ASIA PACIFIC FOOD ANALYSIS NETWORK (APFAN)

APFAN activity: Proficiency Testing 2 (PT-2) to Improve Food Laboratory Analyses in the Asia Pacific Region

Final Report of APFAN PT-2 (2019):

Defatted soybean flour

by

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1. TEST MATERIAL AND PREPARATIONS

Defatted soybean flour contains total fat less than 1.5 g/100g compared to 21-22 g/100 g (Judprasong et al., 2018) in the full-fat soybean flour. To expand the shelf life, the defatted soybean flour was selected to be used as the test material in the APFAN PT-2 programme. Ten kilograms of the fine particles (<250 μm) defatted soybean flour were purchased from Kasetsart University, Thailand. It was mixed in V-shape mixer for 2 hours and then packed under vacuum in aluminum foil bags, about 50 g each. The bags of the test sample were labelled which included name of the test material, date of sample preparation and sample code number. The PT test materials were kept in a freezer at -20°C. They were distributed to registered participants at the APFAN-PT1 Workshop in Indonesia in 2018 or sent by post from representative of each country. The e-documents, i.e,., instruction, participant information , report forms, information on methods used, were sent to the participants by e-mail as attached files.

2. STATISTICAL EVALUATION

The statistical procedure for each step was chosen by professional judgment according to ISO 13528: 2015 (Statistical methods for use in proficiency testing by interlaboratory comparison).

2.1 Homogeneity testing of representative nutrients in test material

Ten packages of test materials were strictly selected at random. Homogeneity of the test material was evaluated by analysing representative nutrients (i.e. moisture, protein, ash, dietary fibre), in duplicate, from each package in a random order, in one setting, and under repeatability conditions, by ISO/IEC 17025:2005 accredited laboratories. For homogeneity testing, SGS Co. Ltd. contributed to the PT-2 programme by analyses of moisture, protein and ash; the Institute of Nutrition, Mahidol University, analysed total dietary fibre. All analytical methods used are based on the international AOAC methods (AOAC, 2016) which were well validated before use, i.e. AOAC 927.05 was used for moisture, AOAC 991.20, for protein, AOAC 930.30 for ash, and AOAC 985.29 for dietary fibre . The results were statistically evaluated for homogeneity testing of the test materials without removal of any value. Within-sample variation using Cochran's maximum range test (ISO 13528:2015, ISO 5725:1994) and between-sample variation using ISO 13528 approach were evaluated. For the first approach, the between-sample variation due to sampling (ss) should be less than 0.3 times of the standard deviation for proficiency testing (□PT), indicating adequate sample homogeneity. Standard deviation for PT of each nutrient was derived from Horwitz's equation (Horwitz W & Albert R, 2006).

2.2 Assigned value (x_{pt}) of measurands and standard uncertainty (u_x)

Assigned values of nutrients in a test material were consensus values derived from participants as robust average (x^*) or median as an x_{pt} value, For standard deviation of proficiency assessment (SDPA or σ_{pt}), it was chosen based on professional judgment using the suitable one of the following statistics namely robust standard deviation (s^*) obtained from Algorithm A of ISO 13528:2015 (s*), predicted standard deviation from Horwitz's equation (SD_p) (Horwitz W & Albert R, 2006) or normalized interquartile range (NIQR).

Assigned values, as mean and standard diviasion (X and SD), for some minerals in defatted soybean flour were determined by the National Institute of Metrology (Thailand), using primary methods. Isotope dilution Inductively Coupled Plasma Mass Spectrometric (ID-ICP-MS) was

used for determination of Cu and Zn and Gravimetric standard addition ICP-MS was used for determination of Ca, Fe, and Mg.

The detailed descriptions of each step for estimating assigned values of a measurand are presented as follows.

1) Visual inspection of the normal distribution of PT participants' results using Kernel density plot (ISO 13528: 2015). The ISO/IEC 17043:2010, B.2.5 and the IUPAC Harmonized Protocol (2006) recommend removing obvious blunders from a data set at an early stage, prior to use of any robust procedure or any test to identify statistical outliers. An example of obvious blunders are reporting results in incorrect units or switching results from different proficiency test items.

2) Use the remaining data for calculation of assigned value as robust average (x^*) and its robust standard deviation (s^*) or predicted standard deviation from Horwitz's equation (SD_p). Median and normalized interquartile range (NIQR) of each data set without outlier removal were also calculated. The decision was made for the suitability of the assigned value (acceptable %CV for each measurand) using professional judgment in combination with experience from previous rounds of a proficiency testing scheme.

3) Calculation of standard uncertainty (u_x) for each type of assigned value was used the following formula:

$$u_x = \frac{1.25\ x\ s * (or\ NIQR)}{\sqrt{p}}\ (\text{for robust values}) = \frac{SD_p}{\sqrt{p}}$$

Where u_x = standard uncertainty of the assigned value x_{pt}
s^* = robust standard deviation
NIQR = normalized interquartile range
SD_p = predicted standard deviation from Horwitz's equation
p = number of data

4) Expanded uncertainty was calculated by multiplying the standard uncertainty by the coverage factor (k=2) with a probability of 95%.

5) Determination of the suitability of the assigned value to be used as assigned value based on ISO13528: 2015 criteria as:

If $u_{x(pt)} \leq 0.3\ \sigma_{pt}$, $u_{x(pt)}$ is negligible, z score can be used.

If $u_{x(pt)} > 0.3\ \sigma_{pt}$, $u_{x(pt)}$ is large in comparison with the performance evaluation criterion, then there is a risk that some participants will receive action and warning signals, z' score can be used.

2.3 Evaluation of laboratory performance

2.3.1 Evaluation of analytical performance by z or z' scores

Laboratory performance on nutrients analyses were evaluated based on z score or z' score depending on standard uncertainty of assigned value of each parameter.

$$z\ score = \frac{(x_i - x_{pt})}{\sigma_{pt}}$$

$$z'score = \frac{(x_i - x_{pt})}{\sqrt{(\sigma_{pt}^2 + u_{x_{pt}}^2)}}$$

where x_i is the participant's result
x_{pt} is the assigned value from PT participants' results derived as robust average
σ_{pt} is the standard deviation for proficiency assessment
$u_{x(pt)}$ is the standard uncertainty of the assigned value

Interpretation of laboratory performance

|z or z' score| ≤ 2.00: Satisfactory result

2.00 < |z or z' score| < 3.00; Questionable result

|z or z' score| ≥ 3.00; Unsatisfactory result

2.3.2 Evaluation of analytical performance using measurement uncertainty by zeta score

To avoid effect of different coverage factors used by each laboratory, the zeta (ζ) score, instead of En score, was applied in this study. The ζ score is useful when an objective of the PT scheme is to evaluate a participant's ability to have results be close to the assigned value within their claimed uncertainty. ζ score was used in conjunction with z score, as an aid for improving the performance of participants and claim standard uncertainty. The ζ score can be calculated as follows:

$$\zeta = \frac{x_i - x_{pt}}{\sqrt{u_{x_i}^2 + u_{x_{pt}}^2}}$$

Where x_i is the participant's result
x_{pt} is the assigned value determined in a reference laboratory
$u_{(xi)}$ is the standard uncertainty of a participant's result x_i
$u_{xpt)}$ is the standard uncertainty of the assigned value x_{pt}

ζ scores differ from Zeta scores by using standard uncertainties $u_{(xi)}$ and $u_{(xpt)}$, rather than expanded uncertainties $U_{(xi)}$ and $U_{(xpt)}$. ζ scores can be interpreted using the same critical values of 2.0 and 3.0 as for z scores. An adverse ζ score may indicate either a large deviation of x_i from x_{pt}, an under-estimate of uncertainty on the part of the participant, or a combination of both. The unsatisfactory ζ score could indicate a need to review the uncertainty estimates, or to correct a measurement issue.

2.3.3 Graphic presentation:

1) Individual results sorting from lowest to highest, with measurement expanded uncertainty.
2) Graph of ordered z or z' scores
3) Graph of Zeta score, sorting based on ordered z or z' scores
4) Graph of ordered z or z' scores, categorised in groups according to analytical methods/parameters used.

3. RESULTS AND DISCUSSION

3.1 Homogeneity of defatted soybean flour

Table 1. Within sample homogeneity checking by Cochran's maximum range test

No.	Moisture (g/100g)				Total protein (g/100g)				Ash (g/100g)				Dietary fibre (g/100g)			
	A	B	Range^2 (R)	R/R-total	A	B	Range^2 (R)	R/R-total	A	B	Range^2 (R)	R/R-total	A	B	Range^2 (R)	R/R-total
1	8.09	8.05	0.002	0.11	49.87	49.97	0.010	0.05	6.28	6.12	0.026	0.07	15.75	16.09	0.115	0.04
2	8.00	8.02	0.000	0.03	49.96	49.79	0.029	0.15	6.25	6.12	0.017	0.04	16.34	16.27	0.004	0.00
3	8.05	8.06	0.000	0.01	49.91	49.87	0.002	0.01	6.22	6.22	0.000	0.00	16.42	16.46	0.001	0.00
4	8.09	8.08	0.000	0.01	49.80	49.77	0.001	0.00	6.37	6.19	0.032	0.08	16.35	16.66	0.096	0.03
5	7.99	7.96	0.001	0.06	49.71	49.69	0.000	0.00	6.40	6.26	0.020	0.05	16.16	16.78	0.386	0.13
6	8.12	8.02	0.010	0.67	49.79	49.92	0.017	0.09	6.41	6.17	0.058	0.15	16.19	16.91	0.508	0.17
7	7.99	7.98	0.000	0.01	49.92	49.82	0.010	0.05	6.44	6.32	0.014	0.04	16.41	15.99	0.176	0.06
8	7.91	7.91	0.000	0.00	49.84	49.74	0.010	0.05	6.17	6.34	0.029	0.07	17.16	16.04	1.241	0.42
9	8.01	7.98	0.001	0.06	49.93	49.63	0.090	0.48	5.96	6.40	0.194	0.49	16.65	16.10	0.309	0.10
10	8.04	8.01	0.001	0.06	49.85	49.71	0.020	0.10	6.29	6.34	0.002	0.01	16.35	15.99	0.124	0.04
R Total			0.015	Max =		R Total	0.188	Max =			0.392	Max =			2.961	Max =
N			10	0.67		N	10	0.48			10	0.49			10	0.42
Cochran critical value[(a)] 95% CI			0.602	Not pass			0.602	Pass			0.602	Pass			0.602	Pass
	99% CI		0.718	Pass			0.718	Pass			0.718	Pass			0.718	Pass

[(a)] critical value for Cochran's maximum range test for 10 sets of data, number of results per set (n) =2, given in the ISO 5725-1981: Precision of test methods – Determination of repeatability and reproducibility by interlaboratory tests.

Table 2. Summary results of homogeneity testing (between sample variation) of defatted soybean flour

Parameter	Mean	Standard deviation (SD_r)	Relative standard deviation (RSD_r), %CV	Sampling standard deviation (*Ss*)	RSDp (%)*	σ_{pt}	0.3 σ_{pt}	Summary
Moisture (g/100g)	8.02	0.06	0.70	0.051	2.92	0.234	0.070	Pass
Protein (g/100g)	49.82	0.10	0.2	0.000	2.22	1.107	0.332	Pass
Ash (mg/kg)	6.26	0.12	1.93	0.000	3.03	0.190	0.057	Pass
Total Dietary fibre (g/100g)	16.35	0.34	2.1	0.000	2.63	0.430	0.129	Pass

***** Target standard deviation using general model: "Horwitz' s equation"

The within-sample variation, evaluated by Cochran's maximum range test, showed that all studied nutrients passed the acceptable criteria. The ratios of the maximum range to the sum of the ranges were less than Cochran's critical values at a 95 % confidence level except moisture. However the ISO 5725-2 stated that an outlier set should not be rejected unless it is significant at 99% level or any permanent analytical errors are found.
These results indicated acceptable precision of the analysts who conducted the homogeneity testing and good homogeneity of the test material in each package.

For between-sample variation, which indicates sample homogeneity, a summary of homogeneity results is shown in **Table 2**. Based on the ISO 13528 approach, the Ss for four components was less than $0.3\sigma_{PT}$ (**Table 2**), which indicated adequate nutrient homogeneity of the test material. These results indicated that all the studied nutrients in the test material were sufficiently homogeneous; thus, the test material was appropriated to be used for this laboratory performance study.

3.2 Determination of the assigned values of defatted soybean flour

Average results of all nutrients submitted by participating laboratory were used to develop assigned values as robust mean and robust standard deviation (x* and s* respectively), based on algorithm A in ISO 13528 (2015). For acceptance of s*, the relative standard deviation (%RSD) of each parameter must be at the same range of predicted relative standard deviation (RSD_p) or RSD from previous studies, as shown for moisture, nitrogen, ash, magnesium, phosphorus and zinc. The RSD_p was applied for total dietary fibre, calcium, sodium, potassium and copper (3, 2, 3, 3, and 1 times of SD_p derived from Horwitz's equation). Median and normalised interquartile range (nIQR) were used as assigned value for iron. Due to participating laboratories reported high variation of fat (with 50%RSD), this component was not included in the performance evaluation.

With the contributions from the National Institute of Metrology (Thailand), the assigned values of Ca, Fe, and Mg were developed by Gravimetric standard addition Inductively Coupled Plasma Mass Spectrometry (ICP-MS) and those of Cu and Zn by Isotope dilution ICP-MS. These assigned values for all minerals–were used as reference values as x_{pt} whereas the values of

appropriate standard deviation for proficiency assessment (σ_{pt}) were established as s*, SD_p, or nIQR based on the suitable RSD, similar to the approach of other nutrients.

Assigned values of all studied parameters are summarized in **Table 3**.

The uncertainty of the assigned values of all parameters derived for the defatted soybean flour were considered to be negligible ($u_{(xpt)} < 0.3\ \sigma_{pt}$). Based on the ISO 13528: 2015, they were not included in the interpretation of the laboratory performance. Thus, z score was used for evaluation of laboratory performance in this PT round.

Table 3. Summary: assigned values of measurands for evaluation of testing parameters in defatted soybean flour

Parameters	Method of assigned value[1]	x_{pt}	σ_{pt}	%RSD	$u_{x(pt)}$	$0.3\sigma_{pt}$	$u_{x(pt)}$ is negligible?
Moisture (g/100g)	x* & s*	7.26	0.71	9.8	0.10	0.21	Yes, use z score
Total nitrogen (g/100g)	x* & s*	7.87	0.16	2.0	0.02	0.05	Yes, use z score
Fat (g/100g)	x* & s*	1.41	0.71	50.4	0.14	0.04	No, Not evaluate
Ash (g/100g)	x* & s*	6.31	0.33	5.2	0.05	0.10	Yes, use z score
Total dietary fibre (g/100g)	x* & 3SD_p	16.44	1.29	7.9	0.10	0.39	Yes, use z score
Calcium (mg/kg)	x* & 2SD_p	2031	207	10.2	35.8	62.0	Yes, use z score
(reference value)	X & 2SD_p	2100	207	9.8	28.6	62.0	Yes, use z score
Magnesium (mg/kg)	x* & s*	2652	343	12.9	62.5	102.8	Yes, use z score
(reference value)	X & SD_p	2650	259	9.8	37.8	102.8	Yes, use z score
Phosphorus (mg/kg)	x* & s*	7787	456	5.9	99.3	136.9	Yes, use z score
Sodium (mg/kg)	x* & 3SD_p	72.5	18.3	25.2	4.0	5.5	Yes, use z score
Potassium (mg/kg)	x* & 3SD_p	23133	2447	10.6	437	734	Yes, use z score
Iron (mg/kg)	Med. & NIQR	75.50	8.78	11.6	1.54	2.63	Yes, use z score
(reference value)	X & SD_p	75.1	6.4	8.5	0.9	2.63	Yes, use z score

Table 3. Summary: assigned values of measurands for evaluation of testing parameters in defatted soybean flour *(continued)*

Parameters	Method of assigned value[1]	x_{pt}	σ_{pt}	%RSD	$u_{x(pt)}$	$0.3\sigma_{pt}$	$u_{x(pt)}$ is negligible?
Zinc (mg/kg)	x* & s*	42.59	5.18	12.2	0.95	1.55	Yes, use z score
(reference value)	X & SD_p	43.1	3.9	9.0	0.6	1.55	Yes, use z score
Copper (mg/kg)	x* & SD_p	12.19	1.34	11.0	0.25	0.40	Yes, use z score
(reference value)	X & SD_p	12.5	1.3	10.7	0.2	0.40	Yes, use z score

[1] x* = Robust average derived from algorithm A of ISO 13538: 2015
*s** = Robust standard deviation derived from algorithm A of ISO 13538: 2015
SD_p = Predicted standard deviation from Horwitz equation
X = Mean derived from gravimetric standard addition IDMS from NIMT
Med. & NIQR = Median and normalised interquartile range

3.3 Laboratory performance on analyses of nutrients in defatted soybean flour

The submitted analytical data from the participants and the evaluated data based on z-score and zeta score are presented in separate tables for each parameter as follows: 1) individual results sorting from lowest to highest level of a nutrient with measurement expanded uncertainty, 2) graph of ordered z scores, 3) graph of zeta scores, sorting based on ordered z scores, and 4) graph of ordered z scores, categorised in groups according to analytical methods/parameters used.

Although the performances of laboratories for nutrients analyses were statistical evaluated using z-score and Zeta score; the discussion on laboratory performances on main nutrients analyses and few points discussion on minerals were based on z-score only. The main results and discussion on minerals are presented at the APFAN PT-2 Workshop.

MOISTURE

The submitted and evaluated data are presented in **Table 4** and **Figures 1 to 4**. As shown in **Table 4**, participants applied three standard methods for determination of moisture content in the defatted soybean flour. About 51% of the participating laboratories (40 out of 78) used drying temperature in an oven at 125-135^{o}C, for 1 to 5 h (AOAC 925.10, 2016). Others applied drying temperature at 100-105^{o}C for 2 to 7.5 h (ISO 6496, and SNI 01-2891-1992). According to the results presented in **Figure 4,** although the difference on the temperatures used for drying the sample has generally no effect on the performance of moisture analysis (Iz-scoresI $\leq$ 2) but it was noticed that those lab who dried the sample at 125-135^{o}C reported lower levels of moisture content.

Drying the sample until constant weight for moisture determination is strongly recommended.

The reference value of moisture content in the defatted soybean flour obtained from 70 good performance laboratories is 7.27 $\pm$ 0.57 g /100 g (Mean $\pm$ SD, %CV= 7.8).

Table 4. Laboratory performance on **moisture** analysis (g/100 g, as received) in defatted soybean flour*

Laboratory Number	Moisture (g/100g)	MU (g/100g)	z score	Zeta score	Sample weight (g)	Temp. (^{o}C)	Time (Hours)	Method Reference
Assigned value obtained from robust average (x) ± robust SD (s*) = 7.26 ± 0.71 g/100 g (CV 9.8%, n= 79) with u_{xpt} = 0.10 g/100 g*								
	Acceptance criteria =		|z score| ≤ 2.00	|ζ score| < 2.00				
2	**7.92**	-	0.93	-	2.00	135	2	AOAC (2016) 930.15
4	**8.07**	0.13	1.14	**6.79**	2.xxxx	130 ± 3	1	Based on AOAC
5	**7.31**	-	0.06	-	5.0000	103	4	ISO 6496:1999
6	**7.08**	0.10	-0.25	-1.62	5.0000	103	4.00	ISO 6496
8	**7.06**	0.21	-0.28	-1.38	5	105 ± 2	2	SLS 898:1990
9	**7.28**	-	0.03	-	5 to 10	105	4	Based on ISO 6496:1999
10	**7.70**	-	0.62	-	2	130	1	AOAC 2012, 32.2.09 A, Chapter 32
11	**8.06**	-	1.13	-	5.0000	103	4	AOAC (2016) 925.10
12	**6.36**	0.03	-1.27	**-8.90**	1	100 ± 1	3	AOAC (2016) 952.08
14	**7.98**	0.30	1.01	**4.00**	2	130 ± 3	1	AOAC 925.10
15	**7.81**	-	0.77	-	2	130	1	AOAC (2016) 925.10
16	**6.33**	0.05	-1.31	**-9.02**	1 to 2	105	3	SNI 01-2891-1992 Food & Beverage
18	**7.19**	-	-0.10	-	2.0	105	3	SNI 01-2891-1992
19	**7.17**	-	-0.12	-	5	105	3	AOAC 934.01
21	**7.85**	0.03	0.83	**5.84**	2	130	2	AOAC 925.10 (2016)
22	**7.46**	-	0.28	-	-	-	-	-
23	**7.47**	-	0.30	-	5.00	103	4	ISO 6496
25	**6.49**	-	-1.08	-	5.1745 / 5.1797	103	4	Laboratory Handbook of Methods of Food Analysis, 3rd Ed, R. Lees

Laboratory Number	Moisture (g/100g)	MU (g/100g)	z score	Zeta score	Sample weight (g)	Temp. (^{o}C)	Time (Hours)	Method Reference
Assigned value obtained from robust average (x) ± robust SD (s*) = 7.26 ± 0.71 g/100 g (CV 9.8%, n= 79) with u_{xpt} = 0.10 g/100 g*								
26	**7.08**	0.04	-0.25	-1.76	2.0	100	5	AOAC No. 925.09B
27	**6.77**	0.26	-0.69	**-2.99**	2	105	22	SNI 2354.2:2015
32	**7.48**	0.15	0.31	-	2.1003	130	3	AOAC 945.39
35	**6.62**	0.36	-0.90	**-3.11**	5.3873	100 ± 5	2	Sri Lanka Standard 1011:1994 specification for Soya Flour
37	**8.17**	-	1.28	-	3	130	3	AOAC (2016) 925.10
38	**7.29**	0.20	0.04	0.21	2.000	130	1, to constant	AOAC 925.10, 19th Ed 2012
39	**7.89**	-	0.89	-	5	130 ± 3	2	AOAC 945.39
40	**7.67**	0.05	0.58	**3.98**	-	-	-	-
41	**7.53**	0.06	0.37	**2.54**	2	135 ± 2	2	AOAC (2016) 930.15
42	**6.65**	0.07	-0.86	**-5.72**	2	105	3	SNI 01-2891-1992. point 5.1
43	**7.80**	0.10	0.76	**4.84**	2	130	2 to constant	AOAC, National Standard
44	**7.53**	0.76	0.38	0.69	2.0056	130	1.0	AOAC 19th Ed, 2012
45	**6.92**	0.18	-0.47	**-2.47**	5 ± 0.3	103 ± 2	4 ± 1	ISO 6496
48	**7.65**	0.19	0.54	**2.79**	2	130	1	SNI 3549 2009
49	**3.67**	0.18	**-5.06**	**-26.68**	2	130	1	AOAC 20th Ed 2016
50	**7.67**	0.23	0.58	**2.71**	2.1804	130	1.0	AOAC 925.10
54	**7.44**	0.08	0.25	1.67	1	105	5	AOAC 927.05
55	**7.90**	0.06	0.90	**6.11**	5	130	1	AOAC (2012) 945.39A
56	**7.59**	0.46	0.46	1.32	2.03250	130 ± 3	1	AOAC Intl 20th Ed, 2016 925.10
58	**7.65**	0.26	0.55	**2.38**	2 to 5	130 / 105	3	Based on AOAC 20th Ed 2016
59	**5.78**	0.02	**-2.08**	**-14.73**	1 to 2	105	3	SNI 01-2891-1992 point 5.1
60	**6.98**	-	-0.39	-	-	-	-	SNI 01-2891-1992 Butir 5.1

Laboratory Number	Moisture (g/100g)	MU (g/100g)	z score	Zeta score	Sample weight (g)	Temp. (^{o}C)	Time (Hours)	Method Reference
Assigned value obtained from robust average (x) ± robust SD (s*) = 7.26 ± 0.71 g/100 g (CV 9.8%, n= 79) with u_{xpt} = 0.10 g/100 g*								
61	**7.46**	1.13	0.28	0.35	3	130	1.5	A6801 130C Air oven
62	**6.15**	-	-1.56	-	-	-	-	-
63	**7.83**	-	0.80	-	-	-	-	-
64	**7.37**	0.06	0.16	1.09	2.0577	130	1	AOAC 925.10
65	**4.98**	-	**-3.21**	-	4.8006	105	2.5	Oven drying
66A	**6.95**	0.25	-0.44	-1.94	10.0032	130.0	0.50	AOCS Official Method Ca 2c-25, 7th Ed., 2017
66B	**5.76**	0.10	**-2.11**	**-13.42**	10.0010	130.0	0.50	AOCS Official Method Ca 2c-25, 7th Ed., 2017
67	**7.84**	0.22	0.82	**3.90**	2.0xxx	130	1.0	AOAC 925.10
68	**8.19**	-	**1.31**	-	2	135	2	AOAC
69	**6.94**	-	-0.45	-	-	-	-	-
70	**4.85**	0.50	**-3.39**	**-8.95**	5	105	5	-
71	**7.08**	0.22	-0.25	-1.20	4.9979, 5.0033	105	3	AOAC 930.15
72	**7.02**	0.01	-0.34	**-2.40**	2	130	1	AOAC 925.10
73A	**7.02**	0.25	-0.34	-1.50	5	105	3	FTC-01.01 (refers to AOAC 945.39)
74	**6.24**	0.25	-1.44	**-6.37**	5.0	105	3	SNI 01-2891-1992 (part 5.1)
75	**7.14**	0.06	-0.17	-1.17	2	105 + 2	4	SNI 01-2891-1992 Butir 5.1
79	**6.46**	0.65	-1.13	**-2.38**	1 to 2	105	3	SNI 01-2891-1992 Butir 5.1
80	**8.04**	-	1.10	-	2.xx	135	2	AOAC 930.15
81	**7.50**	0.06	0.34	**2.30**	2.0309 mean	130	1 then 0.5 until <0.005 mg diff	AOAC 925.10

Laboratory Number	Moisture (g/100g)	MU (g/100g)	z score	Zeta score	Sample weight (g)	Temp. (^{o}C)	Time (Hours)	Method Reference
Assigned value obtained from robust average (x) ± robust SD (s*) = 7.26 ± 0.71 g/100 g (CV 9.8%, n= 79) with u_{xpt} = 0.10 g/100 g*								
82A	**6.71**	0.08	-0.77	**-5.11**	1.00	105	7.5	Drying Oven
82B	**6.71**	0.08	-0.77	**-5.11**	1.00	105	7.5	Drying Oven
83	**6.32**	0.22	-1.33	**-6.31**	2	105	5	SNI-01-2891-1992
84	**7.92**	-	0.93	-	2	130	1	AOAC 945.39
85	**5.86**	0.03	-1.97	**-13.85**	2	105	3	SNI 01-2896-1992
86	**9.30**	0.40	**2.87**	**9.12**	2	130	2	AOAC (2012) 945.39A
87	**6.09**	0.02	-1.64	**-11.60**	1.5	105	3	MTD/FOD/CHM-01
89	**6.56**	0.12	-0.98	**-6.04**	2	100 to 105	4	AOAC 925.23
90	**7.98**	0.40	1.01	**3.21**	2	130	1	AOAC (2016) 930.15
91	**7.53**	0.01	0.38	**2.70**	-	-	-	-
92	**7.47**	-	0.30	-	5	103	4	ISO 6494
93	**7.34**	-	0.11	-	2	130 ± 3	1	AOAC 925.10
94	**8.10**	-	1.18	-	1	130	5	AOAC (2000) 925.10
95	**7.16**	0.04	-0.14	-0.98	-	-	-	-
96	**8.31**	-	1.48	-	3	125	4	TCVN 4846:1989
97	**7.20**	-	-0.08	-	5	105	2	Sri Lanka Standard 1011:1994
98	**7.11**	-	-0.21	-	~2.0	100 ± 5	5	AOAC 930.15
100	**9.03**	0.16	**2.49**	**13.82**	5	130	2	AOAC 945.39

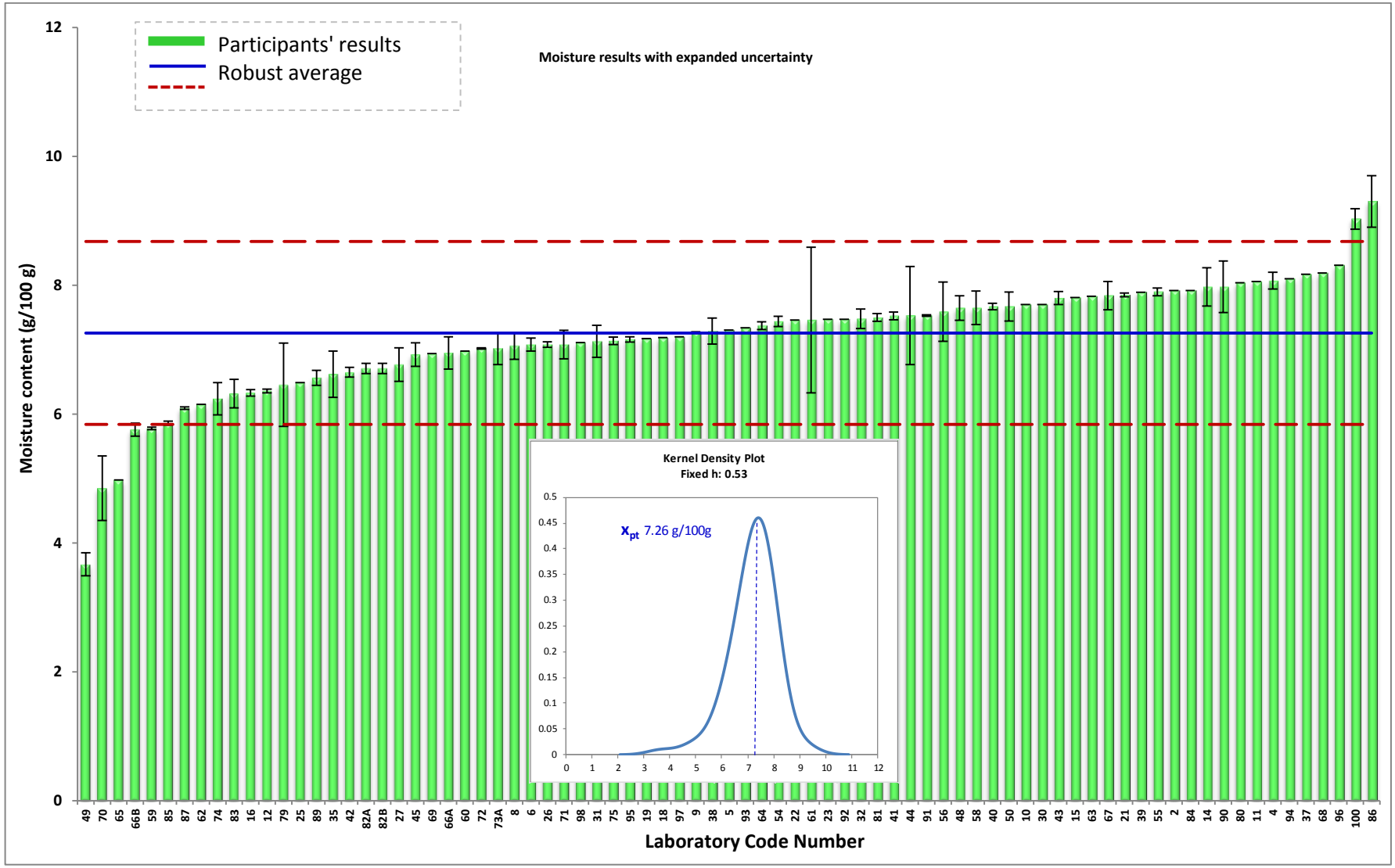

Figure 1. Distribution of **moisture** results (ascending order) in defatted soybean flour with expanded uncertainty

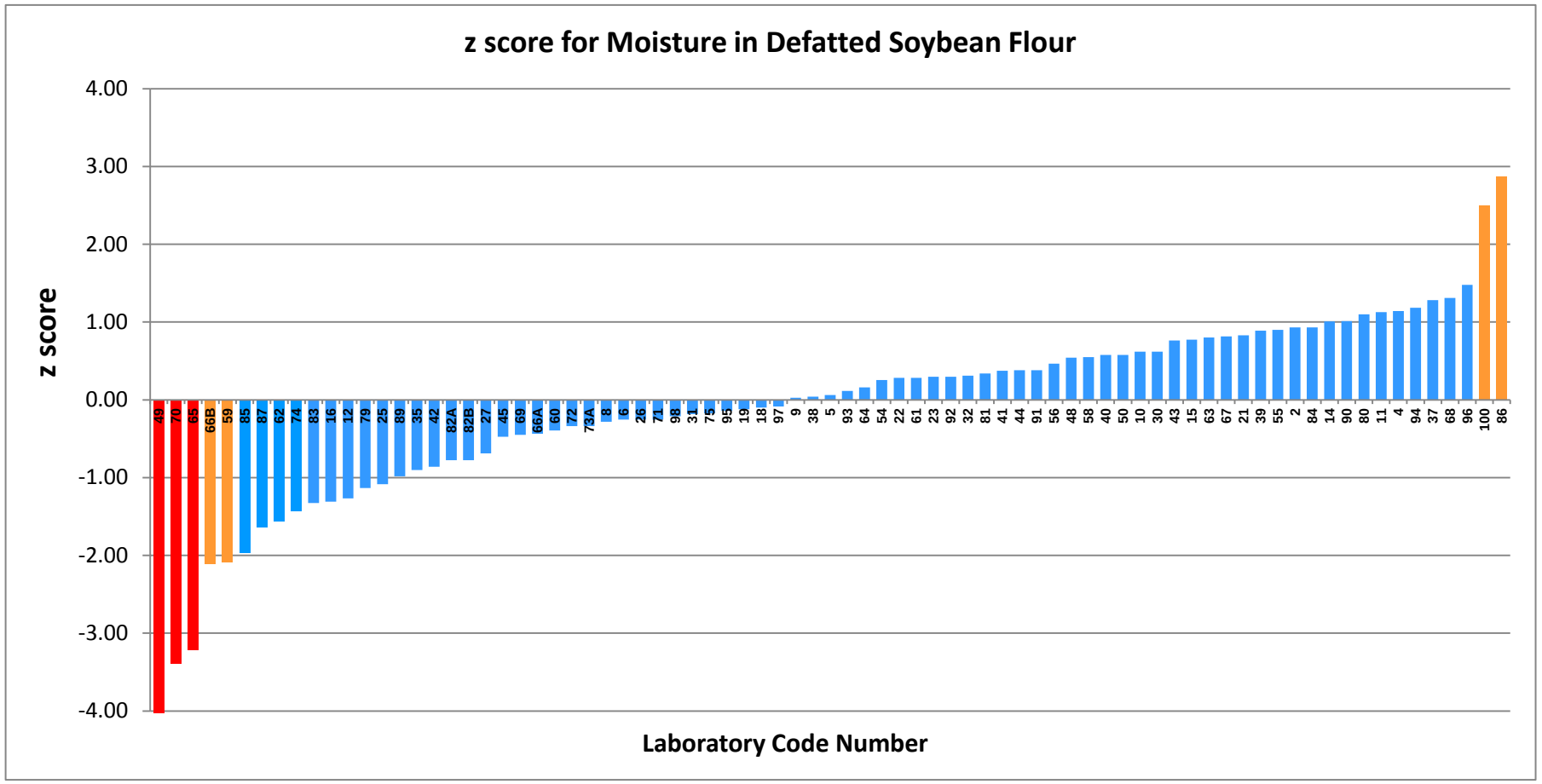

Figure 2. Plot of ordered *z* scores for **moisture** results in defatted soybean flour

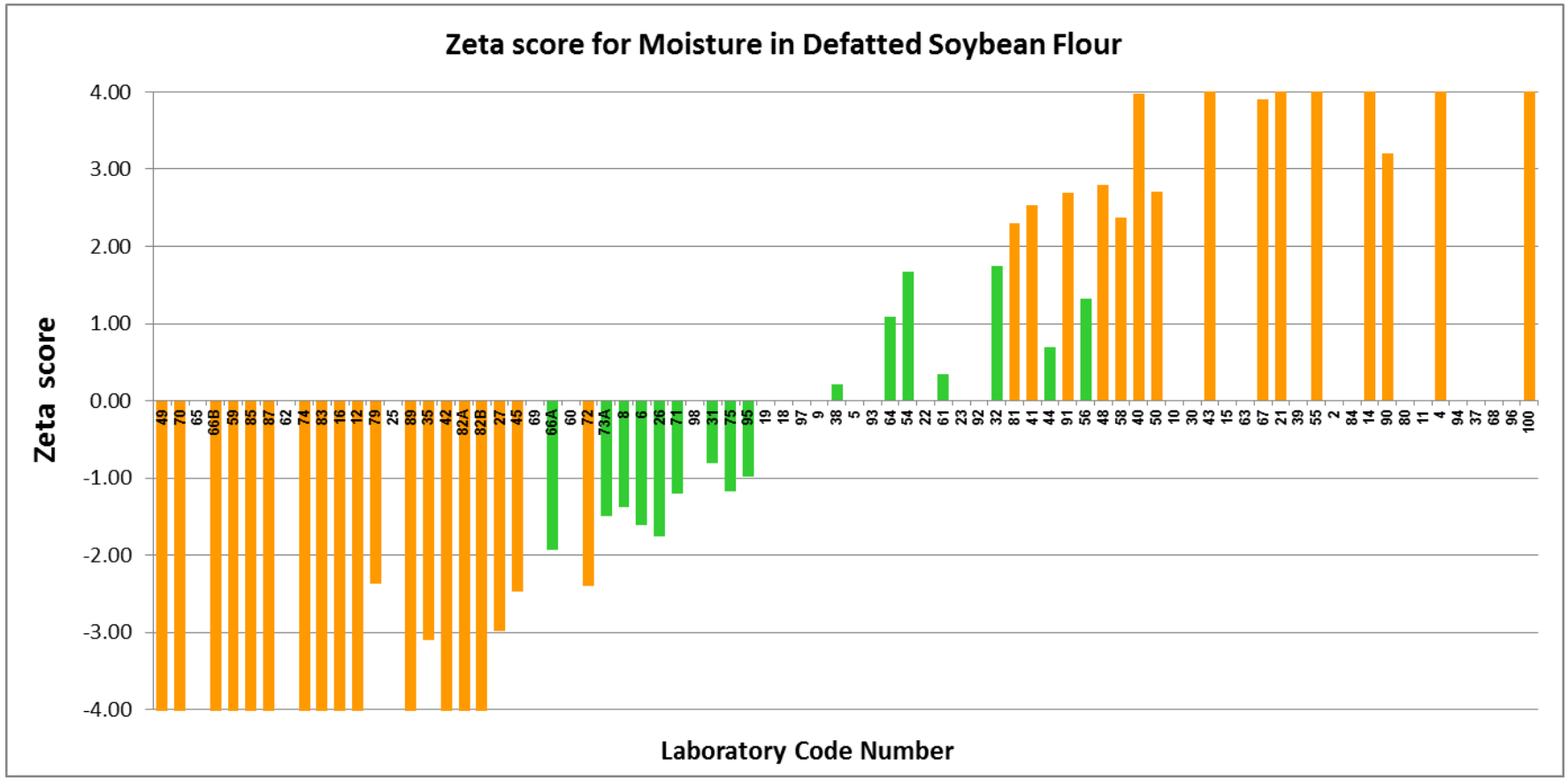

Figure 3. Plot of *Zeta* score for **moisture** in defatted soybean flour, following the ordered z scores in the above Figure 2.

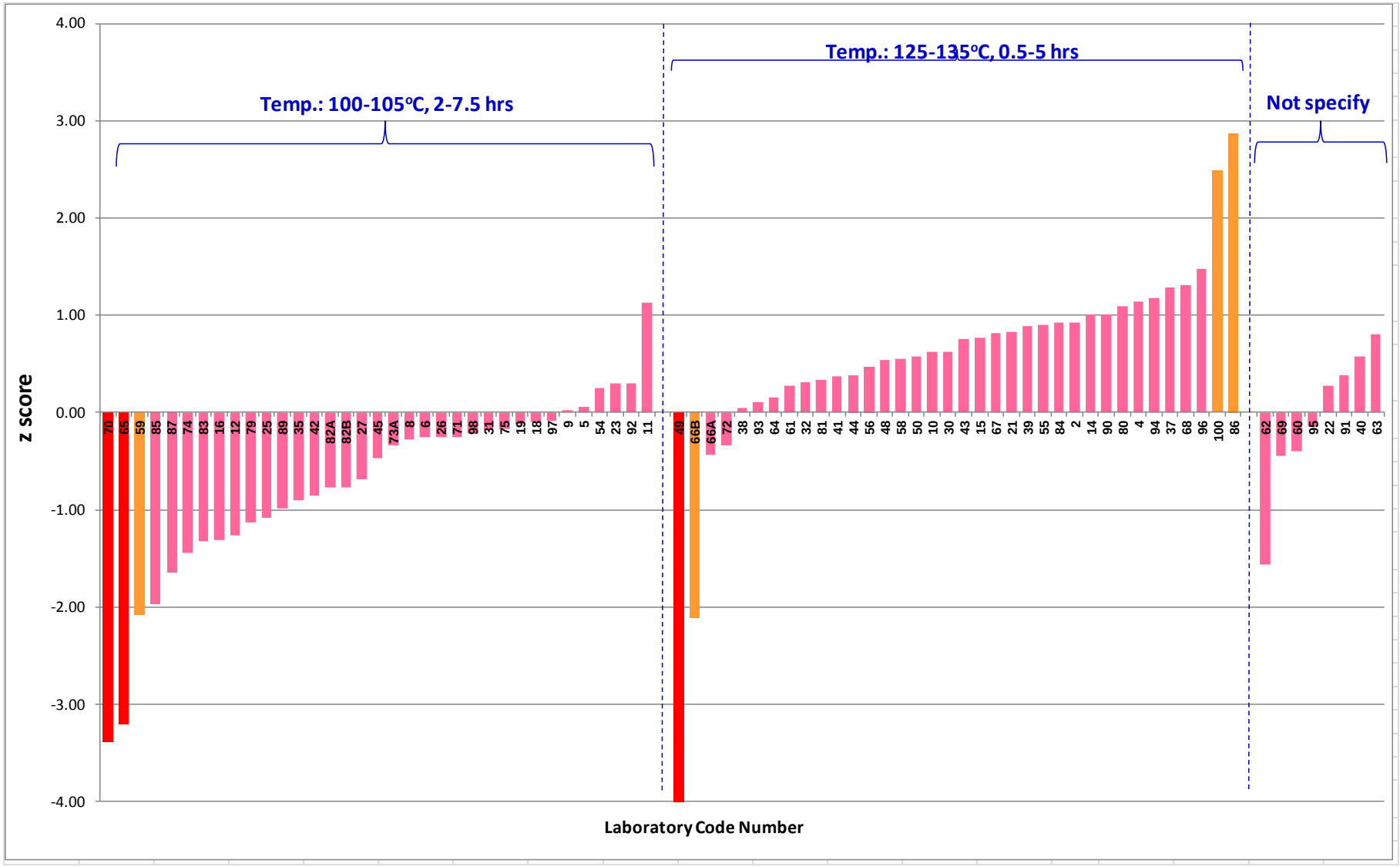

Figure 4. Plot of ordered *z* score for **moisture** in defatted soybean flour, grouping by method - drying temperatures

TOTAL NITROGEN

Defatted soybean flour contains 8.31 g of total N/100 g (Judprasong etal., 2018, adjusted moisture content).

Laboratories who submitted the methods used applied Kjeldahl method for total N determination, except one use combustion method (DuMaster Protein Analyzer, Buchi). To avoid an error in using different conversion factors to calculate protein from total N; this PT study on protein analysis was evaluated from total N values.

As shown in **Figure 8,** 56 out of 66 participants (about 85%) have good performance on total N analyses; two laboratories reported extreme low values of total N and the other two reported extreme high values (6%). In addition another 5 laboratories reported questionable levels of total N. According to the summary of methods used, these laboratories applied Kjeldahl method for total N analysis, following the standard methods of AOAC, ISO and SNI (Soy Nutrition Institute). They conducted the analysis on similar range of sample weight, 0.5 to 2 g (except one lab used sample weight of 0.05 g reported questionable low value). Thus, each step on total N analysis must be critically reviewed and corrective action must be performed at these laboratories.

The reference value of total N content in the defatted soybean flour obtained from 56 good performance laboratories is 7.88 ± 0.12 g /100 g (Mean ± SD, %CV= 1.5).

Note: Although total N was used in this study for assessment of analytical performance, some PT studies may request to report total protein. The usual conversion factors used to convert total N to protein for defatted soybean among these laboratories was therefore collected. Eighteen laboratories out of 27 lab (67%) who submitted the information on methods used, normally use the conversion factor of 6.25; six laboratories (22%) used the conversion factor of 5.71 and few to them (3 out of 27, 11%) use 5.95 to convert total N to protein. This evidence emphasises that the difference in N conversion factor used among participants could be one of the important factors for discrepancy of the reported levels of protein in some PT study.

There are several N-conversion factors for soy protein among various references, i.e., ISO. AOAC and SNI. Thus, the factors for soybean and other protein foods should be harmonised based on the scientific evidence from research. In 2010, the International Network of Food Data System (INFOODS) published a book – FAO / INFOODS Guidelines for Checking Food Composition Data prior to the Publication of a User Table/Database - Version 1.0 <http://www.fao.org/3/ap810e/ap810e.pdf> - which include also the N conversion factors used for various foods. The specific conversion factor for converting total N to protein for defatted soybean flour is 5.71 (Greenfield and Southgate, 2003, FAO/INFOODS Guideline, 2012).

Table 5. Evaluation of laboratory performance on **total nitrogen** analysis (g/100 g, as received) in defatted soybean flour

Laboratory Number	Total Nitrogen (g/100g)	MU (g/100g)	z score	Zeta score	Sample Weight (g)	Catalyst	Acid Volume (mL)	Receiver Solution	Titrant	Conver-sion Factor	Reference
Assigned value obtained from robust average (x*) ± robust SD (s*) = 7.87 ± 0.16 g/100 g (CV 2.0%, n= 66) with u_{xpt} = 0.02 g/100 g											
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00							
2	**8.09**	-	1.38	-	0.50	$CuSO_4$+K_2SO_4	20	50	HCl 0.1 M	6.25	AOAC (2016) 981.10
4	**7.99**	0.22	0.75	1.07	0.9xxx	K_2SO_4 and Selenium	25 mL H_2SO_4	NaOH 100 mL	H_2SO_4 0.08-0.1 N	-	Based on AOAC
5	**7.79**	-	-0.48	-	1.0000	$CuSO_4$+K_2SO_4	13 mL conc H_2SO_4	30 mL 4% Boric Acid	0.5 N H_2SO_4	-	AOAC 20th Ed, 2016, 2001.11, Chapt 4
6	**8.08**	0.65	1.34	0.66	0.5074	K_2PO_4 + $CuPO_4$	15	55 mL 4% Boric Acid	0.2036 N H_2SO_4	6.25	ISO 5983-2
9	**7.81**	-	-0.39	-	1	K_2SO_4/$CuSO_4$	H_2SO_4 / 12.5 mL	Boric acid 30 mL	0.5 N H_2SO_4	-	Based on AOAC 20th Ed, 2016, 2001.11, Chapt 4
10	**7.84**	-	-0.19	-	1	Selenium mixture	25 mL conc H_2SO_4	50 mL 4% H_3BO_4	HCl 0.1 M	-	AOAC 2012, 32.2.09 C, Chapt 32
11	**7.54**	-	**-2.06**	-	0.5000	1 mL	H_2SO_4 / 25 mL	Boric acid solution 25 mL	0.1 N H_2SO_4	6.25	Manual on fertilizer analysis, Arsrod, Doa 12/2551
12	**8.01**	0.03	0.87	**5.33**	1	K_2SO_4, $CuSO_4$	H_2SO_4, 20 mL	H_3BO_4, 100 mL	0.1 M HCl	5.71	AOAC (2016) 992.23
14	**7.87**	0.24	0.02	0.02	1	K_2SO_4:$CuSO_4$.$5H_2O$ (9:1)	H_2SO_4 15 mL	4% Boric Acid 30 mL	0.1 M HCl	-	AOAC 991.20
15	**8.02**	-	0.94	-	0.2	$CuSO_4.5H_2O$, K_2SO_4	H_2SO_4 20 mL	1% H3BO4	0.1 M HCl	6.25	Based on AOAC (2016) 991.20
16	**7.75**	0.22	-0.75	-1.07	0.5 - 1	Selenium	Sulphuric Acid 25 mL	Boric Acid 50 mL	HCl 0.1 N	-	SNI 01-2891-1992 Food & Beverage

Laboratory Number	Total Nitrogen (g/100g)	MU (g/100g)	z score	Zeta score	Sample Weight (g)	Catalyst	Acid Volume (mL)	Receiver Solution	Titrant	Conver-sion Factor	Reference
Assigned value obtained from robust average (x*) ± robust SD (s*) = 7.87 ± 0.16 g/100 g (CV 2.0%, n= 66) with u_{xpt} = 0.02 g/100 g											
18	**7.83**	-	-0.25	-	2.0	$CuSO_4$, SeO_2	H_2SO_4, 25 mL	H_3BO_3 2%, 25 mL	HCl 0.1 M	-	SNI 01-2891-1992
19	**7.90**	-	0.21	-	1	Kjeltabs, 2 pcs	Sulphuric Acid 15 mL	Boric Acid 1%, 15 mL	HCl, 15 mL	6.25	AOAC 988.05, AN300 FOSS 2003
21	**7.76**	-	-0.69	-	0.5 to 1.0	Se, K_2SO_4	H_2SO_4 15 mL	Boric acid (AR) 25 mL	HCl (AR) 0.1 N	-	AOAC 930.29 (2016)
22	**7.66**	-	-1.31	-	-	-	-	-	-	-	-
23	**8.02**	-	0.94	-	0.20	-	-	-	-	-	AOAC 992.15
25	**7.43**	-	**-2.75**	-	0.0521 / 0.0517	Copper Sulphate	Digestion Reagent, 10 mL	Indicating Boric Sol'n, 10 mL	0.02 N H2SO4	-	Laboratory Handbook of Methods of Food Analysis, 3rd Ed, R. Lees
26	**7.88**	0.23	0.06	0.08	2.0	Copper (II) sulphate pentahydrate	Conc H_2SO_4, 15 mL	0.1 N HCl	0.1 N NaOH	5.95	AOAC No. 2001.11
27	**8.70**	0.57	**5.19**	**2.91**	0.5	K_2SO_4 / Se	10	30	0.2000	-	SNI 01-2354.4-2006 Modified
30	**7.18**	-	**-4.30**	-	1.0023, 1.0030	K_2SO_4:$CuSO_4$.$5H_2O$:TiO_2 (10:0.3:0.3)	Sulphuric acid 20 mL	Erlenmeyer Flask 250 mL	H_2SO_4 0.05 N	-	ISO 20483:2006 (E)
31	**7.82**	0.18	-0.31	-0.54	0.3	Selenium	H_2SO_4 (8 mL)	H_3BO_3 3% (50 mL)	HCl 0.05 N	-	SNI 01-2891
32	**7.95**	0.20	0.50	0.77	2.1110	K_2SO_4, $CuSO_4.5H_2O$	Conc H_2SO_4, 25 mL	4% Boric Acid 50 mL	0.50987 M HCl	5.71	Block Digestion - Kjeldahl
35	**7.86**	-	-0.06	-	1.0626	Anhyd Na_2SO_4: Anhyd $CuSO_4$ (97:3)	H_2SO_4 25 mL	2% Boric Acid 100 mL	0.1 N H2SO4	6.25	Sri Lanka Standard 1011:1994 specification for Soya Flour
38	**7.90**	0.55	0.19	0.11	0.400	Salt mixture	H_2SO_4 12 mL	4% H_3BO_3, 20 mL	0.2 N HCl		AOAC 991.2, 19th Ed 2012

Laboratory Number	Total Nitrogen (g/100g)	MU (g/100g)	z score	Zeta score	Sample Weight (g)	Catalyst	Acid Volume (mL)	Receiver Solution	Titrant	Conversion Factor	Reference
Assigned value obtained from robust average (x*) ± robust SD (s*) = 7.87 ± 0.16 g/100 g (CV 2.0%, n= 66) with u_{xpt} = 0.02 g/100 g											
39	**7.93**	-	0.37	-	0.5	Cu	H_2SO_4 / 10	Boric acid 30 mL	0.1 M HCl	-	AOAC 991.20
41	**7.95**	0.28	0.51	0.58	0.5	K_2SO_4:$CuSO_4$	Sulphuric acid 15 mL	Boric acid 25 mL	0.1000	6.25	AOAC 2001.11
42	**7.84**	0.19	-0.19	-0.31	0.5	Selenium	H2SO4	H_3BO_3 1% 30 mL	HCl 0.1 N		SNI 01-2891-1992. point 7.1
43	**7.80**	0.40	-0.43	-0.34	1	Mix selenium	H2SO4, 12 mL	H_3BO_3, 25 mL	HCl 0.2 M	1.4007	National Standard, inhouse method
44	**8.02**	0.01	0.94	**7.13**	0.5063	Na_2SO_4, $CuSO_4$	Conc H_2SO_4 20 mL	50 mL 0.1 N H_2SO_4	0.2 N NaOH	5.71	AOAC 19th Ed, 2012
45	**7.96**	0.52	0.56	0.34	1	7g K_2SO_4 + 0.8 g $CuSO_4.5H_2O$	98% H_2SO_4 15 mL	4.0% Boric acid 30 mL	0.5 N H_2SO_4	6.25	ISO 5983-2
48	**7.72**	0.15	-0.95	-1.96	1	Selenium Mixture reagent	20	30	0.1	1	MU-01/04
49	**6.94**	0.36	**-5.81**	**-5.14**	1, 2	Kjeltabs	Conc H_2SO_4 20 mL	Boric Acid 50 mL	0.2 N H_2SO_4	5.7	AOAC 20th Ed 2016
50	**8.33**	0.18	**2.90**	**5.05**	1.1567	Cu	H_2SO_4, 15.0 mL	Boric Acid, 75.0 mL	HCl, 0.0902	Fish M 6.25, Rice F 5.7	AOAC 984.13
53	**7.86**	0.09	-0.04	-0.14	0.3	Selenium	H_2SO_4 8 mL	H_3BO_3 3% 25 mL	HCl 0.0958 N	5.71	SNI-01-2891-1992
54	**7.51**	0.29	**-2.23**	**-2.48**	1	Kjeltabs	H_2SO_4 12 mL	25 mL 4% Boric Acid	0.3 M H_2SO_4	5.95	AOAC 920.87
55	**7.70**	0.08	-1.06	**-3.80**	1	K2SO4 / Se	H_2SO_4 15 mL	250 mL Erlenmeyer flask, 25 mL 4% Boric Acid	HCl 0.1 M	-	AOAC (2012) 991.20

Laboratory Number	Total Nitrogen (g/100g)	MU (g/100g)	z score	Zeta score	Sample Weight (g)	Catalyst	Acid Volume (mL)	Receiver Solution	Titrant	Conver-sion Factor	Reference
Assigned value obtained from robust average (x*) ± robust SD (s*) = 7.87 ± 0.16 g/100 g (CV 2.0%, n= 66) with u_{xpt} = 0.02 g/100 g											
56	**7.81**	0.20	-0.39	-0.60	1.01193	Kjeltabs (Foss)	H_2SO_4 12.0 mL	Boric Acid Soln 25 mL	0.09729 M HCl	6.25	AOAC Intl 20th Ed, 2016 981.10
58	**7.85**	0.23	-0.13	-0.17	0.5	-	-	Boric Acid	0.25 HCl	1.0	Based on AOAC 20th Ed 2016
60	**7.92**	-	0.31	-	-	-	-	-	-	-	AOAC (2012) 2011.11
61	**7.82**	0.34	-0.31	-0.29	1	K_2SO_4/$CuSO_4$ Kjeltab catalyst tablets	H2SO4 20 mL	Boric Acid 50 mL	0.1 M HCl	6.25	A6501 Kjeldahl/Boric Acid Method
63	**7.97**	-	0.62	-	-	-	-	-	-	-	-
67	**8.03**	0.22	1.00	1.43	0.5xxx	$CuSO_4$+K_2SO_4	H_2SO_4 15 mL	Boric acid 50 mL	0.2 N H_2SO_4	6.25	Inhouse based on ISO 5988.2
68	**7.88**	-	0.06	-	0.5	Kjeltabs	17	50	0.1 N HCl	6.25	AOAC
69	**7.93**	-	0.36	-	-	-	-	-	-	-	-
71	**7.98**	0.24	0.69	0.90	1.0075, 0.9934	Kjeltabs 3.5 g, K_2SO_4 0.4 g, $CuSO_4.5H_2O$	H_2SO_4 15 mL	Boric acid	0.2 N HCl		AOAC 2001.11
72	**8.01**	0.05	0.87	**4.37**	2	K_2SO_4, $CuSO_4$, SeO_2	25	4% Boric acid 25 mL	0.05 M H2SO4	6.25	AOAC 920.87
73A	**8.00**	0.70	0.81	0.37	1	2 Kjeltabs (each 3.5 g K_2SO_4, 0.4 g $CuSO_4.5H_2O$)	H2SO4 15 mL	1% Boric acid, 1% BCG 0.1% soln, 0.7% Methyl Red 0.1% soln (30 mL)	HCl 0.2 M	6.25	FTC-02.01 (refers to AOAC 2001.11, 979.09)
75	**9.61**	0.17	**10.88**	**20.50**	0.5	$CuSO_4$	H_2SO_4, 5 mL	H_3BO_3, 20 mL	HCl, 0.1 M	N/A (report as Nitrogen)	SNI 01-2891-1992 Butir 7.1
78	**7.77**	0.10	-0.63	-1.86	2	Kjeltabs	Conc H_2SO_4, 15 mL	4% Boric acid, 25 mL	0.1 N HCl	-	AOAC 19th Ed

Laboratory Number	Total Nitrogen (g/100g)	MU (g/100g)	z score	Zeta score	Sample Weight (g)	Catalyst	Acid Volume (mL)	Receiver Solution	Titrant	Conversion Factor	Reference
Assigned value obtained from robust average (x*) ± robust SD (s*) = 7.87 ± 0.16 g/100 g (CV 2.0%, n= 66) with u_{xpt} = 0.02 g/100 g											
79	**8.02**	0.28	0.93	1.06	0.1 to 0.15	-	-	-	-	-	IK/02/5.4.1/LDITP/ Analisis Protein
80	**7.86**	-	-0.06	-	0.25 to 0.5	TAP/S3.5	25	40	0.1	6.25	AOAC 984.13
81	**7.82**	0.09	-0.31	-1.02	1.0140 mean	K_2SO_4 and $CuSO_4.5H_2O$	20 mL H_2SO_4	60 mL 2% Boric Acid soln	0.09597 N HCl	5.71	Automated Kjeldahl Method
83	**7.93**	0.05	0.37	1.91	0.5	$CuSO_4$	H_2SO_4	Boric Acid, Bromocresol green, Methanol, Methyl red, 30 mL	HCl (0.1)	6.25	SNI-01-2891-1992
84	**8.12**	-	1.56	-	1	KJELCAT 12-0328	H_2SO_4 98% 20 mL	H_3BO_3 4%, 60 mL	HCl 0.1 M	-	KJELDAHL
85	**7.90**	0.00	0.19	1.50	0.2	-	-	-	-	-	DuMaster Protein Analyzer (Buchi)
86	**7.98**	0.75	0.69	0.29	0.5	3.5g K_2SO_4 + 3.5 mg Se	Conc H_2SO_4 12.5 mL	4% Boric Acid 30 mL	0.1 N HCl	-	AOAC (2012) 981.10
87	**7.67**	0.35	-1.26	-1.14	0.51	K_2SO_4+Se	Sulphuric Acid; 25 mL	Boric Acid; 15 mL	HCl 0.01 N	14	MTD/FOD/CHM-03
89	**7.71**	0.38	-1.02	-0.85	0.5	$CuSO_4$	HCl	25 mL Boric Acid	0.1 N HCl	-	AOAC 991.2
90	**7.68**	0.14	-1.19	**-2.61**	0.3	K_2SO_4, Se	15	-	0.1 M HCl	-	AOAC (2016) 2001.11
91	**7.72**	0.05	-0.94	**-4.69**	-	-	-	-	-	-	-
92	**7.95**	-	0.50	-	1	$CuSO_4$	H_2SO_4	Boric	HCl	6.25	-

Laboratory Number	Total Nitrogen (g/100g)	MU (g/100g)	z score	Zeta score	Sample Weight (g)	Catalyst	Acid Volume (mL)	Receiver Solution	Titrant	Conversion Factor	Reference
Assigned value obtained from robust average (x*) ± robust SD (s*) = 7.87 ± 0.16 g/100 g (CV 2.0%, n= 66) with u_{xpt} = 0.02 g/100 g											
93	**7.61**	-	-1.63	-	2	H_2O_2 5 mL, Kjeltabs: 3.5 g K_2SO_4, 0.4 g $CuSO_4$,$5H_2O$	H_2SO_4 12 mL	Boric acid 25 mL	0.05 N H2SO4	5.95	AOAC 945.18-B
94	**8.24**	-	**2.31**	-	1	$CuSO_4.5H_2O$ and K_2SO_4	Conc H_2SO_4 / 13 mL	1% Boric acid	0.1 M HCl	-	AOAC (2012) 991.20
95	**7.91**	0.07	0.22	0.85	-	-	-	-	-	-	-
98	**7.82**	-	-0.31	-	~1.0	7g K_2SO_4, 0.8 g $CuSO_4$	15 mL H_2SO_4	30 mL 4% Boric Acid	0.2 N HCl	-	AOAC 976.05
100	**7.86**	0.33	-0.06	-0.06	0.5	$CuSO_4$ and K_2SO_4	H_2SO_4 / 15 mL	4% Boric Acid 50 mL	0.2 N H_2SO_4	6.25	Inhouse based on ISO 5983-2 (2009)

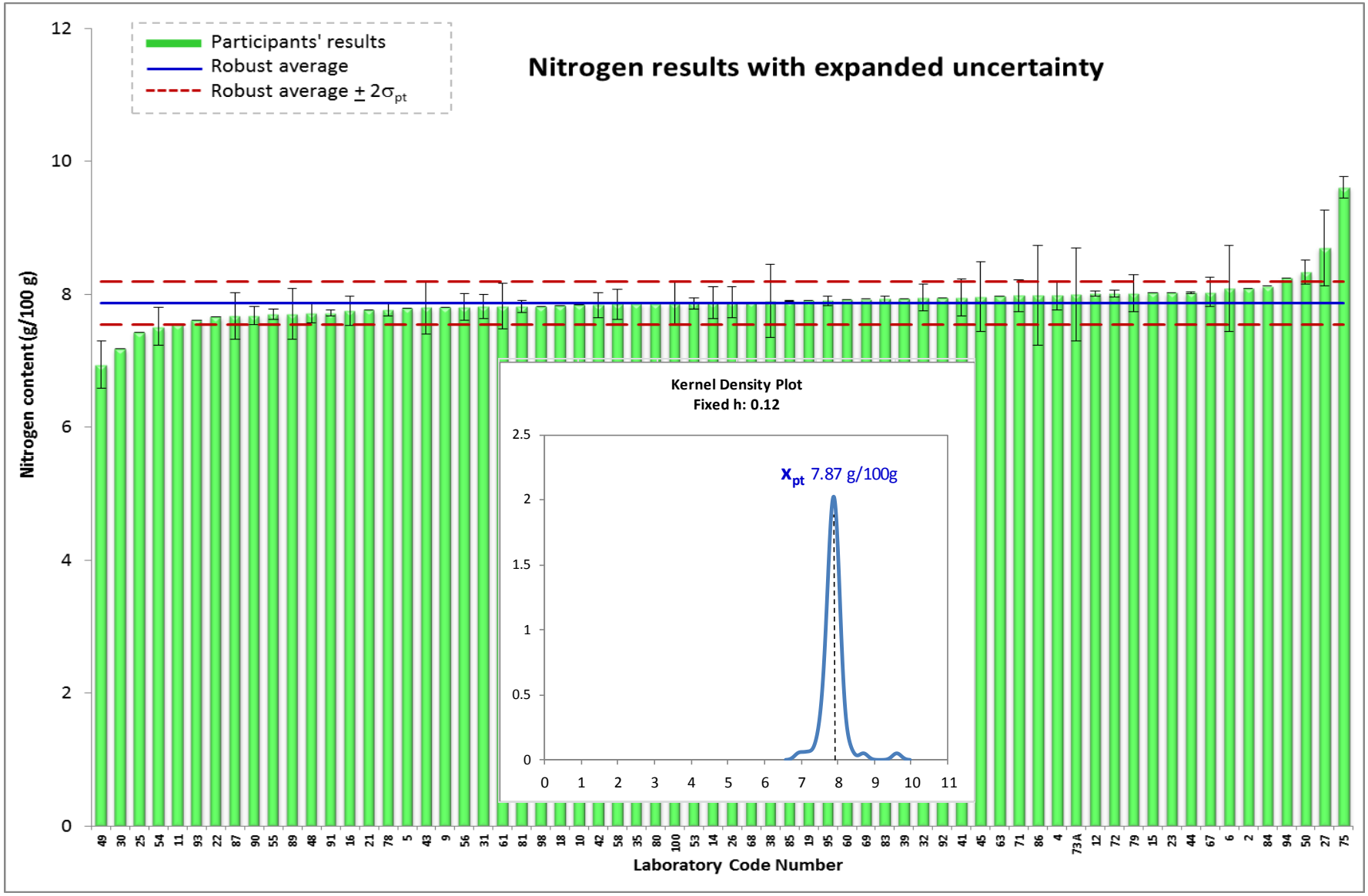

Figure 5. Distribution of **total nitrogen** results (ascending order) in defatted soybean flour with expanded uncertainty

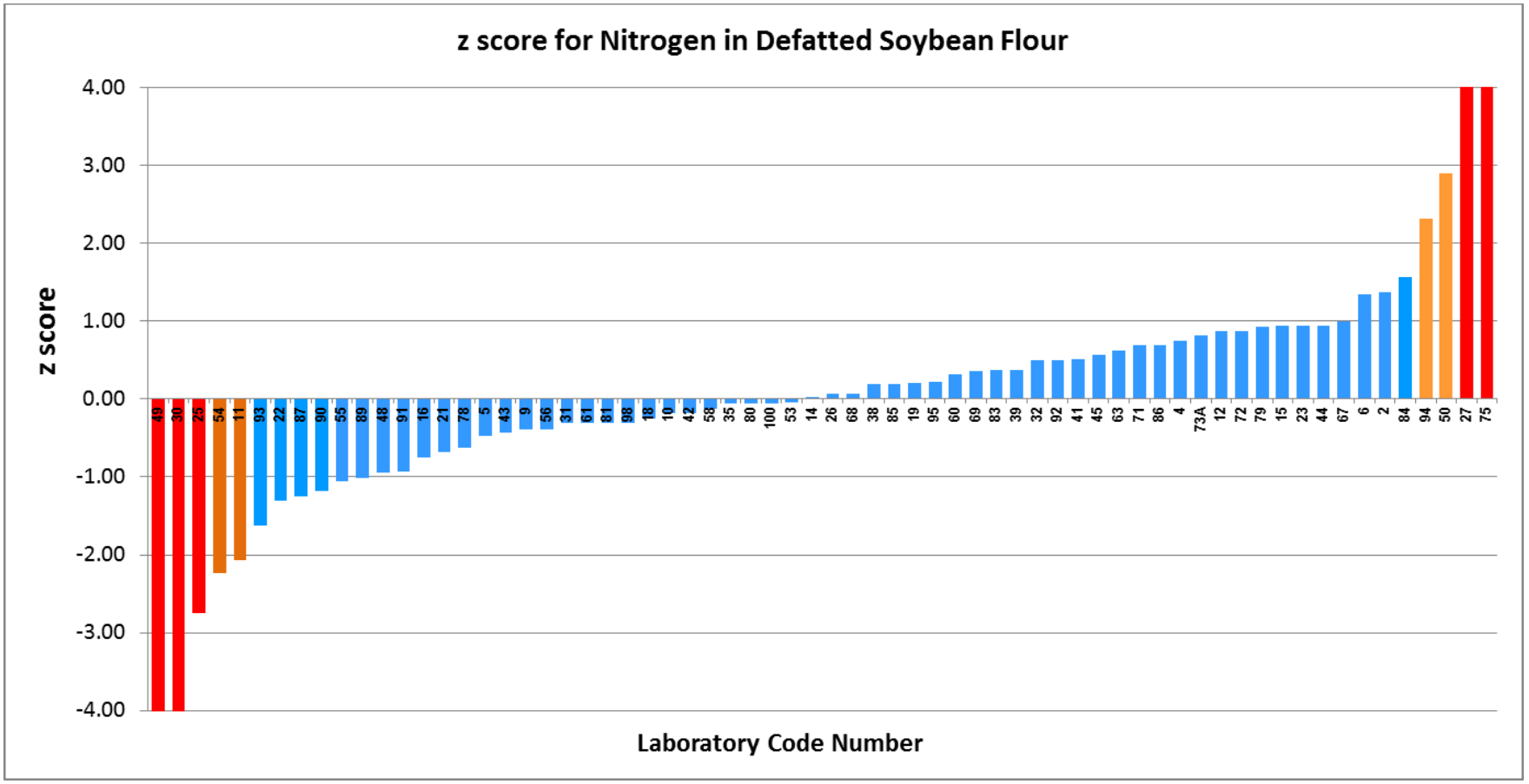

Figure 6. Plot of ordered *z* scores for **total nitrogen** results in defatted soybean flour

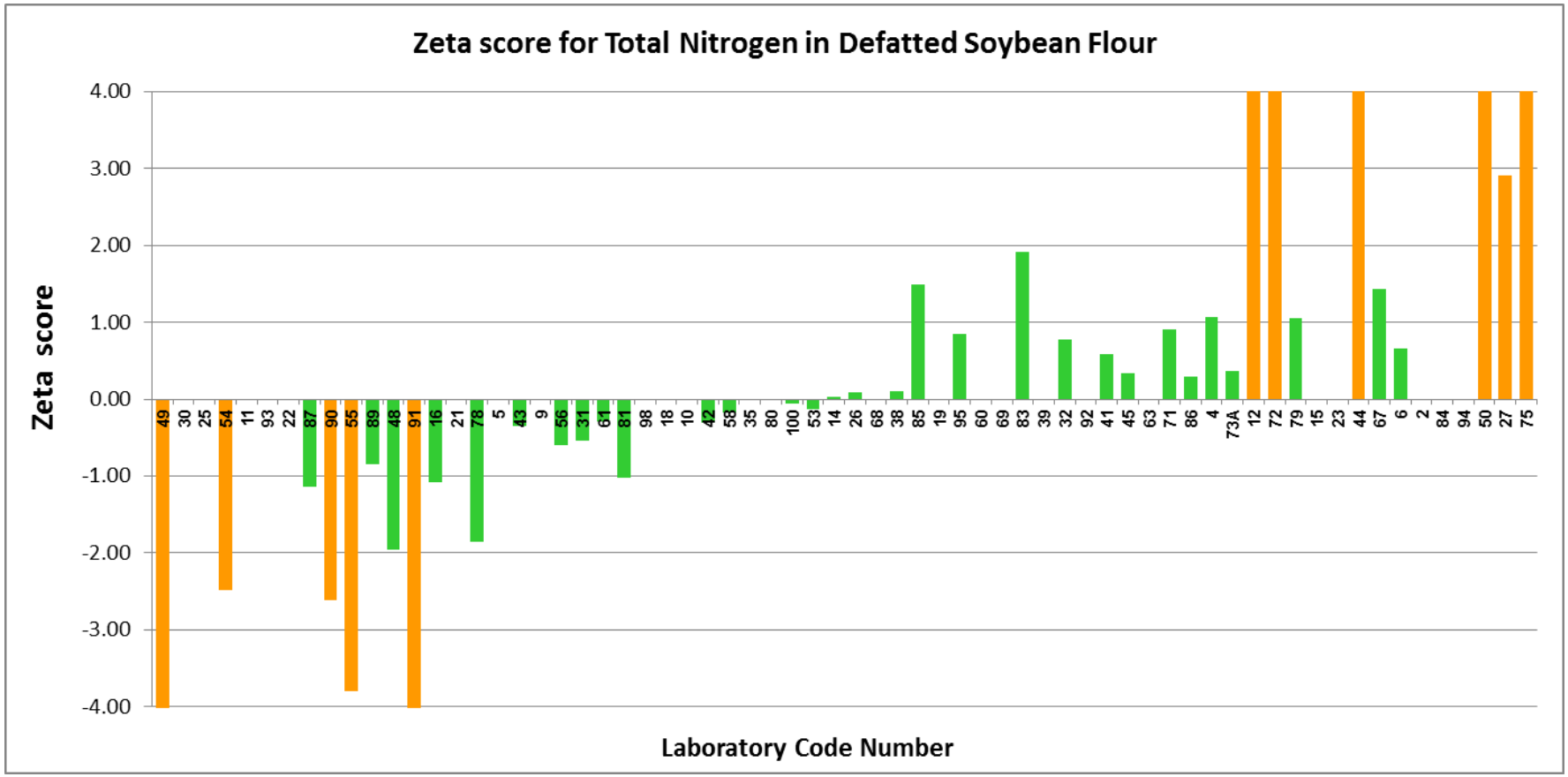

Figure 7. Plot of *Zeta* score for **total nitrogen** in defatted soybean flour, following the ordered z scores in the above Figure 6.

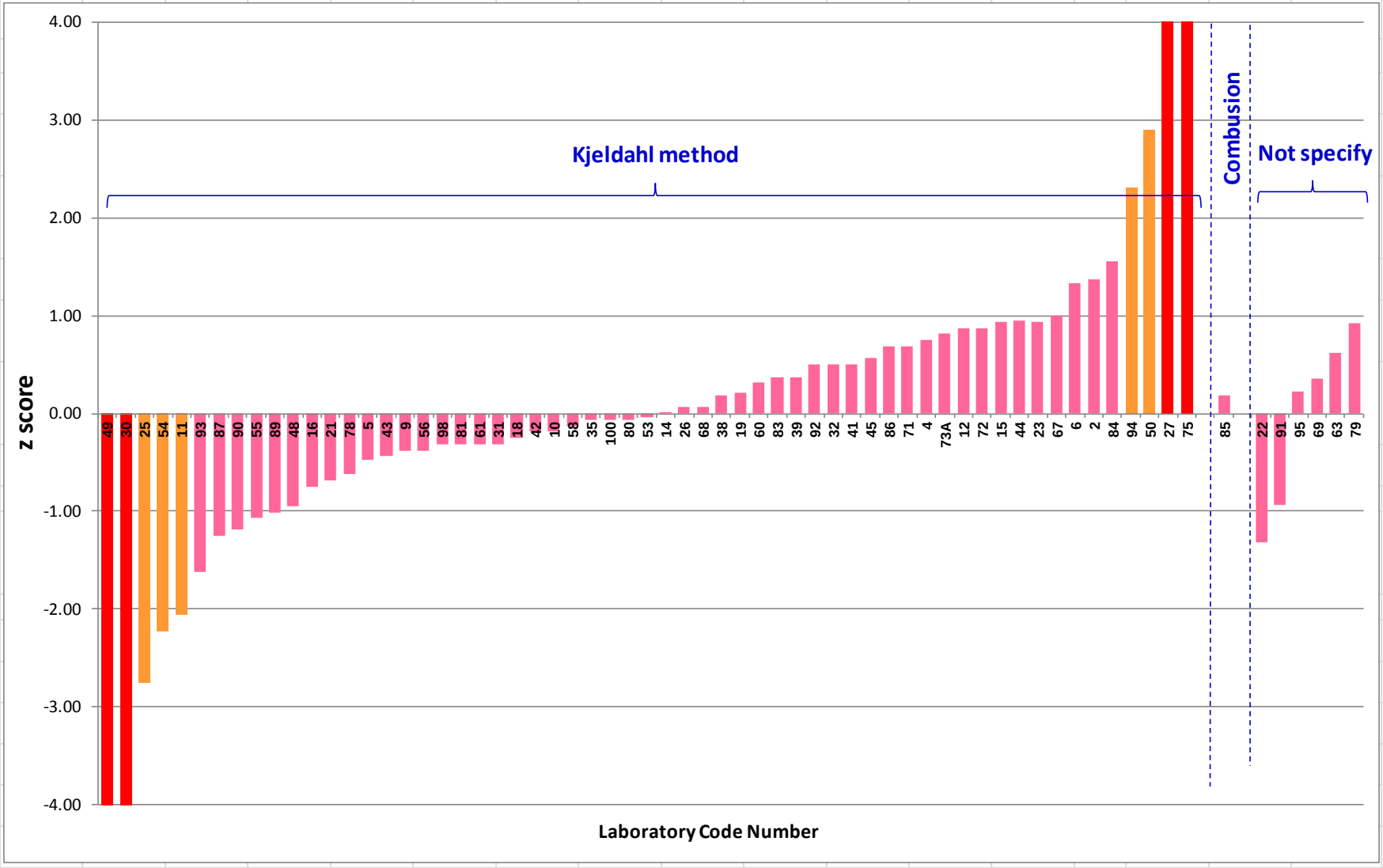

Figure 8. Plot of ordered *z* score for **total nitrogen** in defatted soybean flour, categorised in groups according to analytical methods/parameters used

TOTAL FAT

The selected test material used in this study is the defatted soybean flour which contains low level of total lipid - less than 2 g/100g.

For lipid determination of plant origin, e.g., cereals, legumes seeds. acid hydrolysis prior to direct extraction with non-polar solvent to break the lipid-carbohydrate bond and to hydrolyse protein is required for sample treatment.

In this study, 22 out of 55 of participating laboratories (40 %) did not include the acid hydrolysis in the process for lipid analysis. Most of them reported very low levels of total lipid content, ranged from 0 to less than 0.5 g/100 g. Those who included acid treatment prior to solvent extraction reported higher level of total lipid, 1.49 ± 0.67 g)/100 g (mean±SD) but with wide variation (%CV = 45), due to the low level of fat content in the selected test material. Thus, the laboratory performance on lipid analysis in defatted soybean flour could not be evaluated.

Table 6. Evaluation of laboratory performance on **total fat** analysis (g/100 g, as received) in defatted soybean flour

Laboratory Number	**Total fat** (g/100g)	**MU** (g/100g)	**z score**	**Zeta score**	**Fat weight (g)**	**Hydrolysis (Y/N)**	**Extraction Solvent**	**Extraction Time (hours)**	**Method Reference**
*Assigned value obtained from robust average ± robust SD = **1.48 ± 0.78 g/100 g** (**CV 52.7%**, n= 40 only laboratory who performed hydrolysis before extraction)*									
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00					
2	**0.10**	-	**Not evaluate due to high variation of results**		2.00	N	Petroleum Ether	8	AOAC (2016) 920.39
4	**1.61**	0.12			1.xxxx	Y	Diethyl Ether and Petroleum Ether	-	Based on AOAC
5	**0.52**	-			2.0000	N	Petroleum Ether	1.5	AOAC 20th Ed, 2016, 2003.05, Chapt 4
6	**0.11**	0.00			3.0000	N	Diethyl Ether	80 min	AOAC 2003.05
9	**0.14**	-			2	N	Petroleum Ether	1.5	Based on AOAC 20th Ed, 2016, 2003.05, Chapt 4
10	**0.28**	-			2	N	Petroleum Ether	5	AOAC 2012, 32.2.09 E, Chapt 32

Laboratory Number	Total fat (g/100g)	MU (g/100g)	z score	Zeta score	Fat weight (g)	Hydrolysis (Y/N)	Extraction Solvent	Extraction Time (hours)	Method Reference
11	**0.00**	-	**Not evaluate due to high variation of results**		4.0000	N	Petroleum Ether	16-18 hr	AOAC (2016) 922.06
12	**< LOD**	-			2	Yes, Acid digestion	Petroleum Ether	2	AOAC (2016) 984.15
14	**1.98**	0.02			2	Y	Diethyl Ether and Petroleum Ether	3	AOAC 922.06
15	**0.66**	-			1	Y	Petroleum Ether	1	Based on ISO 1443:1973
16	**2.29**	0.11			2	Y	Diethyl Ether + Petroleum Ether	-	SNI 01-2891-1992 Food & Beverage
18	**1.64**	-			2.0	Y	Petroleum Benzine	6	SNI 01-2891-1992
19	**0.28**	-			1	N	Petroleum Benzene	1	AOAC 2003.05, AN305, FOSS, 2005
21	**1.45**	-			0.5 to 1.0	Y	Diethyl Ether Petroleum Ether	2	AOAC 932.06 (2016)
22	**0.55**	-			-	-	-	-	-
23	**< LOR**	-			1.90	N	Petroleum Ether	1	AOCS Am5-04
25	**0.97**	-			7.1745	N	Hexane	8	Laboratory Handbook of Methods of Food Analysis, 3rd Ed, R. Lees
27	**1.41**	0.17			2	Y	Diethyl Ether	2	SNI 2354-3:2017
30	**2.28**	-			2.0039, 2.0027	Y	Diethyl Ether, Petroleum Ether	3	AOAC (2016) 925.10
31	**2.20**	0.07			3.5	Y	Petroleum Benzene	4	SNI 01-2891
32	**0.82**	0.03			2.0832	Y	Petroleum Ether	20 cycles (2 hours)	Acid Hydrolysis
35	**0.67**	-			5.5793	N	Petroleum Ether 60-40 ^{o}C	16	Sri Lanka Standard 1011:1994 specification for Soya Flour

Laboratory Number	Total fat (g/100g)	MU (g/100g)	z score	Zeta score	Fat weight (g)	Hydrolysis (Y/N)	Extraction Solvent	Extraction Time (hours)	Method Reference
38	**1.88**	0.24	**Not evaluate due to high variation of results**		2.000	Y	Diethyl Ether, Petroleum Ether	1 min each	AOAC 922.06, 19th Ed 2012 (total fat)
39	**1.35**	-			1	Y	Diethyl Ether:Petroleum Ether (1:1)	6 min	AOAC 922.06
41	**2.04**	0.09			2	Y	Petroleum Ether / Diethyl Ether	-	AOAC 954.02
42	**0.78**	0.01			3	Y	Hexane	2	SNI 01-2891-1992. point 8.2
43	**0.41**	0.30			2	N	Hexane	3	National Standard
44	**1.70**	0.07			2.0495	Y	Petroleum Ether, Anhydrous Ether	2 mins	AOAC 19th Ed, 2012
45	**0.18**	0.01			1.5	N	Petroleum Ether	2.25	ISO 11085
48	**1.45**	0.11			1	Y	Diethyl Ether	3 x 20 mins	MU-01/02
49	**4.04**	0.20			2	Y	Petroleum Ether	1 minute	AOAC 20th Ed 2016
50	**2.84**	0.06			2.0000	Y	Pet. Ether	2.0	AOAC 920.85
54	**1.79**	0.04			1	Y	1:1 Mixed ether	5	AOAC 923.03
55	**1.16**	0.04			2	Y	Ether, Petroleum ether	1	AOAC (2012) 992.06
56	**1.54**	0.01			1.01512	Y	Petroleum ether	5 mins	AOAC Intl 20th Ed, 2016 945.44
59	**0.70**	0.01			1 to 2	Y	Petroleum Benzine	4	SNI 01-2891-1992 point 8.2
60	**0.37**	-			-	-	-	-	AOAC (2012) 2003.06
61	**1.50**	0.10			2	N (Y)	Petroleum Spirits (Diethyl	1 (-)	A6301 Soxtec (A6302 Acid Hydrolysis)
63	**1.87**	-			-	-	-	-	-

Laboratory Number	Total fat (g/100g)	MU (g/100g)	z score	Zeta score	Fat weight (g)	Hydrolysis (Y/N)	Extraction Solvent	Extraction Time (hours)	Method Reference
66A	**0.11**	0.08	**Not evaluate due to high variation of results**		3.5586	N	Petroleum Ether	0.75	AOAC 963.15, 20th Ed 2016 (Crude Fat)
66B	**0.06**	0.04			1.5005	N	Petroleum Ether	0.75	AOAC 963.15, 20th Ed 2016 (Crude Fat)
67	**0.11**	0.01			1.0xxx	N	Petroleum Ether	1.30	Inhouse based on AOAC 920.39
68	**1.84**	-			2	N	Petroleum ether	6	AOAC
69	**0.36**	-			-	-	-	-	-
71	**0.22**	0.08			1.0026, 1.0032	N	Petroleum Ether	2	AOAC 2003.05
72	**1.12**	0.12			2	Y	Petroleum ether, Diethyl ether	1 min	AOAC 922.06
73A	**0.26**	0.09			1	N	Petroleum Benzene	1	FTC-06.01 (refers to AOAC 2003.05)
75	**2.22**	0.03			2	Y	Hexane	4	SNI 01-2891-1992 Butir 8.2
79	**1.87**	0.03			1	Y	-	-	SNI 01-2891-1992 Butir 8.2
81	**2.07**	0.08			2.0327 mean	Y	Anhydrous Diethyl ether	20 cycles	Soxhlet Method
83	**11.08**	0.41			1.5	Y	HCC & Petroleum ether	2	SNI-01-2891-1992
84	**0.44**	-			5	Y	Petroleum Ether	4	SNI 01-2891-1992
86	**0.47**	0.05			2	Y	Hexane	0.75	AOAC (2012) 922.06
87	**0.52**	0.20			1.5	Y	Hexane	2	MTD/FOD/CHM-04
89	**0.84**	0.02			5	Y	Petroleum Ether	16	AOAC 963.15
90	**1.30**	0.04			2	-	Diethyl Ether	-	AOAC (2016) 954.02
91	**0.65**	0.03			-	-	-	-	-

Laboratory Number	Total fat (g/100g)	MU (g/100g)	z score	Zeta score	Fat weight (g)	Hydrolysis (Y/N)	Extraction Solvent	Extraction Time (hours)	Method Reference
92	**0.20**	-	**Not evaluate due to high variation of results**		1	N	Petroleum Ether	1	-
93	**1.48**	-			2	-	-	-	-
94	**2.48**	-			2	Y	Diethyl Ether and Petroleum Ether	-	AOAC (2012) 922.06
95	**0.28**	0.01			-	-	-	-	-
98	**0.22**	-			~1.5	N	Petroleum Ether	-	Ankom Filter bag Technique
100	**0.15**	0.01			1.5	N	Petroleum ether	60 mins	AOAC AM5-04 reapprove 2009

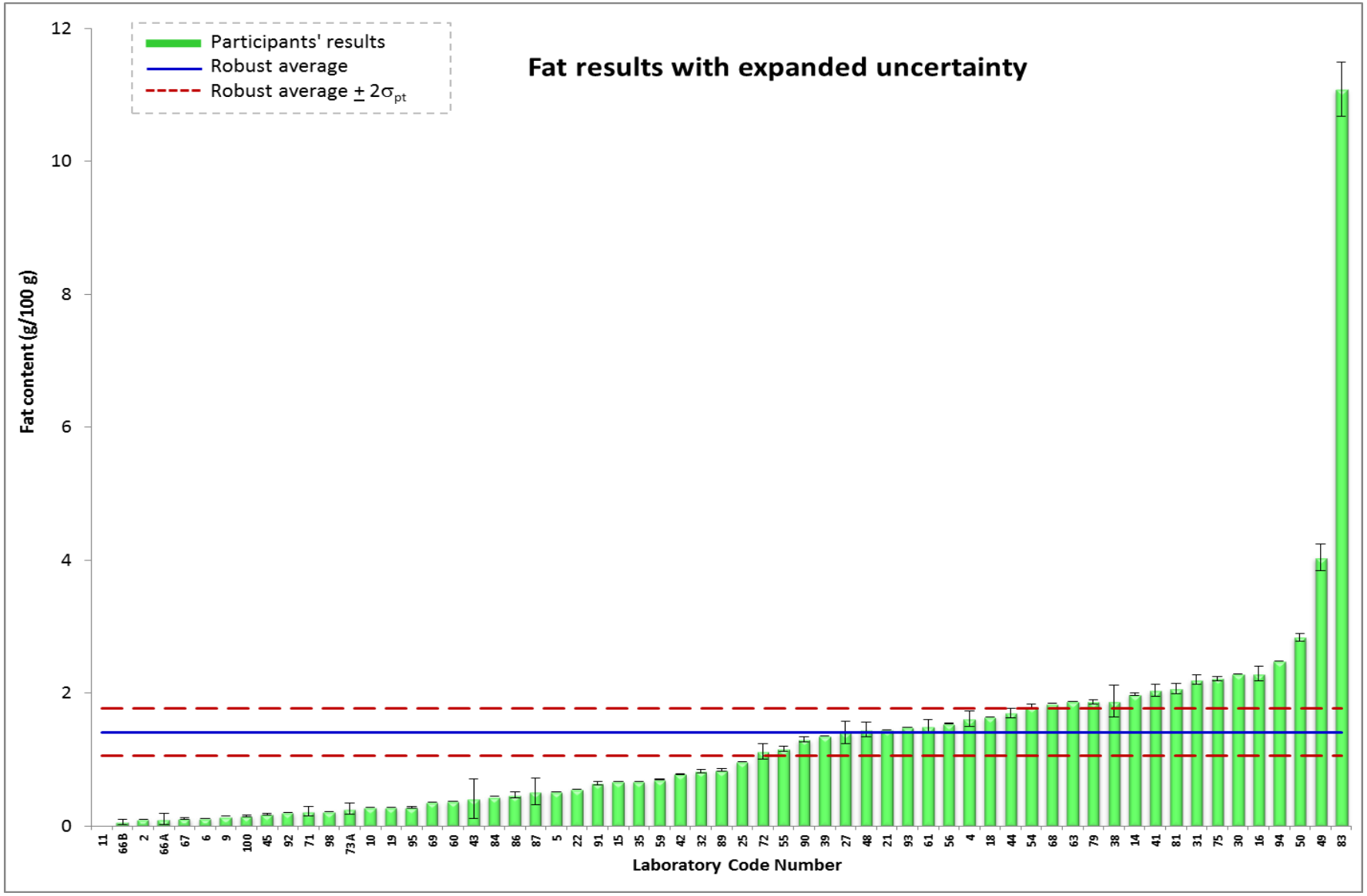

Figure 9. Distribution of **total fat** results (ascending order) in defatted soybean flour with expanded uncertainty

DIETARY FIBRE

Using defatted soybean flour as the test material, the step for fat removal in dietary fibre (DF) determination is omitted.

The AOAC enzymatic gravimetric methods was mainly used by participating laboratories for determination of total DF in 0.5 to 1 g of defatted soybean flour (except one good performance lab used 0.3 g). However, not all of them passed the criteria for good performance.

Thirty laboratories submitted the report on DF determination; 19 laboratories were identified as good performance laboratories (63%); 4 and 5 laboratories (30%) reported extremely high (and low levels, respectively and 2 of them reported questionable levels (**Figure 11**).

Activities and purity of enzymes alpha-amylase, protease and amyloglucosidase which involve in removing starch and protein in the sample are the critical factors in the determination of total DF. To ensure that enzymes have not degraded, the activities must be checked for each of new lot or at maximal interval of 6 months. Applying the degraded enzymes could affect the efficiencies in removing other components from the sample, resulting in extreme high values of the final residue of the DF.

Three laboratories used the AOAC methods for crude fibre or ISO method for neutral detergent dietary fibre determination; as expected they reports extremely low levels of DF.

In addition, it was found in the previous PT programmes, some laboratories did not corrected the amount of protein and ash from the residue; they reported extreme high values of DF.

The reference value of total dietary fibre content in defatted soybean flour obtained from 19 good performance laboratories is 16.80 ± 1.26 g/100 g (mean±SD, %CV=7.5).

Table 7. Evaluation of laboratory performance on **total dietary fibre** analysis (g/100 g, as received) in defatted soybean flour

Laboratory Number	Total dietary fibre g/100g)	MU (g/100g)	z score	Zeta score	Sample weight (g)	Digestion Technique	Digestion Medium	Method Reference
Assigned value obtained from robust average (x*) ± 3SD$_p$ = 16.44 ± 1.29 g/100 g (CV 7.8%, n= 30) with u$_{xpt}$ = 0.64 g/100 g								
		Acceptance criteria =	|z score| ≤ 2.00	|ζ score| ≤ 2.00				
2	**18.50**	-	1.60	-	0.50	Enzyme Digestion		AOAC (2016) 985.29
4	**16.50**	0.42	0.05	0.09	1.xxxx	Enzymatic	Buffer	Based on AOAC
11	**17.99**	-	1.20	-	0.5000	Enzyme	HCl	Based on AOAC (2016) 985.29
12	**18.60**	1.69	1.67	2.04	0.3	Enzymatic	Phosphate buffer	AOAC (2016) 985.29
14	**16.54**	-	0.08	-	0.5	Enzymatic - Gravimetric Method	α - amylase 97.5 ± 2.5°C 30 min, Protease 60 ± 1°C 30 min, Amyloglucosidase 60 ±1 °C 30 min	AOAC 985.29
16	**27.70**	0.27	**8.73**	**17.21**	1	-	-	AOAC 985.29
19	**1.82**	-	**-11.33**	-	1	Acid Digestion H2SO4 1.25%, 95°C 30 min	Base Diestion, NaOH 1, 1.25%, 95°C 30 min	AOAC 978.10, AN 304, FOSS, 2003
21	**9.23**	-	**-5.59**	-	1.0	Water bath	100 °C 15 min	AOAC 985.29 (2016)
31	**15.89**	-	-0.43	-	1	Enzimatik		AOAC 991.43
32	**16.50**	0.26	0.05	0.09	1.0100	Enzymatic Gravimetric	Buffer	AOAC 991.42

Laboratory Number	Total dietary fibre g/100g)	MU (g/100g)	z score	Zeta score	Sample weight (g)	Digestion Technique	Digestion Medium	Method Reference
Assigned value obtained from robust average (x*) ± 3SD$_p$ = 16.44 ± 1.29 g/100 g (CV 7.8%, n= 30) with u$_{xpt}$ = 0.64 g/100 g								
38	**18.50**	3.32	1.60	1.16	1.000	Enzymatic Digestion	Phosphate buffer	AOAC 985.29, 19th Ed 2012
39	**14.20**	-	-1.74	-	1	Enzyme	Buffer solution	AOAC 985.29
43	**31.02**	3.71	**11.30**	**7.43**	1	-	-	AOAC
48	**18.14**	0.43	1.32	2.52	1	Enzymatic	Amylase, protease, amyloglucosidase	AOAC 985.29 19th Ed 2012
49	**24.00**	0.00	**5.86**	**11.81**	0.5	Enzymatic	Alpha-Amylase, Protease, Amyloglucosidase	AOAC 20th Ed 2016 / Sigma Kit
54	**17.60**	0.10	0.90	1.81	1	Enzymatic	Phosphate buffer / Enzyme	AOAC 985.29
55	**13.45**	1.81	**-2.32**	**-2.70**	0.5	Enzymatic-Gravimetric	-	AOAC (2012) 985.29
58	**28.30**	0.17	**9.19**	**18.37**	1.0	-	-	Based on AOAC 20th Ed 2016
59	**7.38**	0.23	**-7.02**	**-13.93**	1 to 2	Enzymatic	-	AOAC 18th Ed 985.29
61	**15.40**	2.16	-0.81	-0.83	0.5	Enzymatically Digested with protease and amyloglucosidase	Methylated spirits	A6234 (ANKOM automated TDF instrument)
63	**16.70**	-	0.20	-	-	-	-	-
67	**14.40**	-	-1.58	-	1.0xxx	Enzyme Digestion	-	AOAC 985.29
68	**16.80**	-	0.28	-	0.5	Enzyme	-	AOAC
69	**16.26**	-	-0.14	-			-	

Laboratory Number	Total dietary fibre g/100g)	MU (g/100g)	z score	Zeta score	Sample weight (g)	Digestion Technique	Digestion Medium	Method Reference
Assigned value obtained from robust average (x*) ± 3SD$_p$ = 16.44 ± 1.29 g/100 g (CV 7.8%, n= 30) with u$_{xpt}$ = 0.64 g/100 g								
71	**5.27**	0.08	**-8.66**	**-17.42**	1.0018, 1.0008	Neutral Detrergent / heat stable alpha amylase	-	ISO 16472:2006
81	**16.90**	0.90	0.36	0.59	mean 1.0003	Enzymatic Digestion (Heat-stable alpha-amylase, protease, amyloglucosidase)	MES-TRIS Buffer	AOAC 991.43 (Modified)
83	**19.70**	1.04	**2.52**	**3.95**	1	Enzimatic gravimetry	-	AOAC 991.43
86	**17.00**	1.15	0.43	0.65	0.5	Enzymatic - Gravimetric Method	-	AOAC (2012) 985.29
90	**6.96**	2.28	**-7.35**	**-7.25**	0.5	Fibertec	-	AOAC (2016) 985.29
94	**16.76**	-	0.25	-	1	-	-	AOAC (2012) 985.29

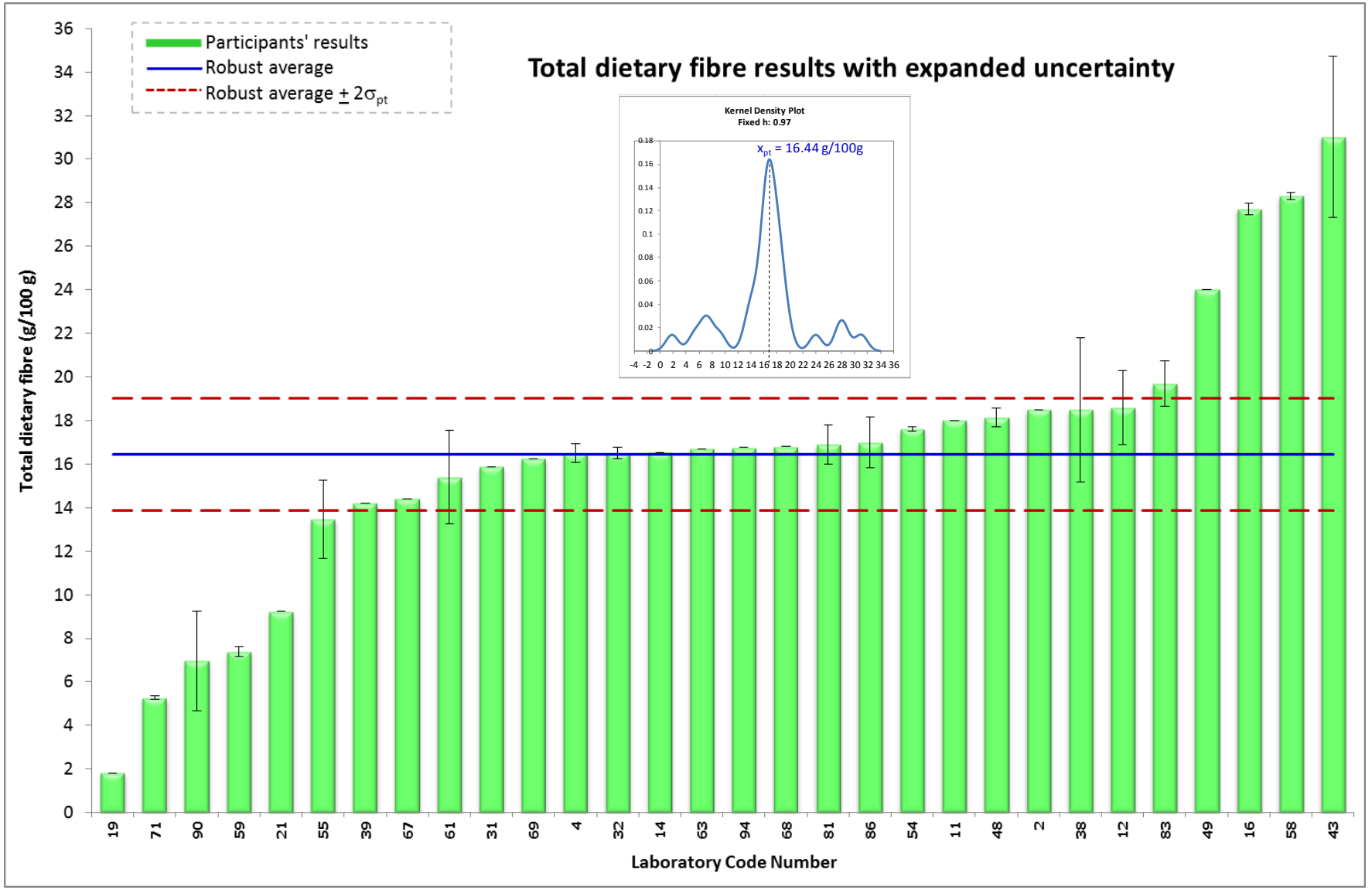

Figure 10. Distribution of **total dietary fibre** results (ascending order) in defatted soybean flour with expanded uncertainty

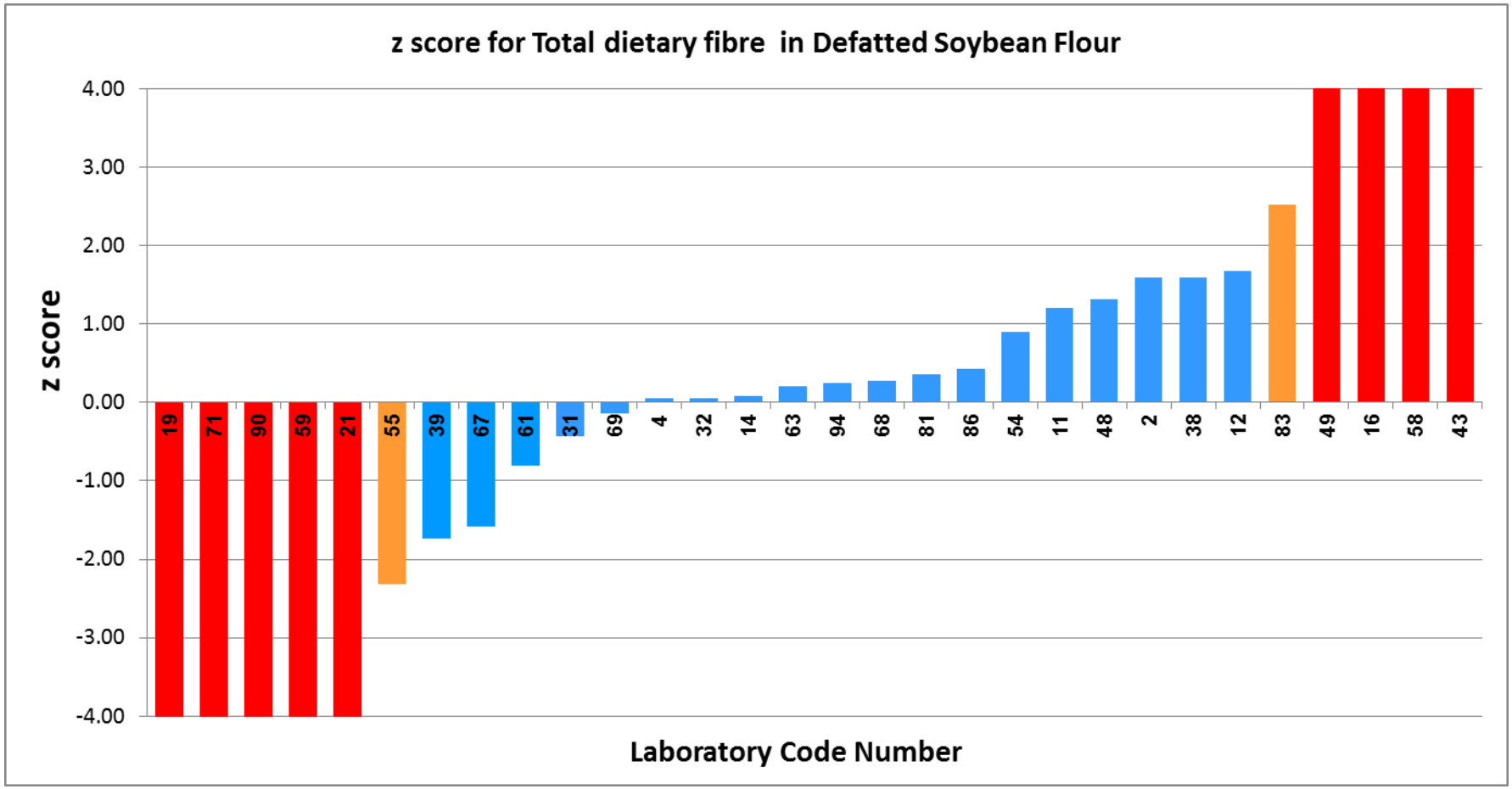

Figure 11. Plot of ordered *z* scores for **total dietary fibre** results in defatted soybean flour

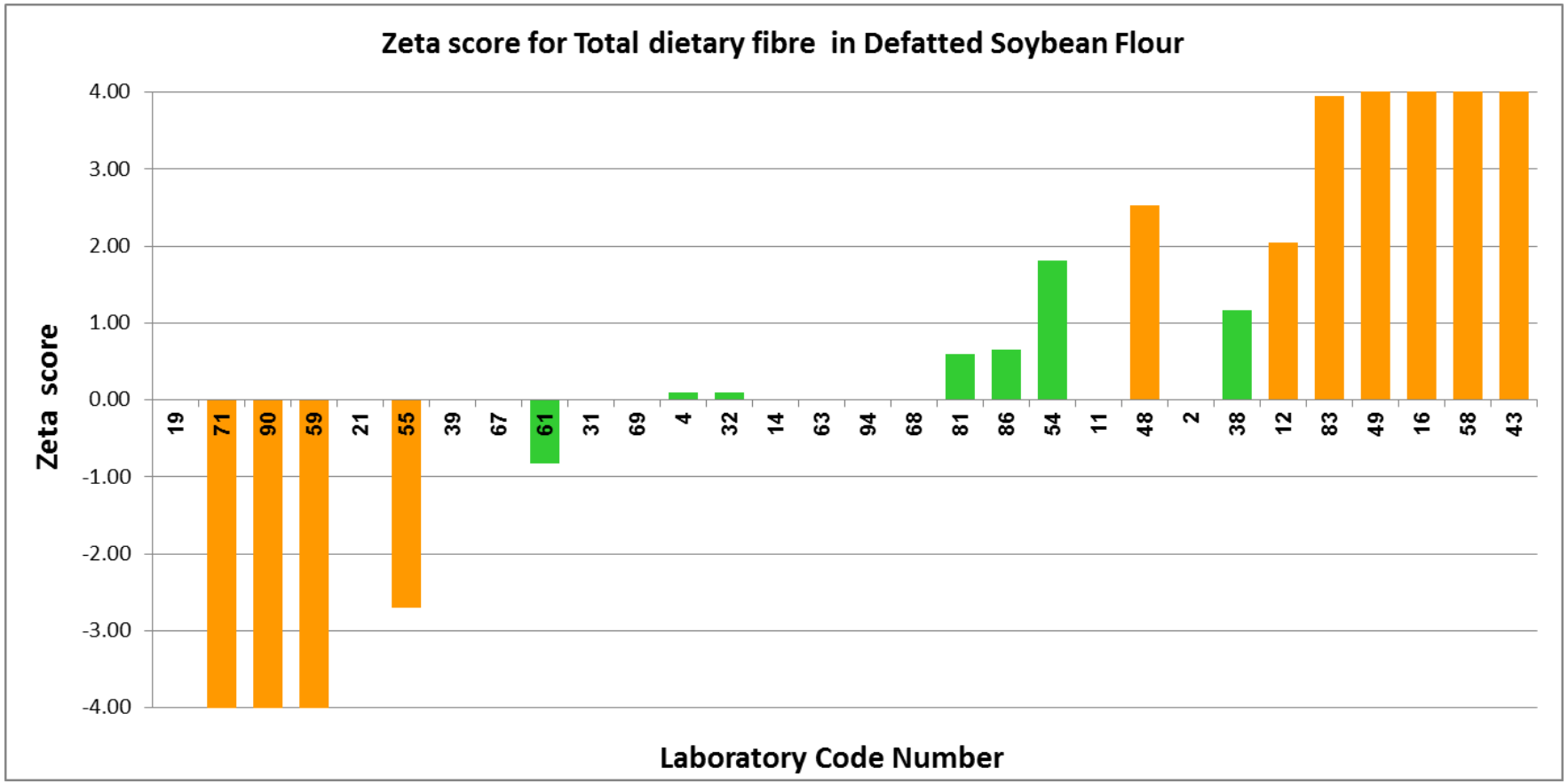

Figure 12. Plot of *Zeta score* for **total dietary fibre** in defatted soybean flour, following the ordered z scores in the above Figure 11

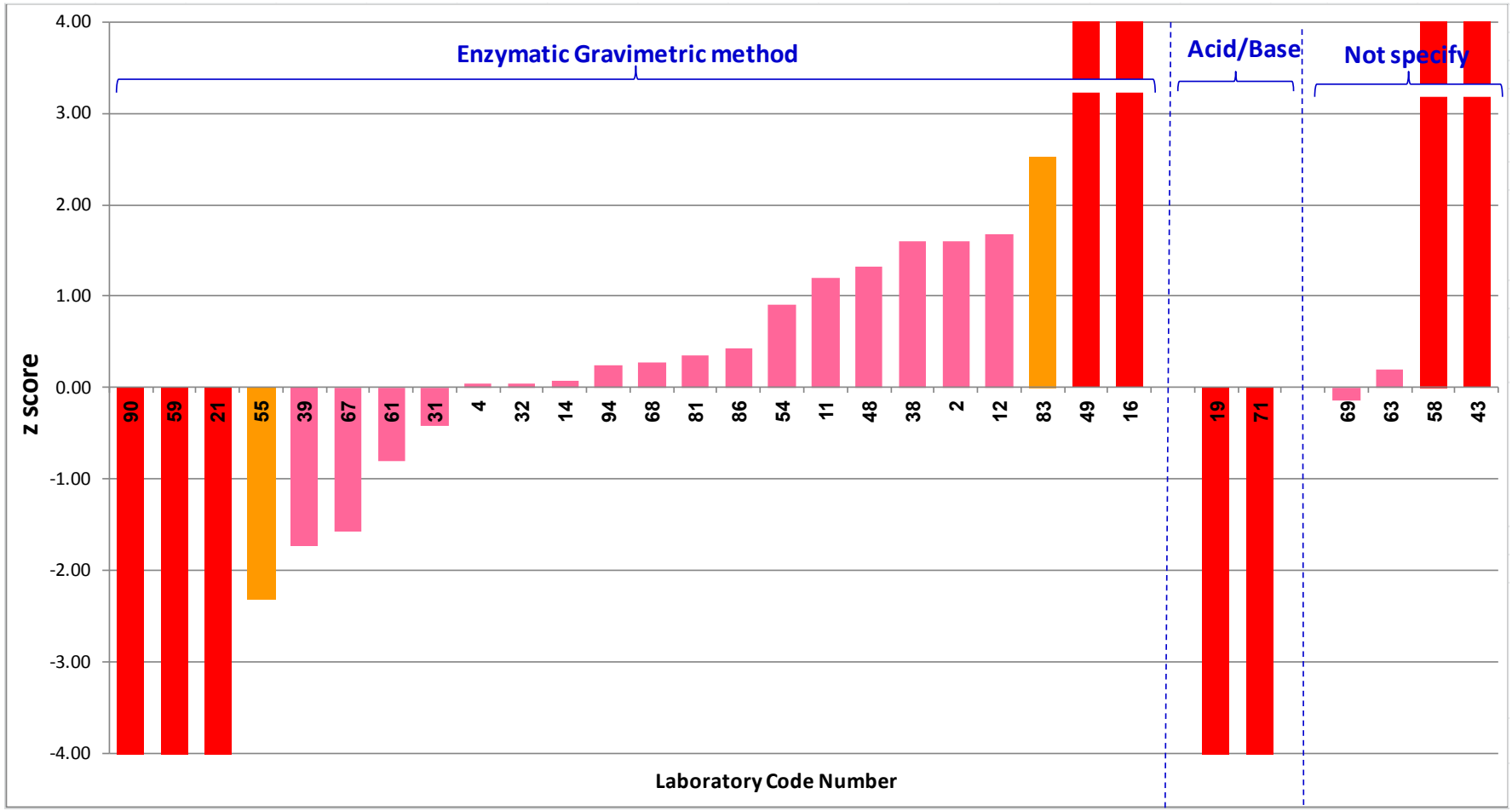

Figure 13. Plot of ordered *z* score for **total dietary fibre** in defatted soybean flour, categorised in groups according to analytical methods/parameters used

ASH

Seventy-three laboratories participated in ash determination. The majority of the participants (47 lab, 64%) used the temperature of 520-550^{o}C for 2 to 24 h for ashing. Others applied higher temperature of 600^{o}C for 2-10 h (**Table 8, Figure 17).**

About 56% of total participants (41 out of 73 lab) included *charring* the sample over a hotplate or a Bunsen burner before incineration in a muffle furnace.

Charring the sample over a hotplate, initially at low temperature to avoid losing ash with flame, then increase the temperature gradually until smoking ceases before incineration in a muffle furnace. If the sample is not completely white, moist ash with a few drops of water or diluted acid. Evaporate on water bath and repeat heating in the muffle furnace for 30 – 60 min until constant weight is obtained. This step is recommended as it could reduce the period for getting completely white ash under the high temperature in the muffle furnace. It could prevent fluffing of ash during opening the furnace

Based on z-scores, most laboratories (65 out of 73, 89%), were identified as good performance on ash analysis. Two lab each reported extreme high values and extreme low values and three reported questionable values; they must review each step of ash determination and do corrective action.

The consensus value of ash content in defatted soybean flour obtained from 65 good performance laboratories is 6.32 ± 0.32 g/100 g (mean±SD, N=65, %CV=5.0)

Table 8. Evaluation of laboratory performance on **ash** analysis (g/100 g, as received) in defatted soybean flour

Laboratory Number	Ash (g/100g)	MU (g/100g)	z score	Zeta score	Sample weight (g)	Pre-Charring (Y/N)	Ash Temperature (°C)	Ash Time (hours)	Method Reference
Assigned value obtained from robust average (x*) ± robust SD (s*) = 6.31 ± 0.33 g/100 g (CV 5.2%, n= 73) with u_{xpt} = 0.05 g/100 g									
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00					
2	**6.59**	-	0.85	-	2	Hot plate	600	2	AOAC (2016) 942.05
4	**6.20**	0.16	-0.33	-1.17	-	-	-	-	-
5	**6.15**	-	-0.49	-	2	-	550	3	ISO 5984:2002
6	**6.06**	0.21	-0.76	**-2.17**	2	Pre-burn on Hotplate	600	4	AOAC (2016) 942.05 4.1.10
8	**6.06**	0.01	-0.76	**-4.98**	5	Char 30 minutes	600 ± 20	Until grey ash	SLS 898:1990
9	**6.12**	-	-0.57	-	1 to 2	-	550	2.5	Based on ISO 5984:2002
10	**6.02**	-	-0.88	-	2	-	600	2	AOAC 2012, 32.2.09 B, Chapt 32
11	**6.69**	-	1.15	-	2	Pre heat 3 hour	550	3	ISO (5984) 2002 (E)
12	**6.44**	0.04	0.39	**2.41**	0.5	Charring on hot plate	550	2	AOAC (2016) 930.30, 945.46
14	**6.06**	0.05	-0.76	**-4.51**	5	Charring	550	5	AOAC 923.03
15	**6.02**	-	-0.88	-	4	-	550	8	AOAC (2016) 923.03
16	**5.97**	0.06	-1.03	**-5.83**	2 to 3	-	550	8	SNI 01-2891-1992 Food & Beverage
18	**6.66**	-	1.06	-	2	Charring	550	4	SNI 01-2891-1992
19	**5.84**	-	-1.43	-	1	-	600	3	AOAC 942.05
21	**6.83**	0.05	1.58	**9.23**	1	4	550	6	AOAC 923.03 (2016)
22	**7.30**	-	**3.00**	-	-	-	-	-	-

Laboratory Number	Ash (g/100g)	MU (g/100g)	z score	Zeta score	Sample weight (g)	Pre-Charring (Y/N)	Ash Temperature (°C)	Ash Time (hours)	Method Reference
Assigned value obtained from robust average (x*) ± robust SD (s*) = 6.31 ± 0.33 g/100 g (CV 5.2%, n= 73) with u_{xpt} = 0.05 g/100 g									
23	**6.16**	-	-0.45	-	2	-	550	3	ISO 5984
25	**6.53**	-	0.67	-	5.0205 / 5.0206	Addition of HNO_3	550	4	Laboratory Handbook of Methods of Food Analysis, 3rd Ed, R. Lees
26	**5.92**	0.01	-1.18	**-7.78**	4	Drying at 150°C	525	24	AOAC No. 923.03
27	**7.79**	0.19	**4.48**	**13.79**	2	Drying in vacuum oven 105°C 22 hours then 2 hours at 300°C in furnace	550	8	SNI 2354.1:2010
30	**6.88**	-	1.74	-	3.5037, 3.5033	-	550	10	AOAC (2016) 923.03
31	**6.65**	0.26	1.03	**2.44**	3	-	600	10	SNI 01-2891
32	**6.69**	0.14	1.15	**4.50**	2.0151	Charring	550	8	AOAC 923.03
35	**7.18**	0.12	**2.64**	**11.14**	5.3337	Charring on Bunsen	550 ± 25	3	Sri Lanka Standard 1011:1994 specification for Soya Flour
37	**6.30**	-	-0.04	-	3	Free flame by hotplate	550	4	AOAC (2016) 938.08
38	**6.49**	0.02	0.55	**3.50**	2	Charring	550	2	AOAC 923.03, 19th Ed 2012
39	**6.21**	-	-0.30	-	2	Charring	550	5 to 6	AOAC 942.05
40	**5.30**	0.03	**-3.06**	**-19.47**	-	-	-	-	-
41	**6.11**	0.30	-0.61	-1.29	2	-	600	2	AOAC
42	**0.13**	0.00	**-18.73**	**-123.60**	3	-	600	5	SNI 01-2891-1992. point 6.1
43	**6.29**	0.03	-0.06	-0.38	2	Charring	550	3 to constant	National Standard
44	**6.76**	0.75	1.36	1.19	1	Charring	520.0	8	AOAC 19th Ed, 2012

Laboratory Number	Ash (g/100g)	MU (g/100g)	z score	Zeta score	Sample weight (g)	Pre-Charring (Y/N)	Ash Temperature (°C)	Ash Time (hours)	Method Reference
Assigned value obtained from robust average (x*) ± robust SD (s*) = 6.31 ± 0.33 g/100 g (CV 5.2%, n= 73) with u_{xpt} = 0.05 g/100 g									
45	**6.72**	0.08	1.23	**6.42**	2	-	550 ± 20	3	ISO 5984
48	**6.48**	0.14	0.51	1.96	3	Charring	550	3 then 1 then 1	SNI 3549 2009
49	**6.67**	0.33	1.09	**2.09**	1, 3	Charring	555	6	AOAC 20th Ed 2016
50	**6.49**	0.12	0.55	**2.36**	2.0118	Charring	550.0	6	AOAC 923.03
54	**6.44**	0.10	0.39	1.84	1	Charring	525	5	AOAC 92.03
55	**6.21**	0.04	-0.32	-1.93	3	Charring	550	2	AOAC (2012) 923.03
56	**6.26**	0.06	-0.15	-0.86	4.02388	None	550	8	AOAC Intl 20th Ed, 2016 923.03
58	**6.46**	0.58	0.45	0.51	3	-	550	8	Based on AOAC 20th Ed 2016
59	**6.16**	0.06	-0.45	**-2.57**	2 to 3	-	550	15	SNI 01-2891-1992 point 6.1
60	**6.34**	-	0.09	-	-	-	-	-	SNI 01-2891-1992 Butir 6
61	**6.08**	0.19	-0.70	**-2.16**	2	N/A	550	15	A6401 550C Ash
62	**6.58**	-	0.82	-	-	-	-	-	-
63	**6.10**	-	-0.64	-	-	-	-	-	-
65	**6.20**	-	-0.35	-	5.1881	-	545	2.5	Drying method
67	**6.99**	0.15	**2.06**	**7.54**	3.0xxx	-	550	2	AOAC 923.03
68	**6.60**	-	0.88	-	2	Y	600	2	AOAC
69	**6.34**	-	0.09	-	-	-	-	-	-
70	**6.64**	0.50	1.00	1.29	1	-	550	5	-
71	**5.92**	0.48	-1.18	-1.59	1.0055, 1.0063	-	600	3	AOAC 942.05

Laboratory Number	Ash (g/100g)	MU (g/100g)	z score	Zeta score	Sample weight (g)	Pre-Charring (Y/N)	Ash Temperature (°C)	Ash Time (hours)	Method Reference
Assigned value obtained from robust average (x*) ± robust SD (s*) = 6.31 ± 0.33 g/100 g (CV 5.2%, n= 73) with u_{xpt} = 0.05 g/100 g									
72	**6.58**	0.05	0.82	**4.83**	2	Charring	550	4	AOAC 923.03
73A	**6.02**	0.13	-0.88	-	1	N	600	3.5	FTC-05.01 (refers to AOAC 942.05)
75	**7.32**	0.40	**3.05**	**4.89**	2	-	550	4	SNI 01-2891-1992 Butir 6.1
79	**5.99**	0.30	-0.96	**-2.02**	2 to 3	-	550	-	SNI 01-2891-1992 Butir 6.1
80	**6.02**	-	-0.88	-	2.xx	N/A	600	Constant weight	AOAC 942.05
81	**6.08**	0.10	-0.70	**-3.25**	3.0341 mean	Charring	550	10	AOAC 923.03
83	**6.10**	0.34	-0.65	-1.22	2	-	550	4	SNI-01-2891-1992
84	**6.16**	-	-0.45	-	2	Charring	600	6	AOAC 945.39
85	**6.45**	0.02	0.42	**2.75**	2	-	550	3	SNI 01-2896-1992
86	**6.24**	0.69	-0.21	-0.20	2	Charring	600	2	AOAC (2012) 945.39B
87	**6.07**	0.18	-0.72	**-2.30**	2.5	Heating on hotplate	550	Overnight	MTD/FOD/CHM-02
89	**6.45**	0.03	0.43	**2.67**	2	Charring on hotplate	550	16	AOAC 930.30
90	**6.06**	0.14	-0.76	**-2.93**	2	-	550	3	AOAC (2016) 942.05
91	**6.28**	0.02	-0.09	-0.59	-	-	-	-	-
92	**6.32**	-	0.03	-	5		550	3	
93	**6.17**	-	-0.42	-	2	Charring	600	2	AOAC 942.05, 945.39
94	**6.31**	-	0.00	-	2	Charring	550	5	AOAC (2012) 945.46
95	**6.04**	0.06	-0.82	**-4.73**	-	-	-	-	-
96	**5.61**	-	**-2.12**	-	3	-	550	4	TCVN 8124:2009
97	**6.00**	-	-0.94	-	3	-	600	5	Sri Lanka Standard 1011:1994
98	**6.31**	-	0.00	-	~2.0	N	600	4	AOAC 942.05
100	**6.13**	0.12	-0.55	-	2	-	600	2	AOAC (2016) 942.05

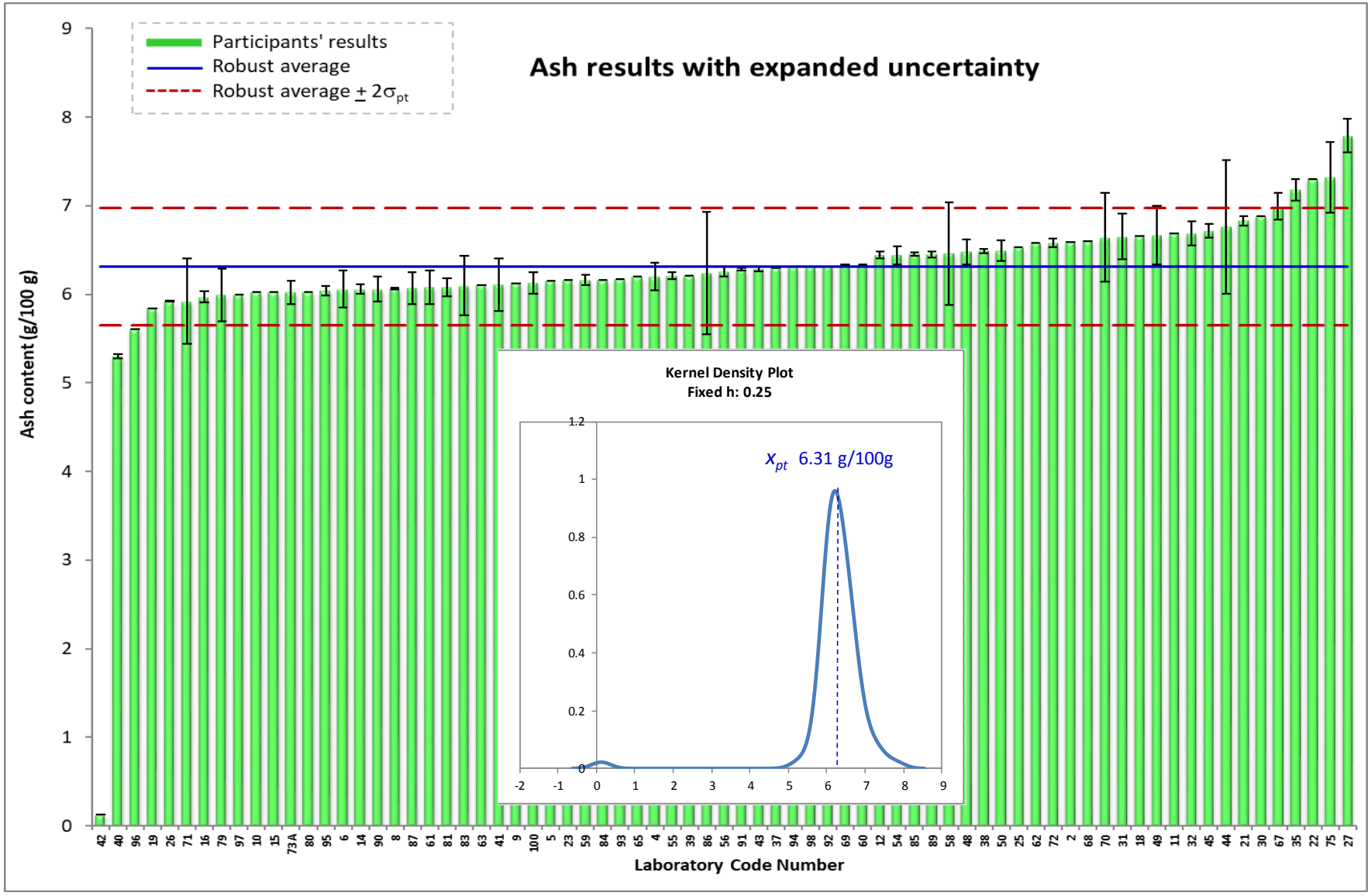

Figure 14. Distribution of **ash** results (ascending order) in defatted soybean flour with expanded uncertainty

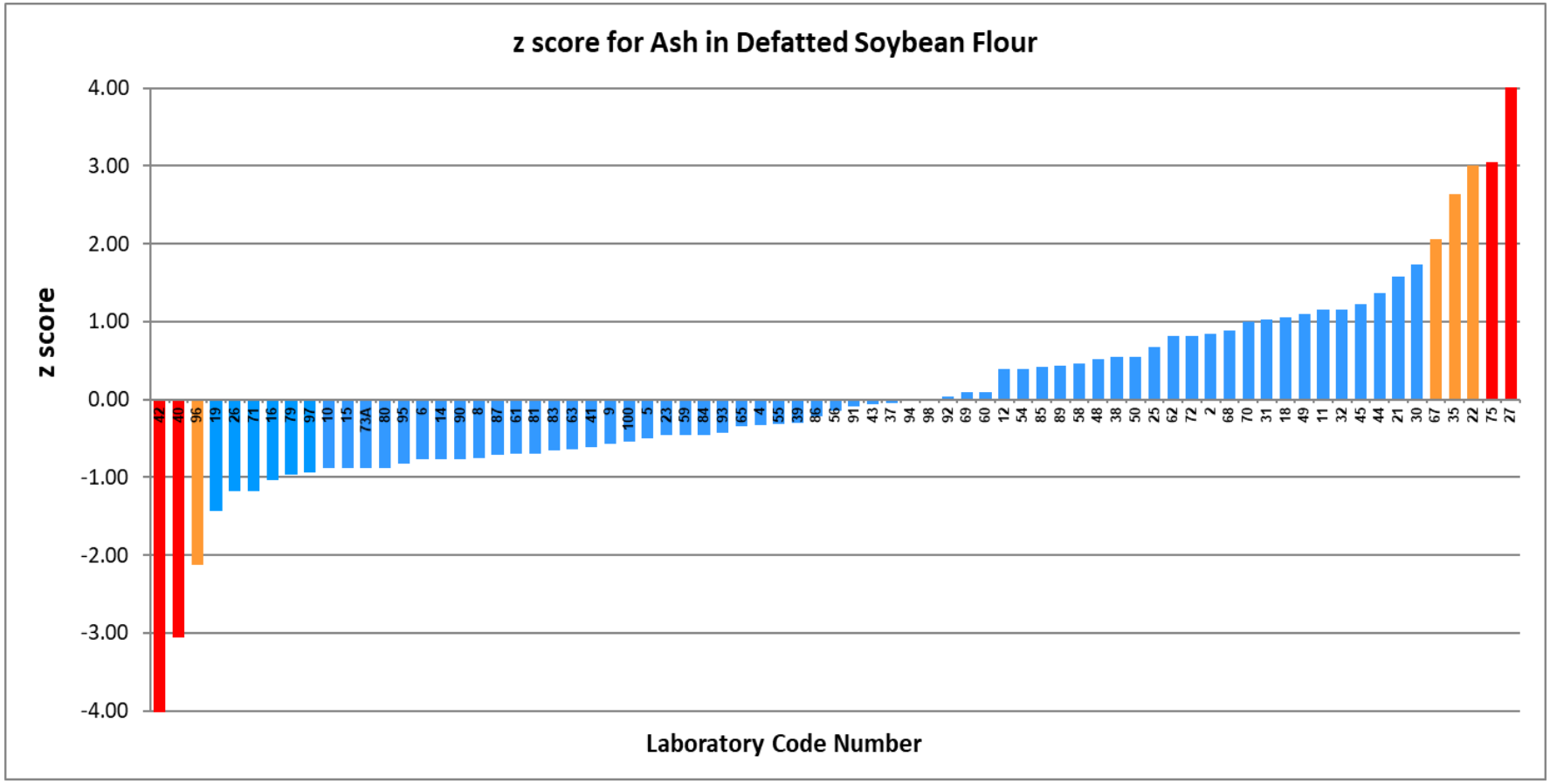

Figure 15. Plot of ordered *z* scores for **ash** results in defatted soybean flour

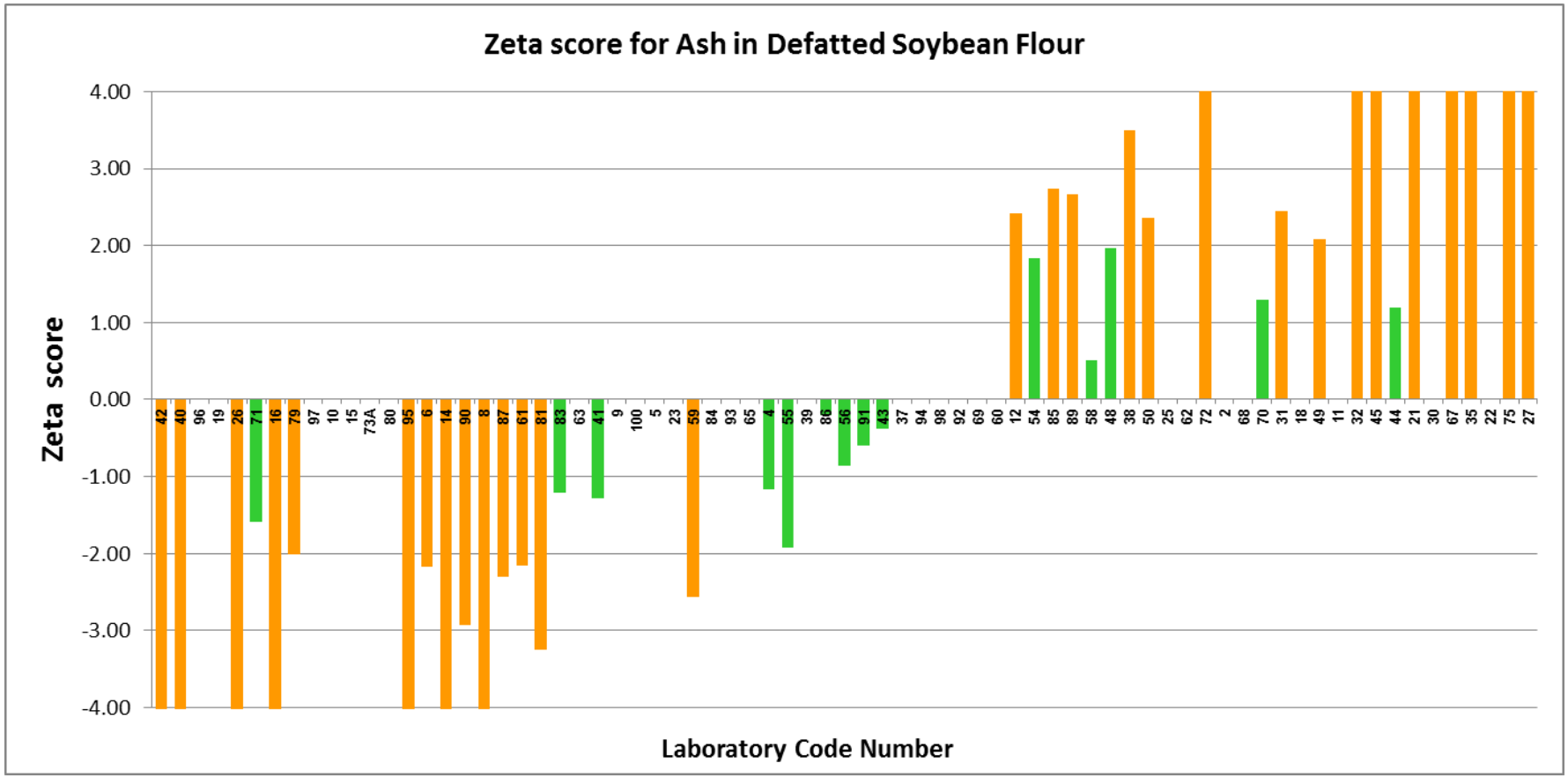

Figure 16. Plot of *Zeta* score for **ash** in defatted soybean flour, following the ordered z scores in the above Figure 15.

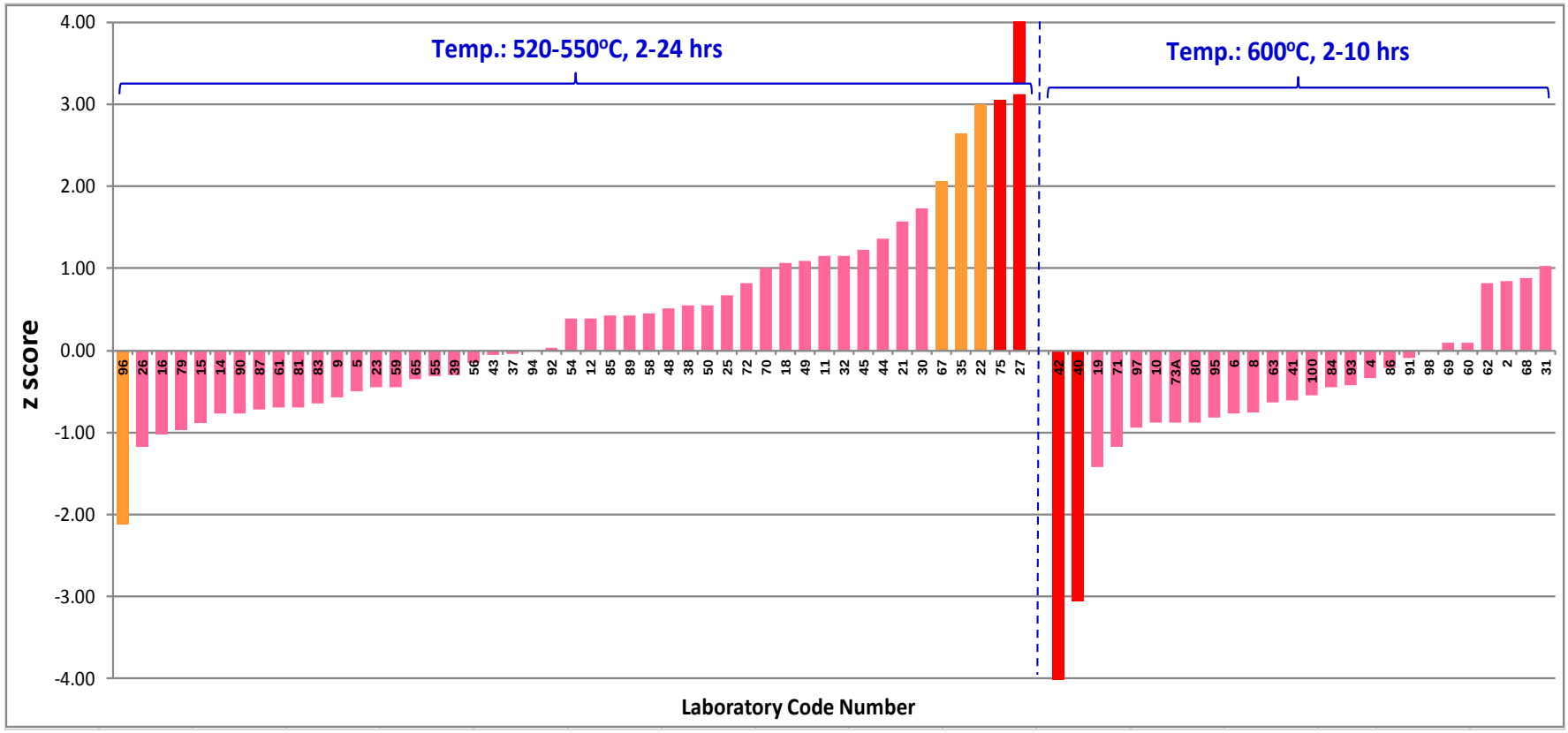

Figure 17. Plot of ordered *z* score for **ash** in defatted soybean flour, grouping by ashing temperatures

MINERALS

Sample preparation by dry ashing or wet digestion was generally conducted for minerals analyses. If dry ashing is used, temperature of the furnace should not exceed 450°C. Too high a temperature may cause the volatilisation of certain elements particularly Fe, K, Na, S, Cl & P.

In this PT prgramme, the ashing tempertures higher than 450°C (520-550°C and 600°C), were used and more than 50% of the Lab prepared the sample for mineral analysis by dry ashing. Laboratory performance on mineral analysis: effects of sample preparation - dry ashing VS wet digestion - were evaluated.

Lab performance on Mineral analysis	Sample preparation	
	Dry ashing	Wet digestion
CALCIUM (Ca)		
Total No. of submitted data (N=47)	28 (60%)	19
% Lab with Good performance	57	63
% Lab with Extreme high/low values	28	26
% Questionable high/low values	14	11
POTASSIUM (K)		
Total No. of submitted data (N=45)	25 (56%)	20
% Lab with Good performance	76	75
% Lab with Extreme high/low values	12	15
% Questionable high/low values	12	10
IRON (Fe)		
Total No. of submitted data (N=48)	26 (54%)	22
% Lab with Good performance	65	82
% Lab with Extreme high/low values	16	9
% Questionable high/low values	19	9

Data evaluation: a trial

For Calcium and Iron:
sample prepared by wet digestion shows higher percentage of laboratory with good performance compared to those prepared by dry ashing.

For Potassium:
No effect of methods used for sample preparation on the laboratory performance

Table 9. Evaluation of laboratory performance **calcium** analysis (mg/kg, as received) in defatted soybean flour

Lab Number	Calcium (mg/kg)	MU (mg/kg)	Based on reference values[1]		Based on x* ± 2SDp[2]		Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wave-length (nm or mass)	Recovery Correction (Y/N)	Method Reference
			z score	Zeta score	z score	Zeta score							
[1]Assigned value obtained from reference values (Gravimetric standard addition ICP-MS as x_{pt} ± $2SD_p$ from Horwitz' s equation) = 2100 ± 207 mg/kg (CV 9.9%) with u_{xpt} = 29 mg/kg; [2]Assigned value obtained from robust average (x) ± $2SD_p$ from Horwitz' s equation = 2031 ± 207 mg/kg (CV 10.2%, n= 52) with u_{xpt} = 36 mg/kg*													
6	**2189**	238.1	0.43	0.72	0.76	1.14	2.0000	Acid	$HCl:HNO_3:H_2O$	AAS	Ca 422.7	Y	AOAC (2016, 20th Ed, 928.08, 985.35 (50.1.14)
11	**43816**	-	**202.00**	-	**202.33**	-	2.0000	Dry Ashing	$HCl:H_2O$	AAS	Ca 239.9	Y	AOAC (2016), 975.03, 985.35
12	**1720**	140.0	-1.84	**-4.83**	-1.51	**-3.11**	0.5	Closed vessel	HNO_3	Flame AAS	Ca 422.7	N	AOAC (2016), 985.35
13	**1120**	-	**-4.75**	-	**-4.41**	-	0.5	Microwave	HNO_3 10 mL + HCl 2 mL	Analytikal Jena ContrAA 800 D	Ca 422	N	Internal Method
14	**1963**	-	-0.66	-	-0.33	-	0.5	Ashing	50% HNO_3, 50% HCl	ICP Horiba Jobin Yvon	Ca 393.366	Y	AOAC 975.03, 984.27
15	**2080**	-	-0.10	-	0.24	-	0.5	Ultrawave Digestion	5% HNO_3 + 0.5% HCl	ICP-MS (7900 Agilent)	Ca 44	N	Based on USFDA 4.7 version 1.1
16	**2253**	23.0	0.74	**4.07**	1.07	**3.06**	0.5	Hot plate	HNO_3+H_2O_2	ICP-OES Optima 7000 DV Perkin Elmer	Ca 317.933	N	In-house Method
18	**1250**	-	**-4.12**	-	**-3.78**	-	2.0	Dry Ashing	HNO3	AAS, Varian	Various	N	AOAC 968.08
19	**3970**	-	**9.05**	-	**9.39**	-	1	Furnace	HNO3:H 2O (1:1)	-	Ca Manual by Buret	N	AOAC 927.02, 944.03, 965.17
21	**2544**	6.6	**2.15**	**12.35**	**2.48**	**7.16**	0.1	Microwave	180oC	Mar Xpress (CEM)	-	Y	AOAC 2011.14 (2016)
22	**741**	-	**-6.58**	-	**-6.25**	-	0.2 to 0.3	Microwave	HNO3	ICP-MS Perkin Elmer	-	-	AOAC 2015.06
23	**2020**	-	-0.39	-	-0.05	-	1.00	Dry Ashing	-	ICP-OES	589, 766, 422, 285, 238	-	AOAC 985.01

Lab Number	Calcium mg/kg	MU mg/kg	Based on reference values[1]		Based on x* ± 2SDp[2]		Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wave-length (nm or mass)	Recovery Correction (Y/N)	Method Reference
			z score	Zeta score	z score	Zeta score							
[1]Assigned value obtained from reference values (Gravimetric standard addition ICP-MS as x_{pt} ± $2SD_p$ from Horwitz' s equation) = 2100 ± 207 mg/kg (CV 9.9%) with u_{xpt} = 29 mg/kg; [2]Assigned value obtained from robust average (x) ± $2SD_p$ from Horwitz' s equation = 2031 ± 207 mg/kg (CV 10.2%, n= 52) with u_{xpt} = 36 mg/kg*													
25	**1230**	0.1	**-4.21**	**-24.30**	**-3.88**	**-11.19**	5.0205 / 5.0206	Wet Digestion	HNO_3-HCl	ICP-OES	Ca 396.847	-	USEPA Method 3050B
26	**1990**	112	-0.53	-1.65	-0.20	-0.45	4.0	Dry ashing	Water & HCl (1+1)	AAS Shimadzu AA-7000	Ca 422.7	N	AOAC No. 975.03
31	**1876**	-	-1.09	-	-0.75	-	5	Dry Ashing	-	AAS, Agilent	-	N	AOAC 985.35
37	**1998**	-	-0.49	-	-0.16	-	1	Wet Digestion	Nitric + perchloric	ICP-OES (Perkin Elmer Optima 8000)	Ca 317.933	N	AOAC (2016) 984.27
38	**1740**	82	-1.74	**-6.61**	-1.41	**-3.53**	1.000	Dry Ashing	1N HNO_3	Flame AAS, Shimadzu AA6300	Ca 422.70	-	AOAC 985.35, 19th Ed 2012
39	**1920**	-	-0.87	-	-0.54	-	0.5	Microwave	-	AAS	Ca 422.7	Y	AOAC 985.35
41	**0.3**	-	**-10.17**	-	**-9.83**	-	2	-	-	-	-	-	-
42	**1970**	126	-0.63	-1.79	-0.30	-0.64	5	Dry Ashing	HNO_3-HCl	Flame AAS, Agilent 280 FS	Ca 422.7	N	AOAC 985.35.2005
43	**2148**	291	0.23	0.32	0.57	0.72	0.5	Microwave	HNO3	ICP-OES	Ca 317.933	N	AOAC
44	**1575**	132	**-2.54**	**-6.99**	**-2.21**	**-4.68**	1.0000	Dry Ashing		AAS, Thermoscientific	Ca 422.7	N	AOAC 19th Ed
45	**1948**	67	-0.74	**-3.12**	-0.40	-1.06	4	Dry Ashing	HCl+HNO_3+DI (2+2+70 mL) on hotplate	AAS (Flame, Varian)	Ca 422.7	N	AOAC 968.08
48	**2482**	24	1.85	**10.15**	**2.19**	**6.22**	5	Dry Digestion		AA800 Perkin Elmer	Ca 422.7	N	MU-03/21 (AAS)
49	**1640**	82	**-2.23**	**-8.45**	-1.89	**-4.74**	1, 3	Dry Ashing	Conc Nitric acid	AAS / AA-7000 Shimadzu	Ca 422.7	N	AOAC 20th Ed 2016
50	**1585**	86	**-2.49**	**-9.20**	**-2.16**	**-5.34**	2.0000	Wet	Acid	Flame AAS (Varian)	422.7	N	AOAC 985.35
53	**2806**	197	**3.42**	**6.74**	**3.75**	**6.36**	0.3	Microwave	4 mL HNO_3, 1 mL HCl, 1 mL H_2O_2	ICPMS Thermo	-	-	In house method

Lab Number	Calcium mg/kg	MU mg/kg	Based on reference values[1]		Based on x* ± 2SDp[2]		Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wave-length (nm or mass)	Recovery Correction (Y/N)	Method Reference
			z score	Zeta score	z score	Zeta score							
[1]Assigned value obtained from reference values (Gravimetric standard addition ICP-MS as x_{pt} ± $2SD_p$ from Horwitz' s equation) = 2100 ± 207 mg/kg (CV 9.9%) with u_{xpt} = 29 mg/kg; [2]Assigned value obtained from robust average (x) ± $2SD_p$ from Horwitz' s equation = 2031 ± 207 mg/kg (CV 10.2%, n= 52) with u_{xpt} = 36 mg/kg*													
54	**1920**	233	-0.87	-1.48	-0.54	-0.81	1	Dry Ashing	HNO_3	ICP / Shimadzu	Ca 317.933	N	AOAC 984.27
55	**204**	-	-0.32	-	0.01	-	1.5	Wet digestion		ICP-OES	Ca 317.933	Y	AOAC (2012) 984.27
58	**2053**	23	-0.23	-1.26	0.10	0.30	3.0	Dry Ash	HCl	ICP-OES			Dry Ashing and Quantitation by ICP-OES
59	**1975**	136	-0.60	-1.62	-0.27	-0.56	1.5	Dry Ashing		AAS, Shimadzu	Ca 422.7	Y	AOAC 18th Ed 985.35
60	**2000**	-	-0.48	-	-0.15	-							AOAC (2012) 968.08 (Ca, Mg)
61	**1970**	540	-0.63	-0.48	-0.30	-0.22	1	Acid block digestion	HNO_3 (HNO_3/$HClO_4$ for P)	Varian AA240 FS Fast Sequential AAS	Ca 422.7	N	A6407-26 AAS
63	**1785**	-	-1.53	-	-1.19	-							
64	**2192**	28	0.44	**2.39**	0.78	**2.20**	0.5070	Dry Ashing	1 N HNO_3	Shimadzu AA6300	Ca 422.7	N	Modified AOAC 985.35
67	**2100**	-	0.00	-	0.33	-	2.0xxx	Dry Ash	Wet chemical	AAS, Perkin Elmer	Ca 422.67	N	AOAC 968.08
69	**2350**	-	1.21	-	1.54	-							
71	**0.2**	0.0	**-10.17**	**-58.65**	**-9.83**	**-28.36**	1.0036, 1.0063	Acid Digestion	HCl (1:3)				AOAC 927.02, Titration
72	**1760**	167	-1.65	**-3.74**	-1.31	**-2.46**	3	Ashing	HNO3	AAS / Analytik Jena	Ca 422.7	N	AOAC 985.35
73A	**0.2**	0.1	**-10.17**	**-58.65**	**-9.83**	**-28.36**	1	Dry ashing	Hot plate	AAS (280FS AA, Agilent Technology)	Ca 422.7	N	FTC-46.01 (refers to AOAC 968.08, 965.09)
73B	**0.2**	0.0	**-10.17**	**-58.65**	**-9.83**	**-28.36**	1	Dry ashing	Hot plate	AAS (280FS AA, Agilent Technology)	Ca 422.7	N	FTC-46.01 (refers to AOAC 968.08, 965.09)
75	**2054**	131	-0.22	-0.62	0.11	0.24	1	Wet digestion (hot block)	HNO3 + H2O2	ICP-OES Agilent 5100	Ca 317.933	N	In House Method ICP-OES

Lab Number	Calcium mg/kg	MU mg/kg	Based on reference values[1] z score	Based on reference values[1] Zeta score	Based on x* ± 2SDp[2] z score	Based on x* ± 2SDp[2] Zeta score	Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wave-length (nm or mass)	Recovery Correction (Y/N)	Method Reference
[1]Assigned value obtained from reference values (Gravimetric standard addition ICP-MS as x_{pt} ± $2SD_p$ from Horwitz' s equation) = 2100 ± 207 mg/kg (CV 9.9%) with u_{xpt} = 29 mg/kg; [2]Assigned value obtained from robust average (x) ± $2SD_p$ from Horwitz' s equation = 2031 ± 207 mg/kg (CV 10.2%, n= 52) with u_{xpt} = 36 mg/kg*													
78	**2529**	120	**2.08**	**6.14**	**2.41**	**5.33**	0.5	Mircowave Digestion	Acid Digestion	Berghof Speedwave 4 Microwave Digestion Unit	Ca 393.366		MP-AES
81	**2150**	123	0.24	0.70	0.58	1.26	mean: Ca 1.0027,	Dry Ashing (Ca, Fe)	1 N HNO_3 (Ca, Fe)	Shimadzu AAS AA 6300	Ca 422.7	N	AOAC 999.10 Mod (Na, K), 985.35 Mod (Ca, Fe)
82A	**2340**	66	1.16	**4.93**	1.50	**3.92**	0.250	none	none	HPGe detector, Canberra			Neutron Activation Analysis (NAA)
82B	**2470**	140	1.79	**4.71**	**2.13**	**4.38**	1.00	Microwave	Nitric Acid	AAS, GBC		Y	Flame SSA
83	**2259**	40	0.77	**3.88**	1.10	**3.07**	0.3	Microwave Digestion with HNO3	-	Microwave digester Mars Xpress, ICP MS Nex Ion (Perkin Elmer)		Y	Application Note, Perkin Elmer
84	**2180**	220	0.39	0.69	0.72	1.13	0.5	Microwave Digestion	HNO_3 / H_2O_2	ICP-OES, ICP-MS	Ca 317.933	N	AOAC 999.10:2005
86	**1958**	115	-0.69	**-2.10**	-0.35	-0.80	1.0000	Wet Digest	-	ICP-OES	Ca 315.8	Y	AOAC (2012) 984.27
87	**2479**	62	1.84	**8.01**	**2.17**	**5.75**	2.5	Dry Ashing	HNO_3	Furnace Thermolyne	ICP-OES	N	MTD/FOD/CHM-09
89	**2442**	37	1.65	**8.49**	1.99	**5.55**	2	Dry Ashing	1.5% HNO_3	AAS Agilent	Various	N	AOAC 985.35
90	**2273**	-	0.84	-	1.17	-	1	Ultrawave	-	ICP-OES	Ca 422.673	-	-
91	**2100**	-	0.00	-	0.33	-	-	-	-	-	-	-	-
92	**215**	-	**-9.13**	-	**-8.79**	-	1	Ashing	HNO_3	ICP-OES	-	-	-
93	**200**	-	**-9.20**	-	**-8.87**	-	0.05	Charring, Dry ashing	Hotplate, Furnace	Flame Photometer, Sherwood	N/A	N/A	AOAC 985.35
94	**2496**	-	1.92	-	**2.25**	-	1.5	Dry ashing	-	ICP-OES / Perkin Elmer	Ca 317.9	Y	AOAC (2012) 984.27
95	**2060**	770	-0.19	-0.10	0.14	0.07	-	-	-	-	-	-	-

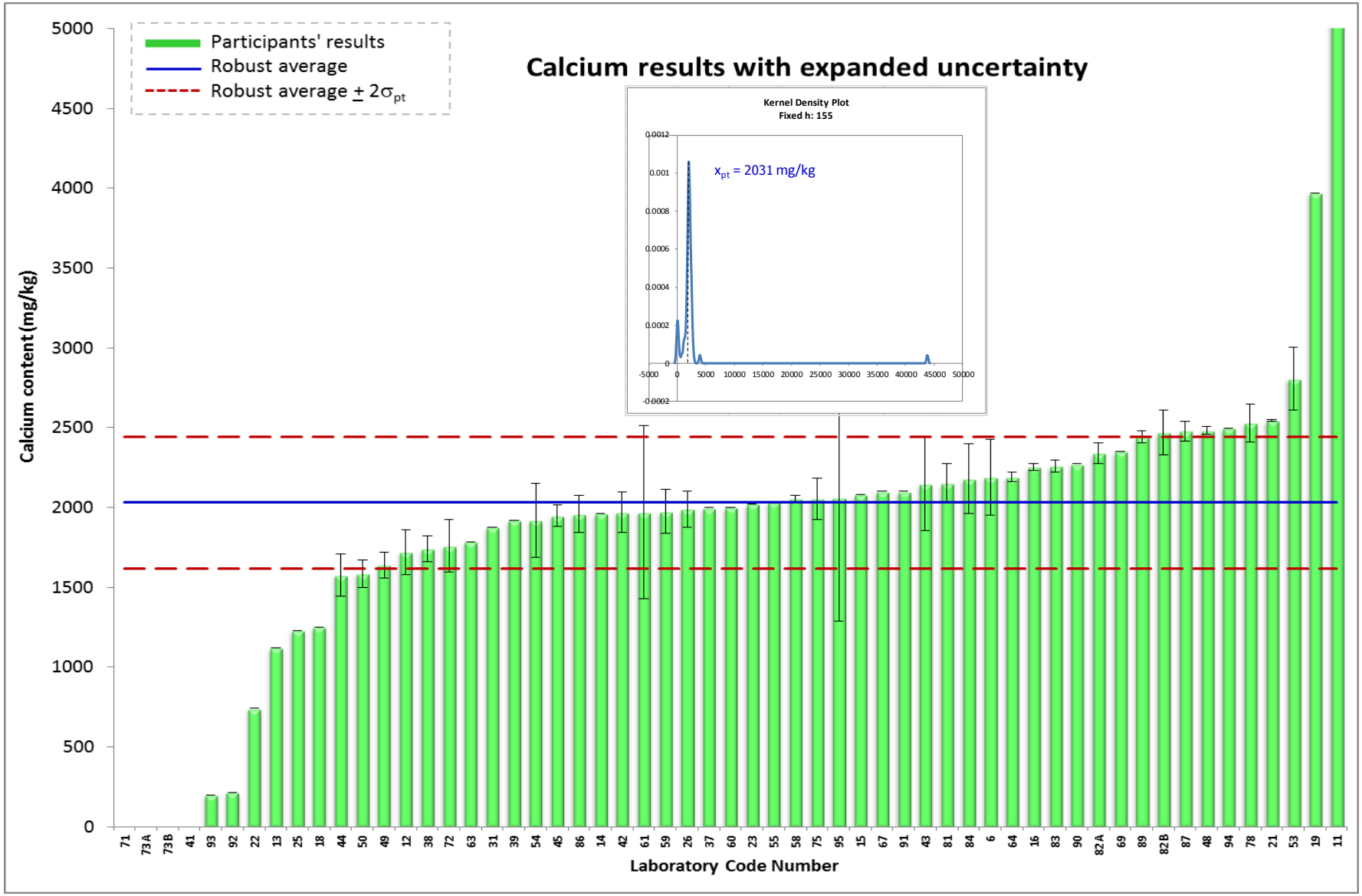

Figure 18. Distribution of **calcium** results (ascending order) in defatted soybean flour with expanded uncertainty

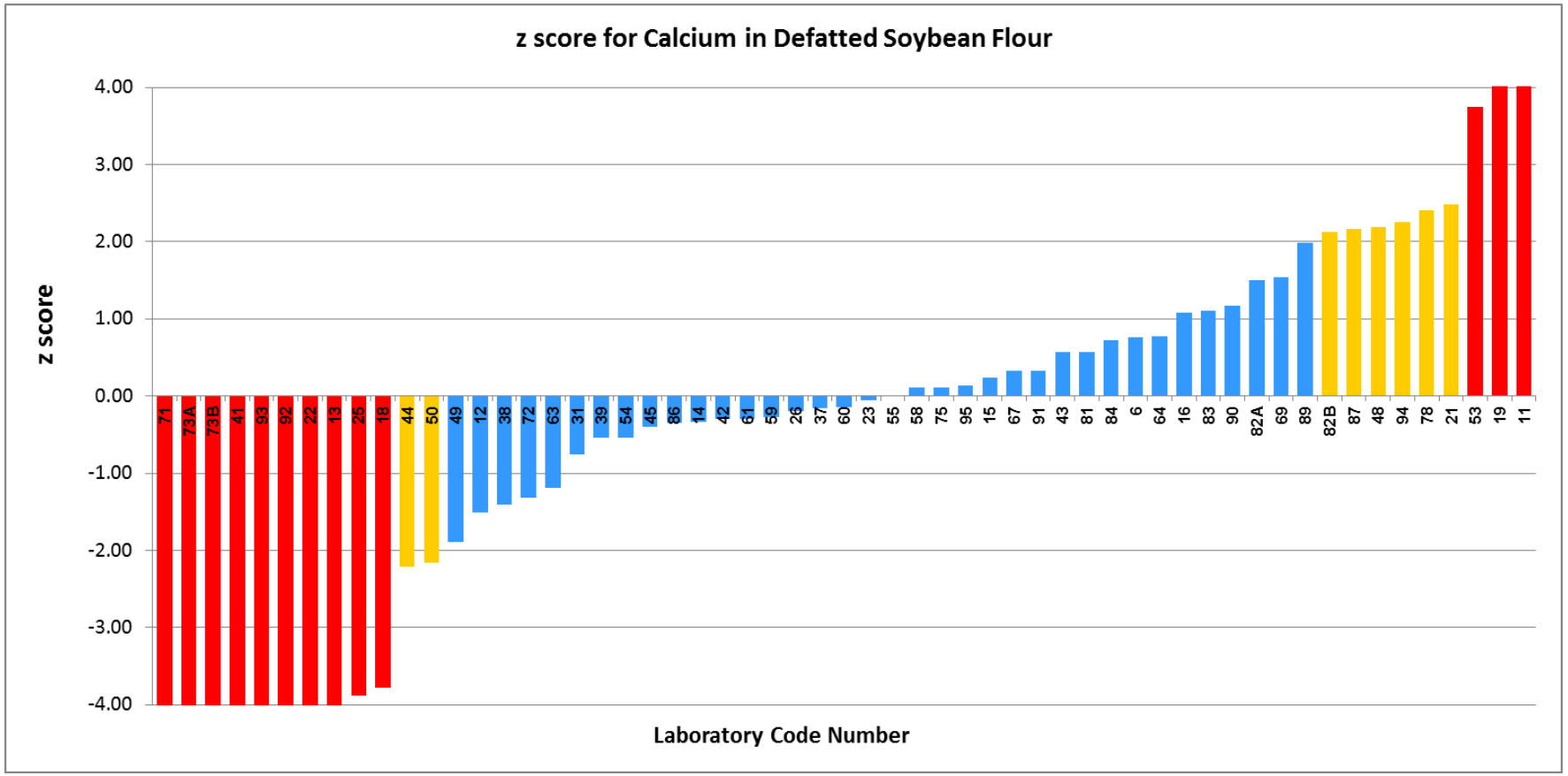

Figure 19. Plot of ordered *z* scores for **calcium** results in defatted soybean flour

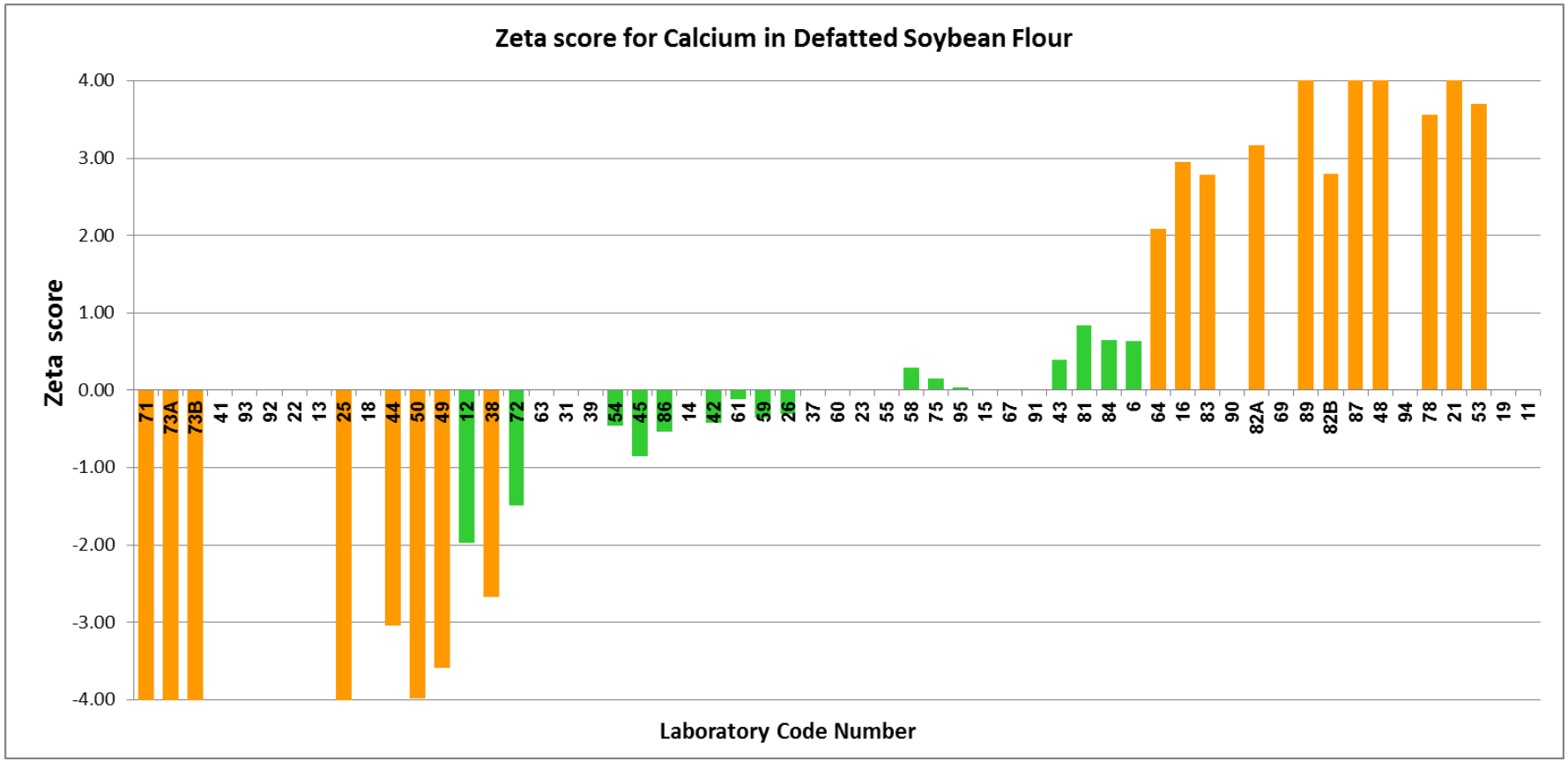

Figure 20. Plot of *Zeta* score for **calcium** in defatted soybean flour, following the ordered z scores in the above Figure 19

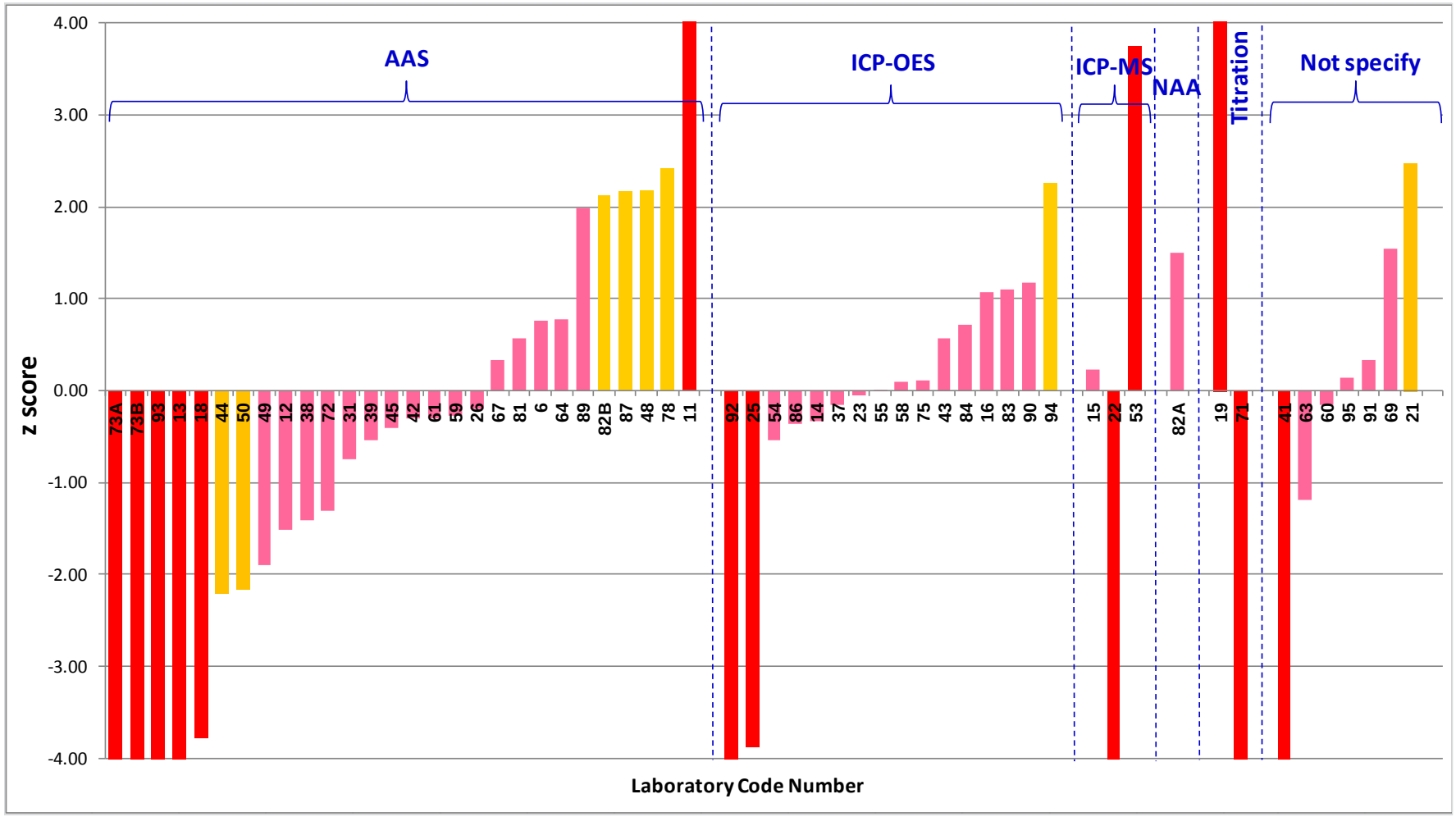

Figure 21. Plot of ordered *z* score for **calcium** in defatted soybean flour, categorised in groups according to analytical methods/parameters used

Table 10. Evaluation of laboratory performance **magnesium** analysis (mg/kg, as received) in defatted soybean flour

Lab Number	Mg (mg/kg)	MU (mg/kg)	Based on reference values[1] z score	Based on reference values[1] Zeta score	Based on x* ± S*[2] z score	Based on x* ± S*[2] Zeta score	Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm or mass)	Recovery Correction (Y/N)	Method Reference
[1]Assigned value obtained from reference values (Gravimetric standard addition ICP-MS ± 2SD$_p$ = 2650 ± 259 mg/kg (CV 9.8%) with u_{xpt} = 38 mg/kg; [2]Assigned value obtained from robust average (x) ± robust SD (s*)= 2652 ± 343 mg/kg (CV 12.9%, n= 47) with u_{xpt} = 63 mg/kg*													
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00	|z score| ≤ 2.00	|ζ score| ≤ 2.00							
6	**2715**	-	0.25	-	0.18	-	2.0000	Acid	HCl:HNO_3: H_2O	AAS	-	Y	AOAC (2016, 20th Ed, 928.08, 985.35 (50.1.14)
11	**2348**	-	-1.16	-	-0.89	-	2.0000	Dry Ashing	HCl:H_2O	AAS	-	Y	AOAC (2016), 975.03, 985.35
12	**3750**	121	**4.25**	**15.42**	**3.20**	**12.63**	0.5	Closed vessel	HNO_3	Fllame AAS	-	N	AOAC (2016), 985.35
13	**1800**	-	**-3.28**	-	**-2.48**	-	0.5	Microwave	HNO_3 10 mL + HCl 2 mL	Analytikal Jena ContrAA 800 D	Mg 285	N	Internal Method
14	**2568**	-	-0.32	-	-0.24	-	0.5	Ashing	50% HNO_3, 50% HCl	ICP Horiba Jobin Yvon	Mg 279.553	Y	AOAC 975.03, 984.27
15	**2540**	-	-0.42	-	-0.33	-	0.5	Ultrawave Digestion	5% HNO_3 + 0.5% HCl	ICP-MS (7900 Agilent)	Mg 24	N	Based on USFDA 4.7 version 1.1
16	**2933**	30	1.09	**6.96**	0.82	**4.37**	0.5	Hot plate	HNO_3+H_2O_2	ICP-OES Optima 7000 DV Perkin Elmer	Mg 279.077	N	In-house Method
18	**2500**	-	-0.58	-	-0.44	-	2.0	Dry Ashing	HNO_3	AAS, Varian	Various	N	AOAC 968.08
21	**2673**	-	0.09	-	0.06	-	0.1	Microwave	180°C	Mar Xpress (CEM)	-	Y	AOAC 2011.14 (2016)
22	**3259**	-	**2.35**	-	1.77	-	0.2 to 0.3	Microwave	HNO_3	ICP-MS Perkin Elmer	-	-	AOAC 2015.06
23	**2620**	-	-0.12	-	-0.09	-	1.00	Dry Ashing	-	ICP-OES	589, 766, 422, 285, 238	-	AOAC 985.01
25	**1800**	0.07	**-3.28**	**-22.49**	**-2.48**	**-13.64**	5.0205 / 5.0206	Wet Digestion	HNO_3-HCl	ICP-OES	Mg 280.27		USEPA Method 3050B
26	**2550**	128	-0.39	-1.35	-0.30	-1.14	4.0	Dry ashing	Water & HCl (1+1)	AAS Shimadzu AA-7000	Mg 285.2	N	AOAC No. 975.03

Lab Number	Mg (mg/kg)	MU (mg/kg)	Based on reference values[1]		Based on x* ± S*[2]		Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm or mass)	Recovery Correction (Y/N)	Method Reference
			z score	Zeta score	z score	Zeta score							
[1]Assigned value obtained from reference values (Gravimetric standard addition ICP-MS ± 2SD$_p$ = 2650 ± 259 mg/kg (CV 9.8%) with u_{xpt} = 38 mg/kg; [2]Assigned value obtained from robust average (x) ± robust SD (s*)= 2652 ± 343 mg/kg (CV 12.9%, n= 47) with u_{xpt} = 63 mg/kg*													
31	**2337**	-	-1.21	-	-0.92	-	5	Dry Ashing	-	AAS, Agilent	-	N	AOAC 985.35
37	**2577**	-	-0.28	-	-0.22	-	1	Wet Digestion	Nitric + perchloric	ICP-OES (Perkin Elmer Optima 8000)	Mg 285.213	N	AOAC (2016) 984.27
38	**2040**	94	**-2.35**	**-10.13**	-1.78	**-7.83**	1.000	Dry Ashing	1N HNO_3 (0.1M HNO_3 for Fe)	Flame AAS, Shimadzu AA6300	Mg 285.20	-	AOAC 985.35, 19th Ed 2012 (Fe modified AOAC 999.11)
39	**2500**	-	-0.58	-	-0.44	-	0.5	Microwave	-	AAS	Mg 285.2	Y	AOAC 985.35
42	**2890**	85	0.93	**4.22**	0.69	**3.15**	5	Dry Ashing	HNO_3-HCl	Flame AAS, Agilent 280 FS	Mg 202.6	N	AOAC 985.35.2005
43	**2746**	200	0.37	0.90	0.27	0.80	0.5	Microwave	HNO_3	ICP-OES	Mg 285.213	N	AOAC
45	**2705**	107	0.21	0.83	0.15	0.64	4	Dry Ashing	HCl+HNO_3+DI (2+2+70 mL) on hotplate	AAS (Flame, Varian)	Mg 285.2	N	AOAC 968.08
48	**4126**	62	**5.70**	**30.24**	**4.30**	**21.15**	5	Dry Digestion		AA800 Perkin Elmer	Mg 285.2	N	MU-03/21 (AAS)
49	**2700**	135	0.19	0.65	0.14	0.52	1, 3	Dry Ashing	Conc Nitric acid	AAS / AA-7000 Shimadzu	Mg 285.2	N	AOAC 20th Ed 2016
50	**2552**	102	-0.38	-1.54	-0.29	-1.24	2.0000	Wet	Acid	Flame AAS (Varian)	330.3, 404.4, 422.7, 248.3	N	AOAC 985.35
53	**2260**	31	-1.51	**-9.55**	-1.14	**-6.09**	0.3	Microwave	4 mL HNO_3, 1 mL HCl, 1 mL H_2O_2	ICPMS Thermo	-	-	In house method
54	**2772**	36	0.47	**2.91**	0.35	1.85	1	Dry Ashing	HNO_3	ICP / Shimadzu	Mg 279.553	N	AOAC 984.27
55	**2710**	-	0.23	-	0.17	-	1.5	Wet digestion		ICP-OES	Mg 280.270	Y	AOAC (2012) 984.27
58	**2649**	49	-0.01	-0.03	-0.01	-0.05	3.0	Dry Ash	HCl	ICP-OES	-	-	Dry Ashing and Quantitation by ICP-OES

Lab Number	Mg (mg/kg)	MU (mg/kg)	Based on reference values[1]		Based on x* ± S*[2]		Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm or mass)	Recovery Correction (Y/N)	Method Reference
			z score	Zeta score	z score	Zeta score							
[1]*Assigned value obtained from reference values (Gravimetric standard addition ICP-MS ± 2SD$_p$ = 2650 ± 259 mg/kg (CV 9.8%) with u_{xpt} = 38 mg/kg;* [2]*Assigned value obtained from robust average (x*) ± robust SD (s*)= 2652 ± 343 mg/kg (CV 12.9%, n= 47) with u_{xpt} = 63 mg/kg*													
59	**3520**	380	**3.36**	**4.49**	**2.53**	**4.33**	1.5	Dry Ashing	-	AAS, Shimadzu	Mg 285.2	Y	AOAC 18th Ed 985.35 (Fe: SNI 3751:2009 point A.10)
60	**100**	-	**-9.84**	-	**-7.44**	-	-	-	-	-	-	-	AOAC (2012) 968.08 (Ca, Mg)
61	**2480**	362	-0.66	-0.92	-0.50	-0.90	1	Acid block digestion	HNO_3 (HNO_3/$HClO_4$ for P)	Varian AA240 FS Fast Sequential AAS	Mg 285.2	N	A6407-26 AAS (A6417 Spectro Method for P)
63	**2642**	-	-0.03	-	-0.03	-	-	-	-	-	-	-	-
67	**3040**	-	1.51	-	1.13	-	2.0xxx	Dry Ash	Wet chemical	AAS, Perkin Elmer	Mg 285.21	N	AOAC 968.08
69	**2370**	-	-1.08	-	-0.82	-	-	-	-	-	-	-	-
72	**2620**	223	-0.12	-0.25	-0.09	-0.25	3	Ashing	HNO3	AAS / Analytik Jena	Mg 285.2	N	AOAC 985.35
75	**4619**	112	**7.60**	**29.16**	**5.73**	**23.45**	1	Wet digestion (hot block)	HNO3 + H2O2	ICP-OES Agilent 5100	Mg 279.078	N	In House Method ICP-OES
78	**2785**	-	0.52	-	0.39	-	0.5	Mircowave Digestion	Acid Digestion	Berghof Speedwave 4 Microwave Digestion	Mg 279.08	-	MP-AES
82A	**2290**	84	-1.39	**-6.37**	-1.06	**-4.81**	0.250	none	none	HPGe detector, Canberra	-	-	Neutron Activation Analysis (NAA)
83	**2830**	34	0.69	**4.34**	0.52	**2.75**	0.3	Microwave Digestion with HNO3	Microwave digester Mars Xpress	ICP MS Nex Ion (Perkin Elmer)	-	Y	Application Note, Perkin Elmer
84	**2910**	290	1.00	1.74	0.75	1.63	0.5	Microwave Digestion	HNO3 / H2O2	ICP-OES, ICP-MS	Mg 285.213	N	AOAC 999.10:2005
86	**2584**	152	-0.25	-0.78	-0.20	-0.69	1.0000	Wet Digest	-	ICP-OES	Mg 280.2	Y	AOAC (2012) 984.27

Lab Number	Mg (mg/kg)	MU (mg/kg)	Based on reference values[1]		Based on x* ± S*[2]		Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm or mass)	Recovery Correction (Y/N)	Method Reference
			z score	Zeta score	z score	Zeta score							
[1]Assigned value obtained from reference values (Gravimetric standard addition ICP-MS ± 2SD$_p$ = 2650 ± 259 mg/kg (CV 9.8%) with u_{xpt} = 38 mg/kg; [2]Assigned value obtained from robust average (x) ± robust SD (s*)= 2652 ± 343 mg/kg (CV 12.9%, n= 47) with u_{xpt} = 63 mg/kg*													
87	**3077**	8	1.65	**11.24**	1.24	**6.79**	2.5	Dry Ashing	HNO_3	Furnace Thermolyne	ICP-OES	N	MTD/FOD/CHM-09
89	**2412**	36	-0.92	**-5.68**	-0.70	**-3.69**	2	Dry Ashing	1.5% HNO_3	AAS Agilent	Various	N	AOAC 985.35
90	**2746**	-	0.37	-	0.27	-	1	Ultrawave	-	ICP-OES	Mg 285.213	-	-
91	**2640**	-	-0.04	-	-0.03	-	-	-	-	-	-	-	-
92	**300**	-	**-9.07**	-	**-6.86**	-	1	Ashing	HNO_3	ICP-OES	-	-	-
94	**3057**	-	1.57	-	1.18	-	1.5	Dry ashing	-	ICP-OES / Perkin Elmer	Mg 383.2	Y	AOAC (2012) 984.27
95	**2810**	310	0.62	1.00	0.46	0.95	-	-	-	-	-	-	-

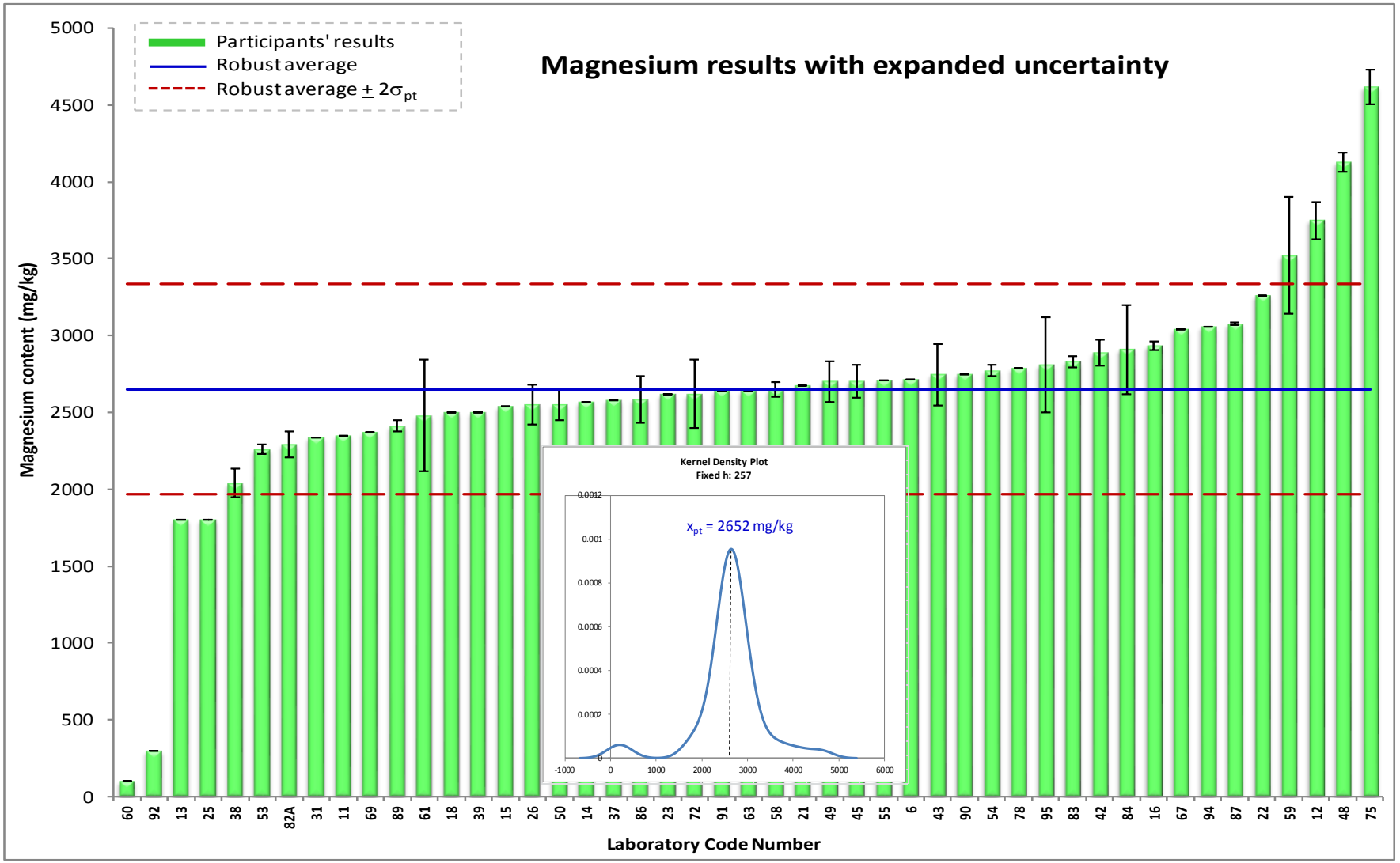

Figure 22. Distribution of **magnesium** results (ascending order) in defatted soybean flour with expanded uncertainty

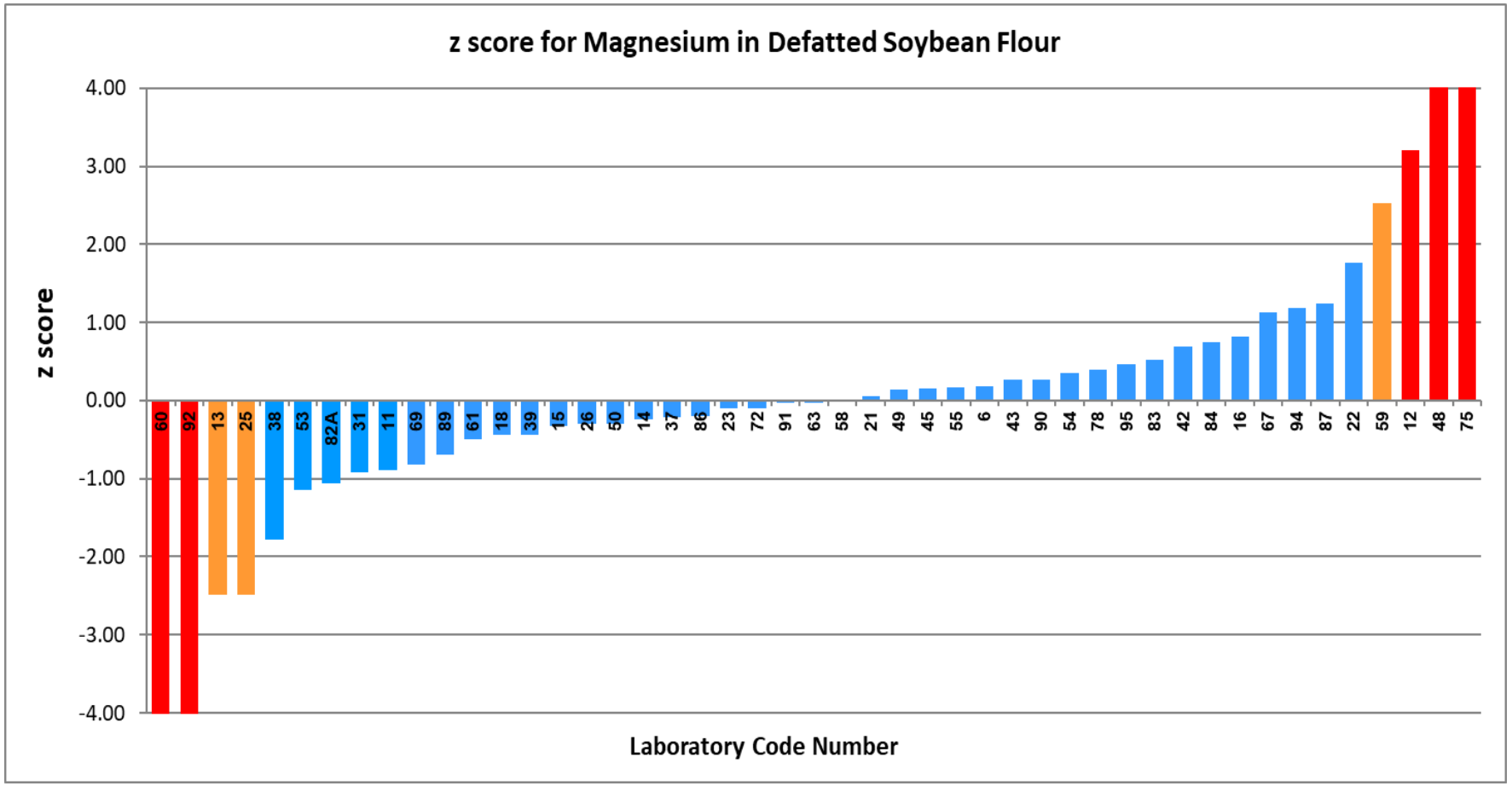

Figure 23. Plot of ordered *z* scores for **magnesium** results in defatted soybean flour

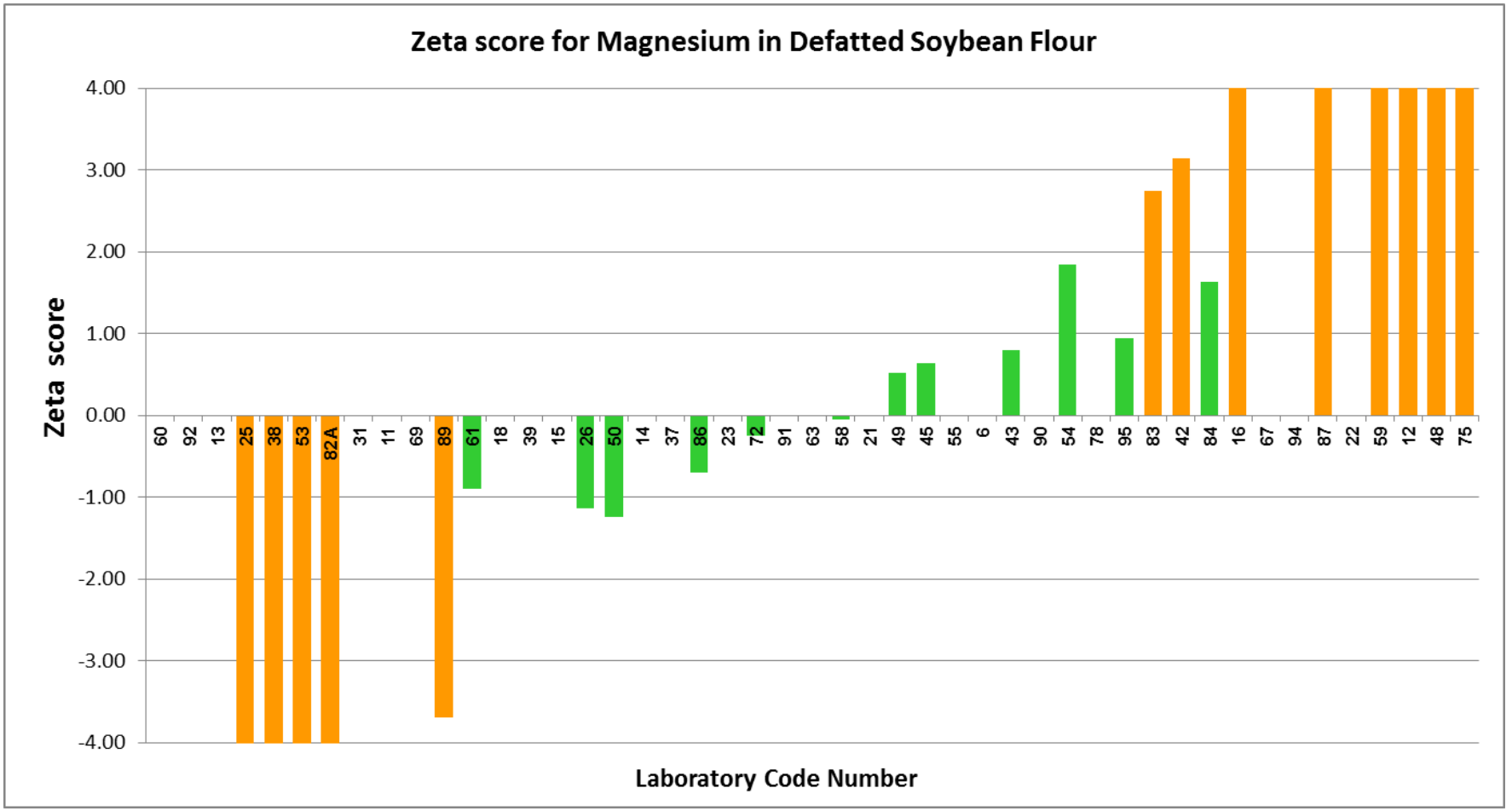

Figure 24. Plot of *Zeta* score for **magnesium** in defatted soybean flour, following the ordered z scores in the above Figure 23

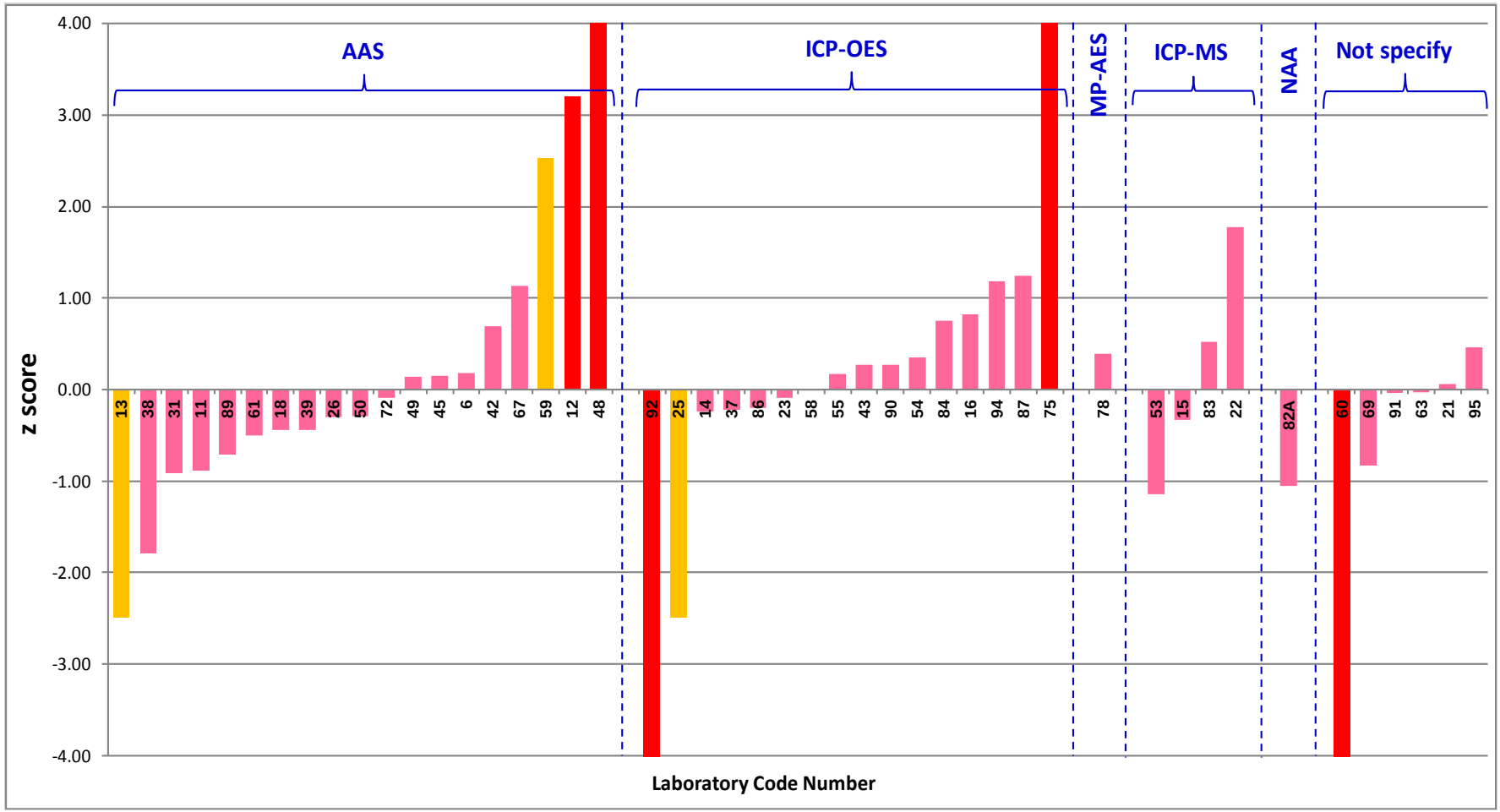

Figure 25. Plot of ordered *z* score for **magnesium** in defatted soybean flour, categorised in groups according to analytical methods/parameters used

Table 11. Evaluation of laboratory performance **phosphorus** analysis (mg/kg, as received) in defatted soybean flour

Laboratory Number	P mg/kg	MU mg/kg	z score (based on x* ± s*)	Zeta score	Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm or mass)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± robust SD (s*) = 7787 ± 456 mg/kg (CV 5.9%, n= 33) with u_{xpt} = 99 mg/kg*											
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00							
2	**0.8**	-	**-17.08**	-	-	-	-	-	-	-	-
6	**7564**	231	-0.49	-1.46	2.0000	Acid	HCl:HNO_3:H_2O	AAS	-	Y	AOAC (2016, 20th Ed, 928.08, 985.35 (50.1.14)
11	**7620**	-	-0.37	-	2.0000	Dry Ashing	HCl:H_2O	AAS	-	Y	AOAC (2016), 975.03, 985.35
12	**8180**	-	0.86	-	0.5	Closed vessel	HNO_3	Fllame AAS	-	N	AOAC (2016), 985.35
14	**7959**	-	0.38	-	0.5	Ashing	50% HNO_3, 50% HCl	ICP Horiba Jobin Yvon	P 213.618	Y	AOAC 975.03, 984.27
16	**7895**	78	0.24	1.01	0.5	Hot plate	HNO_3+H_2O_2	ICP-OES Optima 7000 DV Perkin Elmer	-	N	In-house Method
18	**7740**	-	-0.10	-	2.0	Dry Ashing	HNO_3	AAS, Varian	Various	N	AOAC 968.08
19	**7470**	-	-0.70	-	1	Furnace	HNO_3:H_2O (1:1)	Ca Manual by Buret, P by UV-Vis Spectro.	P 400	N	AOAC 927.02, 944.03, 965.17
21	**52**	-	**-16.96**	-	0.1	Microwave	180°C	Mar Xpress (CEM)	-	Y	AOAC 2011.14 (2016)
22	**741**	-	**-15.45**	-	0.2 to 0.3	Microwave	HNO_3	ICP-MS Perkin Elmer	-	-	AOAC 2015.06
23	**8070**	-	0.62	-	1.00	Dry Ashing	-	ICP-OES	589, 766, 422, 285, 238	-	AOAC 985.01
25	**6150**	-	**-3.59**	-	5.0205 / 5.0206	Wet Digestion	HNO_3-HCl	ICP-OES	-	-	USEPA Method 3050B
31	**7035**	571	-1.65	**-2.49**	5	Dry Ashing	-	AAS, Agilent	-	N	AOAC 985.35
37	**6985**	-	-1.76	-	1	Wet Digestion	Nitric + perchloric	ICP-OES (Perkin Elmer Optima 8000)	-	N	AOAC (2016) 984.27

Laboratory Number	**P** (mg/kg)	**MU** (mg/kg)	**z score** (based on x* ± s*)	**Zeta score**	Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm or mass)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± robust SD (s*) = 7787 ± 456 mg/kg (CV 5.9%, n= 33) with u_{xpt} = 99 mg/kg*											
38	**7970**	17	0.40	1.84	1.000	Dry Ashing	1N HNO_3 (0.1M HNO_3 for Fe)	Flame AAS, Shimadzu AA6300	-	-	AOAC 985.35, 19th Ed 2012 (Fe modified AOAC 999.11)
41	**0.7**	-	**-17.08**	-	2	-	-	-	-	-	-
42	**7980**	144	0.42	1.57	5	Dry Ashing	HNO_3-HCl	Flame AAS, Agilent 280 FS	-	N	AOAC 985.35.2005
43	**8492**	802	1.55	1.71	0.5	Microwave	HNO_3	ICP-OES	-	N	AOAC
45	**7870**	458	0.18	0.33	4	Dry Ashing	HCl+HNO_3+DI (2+2+70 mL) on hotplate	AAS (Flame, Varian)	-	N	AOAC 968.08
48	**7399**	213	-0.85	**-2.67**	5	Dry Digestion		AA800 Perkin Elmer	-	N	MU-03/21 (AAS)
49	**8310**	416	1.15	**2.27**	1, 3	Dry Ashing	Conc Nitric acid	AAS / AA-7000 Shimadzu	-	N	AOAC 20th Ed 2016
54	**7624**	153	-0.36	-1.30	1	Dry Ashing	HNO_3	ICP / Shimadzu	-	N	AOAC 984.27
55	**8026**	-	0.52	-	1.5	Wet digestion		ICP-OES	-	Y	AOAC (2012) 984.27
58	**8016**	131	0.50	1.93	3.0	Dry Ash	HCl	ICP-OES	-		Dry Ashing and Quantitation by ICP-OES
59	**7639**	304	-0.32	-0.82	1.5	Dry Ashing	-	AAS, Shimadzu	-	Y	AOAC 18th Ed 985.35 (Fe: SNI 3751:2009 point A.10)
60	**7300**	-	-1.07	-	-	-	-	-	-	-	AOAC (2012), 965.17 (P)
61	**7770**	1080	-0.04	-0.03	1	Acid block digestion	HNO_3 (HNO_3/$HCLO_4$ for P)	Varian AA240 FS Fast Sequential AAS (Shimadzu UV-2700 for P)	-	N	A6417 Spectro Method for P
67	**7300**	1100	-1.07	-0.87	2.0xxx	Dry Ash	Wet chemical	AAS, Perkin Elmer	-	N	AOAC 968.08

Laboratory Number	P (mg/kg)	MU (mg/kg)	z score (based on x* ± s*)	Zeta score	Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm or mass)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± robust SD (s*) = 7787 ± 456 mg/kg (CV 5.9%, n= 33) with u_{xpt} = 99 mg/kg*											
68	**7867**	-	0.18	-	-	-	-	-	-	-	-
69	**9010**	-	**2.68**	-	-	-	-	-	-	-	-
71	**0.79**	0.29	**-17.08**	**-78.44**	1.0036, 1.0063	Acid Digestion	HCl (1:3)	-	-	-	AOAC 927.02, Titration
72	**7780**	0	-0.02	-0.07	3	Ashing	HNO_3	AAS / Analytik Jena	-	N	AOAC 985.35
73A	**0.81**	0.04	**-17.07**	**-78.44**	1	Dry ashing	Hot plate	AAS (280FS AA, Agilent Technology)	-	N	FTC-46.01 (refers to AOAC 968.08, 965.09)
75	**14096**	383	**13.83**	**29.25**	1	Wet digestion (hot block)	HNO_3 + H_2O_2	ICP-OES Agilent 5100	-	N	In House Method ICP-OES
79	**54**	-	**-16.96**	-	-	-	-	-	-	-	-
80	**7600**	-	-0.41	-	-	-	-	-	-	-	-
83	**8880**	22	**2.40**	**10.95**	0.3	Microwave Digestion with HNO3	-	Microwave digester Mars Xpress, ICP MS Nex Ion (Perkin Elmer)	-	Y	Application Note, Perkin Elmer
86	**7058**	519	-1.60	**-2.62**	1.0000	Wet Digest	-	ICP-OES	-	Y	AOAC (2012) 984.27
87	**8212**	21	0.93	**4.26**	2.5	Dry Ashing	HNO_3	Furnace Thermolyne	ICP-OES	N	MTD/FOD/CHM-09
90	**7994**	-	0.45	-	1	Ultrawave	-	ICP-OES	-	-	-
92	**828**	-	**-15.26**	-	1	Ashing	HNO_3	ICP-OES	-	-	-
95	**8000**	310	0.47	1.16	-	-	-	-	-	-	-

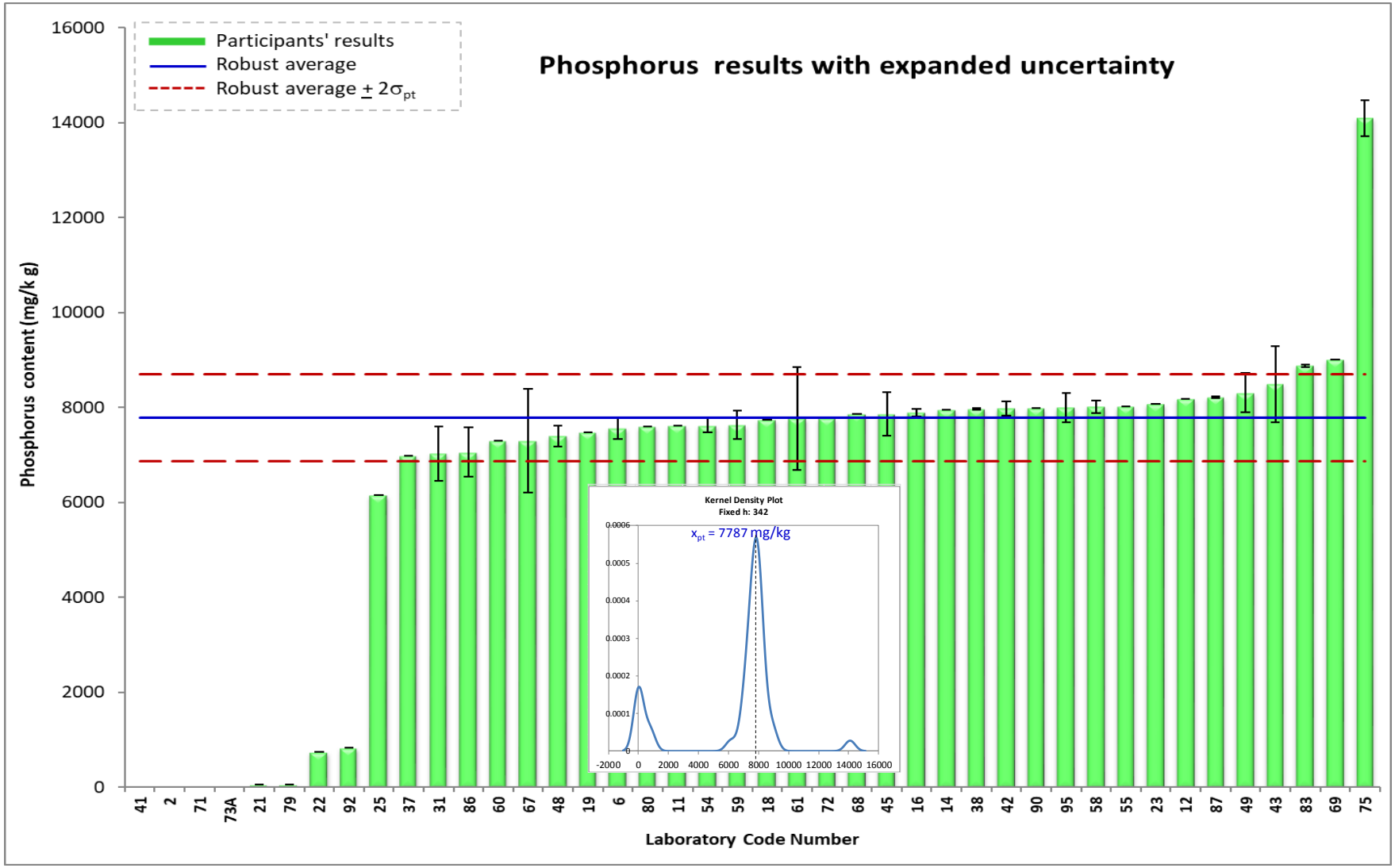

Figure 26. Distribution of **phosphorus** results (ascending order) in defatted soybean flour with expanded uncertainty

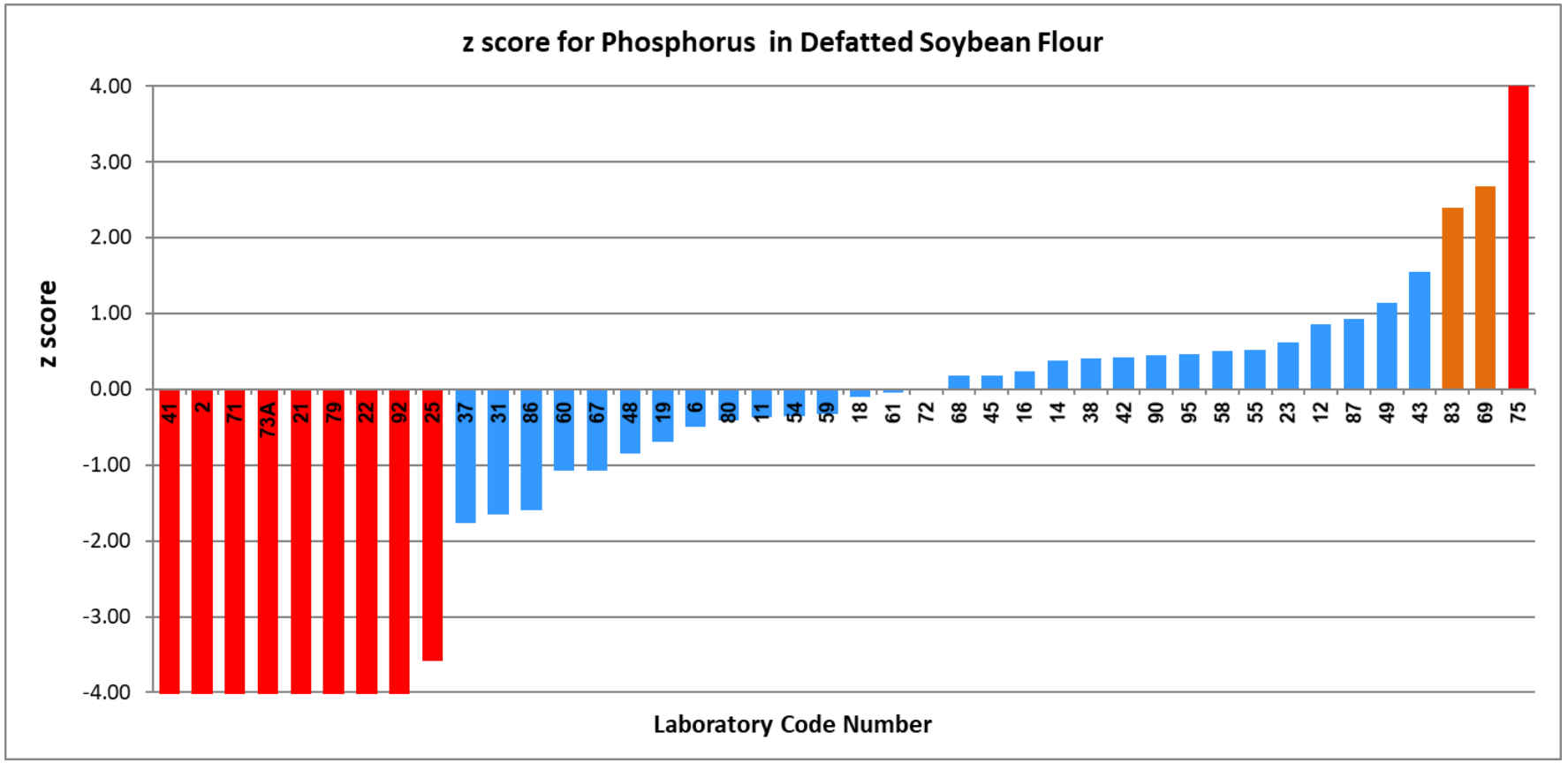

Figure 27. Plot of ordered *z* scores for **phosphorus** results in defatted soybean flour

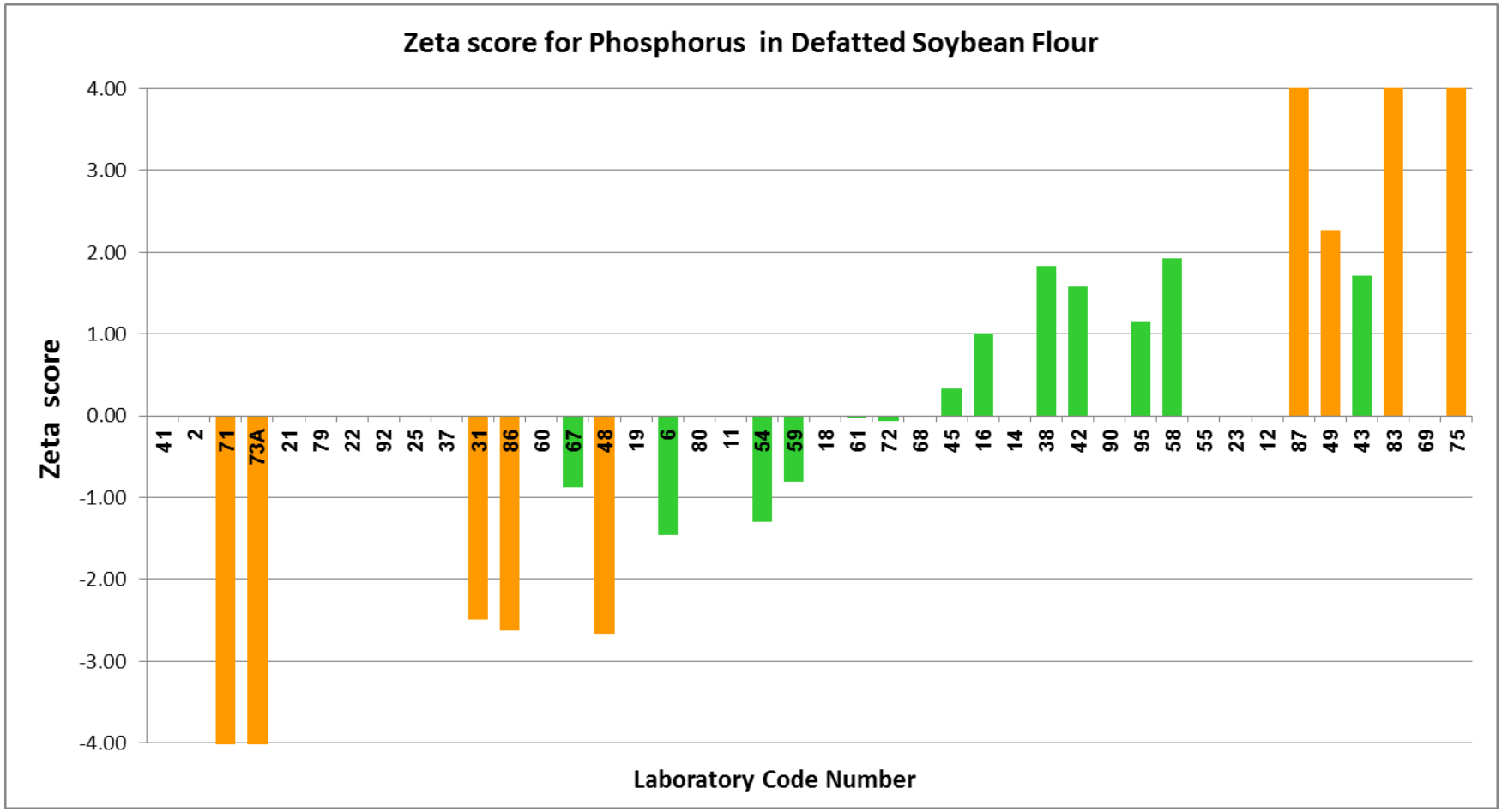

Figure 28. Plot of *Zeta* score for **phosphorus** in defatted soybean flour, following the ordered z scores in the above Figure 27

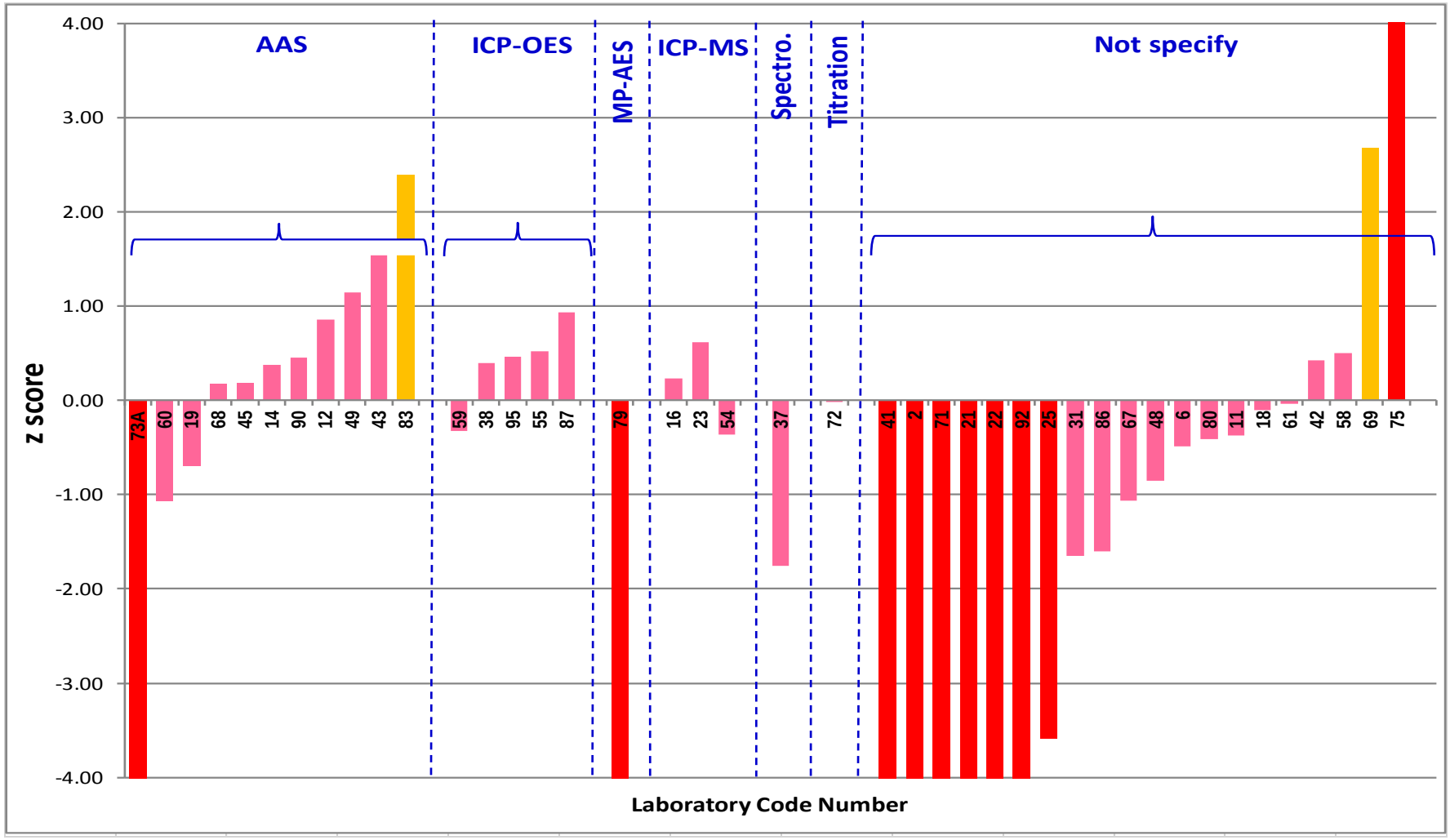

Figure 29. Plot of ordered *z* score for **phosphorus** in defatted soybean flour, categorised in groups according to analytical methods/parameters used

Table 12. Evaluation of laboratory performance **sodium** analysis (mg/kg, as received) in defatted soybean flour

Lab Number	Sodium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm or mass)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± 3SDp from Horwitz' s equation = 72.5 ± 18.3 mg/kg (CV 25.2%, n= 42) with u_{xpt} = 4.0 mg/kg*											
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00							
6	**53.3**	9.14	-1.05	**-3.16**	2.0000	Acid	$HCl:HNO_3:H_2O$	AAS	Na 589.0	Y	AOAC (2016, 20th Ed, 928.08, 985.35 (50.1.14)
11	**406.5**	-	**18.25**	-	2.0000	Dry Ashing	$HCl:H_2O$	AAS	Na 330.3	Y	AOAC (2016), 975.03, 985.35
12	**287.0**	-	**11.72**	-	0.5	Closed vessel	HNO_3	Flame AAS	Na 589.0	N	AOAC (2016), 985.35
13	**15.6**	-	**-3.11**	-	0.5	Microwave	HNO_3 10 mL + HCl 2 mL	Analytikal Jena ContrAA 800 D	Na 588	N	Internal Method
14	**38.9**	-	-1.84	-	0.5	Ashing	50% HNO_3, 50% HCl	ICP Horiba Jobin Yvon	Na 588.995	Y	AOAC 975.03, 984.27
16	**40.0**	2.00	-1.78	**-7.92**	0.5	Hot plate	$HNO_3+H_2O_2$	ICP-OES Optima 7000 DV Perkin Elmer	Na 588.995	N	In-house Method
18	**148.0**	-	**4.13**	-	2.0	Dry Ashing	HNO_3	AAS, Varian	Various	N	AOAC 968.08
21	**819.0**	2.11	**40.79**	**181.28**	0.1	Microwave	180°C	Mar Xpress (CEM)	-	Y	AOAC 2011.14 (2016)
22	**51.2**	-	-1.16	-	0.2 to 0.3	Microwave	HNO_3	ICP-MS Perkin Elmer	-	-	AOAC 2015.06
23	**< LOD**	-	-	-	1.00	Dry Ashing	-	ICP-OES	589	-	AOAC 985.01
25	**119.0**	0.07	**2.54**	**11.68**	5.0205 / 5.0206	Wet Digestion	HNO_3-HCl	ICP-OES	Na 588.995	-	USEPA Method 3050B
26	**392.0**	20.30	**17.46**	**29.31**	4.0	Dry ashing	Water & HCl (1+1)	AAS Shimadzu AA-7000	Na 589.0	N	AOAC No. 975.03
31	**551.5**	-	**26.18**	-	5	Dry Ashing	-	AAS, Agilent		N	AOAC 985.35

Lab Number	Sodium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm or mass)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± 3SDp from Horwitz' s equation = 72.5 ± 18.3 mg/kg (CV 25.2%, n= 42) with u_{xpt} = 4.0 mg/kg*											
37	**33.2**	-	**-2.15**	-	1	Wet Digestion	Nitric + perchloric	ICP-OES (Perkin Elmer Optima 8000)	Na 589.592	N	AOAC (2016) 984.27
38	**303.0**	43.60	**12.60**	**10.40**	1.000	Dry Ashing	1N HNO_3 (0.1M HNO_3 for Fe)	Flame AAS, Shimadzu AA6300	Na 589.0	-	AOAC 985.35, 19th Ed 2012 (Fe modified AOAC 999.11)
39	**30.6**	-	**-2.29**	-	0.5	Microwave	-	AAS	Na 589.0	Y	AOAC 985.35
42	**157.0**	6.38	**4.62**	**16.57**	5	Dry Ashing	HNO_3-HCl	Flame AAS, Agilent 280 FS	Na 589.9	N	AOAC 985.35.2005
43	**6.6**	0.02	**-3.60**	**-16.55**	0.5	Microwave	HNO_3	ICP-OES	Na 568.821	N	AOAC
45	**58.5**	4.35	-0.77	**-3.09**	4	Dry Ashing	HCl+HNO_3+DI (2+2+70 mL) on hotplate	AAS (Flame, Varian)	Na 589.0	N	AOAC 968.08
48	**328.7**	6.96	**14.00**	**48.47**	5	Dry Digestion	-	AA800 Perkin Elmer	Na 330.2	N	MU-03/21 (AAS)
49	**42.4**	2.10	-1.64	**-7.31**	1, 3	Dry Ashing	Conc Nitric acid	AAS / AA-7000 Shimadzu	Na 589.0	N	AOAC 20th Ed 2016
50	**153.0**	35.10	**4.40**	**4.47**	2.0000	Wet	Acid	Flame AAS (Varian)	330.3, 404.4, 422.7, 248.3	N	AOAC 985.35
55	**35.1**	-	**-2.04**	-	1.5	Wet digestion	-	ICP-OES	Na 589.592	Y	AOAC (2012) 984.27
58	**10.2**	2.75	**-3.40**	**-14.80**	3.0	Dry Ash	HCl	ICP-OES	-	-	Dry Ashing and Quantitation by ICP-OES
59	**415.6**	171.66	**18.75**	**3.99**	1.5	Dry Ashing	-	AAS, Shimadzu	Na 589	Y	AOAC 18th Ed 985.35
60	**54.0**	-	-1.01	-	-	-	-	-	-	-	MP37-BPMSP (AAS) (Na, K), SNI 01-2896-1998 (Fe)
61	**< 10.0**	1.38	-	-	1	Acid block digestion	HNO_3	Varian AA240 FS Fast Sequential AAS	Na 589.6	N	A6407-26 AAS

Lab Number	Sodium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm or mass)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± 3SDp from Horwitz' s equation = 72.5 ± 18.3 mg/kg (CV 25.2%, n= 42) with u_{xpt} = 4.0 mg/kg*											
63	**57.1**	-	-0.84	-	-	-	-	-	-	-	-
67	**< 100**	-	-	-	2.0xxx	Dry Ash	Wet chemical	AAS, Perkin Elmer	Na 589.00	N	AOAC 968.08
69	**29.8**	-	**-2.33**	-	-	-	-	-	-	-	-
72	**286.0**	26.10	**11.67**	**15.65**	3	Ashing	HNO_3	AAS / Analytik Jena	Na 589.0	N	AOAC 985.35
75	**78.8**	3.09	0.35	1.48	1	Wet digestion (hot block)	HNO_3 + H_2O_2	ICP-OES Agilent 5100	Na 589.592	N	In House Method ICP-OES
78	**119.0**	4.62	**2.54**	**10.10**	0.5	Mircowave Digestion	Acid Digestion	Berghof Speedwave 4	Na 589.592	-	MP-AES
81	**14.4**	2.60	**-3.17**	**-13.88**	1.0054	Wet Digestion (Na, K)	1 N HNO_3 and 30% H_2O_2 (Na, K)	Shimadzu AAS AA 6300	Na 589.0	N	AOAC 999.10 Mod (Na, K)
82A	**5.7**	0.24	**-3.65**	**-16.77**	0.250	none	none	HPGe detector, Canberra	-	-	Neutron Activation Analysis (NAA)
83	**4.3**	0.59	**-3.73**	**-17.09**	0.3	Microwave Digestion with HNO3	-	Microwave digester Mars Xpress, ICP MS Nex Ion (Perkin Elmer)	-	Y	Application Note, Perkin Elmer
84	**< 50**	-	-		0.5	Microwave Digestion	HNO_3 / H_2O_2	ICP-OES, ICP-MS	Na 589.592	N	AOAC 999.10:2005
86	**7.6**	0.78	**-3.55**	**-16.24**	1.0000	Wet Digest	-	ICP-OES	Na 589.5	Y	AOAC (2012) 984.27
87	**148.1**	0.76	**4.13**	**18.90**	2.5	Dry Ashing	HNO_3	Furnace Thermolyne	ICP-OES	N	MTD/FOD/CHM-09
89	**374.5**	5.62	**16.50**	**62.00**	2	Dry Ashing	1.5% HNO_3	AAS Agilent	Various	N	AOAC 985.35
90	**58.2**	-	-0.78	-	1	Ultrawave	-	ICP-OES	Na 589.592	-	-

Lab Number	Sodium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm or mass)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± 3SDp from Horwitz' s equation = 72.5 ± 18.3 mg/kg (CV 25.2%, n= 42) with u_{xpt} = 4.0 mg/kg*											
91	**1130.0**	14.00	**57.79**	**131.33**	-	-	-	-	-	-	-
92	**16.0**	-	**-3.09**	-	1	Ashing	HNO_3	ICP-OES	-	-	-
93	**600.0**	-	**28.83**	-	0.05	Charring, Dry ashing	Hotplate, Furnace	Flame Photometer, Sherwood	N/A	N/A	AOAC 985.35
94	**28.2**	-	**-2.42**	-	1.5	Dry ashing (Fe: Wet ashing)	-	ICP-OES / Perkin Elmer	Na 589.0	Y	AOAC (2012) 984.27
95	**320.0**	100.00	**13.52**	**4.93**	-	-	-	-	-	-	-

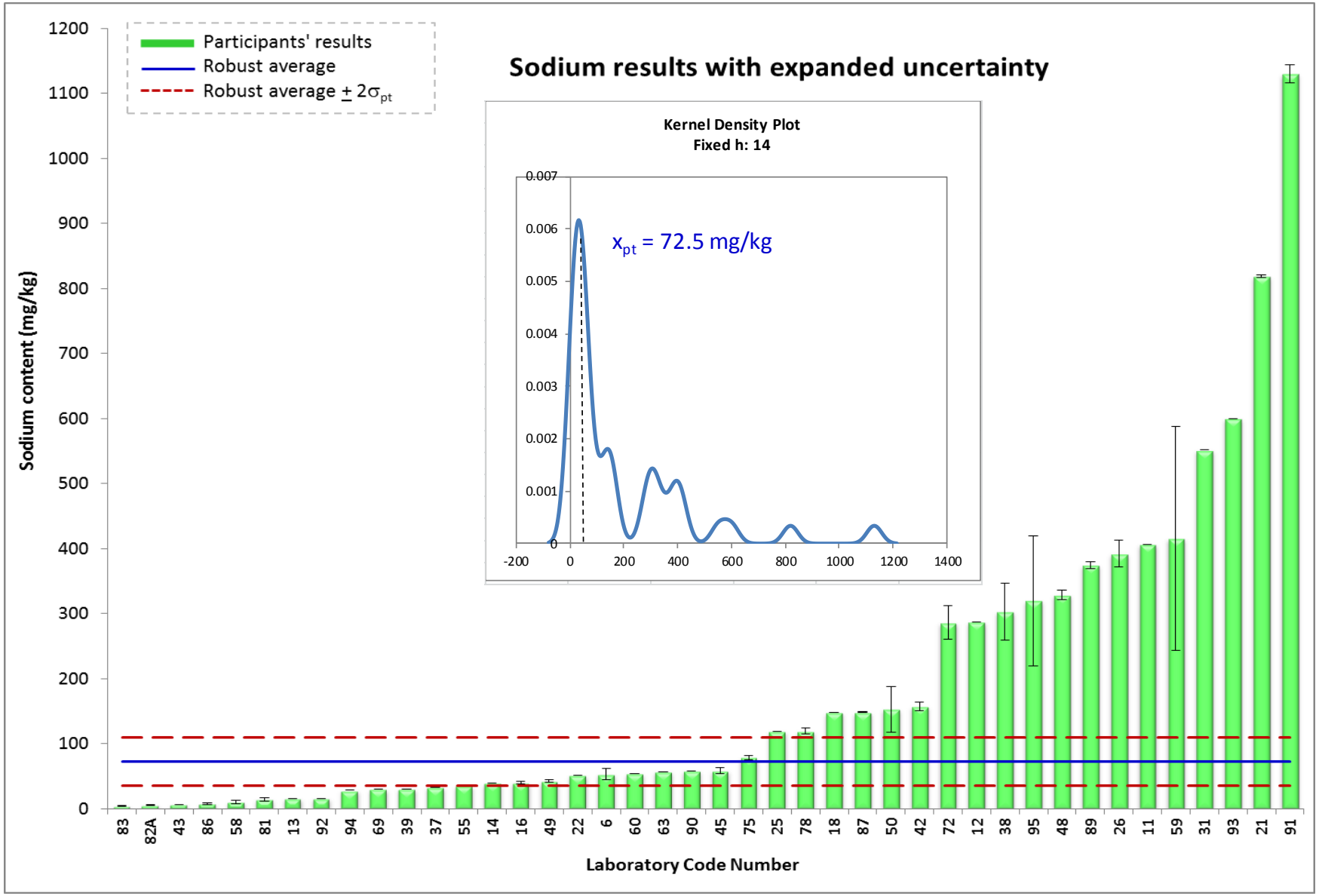

Figure 30. Distribution of **sodium** results (ascending order) in defatted soybean flour with expanded uncertainty

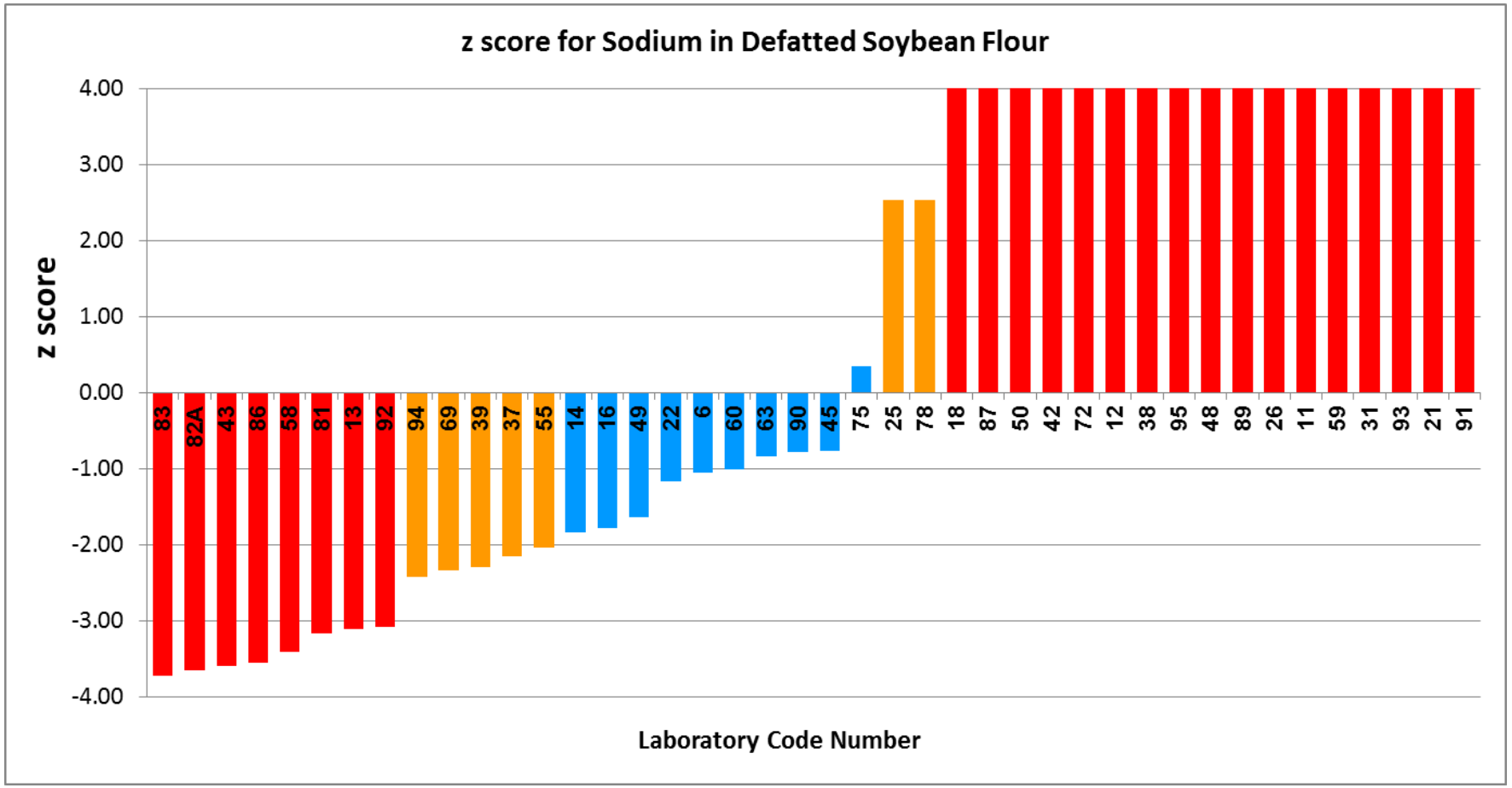

Figure 31. Plot of ordered *z* scores for **sodium** results in defatted soybean flour

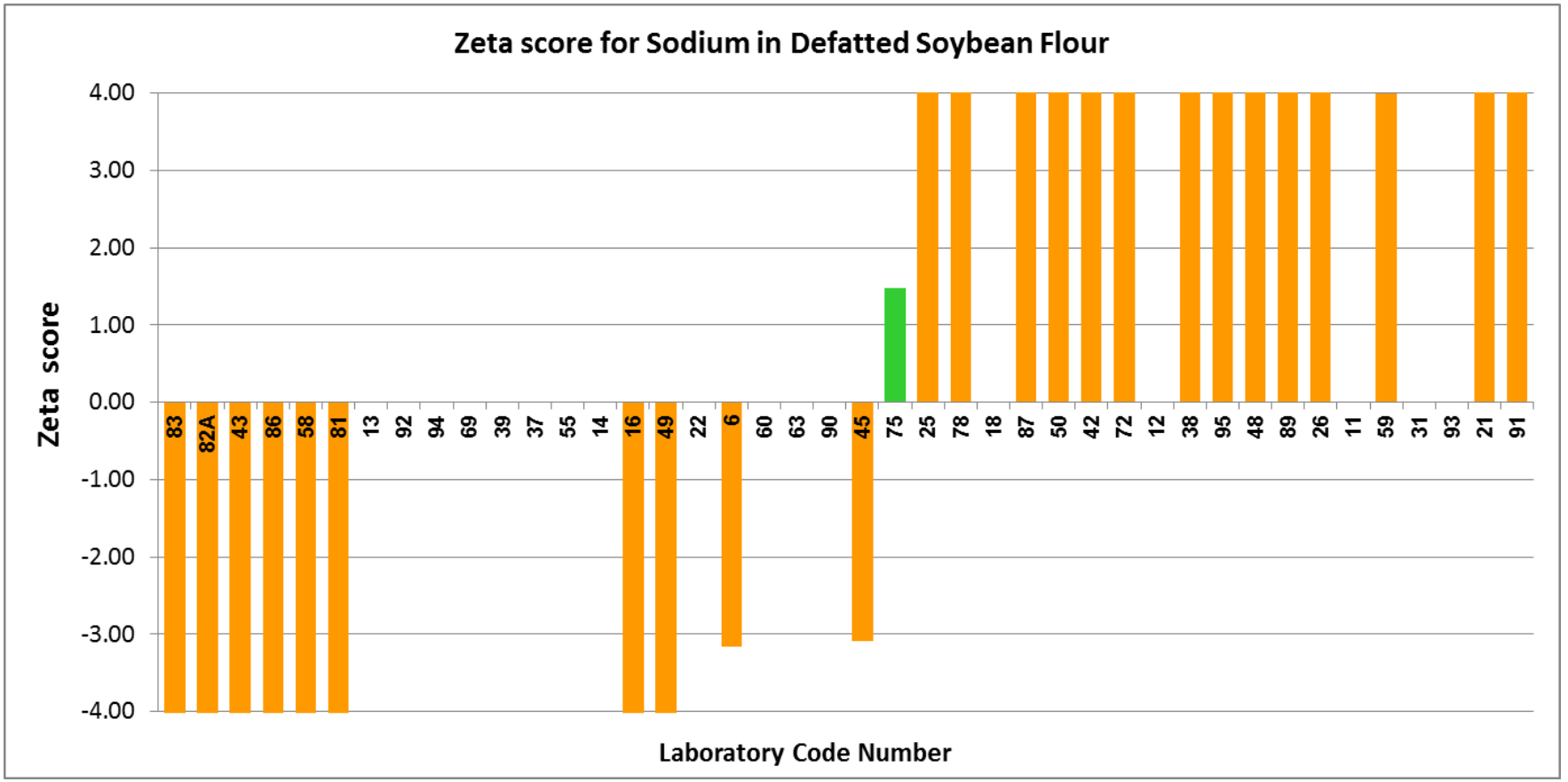

Figure 32. Plot of *Zeta* score for **sodium** in defatted soybean flour, following the ordered z scores in the above Figure 31

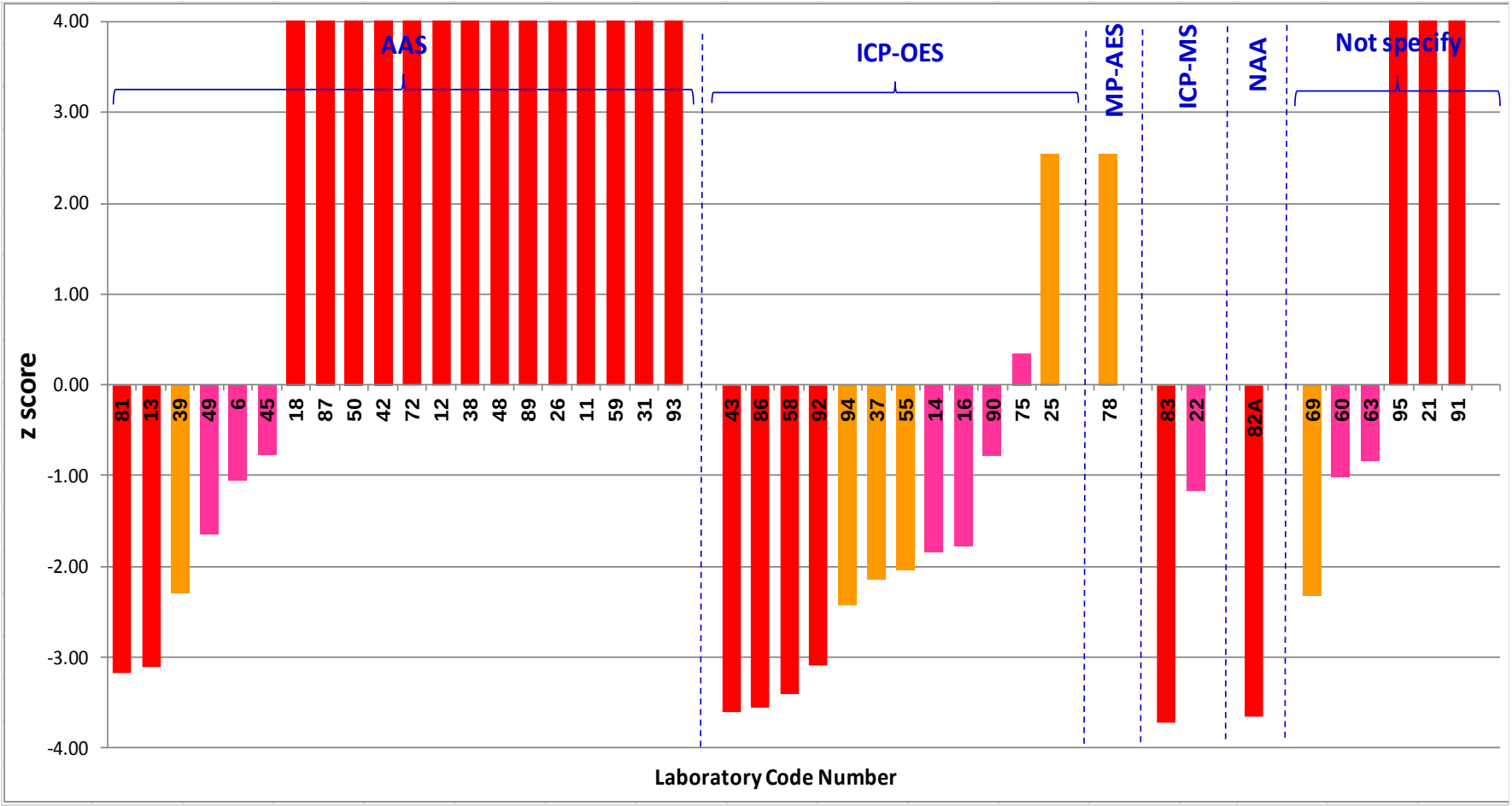

Figure 33. Plot of ordered *z* score for **sodium** in defatted soybean flour, categorised in groups according to analytical methods/parameters used

Table 13. Evaluation of laboratory performance **potassium** analysis (mg/kg, as received) in defatted soybean flour

Lab Number	Potassium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm or mass)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± 3SD from Horwitz' s equation = 23133 ± 2447 mg/kg (CV 10.6%, n= 49) with u_{xpt} = 437 mg/kg*											
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00							
6	**24048**	1826	0.37	0.90	2.0000	Acid	HCl:HNO_3:H_2O	AAS	K 766.5	Y	AOAC (2016, 20th Ed, 928.08, 985.35 (50.1.14)
11	**24082**	-	0.39	-	2.0000	Dry Ashing	HCl:H_2O	AAS	K 404.4	Y	AOAC (2016), 975.03, 985.35
12	**21460**	-	-0.68	-	0.5	Closed vessel	HNO_3	Flame AAS	K 776.5	N	AOAC (2016), 985.35
13	**4980**	-	**-7.42**	-	0.5	Microwave	HNO_3 10 mL + HCl 2 mL	Analytikal Jena ContrAA 800 D	K 766	N	Internal Method
14	**23500**	-	0.15	-	0.5	Ashing	50% HNO_3, 50% HCl	ICP Horiba Jobin Yvon	K 766.4	Y	AOAC 975.03, 984.27
15	**22900**	-	-0.10	-	0.5	Ultrawave Digestion	5% HNO_3 + 0.5% HCl	ICP-MS (7900 Agilent)	K 39	N	Based on USFDA 4.7 version 1.1
16	**26149**	260	1.23	**6.62**	0.5	Hot plate	HNO_3+H_2O_2	ICP-OES Optima 7000 DV Perkin Elmer	K 769.896	N	In-house Method
18	**24600**	-	0.60	-	2.0	Dry Ashing	HNO_3	AAS, Varian	Various	N	AOAC 968.08
21	**22835**	59	-0.12	-0.68	0.1	Microwave	180°C	Mar Xpress (CEM)	-	Y	AOAC 2011.14 (2016)
23	**24400**	-	0.52	-	1.00	Dry Ashing	-	ICP-OES	766	-	AOAC 985.01
25	**17300**	0	**-2.38**	**-13.35**	5.0205 / 5.0206	Wet Digestion	HNO_3-HCl	ICP-OES	K 766.491	-	USEPA Method 3050B
26	**21700**	1900	-0.59	-1.37	4.0	Dry ashing	Water & HCl (1+1)	AAS Shimadzu AA-7000	K 766.5	N	AOAC No. 975.03
31	**18364**	-	-1.95	-	5	Dry Ashing	-	AAS, Agilent	-	N	AOAC 985.35
32	**27953**	4195	1.97	**2.25**	1.0068	Ashing	HCl	Flame AAS, Shimadzu 6300	K: 766.5	N	Modified AOAC 969.32

Lab Number	Potassium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm or mass)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± 3SD from Horwitz' s equation = 23133 ± 2447 mg/kg (CV 10.6%, n= 49) with u_{xpt} = 437 mg/kg*											
37	**22653**	-	-0.20	-	1	Wet Digestion	Nitric + perchloric	ICP-OES (Perkin Elmer Optima 8000)	K 766.490	N	AOAC (2016) 984.27
38	**22000**	869	-0.46	-1.84	1.000	Dry Ashing	1N HNO_3	Flame AAS, Shimadzu AA6300	K 766.50	-	AOAC 985.35, 19th Ed 2012
39	**22600**	-	-0.22	-	0.5	Microwave	-	AAS	K 766.5	Y	AOAC 985.35
42	**30900**	2220	**3.17**	**6.51**	5	Dry Ashing	HNO_3-HCl	Flame AAS, Agilent 280 FS	K 769.9	N	AOAC 985.35.2005
43	**27681**	834	1.86	**7.53**	0.5	Microwave	HNO_3	ICP-OES	K 766.491	N	AOAC
44	**23106**	1178	-0.01	-0.04	1.0000	Dry Ashing	-	AAS, Thermoscientific	K 766.5	N	AOAC 19th Ed
45	**22491**	835	-0.26	-1.06	4	Dry Ashing	HCl+HNO_3+DI (2+2+70 mL) on hotplate	AAS (Flame, Varian)	K 766.5	N	AOAC 968.08
48	**22989**	128	-0.06	-0.33	5	Dry Digestion		AA800 Perkin Elmer	K 766.5	N	MU-03/21 (AAS)
49	**23600**	1180	0.19	0.64	1, 3	Dry Ashing	Conc Nitric acid	AAS / AA-7000 Shimadzu	K 766.5	N	AOAC 20th Ed 2016
50	**24189**	1371	0.43	1.30	2.0000	Wet	Acid	Flame AAS (Varian)	330.3, 404.4, 422.7	N	AOAC 985.35
54	**24000**	2000	0.35	0.79	1	Dry Ashing	HNO_3	ICP / Shimadzu	-	N	AOAC 984.27
55	**13460**	-	**-3.95**	-	1.5	Wet digestion	-	ICP-OES	K 766.491	Y	AOAC (2012) 984.27
58	**22453**	332	-0.28	-1.45	3.0	Dry Ash	HCl	ICP-OES	-	-	Dry Ashing and Quantitation by ICP-OES
59	**43695**	1394	**8.40**	**24.99**	1.5	Dry Ashing		AAS, Shimadzu	K 766.5	Y	AOAC 18th Ed 985.35 (Fe: SNI 3751:2009 point A.10)
60	**18800**	-	-1.77	-	-	-	-	-	-	-	MP37-BPMSP (AAS) (Na, K)

Lab Number	Potassium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm or mass)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± 3SD from Horwitz' s equation = 23133 ± 2447 mg/kg (CV 10.6%, n= 49) with u_{xpt} = 437 mg/kg*											
61	**20700**	7040	-0.99	-0.69	1	Acid block digestion	HNO_3	Varian AA240 FS Fast Sequential AAS	K 769.9	N	A6407-26 AAS
63	**21222**	-	-0.78	-	-	-	-	-	-	-	-
67	**19200**	-	-1.61	-	2.0xxx	Dry Ash	Wet chemical	AAS, Perkin Elmer	K 766.49	N	AOAC 968.08
69	**27000**	-	1.58	-	-	-	-	-	-	-	-
72	**29000**	2470	**2.40**	**4.48**	3	Ashing	HNO_3	AAS / Analytik Jena	K 766.5	N	AOAC 985.35
75	**36878**	1002	**5.62**	**20.68**	1	Wet digestion (hot block)	HNO_3 + H_2O_2	ICP-OES Agilent 5100	K 766.491	N	In House Method ICP-OES
78	**22672**	390	-0.19	-0.96	0.5	Mircowave Digestion	Acid Digestion	Berghof Speedwave 4 Microwave Digestion Unit	K 766.490	-	MP-AES
81	**22500**	2290	-0.26	-0.52	K 1.0034	Wet Digestion (Na, K)	1 N HNO_3 and 30% H_2O_2 (Na, K)	Shimadzu AAS AA 6300	K 766.5	N	AOAC 999.10 Mod (Na, K)
82A	**23500**	1200	0.15	0.49	0.250	none	none	HPGe detector, Canberra	-	-	Neutron Activation Analysis (NAA)
83	**23670**	335	0.22	1.15	0.3	Microwave Digestion with HNO3	-	Microwave digester Mars Xpress, ICP MS Nex Ion (Perkin Elmer)	-	Y	Application Note, Perkin Elmer
84	**26600**	2700	1.42	**2.44**	0.5	Microwave Digestion	HNO_3 / H_2O_2	ICP-OES, ICP-MS	K 766.490	N	AOAC 999.10:2005
86	**23499**	1168	0.15	0.50	1.0000	Wet Digest	-	ICP-OES	K 769.8	Y	AOAC (2012) 984.27
87	**27698**	87	1.87	**10.39**	2.5	Dry Ashing	HNO_3	Furnace Thermolyne	ICP-OES	N	MTD/FOD/CHM-09
89	**16721**	251	**-2.62**	**-14.10**	2	Dry Ashing	1.5% HNO_3	AAS Agilent	Various	N	AOAC 985.35

Lab Number	Potassium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm or mass)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± 3SD from Horwitz' s equation = 23133 ± 2447 mg/kg (CV 10.6%, n= 49) with u_{xpt} = 437 mg/kg*											
90	**17926**	-	**-2.13**	-	1	Ultrawave	-	ICP-OES	K 766.490	-	-
91	**21300**	-	-0.75	-	-	-	-	-	-	-	-
92	**1855**	-	**-8.70**	-	1	Ashing	HNO_3	ICP-OES	-	-	-
93	**23800**	-	0.27	-	0.05	Charring, Dry ashing	Hotplate, Furnace	Flame Photometer, Sherwood	N/A	N/A	AOAC 985.35
94	**29330**	-	**2.53**	-	1.5	Dry ashing (Fe: Wet ashing)	-	ICP-OES / Perkin Elmer	K 766.5	Y	AOAC (2012) 984.27
95	**22340**	2610	-0.32	-0.58	-	-	-	-	-	-	-

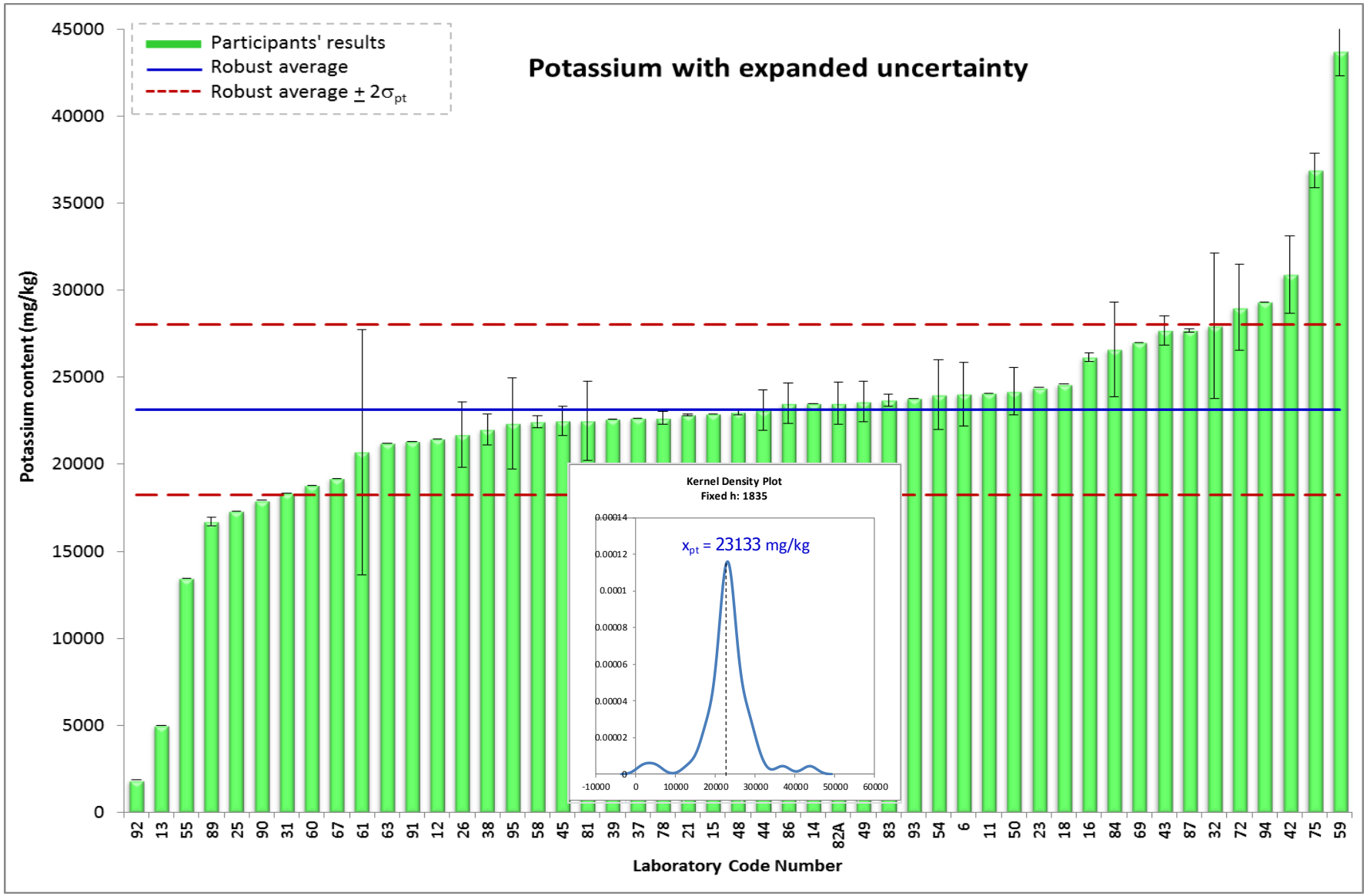

Figure 34. Distribution of **potassium** results (ascending order) in defatted soybean flour with expanded uncertainty

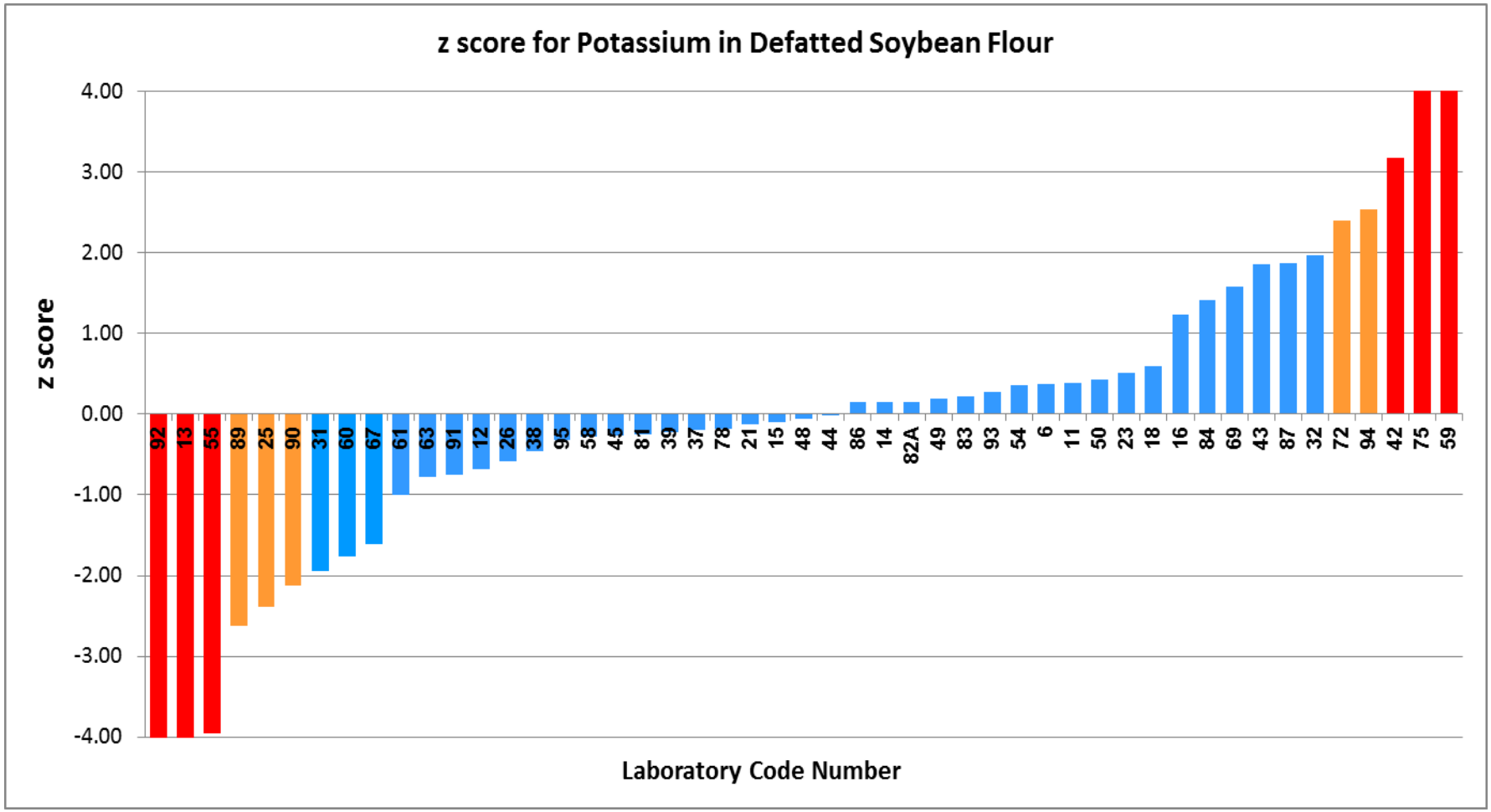

Figure 35. Plot of ordered *z* scores for **potassium** results in defatted soybean flour

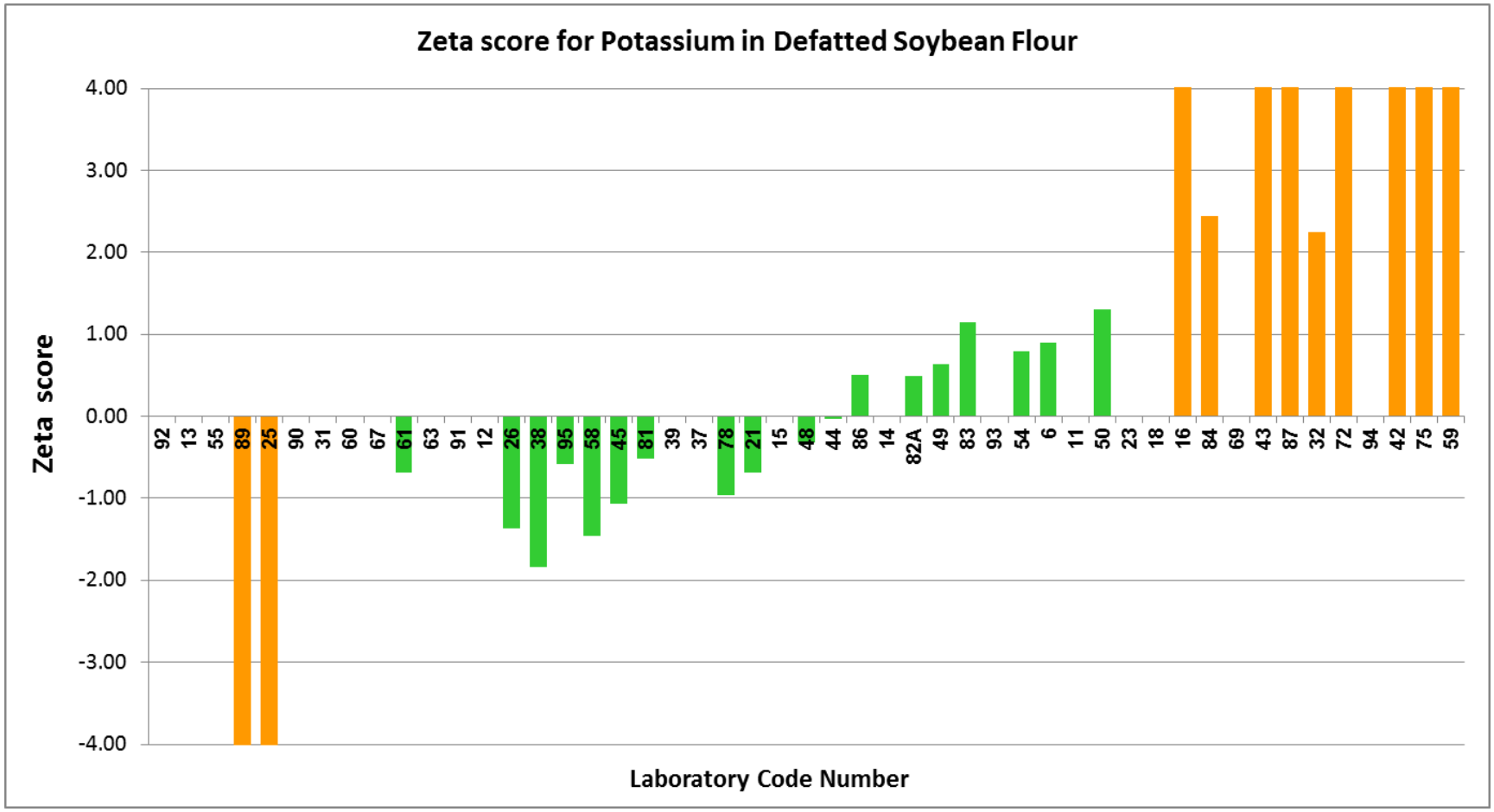

Figure 36. Plot of *Zeta* score for **potassium** in defatted soybean flour, following the ordered z scores in the above Figure 35

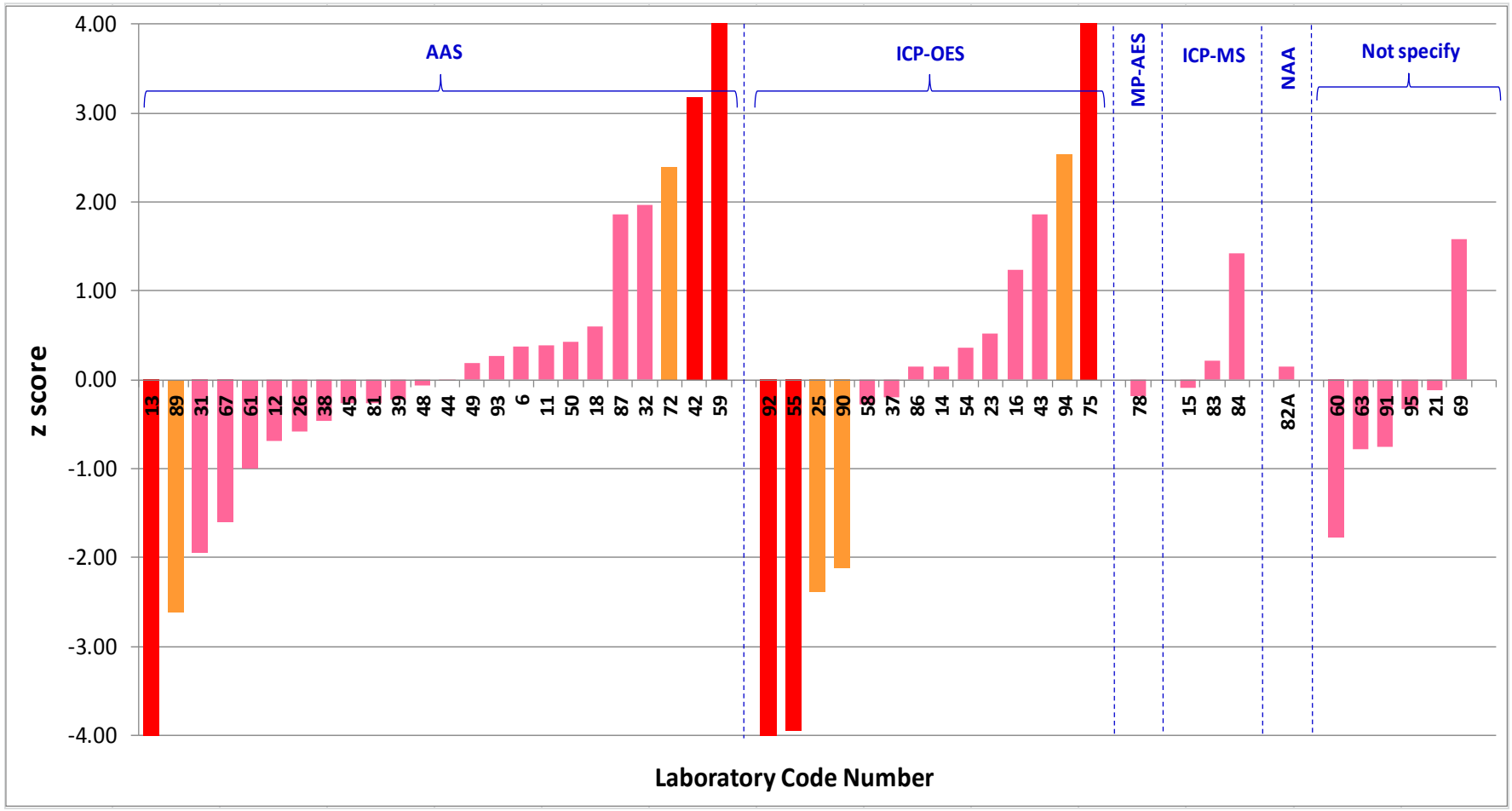

Figure 37. Plot of ordered *z* score for **potassium** in defatted soybean flour, categorised in groups according to analytical methods/parameters used

Table 14. Evaluation of laboratory performance **iron** analysis (mg/kg, as received) in defatted soybean flour

Lab Number	Iron (mg/kg)	MU (mg/kg)	Based on NIMT		Based on median ± NIQR		Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm or mass)	Recovery Correction (Y/N)	Method Reference
			z score	Zeta score	z score	Zeta score							
[1]*Assigned value obtained from reference values (Gravimetric standard addition ICP-MS as x_{pt} ± SD_p from Horwitz' s equation) = 75.1 ± 6.4 mg/kg (CV 8.5%) with u_{xpt} = 0.9 mg/kg;* [2]*Assigned value obtained from median ± normalised IQR = 75.50 ± 8.78 mg/kg (CV 11.6%, n= 51) with u_{xpt} = 1.54 mg/kg*													
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00	|z score| ≤ 2.00	|ζ score| ≤ 2.00							
6	**84.04**	-	1.39	-	0.97	-	2.0000	Acid	$HCl:HNO_3:H_2O$	AAS	-	Y	AOAC (2016, 20th Ed, 928.08, 985.35 (50.1.14)
11	**85.78**	-	1.66	-	1.17	-	2.0000	Dry Ashing	$HCl:H_2O$	AAS	Fe 248.3	Y	AOAC (2016), 975.03, 985.35
12	**74.60**	7.00	-0.08	-0.12	-0.10	-0.24	0.5	Closed vessel	HNO_3	Flame AAS	-	N	AOAC (2016), 985.35
13	**53.20**	-	**-3.41**	-	**-2.54**	-	0.5	Microwave	HNO_3 10 mL + HCl 2 mL	Analytikal Jena ContrAA 800 D	-	N	Internal Method
14	**68.28**	-	-1.06	-	-0.82	-	0.5	Ashing	50% HNO_3, 50% HCl	ICP Horiba Jobin Yvon	Fe 259.94	Y	AOAC 975.03, 984.27
15	**75.50**	-	0.06	-	0.00	-	0.5	Ultrawave Digestion	5% HNO_3 + 0.5% HCl	ICP-MS (7900 Agilent)	Fe 56	N	Based on USFDA 4.7 version 1.1
16	**66.70**	2.05	-1.31	**-3.16**	-1.00	**-4.76**	0.5	Hot plate	HNO_3+H_2O_2	ICP-OES Optima 7000 DV Perkin Elmer	Fe 238.204	N	In-house Method
18	**118.00**	-	**6.69**	-	**4.84**	-	2.0	Dry Ashing	HNO_3	AAS, Varian	Various	N	AOAC 968.08
21	**73.50**	0.19	-0.25	-0.65	-0.23	-1.30	0.1	Microwave	180°C	Mar Xpress (CEM)	-	Y	AOAC 2011.14 (2016)
22	**91.70**	-	**2.59**	-	1.85	-	0.2 to 0.3	Microwave	HNO_3	ICP-MS Perkin Elmer	-	-	AOAC 2015.06
23	**68.30**	-	-1.06	-	-0.82	-	1.00	Dry Ashing	-	ICP-OES	238	-	AOAC 985.01
25	**65.80**	0.07	-1.45	**-3.80**	-1.10	**-6.30**	5.0205 / 5.0206	Wet Digestion	HNO_3-HCl	ICP-OES	Fe 238.204	-	USEPA Method 3050B
26	**82.20**	5.85	1.11	1.86	0.76	**2.03**	4.0	Dry ashing	Water & HCl (1+1)	AAS Shimadzu AA-7000	Fe 248.3	N	AOAC No. 975.03

Lab Number	Iron (mg/kg)	MU (mg/kg)	Based on NIMT		Based on median ± NIQR		Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm or mass)	Recovery Correction (Y/N)	Method Reference
			z score	Zeta score	z score	Zeta score							
[1]*Assigned value obtained from reference values (Gravimetric standard addition ICP-MS as x_{pt} ± SD_p from Horwitz' s equation) = 75.1 ± 6.4 mg/kg (CV 8.5%) with u_{xpt} = 0.9 mg/kg;* [2]*Assigned value obtained from median ± normalised IQR = 75.50 ± 8.78 mg/kg (CV 11.6%, n= 51) with u_{xpt} = 1.54 mg/kg*													
31	**56.06**	-	**-2.97**	-	**-2.21**	-	5	Dry Ashing	-	AAS, Agilent	-	N	AOAC 985.35
36	**71.00**	-	-0.64	-	-0.51	-	15 (Fe)	Dry Ashing	NA	UV-Vis (Agilent 8453)	510	N	AOAC 944.02 18th Ed
37	**77.15**	-	0.32	-	0.19	-	1	Wet Digestion	Nitric + perchloric	ICP-OES (Perkin Elmer Optima 8000)	Fe 238.204	N	AOAC (2016) 984.27
38	**56.20**	4.64	**-2.95**	**-5.60**	**-2.20**	**-6.93**	1.000	Dry Ashing	1N HNO_3 (0.1M HNO_3 for Fe)	Flame AAS, Shimadzu AA6300	Fe 248.30	-	AOAC 985.35, 19th Ed 2012 (Fe modified AOAC 999.11)
39	**74.00**	-	-0.17	-	-0.17	-	0.5	Microwave	-	AAS	Fe 248.3	Y	AOAC 985.35
42	**75.60**	5.45	0.08	0.14	0.01	0.03	5	Dry Ashing	HNO_3-HCl	Flame AAS, Agilent 280 FS	Fe 248.3	N	AOAC 985.35.2005
43	**72.65**	9.74	-0.38	-0.45	-0.32	-0.56	0.5	Microwave	HNO_3	ICP-OES	-	N	AOAC
44	**82.80**	14.20	1.20	1.03	0.83	1.00	1.0000	Dry Ashing	-	AAS, Thermoscientific	Fe 248.3	N	AOAC 19th Ed
45	**89.64**	3.38	**2.27**	**4.89**	1.61	**6.19**	4	Dry Ashing	HCl+HNO_3 +DI (2+2+70 mL) on hotplate	AAS (Flame, Varian)	Fe 248.3	N	AOAC 968.08
48	**73.47**	6.00	-0.25	-0.42	-0.23	-0.60	5	Dry Digestion		AA800 Perkin Elmer	Fe 248.3	N	MU-03/21 (AAS)
49	**102.00**	5.00	**4.19**	**7.68**	**3.02**	**9.03**	1, 3	Dry Ashing	Conc Nitric acid	AAS / AA-7000 Shimadzu	Fe 248.3	N	AOAC 20th Ed 2016
50	**55.30**	1.41	**-3.09**	**-7.77**	**-2.30**	**-11.93**	2.0000	Wet	Acid	Flame AAS (Varian)	248.3	N	AOAC 985.35
53	**83.30**	4.60	1.28	**2.44**	0.89	**2.82**	0.3	Microwave	4 mL HNO_3, 1 mL HCl, 1 mL H_2O_2	ICPMS Thermo	-	-	In house method

Lab Number	Iron (mg/kg)	MU (mg/kg)	Based on NIMT		Based on median ± NIQR		Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm or mass)	Recovery Correction (Y/N)	Method Reference
			z score	Zeta score	z score	Zeta score							
[1]Assigned value obtained from reference values (Gravimetric standard addition ICP-MS as x_{pt} ± SD_p from Horwitz' s equation) = 75.1 ± 6.4 mg/kg (CV 8.5%) with u_{xpt} = 0.9 mg/kg; [2]Assigned value obtained from median ± normalised IQR = 75.50 ± 8.78 mg/kg (CV 11.6%, n= 51) with u_{xpt} = 1.54 mg/kg													
54	**74.10**	2.60	-0.16	-0.36	-0.16	-0.69	1	Dry Ashing	HNO_3	ICP / Shimadzu	Fe 259.940	N	AOAC 984.27
55	**73.01**	-	-0.33	-	-0.28	-	1.5	Wet digestion	-	ICP-OES	Fe 259.940	Y	AOAC (2012) 984.27
58	**79.30**	2.03	0.65	1.58	0.43	**2.06**	3.0	Dry Ash	HCl	ICP-OES	-	-	Dry Ashing and Quantitation by ICP-OES
59	**76.91**	13.81	0.28	0.25	0.16	0.20	1.5	Dry Ashing	-	AAS, Shimadzu	Fe 248.3	Y	Fe: SNI 3751:2009 point A.10
60	**100.00**	-	**3.88**	-	**2.79**	-	-	-	-	-	-	-	SNI 01-2896-1998 (Fe)
61	**74.00**	8.73	-0.17	-0.22	-0.17	-0.32	1	Acid block digestion	HNO_3 (HNO_3/$HCLO_4$ for P)	Varian AA240 FS Fast Sequential AAS	Fe 248.3,	N	A6407-26 AAS
63	**96.60**	-	**3.35**	-	**2.40**	-	-	-	-	-	-	-	-
64	**75.13**	0.24	0.00	0.01	-0.04	-0.24	0.5070	Dry Ashing	1 N HNO_3	Shimadzu AA6300	Fe 248.3	N	Modified AOAC 985.35
67	**91.80**	-	**2.60**	-	1.86	-	2.0xxx	Dry Ash	Wet chemical	AAS, Perkin Elmer	Fe 248.33	N	AOAC 968.08
69	**124.00**	-	**7.62**	-	**5.52**	-	-	-	-	-	-	-	-
72	**80.00**	6.80	0.76	1.17	0.51	1.21	3	Ashing	HNO_3	AAS / Analytik Jena	Fe 589.0	N	AOAC 985.35
73A	**75.98**	15.49	0.14	0.11	0.06	0.06	1	Dry ashing	Hot plate	AAS (280FS AA, Agilent Technology)	Fe 248.3	N	FTC-46.01 (refers to AOAC 968.08, 965.09)
75	**86.92**	7.33	1.84	**2.68**	1.30	**2.87**	1	Wet digestion (hot block)	HNO_3 + H_2O_2	ICP-OES Agilent 5100	Fe 238.204	N	In House Method ICP-OES
81	**96.20**	4.10	**3.29**	**6.61**	**2.36**	**8.07**	Fe 0.5034	Dry Ashing (Ca, Fe)	1 N HNO_3 (Ca, Fe)	Shimadzu AAS AA 6300	Fe 248.3	N	AOAC 985.35 Mod (Ca, Fe)

Lab Number	Iron (mg/kg)	MU (mg/kg)	Based on NIMT		Based on median ± NIQR		Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm or mass)	Recovery Correction (Y/N)	Method Reference
			z score	Zeta score	z score	Zeta score							
[1]Assigned value obtained from reference values (Gravimetric standard addition ICP-MS as x_{pt} ± SD_p from Horwitz' s equation) = 75.1 ± 6.4 mg/kg (CV 8.5%) with u_{xpt} = 0.9 mg/kg; [2]Assigned value obtained from median ± normalised IQR = 75.50 ± 8.78 mg/kg (CV 11.6%, n= 51) with u_{xpt} = 1.54 mg/kg													
82A	**68.90**	9.08	-0.97	-1.20	-0.75	-1.38	0.250	none	none	HPGe detector, Canberra	-	-	Neutron Activation Analysis (NAA)
83	**63.96**	0.85	-1.74	**-4.48**	-1.31	**-7.22**	0.3	Microwave Digestion with HNO3	-	Microwave digester Mars Xpress, ICP MS Nex Ion (Perkin Elmer)	-	Y	Application Note, Perkin Elmer
84	**74.60**	7.50	-0.08	-0.11	-0.10	-0.22	0.5	Microwave Digestion	HNO_3 / H_2O_2	ICP-OES, ICP-MS	-	-	AOAC 999.10:2005
86	**67.90**	4.95	-1.12	**-2.07**	-0.87	**-2.61**	1.0000	Wet Digest		ICP-OES	Fe 259.9	Y	AOAC (2012) 984.27
87	**76.53**	0.19	0.22	0.58	0.12	0.66	2.5	Dry Ashing	HNO_3	Furnace Thermolyne	ICP-OES	N	MTD/FOD/CHM-09
89	**30.56**	0.46	**-6.94**	**-18.10**	**-5.12**	**-28.87**	2	Dry Ashing	1.5% HNO_3	AAS Agilent	Various	N	AOAC 985.35
90	**74.88**	-	-0.03	-	-0.07	-	1	Ultrawave	-	ICP-OES	Fe 238.204	-	-
91	**77.20**	-	0.33	-	0.19	-	-	-	-	-	-	-	-
92	**88.60**	-	**2.10**	-	1.49	-	1	Ashing	HNO_3	ICP-OES	-	-	-
94	**78.10**	-	0.47	-	0.30	-	1.5	Dry ashing (Fe: Wet ashing)	-	ICP-OES / Perkin Elmer	Fe 259.9	Y	AOAC (2012) 984.27
95	**74.50**	3.00	-0.09	-0.21	-0.11	-0.47	-	-	-	-	-	-	-

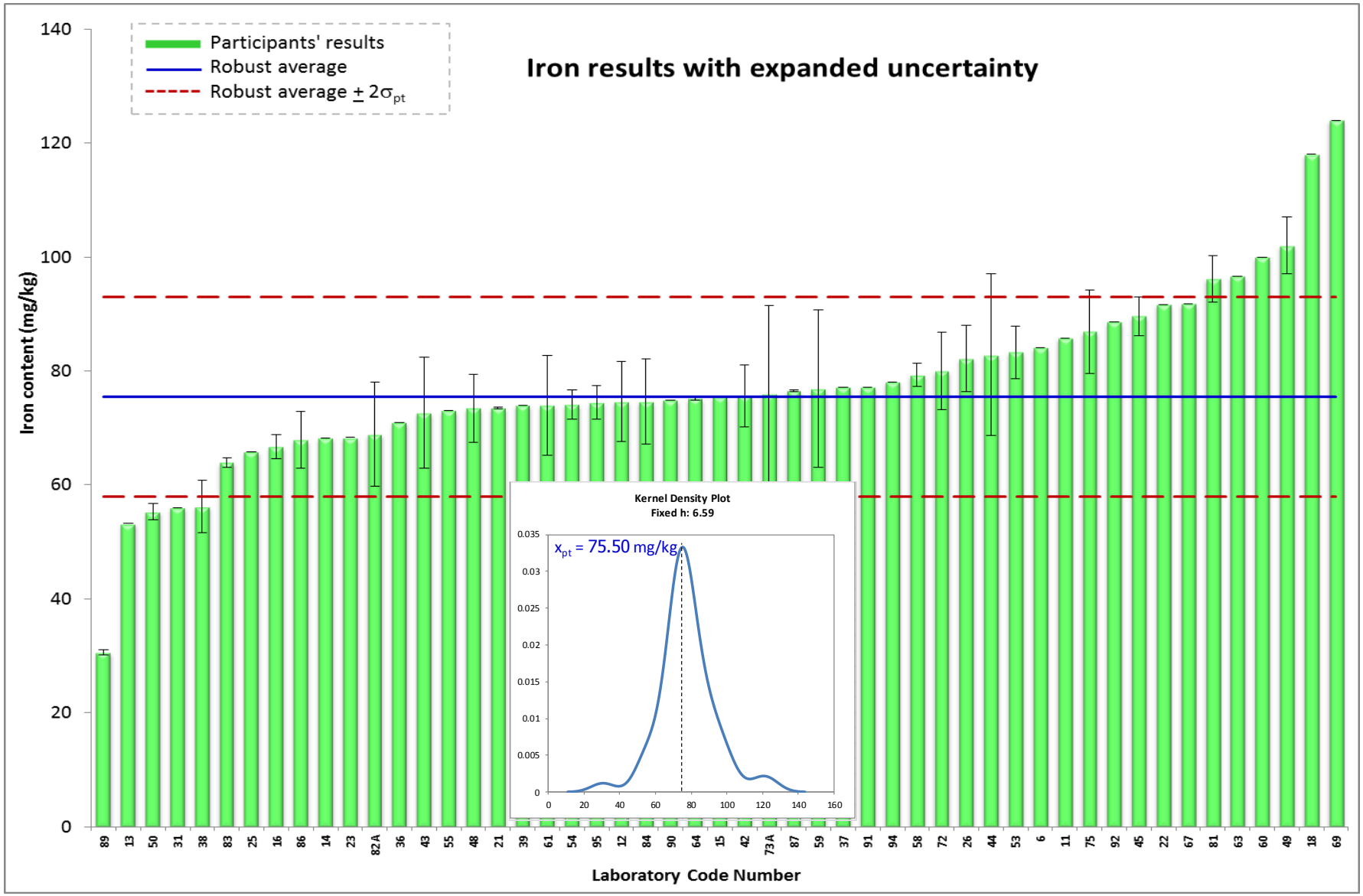

Figure 38. Distribution of **iron** results (ascending order) in defatted soybean flour with expanded uncertainty

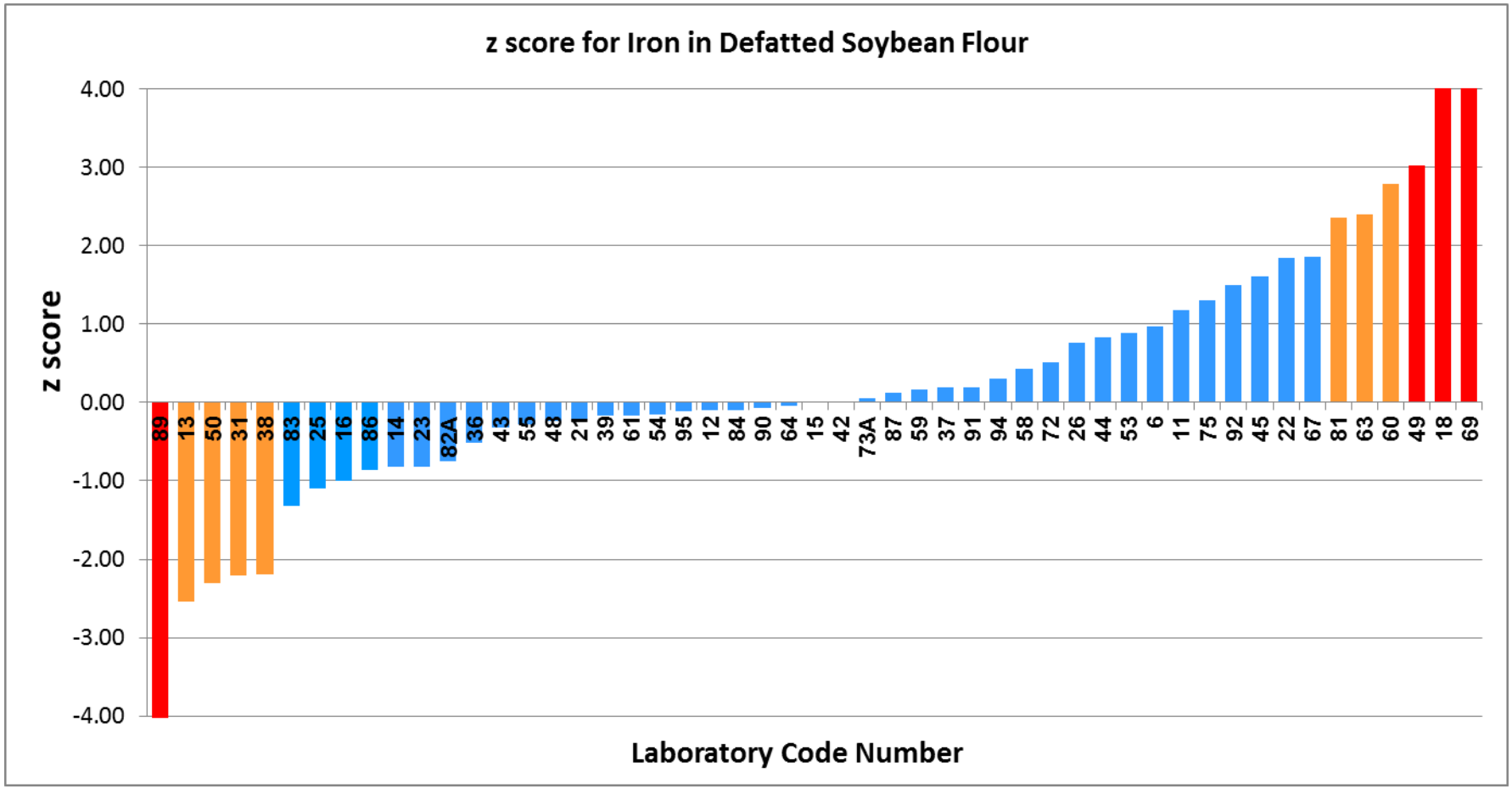

Figure 39. Plot of ordered *z* scores for **iron** results in defatted soybean flour

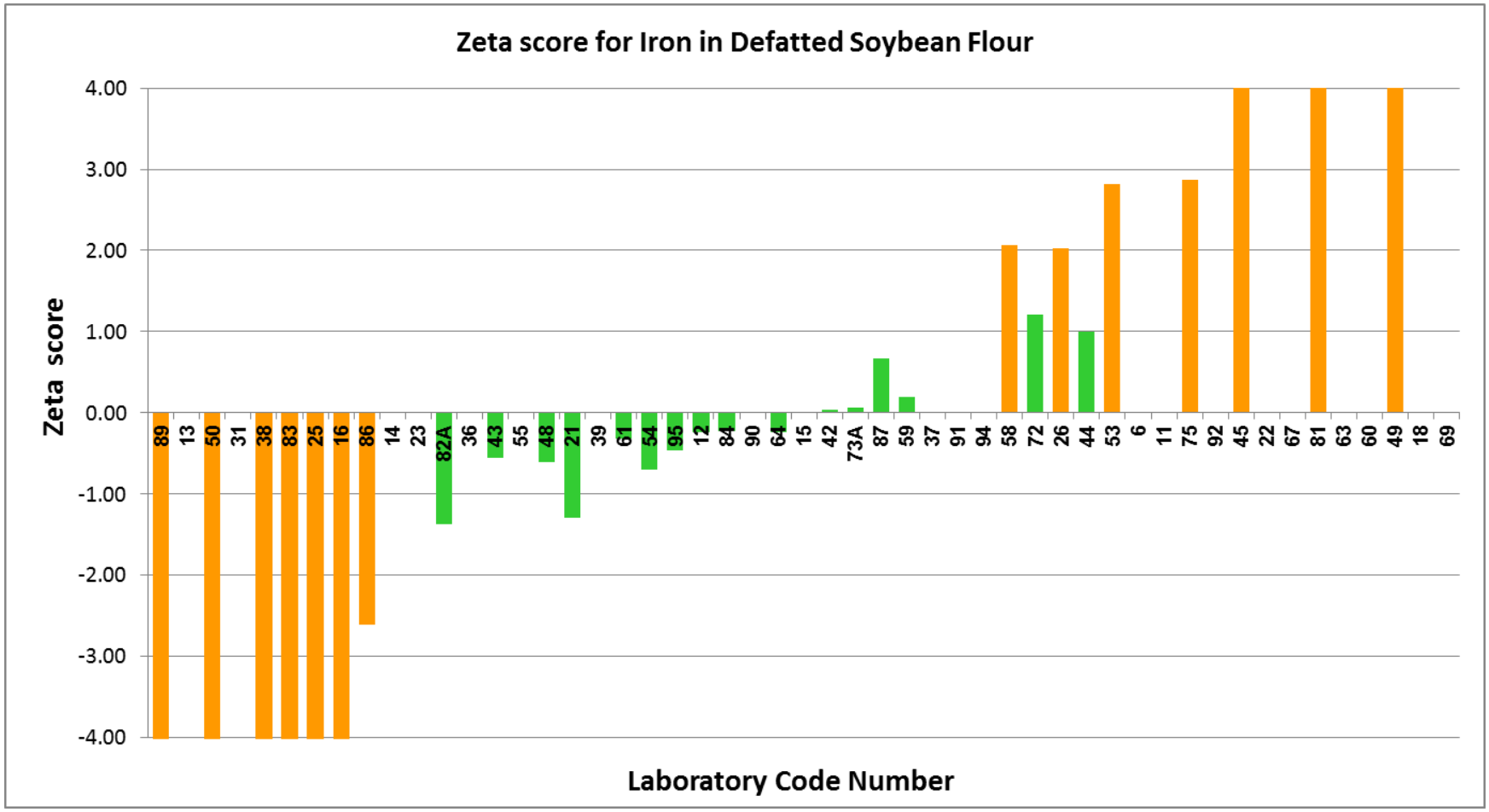

Figure 40. Plot of *Zeta score* for **iron** in defatted soybean flour, following the ordered z scores in the above Figure 39

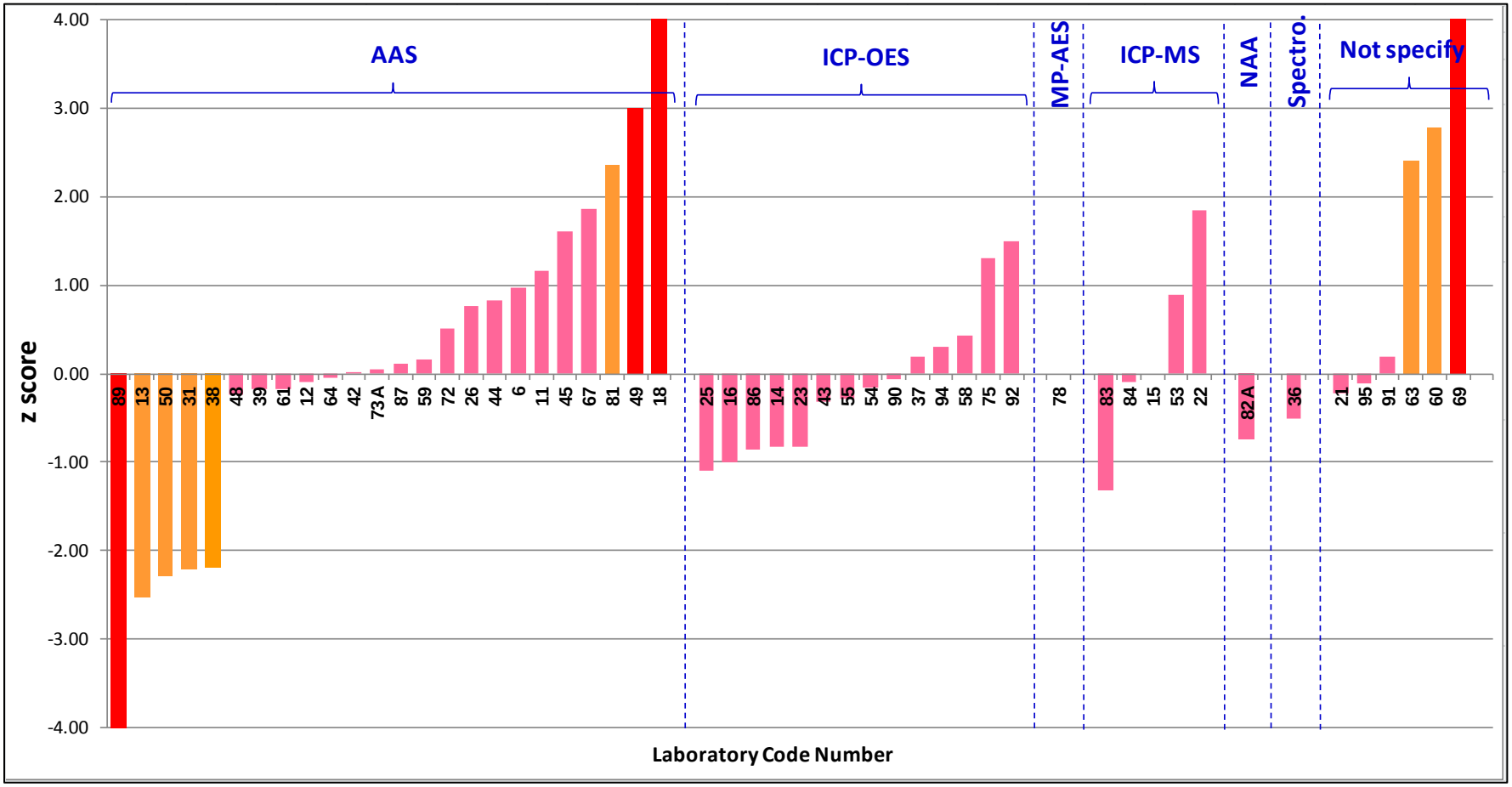

Figure 41. Plot of ordered *z* score for **iron** in defatted soybean flour, categorised in groups according to analytical methods/parameters used

Table 15. Evaluation of laboratory performance **zinc** analysis (mg/kg, as received) in defatted soybean flour

Lab Number	Zinc (mg/kg)	MU (mg/kg)	Based on NIMT		Based on x* ± s*		Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm or mass)	Recovery Correction (Y/N)	Method Reference
			z score	Zeta score	z score	Zeta score							
[1]Assigned value obtained from reference values (Isotope Dilution ICP-MS as x_{pt} ± SD_p from Horwitz' s equation) = 43.1 ± 3.9 mg/kg (CV 9.0%) with u_{xpt} = 0.6 mg/kg; [2]Assigned value obtained from robust average (x) ± robust SD (s*) = 42.59 ± 5.18 mg/kg (CV 12.2%, n= 46) with u_{xpt} = 0.95 mg/kg*													
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00	|z score| ≤ 2.00	|ζ score| ≤ 2.00							
6	**48.61**	-	1.42	-	1.16	-	2.0000	Acid	$HCl:HNO_3:H_2O$	AAS	-	Y	AOAC (2016, 20th Ed, 928.08, 985.35 (50.1.14)
11	**477.57**	-	**112.16**	**-**	**83.97**	-	2.0000	Dry Ashing	$HCl:H_2O$	AAS	-	Y	AOAC (2016), 975.03, 985.35
12	**32.20**	3.00	**-2.81**	**-7.20**	**-2.01**	**-5.85**	0.5	Closed vessel	HNO_3	Flame AAS	-	N	AOAC (2016), 985.35
14	**40.90**	-	-0.57	-	-0.33	-	0.5	Ashing	50% HNO_3, 50% HCl	ICP Horiba Jobin Yvon	Zn 213.856	Y	AOAC 975.03, 984.27
15	**43.30**	-	0.05	-	0.14	-	0.5	Ultrawave Digestion	5% HNO_3 + 0.5% HCl	ICP-MS (7900 Agilent)	-	N	Based on USFDA 4.7 version 1.1
16	**54.10**	5.41	**2.84**	**4.06**	**2.22**	**4.01**	0.5	Hot plate	$HNO_3+H_2O_2$	ICP-OES Optima 7000 DV Perkin Elmer	-	N	In-house Method
18	**37.50**	-	-1.45	-	-0.98	-	2.0	Dry Ashing	HNO_3	AAS, Varian	Various	N	AOAC 968.08
21	**30.50**	-	**-3.25**	**-**	**-2.33**	-	0.1	Microwave	180°C	Mar Xpress (CEM)	-	Y	AOAC 2011.14 (2016)
22	**50.10**	-	1.81	-	1.45	-	0.2 to 0.3	Microwave	HNO_3	ICP-MS Perkin Elmer	-	-	AOAC 2015.06
23	**39.80**	-	-0.85	-	-0.54	-	1.00	Dry Ashing	-	ICP-OES	589, 766, 422, 285, 238	-	AOAC 985.01
25	**34.20**	0.07	**-2.30**	**-43.87**	-1.62	**-8.83**	5.0205 / 5.0206	Wet Digestion	HNO_3-HCl	ICP-OES	-	-	USEPA Method 3050B
26	**39.50**	3.43	-0.93	**-2.08**	-0.60	-1.58	4.0	Dry ashing	Water & HCl (1+1)	AAS Shimadzu AA-7000	-	N	AOAC No. 975.03
31	**33.15**	1.86	**-2.57**	**-10.46**	-1.82	**-7.10**	5	Dry Ashing	-	AAS, Agilent	-	N	AOAC 985.35

Lab Number	Zinc (mg/kg)	MU (mg/kg)	Based on NIMT		Based on x* ± s*		Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm or mass)	Recovery Correction (Y/N)	Method Reference
			z score	Zeta score	z score	Zeta score							
[1]Assigned value obtained from reference values (Isotope Dilution ICP-MS as x_{pt} ± SD_p from Horwitz' s equation) = 43.1 ± 3.9 mg/kg (CV 9.0%) with u_{xpt} = 0.6 mg/kg; [2]Assigned value obtained from robust average (x) ± robust SD (s*) = 42.59 ± 5.18 mg/kg (CV 12.2%, n= 46) with u_{xpt} = 0.95 mg/kg*													
32	**47.00**	3.75	1.01	**2.07**	0.85	**2.10**	1.0068	Ashing	HCl	Flame AAS, Shimadzu 6300	-	N	Modified AOAC 969.32
37	**4.91**	-	**-9.86**	**-**	**-7.27**	-	1	Wet Digestion	Nitric + perchloric	ICP-OES (Perkin Elmer Optima 8000)	-	N	AOAC (2016) 984.27
38	**42.30**	1.69	-0.21	-0.92	-0.06	-0.23	1.000	Dry Ashing	1N HNO_3 (0.1M HNO_3 for Fe)	Flame AAS, Shimadzu AA6300	-	-	AOAC 985.35, 19th Ed 2012 (Fe modified AOAC 999.11)
39	**36.20**	-	-1.78	-	-1.23	-	0.5	Microwave		AAS	-	Y	AOAC 985.35
42	**47.70**	1.04	1.19	**8.26**	0.99	**4.72**	5	Dry Ashing	HNO_3-HCl	Flame AAS, Agilent 280 FS	-	N	AOAC 985.35.2005
43	**45.95**	1.02	0.74	**5.20**	0.65	**3.12**	0.5	Microwave	HNO_3	ICP-OES	-	N	AOAC
45	**41.56**	1.42	-0.40	**-2.09**	-0.20	-0.87	4	Dry Ashing	HCl+HNO_3+ DI (2+2+70 mL) on hotplate	AAS (Flame, Varian)	-	N	AOAC 968.08
48	**45.85**	1.47	0.71	**3.60**	0.63	**2.71**	5	Dry Digestion		AA800 Perkin Elmer	-	N	MU-03/21 (AAS)
49	**40.60**	2.00	-0.65	**-2.45**	-0.38	-1.44	1, 3	Dry Ashing	Conc Nitric acid	AAS / AA-7000 Shimadzu	-	N	AOAC 20th Ed 2016
50	**44.20**	2.43	0.28	0.89	0.31	1.04	2.0000	Wet	Acid	Flame AAS (Varian)	-	N	AOAC 985.35
53	**50.10**	2.30	1.81	**6.00**	1.45	**5.03**	0.3	Microwave	4 mL HNO_3, 1 mL HCl, 1 mL H_2O_2	ICPMS Thermo	-	-	In house method
54	**38.40**	1.00	-1.21	**-8.73**	-0.81	**-3.90**	1	Dry Ashing	HNO_3	ICP / Shimadzu	-	N	AOAC 984.27
55	**45.89**	-	0.72	-	0.64	-	1.5	Wet digestion		ICP-OES	-	Y	AOAC (2012) 984.27

Lab Number	Zinc (mg/kg)	MU (mg/kg)	Based on NIMT		Based on x* ± s*		Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm or mass)	Recovery Correction (Y/N)	Method Reference
			z score	Zeta score	z score	Zeta score							
[1]Assigned value obtained from reference values (Isotope Dilution ICP-MS as x_{pt} ± SD_p from Horwitz' s equation) = 43.1 ± 3.9 mg/kg (CV 9.0%) with u_{xpt} = 0.6 mg/kg; [2]Assigned value obtained from robust average (x) ± robust SD (s*) = 42.59 ± 5.18 mg/kg (CV 12.2%, n= 46) with u_{xpt} = 0.95 mg/kg*													
58	**47.10**	0.16	1.03	**18.57**	0.87	**4.73**	3.0	Dry Ash	HCl	ICP-OES	-	-	Dry Ashing and Quantitation by ICP-OES
59	**35.28**	3.36	**-2.02**	**-4.62**	-1.41	**-3.79**	1.5	Dry Ashing		AAS, Shimadzu	-	Y	AOAC 18th Ed 985.35 (Fe: SNI 3751:2009 point A.10)
60	**2900**	-	**737.51**	**-**	**551.62**	-	-	-	-	-	-	-	AOAC (2012)
61	**40.70**	11.20	-0.62	-0.43	-0.36	-0.33	1	Acid block digestion	HNO_3 (HNO_3/HCL O_4 for P)	Varian AA240 FS Fast Sequential AAS	-	N	A6407-26 AAS
63	**39.50**	-	-0.93	-	-0.60	-	-	-	-	-	-	-	-
64	**45.97**	0.19	0.74	**12.97**	0.65	**3.54**	0.5070	Dry Ashing	1 N HNO_3	Shimadzu AA6300	-	N	Modified AOAC 985.35
67	**48.40**	-	1.37	-	1.12	-	2.0xxx	Dry Ash	Wet chemical	AAS, Perkin Elmer	-	N	AOAC 968.08
69	**44.10**	-	0.26	-	0.29	-	-	-	-	-	-	-	-
72	**45.20**	4.10	0.54	1.02	0.50	1.16	3	Ashing	HNO_3	AAS / Analytik Jena	-	N	AOAC 985.35
73A	**45.93**	7.99	0.73	0.71	0.64	0.81	1	Dry ashing	Hot plate	AAS (280FS AA, Agilent Technology)	-	N	FTC-46.01 (refers to AOAC 968.08, 965.09)
75	**50.76**	3.61	1.98	**4.22**	1.58	**4.01**	1	Wet digestion (hot block)	HNO_3 + H_2O_2	ICP-OES Agilent 5100	-	N	In House Method ICP-OES
78	**36.20**	0.61	-1.78	**-18.92**	-1.23	**-6.40**	0.5	Mircowave Digestion	Acid Digestion	Berghof Speedwave 4	-	-	MP-AES
80	**40.70**	-	-0.62	-	-0.36	-	-	-	-	-	-	-	-

Lab Number	Zinc (mg/kg)	MU (mg/kg)	Based on NIMT		Based on x* ± s*		Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm or mass)	Recovery Correction (Y/N)	Method Reference
			z score	Zeta score	z score	Zeta score							
[1]Assigned value obtained from reference values (Isotope Dilution ICP-MS as x_{pt} ± SD_p from Horwitz' s equation) = 43.1 ± 3.9 mg/kg (CV 9.0%) with u_{xpt} = 0.6 mg/kg; [2]Assigned value obtained from robust average (x) ± robust SD (s*) = 42.59 ± 5.18 mg/kg (CV 12.2%, n= 46) with u_{xpt} = 0.95 mg/kg*													
81	**43.40**	1.90	0.08	0.31	0.16	0.60	0.5034	Dry Ashing (Ca, Fe)	1 N HNO_3 (Ca, Fe)	Shimadzu AAS AA 6300	-	N	AOAC 985.35 Mod (Ca, Fe)
82A	**45.50**	1.42	0.62	**3.25**	0.56	**2.45**	0.250	none	none	HPGe detector, Canberra	-	-	Neutron Activation Analysis (NAA)
83	**41.97**	1.05	-0.29	**-2.02**	-0.12	-0.57	0.3	Microwave Digestion with HNO3	-	Microwave digester Mars Xpress, ICP MS Nex Ion (Perkin Elmer)	-	Y	Application Note, Perkin Elmer
84	**43.70**	4.40	0.15	0.27	0.21	0.46	0.5	Microwave Digestion	HNO_3 / H_2O_2	ICP-OES, ICP-MS	-	N	AOAC 999.10:2005
86	**38.20**	2.19	-1.26	**-4.40**	-0.85	**-3.03**	1.0000	Wet Digest		ICP-OES	-	Y	AOAC (2012) 984.27
87	**44.25**	0.12	0.30	**5.48**	0.32	1.74	2.5	Dry Ashing	HNO_3	Furnace Thermolyne	ICP-OES	N	MTD/FOD/CHM -09
90	**43.20**	-	0.03	-	0.12	-	1	Ultrawave	-	ICP-OES	-	-	-
91	**40.30**	-	-0.72	-	-0.44	-	-	-	-	-	-	-	-
94	**41.20**	-	-0.49	-	-0.27	-	1.5	Dry ashing (Fe: Wet ashing)	-	ICP-OES / Perkin Elmer	-	Y	AOAC (2012) 984.27
95	**43.00**	5.00	-0.03	-0.04	0.08	0.15	-	-	-	-	-	-	-

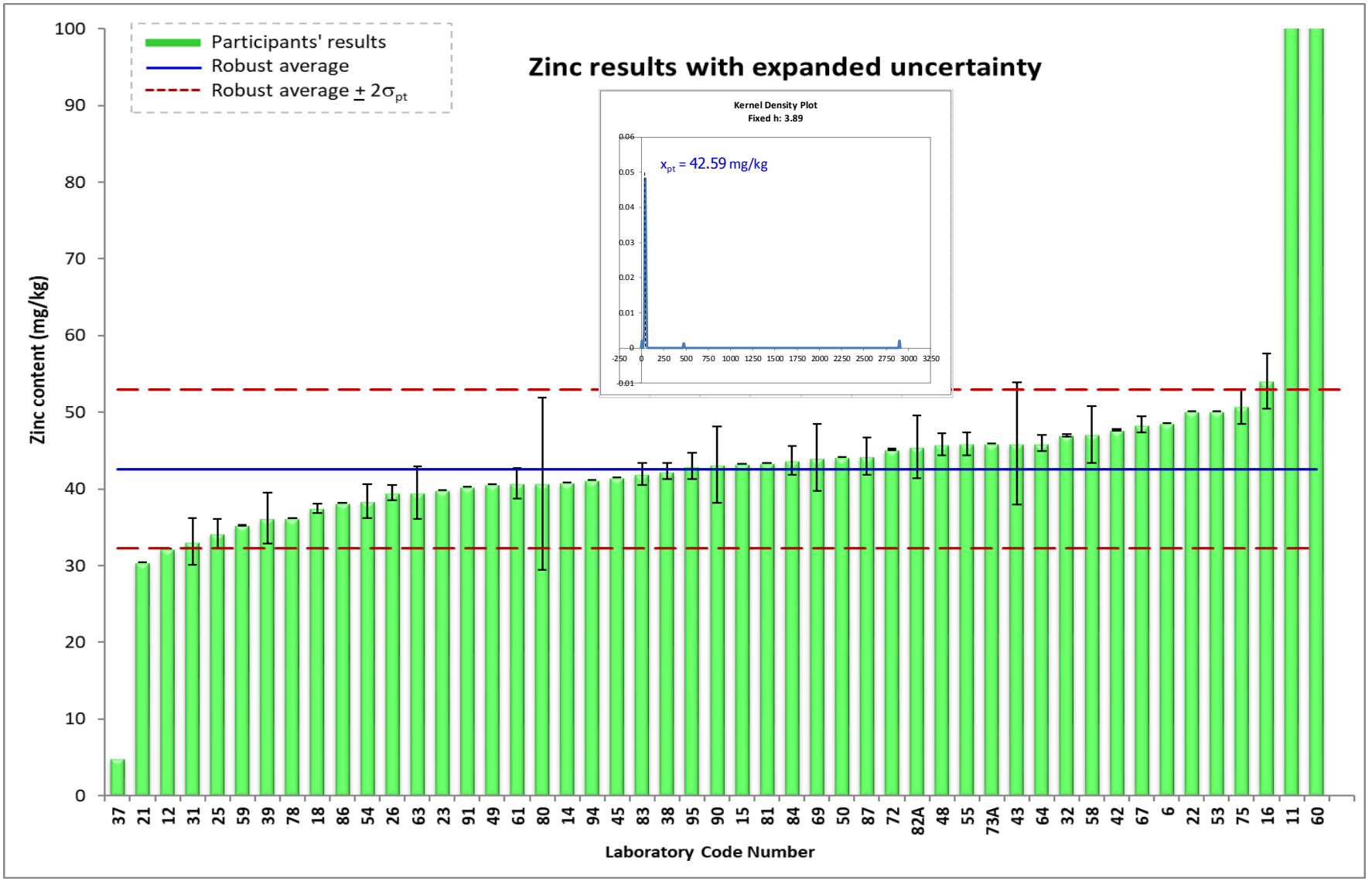

Figure 42. Distribution of **zinc** results (ascending order) in defatted soybean flour with expanded uncertainty

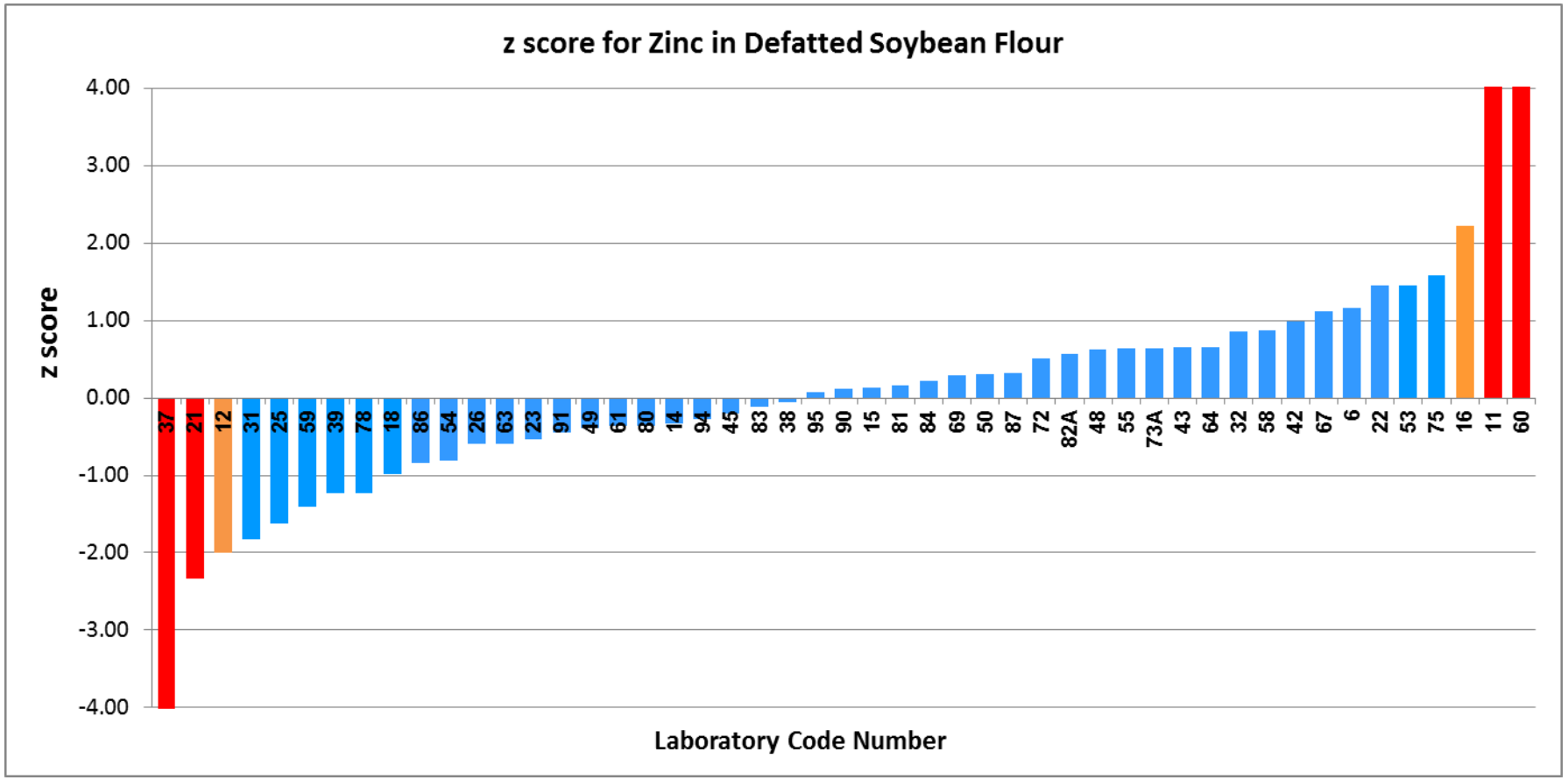

Figure 43. Plot of ordered *z* scores for **zinc** results in defatted soybean flour

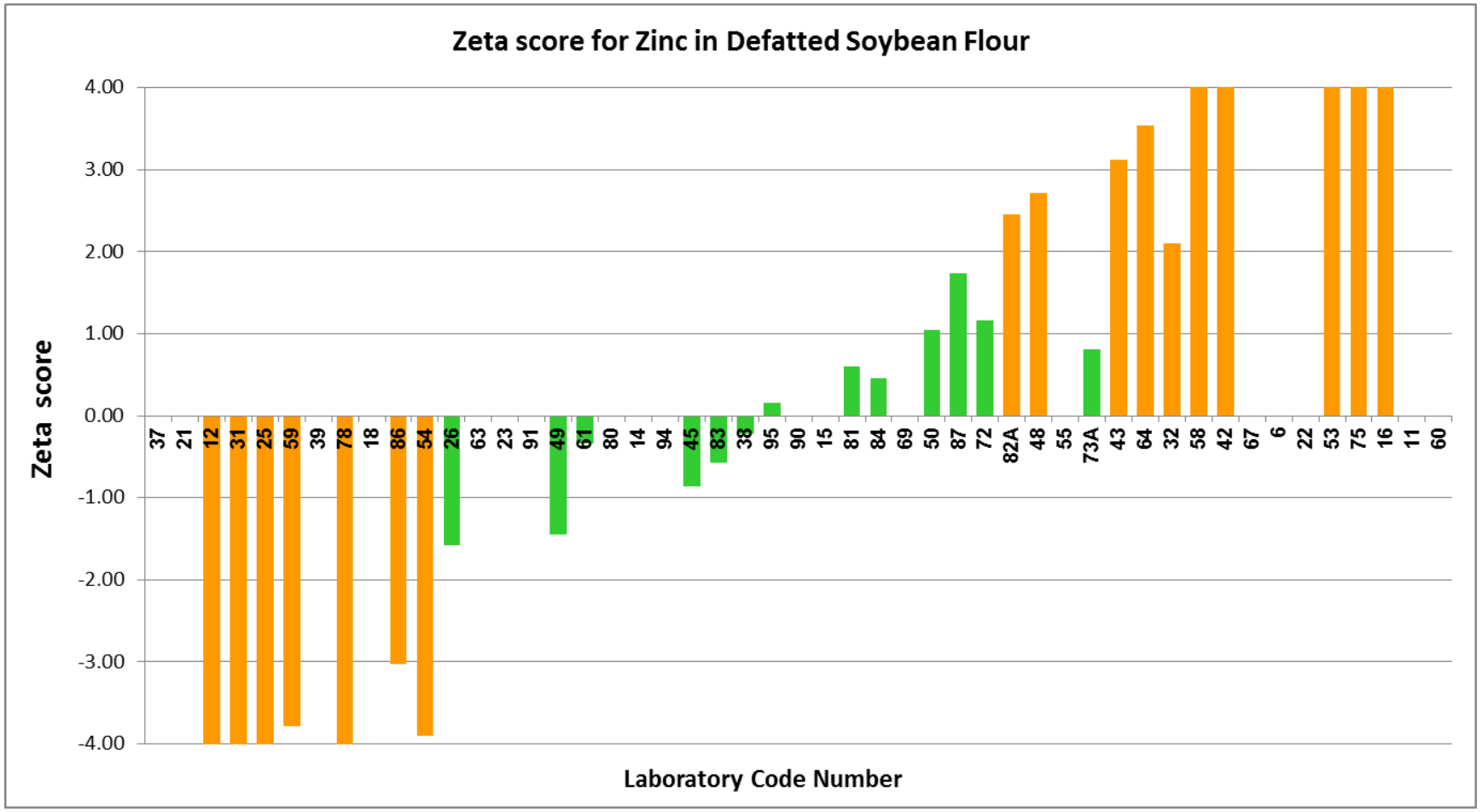

Figure 44. Plot of *Zeta score* for **zinc** in defatted soybean flour, following the ordered z scores in the above Figure 43

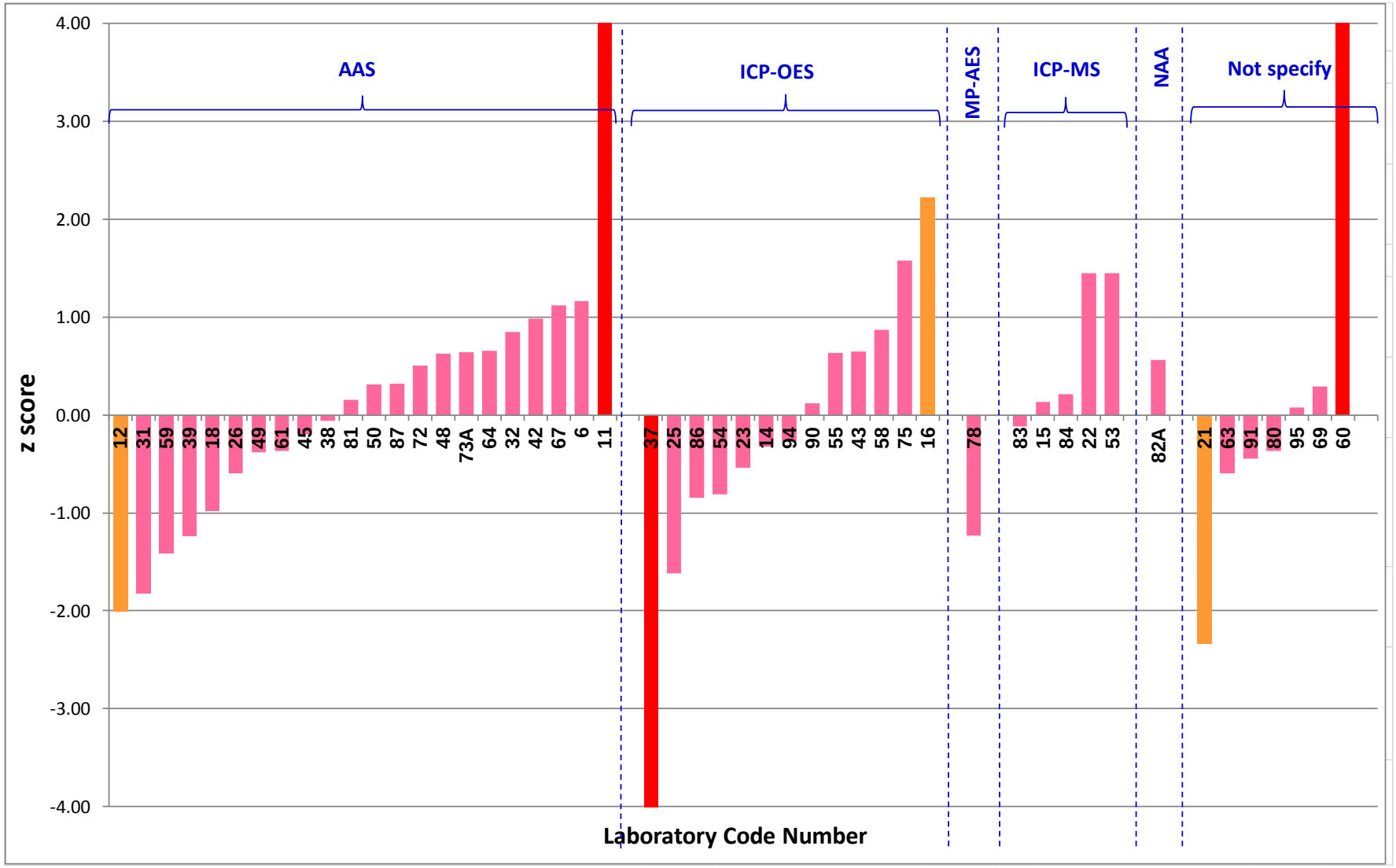

Figure 45. Plot of ordered *z* score for **zinc** in defatted soybean flour, categorised in groups according to analytical methods/parameters used

Table 16. Evaluation of laboratory performance **copper** analysis (mg/kg, as received) in defatted soybean flour

Lab Number	Copper (mg/kg)	MU (mg/kg)	Based on NIMT		Based on x* ± SDp		Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wave-length (nm or mass)	Recovery Correction (Y/N)	Method Reference
			z score	Zeta score	z score	Zeta score							
[1]Assigned value obtained from reference values (Isotope Dilution ICP-MS as x_{pt} ± SD_p from Horwitz' s equation) = 12.5 ± 1.3 mg/kg (CV 10.7%) with u_{xpt} = 0.2 mg/kg; [2]Assigned value obtained from robust average (x) ± SDp from Horwitz' s equation = 12.19 ± 1.34 mg/kg (CV 11.0%, n= 46) with u_{xpt} = 0.25 mg/kg*													
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00	|z score| ≤ 2.00	|ζ score| ≤ 2.00							
6	**13.21**	**-**	0.53	-	0.76	-	2.0000	Acid	$HCl:HNO_3:H_2O$	AAS	-	Y	AOAC (2016, 20th Ed, 928.08, 985.35 (50.1.14)
11	**13.98**	**-**	1.11	-	1.34	-	2.0000	Dry Ashing	$HCl:H_2O$	AAS	-	Y	AOAC (2016), 975.03, 985.35
12	**12.20**	**0.50**	-0.22	-0.94	0.01	0.03	0.5	Closed vessel	HNO_3	Flame AAS	-	N	AOAC (2016), 985.35
14	**11.01**	**-**	-1.11	-	-0.88	-	0.5	Ashing	50% HNO_3, 50% HCl	ICP Horiba Jobin Yvon	Cu 224.70	Y	AOAC 975.03, 984.27
15	**11.60**	**-**	-0.67	-	-0.44	-	0.5	Ultrawave Digestion	5% HNO_3 + 0.5% HCl	ICP-MS (7900 Agilent)	-	N	Based on USFDA 4.7 version 1.1
16	**9.01**	**0.21**	**-2.61**	**-15.45**	**-2.37**	**-11.73**	0.5	Hot plate	$HNO_3+H_2O_2$	ICP-OES Optima 7000 DV Perkin Elmer	-	N	In-house Method
18	**9.49**	**-**	**-2.25**	-	**-2.01**	-	2.0	Dry Ashing	HNO_3	AAS, Varian	Various	N	AOAC 968.08
21	**10.60**	**-**	-1.42	-	-1.19	-	0.1	Microwave	180°C	Mar Xpress (CEM)	-	Y	AOAC 2011.14 (2016)
22	**14.40**	**-**	1.42	-	1.65	-	0.2 to 0.3	Microwave	HNO_3	ICP-MS Perkin Elmer	-	-	AOAC 2015.06

Lab Number	Copper (mg/kg)	MU (mg/kg)	Based on NIMT		Based on x* ± SDp		Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wave-length (nm or mass)	Recovery Correction (Y/N)	Method Reference
			z score	Zeta score	z score	Zeta score							
[1]Assigned value obtained from reference values (Isotope Dilution ICP-MS as x_{pt} ± SD_p from Horwitz' s equation) = 12.5 ± 1.3 mg/kg (CV 10.7%) with u_{xpt} = 0.2 mg/kg; [2]Assigned value obtained from robust average (x) ± SDp from Horwitz' s equation = 12.19 ± 1.34 mg/kg (CV 11.0%, n= 46) with u_{xpt} = 0.25 mg/kg*													
23	**12.00**	**-**	-0.37	-	-0.14	-	1.00	Dry Ashing	-	ICP-OES	589, 766, 422, 285, 238	-	AOAC 985.01
25	**9.74**	**0.07**	**-2.06**	**-13.60**	-1.83	**-9.71**	5.0205 / 5.0206	Wet Digestion	HNO_3-HCl	ICP-OES	-	-	USEPA Method 3050B
26	**10.30**	**0.23**	-1.64	**-9.54**	-1.41	**-6.87**	4.0	Dry ashing	Water & HCl (1+1)	AAS Shimadzu AA-7000	-	N	AOAC No. 975.03
31	**11.26**	**1.51**	-0.93	-1.60	-0.70	-1.18	5	Dry Ashing	-	AAS, Agilent	-	N	AOAC 985.35
37	**12.89**	**-**	0.29	-	0.52	-	1	Wet Digestion	Nitric + perchloric	ICP-OES (Perkin Elmer Optima 8000)	-	N	AOAC (2016) 984.27
38	**11.90**	**1.68**	-0.45	-0.69	-0.22	-0.33	1.000	Dry Ashing	1N HNO_3 (0.1M HNO_3 for Fe)	Flame AAS, Shimadzu AA6300	-	-	AOAC 985.35, 19th Ed 2012 (Fe modified AOAC 999.11)
39	**15.20**	**-**	**2.02**	-	**2.25**	-	0.5	Microwave	-	AAS	-	Y	AOAC 985.35
42	**1.64**	**0.02**	**-8.11**	**-54.23**	**-7.87**	**-42.17**	5	Dry Ashing	HNO_3-HCl	Flame AAS, Agilent 280 FS	-	N	AOAC 985.35.2005
43	**12.21**	**0.53**	-0.22	-0.87	0.01	0.05	0.5	Microwave	HNO_3	ICP-OES	-	N	AOAC

Lab Number	Copper (mg/kg)	MU (mg/kg)	Based on NIMT		Based on x* ± SDp		Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wave-length (nm or mass)	Recovery Correction (Y/N)	Method Reference
			z score	Zeta score	z score	Zeta score							
[1]Assigned value obtained from reference values (Isotope Dilution ICP-MS as x_{pt} ± SD_p from Horwitz' s equation) = 12.5 ± 1.3 mg/kg (CV 10.7%) with u_{xpt} = 0.2 mg/kg; [2]Assigned value obtained from robust average (x) ± SDp from Horwitz' s equation = 12.19 ± 1.34 mg/kg (CV 11.0%, n= 46) with u_{xpt} = 0.25 mg/kg*													
45	**11.21**	**0.79**	-0.97	**-2.91**	-0.73	**-2.10**	4	Dry Ashing	HCl+HNO_3+DI (2+2+70 mL) on hotplate	AAS (Flame, Varian)	-	N	AOAC 968.08
48	**12.38**	**0.58**	-0.09	-0.35	0.14	0.49	5	Dry Digestion		AA800 Perkin	-	N	MU-03/21 (AAS)
49	**9.96**	**0.50**	-1.90	**-7.93**	-1.66	**-6.31**	1, 3	Dry Ashing	Conc Nitric acid	AAS / AA-7000 Shimadzu	-	N	AOAC 20th Ed 2016
50	**11.30**	**0.28**	-0.90	**-4.90**	-0.66	**-3.10**	2.0000	Wet	Acid	Flame AAS (Varian)	330.3, 404.4, 422.7, 248.3	N	AOAC 985.35
53	**12.70**	**0.40**	0.15	0.71	0.38	1.59	0.3	Microwave	4 mL HNO_3, 1 mL HCl, 1 mL H_2O_2	ICPMS Thermo	-		In house method
54	**10.50**	**0.40**	-1.49	**-7.07**	-1.26	**-5.28**	1	Dry Ashing	HNO_3	ICP / Shimadzu	-	N	AOAC 984.27
55	**12.60**	**-**	0.07	-	0.31	-	1.5	Wet digestion		ICP-OES	-	Y	AOAC (2012) 984.27
58	**13.90**	**0.93**	1.05	**2.77**	1.28	**3.24**	3.0	Dry Ash	HCl	ICP-OES	-		Dry Ashing and Quantitation by ICP-OES
59	**1.13**	**0.24**	**-8.50**	**-48.75**	**-8.25**	**-39.88**	1.5	Dry Ashing		AAS, Shimadzu	-	Y	AOAC 18th Ed 985.35 (Fe: SNI 3751:2009 point A.10)

Lab Number	Copper (mg/kg)	MU (mg/kg)	Based on NIMT		Based on x* ± SDp		Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wave-length (nm or mass)	Recovery Correction (Y/N)	Method Reference
			z score	Zeta score	z score	Zeta score							
[1]Assigned value obtained from reference values (Isotope Dilution ICP-MS as x_{pt} ± SD_p from Horwitz' s equation) = 12.5 ± 1.3 mg/kg (CV 10.7%) with u_{xpt} = 0.2 mg/kg; [2]Assigned value obtained from robust average (x) ± SDp from Horwitz' s equation = 12.19 ± 1.34 mg/kg (CV 11.0%, n= 46) with u_{xpt} = 0.25 mg/kg*													
61	**11.60**	**3.61**	-0.67	-0.50	-0.44	-0.32	1	Acid block digestion	HNO_3	Varian AA240 FS Fast Sequential AAS	-	N	A6407-26 AAS
63	**10.50**	**-**	-1.49	-	-1.26	-							
67	**17.50**	**-**	**3.74**	-	**3.96**	-	2.0xxx	Dry Ash	Wet chemical	AAS, Perkin Elmer	-	N	AOAC 968.08
69	**11.80**	**-**	-0.52	-	-0.29	-							
72	**12.70**	**1.10**	0.15	-	0.38	0.84	3	Ashing	HNO_3	AAS / Analytik Jena	-	N	AOAC 985.35
75	**14.11**	**1.31**	1.20	**2.35**	1.43	**2.74**	1	Wet digestion (hot block)	HNO_3 + H_2O_2	ICP-OES Agilent 5100	-	N	In House Method ICP-OES
78	**13.10**	**-**	0.45	-	0.68	-	0.5	Mircowave Digestion	Acid Digestion	Berghof Speedwave	-		MP-AES
80	**15.20**	**-**	**2.02**		**2.25**	-							
82A	**13.90**	**6.05**	1.05	0.46	1.28	0.56	0.250	none	none	HPGe detector, Canberra	-		Neutron Activation Analysis
82B	**13.50**	**1.30**	0.75	1.47	0.98	1.88	1.00	Microwave	Nitric Acid	AAS, GBC	-	Y	Flame SSA
83	**13.00**	**0.05**	0.37	**2.48**	0.60	**3.22**	0.3	Microwave Digestion with HNO_3		ICP MS Nex Ion (Perkin Elmer)	-	Y	Application Note, Perkin Elmer
84	**12.40**	**1.20**	-0.07	-0.16	0.16	0.32	0.5	Microwave Digestion	HNO_3 / H_2O_2	ICP-OES, ICP-MS	-	N	AOAC 999.10:2005

Lab Number	Copper (mg/kg)	MU (mg/kg)	Based on NIMT		Based on x* ± SDp		Sample weight (g)	Digestion Technique	Digestion Medium	Instrument	Wave-length (nm or mass)	Recovery Correction (Y/N)	Method Reference
			z score	Zeta score	z score	Zeta score							
[1]Assigned value obtained from reference values (Isotope Dilution ICP-MS as x_{pt} ± SD_p from Horwitz' s equation) = 12.5 ± 1.3 mg/kg (CV 10.7%) with u_{xpt} = 0.2 mg/kg; [2]Assigned value obtained from robust average (x) ± SDp from Horwitz' s equation = 12.19 ± 1.34 mg/kg (CV 11.0%, n= 46) with u_{xpt} = 0.25 mg/kg*													
86	**11.30**	**0.67**	-0.90	**-3.08**	-0.66	**-2.13**	1.0000	Wet Digest		ICP-OES	-	Y	AOAC (2012) 984.27
87	**12.12**	**0.18**	-0.29	-1.75	-0.05	-0.27	2.5	Dry Ashing	HNO_3	Furnace Thermolyne	ICP-OES	N	MTD/FOD/CHM-09
89	**28.99**	**0.44**	**12.32**	**55.80**	**12.54**	**50.70**	2	Dry Ashing	1.5% HNO_3	AAS Agilent	Various	N	AOAC 985.35
90	**12.67**	**-**	0.13	-	0.36	-	1	Ultrawave	-	ICP-OES	-	-	-
91	**11.70**	**-**	-0.60	-	-0.37	-	-	-	-	-	-	-	-
92	**14.10**	**-**	1.20	-	1.43	-	1	Ashing	HNO_3	ICP-OES	-	-	-
94	**12.80**	**-**	0.22	-	0.46	-	1.5	Dry ashing (Fe: Wet ashing)	-	ICP-OES / Perkin Elmer	Cu 324.7	Y	AOAC (2012) 984.27

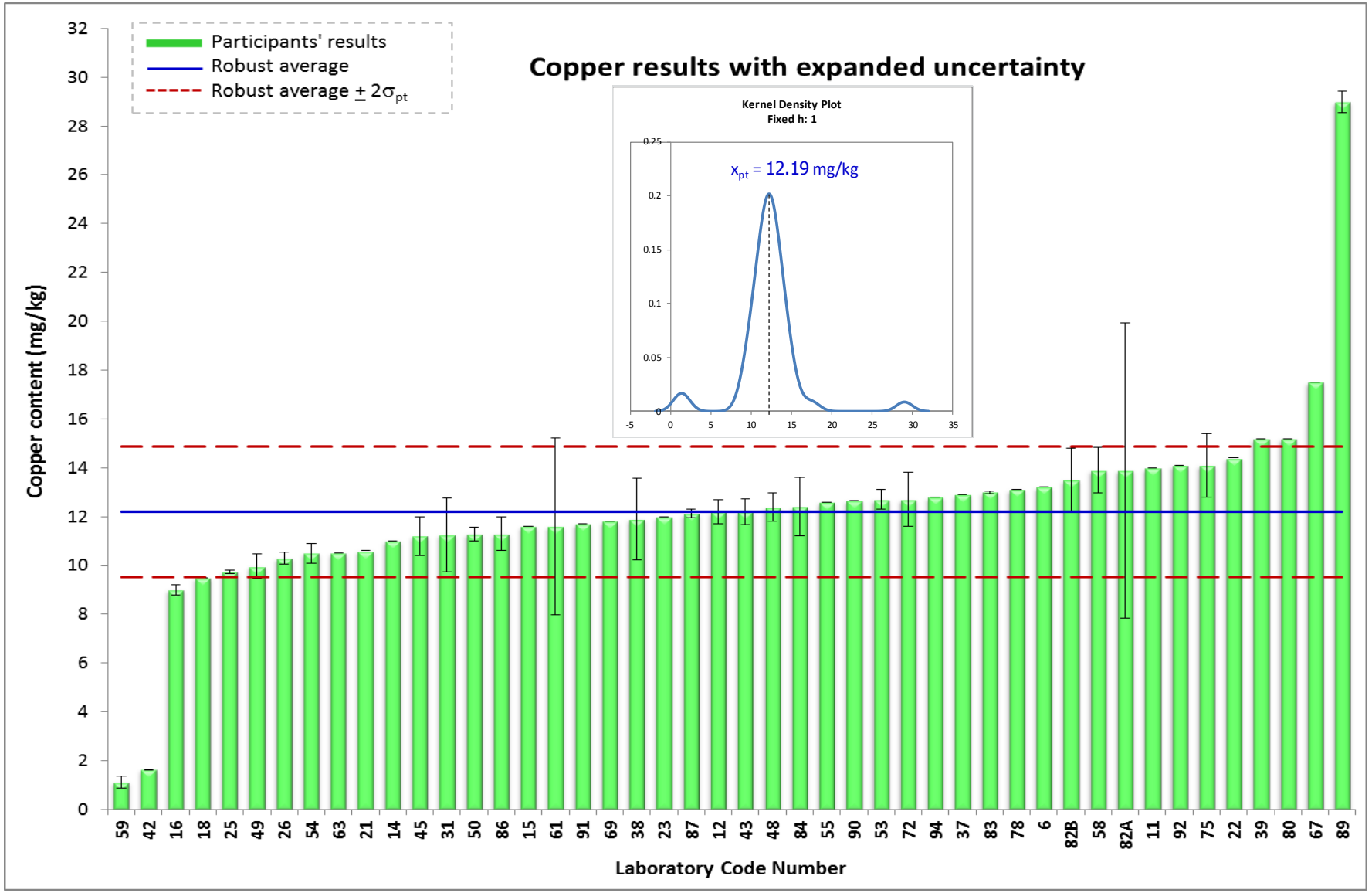

Figure 46. Distribution of **copper** results (ascending order) in defatted soybean flour with expanded uncertainty

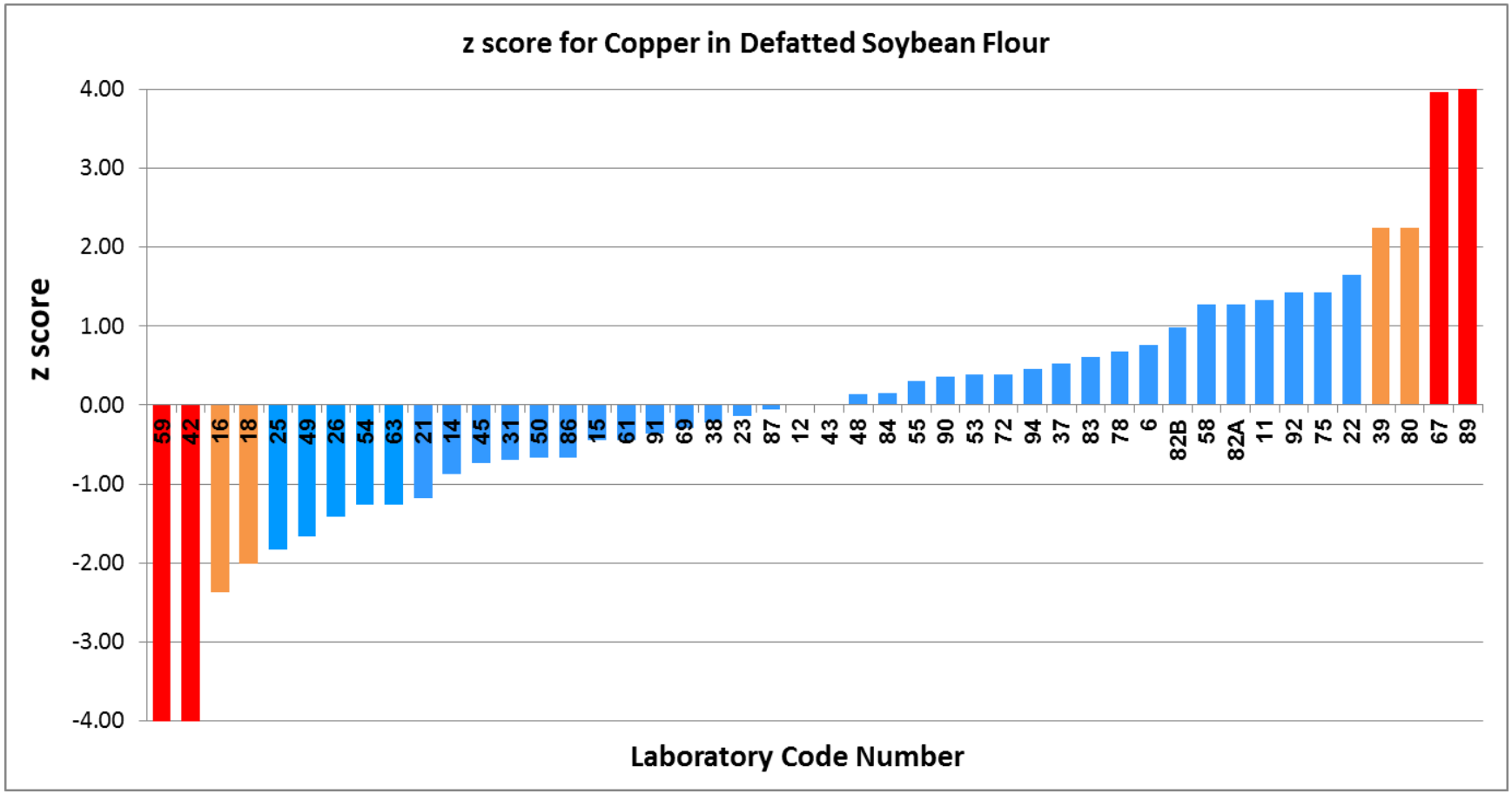

Figure 47. Plot of ordered *z* scores for **copper** results in defatted soybean flour

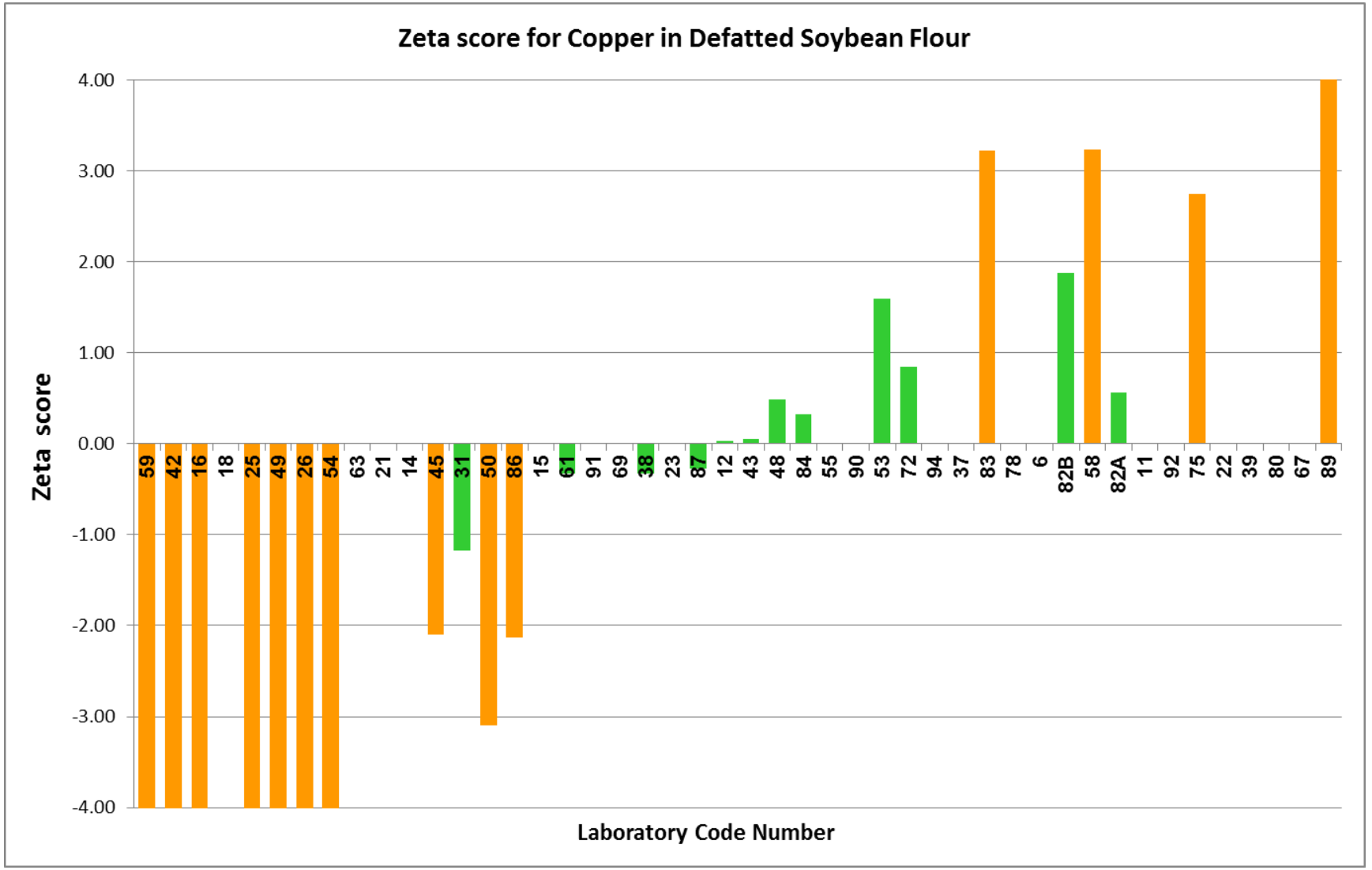

Figure 48. Plot of *Zeta score* for **copper** in defatted soybean flour, following the ordered z scores in the above Figure 47

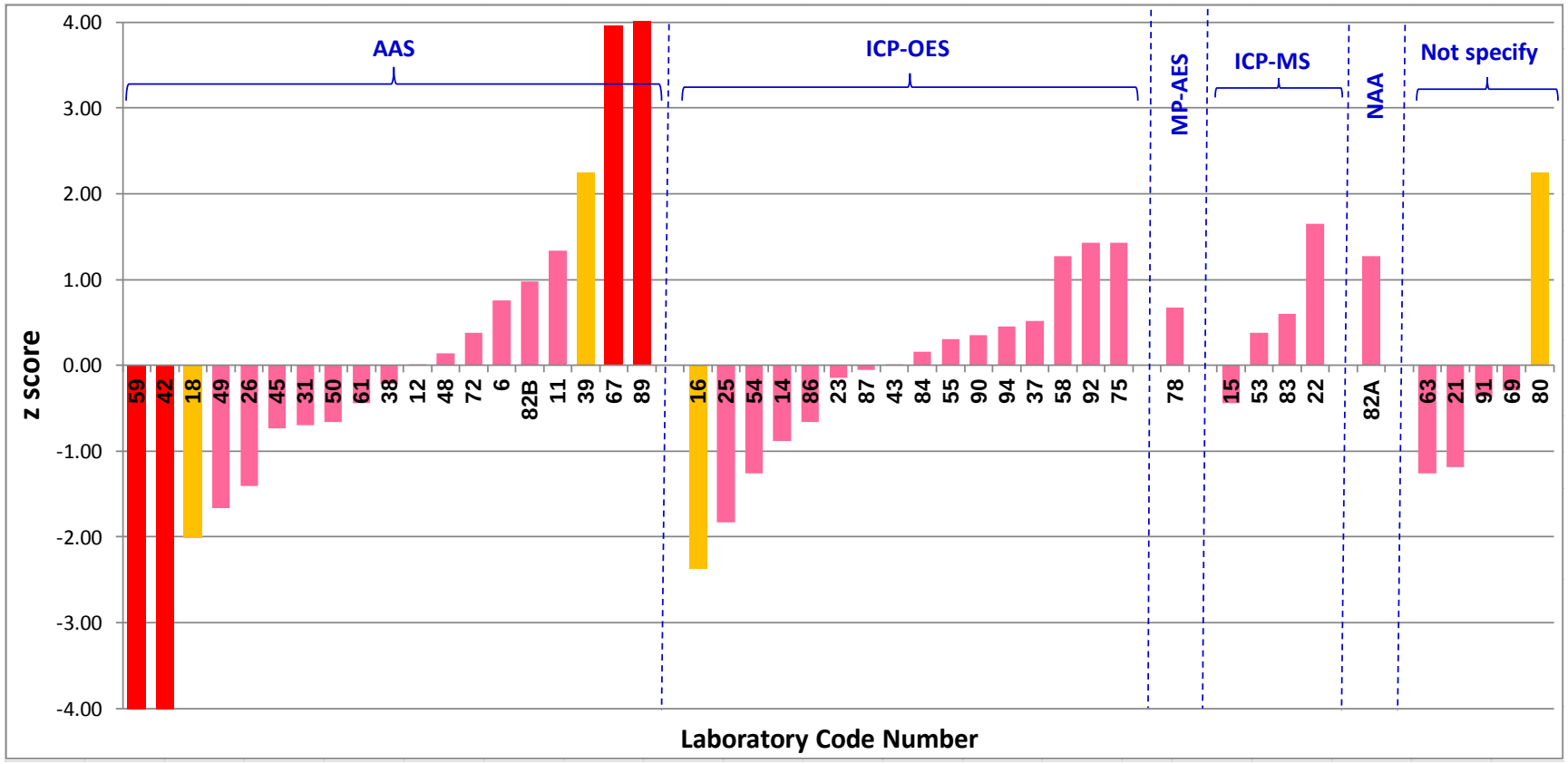

Figure 49. Plot of ordered *z* score for **copper** in defatted soybean flour, categorised in groups according to analytical methods/parameters used

4. SUMMARY OF LABORATORY PERFORMANCE ON ANALYSES OF NUTRIENTS IN DEFATTED SOYBEAN FLOUR

Robust z-scores, plotted as bar-charts in **Figure 50-57**, are used as the demonstrators of participating laboratory analytical performance on analyses of nutrients in defatted soybean flour. None of the participanting laboratories submitted data of all parameters. Twenty-one out of 84 laboratories (25%, Lab nos. 4, 5, 8, 9, 10, 14, 15, 23, 32, 36, 45, 56, 61, 62, 64, 66A, 74, 84, 85, 97 and 98) showed excellent performance ($|z\ score| \leq 2$) for analyses of all nutrients. Twenty-four out of 84 laboratories (29%, Lab nos. 2, 6, 26, 31, 37, 38, 39, 41, 43, 44, 53, 54, 58, 63, 67, 68, 72, 78, 80, 82A, 86, 87, 91 and 95) performed good performance for $\geq$80% of all parameters. The most problematic nutrient ($|z\ score| > 2$) in this study is sodium analysis (good performance was identified for 28 out of 84 laboratories) followed by calcium analysis (21 out of 84 laboratories). Questionable and unsatisfactory results of ash, total dietary fibre, phosphorus, potassium and iron were identified in 9-13 out of 84 laboratories whereas those of moisture, total nitrogen, magnesium, copper and zinc found in some laboratories (6-8 out of 84 laboratories).

Table 17 shows summary of performance on individual nutrients analyses in defatted soybean flour of all laboratories. Good performance for moisture, total nitrogen, ash, magnesium, zinc and copper are achieved ($|z\ score| \leq 2$, satisfactory results) by >80% of all participants). Satisfactory performance for analyses of total dietary fibre, calcium, phosphorus, potassium and iron by participating laboratories are shown in 60-80% of all participants. Sodium analysis was classified as problematic nutrients, only about 24% of all participants submitted values which were identified as satisfactory results. Due to defatted soyben flour contained small amount of total fat, the performance on the analysis cannot be evaluated.

5. REFERENCE VALUES IN DEFATTED SOYBEAN FLOUR

After evaluation of analytical performance of participating laboratories, reference values of all assigned nutrients (except total fat) for defatted soybean flour were developed from participating laboratories with good performance ($|z\ score| \leq 2$). The reference values as mean $\pm$ SD are presented in **Table 18**. The defatted soybean flour with reference values of nutrients becomes a reference material from proficiency testing programme. It can be used as a reference test material for future laboratory performance study or a quality control sample for internal quality control system and/or as a reference material for method validation.

Table 17. Summary: evaluation of laboratory performance in defatted soybean flour

Parameters	Total participants	Evaluation results (number of laboratory, percentage in bracket)		
		Satisfactory	Questionable	Unsatisfactory
Moisture (g/100g)	79	72 (91.1%)	4 (5.1%)	3 (3.8%)
Total nitrogen (g/100g)	66	57 (86.4%)	5 (7.6%)	4 (6.1%)
Fat (g/100g)	40	Not evaluate due to high variation of results (0.0-4.0 g/100g)		
Ash (g/100g)	73	65 (89.0%)	3 (4.1%)	5 (6.8%)
Total dietary fibre (g/100g)	30	19 (63.3%)	2 (6.7%)	9 (30.0%)
Calcium (mg/kg)	57	36 (63.2%)	8 (14.0%)	13 (22.8%)
Magnesium (mg/kg)	47	39 (83.0%)	3 (6.4%)	5 (10.6%)
Phosphorus (mg/kg)	42	30 (71.4%)	2 (4.8%)	10 (23.8%)
Sodium (mg/kg)	42	10 (23.8%)	7 (16.7%)	25 (59.5%)
Potassium (mg/kg)	49	38 (77.6%)	5 (10.2%)	6 (12.2%)
Iron (mg/kg)	51	40 (78.4%)	7 (13.7%)	4 (7.8%)
Zinc (mg/kg)	49	43 (87.8%)	3 (6.1%)	3 (6.1%)
Copper (mg/kg)	46	38 (82.6%)	4 (8.7%)	4 (8.7%)

Table 18. Summary of reference values of nutrients in defatted soybean flour*

Parameters	Number of laboratories (n)	Reference values	
		Mean ± SD	%CV
Moisture (g/100g)	70	7.27 ± 0.57	7.8
Total nitrogen (g/100g)	56	7.88 ± 0.12	1.5
Ash (g/100g)	66	6.32 ± 0.32	5.0
Total dietary fibre (g/100g)	19	16.80 ± 1.26	7.5
Calcium (mg/kg)	36	2039 ± 184	9.0
Magnesium (mg/kg)	39	2652 ± 246	9.3
Phosphorus (mg/kg)	30	7758 ± 377	4.9
Sodium (mg/kg)	10	53.2 ± 11.7	21.9
Potassium (mg/kg)	38	23270 ± 2252	9.7
Iron (mg/kg)	40	76.71 ± 7.02	9.2
Zinc (mg/kg)	42	42.77 ± 4.42	10.3
Copper (mg/kg)	38	12.19 ± 1.23	10.1

*Reference values derived from good performance laboratories (|z score |≤ 2) in each parameter.

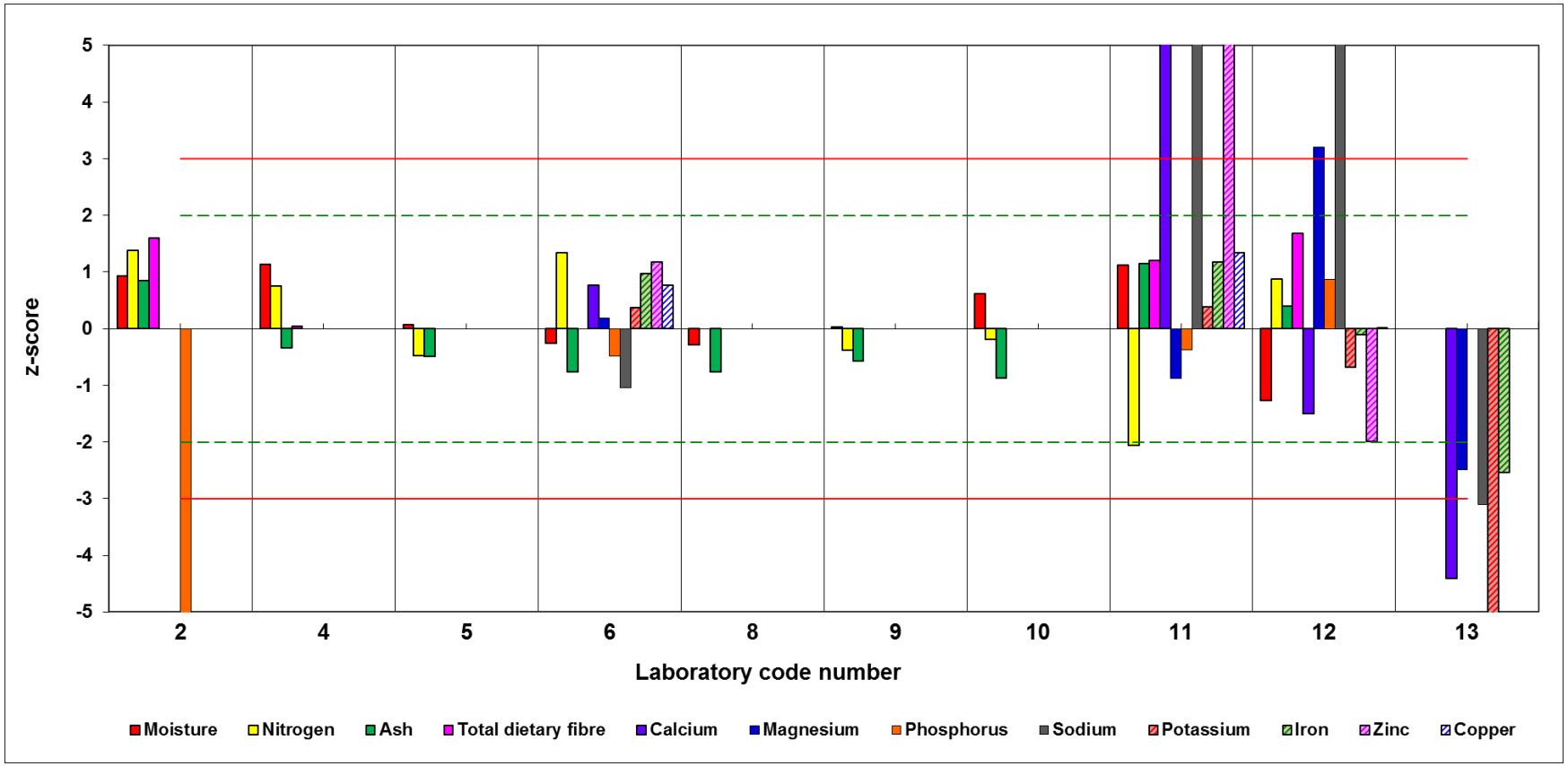

Figure 50. Bar-chart of z-scores for APFAN PT-2 in which the participants determined all nutrients using defatted soybean flour as test material: laboratory code number 2-13.

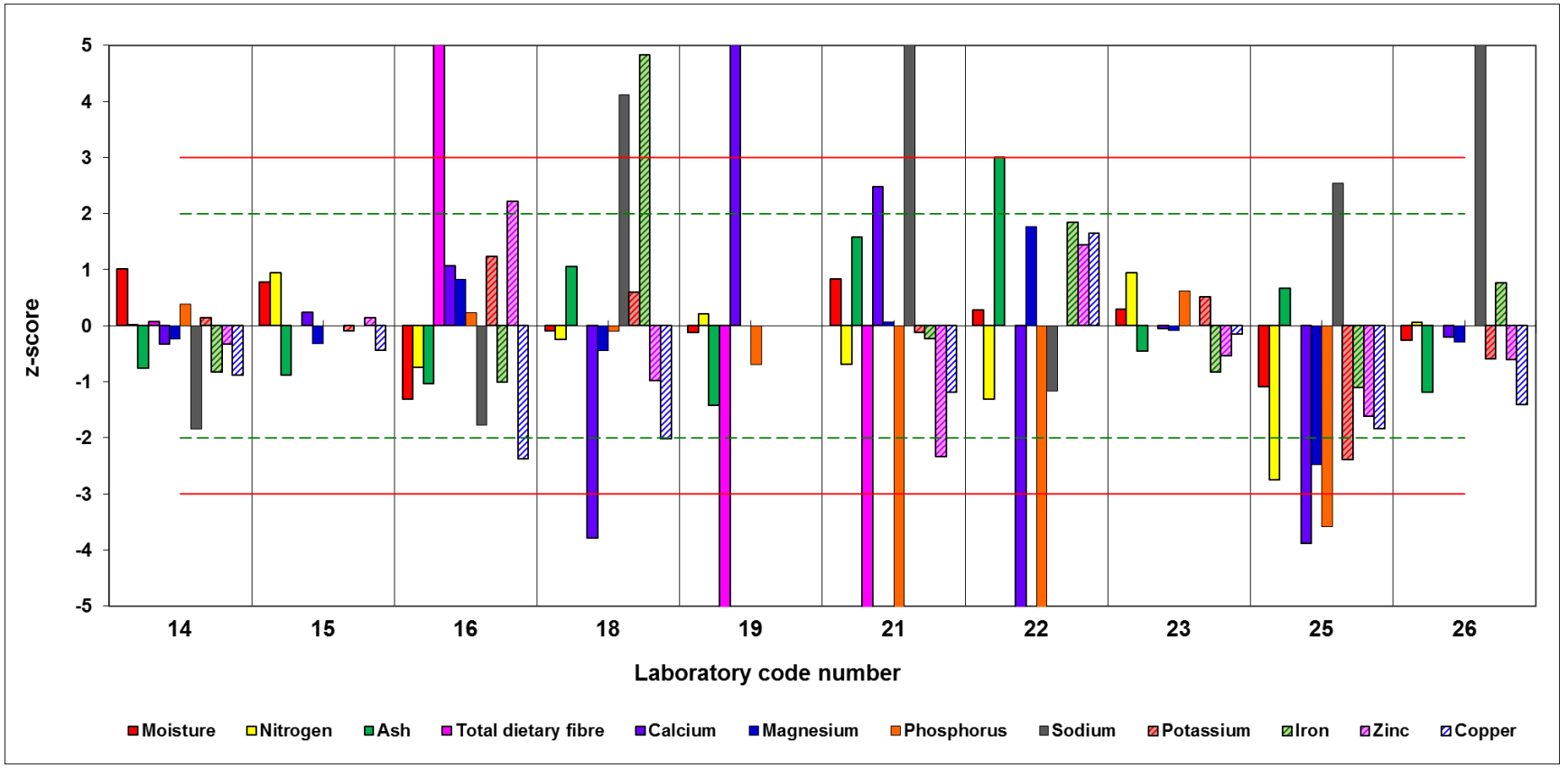

Figure 51. Bar-chart of z-scores for APFAN PT-2 in which the participants determined all nutrients using defatted soybean flour as test material: laboratory code number 14-26.

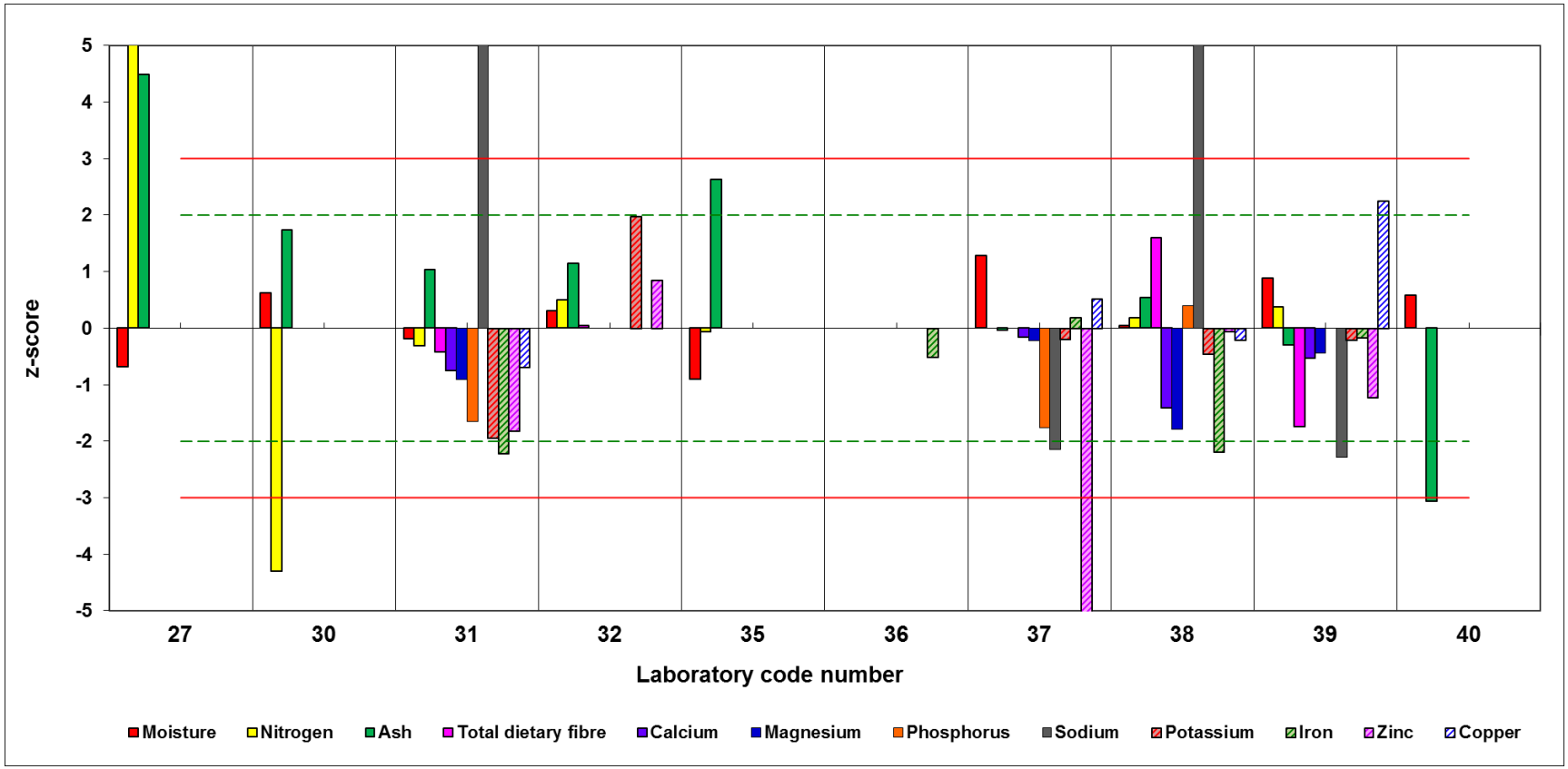

Figure 52. Bar-chart of z-scores for APFAN PT-2 in which the participants determined all nutrients using defatted soybean flour as test material: laboratory code number 27-40.

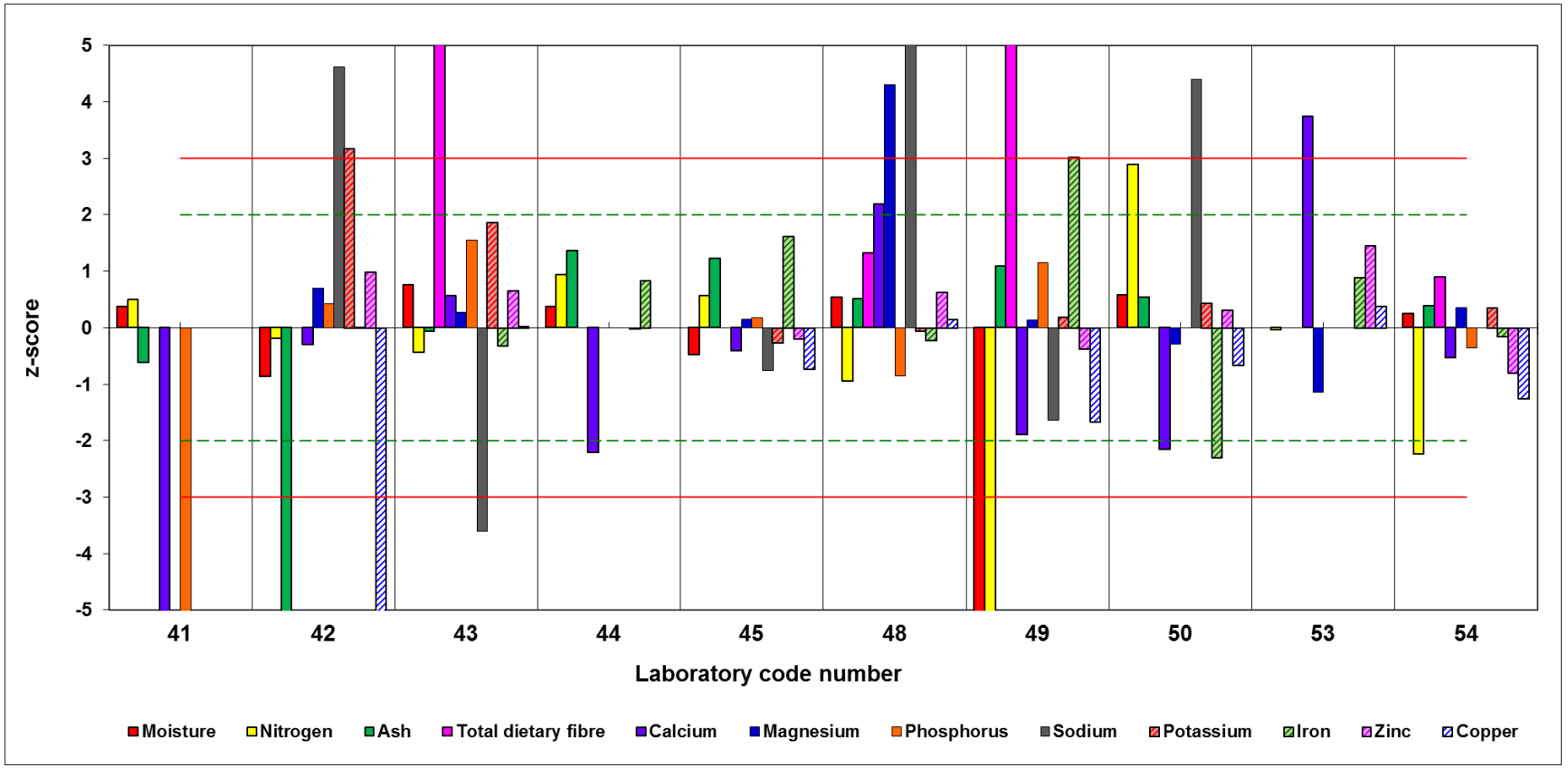

Figure 53. Bar-chart of z-scores for APFAN PT-2 in which the participants determined all nutrients using defatted soybean flour as test material: laboratory code number 41-54.

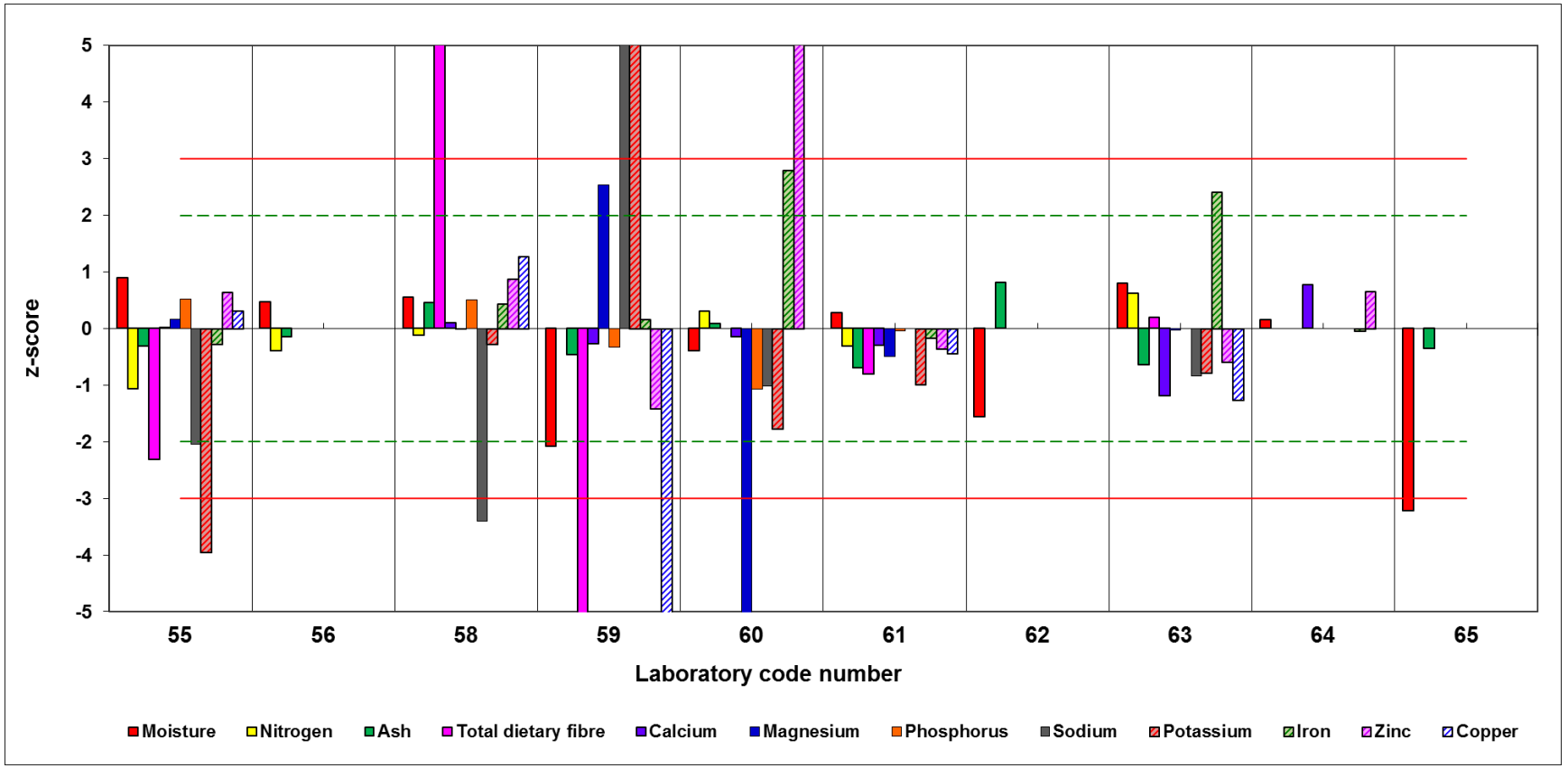

Figure 54. Bar-chart of z-scores for APFAN PT-2 in which the participants determined all nutrients using defatted soybean flour as test material: laboratory code number 55-65.

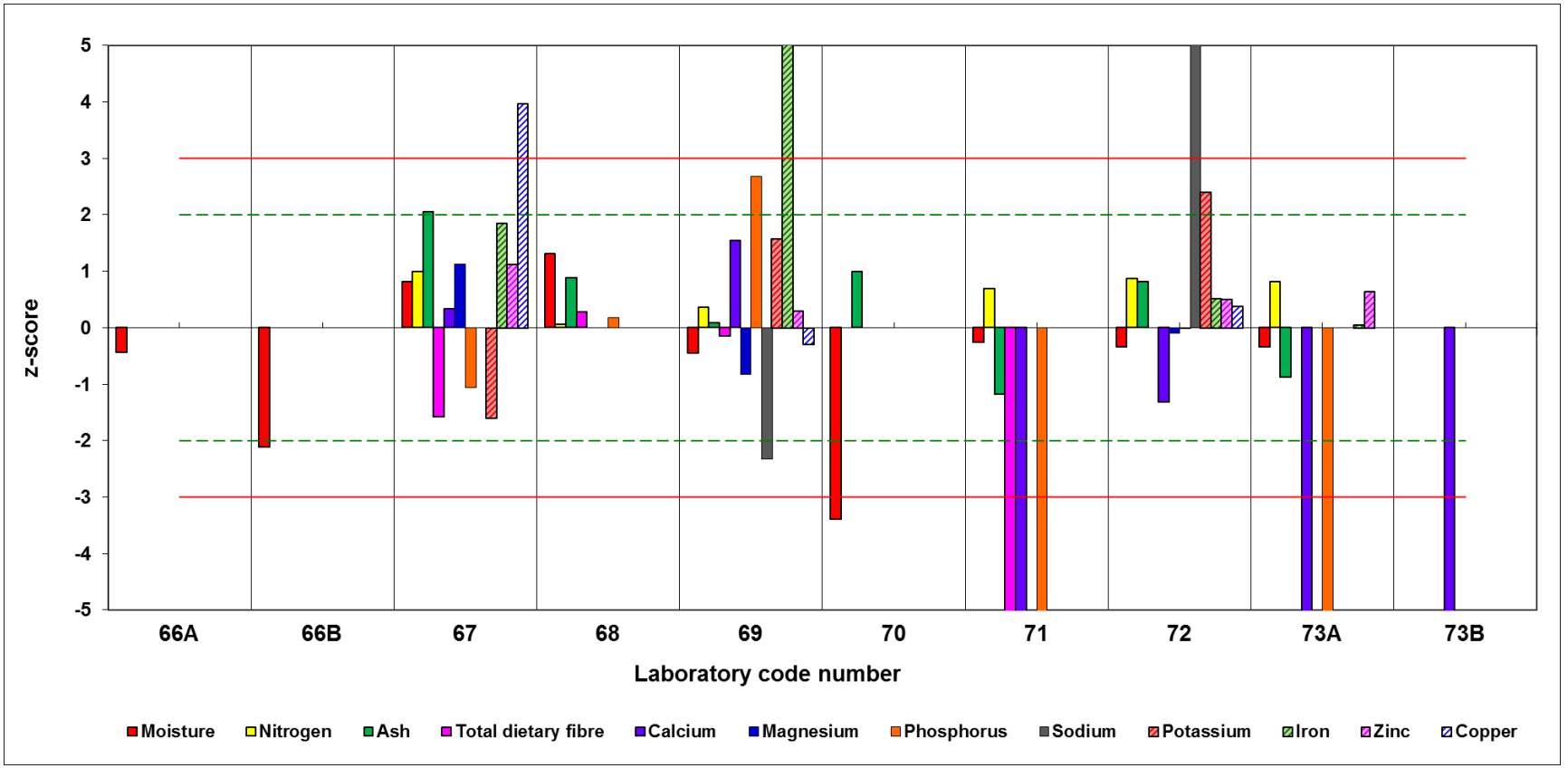

Figure 55. Bar-chart of z-scores for APFAN PT-2 in which the participants determined all nutrients using defatted soybean flour as test material: laboratory code number 66-73.

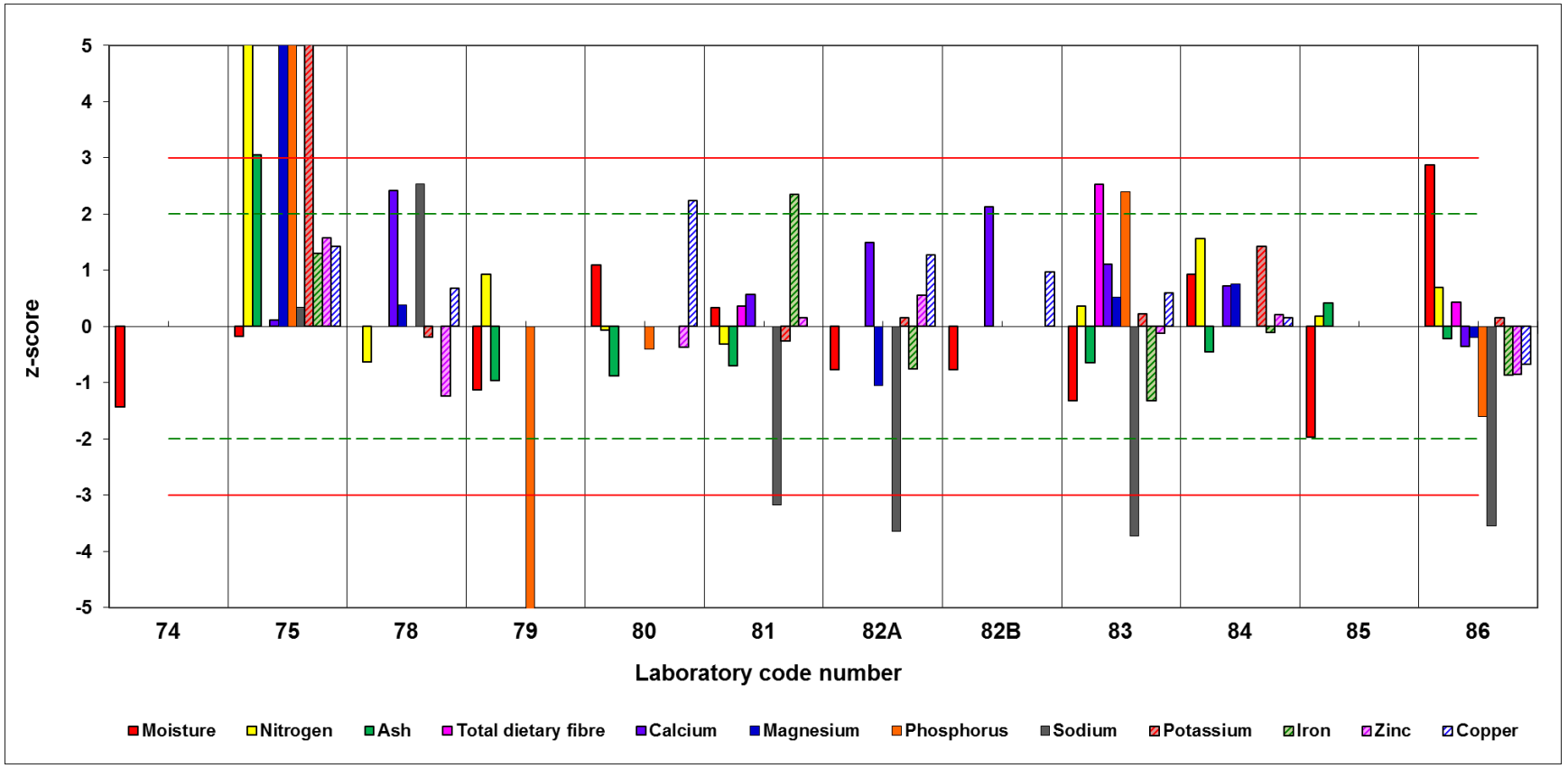

Figure 56. Bar-chart of z-scores for APFAN PT-2 in which the participants determined all nutrients using defatted soybean flour as test material: laboratory code number 74-86.

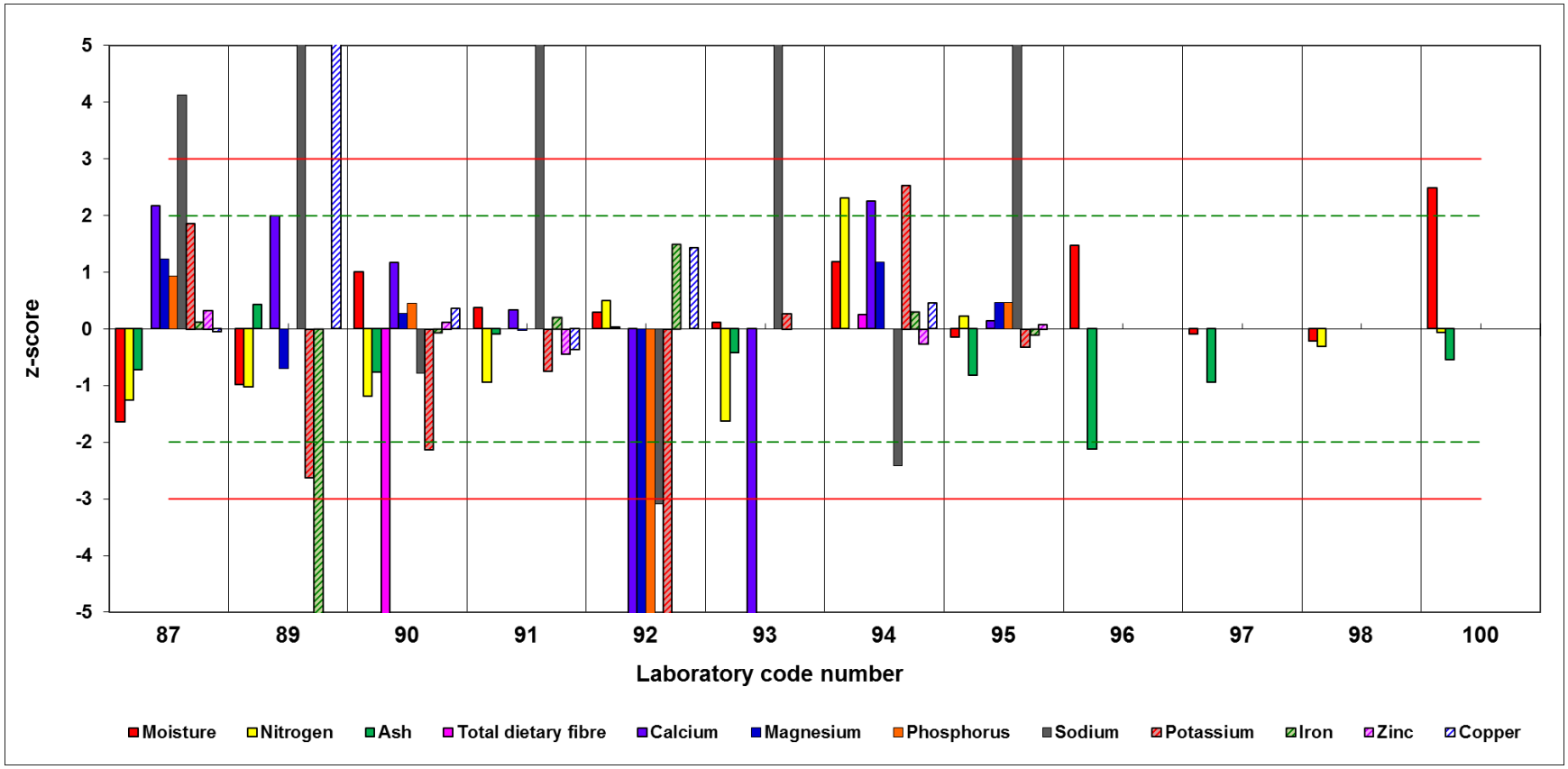

Figure 57. Bar-chart of z-scores for APFAN PT-2 in which the participants determined all nutrients using defatted soybean flour as test material: laboratory code number 87-100.

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