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INTI – OAA – IAAC T 002 2008 PROFICIENCY TESTING PROGRAM

"MEAT ANALYSIS"

FINAL REPORT

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1. INTRODUCTION

Due to the market and quality systems requirements, the testing and calibration laboratories should demonstrate the quality of their measurements participating in proficiency testing (PT) exercises.

Taking into consideration such requirements the IAAC Laboratories Subcommittee is promoting the organization of proficiency testing exercises within the region.

The Argentinean Accreditation Body (OAA) and INTI (National Institute of Industrial Technology), as PT provider, are collaborating with IAAC in this activity.
Until now two exercises have been organized INTI – OAA – IAAC T 001 2006 “Water analysis” and INTI – OAA – IAAC T002 2008 “Meat analysis”.

In 2006 exercise the participant laboratories were 70 laboratories from IAAC region and 30 laboratories from APLAC (Asia – Pacific) region.
In the present exercise the participant laboratories are 53 laboratories from IAAC region, 7 laboratories from APLAC region and 10 laboratories from SADCA (South Africa) region.

2. SAMPLES

2.1. Sample preparation

- Approximately 12 kg of bovine meat was processed.
- 90 g of the meet product is canned in cylindrical containers (diameter 68 mm, height 32 mm). The cans are sealed and after that are heated in autoclave until they are "commercially sterile" it means that after that they can be store at room temperature.
  The thermal process involves heating at 115º C during 70 minutes. In this way a "Value of Lethality" is obtained, calculated in the center of the can that can guaranty that they are innocuous, larger than the required to destroy *Clostridium botulinum.*
  The "core temperature" is in average114º and the period of time that this is maintained during 60 minutes.
- The samples were incubated at 37°C for 15 days to detect any possible problem with the sterilization process. The cans were inspected to detect damages or deformations.

2.2. Reference values

To asses the performance of the participant laboratories the **consensus value**, estimated as described in **4.**, was used.
However, as additional information, in the following table are shown the values obtained by INTI (Physicochemical Laboratory. Meat Center. INTI)

Analyte	Result of analysis (g/100g)	Uncertainty (g/100g)	Method
Moisture	53,96	0,74	AOAC 950.46.B
Ash	3,01	0,13	AOAC 39.1.01
Fat	17,14	0,38	AOAC 39.1.05
Nitrogen	3,69	0,11	AOAC 39.1.15

The uncertainty values were calculated taking into account the recommendations in "Quantifying Uncertainty in Analytical Measurement" (Eurachem, Second edition, 2000).

2.3. Homogeneity

To asses the homogeneity, a representative number of samples was selected and analyzed.
The results obtained were satisfactory compared to the repeatability of the measurement method.

2.4. Stability

The samples used for this intercomparison were prepared in the same way as the samples used in previous ones.
In order to asses the stability of the samples, some of them prepared to be used in previous exercises were maintained and reanalyzed after a period of time. Until now, it has been demonstrated that the samples are stable for at least four years.

3. RESULTS REPORTED BY PARTICIPANT LABORATORIES

3.1. Results

The results reported by participant laboratories are given in Table 1(Annex 1).
The number of significant figures and units appear exactly as they were reported by the laboratories.

In graphs 1 to 4 (Annex 2) are shown the results reported by participant laboratories along with the reported uncertainty and the values obtained by INTI.
It can also be observed in these graphs, the consensus mean value and standard deviation obtained as is described in item. 4.

3.2. Test methods

Participants used the analytical method of their choice to perform the tests. A summary of the methods used by participants is shown in Table 2.

Table 3 shows a list of the accredited tests for each participant.

4. STATISTICAL PROCEDURE FOR THE ANALYSIS OF RESULTS

To estimate the consensus value and the interlaboratory standard deviation three different methods were used:

4.1. Classic Method

The first stage of this procedure consisted in screening and rejecting the obviously non valid data.
After that the statistical procedure started taking into consideration the laboratories that sent triplicate results.
The procedure essentially consisted of sequential application of the Cochran and Grubbs test, which are described in annex 3, to remove outliers. The results obtained applying this procedure are given in Table 4.

This procedure allows selecting the statistically acceptable data. This set of data was used to calculate interlaboratory mean (consensus value) and the interlaboratory standard deviation for each analyte.

4.2. Robust Method ISO 5725

The consensus value was estimated as the robust mean of the results reported by participating laboratories. The robust mean was calculated using Algorithm A as described in ISO 5725. (1994) Part 5 (ref. 1)

The robust standard deviation was estimated using Algorithm A and Algorithm S also described in ISO 5725.

4.3. Robust Method IQR

The consensus value was estimated as the median of interquartile range (IQR).
To estimate the dispersion the normalized interquartile range was used (ref. 7)

A summary of the results obtained by the different methods is given in the following table:

Analyte	Classic Method		Robust Method ISO 5725		Robust Method IQR	
(g/100g)	Mean value	Standard deviation	Mean value	Standard deviation	Mean value	Standard deviation
Moisture	54,36	0,79	54,38	0,85	54,30	0,85
Ash	3,12	0,11	3,11	0,10	3,12	0,10
Fat	17,06	1,22	16,93	1,14	16,95	1,03
Nitrogen	3,66	0,11	3,67	0,14	3,67	0,10

As it can be observed in the table the results obtained by applying the different methods are comparable.

To asses the performance of the participating laboratories we decided to use the mean value and standard desviation obtained by appliying the robust method described in ISO 5725.

The deviations between each laboratory mean value and the consensus value for each analyte can be observed on Table 5.

5. ASSESSMENT OF PERFORMANCE

The assessment of performance for the participating laboratories was realized according to international standards quoted in bibliography.
The performance criterion used was “z score”, defined as:

$$z = (x_{1/2} - x_{ref}) / s_L$$

where:

$x_{1/2}$ = average for each analyte and participant laboratory = $\sum x_i / r$

x_{ref} = consensus value for each analyte in the sample (estimated by robust method ISO 5725, as described in **4**.).

r = number of reported results for each analyte (1, 2, 3)

s_L= standard deviation (s_L was estimated by robust method ISO 5725, as described in **4**.)

The calculated "z scores" for each analyte and participant laboratory are shown in Table 6 and graphs 5 to 8.

According to the definition in annex 3, it is possible to classify scores as:

$|z| \leq 2$ satisfactory, $2 < |z| < 3$ questionable, $|z| \geq 3$ unsatisfactory

6. COMMENTS

A summary of the number of satisfactory, questionable and non satisfactory determinations evaluated by z score is shown in the following table:

	$\|Z\| \leq 2$	$2 < \|Z\| < 3$	$\|Z\| \geq 3$
Moisture	63	4	3
Ash	55	1	9
Fat	56	5	3
Nitrogen	56	1	3

- The participants submitted their results with different number of significant figures. The laboratories should be aware that this number should be in agreement with the uncertainty of that particular measurement. Some examples are Lab 9, 12, 29 and 36 in Table 1.

- In some cases the reported uncertainty seems to be incorrectly evaluated. Examples are Lab 57 and 58 in moisture determination, Lab 20, 25 and 29 in ash determination, Lab 39, 50 and 61 in nitrogen determination, etc.

ANNEX 1
TABLES

TABLE 1
Results reported by participant laboratories

Lab.	Sample	Moisture (g/100g)					Ash (g/100g)					Fat (g/100g)					Total nitrogen (g/100g)				
nº	nº	Result 1	Result 2	Result 3	Mean	U exp	Result 1	Result 2	Result 3	Mean	U exp	Result 1	Result 2	Result 3	Mean	U exp	Result 1	Result 2	Result 3	Mean	U exp
1	ni	55,1	54,82	54,65	ni	ni	ni	ni	ni	ni	ni	ni	ni	ni	ni	ni	ni	ni	ni	ni	ni
2	ni	54,67	54,68	54,43	54,59	0,016	ni	ni	ni	ni	ni	ni	ni	ni	ni	ni	ni	ni	ni	ni	ni
3	ni	53,5	53,9	54,3	53,9	0,16	3,17	3,17	3,14	3,16	0,012	17,0	16,7	16,7	16,8	0,01	3,82	3,81	3,82	3,82	0,090
5	ni	53,59	53,51	53,63	53,58	0,24	3,18	3,17	3,16	3,17	0,14	15,84	15,70	15,56	15,70	0,047	3,67	3,65	3,62	3,65	0,028
6	4	53,2	54,3	53,7	53,7	ni	3,41	3,41	3,50	3,44	ni	17,3	17,9	17,1	17,4	ni	3,70	3,75	3,75	3,73	ni
7	78	54,35	54,24	54,41	54,33	0,50	2,29	2,36	2,40	2,35	0,18	16,46	16,45	16,95	16,62	0,95	3,69	3,60	3,52	3,60	0,13
8	ni	55,49	55,70	54,40	55,20	ni	3,14	3,17	3,13	3,15	ni	18,24	18,52	18,94	18,57	ni	3,59	3,55	3,57	3,57	ni
9	ni	53,9999	53,6607	53,8320	53,8309	ni	3,1916	3,2570	3,3118	3,2535	ni	17,2808	17,2777	17,3267	17,2951	ni	3,6127	3,6279	3,5934	3,6114	0,0778
10	ni	54,64	54,78	54,7	ni	ni	3,01	2,57	2,96	ni	ni	16,61	16,85	16,62	ni	ni	3,77	3,84	3,86	ni	ni
12	ni	55,4887	55,2082	55,2187	55,3052	0,1590	3,1586	3,1338	3,1565	3,1496	0,01375	ni	ni	ni	ni	ni	3,9275	3,8701	3,8386	3,8787	0,04507
13	ni	54,1	54,0	54,2	54,1	3,0	3,1	3,1	3,1	3,1	0,5	16,0	16,3	16,6	16,3	1,5	3,5	3,5	3,4	3,5	0,3
14	ni	54,76	54,77	55,78	54,77	0,87	3,21	3,21	3,18	3,21	0,09	16,80	16,76	17,05	16,87	1,20	3,69	3,64	3,60	3,64	0,10
15	ni	53,5	53,8	54,2	53,8	0,54	3,17	3,16	3,15	3,16	0,36	17,7	17,3	17,8	17,6	0,70	3,68	3,69	3,69	3,69	0,37
16	ni	53,66	53,58	53,68	53,60	ni	3,10	3,08	3,09	3,10	ni	ni	ni	ni	ni	ni	ni	ni	ni	ni	ni
17	ni	56,3	58,7	60,8	58,6	ni	3,16	3,14	3,12	3,23	ni	16,1	17,3	16,7	16,7	ni	4,15	3,56	3,85	3,85	ni
18	ni	52,93	51,86	52,7	52,50	1,05	3,18	3,16	ni	3,17	0,10	17,20	17,81	ni	17,51	0,53	3,64	3,64	3,70	3,66	0,07
19	31	53,21	53,18	53,00	53,13	ni	3,10	3,14	3,11	3,12	ni	18,76	19,06	18,98	18,93	ni	ni	ni	ni	ni	ni
20	ni	52,8	52,5	52,7	52,7	0,22	3,0	2,9	2,9	2,9	0,03	17,4	17,4	17,1	17,3	0,22	3,9	4,0	3,8	3,9	0,09
21	ni	54,51	54,65	54,79	54,65	1,39	3,24	3,26	3,19	3,23	0,31	14,85	14,82	15,02	14,9	0,90	ni	ni	ni	ni	ni
22	37	53,74	53,62	53,98	53,78	ni	3,18	3,02	3,01	3,07	ni	19,80	19,00	19,20	19,33	ni	ni	ni	ni	ni	ni
23	ni	54,2	54,3	54,6	54,4	ni	3,14	3,14	3,20	3,16	ni	17,5	17,5	17,7	17,6	ni	3,67	3,68	3,70	3,68	ni
25	53	54,14	54,12	54,21	54,16	0,6	2,99	3,00	3,01	3,00	1,1	16,96	17,03	17,06	17,02	0,8	3,67	3,67	3,68	3,67	0,6
26	ni	54,1	54,1	54,1	54,1	1,3	3,1	3,0	3,1	3,1	0,1	17,9	17,8	17,7	17,8	0,5	3,63	3,64	3,62	3,63	0,10
27	ni	55,46	55,44	55,46	55,45	0,0184	3,02	3,00	3,02	3,01	0,0141	16,41	16,35	16,36	16,37	0,0190	3,41	3,43	3,43	3,42	0,0083
28	ni	54,28	54,30	54,40	54,32	0,092	3,59	3,65	3,46	3,57	0,126	16,60	16,46	16,57	16,54	0,102	3,46	3,46	3,47	3,46	0,058
29	ni	54,838	54,126	54,344	54,436	3,485	3,219	3,205	3,212	3,212	1,878	17,573	17,0	17,025	17,203	1,242	3,685	3,596	3,664	3,648	0,124
30	ni	54,6	54,7	54,8	54,7	0,7	3,2	3,1	3,1	3,1	0,2	16,8	16,6	16,2	16,5	1,3	3,59	3,64	3,62	3,62	0,13
31	ni	55,2	55,4	55,0	55,2	0,06	0,810	0,813	0,815	0,813	0,03	16,9	16,9	16,8	16,9	0,04	3,65	3,56	3,66	3,62	0,04
32	ni	46,70	47,38	46,48	46,85	ni	3,78	3,36	3,42	3,52	ni	17,68	17,58	18,10	17,78	ni	3,69	3,76	3,73	3,72	ni
33	ni	55,6	54,9	54,8	55,1	ni	3,44	3,48	3,48	3,46	ni	15,0	16,0	15,9	15,6	ni	3,64	3,42	3,53	3,53	ni
34	ni	53,48	53,45	53,34	53,42	0,002	3,08	3,05	3,09	3,07	0,014	20,24	19,85	20,53	20,21	0,0026	3,65	3,65	3,66	3,65	0,0093
35	ni	55,03	55,04	55,01	55,03	0,02	3,22	3,24	3,22	3,23	0,02	17,08	17,06	17,04	17,06	0,03	3,72	3,74	3,74	3,73	0,28
36	ni	53,7245	53,7245	53,6895	53,7128	0,0202	3,0859	3,0988	3,0904	3,0917	0,0066	16,6136	16,5397	16,1714	16,4416	0,2369	3,3334	3,4924	3,5537	3,4598	0,1137
37	45	55,6	55,3	56,1	55,7	0,56	3,0	3,1	3,0	3,0	0,08	18,2	16,1	17	17,1	1,46	3,6	3,6	3,5	3,6	0,08

ni: not informed

TABLE 1 (Cont)
Results reported by participant laboratories

Lab.	Sample	Moisture (g/100g)					Ash (g/100g)					Fat (g/100g)					Total nitrogen (g/100g)				
nº	nº	Result 1	Result 2	Result 3	Mean	U exp	Result 1	Result 2	Result 3	Mean	U exp	Result 1	Result 2	Result 3	Mean	U exp	Result 1	Result 2	Result 3	Mean	U exp
38	ni	64,86	67,82	63,31	65,33	0,41	3,38	3,08	3,17	3,21	0,014	14,84	14,34	ni	14,54	0,25	3,94	3,96	3,98	3,96	0,12
39	ni	54,9	54,8	49,3	53,0	1,1	3,09	3,10	3,14	3,10	0,04	18,2	18,0	16,7	17,6	0,50	3,97	4,40	4,24	4,20	6,0
40	ni	54,95	54,96	54,94	54,95	1,48	3,10	3,07	3,10	3,09	0,17	ni	ni	ni	ni	ni	3,70	3,65	3,70	3,68	0,096
41	55	53,859	54,102	53,650	53,870	0,16	2,46	2,18	2,09	2,24	0,385	21	21	21	21	0	3,21	3,20	3,23	3,213	0,098
42	ni	54,14	54,26	54,24	54,21	1,62	3,10	3,22	3,12	3,15	0,02	16,67	16,52	16,56	16,58	0,52	3,701	3,711	3,714	3,709	0,05
43	ni	55,80	56,70	56,26	56,25	ni	4,22	3,78	4,0	4,0	ni	14,06	16,14	15,10	15,10	ni	ni	ni	ni	ni	ni
44	ni	54,43	54,34	54,31	54,36	ni	3,10	3,09	3,12	3,10	ni	17,32	17,36	17,31	17,33	ni	3,69	3,72	3,68	3,70	ni
45	ni	54,27	54,30	54,27	54,28	ni	3,03	2,90	2,97	2,97	ni	17,93	23,74	17,72	19,80	ni	3,87	3,84	3,79	3,83	ni
46	ni	53,55	53,53	53,79	53,62	0,075	3,20	3,23	3,15	3,19	0,092	18,09	17,95	17,19	17,74	0,217	3,50	3,60	3,70	3,60	0,073
47	ni	54,97	55,07	55,02	55,02	ni	3,16	3,13	3,15	3,15	ni	10,68	11,15	10,64	10,82	ni	3,72	3,79	3,77	3,76	ni
49	100	56,6	56,4	56,1	56,4	0,0116	ni	ni	ni	ni	ni	14,07	14,16	14,54	14,26	0,054	ni	ni	ni	ni	ni
50	ni	54,7	58,6	54,9	56,04	4,13	3,1	2,8	3,3	3,09	0,48	15,9	16,6	16,5	16,34	0,78	3,68	2,82	3,92	3,47	1,16
51	ni	53,3	53,0	53,2	53,2	0,6	3,00	3,27	2,77	3,01	0,30	7,31	7,25	7,33	7,29	0,80	3,62	4,06	3,85	3,84	0,77
52	ni	54,3	54,3	54,3	54,3	2,1	3,18	3,18	3,17	3,18	0,12	16,6	16,6	17,0	16,7	1,3	3,73	3,73	3,70	3,72	0,19
53	ni	53,80	54,27	54,62	54,23	0,001	3,15	3,17	3,16	3,16	0,001	14,75	14,48	14,76	14,66	0,004	3,62	3,66	3,69	3,66	0,013
54	ni	55,00	55,06	54,94	55,00	0,22	3,109	3,04	3,04	3,06	0,017	18,62	19,00	20,50	19,37	0,60	0,89	0,84	0,80	0,84	0,024
55	ni	54,01	54,13	53,98	54,04	0,09	3,09	3,12	3,16	3,12	0,20	16,6	17,58	17,2	17,23	0,50	3,52	3,46	3,60	3,53	0,28
56	ni	54,28	54,32	54,39	54,33	0,1188	2,80	2,79	2,79	2,79	0,0518	16,54	16,34	16,47	16,45	0,2559	3,59	3,58	3,63	3,60	0,878
57	ni	55,38	54,29	55,15	54,94	8,42	3,00	2,96	2,89	2,95	0,34	15,81	16,50	15,81	16,04	2,39	3,61	3,55	3,50	3,55	0,74
58	62	55,1	55,0	55,1	55,1	0,20	3,17	3,15	3,12	3,15	0,15	16,2	16,1	16,4	16,2	0,4	3,65	3,68	3,69	3,67	0,1
59	ni	54,98	55,44	55,38	55,27	0,77	3,18	3,14	3,16	3,16	0,50	15,72	16,22	15,60	15,85	1,02	3,75	3,72	3,70	3,72	0,14
60	ni	53,4	53,4	53,2	53,4	0,32	3,06	3,07	3,05	3,07	0,02	18,5	18,6	18,5	18,53	0,12	3,60	3,65	3,70	3,65	0,10
61	ni	54,33	54,16	54,25	54,25	ni	3,19	3,17	3,12	3,16	ni	17,26	17,54	17,39	17,40	ni	3,52	3,53	3,56	3,54	1,44
62	ni	53,8	53,9	53,3	53,7	0,6	3,00	3,08	3,04	3,1	0,06	18,21	18,05	18,20	18,2	0,2	3,7	3,7	3,7	3,7	0,1
63	ni	55,14	ni	ni	55,14	0,83	3,18	3,18	ni	3,18	0,60	16,39	15,80	ni	16,09	0,87	3,73	ni	ni	3,73	0,16
64	ni	53,67	53,70	ni	53,69	ni	3,12	3,12	ni	3,12	ni	16,46	16,52	ni	16,49	ni	3,80	3,79	ni	3,80	ni
65	ni	54,6	54,5	54,7	54,6	0,2	3,21	3,20	3,18	3,20	0,04	16,6	16,8	16,4	16,600	0,4	3,82	3,84	3,86	3,84	0,04
66	ni	54,36	54,38	54,23	54,32	0,33	3,12	3,13	3,05	3,10	0,11	17,57	17,30	17,75	17,54	0,14	3,47	3,51	3,52	3,50	0,03475
67	ni	54,31	54,41	54,26	54,32	0,20	3,06	3,06	3,07	3,06	0,02	15,53	15,60	15,64	15,59	ni	3,68	3,73	3,76	3,72	0,20
68	ni	54,3	54,0	53,9	54,1	0,2	3,09	3,15	3,08	3,11	0,04	17,3	17,7	17,8	17,6	0,2	4,17	4,19	4,11	4,16	0,04
69	ni	54,06	53,97	53,62	53,88	0,42	3,06	3,07	3,06	3,06	0,02	17,65	17,32	17,29	17,42	0,51	3,71	3,70	3,70	3,70	0,02
70	ni	56,33	56,33	56,32	56,33	0,0301	2,96	2,98	2,99	2,98	0,0274	18,29	18,28	18,28	18,28	0,0616	3,61	3,59	3,63	3,61	0,0371
71	ni	54,64	54,40	54,54	54,53	0,20	3,03	3,13	3,05	3,07	0,06	16,66	16,29	ni	16,48	0,38	3,66	3,64	ni	3,65	0,18
72	55	54,0	53,8	54,0	53,9	0,2	ni	ni	ni	ni	ni	17,0	17,5	17,5	17,3	1,0	ni	ni	ni	ni	ni
73	57	54,70	54,76	54,68	54,72	0,26	ni	ni	ni	ni	ni	ni	ni	ni	ni	ni	ni	ni	ni	ni	ni
74	21	53,10	53,24	53,18	53,17	1,41	3,15	3,11	3,10	3,12	3,45	16,13	16,09	16,19	16,14	2,3	3,64	3,64	3,58	3,62	0,89

ni: not informed

TABLE 2
Methods reported by participant laboratories

Lab nº	Moisture	Ash	Fat	Total Nitrogen
1	Forzed air 100° C-16,5 hr	na	na	na
2	Gravimetry	na	na	na
3	AOAC 950.46 sec 39.1.02 (2005)	AOAC 920.153 sec 39.1.09 (2005)	AOAC 960.39 sec. 39.1.05 (2005)	AOAC 981.10 sec 39.1.19 (2005)
5	AOAC 950.46	AOAC 923.03	Anrom Fat analyzer	AOAC 992.15 LECO combustion method
6	AOAC 950.46	AOAC 920.153	AOAC 960.39	AOAC 981.10
7	ISO 1442:1997	ISO 936:1998	ISO 1444:1996	AOAC 928.08:2000
8	103° C +/-2°C	550° C +/- 20 ° C	Extraction with petroleum ether	Micro Kjeldahl
9	100° C until constant weight	500° C until constant weight	Extraction with petroleum ether	Kjeldahl
10	ni	ni	ni	ni
12	100 - 102° C, 16-18 hs	550° C, 3 hs	na	Micro Kjeldahl
13	105° C until constant weight	550° C until white ashes	Digestion with HCl 25:11 and extraction	DUMAS method
14	AOAC 950.46 B	AOAC 920.153	AOAC 960.30	AOAC 981.10
15	AOAC 950.46	AOAC 923.03	AOAC 960.39	AOAC 981.10
16	100° C, 3 hs	550° C	na	na
17	AOAC 950.46 B	AOAC 900.02	Extraction with petroleum ether	Kjeldahl
18	AOAC 950.46	AOAC 920.153-920.03	AOAC 960.39-920.39 C	AOAC 981.10
19	AOAC 931.04	AOAC - Gravimetry	AOAC 991.36	na
20	AOAC 950.46	AOAC 920.153	PEARSONS CHEMICAL ANALYSIS. LONGMAN SCIENTIFIC & TECHNICAL. UK 1981	AOAC 981.10
21	105° C 12 hs. AOAC 18 th	600° C 4 hs. AOAC 18 th	Extraction with petroleum ether	na
22	AOAC 950.46	AOAC 923.03	Folch modified	na
23	AOAC 950.46 B	AOAC 920.153	AOAC 960.39	AOAC 991.2
25	80 -100° C	Gravimetry	Extraction with acetone	Combustion with high purity oxigen. Reduced nitrogen gases are measured by thermal conductivity detection.
26	103° C +/- 2° C	550° C +/- 25° C	Extraction with petroleum ether	Kjeldahl
27	AOAC 950.46	AOAC 920.153	AOAC 960.39	AOAC 928.08:2000
28	103° C +/- 2° C,1 hr	500 ° C +/- 50°C 1 hr	Soxhlet	Kjeldahl
29	Gravimetry 105° C	Gravimetry 550° C	Extraction with ether	Kjeldahl
30	AOAC 950.46 B	AOAC 920.153	AOAC 960.39 B	AOAC 981.10. Kjeldahl
31	AOAC 950.46	AOAC 920.153	AOAC 991.36	AOAC 928.28
32	AOAC 950.46	AOAC 920.153	AOAC 960.39	AOAC 928.08
33	103° C +/- 2° C until constant weight	Dry. Mg Acetate.	AOAC 960.39	Kjeldahl
34	100° C, 2 hrs.	550° C with Mg acetate	Extraction	Kjeldahl
35	103° C +/- 1° C, 5 hs.	550° C +/- 4 ° C,8 hs.	Extraction with petroleum ether	Kjeldahl.
36	AOAC 950.46	AOAC 920.153	AOAC 960.39	AOAC 976.05
37	AOAC 950.46	AOAC 920.153	ISO 1444:1996	AOAC 928.08

ni: not informed na: not analyzed

TABLE 2 (Cont)
Methods reported by participant laboratories

Lab nº	Moisture	Ash	Fat	Total Nitrogen
38	AOAC 950.46	AOAC 920.154	AOAC 960.39	Kjeldahl
39	Gravimetry	AOAC 920.153	AOAC 928.08	AOAC 960.39
40	AOAC 930.15	AOAC 942.05	na	AOAC 990.03
41	125° C, 2 hrs.	ISO 936: 2006	Gerber Method	Kjeldahl.
42	100° C, 16 hrs.	100° C,16 hs and then 600° C, 4 hrs	Extraction with diethilic ether	Kjeldahl.
43	103° C +/- 2 ° C until constant weight	550° C +/- 50 ° C, 1 hr	Extraction with petroleum ether	na
44	AOAC 950.46	AOAC 920.153	AOAC 991.37	AOAC 981.10
45	AOAC 934.01	AOAC 923.03	AOAC 960.39	Combustion with oxigen and helium at 900° C
46	AOAC 950.46	AOAC 923.03	AOAC 960.39	AOAC 990.03
47	AOAC 950.46 B	AOAC 920.15	AOAC 991.36	AOAC 17 ed.
49	125° C +/- 3 ° C , 3 hs	na	Extraction	na
50	103-105° C, 12 hrs.	550° C, 12 hrs.	Extraction	Kjeldahl
51	AOAC. 16 ed. 1995	AOAC. 16 ed. 1995	AOAC. 16 ed. 1995	AOAC. 16 ed. 1995.
52	105° C +/- 5° C, 16 hs.	525° C, 5 hs	Extraction with petroleum ether	Kjeldahl
53	100-102° C, 16-18 hrs.	550 ° C, 2 hrs.	Extraction with hexane	Kjeldahl
54	ISO 1442:1997	ISO 936:1998	ISO 1443:1973	ISO 1871:1975
55	Gravimetry	Gravimetry	Extraction with ether	Kjeldahl
56	AOAC 925.10	AOAC 923.03	AOAC 920.39 C	AOAC 984.13
57	AOAC Gravimetrical	550°C Gravimetrícal	Extraction with petroleum ether	Kjeldahl
58	AOAC 950.46	AOAC 920.152	AOAC 960.39	AOAC 928.08
59	AOAC 925.10	AOAC 923.03	AOAC 991.36	AOAC 981.10
60	103° C +/- 2° C until constant weight	550° C +/- 25 ° C, 6 hrs.	Gravimetry. Acid hidrolisis. Soxhlet	Kjeldahl.
61	AOAC 950.46 B	AOAC 920.153	AOAC 960.39	AOAC 981.10
62	AOAC 950.46	-	AOAC 960.39	Kjeldahl
63	Gravimetry	550° C	Extraction with petroleum ether	Kjeldahl
64	103° C +/- 2 ° C, 5 hrs	Gravimetry	Extraction with petroleum ether	Kjeldahl
65	103° C +/- 2° C until constant weight	AOAC 930.22	AOAC 960.39.	ISO 5983
66	AOAC 950.46 B.	AOAC 920.153	AOAC 991.36	AOAC 981.10
67	AACC 44-15 A	AACC 08-01	AACC 30-26	AACC 46-12
68	AOAC 950.46 B	AOAC 920.153	AOAC 928.08	AOAC 960.39
69	AOAC 950.46 B	AOAC 920.153	AOAC 960.39	Kjeldahl
70	AOAC 950.46 B	AOAC 923.03	AOAC 960.39	AOAC 981.10
71	ni	ni	ni	ni
72	125° C +/-0.2 ° C, 2 hs	na	Gerber Method	na
73	100° C +/- 2° C	na	na	na
74	AOAC 934.01	AOAC 942.05	Soxtec 2045	AOAC 988.05

ni: not informed na: not analyzed

TABLE 3
Accredited Tests

Lab. nº	Is the Laboratory accredited for Tests?			
	Moisture	**Ash**	**Fat**	**Total nitrogen**
1	YES	na	na	na
2	NO	NO	na	na
3	YES	YES	YES	YES
5	NO	NO	NO	NO
6	NO	NO	NO	NO
7	ni	ni	na	ni
8	NO	NO	NO	NO
9	NO	NO	NO	NO
10	YES	YES	YES	YES
12	NO	NO	NO	NO
13	NO	NO	NO	NO
14	YES	YES	YES	YES
15	YES	YES	YES	YES
16	NO	NO	na	na
17	NO	NO	NO	NO
18	YES	YES	YES	YES
19	NO	NO	NO	NO
20	NO	NO	NO	NO
21	YES	YES	YES	na
22	NO	NO	NO	NO
23	NO	NO	NO	NO
25	YES	YES	YES	YES
26	YES	YES	YES	YES
27	NO	NO	NO	NO
28	NO	NO	NO	NO
29	NO	NO	NO	NO
30	YES	YES	YES	YES
31	NO	NO	NO	NO
32	NO	NO	NO	NO
33	NO	NO	NO	NO
34	YES	YES	YES	YES
35	YES	YES	YES	YES
36	NO	NO	NO	NO
37	NO	NO	NO	NO

ni: not informed na: not analyzed

TABLE 3 (Cont.)
Accredited Tests

Lab. nº	Is the Laboratory accredited for Tests?			
	Moisture	**Ash**	**Fat**	**Total nitrogen**
38	YES	YES	YES	YES
39	NO	NO	NO	YES
40	YES	YES	na	YES
41	NO	NO	NO	NO
42	YES	YES	YES	YES
43	NO	NO	NO	na
44	YES	YES	YES	YES
45	NO	NO	NO	NO
46	YES	YES	YES	YES
47	YES	YES	YES	YES
48	NO	NO	NO	NO
49	NO	na	NO	na
50	NO	NO	NO	NO
51	NO	NO	NO	NO
52	YES	YES	YES	YES
53	NO	NO	NO	NO
54	NO	NO	NO	NO
55	YES	YES	YES	YES
56	YES	YES	YES	YES
57	ni	ni	ni	ni
58	NO	NO	NO	NO
59	YES	YES	YES	YES
60	NO	NO	NO	NO
61	na	NO	na	YES
62	YES	YES	YES	YES
63	YES	YES	YES	YES
64	NO	NO	NO	NO
65	NO	NO	NO	NO
66	YES	YES	YES	YES
67	YES	YES	YES	YES
68	NO	NO	NO	NO
69	YES	YES	YES	YES
70	NO	NO	NO	NO
71	YES	YES	YES	YES
72	NO	NO	NO	na
73	YES	NO	NO	NO
74	NO	NO	NO	NO

ni: not informed na: not analyzed

TABLE 4
Results obtained applying the statistical procedure

Lab. nº	Moisture (g/100g)				Ash (g/100g)				Fat (g/100g)				Total nitrogen (g/100g)			
	Result 1	Result 2	Result 3	T	Result 1	Result 2	Result 3	T	Result 1	Result 2	Result 3	T	Result 1	Result 2	Result 3	T
1	55,1	54,82	54,65		ni	ni	ni		ni	ni	ni		ni	ni	ni	
2	54,67	54,68	54,43		ni	ni	ni		ni	ni	ni		ni	ni	ni	
3	53,5	53,9	54,3		3,17	3,17	3,14		17,0	16,7	16,7		3,82	3,81	3,82	
5	53,59	53,51	53,63		3,18	3,17	3,16		15,84	15,70	15,56		3,67	3,65	3,62	
6	53,2	54,3	53,7		3,41	3,41	3,50		17,3	17,9	17,1		3,70	3,75	3,75	
7	54,35	54,24	54,41		2,29	2,36	2,40	G	16,46	16,45	16,95		3,69	3,60	3,52	
8	55,49	55,70	54,40	C	3,14	3,17	3,13		18,24	18,52	18,94		3,59	3,55	3,57	
9	53,9999	53,6607	53,8320		3,1916	3,2570	3,3118		17,2808	17,2777	17,3267		3,6127	3,6279	3,5934	
10	54,64	54,78	54,7		3,01	2,57	2,96	C	16,61	16,85	16,62		3,77	3,84	3,86	
12	55,4887	55,2082	55,2187		3,1586	3,1338	3,1565		ni	ni	ni		3,9275	3,8701	3,8386	
13	54,1	54,0	54,2		3,1	3,1	3,1		16,0	16,3	16,6		3,5	3,5	3,4	
14	54,76	54,77	55,78		3,21	3,21	3,18		16,80	16,76	17,05		3,69	3,64	3,60	
15	53,5	53,8	54,2		3,17	3,16	3,15		17,7	17,3	17,8		3,68	3,69	3,69	
16	53,66	53,58	53,68		3,10	3,08	3,09		ni	ni	ni		ni	ni	ni	
17	56,3	58,7	60,8	C	3,16	3,14	3,12		16,1	17,3	16,7		4,15	3,56	3,85	C
18	52,93	51,86	52,7		3,18	3,16	ni	<3	17,20	17,81	ni	<3	3,64	3,64	3,70	
19	53,21	53,18	53,00		3,10	3,14	3,11		18,76	19,06	18,98		ni	ni	ni	
20	52,8	52,5	52,7		3,0	2,9	2,9		17,4	17,4	17,1		3,9	4,0	3,8	
21	54,51	54,65	54,79		3,24	3,26	3,19		14,85	14,82	15,02		ni	ni	ni	
22	53,74	53,62	53,98		3,18	3,02	3,01	C	19,80	19,00	19,20		ni	ni	ni	
23	54,2	54,3	54,6		3,14	3,14	3,20		17,5	17,5	17,7		3,67	3,68	3,70	
25	54,14	54,12	54,21		2,99	3,00	3,01		16,96	17,03	17,06		3,67	3,67	3,68	
26	54,1	54,1	54,1		3,1	3,0	3,1		17,9	17,8	17,7		3,63	3,64	3,62	
27	55,46	55,44	55,46		3,02	3,00	3,02		16,41	16,35	16,36		3,41	3,43	3,43	
28	54,28	54,30	54,40		3,59	3,65	3,46	C	16,60	16,46	16,57		3,46	3,46	3,47	
29	54,838	54,126	54,344		3,219	3,205	3,212		17,573	17,0	17,025		3,685	3,596	3,664	
30	54,6	54,7	54,8		3,2	3,1	3,1		16,8	16,6	16,2		3,59	3,64	3,62	
31	55,2	55,4	55,0		0,810	0,813	0,815	G	16,9	16,9	16,8		3,65	3,56	3,66	
32	46,70	47,38	46,48	G	3,78	3,36	3,42	C	17,68	17,58	18,10		3,69	3,76	3,73	
33	55,6	54,9	54,8		3,44	3,48	3,48		15,0	16,0	15,9		3,64	3,42	3,53	
34	53,48	53,45	53,34		3,08	3,05	3,09		20,24	19,85	20,53		3,65	3,65	3,66	
35	55,03	55,04	55,01		3,22	3,24	3,22		17,08	17,06	17,04		3,72	3,74	3,74	
36	53,7245	53,7245	53,6895		3,0859	3,0988	3,0904		16,6136	16,5397	16,1714		3,3334	3,4924	3,5537	
37	55,6	55,3	56,1		3,0	3,1	3,0		18,2	16,1	17	C	3,6	3,6	3,5	

n.i.: not informed
T: results obtained applying the statistical procedure
C: result rejected applying Cochran test
G: result rejected applying Grubbs test.
< 3: not triplicate results
I: result rejected at the initial screening

TABLE 4 (Cont)
Results obtained applying the statistical procedure

Lab.	Moisture (g/100g)				Ash (g/100g)				Fat (g/100g)				Total nitrogen (g/100g)			
nº	Result 1	Result 2	Result 3	T	Result 1	Result 2	Result 3	T	Result 1	Result 2	Result 3	T	Result 1	Result 2	Result 3	T
38	64,86	67,82	63,31	C	3,38	3,08	3,17	C	14,84	14,34	ni	<3	3,94	3,96	3,98	
39	54,9	54,8	49,3	C	3,09	3,10	3,14		18,2	18,0	16,7	C	3,97	4,40	4,24	C
40	54,95	54,96	54,94		3,10	3,07	3,10		ni	ni	ni		3,70	3,65	3,70	
41	53,859	54,102	53,650		2,46	2,18	2,09	C	21	21	21		3,21	3,20	3,23	G
42	54,14	54,26	54,24		3,10	3,22	3,12		16,67	16,52	16,56		3,701	3,711	3,714	
43	55,80	56,70	56,26		4,22	3,78	4,0	C	14,06	16,14	15,10	C	ni	ni	ni	
44	54,43	54,34	54,31		3,10	3,09	3,12		17,32	17,36	17,31		3,69	3,72	3,68	
45	54,27	54,30	54,27		3,03	2,90	2,97		17,93	23,74	17,72	C	3,87	3,84	3,79	
46	53,55	53,53	53,79		3,20	3,23	3,15		18,09	17,95	17,19		3,50	3,60	3,70	
47	54,97	55,07	55,02		3,16	3,13	3,15		10,68	11,15	10,64	G	3,72	3,79	3,77	
49	56,6	56,4	56,1		ni	ni	ni		14,07	14,16	14,54		ni	ni	ni	
50	54,7	58,6	54,9	C	3,1	2,8	3,3	C	15,9	16,6	16,5		3,68	2,82	3,92	C
51	53,3	53,0	53,2		3,00	3,27	2,77	C	7,31	7,25	7,33	G	3,62	4,06	3,85	C
52	54,3	54,3	54,3		3,18	3,18	3,17		16,6	16,6	17,0		3,73	3,73	3,70	
53	53,80	54,27	54,62		3,15	3,17	3,16		14,75	14,48	14,76		3,62	3,66	3,69	
54	55,00	55,06	54,94		3,109	3,04	3,04		18,62	19,00	20,50	C	0,89	0,84	0,80	G
55	54,01	54,13	53,98		3,09	3,12	3,16		16,6	17,58	17,2		3,52	3,46	3,60	
56	54,28	54,32	54,39		2,80	2,79	2,79		16,54	16,34	16,47		3,59	3,58	3,63	
57	55,38	54,29	55,15		3,00	2,96	2,89		15,81	16,50	15,81		3,61	3,55	3,50	
58	55,1	55,0	55,1		3,17	3,15	3,12		16,2	16,1	16,4		3,65	3,68	3,69	
59	54,98	55,44	55,38		3,18	3,14	3,16		15,72	16,22	15,60		3,75	3,72	3,70	
60	53,4	53,4	53,2		3,06	3,07	3,05		18,5	18,6	18,5		3,60	3,65	3,70	
61	54,33	54,16	54,25		3,19	3,17	3,12		17,26	17,54	17,39		3,52	3,53	3,56	
62	53,8	53,9	53,3		3,00	3,08	3,04		18,21	18,05	18,20		3,7	3,7	3,7	
63	55,14	ni	ni	<3	3,18	3,18	ni	<3	16,39	15,80	ni	<3	3,73	ni	ni	<3
64	53,67	53,70	ni	<3	3,12	3,12	ni	<3	16,46	16,52	ni	<3	3,80	3,79	ni	<3
65	54,6	54,5	54,7		3,21	3,20	3,18		16,6	16,8	16,4		3,82	3,84	3,86	
66	54,36	54,38	54,23		3,12	3,13	3,05		17,57	17,30	17,75		3,47	3,51	3,52	
67	54,31	54,41	54,26		3,06	3,06	3,07		15,53	15,60	15,64		3,68	3,73	3,76	
68	54,3	54,0	53,9		3,09	3,15	3,08		17,3	17,7	17,8		4,17	4,19	4,11	G
69	54,06	53,97	53,62		3,06	3,07	3,06		17,65	17,32	17,29		3,71	3,70	3,70	
70	56,33	56,33	56,32		2,96	2,98	2,99		18,29	18,28	18,28		3,61	3,59	3,63	
71	54,64	54,40	54,54		3,03	3,13	3,05		16,66	16,29	ni	<3	3,66	3,64	ni	<3
72	54,0	53,8	54,0		ni	ni	ni		17,0	17,5	17,5		ni	ni	ni	
73	54,70	54,76	54,68		ni	ni	ni		ni	ni	ni		ni	ni	ni	
74	53,10	53,24	53,18		3,15	3,11	3,10		16,13	16,09	16,19		3,64	3,64	3,58	

n.i.: not informed
T: results obtained applying the statistical procedure
C: result rejected applying Cochran test
G: result rejected applying Grubbs test.
< 3: not triplicate results
I: result rejected at the initial screening

TABLE 5
Relative deviation between each laboratory mean value and the consensus value

Lab. nº	Moisture (g/100g)		Ash (g/100g)		Fat (g/100g)		Total nitrogen (g/100g)	
	Mean value	% dev. consensus	Mean value	% dev. consensus	Mean value	% dev. consensus	Mean value	% dev. consensus
1	ni	-	ni	-	ni	-	ni	-
2	54,59	0,42	ni	-	ni	-	ni	-
3	53,90	-0,85	3,16	1,28	16,80	-1,52	3,82	4,37
5	53,58	-1,43	3,17	1,60	15,70	-7,97	3,65	-0,27
6	53,70	-1,21	3,44	10,26	17,40	1,99	3,73	1,91
7	54,33	-0,06	2,35	-24,68	16,62	-2,58	3,60	-1,64
8	55,20	1,55	3,15	0,96	18,57	8,85	3,57	-2,46
9	53,83	-0,97	3,25	4,28	17,30	1,38	3,61	-1,33
10	ni	-	ni	-	ni	-	ni	-
12	55,31	1,74	3,15	0,95	ni	-	3,88	5,98
13	54,10	-0,48	3,10	-0,64	16,30	-4,45	3,50	-4,37
14	54,77	0,75	3,21	2,88	16,87	-1,11	3,64	-0,55
15	53,80	-1,03	3,16	1,28	17,60	3,17	3,69	0,82
16	53,60	-1,40	3,10	-0,64	ni	-	ni	-
17	58,60	7,80	3,23	3,53	16,70	-2,11	3,85	5,19
18	52,50	-3,42	3,17	1,60	17,51	2,64	3,66	0,00
19	53,13	-2,26	3,12	0,00	18,93	10,96	ni	-
20	52,70	-3,05	2,90	-7,05	17,30	1,41	3,90	6,56
21	54,65	0,53	3,23	3,53	14,90	-12,66	ni	-
22	53,78	-1,07	3,07	-1,60	19,33	13,31	ni	-
23	54,40	0,07	3,16	1,28	17,60	3,17	3,68	0,55
25	54,16	-0,37	3,00	-3,85	17,02	-0,23	3,67	0,27
26	54,10	-0,48	3,10	-0,64	17,80	4,34	3,63	-0,82
27	55,45	2,01	3,01	-3,53	16,37	-4,04	3,42	-6,56
28	54,32	-0,07	3,57	14,42	16,54	-3,05	3,46	-5,46
29	54,44	0,14	3,21	2,95	17,20	0,84	3,65	-0,33
30	54,70	0,63	3,10	-0,64	16,50	-3,28	3,62	-1,09
31	55,20	1,55	0,81	-73,94	16,90	-0,94	3,62	-1,09
32	46,85	-13,82	3,52	12,82	17,78	4,22	3,72	1,64
33	55,10	1,36	3,46	10,90	15,60	-8,56	3,53	-3,55
34	53,42	-1,73	3,07	-1,60	20,21	18,46	3,65	-0,27
35	55,03	1,23	3,23	3,53	17,06	0,00	3,73	1,91
36	53,71	-1,19	3,09	-0,91	16,44	-3,62	3,46	-5,47
37	55,70	2,47	3,00	-3,85	17,10	0,23	3,60	-1,64

ni: not informed

TABLE 5 (Cont)
Relative deviation between each laboratory mean value and the consensus value

Lab. nº	Moisture (g/100g)		Ash (g/100g)		Fat (g/100g)		Total nitrogen (g/100g)	
	Mean value	% dev. consensus	Mean value	% dev. consensus	Mean value	% dev. consensus	Mean value	% dev. consensus
38	65,33	20,18	3,21	2,88	14,54	-14,77	3,96	8,20
39	53,00	-2,50	3,10	-0,64	17,60	3,17	4,20	14,75
40	54,95	1,09	3,09	-0,96	ni	-	3,68	0,55
41	53,87	-0,90	2,24	-28,21	21,00	23,09	3,21	-12,21
42	54,21	-0,28	3,15	0,96	16,58	-2,81	3,71	1,34
43	56,25	3,48	4,00	28,21	15,10	-11,49	ni	-
44	54,36	0,00	3,10	-0,64	17,33	1,58	3,70	1,09
45	54,28	-0,15	2,97	-4,81	19,80	16,06	3,83	4,64
46	53,62	-1,36	3,19	2,24	17,74	3,99	3,60	-1,64
47	55,02	1,21	3,15	0,96	10,82	-36,58	3,76	2,73
49	56,40	3,75	ni	-	14,26	-16,41	ni	-
50	56,04	3,09	3,09	-0,96	16,34	-4,22	3,47	-5,19
51	53,20	-2,13	3,01	-3,53	7,29	-57,27	3,84	4,92
52	54,30	-0,11	3,18	1,92	16,70	-2,11	3,72	1,64
53	54,23	-0,24	3,16	1,28	14,66	-14,07	3,66	0,00
54	55,00	1,18	3,06	-1,92	19,37	13,54	0,84	-77,05
55	54,04	-0,59	3,12	0,00	17,23	1,00	3,53	-3,55
56	54,33	-0,06	2,79	-10,58	16,45	-3,58	3,60	-1,64
57	54,94	1,07	2,95	-5,45	16,04	-5,98	3,55	-3,01
58	55,10	1,36	3,15	0,96	16,20	-5,04	3,67	0,27
59	55,27	1,67	3,16	1,28	15,85	-7,09	3,72	1,64
60	53,40	-1,77	3,07	-1,60	18,53	8,62	3,65	-0,27
61	54,25	-0,20	3,16	1,28	17,40	1,99	3,54	-3,28
62	53,70	-1,21	3,10	-0,64	18,20	6,68	3,70	1,09
63	55,14	1,43	3,18	1,92	16,09	-5,69	3,73	1,91
64	53,69	-1,23	3,12	0,00	16,49	-3,34	3,80	3,83
65	54,60	0,44	3,20	2,56	16,60	-2,70	3,84	4,92
66	54,32	-0,07	3,10	-0,64	17,54	2,81	3,50	-4,37
67	54,32	-0,07	3,06	-1,92	15,59	-8,62	3,72	1,64
68	54,10	-0,48	3,11	-0,32	17,60	3,17	4,16	13,66
69	53,88	-0,88	3,06	-1,92	17,42	2,11	3,70	1,09
70	56,33	3,62	2,98	-4,49	18,28	7,15	3,61	-1,37
71	54,53	0,31	3,07	-1,60	16,48	-3,40	3,65	-0,27
72	53,90	-0,85	ni	-	17,30	1,41	ni	-
73	54,72	0,66	ni	-	ni	-	ni	-
74	53,17	-2,19	3,12	0,00	16,14	-5,39	3,62	-1,09

ni: not informed

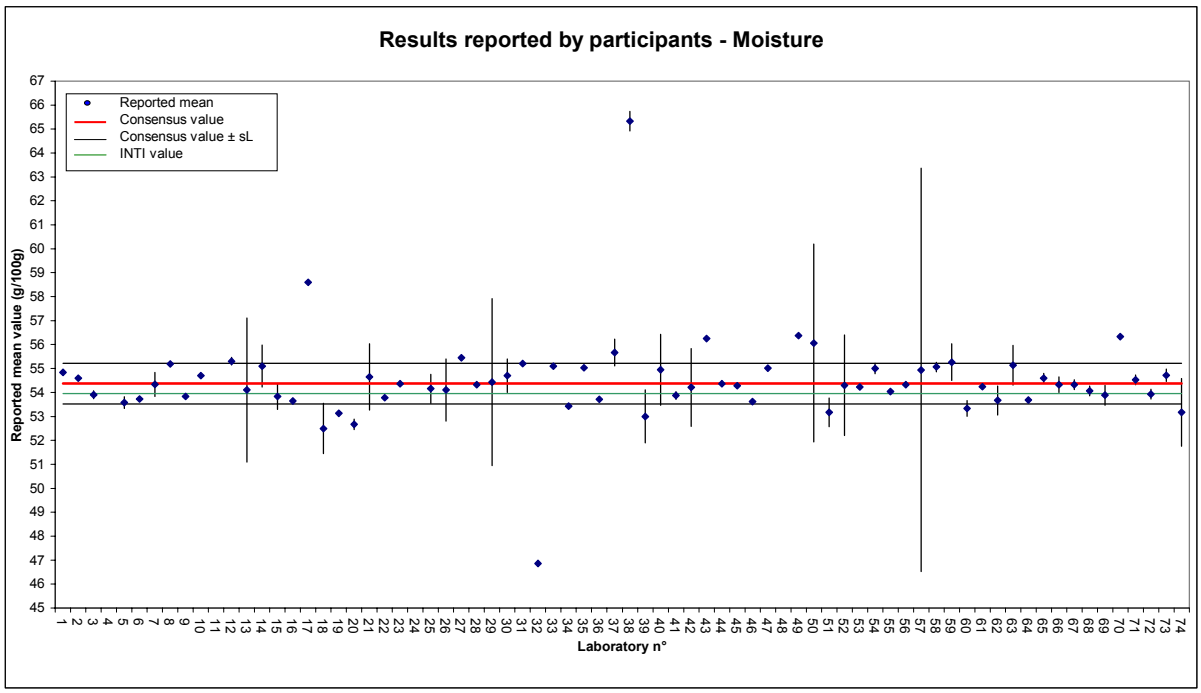
TABLE 6
Z Score

Lab. nº	Moisture	Ash	Fat	Nitrogen
1	0,55	-	-	-
2	0,26	-	-	-
3	-0,56	0,48	-0,11	1,10
5	-0,94	0,58	-1,07	-0,14
6	-0,76	3,35	0,44	0,49
7	-0,05	-7,82	-0,27	-0,46
8	0,97	0,34	1,44	-0,71
9	-0,64	1,44	0,32	-0,40
10	0,39	-2,73	-0,20	1,15
12	1,10	0,37	-	1,55
13	-0,33	-0,14	-0,55	-1,46
14	0,86	0,89	-0,05	-0,17
15	-0,64	0,48	0,59	0,15
16	-0,87	-0,24	-	-
17	4,99	0,27	-0,20	1,37
18	-2,22	0,58	0,51	-0,05
19	-1,47	0,04	1,76	-
20	-2,02	-1,84	0,33	1,71
21	0,32	1,20	-1,78	-
22	-0,70	-0,44	2,11	-
23	-0,01	0,48	0,56	0,12
25	-0,26	-1,16	0,08	0,05
26	-0,33	-0,48	0,76	-0,27
27	1,27	-1,02	-0,48	-1,78
28	-0,06	4,65	-0,34	-1,49
29	0,07	1,01	0,24	-0,13
30	0,38	0,21	-0,34	-0,36
31	0,97	-23,58	-0,05	-0,31
32	-8,88	4,17	0,75	0,44
33	0,85	3,62	-1,13	-1,00
34	-1,12	-0,41	2,87	-0,10
35	0,77	1,16	0,12	0,49
36	-0,78	-0,22	-0,43	-1,51
37	1,52	-0,82	0,15	-0,73
38	12,93	0,99	-2,05	2,15

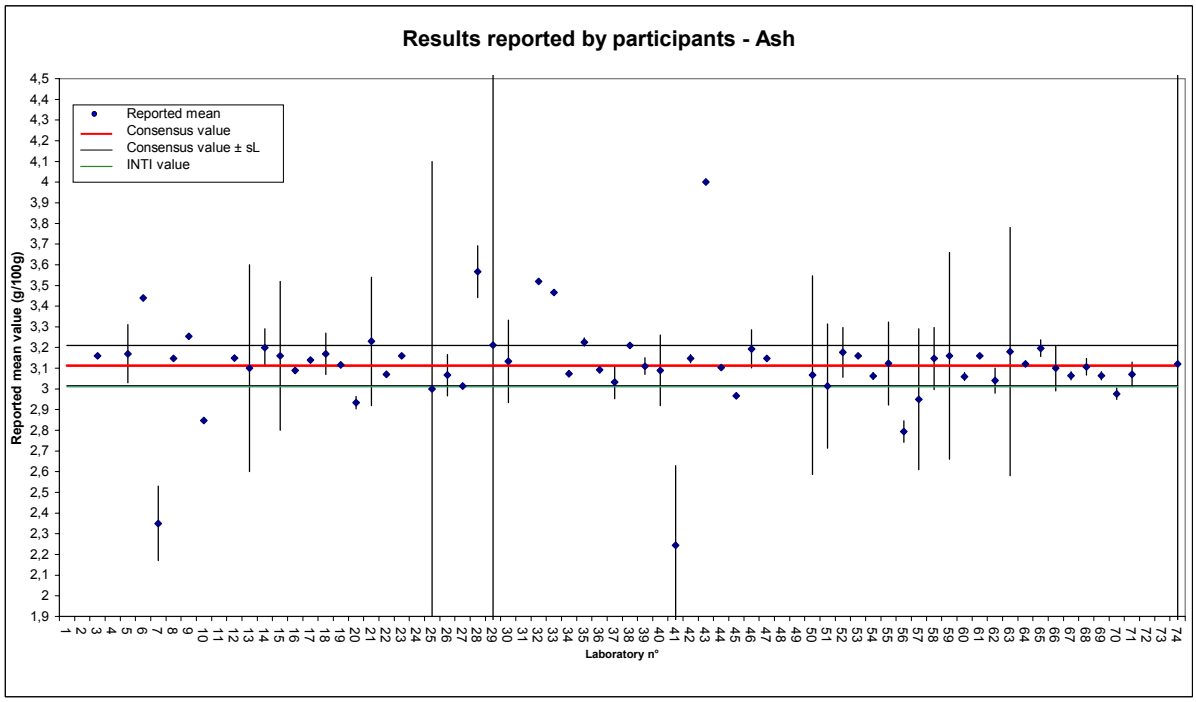
Lab. nº	Moisture	Ash	Fat	Nitrogen
39	-1,62	-0,03	0,62	3,93
40	0,68	-0,24	-	0,12
41	-0,60	-8,92	3,57	-3,32
42	-0,19	0,34	-0,30	0,31
43	2,22	9,09	-1,60	-
44	-0,02	-0,10	0,35	0,22
45	-0,11	-1,50	2,51	1,22
46	-0,89	0,82	0,71	-0,49
47	0,76	0,34	-5,34	0,69
49	2,35	-	-2,34	-
50	2,00	-0,48	-0,52	-1,41
51	-1,43	-1,02	-8,43	1,30
52	-0,09	0,65	-0,17	0,39
53	-0,17	0,48	-1,98	-0,07
54	0,74	-0,51	2,14	-20,67
55	-0,40	0,10	0,17	-1,02
56	-0,05	-3,28	-0,42	-0,49
57	0,67	-1,67	-0,78	-0,83
58	0,81	0,34	-0,61	0,05
59	1,05	0,48	-0,95	0,42
60	-1,23	-0,55	1,41	-0,12
61	-0,15	0,48	0,41	-0,95
62	-0,84	-0,75	1,07	0,25
63	0,90	0,68	-0,73	0,47
64	-0,82	0,07	-0,38	0,94
65	0,26	0,86	-0,29	1,27
66	-0,06	-0,14	0,54	-1,22
67	-0,06	-0,51	-1,17	0,42
68	-0,37	-0,07	0,59	3,59
69	-0,58	-0,51	0,43	0,27
70	2,30	-1,40	1,19	-0,41
71	0,18	-0,44	-0,40	-0,12
72	-0,52	-	0,36	-
73	0,40	-	-	-
74	-1,42	0,07	-0,69	-0,34

ANNEX 2
GRAPHS

Graph 1



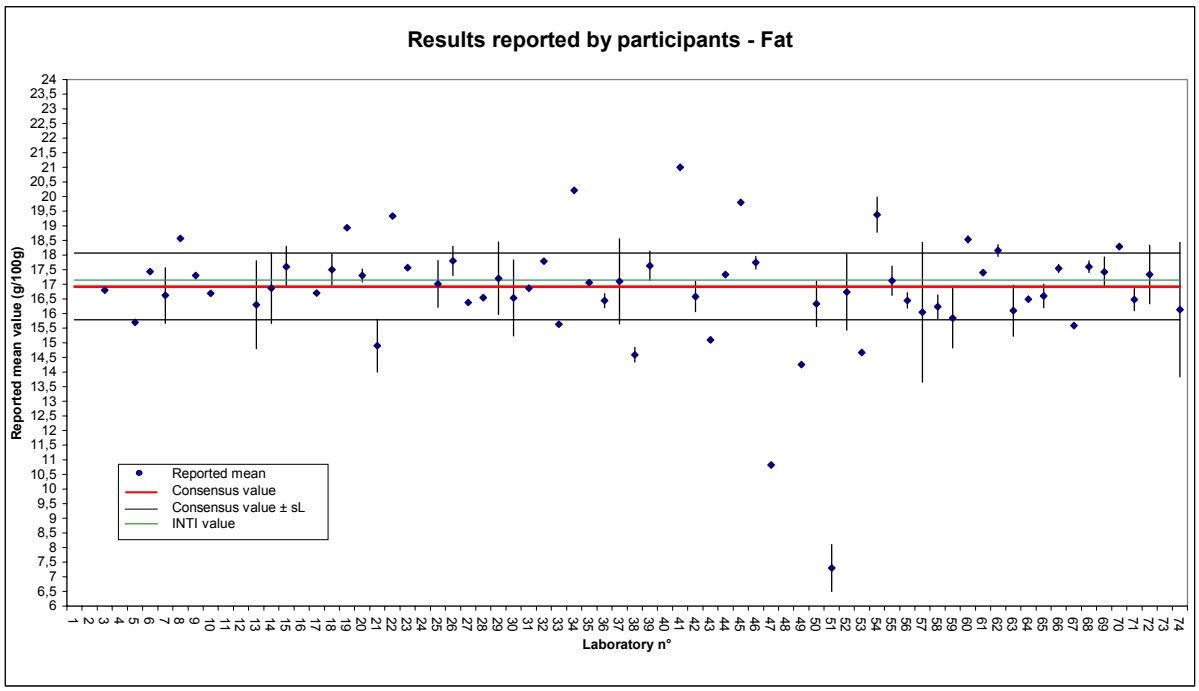
Graph 2



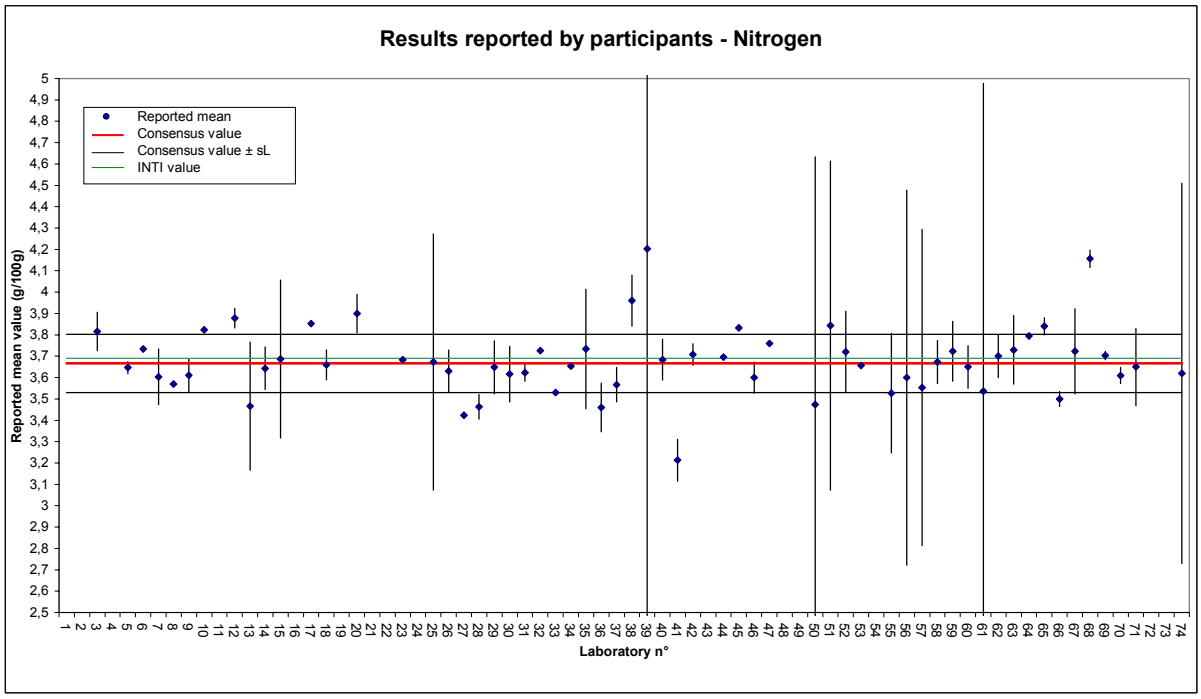
Out of scale participants:

n°	Mean value
31	0,813

Graph 3



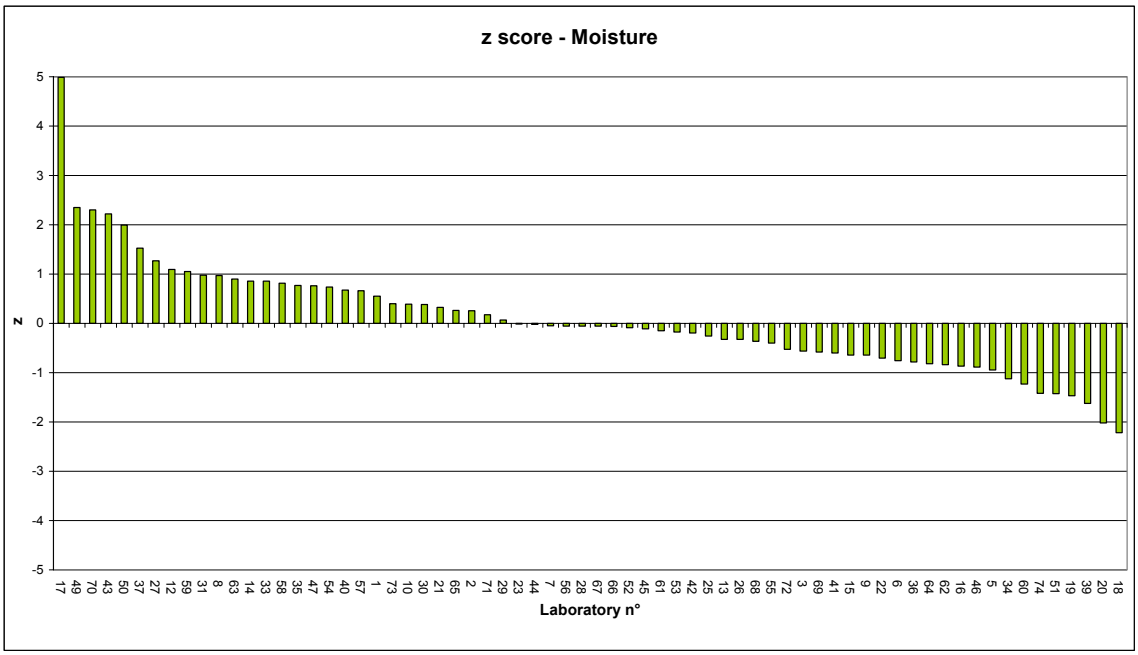
Graph 4



Out of scale participants:

n°	Mean value
54	0,843

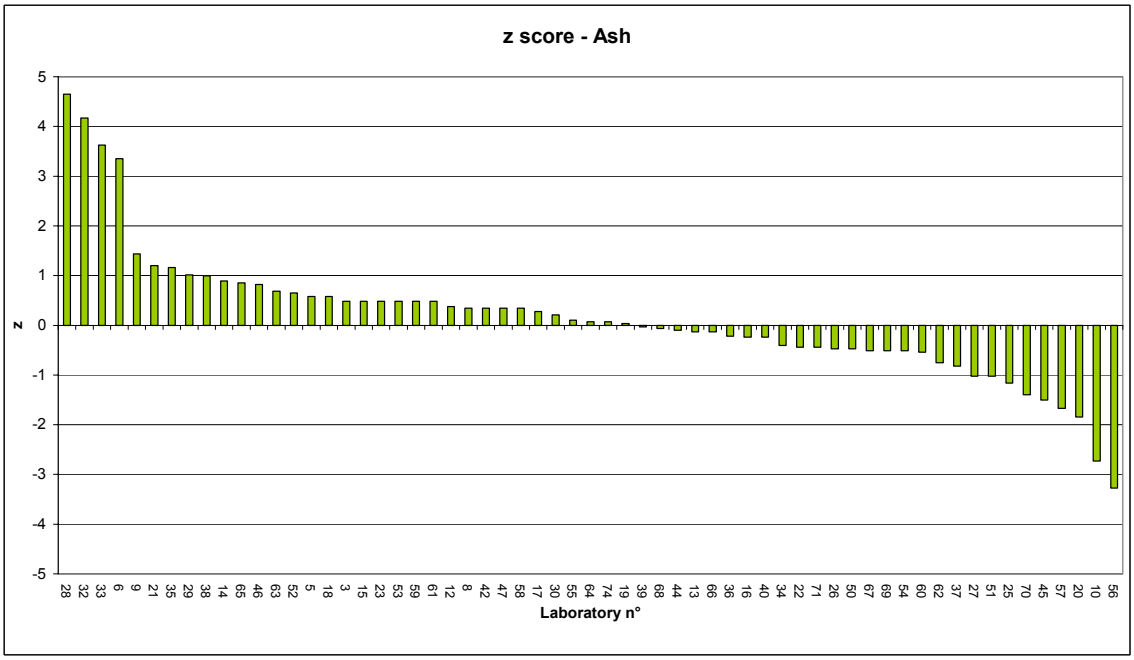
Graph 5



Out of scale participants:

n°	z
38	12,9
32	-8,9

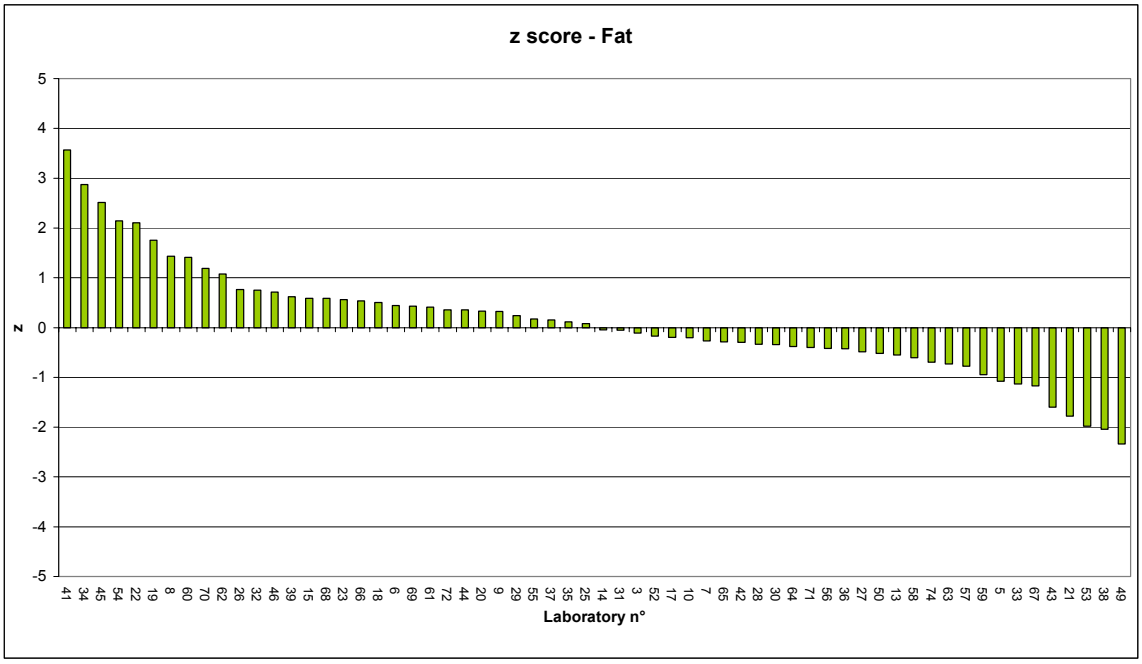
Graph 6



Out of scale participants:

n°	z	n°	z
43	9,1	41	-8,9
7	-7,8	31	-23,6

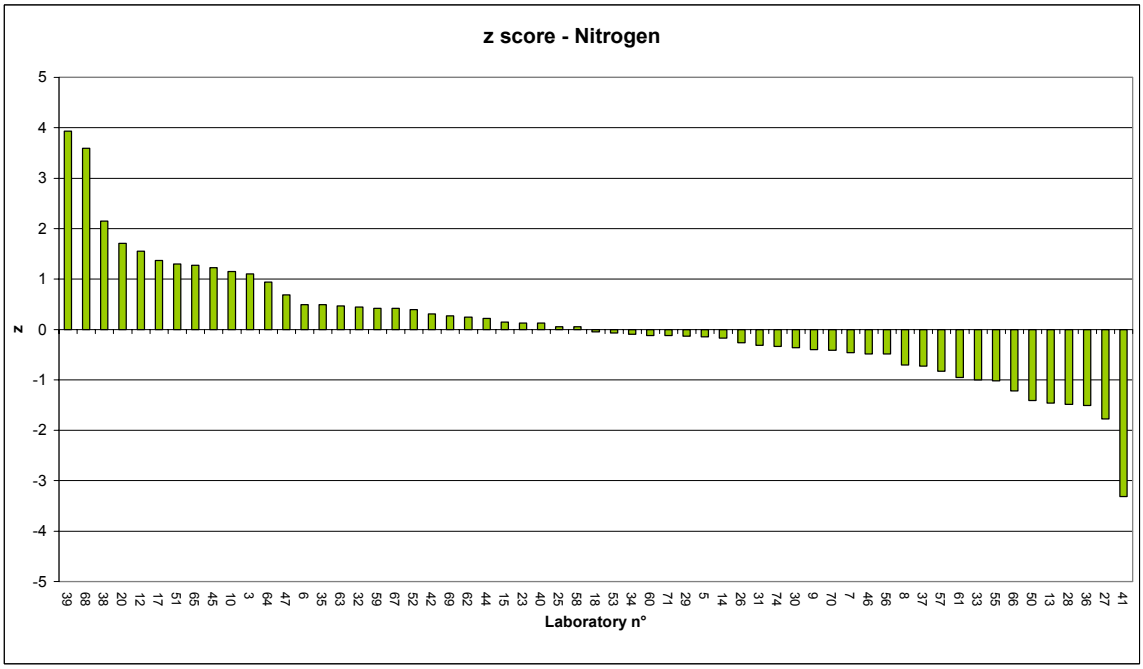
Graph 7



Out of scale participants:

n°	z
47	-5,3
51	-8,4

Graph 8



Out of scale participants:

n°	z
54	-20,7

ANNEX 3
DEFINITIONS

Test result: The value of a characteristic obtained by carrying out a specified test method.

Accepted reference value: A value that serves as an agreed-upon reference for comparison, and which is derived as:

a) a theoretical or established value, based on scientific principles;
b) an assigned or certified value, based on experimental work of some national or international organization;
c) a consensus or certified value, based on collaborative experimental work under the auspices of a scientific or engineering group.
d) When a), b), and c) are not available, the expectation of the (measurable) quantity, i.e. the mean of a specified population of measurements.

Accuracy: the closeness of agreement between a test result and the accepted reference value.

Trueness: The closeness of agreement between the average value obtained from a large series of test results and an accepted reference value.

Bias: The difference between the expectation of the test results and an accepted reference value.

Laboratory bias: The difference between the expectation of the test results from a particular laboratory and an accepted reference value.

Bias of the measurement method: The difference between the expectations of test results obtained from all laboratories using that method and an accepted reference value.

Repeatability: Precision under repeatability conditions.

Repeatability conditions: Conditions where independent test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time.

Repeatability standard deviation: The standard deviation of test results obtained under repeatability conditions.

Reproducibility: Precision under reproducibility conditions.

Reproducibility conditions: Conditions where test results are obtained with the same method on identical test items in different laboratories with different operators using different equipment.

Reproducibility standard deviation: The standard deviation of test results obtained under reproducibility conditions.

Outlier: A member of a set of values which is inconsistent with the other members of that set.

Statistic General Definitions

n = data number

x_i = data

Average = $x_{1/2}$ = mean value = $(\sum x_i) / n$

Standard deviation = $S_d = [\sum (x_i - x_{1/2})^2 / (n - 1)]^{1/2}$

Relative deviation from mean value (%) = $[(x_i - x_{1/2}) / x_{1/2}] *100$

Relative deviation from reference value (%) = $[(x_i - x_{ref}) / x_{ref}] *100$

z score

The first stage in producing a score from a result x (a single measurement of analyte concentration (or amount) in a test material) is obtaining an estimate of the bias, which is defined as:

Bias estimate: $x_i – x_{ref}$

Where x_{ref} is the assigned value for the analyte in the sample.

Most proficiency testing schemes proceed by comparing the bias estimate with a target value for standard deviation that forms the criterion of performance. An obvious approach is to form the z-score given by

$$z = (x_i - x_{ref}) / \sigma$$

In some circumstances the technical panel may decide to use a fixed standard deviation based in several criterion as the expected performance of the laboratories, the precision required for a specific task of data interpretation, etc.

It also can be used an estimate of the actual variation encountered in a particular round of a trial. In that cases should be estimated from the laboratories results after outlier elimination, or by robust methods for each analyte/ material / round combination.

If x_{ref} and σ were good estimates of the population mean and standard deviation, and the underlying distribution were normal, then z would be approximately normally distributed with a mean of zero and a unit standard deviation. An analytical system can be described as "well behaved" when it complies with this conditions.
Under these circumstances an absolute value of z (|z|) greater than three suggests poor performance.

In a well-behaved analytical system z-scores would be expected to fall outside the range -2>z<2 in about 5 % of instances, and outside the range -3>z<3 only in about 0.3 %. In the latter case it could be interpreted that the probability is so small for a "well-behaved" system, that it almost certainly represents a poor performance. It would be therefore be possible to classify scores as:

$|z| \leq 2$ Satisfactory $2 < |z| > 3$ Questionable $|z| \geq 3$ Unsatisfactory

Grubbs test

To calculate the single Grubbs test statistics, compute de average for each laboratory and then calculate the standard deviation (S) of these L averages(designate as the original s). Calculate de S of the set of averages with the highest average removed (s_a); calculate the S of the set of averages with the lowest average removed (s_b).

Then calculate the percentage decrease in S for both as follows:

$100 \times [1 - (s_b / s)]$ and $100 \times [1 - (s_a / s)]$

The higher of these percentage decreases is the single Grubbs test statistic, which signals the presence of an outlier to be omitted at the P=2,5% level, if it exceeds the critical value.

Cochran test

Given a set of standard deviations s_i, all computed from the same number of replicated test results, Cochran´s criterion then becomes:

$$C = s^2_{max} / \sum s^2_i$$

Where s_{max} stand for the highest value in the set.
This value C is compared with the critical value in the Cochran table for the number of replicates and laboratories specified (5% level).
If C exceeds the critical value, s_{max} is classified as a statistical outlier.

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