Asia Pacific Laboratory Accreditation Cooperation

DETERMINATION OF NUTRIENTS IN FOOD

PROFICIENCY TEST PROGRAM

APLAC T073

REPORT

1 March 2010

CONTENTS

Summary of Results

1. This proficiency testing program (APLAC T073) aimed at evaluating the capability of participants to determine the content of seven core nutrients, *viz*. total lipids, saturated fat, trans fat, protein, sugars, total dietary fibre, and sodium in food. Participants were also asked to determine the moisture and ash content in the sample.

2. A total of 50 laboratories from 36 economies enrolled in the program and 48 of them returned results to the organizer.

3. Consensus means and standard deviation (SD) of participants' data are determined by robust statistic. z-Score is used as the numerical indicator to assess individual participant's competence relative to the others in the programme.

4. z-Scores for the eight test parameters are summarized as follows:

Test parameter	$\lvert z \rvert \leq 2$	$2 < \lvert z \rvert < 3$	$\lvert z \rvert \geq 3$	Total
Total lipid	39	1	4	44
	88.6%	2.3%	9.1%	100%
Saturated fat	27	3	4	34
	79.4%	8.8%	11.8%	100%
Trans fat	21	3	2	26
	80.8%	11.5%	7.7%	100%
Protein	41	2	3	46
	89.1%	4.4%	6.5%	100%
Sugars	30	2	7	39
	76.9%	5.1%	18.0%	100%
Total dietary fibre	24	1	8	33
	72.8%	3.0%	24.2%	100%
Sodium	31	1	7	39
	79.5%	2.6%	17.9%	100%
Ash	45	2	0	47
	95.7%	4.3%	0.0%	100%

5. Performance assessment for moisture was not carried out because the stability study indicated that there may be changes of the moisture content during the proficiency test. The changes of the moisture content however should not affect the performance assessment for the other test parameters.

1. Introduction

1.1 Providing nutrition information on food labels is an important public health tool to promote a balanced diet as food labels is an important communication channel whereby consumers can obtain specific information on individual food products.

1.2 A proficiency testing program (APLAC T073) on the determination of the content of seven core nutrients, *viz*. total lipids, saturated fat, trans fat, protein, sugars, total dietary fibre (TDF), and sodium, as well as moisture and ash content in food, was organised by the Centre for Food Safety (CFS), in collaboration with Hong Kong Accreditation Service (HKAS), and under the auspices of the Asia-Pacific Laboratory Accreditation Co-operation (APLAC). The program was approved by APLAC PT Committee in August 2009 and completed in March 2010 (Table I).

1.3 A total of 50 laboratories from 36 economies enrolled in the program and 48 of them returned their results on or before the deadline (Table II). Each participant was confidentially assigned with a unique laboratory code (1 to 48) and the code was used throughout the program.

2. Objectives

2.1 The main objective of this program is to assess the performance of participants in the analysis of seven nutrients and moisture and ash in food through an inter-laboratory comparison.

2.2 Test results from participants are evaluated according to the statistical techniques outlined in ISO13528:2005[9.1] "Statistical methods for use in proficiency testing by inter-laboratory comparisons" and in the "International harmonized protocol for the proficiency testing of analytical chemistry laboratories"[9.2]. z-Score was used as a numerical indicator to assess individual participant's proficiency relative to the others in the program.

3. Test Material

3.1 About 10 kg of biscuit were purchased from local market and were confirmed to contain suitable amount of protein, total lipids, saturated fat, trans fat, sugars, TDF and sodium. The sample was homogenized. The fine powder, in about 120 g portion each, was independently transferred to clean plastic bags. The prepared samples (about 66 bags) were vacuum packed to prevent microbial growth.

3.2 Homogeneity (performed in July to August 2009) and stability tests (November 2009) of the samples were determined in accordance with the procedures stipulated in APPENDICES I and II. Apart from moisture, the results indicate that the samples are suitable to be used as the testing materials for the program.

3.3 Samples together with “Instructions to Accreditation Bodies”, “Receipt Form”, “Instructions to Participating Laboratories”, and “Result Proformas” were sent to participating accreditation bodies (AB) in September 2009. A specimen copy of these instructions was given in APPENDICES III to VII.

3.4 All AB were requested 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.

3.5 Each participant was provided with one bag of 120 g of test sample in the program.

4. Tests Required

4.1 Participants were required to determine the content of total lipids, saturated fat, trans fat, protein, sugars, TDF, moisture, ash and sodium in the PT sample by the most appropriate methods of their choice.

4.2 Participants were requested to report results in g/100g to three significant figures or three decimal places, whichever was appropriate.

4.3 Expanded measurement uncertainty and technical information of the method used were required.

4.4 All the analytical results and information were reported in the Result Proformas provided.

5. Statistical Evaluation

5.1 Participants' test results

5.1.1 Test results received from participants are presented in TABLES III to XI. Their methodologies and techniques employed as well as their accreditation status are tabulated in TABLES XII to XX.

5.2 Performance evaluation

5.2.1 Performance of each participant was evaluated by means of *z*-score, which is calculated as:

$$z_i = \frac{x_i - \bar{x}}{s}$$

where x_i = reported result

$\bar{x}$ = Consensus mean of participants by robust statistic

s = Standard deviation (SD) of participants by robust statistic

5.2.2 z-Score is interpreted as follows:

(a)	$\|z\| \leq 2$	Satisfactory
(b)	$2 < \|z\| < 3$	Questionable
(c)	$\|z\| \geq 3$	Unsatisfactory

5.2.3 Participants having z-score(s) ≥ 3 or ≤ -3 should thoroughly investigate their results and, if appropriate, report the findings to their ABs. Participants having z-score(s) in the range $2 < |z| < 3$ are encouraged to review their results.

5.3 An interim report containing participants' data and the respective z-score results was issued to participants and their ABs on 8 December 2009. Participants were requested to check the correctness of their submitted data and to inform the organizers of any mistakes found on or before 24 December 2009 prior to the issuance of Final Report.

6. Results and Discussions

6.1 Upon checking the interim report, three laboratories informed the organizer that they had mixed up the reporting unit or results of their submitted data for sodium or protein. Since it is the responsibility of participants to ensure the correctness of their submitted results, no correction was made and the results in the interim report for these laboratories remain unchanged in the final report. Only one laboratory (Lab. Code 40) reported one value less than the reporting limit. Such result was treated as non-valid numeric data and removed from statistical evaluation.

6.2 Number of participants' data submitted is summarized as follows:

	Total lipids	Sat fat	Trans fat	Protein	Sugars	TDF	Moisture	Ash	Sodium
No. of data submitted	44	34	27	46	39	33	47	47	39
No. of valid numeric data	44	34	26	46	39	33	47	47	39

6.3 Consensus mean and standard deviation are summarized as follows:

	Total lipids	Sat fat	Trans fat	Protein	Sugars	TDF	Moisture	Ash	Sodium
Consensus mean (g/100g)	20.1	10.1	0.414	6.17	37.1	3.59	2.82	1.74	0.487
Standard deviation (g/100g)	0.62	0.80	0.078	0.22	2.3	0.86	0.29	0.11	0.039

6.3.1 All participants' results, except for the one as indicated in 6.1 above, were included in the computation of the mean and SD by robust statistics The stability on moisture was found not meeting the requirement specified in ISO13528:2005[9.1]. Therefore, the organizer decided that performance assessment should not be carried out for moisture. Also notable is that the standard deviations for trans fat and TDF are larger when compared with the respective SD calculated from the Horwitz equation (which are 0.014 and 0.10 g/100g respectively). This would suggest that participants may encounter difficulties in achieving a reasonable reproducibility precision from a single population for these two parameters.

6.3.2 For total lipids determination, only 4 unsatisfactory results were identified. One participant (Lab. Code 32) used NMR as method of analysis and reported a low result of 17.3 g/100g. This information may suggest that using NMR to determine total lipids content may not be comparable with the other techniques but since there was only one laboratory using this technique, no firm conclusion could be drawn. For the saturated fat determination, there were 4 unsatisfactory participants. Of these four, 3 laboratories reported their results using a different basis from that given in the instructions. These participants reported results as percentage of total fatty acids. When the SD of reported results of saturated fat is compared with that of trans fat, the latter was found to be higher although most participants used the same method for both test parameters. This would indicate that the determination of trans fat still presents greater challenge to testing laboratories than saturated fat.

6.3.3 In general, the participants have no problem in the determination of protein in food. Most participants used Kjeldahl digestion as the method of analysis. The 2 unsatisfactory participants also reported using Kjeldahl method, but with results deviated more than 10% from the consensus mean value. Hence, this information indicated these participants still have difficulties in determining the nitrogen content by classical Kjeldahl method.

6.3.4 For determination of sugars, the technique used varied from titrimetric, gravimetric, enzymatic, colourmetric, ion chromatographic to liquid chromatograph mass spectrometric. Some participants employed classical techniques with reported values closed to the consensus mean while some participants used chromatographic technique with reported values deviated more than 20% from the consensus mean.

6.3.5 Almost all the participants employed AOAC 985.29 or 991.43 as method of determination of TDF. AOAC 991.43 and 985.29 should produce comparable results as their working principles are almost the same. However, a very wide dispersion of results was observed in this exercise. As the sample contained only small amount of water, the dryness of the sample should not affect the test results. Hence, this may be because the test material contained high level of total lipids, which requires to be removed before the analysis for an accurate measurement.

6.3.6 For the determination of ash, even though the temperature used varied from 450 to 600°C, comparable results were obtained. The participants have no problem in the determination of ash in food.

6.3.7 Besides those with reporting errors, the participants have no problem in the determination of sodium in food when inductively coupled plasma optical emission spectrometry was employed. For those participants using atomic emission spectrometry technique, quite a number of participants got higher results. These participants may wish to check whether it was due to ionization enhancement in the present of other alkali salts in food.

6.4 z-Scores of individual participant were presented in TABLE XXI and illustrated graphically in CHART I to IX. The performance of participants was summarized as follows:

In summary, 24 (Lab. Code: 1, 2, 3, 6, 9, 11, 12, 15, 16, 17, 22, 23, 27, 29, 30, 31, 32, 35, 36, 38, 40, 42, 43, and 45) out of the 48 participants (50%) were identified as having reported one or more unsatisfactory results; and 35 out of 308 results submitted, excluding results of moisture, were identified as unsatisfactory results (11.4%).

6.5 The relative standard deviation for trans fat and TDF are high, 19 and 24% respectively, when compared to the tolerance limits of 20% set by many countries. It appears that the analyses of trans fat and TDF in foods still present challenges to testing laboratories and improvement is needed. Otherwise, there could be disputes on the test results of trans fat and/or TDF between laboratories.

6.6 Amongst the test parameters, excluding moisture, which claimed to be accredited, 15 (9%) out of 165 have unsatisfactory z-scores and the remaining achieved satisfactory. For the non-accredited test parameters, 123 out of 143 (86%) achieved satisfactory and 20 (14%) had unsatisfactory z-scores.

6.7 ISO/IEC 17025 requires laboratories to report measurement uncertainty (MU) when requested. Although MU was not used in the performance assessment in the programme, laboratories were requested to report their MU. Similar to other APLAC PT programmes on chemical testing, large variation of the reported MU was found amongst participants (TABLE III to XI). For instance, a minimum of 0.0000308 g/100g (Lab. Code 35) and a maximum of 5.7 g/100g (Lab. Code 15), both as the coverage factor of 2, were reported for the analysis of total lipids. Furthermore, a high proportion of participants (29.5%) did not report the MU of their analyses. The information indicates that some participants have difficulties in estimating MU and training is recommended.

7. Remarks

7.1 Contributions from all ABs and participants to this program are gratefully noted. Special thanks are also extended to APLAC PT Committee, HKAS and the Controller, Centre for Food Safety of Food Research Laboratory for their support of the program.

7.2 For further comments, participants are welcome to contact the organizers in following address:

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Centre for Food Safety
4/F Public Health Laboratory Centre,
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8. Acknowledgement

8.1 Special thanks to the Food Research Laboratory, Centre for Food Safety for their support in providing the suitable biscuit sample and in the design of the homogeneity and stability studies.

9. References

9.1 ISO 13528:2005. Statistical methods for use in proficiency testing by interlaboratory comparisons, **2005**, Geneva, Switzerland.

9.2 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.

TABLE I: Timeline of the Program

EVENT	SCHEDULE
Preparation of sample	Jun 2009
Homogeneity testing	Jul – Aug 2009
Submission of proposal to APLAC PT Committee for approval	Aug 2009
Invitation of participants	Sep 2009
Dispatch of samples	Sep 2009
Submission of results	Nov 2009
Stability testing	Nov 2009
Statistical analysis of results	Nov – Dec 2009
Interim report	Dec 2009
Submission of draft final report to APLAC PT Committee	Jan 2010
Approval of draft final report by APLAC PT Committee	Feb 2010
Distribution of final report	Mar 2010

TABLE II: List of Participating Accreditation Bodies

No.	ECONOMES	PARTICIPANTS ENROLLED	RETURNED RESULTS
1	Argentina	1	1
2	Australia	1	1
3	Belgium	1	1
4	Brazil	1	1
5	Canada	1	1
6	Chile	1	1
7	China	1	1
8	Costa Rica	1	1
9	Cyprus	1	1
10	Czech Repulic	1	1
11	Denmark	1	1
12	Finland	1	1
13	France	1	1
14	Germany	1	1
15	Guatemala	1	1
16	Honduras	1	1
17	Hong Kong	14	13
18	Hungary	1	1
19	Ireland	1	1
20	Israel	1	1
21	Japan	1	1
22	Korea	1	1
23	Mexico	1	1
24	Mongolia	1	1
25	New Zealand	1	1
26	Norway	1	1
27	Paraguay	1	1
28	Philippines	1	1
29	Poland	1	1
30	Portugal	1	1
31	Singapore	1	1
32	Sri Lanka	1	1
33	Sweden	1	1
34	Trinidad & Tabago	2	1
35	USA	1	1
36	Viet Nam	1	1
	Total:	50	48

TABLE III: Participants' results for total lipids

Lab Code	Result (g/100g)	Expanded uncertainty (g/100g)	Coverage factor
1	19.8	---	---
2	---	---	---
3	19.9	1.3	2
4	20.3	---	---
5	20.26	2 %	---
6	20.5	---	---
7	20.073	1.439	2
8	19.5	0.3	2
9	19.7	0.12	4.3
10	19.9*	---	---
11	---	---	---
12	20.01	1.00	2
13	---	---	---
14	20.8	---	---
15	22.8	25 %	2
16	17.0	2.79	2
17	20.8	0.29	2
18	20.3	1.4	2
19	20.03	0.50	2
20	19.46	0.10	2
21	19.8	0.48	2.8
22	21.2	3.18	2
23	20.39	---	---
24	---	---	---
25	20.6	---	---
26	19.4	2.9	2
27	18.80	0.524	2
28	20.5	0.77	2
29	19.9	0.3	2
30	20.5	---	---
31	20.0	0.20	2
32	17.3	0.32% Abs	2
33	20.7	---	---
34	20.687	0.1	2
35	20.00	3.08×10^{-5}	2
36	19.672	---	---
37	20.87	20%	2
38	19.8	0.05	2
39	21.3	24	2
40	22.5	0.338	2
41	20.8	0.990	2
42	20.4	0.25	2
43	19.41	---	---
44	20.1	---	---
45	19.74	0.0045	2
46	20.3	1.25	2
47	20.4	4.41	2
48	19.10	0.27	2

"---" denotes data/information was not provided by participants

"*" denotes mean concentration of reported values

TABLE IV: Participants' results for saturated fat

Lab Code	Result (g/100g)	Expanded uncertainty (g/100g)	Coverage factor
1	10.6	---	---
2	---	---	---
3	10.4	1.3	2
4	---	---	---
5	9.6	5 %	--
6	10.8	---	---
7	10.189	0.815	2
8	10.1	0.1	2
9	8.00	0.26	3.2
10	10.3*	---	---
11	---	---	---
12	10.5	0.52	2
13	---	---	---
14	---	---	---
15	---	---	---
16	8.13	1.88	2
17	9.9922	0.56	2
18	10.7	---	---
19	10.89	0.49	2
20	---	---	---
21	10.9	1.8	2.8
22	7.81	1.56	2
23	10.36	---	---
24	---	---	---
25	10.7	---	---
26	9.52	1.9	2
27	9.22	0.120	2
28	9.78	0.04	2
29	---	---	---
30	50.4	---	---
31	9.61	0.26	2
32	49.8	13% Rel	2
33	9.70	---	---
34	10.833	1.45	2
35	1.63	3.08×10^{-5}	2
36	---	---	---
37	9.59	20%	2
38	10.3	0.05	2
39	---	---	---
40	9.94	1.31	2
41	10.4	1.70	2
42	9.20	0.5	2
43	---	---	---
44	9.64	---	---
45	49.01	2.0	2
46	---	---	---
47	10.4	1.56	2
48	---	---	---

"---" denotes data/information was not provided by participants

"*" denotes mean concentration of reported values

TABLE V: Participants' results for trans fat

Lab Code	Result (g/100g)	Expanded uncertainty (g/100g)	Coverage factor
1	0.531	---	---
2	---	---	---
3	0.239	0.03	2
4	---	---	---
5	0.44	5 %	---
6	0.39	---	---
7	0.425	0.145	2
8	0.409	0.050	2
9	0.288	0.02	3.2
10	0.255*	---	---
11	---	---	---
12	---	---	---
13	---	---	---
14	---	---	---
15	---	---	---
16	0.356	0.0701	2
17	0.849	0.18	2
18	0.375	---	---
19	---	---	---
20	---	---	---
21	---	---	---
22	0.450	0.09	2
23	0.279	---	---
24	---	---	---
25	0.425	---	---
26	---	---	---
27	0.37	0.052	2
28	0.37	0.13	2.4
29	---	---	---
30	---	---	---
31	0.499	0.021	2
32	1.30	13% Rel	2
33	---	---	---
34	0.214	0.04	2
35	---	---	---
36	---	---	---
37	0.40	20%	2
38	0.413	0.05	2
39	---	---	---
40	<0.3	---	2
41	0.458	0.073	2
42	0.40	0.1	2
43	---	---	---
44	0.45	---	---
45	0.50	0.1	2
46	---	---	---
47	0.51	0.15	2
48	---	---	---

"---" denotes data/information was not provided by participants

"*" denotes mean concentration of reported values

TABLE VI: Participants' results for protein

Lab Code	Result (g/100g)	Expanded uncertainty (g/100g)	Coverage factor
1	6.39	---	---
2	20.37	4 %	2
3	6.21	0.4	2
4	6.37	---	---
5	6.2	2 %	---
6	6.26	---	---
7	6.035	0.465	2
8	6.20	0.12	2
9	6.27	0.52	2
10	5.9*	---	---
11	6.82	---	---
12	5.99	0.25	2
13	---	---	---
14	5.94	0.89	2
15	6.11	25 %	2
16	6.63	0.669	2
17	6.07	0.19	2
18	6.00	0.1	2
19	6.29	0.40	2
20	6.59	0.08	2
21	6.01	0.27	2.8
22	5.92	0.32	2
23	6.38	---	---
24	---	---	---
25	5.86	0.02	2
26	5.87	0.59	2
27	6.18	0.38	2
28	6.27	0.17	2
29	6.08	0.01	2
30	6.3	---	---
31	6.05	0.65	2
32	6.23	3.3% Rel	2
33	6.27	---	---
34	6.459	1	2
35	6.06	1.32×10^{-5}	2
36	6.175	---	---
37	5.92	20%	2
38	6.18	0.05	2
39	6.20	18	2
40	5.97	0.190	2
41	6.05	0.340	2
42	6.03	0.25	2
43	5.70	---	---
44	6.09	---	---
45	7.10	0.013	2
46	6.36	0.22	2
47	6.2	0.41	2
48	6.00	0.04	2

"---" denotes data/information was not provided by participants

"*" denotes mean concentration of reported values

TABLE VII: Participants' results for sugars

Lab Code	Result (g/100g)	Expanded uncertainty (g/100g)	Coverage factor
1	36.5	---	---
2	---		
3	38.1	3.8	2
4	---		
5	37.5	---	---
6	46.25	---	---
7	38.710	2.871	2
8	39.6	0.5	2
9	37.1	3.36	4.3
10	35.25*	---	---
11	---	---	---
12	---	---	---
13	37.7	1.2	2
14	36.0	---	---
15	21.7	25 %	2
16	38.2	3.65	2
17	34	3	2
18	39.5	---	---
19	37.14	0.50	2
20	---	---	---
21	40.5	1.6	2.8
22	19.17	3.83	2
23	35.9	---	---
24	---	---	---
25	34.6	---	---
26	39.4	7.9	2
27	35.6	1.5	2
28	36.3	0.99	2
29	70.6	2.2	2
30	37.4	---	---
31	36.9	8.86	2
32	35.6	9% Rel	2
33	35.8	---	---
34	36.030	0.4	2
35	49.20	7.92×10^{-9}	2
36	41.456	---	---
37	30.7	20%	2
38	36.0	0.05	2
39	---	---	---
40	35.9	1.62	3.18
41	35.9	4.60	2
42	44.1	5	2
43	45.07	---	---
44	30.8	---	---
45	36.36	0.37	2
46	---	---	---
47	39.0	10	2
48	---	---	---

"---" denotes data/information was not provided by participants

"*" denotes mean concentration of reported values

TABLE VIII: Participants' results for total dietary fibre

Lab Code	Result (g/100g)	Expanded uncertainty (g/100g)	Coverage factor
1	7.10	---	---
2	---	---	---
3	11.1	1.8	2
4	---	---	---
5	3.1	CV = 5 %	---
6	2.35	---	---
7	2.880	0.432	2
8	3.38	0.13	2
9	2.98	0.88	3.2
10	---	---	---
11	---	---	---
12	---	---	---
13	---	---	---
14	---	---	---
15	3.08	25 %	2
16	12.5	1.21	2
17	3.90	1.7	2
18	3.04	---	---
19	5.92	0.43	2
20	---	---	---
21	2.89	0.35	2.8
22	7.63	1.53	2
23	7.1	---	---
24	---	---	---
25	2.96	0.21	2
26	3.41	0.51	2
27	4.63	0.256	2
28	3.76	0.40	2
29	---	---	---
30	2.7	---	---
31	11.5	2.6	2
32	---	---	---
33	3.46	---	---
34	4.330	0.5	2
35	6.70	1.035×10^{-6}	2
36	---	---	---
37	3.4	20%	2
38	0.9	0.05	2
39	---	---	---
40	3.01	0.255	2
41	3.07	0.320	2
42	2.72	0.2	2
43	5.25	---	---
44	3.5	---	---
45	---	---	---
46	---	---	---
47	3.1	1.49	2
48	3.6	0.3	2

"---" denotes data/information was not provided by participants

TABLE IX: Participants' results for sodium

Lab Code	Result (g/100g)	Expanded uncertainty (g/100g)	Coverage factor
1	0.705	---	---
2	---	---	---
3	0.436	0.06	2
4	---	---	---
5	0.47	7 %	---
6	0.426	---	---
7	---	---	---
8	0.491	0.006	2
9	478	65.5	2
10	0.51*	---	---
11	0.395	---	---
12	484	20	2
13	0.483	0.021	2
14	0.51	---	---
15	0.43	25 %	2
16	0.504	0.0965	2
17	0.475	0.02	2
18	0.493	---	---
19	0.49	0.02	2
20	---	---	---
21	0.487	0.044	2.8
22	0.453	0.091	2
23	0.513	---	---
24	0.492	0.054	2
25	0.529	---	---
26	0.49	0.1	2
27	0.625	0.082	2
28	0.448	0.039	2
29	0.255	0.010	2
30	0.441	---	---
31	---	---	---
32	0.473	5.9% Rel	2
33	0.520	---	---
34	0.419	0.04	2
35	0.47	1.548×10^{-8}	2
36	6.417	---	---
37	0.475	20%	2
38	0.50	0.05	2
39	---	---	---
40	0.506	0.063	2
41	0.503	0.040	2
42	485	32	2
43	---	---	---
44	0.46	---	---
45	---	---	---
46	0.447	0.001	2
47	0.48	0.11	2
48	---	---	---

"---" denotes data/information was not provided by participants

"*" denotes mean concentration of reported values

TABLE X: Participants' results for ash

Lab Code	Result (g/100g)	Expanded uncertainty (g/100g)	Coverage factor
1	1.60	---	---
2	1.72	7 %	2
3	1.93	0.06	2
4	1.63	---	---
5	1.8	CV = 5 %	---
6	1.88	---	---
7	1.673	0.151	2
8	1.86	0.10	2
9	1.73	0.17	2
10	1.6*	---	---
11	1.86	---	---
12	1.73	0.08	2
13	---	---	---
14	1.70	0.09	2
15	1.82	25 %	2
16	1.84	0.110	2
17	1.75	0.22	2
18	1.83	---	---
19	1.68	0.08	2
20	1.84	0.02	2
21	1.64	0.14	2.8
22	1.99	0.29	2
23	1.76	---	---
24	1.86	0.19	2
25	1.77	0.0002	2
26	1.59	0.08	2
27	1.73	0.094	2
28	1.77	0.08	2
29	1.71	0.01	2
30	1.78	---	---
31	1.83	0.20	2
32	1.91	2.7% Rel	2
33	1.80	---	---
34	1.505	0.02	2
35	1.61	4.928×10^{-4}	2
36	1.722	---	---
37	1.67	20%	2
38	1.72	0.025	2
39	1.84	5.1	2
40	1.63	0.115	2
41	1.74	0.026	2
42	1.54	0.1	2
43	1.76	---	---
44	1.63	---	---
45	1.84	0.01	2
46	1.72	0.44	2
47	1.72	0.14	2
48	1.56	0.05	2

"---" denotes data/information was not provided by participants

"*" denotes mean concentration of reported values

TABLE XI: Participants' results for moisture

Lab Code	Result (g/100g)	Expanded uncertainty (g/100g)	Coverage factor
1	2.46	---	---
2	2.71	5 %	2
3	2.86	0.4	2
4	3.03	---	---
5	3.1	CV = 2.5 %	---
6	2.59	---	---
7	2.835	0.354	2
8	2.68	0.21	2
9	3.10	0.46	2
10	2.8*	---	---
11	2.74	---	---
12	2.85	0.15	2
13	---	---	---
14	2.59	0.13	2
15	2.23	25 %	2
16	2.62	0.261	2
17	2.68	0.38	2
18	2.78	0.02	2
19	2.46	0.12	2
20	2.78	0.03	2
21	3.68	0.60	2.8
22	2.96	0.44	2
23	2.63	---	---
24	3.08	0.27	2
25	3.67	---	---
26	2.83	0.14	2
27	3.02	0.012	2
28	2.71	0.08	2
29	3.58	0.02	2
30	1.4	---	---
31	2.87	0.25	2
32	2.87	2.0% Rel	2
33	2.90	---	---
34	2.677	0.1	2
35	1.55	---	2
36	2.561	---	---
37	2.82	20%	2
38	3.11	0.025	2
39	2.83	3.8	2
40	3.27	0.051	2
41	2.90	0.044	2
42	2.72	0.25	2
43	3.24	---	---
44	2.49	---	---
45	2.92	0.015	2
46	2.87	0.40	2
47	1.5	0.44	2
48	3.6	0.4	2

"---" denotes data/information was not provided by participants

"*" denotes mean concentration of reported values

TABLE XII: Technical information for the determination of total lipids

Lab Code	Method	Technique used	Method accredited
1	AOAC 922.06		No
2	---	---	---
3	---	Acid hydrolysis	Yes
4	AOAC 922.06, clause 32.4.02	Acid hydrolysis	No
5	NMKL 131:89	SBR	No
6	AOCS Ce 1h-05, rev 05	Acid hydrolysis/solvent extraction/GC	Yes
7	PA-A-88021:1971	Soxhlet method	Yes
8	AOAC 922.06 – 18 ED	---	Yes
9	---	Acid hydrolysis followed by ether extraction	No
10	---	Hydrolysis with ether extraction	No
11	---	---	---
12	---	Hydrolysis	No
13	---	---	---
14	In-house method	---	No
15	---	Acid hydrolysis	No
16	AOAC 945.44	---	No
17	AOAC 922.06	Acid hydrolysis	Yes
18	AOAC 922.06	---	No
19	---	---	Yes
20	Korea Food Code	Ethyl ether	Yes
21	---	Acid hydrolysis and solvent extraction	Yes
22	GB/T 5009.6-2003	---	Yes
23	ASU 01.00-20, mod./ASU 06.00-6, mod	Gravimetric acc. Weibull-Stoldt	Yes
24	---	---	---
25	AOAC 922.06	---	No
26	---	Acid hydrolysis + extraction	Yes
27	---	Acid hydrolysis + Soxhlet extraction	No
28	---	HCl hydrolysis	Yes
29	---	Soxhlet	No
30	---	HCl hydrolysis	Yes
31	---	Soxhlet extraction with petroleum ether on SOXTEC after acid hydrolysis with HCl	Yes
32	---	NMR	Yes
33	---	HCl hydrolysis, petroleum ether/diethyl ether extraction, gravimetric analysis	Yes
34	AOAC 979.09	Hidolisis acida Mojonnier	Yes
35	AOAC 920.39	Acid hydrolysis followed by Soxhlet extraction	Yes
36	NMX-F-615-NORMEX-2004	---	Yes
37	AOAC 922.06	---	No
38	GB/T 5009.6-2003	---	Yes
39	---	Acid hydrolysis	No
40	---	Acid hydrolysis followed by extraction	No
41	AOAC 992.06	---	No
42	AOAC 922.06	---	Yes
43	AOAC 963.15, Ed. 16	---	No
44	32.1.14 AOAC (2000)	---	No
45	MNS 0342:84	Total lipid determination by Soxhlet	Yes
46	OJ-N^{o} L257/14, 1998	---	Yes
47	---	---	Yes
48	---	Acid hydrolysis and ether extraction	Yes

"---" denotes data/information was not provided by participants

TABLE XIII: Technical information for the determination of saturated fat

Lab Code	Method	Technique used	Method accredited
1	AOAC 963.22	---	Yes
2	---	---	---
3	---	FAP-GC	Yes
4	---	---	---
5	AOAC 1f-96	---	No
6	AOCS Ce 1h-05, rev 05	Acid hydrolysis/solvent extraction/GC	Yes
7	PN-EN ISO 5508:1996 PN-EN ISO 5509:2001	GC method	No
8	AOAC 963.22 – 18 ED	---	Yes
9	---	Hydrolytic extraction followed by GC method	No
10	---	Chromatography gaseous-GC	No
11	---	---	---
12	---	GC	No
13	---	---	---
14	---	---	---
15	---	---	---
16	AOAC 996.09 & AOCS Ce 1h-05	---	No
17	AOAC 991.39/969.33/963.22	---	Yes
18	AOAC 996.06	---	No
19	---	---	Yes
20	---	---	---
21	---	GC-FID	Yes
22	AOAC 996.06	---	No
23	Internal method of lab	GC	Yes
24	---	---	---
25	AOAC 996.06	---	No
26	In-house method	GC-FID	Yes
27	MSZ 5508:1992	GC analysis from fatty acid methyl esters	Yes
28	---	GC-FID	Yes
29	---	---	---
30	---	---	Yes
31	---	GC-FID	Yes
32	---	Gas chromatography	Yes
33	---	GC-FID, calc factor 0.956 to fat	Yes
34	AOAC 969.33	GLC-FID	No
35	AOAC 969.33	Sample prep. – AOAC 969.33 followed by GC analysis	No
36	---	---	---
37	AOAC 996.06	---	No
38	SN/T 1945-2007	---	Yes
39	---	---	---
40	---	GC-FID	No
41	AOAC 996.06	---	No
42	AOCS Ce 1f-96	---	Yes
43	---	---	---
44	Ce 1-62/2-66 1d-91 AOCS (1997)	--	No
45	MNS GOSTR 51698:2004	Saturated fat and trans fatty acid determination by GC	Yes
46	---	---	---
47	---	---	No
48	---	---	---

"---" denotes data/information was not provided by participants

TABLE XIV: Technical information for the determination of trans fat

Lab Code	Method	Technique used	Method accredited
1	AOAC 994.15	---	Yes
2	---	---	---
3	---	FAP-GC	Yes
4	---	---	---
5	AOAC 1f-96	---	No
6	AOCS Ce 1h-05, rev 05	Acid hydrolysis/solvent extraction/GC	Yes
7	PN-EN ISO 5508:1996 PN-EN ISO 5509:2001	GC method	No
8	AOAC 963.22 – 18 ED	---	Yes
9	---	Hydrolytic extraction followed by gas chromatographic method	No
10	---	Chromatographic gaseous-GC	No
11	---	---	---
12	---	---	---
13	---	---	---
14	---	---	---
15	---	---	---
16	AOAC 996.09 & AOCS Ce 1h-05	---	No
17	AOAC 991.39/969.33/963.22	---	Yes
18	AOAC 996.06	---	No
19	---	---	---
20	---	---	---
21	---	---	---
22	AOAC 996.06	---	No
23	Internal method of lab	GC	Yes
24	---	---	---
25	AOAC 996.06	---	No
26	---	---	---
27	MSZ 5508:1992	GC analysis from fatty acid methyl esters	Yes
28	---	GC-FID	Yes
29	---	---	---
30	---	---	---
31	---	GC-FID	Yes
32	---	Gas Chromatography	Yes
33	---	---	---
34	AOAC 969.33	GLC-FID	No
35	---	---	---
36	---	---	---
37	AOAC 996.06	---	No
38	SN/T 1945-2007	---	Yes
39	---	---	---
40	---	GC-FID	No
41	AOAC 996.06	---	No
42	AOCS Ce 1f-96	---	Yes
43	---	---	---
44	Ce 1-62/2-66 1d-91 AOCS (1997)	---	No
45	MNS GOSTR 51698:2004	Saturated fat and trans fatty acid determination by GC	Yes
46	---	---	---
47	---	---	No
48	---	---	---

"---" denotes data/information was not provided by participants

TABLE XV: Technical Information for the determination of protein

Lab Code	Method	Technique used	Method accredited
1	AOAC 960.52	---	No
2	---	Kjeldahl	Yes
3	---	Kjeldahl	Yes
4	AOAC Official Method 920.87, clause 32.4.02 (2007)	---	No
5	NMKL 64:2003	Kjeldahl	No
6	---	---	Yes
7	PN-A-04018:1975+Az3:2002	Kjeldahl method	Yes
8	AOAC 960.25 – 18 ED	---	Yes
9	---	Total Kjeldahl Method	No
10	---	Dumas method-Leco	Yes
11	AOAC 978.04	---	No
12	---	Kjeldahl	No
13	---	---	---
14	In-house method	---	No
15	---	Kjeldahl method	No
16	AOAC 928.08	---	No
17	AOAC 992.15	Protein combustion	Yes
18	AOAC 920.87	---	No
19	---	---	Yes
20	Korea Food Code	Kjeldahl	Yes
21	---	Kjeldahl	Yes
22	GB/T 5009.5-2003	---	Yes
23	ASU L17.00-15, mod/ASU L06.00-7 mod.	Kjeldahl	Yes
24	---	---	---
25	AOAC 981.10	---	Yes
26	ISO 937:1978	Kjeldahl	Yes
27	---	Kjeldahl method	Yes
28	---	Kjeldahl	Yes
29	---	Kjeldahl, distillation	No
30	---	Kjeldahl	Yes
31	---	Kjeldahl method using KJELTEC Analyser	Yes
32	---	Kjeldahl	Yes
33	---	Dumas	Yes
34	AACC Method 46-12-Factor 6.25	---	Yes
35	---	Kjeldahl semi-micro method	Yes
36	NMX-F-608-NORMEX-2002	---	Yes
37	AOAC 928.08	---	No
38	GB/T 5009.5-2003	---	Yes
39	---	Digestion-Distillation-Titration	No
40	---	Kjeldahl method for nitrogen	No
41	NMKL method No. 6, 4th edition	---	No
42	---	Kjeldahl	Yes
43	AOAC 970.22, Ed. 16	---	No
44	39.1.15 II AOAC (2000)	---	No
45	---	Determination of protein by Kjeldahl	Yes
46	OJ-N^{o} L179/9, 1993	---	Yes
47	---	---	Yes
48	AOAC 935.39C 2005	---	Yes

"---" denotes data/information was not provided by participants

TABLE XVI: Technical information for the determination of sugars

Lab Code	Method	Technique used	Method accredited
1	PC/FQ/12 Browne & Zerban	---	No
2	---	---	---
3	---	HPLC	Yes
4	---	---	---
5	---	Enzymatic	---
6	---	LC/Pulse electrochemical detector	No
7	PN-A-88023:1961	Lane Eynon Method	Yes
8	AOAC 923.09 – 18 ED	---	Yes
9	---	Extraction followed by HPLC-RI measurement	No
10	---	Gravimetric method – Munson-Walker	No
11	---	---	---
12	---	---	---
13	---	HPLC	Yes
14	In-house method	---	No
15	---	Water extraction, sugar determined by RI detector through carbohydrate column	No
16	---	LC/MS/MS	No
17	AOAC 980.13	HPLC (ELSD)	Yes
18	AOAC 980.13	---	No
19	---	---	Yes
20	---	---	---
21	---	HPLC	Yes
22	AOAC 982.14	---	No
23	Internal method of lab	HPLC	Yes
24	---	---	---
25	---	HPLC/ELSD	No
26	Mod. NMKL 148/1993	HPLC	No
27	Schoorl method	---	Yes
28	---	Enzymatic	Yes
29	---	Colorimetric method	No
30	---	Luff Schoorl	Yes
31	---	HPAEC/PAD	Yes
32	---	HPLC	Yes
33	---	HPLC, Dionex system	Yes
34	Munson y Walker	Gravimetrico	No
35	Lane and Enyon Titration method	---	No
36	NMX-312-1978	---	Yes
37	In-house method	HPLC-RI	No
38	GB/T 5009.8-2008	---	Yes
39	---	---	---
40	---	HPLC-RI	No
41	AOAC 982.14	---	No
42	AOAC 996.04	IC	Yes
43	AOAC 923.09	---	No
44	4.7.02 AOAC (2000)	---	No
45	MNS 344:84	Determination of total sugars titration method	Yes
46	---	---	---
47	---	---	Yes
48	---	---	---

"---" denotes data/information was not provided by participants

TABLE XVII: Technical information for the determination of total dietary fibre

Lab Code	Method	Technique used	Method accredited
1	AOAC 985.29	---	No
2	---	---	---
3	AOAC 985.29	---	Yes
4	---	---	---
5	AOAC 985.29:1995	---	No
6	AOAC 985.29 (2000)	---	Yes
7	AOAC 991.43 1994	Enzymatic-gravimetric method MES-TRIS buffer	Yes
8	AOAC 991.43 – 18 ED	---	Yes
9	---	Gravimetric determination after defatting and enzymatic hydrolysis of protein and carbohydrate	No
10	---	---	---
11	---	---	---
12	---	---	---
13	---	---	---
14	---	---	---
15	AOAC 985.29	---	No
16	AOAC 983.16	---	No
17	AOAC 985.29	---	Yes
18	AOAC 985.29	---	No
19	---	---	Yes
20	---	---	---
21	---	Gravimetric/Enzymatic	Yes
22	AOAC 985.29	---	No
23	ASU 00.00-18	---	Yes
24	---	---	---
25	AOAC 985.29	---	Yes
26	Mod. AOAC 985.29 (-95)	---	Yes
27	AOAC 985.29	---	No
28	AOAC 985.29	---	Yes
29	---	---	---
30	---	Enzymatic (Bioquant kit)	Yes
31	AOAC 985.29	Enzymatic-gravimetric method	Yes
32	---	---	---
33	AOAC 985.29	---	Yes
34	AOAC 985.29	Enzimatico gravimetrico	Yes
35	AOAC 985.29	---	No
36	---	---	---
37	AOAC 985.29	---	No
38	GB/T 5009.88 – 2008	---	Yes
39	---	---	---
40	---	Gravimetric determination after defatting and enzymatic hydrolysis of protein and carbohydrates	No
41	AOAC 991.43	---	No
42	AOAC 985.29	---	Yes
43	AOAC 985.29	---	No
44	45.4.07 AOAC (2000)	---	No
45	---	---	---
46	---	---	---
47	---	---	Yes
48	AOAC 985.29 2005	---	Yes

"---" denotes data/information was not provided by participants

TABLE XVIII: Technical information for the determination of sodium

Lab Code	Method	Technique used	Method accredited
1	Own method	Atomic Emission Spectroscopy	No
2	---	---	---
3	---	AAS	Yes
4	---		---
5	DS 13805:2002	ICP-OES	No
6	---	Acid digestion/ICP-OES	Yes
7	---	---	---
8	AOAC 969.23 – 18 ED	---	Yes
9	---	Acid digestion followed by ICP-OES measurement	No
10	---	Atomic absorption	No
11	AOAC 969.23 or AOAC 985.35	---	No
12	---	AA-Flame	No
13	---	Atomic absorption spectrophotometric method	Yes
14	In-house method	---	No
15	---	Microwave acid digestion + AAS	No
16	---	AAS	No
17	AOAC 984.27	ICP-OES	Yes
18	AOAC 985.35	---	No
19	---	---	Yes
20	---	---	---
21	---	AAS	Yes
22	GB/T 5009.91-2003	---	No
23	Internal method of lab	ICP-OES	Yes
24	AOAC 969.23	---	No
25	---	ICP-OES	No
26	SFS 3044+3017	Flame AAS	No
27	---	Flame atomic absorption spectrometric method	No
28	---	ICP-OES	Yes
29	---	Atomic absorption	No
30	---	AAS Flame	Yes
31	---	---	---
32	---	Ashing and atomic absorption	Yes
33	---	ICP-AES	Yes
34	AOAC 969.29	---	Yes
35	---	Digestion – two acid (HNO_3/H_2SO_4) and peroxide Analysis – SMEWW 3111 – A, B	No
36	---	Metodo estandar 3500 Na-D-1992	Yes
37	In-house method	ICP-OES	No
38	GB/T 5009.91 – 2003	---	Yes
39	---	---	---
40	---	ICP-OES, Flame photometry	No
41	In-house method	---	No
42	---	Flame AA	Yes
43	---	---	---
44	985.35 AOAC (2005)	---	No
45	---	---	---
46	CYS CEN/TS 15621:2007	---	No
47	---	---	Yes
48	---	---	---

"---" denotes data/information was not provided by participants

TABLE XIX: Technical information for the determination of ash

Lab Code	Method / technique used	Ashing temperature (°C)	Method accredited
1	AOAC 930.30	600	No
2	---	550	Yes
3	---	550	Yes
4	AOAC 920.87, clause 32.4.02 (2007)	550	No
5	NMKL 173:2005	550	No
6	AOAC 930.30 sec 33.5.05 (2000)	550	Yes
7	PN-A-88022:1959	550 – 600	Yes
8	AOAC 900.02 – 18 ED	550	Yes
9	---	550	No
10	---	550	No
11	ISO 936:1998	550	No
12	---	525	No
13	---	---	---
14	---	550	No
15	---	550	No
16	---	550	No
17	AOAC 923.03	550	Yes
18	AOAC 923.03	550	No
19	---	541	Yes
20	Korea Food Code	600	Yes
21	---	550	Yes
22	GB/T 5009.4-2003	550	No
23	Gravimetric (ASU 17.00-3, mod.)	550	Yes
24	GB/T 5009.4 - 2003	550	No
25	AOAC 923.03	550	Yes
26	ISO 936:1978	525	Yes
27	Gravimetric method	550	No
28	---	550	Yes
29	---	550	No
30	---	525	Yes
31	Ashing in muffle furnance	525	Yes
32	18 hours in muffle furnace	450	Yes
33	---	550	Yes
34	Gravimetrico 945.38	550	Yes
35	AOAC 923.03	550	Yes
36	NMX-607-NORMEX-2002	550-600	Yes
37	ISO 936:1998	550	No
38	GB/T 5009.4 – 2003	550	Yes
39	---	550	No
40	Gravimetric determination after dried, carbonized and incinerated at 550 °C	550	No
41	ISO Vol. 936	525	No
42	---	600	Yes
43	---	600	No
44	31.012 AOAC (1980)	525	No
45	MNS 0341:84 Determination of ash by ignition	550	Yes
46	---	550	No
47	---	550	Yes
48	AOAC 935.39A 2005	550	Yes

"---" denotes data/information was not provided by participants

TABLE XX: Technical information for the determination of moisture

Lab Code	Method / technique used	Drying temperature (°C)	Method accredited
1	AOAC 925.04	98	No
2	---	103	Yes
3	---	130	Yes
4	SLS 251:1991	105	No
5	NMKL 14:1953	130	No
6	AOAC 927.05 sec 33.5.05 (2000)	103	Yes
7	PN-A-88027:1984	105	Yes
8	AOAC 971.28 – 18 ED	105	Yes
9	---	105	No
10	---	103	No
11	ISO 1442:1997	104	No
12	---	102	No
13	---	---	---
14	---	105	No
15	---	105	No
16	---	105	No
17	AOAC 925.09	98 – 100, vacuum	Yes
18	AOAC 925.45	100	No
19	---	105	Yes
20	Korea Food Code	105	Yes
21	---	130	Yes
22	GB/T 5009.3-2003	105	No
23	Gravimetric (ASU 06.00-3, mod./ASU 02.09-4, mod./ASU 02.06-E, 1-8; mod.)	102	Yes
24	GB/T 5009.3 - 2003	104	No
25	AOAC 925.10	130	No
26	In-house method	103	Yes
27	Gravimetric method	130	No
28	---	100	Yes
29	---	105	No
30	---	70	Yes
31	Loss on drying in oven	105	Yes
32	4 hours in oven	102	Yes
33	---	103	Yes
34	Gravimetrico NCh841	103	Yes
35	AOAC 925.45	60	Yes
36	NOM-116-SSA1-1994	100	Yes
37	ISO 1442:1997	105	No
38	GB/T 5009.3 – 2003	105	Yes
39	---	102	No
40	Gravimetric determination after dried at 105 °C	105	No
41	AOAC 950.46	105	No
42	---	100	Yes
43	---	100	No
44	27.4.03 AOAC (2000)	102	No
45	MNS 0348:84 Determination of moisture by oven drying method	130	Yes
46	---	107	No
47	---	vacuum, 70	No
48	AOAC 935.36 2005	70	Yes

"---" denotes data/information was not provided by participants

TABLE XXI: z-Scores of participants

Lab Code	Total lipids	Sat fat	Trans fat	Protein	Sugars	TDF	Sodium	Ash
1	-0.55	0.60	1.50	1.03	-0.26	**4.07**	**5.55**	-1.26
2	---	---	---	**65.39**	---	---	---	-0.19
3	-0.39	0.35	-2.24	0.20	0.45	**8.70**	-1.30	1.69
4	0.26	---	---	0.94	---	---	---	-0.99
5	0.19	-0.64	0.34	0.15	0.18	-0.57	-0.43	0.53
6	0.58	0.85	-0.31	0.43	**4.06**	-1.44	-1.55	1.24
7	-0.11	0.09	0.49	-0.61	0.72	-0.83	---	-0.61
8	-1.03	-0.02	-0.06	0.15	1.11	-0.25	0.10	1.06
9	-0.71	-2.63	-1.61	0.48	0.01	-0.71	**12166**	-0.10
10	-0.39	0.23	-2.04	-1.23	-0.81	---	0.59	-1.26
11	---	---	---	**3.01**	---	---	-2.34	1.06
12	-0.21	0.47	---	-0.81	---	---	**12318**	-0.10
13	---	---	---	---	0.27	---	-0.10	---
14	1.06	---	---	-1.04	-0.48	---	0.59	-0.37
15	**4.27**	---	---	-0.26	**-6.81**	-0.60	-1.45	0.70
16	**-5.05**	-2.47	-0.74	2.13	0.49	**10.33**	0.43	0.88
17	1.06	-0.16	**5.59**	-0.44	-1.36	0.36	-0.31	0.08
18	0.26	0.72	-0.50	-0.77	1.07	-0.64	0.15	0.79
19	-0.18	0.96	---	0.57	0.02	2.70	0.08	-0.55
20	-1.09	---	---	1.95	---	---	---	0.88
21	-0.55	0.97	---	-0.72	1.51	-0.82	0.00	-0.90
22	1.70	-2.87	0.46	-1.14	**-7.93**	**4.68**	-0.87	2.22
23	0.40	0.30	-1.73	0.98	-0.52	**4.07**	0.66	0.17
24	---	---	---	---	---	---	0.13	1.06
25	0.74	0.72	0.14	-1.41	-1.10	-0.73	1.07	0.26
26	-1.19	-0.74	---	-1.37	1.02	-0.21	0.08	-1.35
27	-2.15	-1.12	-0.56	0.06	-0.66	1.20	**3.52**	-0.10
28	0.58	-0.42	-0.56	0.48	-0.35	0.19	-0.99	0.26
29	-0.39	---	---	-0.40	**14.83**	---	**-5.91**	-0.28
30	0.58	**50.06**	---	0.61	0.14	-1.04	-1.17	0.35
31	-0.22	-0.63	1.09	-0.54	-0.08	**9.17**	---	0.79
32	**-4.56**	**49.31**	**11.37**	0.29	-0.66	---	-0.36	1.51
33	0.90	-0.52	---	0.48	-0.57	-0.15	0.84	0.53
34	0.88	0.89	-2.56	1.35	-0.47	0.85	-1.73	-2.11
35	-0.22	**-10.55**	---	-0.49	**5.36**	**3.60**	-0.43	-1.17
36	-0.75	---	---	0.04	1.93	---	**151.08**	-0.17
37	1.17	-0.66	-0.18	-1.14	-2.82	-0.22	-0.31	-0.63
38	-0.55	0.23	-0.01	0.06	-0.48	**-3.12**	0.33	-0.19
39	1.86	---	---	0.15	---	---	---	0.88
40	**3.79**	-0.22	---	-0.91	-0.52	-0.68	0.48	-0.99
41	1.06	0.35	0.57	-0.54	-0.66	-0.61	0.41	-0.01
42	0.42	-1.14	-0.18	-0.63	**3.10**	-1.01	**12344**	-1.79
43	-1.17	---	---	-2.15	**3.53**	1.92	---	0.17
44	-0.06	-0.59	0.46	-0.35	-2.78	-0.11	-0.69	-0.99
45	-0.64	**48.33**	1.11	**4.30**	-0.32	---	---	0.88
46	0.26	---	---	0.89	---	---	-1.02	-0.19
47	0.42	0.35	1.23	0.15	0.85	-0.57	-0.18	-0.19
48	-1.67	---	---	-0.77	---	0.01	---	-1.62

"---" denotes data/information was not provided by participants
Unsatisfactory z-score results are bolded
.

CHART I: z-Scores of Participants' for Total Lipids

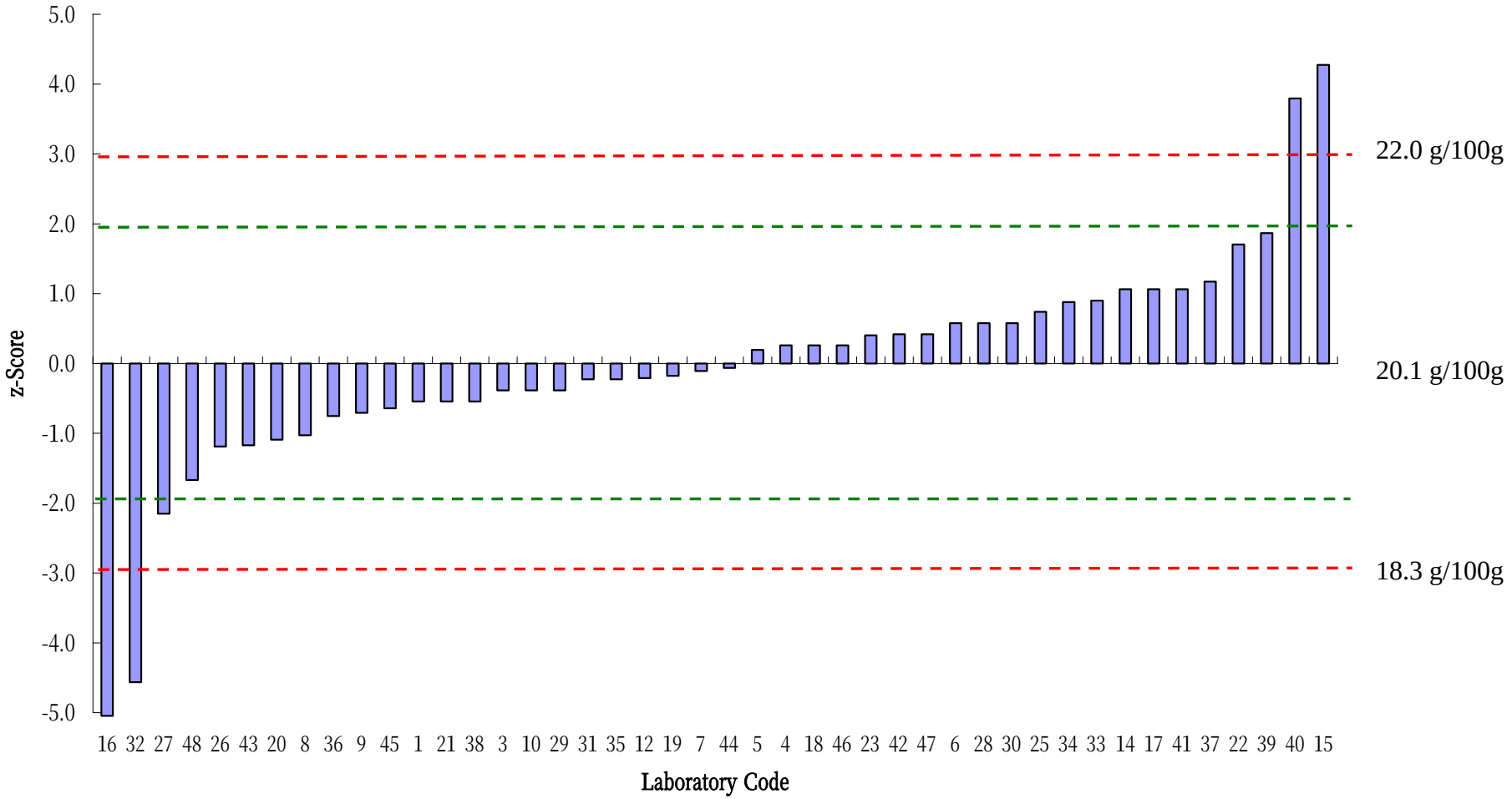


CHART II: z-Scores of Participants' for Saturated Fat

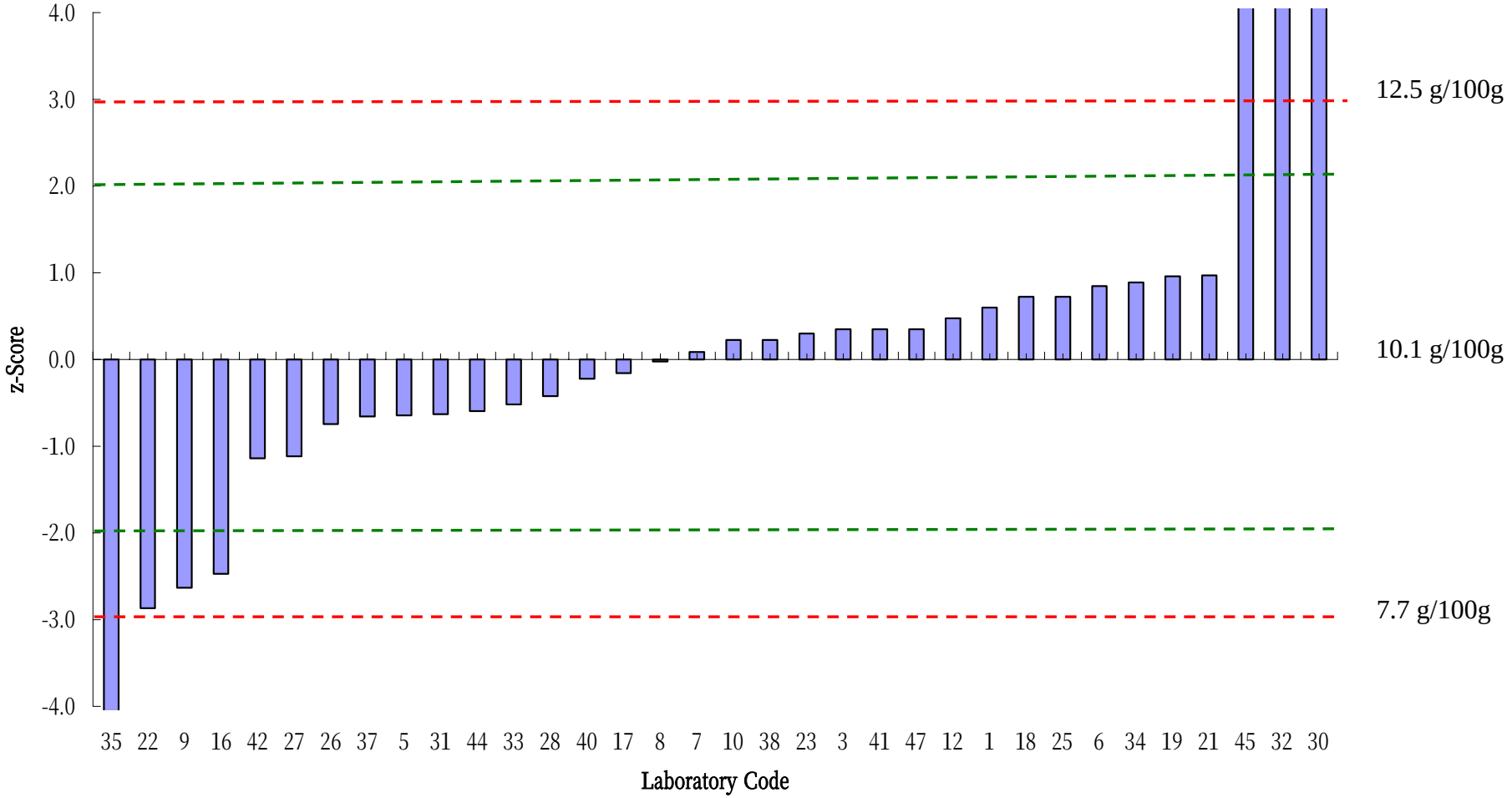


CHART III: z-Scores of Participants' for Trans Fat

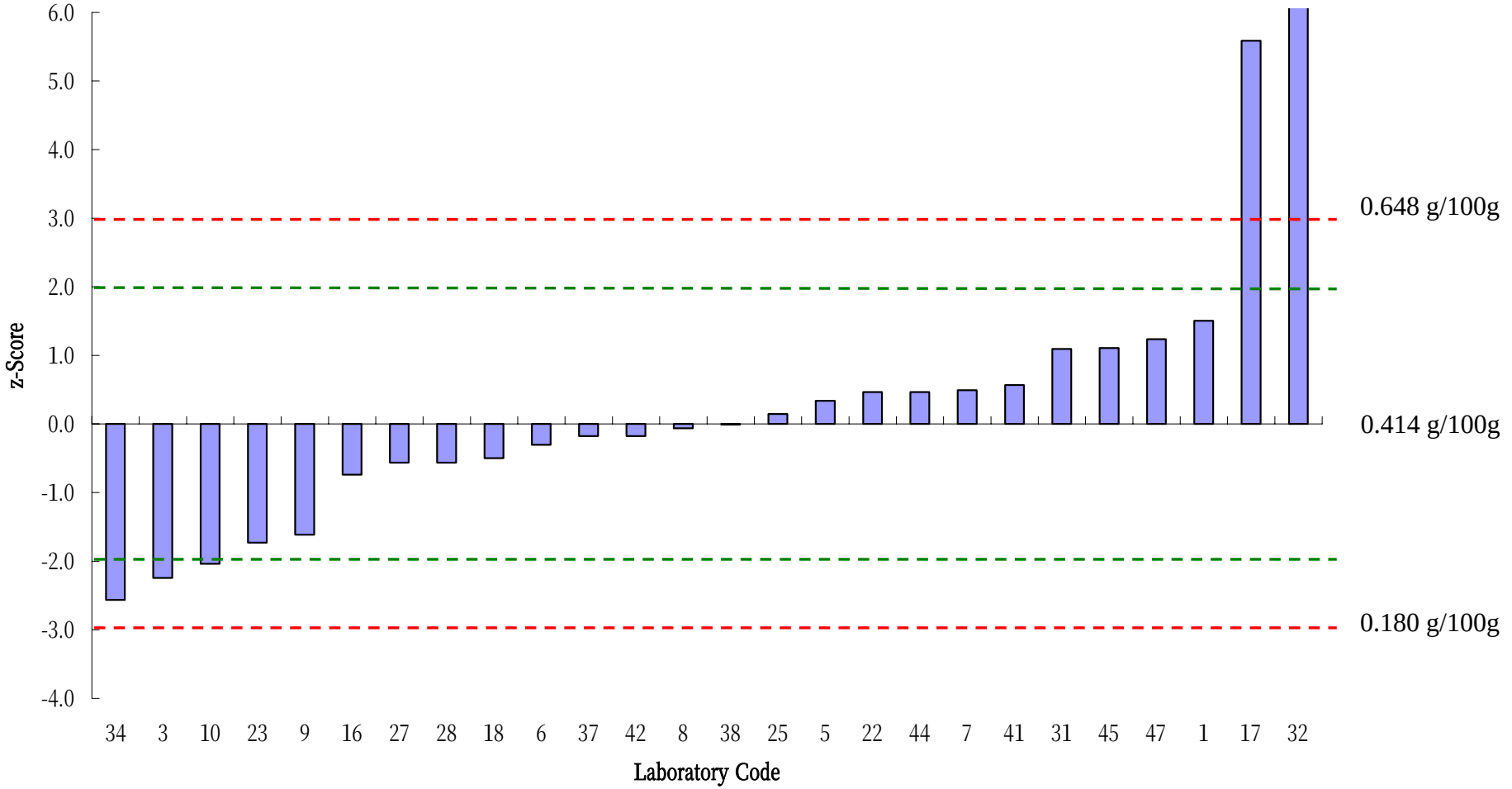


CHART IV: z-Scores of Participants' for Protein

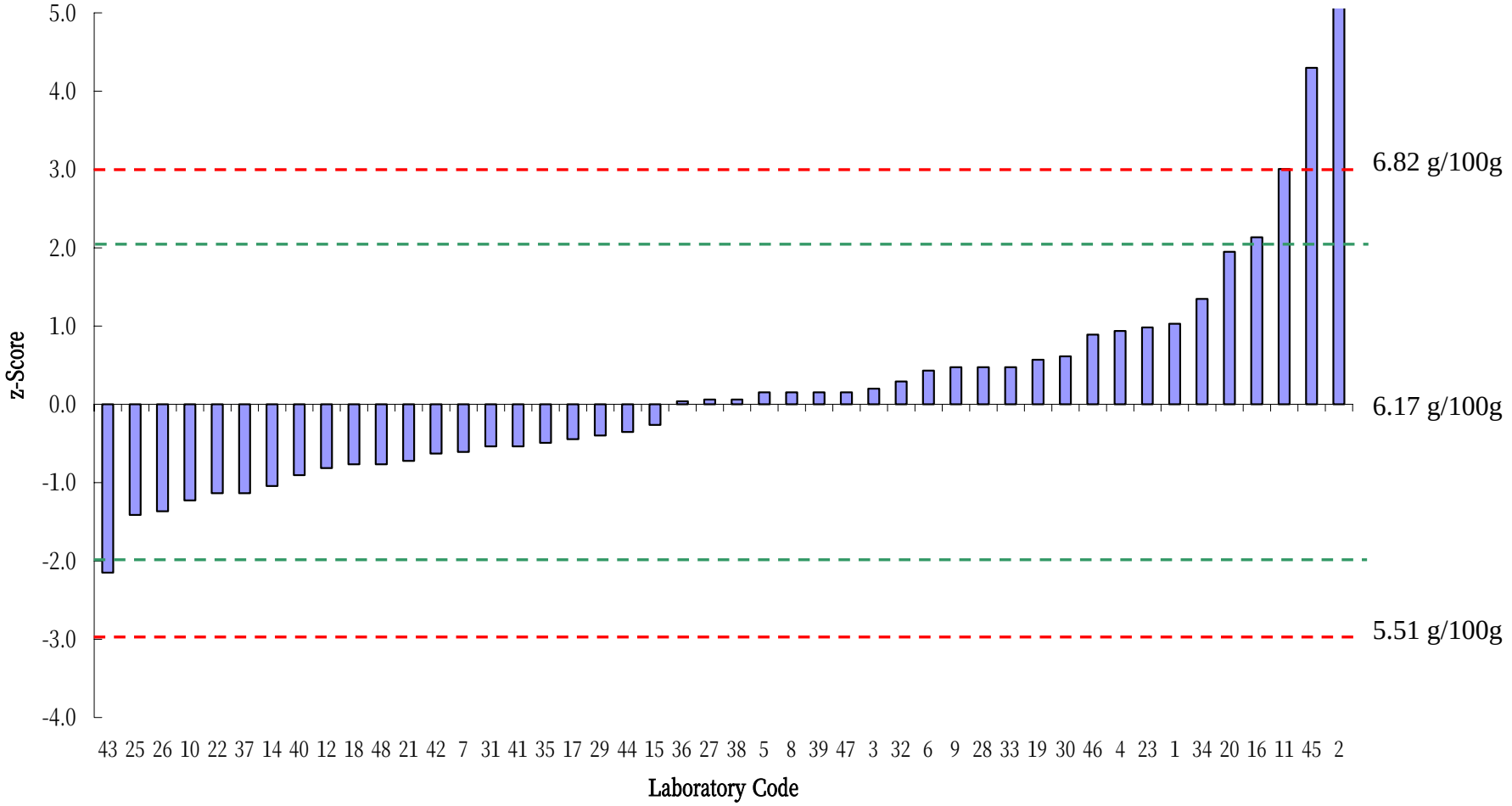


CHART V: z-Scores of Participants' for Sugars

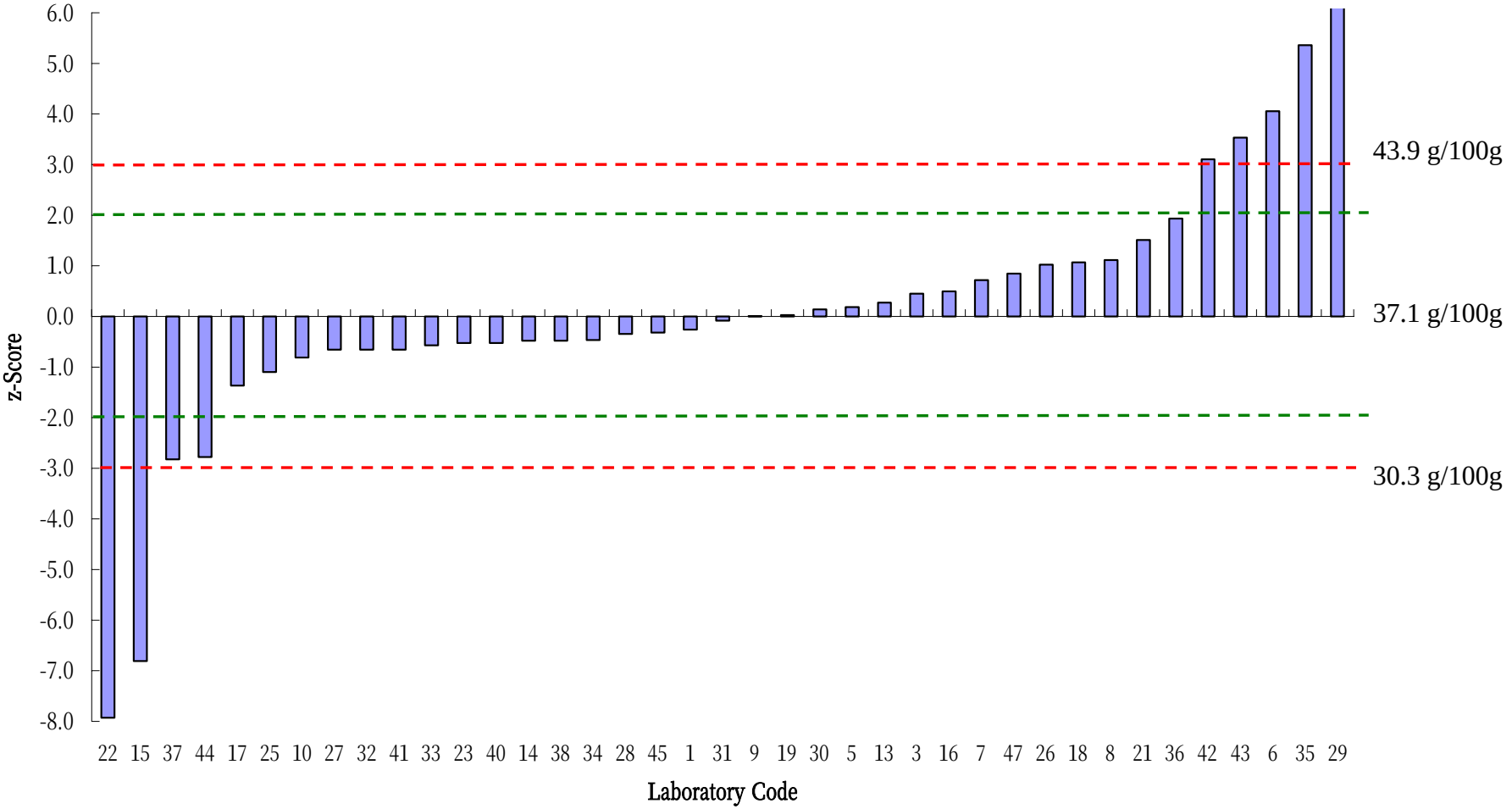


CHART VI: z-Scores of Participants' for Total Dietary Fibre

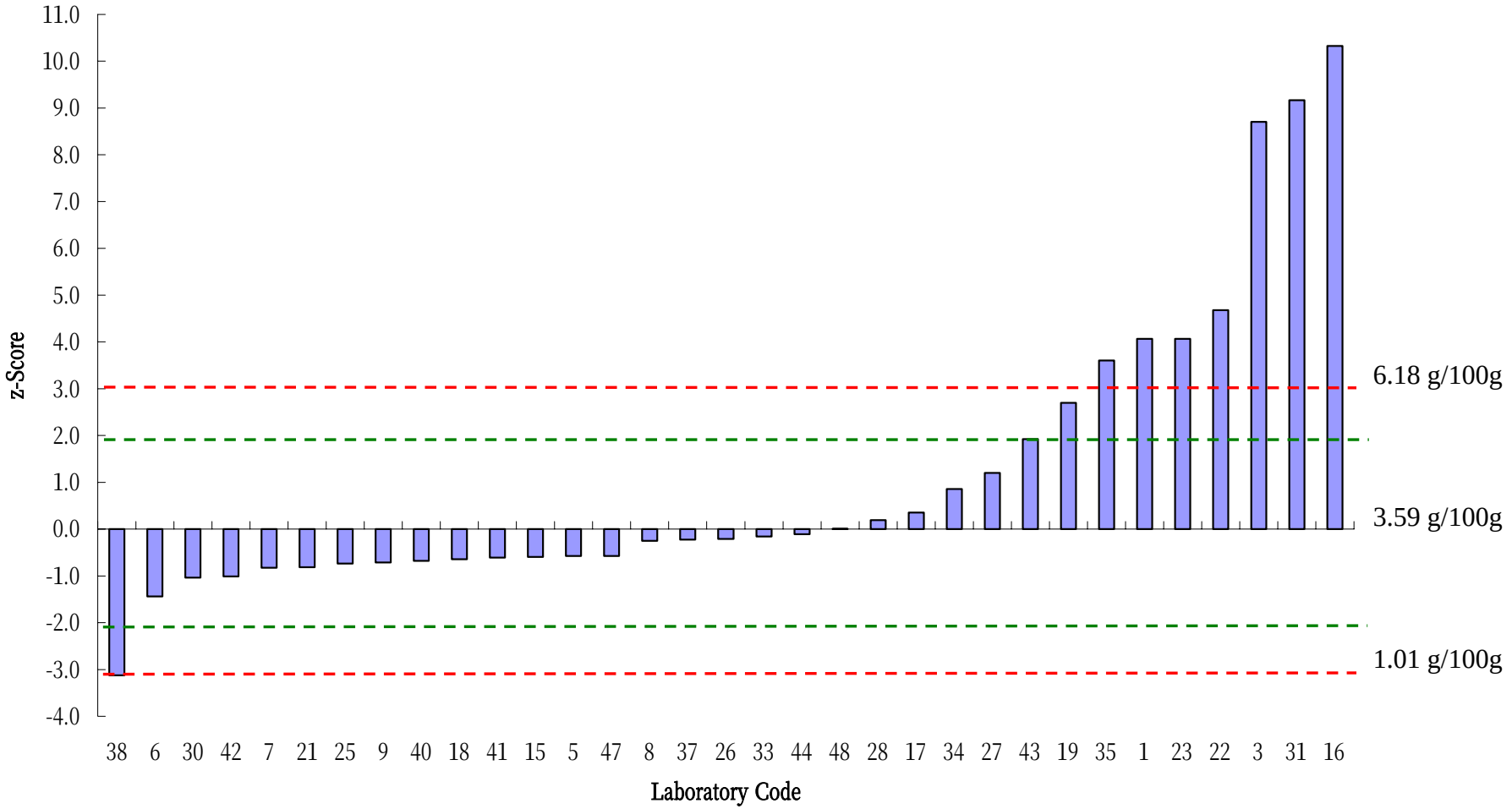


CHART VII: z-Scores of Participants' for Sodium

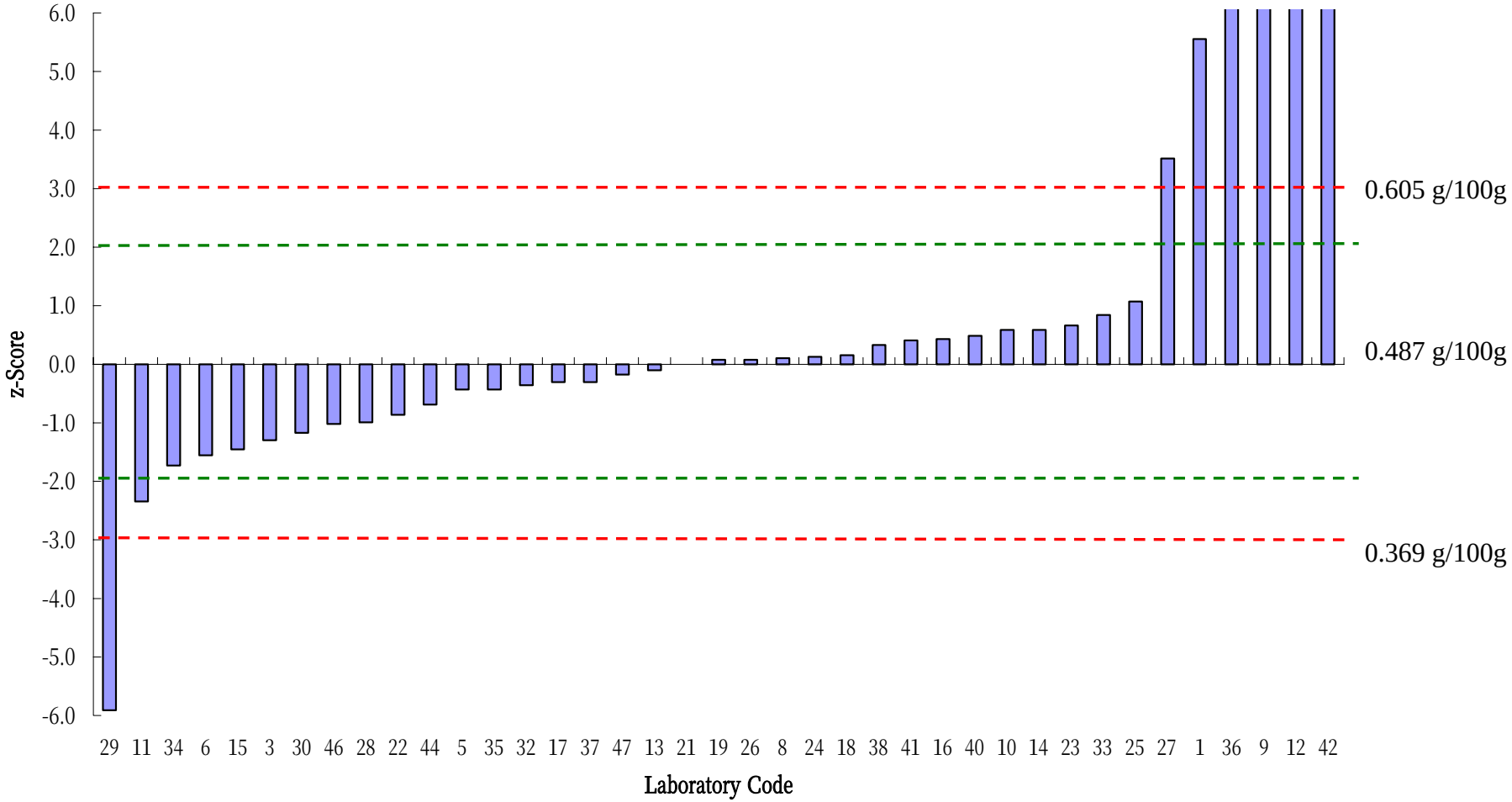
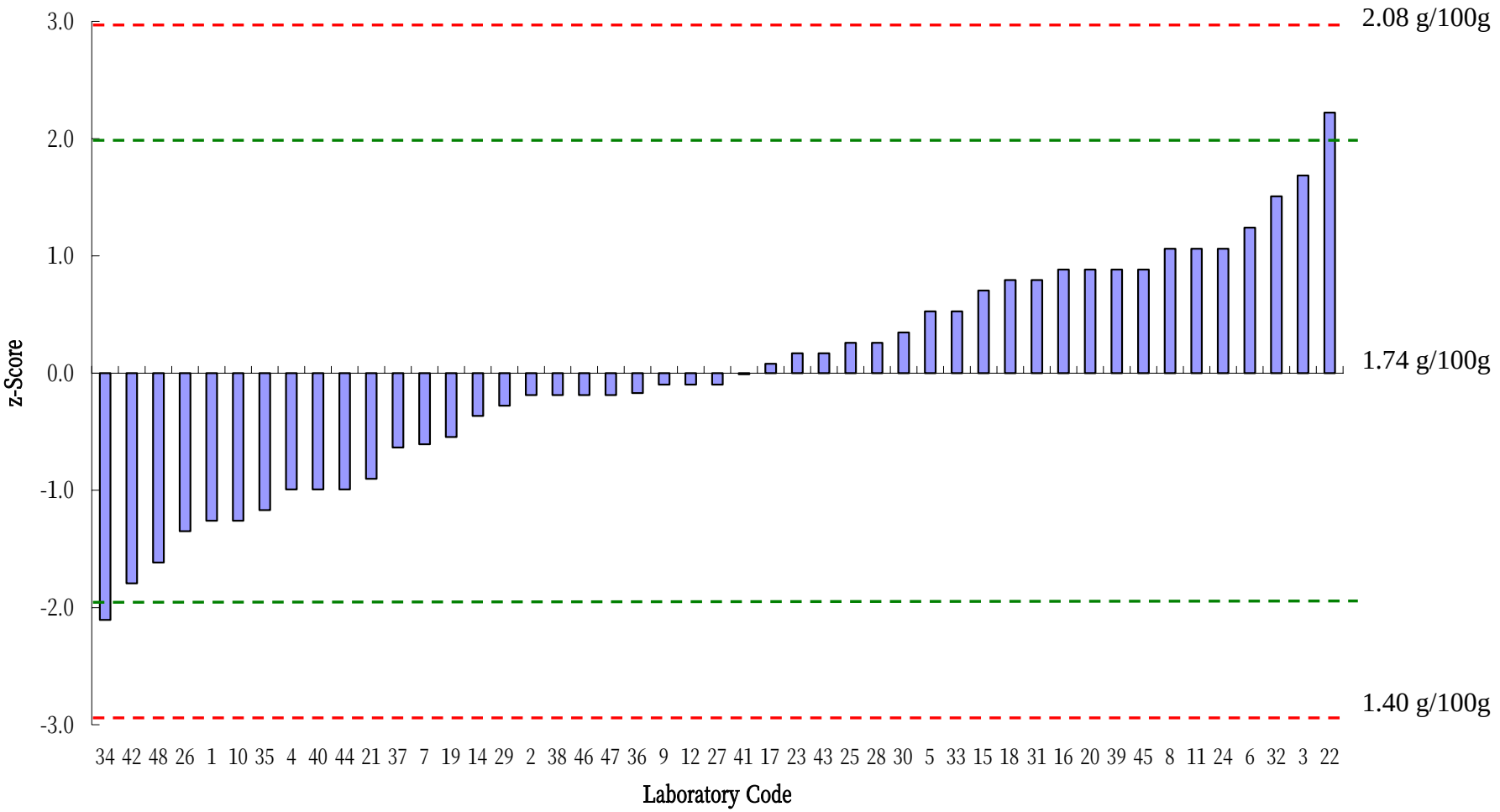


CHART VIII: z-Scores of Participants' for Ash



APPENDIX I: Homogeneity Test

a. A total of 10 bags of sample were randomly selected from a total of 66 bags. Two test portions (sub-samples) were taken from each bag for duplicate analysis of total lipids, saturated fat, trans fat, protein, sugars, total dietary fibre, moisture, ash and sodium between July and August 2009.

b. The results, together with their statistical evaluation, are given below.

Sample code	Concentration (g/100 g) of duplicate analysis					
	Total lipids		Saturated fat		Trans fat	
	#1	#2	#1	#2	#1	#2
29	20.9	20.5	9.41	9.00	0.308	0.271
17	20.1	20.6	9.20	9.24	0.285	0.293
50	20.5	20.5	9.66	9.56	0.301	0.303
57	21.2	20.4	9.25	9.78	0.287	0.305
64	20.1	21.1	9.13	9.27	0.283	0.278
49	20.5	20.4	9.57	9.47	0.299	0.298
2	20.4	20.1	9.12	9.45	0.272	0.276
10	21.3	20.9	8.97	9.64	0.272	0.302
7	20.5	20.2	9.46	9.33	0.298	0.293
18	20.4	21.0	9.23	9.31	0.286	0.291
Mean ($\bar{x}$)	20.6		9.35		0.290	

Sample code	Concentration (g/100 g) of duplicate analysis					
	Protein		Sugars		Total dietary fibre	
	#1	#2	#1	#2	#1	#2
29	6.10	6.08	36.4	36.8	2.85	2.18
17	6.11	6.04	34.9	36.7	3.12	2.81
50	6.02	6.10	36.7	36.0	2.29	2.69
57	6.09	6.11	36.4	36.5	3.49	2.95
64	6.08	6.12	37.1	36.7	3.40	2.88
49	6.08	6.04	36.4	36.2	3.36	2.84
2	6.04	6.04	37.9	36.4	2.86	3.27
10	6.08	6.04	36.7	36.2	3.04	2.12
7	6.04	6.09	36.2	35.6	3.85	2.96
18	6.07	6.14	35.7	36.0	3.40	2.84
Mean ($\bar{x}$)	6.07		36.4		2.96	

Sample code	Concentration (g/100 g) of duplicate analysis					
	Moisture		Ash		Sodium	
	#1	#2	#1	#2	#1	#2
29	1.97	2.34	1.84	1.78	0.490	0.477
17	2.21	2.01	1.79	1.78	0.483	0.480
50	2.05	2.02	1.73	1.81	0.506	0.487
57	1.94	2.18	1.80	1.79	0.501	0.481
64	1.96	1.98	1.81	1.81	0.469	0.478
49	1.96	1.98	1.80	1.79	0.483	0.484
2	2.10	2.28	1.79	1.81	0.488	0.457
10	1.92	1.94	1.80	1.82	0.498	0.489
7	1.90	2.03	1.82	1.80	0.480	0.468
18	2.04	1.95	1.80	1.82	0.486	0.468
Mean ($\bar{x}$)	2.04		1.80		0.483	

c. According to ISO 13528:2005, the samples may be considered to be adequately homogenous if:

$$s_s \leq 0.3\sigma_R$$

where:

s_s = between-samples standard deviation, g/100 g

σ_R = standard deviation for proficiency assessment, g/100 g

Analyte	$\bar{x}$ (g/100 g)	σ_R (g/100 g)	$0.3\,\sigma_R$ (g/100 g)
Total lipids	20.6	0.62	0.186
Sat fat	9.35	0.80	0.240
Trans fat	0.29	0.078	0.023
Protein	6.07	0.22	0.066
Sugars	36.4	2.3	0.69
Total dietary fibre	2.96	0.86	0.258
Moisture	2.04	0.29	0.087
Ash	1.80	0.11	0.033
Sodium	0.483	0.039	0.012

s_s is calculated as:

$$\sqrt{s_x^2 - s_w^2/2}$$

where:

s_x = standard deviation of sample averages

s_w = within-samples standard deviation

s_w is calculated as:

$$\sqrt{\sum w_t^2/2n}$$

where:

w_t = absolute difference of the duplicate sample

n = number of tested samples

The standard deviation of sample average (s_x), within-samples standard deviation (s_w) and between-samples standard deviation (s_s) of the analytes are summarized as follows.

Analyte	s_x	s_w	s_s	$0.3\sigma_R$
Total lipids	0.253	0.373	0.074	0.186
Sat fat	0.148	0.232	0.071	0.240
Trans fat	0.008	0.012	0.001	0.023
Protein	0.022	0.032	0.007	0.066
Sugars	0.448	0.581	0.178	0.69
Total dietary fibre	0.319	0.427	0.103	0.258
Moisture	0.089	0.122	0.021	0.087
Ash	0.013	0.025	0.011	0.033
Sodium	0.009	0.011	0.003	0.012

d. The between-samples standard deviation (s_s) for total lipids, saturated fat, trans fat, protein, sugars, total dietary fibre, moisture, ash and sodium are less than their corresponding $0.3\sigma_R$. It is concluded that the samples are adequately homogenous for use in the testing scheme.

APPENDIX II: Stability Test

a. Stability of total lipids, saturated fat, trans fat, protein, sugars, TDF, moisture, ash and sodium was monitored in mid November 2009, which is closed to the time when the results were submitted by the participants.

b. Samples selected for the stability test were stored in room temperature of 25°C. Three samples were randomly selected and each analysed in duplicate under the same conditions as those in the homogeneity test.

c. Stability of samples was evaluated from the absolute difference between the mean value obtained from homogeneity test ($\bar{x}$) and the mean value ($\bar{y}$) of each stability test. According to ISO 13528:2005[9.1], the absolute difference should not be more than 30% of the standard deviation for proficiency assessment (σ_R)

$$\left|\bar{x} - \bar{y}\right| \leq 0.3\sigma_R$$

d. The stability test was summarized below:

Sample code	Concentration (g/100 g) of duplicate analysis					
	Total lipids		Saturated fat		Trans fat	
	#1	#2	#1	#2	#1	#2
21	20.6	20.5	9.41	8.85	0.311	0.289
33	20.6	20.3	9.29	9.50	0.293	0.303
65	20.9	20.5	9.46	9.44	0.275	0.282
$\bar{y}$	20.6		9.33		0.292	
$\bar{x}$	20.6		9.35		0.290	
$\left\|\bar{x} - \bar{y}\right\|$	0.0		0.02		0.002	
$0.3\sigma_R$	0.186		0.240		0.023	
Status	Pass		Pass		Pass	

Sample code	Concentration (g/100 g) of duplicate analysis					
	Protein		Sugars		Total dietary fibre	
	#1	#2	#1	#2	#1	#2
21	5.98	6.05	36.4	36.5	3.37	3.37
33	6.08	6.03	36.1	36.1	2.93	2.67
65	5.99	6.11	36.4	36.6	2.62	2.94
$\bar{y}$	6.04		36.3		2.98	
$\bar{x}$	6.07		36.4		2.96	
$\lvert\bar{x}-\bar{y}\rvert$	0.033		0.1		0.02	
$0.3\sigma_R$	0.066		0.69		0.258	
Status	Pass		Pass		Pass	

Sample code	Concentration (g/100 g) of duplicate analysis					
	Moisture		Ash		Sodium	
	#1	#2	#1	#2	#1	#2
21	2.54	2.54	1.79	1.78	0.484	0.482
33	2.49	2.53	1.78	1.81	0.463	0.495
65	2.52	2.53	1.80	1.77	0.473	0.483
$\bar{y}$	2.52		1.79		0.480	
$\bar{x}$	2.04		1.80		0.483	
$\lvert\bar{x}-\bar{y}\rvert$	0.48		0.01		0.003	
$0.3\sigma_R$	0.087		0.033		0.012	
Status	Fail		Pass		Pass	

e. Results showed that the content of moisture for the vacuum packed samples increased by about 0.5 g/100g after stored for 3 months.

f. Apart from moisture, the results of the absolute differences between value obtained from homogeneity test ($\bar{x}$) and the mean value ($\bar{y}$) of each stability test for total lipids, saturated fat, trans fat, protein, sugars, total dietary fibre, ash and sodium were less than their corresponding $0.3\sigma_R$. The material was considered to be stable at 25°C during the proficiency test period.

APPENDIX III: Instructions to Accreditation Bodies

**INSTRUCTIONS
TO ACCREDITATION BODY**

1. ORGANISERS

The PT program on "Nutrients in Food (APLAC T073)" is organised by the Centre for Food Safety (CFS) in collaboration with Hong Kong Accreditation Service (HKAS) under the auspices of the Asia-Pacific Laboratory Accreditation Cooperation (APLAC). The main objectives of this proposal are to provide an analytical forum for participants in the quantitative analysis of nutrients in food sample and to evaluate the measurement capability of participating laboratories that providing such testing services.

2. SAMPLE

Accreditation bodies (AB) should receive the PT samples and instruction information (hard copy and soft copy) from the organisers. The PT sample consists of **ONE** plastic bag, and each containing about 120 g of fine biscuit powder. Upon receipt, AB should carefully check the samples (number of plastic bags, physical conditions, etc.) and shall promptly acknowledge the organisers by returning the "Receipt Form (for Accreditation Bodies)" electronically to calvinho@itc.gov.hk. New sample(s) will be replaced for any missing and damaged bags.

AB shall distribute the samples to their nominated laboratories as soon as possible. The samples are recommended to be stored in a secure environment at room temperature (around 25°C) if immediate dispatch is not possible.

AB shall ask their nominated laboratories to check the samples carefully upon receipt and promptly acknowledge the organisers by returning the "Receipt Form (for Participating Laboratories)" electronically to calvinho@itc.gov.hk

3. RESULTS

AB shall remind their nominated laboratories to complete the "Result Proforma" and submit electronically to the organisers CFS on or before **14 Nov 2009** at chtran@fehd.gov.hk. (Notes: 1. Under normal circumstances, results submitted after the deadline will not be accepted. 2. Owing to possible poor transmission quality, results by fax are generally not acceptable.)

4. ENQUIRIES

Please contact the organizers for further enquiries at chtran@fehd.gov.hk

APPENDIX IV: Receipt Form for Accreditation Bodies

RECEIPT FORM
(FOR ACCREDITATION BODY)

Hong Kong Accreditation Service (HKAS)

Contact Person: Mr Calvin Ho

Phone: +852 2829 4804

Fax: +852 2824 1302

Email: calvinho@itc.gov.hk

Accreditation Body Information

Name: _____

Contact Person: _____

Phone: _____ Fax: _____

E-mail: _____

Sample received date: _____

I hereby acknowledge the receipt of _____ sample(s) for the APLAC T073 programme.

The sample(s) is/are found (damaged / satisfactory)* and should be (suitable / not suitable)* for laboratory testing.

Problem with sample(s), if any:

---

*Please delete as inappropriate

APPENDIX V: Instruction to Participating Laboratories

INSTRUCTIONS TO PARTICIPATING LABORATORIES

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

1. Participating laboratories are supplied with a bag of powered biscuit sample, which is labelled as “Test Sample”.

2. Upon receipt of the samples, participating laboratories should carefully inspect the samples for any physical damages and defects. If presence of abnormality is detected, please contact the organizers at calvinho@itc.gov.hk immediately. New sample will be replaced if necessary.

3. If the conditions of the samples are satisfactory, participating laboratories shall promptly acknowledge the receipt of the samples by returning the “Receipt Form” electronically to the organizers at calvinho@itc.gov.hk.

4. Analysis may commence as soon as the sample is received. The sample should be stored in the original packaging at room temperature until analysis commences.

5. The following analyses are to be performed. Analysis results, in g/100g on as-received basis, and other information should be reported on the given Result Proforma.

   i. Protein [multiply percent nitrogen by factor 6.25 to calculate percent protein]
   ii. Total lipids [total amount of fats (free and bound) and fat soluble substances]
   iii. Saturated fat [sum of saturated fatty acids, e.g. $C_{4:0}$, $C_{6:0}$, $C_{8:0}$, $C_{10:0}$, $C_{12:0}$, $C_{14:0}$, $C_{15:0}$, $C_{17:0}$, $C_{18:0}$, $C_{20:0}$, $C_{24:0}$, etc.]
   iv. Trans fatty acids [sum of all the geometrical isomers of monounsaturated and polyunsaturated fatty acids having non-conjugated, interrupted by at least one methylene group, carbon-carbon double bonds in the trans configuration, e.g. $C_{14:1T(9\text{-}trans)}$, $C_{16:1T(9\text{-}trans)}$, $C_{18:1T(total)}$, $C_{18:2TT(9,\ 12\text{-}trans)}$, $C_{18,2T(9\text{-}cis,\ 12\text{-}trans)}$, $C_{18:2T(9\text{-}trans,\ 12\text{-}cis)}$, $C_{20:1T(11\text{-}trans)}$, $C_{22:1T(13\text{-}trans)}$, etc.]
   v. Sugars [sum of monosaccharides and disaccharides present]
   vi. Sodium
   vii. Moisture
   viii. Ash
   ix. Total dietary fibre

   Participating laboratories should be aware of analyte stability and perform the analyses in an appropriate order.

6. Except the analysis of total dietary fibre, participating laboratories should use the most appropriate analytical methods which would normally be used to analyse customer samples. For “sugars”and “trans fatty acids”, the analysis results should be conforming to the respective definitions given by Codex Alimentarius (CAC/GL 2-1985). AOAC Official Method should be used for the analysis of total dietary fibre.

7. Estimate and report the expanded measurement uncertainty (± % base) for each result.

8. Send the completed results sheet to the organizers on or before 14 November 2009 at chtran@fehd.gov.hk using the electronic copy of the “Result Proforma” provided.

   (Notes: 1. Under normal circumstances, results submitted after the deadline will not be accepted. 2. Owing to possible poor transmission quality, results submitted by fax are generally not acceptable.)

9. For enquires, please contact the organizers at swcchung@fehd.gov.hk.

APPENDIX VI: Receipt Form for Participating Laboratories

RECEIPT FORM
(FOR PARTICIPATING LABORATORY)

Hong Kong Accreditation Service (HKAS)

Contact Person: Mr Calvin Ho

Phone: +852 2829 4804

Fax: +852 2824 1302

Email: calvinho@itc.gov.hk

Participating Laboratory Information

Name: _____

Contact Person: _____

Phone: _____ Fax: _____

E-mail: _____

Sample received date: _____

I hereby acknowledge the receipt of **ONE** sample for the APLAC T073 programme.

The sample is found (damaged / satisfactory)* and should be (suitable / not suitable)* for laboratory testing.

Problem with the sample, if any:

---

*Please delete as inappropriate

APPENDIX VII: Results Proforma

Laboratory Code: [ ]
(will be assigned later)

Name of participating laboratory: _____

Contact person / e-mail: _____

Test	Result (g/100 g)	Measurement uncertainty		Method / technique used	Method accredited (Yes/No)
		Expanded uncertainty (g / 100 g)	Coverage factor (k)		
Protein					
Total lipids					
Saturated fat					
Trans fatty acids					
Sugars					
Sodium					
Moisture				(Drying Temp: °C)	
Ash				(Ashing Temp: °C)	
Total dietary fibre					

Note: If the concentration is less than your limit of quantification (LOQ), please specify the LOQ, e.g. <0.50 g /100g

§ Report all values to 3 significant figures.

◗ It is the responsibility of the participants to avoid collusion and falsification of result so as to ensure a reliable assessment to be made in this proficiency testing program. Information on the identities and results of the participating laboratories will be kept confidential to the respective nominating accreditation bodies and the organisers (APLAC, HKAS and CFS). If laboratories submit their results through their accreditation bodies, their results may be disclosed to and released through their accreditation bodies.

◆ Please submit this "Results Proforma" electronically to the organisers on or before 14 November 2009 at chtran@fehd.gov.hk.