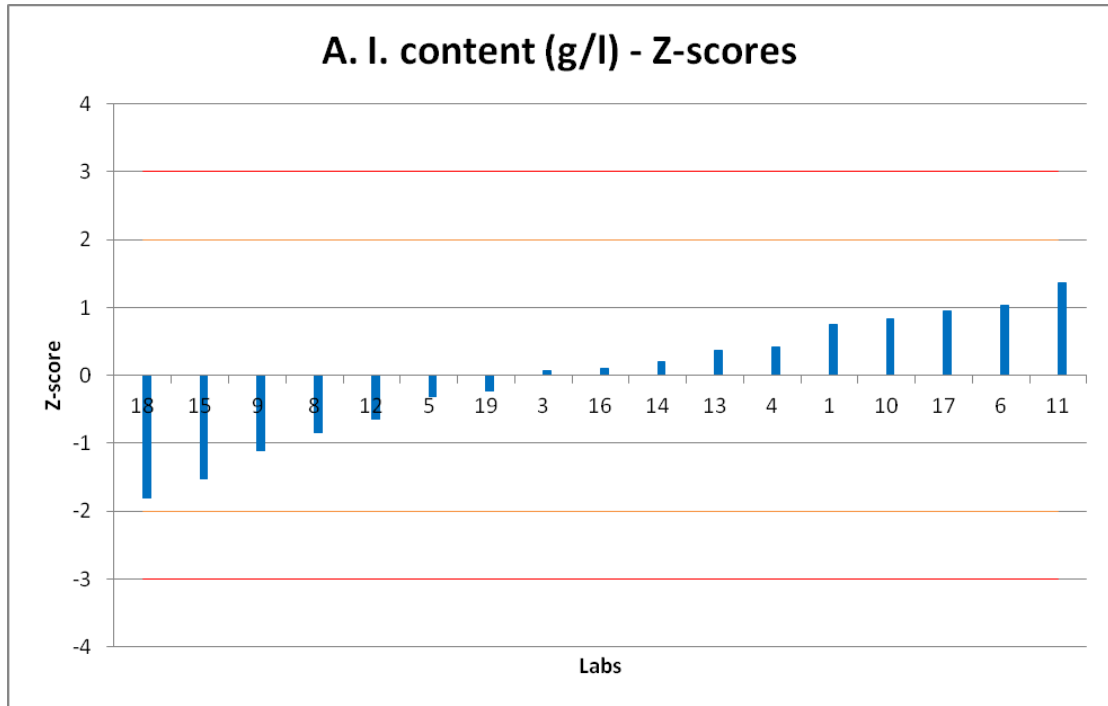
Due to some surprising replies about the number of repeat tests (until 22 repetitions for A.I. content determination), the organizer wants to reminder that **the PT samples must be analyzed in the same way that the routine samples** to give the most useful information to the participant.



4.2.1.2 A.I. content in g/l

Labs Number	Active ingredient (g/l)	Z-score
1	317.17	0.75
2		
3	312.92	0.08
4	315.08	0.42
5	310.50	-0.31
6	319.00	1.04
7		
8	307.10	-0.84
9	305.46	-1.10
10	317.71	0.83
11	321.09	1.37
12	308.36	-0.64
13	314.81	0.37
14	313.74	0.21
15	302.80	-1.52
16	313.13	0.11
17	318.45	0.95
18	301.00	-1.80
19	311.00	-0.23

Number of results	17
Number of results used for the statistical evaluation	17
Minimum value	301.00
maximum value	321.09
Stat. minimum value	301.00
Stat. maximum value	321.09
Median	313.13
Average	312.31
Robust mean (algorithm A, ISO 13528)	312.43
Expanded uncertainty of robust mean	3.84
Robust standard deviation (algorithm A, ISO 13528)	6.338



All the results are statistically and analytically satisfactory.

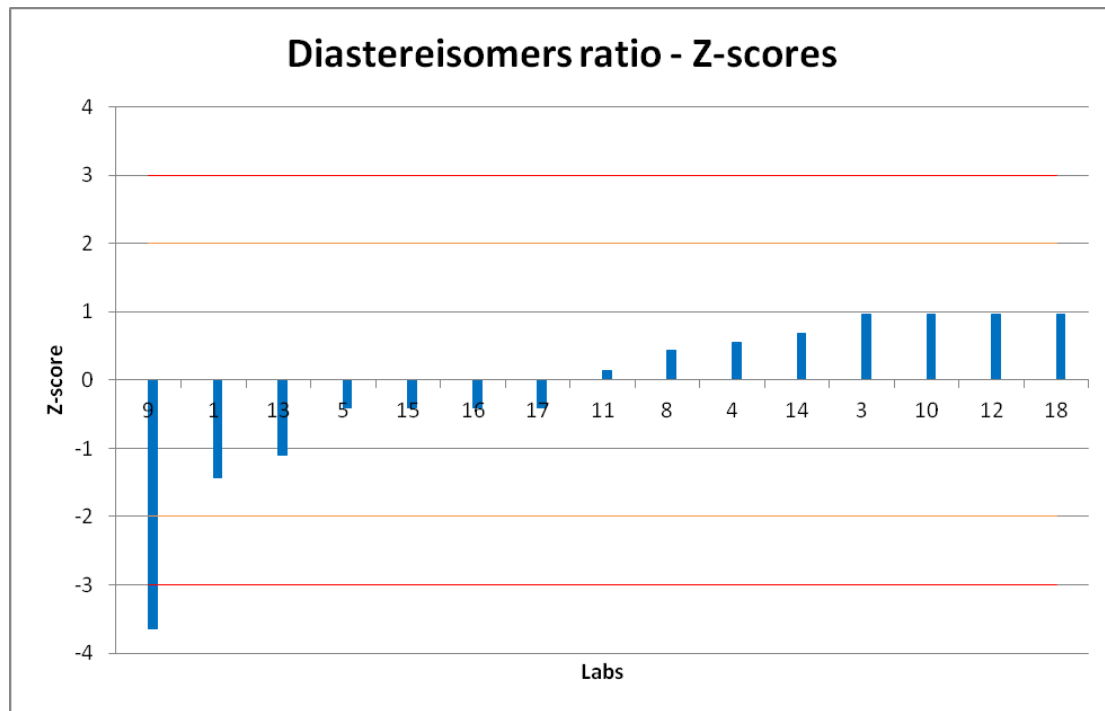


4.2.1.3 Diastereoisomers ratio

Labs Number	Diastereoisomers ratio		Z-score
1	84.4 : 15.6	5.42	-1.43
2			
3	85.7 : 14.3	6.00	0.97
4	85.5 : 14.5	5.90	0.55
5	85 : 15	5.67	-0.41
6	85.4 : 14.6	5.87	0.43
7			
8	83 : 17	4.88	-3.65*
9	85.7 : 14.3	6.00	0.97
10	85.3 : 14.7	5.80	0.14
11	85.7 : 14.3	6.00	0.97
12	84.6 : 15.4	5.50	-1.10
13	85.6 : 14.4	5.93	0.68
14	85 : 15	5.67	-0.41
15	85 : 15	5.67	-0.41
16	85 : 15	5.67	-0.41
17	85.7 : 14.3	6.00	0.97
18			
19	80 : 20* (83 : 17)	4.00	

* considered as satisfactory in practice (see hereunder).

Number of results	16
Number of results used for the statistical evaluation	15
Minimum value	4.00
maximum value	6.00
Stat. minimum value	4.88
Stat. maximum value	6.00
Median	5.80
Average	5.73
Robust mean (algorithm A, ISO 13528)	5.77
Expanded uncertainty of robust mean	0.16
Robust standard deviation (algorithm A, ISO 13528)	0.242



All the results are close to each other except for Lab 19 (using a GC-MS method). This result has been discarded from the statistical evaluation because it was pointed as outliers according to Grubbs' test. He recalculated the ratio and found 83 : 17 as Lab 8.

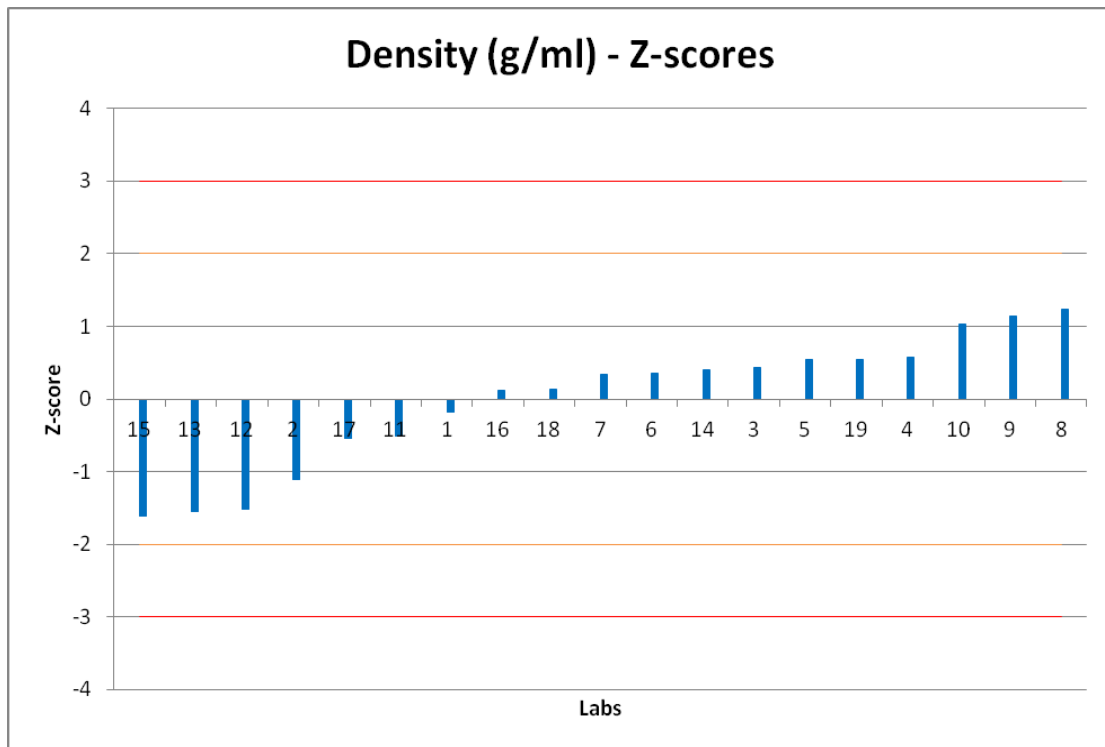
The result of Lab 8 is statistically considered as unsatisfactory.

However, in practice, the acceptable range for the isomers ratio (mean value \pm 15-20%) is larger than this statistically calculated. **So, all the results are acceptable.**

**4.2.2. Density (CIPAC MT 3.3.2, CIPAC Handbook F, pp. 19 – 21)**

Labs Number	Density (g/ml)	Z-score
1	1.1082	-0.18
2	1.1000	-1.11
3	1.1137	0.43
4	1.1150	0.58
5	1.1147	0.55
6	1.1128	0.33
7	1.1130	0.35
8	1.1209	1.24
9	1.1200	1.14
10	1.1190	1.03
11	1.1053	-0.51
12	1.0963	-1.52
13	1.0960	-1.55
14	1.1134	0.40
15	1.0955	-1.61
16	1.1109	0.12
17	1.1050	-0.54
18	1.1110	0.13
19	1.1147	0.55

Number of results	19
Number of results used for the statistical evaluation	19
Minimum value	1.0955
maximum value	1.1209
Stat. minimum value	1.0955
Stat. maximum value	1.1209
Median	1.1128
Average	1.1098
Robust mean (algorithm A, ISO 13528)	1.1098
Expanded uncertainty of robust mean	0.0051
Robust standard deviation (algorithm A, ISO 13528)	0.0089



The Labs 2, 3, 5, 6, 10 and 19 used an alternative method. Their results were in the same range than those provided by recommended method, so all of them were included in the statistical evaluation.

All the results are statistically and analytically satisfactory.



4.2.3. Acidity of formulation (CIPAC MT 191, CIPAC Handbook L, pp. 143 – 144)

Labs Number	Acidity (% H ₂ SO ₄)
1	0.1300
2	
3	0.0025
4	0.0020
5	0.0100
6	
7	0.0020
8	0.0010
9	
10	0.0020
11	
12	
13	0.0080
14	
15	
16	0.0024
17	0.0010
18	
19	0.0007

Due to the non normality of the distribution, it is not possible to evaluate the results statistically.

All the results are close to each other except for Lab 1 (highest result). No reason could be detected for this high acidity.



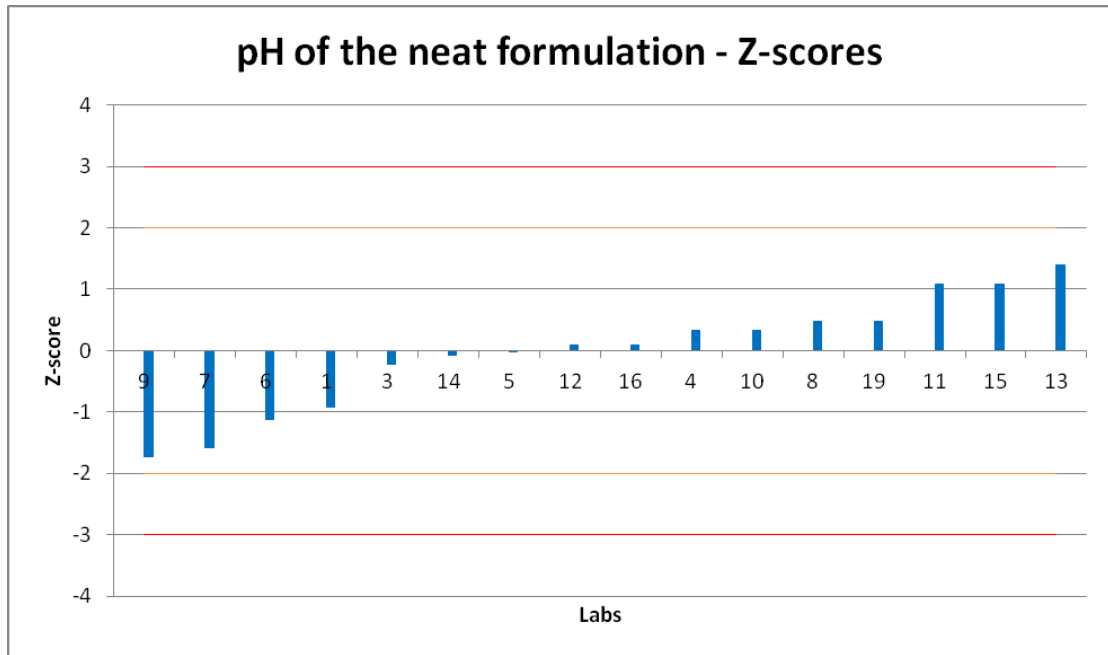
4.2.4. pH of neat formulation (CIPAC MT 75.3, CIPAC Handbook J, pp. 131 – 132)

Labs Number	pH formulation	Z-score
1	6.62	-0.92
2		
3	6.76	-0.22
4	6.87	0.33
5	6.80	-0.02
6	6.58	-1.12
7	6.49	-1.58
8	6.90	0.48
9	6.46	-1.73
10	6.87	0.33
11	7.02	1.09
12	6.82	0.08
13	7.08	1.39
14	6.79	-0.07
15	7.02	1.09
16	6.82	0.08
17	6.30* (6.77)	
18		
19	6.90	0.48

*The erroneous result of Lab 17 was discarded from the statistical evaluation not to distort it (see the participant's comment).

Note: for the Labs 9 and 15, the results of the pH of the neat formulation are lower than those of the pH of the diluted formulation.

Number of results	17
Number of results used for the statistical evaluation	16
Minimum value	6.46
maximum value	7.08
Stat. minimum value	6.46
Stat. maximum value	7.08
Median	6.82
Average	6.80
Robust mean (algorithm A, ISO 13528)	6.80
Expanded uncertainty of robust mean	0.124
Robust standard deviation (algorithm A, ISO 13528)	0.199



All the results are statistically and analytically satisfactory.



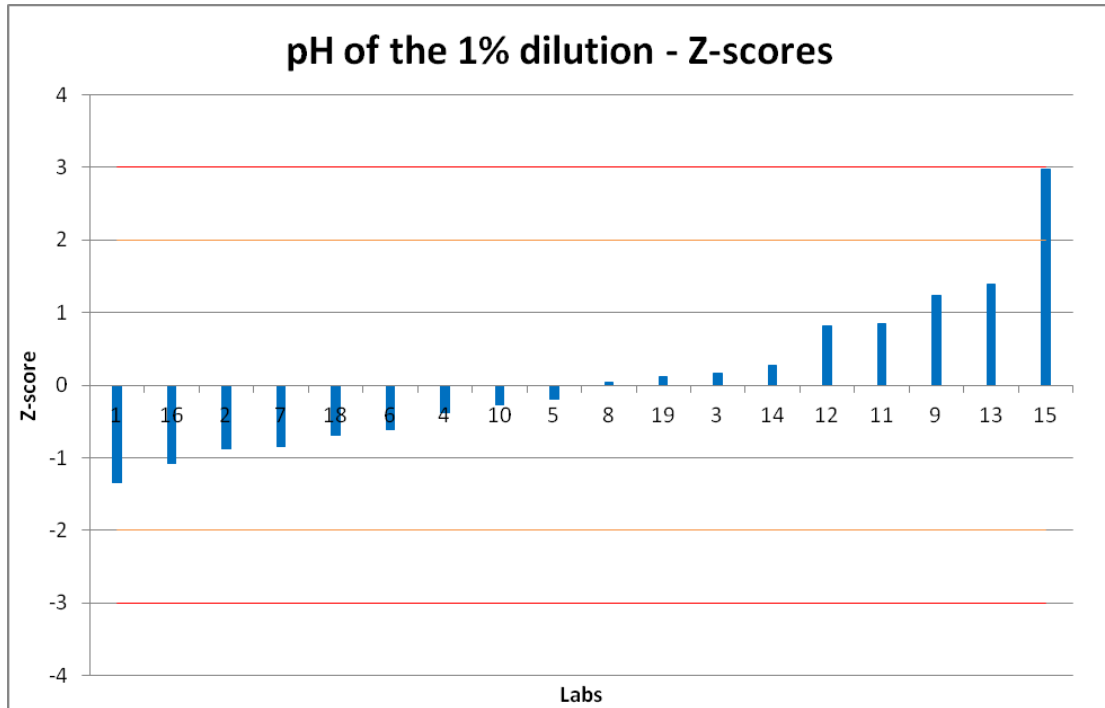
4.2.5. pH of 1% dilution (CIPAC MT 75.3, CIPAC Handbook J, pp. 131 – 132)

Labs Number	pH dilution 1%	Z-score
1	5.98	-1.34
2	6.1	-0.88
3	6.37	0.16
4	6.23	-0.38
5	6.28	-0.19
6	6.17	-0.61
7	6.11	-0.84
8	6.34	0.05
9	6.65	1.24
10	6.26	-0.26
11	6.55	0.85
12	6.54	0.82
13	6.69	1.39
14	6.4	0.28
15	7.1 (6.80)	2.97
16	6.05	-1.07
17	6.77 * (6.30)	
18	6.15	-0.69
19	6.36	0.12

*The erroneous result of Lab 17 was discarded from the statistical evaluation not to distort it (see the participant's comment).

Note: see 4.2.4.

Number of results	19
Number of results used for the statistical evaluation	18
Minimum value	5.98
maximum value	7.1
Stat. minimum value	5.98
Stat. maximum value	7.1
Median	6.31
Average	6.35
Robust mean (algorithm A, ISO 13528)	6.33
Expanded uncertainty of robust mean	0.15
Robust standard deviation (algorithm A, ISO 13528)	0.260



The result of Lab 15 is the highest and statistically considered as questionable. The participant has repeated the test and found 6.85. In practice, the difference of 0.5 pH unit from the robust mean is analytically significant.

All the other results are statistically and analytically satisfactory.

However, as previously seen in the PT's 2010 and 2012 with the same type of formulation, the range of results for the dilution (1.12 unit of pH) is almost twice larger than the range for the formulation (0.62 unit of pH). If we assume that the same pHmeter was used for the two determinations, this difference could be due to the dilution step.

The participants are encouraged to take a particular attention to this step.



4.2.6. Wet sieve test (CIPAC MT 185, CIPAC Handbook K, pp. 149-150)

Labs Number	Wet sieve test (%)
1	0.00
2	
3	0.001
4	0.00
5	0.02
6	0.03
7	0.002
8	0.10
9	0.10
10	0.00
11	0.00
12	0.10
13	0.00
14	0.00
15	0.22 (0.1)
16	0.014
17	0.00
18	0.015
19	

Due to the non normality of the distribution, it is not possible to evaluate the results statistically.

The Lab 3 used a 0.063 mm mesh size sieve (see comment).

Except of Lab 15, all the results are close to each other and considered as satisfactory.

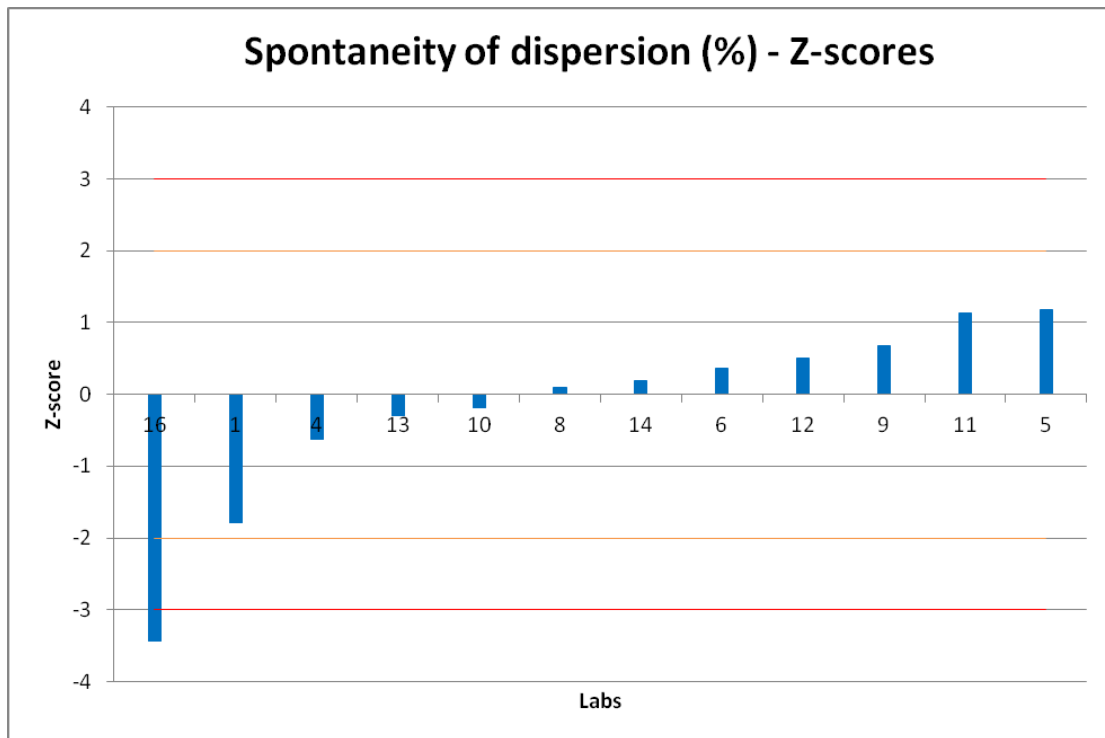
The result of Lab 15 is out of FAO particular specification (max. 0.2%). This high result may be due to the presence of impurity in the sample, in the vessel or in the tap water. The participant has repeated the test and found 0.1% (see comment).



4.2.7. Spontaneity of dispersion (CIPAC MT 160, CIPAC Handbook F, pp. 391 - 394)

Labs Number	Spontaneity of Dispersion (%)	Z-score
1	89.6	-1.79
2		
3		
4	93.7	-0.62
5	100	1.19
6	97.1	0.35
7		
8	96.2	0.10
9	98.2	0.67
10	95.2	-0.19
11	99.8	1.13
12	97.6	0.50
13	94.8	-0.30
14	96.5	0.18
15		
16	83.9	-3.43
17	105.8	
18		
19		

Number of results	13
Number of results used for the statistical evaluation	12
Minimum value	83.9
maximum value	105.8
Stat. minimum value	83.9
Stat. maximum value	100
Median	96.4
Average	95.2
Robust mean (algorithm A, ISO 13528)	95.9
Expanded uncertainty of robust mean	2.5
Robust standard deviation (algorithm A, ISO 13528)	3.49



The result of Lab 17 has been discarded from the statistical evaluation because it is not consistent according to the advice of the technical group.

The result of Lab 16 is the lowest and statistically and analytically considered as unsatisfactory. This participant found also the lowest suspensibility (see 4.2.8). This participant is invited to investigate what could be the reason.

Except those, the results are considered statistically and analytically as satisfactory.

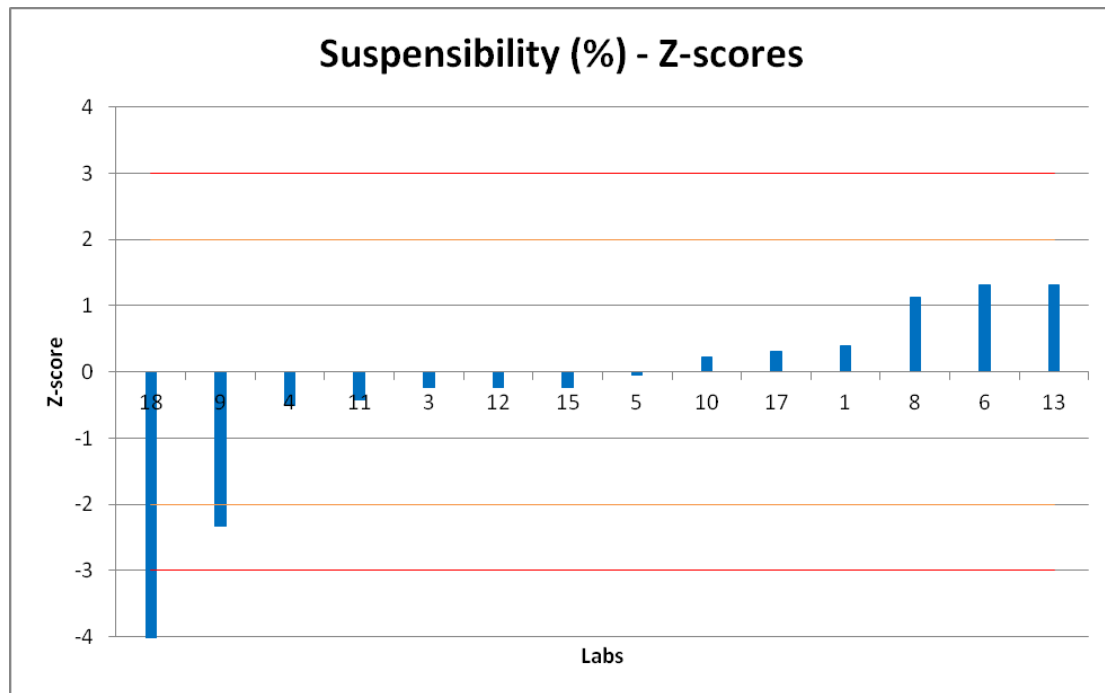


4.2.8. Suspensibility without creaming (chemical assay) (CIPAC MT 184, CIPAC Handbook K, pp. 142-148 and CIPAC 398 (CIPAC Handbook N, pp. 134 - 144)

Labs Number	Suspensibility (%)	Z-score
1	100.6	0.40
2		
3	99.9	-0.23
4	99.6	-0.51
5	100.1	-0.05
6	101.6	1.31
7		
8	101.4	1.13
9	97.6	-2.32*
10	100.4	0.22
11	99.7	-0.42
12	99.9	-0.23
13	101.6	1.31
14	91.2	
15	99.9	-0.23
16	86.6	
17	100.5	0.31
18	95.6	-4.14*
19		

*considered as satisfactory in practice (see hereunder).

Number of results	16
Number of results used for the statistical evaluation	14
Minimum value	86.6
maximum value	101.6
Stat. minimum value	95.6
Stat. maximum value	101.6
Median	100
Average	99.89
Robust mean (algorithm A, ISO 13528)	100.16
Expanded uncertainty of robust mean	0.74
Robust standard deviation (algorithm A, ISO 13528)	1.100



The results of Labs 14 and 16 have been discarded from the statistical evaluation because they are pointed as outliers according to Grubbs' test. They are responsible for the non-normality of the distribution. In practice, these results are also considered as unsatisfactory.

The Lab 3 used a HPLC method for the A.I. content determination. This change is considered as significant but, as his result is in the same range than those provided by the other participants, it was included in the global statistical evaluation.

The results of Labs 18 and 9 are the lowest and statistically considered respectively as unsatisfactory and questionable. **In practice, these results are however considered as analytically satisfactory.**

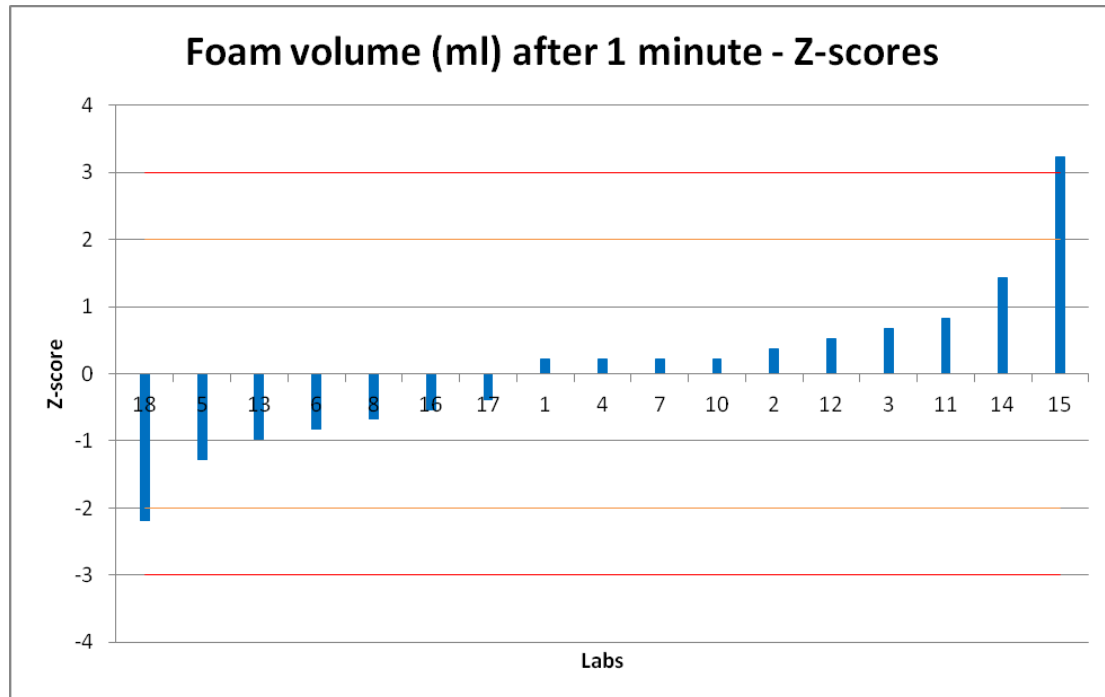
**4.2.9. Foaming properties (CIPAC MT 47.3, CIPAC prepublished method)**

Labs Number	Foaming properties at dilution 1 ml/l				Z-score (1 min)
	10 sec	1 min	3 min	12 min	
1		18.0		10.0	0.22
2	21.0	19.0	16.0	14.0	0.37
3	28.0	21.0	17.0	15.0	0.67
4	25.0	18.0	14.0	10.0	0.22
5	10.0	8.0	6.0	5.0	-1.28
6	12.0	11.0	10.0	10.0	-0.83
7	20.0	18.0	10.0	10.0	0.22
8	16.0	12.0	10.0	8.0	-0.68
9	6.0	4.0	2.0	1.0	
10	23.0	18.0	15.0	12.0	0.22
11	26.0	22.0	16.0	12.0	0.82
12	34.0	20.0	12.0	6.0	0.52
13	10.0	10.0	9.0	6.0	-0.98
14	30.0	26.0	20.0	14.0	1.42
15	44.0 (34)	38.0 (26.0)	26.0 (16.0)	20.0 (14.0)	3.23
16	16.0	13.0	12.0	10.0	-0.53
17	20.0	14.0	10.0	8.0	-0.38
18	4.0	2.0	0.0	0.0	-2.18
19					

*Comment of Lab 1: not asked in CIPAC MT 47.3.

Only the results after 1 minute have been evaluated. The z-scores are informal for this type of parameter.

Number of results	18
Number of results used for the statistical evaluation	17
Minimum value	2
maximum value	38
Stat. minimum value	2
Stat. maximum value	38
Median	18
Average	16.94
Robust mean (algorithm A, ISO 13528)	16.53
Expanded uncertainty of robust mean	4.03
Robust standard deviation (algorithm A, ISO 13528)	6.654



Almost all the labs used the same conditions except Lab 9 (MT 47.1, 100 ml, 21°C).

The results of Labs 18 and 15 are statistically considered respectively as questionable (the lowest one) and unsatisfactory (the highest one).

The Lab 15 has repeated the test and obtain lower results.

The results of Lab 18 are clearly lower than these of other participants. The participant is invited to investigate the possible reasons (dilution rate, water quality, cleanliness of vessel, ...).

5. OTHER INFORMATIONS

5.1. Confidentiality

The report will be distributed via e-mail (PDF-file) to the participants (with their own identification) and to the FASFC (with the identification of all the participants approved by the Agency).

5.2. Comments of Technical Group.

The results are in general very good. The participants are invited to have particular attention to the dilution step for pH determination of the diluted formulation and to their "unexpected" results.

The organizer wants to reminder that **the PT samples must be analyzed in the same way that the routine samples** to give the most useful information to the participant.



5.3. Accreditation

This proficiency testing schemes is accredited by BELAC, Certificate No. 014-PT. This accreditation confirms that the BU PT Schemes complies with the requirements of International Standard ISO/IEC 17043:2010.

6. LIST OF PARTICIPATING LABORATORIES (IN ALPHABETICAL ORDER)

Agence Fédérale pour la Sécurité de la Chaîne Alimentaire – Laboratoire fédéral pour la Sécurité alimentaire – LIEGE – Section Phyto/Résidus, Liège – Belgium.

Agri Sciences LTD – LAB, IZMIR – Turkey.

Agrofina S.A. – Analytical Development Laboratory of Agrofina S.A., Buenos Aires – Argentina.

Agroscope – Department of Plant Protection Chemistry, Wädenswil – Switzerland.

Austrian Agency for Health and Food Safety – Group for Contaminant and Special Analysis, Wien – Austria.

BASF SE, Limburgerhof – Germany.

Benaki Phytopatological Institute – Pesticide Control and Phytopharmacy, Kifissia Athens – Greece.

Central Controlling and Testing Institute in Agriculture – Department of Laboratory Testing, Bratislava – Slovak Republic.

Central Institute for Supervising and Testing in Agriculture – National Reference Laboratory, Department of Testing Plant Protection Products, Brno – Czech Republic.

Centre Wallon de Recherches Agronomiques (CRA-W) - Département Agriculture et Milieu Naturel - Unité Physico-chimie et Résidus des Produits Phytopharmaceutiques et des Biocides, Gembloux – Belgium.

Currenta GmbH & Co. OHG – ANT - Production Analytics, Dormagen - Germany

Danish Technological Institute – Laboratory for Chemistry and Microbiology, Aarhus – Denmark.

FARMEX S.A. – Quality, Lima – Peru.

Federal Office of Consumer Protection and Food Safety – Department of Plant Protection Products, Braunschweig – Germany.

Kmetijski inštitut Slovenije (Agricultural Institute of Slovenia) – Central Laboratory, Ljubljana – Slovenia.

Laboratorio Arbitral Agroalimentario (MAGRAMA) – Productos Fitosanitarios, Madrid – Spain.

Laboratorios Munuera SLU – Special Studies Division - GLP, San Ginés – Murcia – Spain.

National Food Chain Safety Office, Directorate of Plant Protection, Soil Conservation and Agri-environment, Pesticide Analytical Laboratory, Velence – Hungary.

National Phytosanitary Laboratory – Laboratory for Quality Control of Pesticides, Voluntari – Romania.

7. LIST OF ABBREVIATIONS

BELAC : Belgian Accreditation Body.

BU : Business Unit.



CIPAC : Collaborative International Pesticides Analytical Council.

FAFSC : Federal Agency for the Safety of the Food Chain.

FLSF : Federal Laboratory for the Safety of the Food.

FAO : Food and Agriculture Organization of the United Nations.

HDPE : High-Density polyethylene.

PT : Proficiency test.

8. ACKNOWLEDGEMENTS

We thank all the participating laboratories that trusted in us. All our gratitude goes also to the members of Technical Group and the collaborators of the FLSF-Gembloux involved in the preparation of this test.