

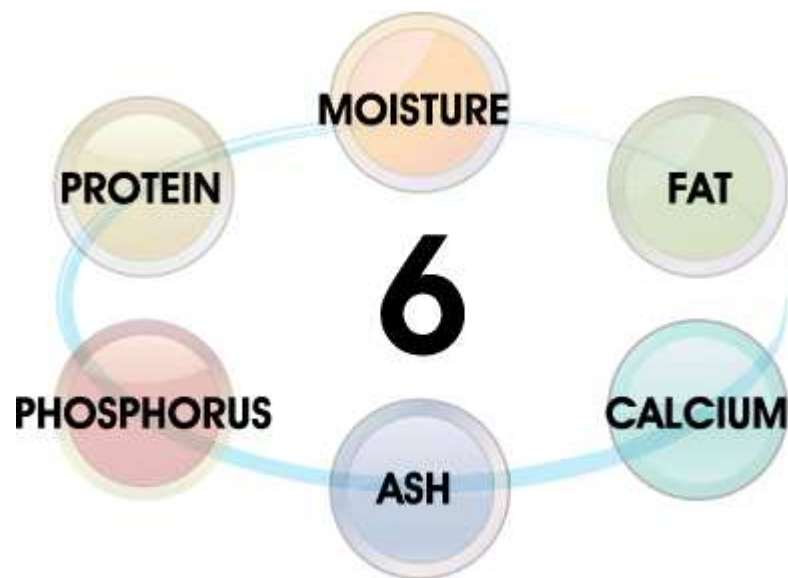


Asia Pacific Laboratory Accreditation Cooperation

**PROFICIENCY TESTING PROGRAM
DETERMINATION OF MOISTURE, PROTEIN, FAT, ASH,
CALCIUM AND PHOSPHORUS
IN FEEDING STUFFS
[**APLAC T060**](#)**

[FINAL REPORT**](#)**

February 2010



**The Programme was organized by
Department of Science Service (DSS)
Ministry of Science and Technology, Thailand**

SUMMARY

1. This report summarized the results of the proficiency testing programme (APLAC T060) on the proximate analysis (namely moisture, protein, fat, ash, calcium and phosphorus) in Feeding stuffs. A total of 27 laboratories from 7 economies registered in this program and returned their results to the organizer within the scheduled timeline.

2. The samples for this proficiency testing programme were Pig Feed. The assigned values for all analytes were calculated from the most appropriate measure of central tendency of participants' results which were evaluated according to the robust statistical techniques outlined in ISO 13528:2005 "Statistical methods for use in proficiency testing by interlaboratory comparisons"

3. Summary of participants' results:

Test items	No. of data submitted	No. of Participants (percentage)		
		$ Z \leq 2$	$2 < Z < 3$	$ Z \geq 3$
Moisture (Temp 103±2 °C)	15	14 (93.3 %)	0 (0 %)	1 (6.7 %)
Moisture (Temp 135±2 °C)	13	13 (100 %)	0 (0 %)	0 (0 %)
Protein (Nx6.25)	27	25 (92.6 %)	2 (7.4 %)	0 (0%)
Fat	27	25 (92.6 %)	2 (7.4 %)	0 (0 %)
Ash	27	24 (88.9 %)	1 (3.7 %)	2 (7.4 %)
Calcium	27	24 (88.9 %)	1 (3.7 %)	2 (7.4 %)
Phosphorus	27	20 (74.1 %)	1 (3.7 %)	6 (22.2 %)

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1. Introduction

1.1 APLAC T060 was organized by the Department of Science Service (DSS), under the auspices of the Asia – Pacific Laboratory Accreditation Co-operation (APLAC).

1.2 A total of 27 laboratories from 7 economies registered in this program and returned their results to the organizer within the scheduled timeline. Summary of Economies and Participants is listed below.

Economy	No. of Participants
Japan	4
Korea	1
New Zealand	2
Singapore	2
Thailand	12
USA	2
Vietnam	4
Total	27

2. Objectives

2.1 The aim of the program was to assess laboratories' ability to competently perform the analysis for the contents of moisture, protein, fat, ash, calcium and phosphorus in feeding stuffs through interlaboratory comparison.

2.2 Test results from participants were evaluated according to the robust statistical techniques that are outlined in ISO 13528:2005 "Statistical methods for use in proficiency testing by interlaboratory comparisons" and the IUPAC International Harmonized Protocol for Proficiency Testing of analytical chemistry laboratories. The z-score was provided as a numerical indicator to reveal individual participant's analytical competence relative to others in the programme.

3. Test material

3.1 Preparation

About 10 kg of commercially available pig feed were bulked together. The samples were milled in a stainless steel grinding machine until the fine particles passed completely through sieve 500 μm and mixed thoroughly in a rotating mixer. Aliquots of about 45 g of powder were packed into laminated bag, which were then stored at ambient temperature prior to dispatch.

3.2 Homogeneity testing

Prior to distribution, test samples were checked for homogeneity. Ten randomly selected samples were analyzed in duplicate for moisture, protein, fat, ash, calcium and phosphorus under repeatability conditions; i.e., same laboratory, same operator, same method, and same apparatus. The results, together with their statistical evaluations are given in Appendix I (Table 1-2). The results of homogeneity testing indicated that there no significant variability existed in the samples. Therefore, any extreme results subsequently identified should not be attributed to sample variability.

3.3 Stability Testing

Assessment of the adequacy of stability was calculated by comparing the general average of the measurements obtained in the homogeneity testing with the general average of the results obtained in the stability testing (ISO 13528:2005). Statistical analysis showed that the sample was stable over the time of preparation to the last day analysis. The results are shown in Appendix I (Table 3).

3.4 Distribution of samples and documents

3.4.1 Samples together with “Instructions to Accreditation Bodies” “Participating Laboratories Nomination Form”, “Receipt Form for Accreditation Bodies”, “Instructions to participating Laboratories”, “Receipt Form for Participating Laboratories” and “Results Proforma” were sent to participating accreditation bodies (AB) in August 2009. A sample copy of these instructions is given in APPENDIX V.

3.4.2 All ABs were informed to read the “Instructions to Accreditation Bodies” and to redirect the sample (s) and the attached documents to their nominated laboratories as soon as possible.

4. Test Procedures

- 4.1. Each participant was provided with one sample. A single result was submitted for each sample for each measurand. Participants were required to determine the concentration of moisture, protein, fat, ash, calcium and phosphorus in feeding stuffs.
- 4.2. Participants should use routine test methods (could be accredited, in-house, modified, etc.) which would normally be used to test customer supplied samples and record the method used on the result sheet. Participants were requested to give their method Information in Part II of RESULTS PROFORMA.
- 4.3 Report the moisture, protein, fat, ash, calcium and phosphorus in g/100g (as received sample), express the results to two decimal places.
- 4.4 Report the measurement uncertainty for each result as expanded uncertainty (g/100g) at 95% confident level.
- 4.5 Record the requested information in the results sheet (weight sample, temperature and drying time, ashing time and ashing temp etc.)

5. Statistical Evaluation of Participants' Data

5.1 Participants' test results

All participants (27 laboratories) reported their results with the associated measurement uncertainty. Addition information on the analysis method used for each measurand was also reported. All testing results were recorded in order to evaluate differences using the statistical analysis.

5.2. Performance evaluation

5.2.1. The assigned value for test material used in the proficiency testing scheme is the robust average (x^*) and the robust standard deviation (s^*) of the results reported by all the participants in the PT-program, calculated using Algorithm A from ISO 13528:2005. With those values, the participant's results are evaluated using the robust z-score as follow:

$$Z = \frac{(X_i - x^*)}{s^*}$$

Where X_i is the participant's result
 x^* is the robust average of the results calculated by using
Algorithm A
 s^* is the robust standard deviation of the results calculated by using
Algorithm A

5.2.2 The criteria for laboratory's performance are interpreted as follows:

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

An unacceptable result is defined as $|z|$ score equal or larger than 3 (i.e. $|z| \geq 3$). All unacceptable results should be investigated by the participant laboratories. Laboratories having $|z|$ score in the range $2 < |z| < 3$ are also encouraged to review their results.

5.2.3 When the assigned value is derived as a robust average of the results calculated by using Algorithm A, the standard uncertainty (u_x) of the assigned value is estimated as:

$$u_x = 1.25 \times \frac{s^*}{\sqrt{n}}$$

where s^* is the robust standard deviation of the results calculated by using
Algorithm A
 n is the number of results

The expanded uncertainty (U_x) of the assigned value is

$$U_x = 2u_x$$

Where 2 is the coverage factor for level of Confidence ~ 95 %

5.2.4 Laboratories who reported the expanded uncertainty of their results will be scored by E_n numbers also (apart from z-scores). E_n numbers are calculated as:

$$E_n = \frac{(x_{Lab} - X_{ref})}{\sqrt{U_{lab}^2 + U_{ref}^2}}$$

where x_{lab} is the participant's result

X_{ref} is the robust average of the results calculated by using Algorithm A (x^*)

U_{Lab} is the expanded uncertainty of participant's result

U_{ref} is the expanded uncertainty of X_{ref}

The criteria for laboratory's performance are interpreted as follows:

$ E_n \leq 1$	Satisfactory
$ E_n > 1$	Unsatisfactory

An absolute E_n number of ≤ 1 indicates that the reported results and assigned value are in agreement (within their respective uncertainties). An absolute E_n number of >1 indicates that the reported result is different from the assigned value and that the uncertainty associated with the result has been understated.

6. Testing Results and Assessment

From the reported testing results of moisture, protein, fat, ash, calcium and phosphorus in feeding stuffs the distribution of the results for analyzing moisture was found to be bimodal so the evaluation of the laboratory efficiencies were separated in two groups. The first group was the 15 laboratories which used temperature between 101 – 105 °C. (103 ± 2 °C). The second group was the 13 laboratories which used temperature between 133-137 °C (135 ± 2 °C) [Histogram of moisture, Protein, fat, Ash, Calcium and Phosphorus are shown in APPENDIX IV (Figures 15-20)]

Analytical results from participants, z-score are presented in APPENDIX II (Tables 4-10 and Figures 1-7). Their methodologies and techniques employed are tabulated in APPENDIX II (Tables 11-17). Laboratories who also reported the expanded uncertainty of their results are scored by E_n numbers also. Diagrams of Testing results related with measurement uncertainty of laboratories are shown in APPENDIX III (Figures 8-14).

6.1. Robust statistical procedures were used to generate the z-scores and the standard uncertainties (u_x) of the assigned values. The summary statistics are listed as follows:

Test items	No. of results	Robust average (x^*) ¹	Robust standard deviation (s^*) ²	Standard uncertainty of the assigned value (u_x)	Expanded uncertainty of the assigned value (U_x)
Moisture (g/100g) (Temp 103±2 °C)	15	9.005	0.228	0.074	0.148
Moisture (g/100g) (Temp 135±2 °C)	13	9.984	0.461	0.160	0.320
Protein (Nx6.25) (g/100g)	27	19.315	0.323	0.078	0.156
Fat (g/100g)	27	4.683	0.346	0.083	0.166
Ash (g/100g)	27	8.420	0.188	0.045	0.090
Calcium (g/100g)	27	1.036	0.064	0.015	0.030
Phosphorus (g/100g)	27	0.865	0.034	0.008	0.016

Note (x^*)¹ = Robust average (x^*) calculated by Algorithm A.

(s^*)² = Robust standard deviation (s^*) calculated by Algorithm A

6.2 The z-scores performance of participants are summarized as follows :

Test items	No. of data submitted	No. of Participants (percentage)		
		$ Z \leq 2$	$2 < Z < 3$	$ Z \geq 3$
Moisture (Temp 103±2 °C)	15	14 (93.3 %)	0 (0 %)	1 (6.7 %)
Moisture (Temp 135±2 °C)	13	13 (100 %)	0 (0 %)	0 (0 %)
Protein (Nx6.25)	27	25 (92.6 %)	2 (7.4 %)	0 (0%)
Fat	27	25 (92.6 %)	2 (7.4 %)	0 (0 %)
Ash	27	24 (88.9 %)	1 (3.7 %)	2 (7.4 %)
Calcium	27	24 (88.9 %)	1 (3.7 %)	2 (7.4 %)
Phosphorus	27	20 (74.1 %)	1 (3.7 %)	6 (22.2 %)

6.3 The E_n number performance of participants are summarized as follows :

Test items	No. of data submitted	No. of Participants (percentage)	
		$ E_n \leq 1$	$ E_n > 1$
Moisture (Temp 103±2 °C)	12	7 (58.3 %)	5 (41.7 %)
Moisture (Temp 135±2 °C)	7	5 (71.4 %)	2 (28.6 %)
Protein (Nx6.25)	20	16 (80.0 %)	4 (20.0 %)
Fat	17	9 (52.9 %)	8 (47.1 %)
Ash	17	11 (64.7 %)	6 (35.3 %)
Calcium	15	10 (66.7 %)	5 (33.3 %)
Phosphorus	14	10 (71.4 %)	4 (28.6 %)

6.4 The lab codes with questionable or unsatisfactory results are listed as follows :

Test items	z-score		E_n number
	$2 < Z < 3$	$ Z \geq 3$	$ E_n > 1$
Moisture (Temp 103±2 °C)	-	016	001, 004, 013, 016, 023
Moisture (Temp 135±2 °C)	-	-	019, 025
Protein(Nx6.25)	017, 022	-	012, 013, 017, 025
Fat	019,021	-	003, 004, 005, 011, 013, 016, 017, 019
Ash	019	002, 010	004, 007, 010, 011, 016, 019
Calcium	020	002, 010	001, 005, 008, 013, 016
Phosphorus	022	006, 010, 017, 018, 019, 020	008, 017, 019, 022

6.5 Technical comments

For this PT program, the performance evaluation of moisture was separated in two groups. The first group was the laboratories which used temperature between 101 – 105 °C. (103 ± 2 °C). The second group was the laboratories which used temperature between 133-137 °C (135 ± 2 °C). The differences in analytical methods used for proximate analysis showed significant effect on their analytical results. Therefore the laboratories should strictly follow the standard method. Any deviations from the standard method should be avoided, and any deviations shall be validated before using them as routine methods.

7. Remarks

7.1 The Proficiency Testing Section, Bureau of Laboratory Accreditation, Department of Science Service (DSS) is responsible for organizing Proficiency Testing (PT) programmes. The programmes include several fields such as food chemistry, food microbiology and environmental field. Since 2005, the DSS has offered the following programmes:

- aerobic plate count in starch;
- moisture, polarization, colour and reducing sugars in sugar;
- moisture, protein, ash and pH in flour;
- moisture, protein, fat, crude fiber and ash in feeding stuffs;
- Ca, Fe, K, Mg, Na, P and Zn in feeding stuffs;
- heavy metals in water;
- Total Dissolved Solids (TDS) and Suspended Solids (SS) in water;
- pH value in water;
- Chemical Oxygen Demand of water.

The participants are mostly from within Thailand. However, we have offered PT programmes to a number of laboratories in Pakistan since both countries have signed a Memorandum of Understanding (MOU) for scientific and technological cooperation.

7.2 Department of Science Service (DSS) wishes to acknowledge the financial support provided by Asia Pacific Laboratory Accreditation Cooperation (APLAC). Special thanks are also extended to the Betagro Public Company Limited (Phrapradaeng) in Thailand for supplying the samples and checking the homogeneity and stability of the sample.

7.3 Efforts and supports from all AB and participants in this programme are gratefully noted. Participants are welcome to forward any comments on this PT to the following address:

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8. References

- 8.1. Statistical methods for use in proficiency testing by interlaboratory comparisons, **ISO/13528**. 2005.
- 8.2. M. Thompson, S.L. Ellison, R. Wood. The International harmonized protocol for the proficiency testing of analytical chemistry laboratories (IUPAC technical report), Pure Appl. Chem. 2006,78:146-196.
- 8.3. J.N. Miller, J.C. Miller, Statistics and Chemometrics for Analytical Chemistry, 4th edition, 2000, prentice Hill.
- 8.4 The International Organization for Standardization/The International Electrotechnical Commission. Proficiency testing by interlaboratory comparisons-Part 1: Development and operation of proficiency testing schemes, **ISO/IEC Guide 43-1**. 1997.
- 8.5 Guidelines for the requirements for the competence of providers of proficiency testing schemes, **ILAC-G 13**. 2007.

APPENDIX I

Homogeneity and Stability testing

1. Homogeneity testing

Table 1 : Homogeneity data of Feeding stuffs for moisture, protein and fat (tested March 2009).

Sample	Moisture, g/100g		Protein , g/100g		Fat , g/100g	
	replicate 1	replicate 2	replicate 1	replicate 2	replicate 1	replicate 2
1	8.94	8.95	19.59	19.34	4.71	4.59
2	8.97	8.94	19.55	19.54	4.29	4.61
3	8.94	8.93	19.49	19.45	4.60	4.31
4	8.93	8.97	19.50	19.57	4.32	4.48
5	8.96	8.93	19.51	19.37	4.57	4.45
6	8.95	8.96	19.54	19.37	4.44	4.56
7	8.95	8.93	19.51	19.30	4.51	4.65
8	8.92	8.94	19.58	19.61	4.66	4.71
9	8.95	8.94	19.28	19.39	4.44	4.48
10	8.93	8.94	19.61	19.36	4.48	4.52
Mean		8.944	19.473		4.519	
Between Sample analysis	S_s	0.007	0.021		0.037	
	$\hat{\sigma}$	0.228	0.323		0.285	
	$0.3 \hat{\sigma}$	0.068	0.097		0.086	
	$S_s \leq 0.3 \hat{\sigma}$	SUFFICIENT	SUFFICIENT		SUFFICIENT	

For this PT scheme : $\hat{\sigma} = s^*$ (s^* = Robust standard deviation for proficiency testing)

Table 2 : Homogeneity data of feeding stuffs for ash, calcium and phosphorus
(tested March 2009).

Sample	Ash, g/100g		Calcium , g/100g		Phosphorus , g/100g	
	replicate 1	replicate 2	replicate 1	replicate 2	replicate 1	replicate 2
1	8.290	8.310	0.93	0.96	0.84	0.88
2	8.290	8.300	0.94	0.91	0.85	0.83
3	8.320	8.160	0.91	0.91	0.84	0.85
4	8.360	8.340	0.92	0.91	0.85	0.89
5	8.200	8.330	0.92	0.89	0.84	0.85
6	8.270	8.300	0.94	0.91	0.88	0.87
7	8.280	8.160	0.96	0.91	0.89	0.86
8	8.290	8.400	0.89	0.86	0.84	0.85
9	8.250	8.240	0.84	0.91	0.86	0.84
10	8.220	8.250	0.87	0.99	0.83	0.87
Mean		8.278	0.914		0.855	
Between Sample analysis	S_s	0.016	0.010		0.004	
	$\hat{\sigma}$	0.188	0.064		0.034	
	$0.3 \hat{\sigma}$	0.056	0.019		0.010	
	$S_s \leq 0.3 \hat{\sigma}$	SUFFICIENT	SUFFICIENT		SUFFICIENT	

For this PT scheme : $\hat{\sigma} = s^*$ (s^* = Robust standard deviation for proficiency testing)

Assessment of the adequacy of homogeneity was calculated by comparing the between samples standard deviation (S_s) with the standard deviation for proficiency assessment ($\hat{\sigma}$) (ISO 13528:2005). The between samples standard deviation should be less than $0.3 \hat{\sigma}$ in order for the materials to be considered homogeneous.

$$S_s \leq 0.3 \hat{\sigma}$$

where

S_s is the between samples standard deviation

$\hat{\sigma}$ is the standard deviation for proficiency assessment

2. Stability testing

Table 3 : Stability data of feeding stuffs for moisture, protein, fat, ash, calcium and phosphorus
(tested September 2009)

Sample	Moisture (g/100 g)	Protein(Nx6.25) (g/100g)	Fat (g/100g)	Ash (g/100g)	Calcium (g/100g)	Phosphorus (g/100g)
1	8.91	19.55	4.60	8.26	0.97	0.86
2	8.93	19.50	4.45	8.27	0.95	0.86
3	8.93	19.69	4.61	8.24	0.96	0.87
4	8.95	19.51	4.55	8.31	0.97	0.91
5	8.91	19.29	4.60	8.40	0.99	0.82
Mean (B)	8.926	19.508	4.562	8.296	0.968	0.864
Mean (A)	8.944	19.473	4.519	8.278	0.950	0.855
 A – B 	0.018	0.035	0.043	0.018	0.018	0.009
$\hat{\sigma}$	0.228	0.323	0.285	0.188	0.064	0.034
0.3 $\hat{\sigma}$	0.068	0.097	0.086	0.056	0.019	0.010
 A – B $\leq$ 0.3 $\hat{\sigma}$	SUFFICIENT	SUFFICIENT	SUFFICIENT	SUFFICIENT	SUFFICIENT	SUFFICIENT

For this PT scheme : $\hat{\sigma} = s^*$ (s^* = Robust standard deviation for proficiency testing)

Assessment of the adequacy of stability was calculated by comparing the general average of the measurements obtained in the homogeneity testing with the general average of the results obtained in the stability testing (ISO 13528:2005). The absolute difference should be less than $0.3 \hat{\sigma}$, the materials were considered to be stable over the time of preparation to the last day analysis

$$| A - B | \leq 0.3 \hat{\sigma}$$

where **A** is the general average of the measurements obtained in the homogeneity testing

B is the general average of the measurements obtained in the stability testing

$\hat{\sigma}$ is the standard deviation for proficiency assessment

APPENDIX II

Results, z-score and Histogram

Table 4 : Results and z-scores for moisture in feeding stuffs (Temp. 103 ± 2 °C)

Laboratory Code No.	Results (g/100g)		z-score	E _n number
	Value	MU*		
T060 - 001	9.35	0.06	1.51	2.17
T060 - 003	9.06	0.18	0.24	0.24
T060 - 004	9.32	0.21	1.38	1.23
T060 - 006	8.92	-	- 0.37	-
T060 - 007	9.23	0.20	0.99	0.91
T060 - 009	9.09	0.11	0.37	0.46
T060 - 010	8.89	0.34	- 0.50	0.31
T060 - 011	8.9	0.08	- 0.46	0.63
T060 - 012	9.10	0.2	0.42	0.38
T060 - 013	8.8	0.05	- 0.90	1.32
T060 - 014	9.03	1.20	0.11	0.02
T060 - 016	7.80	0.18	- 5.29 A	5.18
T060 - 018	9.12	-	0.50	-
T060 - 023	8.66	0.2	- 1.51	1.39
T060 - 024	8.83	-	- 0.77	-

Summary statistics	Sample
No. of results	15
Robust average ($\bar{x}^*$) (g/100g)	9.005
Robust standard deviation (s^*) g/100g)	0.228

- Note
1. “A” = “Action signal” If $|Z| \geq 3$
 2. “W” = “Warning signal” If $2 < |Z| < 3$
 3. MU* = Measurement Uncertainty

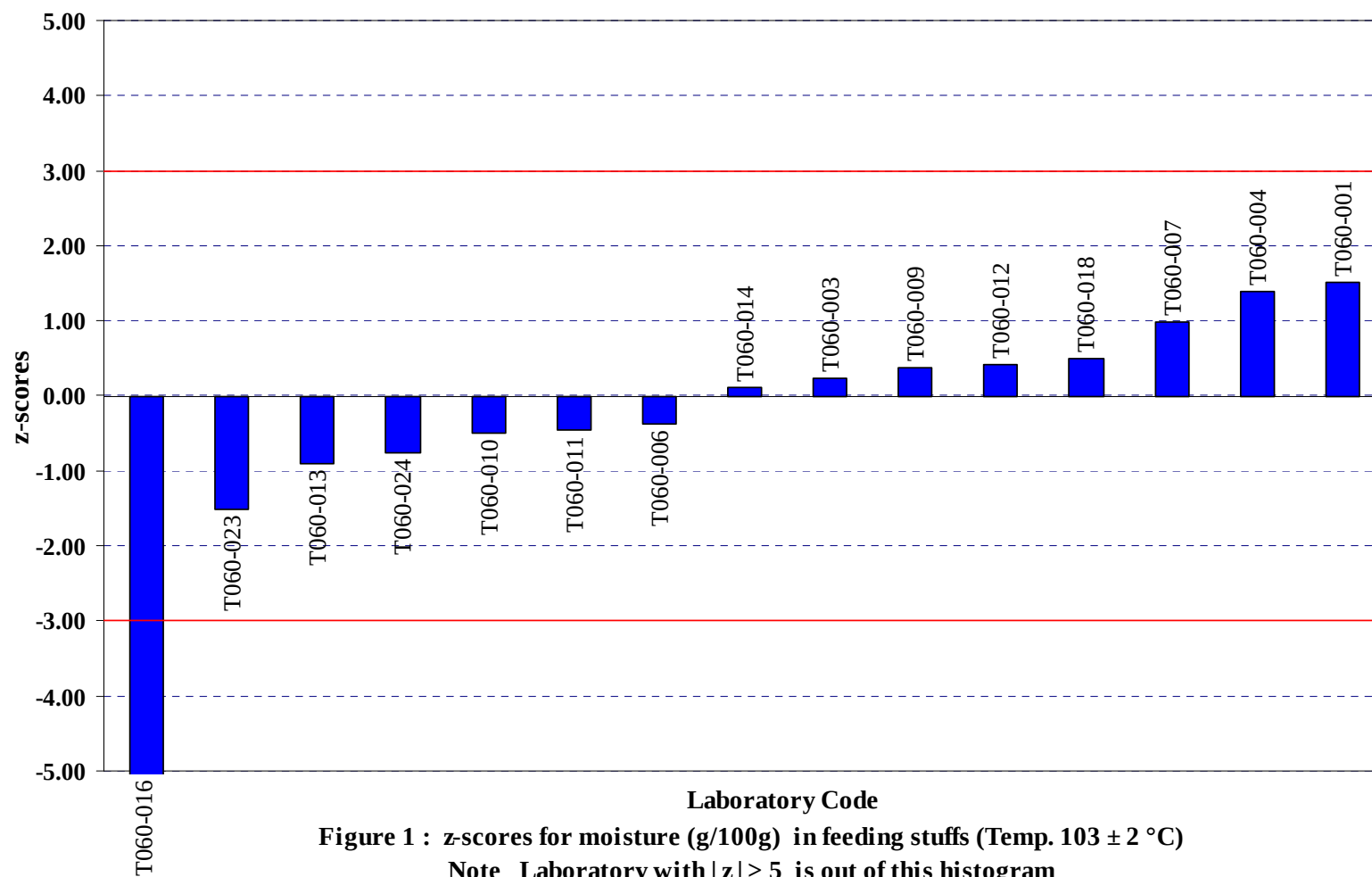


Table 5 : Results and z-scores for moisture in feeding stuffs (Temp.135 ± 2 °C)

Laboratory Code No.	Results (g/100g)		z-score	E _n number
	Value	MU*		
T060 - 002	9.70	-	- 0.62	-
T060 - 005	10.04	0.05	0.12	0.17
T060 - 008	10.23	-	0.53	-
T060 - 013	9.66	±0.05	- 0.70	1.00
T060 - 015	10.52	-	1.16	-
T060 - 017	10.08	0.45	0.21	0.17
T060 - 019	9.33	0.08	- 1.42	1.98
T060 - 020	10.21	-	0.49	-
T060 - 021	10.64	-	1.42	-
T060 - 022	9.59	-	- 0.85	-
T060 - 025	9.44	0.25	- 1.18	1.34
T060 - 026	10.19	0.10	0.45	0.62
T060 - 027	10.16	0.48	0.38	0.31

Summary statistics	Sample
No. of results	13
Robust average (x*) (g/100g)	9.984
Robust standard deviation (s*) g/100g)	0.461

- Note
1. “A” = “Action signal” If $|Z| \geq 3$
 2. “W” = “Warning signal” If $2 < |Z| < 3$
 3. MU* = Measurement Uncertainty

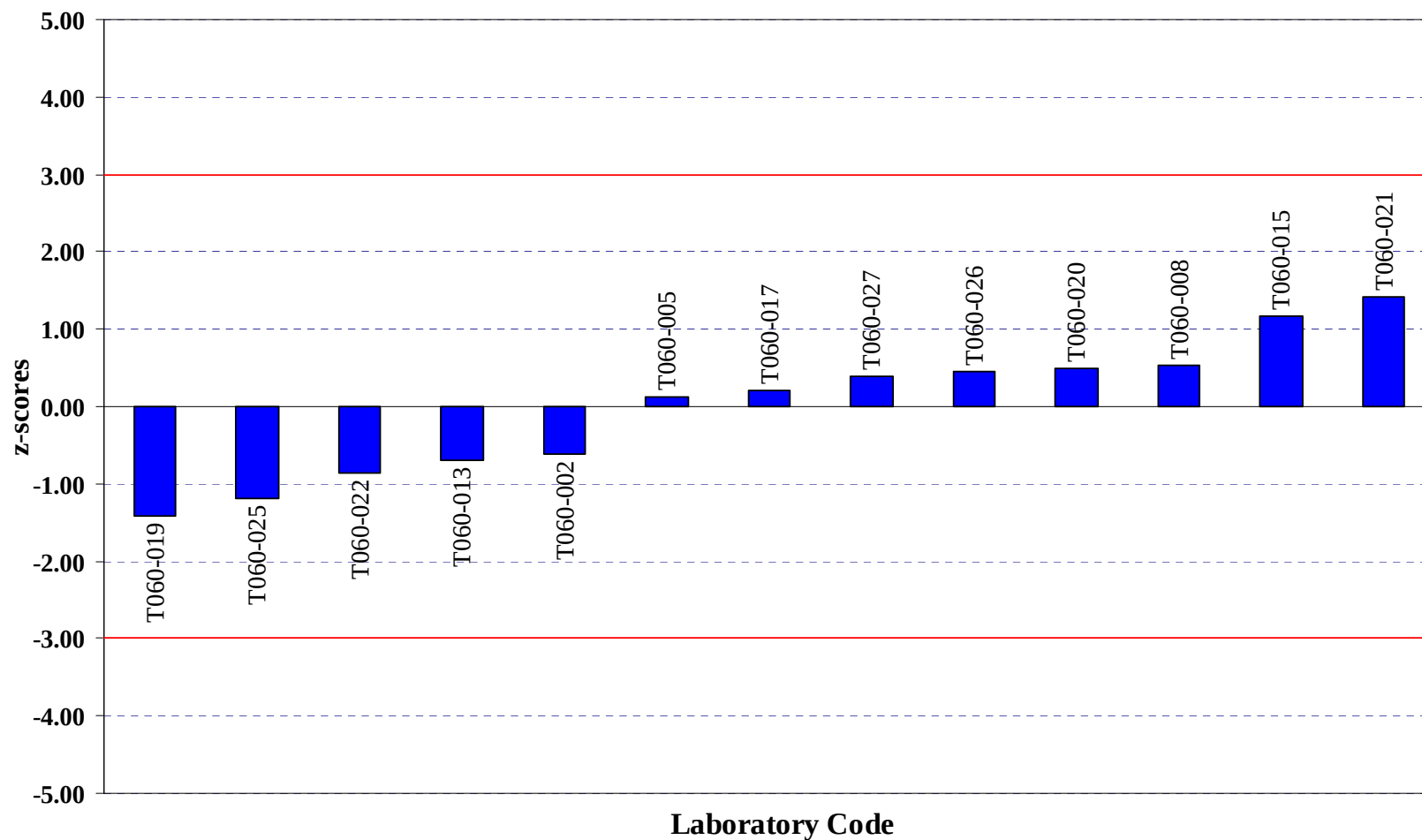


Figure 2 : z-scores for moisture (g/100g) in Feeding stuffs (Temp. 135 ± 2 °C)

Table 6 : Results and z-scores for protein in feeding stuffs

Laboratory Code No.	Results (g/100g)		z-score	E _n number
	Value	MU*		
T060 - 001	19.31	0.45	- 0.02	0.01
T060 - 002	19.82	-	1.56	-
T060 - 003	19.37	0.19	0.17	0.22
T060 - 004	19.10	0.23	- 0.67	0.77
T060 - 005	19.25	0.35	- 0.20	0.17
T060 - 006	18.89	-	- 1.32	-
T060 - 007	19.24	0.30	- 0.23	0.22
T060 - 008	19.18	0.56	- 0.42	0.23
T060 - 009	19.34	0.14	0.08	0.12
T060 - 010	19.17	0.24	- 0.45	0.51
T060 - 011	18.90	1.14	- 1.28	0.36
T060 - 012	19.55	0.14	0.73	1.12
T060 - 013	18.80	0.13	- 1.59	2.54
T060 - 014	19.21	0.44	-0.33	0.23
T060 - 015	19.15	-	- 0.51	-
T060 - 016	19.32	0.34	0.02	0.01
T060 - 017	20.00	0.49	2.12 W	1.33
T060 - 018	19.74	-	1.32	-
T060 - 019	19.52	0.36	0.63	0.52
T060 - 020	19.13	-	- 0.57	-
T060 - 021	19.45	-	0.42	-
T060 - 022	20.20	-	2.74 W	-
T060 - 023	19.08	0.6	- 0.73	0.38
T060 - 024	19.65	0.54	1.04	0.60
T060 - 025	18.86	0.25	- 1.41	1.55
T060 - 026	19.52	0.25	0.63	0.70
T060 - 027	19.41	0.28	0.29	0.30

Summary statistics	Sample
No. of results	27
Robust average (x*) (g/100g)	19.315
Robust standard deviation (s*) g/100g)	0.323

- Note
1. “A” = “Action signal” If $|Z| \geq 3$
 2. “W” = “Warning signal” If $2 < |Z| < 3$
 3. MU* = Measurement Uncertainty

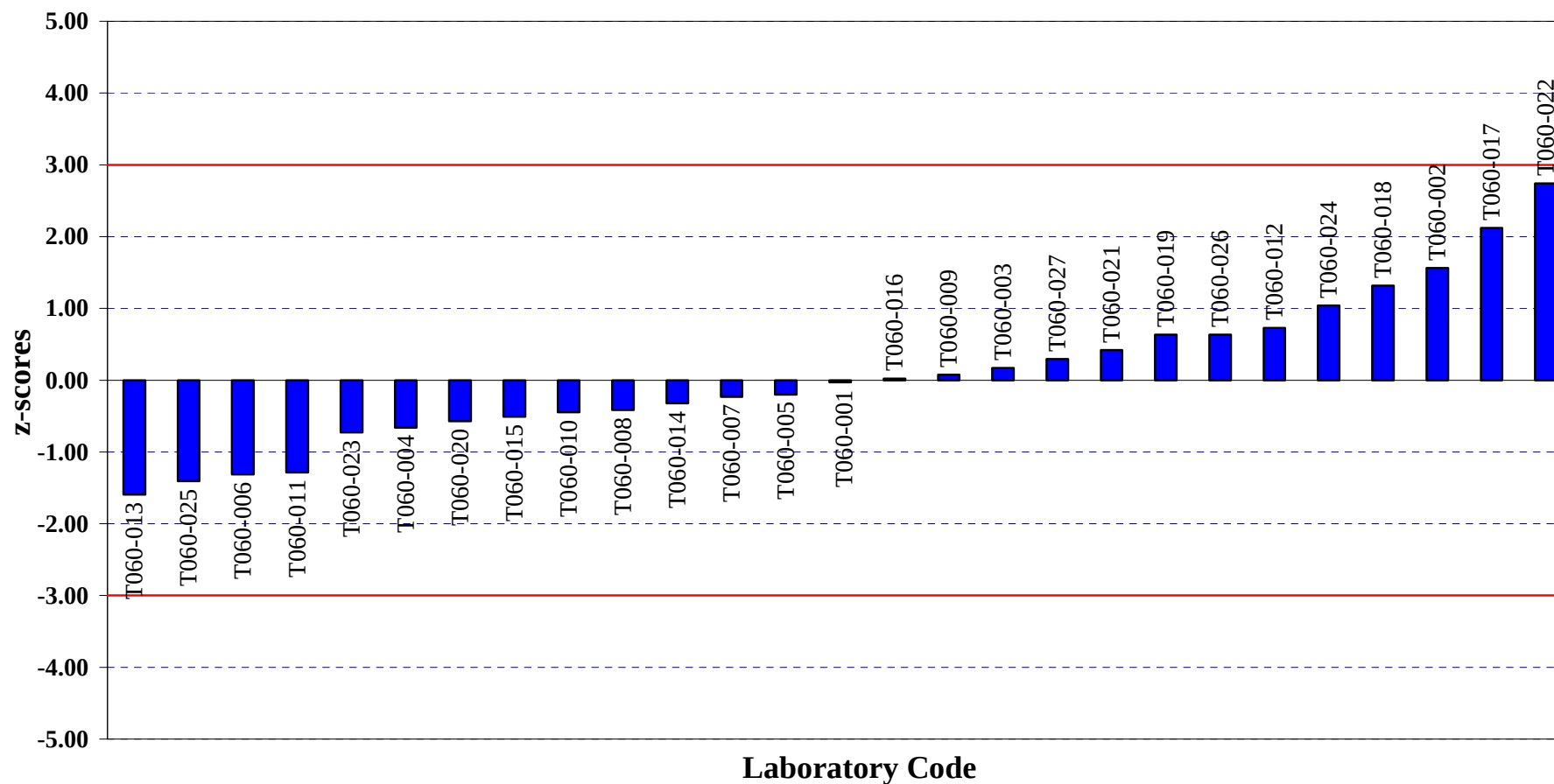


Figure 3 : z-scores for protein (g/100g) in Feeding stuffs

Table 7 : Results and z-score for fat in feeding stuffs

Laboratory Code No.	Results (g/100g)		z-score	E _n number
	Value	MU*		
T060 - 001	4.78	0.17	0.28	0.41
T060 - 002	5.04	-	1.03	-
T060 - 003	4.40	0.12	- 0.82	1.38
T060 - 004	4.94	0.10	0.74	1.32
T060 - 005	4.38	0.04	- 0.88	1.77
T060 - 006	4.66	-	- 0.07	-
T060 - 007	4.87	0.20	0.54	0.72
T060 - 008	4.36	0.28	- 0.93	0.99
T060 - 009	4.22	0.57	- 1.34	0.78
T060 - 010	4.80	0.10	0.34	0.60
T060 - 011	4.38	0.086	- 0.88	1.62
T060 - 012	4.46	-	- 0.64	-
T060 - 013	4.96	0.02	0.80	1.65
T060 - 014	4.45	2.92	- 0.67	0.08
T060 - 015	4.65	-	- 0.10	-
T060 - 016	5.31	0.06	1.81	3.54
T060 - 017	4.38	0.22	- 0.88	1.10
T060 - 018	4.63	-	- 0.15	-
T060 - 019	5.52	0.15	2.42 W	3.74
T060 - 020	4.34	-	- 0.99	-
T060 - 021	5.51	-	2.39 W	-
T060 - 022	4.99	-	0.89	-
T060 - 023	4.73	0.2	0.14	0.18
T060 - 024	4.52	-	- 0.47	-
T060 - 025	4.43	0.25	- 0.73	0.84
T060 - 026	4.43	-	- 0.73	-
T060 - 027	5.04	0.33	1.03	0.97

Summary statistics	Sample
No. of results	27
Robust average (x*) (g/100g)	4.683
Robust standard deviation (s*) g/100g)	0.346

- Note
1. “A” = “Action signal” If $|Z| \geq 3$
 2. “W” = “Warning signal” If $2 < |Z| < 3$
 3. MU* = Measurement Uncertainty

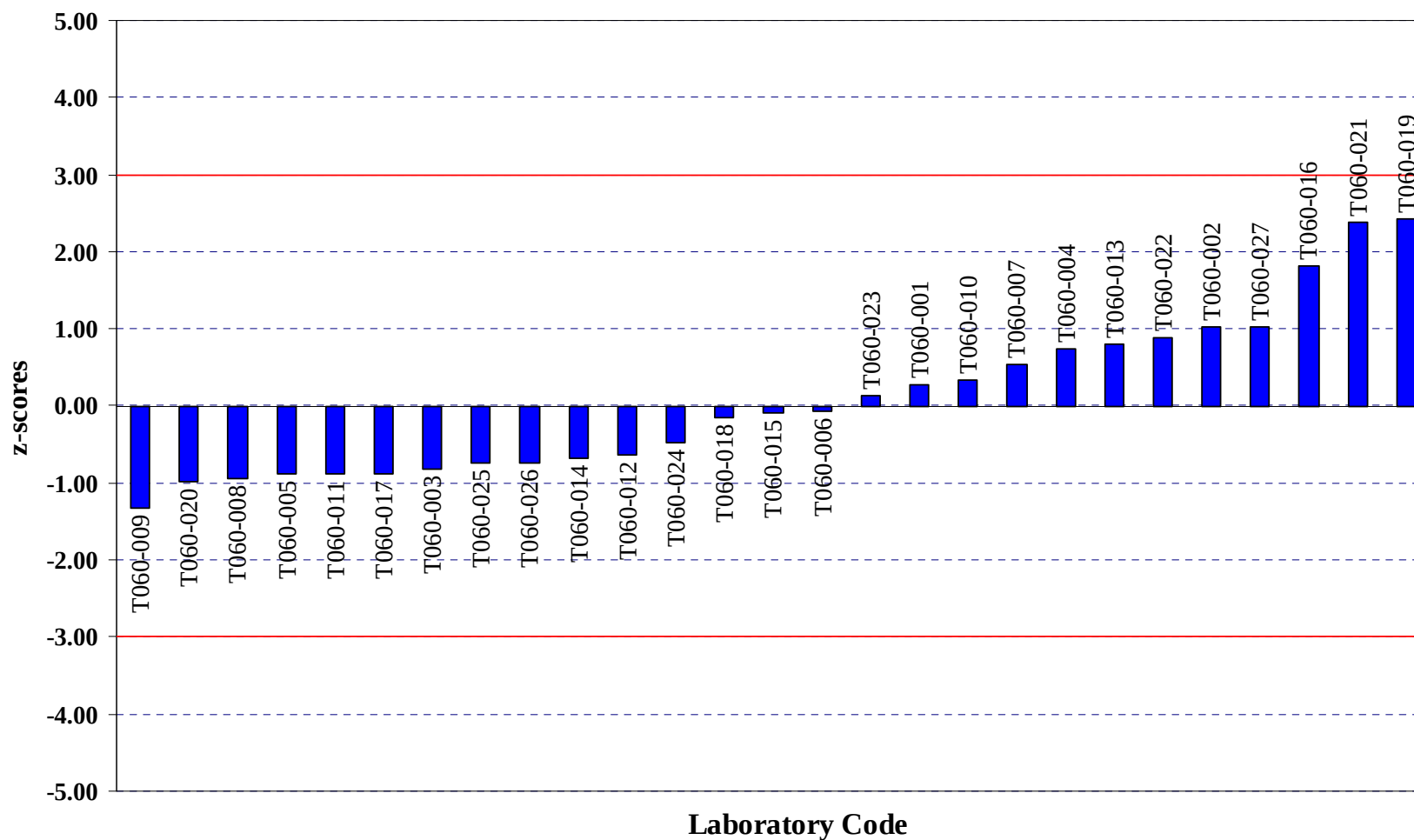


Figure 4 : z-scores for fat (g/100g) in Feeding stuffs

Table 8 : Results and z-scores for ash in feeding stuffs

Laboratory Code No.	Results (g/100g)		z-score	E _n number
	Value	MU*		
T060 - 001	8.29	0.40	- 0.69	0.32
T060 - 002	7.40	-	- 5.43 A	-
T060 - 003	8.48	0.15	0.32	0.34
T060 - 004	8.22	0.12	- 1.06	1.33
T060 - 005	8.37	0.03	- 0.27	0.52
T060 - 006	8.46	-	0.21	-
T060 - 007	8.75	0.25	1.76	1.24
T060 - 008	8.32	0.40	- 0.53	0.24
T060 - 009	8.56	0.18	0.74	0.69
T060 - 010	3.44	0.01	- 26.49 A	54.72
T060 - 011	8.56	0.048	0.74	1.37
T060 - 012	8.57	0.2	0.80	0.68
T060 - 013	8.50	0.07	0.43	0.70
T060 - 014	8.25	-	- 0.90	-
T060 - 015	8.37	-	- 0.27	-
T060 - 016	8.75	0.12	1.76	2.20
T060 - 017	8.32	0.18	- 0.53	0.50
T060 - 018	8.45	-	0.16	-
T060 - 019	8.01	0.07	- 2.18 W	3.58
T060 - 020	8.23	-	- 1.01	-
T060 - 021	8.48	-	0.32	-
T060 - 022	8.66	-	1.28	-
T060 - 023	8.28	0.2	- 0.74	0.64
T060 - 024	8.50	-	0.43	-
T060 - 025	8.53	0.25	0.59	0.41
T060 - 026	8.61	-	1.01	-
T060 - 027	8.44	0.31	0.11	0.06

Summary statistics	Sample
No. of results	27
Robust average (x*) (g/100g)	8.420
Robust standard deviation (s*) g/100g)	0.188

- Note
1. “A” = “Action signal” If $|Z| \geq 3$
 2. “W” = “Warning signal” If $2 < |Z| < 3$
 3. MU* = Measurement Uncertainty

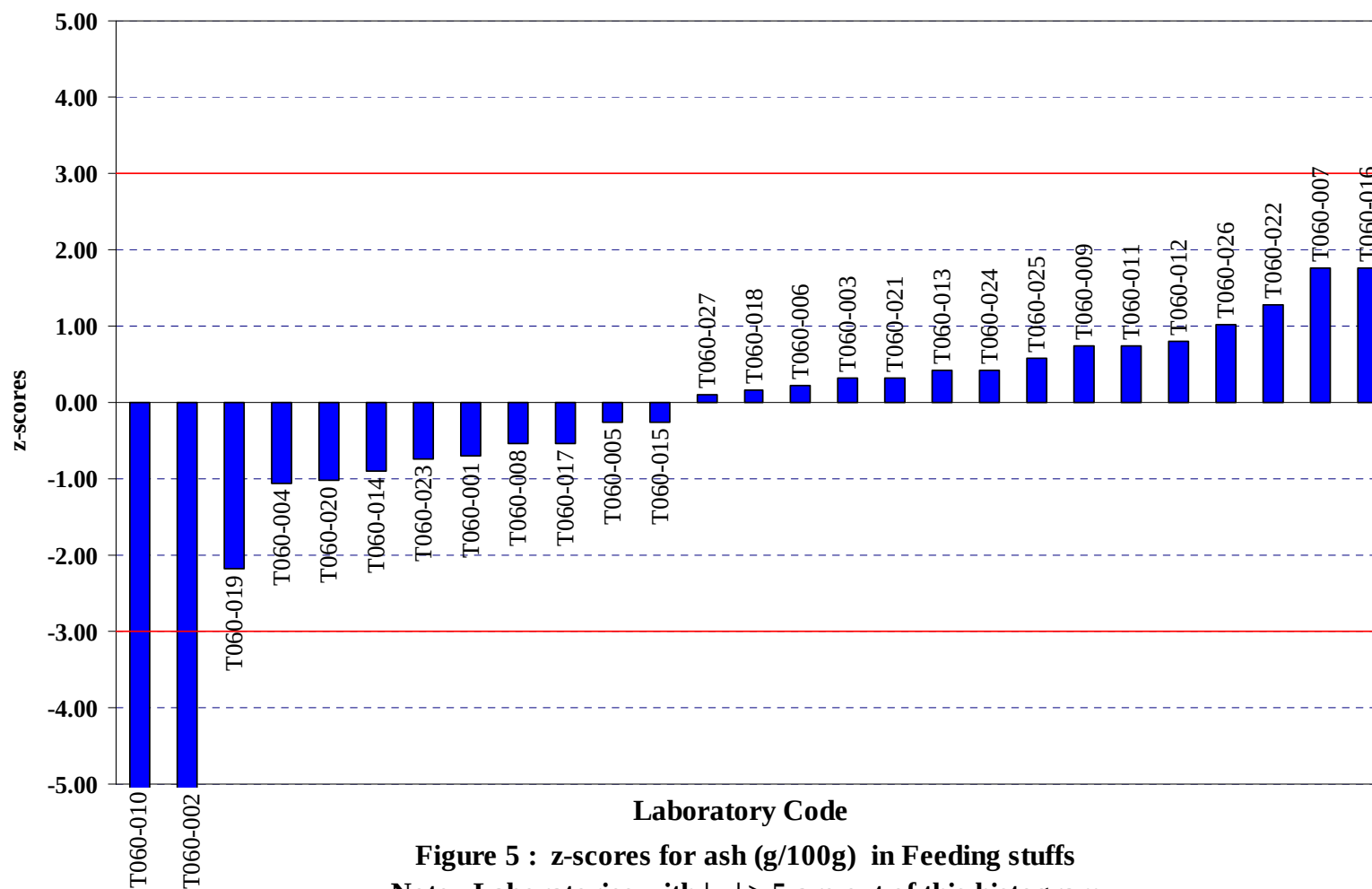


Figure 5 : z-scores for ash (g/100g) in Feeding stuffs
Note Laboratories with $|z| > 5$ are out of this histogram

Table 9 : Results and z-scores for calcium in feeding stuffs

Laboratory Code No.	Results (g/100g)		z-score	E _n number
	Value	MU*		
T060 - 001	1.08	0.03	0.69	1.02
T060 - 002	1.30	-	4.13 A	-
T060 - 003	0.99	-	- 0.72	-
T060 - 004	1.11	0.10	1.16	0.71
T060 - 005	1.12	0.06	1.31	1.25
T060 - 006	1.08	-	0.69	-
T060 - 007	1.02	0.04	- 0.25	0.32
T060 - 008	0.962	0.047	- 1.16	1.32
T060 - 009	1.03	-	-0.09	-
T060 - 010	0.42	-	- 9.63 A	-
T060 - 011	1.04	0.13	0.06	0.03
T060 - 012	1.03	0.05	- 0.09	0.10
T060 - 013	1.07	0.01	0.53	1.05
T060 - 014	0.98	-	- 0.88	-
T060 - 015	1.03	-	- 0.09	-
T060 - 016	1.16	0.10	1.94	1.19
T060 - 017	1.00	0.12	- 0.56	0.29
T060 - 018	0.94	-	- 1.50	-
T060 - 019	0.93	0.17	- 1.66	0.61
T060 - 020	1.18	-	2.25 W	-
T060 - 021	1.04	-	0.06	-
T060 - 022	1.03	-	- 0.09	-
T060 - 023	1.02	0.06	- 0.25	0.24
T060 - 024	1.00	-	- 0.56	-
T060 - 025	1.07	0.10	0.53	0.32
T060 - 026	1.07	0.07	0.53	0.44
T060 - 027	1.02	0.04	- 0.25	0.32

Summary statistics	Sample
No. of results	27
Robust average (x*) (g/100g)	1.036
Robust standard deviation (s*) g/100g)	0.064

- Note
1. “A” = “Action signal” If $|Z| \geq 3$
 2. “W” = “Warning signal” If $2 < |Z| < 3$
 3. MU* = Measurement Uncertainty

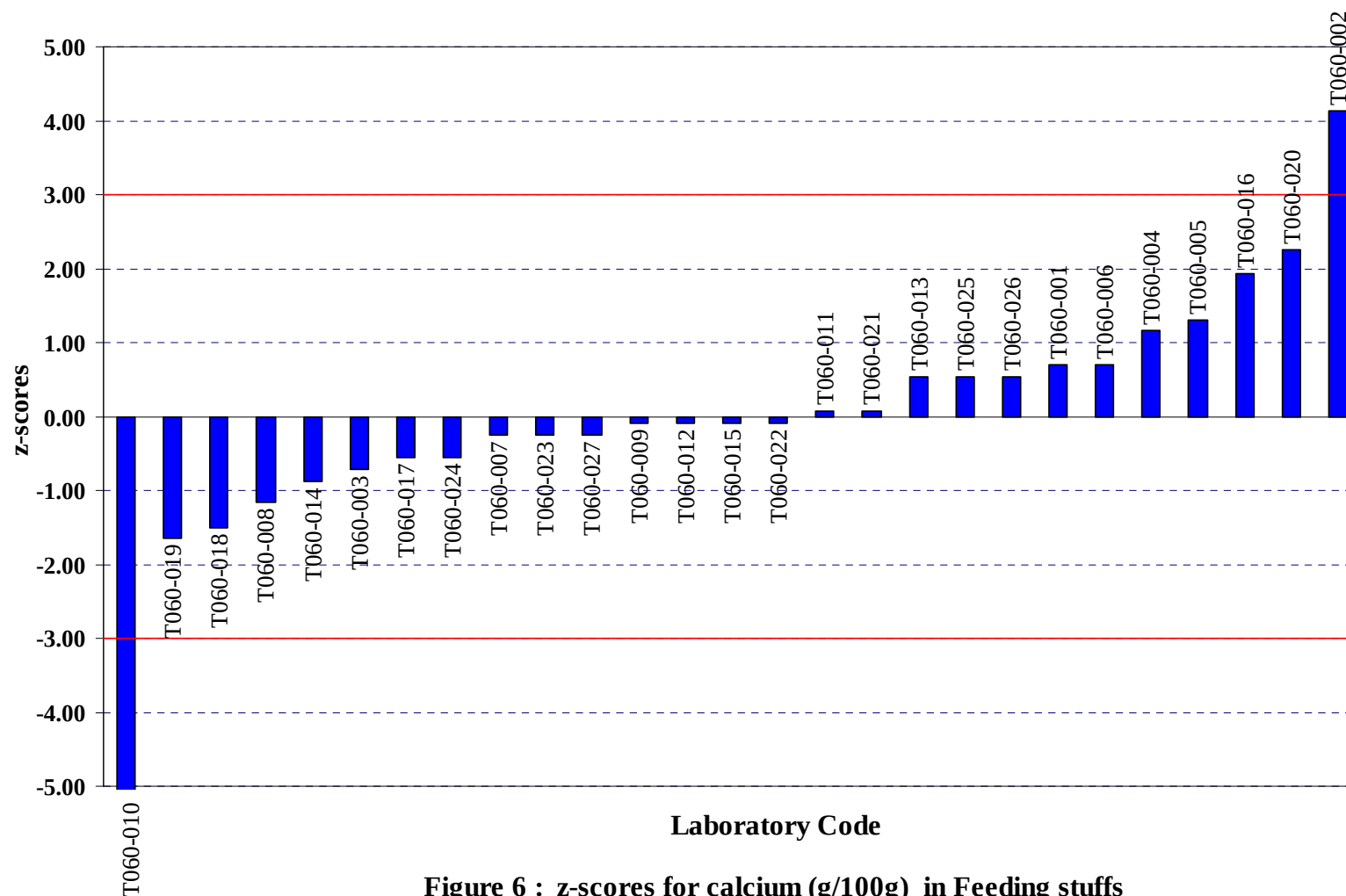


Figure 6 : z-scores for calcium (g/100g) in Feeding stuffs

Note Laboratory with $|z| > 5$ is out of this histogram

Table 10 : Results and z-scores for phosphorus in feeding stuffs

Laboratory Code No.	Results (g/100g)		z-score	E _n number
	Value	MU*		
T060 - 001	0.86	0.05	- 0.15	0.10
T060 - 002	0.87	-	0.15	-
T060 - 003	0.90	-	1.03	-
T060 - 004	0.87	0.02	0.15	0.19
T060 - 005	0.85	0.05	- 0.44	0.29
T060 - 006	1.12	-	7.50 A	-
T060 - 007	0.88	0.03	0.44	0.44
T060 - 008	0.814	0.033	- 1.50	1.38
T060 - 009	0.88	-	0.44	-
T060 - 010	0.36	-	- 14.85 A	-
T060 - 011	0.88	0.072	0.44	0.20
T060 - 012	0.849	-	- 0.47	-
T060 - 013	0.85	0.02	- 0.44	0.58
T060 - 014	0.93	-	1.91	-
T060 - 015	0.87	-	0.15	-
T060 - 016	0.85	0.02	- 0.44	0.58
T060 - 017	1.06	0.04	5.74 A	4.51
T060 - 018	0.75	-	- 3.38 A	-
T060 - 019	0.44	0.005	- 12.50 A	24.85
T060 - 020	1.00	-	3.97 A	-
T060 - 021	0.84	-	-0.74	-
T060 - 022	0.787	0.046	- 2.29 W	1.60
T060 - 023	0.89	0.06	0.74	0.40
T060 - 024	0.87	-	0.15	-
T060 - 025	0.81	0.10	- 1.62	0.54
T060 - 026	0.86	-	- 0.15	-
T060 - 027	0.88	0.04	0.44	0.35

Summary statistics	Sample
No. of results	27
Robust average (x*) (g/100g)	0.865
Robust standard deviation (s*) g/100g)	0.034

- Note
1. “A” = “Action signal” If $|Z| \geq 3$
 2. “W” = “Warning signal” If $2 < |Z| < 3$
 3. MU* = Measurement Uncertainty

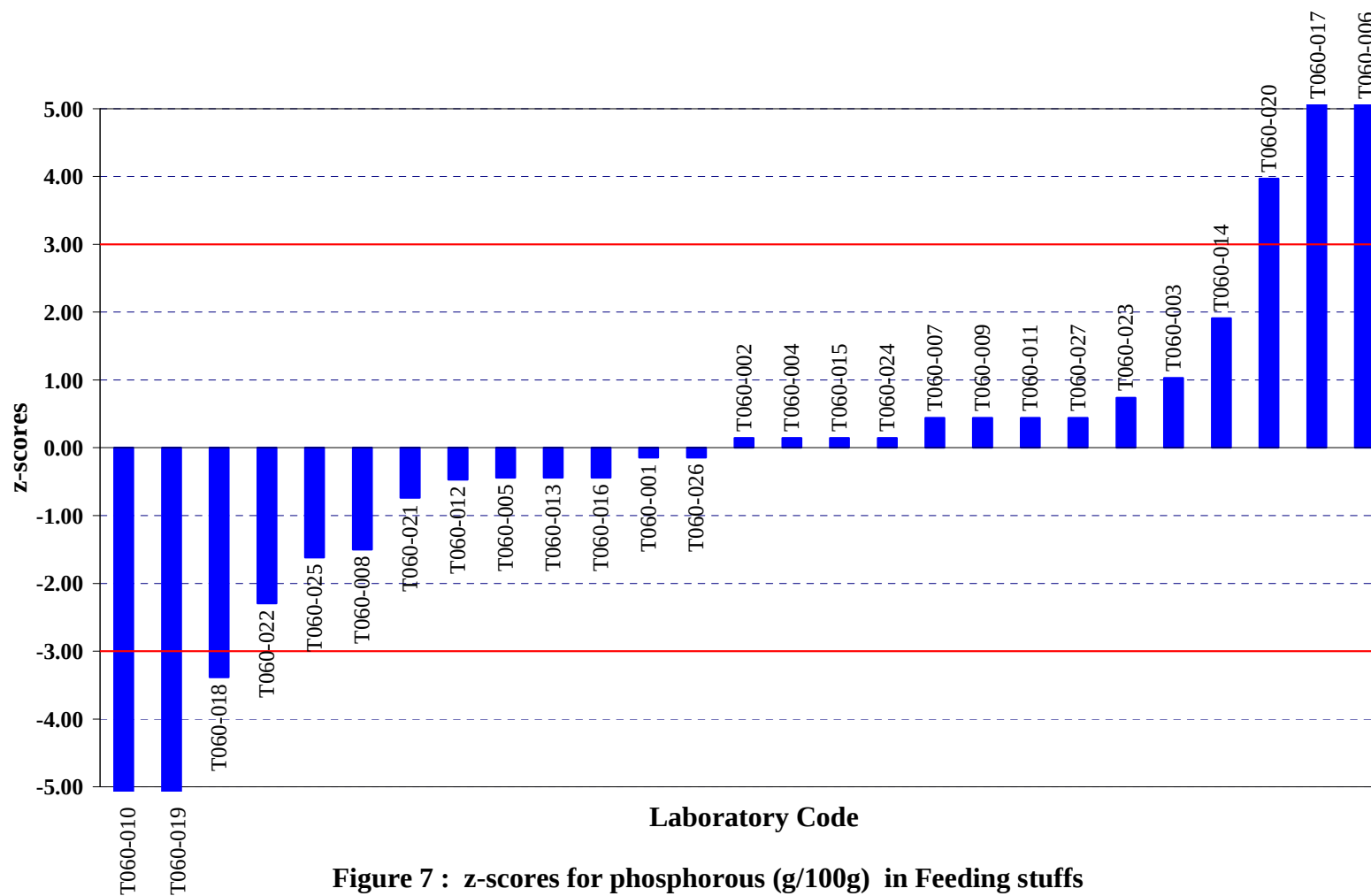


Figure 7 : z-scores for phosphorous (g/100g) in Feeding stuffs

Note Laboratories with $|z| > 5$ are out of this histogram

Table 11 : Moisture test conditions for each laboratory

Laboratory Code No.	Reference	Weight (g)	Temp. (°C)	Drying Times (hrs.)
T060 - 001	AOAC 930.15, 925.10	1	105	16
T060 - 002	Oven drying method	5	135	1
T060 - 003	ISO 6496	5.0000	103±2	4
T060 - 004	AOAC 18 th Edn. 2005 Chapter 4, p1, 4.1.03	2	103	4
T060 - 005	AOAC Official Method 930.15 Loss on Drying (Moisture) for Feeds	2	135	2
T060 - 006	ISO 6496:1999	5	103	4
T060 - 007	TCVN 4326 : 2001 (ISO 6496:1999)	~4	103±2	8
T060 - 008	AOAC Method 930.15 Loss on Drying	1.0495 & 1.1975	135	2
T060 - 009	Based on ISO 6496 : 1999	2	103-105	4
T060 - 010	ISO 6496 Second Edition 1999-08-01	5.0	103±2	4
T060 - 011	ISO 6496-1999	5	103	4
T060 - 012	ISO 6496	5	103±2	4
T060 - 013	AOAC 930.15, ISO 6496:1999	2.0128, 5.0085	135±2, 103±2	2h±5min, 4h±0.1h
T060 - 014	ISO 6496 : 1999 (E) Animal feeding stuffs-Determination of moisture and other volatile matter content	2	103±2	4±0.1
T060 - 015	STANDARD TABLES OF FOOD COMPOSITION IN JAPAN Fifth Revised and Enlarged Edition -2005	3	135	2
T060 - 016	AOAC 950.46	5.27, 5.66	101	5
T060 - 017	AOAC 930.15	5	135	2
T060 - 018	ISO 6496 : 1999	5.0000	103±2	4
T060 - 019	AOAC (2005), 930.15	2	135	2
T060 - 020	AOAC 930.15	2±0.0001	135±2	2
T060 - 021	Standard tables of food composition in JAPAN Fifth revised enlarged edition	3.0226	135	3
T060 - 022	AOAC Official Methods, 930.15, 2005	2.0	135	2
T060 - 023	TCVN 4326:2001	1-2	105	3-8
T060 - 024	Hot air oven based on ISO 6496 (1999)	2	103±2	4

Table 11 : Moisture test conditions for each laboratory (continued)

Laboratory Code No.	Reference	Weight (g)	Temp. (°C)	Drying Times (hrs.)
T060 - 025	AOAC 930.15	2	135	2
T060 - 026	AOAC 930.15 (2005)	2	135	2
T060 - 027	Method of EISHIN No.13, April 26, 1999, "Analytical Methods for Nutrients on the Standards for Nutrition Labeling", Ministry of Health, Labour and Welfare of Japan.	0.4011~0.4087	135	2

Table 12 : Protein test conditions for each laboratory

Laboratory Code No.	Method	Weight (g)	Acid Titration	Determination method
T060 - 001	AOAC 968.06	0.2	-	Leco, Total combustion
T060 - 002	Duma's method	0.3	-	Duma's method
T060 - 003	ISO 5983-2	1 g for protein 1-30%, 0.5 g for protein 30-50%, 0.3 g for protein > 80 %	0.2 N H ₂ SO ₄	Titrate Method
T060 - 004	AOAC 18 th Edn, 2005. Chapter 4, p3, 4.2.09	1	Yes	-
T060 - 005	AOAC Official Method 976.05 Protein (Crude) in Animal Feed and Pet Food	1	0.1 N HCl	Kjeltec
T060 - 006	ISO/WD 5983-1 (2003)	0.5	0.1 N H ₂ SO ₄	Kjeldahl method
T060 - 007	TCVN 4328 : 2007	0.7-0.8	0.1 N H ₂ SO ₄	Kjeldahl
T060 - 008	AOAC Method 990.03 Protein (Crude) in Animal Feed	0.1235 & 0.1243	N/A	Combustion Analysis
T060 - 009	Based on AOAC (2005), 981.10	0.5	0.2 N HCl	Kjeldahl
T060 - 010	AOAC 17 Edition, 2003 part 4.2.11	0.3	0.1 M HCl	Block digestion method
T060 - 011	ISO 5983-1:2005	0.5	H ₂ SO ₄	Kjeldahl
T060 - 012	ISO-5983-2 (modified)	0.9	HCl	Block digestion

Table 12 : Protein test conditions for each laboratory (continued)

Laboratory Code No.	Method	Weight (g)	Acid Titration	Determination method
T060 - 013	AOAC 984.13	0.5757	0.1 N HCl	Kjeldahl
T060 - 014	In-house method base on ISO 5983-2:2005 (E) Animal feeding stuffs-Determination of nitrogen content and calculation of crude protein content	1	2 N H ₂ SO ₄	Block digestion / steam distillation method
T060 - 015	STANDARD TABLES OF FOOD COMPOSITION IN JAPAN Fifth Revised and Enlarged Edition -2005	1	0.05 mol/L sulfuric acid	Automated Kjeldahl method
T060 - 016	AOAC 992.15	0.14 , 0.12	-	Leco Nitrogen Analyzer
T060 - 017	AOAC 990.03	0.30	-	Leco Nitrogen Analyzer
T060 - 018	AOAC official Methods of Analysis, 16 th ed.,1996 (rev.), part 990.03	0.2500	-	Combustion
T060 - 019	AOAC (2005), 954.01	0.5	0.1 N HCl	Kjeldahl method
T060 - 020	ISO 5983-2	0.5±0.0001	0.2 N HCl	Kjeldahl method (Manual)
T060 - 021	Standard tables of food composition in JAPAN Fifth revised enlarged edition	1.1041	0.05 (mol/l)-Sulfuric acid, factor=1.008	Kjeldahl method
T060 - 022	AOAC Official Methods, 992.15, 2005	0.3	-	Dumas
T060 - 023	TCVN 4328-1 : 2007	0.5-1	H ₂ SO ₄	Kjeldahl
T060 - 024	In-house method based on ISO 5983-2 (2005)	0.5	0.1 N HCl	Kjeldahl method
T060 - 025	AOAC 984.13	0.5	0.1 N HCl	Kjeldahl method
T060 - 026	AOAC 2001.11 (2005)	1	0.1 N HCl	Kjeldahl
T060 - 027	Method of EISHIN No.13, April 26, 1999, "Analytical Methods for Nutrients on the Standards for Nutrition Labeling", Ministry of Health, Labour and Welfare of Japan.	0.3982~0.4156	0.05 mol/L sulfuric acid	Kjeldahl method

Table 13 : Fat test conditions for each laboratory

Laboratory Code No.	Reference	Weight (g)	Extraction solvent	Extraction time (hrs.)
T060 - 001	AOAC 991.36	1	Petroleum ether 60-80 °C	40 min-Soxtec apparatus
T060 - 002	ether extraction method	7.5	Diethyl Ether	5
T060 - 003	AOAC 920.39	1.0000	Petroleum ether	1.35
T060 - 004	AOAC 18 th Edn, 2005. Chapter 4, p39, 4.5.01	2	Diethyl Ether	4
T060 - 005	AOAC Official Method 2003.05. Crude fat in Feed, Cereal Grains and Forages	2	Ethyl Ether	1
T060 - 006	AOAC (2005) 954.02	4	Petroleum ether	16
T060 - 007	TCVN 4331 : 2001	2.5-3	Ete petroleum	12
T060 - 008	AOAC Method 2003.05 Crude Fat in Feeds, Cereal Grains, & Forages	2.0146 & 2.0166	Petroleum Ether	1.5
T060 - 009	AOAC (2005),920.39	2	Petroleum Ether	1
T060 - 010	ISO 6492 First Edition 1999-08-01	5.0	Petroleum Ether	6
T060 - 011	AOAC 920.39	3	Petroleum ether	6
T060 - 012	AOAC 920.39 (modified)	1.5	Petroleum Ether	1.30
T060 - 013	AOAC 920.39	2.0172	Ether	4
T060 - 014	In-house method base on AOAC :2005 18 th Edition, 920.39, Fat (Crude) or Ether Extract in Animal Feed	2	Diethyl ether	4
T060 - 015	STANDARD TABLES OF FOOD COMPOSITON IN JAPAN Fifth Revised and Enlarged Edition -2005	2.5	diethyl ether	16
T060 - 016	AOAC 922.06	1.89, 1.88	Ethyl Ether + Petroleum ether	-
T060 - 017	AOAC 991.36	3.0	Petroleum ether 60-80	2
T060 - 018	AOAC 2003.06 Soxtec, Hexanes	3.0000	Hexanes	60 min
T060 - 019	AOAC (2005),920.39	2	Ethanol, Diethyl ether, Petroleum ether	2 hr. (only extraction step)
T060 - 020	AOAC 920.39	1±0.0001	Petroleum ether	2
T060 - 021	Standard tables of food composition in JAPAN Fifth revised enlarged edition	1.5295	Diethyl Ether	2

Table 13 : Fat test conditions for each laboratory (continued)

Laboratory Code No.	Reference	Weight (g)	Extraction solvent	Extraction time (hrs.)
T060 - 022	AOCS Standard Procedure Am 5-04,2007	1.9	Petroleum ether	1
T060 - 023	TCVN 4331 : 2001	1-2	Diethyl ether	8
T060 - 024	Soxtec based on AOAC 920.39 B,C (2005)	2	Petroleum ether	2
T060 - 025	AOAC 920.39	3	Petroleum Ether	2
T060 - 026	AOAC 2003.05	2	Petroleum ether	1
T060 - 027	Method of EISHIN No.13, April 26, 1999, "Analytical Methods for Nutrients on the Standards for Nutrition Labeling", Ministry of Health, Labour and Welfare of Japan.	0.4145~ 0.4577	diethyl ether	16

Table 14 : Ash test conditions for each laboratory

Laboratory Code No.	Reference	Sample Weight (g)	Ashing Time (hrs.)	Ashing Temp. (°c)
T060 - 001	AOAC 942.05	1	16	550
T060 - 002	Direct ashing method	3	2.5	900
T060 - 003	AOAC 942.05	2.0000	2	600
T060 - 004	AOAC 18 th Edn, 2005. Chapter 4, p8, 4.1.10	1	4	600
T060 - 005	AOAC Official Method 942.05 Ash of Animal Feed	2	2	600
T060 - 006	ISO (5984) (2002) (E)	2	3	550
T060 - 007	TCVN 4327 : 2007	~3	14	550
T060 - 008	AOAC Method 942.05 Ash of Animal Feed	2.0179&2.0233	2	600
T060 - 009	AOAC (2005), 942.05	2	2	600
T060 - 010	ISO 5984 Second Edition 2002-11-15	5.0	3	550
T060 - 011	ISO 5984-2002	5	4	550
T060 - 012	ISO 5984	5	3	550
T060 - 013	AOAC 942.05	2.0385	2	600
T060 - 014	AOAC 2005: 18 th Edition 942.05 Ash of Animal Feed	2	2	600
T060 - 015	STANDARD TABLES OF FOOD COMPOSITON IN JAPAN Fifth Revised and Enlarged Edition -2005	2.5	2	550
T060 - 016	AOAC 923.03	1.33, 2.03	2	600
T060 - 017	AOAC 942.05	3	6 hours minimum usually over night	550
T060 - 018	AOAC official Method of Analysis, 18 th ed,2005, part 942.05	2.0000	2	600
T060 - 019	AOAC (2005),942.05	2	4	600
T060 - 020	AOAC 942.05	2±0.0001	2	600±5
T060 - 021	Standard table of food composition in JAPAN Fifth revised enlarged edition	2.0034	3	550
T060 - 022	ISO 5984, 2002	2	4	550
T060 - 023	TCVN 4327 : 1993	1-2	8-16	450
T060 - 024	ISO 5984 (2002)	5	3	550
T060 - 025	AOAC 942.05	2	4	550

Table 14 : Ash test conditions for each laboratory (continued)

Laboratory Code No.	Reference	Sample Weight (g)	Ashing Time (hrs.)	Ashing Temp. (°c)
T060 - 026	AOAC 942.05	2	5	550
T060 - 027	Method of EISHIN No.13, April 26, 1999,"Analytical Methods for Nutrients on the Standards for Nutrition Labeling", Ministry of Health , Labour and Welfare of Japan.	0.4001~0.4075	5	550

Table 15 : Calcium test conditions for each laboratory

Laboratory Code No.	Reference	Sample Weight (g)	Sample preparation	Analytical Instrument (s)
T060 - 001	AOAC 968.08 D (prep only) followed by colorimetric	1	wet digestion, Dry ashing	Cobas Fara-colorimetric
T060 - 002	-	1	wet digestion	ICP
T060 - 003	AOAC 927.02	2.0000	Dry ashing	Titrate by burette
T060 - 004	AOAC 18 th Edn,2005. Chapter 4, p56-57, 4.8.02	1	Dry ashing	AAS
T060 - 005	AOAC Official Method 968.08 Minerals in Animal feed and Pet Food	2	Dry ashing	AAS
T060 - 006	Based on AOAC (2006) 975.03, 985.35	2	Dry ashing	AA
T060 - 007	TCVN 1526-1 : 2001	~3	Dry ashing	Furnace
T060 - 008	AOAC Method 968.08 Minerals in Animal Feeds - Atomic Absorption Method	2.0001 & 2.0224	Dry ashing	ICP-OES (Varian)
T060 - 009	AOAC (2000), 927.02	2	Dry ashing	Burette (Titration method)
T060 - 010	AOAC 18 Edition, 2005, 927.02	5.0	Dry ashing	titration
T060 - 011	ISO 6490/1-1985	5	Dry ashing	Furnace, analytical balance, Hot plate
T060 - 012	AOAC 968.08 (modified)	1	Dry ashing	Atomic Absorption Spectrophotometer
T060 - 013	AOAC 927.02	2.0385	Dry ashing	burette, pipette, ester
T060 - 014	AOAC official Method 18 th Edition 927.02 Calcium in Animal Feed	2	Dry ashing	-

Table 15 : Calcium test conditions for each laboratory (continued)

Laboratory Code No.	Reference	Sample Weight (g)	Sample preparation	Analytical Instrument (s)
T060 - 015	STANDARD TABLES OF FOOD COMPOSITION IN JAPAN Fifth Revised and Enlarged Edition -2005	2.5	Dry ashing	ICP-AES at the wave length of 317.9 nm
T060 - 016	AOAC 984.27	1.02, 1.02	wet digestion	ICP
T060 - 017	AOAC 975.03Ba (modified)	0.5	Dry ashing	AAS-GBC 908
T060 - 018	OX-MnO ₄ Volumetric Method	2.0000	Dry ashing	Titration
T060 - 019	In-hose Method TM-CH-070 based on AOAC (2005), 968.08	1	wet digestion	AAS
T060 - 020	AOAC 962.01	2±0.0001	Dry ashing	Burette
T060 - 021	Standard tables of food composition in JAPAN Fifth revised enlarged edition	1.0482	Dry ashing	Atomic Absorption Spectrophotometers (HITACHI Z-2000)
T060 - 022	AOAC Official Methods, 985.01, 2005	1.0	Dry ashing	ICP-OES
T060 - 023	AOAC 2002 (935.13)	1-2	Dry ashing	Burette
T060 - 024	AOAC 935.13 (2005)	0.5	wet digestion	ICP-OES
T060 - 025	AOAC 965.09	2	wet digestion	AAS
T060 - 026	ISO 6896 (2000)	2	Dry ashing	Flame AAS
T060 - 027	Method of EISHIN No.13, April 26, 1999, "Analytical Methods for Nutrients on the Standards for Nutrition Labeling", Ministry of Health, Labour and Welfare of Japan	1.007~1.080	Dry ashing	ICP Atomic Emission Spectrometry

Table 16 : Phosphorus test conditions for each laboratory

Laboratory Code No.	Reference	Sample Weight (g)	Sample preparation	Analytical Instrument (s)	Wavelength (nm)
T060 - 001	AOAC 968.08 D (prep only) followed by colorimetric	1	wet digestion, Dry ashing	Shimadzu Spectrophotometer	680
T060 - 002	-	1	wet digestion	ICP (Inductively Coupled Plasma)	178.287
T060 - 003	AOAC 956.17	2.0000	Dry ashing	UV-1800 (spectrophotometer)	400
T060 - 004	AOAC 18 th Edn, 2005. Chapter 4, p61, 4.8.14	1	Dry ashing	UV/Visible Spectrophotometer	400
T060 - 005	AOAC Official Method 965.17 Phosphorus in Animal feed and Pet Food	2	Dry ashing	UV-spectrophotometer	420
T060 - 006	Based on AOAC (2005) 965.17, 965.05	1	Dry ashing	spectrophotometer	830
T060 - 007	TCVN 1525 : 2001	~3	Dry ashing	UV-VIS	430
T060 - 008	AOAC Method 968.08 Minerals in Animal Feeds-Atomic Absorption Method	2.0001&2.0224	Dry ashing	ICP-OES (Varian)	
T060 - 009	Based on AOAC (2000), 965.17	-	Dry ashing	Spectrophotometer	400
T060 - 010	AOAC 18 Edition, 2005, 965.17	5.0	Dry ashing	Spectrophotometer	400
T060 - 011	AOAC 965.17	5	Dry ashing	Furnace, analytical balance, Hot plate, spectrophotometer	430
T060 - 012	AOAC 965.17 (modified)	1	Dry ashing	Spectrophotometer	400
T060 - 013	AOAC 965.17	2.0385	Dry ashing	UV-1800 (Shimadzu)	520
T060 - 014	AOAC official Method 18 th Edition 969.10 Chlorine (Soluble) in Animal Feed/ Potentiometric Method	2	Dry ashing	UV-VIS Spectrophotometer	400
T060 - 015	STANDARD TABLES OF FOOD COMPOSITON IN JAPAN Fifth Revised and Enlarged Edition -2005	2.5	Dry ashing	ICP - AES	214.9
T060 - 016	AOAC 962.02	1.08, 1.13	Dry ashing	N/A	
T060 - 017	AOAC 975.03 Ba (modified)+ 965.17	0.5	Dry ashing	UV-VIS Spectro	420
T060 - 018	Photometric Method	2.0000	Dry ashing	Spectrophotometer	646

Table 16 : Phosphorus test conditions for each laboratory (continued)

Laboratory Code No.	Reference	Sample Weight (g)	Sample preparation	Analytical Instrument (s)	Wavelength (nm)
T060 - 019	AOAC (2005), 965.17	2	Dry ashing	Spectrophotometer	400
T060 - 020	AOAC 965.17	2±0.0001	Dry ashing	Spectrophotometer	400
T060 - 021	Standard tables of food composition in JAPAN Fifth revised enlarged edition	1.0482	Dry ashing	Absorption spectrophotometer (SHIMAZU uv-1800)	410
T060 - 022	AOAC Official Methods, 965.17,2005	1.0	Dry ashing	Spectrophotometer	400
T060 - 023	AOAC (995.11) 2002	1-2	Dry ashing	(Spectrophotometer)	823
T060 - 024	AOAC 935.13 (2005)	0.5	wet digestion	ICP-OES	
T060 - 025	AOAC 958.01	2	Dry ashing	Spectrometry C	400
T060 - 026	AOAC 965.17 (2005)	2	Dry ashing	Spectrophotometer	400
T060 - 027	Method of EISHIN No.13, April 26, 1999, "Analytical Methods for Nutrients on the Standards for Nutrition Labeling", Ministry of Health, Labour and Welfare of Japan.	1.007~1.080	Dry ashing	ICP Atomic Emission Spectrometry	

APPENDIX III

Diagrams of Testing results related with measurement uncertainty of laboratories

Figure 8 Testing results on Moisture (Temp 103 ± 2 °C) related with measurement uncertainty of laboratories

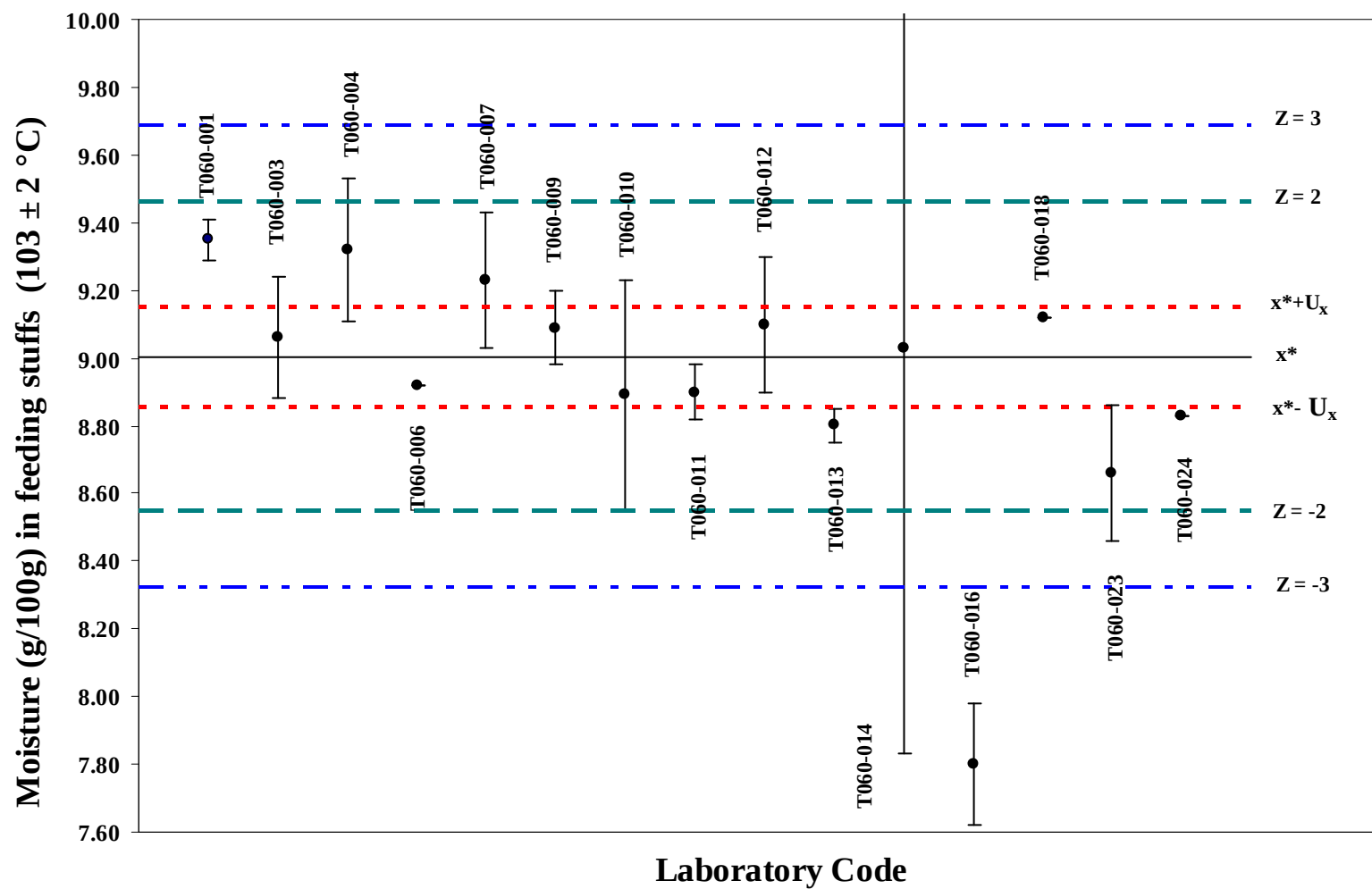


Figure 9 Testing results on Moisture (Temp 135 ± 2 °C) related with measurement uncertainty of laboratories

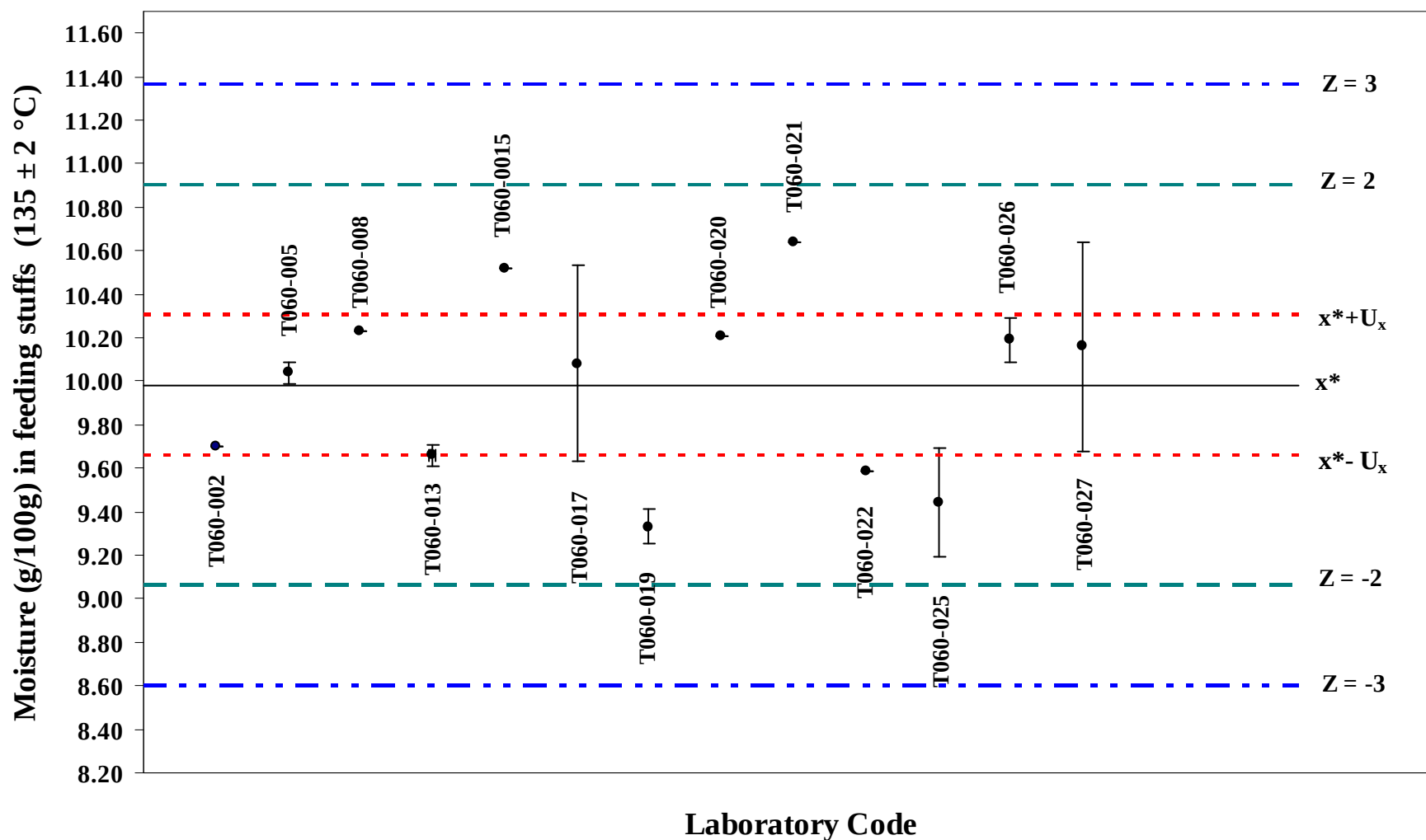


Figure 10 Testing results on Protein related with measurement uncertainty of laboratories

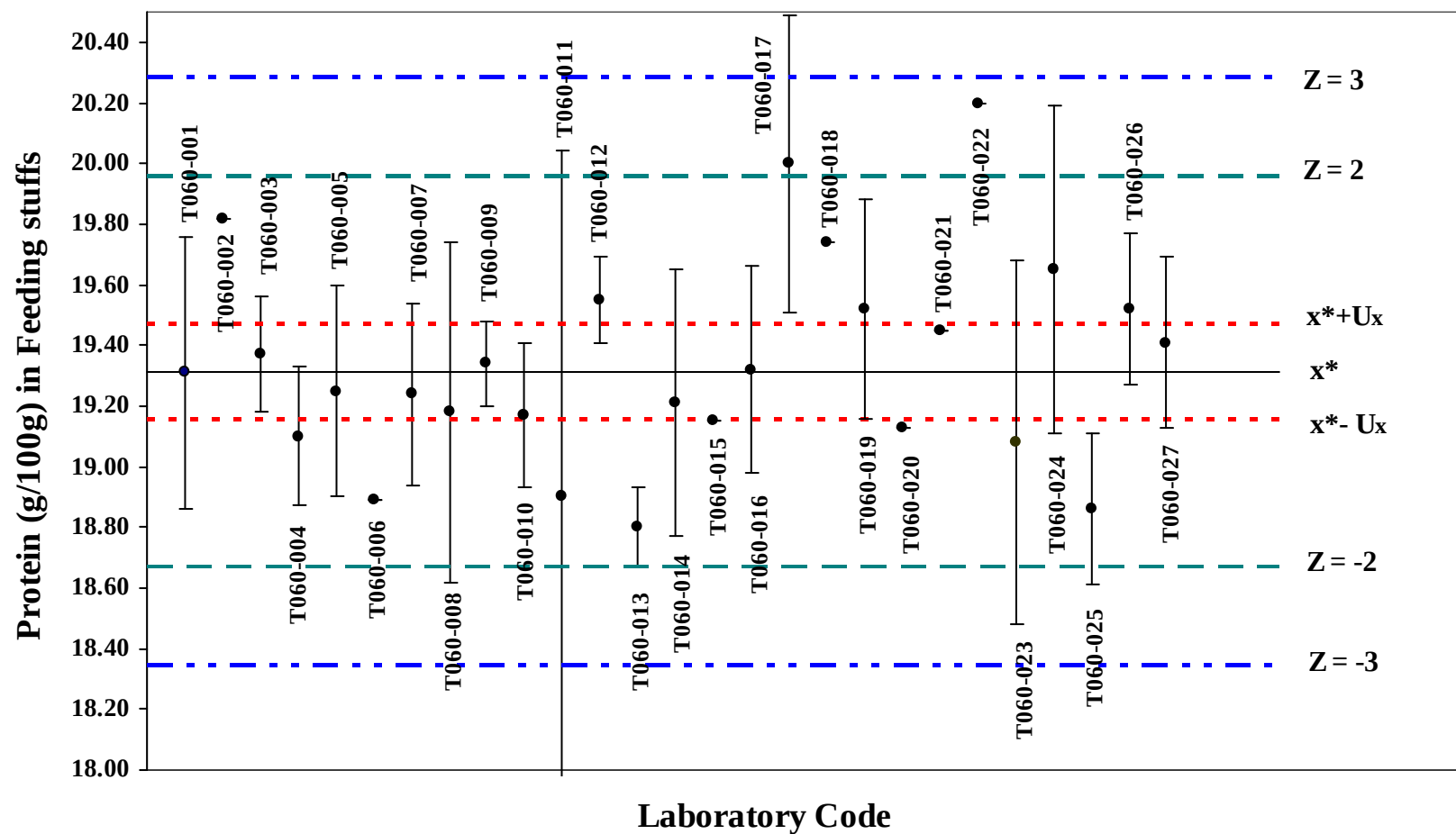


Figure 11 Testing results on Fat related with measurement uncertainty of laboratories

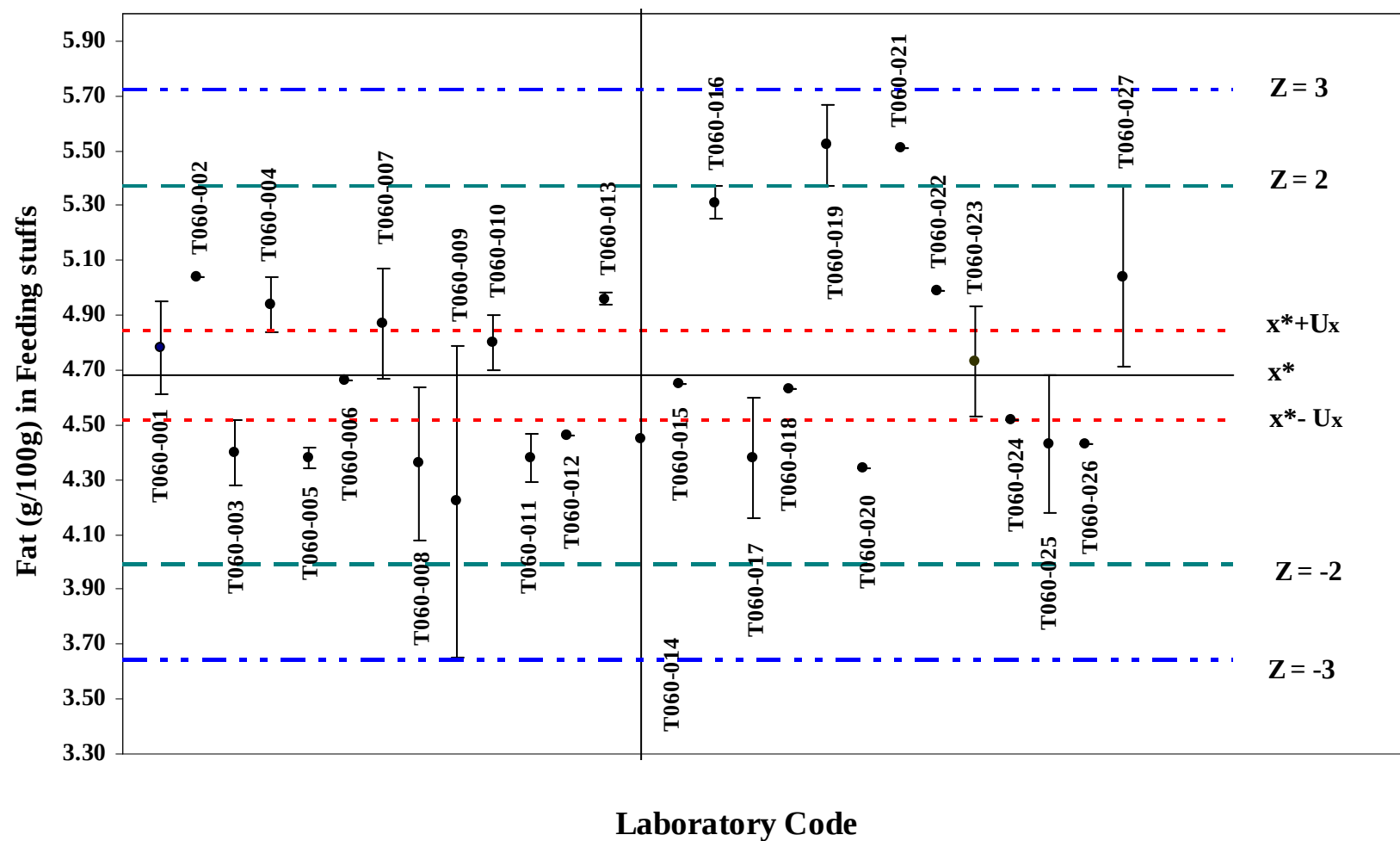


Figure 12 Testing results on Ash related with measurement uncertainty of laboratories

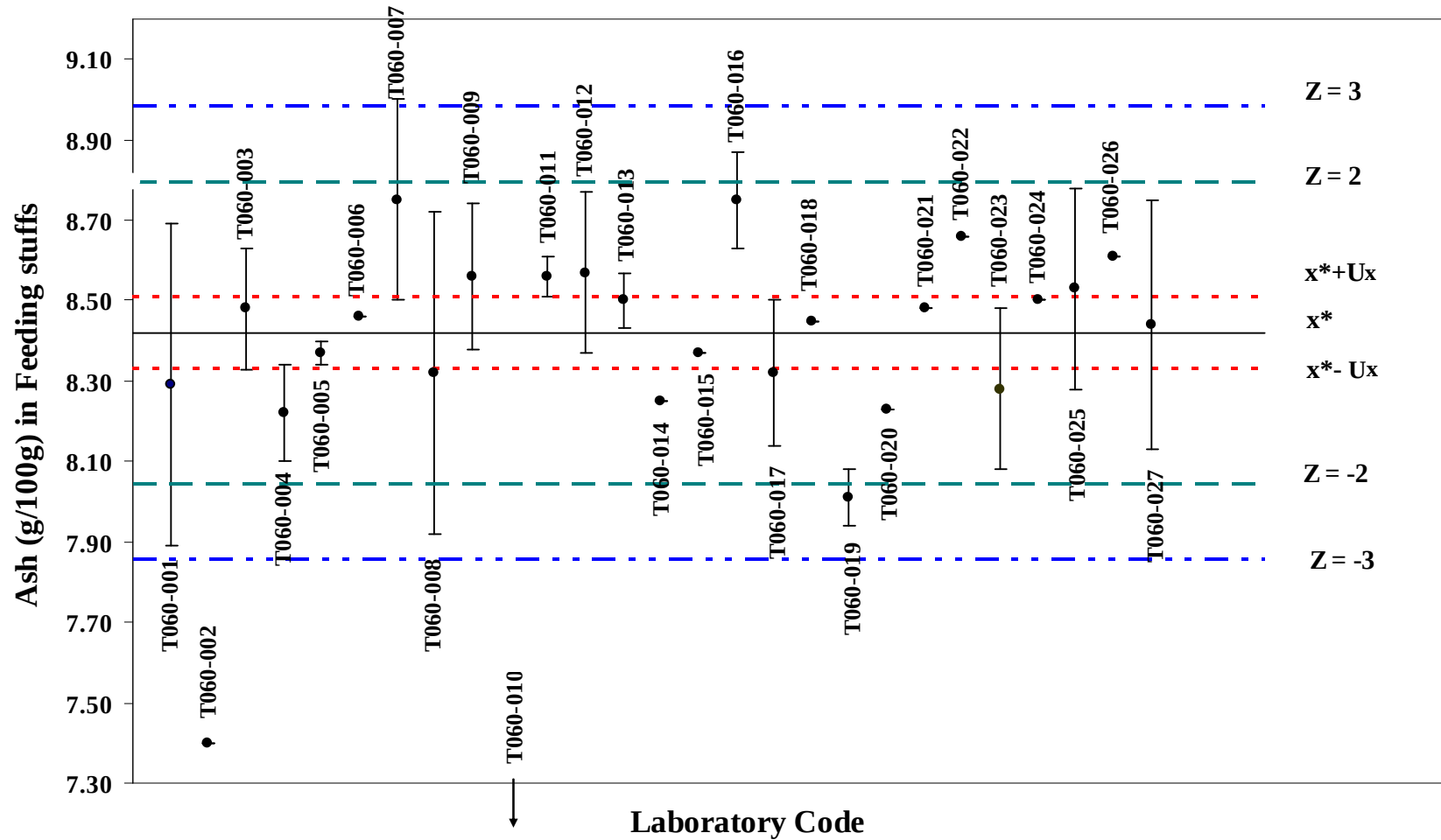


Figure 13 Testing results on Calcium related with measurement uncertainty of laboratories

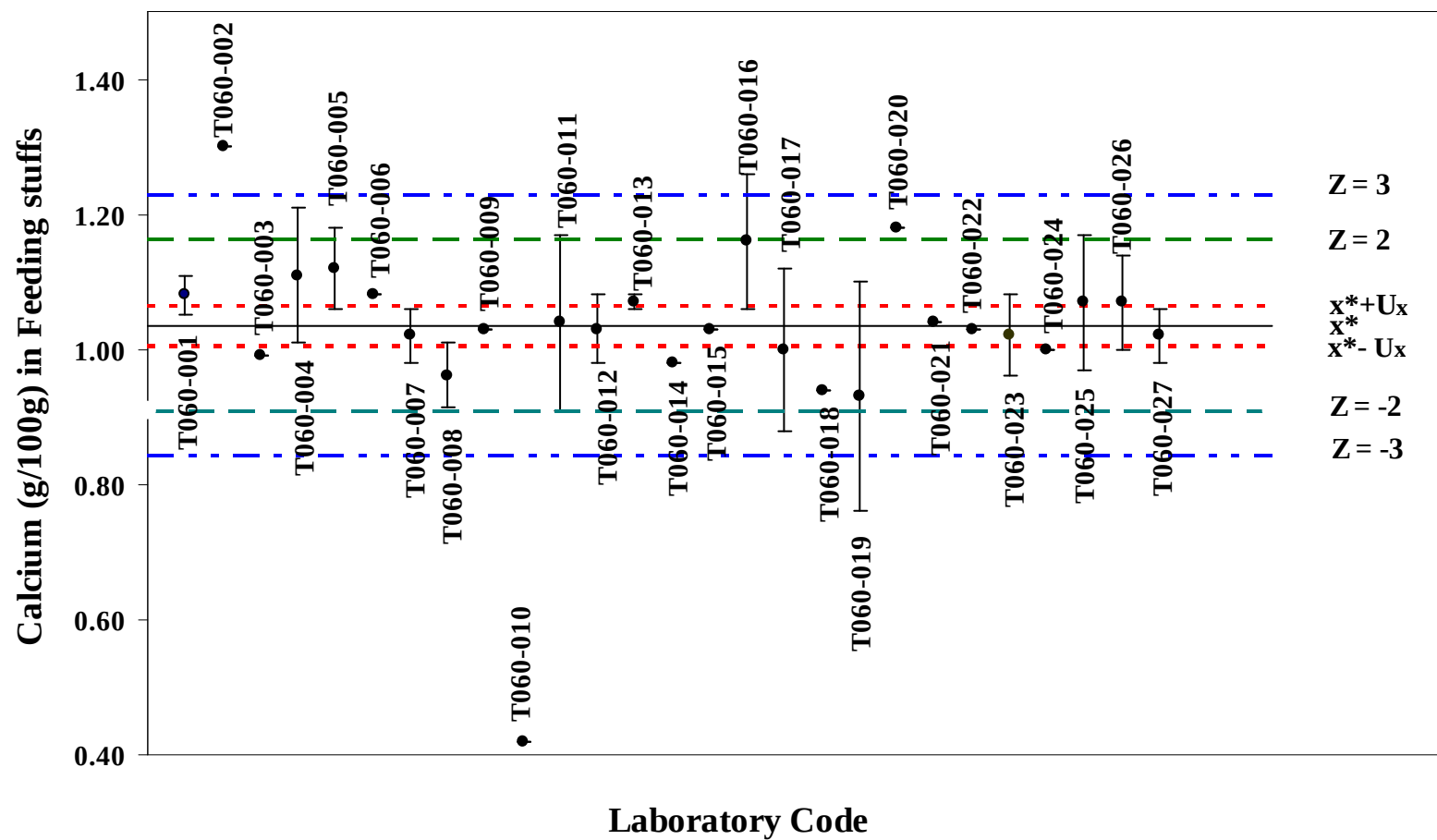
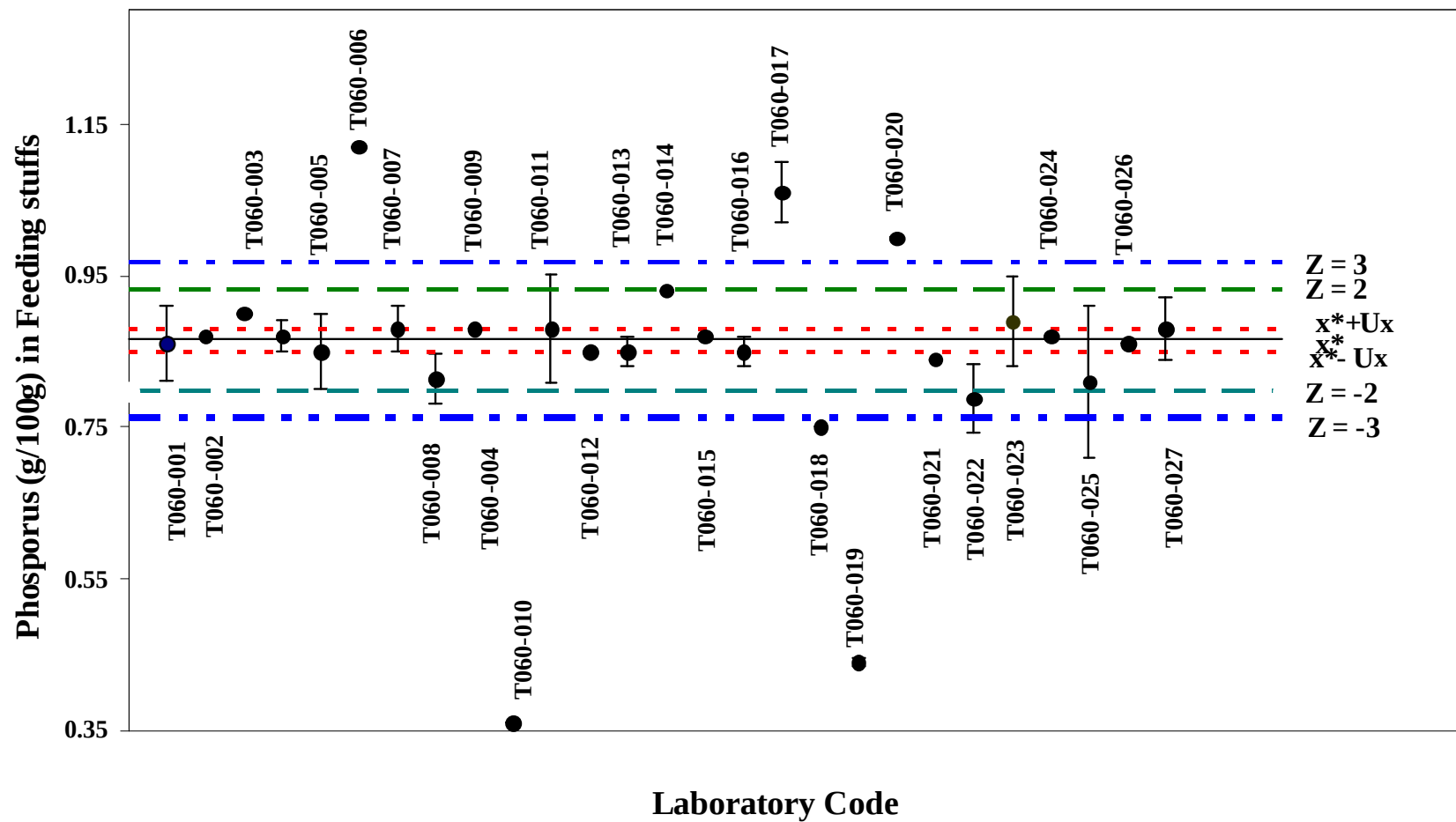


Figure 14 Testing results on Phosphorus related with measurement uncertainty of laboratories



APPENDIX IV

Histogram

Figure 15: Histogram of the moisture content (27 laboratory results)

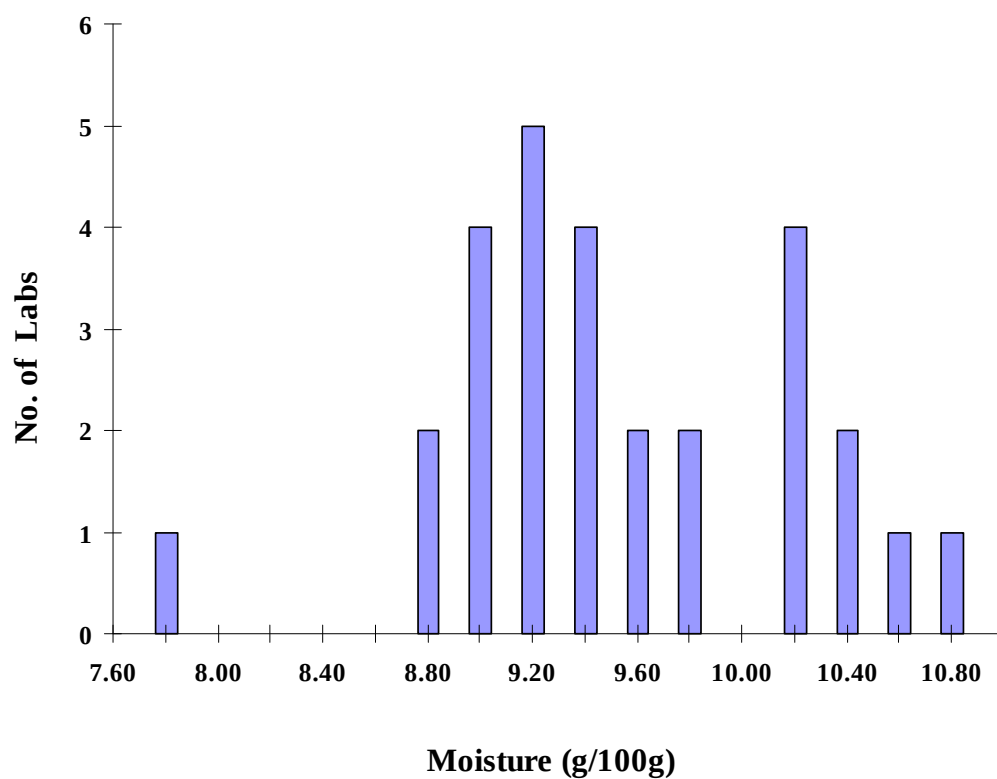


Figure 16: Histogram of the Protein content (27 laboratory results)

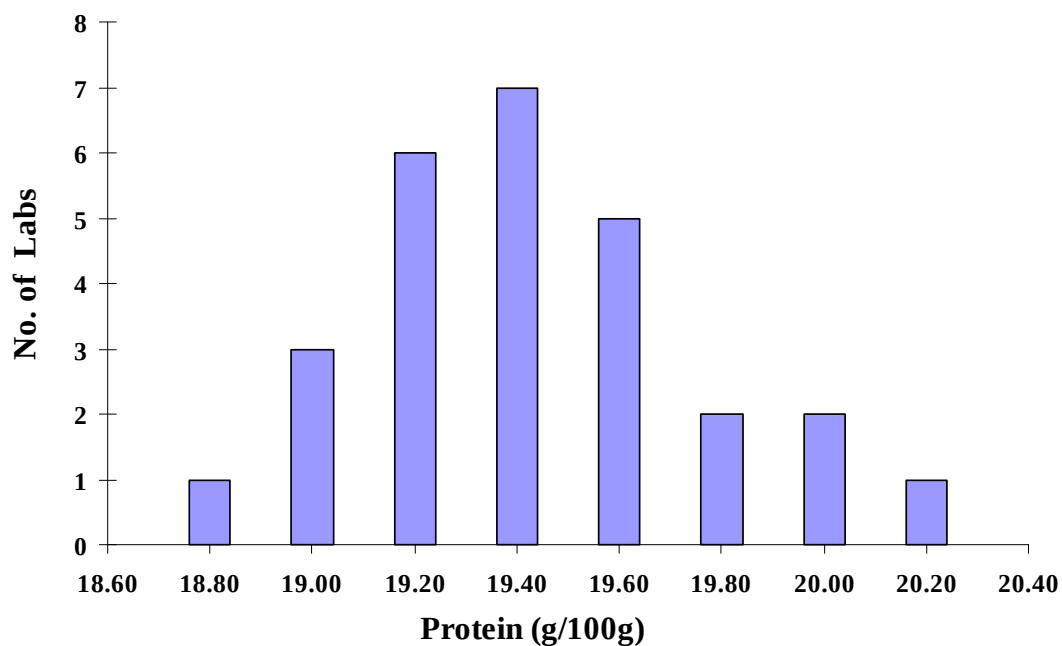


Figure 17: Histogram of the Fat content (27 laboratory results)

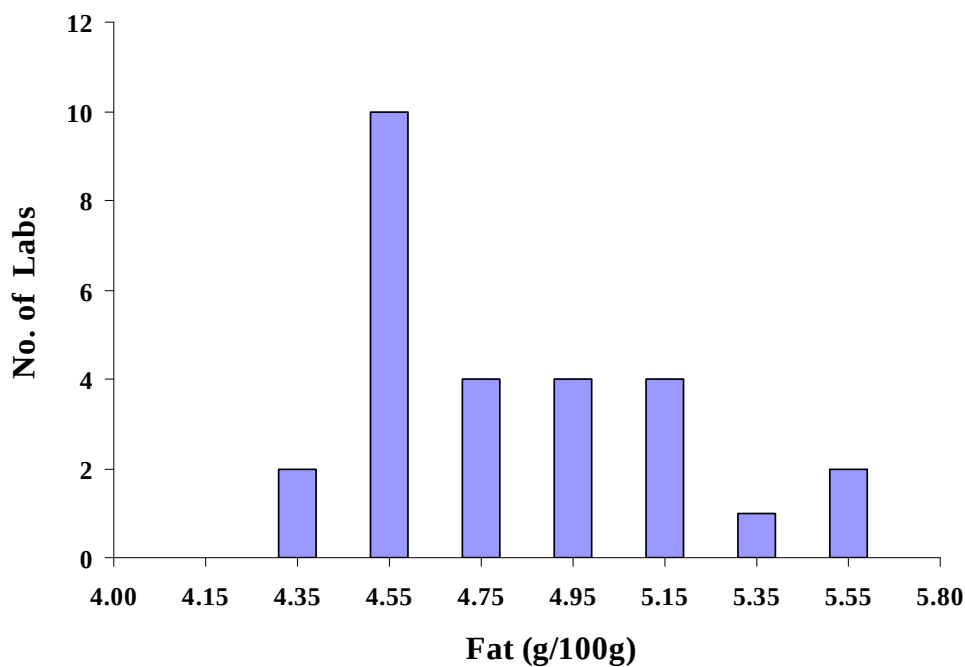


Figure 18 : Histogram of the Ash content (27 laboratory results)

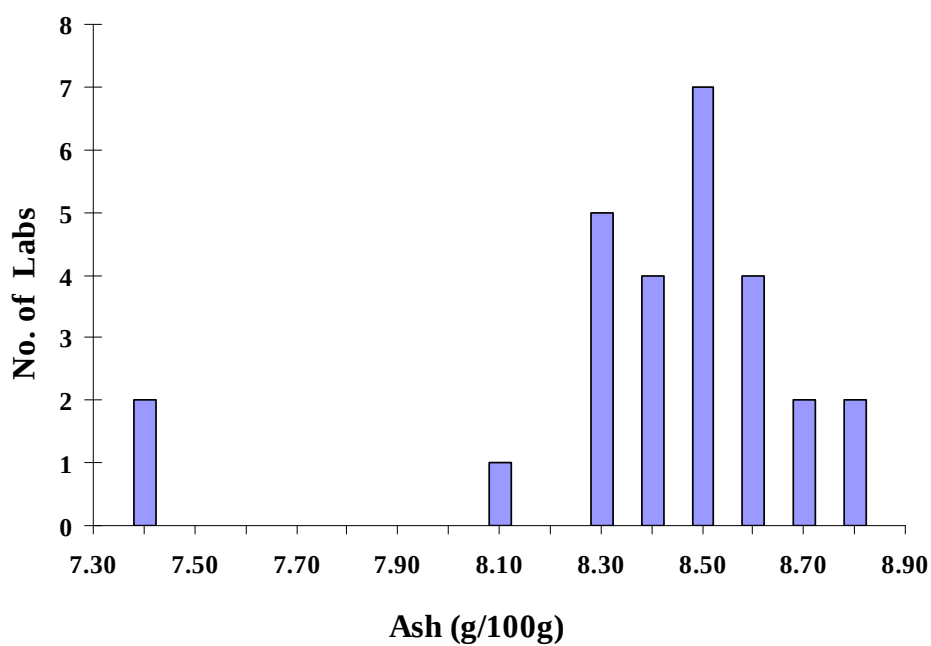


Figure 19 : Histogram of the Calcium content (27 laboratory results)

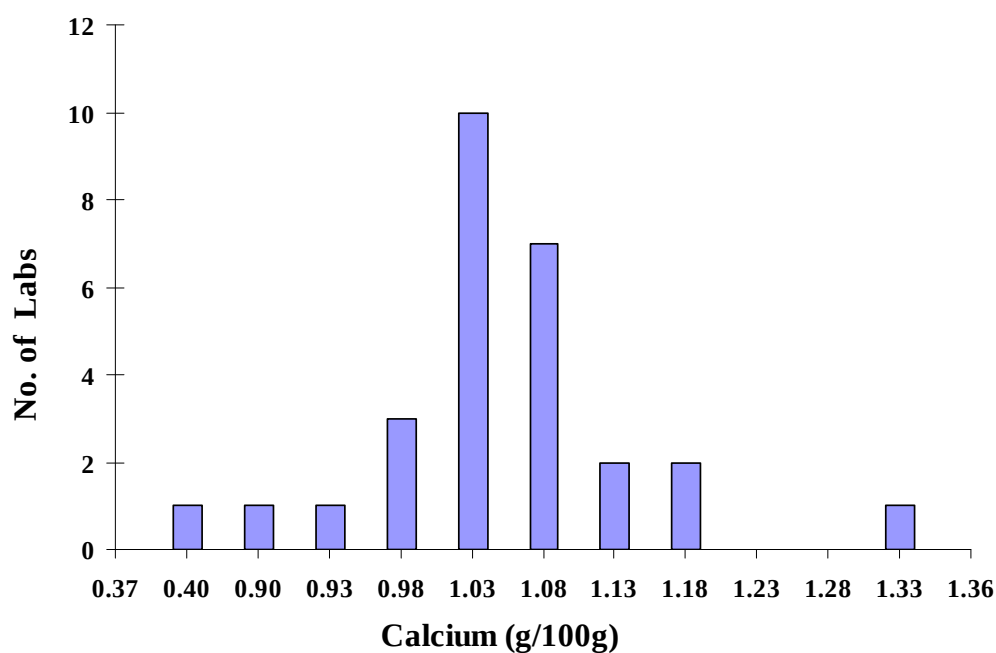
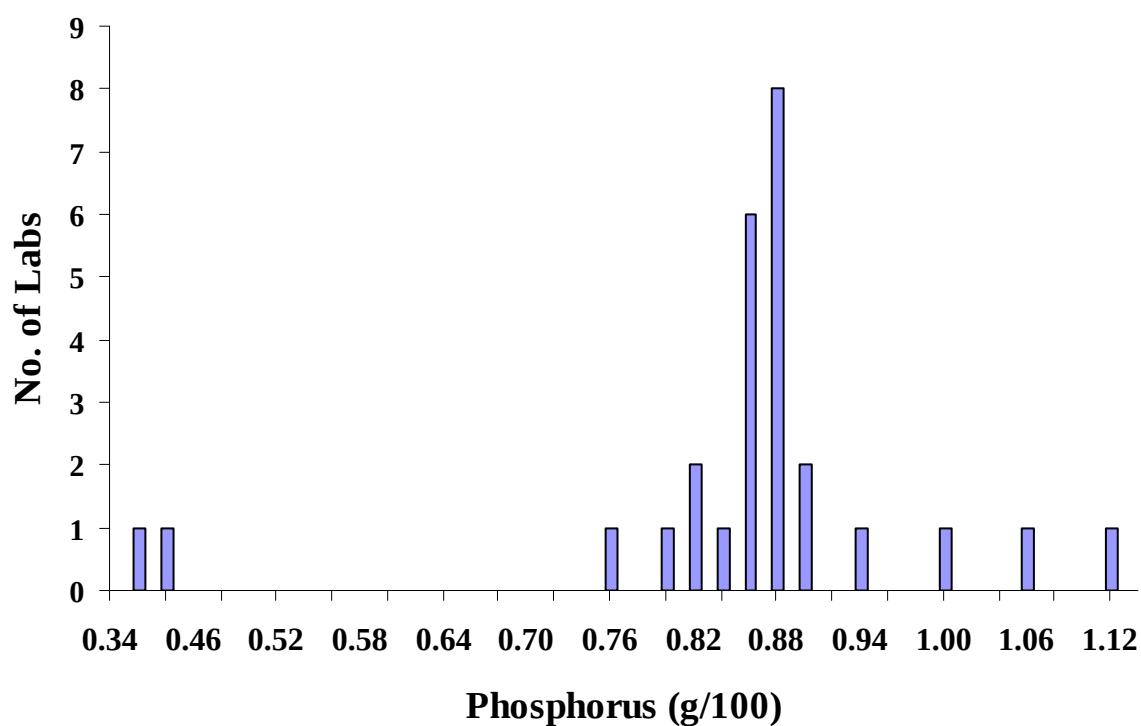


Figure 20: Histogram of the Phosphorus content (27 laboratory results)



APPENDIX V

INSTRUCTIONS TO ACCREDITATION BODIES
PARTICIPATING LABORATORIES NOMINATION FORM
RECEIPT FORM FOR ACCREDITATION BODIES
INSTRUCTIONS TO PARTICIPATING LABORATORIES
RECEIPT FORM
RESULTS PROFORMA



APLAC T060: Feeding Stuffs Proficiency Testing Program

INSTRUCTIONS TO ACCREDITATION BODIES

1. INTRODUCTION

The APLAC T060 : Feeding Stuffs is organized by Bureau of Laboratory Accreditation (BLA), Department of Science Service (DSS), Thailand. The objective of this proficiency testing program is to evaluate the competence of laboratories for quantitative testing of moisture, protein, fat, ash, calcium and phosphorus in feeding stuffs.

2. SAMPLES

Each laboratory is supplied with one feeding stuff sample about 40 g packed in laminated sealed bag. Upon receipt, AB should check the samples (number of bags, physical conditions, etc) and kindly complete the enclosed "RECEIPT FORM" (for Accreditation Bodies) and send to email raviwan@dss.go.th or fax to +662 201-7239.

3. SAMPLE DISTRIBUTION

AB should distribute samples to their nominated laboratories within one week of receipt. The samples shall be stored at room temperature (around 25° C) before dispatch.

4. RESULTS

The AB shall inform their participating laboratories to send results and submit to the organizer DSS **no later than 17 September 2009** at raviwan@dss.go.th.

5. ENQUIRIES

Please contact BLA-DSS for further enquiries at raviwan@dss.go.th.



APLAC T060: Feeding Stuffs Proficiency Testing Program

PARTICIPATING LABORATORIES NOMINATION FORM

Laboratory Name:	
Contact Person:	
Physical Address:	
Phone:	
Fax:	
E-mail:	
Accredited for test:	Y / N

Laboratory Name:	
Contact Person:	
Physical Address:	
Phone:	
Fax:	
E-mail:	
Accredited for test:	Y / N

Laboratory Name:	
Contact Person:	
Physical Address:	
Phone:	
Fax:	
E-mail:	
Accredited for test:	Y / N

From:

(ACCREDITATION BODY)

Name : _____

Organisation : _____

Address : _____

Email : _____

Fax : _____



APLAC T060: Feeding Stuffs Proficiency Testing Program

RECEIPT FORM FOR ACCREDITATION BODIES

In order to monitor the progress of this proficiency testing program, we kindly ask each participating accreditation body to fill out this Receipt Form and send it by fax or e-mail to:

Dr. Raviwan Artsamang
Department of Science Service
Fax: +662 2017239
Phone: +662 2017333
E-mail: raviwan@dss.go.th

The APLAC T060 Samples were received on :

Comments

.....

.....

Name of Accreditation Body :

Country : :

Contact Person :

E-mail :

Thank you in advance for your cooperation.



APLAC T060: Feeding Stuffs Proficiency Testing Program

RECEIPT FORM FOR PARTICIPATING LABORATORIES

In order to monitor the progress of this proficiency testing program, we kindly ask each participating laboratory to fill out this Receipt Form and send it by fax or e-mail to:

Dr. Raviwan Artsamang
Department of Science Service
Fax: +662 2017239
Phone: +662 2017333
E-mail: raviwan@dss.go.th

The APLAC T060 Samples were received on :

Comments:

.....

.....

Participating Laboratory:.....

Accreditation Body:

Country :

Contact Person :

E-mail :

Thank you in advance for your cooperation.



APLAC T060: Feeding Stuffs

Proficiency Testing Program

INSTRUCTIONS TO PARTICIPATING LABORATORIES

To ensure that results from this program can be analysed properly, participants are asked to adhere to the following instructions.

1. SAMPLE

- (a) Participants will receive one sample bag of about 40 g of feeding stuffs. The sample supplies to each laboratory is packed into laminated bag.
- (b) Store your sample in the original package at room temperature around 25°C (away from heat) until testing commences. After opening, the sample has to be used immediately.

2. ANALYSIS

- (a) Participants are required to determine the concentration of moisture, protein, fat, ash, calcium and phosphorus in feeding stuffs.
- (b) Participants should use routine test methods (could be accredited, in-house, modified, etc.) which would normally be used to test customer supplied samples and record the method used on the result sheet. Participants are requested to fill Method Information in Part II of RESULTS PROFORMA.

3. RESULTS

- (a) Report the moisture, protein, fat, ash, calcium and phosphorus in g/100g (as received sample), express the results to two decimal places.
- (b) Report the measurement uncertainty for each result as expanded uncertainty (g/100g) at 95% confident level.
- (c) Record the requested information in the results sheet (weight sample, temperature and drying time, ashing time and ashing temp etc.)

4. DOCUMENT TO BE SUBMITTED

Submit the completed RESULTS PROFORMA (Part I and Part II) to your accreditation body, and to Department of Science Service (DSS) on or **before 17 September 2009** to email raviwan@dss.go.th.

5. ENQUIRES

Additional information may be obtained from:

Dr. Raviwan Artsamang
Department of Science Service
75/7 Rama 6 Road Rachathewi District,
Bangkok 10400 Thailand
E-mail: raviwan@dss.go.th
Phone: +662 2017333 Fax: +662 2017239



APLAC T060: Feeding Stuffs Proficiency Testing Program

RESULTS PROFORMA

< Both Part I & II shall be completed by Participating Laboratory >

Laboratory Code Number : Date of Analysis :

Name of Laboratory :

Part I : Analytical Results

Test	Result (g/100g)	Expanded Measurement Uncertainty (g/100g)*
Moisture		
Protein (N x 6.25)		
Fat		
Ash		
Calcium (Ca)		
Phosphorus (P)		

* Confidence level ~ 95 %

Report values to 2 significant figure or 2 decimal places, whichever is appropriate

Note : It is the responsibility of the participants to avoid collusion and falsification of result so as to ensure a reliable and effective assessment to be made by this proficiency testing programme



APLAC T060: Feeding Stuffs Proficiency Testing Program

RESULTS PROFORMA (Continued)

Part II : Method Information

1. Moisture

Is the method used Accredited? ☐ YES ☐ NO

Reference :

Sample weight (g) :

Oven temperature (°C) :

Duration time (hours) :

Desiccator used to cool the sample? ☐ YES ☐ NO

In case we have to separate the data in two groups.

☐ group I : drying temperature 103 ± 2 °C

☐ group II : drying temperature 135 ± 2 °C

2. Protein

Is the method used accredited? ☐ YES ☐ NO

Reference :

Sample weight (g) :

Determination method :

Acid titration :

Catalyst :

3. Fat

Is the method used accredited? ☐ YES ☐ NO

Reference :

Sample weight (g) :

Extraction

☐ Acid hydrolysis prior to solvent extract

☐ Solvent extract

Extraction solvent :

Total extraction time (hours) :



APLAC T060: Feeding Stuffs Proficiency Testing Program

RESULTS PROFORMA (Continued)

4. Ash

Is the method used accredited? ☐ YES ☐ NO

Reference :

Sample weight (g) :

Steps taken to avoid spattering :

Ashing temperature (°C) :

Ashing time (hours) :

5. Calcium

Is the method used accredited? ☐ YES ☐ NO

Reference :

Sample weight (g) :

Sample preparation : ☐ Wet digestion
☐ Dry ashing
☐ Other.....
(please specify)

Releasing agent :

Analytical Instrument(s) :

6. Phosphorus

Is the method used accredited? ☐ YES ☐ NO

Reference :

Sample weight (g) :

Sample preparation : ☐ Wet digestion
☐ Dry ashing
☐ Other.....
(please specify)

Analytical Instrument(s):.....

(Please specify wavelength in case of spectrophotometer is used)

☐ Tick as appropriate