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):

Fish Meal

by

Kunchit Judprasong, Prapasri Puwastien,

Stewart Jones, Piyanut Sridonpai, Preecha Saetang

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Table 19. Summary: assigned values of measurands for evaluation of testing parameters in fish meal

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*	9.26	0.72	7.8	0.10	0.22	Yes, use z score
Nitrogen (g/100g)	x* & s*	8.89	0.20	2.2	0.03	0.06	Yes, use z score
Fat (g/100g)	x* & $3SD_p$	7.30	0.65	8.9	0.10	0.19	Yes, use z score
Ash (g/100g)	x* & s*	27.27	0.28	1.0	0.04	0.08	Yes, use z score
Calcium (mg/kg)	x* & SD_p	76533	6761	8.8	1172	2028	Yes, use z score
Magnesium (mg/kg)	x* & s*	3130	443	14.3	85	133	Yes, use z score
Phosphorus (mg/kg)	x* & s*	43451	2497	5.7	543	749	Yes, use z score
Sodium (mg/kg)	x* & s*	9943	1367	13.7	249	410	Yes, use z score
Potassium (mg/kg)	x* & s*	7276	724	10.0	135	217	Yes, use z score
Iron (mg/kg)	x* & $2SD_p$	941.9	107.5	11.4	15.2	32.3	Yes, use z score
Zinc (mg/kg)	x* & s*	86.69	12.57	14.5	2.45	3.77	Yes, use z score
Copper (mg/kg)	x* & $2SD_p$	3.70	0.97	26.3	0.15	0.29	Yes, use z score
Lead (mg/kg)	x* & $2SD_p$	1.60	0.48	29.8	0.09	0.14	Yes, use z score
Arsenic (mg/kg)	x* & $2SD_p$	6.84	1.20	24.0	0.28	0.36	Yes, use z score
Cholesterol (mg/100g)	x* & s*	496.80	63.10	12.7	15.18	18.93	Yes, use z score
TVBN (mg/100g)	x* & s*	227.92	12.35	5.4	3.45	3.71	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 $\pm$ SD = Mean and standard deviation derived from gravimetric standard addition IDMS from NIMT
Med. & NIQR = Median and normalised interquartile range

Table 20. Evaluation of laboratory performance on **moisture** analysis (g/100 g, as received) in fish meal

Laboratory Number	Moisture (g/100g)	MU (g/100g)	z score	Zeta score	Sample weight (g)	Temp. (°C)	Time (Hours)	Method Reference
Assigned value obtained from robust average (x) ± robust SD (s*) = 9.26 ± 0.72 g/100 g (CV 7.8%, n= 76) with standard uncertainty of 0.10 g/100g*								
	Acceptance criteria =		|z score| ≤ 2.00	| ζ score| ≤ 2.00				
2	**9.85**	0.30	0.82	**3.27**	2.00	125	2	AOAC (2016) 950.46 (B)
5	**9.29**	-	0.04	-	5.0000	103	4	ISO 6496:1999
6	**9.21**	0.13	-0.06	-0.38	5.0000	103	4.00	ISO 6496
7	**8.90**	0.10	-0.50	**-3.22**	1.0306/1.1313	105	5	AOAC
9	**9.31**	-	0.07	-	5 to 10	105	4	Based on ISO 6496:1999
11	**9.30**	-	0.06	-	5.0000	103	4	AOAC (2016) 925.10
14	**9.22**	0.58	-0.06	-0.14	5	103 + 2	4	ISO6496:1999
15	**9.26**	-	0.00	-	5	103	4	ISO 6496:1999
16	**9.20**	0.16	-0.08	-0.47	1 to 2	105	3	SNI 01-2891-1992 Food & Beverage
17	**9.29**	0.11	0.05	0.30	2.000	125	2	AOAC
18	**9.18**	-	-0.11	-	2.0	105	3	SNI 01-2891-1992
19	**9.29**	-	0.05	-	5	105	3	AOAC 934.01
21	**10.50**	0.04	1.72	**12.18**	2	130	2	AOAC 925.10 (2016)
22	**8.42**	-	-1.17	-				
23	**9.17**	-	-0.13	-	5.00	103	4	ISO 6496
24	**8.78**	0.14	-0.67	**-3.93**	2.000	105	3 until constant	SNI 01-2891-1992
25	**9.58**	-	0.44	-	5.1206 / 5.1702	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*) = 9.26 ± 0.72 g/100 g (CV 7.8%, n= 76) with standard uncertainty of 0.10 g/100g								
26	**9.47**	0.05	0.29	**2.03**	5.0	100	4	AOAC No. 952.08 A
27	**9.17**	0.26	-0.13	-0.55	2	105	22	SNI 2354.2:2015
31	**9.14**	0.32	-0.17	-0.64	3	105	3	SNI
32	**8.92**	0.18	-0.47	**-2.53**	1.9794	100	8	AOAC 934.04
33	**10.80**	1.51	**2.14**	**2.02**	2.0139, 2.0447,	135	2	Method 930.15, OMA AOAC, 18th Ed
34	**9.00**	-	-0.36	-	2.000	105	18	SNI 01.2354.2:2006
37	**9.14**	-	-0.17	-	3	105	4	AOAC (2016) 925.45
38	**9.62**	0.20	0.50	**2.53**	2.000	135	2	AOAC 930.15, 19th Ed 2012
39	**7.78**	-	**-2.06**	-	1	100 + 2	4	AOAC 952.08
41	**9.98**	0.08	1.00	**6.67**	2	135 + 2	2	AOAC (2016) 930.15
42	**9.76**	0.11	0.69	**4.39**	2	135	2	AOAC 2005
43	**10.21**	0.33	1.32	**4.88**	2	130	2 to constant	AOAC, National Standard
44	**9.64**	0.68	0.53	1.07	2.0034	135	2.0	AOAC 19th Ed, 2012
45	**9.11**	0.11	-0.20	-1.28	5 + 0.3	103 + 2	4 + 1	ISO 6496
46	**7.68**	0.06	**-2.19**	**-15.19**	3.00	120.0	NA	In House Developed Method
48	**9.09**	0.16	-0.24	-1.34	1	105	3 then 1 then 1	SNI 01-2891-1992
49	**10.20**	0.20	1.31	**6.65**	2	135	2	AOAC 20th Ed 2016
50	**10.50**	0.23	1.72	**8.20**	2.1804	130	1.0	AOAC 925.10
52	**11.53**	0.10	**3.15**	**20.34**	2.0	135	2.0	AOAC 930.15
54	**9.32**	0.09	0.08	0.55	1	105	5	AOAC 927.05

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*) = 9.26 ± 0.72 g/100 g (CV 7.8%, n= 76) with standard uncertainty of 0.10 g/100g								
55	**8.96**	0.02	-0.42	**-3.03**	10	100	4	AOAC (2012) 952.08
58	**8.77**	0.18	-0.68	**-3.64**	2 to 5	130 / 105	3	Based on AOAC 20th Ed 2016
60	**9.08**	-	-0.25	-				SNI 01-2891-1992 Butir 5.1
61	**9.56**	1.45	0.42	0.41	3	130	1.5	A6801 130C Air oven
63	**9.19**	-	-0.10	-				
64	**13.05**	0.14	**5.26**	**31.05**	2.0356	135	2 hours + 5 mins	AOAC 930.15
66A	**9.14**	0.12	-0.17	-1.03	10.0029	130.0	0.50	AOCS Official Method Ca 2c-25, 7th Ed., 2017
66B	**8.64**	0.12	-0.86	**-5.32**	10.0637	130.0	0.50	AOCS Official Method Ca 2c-25, 7th Ed., 2017
66C	**8.80**	0.12	-0.64	**-3.94**	10.0124	130.0	0.50	AOCS Official Method Ca 2c-25, 7th Ed., 2017
67	**9.09**	0.25	-0.24	-1.06	5.0xxx	103	4.0	ISO 6496
68	**11.00**	-	**2.42**	-	2	135	2	AOAC
69	**8.69**	-	-0.79	-				
70	**8.08**	0.50	-1.64	**-4.38**	5	105	5	
71	**9.20**	0.29	-0.08	-0.34	4.9997, 4.9989	105	3	AOAC 934.01
72	**8.19**	0.01	-1.49	**-10.69**	2	130	1	AOAC 925.10
73A	**8.98**	0.38	-0.39	-1.30	5	105	3	FTC-01.01 (refers to AOAC 945.39)
74	**8.48**	-	-1.08	-	5.0	105	3	SNI 01-2891-1992 (part 5.1)
75	**7.92**	0.21	-1.86	**-9.17**	2	105 + 2	4	SNI 01-2891-1992 Butir 5.1
77	**9.20**	-	-0.08	-				
79	**8.30**	0.74	-1.34	**-2.52**	1 to 2	105	3	SNI 01-2891-1992 Butir 5.1
80	**10.60**	-	1.86	-	2.xx	135	2	AOAC 930.15

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*) = 9.26 ± 0.72 g/100 g (CV 7.8%, n= 76) with standard uncertainty of 0.10 g/100g								
81	**8.97**	0.01	-0.40	**-2.90**	4.0921 mean	100	4 then 0.5 until <0.005	AOAC 952.08A Modified
82A	**9.57**	0.13	0.43	**2.60**	1.00	105	7.5	Drying Oven
82B	**9.57**	0.13	0.43	**2.60**	1.00	105	7.5	Drying Oven
83	**8.54**	0.24	-1.00	**-4.67**	2	105	5	SNI-01-2891-1992
84	**9.05**	-	-0.29	-	2	100	5	AOAC 934.01
85	**8.88**	0.01	-0.53	**-3.80**	2	105	3	SNI 01-2896-1992
86	**11.10**	0.53	**2.56**	**6.50**	2	135	2	AOAC (2012) 930.15
87	**8.65**	0.02	-0.85	**-6.11**	1.5	105	3	MTD/FOD/CHM-01
88	**8.30**	0.04	-1.34	**-9.47**	1.5	105	3	SM 01-2891-1992 Point 5.1
89	**8.57**	0.15	-0.96	**-5.50**	2	100 to 105	4	AOAC 925.23
90	**11.72**	0.28	**3.41**	**14.29**	2	130	1	AOAC (2016) 930.15
91	**9.82**	0.01	0.78	**5.59**				
92	**9.62**	-	0.50	-	5	103	4	ISO 6494
93	**7.91**	-	-1.88	-	2	95 to 100	5	AOAC 934.01
94	**11.20**	-	**2.69**	-	1	130	5	AOAC (2000) 925.10
95	**9.04**	0.06	-0.31	**-2.16**				
96	**10.00**	-	1.03	-	3	125	4	TCVN 4846:1989
100	**9.35**	0.16	0.13	0.70	5	103	4	ISO 6496

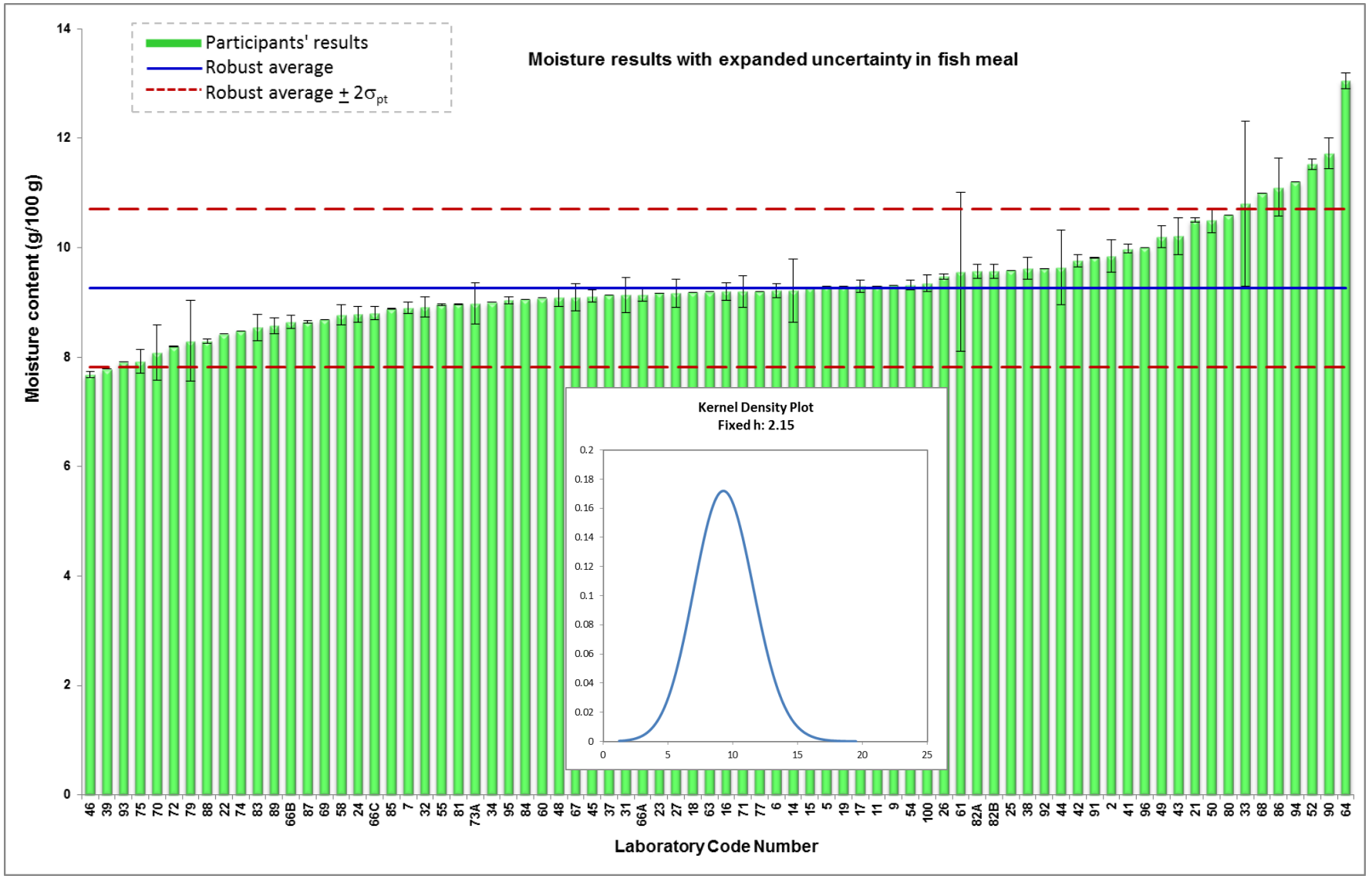

Figure 58. Distribution of **moisture** results (ascending order) in fish meal with expanded uncertainty

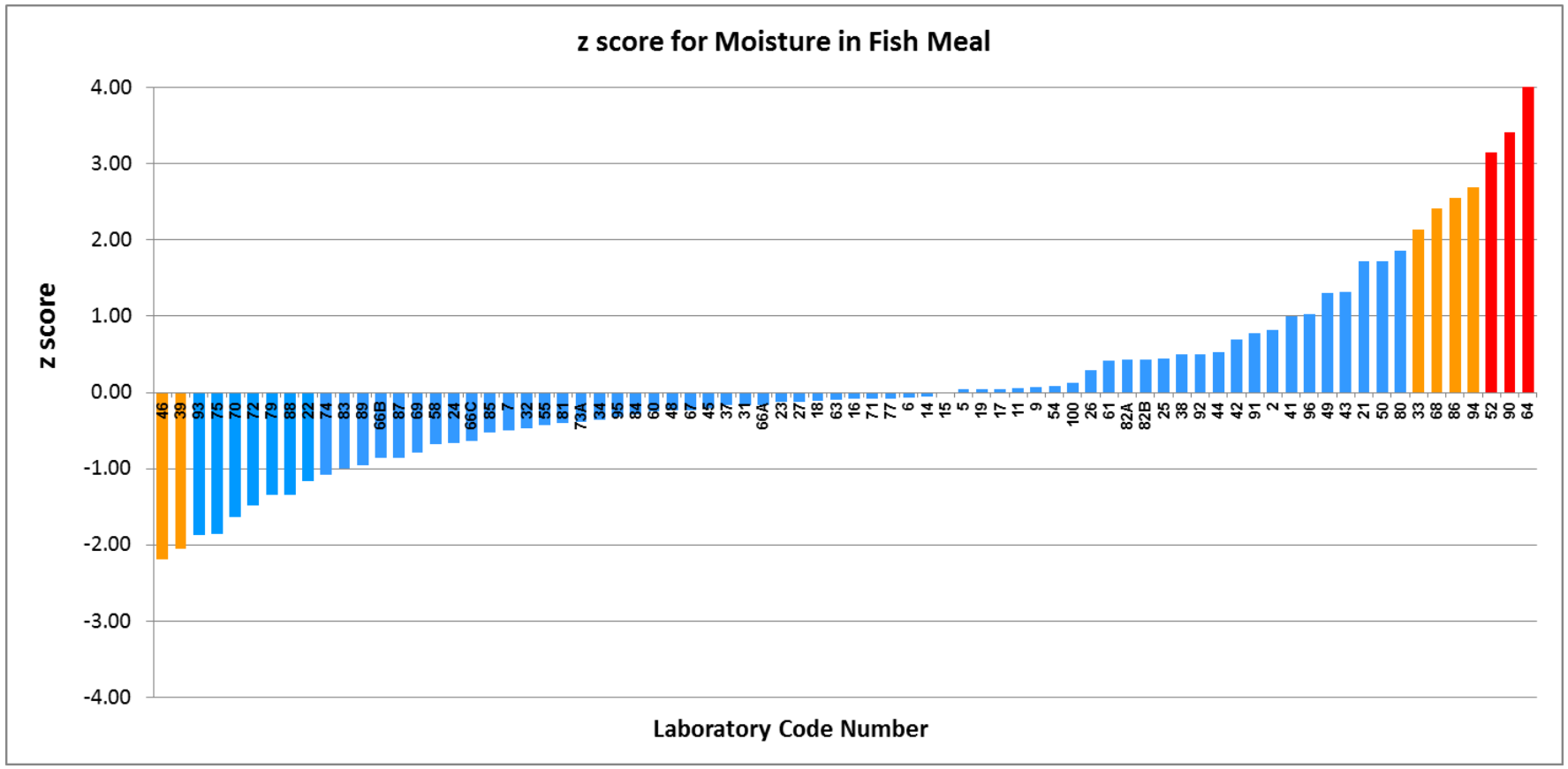

Figure 59. Plot of ordered *z* scores for **moisture** results in fish meal

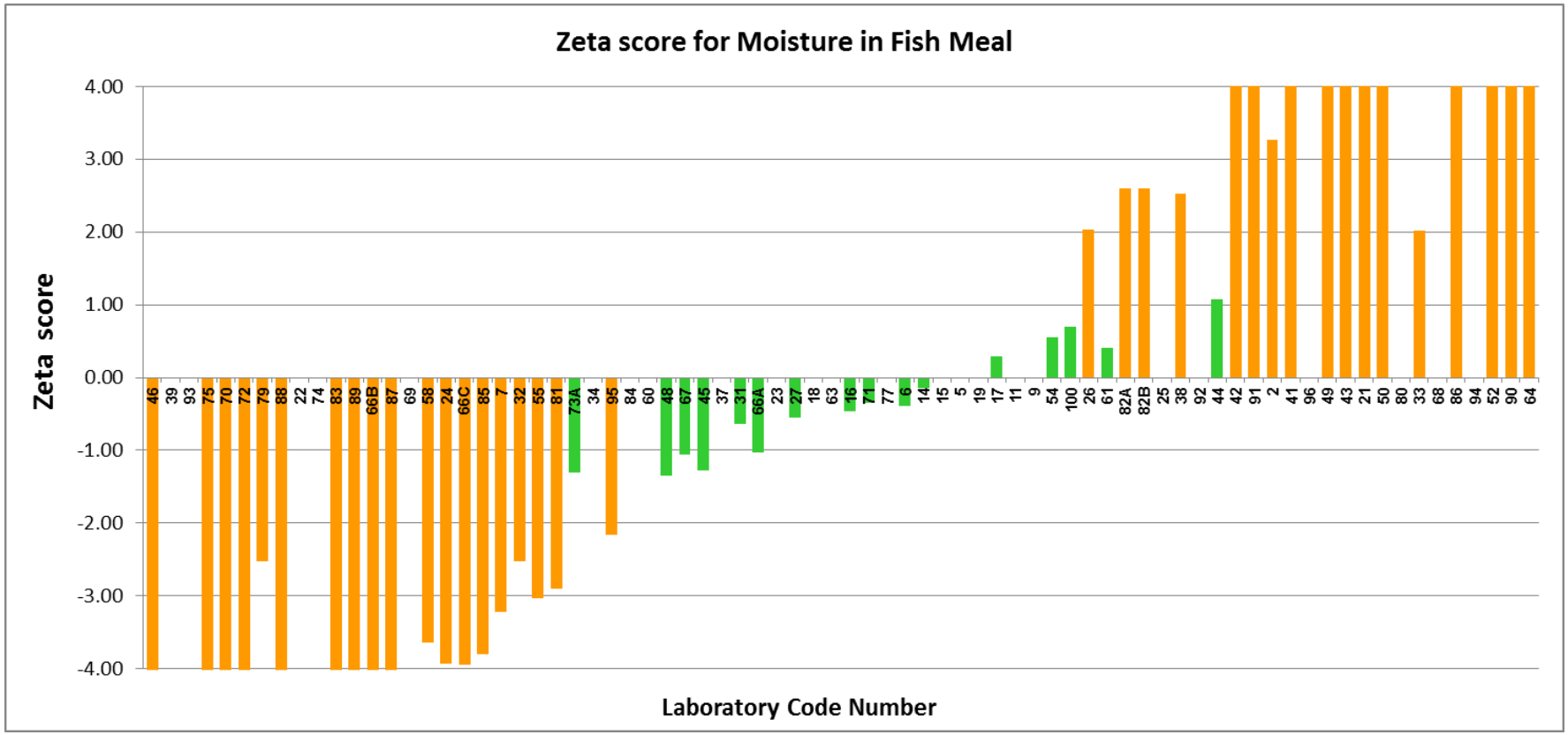

Figure 60. Plot of *Zeta* score for **moisture** fish meal, following the ordered z scores in the above Figure 59.

Table 21. Evaluation of laboratory performance on **total nitrogen** analysis (g/100 g, as received) in fish meal

Laboratory Number	Total Nitrogen	MU	z score	Zeta score	Nitrogen Weight	Catalyst	Acid Volume (mL)	Receiver Solution and Vol	Titrant	Conversion Factor	Nitrogen Reference
Assigned value obtained from robust average (x) ± robust SD (s*) = 8.89 ± 0.20 g/100 g (CV 2.2%, n= 67) with standard uncertainty 0.03 g/100g*											
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| < 2.00							
2	**9.01**	0.27	0.60	0.87	0.50	CuSO4+K2SO4	20	50	HCl 0.1 M	6.25	AOAC (2016) 981.10
5	**8.61**		-1.42	-	1.0000	CuSO4+K2SO4	13 mL conc H2SO4	30 mL 4% Boric Acid	0.5 N H2SO4	-	AOAC 20th Ed, 2016, 2001.11
6	**9.05**	0.73	0.79	0.43	0.5074	K2PO4 + CuPO4	15	55 mL 4% Boric Acid	0.2036 N H2SO4	6.25	ISO 5983-2
7	**5.10**	-	**-18.95**	-	1.0006/ 1.0116	Kjeltab	25	25	HCL/0.1097	-	AOAC
9	**8.67**	-	-1.13	-	1	K2SO4/CuSO4	H2SO4 / 12.5 mL	Boric acid 30 mL	0.5 N H2SO4	-	Based on AOAC 20th Ed, 2016, 2001.11, Chapter 4
11	**8.60**	-	-1.45	-	0.5000	1 mL	H2SO4 / 25 mL	Boric acid solution 25 mL	0.1 N H2SO4	6.25	Manual on fertilizer analysis, Arsrod, Doa 12/2551
14	**8.98**	0.17	0.45	0.99	0.5	K2SO4:CuSO4.5 H2O (9:1)	H2SO4 12 mL	4% Boric Acid 30 mL	0.1 M HCl	6.25	ISO 5983-2:2005
15	**8.98**	-	0.45	-	0.13	CuSO4.5H2O, K2SO4	H2SO4 12 mL	1% H3BO4	0.1 M HCl	6.25	ISO 5983-2:2009
16	**9.02**	0.27	0.65	0.94	0.5 - 1	Selenium	Sulphuric Acid 25 mL	Boric Acid 50 mL	HCl 0.1 N	-	SNI 01-2891-1992 Food & Beverage
17	**9.66**	0.18	**3.85**	**8.12**	-	-	-	-	-	-	-
18	**8.99**	-	0.50	-	2.0	CuSO4, SeO2	H2SO4, 25 mL	H3BO3 2%, 25 mL	HCl 0.1 M	-	SNI 01-2891-1992
19	**8.92**	-	0.14	-	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	**8.75**	-	-0.70	-	0.5 to 1.0	Se, K2SO4	H2SO4 15 mL	Boric acid (AR) 25 mL	HCl (AR) 0.1 N	-	AOAC 930.29 (2016)
22	**8.79**	-	-0.50	-	-	-	-	-	-	-	-
23	**8.92**	-	0.15	-	0.20	-	-	-	-	-	AOAC 992.15
24	**9.16**	0.30	1.33	1.76	1.000	Kjeltab Se/3.5	Sulphuric Acid 12.5 mL	H3BO3 1%, 25 mL	HCl-Titrisol 0.2 N	6.25	SNI 01-2891-1992
25	**8.47**	-	**-2.10**	-	0.0524 / 0.0532	Copper Sulphate	Digestion Reagent, 10	Indicating Boric Sol'n, 10 mL	0.02 N H2SO4	-	Laboratory Handbook of Methods of Food

Laboratory Number	Total Nitrogen	MU	z score	Zeta score	Nitrogen Weight	Catalyst	Acid Volume (mL)	Receiver Solution and Vol	Titrant	Conversion Factor	Nitrogen Reference
Assigned value obtained from robust average (x) ± robust SD (s*) = 8.89 ± 0.20 g/100 g (CV 2.2%, n= 67) with standard uncertainty 0.03 g/100g*											
26	**8.85**	0.07	-0.20	-0.90	5.0	Copper (II) sulphate	Conc H2SO4, 15	0.1 N HCl	0.1 N NaOH	6.25	AOAC No. 2001.11
27	**8.99**	0.57	0.50	0.35	0.5	K2SO4 / Se	10	30	0.2000	-	SNI 01-2354.4-2006 Modified
31	**8.78**	0.21	-0.55	-1.01	0.3	Selenium	H2SO4 (8 mL)	H3BO3 3% (50 mL)	HCl 0.05 N	-	SNI 01-2891
32	**8.87**	0.11	-0.10	-0.33	1.0789	K2SO4, CuSO4.5H2O	Conc H2SO4, 25	4% Boric Acid 50 mL	0.50987 M HCl	6.25	Block Digestion - Kjeldahl
33	**8.71**	0.24	-0.90	-1.46	0.7437, 0.7455	Kjeltabs	36 N H2SO4, 15 mL	Boric acid soln, 25 mL	0.1040	None	Method 976.05, OMA AOAC 18th Ed
34	**9.01**	0.03	0.60	**3.65**	0.750	Kjeldahl	H2SO4 15 mL plus	H3BO3 25 mL	HCl 0.1952 N	-	SNI 01.2354.4:2017
38	**8.88**	0.60	-0.05	-0.03	0.400	Salt mixture	H2SO4 12 mL	4% H3BO3, 20 mL	0.2 N HCl	-	AOAC 991.2, 19th Ed 2012
39	**8.87**	-	-0.10	-	0.5	Cu	H2SO4 / 10	Boric acid 30 mL	0.1 M HCl	-	AOAC 991.20
41	**8.95**	0.31	0.28	0.36	0.5	K2SO4:CuSO4	Sulphuric acid 15 mL	Boric acid 25 mL	0.1000	6.25	AOAC 2001.11
42	**9.15**	0.22	1.30	**2.28**	0.1	Selenium	H2SO4	H3BO3 1% 30 mL	HCl 0.1 N	-	SNI 01-2891-1992. point 7.1
43	**8.81**	0.45	-0.40	-0.35	1	Mix selenium	H2SO4, 12 mL	H3BO3, 25 mL	HCl 0.2 M	1.4007	National Standard, inhouse method
44	**8.99**	0.00	0.51	**3.40**	0.5062	Na2SO4, CuSO4	Conc H2SO4 20 mL	50 mL 0.1 N H2SO4	0.2 N NaOH	6.25	AOAC 19th Ed, 2012
45	**8.92**	0.34	0.17	0.20	1	7g K2SO4 + 0.8 g CuSO4.5H2O	98% H2SO4 15 mL	4.0% Boric acid 30 mL	0.5 N H2SO4	6.25	ISO 5983-2
48	**8.74**	0.13	-0.75	**-2.11**	0.5	Selenium Mixture reagent	20	30	0.1	1	MU-01/04
49	**8.51**	0.42	-1.90	-1.79	0.5, 1	Kjeltabs	Conc H2SO4 20 mL	Boric Acid 50 mL	0.2 N H2SO4	6.25	AOAC 20th Ed 2016
50	**8.99**	0.19	0.51	1.01	1.1567	Cu	H2SO4, 15.0 mL	Boric Acid, 75.0 mL	HCl, 0.0902	Fish M 6.25, Rice	AOAC 984.13
52	**8.60**	0.02	-1.47	**-9.08**	1.0	K2SO4, CuSO4	9.711, 11.164	Boric Acid (60mL)	0.09788 N HCl	6.25	Automated Kjeldahl Method
53	**8.85**	0.06	-0.21	-0.96	0.2	Selenium	H2SO4 8 mL	H3BO3 3% 25 mL	HCl 0.0958 N	6.25	SNI-01-2891-1992
54	**9.33**	0.35	**2.19**	**2.45**	1	Kjeltabs	H2SO4 12 mL	25 mL 4% Boric Acid	0.3 M H2SO4	5.95	AOAC 920.87
55	**8.79**	0.09	-0.51	-1.87	0.5	K2SO4 / Se	H2SO4 15 mL	250 mL Erlenmeyer	HCl 0.1 M		AOAC (2012) 991.20

Laboratory Number	Total Nitrogen	MU	z score	Zeta score	Nitrogen Weight	Catalyst	Acid Volume (mL)	Receiver Solution and Vol	Titrant	Conversion Factor	Nitrogen Reference
Assigned value obtained from robust average (x) ± robust SD (s*) = 8.89 ± 0.20 g/100 g (CV 2.2%, n= 67) with standard uncertainty 0.03 g/100g*											
58	**9.13**	0.26	1.20	1.80	0.5	-	-	Boric Acid	0.25 HCl	1.0	Based on AOAC 20th Ed 2016
60	**8.99**	-	0.50	-	-	-	-	-	-	-	AOAC (2012) 2011.11
61	**8.82**	0.39	-0.35	-0.36	1	K2SO4/CuSO4 Kjeltab catalyst	H2SO4 20 mL	Boric Acid 50 mL	0.1 M HCl	6.25	A6501 Kjeldahl/Boric Acid Method
63	**8.83**	-	-0.30	-	-	-	-	-	-	-	-
67	**9.01**	0.24	0.60	0.97	0.5xxx	CuSO4+K2SO4	H2SO4 15 mL	Boric acid 50 mL	0.2 N H2SO4	6.25	Inhouse based on ISO 5988.2
68	**8.90**	-	0.05	-	0.5	Kjeltabs	17	50	0.1 N HCl	6.25	AOAC
69	**8.76**	-	-0.67	-	-	-	-	-	-	-	-
71	**8.98**	0.27	0.45	0.65	1.0038, 0.9934	Kjeltabs 3.5 g, K2SO4 0.4 g,	H2SO4 15 mL	Boric acid	0.2 N HCl		AOAC 2001.11
72	**9.80**	0.05	**4.55**	**23.30**	2	K2SO4, CuSO4, SeO2	25	4% Boric acid 25 mL	0.05 M H2SO4	6.25	AOAC 920.87
73A	**9.01**	0.80	0.60	0.30	1	2 Kjeltabs (each 3.5 g K2SO4, 0.4	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	**9.44**	0.14	**2.76**	**7.20**	0.5	CuSO4	H2SO4, 5 mL	H3BO3, 20 mL	HCl, 0.1 M	N/A (report as	SNI 01-2891-1992 Butir 7.1
77	**9.66**	-	**3.85**	-	-	-	-	-	-	-	-
78	**8.80**	0.10	-0.45	-1.54	2	Kjeltabs	Conc H2SO4, 15	4% Boric acid, 25 mL	0.1 N HCl	-	AOAC 19th Ed
79	**8.85**	0.31	-0.18	-0.23	0.1 to 0.15	-	-	-	-		IK/02/5.4.1/LDITP/Analisis Protein
80	**8.98**	-	0.45	-	0.25 to 0.5	TAP/S3.5	25	40	0.1	6.25	AOAC 984.13
81	**8.60**	0.10	-1.45	**-4.97**	0.5247 mean	K2SO4 and CuSO4.5H2O	20 mL H2SO4	60 mL 2% Boric Acid soln	0.09597 N HCl	6.25	Automated Kjeldahl Method
83	**8.86**	0.10	-0.14	-0.46	0.5	CuSO4	H2SO4	Boric Acid, Bromocresol	HCl (0.1)	6.25	SNI-01-2891-1992
84	**8.98**	-	0.45		1	KJELCAT 12-0328	H2SO4 98% 20 mL	H3BO3 4%, 60 mL	HCl 0.1 M	-	KJELDAHL
85	**9.01**	0.02	0.60	**3.79**	0.2	-	-	-	-	-	DuMaster Protein Analyzer (Buchi)
86	**9.00**	0.90	0.55	0.24	0.5	3.5g K2SO4 + 3.5 mg Se	Conc H2SO4 12.5 mL	4% Boric Acid 30 mL	0.1 N HCl	-	AOAC (2012) 981.10

Laboratory Number	Total Nitrogen	MU	z score	Zeta score	Nitrogen Weight	Catalyst	Acid Volume (mL)	Receiver Solution and Vol	Titrant	Conversion Factor	Nitrogen Reference
Assigned value obtained from robust average (x) ± robust SD (s*) = 8.89 ± 0.20 g/100 g (CV 2.2%, n= 67) with standard uncertainty 0.03 g/100g*											
87	**8.63**	0.35	-1.31	-1.47	0.51	K2SO4+Se	Sulphuric Acid; 25 mL	Boric Acid; 15 mL	HCl 0.01 N	14	MTD/FOD/CHM-03
88	**8.77**	0.10	-0.59	**-2.04**	0.5	Tablet Kjeldahl	Conc H2SO4 10 mL	H3BO3 2% 50 mL	HCl 0.0515 N	-	SM 3751-2009
89	**8.76**	0.44	-0.64	-0.58	0.5	CuSO4	HCl	25 mL Boric Acid	0.1 N HCl	-	AOAC 991.2
90	**8.52**	0.12	-1.86	**-5.55**	0.3	K2SO4, Se	15	-	0.1 M HCl	-	AOAC (2016) 2001.11
91	**8.70**	0.05	-0.95	**-4.87**	-	-	-	-	-	-	-
92	**9.06**	-	0.85	-	1	CuSO4	H2SO4	Boric	HCl	6.25	-
93	**8.71**	-	-0.90	-	2	H2O2 5 mL, Kjeltabs: 3.5 g	H2SO4 12 mL	Boric acid 25 mL	0.05 N H2SO4	6.25	AOAC 945.18-B
94	**9.21**	-	1.60	-	1	CuSO4.5H2O and K2SO4	Conc H2SO4 / 13 mL	1% Boric acid	0.1 M HCl	-	AOAC (2012) 991.20
95	**8.93**	0.08	0.18	0.69	-	-	-	-	-	-	-
100	**8.80**	0.33	-0.45	-0.54	0.5	CuSO4and K2SO4	H2SO4 / 15 mL	4% Boric Acid 50 mL	0.2 N H2SO4	6.25	Inhouse based on ISO 5983-2 (2009)

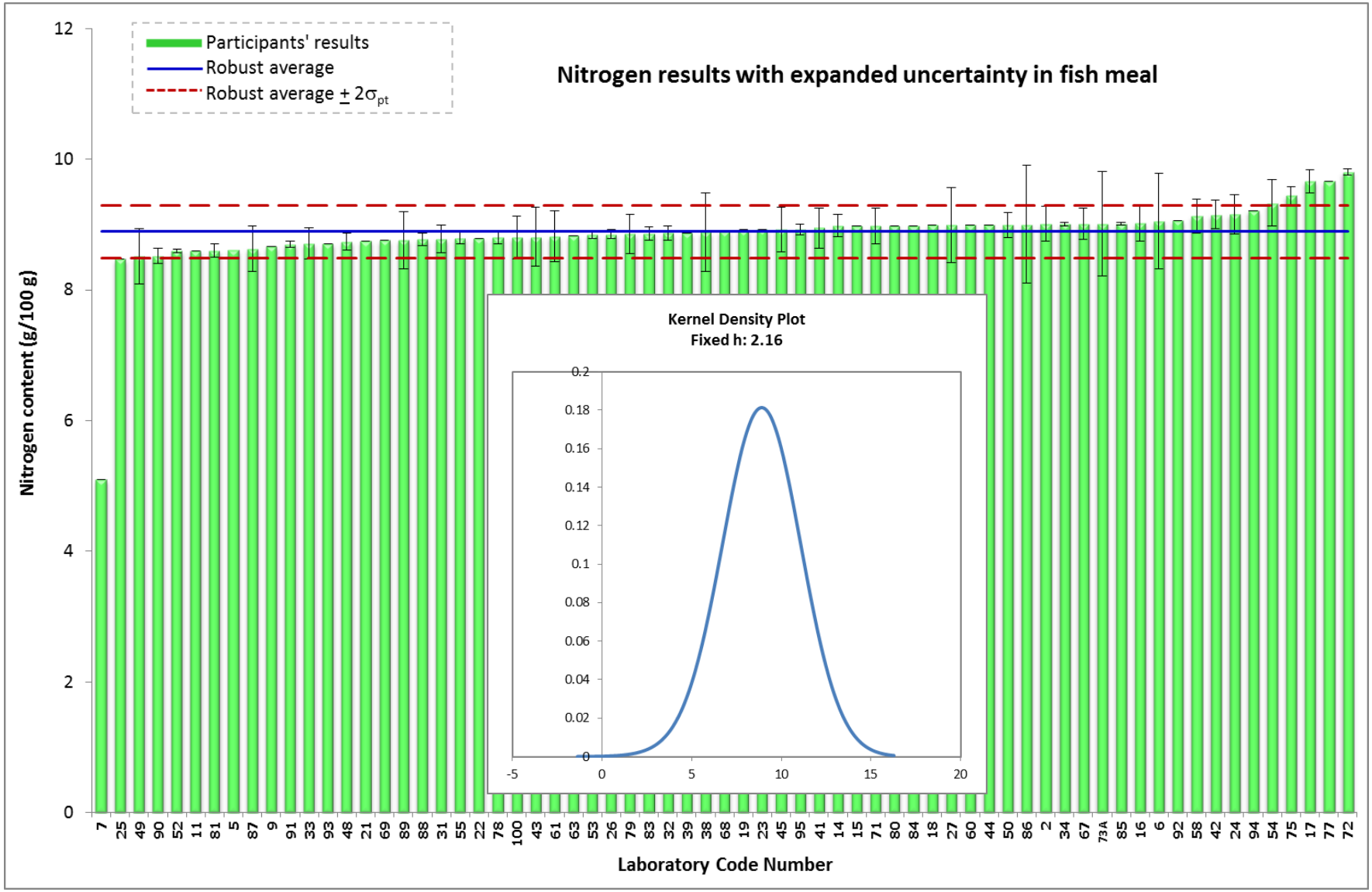

Figure 61. Distribution of **total nitrogen** results (ascending order) in fish meal with expanded uncertainty

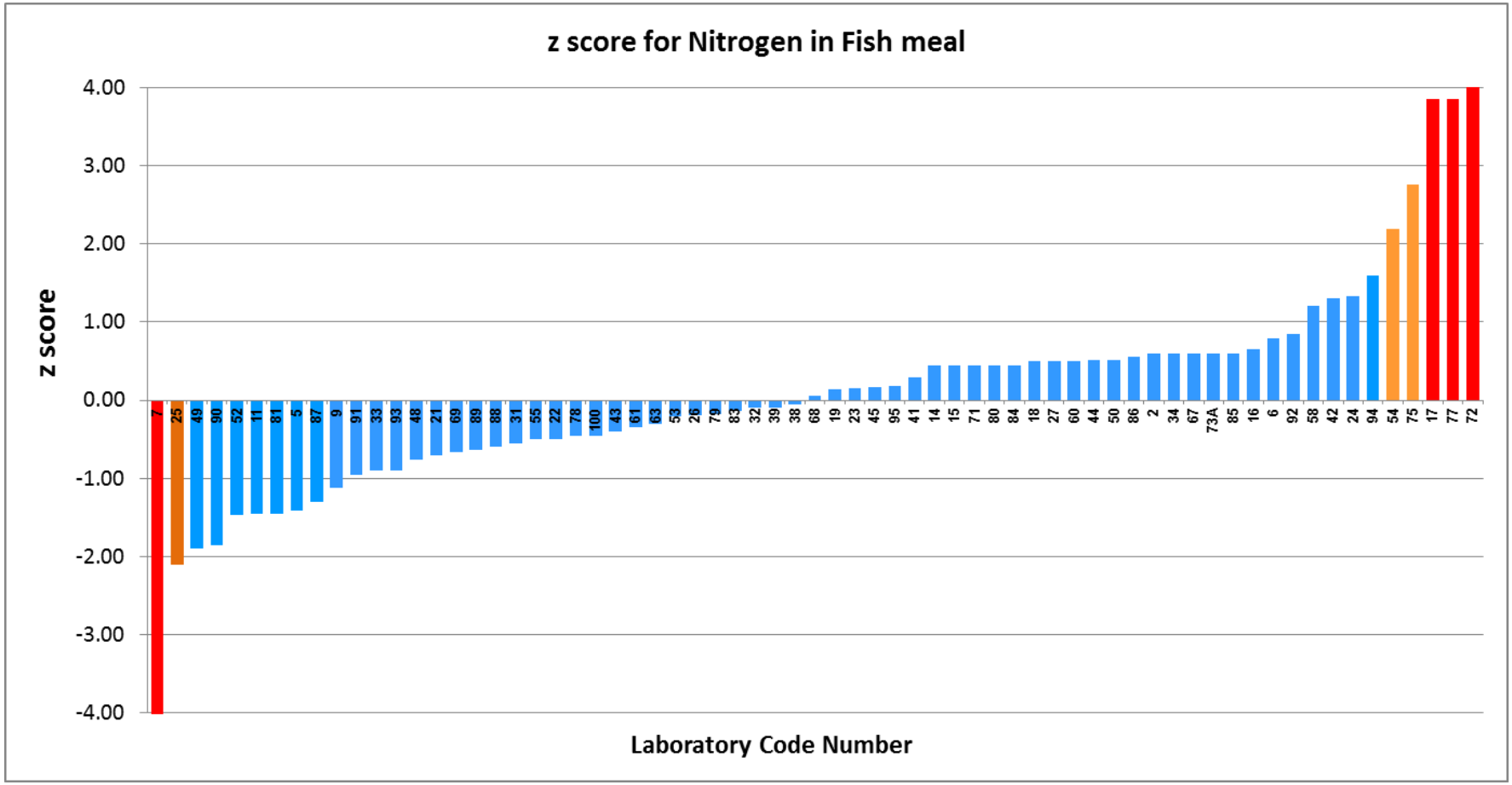

Figure 62. Plot of ordered *z* scores for **total nitrogen** results in fish meal

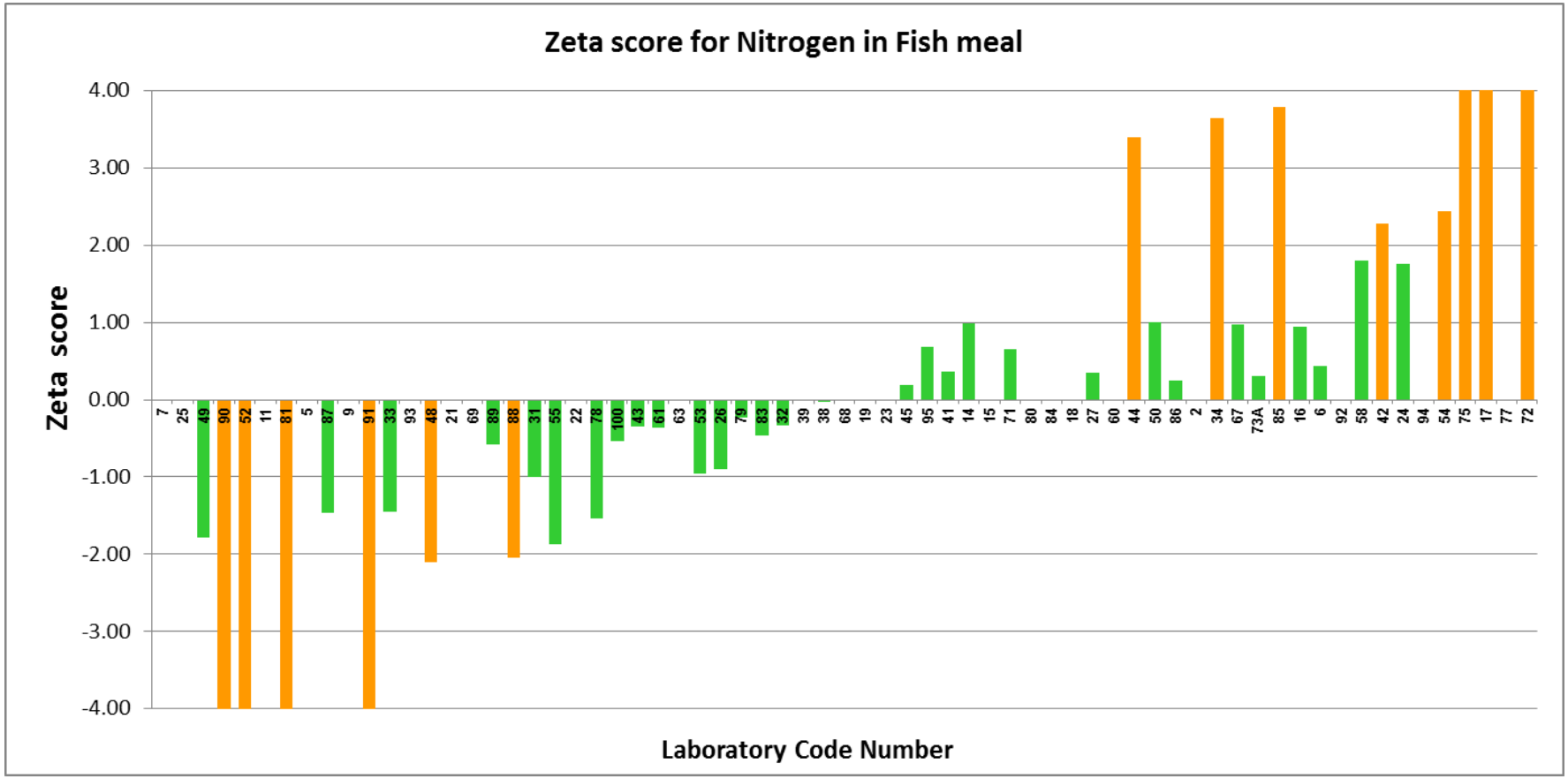

Figure 63. Plot of *Zeta* score for **total nitrogen** in fish meal, following the ordered z scores in the above Figure 62.

Table 22. Evaluation of laboratory performance on **total fat** analysis (g/100 g, as received) in fish meal

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 (x) ± 3SDp from Horwitz's equation = 7.30 ± 0.65 g/100 g (CV 8.9%, n= 54) with standard uncertainty 0.10 g/100g*									
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00					
2	**4.39**	0.21	**-4.48**	**-20.07**	2.00	N	Petroleum Ether	8	AOAC (2016) 920.39
5	**6.38**	-	-1.41	-	2.0000	N	Petroleum Ether	1.5	AOAC 20th Ed, 2016, 2003.05
6	**6.54**	0.24	-1.17	**-4.93**	3.0000	N	Diethyl Ether	80 min	AOAC 2003.05
7	**7.80**	0.20	0.77	**3.54**	1.0051	N	Pet Ether	1	AOAC
9	**6.87**	-	-0.67	-	2	N	Petroleum Ether	1.5	Based on AOAC 20th Ed, 2016, 2003.05, Chapter 4
11	**6.16**	-	-1.75	-	4.0000	N	Petroleum Ether	16-18 hr	AOAC (2016) 922.06
14	**8.87**	0.02	**2.42**	**15.68**	2	Y	Diethyl Ether and Petroleum Ether	3	AOAC 954.02
15	**5.81**	-	**-2.29**	-	1	Y	Petroleum Ether	1	ISO 11085:2015
16	**7.82**	0.12	0.80	**4.46**	2	Y	Diethyl Ether + Petroleum Ether	-	SNI 01-2891-1992 Food & Beverage
17	**7.02**	0.50	-0.43	-1.04	3.000	N	Petroleum Ether	110 minutes	AOAC
18	**8.23**	-	1.43	-	2.0	Y	Petroleum Benzine	6	SNI 01-2891-1992
19	**7.27**	-	-0.05	-	1	N	Petroleum Benzene	1	AOAC 2003.05, AN305, FOSS, 2005
21	**9.07**	-	**2.72**	-	0.5 to 1.0	Y	Diethyl Ether Petroleum Ether	2	AOAC 932.06 (2016)

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 (x) ± 3SDp from Horwitz's equation = 7.30 ± 0.65 g/100 g (CV 8.9%, n= 54) with standard uncertainty 0.10 g/100g*									
22	**7.54**	-	0.37	-	-	-	-	-	-
23	**6.97**	-	-0.51	-	1.90	N	Petroleum Ether	1	AOCS Am5-04
24	**7.26**	0.11	-0.06	-0.35	1.000	N	Petroleum Benzine	20 mins	SNI 01-2891-1992
25	**5.50**	-	**-2.77**	-	5.1206	N	Hexane	8	Laboratory Handbook of Methods of Food Analysis, 3rd Ed, R. Lees
26	**8.44**	0.05	1.75	**11.03**	4.0	Y	Diethyl Ether, anhydrous and Petroleum Ether	4.0	AOAC No.948.15
27	**8.20**	0.17	1.38	**6.86**	2	Y	Diethyl Ether	2	SNI 2354-3:2017
31	**9.65**	0.29	**3.62**	**13.34**	3.5	Y	Petroleum Benzene	4	SNI 01-2891
32	**7.25**	0.15	-0.08	-0.40	2.0528	Y	Petroleum Ether	20 cycles (2 hours)	Acid Hydrolysis
33	**7.11**	0.55	-0.29	-0.65	2.5122, 2.5207, 2.5241	Y	Petroleum Ether	1.5	Method 932.06, OMA AOAC, 18th Ed
34	**7.19**	0.01	-0.17	-1.10	1.000	Y	CHCl3	1.5	SNI 2354.3:2017
38	**9.03**	1.15	**2.66**	**2.96**	2.000	Y	Diethyl Ether, Petroleum Ether	1 min each	AOAC 922.06, 19th Ed 2012 (total fat)
39	**8.16**	-	1.32	-	1	Y	Diethyl Ether:Petroleum Ether (1:1)	6 min	AOAC 922.06
41	**8.40**	0.38	1.69	**5.10**	2	Y	Petroleum Ether / Diethyl Ether	-	AOAC 954.02

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 (x) ± 3SDp from Horwitz's equation = 7.30 ± 0.65 g/100 g (CV 8.9%, n= 54) with standard uncertainty 0.10 g/100g*									
42	**8.26**	0.09	1.48	**8.82**	3	Y	Hexane	2	SNI 01-2891-1992. point 8.2
43	**6.68**	0.69	-0.95	-1.72	2	N	Hexane	3	National Standard
44	**3.94**	0.07	**-5.17**	**-31.71**	2.0066	N	Anhydrous Ether	16.0	AOAC 19th Ed, 2012
45	**6.86**	0.27	-0.68	**-2.64**	1.5	N	Petroleum Ether	2.25	ISO 11085
48	**8.82**	0.23	**2.34**	**9.99**	1	Y	Diethyl Ether	3 x 20 mins	MU-01/02
49	**6.82**	0.34	-0.74	-2.43	2	Y	Petroleum Ether	1 minute	AOAC 20th Ed 2016
52	**8.63**	0.06	**2.04**	**12.67**	2.0	Y	Diethyl Ether	3	Indirect Solvent Extraction (Soxhlet) and Acid Hydrolysis
53	**7.44**	1.36	0.22	0.20	1.0	Y	Petroleum Benzene	80 mins	SNI-01-2891-1992
54	**8.90**	0.04	**2.46**	**15.69**	1	Y	1:1 Mixed ether	5	AOAC 923.03
55	**7.29**	0.17	-0.02	-0.11	8	Y	Ether, Petroleum ether	1	AOAC (2012) 948.15
60	**6.94**	-	-0.55	-	-	-	-	-	AOAC (2012) 2003.06
61	**7.30**	0.93	0.00	0.00	2	N (Y)	Petroleum Spirits (Diethyl Ether:Hexane)	1 (-)	A6301 Soxtec (A6302 Acid Hydrolysis)
63	**8.49**	-	1.83	-	-	-	-	-	-

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 (x) ± 3SDp from Horwitz's equation = 7.30 ± 0.65 g/100 g (CV 8.9%, n= 54) with standard uncertainty 0.10 g/100g*									
66A	**3.17**	0.11	**-6.35**	**-36.19**	1.5101	N	Petroleum Ether	0.75	AOAC 963.15, 20th Ed 2016 (Crude Fat)
66B	**3.02**	0.08	**-6.58**	**-39.74**	1.5005	N	Petroleum Ether	0.75	AOAC 963.15, 20th Ed 2016 (Crude Fat)
66C	**6.49**	0.11	-1.25	**-7.10**	3.5127	N	Petroleum Ether	0.75	AOAC 963.15, 20th Ed 2016 (Crude Fat)
67	**6.62**	0.29	-1.05	**-3.86**	1.0xxx	N	Petroleum Ether	1.30	Inhouse based on AOAC 920.39
68	**4.98**	-	**-3.57**	-	2	N	Petroleum ether	6	AOAC
69	**7.42**	-	0.18	-	-	-	-	-	-
71	**6.54**	0.23	-1.18	**-4.97**	1.0032, 1.0039	N	Petroleum Ether	2	AOAC 2003.05
72	**6.83**	0.12	-0.72	**-4.03**	2	Y	Petroleum ether, Diethyl ether	1 min	AOAC 922.06
73A	**7.07**	0.29	-0.35	-1.31	1	N	Petroleum Benzine	1	FTC-06.01 (refers to AOAC 2003.05)
75	**6.04**	0.09	-1.94	**-11.45**	2	Y	Hexane	4	SNI 01-2891-1992 Butir 8.2
77	**9.30**	-	**3.08**	-	-	-	-	-	-
79	**8.17**	0.14	1.34	**7.13**	1	Y	-	-	SNI 01-2891-1992 Butir 8.2
81	**8.67**	0.14	**2.11**	**11.22**	2.0211 mean	Y	Anhydrous Diethyl ether	20 cycles	Soxhlet Method
83	**8.55**	0.12	1.92	**10.68**	1.5	Y	HCC & Petroleum ether	2	SNI-01-2891-1992

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 (x) ± 3SDp from Horwitz's equation = 7.30 ± 0.65 g/100 g (CV 8.9%, n= 54) with standard uncertainty 0.10 g/100g*									
84	**7.18**	-	-0.18	-	2	N	Petroleum Ether	16	AOAC 920.39
86	**8.34**	0.86	1.60	**2.36**	2	Y	Ether / Petroleum ether	0.75	AOAC (2012) 954.02
87	**7.28**	0.33	-0.03	-0.10	1.5	Y	Hexane	2	MTD/FOD/CHM-04
88	**4.01**	0.07	**-5.06**	**-30.88**	5	Y	Hexane	3	In house method (soxhlet)
89	**7.02**	0.18	-0.44	**-2.11**	5	Y	Petroleum Ether	16	AOAC 963.15
90	**3.60**	0.05	**-5.69**	**-35.87**	2	-	Diethyl Ether	-	AOAC (2016) 954.02
91	**8.15**	0.03	1.31	**8.41**	-	-	-	-	-
92	**7.45**	-	0.23	-	1	N	Petroleum Ether	1	-
94	**9.42**	-	**3.26**	-	2	Y	Diethyl Ether and Petroleum Ether	-	AOAC (2012) 922.06
95	**6.57**	0.21	-1.12	**-5.03**	-	-	-	-	-
100	**6.44**	0.30	-1.32	**-4.77**	1.5	N	Petroleum ether	60 mins	AOAC AM5-04 reapprove 2009

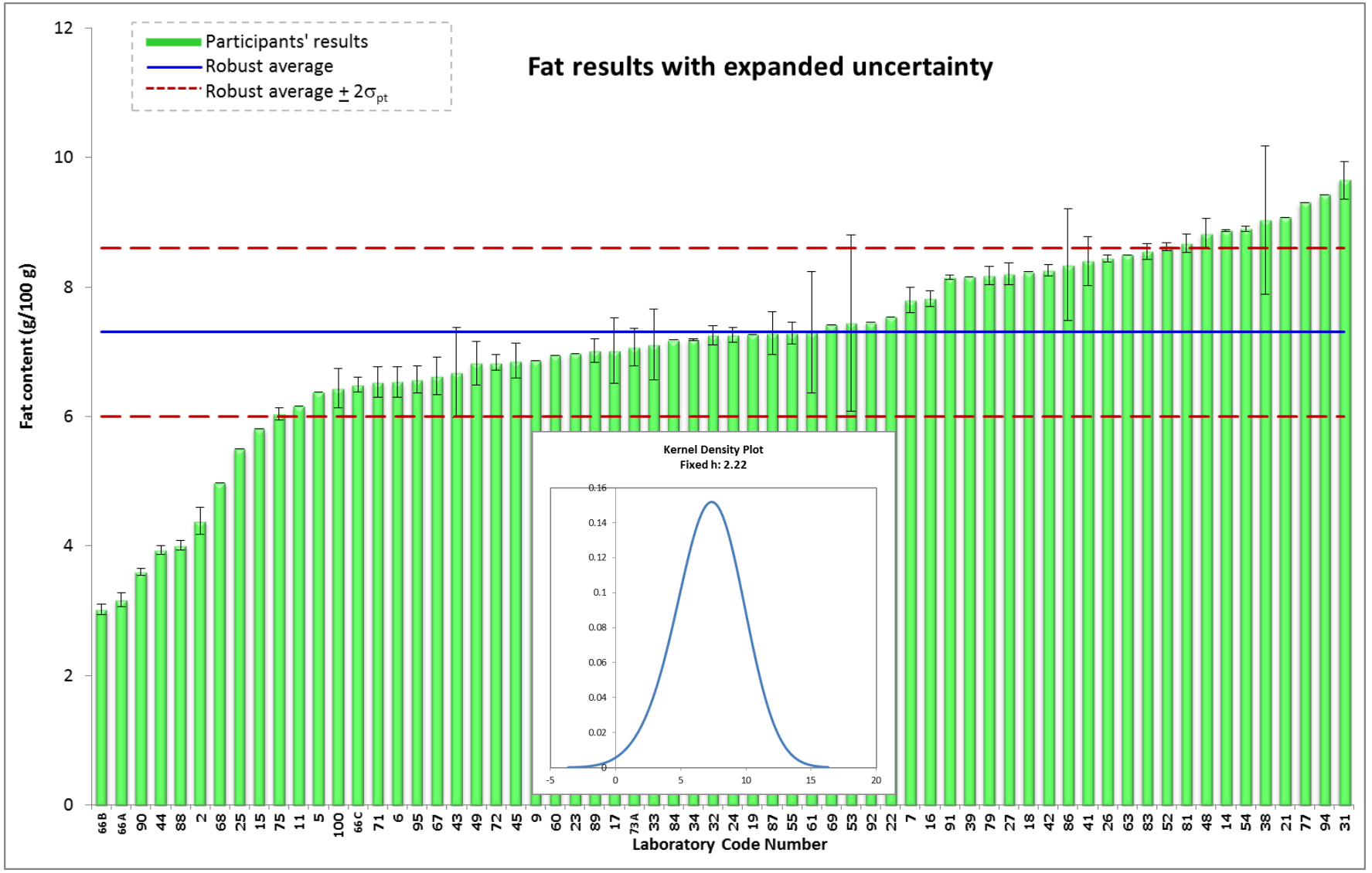

Figure 64. Distribution of **total fat** results (ascending order) in fish meal with expanded uncertainty

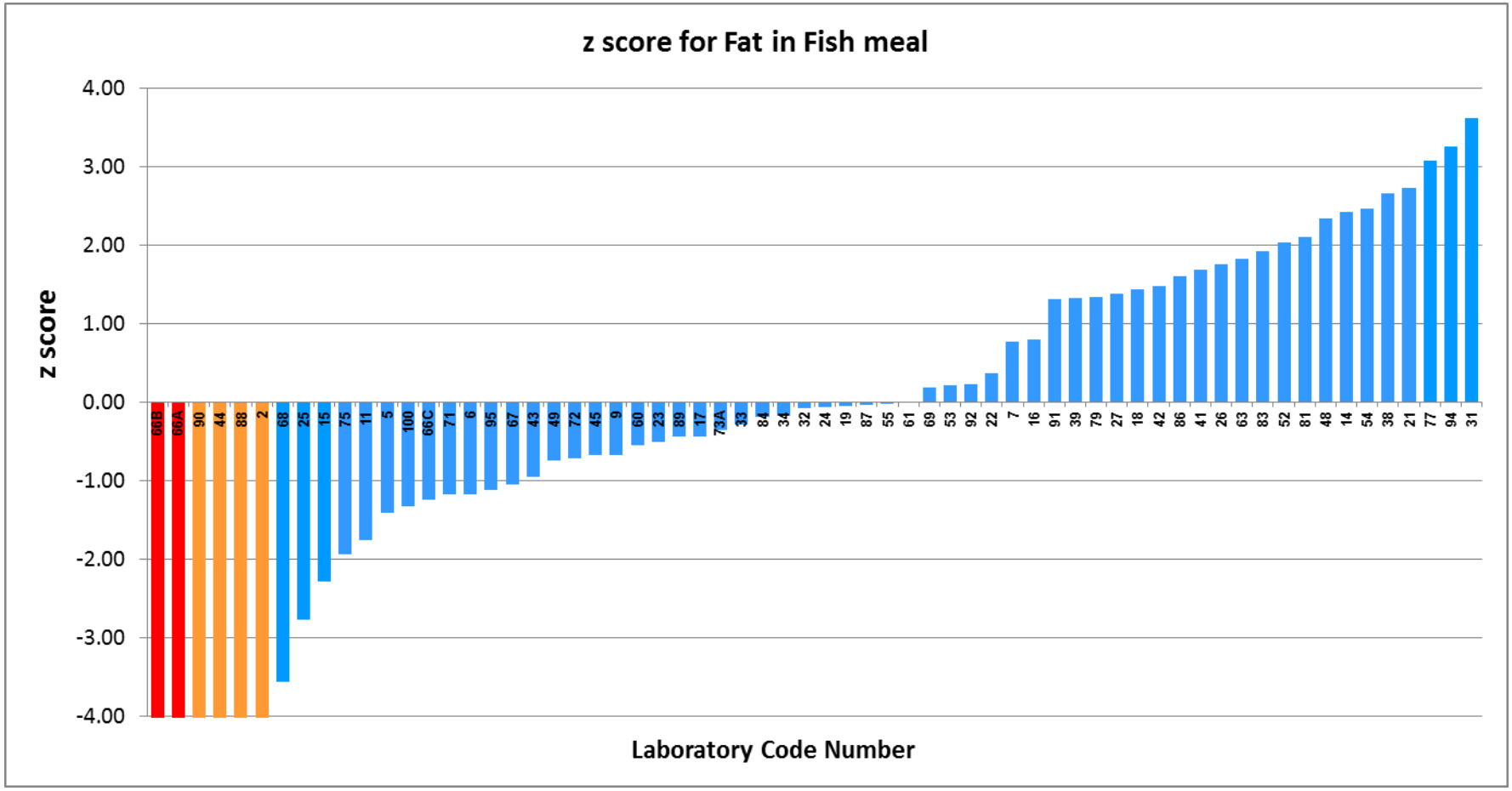

Figure 65. Plot of ordered z scores for **total fat** results in fish meal

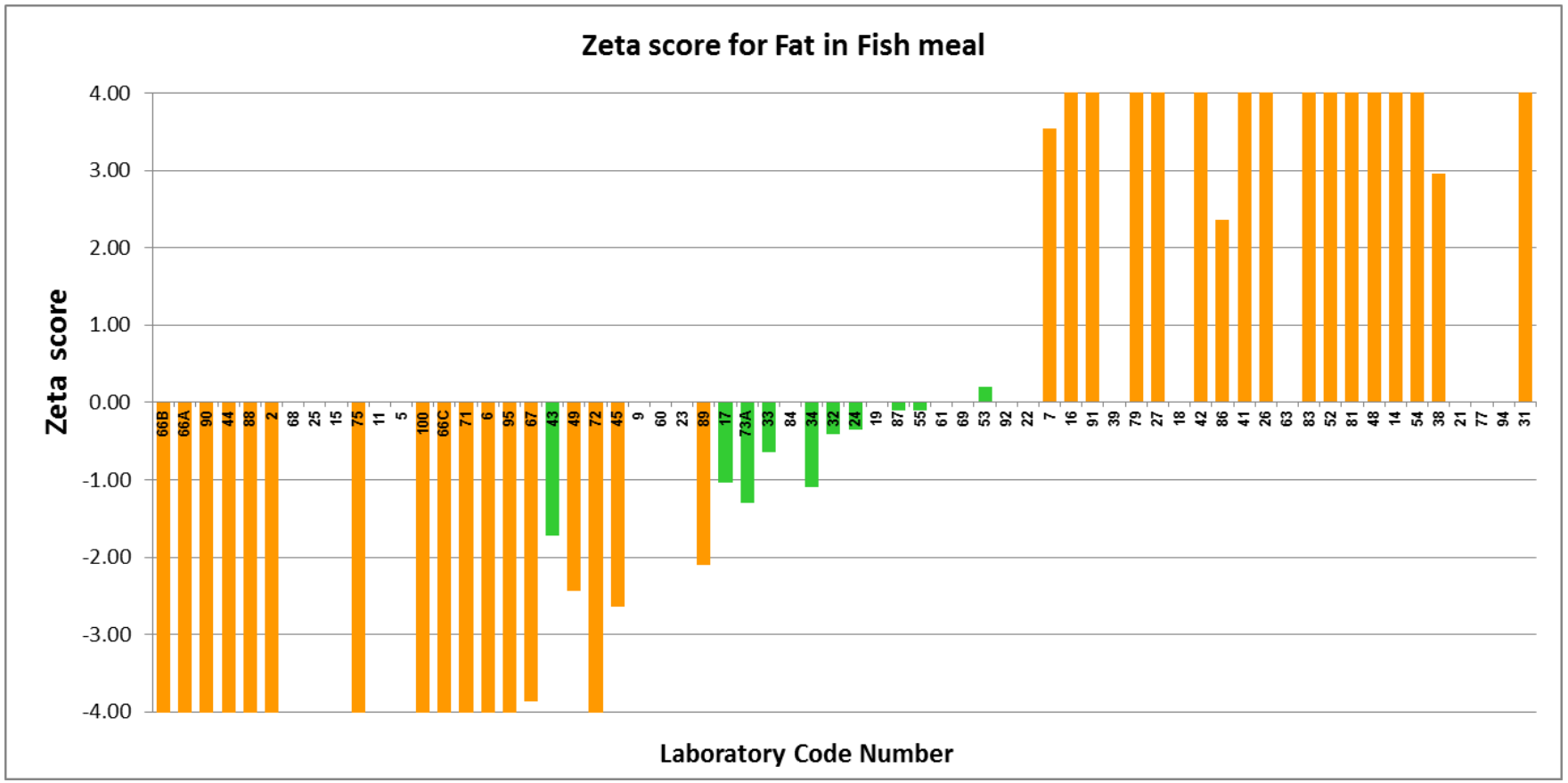

Figure 66. Plot of *Zeta* score for **total fat** in fish meal, following the ordered z scores in the above Figure 65.

Table 23. Evaluation of laboratory performance on **ash** analysis (g/100 g, as received) in fish meal

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*) = 27.27 ± 0.28 g/100 g (CV 1.0%, n= 68) with standard uncertainty 0.04 g/100g									
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00					
1	-	-	-	-	-	-	-	-	-
2	**27.40**	-	0.46	-	4.00	Hot plate	550	4	AOAC (2016) 923.03
5	**27.19**	-	-0.30	-	2.0000		550	3	ISO 5984:2002
6	**27.17**	0.97	-0.36	-0.21	1.0000	Pre-burn on Hotplate	600	4	AOAC (2016) 942.05 4.1.10
7	**27.90**	0.40	**2.25**	**3.09**	1.0039/1.0316	None	550	5 or until white ash	AOAC
9	**27.36**	-	0.33	-	1 to 2	-	550	2.5	Based on ISO 5984:2002
11	**27.48**	-	0.75	-	2.0000	Pre heat 3 hour	550	3	ISO (5984) 2002 (E)
14	**27.55**	1.06	0.99	0.52	2	Charring	600	2	AOAC 942.05
15	**27.00**	-	-0.96	-	2	-	600	2	AOAC (2016) 942.05
16	**27.30**	0.01	0.11	0.74	2 to 3	-	550	8	SNI 01-2891-1992 Food & Beverage
17	**27.17**	0.32	-0.36	-0.62	2.000	Charring	550	2	AOAC
18	**27.40**	-	0.46	-	2.0	Charring	550	4	SNI 01-2891-1992
19	**27.18**	-	-0.31	-	1	-	600	3	AOAC 942.05
21	**27.50**	0.21	0.82	**2.06**	1	4	550	6	AOAC 923.03 (2016)
22	**27.30**	-	0.11	-		-	-	-	-
23	**27.90**	-	**2.25**	-	2.00	-	550	3	ISO 5984
24	**27.24**	0.21	-0.11	-0.27	2.000	N	550	8	SNI 01-2891-1992

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*) = 27.27 ± 0.28 g/100 g (CV 1.0%, n= 68) with standard uncertainty 0.04 g/100g*									
25	**27.00**	-	-0.96	-	5.0208 / 5.0213	Addition of HNO3	550	4	Laboratory Handbook of Methods of Food Analysis, 3rd Ed, R. Lees
26	**27.40**	0.03	0.46	**3.02**	4.0	Drying at 150°C	525	24	AOAC No. 900.02A
27	**27.39**	0.19	0.43	1.16	2	Drying in vacuum oven 105°C 22 h then 2 h at 300°C in furnace	550	8	SNI 2354.1:2010
31	**25.00**	0.98	**-8.11**	**-4.62**	3	-	600	10	SNI 01-2891
32	**27.20**	0.55	-0.25	-0.25	2.0391	Charring	600	8	AOAC 942.05
33	**26.90**	0.58	-1.32	-1.26	2.1007, 2.0308, 2.0282	Charring	600	2	Method 942.05, OMA AOAC, 18th Ed
34	**27.70**	0.03	1.54	**10.23**	2.000	-	550	18	SNI 2354.1:2010
37	**27.53**	-	0.92	-	3	Free flame by hotplate	550	4	AOAC (2016) 938.08
38	**27.14**	0.05	-0.46	**-2.78**	2.000	NA	600	2	AOAC 942.05, 19th Ed 2012
39	**27.50**	-	0.82	-	2	Charring	550	5 to 6	AOAC 942.05
41	**26.78**	1.32	-1.76	-0.74	2	-	600	2	AOAC
42	**24.00**	0.16	**-11.68**	**-36.93**	3	-	600	5	AOAC 942.05 2005
43	**27.45**	0.41	0.64	0.85	2	Charring	550	3 to constant weight	National Standard
44	**27.10**	1.25	-0.61	-0.27	1.0000	Charring	520	8	AOAC 19th Ed, 2012
45	**27.46**	0.08	0.67	**3.37**	2	-	550 ± 20	3	ISO 5984
48	**27.41**	0.11	0.48	1.99	2	Charring	550	3 then 1 then 1	SNI 01-2891-1992

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*) = 27.27 ± 0.28 g/100 g (CV 1.0%, n= 68) with standard uncertainty 0.04 g/100g*									
49	**27.50**	1.40	0.82	0.33	1, 2	Charring	555	6	AOAC 20th Ed 2016
50	**26.90**	0.12	-1.32	**-5.28**	2.0118	Charring	550	6.0	AOAC 923.03
52	**27.24**	0.15	-0.11	-0.36	2.0	Charring	600	8	AOAC 942.05
54	**27.50**	0.10	0.82	**3.59**	1	Charring	525	5	AOAC 92.03
55	**27.62**	0.14	1.26	**4.38**	2	Charring	550	2	AOAC (2012) 920.153
58	**27.60**	6.35	1.18	0.10	3.0	-	550	8	Based on AOAC 20th Ed 2016
60	**27.39**	-	0.43	-	-	-	-	-	SNI 01-2891-1992 Butir 6
61	**26.96**	0.84	-1.11	-0.74	2	N/A	550	15	A6401 550C Ash
63	**27.20**	-	-0.25	-	-	-	-	-	-
67	**27.20**	0.59	-0.25	-0.24	2.0xxx	-	600	2	AOAC 942.05
68	**27.30**	-	0.11	-	2	Y	600	2	AOAC
69	**26.89**	-	-1.36	-	-	-	-	-	-
70	**27.30**	2.00	0.11	0.03	1	-	550	5	-
71	**27.03**	2.17	-0.86	-0.22	1.0019, 1.0048	-	600	3	AOAC 942.05
72	**27.32**	0.05	0.18	1.06	2	Charring	550	4	AOAC 923.03
73A	**27.03**	0.20	-0.86	-	1	N	600	3.5	FTC-05.01 (refers to AOAC 942.05)
75	**29.83**	0.39	**9.13**	**12.77**	2	-	550	4	SNI 01-2891-1992 Butir 6.1
77	**20.63**	-	**-23.71**	-	-	-	-	-	-
79	**27.26**	0.34	-0.03	-0.05	2 to 3	-	550	-	SNI 01-2891-1992 Butir 6.1
80	**26.80**	-	-1.68	-	2.xx	N/A	600	Constant weight	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*) = 27.27 ± 0.28 g/100 g (CV 1.0%, n= 68) with standard uncertainty 0.04 g/100g*									
81	**27.20**	0.50	-0.25	-0.28	2.0678 mean	Charring	600	10	AOAC 942.05
83	**27.30**	0.04	0.09	0.58	2	-	550	4	SNI-01-2891-1992
84	**27.20**	-	-0.25	-	2	Charring	600	2	AOAC 942.05
85	**27.47**	0.01	0.71	**4.96**	2	-	550	3	SNI 01-2896-1992
86	**27.10**	2.41	-0.61	-0.14	2	Charring	600	2	AOAC (2012) 942.05
87	**27.21**	0.11	-0.22	-0.93	2.50	Heating on hotplate	550	Overnight	MTD/FOD/CHM-02
88	**27.56**	1.11	1.05	0.53	2.5	-	550	24	SM 01-2891-1997
89	**27.50**	0.15	0.83	**2.80**	2	Charring on hotplate	550	16	AOAC 930.30
90	**26.89**	1.91	-1.36	-0.40	2	-	550	3	AOAC (2016) 942.05
91	**27.10**	0.02	-0.61	**-4.17**	-	-	-	-	-
92	**27.30**	-	0.11	-	5	-	550	3	-
93	**27.16**	-	-0.39	-	2	Charring	600	2	AOAC 942.05
94	**27.70**	-	1.54	-	2	Charring	550	5	AOAC (2012) 945.46
95	**27.13**	0.25	-0.52	-1.10	-	-	-	-	-
96	**26.60**	-	**-2.39**	-	3	-	550	4	TCVN 8124:2009
100	**27.50**	0.12	0.82	**3.19**	2	-	600	2	AOAC (2016) 942.05

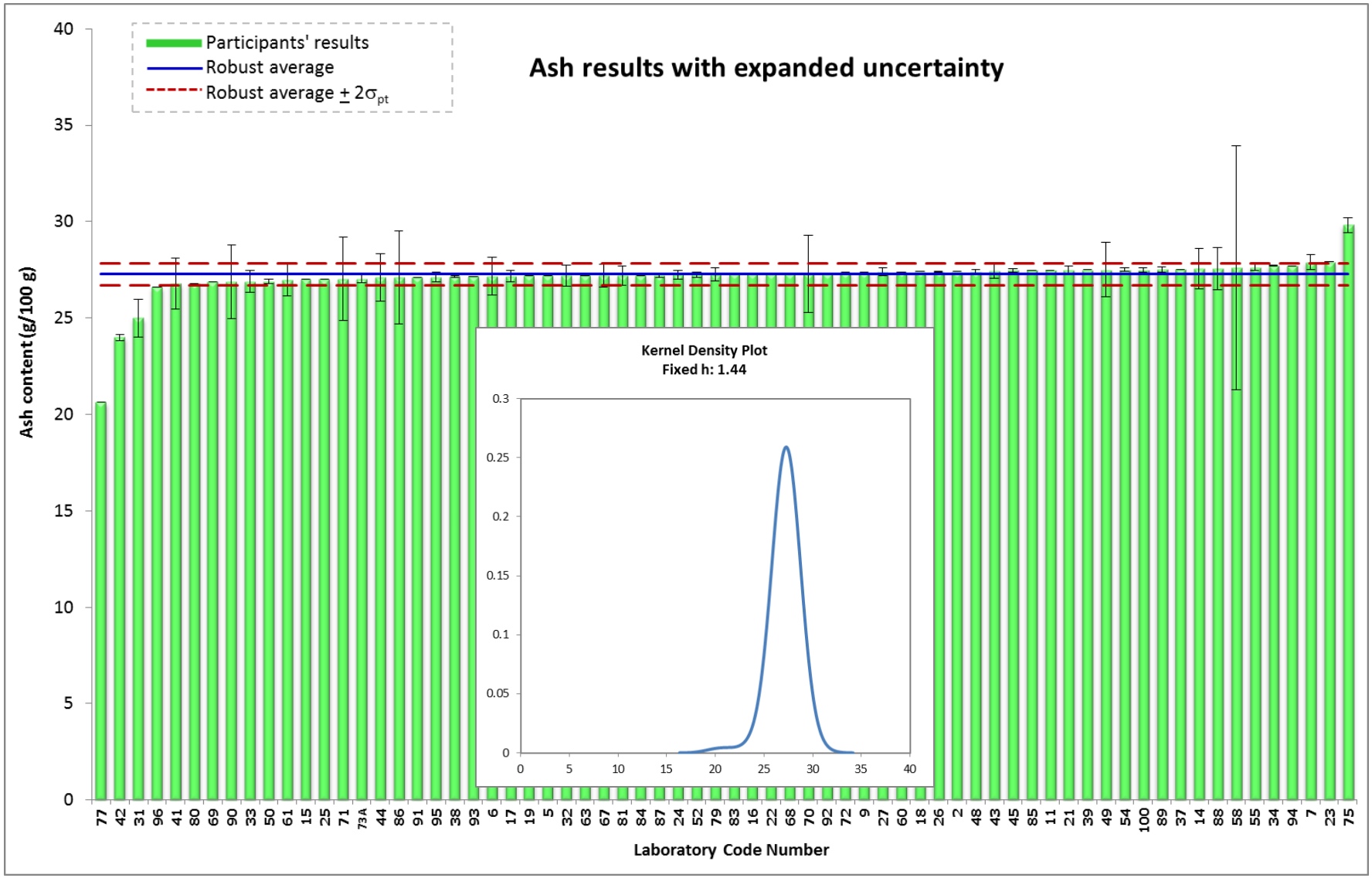

Figure 67. Distribution of **ash** results (ascending order) in fish meal with expanded uncertainty

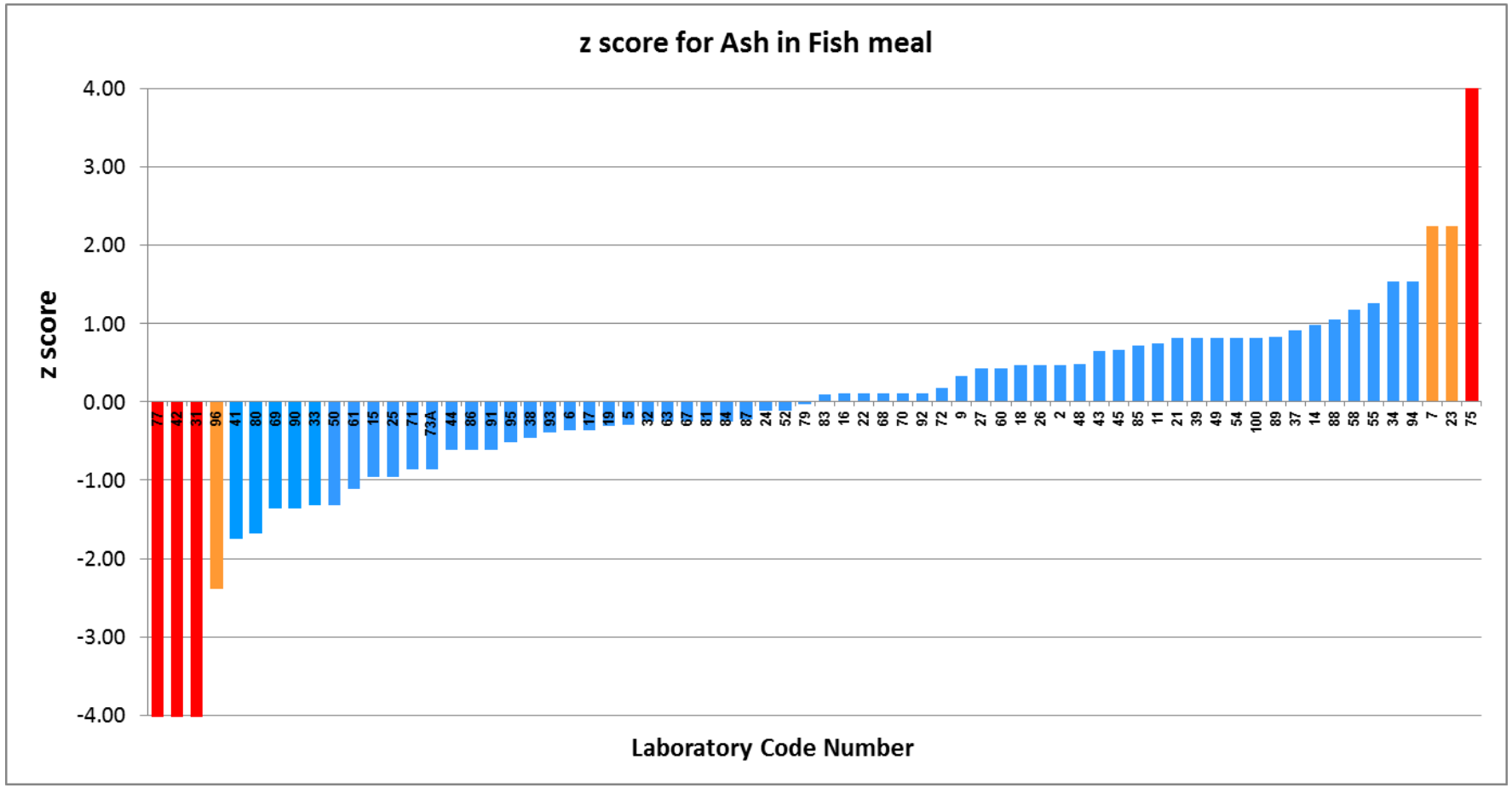

Figure 68. Plot of ordered *z* scores for **ash** results in fish meal

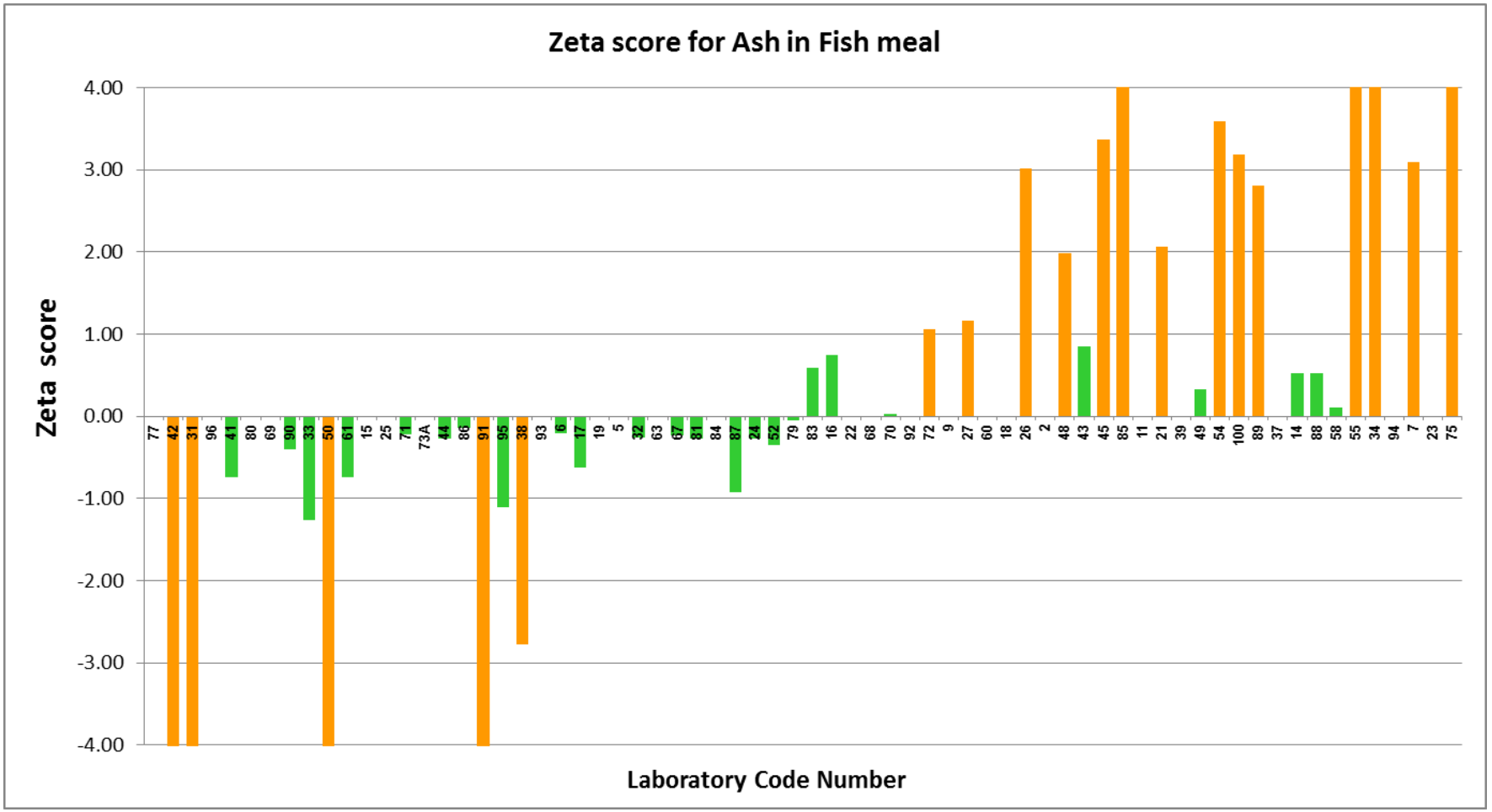

Figure 69. Plot of *Zeta* score for **ash** in fish meal, following the ordered z scores in the above Figure 68.

Table 24. Evaluation of laboratory performance **calcium** analysis (mg/kg, as received) in fish meal

Laboratory Number	Calcium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x*) ± SD from Horwitz' s equation = 76533 ± 6761 mg/kg (CV 8.8%, n= 52) with u_{xpt} 1172 mg/kg											
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| < 2.00							
6	**80262**	4356	0.55	1.51	1.0000	Acid	HCl:HNO_3: H_2O	AAS	Ca 422.7	Y	AOAC (2016), 20th Ed, 928.08, 985.35 (50.1.14)
11	**88733**	-	1.80	-	2.0000	Dry Ashing	HCL:H_2O	AAS	Ca 239.9	Y	AOAC (2016), 975.03, 985.35
13	**65100**	-	-1.69	-	0.5	Microwave	HNO_3 10 mL + HCl 2 mL	Analytikal Jena ContrAA 800 D	Ca 422	N	Internal Method
14	**85718**	-	1.36	-	0.1 - 0.2	Ashing	Conc HNO_3, Conc $HClO_4$	ICP Horiba Jobin Yvon	Ca 393.366	Y	AOAC 975.03, 984.27
16	**96760**	967	**2.99**	**15.95**	0.5	Hot plate	HNO_3+H_2O_2	ICP-OES Optima 7000 DV Perkin Elmer	Ca 317.933,	N	In-house Method
18	**31900**	-	**-6.60**	-	2.0	Dry Ashing	HNO_3	AAS, Varian	Various	N	AOAC 968.08
19	**77300**	-	0.11	-	1	By Furnace	HNO_3:H_2O (1:1)	Ca Manual by Buret	P 400	N	AOAC 927.02, 944.03, 965.17
21	**109689**	283	**4.90**	**28.09**	0.1	Microwave	180°C	Mar Xpress (CEM)	-	Y	AOAC 2011.14 (2016)
22	**46298**	-	**-4.47**	-	0.2 to 0.3	Microwave	HNO_3	ICP-MS Perkin Elmer	-	-	AOAC 2015.06
23	**84500**	-	1.18	-	1.00	Dry Ashing		ICP-OES	422	-	AOAC 985.01
25	**64500**	0	-1.78	**-10.27**	5.0208 / 5.0213	HNO_3-HCl	Water	ICP-OES	Ca 396.847	-	USEPA Method 3050B
26	**69800**	3710	-1.00	**-3.07**	4.0	Dry ashing	Water & HCl (1+1)	AAS Shimadzu AA-7000	Ca 422.7	N	AOAC No. 975.03

Laboratory Number	Calcium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x) ± SD from Horwitz' s equation = 76533 ± 6761 mg/kg (CV 8.8%, n= 52) with u_{xpt} 1172 mg/kg*											
31	**15721**	-	**-8.99**	-	5	Dry Ashing	-	AAS, Agilent		N	AOAC 985.35
34	**655**	-	**-11.22**	-	2.000	Dry Ashing	HNO_3 and HCl	AAS Flame	Ca 422.7	92% (Na)	AOAC
37	**82823**	-	0.93	-	1	Wet Digestion	Nitric + perchloric	ICP-OES (Perkin Elmer Optima 8000)	Ca 317.933	N	AOAC (2016) 984.27
38	**74400**	2760	-0.32	-1.18	1.000	Dry Ashing	1N HNO_3	Flame AAS, Shimadzu AA6300	Ca	N	AOAC 985.35, 19th Ed 2012
39	**88800**	-	1.81	-	0.5	Microwave	-	AAS	Ca 422.7	Y	AOAC 985.35
41	**8**	-	**-11.32**	-	2	-	-	-	-	-	-
42	**72300**	4620	-0.63	-1.63	5	Dry Ashing	HNO_3-HCl	Flame AAS, Agilent 280 FS	Ca 422.7	N	AOAC 985.35.2005
43	**82241**	3938	0.84	**2.49**	0.5	Microwave	HNO_3	ICP-OES	Ca 317.933	N	AOAC
44	**81739**	6224	0.77	1.57	1.0000	Dry Ashing	-	AAS, Thermoscientific	Ca 422.7	N	AOAC 19th Ed
45	**78682**	3627	0.32	1.00	4	Dry Ashing	HCl+HNO_3+ DI (2+2+70 mL) on hotplate	AAS (Flame, Varian)	Ca 422.7	N	AOAC 968.08
48	**115695**	207	**5.79**	**33.28**	5	Dry Digestion	-	AA800 Perkin Elmer	Ca 422.7	N	MU-03/21 (AAS)
49	**82100**	3510	0.82	**2.64**	1, 2	Dry Ashing	Conc Nitric acid	AAS / AA-7000 Shimadzu	Ca 422.7	N	AOAC 20th Ed 2016
52	**84064**	-	1.11	-	1.0 (Ca, Fe)	Dry Ashing (Ca, Fe)	1 N HNO_3 (Ca, Fe)	Flame AAS (Shimadzu AA6300)	Ca 422.7	N	Modified AOAC 985.35 (Ca, Fe)

Laboratory Number	Calcium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x*) ± SD from Horwitz' s equation = 76533 ± 6761 mg/kg (CV 8.8%, n= 52) with u_{xpt} 1172 mg/kg											
53	**75807**	4599	-0.11	-0.28	0.3	Microwave	4 mL HNO_3, 1 mL HCl, 1 mL H_2O_2	ICPMS Thermo	-	-	In house method
54	**79100**	10000	0.38	0.50	1	Dry Ashing	HNO_3	ICP / Shimadzu	Ca 317.933	N	AOAC 984.27
55	**83912**	-	1.09	-	1.5	Wet digestion	-	ICP-OES	Ca 317.933	Y	AOAC (2012) 984.27
58	**85777**	3950	1.37	**4.03**	3.0	Dry Ash	HCl	ICP-OES	-	-	Dry Ashing and Quantitation by ICP-OES
60	**91100**	-	**2.15**	-	-	-	-	-	-	-	AOAC (2012) 968.08 (Ca, Mg)
61	**77500**	21200	0.14	0.09	1	Acid block digestion	HNO_3	Varian AA240 FS Fast Sequential AAS	Ca 422.7	N	A6407-26 AAS
63	**65787**	-	-1.59	-	-	-	-	-	-	-	-
64	**81343**	488	0.71	**4.02**	0.5048	Dry Ashing	1 N HNO3	Shimadzu AA6300	Ca 422.7	N	Modified AOAC 985.35
67	**90300**	-	**2.04**	-	2.0xxx	Dry Ash	Wet chemical	AAS, Perkin Elmer	Ca 422.67	N	AOAC 968.08
69	**58200**	-	**-2.71**	-	-	-	-	-	-	-	-
71	**8.36**	0.50	**-11.32**	**-65.29**	1.0020, 1.0062	Acid Digestion	HCl (1:3)	-	-	-	AOAC 927.02, Titration
72	**72800**	6920	-0.55	-1.02	3	Ashing	HNO_3	AAS / Analytik Jena	Ca 422.7	N	AOAC 985.35
73A	**8.36**	0.38	**-11.32**	**-65.29**	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	**8.44**	0.38	**-11.32**	**-65.29**	1	Dry ashing	Hot plate	AAS (280FS AA, Agilent Technology)	Ca 422.7	N	FTC-46.01 (refers to AOAC 968.08, 965.09)

Laboratory Number	Calcium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x*) ± SD from Horwitz' s equation = 76533 ± 6761 mg/kg (CV 8.8%, n= 52) with u_{xpt} 1172 mg/kg											
75	**92126**	3488	**2.31**	**7.42**	1	Wet digestion (hot block)	HNO_3 + H_2O_2	ICP-OES Agilent 5100	Ca 317.933	N	In House Method ICP-OES
77	**58955**	-	**-2.60**	-	-	-	-	-	-	-	-
78	**83871**	2516	1.09	**4.27**	0.5	Mircowave Digestion	Acid Digestion	Berghof Speedwave 4 Microwave Digestion Unit	Ca 393.366	-	MP-AES
81	**83700**	2910	1.06	**3.84**	1.0024	Dry Ashing (Ca, Fe)	1 N HNO_3 (Ca, Fe)	Shimadzu AAS AA 6300	Ca 422.7	N	AOAC 985.35 Mod (Ca, Fe)
82A	**60700**	1040	**-2.34**	**-12.35**	0.250	none	none	HPGe detector, Canberra	-	-	Neutron Activation Analysis (NAA)
82B	**106000**	5500	**4.36**	**9.86**	1.00	Microwave	Nitric Acid	AAS, GBC	-	Y	Flame SSA
83	**82605**	3004	0.90	**3.19**	0.3	Microwave Digestion with HNO_3	-	Microwave digester Mars Xpress, ICP MS Nex Ion (Perkin Elmer)	-	Y	Application Note, Perkin Elmer
84	**110000**	11000	**4.95**	**5.95**	0.5	Microwave Digestion	HNO_3 / H_2O_2	ICP-OES, ICP-MS	Ca 317.933	N	AOAC 999.10:2005
86	**85816**	4944	1.37	**3.39**	1.0000	Wet Digest	-	ICP-OES	Ca 315.8	Y	AOAC (2012) 984.27
87	**65800**	1710	-1.59	**-7.40**	2.5	Dry Ashing	HNO_3	Furnace Thermolyne	ICP-OES	N	MTD/FOD/CHM-09
88	**51645**	2	**-3.68**	**-21.24**	3	Dry Ashing	HNO_3 conc 10 mL	AAS GBC Flame	Ca 422.3	N	In house method (AAS)
89	**16307**	245	**-8.91**	**-51.11**	2	Dry Ashing	1.5% HNO_3	AAS Agilent	Various	N	AOAC 985.35
90	**52977**	-	**-3.48**	-	1	Ultrawave	-	ICP-OES	Ca 422.673	-	-

Laboratory Number	Calcium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x*) ± SD from Horwitz' s equation = 76533 ± 6761 mg/kg (CV 8.8%, n= 52) with u_{xpt} 1172 mg/kg											
91	**68100**	-	-1.25	-	-	-	-	-	-	-	-
92	**9140**	-	**-9.97**	-	1	Ashing	HNO_3	ICP-OES	-	-	-
93	**9800**	-	**-9.87**	-	0.05	Charring, Dry ashing	Hotplate, Furnace	Flame Photometer, Sherwood	N/A	N/A	AOAC 985.35
94	**94926**	-	**2.72**	-	1.5	Dry ashing	-	ICP-OES / Perkin Elmer	Ca 317.9	Y	AOAC (2012) 984.27
95	**84100**	2800	1.12	**4.14**	-	-	-	-	-	-	-

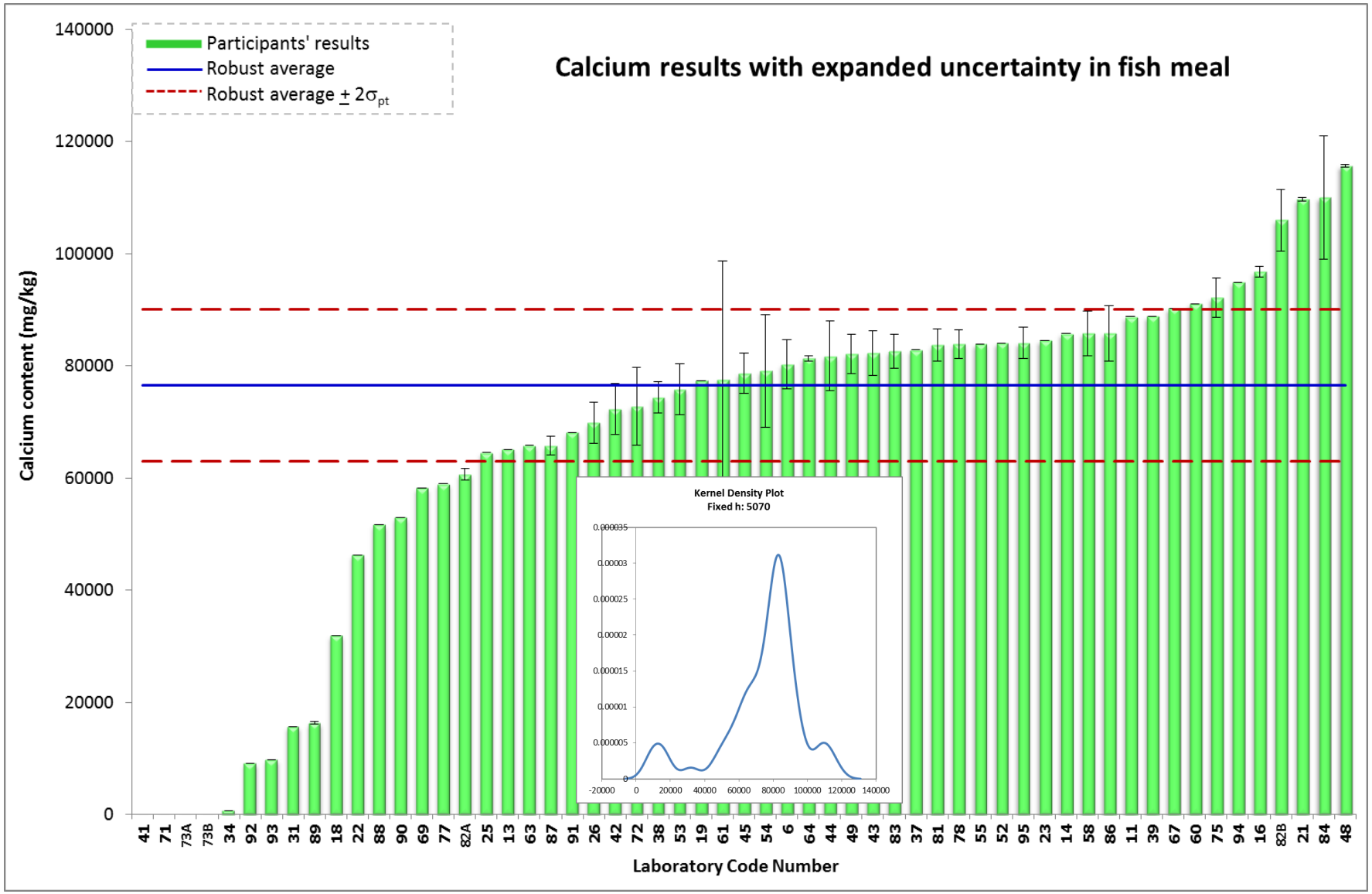

Figure 70. Distribution of **calcium** results (ascending order) in fish meal with expanded uncertainty

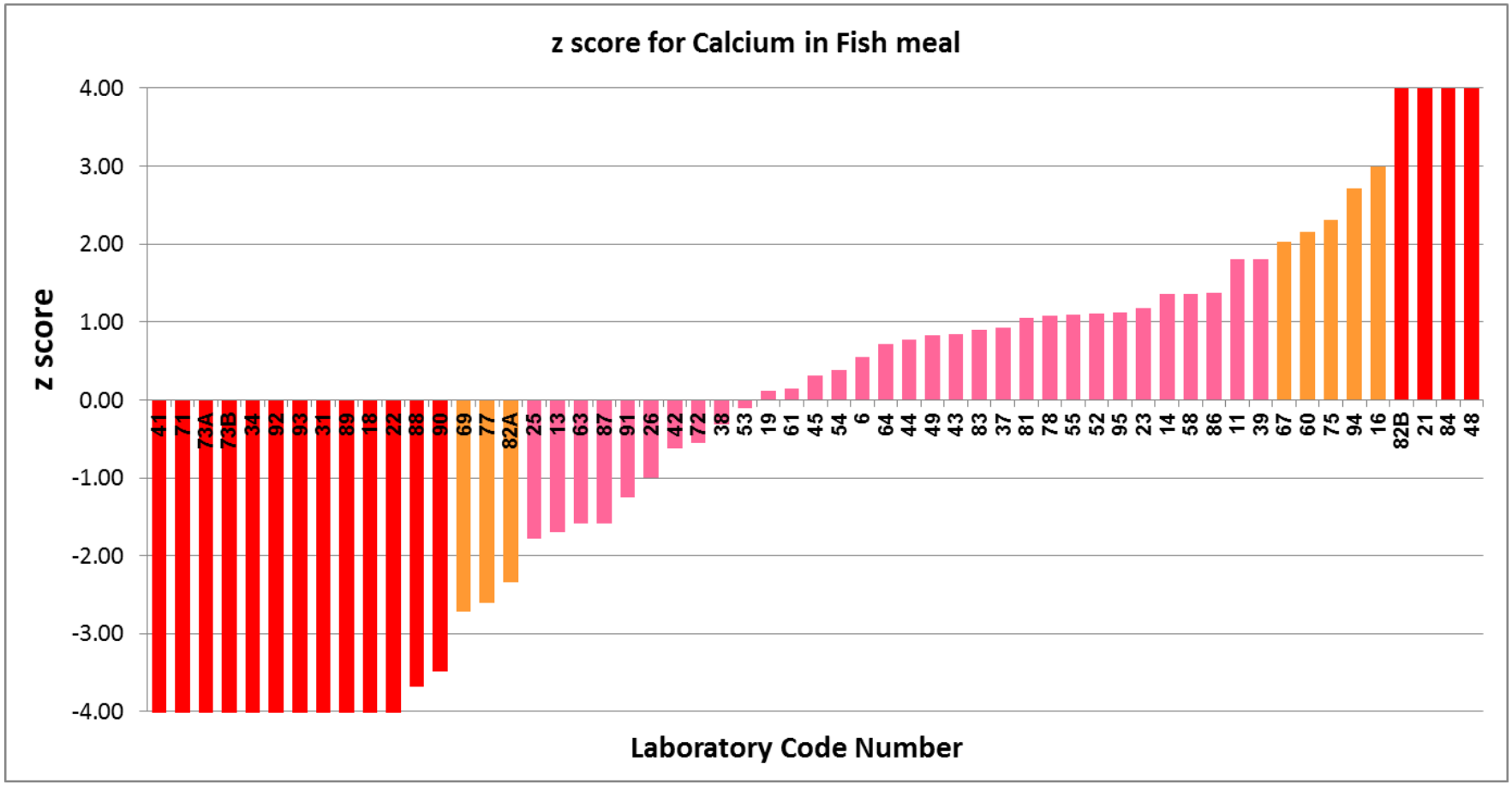

Figure 71. Plot of ordered *z* scores for **calcium** results in fish meal

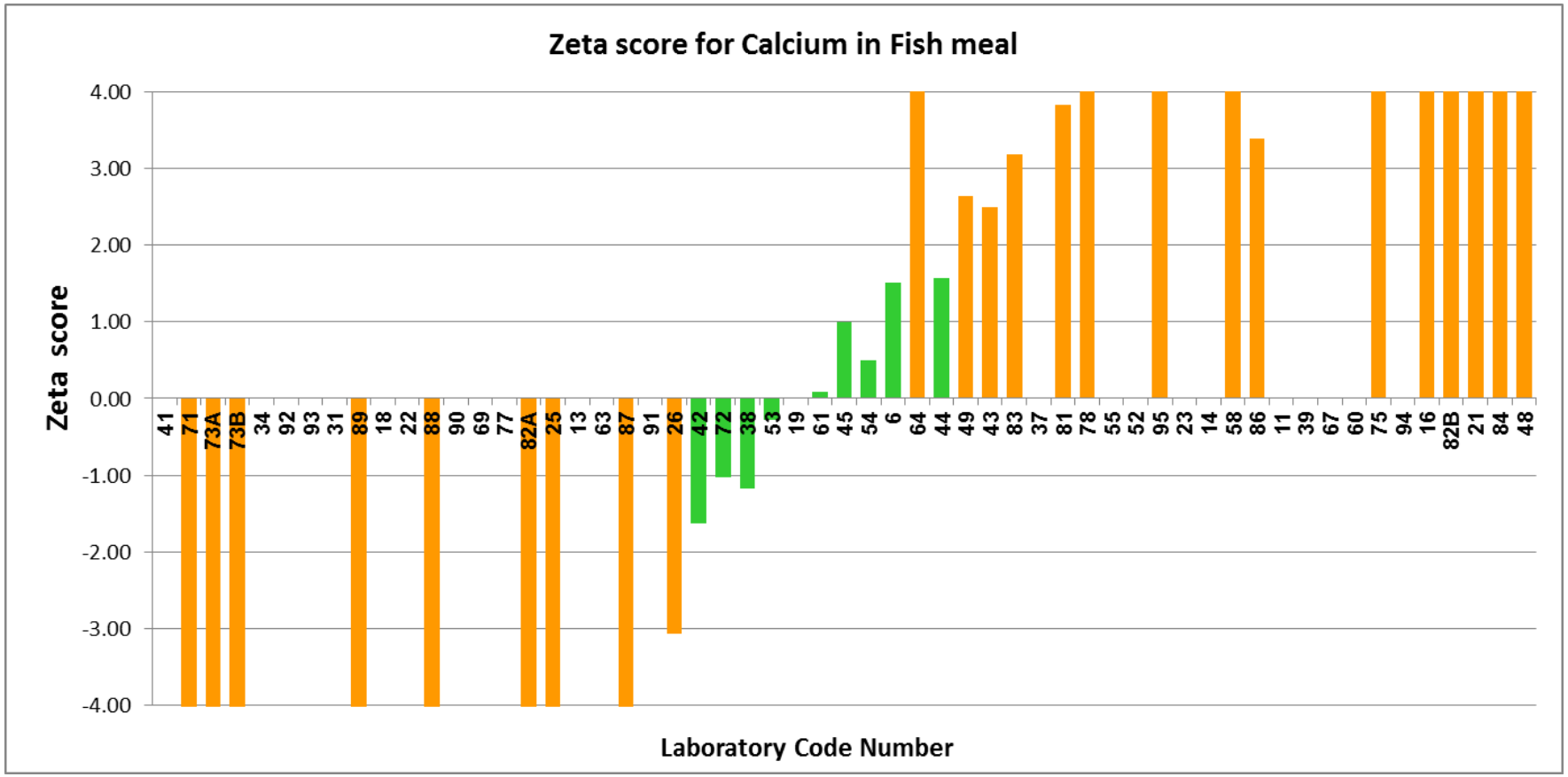

Figure 72. Plot of *Zeta* score for **calcium** in fish meal, following the ordered z scores in the above Figure 71.

Table 25. Evaluation of laboratory performance **magnesium** analysis (mg/kg, as received) in fish meal

Laboratory Number	Magnesium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x*) ± robust SD (s*) = 3101 ± 443 mg/kg (CV 14.3%, n= 42), u_{xpt} = 85 mg/kg											
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00							
6	**3353**	-	0.57	-	1.0000	Acid	HCl:HNO_3:H_2O	AAS	-	Y	AOAC (2016), 20th Ed, 928.08, 985.35 (50.1.14)
11	**2811**	-	-0.66	-	2.0000	Dry Ashing	HCL:H_2O	AAS	-	Y	AOAC (2016), 975.03, 985.35
13	**978**	-	**-4.80**	-	0.5	Microwave	HNO_3 10 mL + HCl 2	Analytikal Jena ContrAA 800 D	Mg 285	N	Internal Method
14	**2993**	-	-0.24	-	0.1 - 0.2	Ashing	Conc HNO_3, Conc $HClO_4$	ICP Horiba Jobin Yvon	Mg 279.553	Y	AOAC 975.03, 984.27
16	**3514**	35	0.93	**4.74**	0.5	Hot plate	HNO_3+H_2O_2	ICP-OES Optima 7000	Mg 279.077	N	In-house Method
18	**2710**	-	-0.88	-	2.0	Dry Ashing	HNO_3	AAS, Varian	Various	N	AOAC 968.08
21	**3549**	-	1.01	-	0.1	Microwave	180°C	Mar Xpress (CEM)	-	Y	AOAC 2011.14 (2016)
22	**2868**	-	-0.53	-	0.2 to 0.3	Microwave	HNO_3	ICP-MS Perkin Elmer	-		AOAC 2015.06
23	**2960**	-	-0.32	-	1.00	Dry Ashing	-	ICP-OES	285		AOAC 985.01
25	**3000**	0	-0.23	-1.19	5.0208 / 5.0213	HNO_3-HCl	Water	ICP-OES	Mg 280.27		USEPA Method 3050B
26	**2830**	142	-0.61	**-2.44**	4.0	Dry ashing	Water & HCl (1+1)	AAS Shimadzu AA-7000	Mg 285.2	N	AOAC No. 975.03
31	**838**	-	**-5.12**	-	5	Dry Ashing	-	AAS, Agilent	-	N	AOAC 985.35
37	**3286**	-	0.42	-	1	Wet Digestion	Nitric + perchloric	ICP-OES (Perkin Elmer	Mg 285,213	N	AOAC (2016) 984.27
38	**2870**	113	-0.52	**-2.26**	1.000	Dry Ashing	1N HNO_3 (0.1M	Flame AAS, Shimadzu	Mg 285.20	N	AOAC 985.35, 19th Ed 2012
39	**3480**	-	0.86	-	0.5	Microwave	-	AAS	Mg 285.2	Y	AOAC 985.35
42	**2360**	69	-1.68	**-8.05**	5	Dry Ashing	HNO_3-HCl	Flame AAS, Agilent 280 FS	Mg 202.6	N	AOAC 985.35.2005
43	**3332**	167	0.52	1.94	0.5	Microwave	HNO_3	ICP-OES	Mg 285.213	N	AOAC

Laboratory Number	Magnesium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x*) ± robust SD (s*) = 3101 ± 443 mg/kg (CV 14.3%, n= 42), u_{xpt} = 85 mg/kg											
45	**3138**	124	0.08	0.34	4	Dry Ashing	HCl+HNO_3+ DI (2+2+70	AAS (Flame, Varian)	Mg 285.2	N	AOAC 968.08
48	**5212**	29	**4.77**	**24.39**	5	Dry Digestion		AA800 Perkin Elmer	Mg 285.2	N	MU-03/21 (AAS)
49	**3000**	151	-0.23	-0.89	1, 2	Dry Ashing	Conc Nitric acid	AAS / AA-7000 Shimadzu	Mg 285.2	N	AOAC 20th Ed 2016
53	**1988**	56	**-2.52**	**-12.39**	0.3	Microwave	4 mL HNO_3, 1 mL HCl, 1	ICPMS Thermo	-	-	In house method
54	**3290**	58	0.43	**2.09**	1	Dry Ashing	HNO_3	ICP / Shimadzu	Mg 279.553	N	AOAC 984.27
55	**3255**	-	0.35	-	1.5	Wet digestion		ICP-OES	Mg 280.270	Y	AOAC (2012) 984.27
58	**3247**	8	0.33	1.70	3.0	Dry Ash	HCl	ICP-OES	-		
60	**3300**	-	0.45	-	-	-	-	-	-		AOAC (2012) 968.08 (Ca, Mg)
61	**2990**	437	-0.25	-0.47	1	Acid block digestion	HNO_3	Varian AA240 FS Fast	Mg 285.2	N	A6407-26 AAS (A6417 Spectro Method for P)
63	**3006**	-	-0.22	-							
67	**3640**	-	1.22	-	2.0xxx	Dry Ash	Wet chemical	AAS, Perkin Elmer	Mg 285.21	N	AOAC 968.08
69	**2570**	-	-1.20	-	-	-	-	-	-	-	-
72	**3010**	256	-0.21	-0.59	3	Ashing	HNO_3	AAS / Analytik Jena	Mg 285.2	N	AOAC 985.35
75	**4718**	27	**3.65**	**18.72**	1	Wet digestion	HNO_3 + H_2O_2	ICP-OES Agilent 5100	Mg 279.078	N	In House Method ICP-OES
77	**2489**	-	-1.38	-	-	-	-	-	-	-	-
78	**3293**	-	0.43	-	0.5	Mircowave Digestion	Acid Digestion	Berghof Speedwave 4	Mg 279.08	-	MP-AES
82A	**2340**	600	-1.72	**-2.44**	0.250	none	none	HPGe detector, Canberra	-		Neutron Activation Analysis (NAA)
83	**3288**	4.86	0.42	**2.19**	0.3	Microwave Digestion	-	Microwave digester Mars	-	Y	Application Note, Perkin Elmer
84	**3630**	363	1.19	**2.64**	0.5	Microwave Digestion	HNO_3 / H_2O_2	ICP-OES, ICP-MS	Mg 285.213	N	AOAC 999.10:2005
86	**3294**	212	0.44	1.42	1.0000	Wet Digest	-	ICP-OES	Mg 280.2	Y	AOAC (2012) 984.27

Laboratory Number	Magnesium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x*) ± robust SD (s*) = 3101 ± 443 mg/kg (CV 14.3%, n= 42), u_{xpt} = 85 mg/kg											
87	**3587**	9.14	1.10	**5.68**	2.5	Dry Ashing	HNO_3	Furnace Thermolyne	ICP-OES	N	MTD/FOD/CHM-09
88	**1529**	0.04	**-3.55**	**-18.42**	3	Dry Ashing	HNO_3 conc 10 mL	AAS GBC Flame	Mg 285.20	N	In house method (AAS)
89	**2058**	31	**-2.36**	**-12.03**	2	Dry Ashing	1.5% HNO_3	AAS Agilent	Various	N	AOAC 985.35
90	**3069**	-	-0.07	-	1	Ultrawave		ICP-OES	Mg 285.213	-	
91	**3010**	-	-0.21	-	-	-	-	-	-	-	-
92	**341**	-	**-6.24**	-	1	Ashing	HNO_3	ICP-OES	-	-	
94	**3618**	-	1.17	-	1.5	Dry ashing (Fe: Wet	-	ICP-OES / Perkin Elmer	Mg 383.2	Y	AOAC (2012) 984.27
95	**3260**	360	0.36	0.80	-	-	-	-	-	-	-

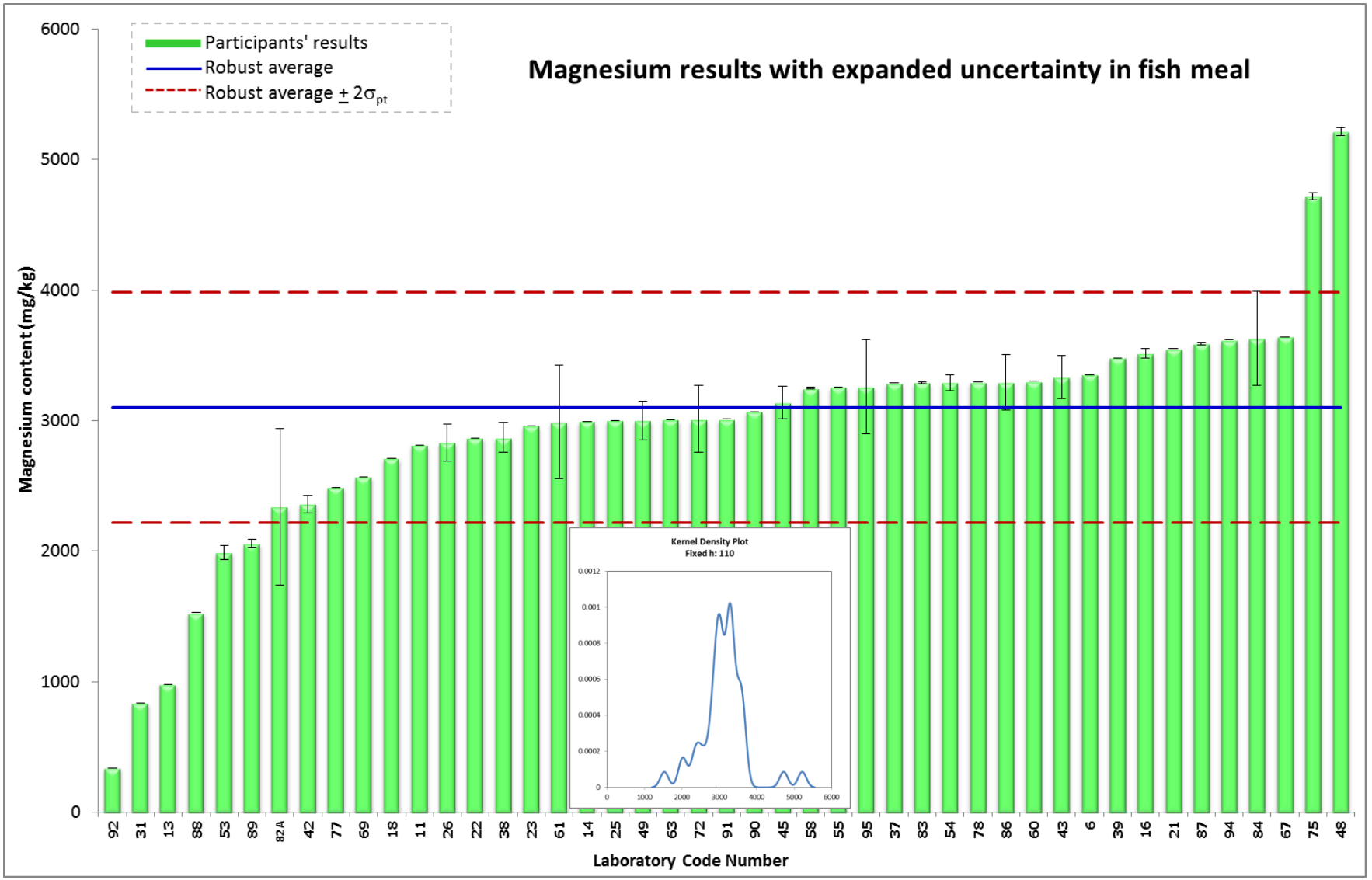

Figure 73. Distribution of **magnesium** results (ascending order) in fish meal with expanded uncertainty

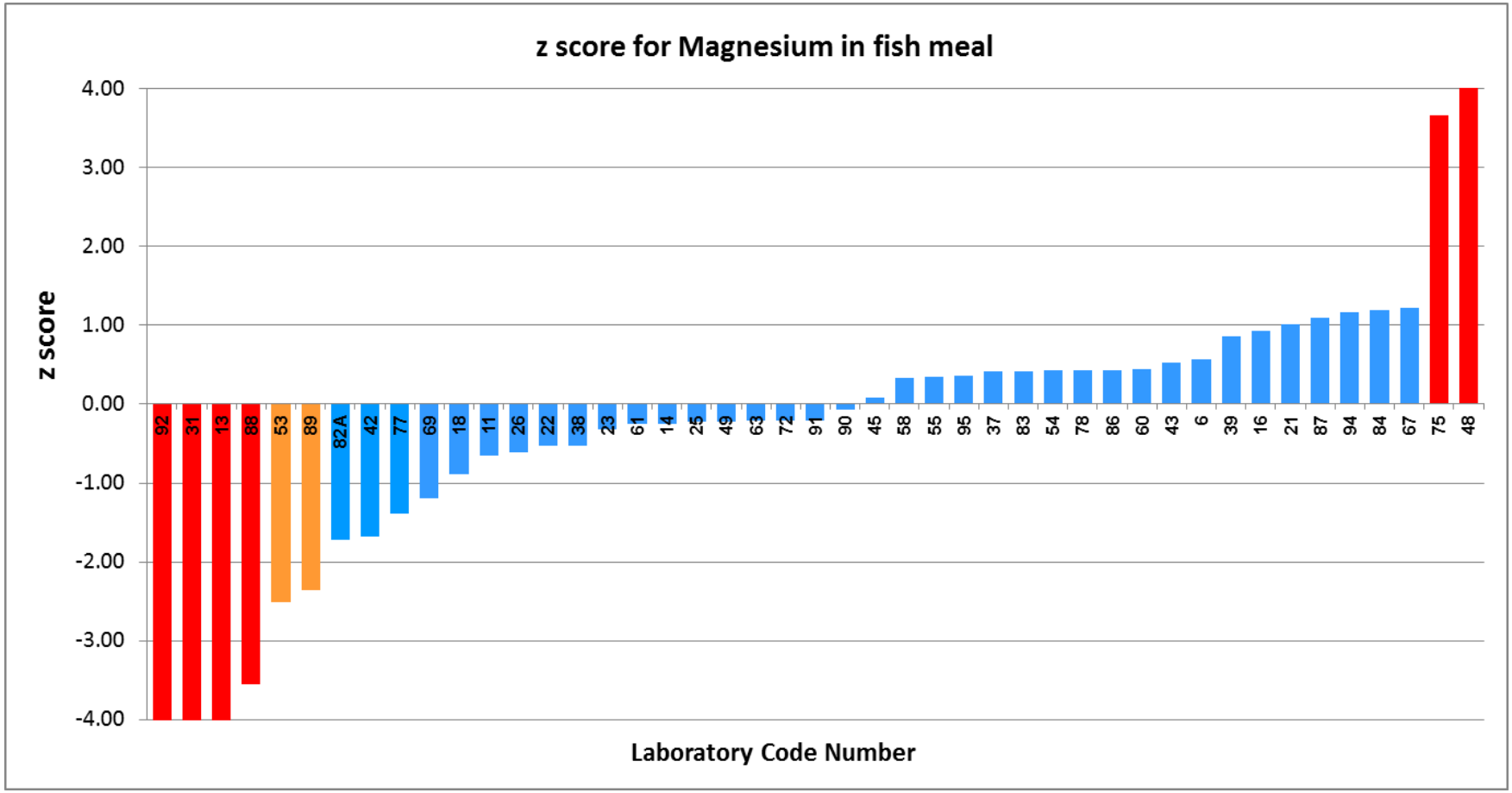

Figure 74. Plot of ordered *z* scores for **magnesium** results in fish meal

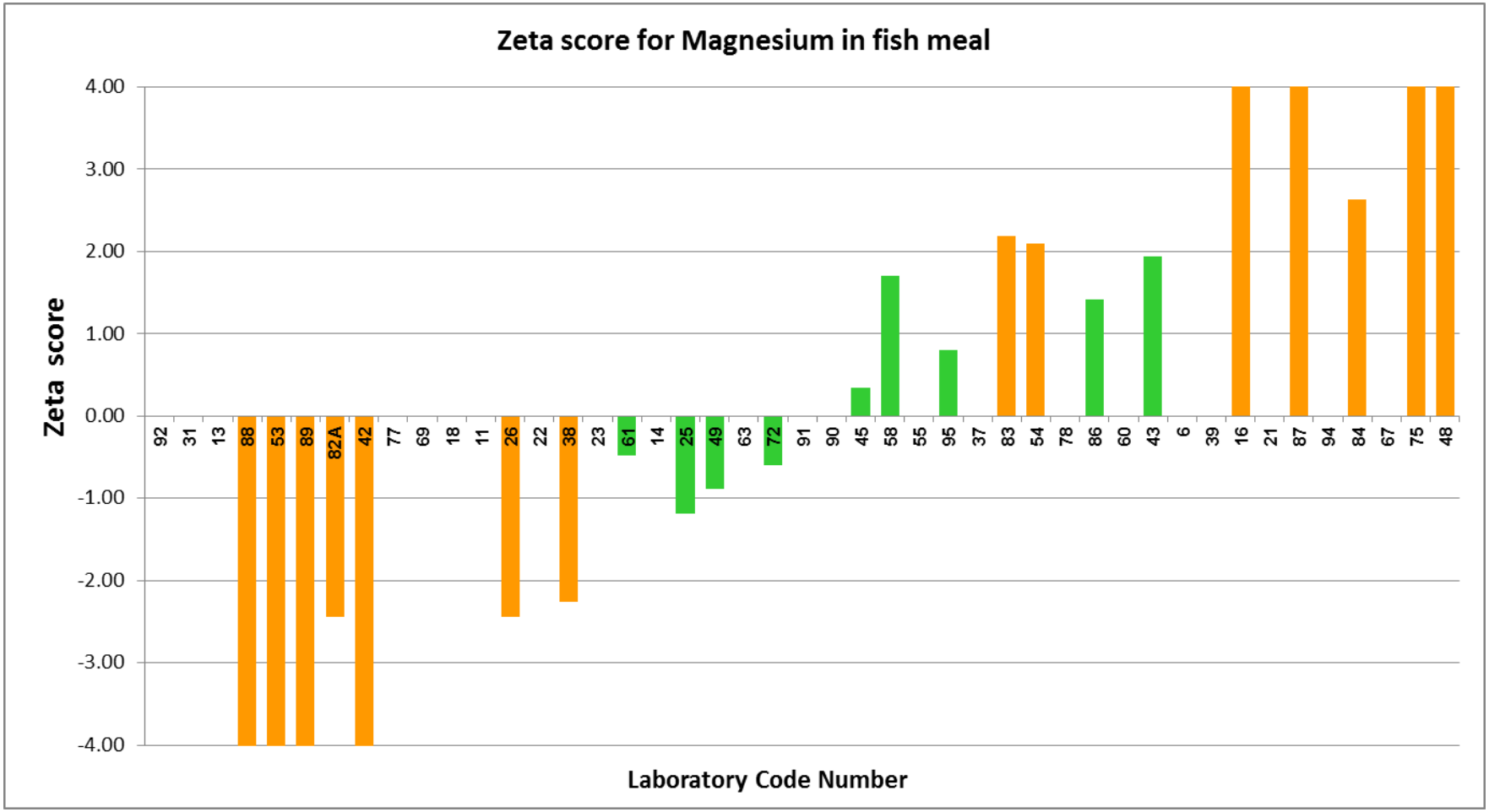

Figure 75. Plot of *Zeta* score for **magnesium** in fish meal, following the ordered z scores in the above Figure 74.

Table 26. Evaluation of laboratory performance **phosphorus** analysis (mg/kg, as received) in fish meal

Laboratory Number	P 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*) ± robust SD (s*) = 43451 ± 2497 mg/kg (CV 5.7%, n= 33) with u_{xpt} = 543 mg/kg											
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00							
2	**7820**	-	**-14.27**	-	-	-	-	-	-	-	-
6	**42734**	1234	-0.29	-0.87	1.0000	Acid	HCl:HNO_3:H_2O	AAS	-	Y	AOAC (2016), 20th Ed, 928.08, 985.35 (50.1.14)
11	**40554**	-	-1.16	-	2.0000	Dry Ashing	HCL:H_2O	AAS	-	Y	AOAC (2016), 975.03, 985.35
14	**44783**	-	0.53	-	0.1 - 0.2	Ashing	Conc HNO_3, Conc $HClO_4$	ICP Horiba Jobin Yvon	P 213.618	Y	AOAC 975.03, 984.27
15	**44000**	-	0.22	-	-	-	-	-	-	-	-
16	**44538**	445	0.44	1.85	0.5	Hot plate	HNO_3+H_2O_2	ICP-OES Optima 7000 DV Perkin Elmer	-	N	In-house Method
18	**43800**	-	0.14	-	2.0	Dry Ashing	HNO_3	AAS, Varian	-	N	AOAC 968.08
19	**43410**	-	-0.02	-	1	By Furnace	HNO_3:H_2O (1:1)	Ca Manual by Buret	P 400	N	AOAC 927.02, 944.03, 965.17
21	**115**	-	**-17.36**	-	0.1	Microwave	180°C	Mar Xpress (CEM)	-	Y	AOAC 2011.14 (2016)
22	**4061**	-	**-15.78**	-	0.2 to 0.3	Microwave	HNO_3	ICP-MS Perkin Elmer	-	-	AOAC 2015.06
23	**43400**	-	-0.02	-	1.00	Dry Ashing	-	ICP-OES	-	-	AOAC 985.01
25	**42200**	-	-0.50	-	5.0208 / 5.0213	HNO_3-HCl	Water	ICP-OES	-	-	USEPA Method 3050B
31	**38514**	3127	-1.98	**-2.98**	5	Dry Ashing	-	AAS, Agilent	-	N	AOAC 985.35
37	**41268**	-	-0.87	-	1	Wet Digestion	Nitric + perchloric	ICP-OES (Perkin Elmer Optima 8000)	P 213.617	N	AOAC (2016) 984.27

Laboratory Number	P 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*) ± robust SD (s*) = 43451 ± 2497 mg/kg (CV 5.7%, n= 33) with u_{xpt} = 543 mg/kg											
38	**47400**	100	1.58	**7.24**	1.000	Dry Ashing	1N HNO_3 (0.1M HNO3 for Fe)	Flame AAS, Shimadzu AA6300	-	N	AOAC 985.35, 19th Ed 2012
41	**3**	-	**-17.40**	-	2	-	-	-	-	-	-
42	**42600**	768	-0.34	-1.28	5	Dry Ashing	HNO_3-HCl	Flame AAS, Agilent 280 FS	-	N	AOAC 985.35.2005
43	**43997**	6525	0.22	0.17	0.5	Microwave	HNO_3	ICP-OES	-	N	AOAC
45	**43951**	2577	0.20	0.36	4	Dry Ashing	HCl+HNO_3+DI (2+2+70 mL) on hotplate	AAS (Flame, Varian)	-	N	AOAC 968.08
48	**44803**	384	0.54	**2.35**	5	Dry Digestion	-	AA800 Perkin Elmer	-	N	MU-03/21 (AAS)
49	**44100**	367	0.26	**1.13**	1, 2	Dry Ashing	Conc Nitric acid	AAS / AA-7000 Shimadzu	-	N	AOAC 20th Ed 2016
54	**42441**	299	-0.40	-1.79	1	Dry Ashing	HNO_3	ICP / Shimadzu	-	N	AOAC 984.27
55	**43912**	-	0.18	-	1.5	Wet digestion	-	ICP-OES	-	Y	AOAC (2012) 984.27
58	**41706**	330	-0.70	-3.07	3.0	Dry Ash	HCl	ICP-OES	-	-	-
60	**35500**	-	**-3.18**	-	-	-	-	-	-		AOAC (2012) 968.08 (Ca, Mg)
61	**33900**	4710	**-3.83**	-3.95	1	Acid block digestion	HNO_3	Shimadzu UV-2700 for P	-	N	A6407-26 AAS (A6417 Spectro Method for P)
67	**43600**	6100	0.06	0.05	2.0xxx	Dry Ash	Wet chemical	AAS, Perkin Elmer	-	N	AOAC 968.08
68	**45703**	-	0.90	-	-	-	-	-	-	-	-
69	**51400**	-	**3.18**	-	-	-	-	-	-	-	-
71	**4**	0	**-17.40**	**-79.97**	1.0020, 1.0062	Acid Digestion	HCl (1:3)	-	-	-	AOAC 927.02, Titration

Laboratory Number	P 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*) ± robust SD (s*) = 43451 ± 2497 mg/kg (CV 5.7%, n= 33) with u_{xpt} = 543 mg/kg											
73A	**4**	0	**-17.40**	**-79.97**	1	Dry ashing	Hot plate	AAS (280FS AA, Agilent Technology)	-	N	FTC-46.01 (refers to AOAC 968.08, 965.09)
75	**62797**	607	**7.75**	**31.09**	1	Wet digestion (hot block)	HNO_3 + H_2O_2	ICP-OES Agilent 5100	-	N	In House Method ICP-OES
77	**28245**	-	**-6.09**	-	-	-	-	-	-	-	-
79	**277**	-	**-17.29**	-	-	-	-	-	-	-	-
80	**44600**	-	0.46	-	-	-	-	-	-	-	-
83	**46338**	1033	1.16	**3.85**	0.3	Microwave Digestion with HNO_3	-	Microwave digester Mars Xpress,	ICP MS Nex Ion (Perkin Elmer)	Y	Application Note, Perkin Elmer
86	**41864**	2550	-0.64	-1.15	1.0000	Wet Digest	-	ICP-OES	-	Y	AOAC (2012) 984.27
87	**46994**	137	1.42	**6.47**	2.5	Dry Ashing	HNO_3	Furnace Thermolyne	ICP-OES	N	MTD/FOD/CHM-09
88	**4480**	2.14	**-15.61**	**-71.74**	3	Dry Ashing	HNO_3 conc 10 mL	AAS GBC Flame	-	N	In house method (AAS)
90	**42624**	-	-0.33	-	1	Ultrawave		ICP-OES	-	-	-
92	**4355**	-	**-15.66**	-	1	Ashing	HNO_3	ICP-OES	-	-	-
95	**43550**	1700	0.04	0.10	-	-	-	-	-	-	-

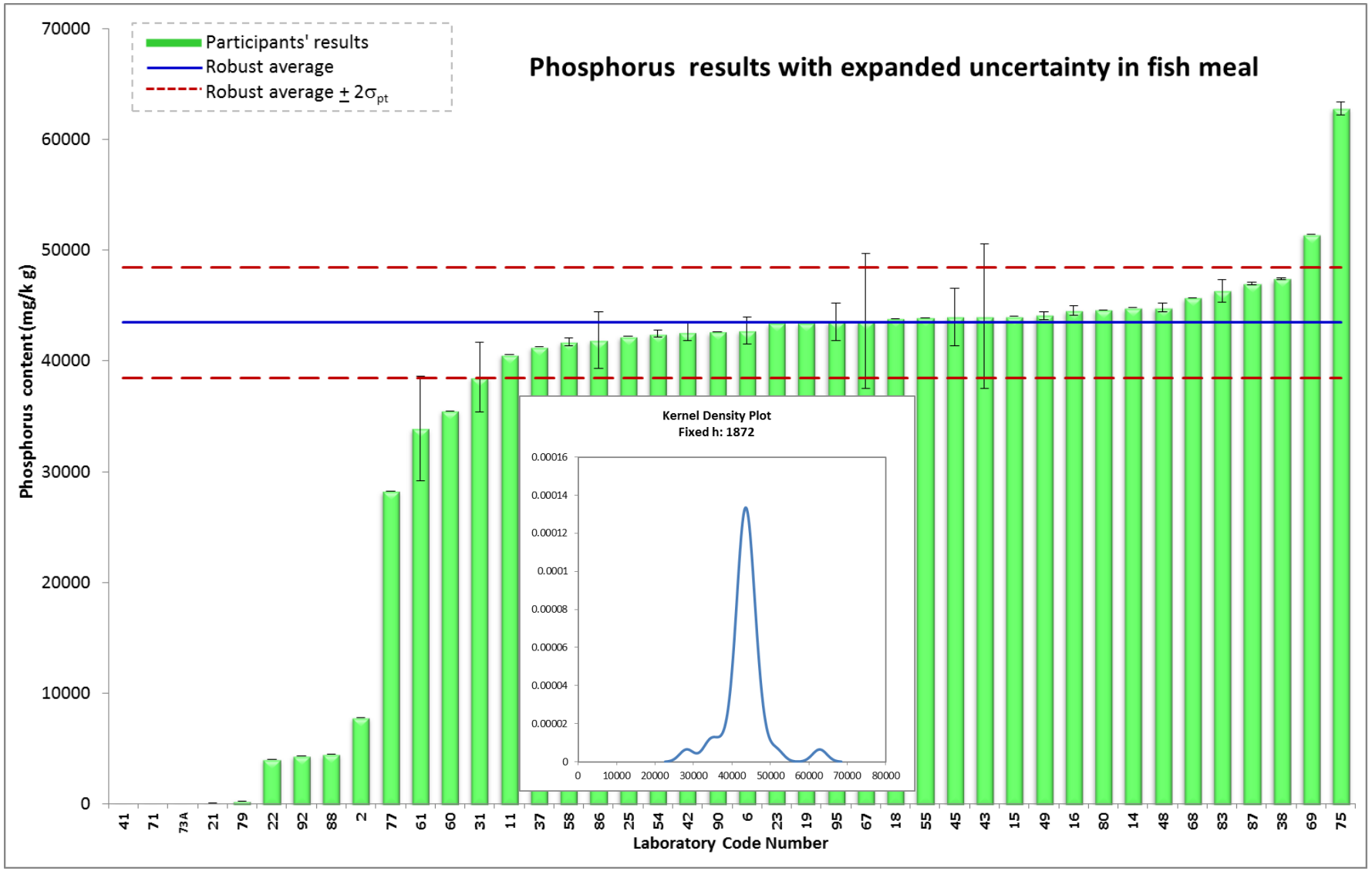

Figure 76. Distribution of **phosphorus** results (ascending order) in fish meal with expanded uncertainty

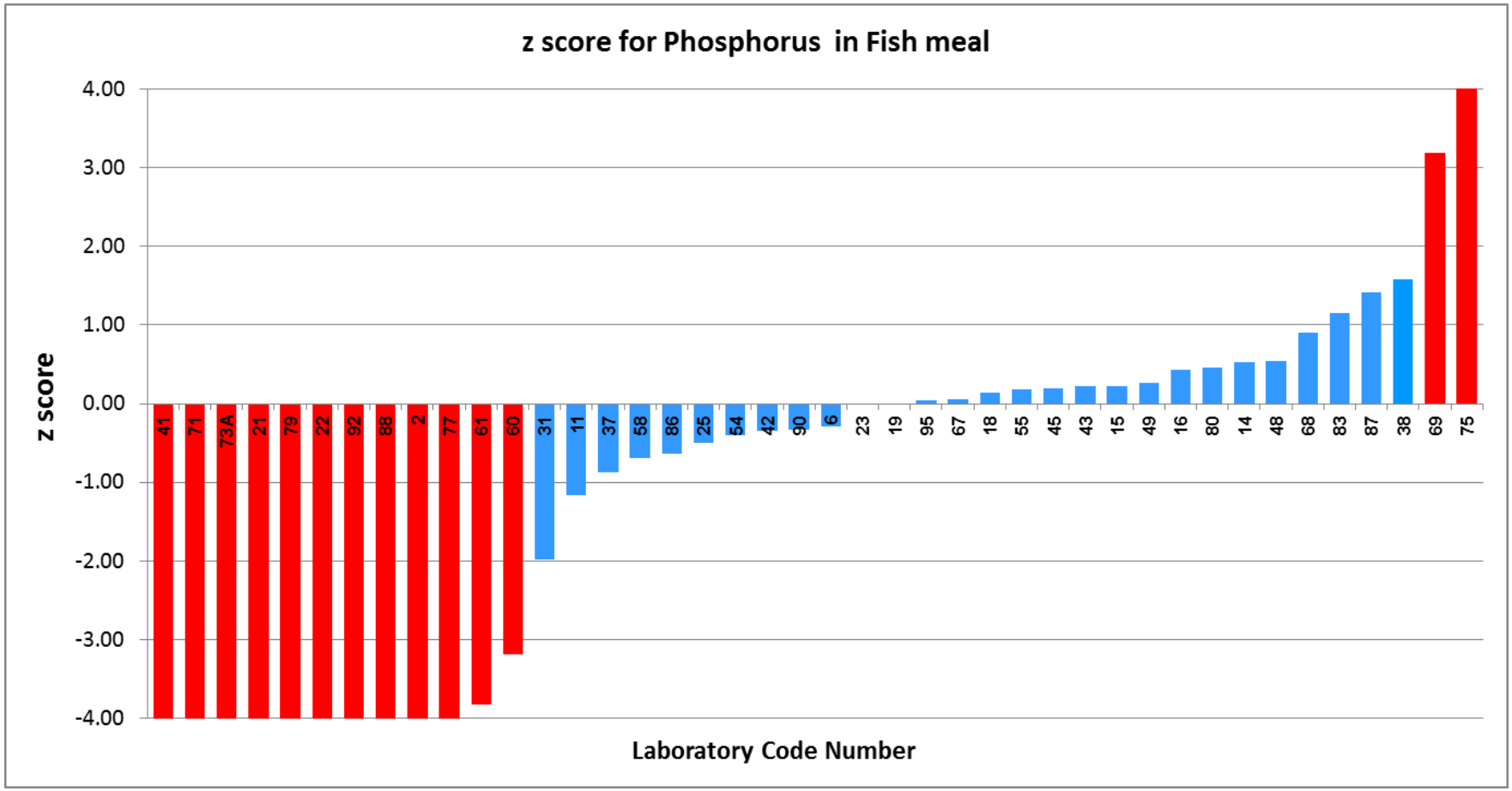

Figure 77. Plot of ordered *z* scores for **phosphorus** results in fish meal

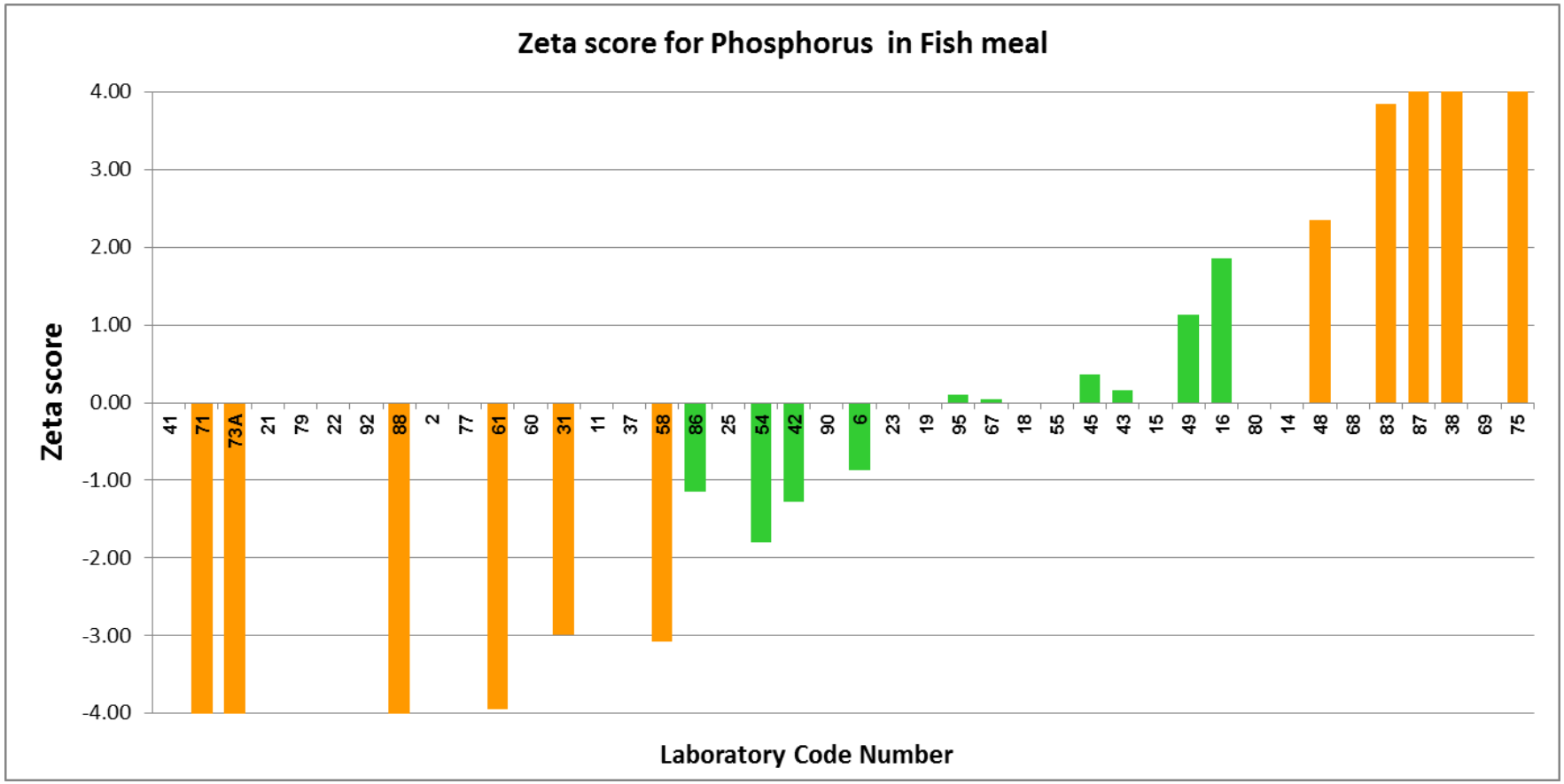

Figure 78. Plot of *Zeta* score for **phosphorus** in fish meal, following the ordered z scores in the above Figure 77.

Table 27. Evaluation of laboratory performance **sodium** analysis (mg/kg, as received) in fish meal

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*) ± robust SD (s*) = 9943 ± 1367 mg/kg (CV 13.7%, n= 52) with u_{xpt} = 249 mg/kg											
	Acceptance criteria =		|z score| ≤ 2.00	|ζ score| ≤ 2.00							
6	**9326**	1412	-0.45	-0.82	1.0000	Acid	$HCl:HNO_3:H_2O$	AAS	Na 589.0	Y	AOAC (2016), 20th Ed, 928.08, 985.35
7	**515**	-	**-6.90**	-	1.0039/1.0316	Ashing	HCL	Flame Photometer	-	N	-
11	**10102**	-	0.12	-	2.0000	Dry Ashing	$HCL:H_2O$	AAS	Na 330.3	Y	AOAC (2016), 975.03, 985.35
13	**7520**	-	-1.77	-	0.5	Microwave	HNO_3 10 mL + HCl 2 mL	Analytikal Jena ContrAA	K 766	N	Internal Method
14	**9865**	-	-0.06	-	0.1 - 0.2	Ashing	Conc HNO_3, Conc $HClO_4$	ICP Horiba Jobin Yvon	Na 588.995	Y	AOAC 975.03, 984.27
16	**11380**	114	1.05	**5.62**	0.5	Hot plate	$HNO_3+H_2O_2$	ICP-OES Optima 7000	Na 588.995	N	In-house Method
18	**10300**	-	0.26	-	2.0	Dry Ashing	HNO_3	AAS, Varian	Various	N	AOAC 968.08
21	**11289**	29	0.98	**5.39**	0.1	Microwave	180°C	Mar Xpress (CEM)	-	Y	AOAC 2011.14 (2016)
22	**9589**	-	-0.26	-	0.2 to 0.3	Microwave	HNO_3	ICP-MS Perkin Elmer	-	-	AOAC 2015.06
23	**9690**	-	-0.19	-	1.00	Dry Ashing	-	ICP-OES	589	-	AOAC 985.01
25	**13000**	0	**2.24**	**12.26**	5.0208 / 5.0213	HNO_3-HCl	Water	ICP-OES	Na 588.995	-	USEPA Method 3050B
26	**8220**	518	-1.26	**-4.79**	4.0	Dry ashing	Water & HCl (1+1)	AAS Shimadzu AA-	Na 589.0	N	AOAC No. 975.03
31	**7712**	-	-1.63	-	5	Dry Ashing	-	AAS, Agilent		N	AOAC 985.35
34	**10400**	-	0.33	-	2.000	Dry Ashing	HNO_3 and HCl	AAS Flame	Na 589	92% (Na)	AOAC
37	**10181**	-	0.17	-	1	Wet Digestion	Nitric + perchloric	ICP-OES (Perkin Elmer	Na 589.592	N	AOAC (2016) 984.27
38	**9860**	1390	-0.06	-0.11	1.000	Dry Ashing	1N HNO_3 (0.1M HNO3 for Fe)	Flame AAS, Shimadzu	Na 589.0	N	AOAC 985.35, 19th Ed 2012
39	**10200**	-	0.19	-	0.5	Microwave	-	AAS	Na 589.0	Y	AOAC 985.35
42	**2530**	102	**-5.42**	**-29.14**	5	Dry Ashing	HNO_3-HCl	Flame AAS, Agilent 280 FS	Na 589.9	N	AOAC 985.35.2005

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) ± robust SD (s*) = 9943 ± 1367 mg/kg (CV 13.7%, n= 52) with u_{xpt} = 249 mg/kg*											
43	**10456**	145	0.37	1.97	0.5	Microwave	HNO_3	ICP-OES	Na 568.821	N	AOAC
44	**9881**	143	-0.05	-0.24	1.0000	Dry Ashing	-	AAS, Thermoscientif	Na 589	N	AOAC 19th Ed
45	**8195**	625	-1.28	**-4.37**	4	Dry Ashing	$HCl+HNO_3$+DI (2+2+70 mL)	AAS (Flame, Varian)	Na 589.0	N	AOAC 968.08
48	**9877**	42	-0.05	-0.26	5	Dry Digestion		AA800 Perkin Elmer	Na 330.2	N	MU-03/21 (AAS)
49	**8950**	448	-0.73	**-2.96**	1, 2	Dry Ashing	Conc Nitric acid	AAS / AA-7000	Na 589.0	N	AOAC 20th Ed 2016
50	**10311**	1126	0.27	0.60	2.0000	Wet	Acid	Flame AAS (Varian)	330.3	N	AOAC 985.35
52	**9345**	-	-0.44	-	1.0 (Ca, Fe)	Dry Ashing (Ca, Fe)	1 N HNO_3 (Ca, Fe)	Flame AAS (Shimadzu	Na 589.0	N	Modified AOAC 985.35 (Ca, Fe),
53	**6687**	66	**-2.38**	**-12.95**	0.3	Microwave	4 mL HNO_3, 1 mL HCl, 1 mL	ICPMS Thermo	-	-	In house method
54	**11000**	412	0.77	**3.27**	1	Dry Ashing	HNO_3	ICP / Shimadzu	Na 589.592	N	AOAC 984.27
55	**9905**	-	-0.03	-	1.5	Wet digestion	-	ICP-OES	Na 589.592	Y	AOAC (2012) 984.27
58	**9184**	87	-0.56	**-3.00**	3.0	Dry Ash	HCl	ICP-OES	-	-	-
60	**100**	-	**-7.20**	-	-	-	-	-	-	-	AOAC (2012) 968.08 (Ca, Mg)
61	**9360**	1290	-0.43	-0.84	1	Acid block digestion	HNO_3	Varian AA240 FS Fast	Na 589.6	N	A6407-26 AAS (A6417 Spectro
63	**10418**	-	0.35	-	-	-	-	-	-	-	-
67	**1020**	-	**-6.53**	-	2.0xxx	Dry Ash	Wet chemical	AAS, Perkin Elmer	Na 589.00	N	AOAC 968.08
69	**9950**	-	0.01	-	-	-	-	-		-	-
72	**13600**	1160	**2.68**	**5.79**	3	Ashing	HNO_3	AAS / Analytik Jena	Na 589.0	N	AOAC 985.35
75	**14445**	739	**3.29**	**10.10**	1	Wet digestion (hot	$HNO_3 + H_2O_2$	ICP-OES Agilent 5100	Na 589.592	N	In House Method ICP-OES
77	**7199**	-	**-2.01**	-	-	-	-	-		-	-
78	**10367**	402	0.31	1.32	0.5	Mircowave Digestion	Acid Digestion	Berghof Speedwave 4	Na 589.592	-	MP-AES

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*) ± robust SD (s*) = 9943 ± 1367 mg/kg (CV 13.7%, n= 52) with u_{xpt} = 249 mg/kg											
81	**9410**	179	-0.39	**-2.01**	1.0024	Dry Ashing (Ca, Fe)	1 N HNO_3 (Ca, Fe)	Shimadzu AAS AA 6300	Na 589.0	N	AOAC 999.10 Mod (Na, K), 985.35
82A	**7880**	570	-1.51	**-5.45**	0.250	none	none	HPGe detector,	-	-	Neutron Activation Analysis (NAA)
83	**9633**	389	-0.23	-0.98	0.3	Microwave Digestion	-	Microwave digester Mars	ICP MS Nex Ion (Perkin	Y	Application Note, Perkin Elmer
84	**11600**	1160	1.21	**2.62**	0.5	Microwave Digestion	HNO_3 / H_2O_2	ICP-OES, ICP-MS	Na 559.592	N	AOAC 999.10:2005
86	**10261**	614	0.23	0.80	1.0000	Wet Digest	-	ICP-OES	Na 589.5	Y	AOAC (2012) 984.27
87	**9754**	63	-0.14	-0.75	2.5	Dry Ashing	HNO_3	Furnace Thermolyne	ICP-OES	N	MTD/FOD/CHM-09
88	**14256**	0	**3.15**	**17.30**	3	Dry Ashing	HNO_3 conc 10 mL	AAS GBC Flame	Na 587.60	N	In house method (AAS)
89	**6549**	98	**-2.48**	**-13.36**	2	Dry Ashing	1.5% HNO_3	AAS Agilent	Various	N	AOAC 985.35
90	**10014**	-	0.05	-	1	Ultrawave	-	ICP-OES	Na 589.592	-	-
91	**10700**	14	0.55	**3.04**	-	-	-	-	-	-	-
92	**1030**	-	**-6.52**	-	1	Ashing	HNO_3	ICP-OES			
93	**11200**	-	0.92	-	0.05	Charring, Dry ashing	Hotplate, Furnace	Flame Photometer,	N/A	N/A	AOAC 985.35
94	**12906**	-	**2.17**	-	1.5	Dry ashing (Fe: Wet		ICP-OES / Perkin Elmer	Na 589.0	Y	AOAC (2012) 984.27
95	**9830**	1700	-0.08	-0.13	-	-	-	-	-	-	-

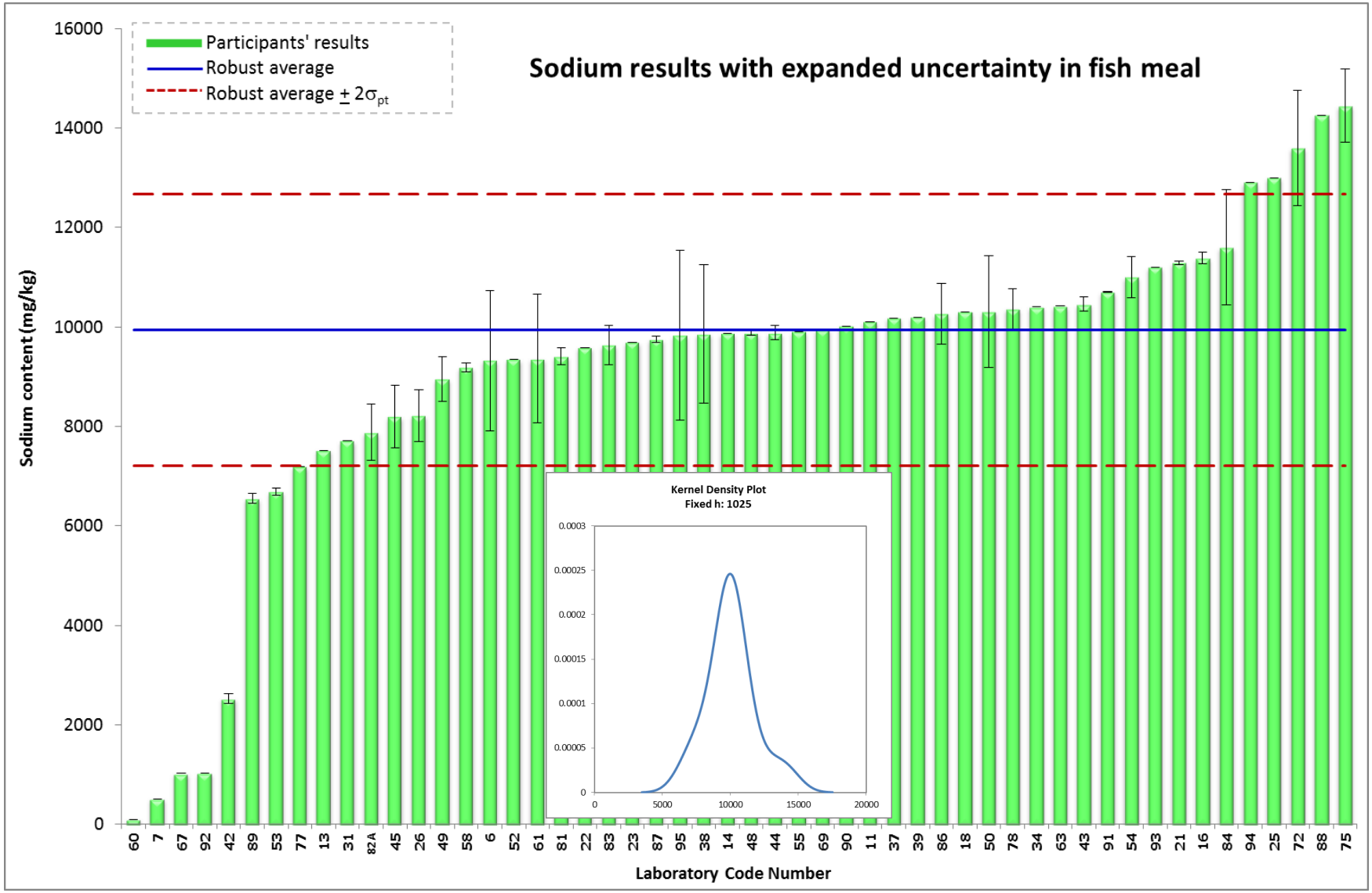

Figure 79. Distribution of **sodium** results (ascending order) in fish meal with expanded uncertainty

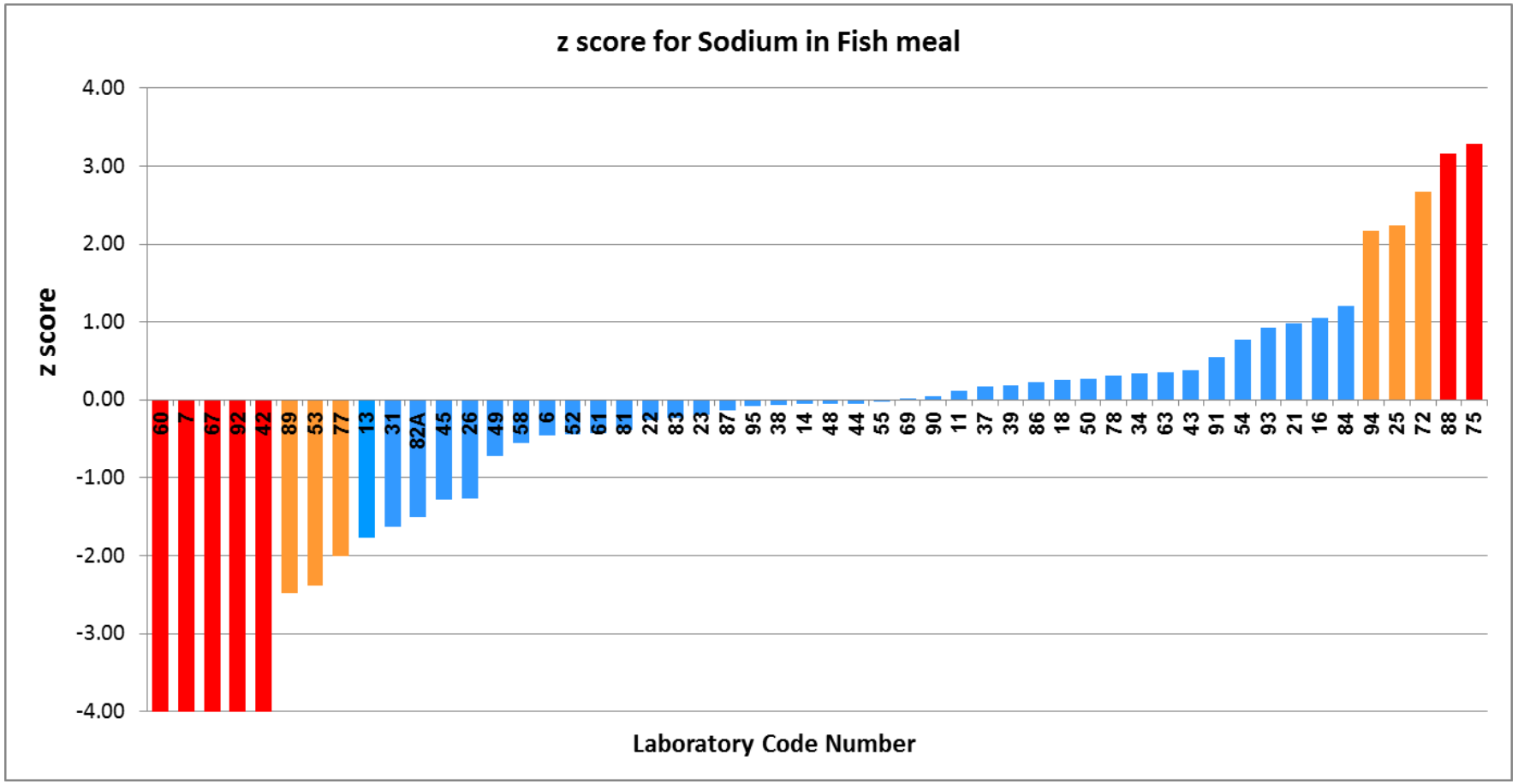

Figure 80. Plot of ordered *z* scores for **sodium** results in fish meal

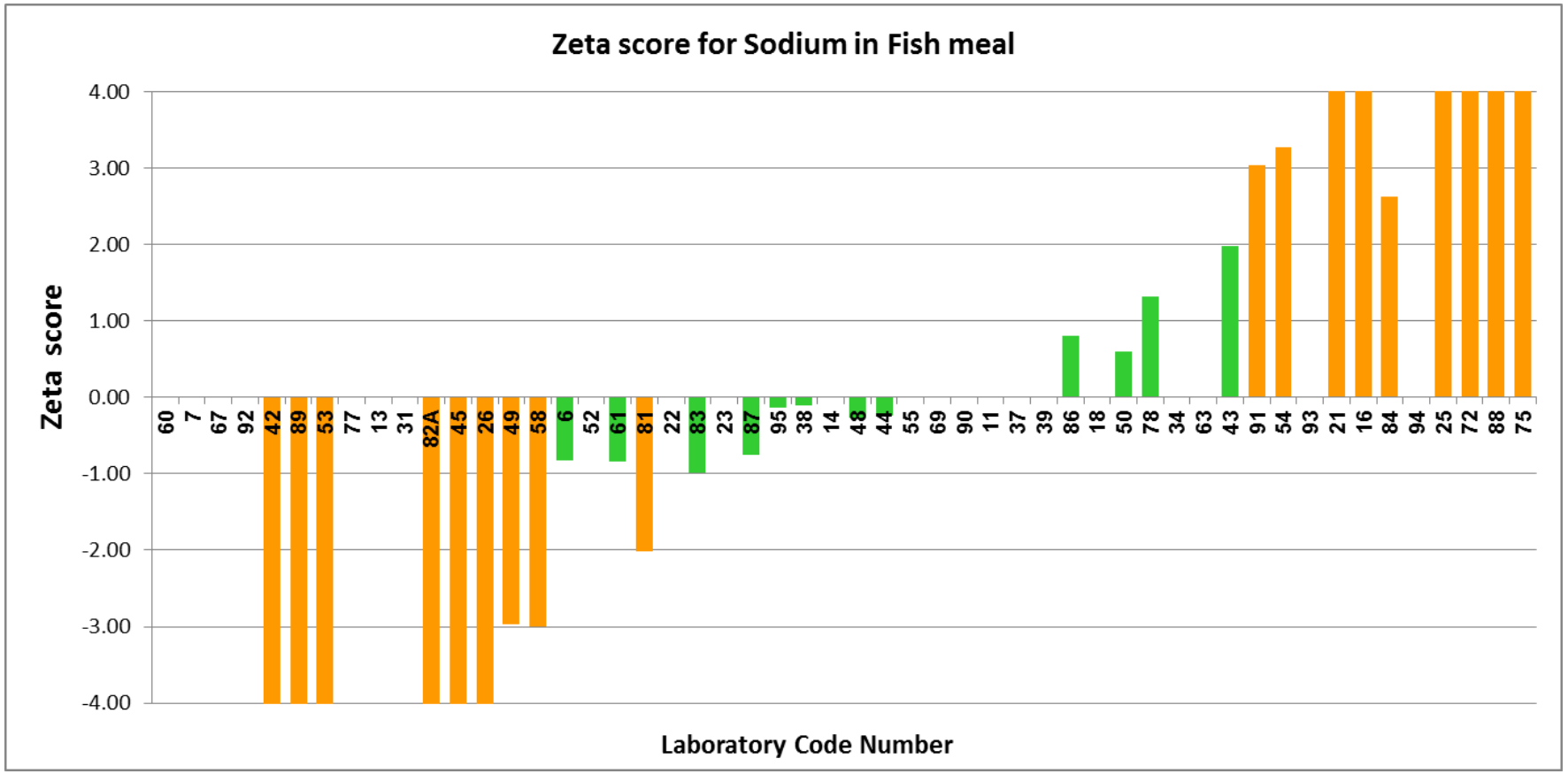

Figure 81. Plot of *Zeta* score for **sodium** in fish meal, following the ordered z scores in the above Figure 80.

Table 28. Evaluation of laboratory performance **potassium** analysis (mg/kg, as received) in fish meal

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) ± robust SD (s*) = 7276 ± 724 mg/kg (CV 9.9%, n= 45) with u_{xpt} = 135 mg/kg*											
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00							
6	**7906**	863	0.87	1.39	1.0000	Acid	$HCl:HNO_3:H_2O$	AAS	-	Y	AOAC (2016), 20th Ed, 928.08, 985.35
11	**7153**	-	-0.17	-	2.0000	Dry Ashing	$HCL:H_2O$	AAS	-	Y	AOAC (2016), 975.03, 985.35
13	**3410**	-	**-5.34**	-	0.5	Microwave	HNO_3 10 mL + HCl 2 mL	Analytikal Jena ContrAA 800 D	K 766	N	Internal Method
14	**6827**	-	-0.62	-	0.1 - 0.2	Ashing	Conc HNO_3, Conc $HClO_4$	ICP Horiba Jobin Yvon	-	Y	AOAC 975.03, 984.27
16	**7965**	80	0.95	**4.90**	0.5	Hot plate	HNO_3+H_2O_2	ICP-OES Optima 7000 DV Perkin	-	N	In-house Method
18	**7490**	-	0.30	-	2.0	Dry Ashing	HNO_3	AAS, Varian	-	N	AOAC 968.08
21	**6271**	16	-1.39	**-7.43**	0.1	Microwave	180°C	Mar Xpress (CEM)	-	Y	AOAC 2011.14 (2016)
23	**7240**	-	-0.05	-	1.00	Dry Ashing	-	ICP-OES	-	-	AOAC 985.01
25	**7540**	0	0.37	1.96	5.0208 / 5.0213	HNO_3-HCl	Water	ICP-OES	-	-	USEPA Method 3050B
26	**6340**	394	-1.29	**-3.92**	4.0	Dry ashing	Water & HCl (1+1)	AAS Shimadzu AA-7000	K 766.5	N	AOAC No. 975.03
31	**5594**	-	**-2.32**	-	5	Dry Ashing	-	AAS, Agilent	-	N	AOAC 985.35
37	**7326**	-	0.07	-	1	Wet Digestion	Nitric + perchloric	ICP-OES (Perkin Elmer Optima	-	N	AOAC (2016) 984.27
38	**7140**	251	-0.19	-0.74	1.000	Dry Ashing	1N HNO_3 (0.1M HNO3	Flame AAS, Shimadzu	-	N	AOAC 985.35, 19th Ed 2012
39	**7340**	-	0.09	-	0.5	Microwave		AAS	K 766.5	Y	AOAC 985.35
42	**9230**	662	**2.70**	**5.47**	5	Dry Ashing	HNO_3-HCl	Flame AAS, Agilent 280 FS	-	N	AOAC 985.35.2005
43	**7304**	891	0.04	0.06	0.5	Microwave	HNO_3	ICP-OES	-	N	AOAC
44	**7131**	356	-0.20	-0.65	1.0000	Dry Ashing	-	AAS, Thermoscientific	K 766.5	N	AOAC 19th Ed

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) ± robust SD (s*) = 7276 ± 724 mg/kg (CV 9.9%, n= 45) with u_{xpt} = 135 mg/kg*											
45	**7108**	256	-0.23	-0.90	4	Dry Ashing	HCl+HNO_3+DI (2+2+70 mL)	AAS (Flame, Varian)	-	N	AOAC 968.08
48	**8230**	123	1.32	**6.44**	5	Dry Digestion	-	AA800 Perkin Elmer	-	N	MU-03/21 (AAS)
49	**6850**	343	-0.59	-1.95	1, 2	Dry Ashing	Conc Nitric acid	AAS / AA-7000 Shimadzu	-	N	AOAC 20th Ed 2016
52	**6827**	-	-0.62	-	1.0 (Ca, Fe)	Dry Ashing (Ca, Fe)	1 N HNO_3 (Ca, Fe)	Flame AAS (Shimadzu	K 766.5	N	Modified AOAC 985.35 (Ca, Fe),
53	**7259**	436	-0.02	-0.06	0.3	Microwave	4 mL HNO_3, 1 mL HCl, 1 mL	ICPMS Thermo			In house method
54	**7360**	600	0.12	0.26	1	Dry Ashing	HNO_3	ICP / Shimadzu	-	N	AOAC 984.27
55	**7595**	-	0.44	-	1.5	Wet digestion	-	ICP-OES	-	Y	AOAC (2012) 984.27
58	**7845**	296	0.79	**2.84**	3.0	Dry Ash	HCl	ICP-OES	-	-	-
60	**23100**	-	**21.86**	-	-	-	-	-	-		AOAC (2012) 968.08 (Ca, Mg)
61	**6680**	2270	-0.82	-0.52	1	Acid block digestion	HNO_3	Varian AA240 FS Fast Sequential	-	N	A6407-26 AAS (A6417 Spectro
63	**7548**	-	0.38	-							
67	**6750**	-	-0.73	-	2.0xxx	Dry Ash	Wet chemical	AAS, Perkin Elmer	-	N	AOAC 968.08
69	**8010**	-	1.01	-	-	-	-	-	-	-	-
72	**91600**	7790	**116.49**	**21.64**	3	Ashing	HNO_3	AAS / Analytik Jena	K 766.5	N	AOAC 985.35
75	**8333**	161	1.46	**6.73**	1	Wet digestion (hot block)	HNO_3 + H_2O_2	ICP-OES Agilent 5100	-	N	In House Method ICP-OES
77	**6154**	-	-1.55	-	-	-	-	-	-	-	-
78	**8025**	148	1.04	**4.87**	0.5	Mircowave Digestion	Acid Digestion	Berghof Speedwave 4	K 766.490		MP-AES
81	**6760**	222	-0.71	**-2.95**	1.0024	Dry Ashing (Ca, Fe)	1 N HNO_3 (Ca, Fe)	Shimadzu AAS AA 6300	K 766.5	N	AOAC 999.10 Mod (Na, K), 985.35 Mod
82A	**7600**	600	0.45	0.99	0.250	none	none	HPGe detector, Canberra	-		Neutron Activation Analysis (NAA)
83	**7253**	138	-0.03	-0.15	0.3	Microwave Digestion	-	Microwave digester Mars	-	Y	Application Note, Perkin Elmer

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) ± robust SD (s*) = 7276 ± 724 mg/kg (CV 9.9%, n= 45) with u_{xpt} = 135 mg/kg*											
84	**7680**	768	0.56	0.99	0.5	Microwave Digestion	HNO_3 / H_2O_2	ICP-OES, ICP-MS	K 766.490	N	AOAC 999.10:2005
86	**7742**	452	0.64	1.77	1.0000	Wet Digest	-	ICP-OES	-	Y	AOAC (2012) 984.27
87	**8574**	25	1.79	**9.58**	2.5	Dry Ashing	HNO_3	Furnace Thermolyne	ICP-OES	N	MTD/FOD/CHM-09
88	**6153**	0	-1.55	**-8.32**	3	Dry Ashing	HNO_3 conc 10 mL	AAS GBC Flame	-	N	In house method (AAS)
89	**6403**	96	-1.20	**-6.09**	2	Dry Ashing	1.5% HNO_3	AAS Agilent	Various	N	AOAC 985.35
90	**6753**	-	-0.72	-	1	Ultrawave		ICP-OES	-	-	-
91	**7530**	-	0.35	-						-	-
92	**833**	-	**-8.90**	-	1	Ashing	HNO_3	ICP-OES	-	-	-
93	**7200**	-	-0.10	-	0.05	Charring, Dry ashing	Hotplate, Furnace	Flame Photometer,	-	N/A	AOAC 985.35
94	**12408**	-	**7.09**	-	1.5	Dry ashing		ICP-OES / Perkin Elmer	-	Y	AOAC (2012) 984.27
95	**7240**	830	-0.05	-0.08	-	-	-	-	-	-	-

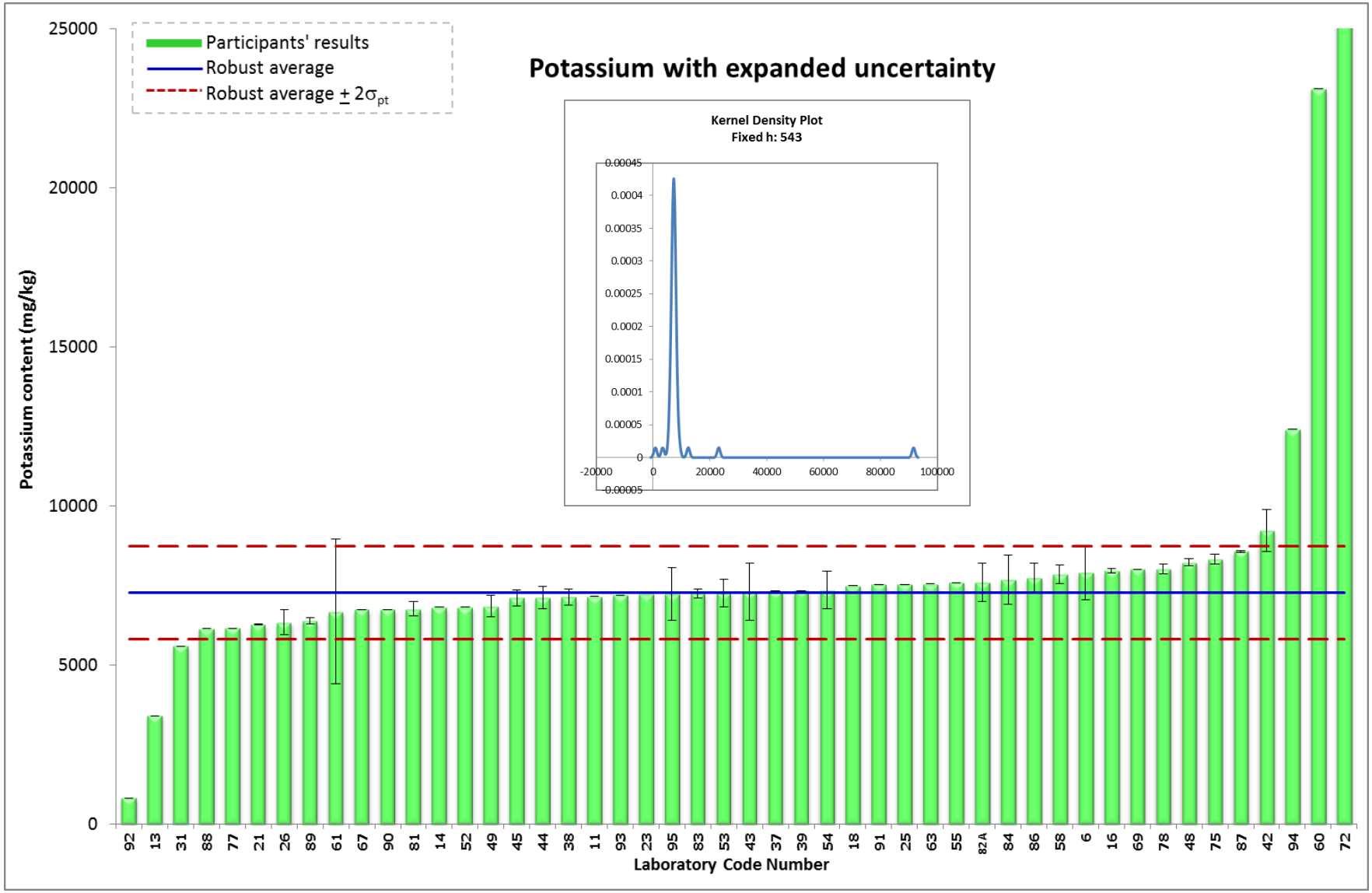

Figure 82. Distribution of **potassium** results (ascending order) in fish meal with expanded uncertainty

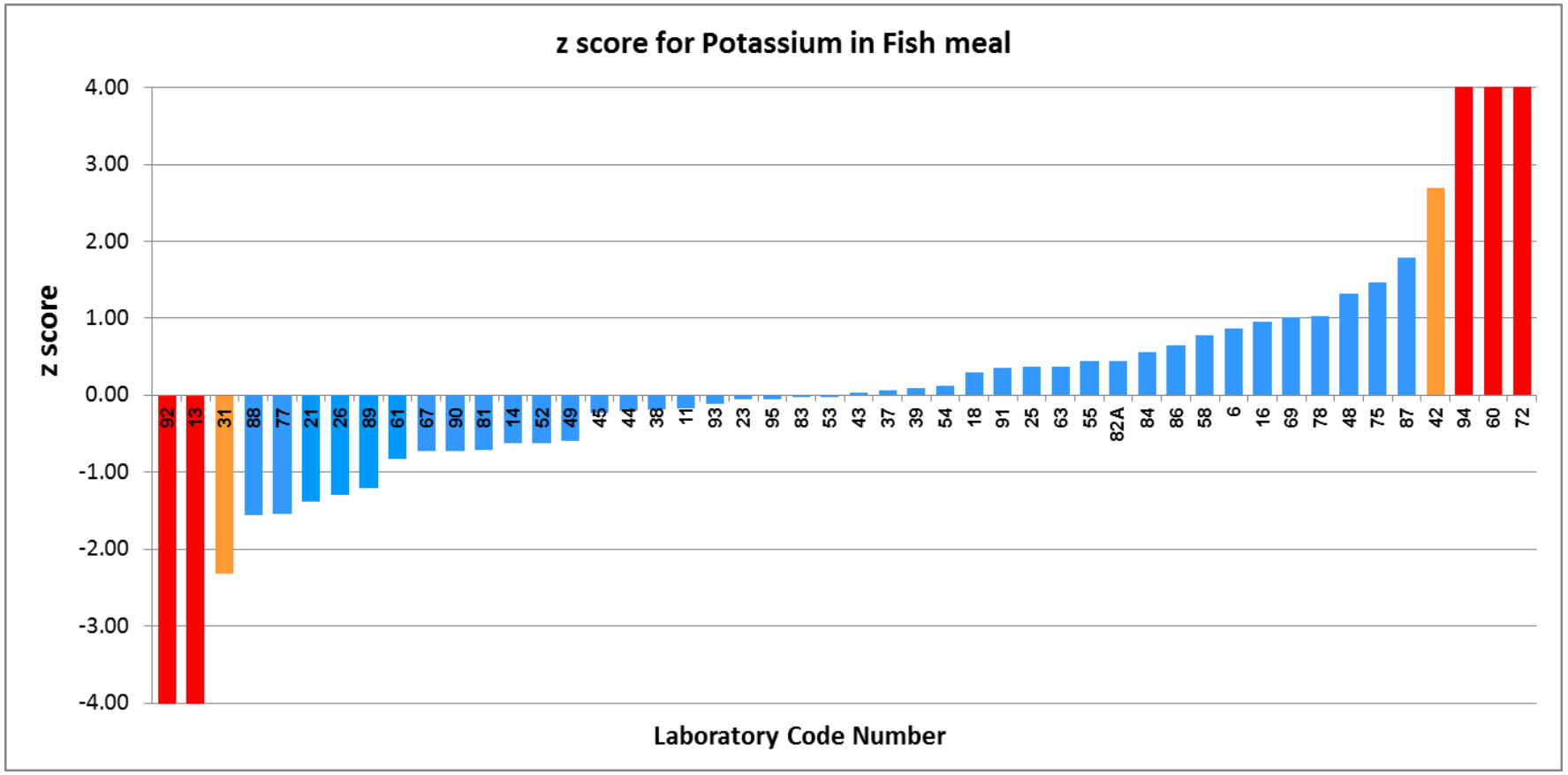

Figure 83. Plot of ordered *z* scores for **potassium** results in fish meal

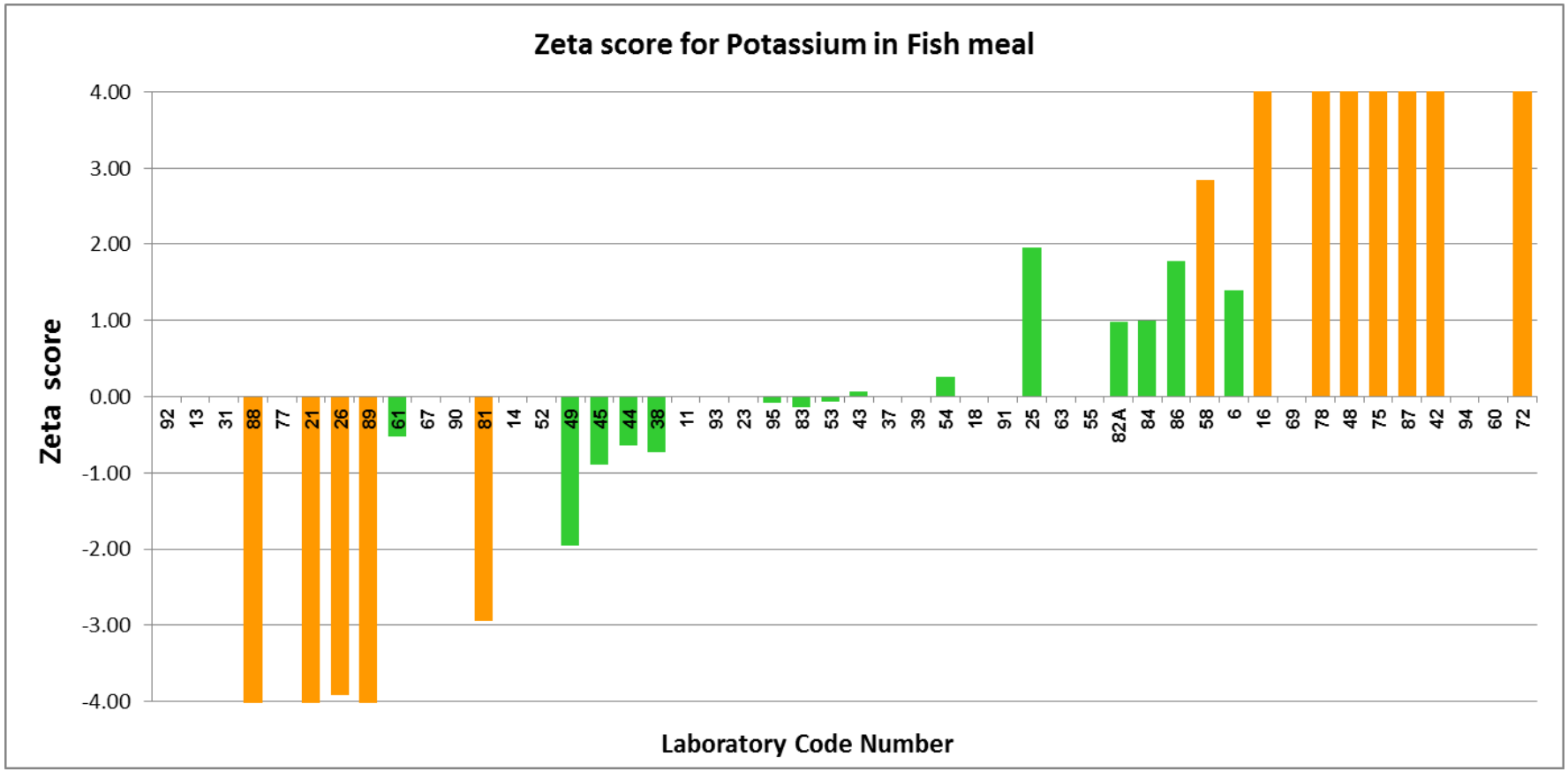

Figure 84. Plot of *Zeta* score for **potassium** in fish meal, following the ordered z scores in the above Figure 83.

Table 29. Evaluation of laboratory performance **iron** analysis (mg/kg, as received) in fish meal

Lab Number	Iron (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x*) ± SDp from Horwitz's equation = 941.9 ± 107.5 mg/kg (CV 11.4%, n= 50) with u_{xpt} 15.2 mg/kg											
Acceptance criteria			**|z score| ≤ 2.00**	|ζ score| ≤ 2.00							
6	**1088.0**	-	1.36	-	1.0000	Acid	$HCl:HNO_3:H_2O$	AAS	-	Y	AOAC (2016), 20th Ed, 928.08, 985.35 (50.1.14)
11	**1059.9**	-	1.10	-	2.0000	Dry Ashing	$HCL:H_2O$	AAS	Fe 248.3	Y	AOAC (2016), 975.03, 985.35
13	**670.0**	-	**-2.53**	-	0.5	Microwave	HNO_3 10 mL + HCl 2 mL	Analytikal Jena ContrAA 800 D	-	N	Internal Method
14	**558.6**	-	**-3.57**	-	0.1 - 0.2	Ashing	Conc HNO_3, Conc $HClO_4$	ICP Horiba Jobin Yvon	Fe 259.94	Y	AOAC 975.03, 984.27
16	**1012.0**	93.0	0.65	1.43	0.5	Hot plate	HNO_3+H_2O_2	ICP-OES Optima 7000 DV Perkin	Fe 238.204	N	In-house Method
18	**1320.0**	-	**3.52**	-	2.0	Dry Ashing	HNO_3	AAS, Varian	Various	N	AOAC 968.08
21	**842.0**	2.2	-0.93	**-6.55**	0.1	Microwave	180°C	Mar Xpress (CEM)	-	Y	AOAC 2011.14 (2016)
22	**1141.0**	-	1.85	-	0.2 to 0.3	Microwave	HNO_3	ICP-MS Perkin Elmer	-	-	AOAC 2015.06
23	**846.0**	-	-0.89	-	1.00	Dry Ashing	-	ICP-OES	238.00	-	AOAC 985.01
25	**992.0**	0.1	0.47	**3.30**	5.0208 / 5.0213	HNO_3-HCl	Water	ICP-OES	Fe 238.204	-	USEPA Method 3050B
26	**1020.0**	76.1	0.73	1.91	4.0	Dry ashing	Water & HCl (1+1)	AAS Shimadzu AA-7000	Fe 248.3	N	AOAC No. 975.03
31	**22.7**	-	**-8.55**	-	5	Dry Ashing	-	AAS, Agilent	-	N	AOAC 985.35
34	**446.0**	-	**-4.61**	-	2.000	Dry Ashing	HNO_3 and HCl	AAS Flame	Fe 372	92% (Na)	AOAC
37	**1057.0**	-	1.07	-	1	Wet Digestion	Nitric + perchloric	ICP-OES (Perkin Elmer Optima	Fe 238.204	N	AOAC (2016) 984.27
38	**1150.0**	93.0	1.94	**4.25**	1.000	Dry Ashing	1N HNO_3 (0.1M HNO3	Flame AAS, Shimadzu	Fe 248.30	N	AOAC 985.35, 19th Ed 2012
39	**1120.0**	-	1.66	-	0.5	Microwave		AAS	Fe 248.3	Y	AOAC 985.35
42	**1060.0**	76.7	1.10	**2.86**	5	Dry Ashing	HNO_3-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	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x*) ± SDp from Horwitz's equation = 941.9 ± 107.5 mg/kg (CV 11.4%, n= 50) with u_{xpt} 15.2 mg/kg											
43	**797.5**	124.0	-1.34	**-2.26**	0.5	Microwave	HNO_3	ICP-OES	-	N	AOAC
44	**837.0**	94.0	-0.98	**-2.12**	1.0000	Dry Ashing	-	AAS, Thermoscientific	Fe 248.3	N	AOAC 19th Ed
45	**970.5**	33.6	0.27	1.26	4	Dry Ashing	$HCl+HNO_3$+DI (2+2+70 mL)	AAS (Flame, Varian)	Fe 248.3	N	AOAC 968.08
48	**1120.7**	27.8	1.66	**8.68**	5	Dry Digestion	-	AA800 Perkin Elmer	Fe 248.3	N	MU-03/21 (AAS)
49	**1030.0**	52.0	0.82	**2.93**	1, 2	Dry Ashing	Conc Nitric acid	AAS / AA-7000 Shimadzu	Fe 248.3	N	AOAC 20th Ed 2016
50	**700.0**	86.6	**-2.25**	**-5.27**	2.0000	Wet	Acid	Flame AAS (Varian)	248.3	N	AOAC 985.35
52	**1175.2**	-	**2.17**	-	1.0 (Ca, Fe)	Dry Ashing (Ca, Fe)	1 N HNO_3 (Ca, Fe)	Flame AAS (Shimadzu	Fe 248.3	N	Modified AOAC 985.35 (Ca, Fe), 999.10 (K, Na)
53	**860.0**	92.0	-0.76	-1.69	0.3	Microwave	4 mL HNO_3, 1 mL HCl, 1 mL	ICPMS Thermo	-		In house method
54	**874.0**	116.0	-0.63	-1.13	1	Dry Ashing	HNO_3	ICP / Shimadzu	Fe 259.940	N	AOAC 984.27
55	**855.6**	-	-0.80	-	1.5	Wet digestion	-	ICP-OES	Fe 259.940	Y	AOAC (2012) 984.27
58	**1145.1**	36.2	1.89	**8.60**	3.0	Dry Ash	HCl	ICP-OES	-	-	-
60	**1100.0**	-	1.47	-	-	-	-	-	-		AOAC (2012) 968.08 (Ca, Mg)
61	**1030.0**	122.0	0.82	1.40	1	Acid block digestion	HNO_3	Varian AA240 FS Fast	Fe 248.3	N	A6407-26 AAS (A6417 Spectro Method for P)
63	**971.0**	-	0.27	-	-	-	-	-	-	-	-
64	**1063.0**	3.0	1.13	**7.93**	0.5048	Dry Ashing	1 N HNO3	Shimadzu AA6300	Fe 248.3	N	Modified AOAC 985.35
67	**1160.0**	-	**2.03**	-	2.0xxx	Dry Ash	Wet chemical	AAS, Perkin Elmer	Fe 248.33	N	AOAC 968.08
69	**403.0**	-	**-5.01**	-	-	-	-	-	-	-	-
72	**238.0**	21.1	**-6.55**	**-38.04**	3	Ashing	HNO_3	AAS / Analytik Jena	Fe 589.0	N	AOAC 985.35
73A	**887.5**	151.2	-0.51	-0.71	1	Dry ashing	Hot plate	AAS (280FS AA, Agilent	Fe 248.3	N	FTC-46.01 (refers to AOAC 968.08, 965.09)

Lab Number	Iron (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x*) ± SDp from Horwitz's equation = 941.9 ± 107.5 mg/kg (CV 11.4%, n= 50) with u_{xpt} 15.2 mg/kg											
75	**1005.9**	84.9	0.60	1.42	1	Wet digestion	HNO_3 + H_2O_2	ICP-OES Agilent 5100	Fe 238.204	N	In House Method ICP-OES
77	**500.5**	-	**-4.11**	-	-	-	-	-	-	-	-
78	**1085.0**	19.0	1.33	**7.98**	0.5	Mircowave Digestion	Acid Digestion	Berghof Speedwave 4	Fe 371.993	-	MP-AES
81	**1160.0**	129.0	**2.03**	**3.29**	1.0024	Dry Ashing (Ca, Fe)	1 N HNO_3 (Ca, Fe)	Shimadzu AAS AA 6300	Fe 248.3	N	AOAC 999.10 Mod (Na, K), 985.35 Mod (Ca, Fe)
82A	**828.0**	54.5	-1.06	**-3.65**	0.250	none	none	HPGe detector, Canberra	-	-	Neutron Activation Analysis (NAA)
83	**931.4**	29.9	-0.10	-0.49	0.3	Microwave Digestion	-	Microwave digester Mars	-	Y	Application Note, Perkin Elmer
84	**943.0**	94.0	0.01	0.02	0.5	Microwave Digestion	HNO_3 / H_2O_2	ICP-OES, ICP-MS	-	N	AOAC 999.10:2005
86	**984.0**	72.8	0.39	1.07	1.0000	Wet Digest	-	ICP-OES	Fe 259.9	Y	AOAC (2012) 984.27
87	**808.5**	2.7	-1.24	**-8.74**	2.5	Dry Ashing	HNO_3	Furnace Thermolyne	ICP-OES	N	MTD/FOD/CHM-09
88	**362.6**	2.5	**-5.39**	**-37.97**	3	Dry Ashing	HNO_3 conc 10 mL	AAS GBC Flame	-	N	In house method (AAS)
89	**27.0**	0.4	**-8.51**	**-60.17**	2	Dry Ashing	1.5% HNO_3	AAS Agilent	Various	N	AOAC 985.35
90	**866.0**	-	-0.71	-	1	Ultrawave		ICP-OES	Fe 238.204	-	-
91	**991.0**	-	0.46	-	-	-	-	-	-	-	-
92	**549.0**	-	**-3.65**	-	1	Ashing	HNO_3	ICP-OES	-	-	-
94	**1226.0**	-	**2.64**	-	1.5	Dry ashing		ICP-OES / Perkin Elmer	Fe 259.9	Y	AOAC (2012) 984.27
95	**914.5**	37.0	-0.25	-1.14	-	-	-	-	-	-	-

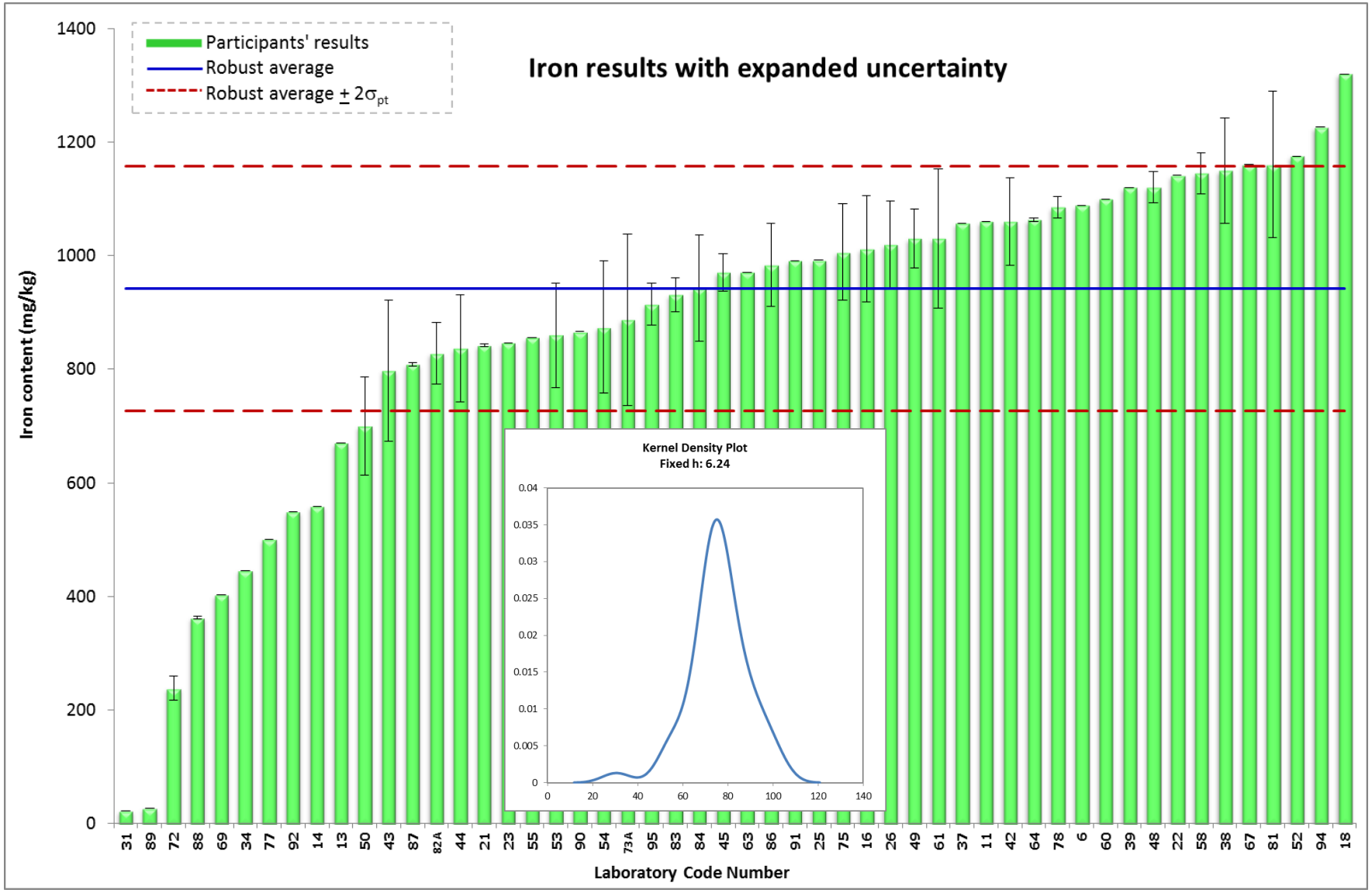

Figure 85. Distribution of **iron** results (ascending order) in fish meal with expanded uncertainty

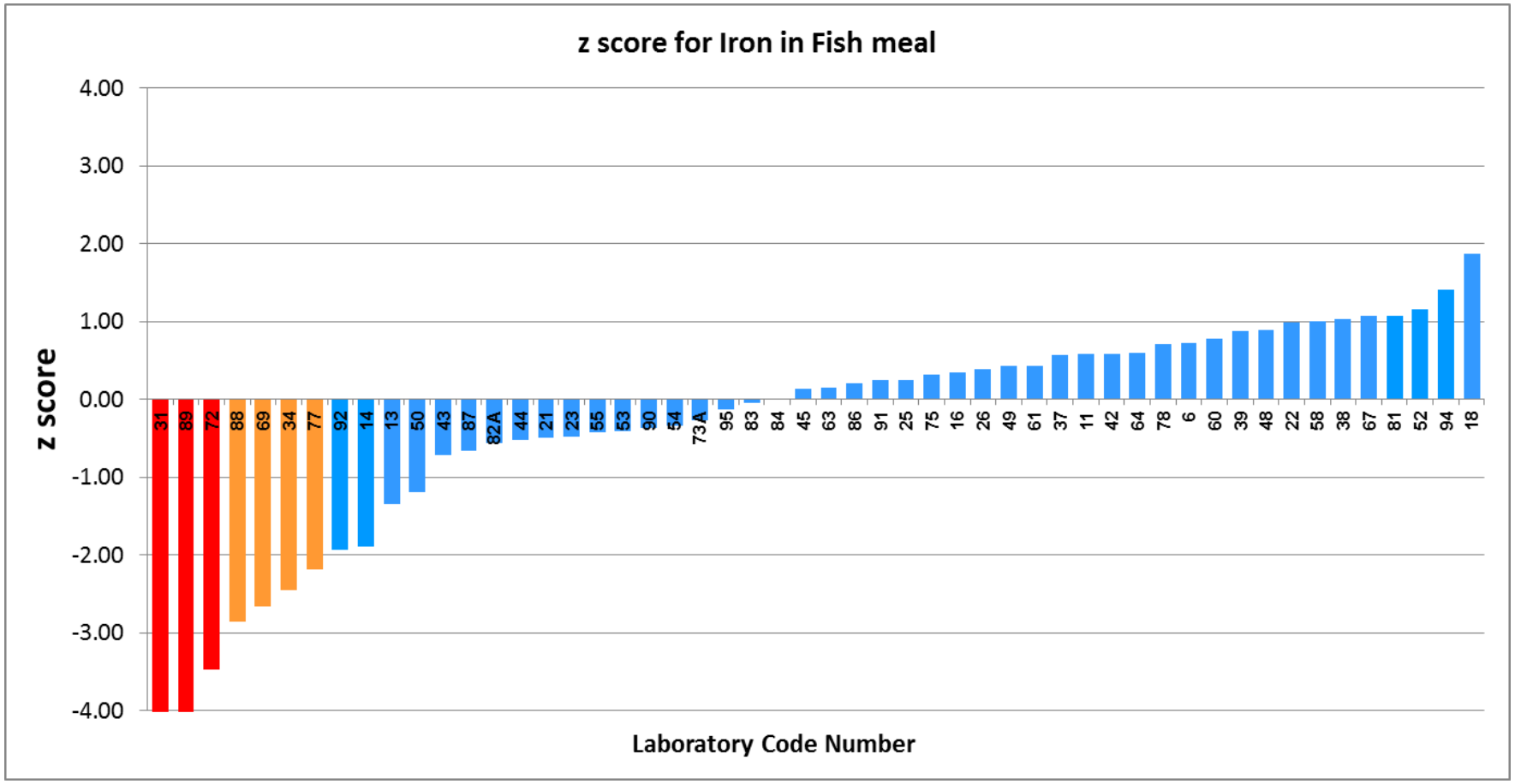

Figure 86. Plot of ordered *z* scores for **iron** results in fish meal

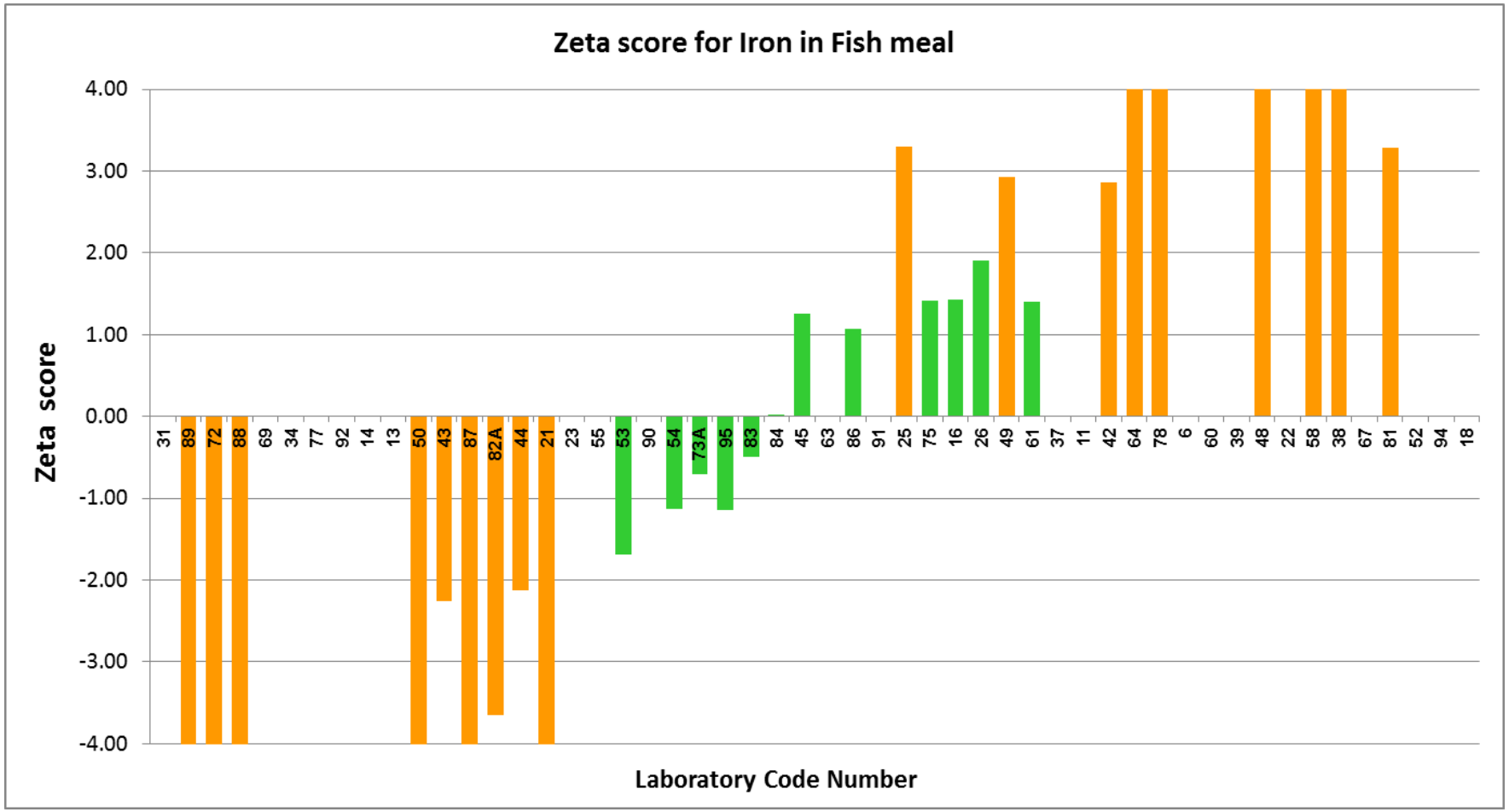

Figure 87. Plot of *Zeta score* for **iron** in fish meal, following the ordered z scores in the above Figure 86.

Table 30. Evaluation of laboratory performance **zinc** analysis (mg/kg, as received) in fish meal

Lab Number	Zinc (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wave-length	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x*) ± robust SD (s*) = 86.69 ± 12.57 mg/kg (CV 14.5%, n= 41) with u_{xpt} 2.45 mg/kg											
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00							
6	**97.12**	-	0.83	-	1.00	Acid	$HCl:HNO_3:H_2O$	AAS	-	Y	AOAC (2016), 20th Ed, 928.08, 985.35 (50.1.14)
11	**969.01**	-	**70.19**	-	2.00	Dry Ashing	$HCL:H_2O$	AAS	Zn 213.9	Y	AOAC (2016), 975.03, 985.35
13	**21.40**	-	**-5.19**	-	0.50	Microwave	HNO_3 10 mL + HCl 2 mL	ICP-MS Thermo Scientific (iCAP	M/z Zn 66	N	Internal Method
14	**76.12**	-	-0.84	-	0.1 - 0.2	Ashing	Conc HNO_3, Conc $HClO_4$	ICP Horiba Jobin Yvon	-	Y	AOAC 975.03, 984.27
16	**125.00**	6.00	**3.05**	**9.89**	0.50	Hot plate	HNO_3+H_2O_2	ICP-MS 7700X Agilent	-	N	In-house Method
18	**66.10**	-	-1.64	-	2.00	Dry Ashing	HNO_3	ICP-OES Agilent	-	N	AOAC 968.08
21	**79.60**	-	-0.56	-	0.10	Microwave	180°C	Mar Xpress (CEM)	Zn 213.228	Y	AOAC 2011.14 (2016)
22	**91.70**	-	0.40	-	0.2 to 0.3	Microwave	HNO_3	ICP-MS Perkin Elmer	-	-	AOAC 2015.06
23	**72.10**	-	-1.16	-	1.00	Dry Ashing	-	ICP-OES	213.00	-	AOAC 985.01
25	**78.70**	0.07	-0.64	**-3.26**	5.0208 / 5.0213	HNO_3-HCl	Water	ICP-OES	Zn 202.548	-	USEPA Method 3050B
26	**73.50**	6.37	-1.05	**-3.28**	4.00	Dry ashing	Water & HCl (1+1)	AAS Shimadzu AA-7000	-	N	AOAC No. 975.03
27	**28.56**	4.52	**-4.62**	**-17.44**	-	-	-	ICP-MS 7700x Agilent	-	-	-
31	**21.13**	1.19	**-5.22**	**-26.01**	5.00	Dry Ashing	-	AAS	-	N	AOAC 985.35
37	**87.37**	-	0.05	-	1.00	Wet Digestion	Nitric + perchloric	ICP-OES (Perkin Elmer	Zn 206.200	N	AOAC (2016) 984.27
38	**67.10**	3.17	-1.56	**-6.71**	1.00	Dry Ashing	1N HNO_3 (0.1M HNO3 for Fe)	Flame AAS, Shimadzu	-	N	AOAC 985.35, 19th Ed 2012
39	**79.70**	-	-0.56	-	0.50	Microwave	-	AAS	-	Y	AOAC 985.35
42	**83.60**	1.82	-0.25	-1.18	5.00	Dry Ashing	HNO_3-HCl	Cu, Zn: Flame AAS 280 FS	Zn 213.9	N	AOAC 985.35.2005

Lab Number	Zinc (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wave-length	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x*) ± robust SD (s*) = 86.69 ± 12.57 mg/kg (CV 14.5%, n= 41) with u_{xpt} 2.45 mg/kg											
43	**103.90**	5.57	1.37	**4.64**	0.50	Microwave	HNO_3	ICP-OES, ICP-MS	Zn 213.857	N	AOAC
45	**90.69**	3.67	0.32	1.31	4.00	Dry Ashing	HCl+HNO_3+DI (2+2+70 mL) on	AAS (Flame, Varian)	Zn 213.9	N	AOAC 968.08
48	**102.96**	1.19	1.29	**6.45**	5.00	Dry Digestion	-	AA800 Perkin Elmer	Zn 213.9	N	MU-03/21 (AAS)
49	**104.00**	5.00	1.38	**4.95**	1, 2	Dry Ashing	Conc Nitric acid	AAS / AA-7000 Shimadzu for	Zn 213.9	N	AOAC 20th Ed 2016
52	**100.75**	-	1.12	-	1.0 (Ca, Fe)	Dry Ashing (Ca, Fe)	1 N HNO_3 (Ca, Fe)	Flame AAS - Shimadzu	Zn 213.9	N	Modified AOAC 985.35 (Ca, Fe), 999.10 (K, Na)
53	**75.90**	9.70	-0.86	-1.99	0.30	Microwave	4 mL HNO_3, 1 mL HCl, 1 mL	ICPMS Thermo	-	-	In house method
54	**75.00**	2.90	-0.93	**-4.11**	1.00	Dry Ashing	HNO_3	ICP / Shimadzu	Zn 213.856	N	AOAC 984.27
55	**83.23**	-	-0.28	-	1.50	Wet digestion	-	ICP-OES	-	Y	AOAC (2012) 984.27
58	**98.60**	1.69	0.95	**4.60**	3.00	Dry Ash	HCl	ICP-OES (AAS-	-	-	-
60	**800.00**	-	**56.75**	-	-	-	-	-	-	-	AOAC (2012) 968.08 (Ca, Mg)
61	**99.30**	27.20	1.00	0.91	1.00	Acid block digestion	HNO_3	Varian AA240 FS Fast	Zn 213.9	N	A6407-26 AAS (A6417 Spectro Method for P)
63	**88.10**	-	0.11	-	-	-	-	-	-	-	-
64	**95.50**	1.00	0.70	**3.52**	0.50	Dry Ashing	1 N HNO3	Shimadzu AA6300	Zn 213.9	N	Modified AOAC 985.35
67	**98.60**	-	0.95	-	2.0xxx	Dry Ash	Wet chemical	AAS, Perkin Elmer	Zn 213.86	N	AOAC 968.08
69	**89.50**	-	0.22	-	-	-	-	-	-	-	-
72	**70.70**	6.12	-1.27	**-4.08**	3.00	Ashing	HNO_3	ICP-OES, JY Ultima	Zn 213.9	N	AOAC 985.35
73A	**88.67**	15.93	0.16	0.24	1.00	Dry ashing	Hot plate	AAS (280FS AA, Agilent	213.90	N	FTC-46.01 (refers to AOAC 968.08, 965.09)
75	**102.41**	7.27	1.25	**3.58**	1.00	Wet digestion	HNO_3 + H_2O_2	ICP-OES Agilent 5100,	Zn 213.857	N	In House Method ICP-OES
77	**92.96**	-	0.50	-	-	-	-	-	-	-	-
81	**91.50**	7.70	0.38	1.05	1.00	Dry Ashing (Ca, Fe)	1 N HNO_3 (Ca, Fe)	Zn: Shimadzu AAS AA 6300	Zn 213.9	N	AOAC 999.10 Mod (Na, K), 985.35 Mod (Ca, Fe)

Lab Number	Zinc (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wave-length	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x*) ± robust SD (s*) = 86.69 ± 12.57 mg/kg (CV 14.5%, n= 41) with u_{xpt} 2.45 mg/kg											
82A	**80.90**	4.61	-0.46	-1.72	0.25	none	none	HPGe detector, Canberra	-	-	Neutron Activation Analysis (NAA)
83	**81.22**	0.47	-0.44	**-2.22**	0.30	Microwave Digestion	-	Microwave digester Mars	-	Y	Application Note, Perkin Elmer
84	**91.40**	0.91	0.37	1.89	0.50	Microwave Digestion	HNO_3 / H_2O_2	ICP-MS	-	N	AOAC 999.10:2005
86	**84.00**	4.09	-0.21	-0.84	1.00	Wet Digest	-	ICP-OES (As: ICP-MS)	Zn 206.2	Y	AOAC (2012) 984.27
87	**90.95**	0.27	0.34	1.74	2.50	Dry Ashing	HNO_3	Microwave Digestion,	ICP-OES	N	MTD/FOD/CHM-09
88	**239.59**	0.55	**12.16**	**62.02**	3.00	Dry Ashing	HNO_3 conc 10 mL	AAS GBC Hydride vapour	Zn 213.90	N	In house method (AAS)
90	**86.18**	-	-0.04	-	1.00	Ultrawave		ICP-OES	Zn 213.857	-	-
91	**83.20**	-	-0.28	-	-	-	-			-	-
94	**58.00**	-	**-2.28**	-	1.50	Dry ashing				Y	AOAC (2012) 984.27
95	**75.50**	9.00	-0.89	**-2.18**	-	-	-			-	-

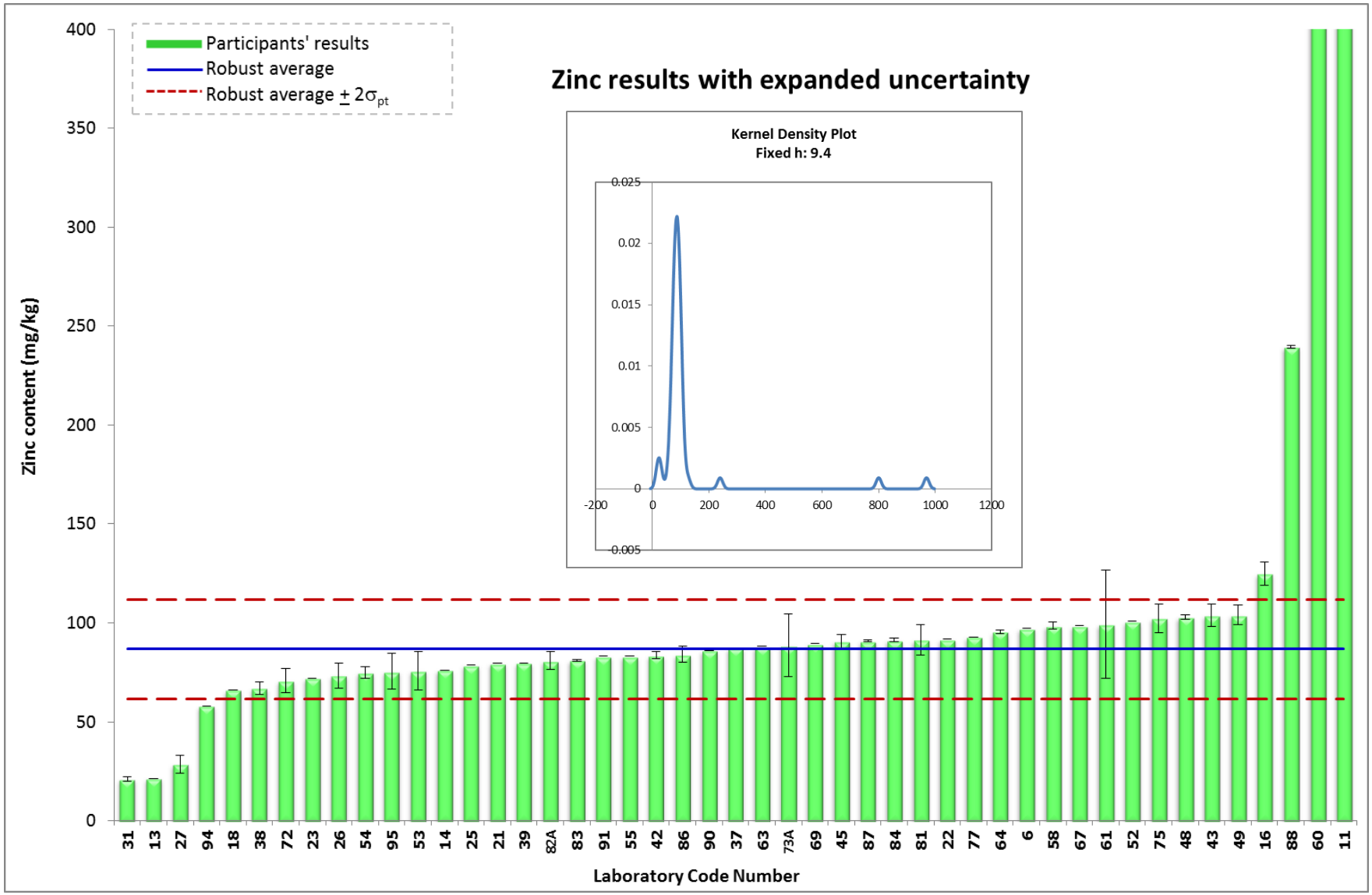

Figure 88. Distribution of **zinc** results (ascending order) in fish meal with expanded uncertainty

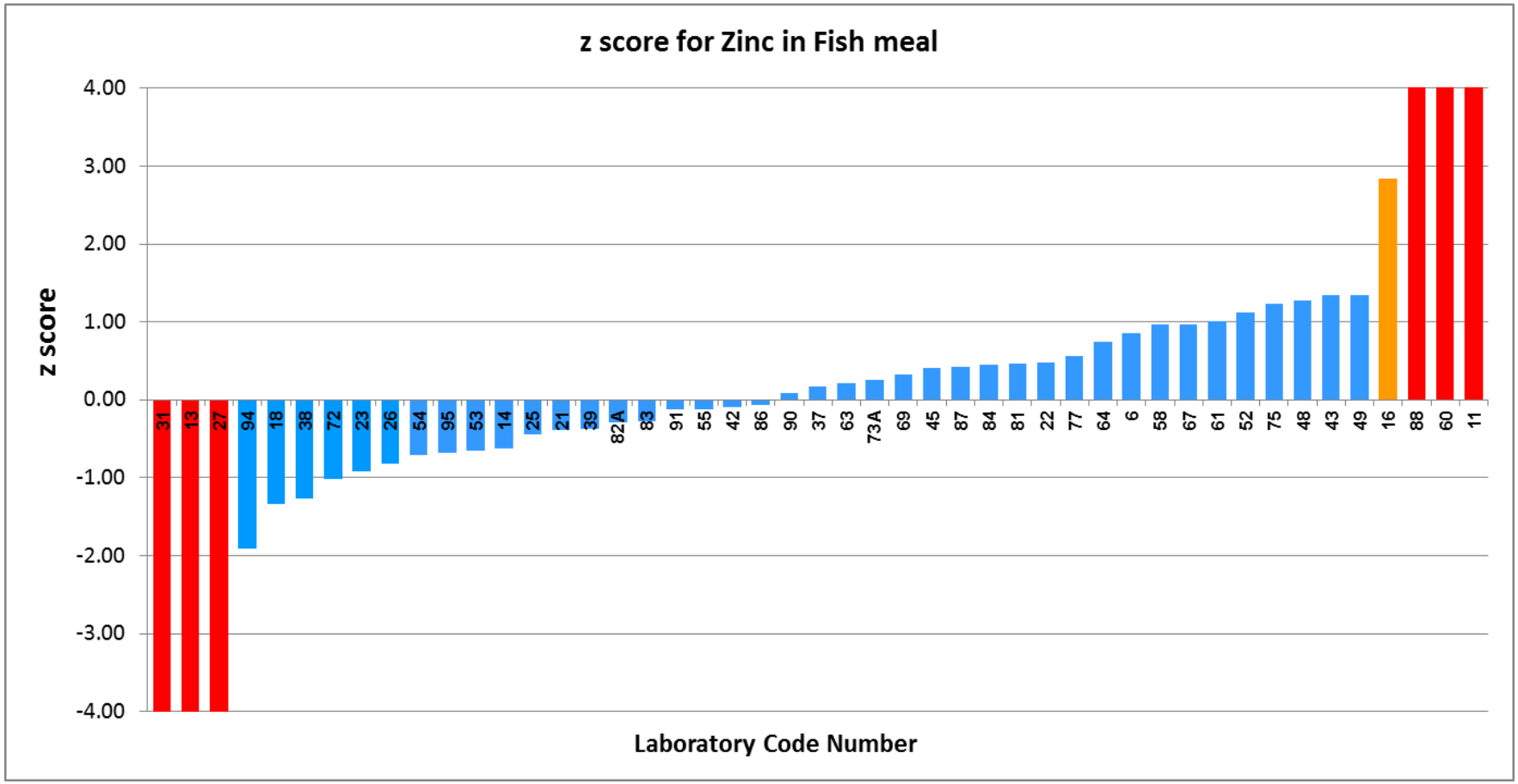

Figure 89. Plot of ordered *z* scores for **zinc** results in fish meal

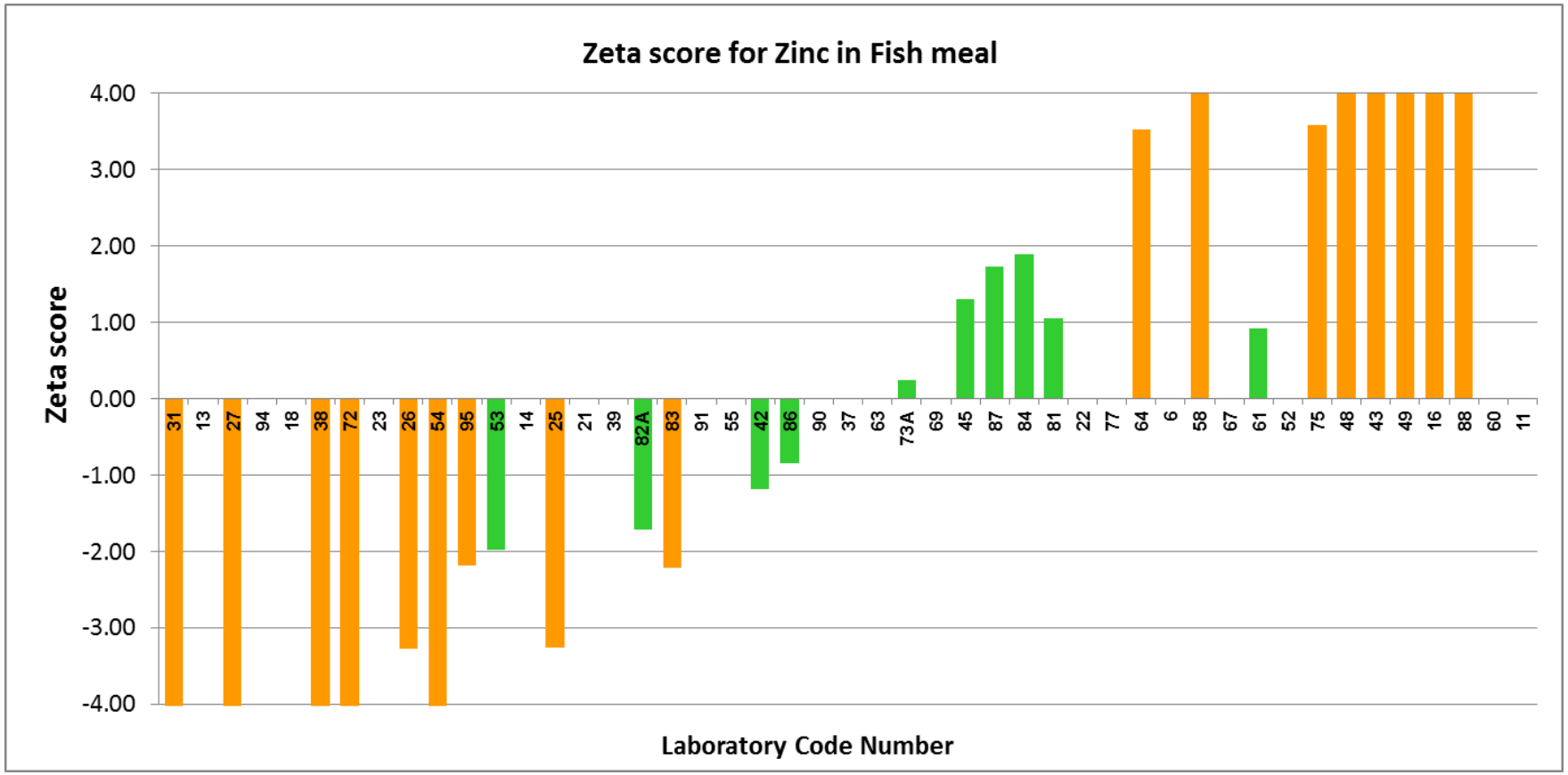

Figure 90. Plot of *Zeta score* for **zinc** in fish meal, following the ordered z scores in the above Figure 89.

Table 31. Evaluation of laboratory performance **copper** analysis (mg/kg, as received) in fish meal

Lab Number	Copper (mg/kg)	MU (mg/kg)	z score	Zeta score	Macro Metals Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wave-length	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x*) ± robust SD (s*) = 3.70 ± 0.97 mg/kg (CV 26.2%, n= 43) with u_{xpt} 0.15 mg/kg											
Acceptance criteria =			|z score| ≤ 2.00	|ζ score| ≤ 2.00							
6	**5.17**	-	1.52	-	1.00	Acid	HCl:HNO_3:H_2O	AAS	-	Y	AOAC (2016), 20th Ed, 928.08, 985.35 (50.1.14)
11	**6.42**	-	**2.80**	-	2.00	Dry Ashing	HCL:H_2O	AAS	Cu 324.8	Y	AOAC (2016), 975.03, 985.35
13	**1.61**	-	**-2.15**	-	0.50	Microwave	HNO_3 10 mL + HCl 2 mL	ICP-MS Thermo Scientific (iCAP RQ)	M/z Cu: 63	N	Internal Method
14	**2.36**	-	-1.38	-	0.1 - 0.2	Ashing	Conc HNO_3, Conc $HClO_4$	ICP Horiba Jobin Yvon	-	Y	AOAC 975.03, 984.27
16	**3.84**	0.25	0.14	0.72	0.50	Hot plate	HNO_3+H_2O_2	ICP-MS 7700X Agilent	-	N	In-house Method
18	**2.47**	-	-1.27	-	2.00	Dry Ashing	HNO_3	ICP-OES Agilent	-	N	AOAC 968.08
21	**0.00**	-	**-3.81**	-	0.10	Microwave	180°C	Mar Xpress (CEM)	Cu 324.754	Y	AOAC 2011.14 (2016)
22	**1.65**	-	**-2.11**	-	0.2 to 0.3	Microwave	HNO_3	ICP-MS Perkin Elmer	-	-	AOAC 2015.06
23	**3.57**	-	-0.13	-	1.00	Dry Ashing	-	ICP-OES	327	-	AOAC 985.01
25	**6.33**	0.07	**2.71**	**17.10**	5.0208 / 5.0213	HNO_3-HCl	Water	ICP-OES	Cu 327.395	-	USEPA Method 3050B
26	**2.94**	0.07	-0.78	**-4.95**	4.00	Dry ashing	Water & HCl (1+1)	AAS Shimadzu AA-7000	-	N	AOAC No. 975.03
27	**1.43**	0.13	**-2.34**	**-13.89**	-	-	-	ICP-MS 7700x Agilent	-	-	-
31	**1.51**	0.20	**-2.26**	**-12.11**	5.00	Dry Ashing	-	AAS	-	N	AOAC 985.35
34	**6.36**	-	**2.74**	-	2.00	Dry Ashing	HNO_3 and HCl	Cu: AAS Flame	Cu 324.70	92% (Na)	AOAC
37	**2841.00**	-	**2925.05**	-	1.00	Wet Digestion	Nitric + perchloric	ICP-OES (Perkin Elmer Optima 8000)	Cu 327.393	N	AOAC (2016) 984.27

Lab Number	Copper (mg/kg)	MU (mg/kg)	z score	Zeta score	Macro Metals Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wave-length	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x*) ± robust SD (s*) = 3.70 ± 0.97 mg/kg (CV 26.2%, n= 43) with u_{xpt} 0.15 mg/kg											
38	**1.04**	0.78	**-2.74**	**-6.38**	1.00	Dry Ashing	1N HNO_3	Flame AAS, Shimadzu AA6300	Cu 324.80	N	AOAC 985.35, 19th Ed 2012
39	**5.87**	-	**2.24**	-	0.50	Microwave		AAS	324.80	Y	AOAC 985.35
42	**1.64**	0.02	**-2.12**	**-13.70**	5.00	Dry Ashing	HNO_3-HCl	Cu, Zn: Flame AAS 280 FS	Cu 324.8	N	AOAC 985.35.2005
43	**3.47**	0.40	-0.24	-0.92	0.50	Microwave	HNO_3	ICP-OES, ICP-MS	Cu 327.395	N	AOAC
45	**4.98**	0.27	1.32	**6.33**	4.00	Dry Ashing	HCl+HNO_3+DI (2+2+70 mL) on hotplate	AAS (Flame, Varian)	Cu 324.8	N	AOAC 968.08
46	**0.65**	0.01	**-3.14**	**-20.32**	-	-	-	GFAAS (Agilent)	324.80	-	-
48	**6.52**	0.72	**2.90**	**7.25**	5.00	Dry Digestion	-	AA800 Perkin Elmer	Cu 324.8	N	MU-03/21 (AAS)
49	**0.28**	0.01	**-3.53**	**-22.78**	1, 2	Dry Ashing	Conc Nitric acid	ICP-OES 5110 Agilent	Cu 327.395	N	AOAC 20th Ed 2016
50	**5.51**	0.71	1.87	**4.72**	2.00	Wet	Acid	Flame AAS (Varian)	-	N	AOAC 985.35
53	**3.10**	0.77	-0.62	-1.45	0.30	Microwave	4 mL HNO_3, 1 mL HCl, 1 mL H_2O_2	ICPMS Thermo	-		In house method
54	**2.08**	0.17	-1.67	**-9.40**	1.00	Dry Ashing	HNO_3	ICP / Shimadzu	Cu 324	N	AOAC 984.27
55	**3.46**	-	-0.25	-	1.50	Wet digestion	-	ICP-OES	324.75	Y	AOAC (2012) 984.27
61	**6.50**	2.02	**2.89**	**2.74**	1.00	Acid block digestion	HNO_3	Varian AA240 FS Fast Sequential AAS	Cu 324.8	N	A6407-26 AAS (A6417 Spectro Method for P)
63	**2.55**	-	-1.19	-	-	-	-	-	-	-	-
67	**7.87**	-	**4.30**	-	2.0xxx	Dry Ash	Wet chemical	AAS, Perkin Elmer	Cu 324.75	N	AOAC 968.08
69	**5.76**	-	**2.12**	-	-	-	-	-	-	-	-
72	**3.83**	0.30	0.13	0.61	3.00	Ashing	HNO_3	ICP-OES, JY Ultima	Cu 324.754	N	AOAC 985.35

Lab Number	Copper (mg/kg)	MU (mg/kg)	z score	Zeta score	Macro Metals Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wave-length	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x*) ± robust SD (s*) = 3.70 ± 0.97 mg/kg (CV 26.2%, n= 43) with u_{xpt} 0.15 mg/kg											
75	**2.45**	0.23	-1.29	**-6.68**	1.00	Wet digestion (hot block)	HNO_3 + H_2O_2	ICP-OES Agilent 5100, ICP-MS Agilent 7700x	63 Cu 324.754	N	In House Method ICP-OES
77	**6.12**	-	**2.49**	-	-	-	-	-	-	-	-
78	**2.64**	0.40	-1.09	-	0.50	Mircowave Digestion	Acid Digestion	-	-	-	MP-AES
80	**3.36**	-	-0.35	-	-	-	-	-	-	-	-
82B	**3.90**	0.46	0.21	0.73	1.00	Microwave	Nitric Acid	Cu: AAS GBC	-	Y	Flame SSA
83	**4.01**	0.04	0.32	**2.02**	0.30	Microwave Digestion with HNO_3	-	Microwave digester Mars Xpress, ICP MS Nex Ion (Perkin Elmer)	-	Y	Application Note, Perkin Elmer
84	**2.95**	0.30	-0.77	**-3.54**	0.50	Microwave Digestion	HNO_3 / H_2O_2	ICP-MS	-	N	AOAC 999.10:2005
86	**3.70**	0.23	0.00	0.00	1.00	Wet Digest	-	ICP-OES	Cu 324.7	Y	AOAC (2012) 984.27
87	**3.50**	0.19	-0.21	-1.15	2.50	Dry Ashing	HNO_3	Microwave Digestion, Berghoff, Speedwave ENTRY	ICP-OES	N	MTD/FOD/CHM-09
89	**2.36**	0.04	-1.38	**-8.86**	2.00	Dry Ashing	1.5% HNO_3	AAS Agilent	Various	N	AOAC 985.35
90	**4.39**	-	0.71	-	1.00	Ultrawave	-	ICP-OES	Cu 324.752	-	-
91	**2.70**	-	-1.03	-	-	-	-	-	-	-	-
92	**8.06**	-	**4.49**	-	1.00	Ashing	HNO_3	ICP-OES	-	-	-
94	**36.00**	-	**33.30**	-	1.50	Dry ashing	-	-	-	Y	AOAC (2012) 984.27

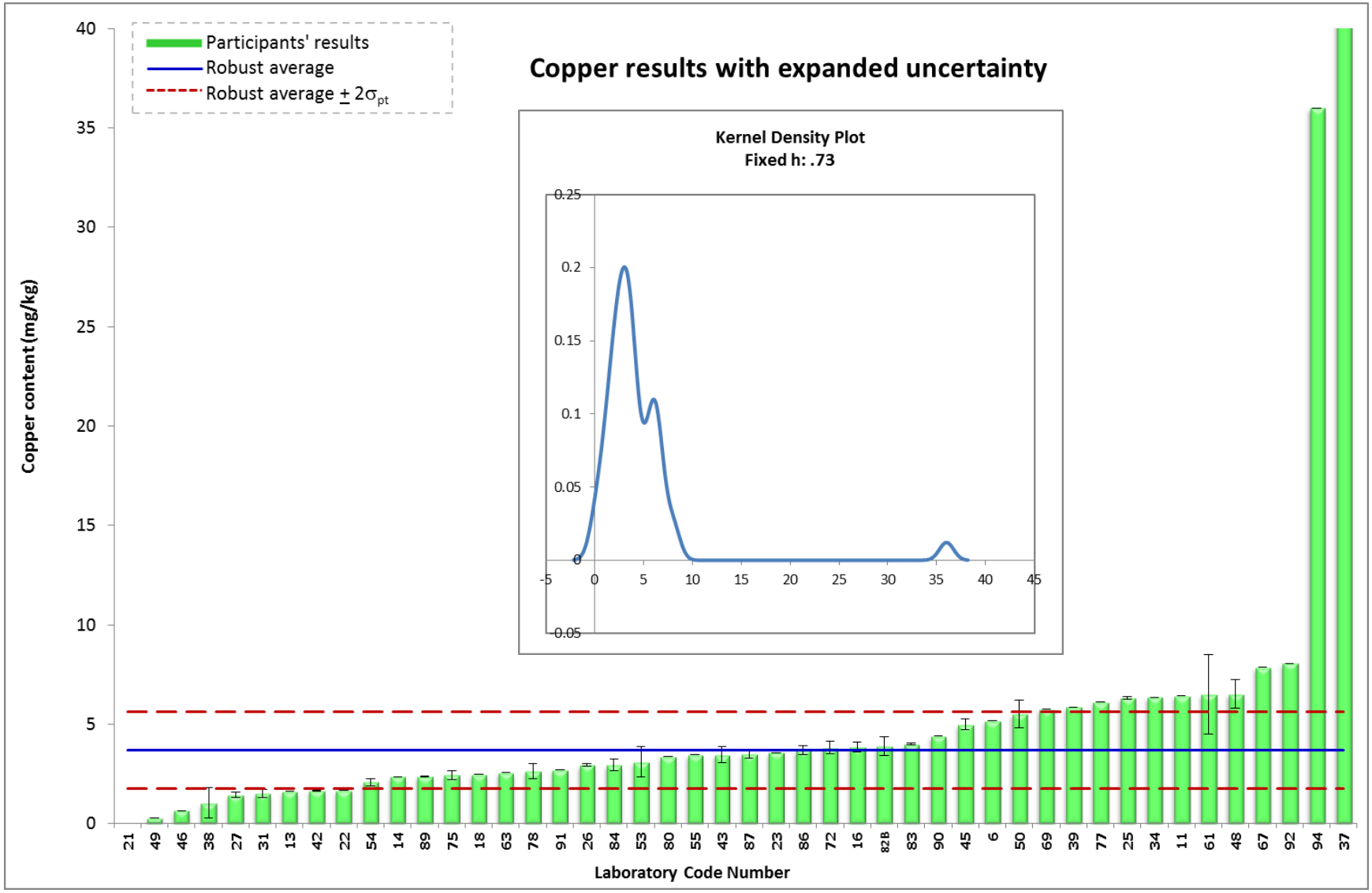

Figure 91. Distribution of **copper** results (ascending order) in fish meal with expanded uncertainty

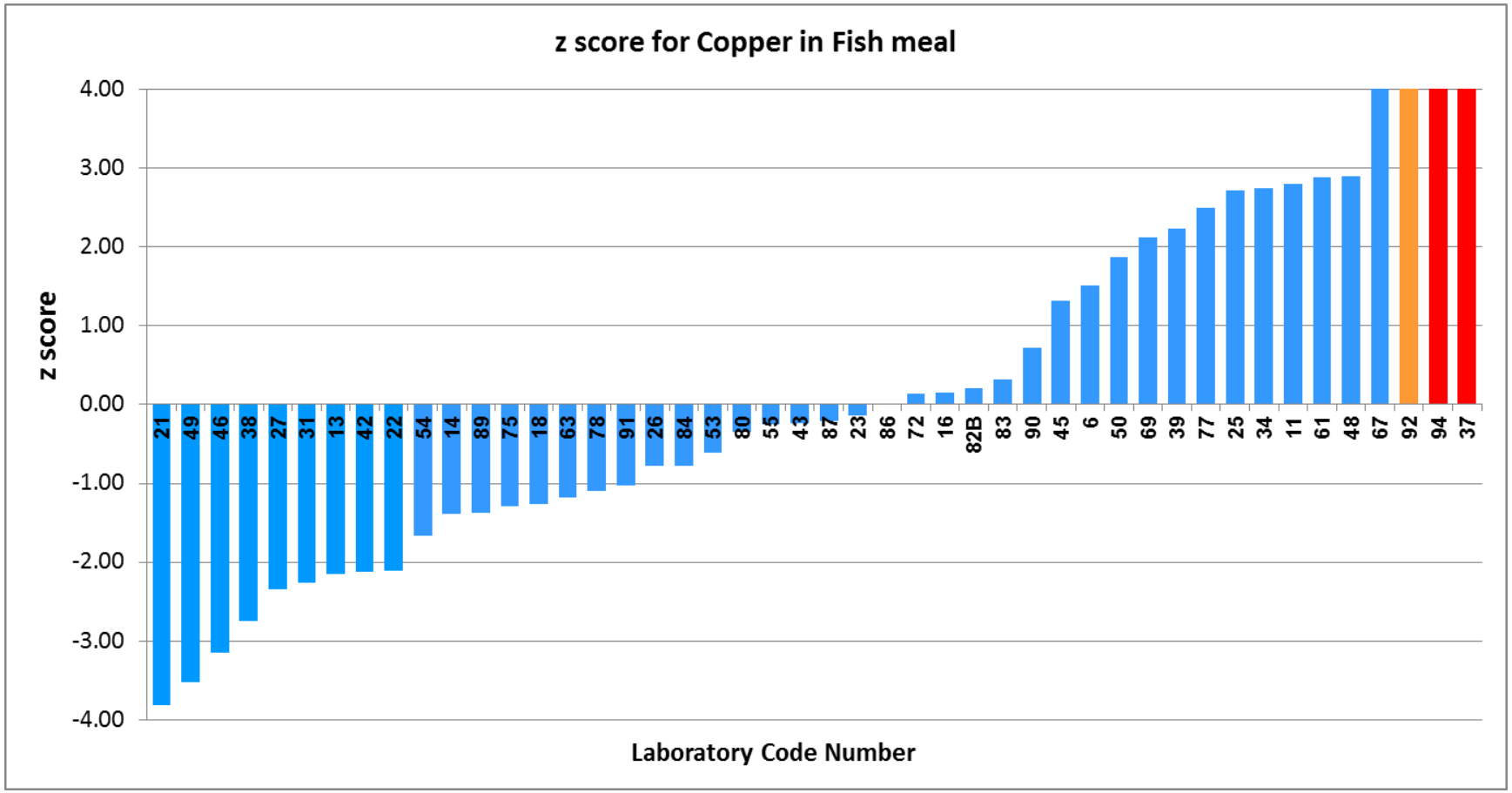

Figure 92. Plot of ordered *z* scores for **copper** results in fish meal

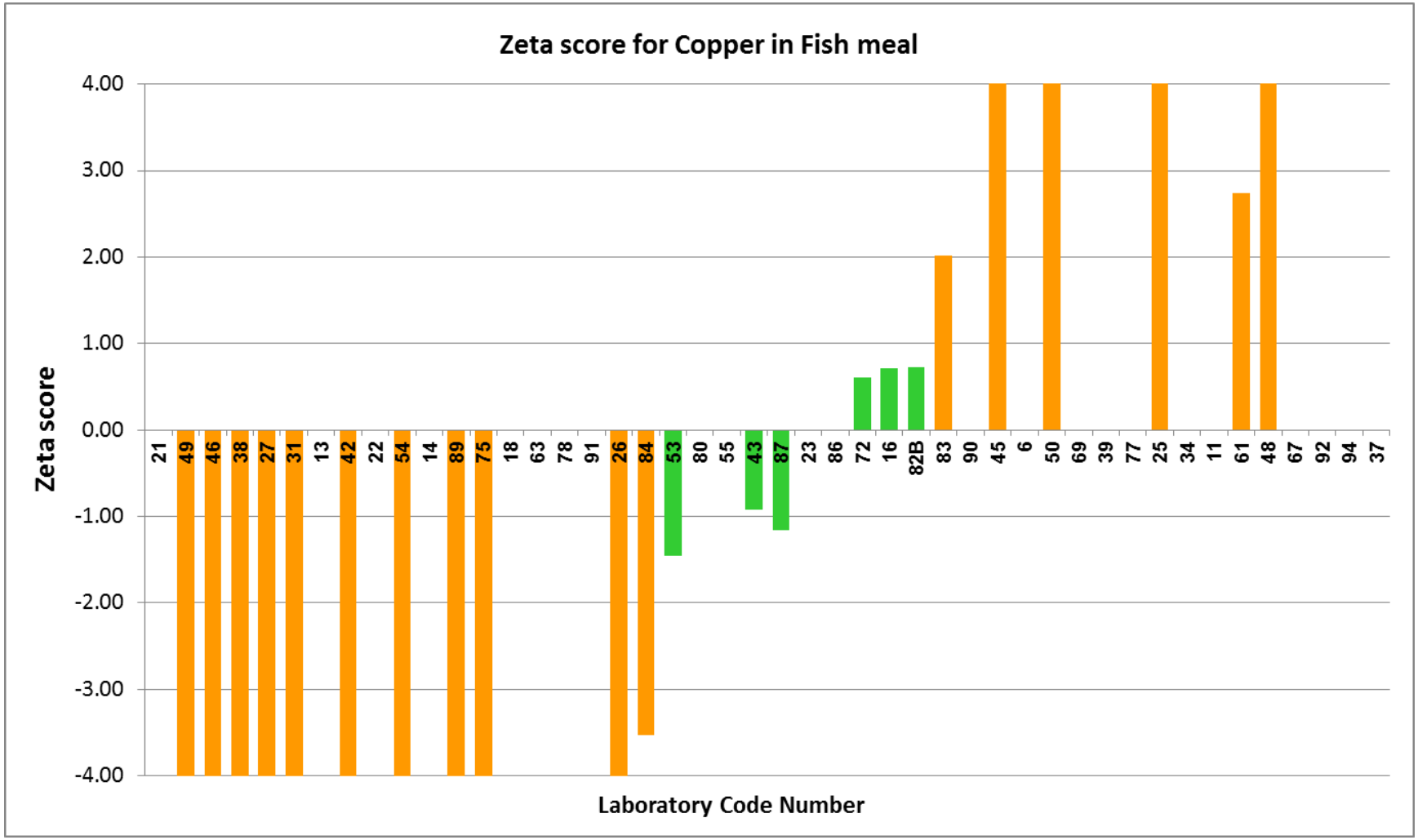

Figure 93. Plot of *Zeta score* for **copper** in fish meal, following the ordered z scores in the above Figure 92.

Table 32. Evaluation of laboratory performance **lead** analysis (mg/kg, as received) in fish meal

Lab Number	Lead (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x*) ± SDp from Horwitz's equation = 1.60±0.48 mg/kg (CV 30.0%,n= 29) with u_{xpt} 0.09 mg/kg											
Acceptance criteria			|z score| ≤ 2.00	|ζ score| ≤ 2.00							
6	**2.57**	-	**2.01**	-	1.0000	Acid	$HCl:HNO_3:H_2O$	AAS	-	Y	AOAC (2016), 20th Ed, 928.08, 985.35 (50.1.14)
11	**1.72**	-	0.25	-	2.0000	Dry Ashing	$HCL:H_2O$	AAS	-	Y	AOAC (2016), 975.03, 985.35
13	**0.97**	-	-1.31	-	0.5	Microwave	HNO_3 10 mL + HCl 2 mL	ICP-MS Thermo Scientific (iCAP	Pb 220.353	N	Internal Method
14	**2.08**	-	1.00	-	0.1 - 0.2	Ashing	Conc HNO_3, Conc $HClO_4$	ICP Horiba Jobin Yvon	-	Y	AOAC 975.03, 984.27
16	**1.39**	0.14	-0.44	-1.84	0.5	Hot plate	$HNO_3+H_2O_2$	ICP-MS 7700X Agilent	-	N	In-house Method
18	**4.81**	-	**6.69**	-	2.0	Dry Ashing	HNO_3	ICP-OES Agilent	-	N	AOAC 968.08
20	**0.87**	-	-1.52	-				AAS Analytic Jena, ZEEnit 700	Pb 405.781		-
21	**7.74**	-	**12.79**	-	0.1	Microwave	180°C	Mar Xpress (CEM)	-	Y	AOAC 2011.14 (2016)
22	**2.03**	-	0.90	-	0.2 to 0.3	Microwave	HNO_3	ICP-MS Perkin Elmer	220.00	-	AOAC 2015.06
23	**2.07**	-	0.98	-	1.00	Dry Ashing	-	ICP-OES	-	-	AOAC 985.01
25	**0.84**	0.07	-1.58	**-7.86**	5.0208 / 5.0213	HNO_3-HCl	Water	ICP-OES	-	-	USEPA Method 3050B
27	**0.63**	0.16	**-2.02**	**-8.06**				ICP-MS 7700x Agilent	-		
31	**< 0.011**	-	**-3.31**	-	5	Dry Ashing	-	AAS	-	N	AOAC 985.35
34	**1.04**	0.16	-1.17	**-4.60**	2.000	Dry Ashing	HNO_3 and HCl	Pb: AAS Agilent S200 Graphite	-	92% (Na)	AOAC
38	**4.65**	1.49	**6.35**	**4.06**	1.000	Dry Ashing	1N HNO_3 (0.1M HNO3	Flame AAS, Shimadzu	324.80	N	AOAC 985.35, 19th Ed 2012
42	**< 0.040**	-	**-3.25**		5	Dry Ashing	HNO_3-HCl	Pb, Cd: GFAAS Agilent 240 FS	Pb m/z 208	N	AOAC 985.35.2005
43	**2.63**	0.28	**2.15**	**6.19**	0.5	Microwave	HNO_3	ICP-OES, ICP-MS	-	N	AOAC

Lab Number	Lead (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x*) ± SDp from Horwitz's equation = 1.60±0.48 mg/kg (CV 30.0%,n= 29) with u_{xpt} 0.09 mg/kg											
48	**9.51**	1.12	**16.48**	**13.97**	5	Dry Digestion	-	AA800 Perkin Elmer	Pb 220.353	N	MU-03/21 (AAS)
49	**1.52**	0.08	-0.17	-0.81	1, 2	Dry Ashing	Conc Nitric acid	ICP-OES 5110 Agilent	-	N	AOAC 20th Ed 2016
50	**29.40**	9.28	**57.92**	**5.99**	2.0000	Wet	Acid	Flame AAS (Varian)	-	N	AOAC 985.35
52	**1.88**	-	0.59	-	1.0 (Ca, Fe)	Dry Ashing (Ca, Fe)	1 N HNO_3 (Ca, Fe)	Graphite Furnace AAS - Thermo	-	N	Modified AOAC 985.35 (Ca, Fe), 999.10 (K, Na)
53	**1.43**	0.14	-0.35	-1.49	0.3	Microwave	4 mL HNO_3, 1 mL HCl, 1 mL	ICPMS Thermo	Pb 220.353		In house method
54	**2.27**	0.16	1.40	**5.56**	1	Dry Ashing	HNO_3	ICP / Shimadzu	-	N	AOAC 984.27
58	**10.20**	1.78	**17.92**	**9.61**	3.0	Dry Ash	HCl	ICP-OES	-	-	-
63	**2.30**	-	1.46	-	-	-	-	-	-	-	-
67	**1.60**	-	0.00	-	2.0xxx	Dry Ash	Wet chemical	AAS, Perkin Elmer	-	N	AOAC 968.08
69	**1.69**	-	0.19	-	-	-	-	-	Pb 283.3	-	-
70	**1.20**	0.40	-0.83	-1.82				Z-2700 Hitachi	-		
72	**5.22**	0.40	**7.54**	**16.51**	3	Ashing	HNO_3	ICP-OES, JY Ultima	213.90	N	AOAC 985.35
75	**1.38**	0.03	-0.45	**-2.37**	1	Wet digestion (hot	HNO_3 + H_2O_2	ICP-OES Agilent 5100, ICP-MS	-	N	In House Method ICP-OES
77	**< 0.01**	-	**-3.31**	-	-	-	-	-	-	-	-
80	**1.50**	-	-0.21	-	-	-	-	-	Pb 217.0	-	-
81	**1.60**	-	0.00	-	1.0024	Dry Ashing (Ca, Fe)	1 N HNO_3 (Ca, Fe)	Pb: Thermo iCe 3500	-	N	AOAC 999.10 Mod (Na, K), 985.35 Mod (Ca, Fe)
82B	**0.81**	0.12	-1.64	**-7.29**	1.00	Microwave	Nitric Acid	Pb, Cd, As: AAS, Agilent	-	Y	Flame SSA
83	**1.77**	0.11	0.36	1.65	0.3	Microwave Digestion	-	Microwave digester Mars	-	Y	Application Note, Perkin Elmer
84	**1.67**	0.17	0.15	0.57	0.5	Microwave Digestion	HNO_3 / H_2O_2	ICP-MS	-	N	AOAC 999.10:2005

Lab Number	Lead (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x*) ± SDp from Horwitz's equation = 1.60±0.48 mg/kg (CV 30.0%,n= 29) with u_{xpt} 0.09 mg/kg											
86	**1.88**	0.16	0.58	**2.33**	1.0000	Wet Digest	-	ICP-OES	ICP-OES	Y	AOAC (2012) 984.27
87	**5.29**	0.14	**7.69**	**32.36**	2.5	Dry Ashing	HNO_3	Microwave Digestion,	Pb 217.0	N	MTD/FOD/CHM-09
90	**0.34**	-	**-2.62**	-	1	Ultrawave	-	ICP-OES	-	-	-
91	**4.14**	-	**5.29**	-	-	-	-	-	-	-	-

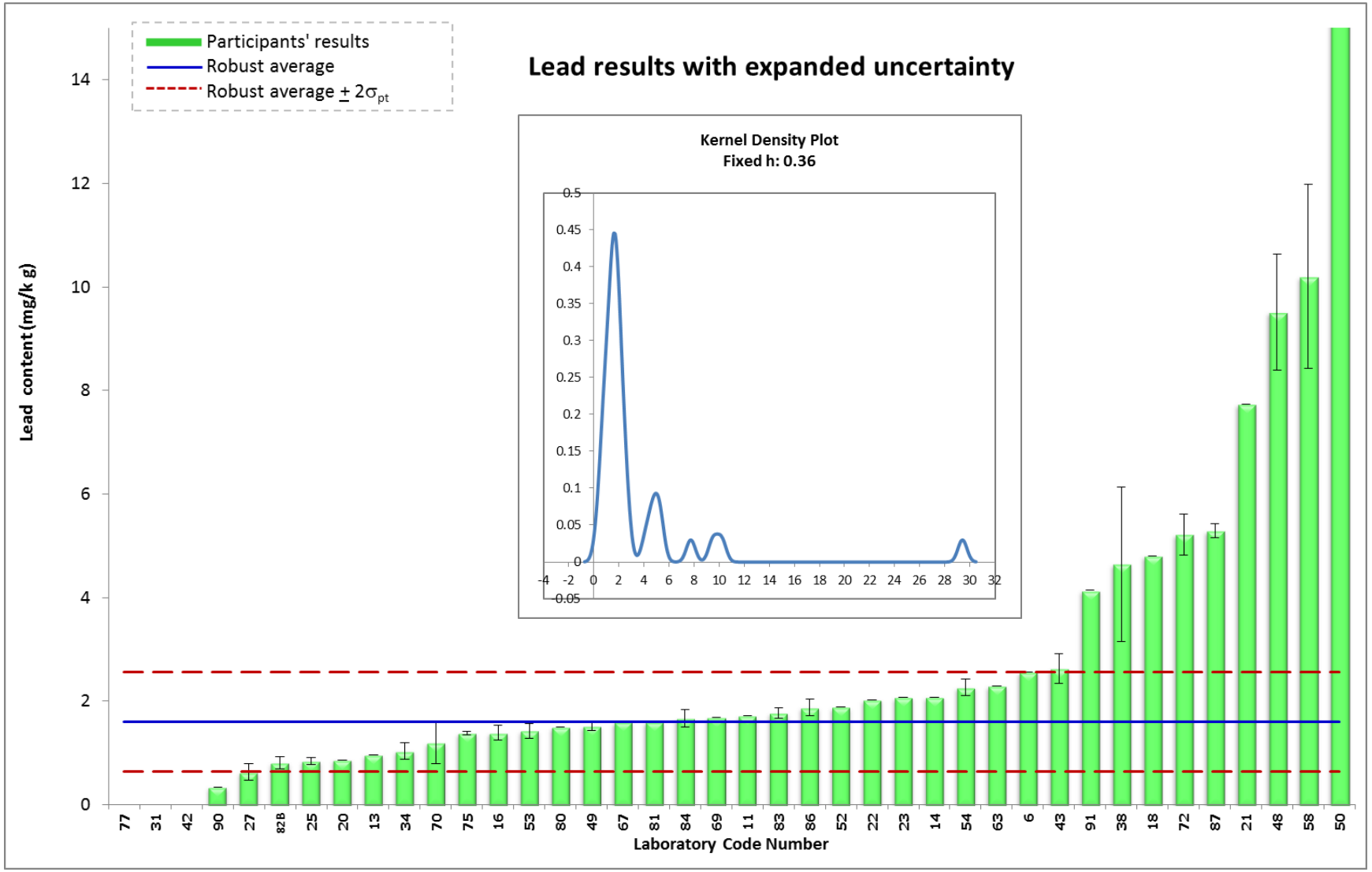

Figure 94. Distribution of **lead** results (ascending order) in fish meal with expanded uncertainty

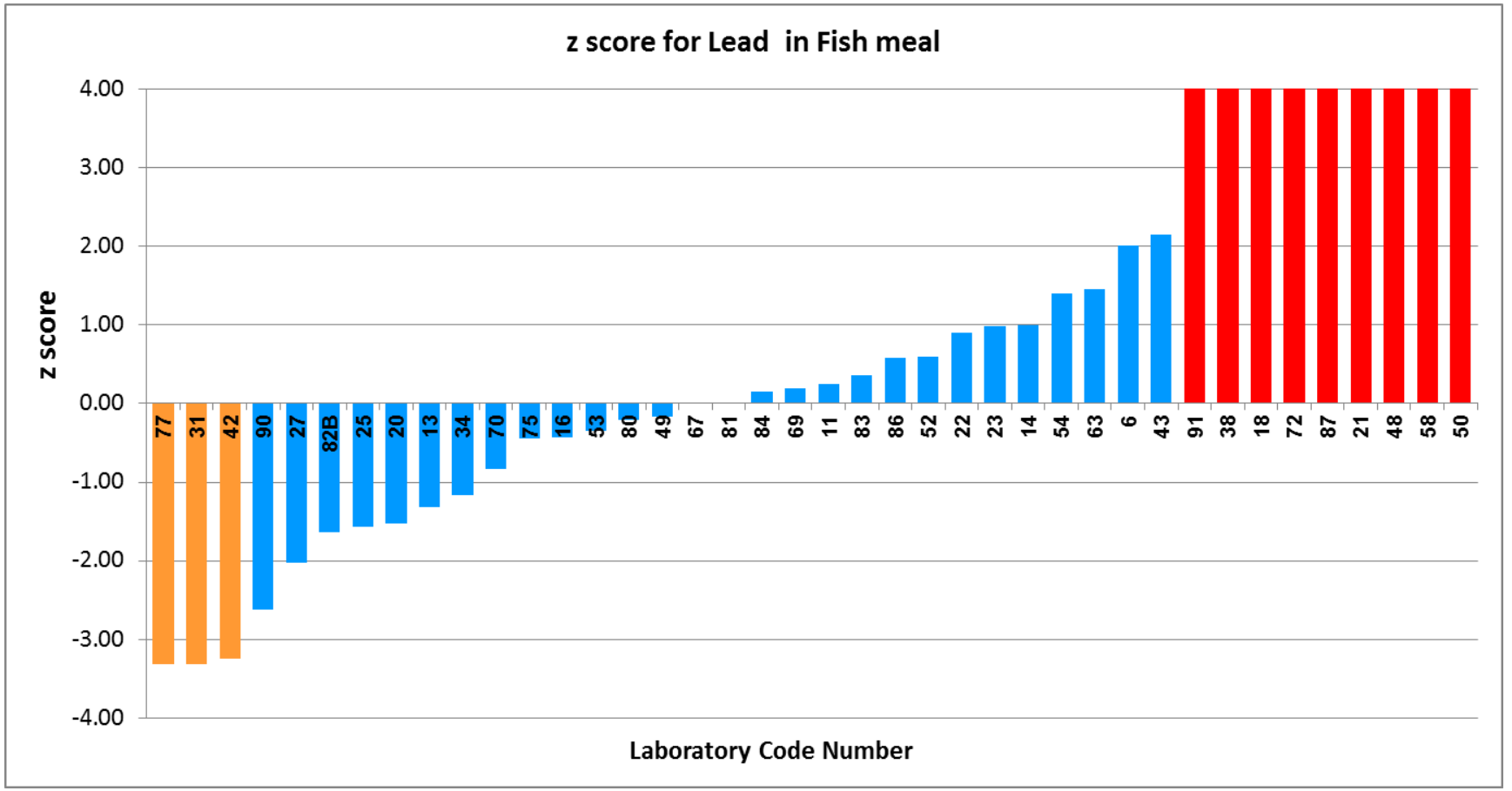

Figure 95. Plot of ordered *z* scores for **lead** results in fish meal

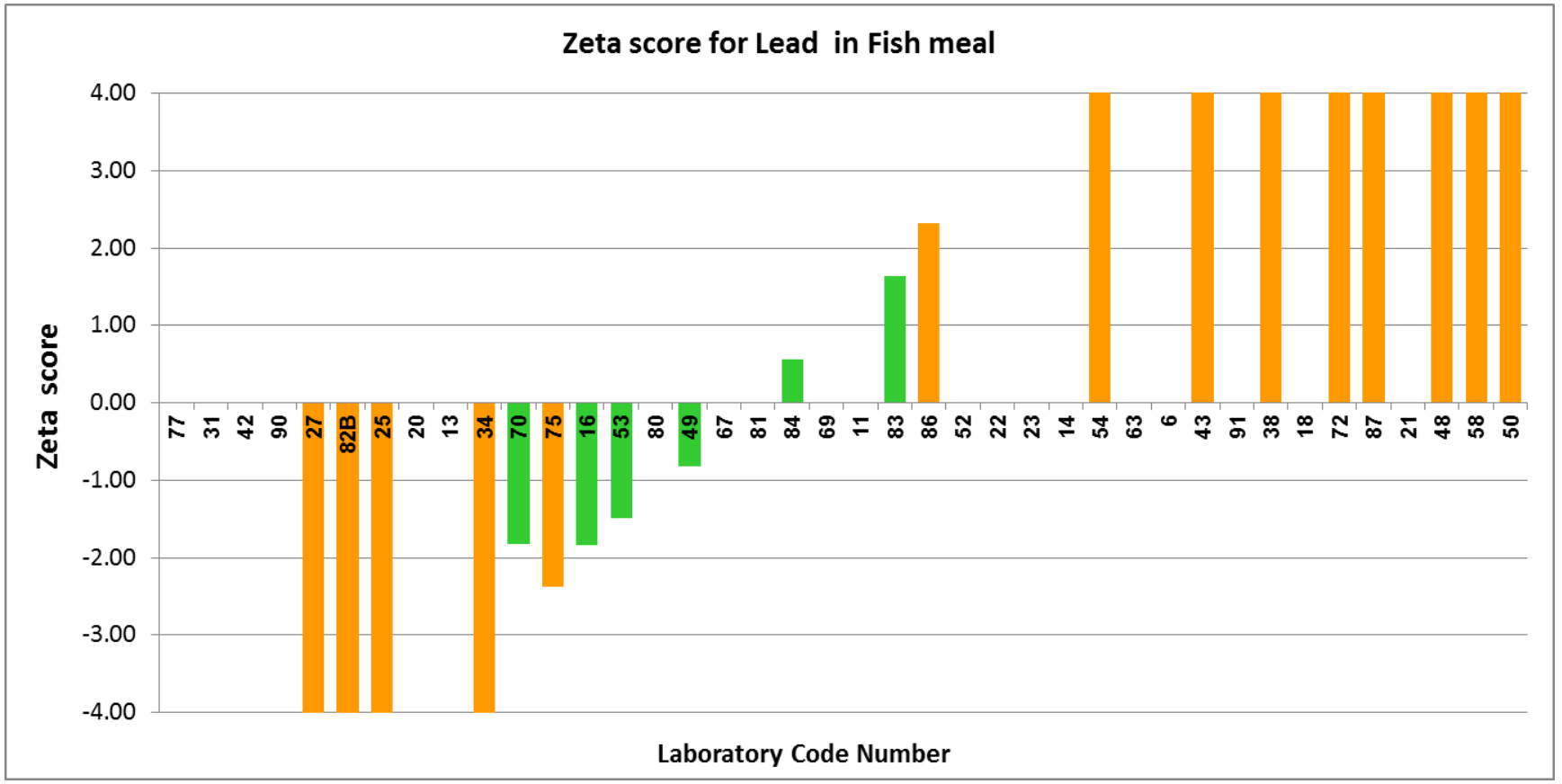

Figure 96. Plot of *Zeta score* for **lead** in fish meal, following the ordered z scores in the above Figure 95.

Table 33. Evaluation of laboratory performance **arsenic** analysis (mg/kg, as received) in fish meal

Lab Number	Arsenic (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x*) ± 2SDp from Horwitz's equation = 6.84±1.20 mg/kg (CV 24.0%,n= 34) with u_{xpt} 0.28 mg/kg											
Acceptance criteria			|z score| ≤ 2.00	|ζ score| ≤ 2.00							
6	0.29	-	-5.46	-	1.0000	Acid	HCl:HNO_3:H_2O	AAS	-	Y	AOAC (2016), 20th Ed, 928.08, 985.35 (50.1.14)
11	0.75	-	-5.08	-	2.0000	Dry Ashing	HCL:H_2O	AAS	As 193.7	Y	AOAC (2016), 975.03, 985.35
13	6.62	-	-0.18	-	0.5	Microwave	HNO_3 10 mL + HCl 2 mL	ICP-MS Thermo Scientific (iCAP	As 75	N	Internal Method
14	5.73	-	-0.93	-	0.1 - 0.2	Ashing	Conc HNO_3, Conc $HClO_4$	ICP Horiba Jobin Yvon	As 189.042	Y	AOAC 975.03, 984.27
16	7.97	0.79	0.94	2.33	0.5	Hot plate	HNO_3+H_2O_2	ICP-MS 7700X Agilent		N	In-house Method
18	1.29	-	-4.63	-	2.0	Dry Ashing	HNO_3	ICP-OES Agilent for Hg, As)	As 193.7	N	AOAC 968.08
21	3.35	-	-2.91	-	0.1	Microwave	180°C	Mar Xpress (CEM)	As 193.695	Y	AOAC 2011.14 (2016)
22	4.19	-	-2.21	-	0.2 to 0.3	Microwave	HNO_3	ICP-MS Perkin Elmer		-	AOAC 2015.06
23	6.17	-	-0.56	-	1.00	Dry Ashing	-	ICP-OES	188.00	-	AOAC 985.01
27	2.63	0.34	-3.51	-12.85	-	-	-	ICP-MS 7700x Agilent	-	-	-
31	0.49	0.05	-5.29	-22.60	5	Dry Ashing	-	AAS	-	N	AOAC 985.35
34	11.60	-	3.97	-	2.000	Dry Ashing	HNO_3 and HCl	As: HG AAS	As 193.7	92% (Na)	AOAC
38	0.80	0.03	-5.04	-21.55	1.000	Dry Ashing	1N HNO_3 (0.1M HNO3	Flame AAS, Shimadzu AA6300	As 193.70	N	AOAC 985.35, 19th Ed 2012
43	9.16	0.48	1.93	6.29	0.5	Microwave	HNO_3	ICP-OES, ICP-MS	As m/z 75	N	AOAC
48	9.48	0.27	2.20	8.48	5	Dry Digestion	-	AA800 Perkin Elmer	As 193.7	N	MU-03/21 (AAS)
49	0.15	0.01	-5.58	-23.90	1, 2	Dry Ashing	Conc Nitric acid	ICP-OES 5110 Agilent	As 188.98	N	AOAC 20th Ed 2016
52	7.28	-	0.37	-	1.0 (Ca, Fe)	Dry Ashing (Ca, Fe)	1 N HNO_3 (Ca, Fe)	Graphite Furnace AAS - Thermo iCE	As 193.7	N	Modified AOAC 985.35 (Ca, Fe)

Lab Number	Arsenic (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Instrument	Wavelength	Recovery Correction (Y/N)	Method Reference
Assigned value obtained from robust average (x*) ± 2SDp from Horwitz's equation = 6.84±1.20 mg/kg (CV 24.0%,n= 34) with u_{xpt} 0.28 mg/kg											
53	**7.66**	0.25	0.68	**2.67**	0.3	Microwave	4 mL HNO_3, 1 mL HCl, 1 mL	ICP-MS Thermo	-		In house method
54	**6.05**	1.70	-0.66	-0.88	1	Dry Ashing	HNO_3	ICP / Shimadzu	-	N	AOAC 984.27
58	**0.32**	0.04	**-5.43**	**-23.22**	3.0	Dry Ash	HCl	AAS-Hydride for As	As 189	-	-
63	**2.83**	-	**-3.34**	-	-	-	-	-	-	-	-
67	**0.37**	-	**-5.39**	-	2.0xxx	Dry Ash	Wet chemical	AAS, Perkin Elmer	As 193.70	N	AOAC 968.08
69	**7.92**	-	0.90	-	-	-	-	-	-	-	-
70	**0.12**	0.04	**-5.60**	**-23.93**	-	-	-	Z-2700 Hitachi	As 193.7	-	-
72	**4.11**	0.40	**-2.28**	**-7.93**	3	Ashing	HNO_3	ICP-OES, JY Ultima	As 193.695	N	AOAC 985.35
75	**8.64**	0.48	1.50	**4.86**	1	Wet digestion (hot block)	HNO_3 + H_2O_2	ICP-OES Agilent 5100, ICP-MS	As 193.696	N	In House Method ICP-OES
77	**< 0.001**	-	**-5.70**	-	-	-	-	-	-	-	-
80	**7.88**	-	0.87	-	-	-	-	-	-	-	-
82A	**5.98**	0.55	-0.72	**-2.19**	0.250	none	none	HPGe detector, Canberra	-	-	Neutron Activation Analysis (NAA)
83	**8.48**	0.08	1.37	**5.81**	0.3	Microwave Digestion with	-	Microwave digester Mars Xpress, ICP	-	Y	Application Note, Perkin Elmer
84	**8.27**	0.83	1.19	**2.86**	0.5	Microwave Digestion	HNO_3 / H_2O_2	ICP-MS	-	N	AOAC 999.10:2005
86	**6.96**	0.41	0.10	0.35	1.0000	Wet Digest	-	As: ICP-MS	As Mass 75	Y	AOAC (2012) 984.27
87	**8.38**	0.10	1.29	**5.42**	2.5	Dry Ashing	HNO_3	Microwave Digestion,	ICP-OES	N	MTD/FOD/CHM-09
88	**0.33**	0.01	**-5.42**	**-23.24**	3	Dry Ashing	HNO_3 conc 10 mL	AAS GBC Hydride vapour	As 193.70	N	In house method (AAS)
90	**0.02**	-	**-5.69**	-	1	Ultrawave	-	ICP-OES	-	-	-

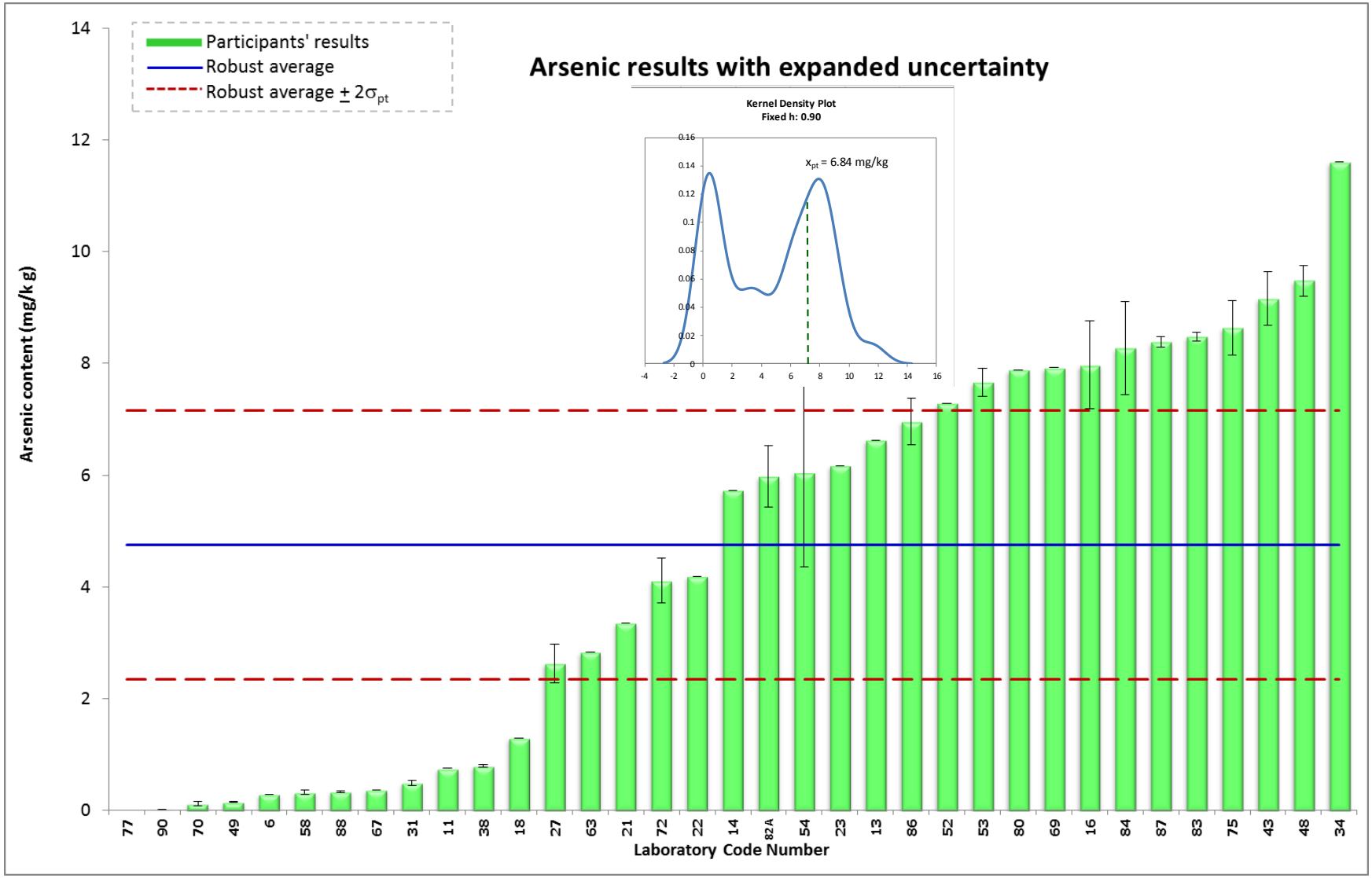

Figure 97. Distribution of **arsenic** results (ascending order) in fish meal with expanded uncertainty

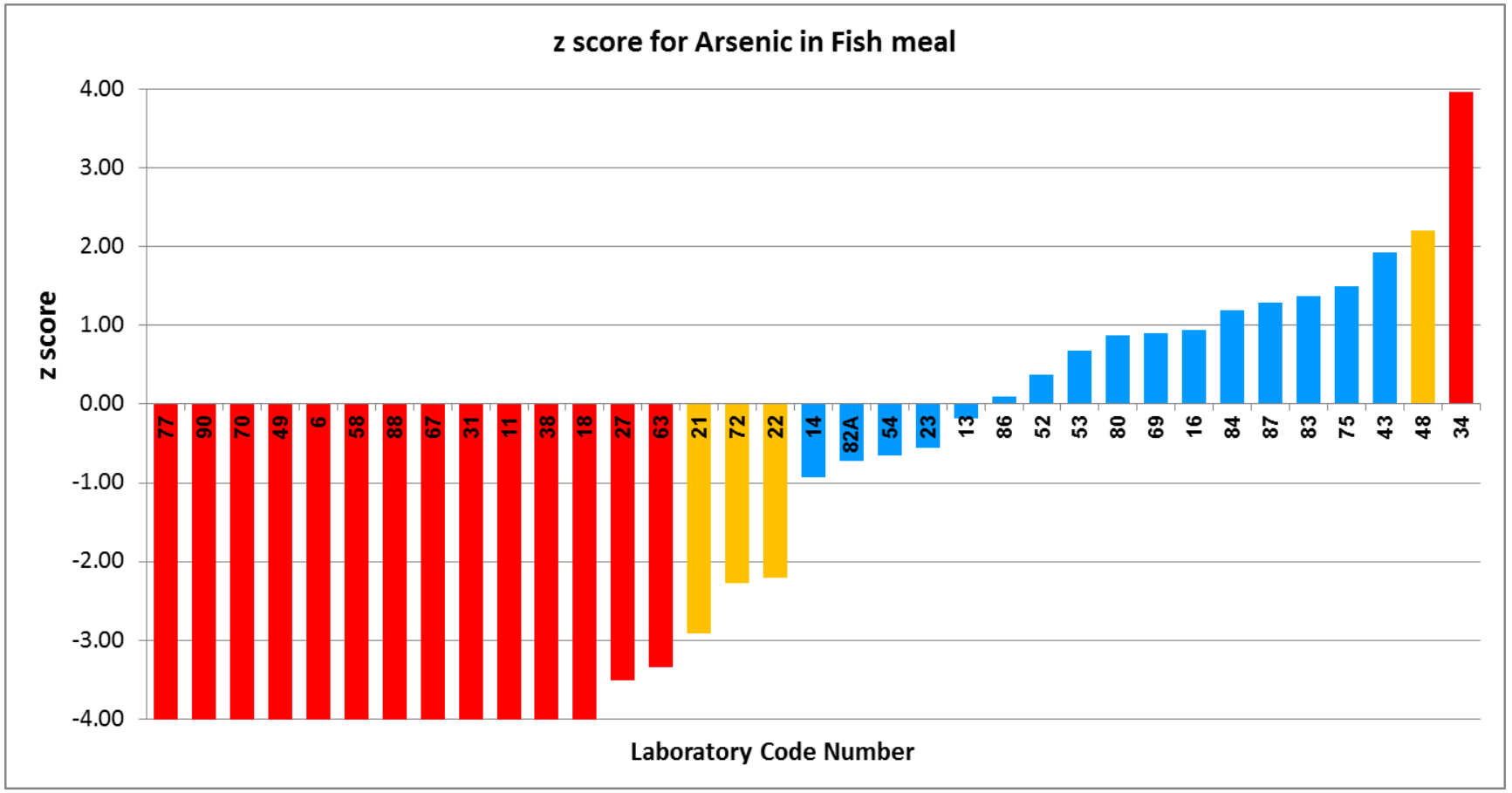

Figure 98. Plot of ordered *z* scores for **arsenic** results in fish meal

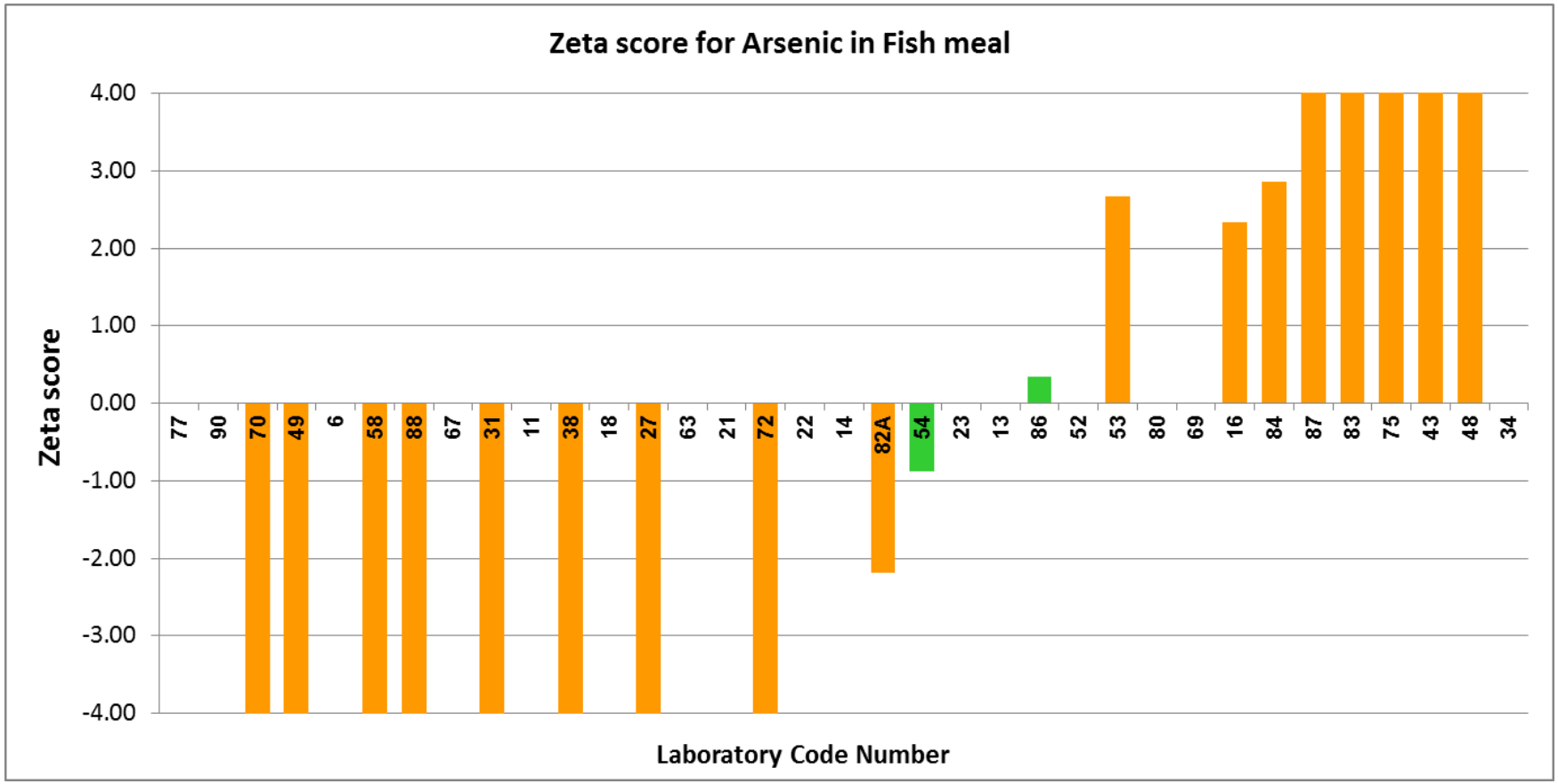

Figure 99. Plot of *Zeta score* for **arsenic** in fish meal, following the ordered z scores in the above Figure 98.

Table 34. Evaluation of laboratory performance **cholesterol** analysis (mg/kg, as received) in fish meal

Lab Number	Cholesterol (mg/100g)	MU (mg/100g)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Derivatization	Method Reference
Assigned value obtained from robust average (x*) ± robust SD (s*) = 496.8 ± 63.1 mg/100g (CV 12.7%, n= 27) with u_{xpt} 15.2 mg/100g									
Acceptance criteria			|z score| ≤ 2.00	|ζ score| ≤ 2.00					
2	**526**	-	0.46	-	-	-	-	-	-
11	**514**	-	0.28	-	-	-	-	-	-
12	**244**	30.0	**-4.01**	**-11.85**	-	-	-	-	-
14	**578**	-	1.28	-	0.25	Saponification	95% Ethanol, 50% KOH	Hexamethyldisilazane, Trimethylchlorosilane	AOAC 994.10
16	**89**	0.4	**-6.47**	**-26.90**	-	-	-	-	-
21	**491**	-	-0.09	-	-	-	-	-	-
23	**488**	-	-0.14	-	-	-	-	-	-
31	**< 1.1**	-	**-7.86**	-	-	-	-	-	-
32	**457**	18.0	-0.63	**-2.26**	-	-	-	-	-
37	**559**	-	0.98	-	-	-	-	-	-
39	**554**	-	0.91	-	-	-	-	-	-
42	**724**	25.0	**3.60**	**11.55**	-	-	-	-	-
43	**646**	138.6	**2.36**	**2.10**	-	-	-	-	-
48	**537**	20.2	0.64	**2.21**	-	-	-	-	-
49	**542**	27.0	0.72	**2.23**	-	-	-	-	-
54	**476**	14.0	-0.33	-1.24	-	-	-	-	-

Lab Number	Cholesterol (mg/100g)	MU (mg/100g)	z score	Zeta score	Sample Weight (g)	Digestion Technique	Digestion Medium	Derivatization	Method Reference
Assigned value obtained from robust average (x*) ± robust SD (s*) = 496.8 ± 63.1 mg/100g (CV 12.7%, n= 27) with u_{xpt} 15.2 mg/100g									
55	**442**	11.0	-0.87	**-3.40**	-	-	-	-	-
58	**565**	16.9	1.08	**3.94**	-	-	-	-	-
61	**461**	83.0	-0.57	-0.81	-	-	-	-	-
63	**526**	-	0.46	-	-	-	-	-	-
68	**522**	-	0.40	-	-	-	-	-	-
69	**421**	-	-1.20	-	-	-	-	-	-
72	**471**	10.0	-0.41	-1.61	-	-	-	-	-
79	**352**	-	**-2.29**	-	-	-	-	-	-
81	**558**	-	0.97	-	-	-	-	-	-
83	**454**	12.5	-0.69	**-2.64**	-	-	-	-	-
84	**433**	13.0	-1.01	**-3.86**	-	-	-	-	-
86	**422**	9.5	-1.19	**-4.70**	-	-	-	-	-
87	**349**	34.8	**-2.35**	**-6.42**	-	-	-	-	-
88	**495**	0.1	-0.03	-0.14	-	-	-	-	-
94	**527**	-	0.48	-	-	-	-	-	-

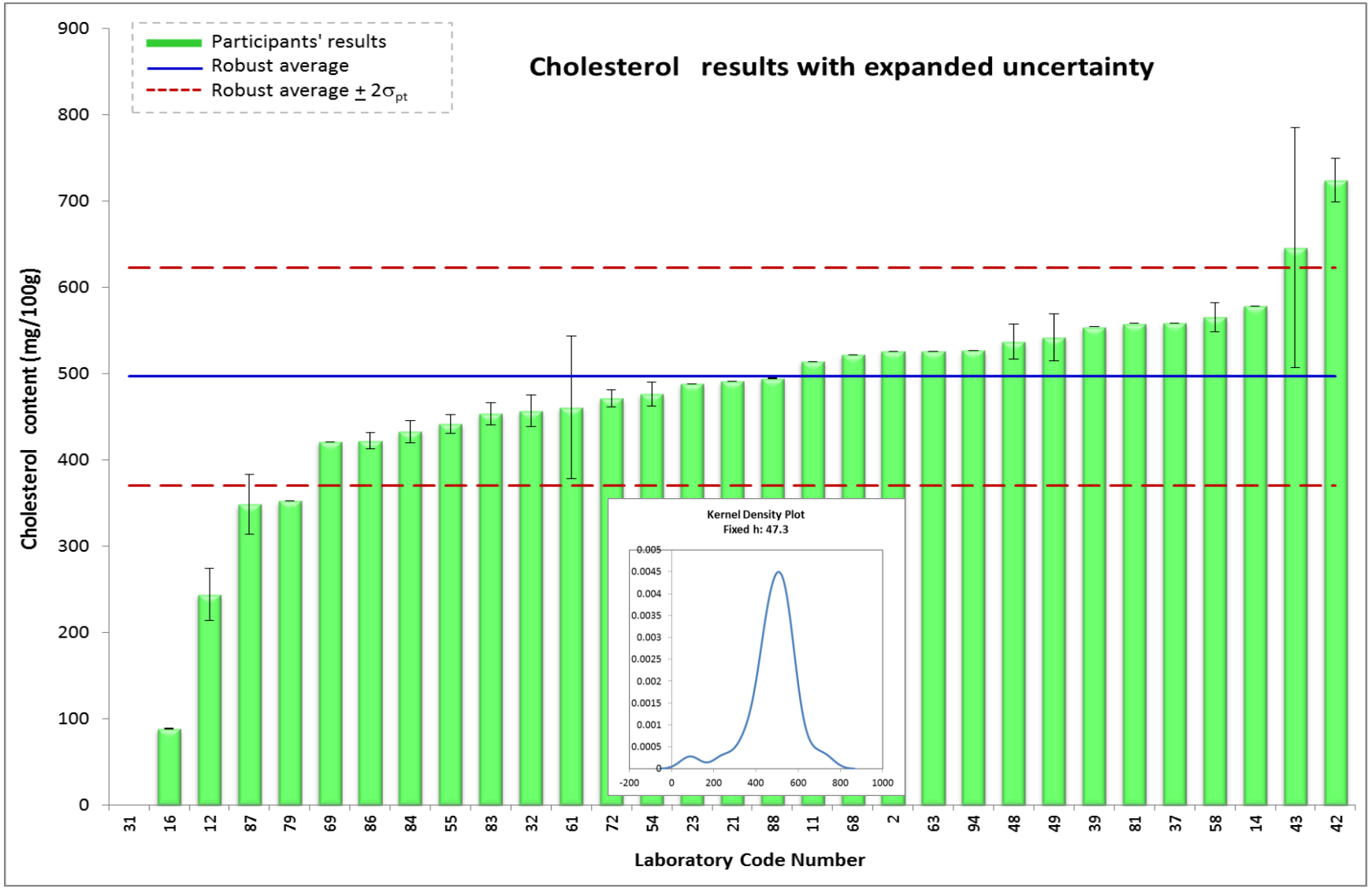

Figure 100. Distribution of **cholesterol** results (ascending order) in fish meal with expanded uncertainty

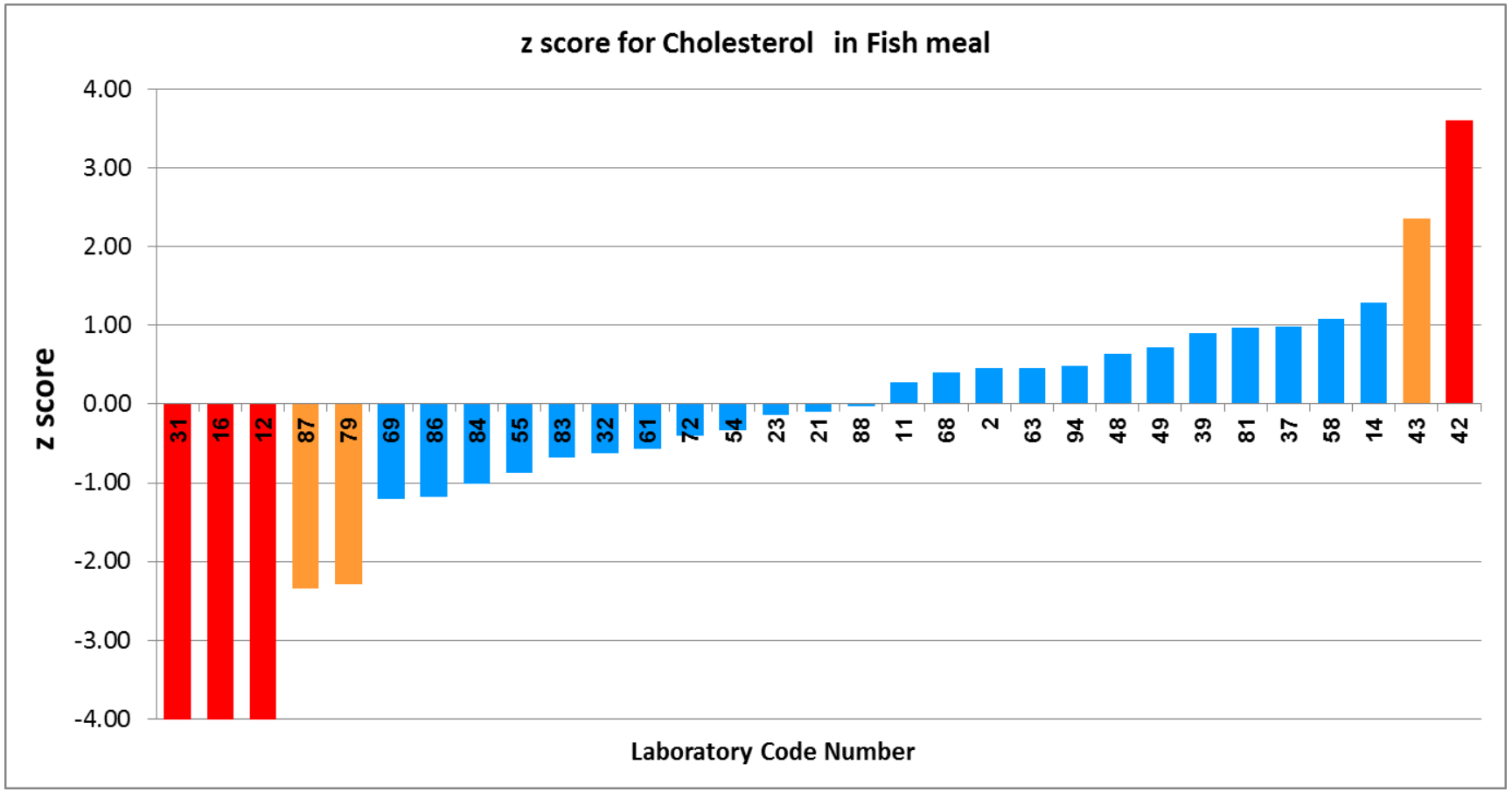

Figure 101. Plot of ordered *z* scores for **cholesterol** results in fish meal

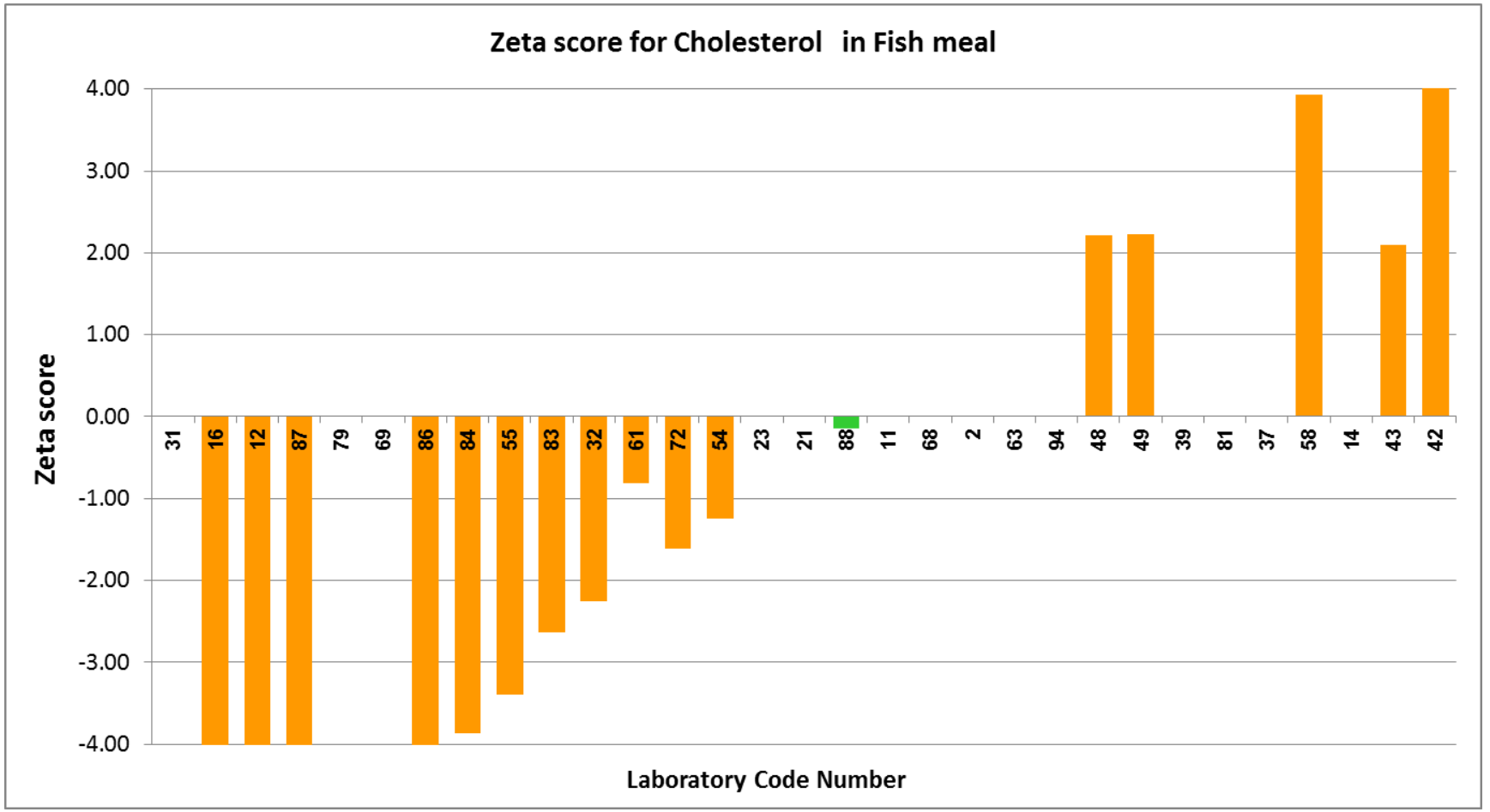

Figure 102. Plot of *Zeta score* for **cholesterol** in fish meal, following the ordered z scores in the above Figure 101.

Table 35. Evaluation of laboratory performance **TVBN** analysis (mg/kg, as received) in fish meal

Lab Number	TVBN (mg/100g)	MU (mg/100g)	z score	Zeta score	Method Reference
Assigned value obtained from robust average (x*) ± robust SD (s*) = 227.92 ± 12.35 mg/100g (CV 5.42%, n= 25) with u_{xpt} 3.45 mg/100g					
		Acceptance criteria	|z score| ≤ 2.00	|ζ score| ≤ 2.00	All laboratories did not submit method of TVBN analysis.
2	**187.00**	-	**-3.31**	-	
6	**222.53**	-	-0.44	-	
15	**232.00**	-	0.33	-	
19	**223.90**	-	-0.33	-	
20	**262.32**	-	**2.79**	-	
21	**312.00**	-	**6.81**	-	
23	**221.00**	-	-0.56	-	
27	**159.23**	1.39	**-5.56**	**-19.51**	
43	**244.03**	16.78	1.30	1.78	
45	**228.59**	10.63	0.05	0.11	
49	**219.00**	0.00	-0.72	**-2.58**	
58	**238.78**	6.91	0.88	**2.22**	
60	**233.12**	-	0.42	-	
63	**282.00**	-	**4.38**	-	
67	**214.00**	-	-1.13	-	

Lab Number	TVBN (mg/100g)	MU (mg/100g)	z score	Zeta score	Method Reference
Assigned value obtained from robust average (x*) ± robust SD (s*) = 227.92 ± 12.35 mg/100g (CV 5.42%, n= 25) with u_{xpt} 3.45 mg/100g					
68	**222.00**	-	-0.48	-	All laboratories did not submit method of TVBN analysis.
71	**224.88**	6.74	-0.25	-0.63	
73A	**216.82**	4.72	-0.90	**-2.65**	
77	**35.42**	-	**-15.59**	-	
83	**229.34**	6.12	0.12	0.31	
86	**241.00**	-	1.06	-	
87	**764.36**	76.44	**43.44**	**13.98**	
88	**248.54**	0.24	1.67	**5.97**	
92	**227.00**	-	-0.07	-	
95	**218.19**	9.94	-0.79	-1.61	

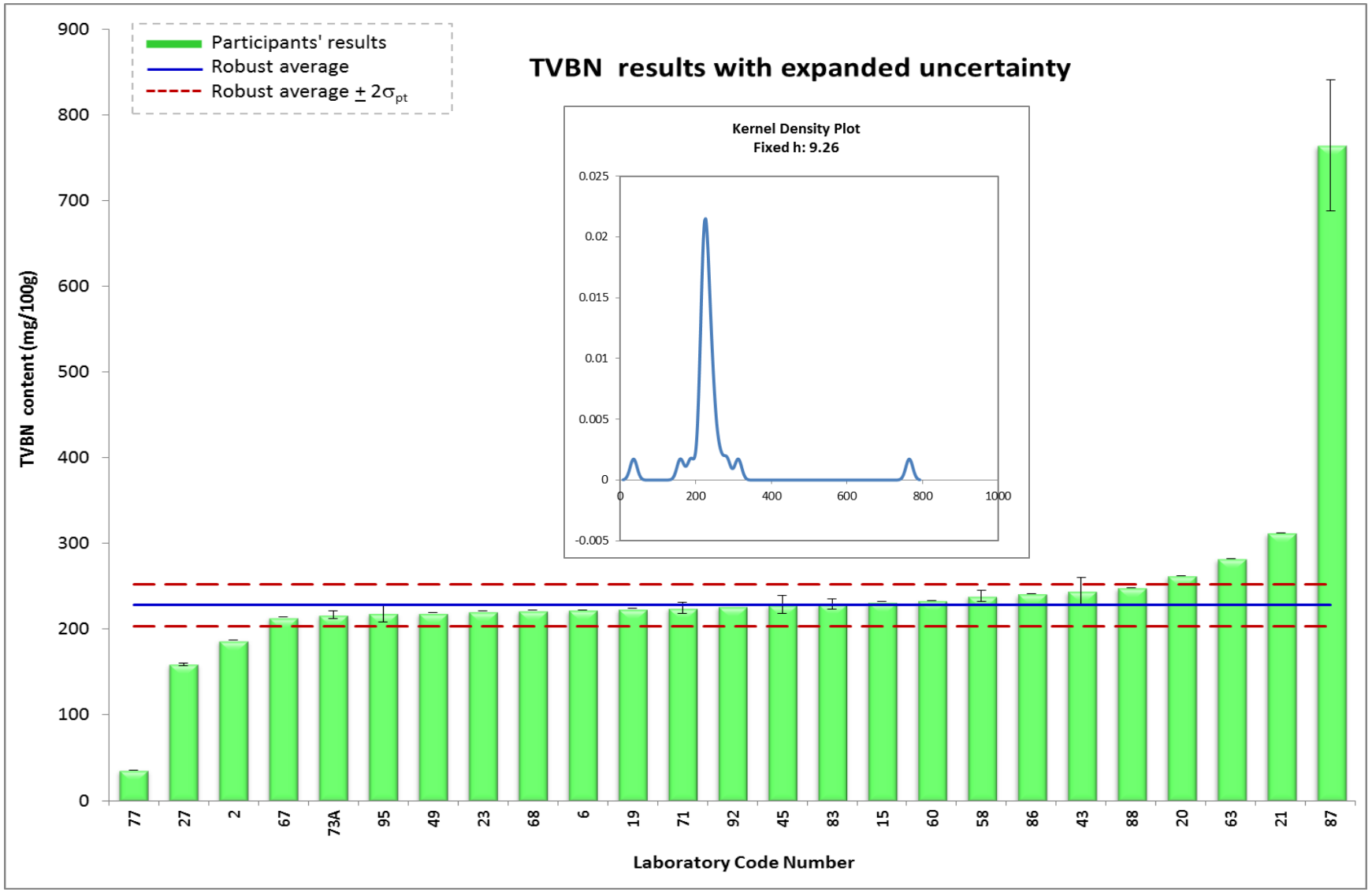

Figure 103. Distribution of **TVBN** results (ascending order) in fish meal with expanded uncertainty

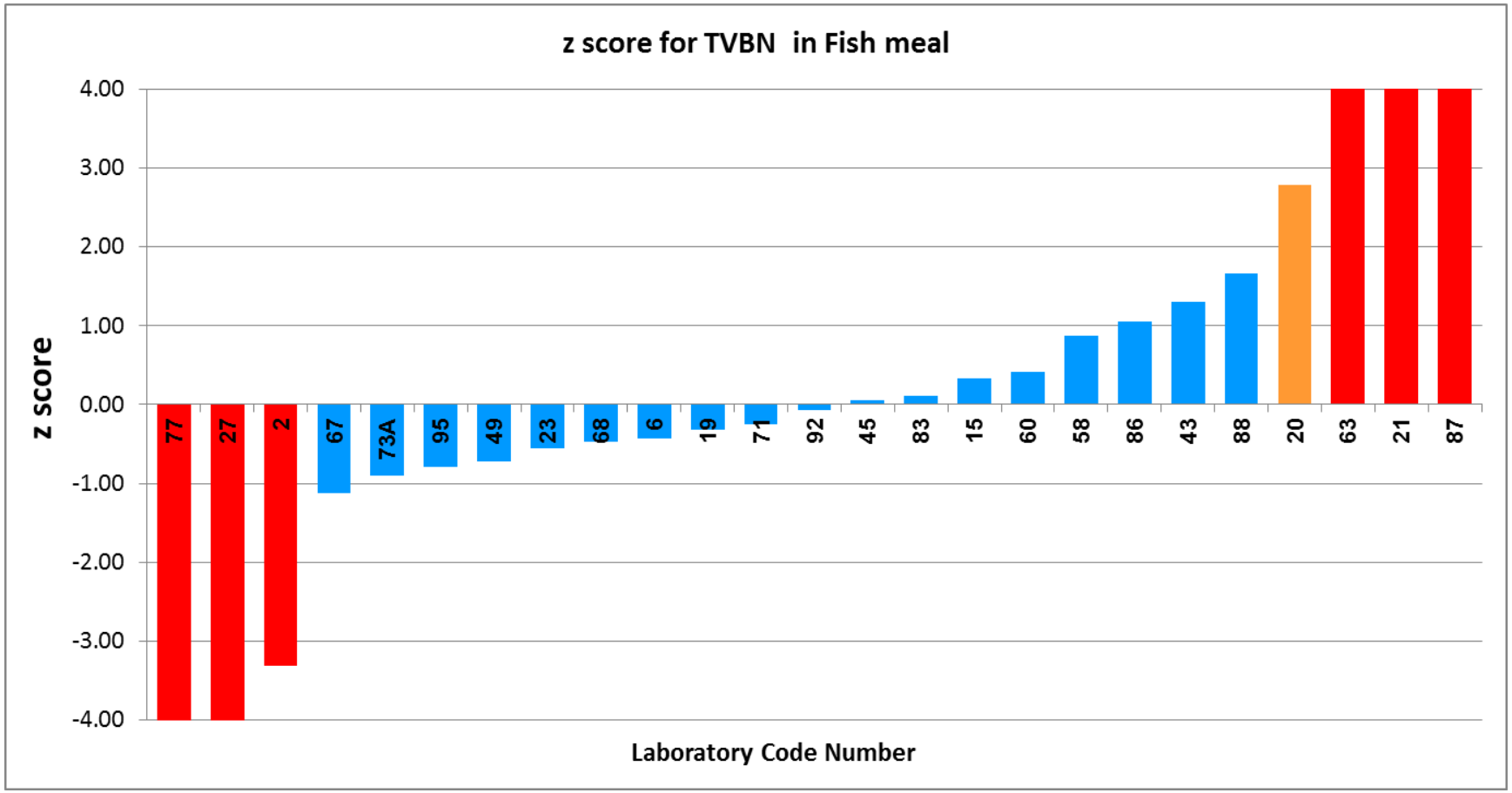

Figure 104. Plot of ordered *z* scores for **TVBN** results in fish meal

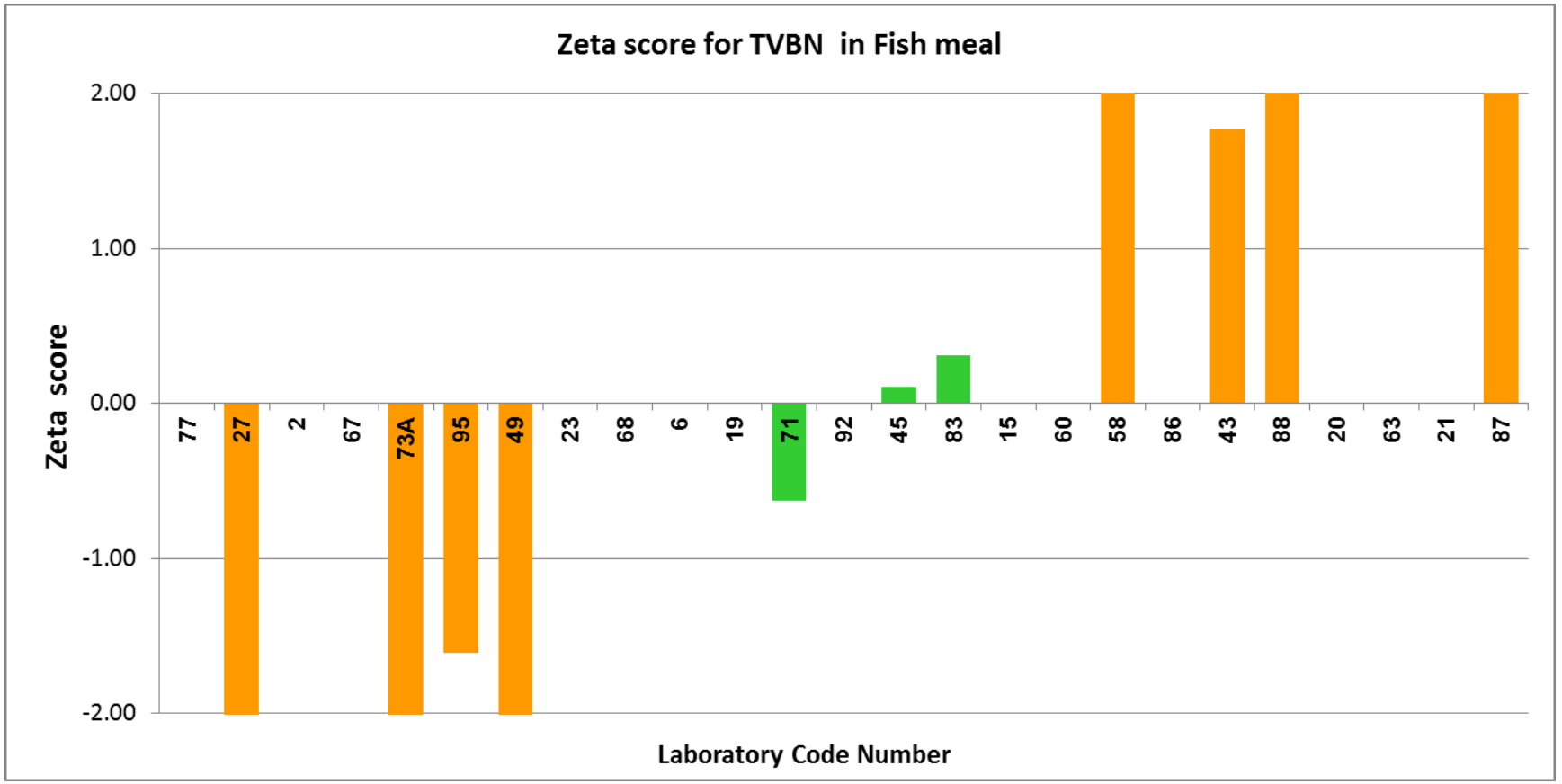

Figure 105. Plot of *Zeta score* for **TVBN** in fish meal, following the ordered z scores in the above Figure 104.

Table 36. Summary: evaluation of laboratory performance in fish meal

Parameters	Total participating laboratory	Evaluation results (number of laboratory, percentage in bracket)		
		Satisfactory	Questionable	Unsatisfactory
Moisture (g/100g)	76	67 (88.2%)	6 (7.9%)	3 (3.9%)
Nitrogen (g/100g)	67	60 (89.6%)	3 (4.5%)	4 (6.0%)
Fat (g/100g)	64	45 (70.3%)	9 (14.1%)	10 (15.6%)
Ash (g/100g)	68	61 (89.7%)	3 (4.4%)	4 (5.9%)
Calcium (mg/kg)	57	32 (56.1%)	8 (14.0%)	17 (29.8%)
Magnesium (mg/kg)	45	37 (82.2%)	2 (4.4%)	6 (13.3%)
Phosphorus (mg/kg)	42	28 (66.7%)	0 (0%)	14 (33.3%)
Sodium (mg/kg)	52	39 (75.0%)	6 (11.5%)	7 (13.5%)
Potassium (mg/kg)	48	41 (85.4%)	2 (4.2%)	5 (10.4%)
Iron (mg/kg)	52	36 (69.2%)	6 (11.5%)	10 (19.2%)
Zinc (mg/kg)	47	39 (83.0%)	1 (2.1%)	7 (14.9%)
Copper (mg/kg)	46	25 (54.3%)	14 (30.4%)	7 (15.2%)
Lead (mg/kg)	40	24 (60.0%)	4 (10.0%)	12 (30.0%)
Arsenic (mg/kg)	35	16 (45.7%)	4 (11.4%)	15 (42.9%)
Cholesterol (mg/100g)	31	24 (77.4%)	3 (9.7%)	4 (12.9%)
TVBN (mg/100g)	25	18 (72.0%)	1 (4.0%)	6 (24.0%)