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):

Rice flour (natural)

by

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June, 2019

Table 37. Summary: assigned values of measurands for evaluation of testing parameters in rice 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*	12.92	0.66	5.1	0.11	0.20	Yes, use z score
Nitrogen (g/100g)	x* & s*	1.15	0.04	3.5	0.01	0.01	Yes, use z score
Ash (g/100g)	x* & SD_p	0.32	0.02	6.2	0.003	0.005	Yes, use z score
Dietary fibre (g/100g)	x* & $3SD_p$	1.23	0.14	11.4	0.03	0.04	Yes, use z score
Calcium (mg/kg)	x* & $2SD_p$	94.40	15.23	16.1	2.25	4.57	Yes, use z score
Phosphorus (mg/kg)	x* & s*	800.56	140.47	15.1	29.95	42.14	Yes, use z score
Sodium (mg/kg)	x* & $2SD_p$	319.11	42.87	13.4	6.54	12.86	Yes, use z score
Potassium (mg/kg)	x* & $2SD_p$	158.24	23.63	14.9	3.69	7.09	Yes, use z score
Iron (mg/kg)	x* & $2SD_p$	6.40	1.55	24.2	0.26	0.46	Yes, use z score
Zinc (mg/kg)	x* & SD_p	11.88	1.31	11.0	0.27	0.39	Yes, use z score
Copper (mg/kg)	x* & SD_p	1.77	0.26	14.7	0.06	0.08	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

Table 38. Evaluation of laboratory performance on **moisture** analysis (g/100 g, as received) in rice flour

Lab Number	Moisture g/100g	MU g/100g	z score	Zeta score	Sample weight (g)	Temperature (°C)	Time (Hours)	Method Reference
Assigned value obtained from robust average (x) ± robust SD (s*) = 12.92 ± 0.66 g/100 g (CV 5.1%, n= 59)*								
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00				
3	**11.88**	0.21	-1.57	**-6.91**	5.000 ± 0.001	105	18	AOAC International (2001)
7	**12.40**	0.10	-0.79	**-4.30**	1.0306/1.1313	105	5	AOAC
10	**13.68**	-	1.15	-	2	130	1	AOAC 2012, 32.2.09 A, Chapt 32
14	**13.40**	-	0.73	-	2	130 ± 3	1	AOAC 925.10
15	**13.50**	-	0.88	-	2	130	1	AOAC (2016) 925.10
16	**12.30**	0.08	-0.94	**-5.30**	1 to 2	105	3	SNI 01-2891-1992 Food & Beverage
18	**12.90**	-	-0.03	-	2.0	105	3	SNI 01-2891-1992
19	**12.95**	-	0.05	-	5	105	3	AOAC 934.01
25	**11.80**	-	-1.70	-	5.1390 / 5.1625	103	4	Laboratory Handbook of Methods of Food Analysis, 3rd Ed, R. Lees
26	**13.00**	0.08	0.12	0.68	2.0	100	5	AOAC No. 925.09B
28	**13.10**	0.77	0.27	0.45	2.0	130	1	AOAC 925.10 (Air Oven Method)
29	**13.26**	0.06	0.52	**2.99**	5	105	16	AOAC (1984)
31	**12.85**	0.45	-0.11	-0.28	3	105	3	SNI
32	**13.60**	0.31	1.03	**3.58**	1.100	130	3	AOAC 925.10
36	**13.40**	0.10	0.73	**3.97**	2.00	130	1	AOAC 925.10 18th Ed

Lab Number	Moisture g/100g	MU g/100g	z score	Zeta score	Sample weight (g)	Temperature (°C)	Time (Hours)	Method Reference
Assigned value obtained from robust average (x) ± robust SD (s*) = 12.92 ± 0.66 g/100 g (CV 5.1%, n= 59)*								
38	**12.50**	0.21	-0.64	**-2.79**	2.000	130	1 until constant	AOAC 925.10, 19th Ed 2012
39	**13.50**	-	0.88	-	2	130 ± 3	1	AOAC 925.10
41	**13.46**	0.11	0.82	**4.40**	2	135 ± 2	2	AOAC (2016) 930.15
42	**13.20**	0.22	0.42	1.80	2	130	1	SNI 3549:2009 Lampiran A.10
43	**13.75**	0.22	1.26	**5.34**	2	130	2 to constant weight	AOAC, National Standard
44	**13.20**	0.20	0.42	1.88	2.0314	130	1.0	AOAC 19th Ed, 2012
48	**13.48**	0.13	0.85	**4.39**	2	130	1	SNI 3549 2009
49	**13.32**	0.67	0.61	1.13	2	130	1	AOAC 20th Ed 2016
50	**13.60**	0.23	1.03	**4.30**	2.1804	130	1.0	AOAC 925.10
52	**13.77**	0.12	1.29	**6.76**	2.0	130	1.0	AOAC 925.10
54	**12.40**	0.10	-0.79	**-4.30**	1	105	5	AOAC 927.05
57	**13.40**	0.18	0.73	**3.39**	2.0059	130	5	AOAC 925.10
58	**13.40**	0.89	0.73	1.05	2 to 5	130 / 105	3	Based on AOAC 20th Ed 2016
59	**11.37**	0.01	**-2.35**	**-14.08**	1 to 2	105	3	SNI 01-2891-1992 point 5.1
60	**12.67**	-	-0.38	-				SNI 01-2891-1992 Butir 5.1
61	**13.20**	2.00	0.42	0.28	3	130	1.5	A6801 130C Air oven
64	**13.49**	0.10	0.86	**4.71**	2.0216	130	1	AOAC 925.10

Lab Number	Moisture g/100g	MU g/100g	z score	Zeta score	Sample weight (g)	Temperature (°C)	Time (Hours)	Method Reference
Assigned value obtained from robust average (x) ± robust SD (s*) = 12.92 ± 0.66 g/100 g (CV 5.1%, n= 59)*								
66A	**10.30**	0.50	**-3.97**	**-9.59**	10.0016	130.0	0.50	AOCS Official Method Ca 2c-25, 7th Ed., 2017
66B	**11.00**	0.20	**-2.91**	**-12.92**	10.0032	130.0	0.50	AOCS Official Method Ca 2c-25, 7th Ed., 2017
66C	**11.20**	0.30	**-2.61**	**-9.25**	10.0000	130.0	0.50	AOCS Official Method Ca 2c-25, 7th Ed., 2017
69	**12.60**	-	-0.48	-	-	-	-	-
71	**12.70**	0.40	-0.33	-0.97	4.9991, 4.9956	105	3	AOAC 925.45
72	**13.10**	0.01	0.27	1.63	2	130	1	AOAC 925.10
73A	**12.87**	0.51	-0.08	-0.18	5	105	3	FTC-01.01 (refers to AOAC 945.39)
74	**11.79**	-	-1.71	-	5.0	105	3	SNI 01-2891-1992 (part 5.1)
75	**10.61**	0.09	**-3.50**	**-19.61**	2	105 ± 2	4	SNI 01-2891-1992 Butir 5.1
76	**13.65**	0.36	1.11	**3.46**	-	-	-	-
77	**13.44**	-	0.79	-	-	-	-	-
78	**13.17**	1.20	0.38	0.41	2	100	1, to constant	AOAC 19th Ed
79	**12.79**	0.83	-0.20	-0.31	1 to 2	105	3	SNI 01-2891-1992 Butir 5.1
81	**13.20**	0.10	0.42	**2.32**	2.0629 mean	130	1 then 0.5 until <0.005	AOAC 925.10
82A	**12.80**	0.12	-0.18	-0.96	1.00	105	7.5	Drying Oven
82B	**12.80**	0.12	-0.18	-0.96	1.00	105	7.5	Drying Oven
84	**13.63**	-	1.08	-	2	130	1	SNI 3549.2009
85	**12.24**	0.04	-1.03	**-6.08**	2	105	3	SNI 01-2896-1992

Lab Number	Moisture g/100g	MU g/100g	z score	Zeta score	Sample **weight** (g)	Temperature (^{o}C)	Time (Hours)	Method Reference
Assigned value obtained from robust average (x) ± robust SD (s*) = 12.92 ± 0.66 g/100 g (CV 5.1%, n= 59)*								
88	**10.85**	0.03	**-3.13**	**-18.62**	1.5	105	3	SM 01-2891-1992 Point 5.1
89	**12.50**	0.22	-0.64	**-2.72**	2	100 to 105	4	AOAC 925.23
91	**13.40**	0.01	0.73	**4.36**	-	-	-	-
93	**12.71**	-	-0.32	-	2	130 ± 3	1	AOAC 925.10
94	**13.49**	-	0.86	-	1	130	5	AOAC (2000) 925.10
95	**12.83**	0.07	-0.14	-0.82	-	-	-	-
96	**13.50**	-	0.88	-	3	125	4	TCVN 4846:1989
98	**12.60**	-	-0.48	-	~2.0	100 ± 5	5	AOAC 930.15
99	**12.30**	1.03	-0.94	-1.18	3.00 ± 0.02	100 to 102	Min 8 and repeat until constant weight	AOAC 16th Ed 950.46

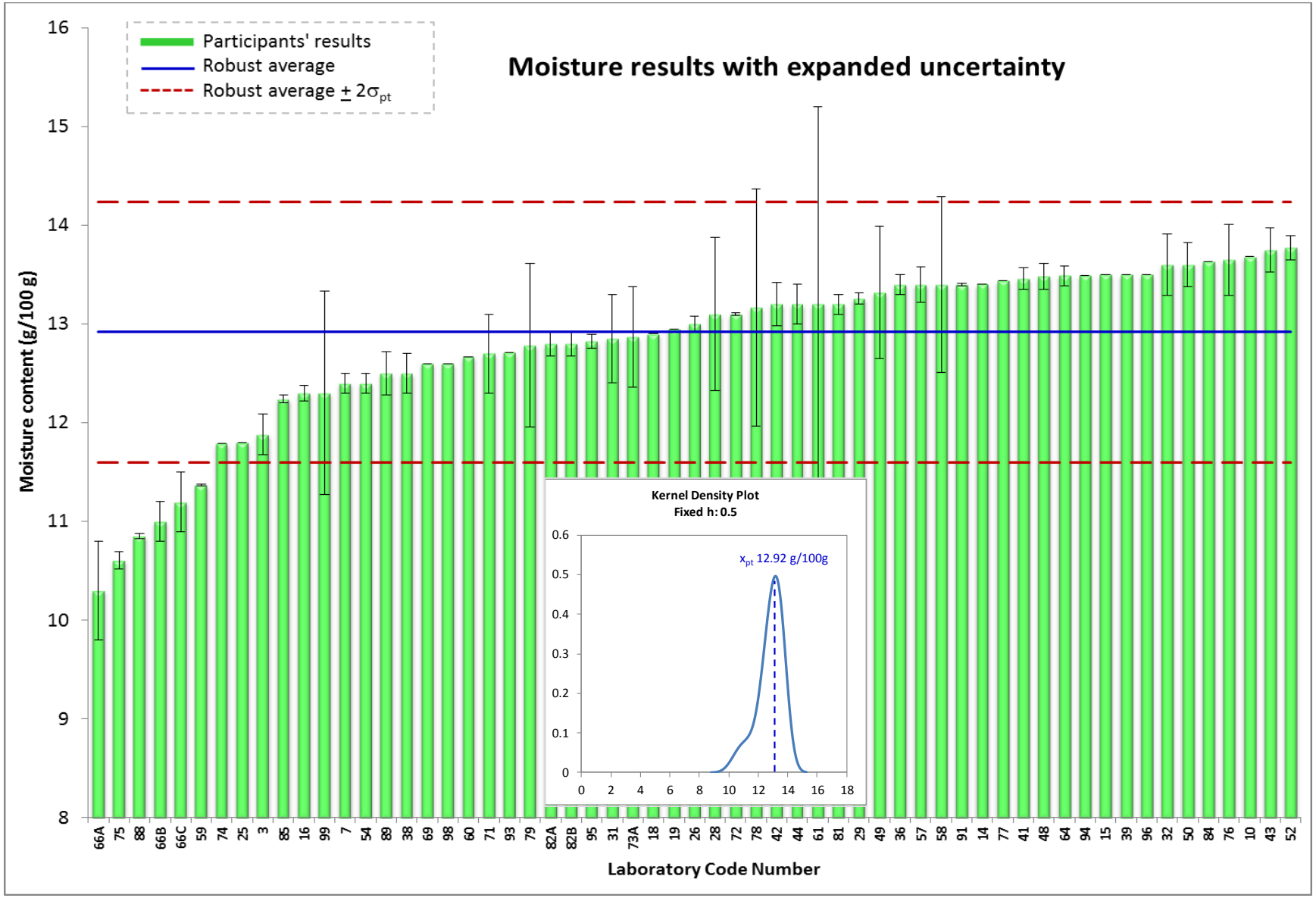

Figure 106. Distribution of **moisture** results (ascending order) in rice flour with expanded uncertainty

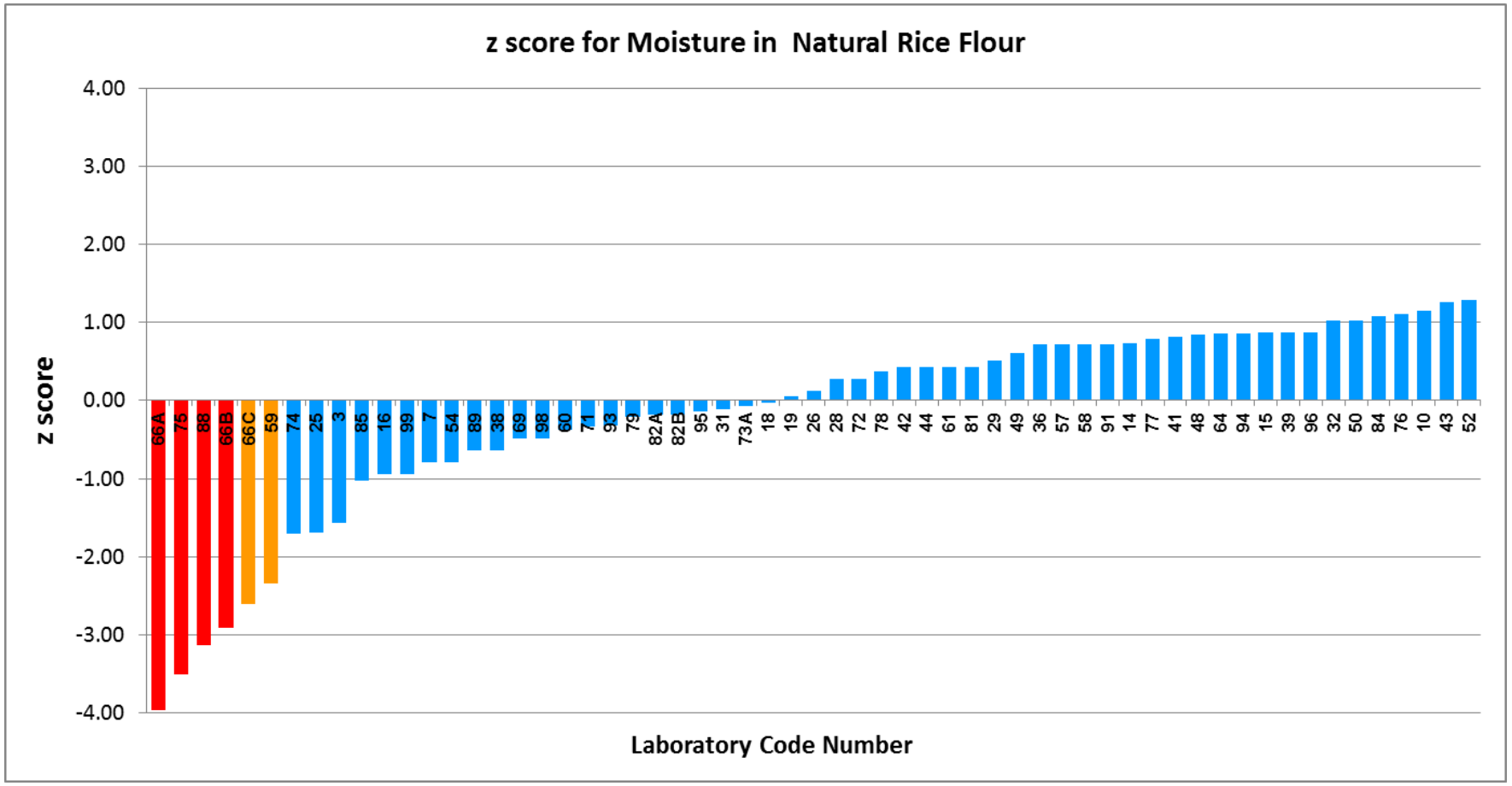

Figure 107. Plot of ordered *z* scores for **moisture** results in rice flour

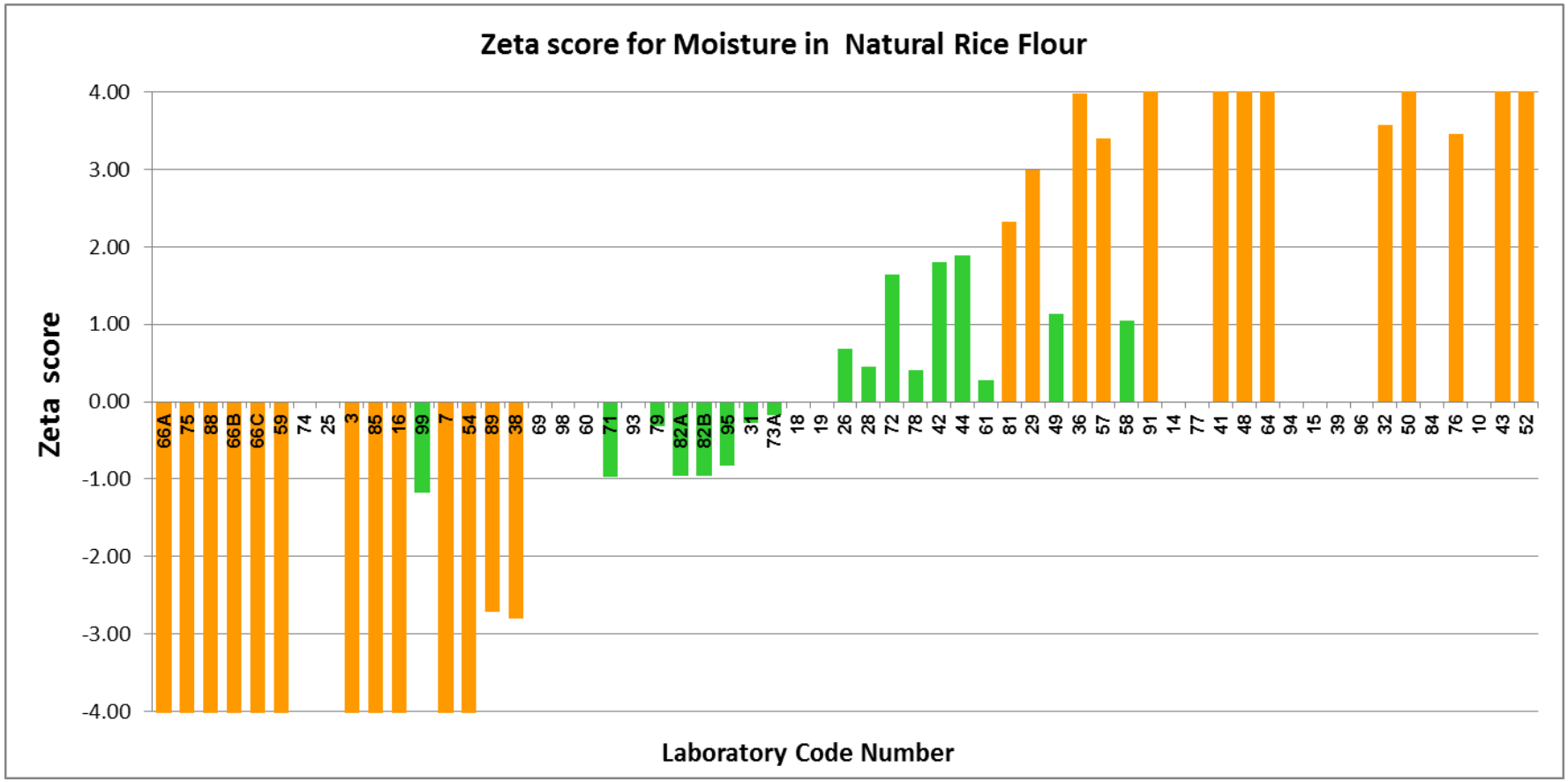

Figure 108. Plot of *Zeta* score for **moisture** rice flour, following the ordered z scores in the above Figure 107.

Table 39. Evaluation of laboratory performance on **total nitrogen** analysis (g/100 g, as received) in rice flour

Lab Number	Total Nitrogen (g/100g)	MU (g/100g)	z score	Zeta score	Sample Weight (g)	Catalyst	Acid Volume (mL)	Receiver Solution	Titrant	Conversion Factor	Method Reference
Assigned value obtained from robust average (x) ± robust SD (s*) = 1.15 ± 0.04 g/100 g (CV 3.5%, n= 48) with u_{xpt} 0.01 g/100g*											
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00							
3	**1.11**	0.03	-1.03	**-2.17**	2.000 ± 0.001	CaSO4, CuSO4	12 mL H2SO4	25 mL 4% Boric Acid	0.2 M HCl	-	AOAC International (2001)
7	**0.8**	-	**-8.75**	-	1.0006/1.0116	Kjeltab	25	25	HCL/0.1097	-	AOAC
10	**1.15**	-	0.00	-	1	Selenium mixture	25 mL conc H2SO4	50 mL 4% H3BO4	HCl 0.1 M	-	AOAC 2012, 32.2.09 C, Chapt 32
14	**1.15**	0.01	-0.05	-0.17	1	K2SO4:CuSO 4.5H2O (9:1)	H2SO4 15 mL	4% Boric Acid 30 mL	0.1 M HCl	-	AOAC 991.20
16	**1.15**	0.12	0.00	0.00	0.5 - 1	Selenium	Sulphuric Acid 25 mL	Boric Acid 50 mL	HCl 0.1 N	-	SNI 01-2891-1992 Food & Beverage
18	**1.32**	-	**4.25**	-	2.0	CuSO4, SeO2	H2SO4, 25 mL	H3BO3 2%, 25 mL	HCl 0.1 M	-	SNI 01-2891-1992
19	**1.16**	-	0.30	-	1	Kjeltabs, 2 pcs	Sulphuric Acid 15 mL	Boric Acid 1%, 15 mL	HCl, 15 mL	6.25	AOAC 988.05, AN300 FOSS 2003
25	**1.15**	-	0.00	-	0.0517 / 0.0511	Copper Sulphate	Digestion Reagent, 10	Indicating Boric Sol'n, 10	0.02 N H2SO4	-	Laboratory Handbook of Methods of Food
26	**1.14**	0.01	-0.25	-0.89	2.0	Copper (II) sulphate	Conc H2SO4, 15	0.1 N HCl	0.1 N NaOH	5.71	AOAC No. 2001.11
28	**1.14**	0.0734	-0.25	-0.26	1.0	CuSO4 / Na2SO4	H2SO4 15 mL	50 mL 0.1 N HCl	0.1 N NaOH	-	Kjeldahl Method
29	**2.16**	0.23	**25.35**	**8.67**	2	CuSO4:K2SO 4	15 mL conc H2SO4	1% boric acid with indicators	0.1 N HCl	-	AOAC (2005)
31	**1.19**	0.03	1.00	**2.22**	0.3	Selenium	H2SO4 (8 mL)	H3BO3 3% (50 mL)	HCl 0.05 N	-	SNI 01-2891
32	**1.10**	0.03	-1.25	**-3.12**	1.3500	K2SO4, CuSO4.5H2O	Conc H2SO4, 25	4% Boric Acid 50 mL	0.099970 M HCl	NA	Block Digestion - Kjeldahl
36	**1.13**	0.03	-0.50	-1.08	0.500	CuSO4	H2SO4 20 mL	Erlenmeyer Flask 500 mL	0.2 M H2SO4	NA	Buchi Nitrogen and Protein Determination
38	**1.12**	0.30	-0.75	-0.20	1.000	Salt mixture	H2SO4 12 mL	4% H3BO3, 20 mL	0.2 N HCl	-	AOAC 991.2, 19th Ed 2012
39	**1.14**	-	-0.25	-	0.5	Cu	H2SO4 / 10	Boric acid 30 mL	0.1 M HCl	-	AOAC 991.20

Lab Number	Total Nitrogen (g/100g)	MU (g/100g)	z score	Zeta score	Sample Weight (g)	Catalyst	Acid Volume (mL)	Receiver Solution	Titrant	Conversion Factor	Method Reference
Assigned value obtained from robust average (x) ± robust SD (s*) = 1.15 ± 0.04 g/100 g (CV 3.5%, n= 48) with u_{xpt} 0.01 g/100g*											
41	**1.15**	0.04	-0.07	-0.13	0.5	K2SO4:CuSO4	Sulphuric acid 15 mL	Boric acid 25 mL	0.1000	6.25	AOAC 2001.11
42	**1.12**	0.01	-0.75	**-2.81**	0.5	Selenium	H2SO4	H3BO3 1% 30 mL	HCl 0.1 N	-	SNI 01-2891-1992. point 7.1
43	**1.17**	0.08	0.50	0.49	1	Mix selenium	H2SO4, 12 mL	H3BO3, 25 mL	HCl 0.2 M	1.4007	National Standard, inhouse method
44	**1.15**	0.01	0.01	0.04	2.0120	Na2SO4, CuSO4	Conc H2SO4 20 mL	50 mL 0.1 N H2SO4	0.2 N NaOH	6.25	AOAC 19th Ed, 2012
48	**1.19**	0.10	0.88	0.69	1	Selenium Mixture	20	30	0.1	1	MU-01/04
49	**0.99**	0.05	**-4.00**	**-5.94**	1, 2	Kjeltabs	Conc H2SO4 20 mL	Boric Acid 50 mL	0.2 N H2SO4	5.7	AOAC 20th Ed 2016
50	**1.14**	0.02	-0.15	-0.39	1.1567	Cu	H2SO4, 15.0 mL	Boric Acid, 75.0 mL	HCl, 0.0902	Fish M 6.25, Rice F 5.7	AOAC 984.13
52	**1.121**	0.11	-0.72	-0.51	1.0	K2SO4, CuSO4	9.711, 11.164	Boric Acid (60mL)	0.09788 N HCl	5.95	Automated Kjeldahl Method
54	**1.12**	0.10	-0.72	-0.55	1	Kjeltabs	H2SO4 12 mL	25 mL 4% Boric Acid	0.3 M H2SO4	5.95	AOAC 920.87
57	**1.11**	0.11	-1.00	-0.70	1.0064	Kjeldahl Tablet	H2SO4, 10 mL	20 mL Boric Acid	HCl, 0.10 M	6.25	AOAC 979.09
58	**1.17**	0.03	0.50	1.11	0.5	-	-	Boric Acid	0.25 HCl	1.0	Based on AOAC 20th Ed 2016
60	**1.17**	-	0.50	-	-	-	-	-	-	-	AOAC (2012) 2011.11
61	**1.14**	0.05	-0.25	-0.37	1	K2SO4/CuSO4 Kjeltab	H2SO4 20 mL	Boric Acid 50 mL	0.1 M HCl	6.25	A6501 Kjeldahl/Boric Acid Method
69	**1.105**	-	-1.13	-	-	-	-	-	-	-	-
71	**1.18**	0.04	0.75	1.47	1.0050, 1.0048	Kjeltabs 3.5 g, K2SO4 0.4 g,	H2SO4 15 mL	Boric acid	0.2 N HCl	-	AOAC 2001.11
72	**1.25**	0.05	**2.50**	**3.71**	2	K2SO4, CuSO4, SeO2	25	4% Boric acid 25 mL	0.05 M H2SO4	6.25	AOAC 920.87
73A	**1.18**	0.12	0.75	0.49	1	2 Kjeltabs (each 3.5 g	H2SO4 15 mL	1% Boric acid, 1% BCG 0.1%	HCl 0.2 M	6.25	FTC-02.01 (refers to AOAC 2001.11, 979.09)
75	**< 0.100**	-	**-26.25**	-	0.5	CuSO4	H2SO4, 5 mL	H3BO3, 20 mL	HCl, 0.1 M	N/A (report as Nitrogen)	SNI 01-2891-1992 Butir 7.1

Lab Number	Total Nitrogen (g/100g)	MU (g/100g)	z score	Zeta score	Sample Weight (g)	Catalyst	Acid Volume (mL)	Receiver Solution	Titrant	Conversion Factor	Method Reference
Assigned value obtained from robust average (x) ± robust SD (s*) = 1.15 ± 0.04 g/100 g (CV 3.5%, n= 48) with u_{xpt} 0.01 g/100g*											
76	**1.2**	0.01	1.25	**4.47**	-	-	-	-	-	-	-
77	**1.27**	-	**3.00**	-	-	-	-	-	-	-	-
78	**1.13**	0.10	-0.50	-0.39	2	Kjeltabs	Conc H2SO4, 15	4% Boric acid, 25 mL	0.1 N HCl	-	AOAC 19th Ed
79	**1.15**	0.04	0.00	0.00	0.1 to 0.15	-	-	-	-	-	IK/02/5.4.1/LDITP/Analisis Protein
81	**1.12**	0.01	-0.75	**-2.68**	1.0324 mean	K2SO4 and CuSO4.5H2O	20 mL H2SO4	60 mL 2% Boric Acid soln	0.09597 N HCl	5.95	Automated Kjeldahl Method
84	**1.22**	-	1.75	-	1	KJELCAT 12-0328	H2SO4 98% 20 mL	H3BO3 4%, 60 mL	HCl 0.1 M	-	KJELDAHL
85	**1.03**	0.01	**-3.00**	**-10.73**	0.2	-	-	-	-	-	DuMaster Protein Analyzer (Buchi)
88	**1.14**	0.10	-0.35	-0.27	0.5	Tablet Kjeldahl	Conc H2SO4 10 mL	H3BO3 2% 50 mL	HCl 0.0515 N	-	SM 3751-2009
89	**1.05**	0.05	**-2.40**	**-3.39**	0.5	CuSO4	HCl	25 mL Boric Acid	0.1 N HCl	-	AOAC 991.2
91	**1.13**	0.05	-0.50	-0.74	-	-	-	-	-	-	
93	**1.17**	-	0.50	-	2	H2O2 5 mL, Kjeltabs: 3.5 g K2SO4, 0.4 g CuSO4,5H2O	H2SO4 12 mL	Boric acid 25 mL	0.05 N H2SO4	5.95	AOAC 945.18-B
94	**1.16**	-	0.35	-	1	CuSO4.5H2O and K2SO4	Conc H2SO4 / 13 mL	1% Boric acid	0.1 M HCl	-	AOAC (2012) 991.20
95	**1.15**	0.11	0.00	0.00	-	-	-	-	-	-	
98	**1.17**	-	0.50	-	~1.0	7g K2SO4, 0.8 g CuSO4	15 mL H2SO4	30 mL 4% Boric Acid	0.2 N HCl	-	AOAC 976.05
99	**1.25**	0.02	**2.50**	**7.07**	1.00 ± 0.02	Kjeltab Cu 3/5	12	1% Boric Acid	0.1 M HCl	-	AOAC 16th Ed 981.10

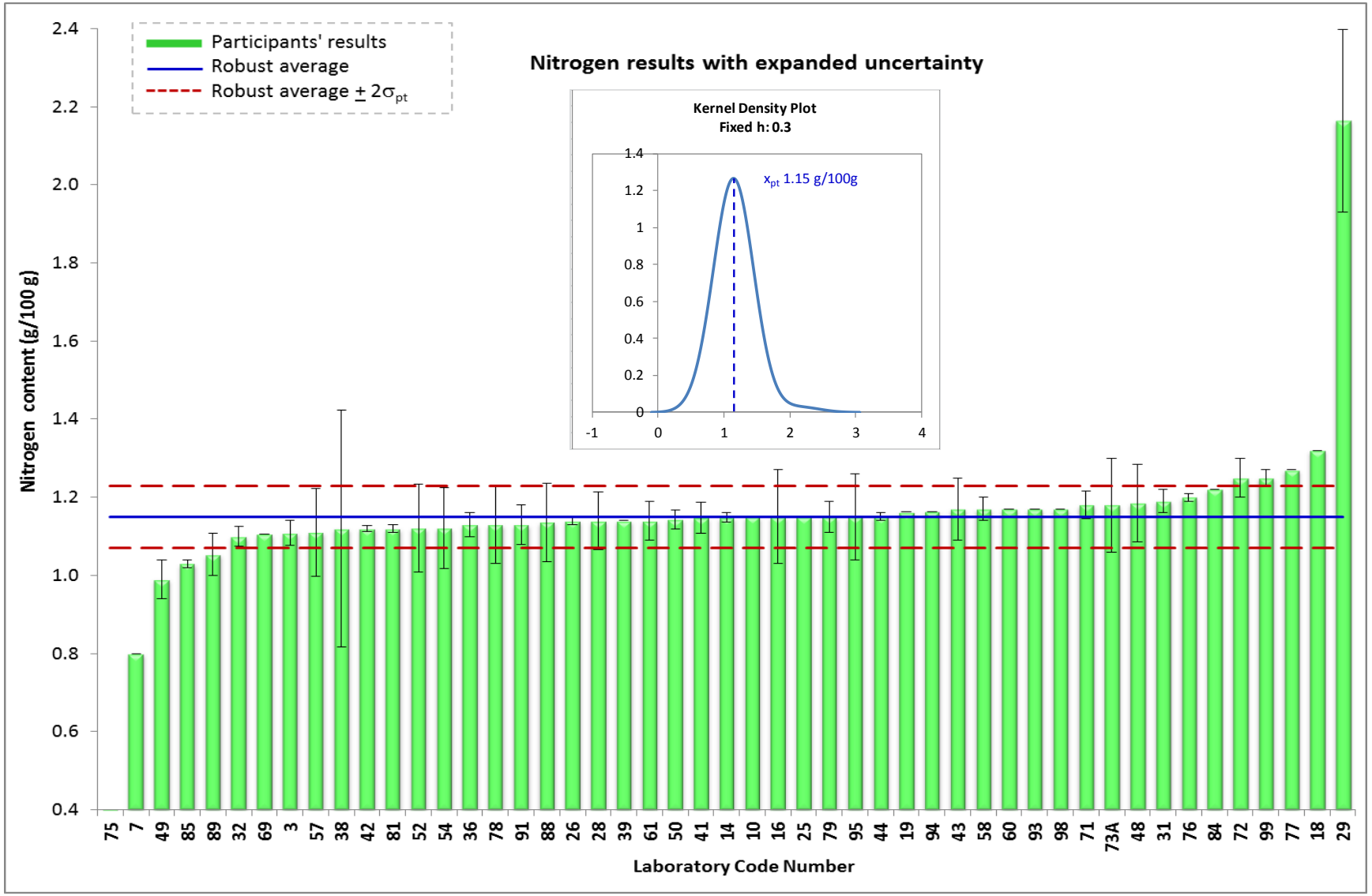

Figure 109. Distribution of **total nitrogen** results (ascending order) in rice flour with expanded uncertainty

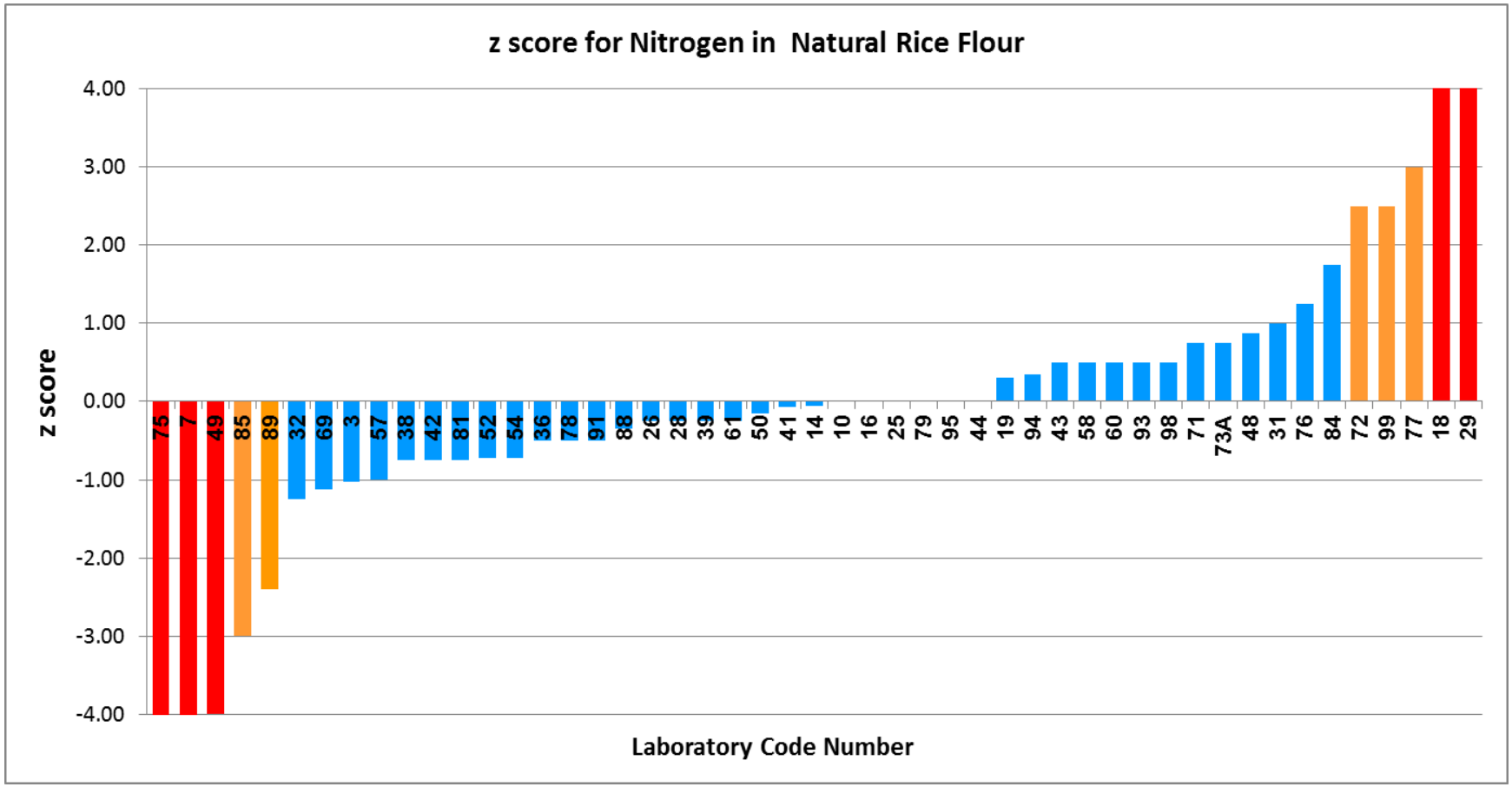

Figure 110. Plot of ordered *z* scores for **total nitrogen** results in rice flour

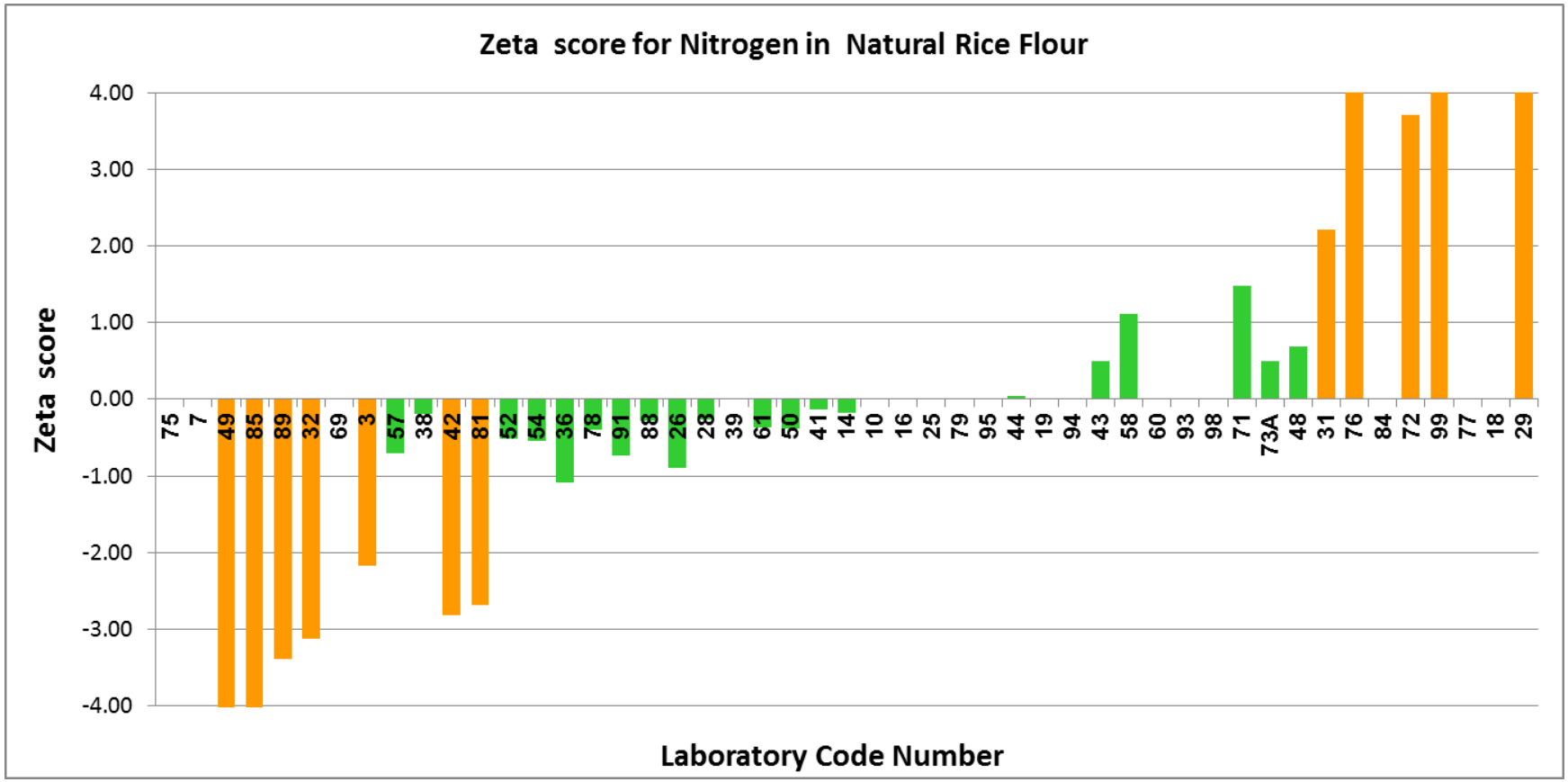

Figure 111. Plot of *Zeta* score for **total nitrogen** in rice flour, following the ordered z scores in the above Figure 110.

Table 40. Evaluation of laboratory performance **dietary fibre** analysis (g/100g, as received) in rice flour

Lab Number	Dietary fibre (g/100g)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Method Reference
Assigned value obtained from robust average (x) ± 3SDp from Horwitz's equation = 1.23 ± 0.15 g/100g (CV 12.2%, n= 20) with u_{xpt} 0.03 mg/kg*								
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00				
14	**0.48**	-	**-4.97**	-	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	**3.17**	0.32	**12.93**	**11.92**	1	-	-	AOAC 985.29
19	**0.11**	-	**-7.47**	-	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
29	**6.85**	0.11	**37.48**	**89.74**	0.5	Enzymatic-Gravimetric Method	MES-TRIS Buffer	AOAC (2005)
31	**0.33**	-	**-6.00**	-	-	-	-	-
32	**1.42**	0.05	1.27	**5.03**	1.150	Enzymatic Gravimetric	Buffer	AOAC 991.42
38	**2.35**	0.40	**7.47**	**5.50**	1.000	Enzymatic Digestion	Phosphate buffer	AOAC 985.29 19th Ed 2012
39	**0.55**	-	**-4.51**	-	1	Enzyme	Buffer solution	AOAC 985.29
43	**7.35**	0.73	**40.80**	**16.73**	1	-	-	AOAC
48	**0.93**	0.15	**-2.02**	**-3.75**	1	Enzymatic	Amylase, protease, amyloglucosidase	AOAC 985.29 19th Ed 2012
49	**1.65**	0.00	**2.80**	**14.00**	0.5	Enzymatic	Alpha-Amylase, Protease, Amyloglucosidase	AOAC 20th Ed 2016 / Sigma Kit
54	**0.98**	0.06	-1.67	**-6.02**	1	Enzymatic	Phosphate buffer / Enzyme	AOAC 985.29
58	**4.55**	1.94	**22.13**	**3.42**	1.0	-	-	Based on AOAC 20th Ed 2016

Lab Number	Dietary fibre (g/100g)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Method Reference
Assigned value obtained from robust average (x) ± 3SDp from Horwitz's equation = 1.23 ± 0.15 g/100g (CV 12.2%, n= 20) with u_{xpt} 0.03 mg/kg*								
59	**0.29**	0.01	**-6.27**	**-30.91**	1 to 2	Enzymatic	-	AOAC 18th Ed 985.29
61	**1.10**	0.15	-0.87	-1.57	0.5	Enzymatically Digested with protease and amyloglucosidase	Methylated spirits	A6234 (ANKOM automated TDF instrument)
69	**8.572**	-	**48.95**	-	-	-	-	-
81	**2.31**	0.19	**7.20**	**10.84**	mean 0.9985	Enzymatic Digestion (Heat-stable alpha-amylase, protease, amyloglucosidase)	MES-TRIS Buffer	AOAC 991.43 (Modified)
88	**0.42**	-	**-5.40**	-	1	Enzymatic	Buffer Phosphat, HCl & NaOH	In house method (enzymatic)
94	**1.02**	-	-1.40	-	1	-	-	AOAC (2012) 985.29
99	**1.52**	0.12	1.93	**4.32**	1.0000 ± 0.005	Enzymetic Technique	MES-TRIS	AOAC 16th Ed 991.43

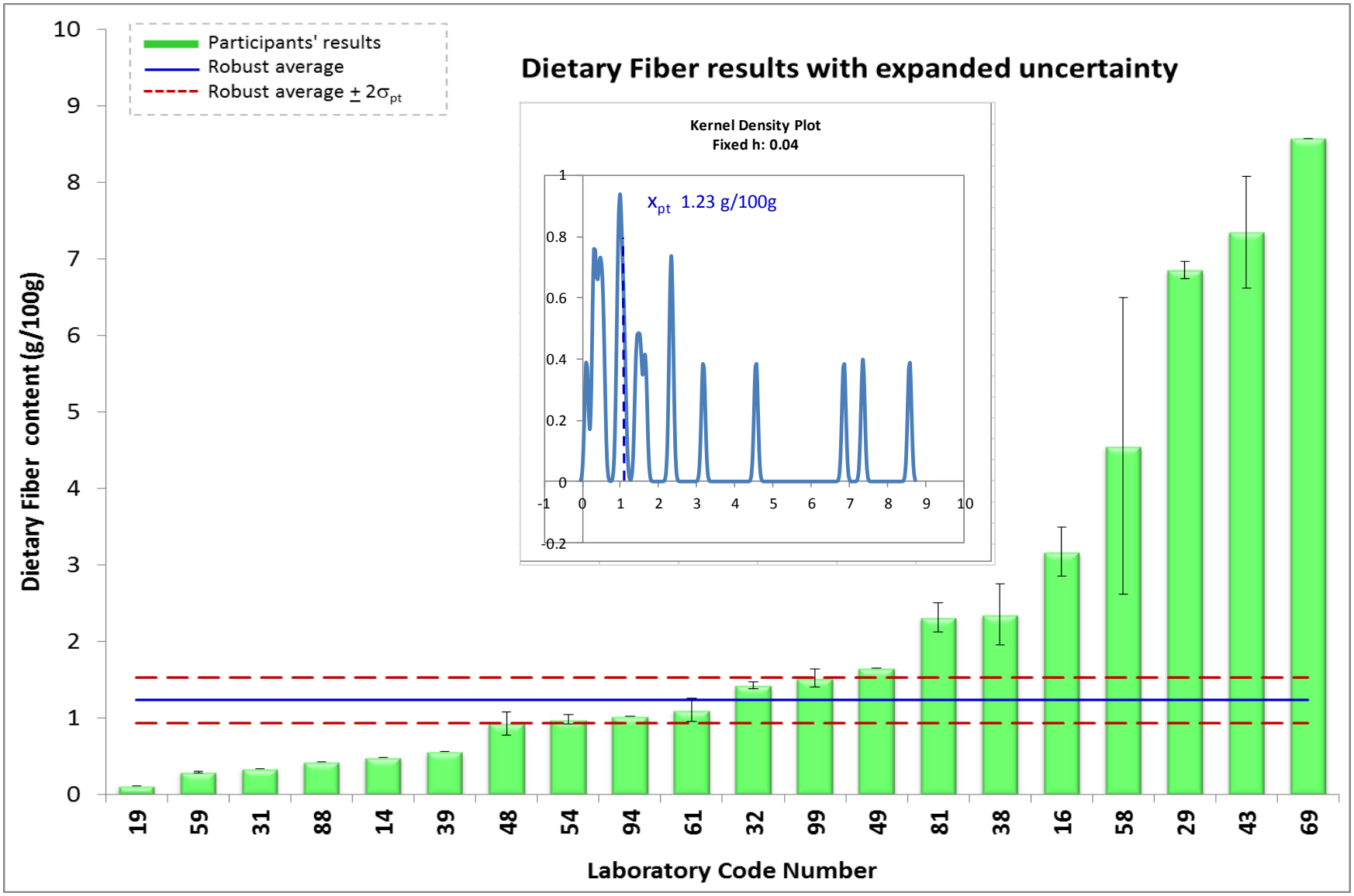

Figure 112. Distribution of **dietary fibre** results (ascending order) in rice flour with expanded uncertainty

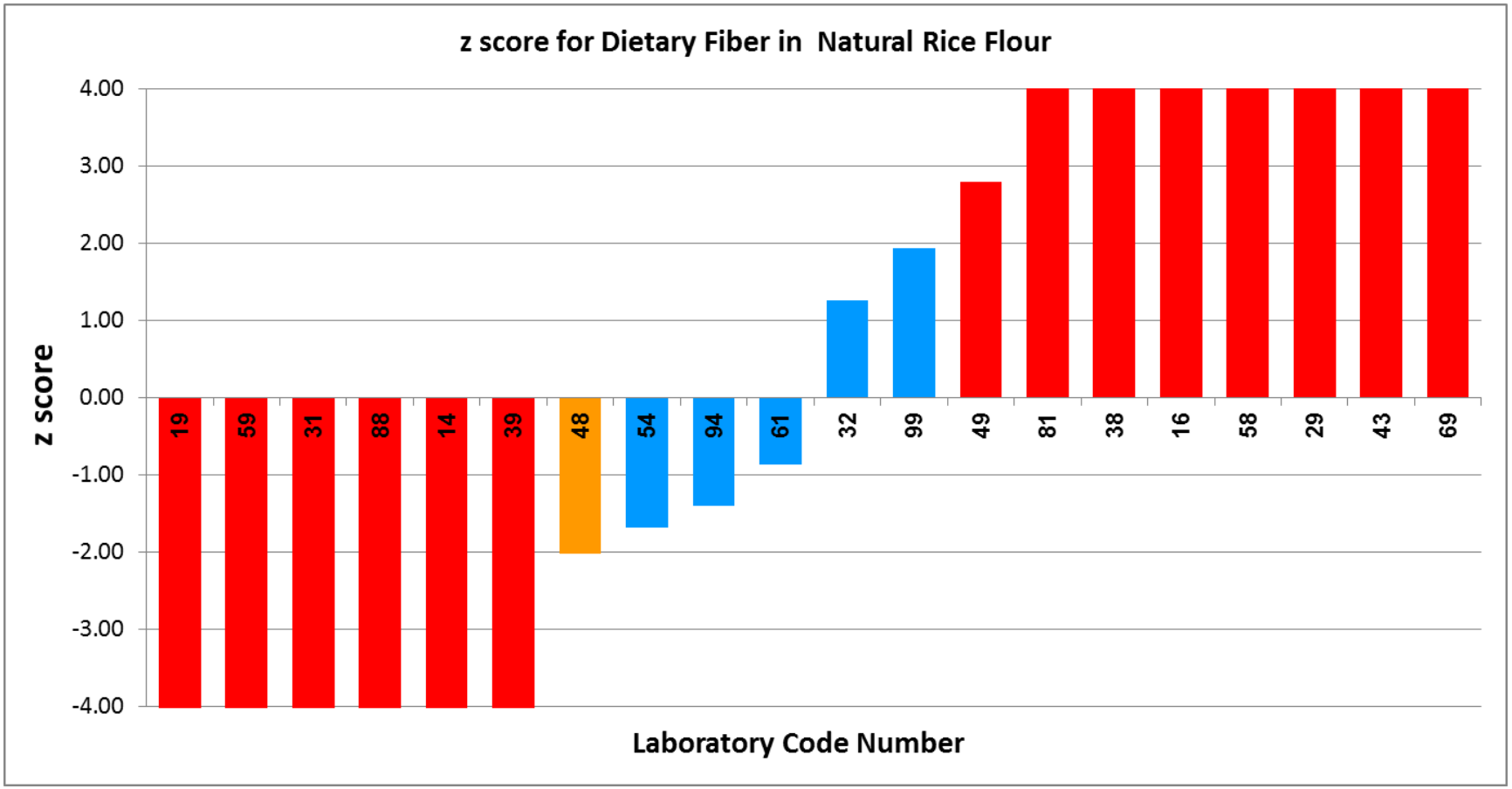

Figure 113. Plot of ordered *z* scores for **dietary fibre** results in rice flour

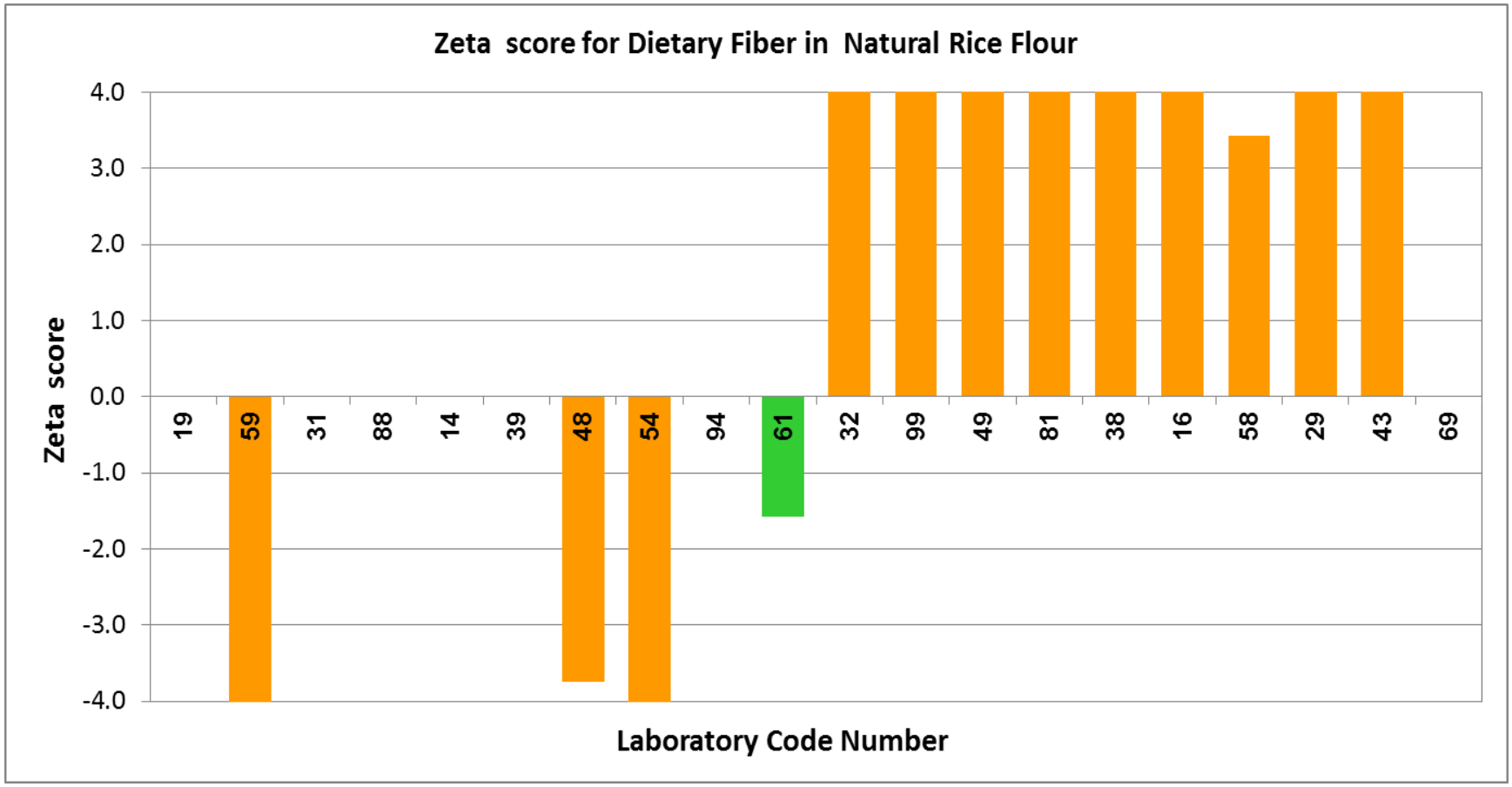

Figure 114. Plot of *Zeta* score for **dietary fibre** in rice flour, following the ordered z scores in the above Figure 113.

Table 41. Evaluation of laboratory performance on **ash** analysis (g/100 g, as received) in rice flour

Lab Number	Ash (g/100g)	MU (g/100g)	z score	Zeta score	Sample Weight (g)	Pre-Charring (Y/N)	Ash Temperature (°C)	Ash Time (hours)	Ash Reference
Assigned value obtained from robust average (x) ± SDp from Horwitz' s equation = 0.32 ± 0.02 g/100 g (CV 6.2%, n= 49) with u_{xpt} 0.01 g/100g*									
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00					
3	**0.30**	0.00	-1.00	-1.96	5.000 + 0.001	On Hotplate until black	550	18	AOAC International (2001) modified
7	**0.50**	0.00	**9.00**	**18.00**	1.0039/1.0316	None	550	5 or until white ash	AOAC
10	**0.32**	-	0.00	-	2	-	600	2	AOAC 2012, 32.2.09 B, Chapt 32
14	**0.32**	0.01	0.10	-	5	Charring	559	5	AOAC 923.03
16	**0.34**	0.01	1.00	1.79	2 to 3		550	8	SNI 01-2891-1992 Food & Beverage
18	**0.20**	-	**-6.00**	-	2.0	Charring	550	4	SNI 01-2891-1992
19	**0.26**	-	**-3.25**	-	1	-	600	3	AOAC 942.05
25	**0.37**	-	**2.40**	-	5.0202 / 5.0205	Addition of HNO3	550	8	Laboratory Handbook of Methods of Food Analysis, 3rd Ed, R. Lees
26	**0.31**	0.00	-0.40	-0.80	4.0	Drying at 150°C	525	24	AOAC No. 923.03
28	**0.33**	0.10	0.40	0.16	2.0	Charring	550	4	AOAC Method 923.05 (Direct Method)
29	**0.35**	0.03	1.50	1.74	3	Charring	550	16	AOAC (1984)
31	**0.45**	0.02	**6.50**	**9.19**	3	-	600	10	SNI 01-2891
32	**0.34**	0.14	1.10	0.30	1.1000	Charring	550	8	AOAC 923.03
36	**0.30**	0.04	-0.90	-0.77	3.00	Charring	550	8	AOAC 923.10 18th Ed
38	**0.30**	0.02	-1.15	-1.56	2.000	Charring	550	2 until light grey	AOAC 923.03, 19th Ed 2012
39	**0.29**	-	-1.30	-	2	Charring	550	5 to 6	AOAC 923.03

Lab Number	Ash (g/100g)	MU (g/100g)	z score	Zeta score	Sample Weight (g)	Pre-Charring (Y/N)	Ash Temperature (°C)	Ash Time (hours)	Ash Reference
Assigned value obtained from robust average (x) ± SDp from Horwitz' s equation = 0.32 ± 0.02 g/100 g (CV 6.2%, n= 49) with u_{xpt} 0.01 g/100g*									
41	**0.30**	0.02	-0.90	-1.44	2	-	600	2	AOAC
42	**0.30**	0.01	-1.00	-1.76	3	-	550	5	SNI 3549:2009 Lampiran A.11
43	**0.51**	0.03	**9.35**	**9.91**	2	Charring	550	3 to constant	National Standard
44	**0.34**	0.17	0.85	0.20	1.000	Charring	520	8.0	AOAC 19th Ed, 2012
48	**0.30**	0.05	-1.25	-0.93	3	Charring	550	3 then 1 then 1	SNI 3549 2009
49	**0.26**	0.02	**-3.00**	**-4.24**	1, 3	Charring	555	6	AOAC 20th Ed 2016
51	**0.30**	0.02	-0.95	-1.47	3.00	Charring	550	To grey ash + contsant	AOAC 923.03
52	**0.37**	0.06	**2.65**	1.59	3.0	Charring	550	8	AOAC 923.03
54	**0.53**	0.08	**10.60**	**5.08**	1	Charring	525	5	AOAC 92.03
58	**0.32**	0.01	-0.10	-0.18	3.0	-	550	8	Based on AOAC 20th Ed 2016
59	**0.34**	0.03	1.00	1.11	2 to 3	-	550	15	SNI 01-2891-1992 point 6.1
60	**0.30**	-	-1.00	-	-	-	-	-	SNI 01-2891-1992 Butir 6
61	**0.31**	0.01	-0.50	-0.90	2	N/A	550	15	A6401 550C Ash
69	**0.528**	-	**10.40**	-	-	-	-	-	-
71	**0.28**	0.08	-2.00	-0.98	1.0039, 1.0018	-	600	3	AOAC 942.05
72	**0.33**	0.05	0.50	0.37	2	Charring	550	4	AOAC 923.03
73A	**0.27**	0.07	**-2.50**	-1.37	1	N	600	3.5	FTC-05.01 (refers to AOAC 942.05)
75	**0.44**	0.02	**6.10**	-	2	-	550	4	SNI 01-2891-1992 Butir 6.1

Lab Number	Ash (g/100g)	MU (g/100g)	z score	Zeta score	Sample Weight (g)	Pre-Charring (Y/N)	Ash Temperature (°C)	Ash Time (hours)	Ash Reference
Assigned value obtained from robust average (x) ± SDp from Horwitz' s equation = 0.32 ± 0.02 g/100 g (CV 6.2%, n= 49) with u_{xpt} 0.01 g/100g*									
76	**0.31**	0.01	-0.50	-	-	-	-	-	-
78	**0.34**	0.10	1.00	0.39	2	Gradual increase of temp	550	1	AOAC 19th Ed
79	**0.27**	0.32	**-2.65**	-0.33	2 to 3	-	550	-	SNI 01-2891-1992 Butir 6.1
81	**0.30**	0.02	-1.00	-	3.0873 mean	Charring	550	10	AOAC 923.03
84	**0.33**	-	0.50	-	2	Charring	550	6	SNI 3549.2009
85	**0.67**	0.07	**17.50**	**9.62**	2	-	550	3	SNI 01-2896-1992
88	**0.30**	0.10	-1.05	-0.40	2.5	-	550	24	SM 01-2891-1997
89	**0.34**	0.00	0.80	1.59	2	Charring on hotplate	550	16	AOAC 930.30
91	**0.32**	0.02	0.20	0.31	-	-	-	-	-
93	**0.44**	-	**6.00**	-	3 to 5	Charring	550	18	AOAC 923.03
94	**0.31**	-	-0.55	-	2	Charring	550	5	AOAC (2012) 945.46
95	**0.21**	0.00	**-5.75**	**-11.44**	-	-	-	-	-
96	**0.35**	-	1.45	-	3	-	550	4	TCVN 8124:2009
98	**0.27**	-	**-2.50**	-	~2.0	N	600	4	AOAC 942.05
99	**0.31**	0.01	-0.65	-1.16	3.00 ± 0.02	Y	550	Until constant	AOAC 16th Ed 923.03

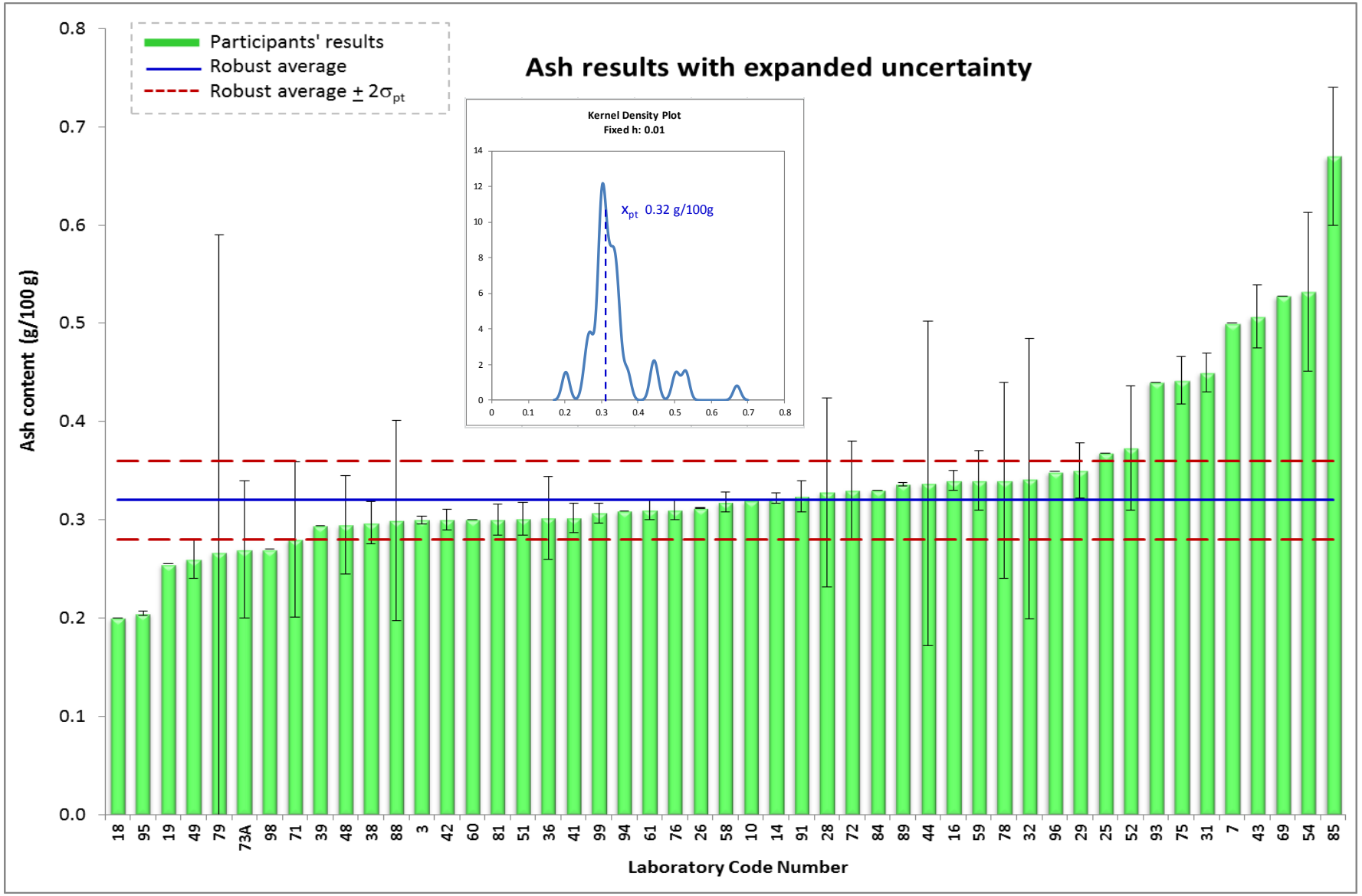

Figure 115. Distribution of **ash** results (ascending order) in rice flour with expanded uncertainty

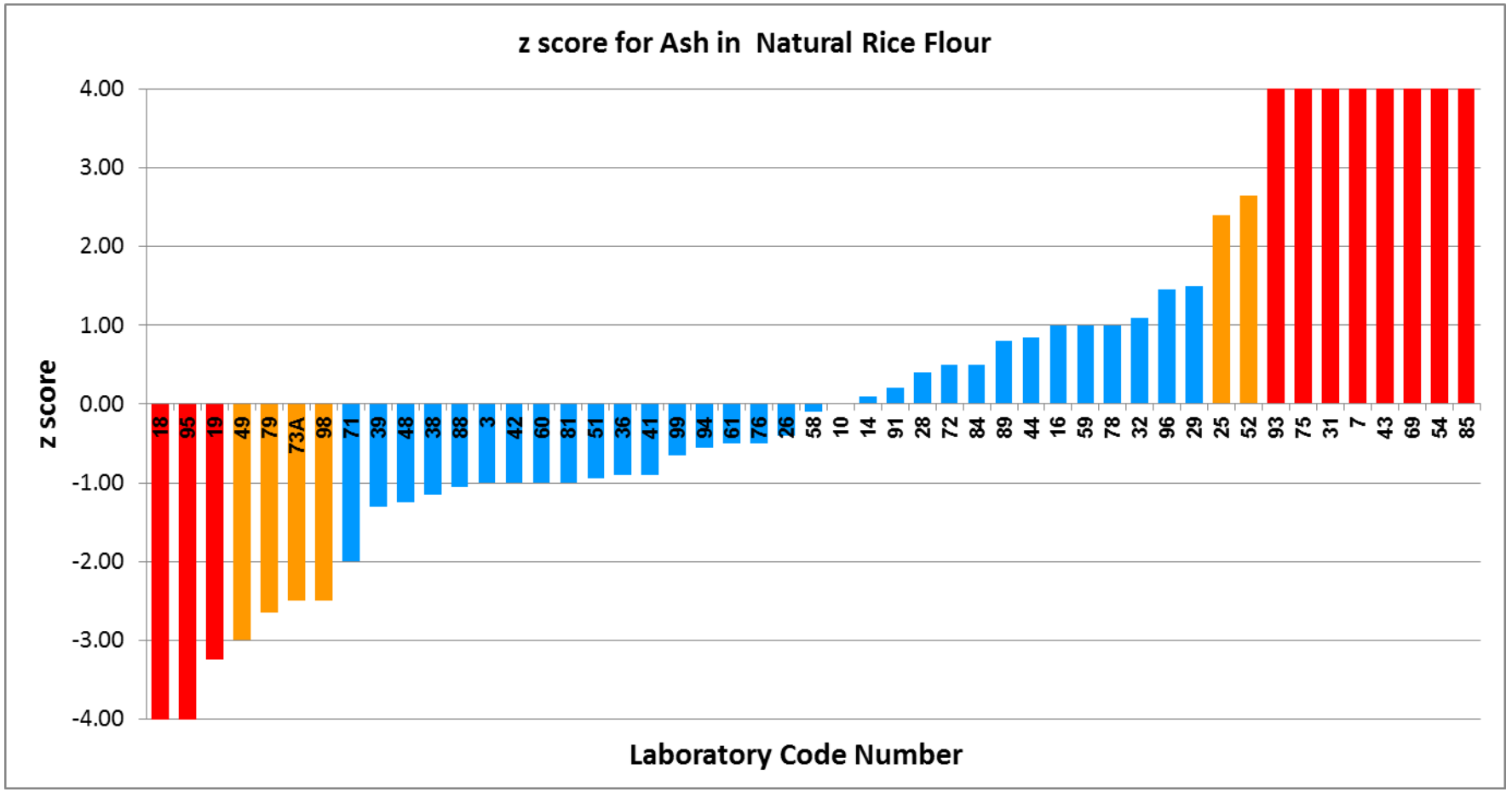

Figure 116. Plot of ordered *z* scores for **ash** results in rice flour

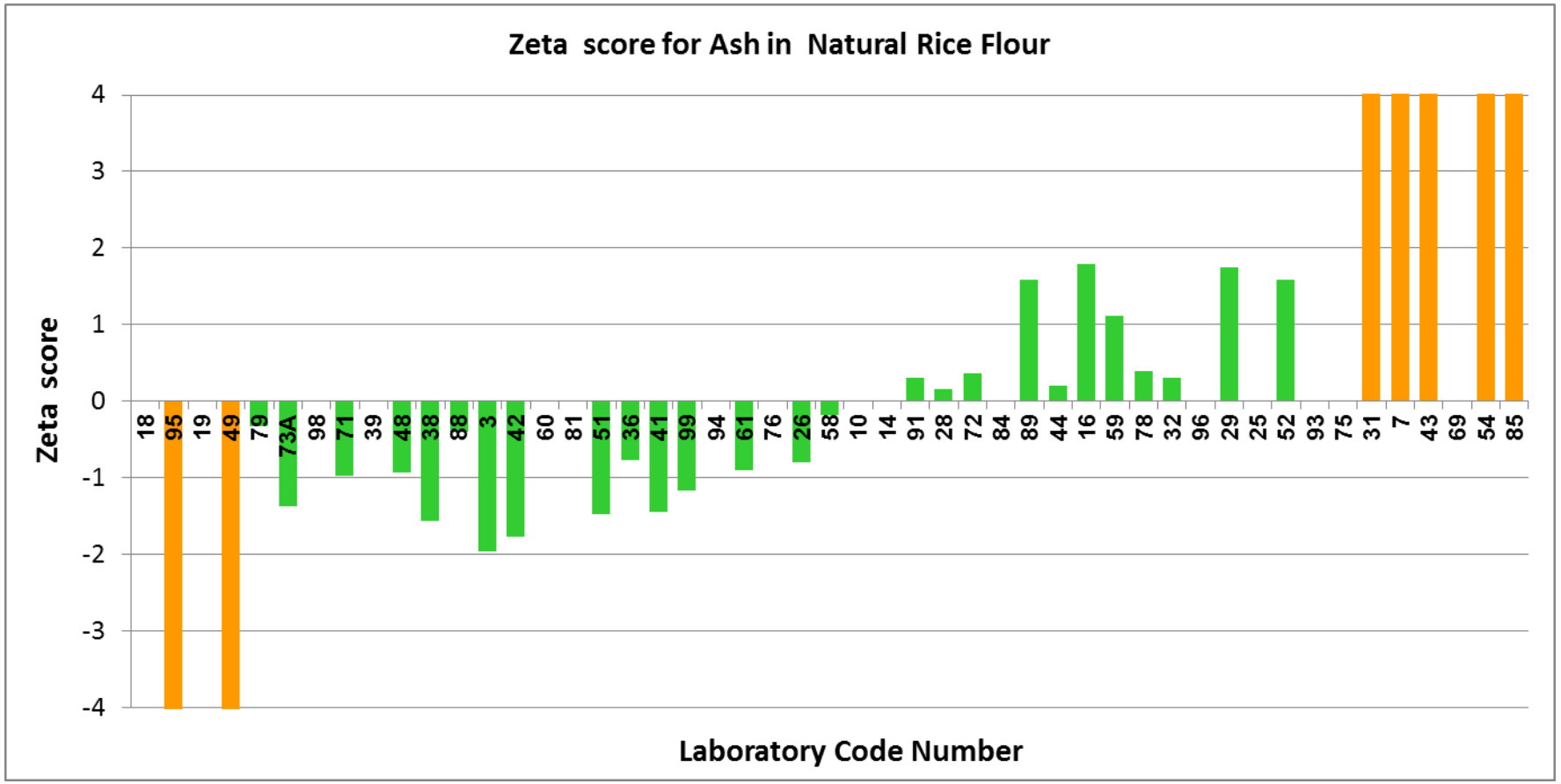

Figure 117. Plot of *Zeta* score for **ash** in rice flour, following the ordered z scores in the above Figure 116.

Table 42. Evaluation of laboratory performance **calcium** analysis (mg/kg, as received) in rice flour

Lab Number	Calcium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± 2SDp from Horwitz's equation = 94.40 ± 15.23 mg/kg (CV 16.1%, n= 45) with u_{xpt} 2.25 mg/kg*											
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00							
13	**28.20**	-	**-4.35**	-	0.5	Microwave	HNO3 10 mL + HCl 2 mL	Analytikal Jena ContrAA 800 D	Ca 422	N	Internal Method
14	**89.34**	-	-0.33	-	0.8	Ashing	50% HNO3, 50% HCl	ICP Horiba Jobin Yvon	Ca 393.366	Y	AOAC 975.03, 984.27
15	**88.60**	-	-0.38	-	0.5	Ultrawave Digestion	5% HNO3 + 0.5% HCl	ICP-MS (7900 Agilent)	Ca 44	N	Based on USFDA 4.7 version 1.1
16	**114.00**	11.00	1.29	**3.30**	0.5	Hot plate	HNO3+H2O 2	ICP-OES Optima 7000 DV Perkin Elmer	Ca 317.933	N	In-house Method
18	**77.40**	-	-1.12	-	2.0	Dry Ashing	HNO3	AAS, Varian	Various	N	AOAC 968.08
19	**1330.00**	-	**81.13**	-	1	By Furnace	HNO3:H2O (1:1)	Ca Manual by Buret	P 400	N	AOAC 927.02, 944.03, 965.17
25	**17.70**	0.07	**-5.04**	**-34.08**	5.0202 / 5.0205	HNO3-HCl Digestion	Water	ICP-OES	Ca 396.847	-	USEPA Method 3050B
26	**143.00**	7.72	**3.19**	**10.88**	4.0	Dry ashing	Water & HCl (1+1)	AAS Shimadzu AA-7000	Ca 422.7	N	AOAC No. 975.03
31	**128.84**	-	**2.26**	-	5	Dry Ashing		AAS, Agilent		N	AOAC 985.35
32	**84.30**	7.12	-0.66	**-2.40**	2.0541	Ashing	HCl	Flame AAS, Shimadzu 6300	Ca 422.7	N	Modified AOAC 969.32
38	**139.00**	17.00	**2.93**	**5.07**	1.000	Dry Ashing	1N HNO3 (0.1M HNO3 for Fe)	Flame AAS, Shimadzu AA6300	Ca 422.70	-	AOAC 985.35, 19th Ed 2012 (Fe modified AOAC 999.11)
39	**79.90**	-	-0.95	-	0.5	Microwave	-	AAS	Ca 422.7	Y	AOAC 985.35
41	**0.00**	-	**-6.20**	-	2	-	-	-	-	-	-

Lab Number	Calcium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± 2SDp from Horwitz's equation = 94.40 ± 15.23 mg/kg (CV 16.1%, n= 45) with u_{xpt} 2.25 mg/kg*											
42	**862.00**	55.20	**50.40**	**27.72**	5	Dry Ashing	HNO3-HCl	Flame AAS, Agilent 280 FS	Ca 422.7	N	AOAC 985.35.2005
43	**98.44**	18.88	0.27	0.42	0.5	Microwave	HNO3	ICP-OES	Ca 317.933	N	AOAC
44	**141.00**	17.00	**3.06**	**5.30**	1.0000	Dry Ashing	-	AAS, Thermoscientific	Ca 422.7	N	AOAC 19th Ed, 2012
47	**80.30**	0.73	-0.93	**-6.19**	5	Dry Ashing	Nitric Acid	AAS - Shimadzu 7000	Ca 422.7	-	AOAC 985.35 (Fe 999.11)
48	**108.81**	4.36	0.95	**4.60**	5	Dry Digestion	-	AA800 Perkin Elmer	Ca 422.7	N	MU-03/21 (AAS)
49	**104.00**	5.00	0.63	**2.85**	1, 3	Dry Ashing	Conc Nitric acid	AAS / AA-7000 Shimadzu	Ca 422.7	N	AOAC 20th Ed 2016
51	**106.00**	4.34	0.76	**3.71**	1	Dry Ashing	Nitric - HCl	AAS Shimadzu 7000	Ca 422.7	N	AOAC 999.11
52	**112.30**		1.18	-	0.5 (Ca, Fe)	Dry Ashing (Ca, Fe)	1 N HNO3 (Ca, Fe)	Flame AAS (Shimadzu AA6300)	Ca 422.7	N	Modified AOAC 985.35 (Ca, Fe)
53	**103.00**	6.00	0.56	**2.29**	0.3	Microwave	4 mL HNO3, 1 mL HCl, 1 mL H2O2	ICPMS Thermo	-	-	In house method
54	**78.80**	9.87	-1.02	**-2.88**	1	Dry Ashing	HNO3	ICP / Shimadzu	Ca 317.933	N	AOAC 984.27
59	**385.59**	26.55	**19.12**	**21.63**	1.5	Dry Ashing		AAS, Shimadzu	Ca 422.7	Y	AOAC 18th Ed 985.35
60	**100.00**	-	0.37	-	-	-	-	-	-	-	AOAC (2012) 968.08 (Ca, Mg),
61	**78.00**	21.40	-1.08	-1.50	1	Acid block digestion	HNO3	Varian AA240 FS Fast Sequential AAS	Ca 422.7	N	A6407-26 AAS
64	**120.80**	1.00	1.73	**11.45**	0.5063	Dry Ashing	1 N HNO3	Shimadzu AA6300	Ca 422.7	N	Modified AOAC 985.35

Lab Number	Calcium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± 2SDp from Horwitz's equation = 94.40 ± 15.23 mg/kg (CV 16.1%, n= 45) with u_{xpt} 2.25 mg/kg*											
69	**424.00**	-	**21.64**	-	-	-	-	-	-	-	-
71	**0.04**	0.02	**-6.20**	**-41.94**	1.0018, 1.0039	Acid Digestion	HCl (1:3)	-	-	-	AOAC 927.02, Titration
72	**65.30**	5.10	-1.91	**-8.56**	3	Ashing	HNO3	AAS / Analytik Jena	Ca 422.7	N	AOAC 985.35
73A	**0.02**	0.00	**-6.20**	**-41.95**	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	**0.02**	0.00	**-6.20**	**-41.95**	1	Dry ashing	Hot plate	AAS (280FS AA, Agilent Technology)	Ca 422.7	-	-
75	**91.13**	5.83	-0.21	-0.89	1	Wet digestion (hot block)	HNO3 + H2O2	ICP-OES Agilent 5100	Ca 317.933	N	In House Method ICP-OES
76	**36.84**	3.81	**-3.78**	**-19.52**	-	-	-	-	-	-	-
77	**152.34**	-	**3.80**	-	-	-	-	-	-	-	-
78	**160.00**	7.61	**4.31**	**14.84**	0.5	Mircowave Digestion	Acid Digestion	Berghof Speedwave 4 Microwave Digestion Unit	Ca 393.366	-	MP-AES
81	**71.80**	3.10	-1.48	**-8.27**	mean:, Ca 2.0042	Dry Ashing (Ca, Fe)	1 N HNO3 (Ca, Fe)	Shimadzu AAS AA 6300	Ca 422.7	N	AOAC 985.35 Mod (Ca, Fe)
82A	**116.00**	36.00	1.42	1.19	0.250	none	none	HPGe detector, Canberra	-		Neutron Activation Analysis (NAA)
82B	**89.60**	8.34	-0.32	-1.01	1.00	Microwave	Nitric Acid	AAS, GBC	-	Y	Flame SSA
84	**82.30**	8.20	-0.79	**-2.59**	0.5	Microwave Digestion	HNO3 / H2O2	ICP-OES, ICP-MS	Ca 317.933	N	AOAC 999.10:2005
88	**143.76**	1.68	**3.24**	**20.55**	3	Dry Ashing	HNO3 conc 10 mL	AAS GBC Flame	Ca 422.3	N	In house method (AAS)

Lab Number	Calcium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± 2SDp from Horwitz's equation = 94.40 ± 15.23 mg/kg (CV 16.1%, n= 45) with u_{xpt} 2.25 mg/kg*											
89	**26.39**	0.40	**-4.47**	**-30.11**	2	Dry Ashing	1.5% HNO3	AAS Agilent	Various	N	AOAC 985.35
91	**58.30**	-	**-2.37**	-	-	-	-	-	-	-	-
93	**< 0.10**	-	**-6.19**	-	0.05	Charring, Dry ashing	Hotplate, Furnace	Flame Photometer, Sherwood	N/A	N/A	AOAC 985.35
94	**9.10**	-	**-5.60**	-	1.5	Dry ashing	-	ICP-OES / Perkin Elmer	Ca 317.9	Y	AOAC (2012) 984.27
95	**180.00**	10.00	**5.62**	**15.61**	-	-	-	-	-	-	-
99	**82.70**	2.48	-0.77	**-4.55**	0.3 ± 0.001	Microwave	Nitric Acid & Hydrogen Peroxide	Digestion (MULTI GO Anton Paar) Determination (ICP/MS, PE)	Refer Mass each element	N	EPA 3015A

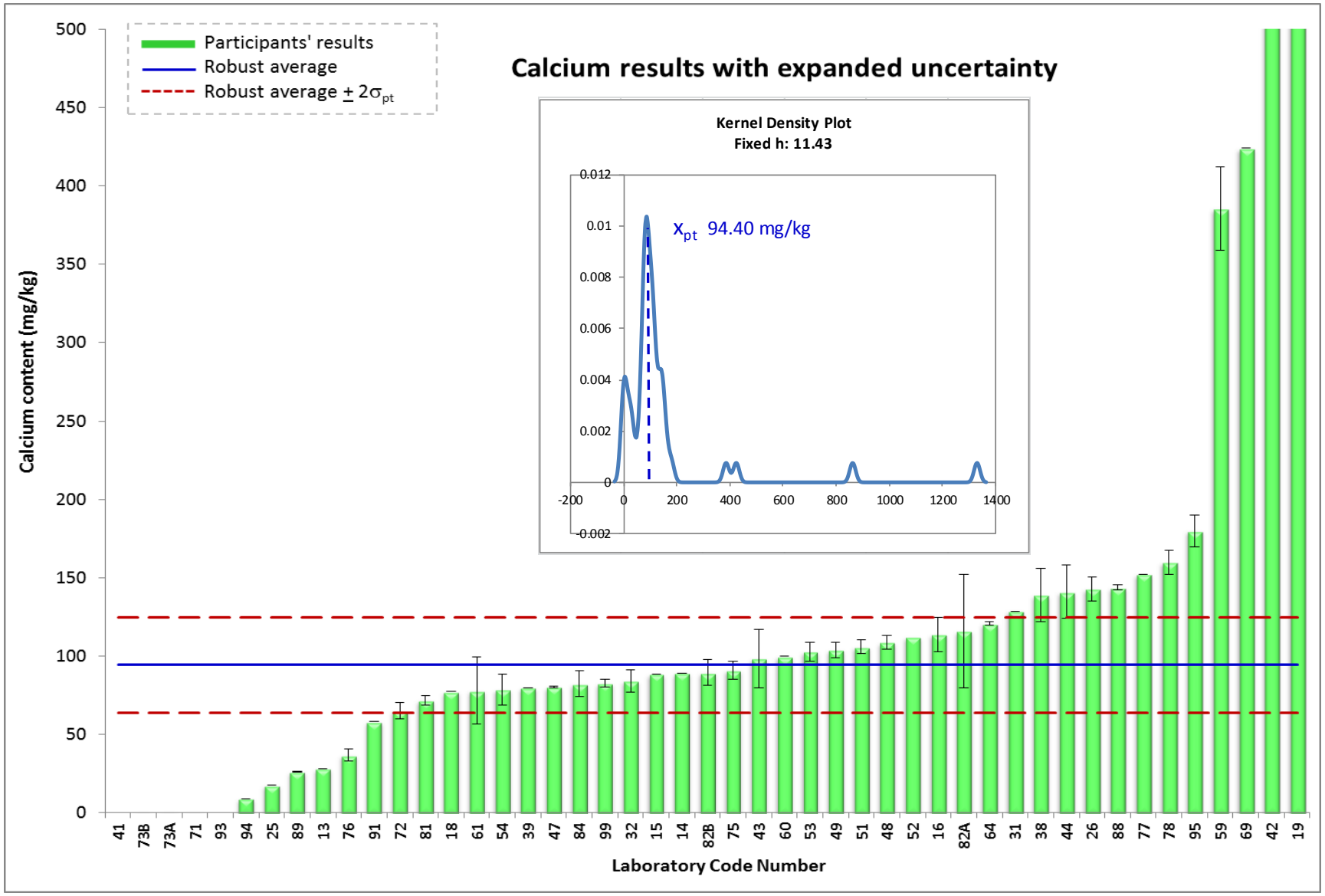

Figure 118. Distribution of **calcium** results (ascending order) in rice flour with expanded uncertainty

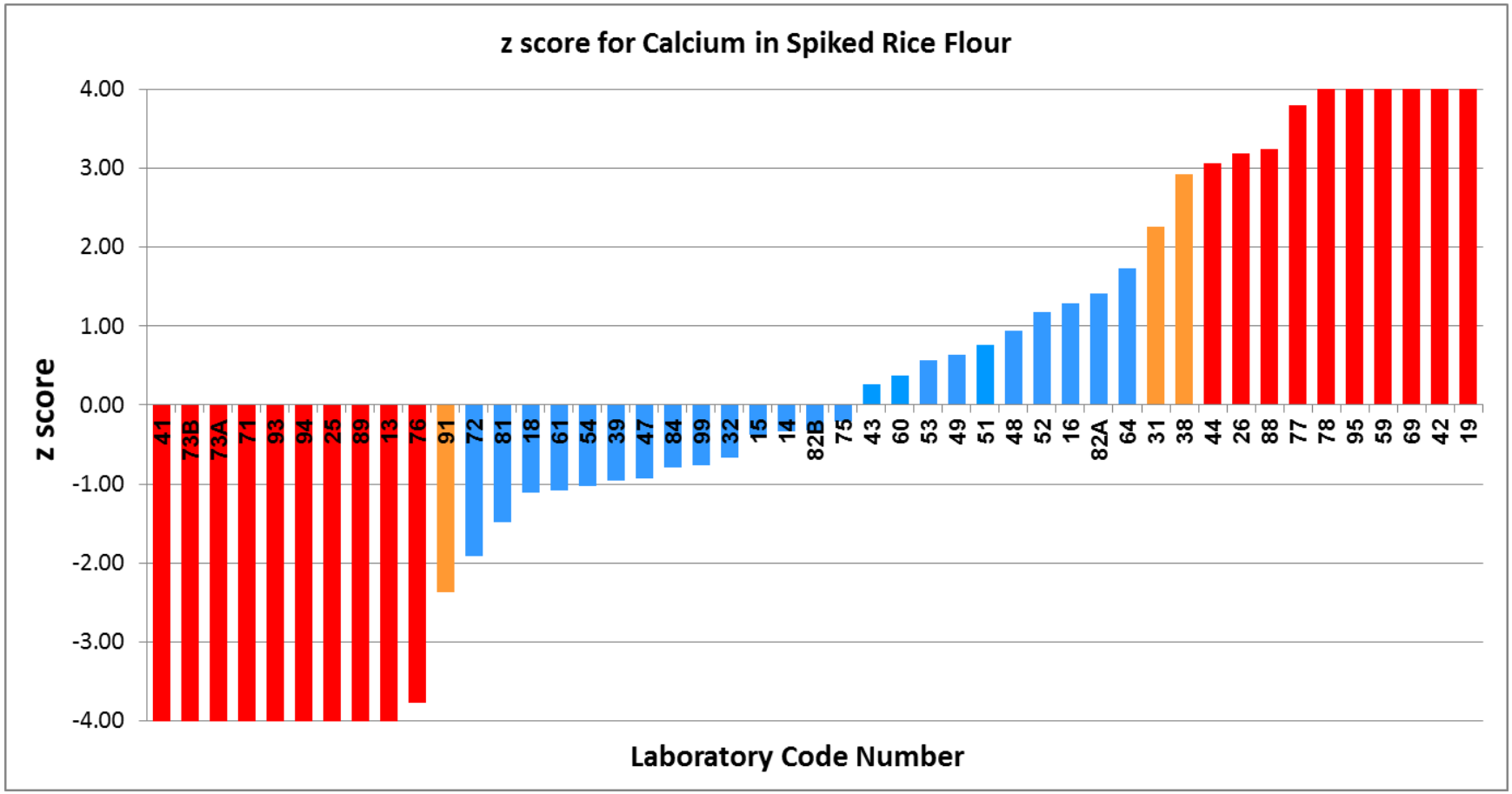

Figure 119. Plot of ordered *z* scores for **calcium** results in rice flour

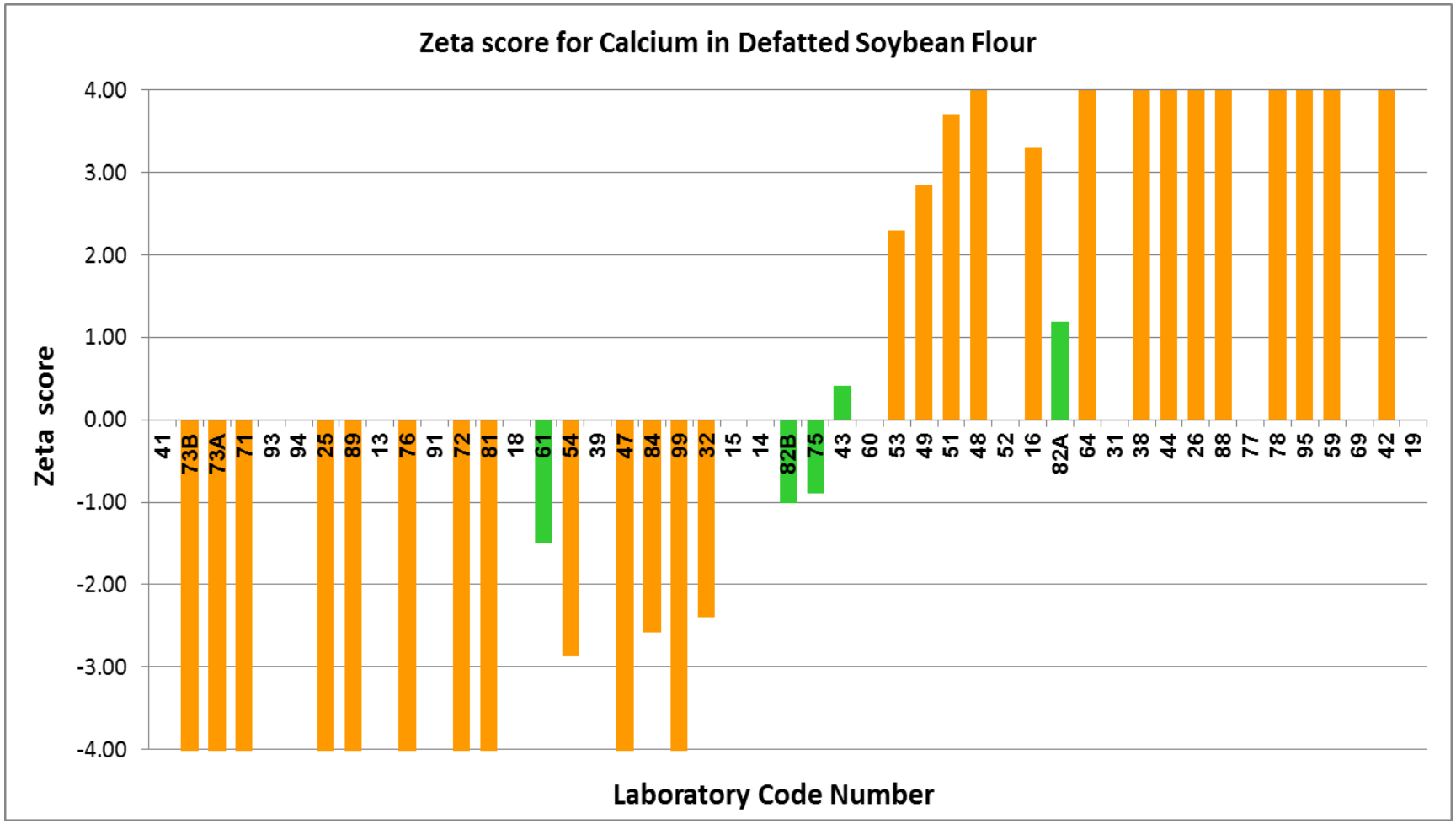

Figure 120. Plot of *Zeta* score for **calcium** in rice flour, following the ordered z scores in the above Figure 119.

Table 43. Evaluation of laboratory performance **phosphorus** analysis (mg/kg, as received) in rice flour

Lab Number	Phosphorus (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± robust SD (s*) = 800.56 ± 120.80 mg/kg (CV 15.1%, n= 28) with u_{xpt} 32.19 mg/kg*											
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00							
14	**694.56**	-	-0.88	-	0.8	Ashing	50% HNO3, 50% HCl	ICP Horiba Jobin Yvon	P 213.618	Y	AOAC 975.03, 984.27
16	**865.00**	9.00	0.53	1.98	0.5	Hot plate	HNO3+H2O2	ICP-OES Optima 7000 DV Perkin Elmer	-	N	In-house Method
18	**862.00**	-	0.51	-	2.0	Dry Ashing	HNO3	AAS, Varian	Various	N	AOAC 968.08
19	**350.00**	-	**-3.73**	-	1	By Furnace	HNO3:H2O (1:1)	P by UV-Vis Spectro.	P 400	N	AOAC 927.02, 944.03, 965.17
25	**76.80**	-	**-5.99**	**-**	5.0202 / 5.0205	HNO3-HCl Digestion	Water	ICP-OES	-	-	USEPA Method 3050B
29	**275.42**	6.14	**-4.35**	**-16.24**	3	Dry Ashing	HCl	-	-	-	-
31	**557.67**	45.28	**-2.01**	**-6.17**	5	Dry Ashing		AAS, Agilent		N	AOAC 985.35
38	**859.00**	1.77	0.48	1.81	1.000	Dry Ashing	1N HNO3	Flame AAS, Shimadzu AA6300	-	-	AOAC 985.35, 19th Ed 2012
41	**0.05**	-	**-6.63**	-	2	-	-	-	-	-	-
42	**644.00**	11.60	-1.30	**-4.79**	5	Dry Ashing	HNO3-HCl	Flame AAS, Agilent 280 FS	-	N	AOAC 985.35.2005
43	**939.28**	165.28	1.15	1.56	0.5	Microwave	HNO3	ICP-OES	-	N	AOAC
48	**754.99**	32.04	-0.38	-1.27	5	Dry Digestion		AA800 Perkin Elmer	-	N	MU-03/21 (AAS)

Lab Number	Phosphorus (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± robust SD (s*) = 800.56 ± 120.80 mg/kg (CV 15.1%, n= 28) with u_{xpt} 32.19 mg/kg*											
49	**818.00**	41.00	0.14	0.46	1, 3	Dry Ashing	Conc Nitric acid	AAS / AA-7000 Shimadzu	-	N	AOAC 20th Ed 2016
54	**814.00**	9.00	0.11	0.41	1	Dry Ashing	HNO3	ICP / Shimadzu	-	N	AOAC 984.27
58	**761.80**	72.51	-0.32	-0.80	3.0	Dry Ash	HCl	ICP-OES	-	-	Dry Ashing and Quantitation by ICP-OES
59	**830.94**	32.22	0.25	0.84	1.5	Dry Ashing	-	AAS, Shimadzu	-	Y	AOAC 18th Ed 985.35
60	**800.00**	-	0.00	-	-	-	-	-	-	-	AOAC (2012) 965.17 (P)
61	**910.00**	126.00	0.91	1.55	1	Acid block digestion	HNO3 (HNO3/HCLO4 for P)	Shimadzu UV-2700 for P	-	N	A6407-26 AAS (A6417 Spectro Method for P)
69	**895.00**	-	0.78	-	-	-	-	-	-	-	-
71	**0.07**	0.07	**-6.63**	**-24.87**	1.0018, 1.0039	Acid Digestion	HCl (1:3)	-	-	-	AOAC 927.02, Titration
73A	**0.09**	0.02	**-6.63**	**-24.87**	1	Dry ashing	Hot plate	AAS (280FS AA, Agilent Technology)	-	N	FTC-46.01 (refers to AOAC 968.08, 965.09)
75	**1092.72**	29.69	**2.42**	**8.24**	1	Wet digestion (hot block)	HNO3 + H2O2	ICP-OES Agilent 5100	-	N	In House Method ICP-OES
76	**848.69**	0.47	0.40	1.50	-	-	-	-	-	-	-
77	**3.09**	-	**-6.60**	-	-	-	-	-	-	-	-
79	**907.16**	-	0.88	-	-	-	-	-	-	-	-

Lab Number	Phosphorus (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± robust SD (s*) = 800.56 ± 120.80 mg/kg (CV 15.1%, n= 28) with u_{xpt} 32.19 mg/kg*											
88	**11.01**	0.12	**-6.54**	**-24.53**	3	Dry Ashing	HNO3 conc 10 mL	AAS GBC Flame	-	N	In house method (AAS)
95	**800.00**	800.00	0.00	0.00	-	-	-	-	-	-	-
99	**768.00**	38.40	-0.27	-0.87	0.3 ± 0.001	Microwave	Nitric Acid & Hydrogen Peroxide	Digestion (MULTI GO Anton Paar) Determination (ICP/MS, PE)	Refer Mass each element	N	EPA 3015A

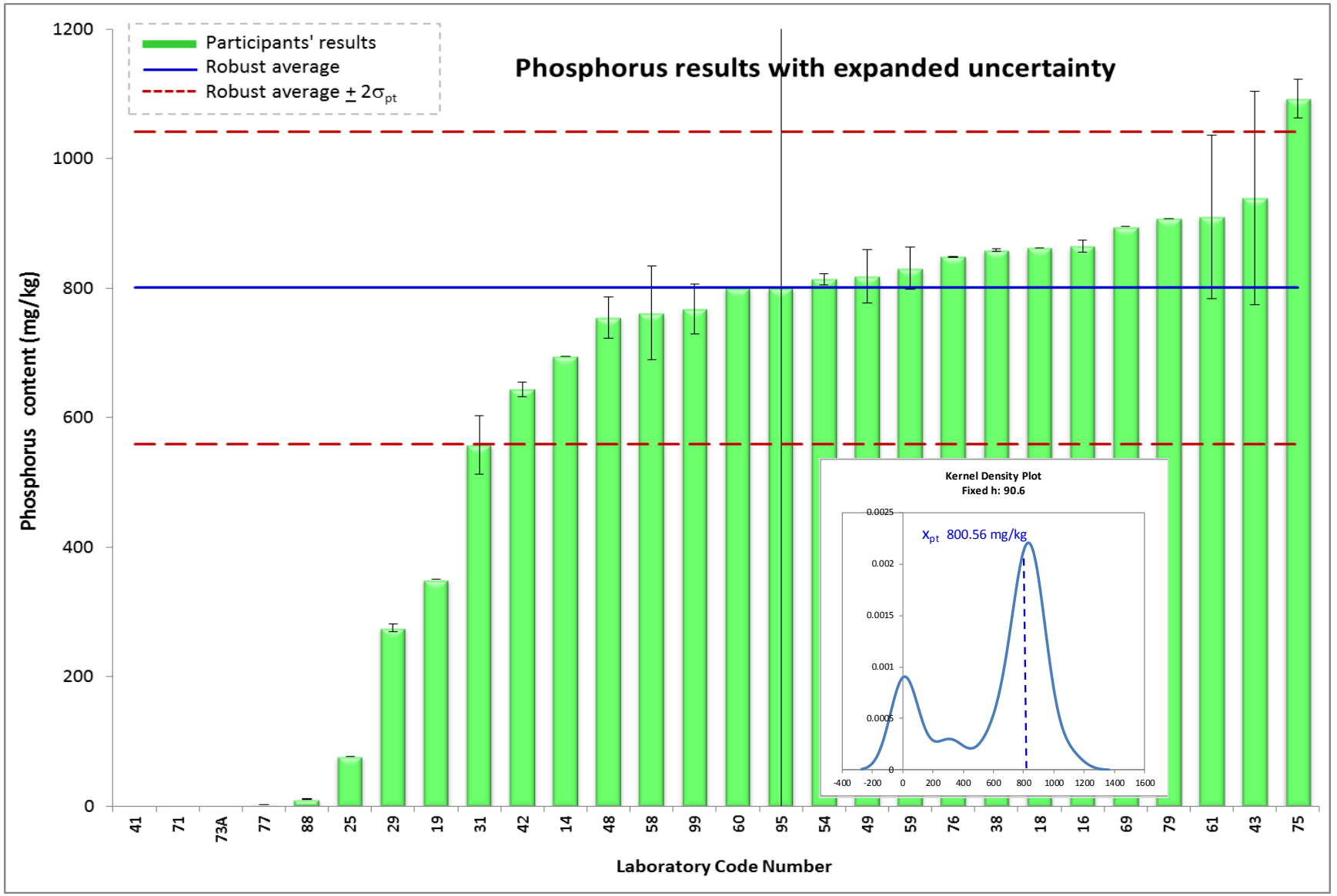

Figure 121. Distribution of **phosphorus** results (ascending order) in rice flour with expanded uncertainty

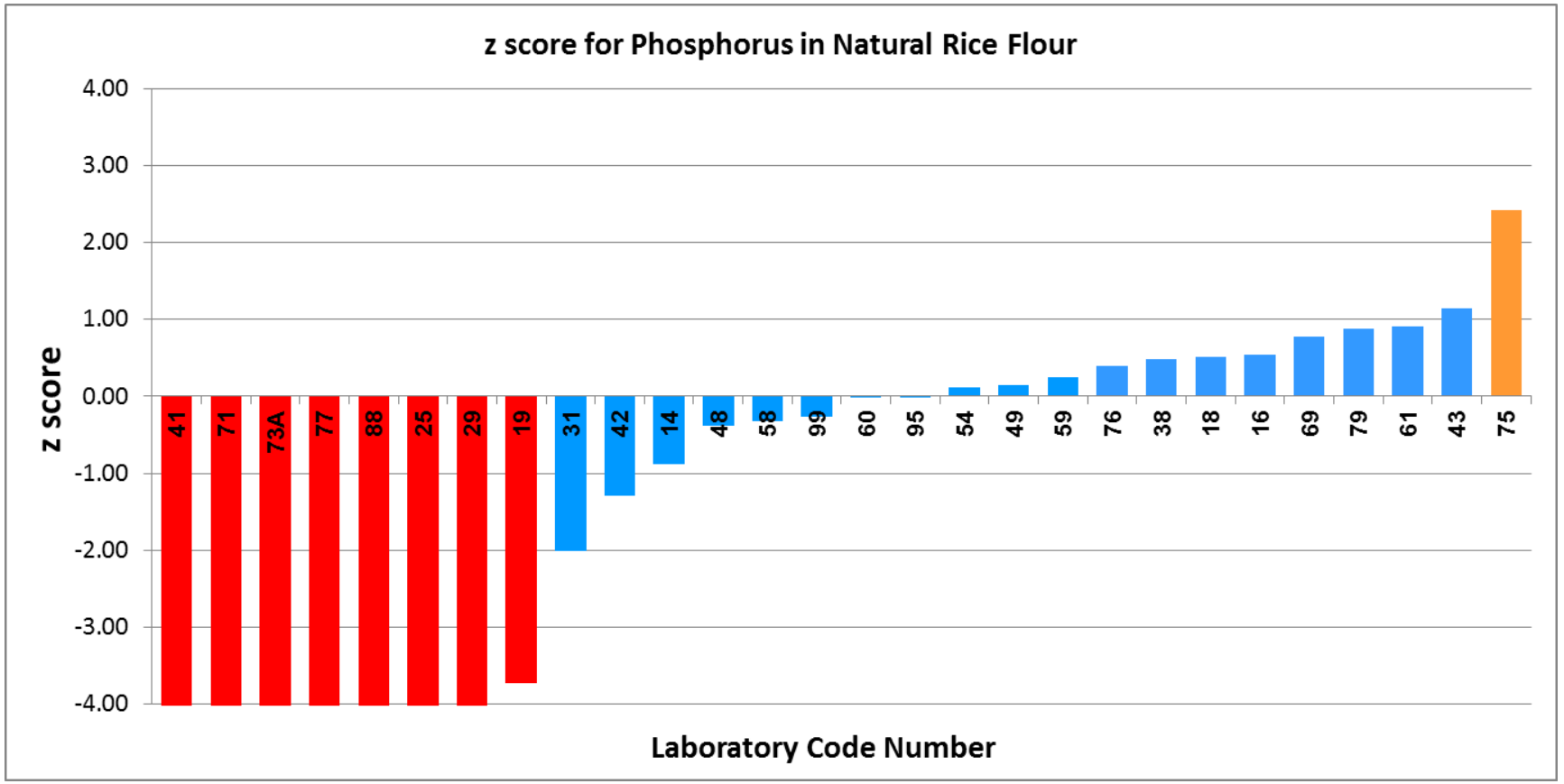

Figure 122. Plot of ordered *z* scores for **phosphorus** results in rice flour

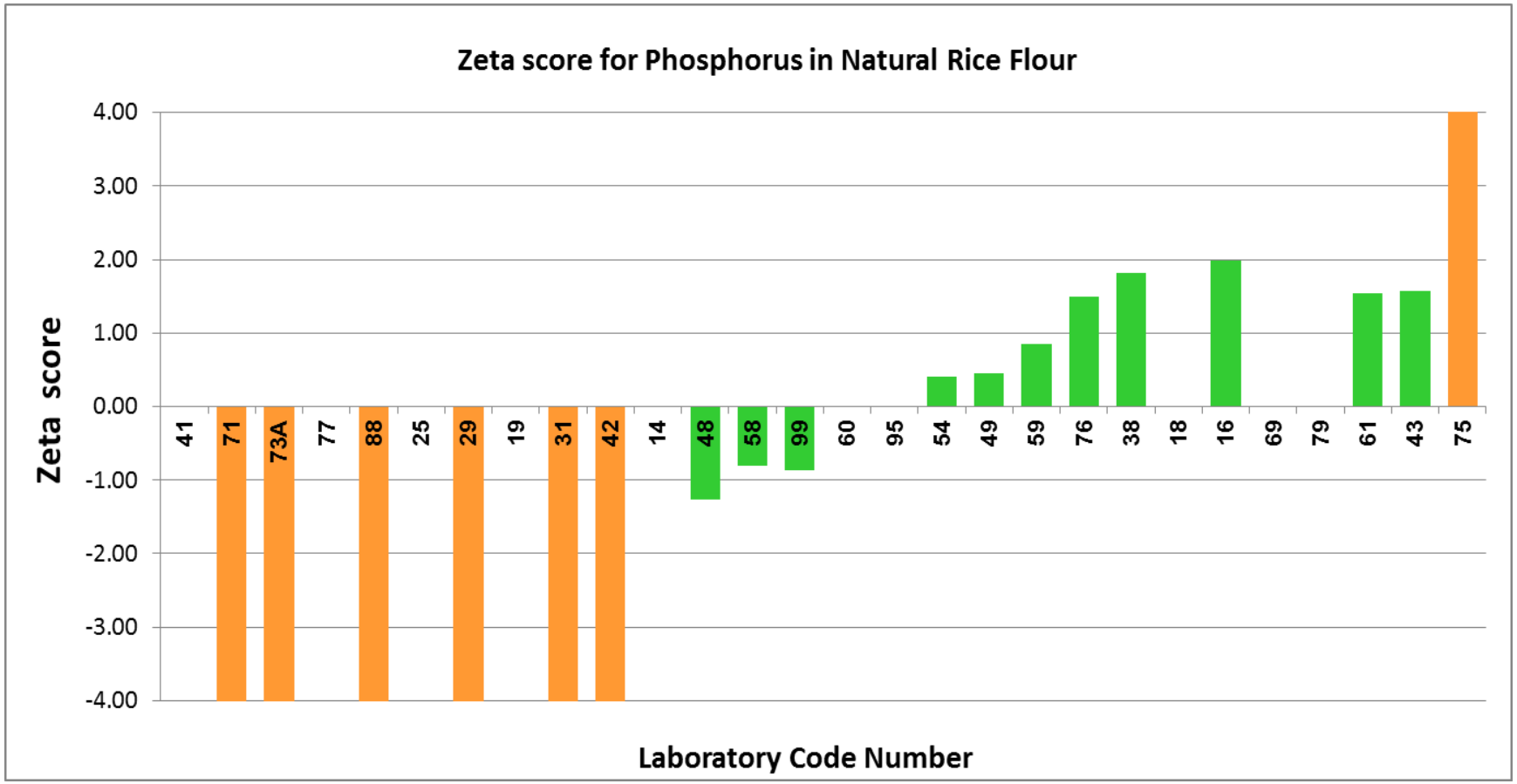

Figure 123. Plot of *Zeta* score for **phosphorus** in rice flour, following the ordered z scores in the above Figure 122.

Table 25. Evaluation of laboratory performance **sodium** analysis (mg/kg, as received) in rice flour

Lab Number	Sodium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± 2SDp from Horwitz's equation = 319.11 ± 42.87 mg/kg (CV 13.4%, n= 43) with u_{xpt} 6.54 mg/kg*											
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00							
7	**0.00**	0.00	**-7.44**	-	1.0039/1.0316	Ashing	HCL	Flame Photometer	-	N	-
13	**111.00**	-	**-4.85**	-	0.5	Microwave	HNO3 10 mL + HCl 2 mL	Analytikal Jena ContrAA 800 D	Na 588	N	Internal Method
14	**241.89**	-	-1.80	-	0.8	Ashing	50% HNO3, 50% HCl	ICP Horiba Jobin Yvon	Na 588.995	Y	AOAC 975.03, 984.27
15	**319.00**	-	0.00	-	0.5	Ultrawave Digestion	5% HNO3 + 0.5% HCl	ICP-MS (7900 Agilent)	-	N	Based on USFDA 4.7 version 1.1
16	**343.00**	22.00	0.56	1.15	0.5	Hot plate	HNO3+H2O2	ICP-OES Optima 7000 DV Perkin Elmer	Na 588.995	N	In-house Method
18	**355.00**	-	0.84	-	2.0	Dry Ashing	HNO3	AAS, Varian	Various	N	AOAC 968.08
25	**79.70**	0.07	**-5.58**	**-13.52**	5.0202 / 5.0205	HNO3-HCl Digestion	Water	ICP-OES	Na 588.995		USEPA Method 3050B
26	**383.00**	24.50	1.49	**2.97**	4.0	Dry ashing	Water & HCl (1+1)	AAS Shimadzu AA-7000	Na 589.0	N	AOAC No. 975.03
29	**158.05**	13.36	**-3.76**	**-8.51**	3	Dry Ashing	HCl	-	-	-	-
31	**295.00**	-	-0.56	-	5	Dry Ashing	-	AAS, Agilent	-	N	AOAC 985.35
32	**369.00**	15.20	1.16	**2.59**	2.0541	Ashing	HCl	Flame AAS, Shimadzu 6300	Na 589.0	N	Modified AOAC 969.32
38	**553.00**	75.70	**5.46**	**5.60**	1.000	Dry Ashing	1N HNO3	Flame AAS, Shimadzu AA6300	Na 589.0	-	AOAC 985.35, 19th Ed 2012 (Fe modified AOAC
39	**365.00**	-	1.07	-	0.5	Microwave	-	AAS	Na 589.0	Y	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)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± 2SDp from Horwitz's equation = 319.11 ± 42.87 mg/kg (CV 13.4%, n= 43) with u_{xpt} 6.54 mg/kg*											
42	**260.00**	9.88	-1.38	**-3.21**	5	Dry Ashing	HNO3-HCl	Flame AAS, Agilent 280 FS	Na 589.9	N	AOAC 985.35.2005
43	**296.23**	6.18	-0.53	-1.27	0.5	Microwave	HNO3	ICP-OES	Na 568.821	N	AOAC
44	**380.00**	42.00	1.42	**2.22**	1.0000	Dry Ashing	-	AAS, Thermoscientific	Na 589	N	AOAC 19th Ed, 2012
47	**310.00**	3.49	-0.21	-0.51	5	Dry Ashing	Nitric Acid	AAS - Shimadzu 7000	Na 589	-	AOAC 985.35 (Fe 999.11)
48	**281.16**	3.07	-0.89	**-2.13**	5	Dry Digestion	-	AA800 Perkin Elmer	Na 330.2	N	MU-03/21 (AAS)
49	**252.00**	13.00	-1.57	**-3.56**	1, 3	Dry Ashing	Conc Nitric acid	AAS / AA-7000 Shimadzu	Na 589.0	N	AOAC 20th Ed 2016
51	**297.00**	23.50	-0.52	-1.04	1	Dry Ashing	Nitric - HCl	AAS Shimadzu 7000	Na 589	N	AOAC 999.11
52	**321.66**	-	0.06	-	1.0 (K, Na)	Wet Digestion (K, Na)	2% HNO3 (K, Na)	Flame AAS (Shimadzu AA6300)	Na 589.0	N	Modified AOAC 999.10 (K, Na)
53	**327.00**	3.00	0.18	0.44	0.3	Microwave	4 mL HNO3, 1 mL HCl, 1 mL H2O2	ICPMS Thermo	-	-	In house method
54	**2750.0**	105.00	**56.70**	**43.87**	1	Dry Ashing	HNO3	ICP / Shimadzu	Na 589.592	N	AOAC 984.27
58	**271.80**	21.10	-1.10	**-2.30**	3.0	Dry Ash	HCl	ICP-OES	-	-	Dry Ashing and Quantitation by ICP-OES
59	**590.35**	171.90	**6.33**	**3.09**	1.5	Dry Ashing	-	AAS, Shimadzu	Na 589	Y	AOAC 18th Ed 985.35

Lab Number	Sodium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± 2SDp from Horwitz's equation = 319.11 ± 42.87 mg/kg (CV 13.4%, n= 43) with u_{xpt} 6.54 mg/kg*											
60	**400.00**	-	1.89	-	-	-	-	-	-	-	AOAC (2012) MP37-BPMSP (AAS) (Na, K)
61	**288.00**	39.70	-0.73	-1.17	1	Acid block digestion	HNO3	Varian AA240 FS Fast Sequential AAS	Na 589.6	N	A6407-26 AAS
69	**315.00**	-	-0.10	-	-	-	-	-	-	-	-
72	**444.00**	38.10	**2.91**	**4.80**	3	Ashing	HNO3	AAS / Analytik Jena	Na 589.0	N	AOAC 985.35
75	**405.10**	15.90	**2.01**	**4.43**	1	Wet digestion (hot block)	HNO3 + H2O2	ICP-OES Agilent 5100	Na 589.592	N	In House Method ICP-OES
76	**209.84**	11.33	**-2.55**	**-5.88**	-	-	-	-	-	-	-
77	**103.62**		**-5.03**	-	-	-	-	-	-	-	-
78	**371.00**	14.00	1.21	**2.72**	0.5	Mircowave Digestion	Acid Digestion	Berghof Speedwave 4 Microwave Digestion Unit	Na 589.592	-	MP-AES
81	**317.00**	7.00	-0.05	-0.12	mean: Na, K 1.0025	Wet Digestion (Na, K)	1 N HNO3 and 30% H2O2 (Na, K)	Shimadzu AAS AA 6300	Na 589.0	N	AOAC 999.10 Mod (Na, K)
82A	**124.00**	0.40	**-4.55**	**-11.02**	0.250	none	none	HPGe detector, Canberra	-	-	Neutron Activation Analysis (NAA)
84	**323.00**	32.00	0.09	0.16	0.5	Microwave Digestion	HNO3 / H2O2	ICP-OES, ICP-MS	Na 589.592	N	AOAC 999.10:2005
88	**394.99**	0.13	1.77	**4.28**	3	Dry Ashing	HNO3 conc 10 mL	AAS GBC Flame	Na 587.60	N	In house method (AAS)

Lab Number	Sodium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± 2SDp from Horwitz's equation = 319.11 ± 42.87 mg/kg (CV 13.4%, n= 43) with u_{xpt} 6.54 mg/kg*											
89	**1706.5**	25.60	**32.36**	**63.49**	2	Dry Ashing	1.5% HNO3	AAS Agilent	Various	N	AOAC 985.35
91	**323.00**	14.00	0.09	-	-	-	-	-	-	-	-
93	**400.00**	-	1.89	-	0.05	Charring, Dry ashing	Hotplate, Furnace	Flame Photometer, Sherwood	N/A	N/A	AOAC 985.35
94	**303.00**	-	-0.38	-	1.5	Dry ashing (Fe: Wet ashing)	-	ICP-OES / Perkin Elmer	Na 589.0	Y	AOAC (2012) 984.27
95	**490.00**	100.00	**3.99**	**3.22**	-	-	-	-	-	-	-
99	**448.00**	13.40	**3.01**	**6.81**	0.3 ± 0.001	Microwave	Nitric Acid & Hydrogen Peroxide	Digestion (MULTI GO Anton Paar) Determination (ICP/MS, PE)	Refer Mass each element	N	EPA 3015A

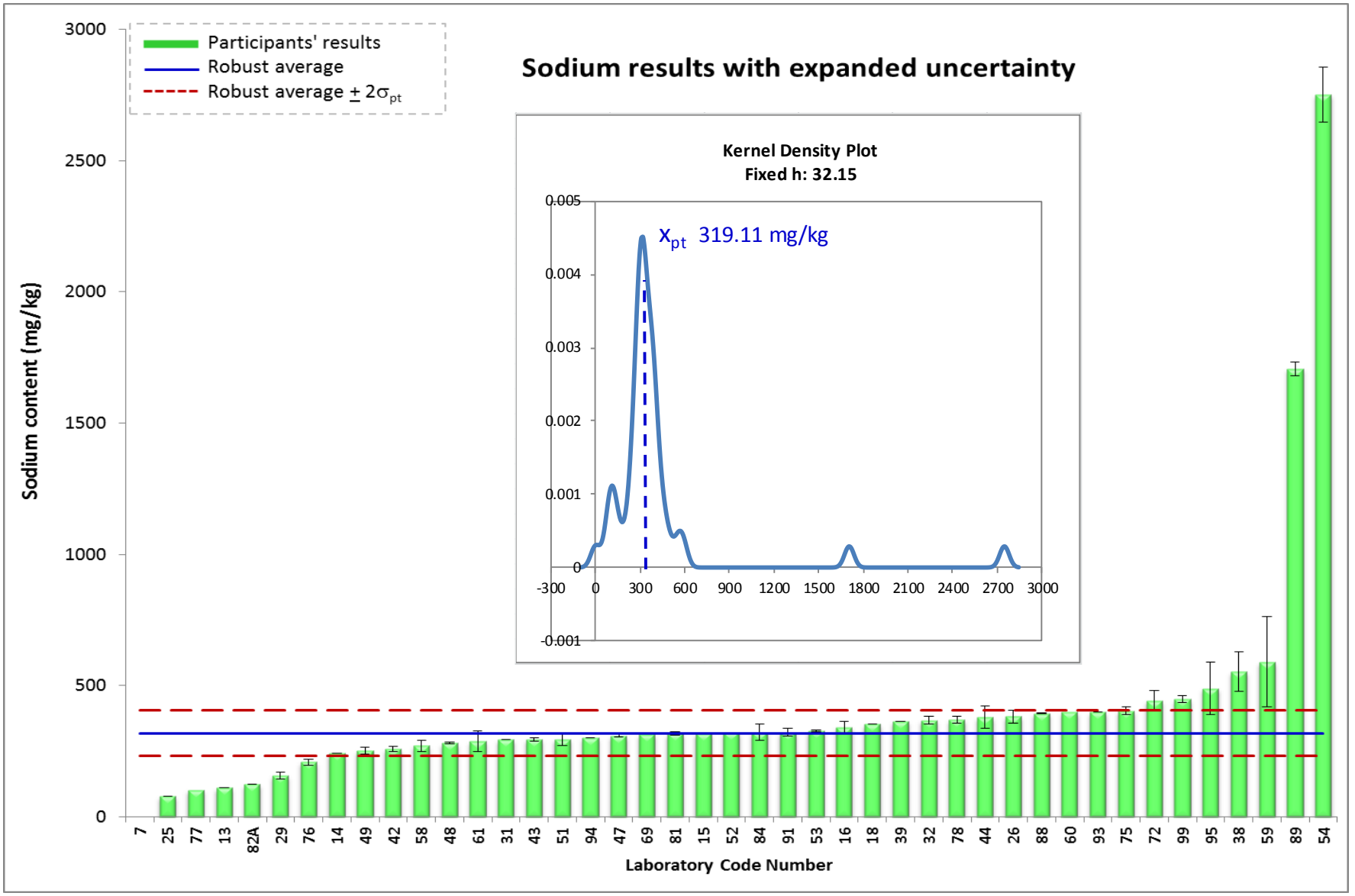

Figure 124. Distribution of **sodium** results (ascending order) in rice flour with expanded uncertainty

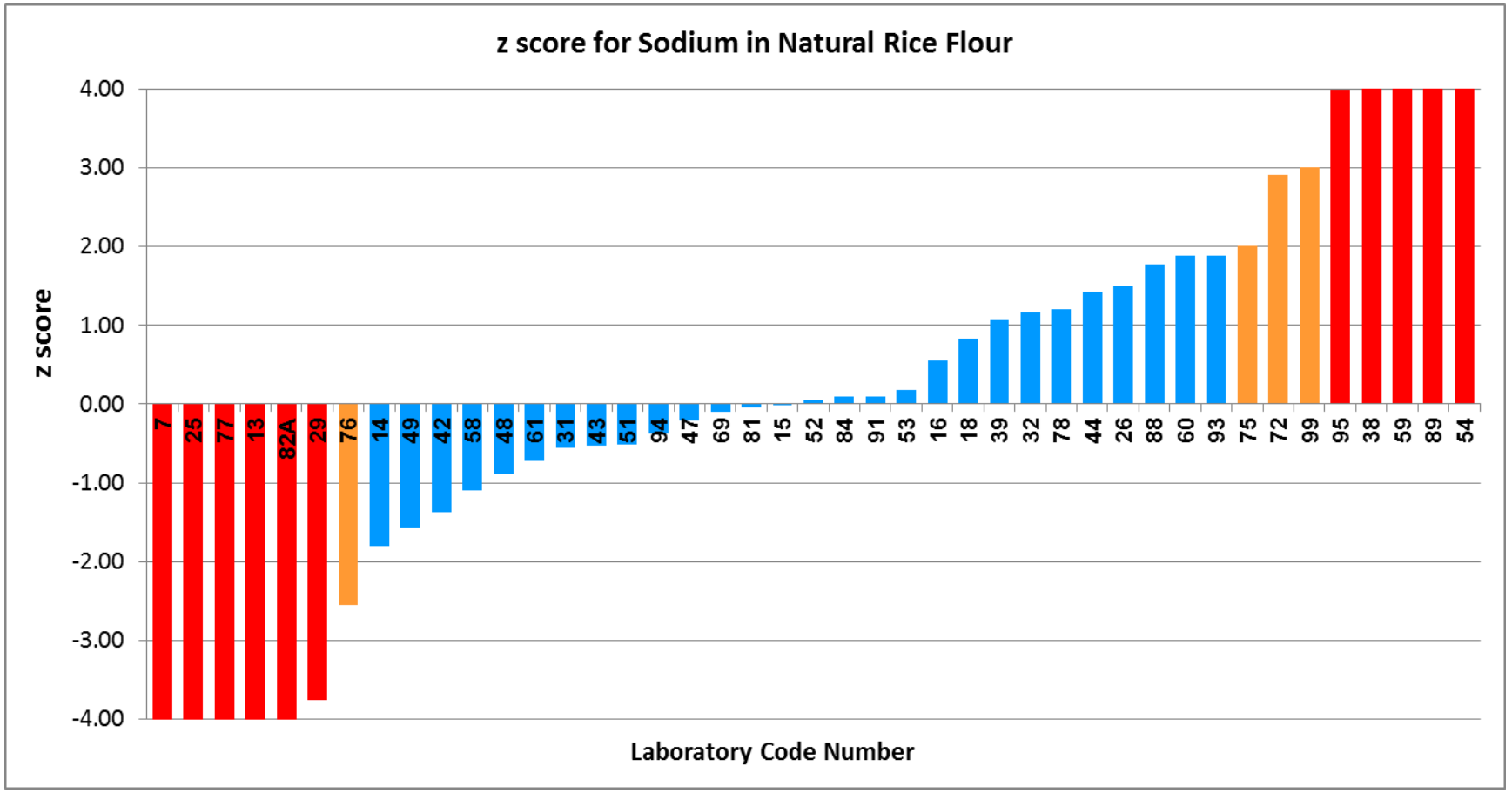

Figure 125. Plot of ordered *z* scores for **sodium** results in rice flour

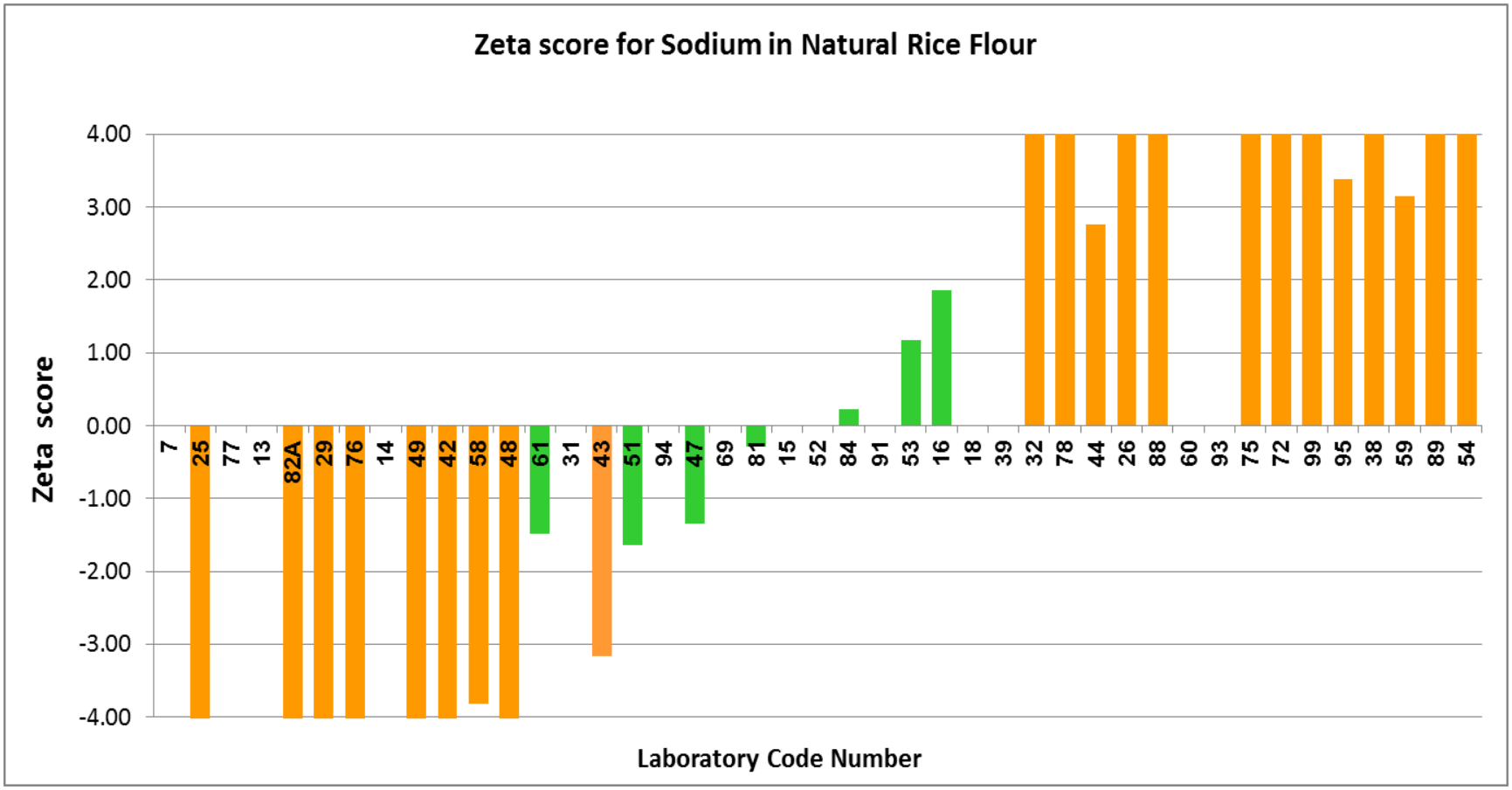

Figure 126. Plot of *Zeta* score for **sodium** in rice flour, following the ordered z scores in the above Figure 125.

Table 45. Evaluation of laboratory performance **potassium** analysis (mg/kg, as received) in rice flour

Lab Number	Potassium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± 2SDp from Horwitz's equation = 158.24 ± 23.63 mg/kg (CV 14.9%, n= 41) with u_{xpt} 3.69 mg/kg*											
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00							
13	**110.00**	-	**-2.04**	-	0.5	Microwave	HNO3 10 mL + HCl 2 mL	Analytikal Jena ContrAA 800 D	K 766	N	Internal Method
14	**174.50**	-	0.69	-	0.8	Ashing	50% HNO3, 50% HCl	ICP Horiba Jobin Yvon	K 766.49	Y	AOAC 975.03, 984.27
15	**162.00**	-	0.16	-	0.5	Ultrawave Digestion	5% HNO3 + 0.5% HCl	ICP-MS (7900 Agilent)	K 39	N	Based on USFDA 4.7 version 1.1
16	**164.00**	16.00	0.24	0.65	0.5	Hot plate	HNO3+H2O2	ICP-OES Optima 7000 DV Perkin Elmer	K 769.896	N	In-house Method
18	**178.00**	-	0.84	-	2.0	Dry Ashing	HNO3	AAS, Varian	Various	N	AOAC 968.08
25	**29.90**	0.07	**-5.43**	**-34.78**	5.0202 / 5.0205	HNO3-HCl Digestion	Water	ICP-OES	K 766.491	-	USEPA Method 3050B
26	**219.00**	13.10	**2.57**	**8.08**	4.0	Dry ashing	Water & HCl (1+1)	AAS Shimadzu AA-7000	K 766.5	N	AOAC No. 975.03
29	**91.62**	8.40	**-2.82**	**-11.92**	3	Dry Ashing	HCl	-	-	-	-
31	**159.11**	-	0.04	-	5	Dry Ashing	-	AAS, Agilent	-	N	AOAC 985.35
32	**93.90**	10.20	**-2.72**	**-10.22**	2.0541	Ashing	HCl	Flame AAS, Shimadzu 6300	K 766.5	N	Modified AOAC 969.32
38	**171.00**	5.73	0.54	**2.73**	1.000	Dry Ashing	1N HNO3 (0.1M HNO3 for Fe)	Flame AAS, Shimadzu AA6300	K 766.50	-	AOAC 985.35, 19th Ed 2012
39	**166.00**	-	0.33	-	0.5	Microwave	-	AAS	K 766.5	Y	AOAC 985.35
42	**199.00**	14.30	1.72	**5.07**	5	Dry Ashing	HNO3-HCl	Flame AAS, Agilent 280 FS	K 769.9	N	AOAC 985.35.2005

Lab Number	Potassium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± 2SDp from Horwitz's equation = 158.24 ± 23.63 mg/kg (CV 14.9%, n= 41) with u_{xpt} 3.69 mg/kg*											
43	**146.15**	0.86	-0.51	**-3.25**	0.5	Microwave	HNO3	ICP-OES	K 766.491	N	AOAC
44	**122.00**	4.10	-1.53	**-8.59**	1.0000	Dry Ashing	-	AAS, Thermoscientific	K 766.5	N	AOAC 19th Ed, 2012
48	**139.50**	8.53	-0.79	**-3.32**	5	Dry Digestion	-	AA800 Perkin Elmer	K 766.5	N	MU-03/21 (AAS)
49	**148.00**	7.00	-0.43	**-2.01**	1, 3	Dry Ashing	Conc Nitric acid	AAS / AA-7000 Shimadzu	K 766.5	N	AOAC 20th Ed 2016
51	**143.00**	5.86	-0.64	**-3.23**	1	Dry Ashing	Nitric - HCl	AAS Shimadzu 7000	-	N	AOAC 999.11
52	**156.04**	-	-0.09	-	0.5 (Ca, Fe), 1.0 (K, Na)	Wet Digestion (K, Na)	2% HNO3 (K, Na)	Flame AAS (Shimadzu AA6300)	K 766.5	N	Modified AOAC 999.10 (K, Na)
53	**163.00**	1.55	0.20	1.26	0.3	Microwave	4 mL HNO3, 1 mL HCl, 1 mL H2O2	ICPMS Thermo	-	-	In house method
54	**138.00**	39.00	-0.86	-1.02	1	Dry Ashing	HNO3	ICP / Shimadzu	-	N	AOAC 984.27
58	**183.50**	2.60	1.07	**6.46**	3.0	Dry Ash	HCl	ICP-OES	-	-	Dry Ashing and Quantitation by ICP-OES
59	**421.09**	69.58	**11.12**	**7.51**	1.5	Dry Ashing	-	AAS, Shimadzu	K 766.5	Y	AOAC 18th Ed 985.35
60	**700.00**	-	**22.93**	-	-	-	-	-	-	-	MP37-BPMSP (AAS) (Na, K)
61	**144.00**	49.00	-0.60	-0.57	1	Acid block digestion	HNO3 (HNO3/HCLO4 for P)	Varian AA240 FS Fast Sequential AAS (Shimadzu UV-2700 for P)	K 769.9	N	A6407-26 AAS (A6417 Spectro Method for P)
69	**153.00**	-	-0.22	-	-	-	-	-	-	-	-
72	**181.00**	16.00	0.96	**2.58**	3	Ashing	HNO3	AAS / Analytik Jena	K 766.5	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)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± 2SDp from Horwitz's equation = 158.24 ± 23.63 mg/kg (CV 14.9%, n= 41) with u_{xpt} 3.69 mg/kg*											
75	**153.75**	4.18	-0.19	-1.06	1	Wet digestion (hot block)	HNO3 + H2O2	ICP-OES Agilent 5100	K 766.491	N	In House Method ICP-OES
76	**128.51**	2.65	-1.26	**-7.58**	-	-	-	-	-	-	-
77	**87.55**	-	**-2.99**	-	-	-	-	-	-	-	-
78	**175.00**	3.00	0.71	**4.21**	0.5	Mircowave Digestion	Acid Digestion	Berghof Speedwave 4 Microwave Digestion Unit	K 766.490	-	MP-AES
81	**163.00**	6.00	0.20	1.00	mean: Na, K 1.0025	Wet Digestion (Na, K)	1 N HNO3 and 30% H2O2 (Na, K)	Shimadzu AAS AA 6300	K 766.5	N	AOAC 999.10 Mod (Na, K)
82A	**165.00**	15.60	0.29	0.78	0.250	none	none	HPGe detector, Canberra	-	-	Neutron Activation Analysis (NAA)
84	**115.00**	12.00	-1.83	**-6.14**	0.5	Microwave Digestion	HNO3 / H2O2	ICP-OES, ICP-MS	K 766.490	N	AOAC 999.10:2005
88	**128.99**	0.25	-1.24	**-7.92**	3	Dry Ashing	HNO3 conc 10 mL	AAS GBC Flame	K 765.20	N	In house method (AAS)
89	**674.76**	10.12	**21.86**	**82.47**	2	Dry Ashing	1.5% HNO3	AAS Agilent	Various	N	AOAC 985.35
91	**34.80**	-	**-5.22**	-	-	-	-	-	-	-	-
93	**200.00**	-	1.77	-	0.05	Charring, Dry ashing	Hotplate, Furnace	Flame Photometer, Sherwood	N/A	N/A	AOAC 985.35
94	**198.00**	-	1.68	-	1.5	Dry ashing	-	ICP-OES / Perkin Elmer	K 766.5	Y	AOAC (2012) 984.27
95	**370.00**	50.00	**8.96**	**8.38**	-	-	-	-	-	-	-
99	**181.00**	5.43	0.96	**4.97**	0.3 ± 0.001	Microwave	Nitric Acid & Hydrogen Peroxide	Digestion (MULTI GO Anton Paar) Determination (ICP/MS, PE)	Refer Mass each element	N	EPA 3015A

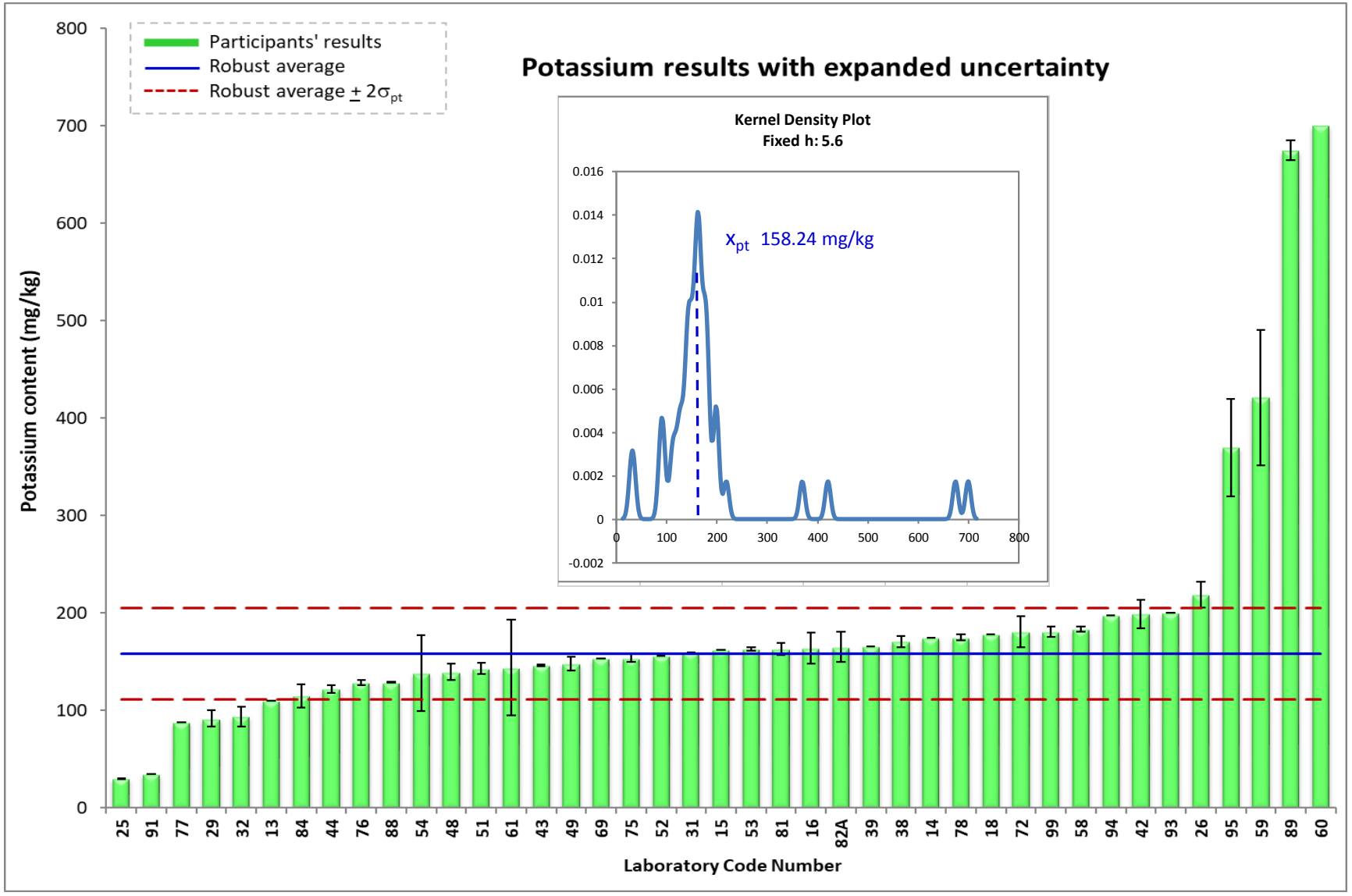

Figure 127. Distribution of **potassium** results (ascending order) in rice flour with expanded uncertainty

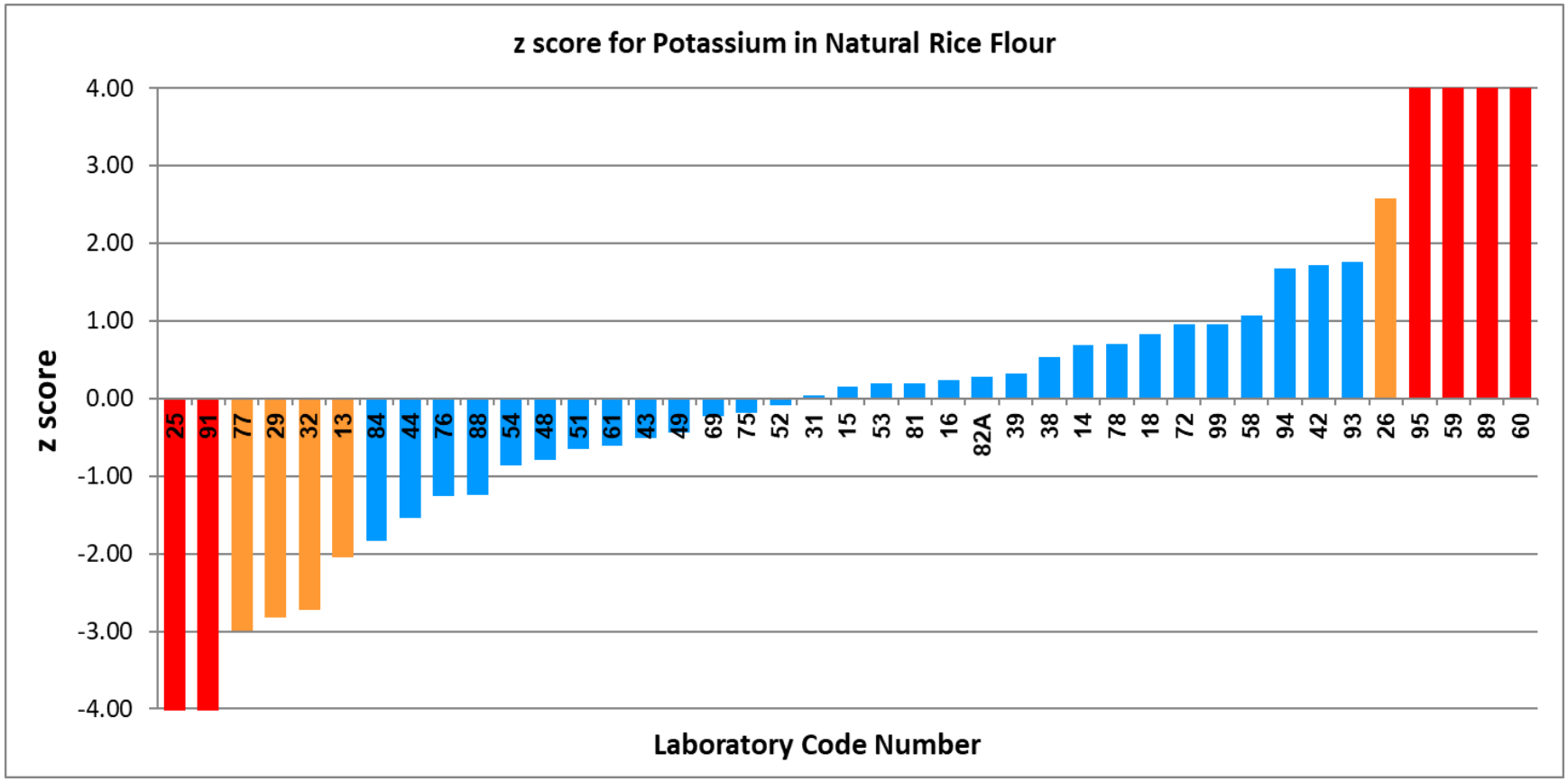

Figure 128. Plot of ordered *z* scores for **potassium** results in rice flour

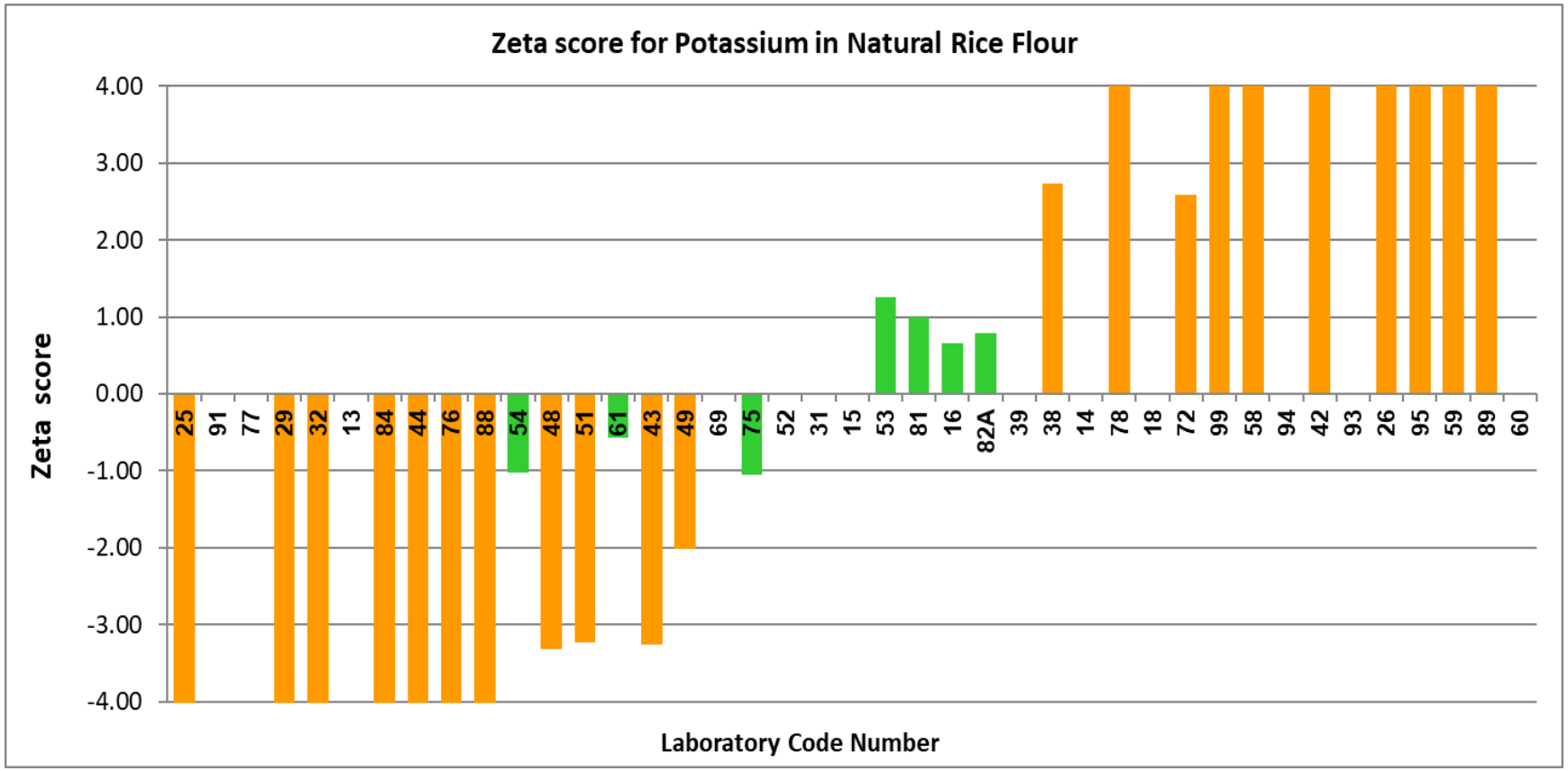

Figure 129. Plot of *Zeta* score for **potassium** in rice flour, following the ordered z scores in the above Figure 128.

Table 46. Evaluation of laboratory performance **iron** analysis (mg/kg, as received) in rice flour

Lab Number	Iron (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± 2SDp from Horwitz's equation = 6.40 ± 1.55 mg/kg (CV 24.2%, n= 38) with u_{xpt} 0.26 mg/kg*											
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00							
13	**9.76**	-	**2.17**	-	0.5	Microwave	HNO3 10 mL + HCl 2 mL	Analytikal Jena ContrAA 800 D	-	N	Internal Method
14	**4.64**	-	-1.14	-	0.8	Ashing	50% HNO3, 50% HCl	ICP Horiba Jobin Yvon	Fe 259.94	Y	AOAC 975.03, 984.27
15	**4.88**	-	-0.98	-	0.5	Ultrawave Digestion	5% HNO3 + 0.5% HCl	ICP-MS (7900 Agilent)	Fe 56	N	Based on USFDA 4.7 version 1.1
16	**6.18**	0.62	-0.14	-0.63	0.5	Hot plate	HNO3+ H2O2	ICP-OES Optima 7000 DV Perkin Elmer	Fe 238.204	N	In-house Method
18	**10.30**	-	**2.52**	-	2.0	Dry Ashing	HNO3	AAS, Varian	Various	N	AOAC 968.08
25	**2.46**	0.07	**-2.54**	**-24.09**	5.0202 / 5.0205	HNO3-HCl Digestion	Water	ICP-OES	Fe 238.204	-	USEPA Method 3050B
26	**7.55**	0.53	0.74	**3.72**	4.0	Dry ashing	Water & HCl (1+1)	AAS Shimadzu AA-7000	Fe 248.3	N	AOAC No. 975.03
29	**4.98**	0.37	-0.92	**-5.81**	3	Dry Ashing	HCl	-	-	-	-
31	**6.43**	-	0.02	-	5	Dry Ashing	-	AAS, Agilent	-	N	AOAC 985.35
32	**3.55**	0.98	-1.84	**-5.51**	2.0541	Ashing	HCl	Flame AAS, Shimadzu 6300	Fe 248.3	N	Modified AOAC 969.32
38	**4.53**	0.13	-1.21	**-10.78**	1.000	Dry Ashing	0.1M HNO3 for Fe	Flame AAS, Shimadzu AA6300	Fe 248.30	-	AOAC 19th Ed 2012, Fe modified AOAC 999.11
39	**4.67**	-	-1.12	-	0.5	Microwave	-	AAS	Fe 248.3	Y	AOAC 985.35
42	**5.68**	0.41	-0.46	**-2.77**	5	Dry Ashing	HNO3-HCl	Flame AAS, Agilent 280 FS	Fe 248.3	N	AOAC 985.35.2005

Lab Number	Iron (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± 2SDp from Horwitz's equation = 6.40 ± 1.55 mg/kg (CV 24.2%, n= 38) with u_{xpt} 0.26 mg/kg*											
43	**4.06**	0.03	-1.51	**-14.56**	0.5	Microwave	HNO3	ICP-OES	-	N	AOAC
44	**10.10**	3.70	**2.39**	1.99	1.0000	Dry Ashing	-	AAS, Thermoscientific	Fe 248.3	N	AOAC 19th Ed, 2012
47	**4.62**	0.06	-1.15	**-10.91**	5	Dry Ashing	Nitric Acid	AAS - Shimadzu 7000	Fe 248.3		AOAC 985.35 (Fe 999.11)
48	**4.94**	0.37	-0.94	**-5.95**	5	Dry Digestion	-	AA800 Perkin Elmer	Fe 248.3	N	MU-03/21 (AAS)
49	**34.70**	1.70	**18.26**	**32.72**	1, 3	Dry Ashing	Conc Nitric acid	AAS / AA-7000 Shimadzu	Fe 248.3	N	AOAC 20th Ed 2016
52	**8.27**	-	1.21	-	0.5 (Ca, Fe)	Dry Ashing (Ca, Fe)	1 N HNO3 (Ca, Fe)	Flame AAS (Shimadzu AA6300)	Fe 248.3	N	Modified AOAC 985.35 (Ca, Fe)
53	**5.21**	0.07	-0.77	**-7.27**	0.3	Microwave	4 mL HNO3, 1 mL HCl, 1 mL H2O2	ICPMS Thermo	-	-	In house method
54	**4.34**	1.12	-1.33	**-3.54**	1	Dry Ashing	HNO3	ICP / Shimadzu	Fe 259.940	N	AOAC 984.27
58	**9.77**	1.98	**2.17**	**3.36**	3.0	Dry Ash	HCl	ICP-OES	-	-	Dry Ashing and Quantitation by ICP-OES
59	**7.28**	1.38	0.57	1.24	1.5	Dry Ashing	-	AAS, Shimadzu	Fe 248.3	Y	AOAC 18th Ed 985.35 (Fe: SNI 3751:2009 point A.10)
60	**200.00**	-	**124.90**	-	-	-	-	-	-	-	SNI 01-2896-1998 (Fe)
61	**< 4.00**	0.47	**-2.84**	**-15.43**	1	Acid block digestion	HNO3	Varian AA240 Sequential AAS	Fe 248.3	N	A6407-26 AAS
64	**8.19**	0.26	1.15	**8.66**	0.5063	Dry Ashing	1 N HNO3	Shimadzu AA6300	Fe 248.3	N	Modified AOAC 985.35

Lab Number	Iron (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± 2SDp from Horwitz's equation = 6.40 ± 1.55 mg/kg (CV 24.2%, n= 38) with u_{xpt} 0.26 mg/kg*											
69	**5.18**	-	-0.79	-	-	-	-	-	-	-	-
72	**5.98**	0.50	-0.27	-1.42	3	Ashing	HNO3	AAS / Analytik Jena	Fe 589.0	N	AOAC 985.35
75	**5.61**	0.47	-0.51	**-2.76**	1	Wet digestion (hot block)	HNO3 + H2O2	ICP-OES Agilent 5100	Fe 238.204	N	In House Method ICP-OES
76	**3.70**	0.18	-1.74	**-14.71**	-	-	-	-	-	-	-
77	**10.79**	-	**2.83**	-	-	-	-	-	-	-	-
81	**6.25**	1.07	-0.10	-0.27	Fe 2.0027	Dry Ashing (Ca, Fe)	1 N HNO3 (Ca, Fe)	Shimadzu AAS AA 6300	Fe 248.3	N	AOAC 985.35 Mod (Ca, Fe)
82A	**13.20**	2.83	**4.39**	**4.78**	0.250	none	none	HPGe detector, Canberra	-	-	Neutron Activation Analysis (NAA)
84	**4.68**	0.47	-1.11	**-6.05**	0.5	Microwave Digestion	HNO3 / H2O2	ICP-OES, ICP-MS	-	N	AOAC 999.10:2005
88	**5.11**	0.10	-0.84	**-7.73**	3	Dry Ashing	HNO3 conc 10 mL	AAS GBC Flame	Fe 248.3	N	In house method (AAS)
89	**28.20**	0.42	**14.07**	**82.21**	2	Dry Ashing	1.5% HNO3	AAS Agilent	Various	N	AOAC 985.35
91	**12.10**	-	**3.68**	-	-	-	-	-	-	-	-
94	**2.60**	-	**-2.45**	-	1.5	Fe: Wet ashing	-	ICP-OES / Perkin Elmer	Fe 259.9	Y	AOAC (2012) 984.27
99	**10.40**	0.31	**2.58**	**17.96**	0.3 ± 0.001	Microwave	Nitric Acid & Hydrogen Peroxide	Digestion (MULTI GO Anton Paar) Determination (ICP/MS, PE)	Refer Mass each element	N	EPA 3015A

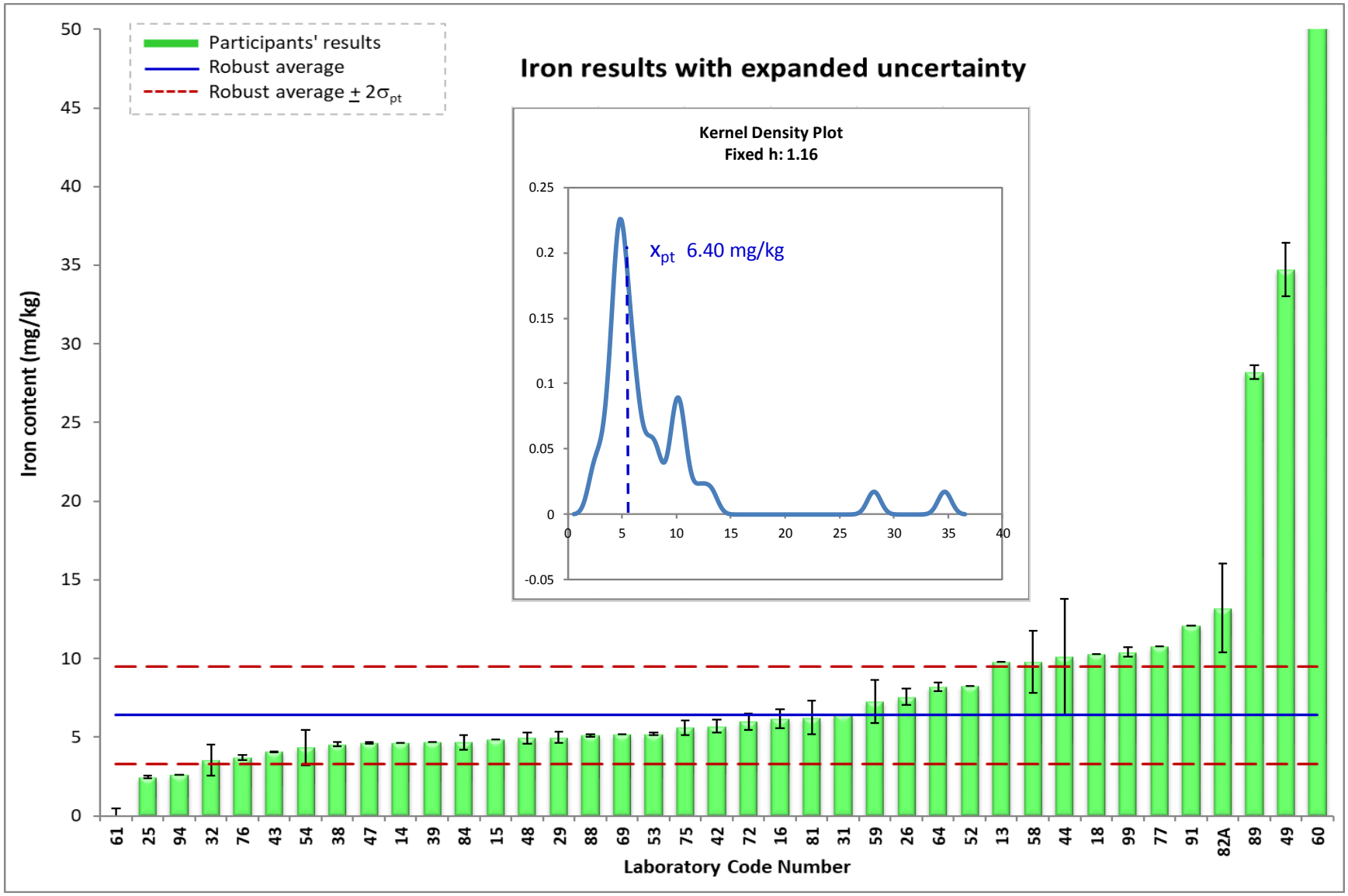

Figure 130. Distribution of **iron** results (ascending order) in rice flour with expanded uncertainty

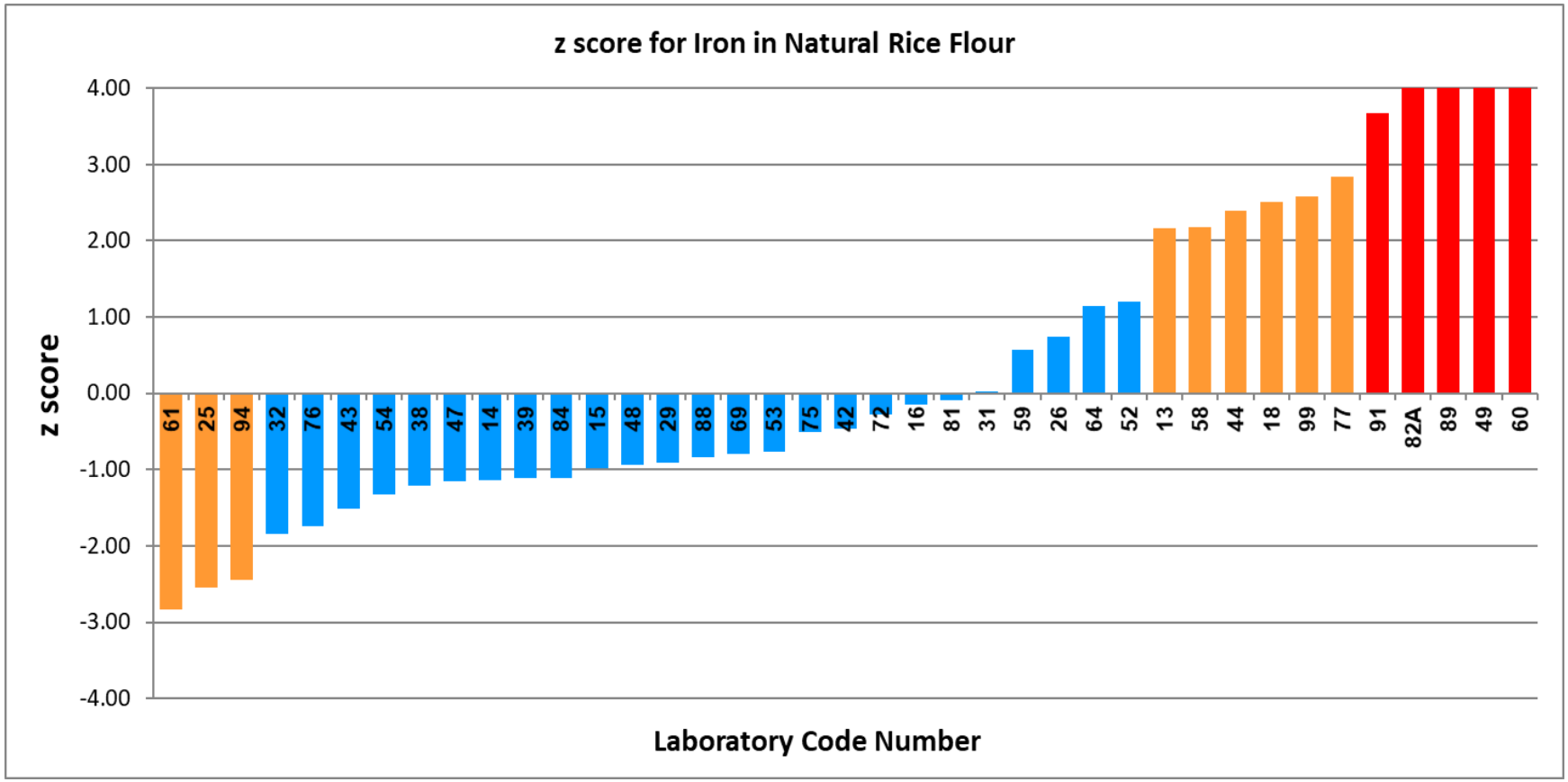

Figure 131. Plot of ordered *z* scores for **iron** results in rice flour

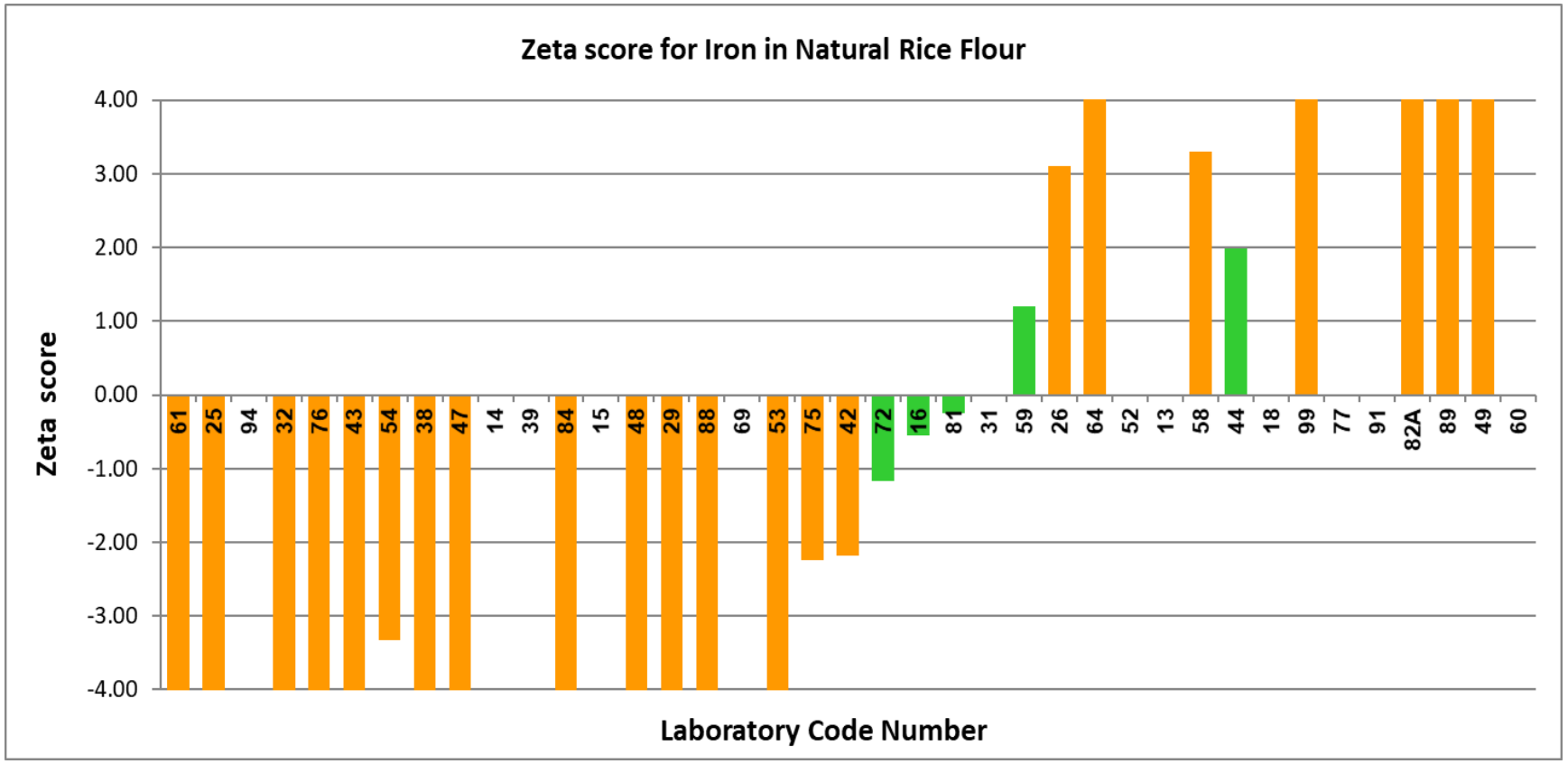

Figure 132. Plot of *Zeta score* for **iron** in rice flour, following the ordered z scores in the above Figure 131.

Table 47. Evaluation of laboratory performance **zinc** analysis (mg/kg, as received) in rice flour

Lab Number	Zinc (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± SDp from Horwitz's equation = 11.88 ± 1.31 mg/kg (CV 11.0%, n= 42) with u_{xpt} 0.27 mg/kg*											
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00							
13	**5.21**	-	**-5.09**	-	0.5	Microwave	HNO3 10 mL + HCl 2 mL	ICP-MS Thermo Scientific (iCAP RQ)	Zn 66	N	Internal Method
14	**11.25**	-	-0.48	-	0.8	Ashing	50% HNO3, 50% HCl	ICP Horiba Jobin Yvon	Zn 213.856	Y	AOAC 975.03, 984.27
15	**11.30**	-	-0.44	-	0.5	Ultrawave Digestion	5% HNO3 + 0.5% HCl	ICP-MS (7900 Agilent)	Zn 66	N	Based on USFDA 4.7 version 1.1
16	**13.20**	1.30	1.01	1.88	2	Microwave	HNO3+H2O2	ICP-MS 7700X Agilent	-	N	In-house Method
18	**11.80**	-	-0.06	-	2.0	Dry Ashing	HCl (HNO3 for Hg, Sn)	ICP-OES Agilent	-	N	SNI 19-2896-1998
25	**2.91**	0.07	**-6.85**	**-32.96**	5.0202 / 5.0205	HNO3-HCl	Water	ICP-OES	Zn 202.548	-	USEPA Method 3050B
26	**11.60**	1.01	-0.21	-0.49	4.0 for Cu, Zn	Dry Ashing	Water & HCl (1+1)	AAS Shimadzu AA-7000	-	N	AOAC No. 975.03
29	**9.79**	0.52	-1.59	**-5.56**	3	Dry Ashing	HCl	-	-	-	-
31	**9.65**	0.54	-1.70	**-5.84**	5	Dry Ashing	-	AAS	-	N	AOAC 999.11
32	**12.70**	0.92	0.63	1.54	2.0541 (Zn)	Ashing	HCl	Flame AAS, Shimadzu 6300	213.9	N	Modified AOAC 969.32
38	**10.90**	0.56	-0.75	**-2.52**	5.000	Dry Ashing	1 N HNO3	Flame AAS, Shimadzu AA6300	Zn 213.90	-	AOAC 985.35 19th Ed 2012

Lab Number	Zinc (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± SDp from Horwitz's equation = 11.88 ± 1.31 mg/kg (CV 11.0%, n= 42) with u_{xpt} 0.27 mg/kg*											
39	**9.69**	-	-1.67	-	0.3	Microwave	-	AAS	324.8	Y	AOAC 985.35
42	**12.10**	0.26	0.17	0.73	10	Dry Ashing	HNO3-HCl	Cu, Zn: Flame AAS 280 FS	Zn 213.9	N	AOAC 999.11.2005
43	**12.26**	1.45	0.29	0.49	0.5	Microwave	HNO3	ICP-OES, ICP-MS	Zn 213.857	N	AOAC
47	**10.90**	0.12	-0.75	**-3.54**	5	Dry Ashing	Nitric Acid	AAS - Shimadzu 7000	Zn 213.9	-	AOAC 999.11
48	**11.63**	0.21	-0.19	-0.87	5	Dry Digestion	-	AA800 Perkin Elmer	Zn 213.9	N	MU-03/20 (AAS)
49	**16.10**	0.80	**3.22**	**8.74**	2 (1, 3 for Zn)	Dry Ashing	6 M HCl (Conc HNO3 for Zn)	AAS / AA-7000 Shimadzu	Zn 213.9	N	AOAC 20th Ed 2016
51	**12.70**	0.58	0.63	**2.07**	5	Dry Ashing	HCl-HNO3	AAS Shimadzu 7000	Zn 213.9	N	AOAC 999.11
52	**22.21**		**7.89**	-	0.5	(Dry Ashing for Zn	5 mL 1N HNO3 for Zn	Flame AAS - Shimadzu AA6300 for Zn	Zn 213.9	N	Modified AOAC 985.35 for Zn
53	**14.78**	0.60	**2.21**	**7.19**	0.3	Microwave	4 mL HNO3, 1 mL HCl, 1 mL H2O2	ICPMS Thermo			In house method
54	**10.70**	0.60	-0.90	**-2.92**	1	Microwave digestion	HNO3 / H2O2	ICP / Shimadzu	Zn 213.856	N	AOAC 984.27
57	**9.10**	1.99	**-2.12**	**-2.70**	3.0255	Dry Ashing	HCl, HNO3	AAS	213.9	Y	AOAC 999.11
58	**39.30**	2.32	**20.93**	**23.02**	1.0	Acid Digestion	HNO3, H2O2	ICP-OES	-	-	Acid Digestion and Quantitation by ICP-OES

Lab Number	Zinc (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± SDp from Horwitz's equation = 11.88 ± 1.31 mg/kg (CV 11.0%, n= 42) with u_{xpt} 0.27 mg/kg*											
59	**7.38**	0.70	**-3.44**	**-10.18**	Cu, Zn: 10	Dry Ashing	-	AAS, Shimadzu	Zn 213.9	Y	Pb+Cd+Zn: SNI 3751:2009 point A.14.1 (11 for Zn)
60	**300.00**	-	**219.94**	-	-	-	-	-	-	-	SNI 01-2896-1998 (Zn)
61	**12.00**	3.29	0.09	0.07	1	Acid block digestion	HNO3	Varian AA240 FS Fast Sequential AAS	Zn 213.9	N	A6407-26 AAS
64	**13.89**	0.08	1.53	**7.36**	0.5063	Dry Ashing	1 N HNO3	Shimadzu AA6300	Zn 213.9	N	Modified AOAC 985.35
69	**12.30**	-	0.32	-							
72	**15.40**	1.30	**2.69**	**5.00**	4	Ashing	HNO3	ICP-OES, JY Ultima	Zn 213.9	N	AOAC 999.11
73A	**25.39**	7.87	**10.31**	**3.43**	1	Dry Ashing	Hot plate	AAS (280FS AA, Agilent Technology)	213.9	N	FTC-46.01 (refers to AOAC 968.08, 965.09)
75	**13.56**	0.96	1.28	3.04	1	Wet digestion (hot block)	HNO3 + H2O2	ICP-OES Agilent 5100, ICP-MS Agilent 7700x	Zn 213.857	N	In House Method ICP-MS & ICP-OES
76	**11.15**	1.30	-0.56	-1.04	-	-	-	-	-	-	-
77	**9.63**	-	-1.72	-	-	-	-	-	-	-	-
78	**11.90**	0.20	0.02	0.07	-	-	-	-	-	-	-
81	**11.90**	1.80	0.02	0.02	mean 2.0042 (Zn)	Zn: Dry Ashing	Zn: 1 N HNO3	Zn: Shimadzu AAS AA 6300	Zn 213.9	N	AOAC 985.35 Mod (Zn)

Lab Number	Zinc (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± SDp from Horwitz's equation = 11.88 ± 1.31 mg/kg (CV 11.0%, n= 42) with u_{xpt} 0.27 mg/kg*											
82A	**14.20**	0.42	1.77	**6.78**	0.250	none	none	HPGe detector, Canberra	-	N	Neutron Activation Analysis (NAA)
84	**11.40**	0.11	-0.37	-1.74	0.5	Microwave Digestion	HNO3 / H2O2	ICP-MS	-	N	AOAC 999.10:2005
88	**36.90**	-	**19.10**	-	0.3 (Cu, Zn 3)	Microwave (Cu, Zn Dry Ashing)	H2O2 2 ml + HNO3 8 mL (Cu, Zn: HNO3 10 mL)	AAS GBC Hydride vapour (Cu, Zn: AAS GBC Flame)	Zn 213.90	N	In house method (AAS)
91	**9.33**	-	-1.95	-	-	-	-	-	-	-	-
94	**10.10**	-	-1.36	-	-	-	-	-	-	-	-
95	**19.50**	2.00	**5.82**	**7.36**	-	-	-	-	-	-	-
99	**10.50**	0.02	-1.05	**-5.11**	0.3 ± 0.001	Microwave	Nitric Acid & Hydrogen Peroxide	Digestion (MULTI GO Anton Paar) Determination (ICP/MS, PE)	Refer Mass each element	N	EPA 3015A

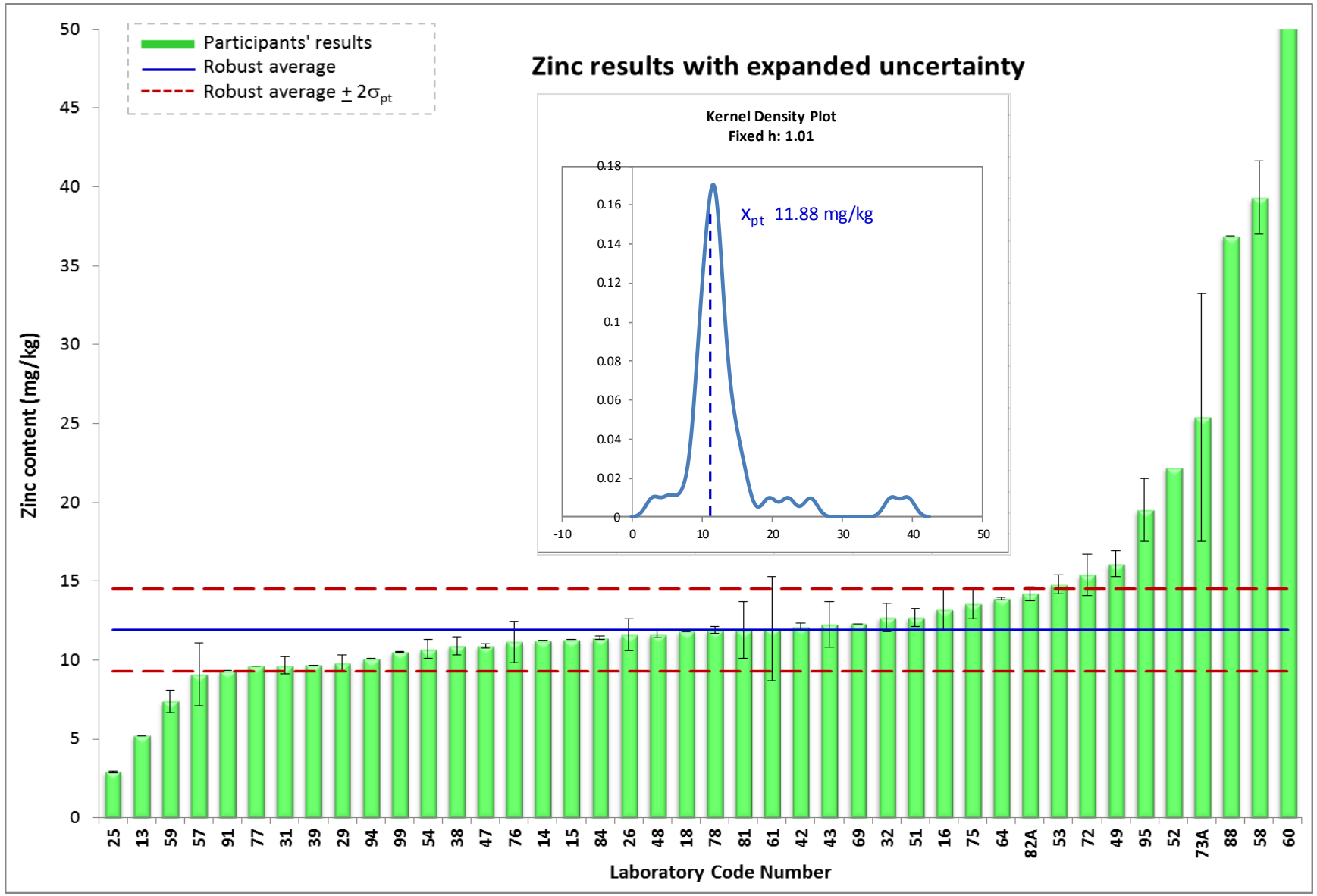

Figure 133. Distribution of **zinc** results (ascending order) in rice flour with expanded uncertainty

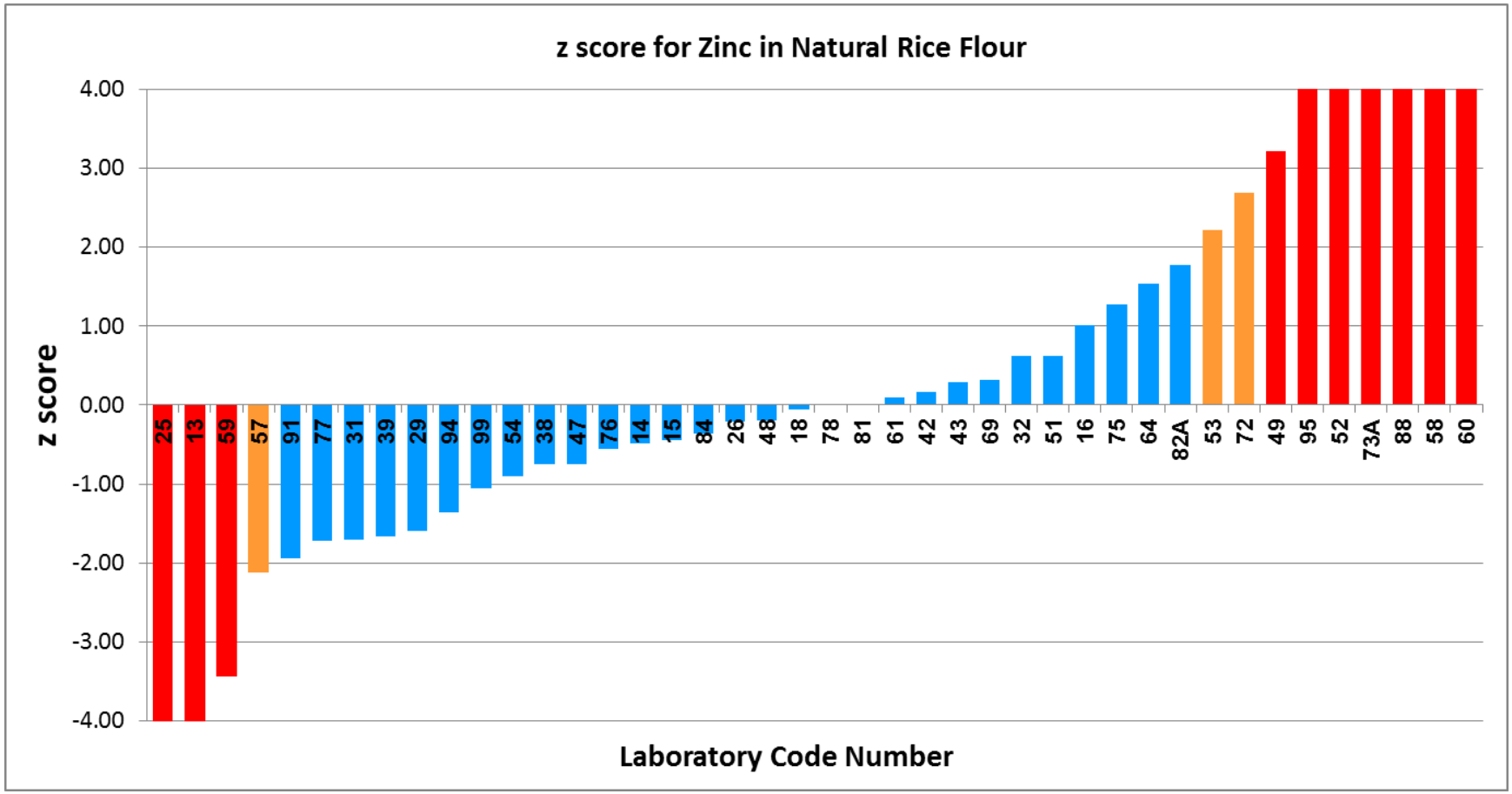

Figure 134. Plot of ordered *z* scores for **zinc** results in rice flour

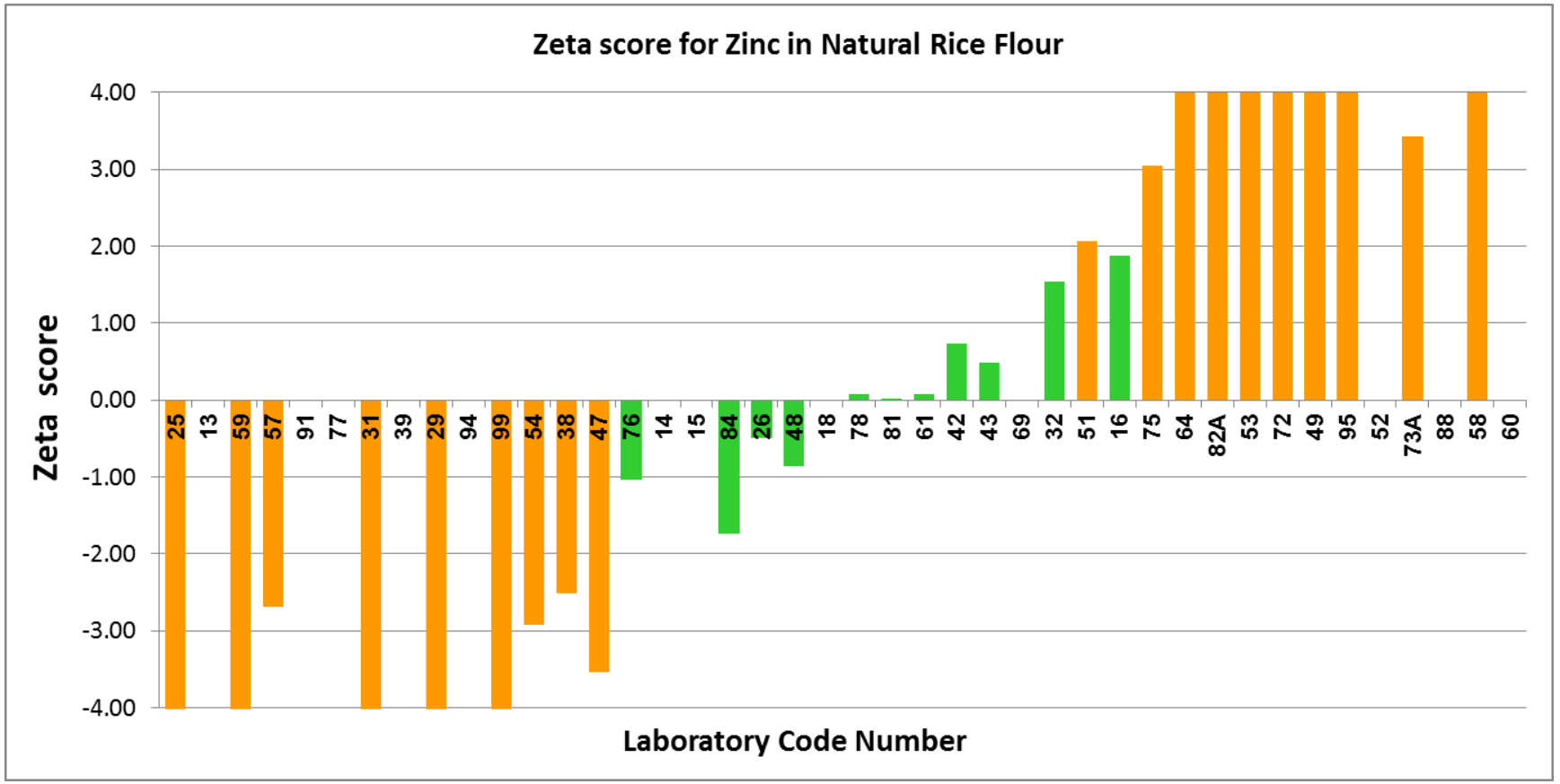

Figure 135. Plot of *Zeta score* for **zinc** in rice flour, following the ordered z scores in the above Figure 134.

Table 48. Evaluation of laboratory performance **copper** analysis (mg/kg, as received) in rice flour

Lab Number	Copper (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± SDp from Horwitz's equation = 1.77 ± 0.26 mg/kg (CV 14.7%, n= 34) with u_{xpt} 0.06 mg/kg*											
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00							
13	**1.07**		**-2.69**	-	0.5	Microwave	HNO3 10 mL + HCl 2 mL	ICP-MS Thermo Scientific (iCAP RQ)	M/z Cu: 63	N	Internal Method
14	**1.66**		-0.43	-	0.8	Ashing	50% HNO3, 50% HCl	ICP Horiba Jobin Yvon	Cu 224.70	Y	AOAC 975.03, 984.27
15	**1.88**		0.42	-	0.5	Ultrawave Digestion	5% HNO3 + 0.5% HCl	ICP-MS (7900 Agilent)	Cu 65	N	Based on USFDA 4.7 version 1.1
16	**1.45**	0.15	-1.23	**-3.33**	2	Microwave	HNO3+H2O 2	ICP-MS 7700X Agilent		N	In-house Method
18	**1.45**		-1.23	-	2.0	Dry Ashing	HCl (HNO3 for Hg, Sn)	ICP-OES Agilent	-	N	SNI 19-2896-1998
25	**1.35**	0.07	-1.62	**-6.09**	5.0202 / 5.0205	HNO3-HCl	Water	ICP-OES	Cu 327.395		USEPA Method 3050B
26	**2.14**	0.05	1.42	**5.74**	4.0 for Cu, Zn	Dry Ashing	Water & HCl (1+1)	AAS Shimadzu AA-7000	-	N	AOAC No. 975.03
29	**0.67**	0.17	**-4.22**	**-10.51**	3	Dry Ashing	HCl	-			
31	**0.72**	0.10	**-4.05**	**-13.72**	5	Dry Ashing	-	AAS		N	AOAC 999.11
38	**4.65**	1.76	**11.08**	**3.27**	5.000	Dry Ashing	1 N HNO3	Flame AAS, Shimadzu AA6300	Cu 324.80		AOAC 985.35 19th Ed 2012
39	**1.14**		**-2.42**	-	0.3	Microwave	-	AAS	324.8	Y	AOAC 985.35
42	**1.60**	0.02	-0.65	**-2.80**	10	Dry Ashing	HNO3-HCl	Cu, Zn: Flame AAS 280 FS	Cu 324.8	N	AOAC 999.11.2005

Lab Number	Copper (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± SDp from Horwitz's equation = 1.77 ± 0.26 mg/kg (CV 14.7%, n= 34) with u_{xpt} 0.06 mg/kg*											
43	**1.69**	0.10	-0.31	-1.02	0.5	Microwave	HNO3	ICP-OES, ICP-MS	Cu 327.395	N	AOAC
47	**1.72**	0.03	-0.19	-0.81	5	Dry Ashing	Nitric Acid	AAS - Shimadzu 7000	Cu 324.8		AOAC 999.11
48	**1.72**	0.19	-0.18	-0.43	5	Dry Digestion	-	AA800 Perkin Elmer	Cu 324.8	N	MU-03/20 (AAS)
49	**0.27**	0.01	**-5.77**	**-24.82**	2 (1, 3 for Zn)	Dry Ashing	6 M HCl (Conc HNO3 for Zn)	AAS / AA-7000 Shimadzu	Cu 327.395	N	AOAC 20th Ed 2016
51	**4.16**	0.37	**9.19**	**12.41**	5	Dry Ashing	HCl-HNO3	AAS Shimadzu 7000	Cu 324.8	N	AOAC 999.11
53	**2.07**	0.10	1.15	**3.84**	0.3	Microwave	4 mL HNO3, 1 mL HCl, 1 mL H2O2	ICPMS Thermo			In house method
54	**0.69**	0.04	**-4.17**	**-17.10**	1	Microwave digestion	HNO3 / H2O2	ICP / Shimadzu	Cu 324	N	AOAC 984.27
59	**1.82**	0.39	0.19	0.25	Cu, Zn: 10	Dry Ashing	-	AAS, Shimadzu	Cu 324	Y	Cu+As: IK A2-LM06 (AAS)
61	**0.90**	0.28	**-3.35**	**-5.71**	1	Acid block digestion	HNO3	Varian AA240 FS Fast Sequential AAS	Cu 324.8	N	A6407-26 AAS
69	**2.60**		**3.19**	-							

Lab Number	Copper (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength (nm)	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± SDp from Horwitz's equation = 1.77 ± 0.26 mg/kg (CV 14.7%, n= 34) with u_{xpt} 0.06 mg/kg*											
72	**5.50**	0.50	**14.35**	**14.51**	4	Ashing	HNO3	ICP-OES, JY Ultima	Cu 324.754	N	AOAC 999.11
75	**1.47**	0.14	-1.15	-3.30	1	Wet digestion (hot block)	HNO3 + H2O2	ICP-OES Agilent 5100, ICP-MS Agilent 7700x	63 Cu 324.754	N	In House Method ICP-MS & ICP-OES
76	**1.90**	0.24	0.50	0.97	-	-	-	-	-	-	-
77	**1.58**	-	-0.73	-	-	-	-	-	-	-	-
78	**2.58**	-	**3.12**	-	-	-	-	-	-	-	-
84	**1.63**	0.16	-0.54	-1.40	0.5	Microwave Digestion	HNO3 / H2O2	ICP-MS	-	N	AOAC 999.10:2005
88	**8.41**	0.04	**25.55**	**105.04**	0.3 (Cu, Zn 3)	Cu, Zn Dry Ashing	Cu, Zn: HNO3 10 mL	Cu, Zn: AAS GBC Flame	Cu 324.70	N	In house method (AAS)
91	**0.59**	-	**-4.53**	**-19.65**	-	-	-	-	-	-	-
94	**1.80**	-	0.12	0.50	-	-	-	-	-	-	-
99	**2.55**	0.05	**3.00**	**11.96**	0.3 ± 0.001	Microwave	Nitric Acid & Hydrogen Peroxide	Digestion (MULTI GO Anton Paar) Determination (ICP/MS, PE)	Refer Mass each element	N	EPA 3015A

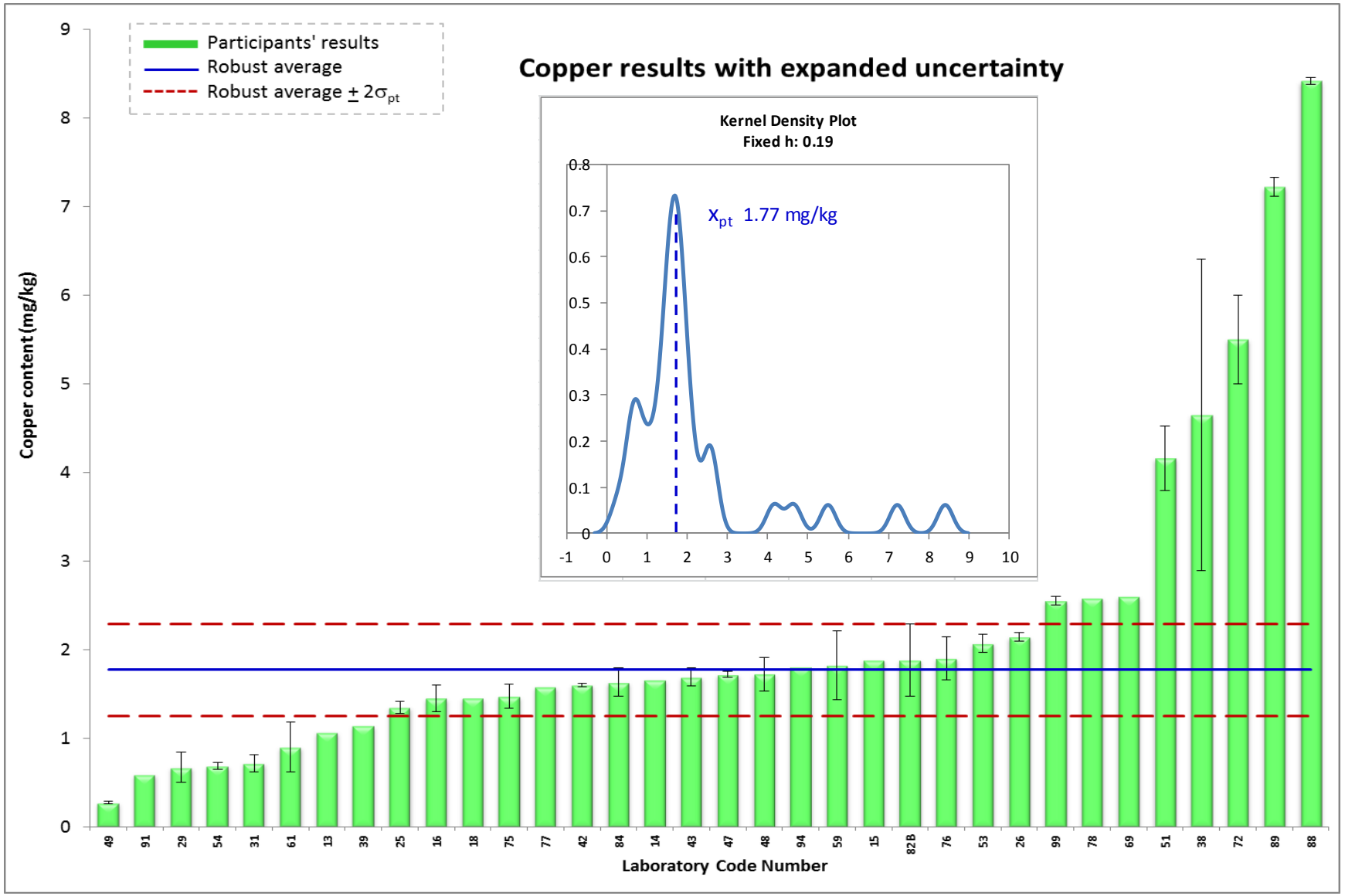

Figure 136. Distribution of **copper** results (ascending order) in rice flour with expanded uncertainty

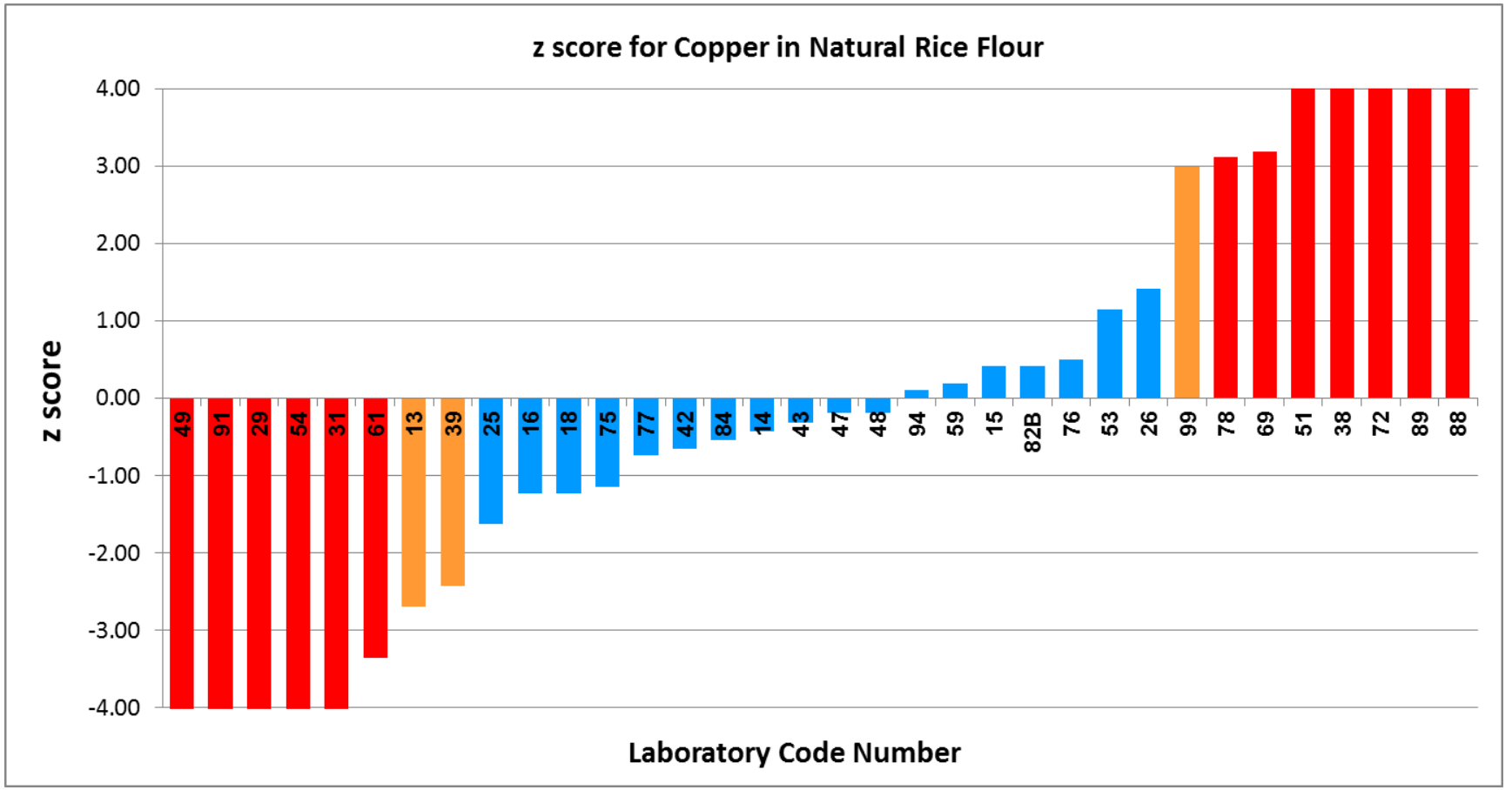

Figure 137. Plot of ordered *z* scores for **copper** results in rice flour

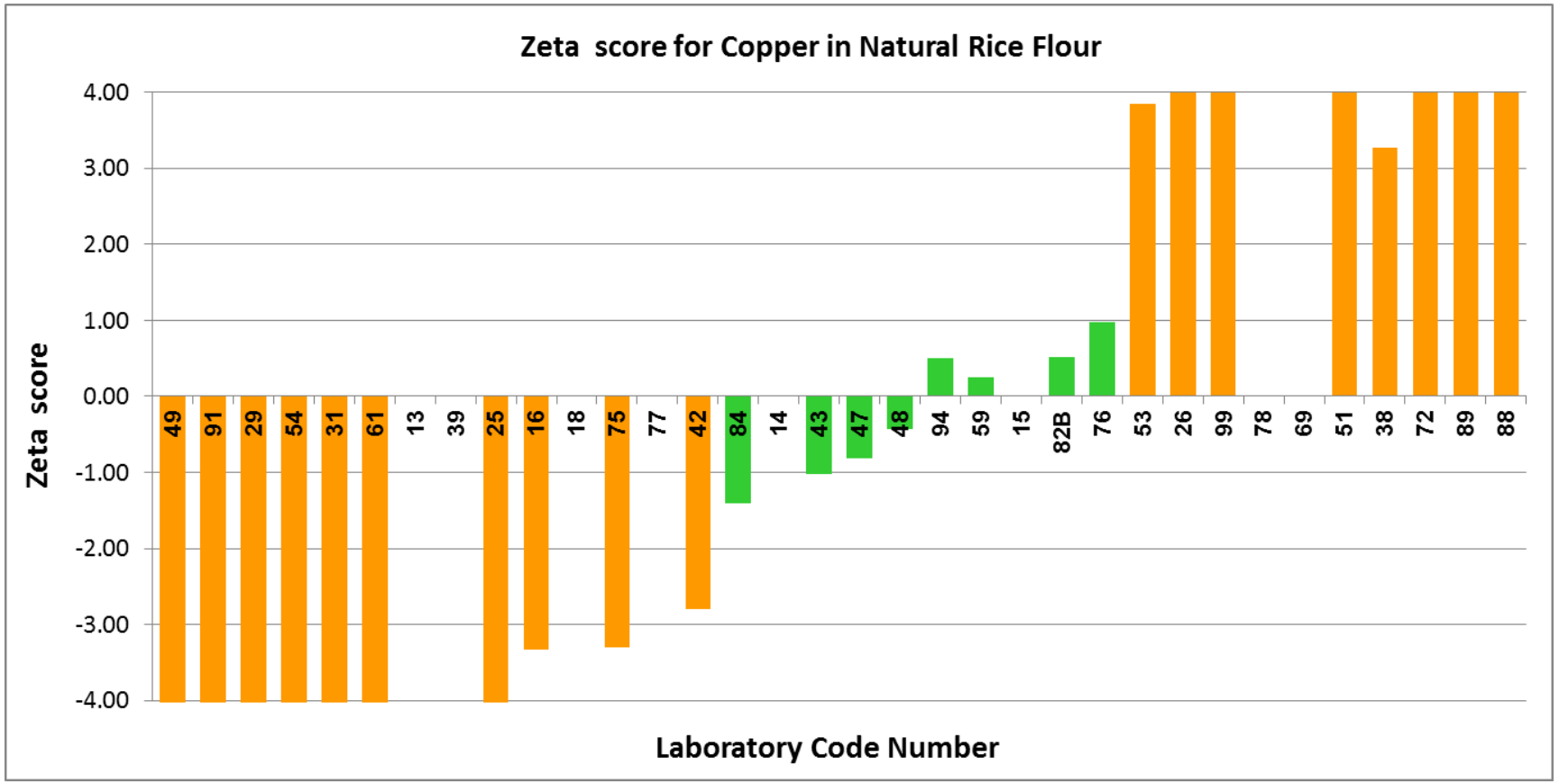

Figure 138. Plot of *Zeta score* for **copper** in rice flour, following the ordered z scores in the above Figure 137.

Table 49. Summary: evaluation of laboratory performance in rice flour

Parameters	Total participating laboratory	Evaluation results (number of laboratory, percentage in bracket)		
		Satisfactory	Questionable	Unsatisfactory
Moisture (g/100g)	59	53 (89.8%)	3 (5.1%)	3 (5.1%)
Nitrogen (g/100g)	49	39 (79.6%)	3 (6.1%)	7 (14.3%)
Ash (g/100g)	49	32 (65.3%)	5 (10.2%)	12 (24.5%)
Dietary fibre (g/100g)	20	5 (25.0%)	2 (10.0%)	13 (65.0%)
Calcium (mg/kg)	47	24 (51.1%)	3 (6.4%)	20 (42.6%)
Phosphorus (mg/kg)	28	18 (64.3%)	2 (7.1%)	8 (28.6%)
Sodium (mg/kg)	43	28 (65.1%)	3 (7.0%)	12 (27.9%)
Potassium (mg/kg)	41	30 (73.2%)	5 (12.2%)	6 (14.6%)
Iron (mg/kg)	39	25 (64.1%)	9 (23.1%)	5 (12.8%)
Zinc (mg/kg)	42	29 (69.0%)	3 (7.1%)	10 (23.8%)
Copper (mg/kg)	34	18 (52.9%)	2 (5.9%)	14 (41.2%)