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):

Spiked Rice Flour

by

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Table 50. Summary: assigned values of measurands for evaluation of testing parameters in spiked rice flour

Parameters	Method of assigned value[1]	x_{pt}	σ_{pt}	%RSD	$u_{x(pt)}$	$0.3\sigma_{pt}$	$u_{x(pt)}$ is negligible?
Moisture (g/100g)	x* & *s**	9.80	0.62	6.3	0.16	0.19	Yes, use z score
Arsenic (mg/kg)	x* & SDp	0.69	0.12	16.9	0.03	0.04	Yes, use z score
Cadmium (mg/kg)	x* & SDp	0.48	0.09	17.9	0.02	0.03	Yes, use z score
Lead (mg/kg)	x* & SDp	1.12	0.18	15.7	0.04	0.05	Yes, use z score
Mercury (mg/kg)	x* & SDp	0.45	0.08	18.0	0.02	0.02	Yes, use z score
Tin (mg/kg)	x* & $2SD_p$	10.27	2.31	22.5	0.64	0.69	Yes, use z score

[1] x* = Robust average derived from algorithm A of ISO 13538: 2015
*s** = Robust standard deviation derived from algorithm A of ISO 13538: 2015
SD_p = Predicted standard deviation from Horwitz equation

Table 51. Evaluation of laboratory performance on moisture analysis (mg/kg) in spiked rice flour

Lab Number	Moisture (g/100g)	MU (g/100g)	z score	Zeta score	Sample weight (g)	Temperature (^{o}C)	Time (Hours)	Method Reference
Assigned value obtained from robust average (x*) ± robust SD (s*) = 9.80 ± 0.62 g/100 g (CV 6.3%, n= 23) with u_{xpt} 0.16 mg/kg								
Acceptance criteria			**|z score| ≤ 2.00**	**|ζ score| ≤ 2.00**				
3	**9.16**	0.23	-1.04	**-3.25**	5.000 ± 0.001	105	18	AOAC International (2001)
14	**11.48**	0.89	**2.71**	**3.56**	2	130 ± 3	1	AOAC 925.10
19	**9.92**	-	0.20	-	5.000	105	3	AOAC 934.01
25	**7.72**	-	**-3.35**	-	4.0365 / 4.0393	103	4	Laboratory Handbook of Methods of Food Analysis, 3rd Ed, R. Lees
29	**10.08**	0.07	0.45	1.69	5.000	105	16	AOAC (1984)
31	**10.56**	0.37	1.23	**3.11**	3	105	3	SNI
38	**9.18**	0.20	-1.00	**-3.27**	2	130	1 until constant weight	AOAC 925.10, 19th Ed 2012
41	**10.28**	0.08	0.77	**2.90**	2.0	135 ± 2	2	AOAC (2016) 930.15
42	**10.00**	0.17	0.32	1.11	2	130	1	SNI 3549:2009 Lampiran A.10
48	**10.32**	0.11	0.83	**3.05**	2.000	130	1	SNI 3549 2009
49	**9.83**	0.49	0.05	0.10	2	130	1	AOAC 20th Ed 2016
50	**10.40**	0.23	0.97	**3.06**	2.1804	130	1	AOAC 925.10
54	**9.43**	0.08	-0.60	**-2.24**	1	105	5	AOAC 927.05
69	**10.37**	-	0.92	-				
72	**10.00**	0.01	0.32	1.25	2	130	1	AOAC 925.10
73A	**9.53**	0.38	-0.44	-1.09	5	105	3	FTC-01.01 (refers to AOAC 945.39)

Lab Number	Moisture (g/100g)	MU (g/100g)	z score	Zeta score	Sample weight (g)	Temperature (^{o}C)	Time (Hours)	Method Reference
Assigned value obtained from robust average (x*) ± robust SD (s*) = 9.80 ± 0.62 g/100 g (CV 6.3%, n= 23) with u_{xpt} 0.16 mg/kg								
74	**10.06**	-	0.42	-	5	105	3	SNI 01-2891-1992 (part 5.1)
75	**7.15**	0.06	**-4.27**	**-16.29**	2.00	105 ± 2	4	SNI 01-2891-1992 Butir 5.1
81	**10.30**	0.00	0.81	**3.13**	2.0293	130	1 then 0.5 until <0.005 mg diff	AOAC 925.10
82A	**9.37**	0.13	-0.69	**-2.49**	1.0	105	7.5	Drying Oven
82B	**9.37**	0.13	-0.69	**-2.49**	1	105	7.5	Drying Oven
85	**9.24**	0.01	-0.90	**-3.50**	2	105	3	SNI 01-2896-1992
99	**9.59**	0.81	-0.34	-0.48	3.00 ± 0.02	100 to 102	Min 8 and repeat until constant	AOAC 16th Ed 950.46

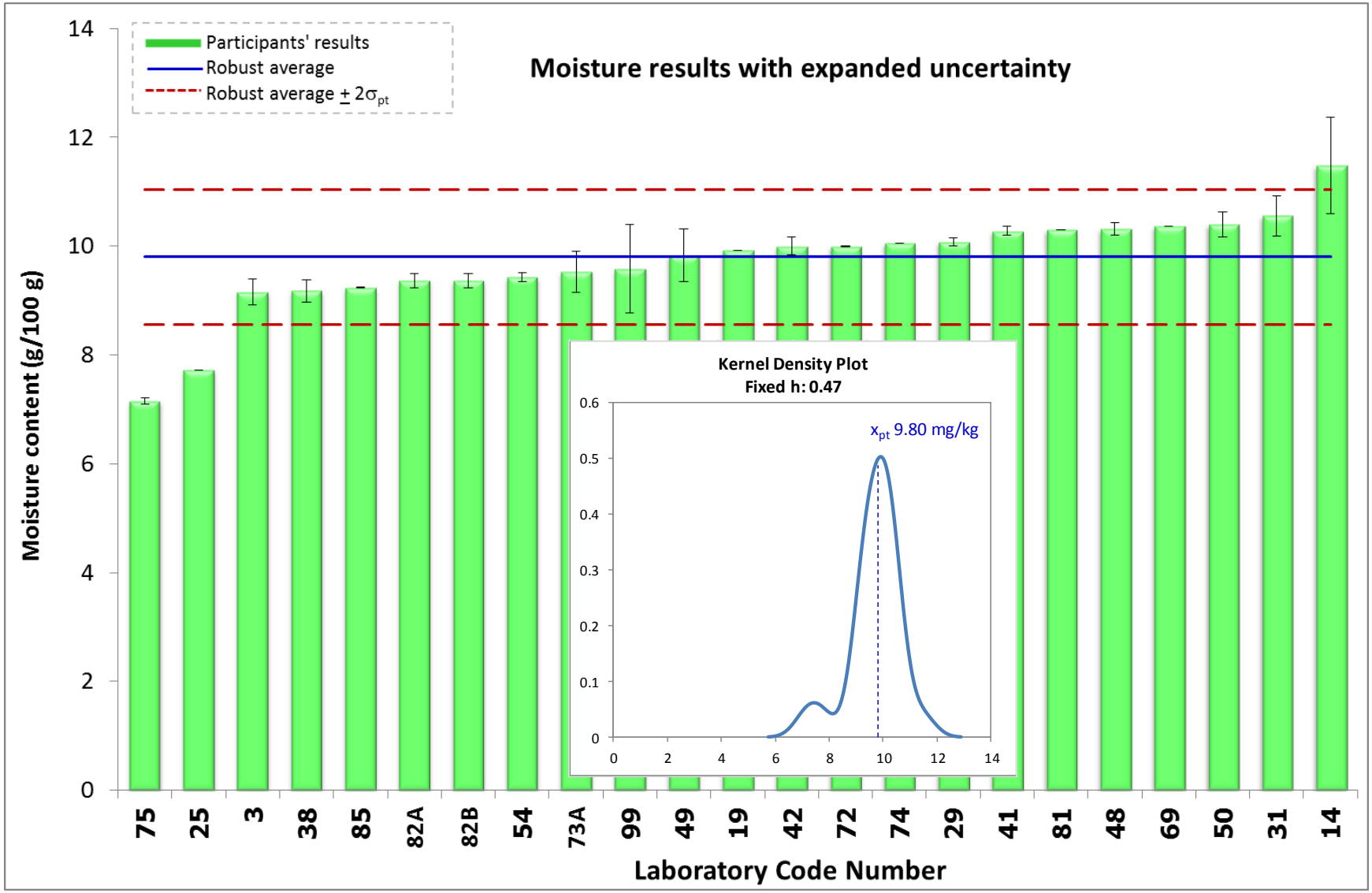

Figure 139. Distribution of **moisture** results (ascending order) in spiked rice flour with expanded uncertainty

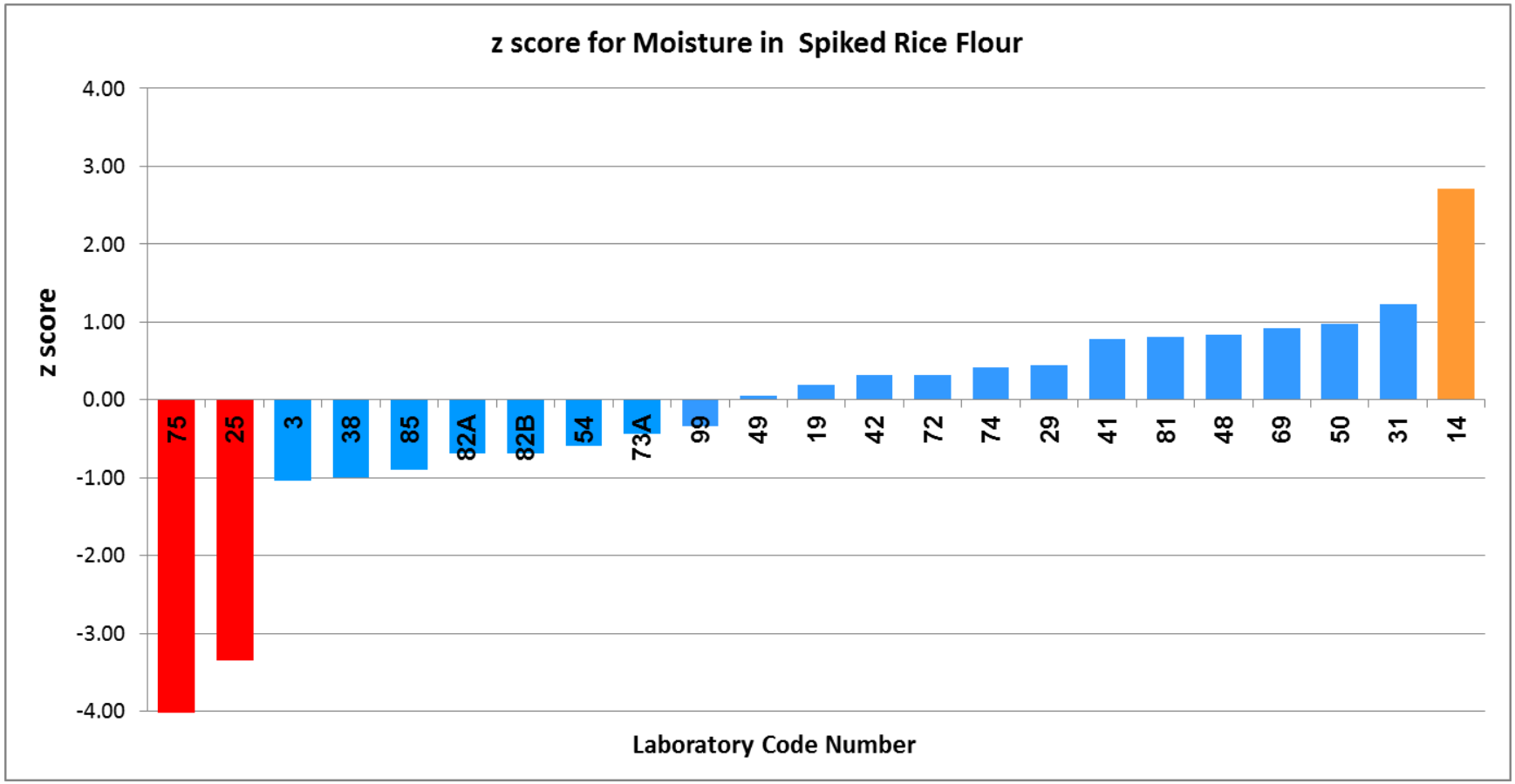

Figure 140. Plot of ordered *z* scores for **moisture** results in spiked rice flour

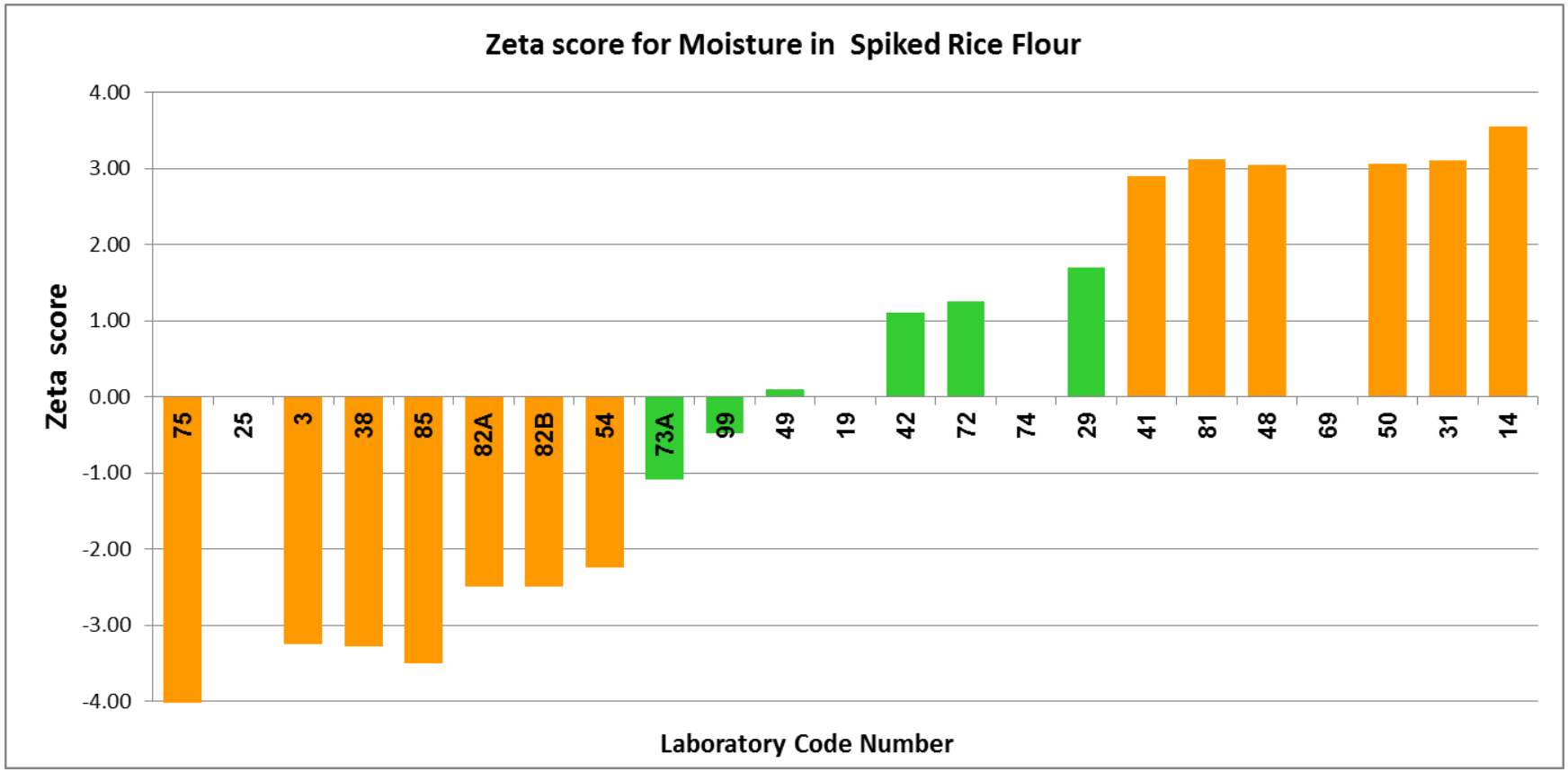

Figure 141. Plot of *Zeta* score for **moisture** spiked rice flour, following the ordered z scores in the above Figure 140.

Table 52. Evaluation of laboratory performance on arsenic analysis (mg/kg) in spiked rice flour

Lab Number	Arsenic (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight g	Digestion Technique	Digestion Medium	Instrument	Wavel-ength (nm or mass)	Recovery correction	Method Reference
Assigned value obtained from robust average (x) ± SDp from Horwitz' s equation = 0.69 ± 0.12 g/100 g (CV 17.4%, n= 26) with u_{xpt} 0.03 mg/kg*											
Acceptance criteria			|z score| ≤ 2.00	|ζ score| ≤ 2.00							
1	**0.65**	0.19	-0.33	-0.40	0.5071	Microwave, Nilestone Srl MA 176-0020SK15	-	ICP-MS 7800	-	N	AOAC 2015.01
13	**1.30**	-	**5.08**	-	0.5	Microwave	HNO3 10 mL + HCl 2 mL	ICP-MS Thermo Scientific (iCAP RQ)	M/z: As 75	N	Internal Method
15	**0.58**	-	-0.96	-	0.5	Ultrawave Digestion	5% HNO3 + 0.5% HCl	ICP-MS (7900 Agilent)	As 75	N	Based on USFDA 4.7 version 1.1
16	**0.54**	0.05	-1.25	**-3.84**	2	Microwave	HNO3+H2O2	ICP-MS 7700X Agilent	-	N	In-house Method
18	**0.93**	-	2.00	-	2.0	Dry Ashing (Wet	HCl (HNO3 for Hg, Sn)	Agilent for Hg, As)	As 193.7	N	SNI 19-2896-1998
25	**1.45**	0.07	**6.33**	**16.76**	5.0160 / 5.0123 (Hg 0.2244 / 0.2245	HNO3-HCl (Hg Cold Vapour)	Water	ICP-OES (Hg Hydra IIAA)	As 188.98		USEPA Method 3050B (Hg EPA-SW 846, Method 7470A
27	**0.14**	0.02	**-4.58**	**-17.39**	0.1	Microwave digestion	HNO3 65% suprapure	ICP-MS 7700x Agilent	-	N	Gray J.P., Mindak R.W., Cheng J., 2015, Elemental Analysis Manual for Food and Related Product, USFDA Version 1.1

Lab Number	Arsenic (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight g	Digestion Technique	Digestion Medium	Instrument	Wavel-ength (nm or mass)	Recovery correctio n	Method Reference
Assigned value obtained from robust average (x) ± SDp from Horwitz' s equation = 0.69 ± 0.12 g/100 g (CV 17.4%, n= 26) with u_{xpt} 0.03 mg/kg*											
31	**0.10**	0.01	**-4.93**	**-19.48**	5 (1 for Hg)	Dry Ashing (Microwave for Hg)	-	AAS (Cold Vapour for Hg)	-	N	AOAC 999.11, (Hg: SNI 01-3751, As: AOAC 986.15, Sn: SNI 3551:2012)
32	**0.55**	0.03	-1.19	-4.29	0.5117 (As)	Microwave	HNO3	Hydride Vapour-AAS, Perkin Elmer	193.7	N	Modified AOAC 986.15
38	**0.33**	0.02	**-3.03**	**-11.18**	1.000	Dry Ashing	0.1 M HNO3	Flame AAS, Shimadzu AA6300	As 193.70		Modified AOAC 999.11
39	**0.71**	0.11	0.13	0.24	0.5	Microwave		GF-AAS (Hg: Mercury analyzer, As: Hydride AAS)	As 193.7	Y	AOAC 999.10 (Hg: 977.15, As: plus 986.15)
43	**0.60**	0.04	-0.78	-2.58	0.5	Microwave	HNO3	ICP-OES, ICP-MS	As m/z 75	N	AOAC
47	**1.08**	0.20	**3.25**	**3.74**	5 (2 for As)	Dry Ashing (Colorimetric for As)	Nitric Acid (H2SO4-H2O2 for As)	AAS - Shimadzu 7000 (Uv-Vis Hanon i3 for As)	As 535	-	AOAC 999.11 (Modified method for As)
48	**< 0.020**	-	**-5.67**	-	5 (0.5 for Hg and As)	Dry Digestion (Hg, As Microwave Digestion)	-	AA800 Perkin Elmer	As 193.7	N	MU-03/20 (AAS)
49	**0.60**	0.03	-0.73	-2.59	2	Dry Ashing	6 M HCl	ICP-OES 5110 Agilent Technologies	As 188.980	N	AOAC 20th Ed 2016
53	**0.71**	0.01	0.17	0.66	0.3	Microwave	4 mL HNO3, 1 mL HCl, 1 mL H2O2	ICPMS Thermo	-	-	In house method
54	**0.57**	0.05	-0.97	-2.97	1	Microwave digestion	HNO3 / H2O2	ICP / Shimadzu	As 193.7	N	AOAC 984.27

Lab Number	Arsenic (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight g	Digestion Technique	Digestion Medium	Instrument	Wavel-ength (nm or mass)	Recovery correctio n	Method Reference
Assigned value obtained from robust average (x) ± SDp from Horwitz' s equation = 0.69 ± 0.12 g/100 g (CV 17.4%, n= 26) with u_{xpt} 0.03 mg/kg*											
58	**0.83**	0.07	1.18	3.06	1.0	Acid Digestion	HNO3, H2O2	AAS-Hydride for As, Hg	As 189	-	Acid Digestion and Quantitation by AAS-hydride for As, Hg
59	**0.40**	0.00	**-2.42**	**-9.66**	As: 12.5	Dry Ashing	-	AAS, Shimadzu	As 193.7,	Y	As: IK A2-LM10 (AAS)
69	**0.81**	-	1.03	-	-	-	-	-	-	-	-
72	**0.95**	0.08	**2.14**	**5.14**	4 (Hg 0.5)	Ashing (Hg Acid Digestion)	HNO3 (Hg Aqua regia)	ICP-OES, JY Ultima (Hg FIMS 400, Perkin Elmer)	As 193.695,	N	AOAC 999.11 (Hg EPA 7471)
75	**0.74**	0.04	0.43	1.39	1	Wet digestion (hot block)	HNO3 + H2O2	ICP-OES Agilent 5100, ICP-MS Agilent 7700x	As 193.696	N	In House Method ICP-MS & ICP-OES
76	**0.87**	0.17	1.50	2.00	-	-	-	-	-	-	-
82A	**0.50**	0.09	-1.58	**-3.51**	0.250	none	none	HPGe detector, Canberra	-	N	Neutron Activation Analysis (NAA)
84	**0.66**	0.07	-0.22	-0.61	0.5	Microwave Digestion	HNO3 / H2O2	ICP-MS	-	N	AOAC 999.10:2005
88	**0.72**	0.00	0.28	1.13	0.3 (Cu, Zn 3)	Microwave	H2O2 2 ml + HNO3 8 mL \	AAS GBC Hydride vapour (Cu, Zn: AAS GBC Flame, Cd: AAS-GF)	As 193.70	N	In house method (AAS)
99	**0.91**	-	1.81	-	0.3 ± 0.001	Microwave	Nitric Acid & Hydrogen Peroxide	Digestion (MULTI GO Anton Paar) Determinatio n (ICP/MS, PE)	Refer Mass each element	N	EPA 3015A

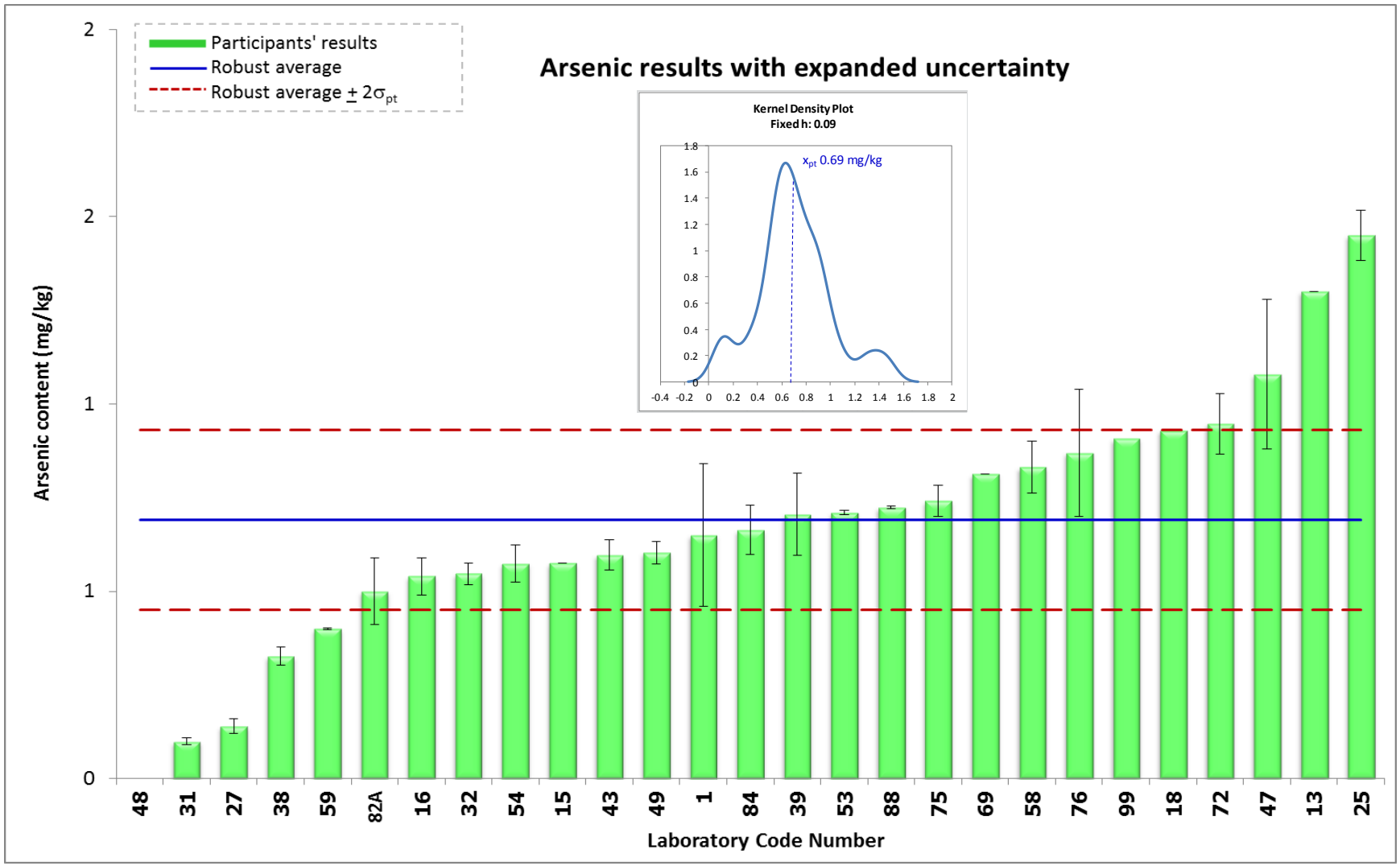

Figure 142. Distribution of **arsenic** results (ascending order) in spiked rice flour with expanded uncertainty

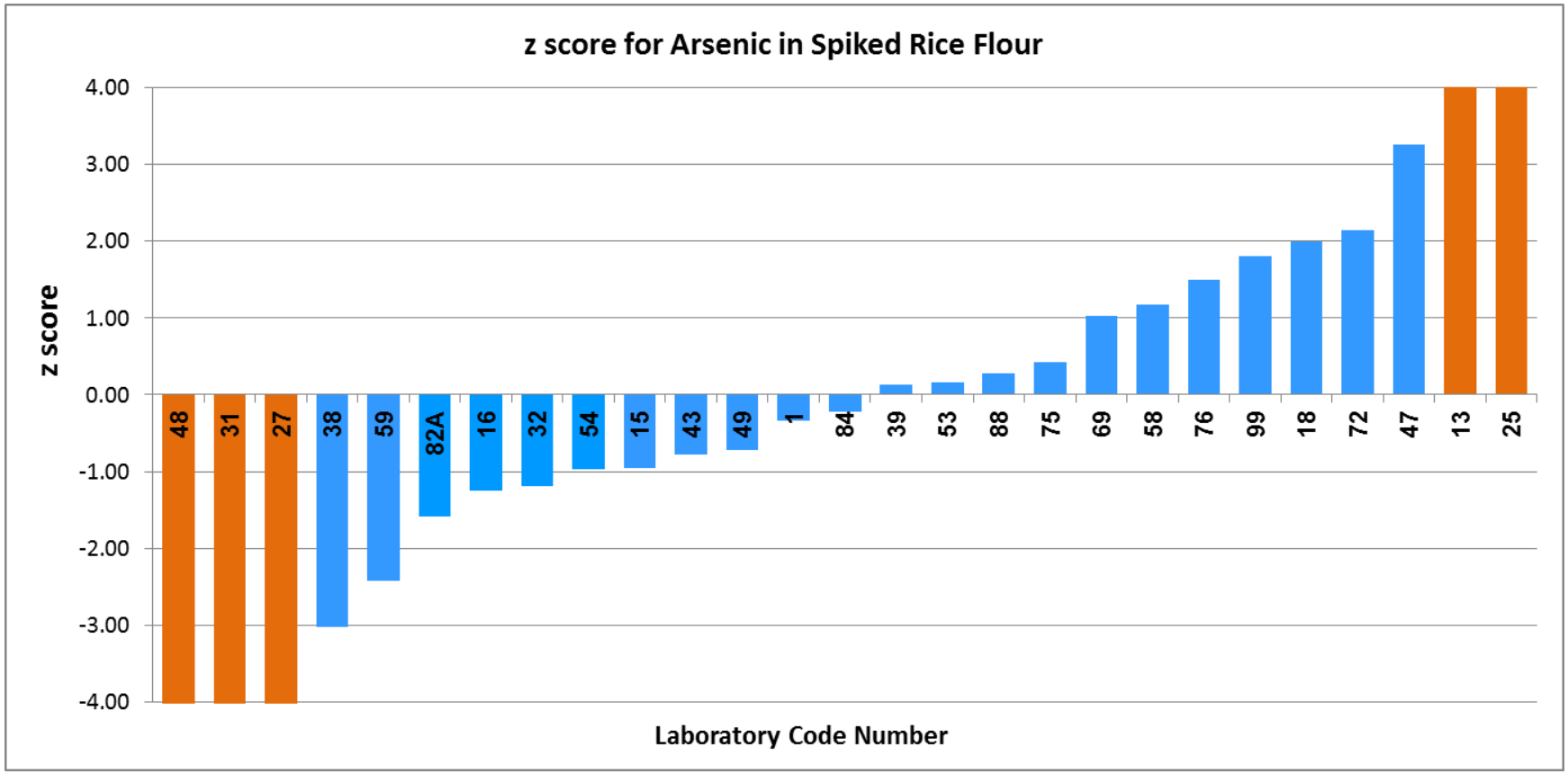

Figure 143. Plot of ordered *z* scores for **arsenic** results in spiked rice flour

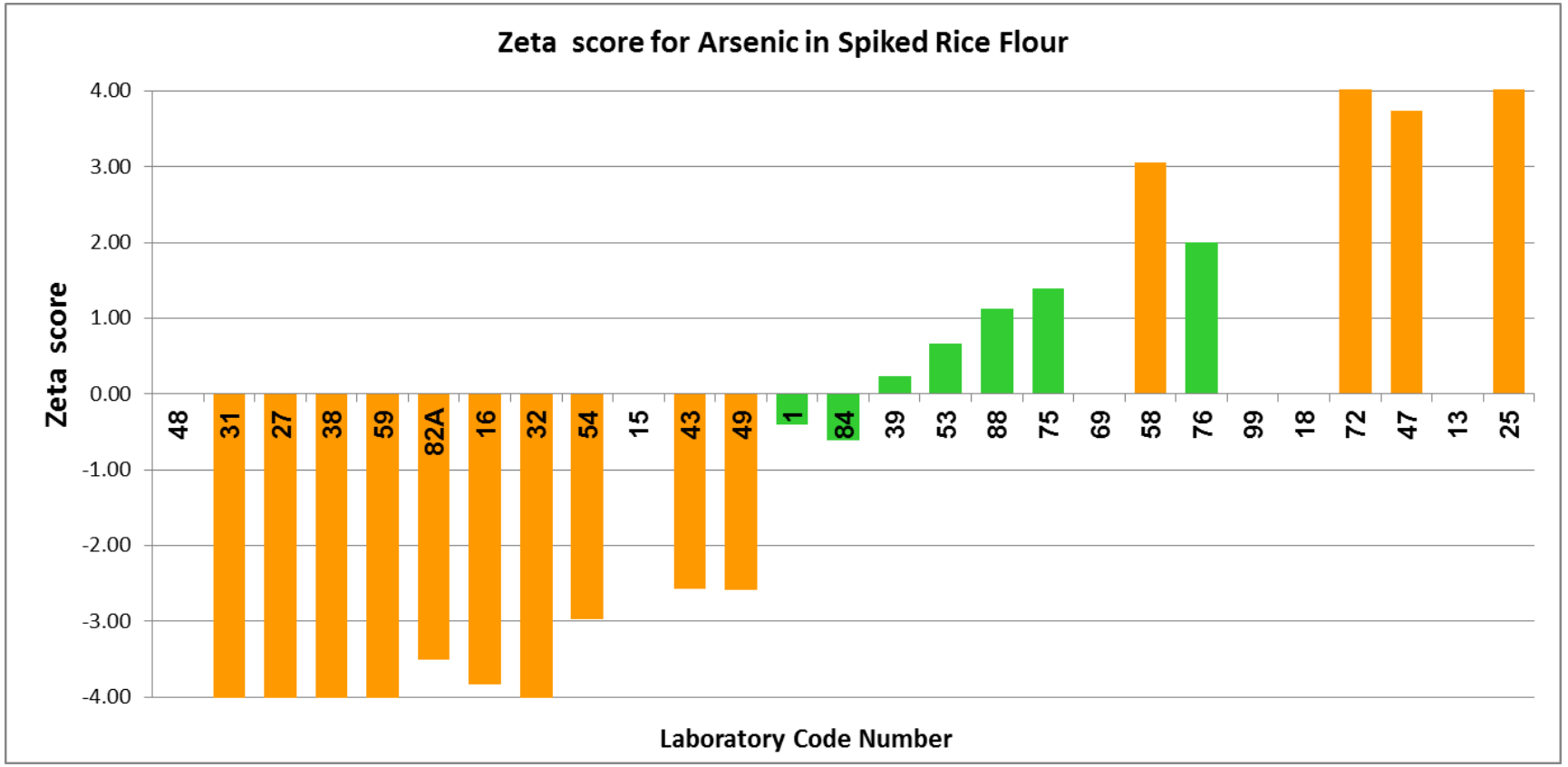

Figure 144. Plot of *Zeta* score for **arsenic** spiked rice flour, following the ordered z scores in the above Figure 143.

Table 53. Evaluation of laboratory performance on **cadmium** analysis (mg/kg) in spiked rice flour

Lab Number	Cadmium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight g	Digestion Technique	Digestion Medium	Instrument	Wave-length (nm or mass)	Recovery correction	Method Reference
Assigned value obtained from robust average (x) ± SDp from Horwitz' s equation = 0.48 ± 0.09 g/100 g (CV 18.8%, n= 29) with u_{xpt} 0.02 mg/kg*											
Acceptance criteria			|z score| ≤ 2.00	|ζ score| ≤ 2.00							
1	**0.59**	0.11	1.23	1.86	0.5071	Microwave, Nilestone Srl MA 176-0020SK15	-	ICP-MS 7800	-	N	AOAC 2015.01
13	**0.28**	-	**-2.24**	-	0.5	Microwave	HNO3 10 mL + HCl 2 mL	ICP-MS Thermo Scientific (iCAP RQ)	Cd 111	N	Internal Method
14	**0.50**	-	0.18	-	0.5	Wet Digestion	HNO3, H2O2	ICP Horiba Jobin Yvon	Cd 228.802	Y	AOAC 999.10
15	**0.56**	-	0.86	-	0.5	Ultrawave Digestion	5% HNO3 + 0.5% HCl	ICP-MS (7900 Agilent)	Cd 111	N	Based on USFDA 4.7 version 1.1
16	**0.46**	0.04	-0.22	-0.71	2	Microwave	HNO3+H2O 2	ICP-MS 7700X Agilent	-	N	In-house Method
18	**1.11**	-	**7.00**	-	2.0	Dry Ashing	HCl (HNO3 for Hg, Sn)	ICP-OES Agilent	-	N	SNI 19-2896-1998
25	**0.52**	0.07	0.40	0.91	5.0160 / 5.0123 (Hg 0.2244 / 0.2245	HNO3-HCl (Hg Cold Vapour)	Water	ICP-OES (Hg Hydra IIAA)	Cd 226.502		USEPA Method 3050B (Hg EPA-SW 846, Method 7470A
27	**0.18**	0.02	**-3.33**	**-13.42**	0.1	Microwave digestion	HNO3 65% suprapure	ICP-MS 7700x Agilent	-	N	Gray J.P., Mindak R.W., Cheng J., 2015, Elemental Analysis Manual for Food and Related Product, USFDA Version 1.1
31	**0.33**	-	-1.63	-	5 (1 for Hg)	Dry Ashing (Microwave for Hg)	-	AAS (Cold Vapour for Hg)	-	N	AOAC 999.11, (Hg: SNI 01-3751, As: AOAC 986.15, Sn: SNI 3551:2012)

Lab Number	Cadmium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight g	Digestion Technique	Digestion Medium	Instrument	Wave-length (nm or mass)	Recovery correction	Method Reference
Assigned value obtained from robust average (x) ± SDp from Horwitz' s equation = 0.48 ± 0.09 g/100 g (CV 18.8%, n= 29) with u_{xpt} 0.02 mg/kg*											
38	< 0.150	-	-4.50	-	1.000	Dry Ashing	0.1 M HNO3	Flame AAS, Shimadzu AA6300	Cd 228.8		Modified AOAC 999.11
39	**0.47**	0.03	-0.17	-0.64	0.5	Microwave		GF-AAS	Cd 228.8	Y	AOAC 999.10 (Hg: 977.15, As: plus 986.15)
42	0.71	0.01	2.59	10.99	10 (0.5 for Hg, Sn)	Dry Ashing (Microwave Digestion for Hg, Sn)	HNO3-HCl (HNO3 for Hg, Sn)	Pb, Cd, Sn: GFAAS Agilent 240 FS	Cd 228.8	N	AOAC 999.11.2005 (Hg: SNI 3549:2009. Lampiran A.16, Sn: BS EN 15764:2009)
43	**0.51**	0.03	0.34	1.24	0.5	Microwave	HNO3	ICP-OES, ICP-MS	Cd m/z 111	N	AOAC
44	**0.51**	0.53	0.37	0.12	1.0	Dry Ashing	-	AAS, Thermoscientific	Cd 228.8	N	AOAC 19th Ed, 2012
47	**0.43**	0.03	-0.59	-2.21	5 (2 for As)	Dry Ashing	Nitric Acid	AAS - Shimadzu 7000	Cd 228.8	-	AOAC 999.11 (Modified method for As)
48	0.72	0.07	2.70	6.37	5 (0.5 for Hg and As)	Dry Digestion (Hg, As Microwave Digestion)	-	AA800 Perkin Elmer	Cd 228.8	N	MU-03/20 (AAS)
49	**0.48**	0.02	-0.04	-0.17	2	Dry Ashing	6 M HCl	ICP-OES 5110 Agilent Technologies	Cd 226.502	N	AOAC 20th Ed 2016
53	**0.47**	0.04	-0.09	-0.30	0.3	Microwave	4 mL HNO3, 1 mL HCl, 1 mL H2O2	ICPMS Thermo	-	-	In house method
54	**0.48**	0.01	0.01	0.05	1	Microwave digestion	HNO3 / H2O2	ICP / Shimadzu	-	N	AOAC 984.27
58	**0.47**	0.06	-0.16	-0.39	1.0	Acid Digestion	HNO3, H2O2	ICP-OES	-	-	Acid Digestion and Quantitation by ICP-OES

Lab Number	Cadmium (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight g	Digestion Technique	Digestion Medium	Instrument	Wave-length (nm or mass)	Recovery correction	Method Reference
Assigned value obtained from robust average (x) ± SDp from Horwitz' s equation = 0.48 ± 0.09 g/100 g (CV 18.8%, n= 29) with u_{xpt} 0.02 mg/kg*											
59	**0.39**	0.09	-1.00	-1.83	Pb, Cd, Sn: 2.5, Hg: 1, As: 12.5	Dry Ashing	-	AAS, Shimadzu	Cd 228.8	Y	Pb+Cd: SNI 3751:2009 point A.14.1
69	**0.39**	-	-0.99	-	-	-	-	-	-	-	-
72	**0.18**	0.02	**-3.31**	**-13.33**	4 (Hg 0.5)	Ashing (Hg Acid Digestion)	HNO3 (Hg Aqua regia)	ICP-OES, JY Ultima (Hg FIMS 400, Perkin Elmer)	Cd 228.802	N	AOAC 999.11 (Hg EPA 7471)
75	**0.47**	0.02	-0.09	-0.36	1	Wet digestion (hot block)	HNO3 + H2O2	ICP-MS Agilent 7700x	111 Cd	N	In House Method ICP-MS & ICP-OES
76	**0.53**	0.02	0.56	**2.24**	-	-	-	-	-	-	-
82B	**0.49**	0.08	0.14	0.30	1.00	Microwave	Nitric Acid	Pb, Cd, As: AAS, Agilent,	-	Y	Pb, Cd, As: Graphite SSA
84	**0.56**	0.06	0.91	**2.38**	0.5	Microwave Digestion	HNO3 / H2O2	ICP-MS	-	N	AOAC 999.10:2005
88	**0.07**	0.00	**-4.51**	**-20.27**	0.3 (Cu, Zn 3)	Microwave (Cu, Zn Dry Ashing)	H2O2 2 ml + HNO3 8 mL (Cu, Zn: HNO3 10 mL)	Cd: AAS-GF	Cd 228.8	N	In house method (AAS)
91	**0.58**	-	1.07	-	-	-	-	-	-	-	-
99	**0.59**	-	1.21	-	0.3 ± 0.001	Microwave	Nitric Acid & Hydrogen Peroxide	Digestion (MULTI GO Anton Paar) Determination (ICP/MS, PE)	Refer Mass each element	N	EPA 3015A

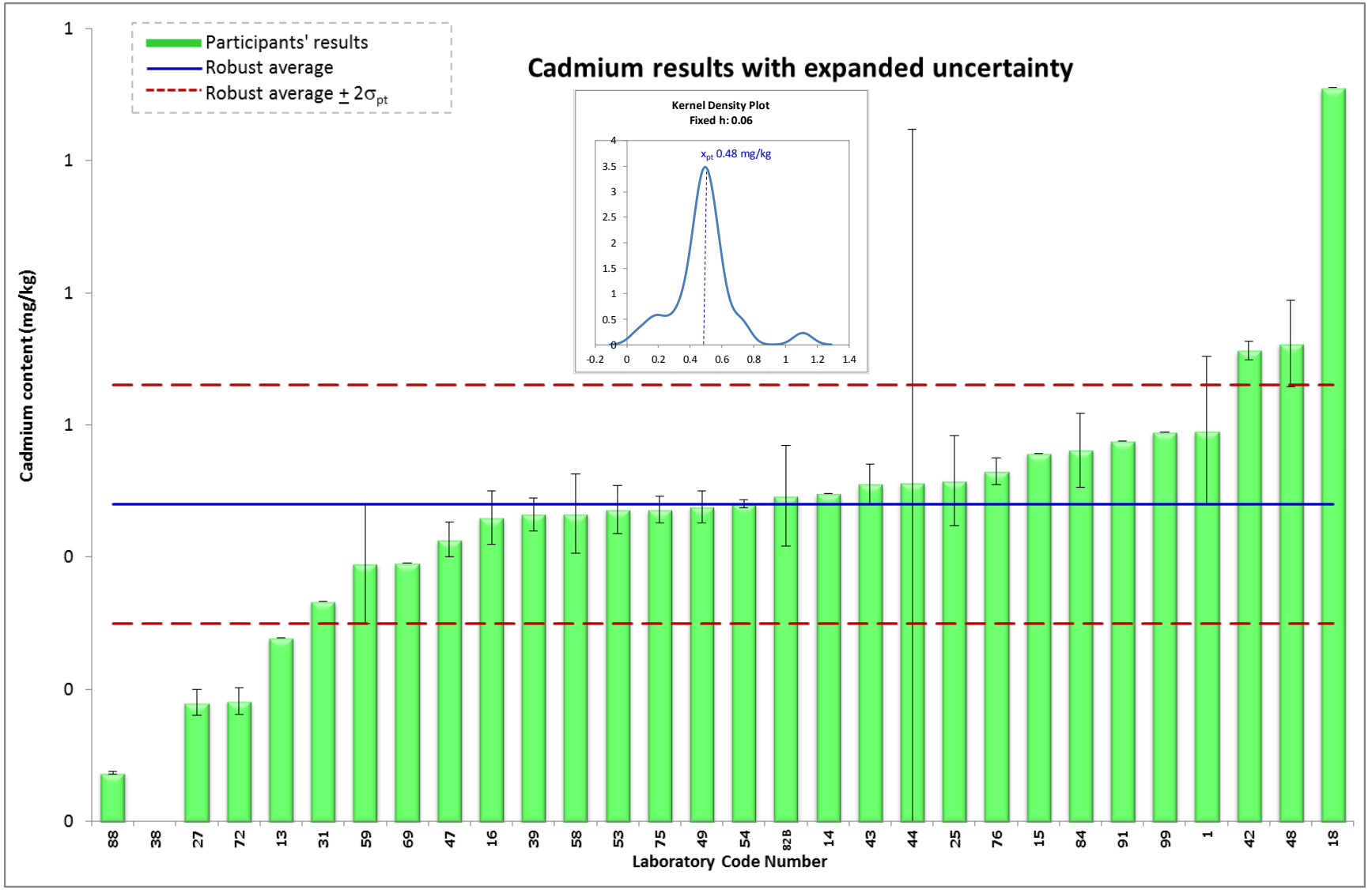

Figure 145. Distribution of **cadmium** results (ascending order) in spiked rice flour with expanded uncertainty

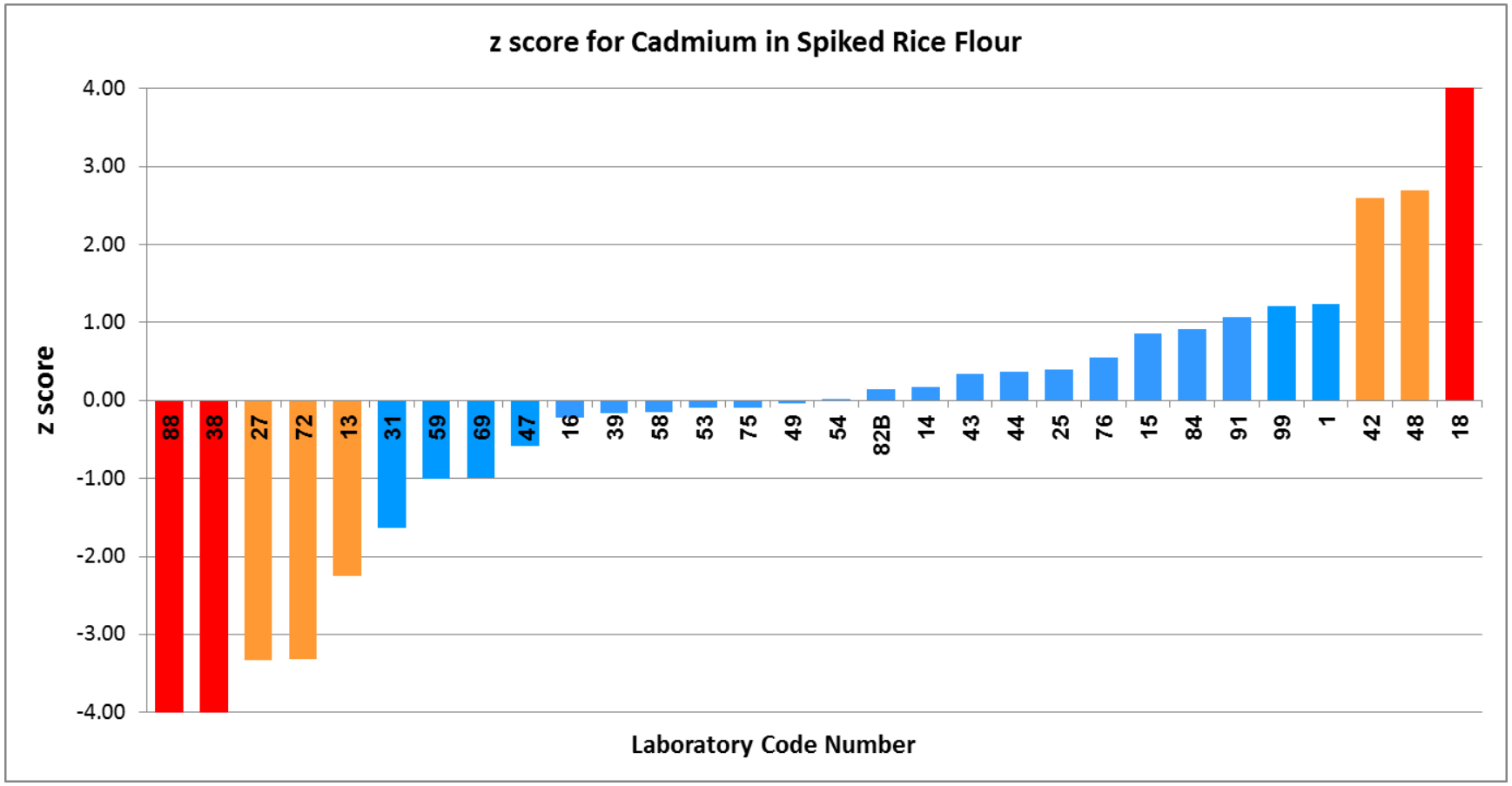

Figure 146. Plot of ordered *z* scores for **cadmium** results in spiked rice flour

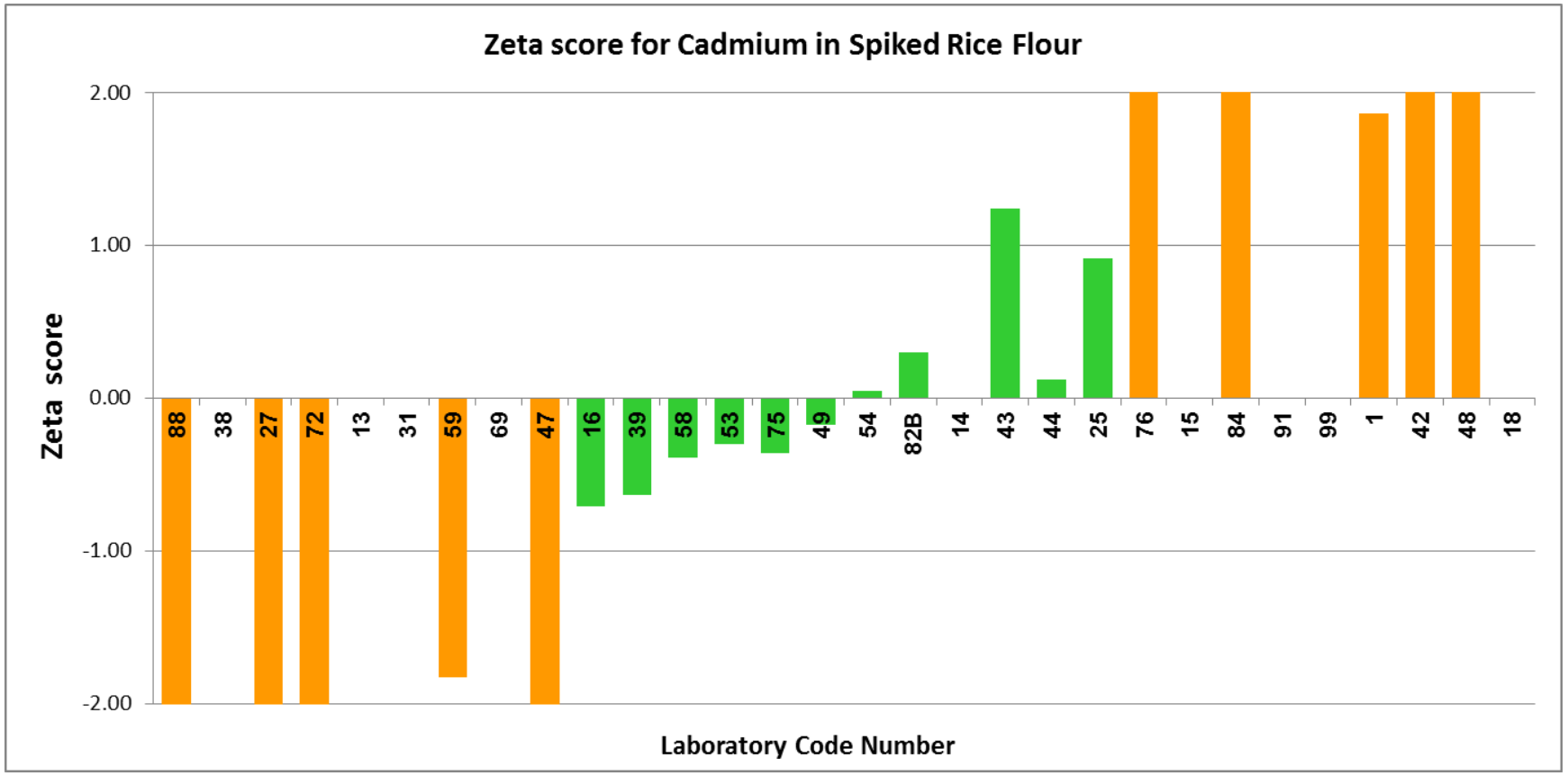

Figure 147. Plot of *Zeta* score for **cadmium** spiked rice flour, following the ordered z scores in the above Figure 146.

Table 54. Evaluation of laboratory performance on **lead** analysis (mg/kg) in spiked rice flour

Lab Number	Lead (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight g	Digestion Technique	Digestion Medium	Instrument	Wave-length (nm or mass)	Recovery correction	Method Reference
Assigned value obtained from robust average (x) ± SDp from Horwitz' s equation = 1.12 ± 0.18 g/100 g (CV 16.1%, n= 29) with u_{xpt} 0.04 mg/kg*											
Acceptance criteria			|z score| ≤ 2.00	|ζ score ≤ 2.00							
1	**1.05**	0.33	-0.37	-0.38	0.5071	Microwave, Nilestone Srl MA 176-0020SK15	-	ICP-MS 7800	-	N	AOAC 2015.01
13	**1.49**	-	**2.06**	-	0.5	Microwave	HNO3 10 mL + HCl 2 mL	ICP-MS Thermo Scientific (iCAP RQ)	M/z: Pb 208	N	Internal Method
14	**0.55**	-	**-3.19**	-	0.5	Wet Digestion	HNO3, H2O2	ICP Horiba Jobin Yvon	Pb 220	Y	AOAC 999.10
15	**1.19**	-	0.39	-	0.5	Ultrawave Digestion	5% HNO3 + 0.5% HCl	ICP-MS (7900 Agilent)	Pb 238	N	Based on USFDA 4.7 version 1.1
16	**0.94**	0.09	-1.00	**-2.99**	2	Microwave	HNO3+H2O2	ICP-MS 7700X Agilent	-	N	In-house Method
18	**3.10**	-	**11.00**	-	2.0	Dry Ashing (Wet Digestion for Hg, Sn)	HCl (HNO3 for Hg, Sn)	ICP-OES	-	N	SNI 19-2896-1998
25	**0.90**	0.07	-1.22	**-4.19**	5.0160 / 5.0123 (Hg 0.2244 / 0.2245	HNO3-HCl (Hg Cold Vapour)	Water	ICP-OES (Hg Hydra IIAA)	Pb 220.353	-	USEPA Method 3050B (Hg EPA-SW 846, Method 7470A
26	**1.12**	0.09	0.00	0.00	5.0	Dry Ashing (Wet Ashing for Hg)	Water & HCl (1+1) (Nitric:Perchloric (1+1) for Hg)	AAS Shimadzu AA-7000-	-	N	AOAC No. 999.11 using flame AAS, 977.15 for Hg

Lab Number	Lead (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight g	Digestion Technique	Digestion Medium	Instrument	Wave-length (nm or mass)	Recovery correction	Method Reference
Assigned value obtained from robust average (x) ± SDp from Horwitz' s equation = 1.12 ± 0.18 g/100 g (CV 16.1%, n= 29) with u_{xpt} 0.04 mg/kg*											
27	**0.64**	0.05	**-2.67**	**-10.18**	0.1	Microwave digestion	HNO3 65% suprapure	ICP-MS 7700x Agilent	-	N	Gray J.P., Mindak R.W., Cheng J., 2015, Elemental Analysis Manual for Food and Related Product, USFDA Version 1.1
31	**0.68**	0.00	**-2.44**	**-10.97**	5 (1 for Hg)	Dry Ashing (Microwave for Hg)	-	AAS (Cold Vapour for Hg)	-	N	AOAC 999.11, (Hg: SNI 01-3751, As: AOAC 986.15, Sn: SNI 3551:2012)
38	**2.77**	0.31	**9.17**	**10.21**	1.000	Dry Ashing	0.1 M HNO3	Flame AAS, Shimadzu AA6300	Pb 283.30	-	Modified AOAC 999.11
39	**0.66**	0.05	**-2.53**	**-9.83**	0.5	Microwave	-	GF-AAS	Pb 283.3	Y	AOAC 999.10 (Hg: 977.15, As: plus 986.15)
42	**1.84**	0.11	**4.00**	**10.52**	10 (0.5 for Hg, Sn)	Dry Ashing (Microwave Digestion for Hg, Sn)	HNO3-HCl (HNO3 for Hg, Sn)	Pb, Cd, Sn: GFAAS Agilent 240 FS	Pb 217.0	N	AOAC 999.11.2005 (Hg: SNI 3549:2009. Lampiran A.16, Sn: BS EN 15764:2009)
43	**1.12**	0.05	0.00	0.00	0.5	Microwave	HNO3	ICP-OES, ICP-MS	Pb m/z 208	N	AOAC
44	**1.32**	0.06	1.11	**3.93**	1.0	Dry Ashing		AAS, Thermoscientific	Pb 217.0	N	AOAC 19th Ed, 2012
47	**0.99**	0.03	-0.75	**-3.21**	5 (2 for As)	Dry Ashing	Nitric Acid	AAS - Shimadzu 7000	Pb 217	-	AOAC 999.11
48	**9.97**	1.12	**49.17**	**15.79**	5 (0.5 for Hg and As)	Dry Digestion	-	AA800 Perkin Elmer	Pb 283.3	N	MU-03/20 (AAS)
49	**1.10**	0.06	-0.11	-0.40	2	Dry Ashing	6 M HCl	ICP-OES 5110 Agilent Technologies	Pb 220.353	N	AOAC 20th Ed 2016

Lab Number	Lead (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight g	Digestion Technique	Digestion Medium	Instrument	Wave-length (nm or mass)	Recovery correction	Method Reference
Assigned value obtained from robust average (x) ± SDp from Horwitz' s equation = 1.12 ± 0.18 g/100 g (CV 16.1%, n= 29) with u_{xpt} 0.04 mg/kg*											
53	**0.99**	0.05	-0.74	**-2.85**	0.3	Microwave	4 mL HNO3, 1 mL HCl, 1 mL H2O2	ICPMS Thermo	-	-	In house method
54	**1.23**	0.21	0.61	0.98	1	Microwave digestion	HNO3 / H2O2	ICP / Shimadzu	Pb 220.353	N	AOAC 984.27
58	**0.50**	0.08	**-3.47**	**-11.05**	1.0	Acid Digestion	HNO3, H2O2	ICP-OES	-	-	Acid Digestion and Quantitation by ICP-OES
59	**< 0.05**		**-6.08**	-	Pb, Cd, Sn: 2.5	Dry Ashing	-	AAS, Shimadzu	Pb 283	Y	Pb+Cd: SNI 3751:2009 point A.14.1,
69	**0.96**	-	-0.87	-	-	-	-	-	-	-	-
72	**0.23**	0.02	**-4.96**	**-21.63**	4 (Hg 0.5)	Ashing (Hg Acid Digestion)	HNO3 (Hg Aqua regia)	ICP-OES, JY Ultima	Pb 220.353	N	AOAC 999.11 (Hg EPA 7471)
75	**1.04**	0.03	-0.47	**-2.03**	1	Wet digestion (hot block)	HNO3 + H2O2	ICP-OES Agilent 5100, ICP-MS Agilent 7700x	208 Pb	N	In House Method ICP-MS & ICP-OES
76	**0.97**	0.11	-0.83	**-2.21**	-	-	-	-	-	-	-
81	**1.10**	-	-0.11	-	mean 0.5229 (Pb)	Pb: Microwave Digestion	Pb: conc HNO3 + 30% H2O2	Pb: Thermo iCe 3500	Pb 217.0	N	AOAC 999.10 Mod (Pb)
82B	**1.11**	0.27	-0.06	-0.07	1.00	Microwave	Nitric Acid	Pb, Cd, As: AAS, Agilent,	-	Y	Pb, Cd, As: Graphite SSA, Cu: Flame SSA
84	**0.95**	0.10	-0.94	**-2.74**	0.5	Microwave Digestion	HNO3 / H2O2	ICP-MS	-	N	AOAC 999.10:2005
91	**3.56**	-	**13.56**	-	-	-	-	-	-	-	-
99	**1.55**	-	**2.39**	-	0.3 ± 0.001	Microwave	Nitric Acid & Hydrogen Peroxide	Determination (ICP/MS, PE)	Refer Mass each element	N	EPA 3015A

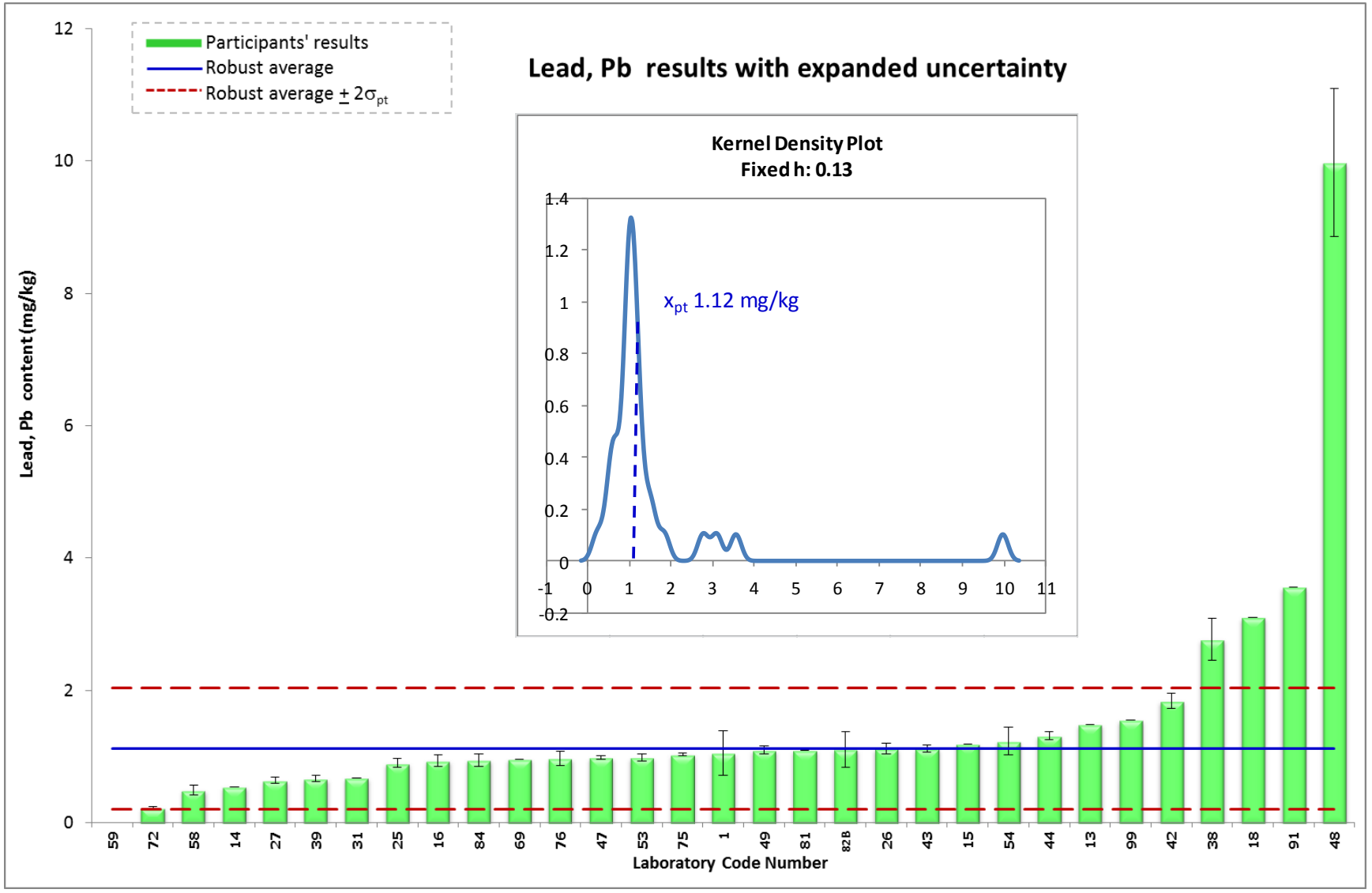

Figure 148. Distribution of **lead** results (ascending order) in spiked rice flour with expanded uncertainty

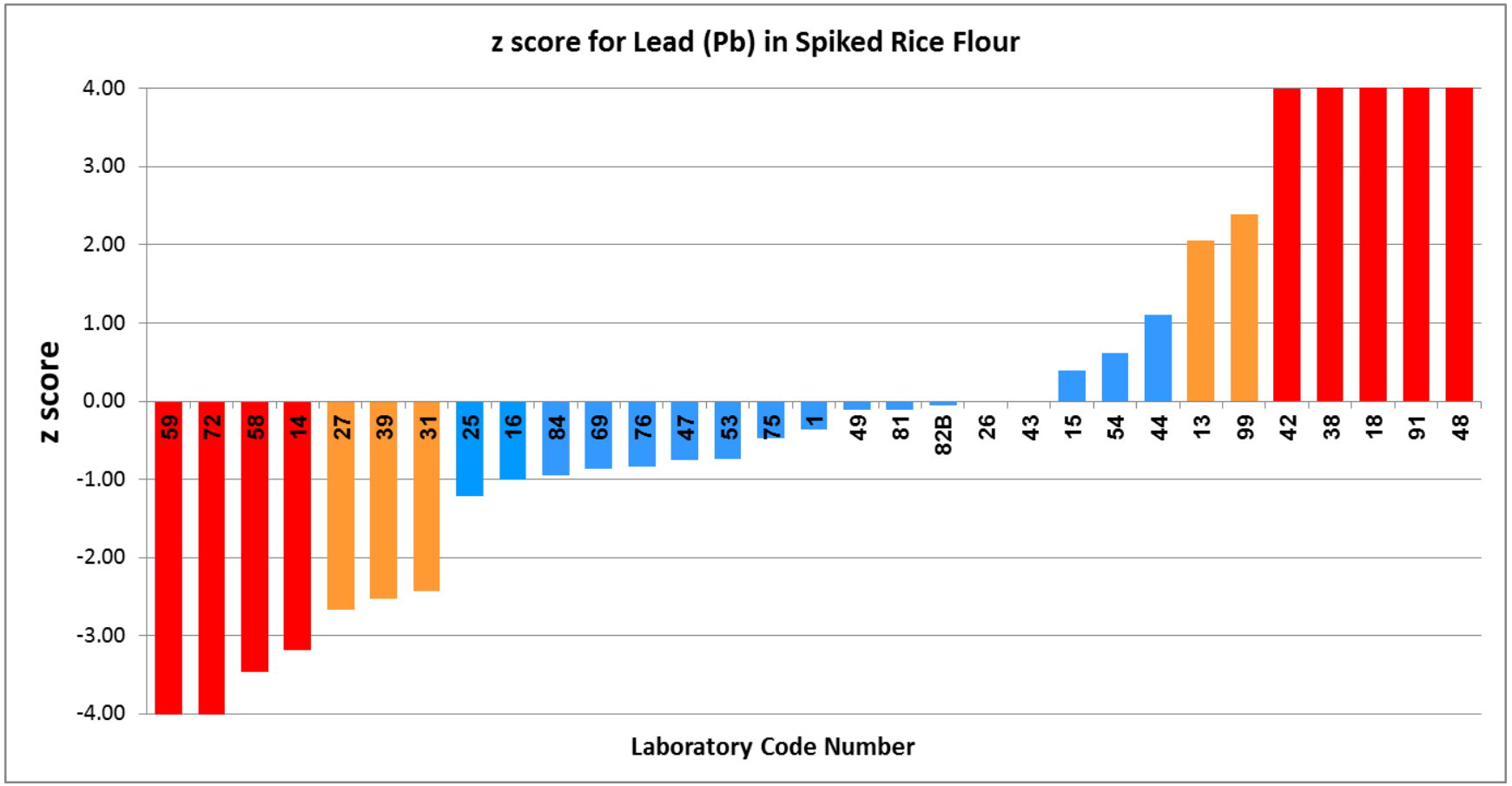

Figure 149. Plot of ordered *z* scores for **lead** results in spiked rice flour

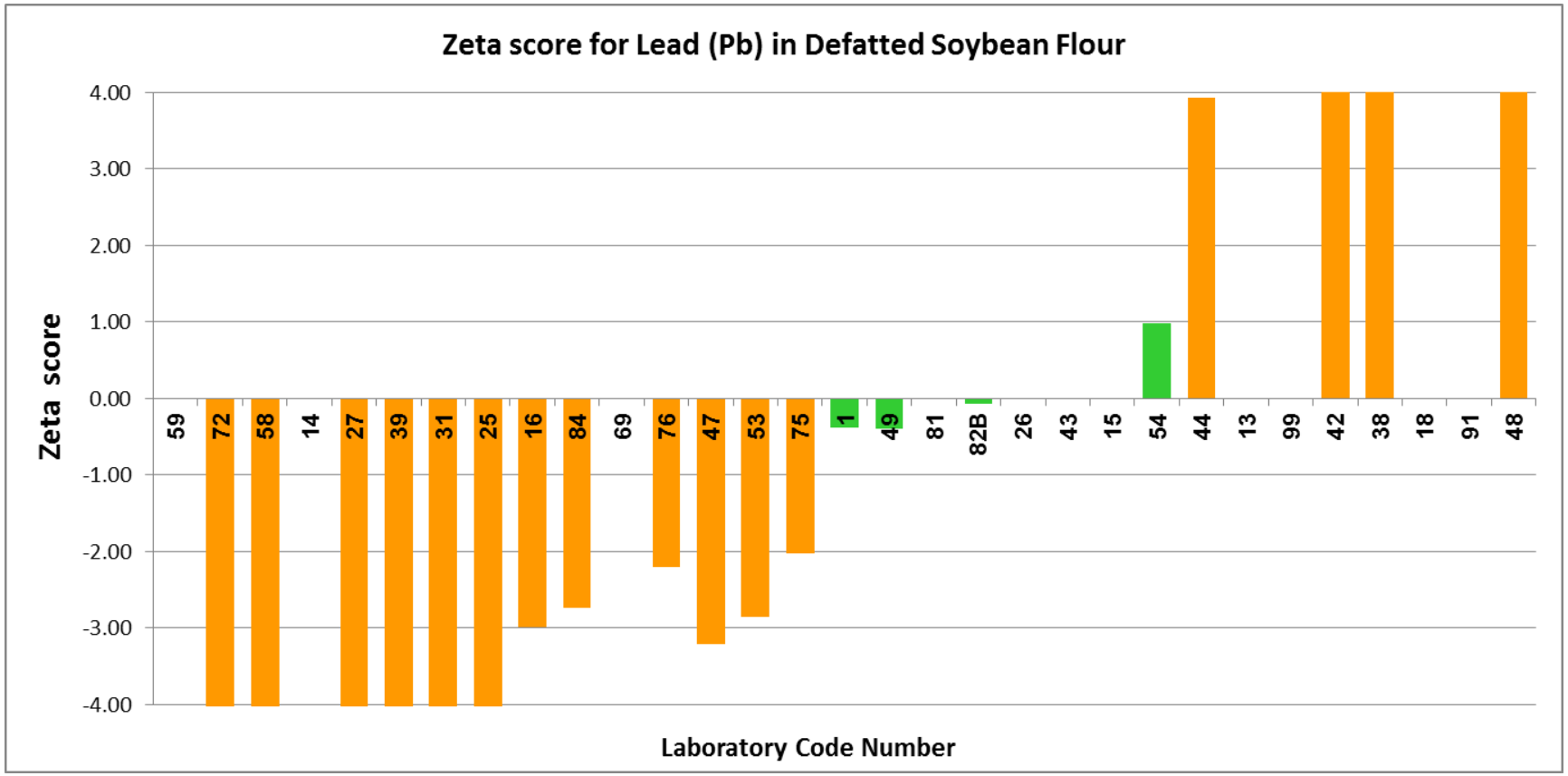

Figure 150. Plot of *Zeta* score for **lead** spiked rice flour, following the ordered z scores in the above Figure 149.

Table 55. Evaluation of laboratory performance on **mercury** analysis (mg/kg) in spiked rice flour

Lab Number	Mercury (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight g	Digestion Technique	Digestion Medium	Instrument	Wave-length (nm or mass)	Recovery correction	Method Reference
Assigned value obtained from robust average (x) ± SDp from Horwitz' s equation = 0.45 ± 0.08 g/100 g (CV 17.8%, n= 26) with u_{xpt} 0.02 mg/kg*											
Acceptance criteria			|z score| ≤ 2.00	|ζ score| ≤ 2.00							
1	0.56	0.19	1.33	1.12	0.5071	Microwave, Nilestone Srl MA 176-0020SK15	-	ICP-MS 7800	-	N	AOAC 2015.01
13	0.33	-	-1.49	-	0.5	Microwave	HNO3 10 mL + HCl 2 mL	ICP-MS Thermo Scientific (iCAP RQ)	M/z: Hg 202	N	Internal Method
15	0.52	-	0.91	-	0.5	Ultrawave Digestion	5% HNO3 + 0.5% HCl	ICP-MS (7900 Agilent)	Hg 202	N	Based on USFDA 4.7 version 1.1
16	0.54	0.05	1.13	2.81	2	Microwave	HNO3+H 2O2	ICP-MS 7700X Agilent		N	In-house Method
18	0.54	-	1.13	-	2.0	Dry Ashing (Wet Digestion for Hg, Sn)	HCl (HNO3 for Hg, Sn)	ICP-OES Agilent (AAS-VGA Varian, Agilent for Hg, As)	Hg 253.7	N	SNI 19-2896-1998
25	0.44	0.07	-0.14	-0.28	Hg 0.2244	HNO3-HCl (Hg Cold Vapour)	Water	ICP-OES (Hg Hydra IIAA)	Hg 254		USEPA Method 3050B (Hg EPA-SW 846, Method 7470A
26	0.01	0.00	-5.48	-21.92	5.0	Dry Ashing (Wet Ashing for Hg)	Water & HCl (1+1) (Nitric:Perchloric (1+1) for Hg)	AA-7000 (plus MVU-1A)	253.7 Hg	N	AOAC No. 999.11 using flame AAS, 977.15 for Hg
31	0.42	-	-0.36	-	5 (1 for Hg)	Dry Ashing (Microwave for Hg)	-	AAS (Cold Vapour for Hg)	-	N	AOAC 999.11, (Hg: SNI 01-3751)

Lab Number	Mercury (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight g	Digestion Technique	Digestion Medium	Instrument	Wave-length (nm or mass)	Recovery correction	Method Reference
Assigned value obtained from robust average (x) ± SDp from Horwitz' s equation = 0.45 ± 0.08 g/100 g (CV 17.8%, n= 26) with u_{xpt} 0.02 mg/kg*											
38	**0.22**	0.11	**-4.69**	**-3.86**	1.000	Dry Ashing	0.1 M HNO3	Flame AAS, Shimadzu AA6300	Hg 253.7		Modified AOAC 999.11
39	**0.49**	0.04	0.54	1.54	0.5	Microwave	-	GF-AAS (Hg: Mercury analyzer)	Hg 253.7	Y	AOAC 999.10 (Hg: 977.15, As: plus 986.15)
42	**0.39**	0.02	-0.79	**-2.88**	10 (0.5 for Hg, Sn)	Dry Ashing (Microwave Digestion for Hg, Sn)	HNO3-HCl (HNO3 for Hg, Sn)	Hg: HG AAS Perkin Elmer	Hg 253.7	N	AOAC 999.11.2005 (Hg: SNI 3549:2009. Lampiran A.16, Sn: BS EN 15764:2009)
43	**0.51**	0.01	0.80	**3.10**	0.5	Microwave	HNO3	ICP-OES, ICP-MS	Hg m/z 202	N	AOAC
48	**0.56**	0.09	1.38	**2.23**	5 (0.5 for Hg and As)	Dry Digestion (Hg, As Microwave Digestion)		AA800 Perkin Elmer	Hg 253.7	N	MU-03/20 (AAS)
53	**0.57**	0.06	1.50	**3.22**	0.3	Microwave	4 mL HNO3, 1 mL HCl, 1 mL H2O2	ICPMS Thermo			In house method
54	**0.55**	0.05	1.21	**3.03**	1	Microwave digestion	HNO3 / H2O2	ICP / Shimadzu		N	AOAC 984.27
58	**0.32**	0.13	-1.65	-1.94	1.0	Acid Digestion	HNO3, H2O2	ICP-OES (AAS-Hydride for As, Hg)	Hg 253.7	-	Acid Digestion and Quantitation by ICP-OES (AAS-hydride for As, Hg)

Lab Number	Mercury (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight g	Digestion Technique	Digestion Medium	Instrument	Wave-length (nm or mass)	Recovery correction	Method Reference
Assigned value obtained from robust average (x) ± SDp from Horwitz' s equation = 0.45 ± 0.08 g/100 g (CV 17.8%, n= 26) with u_{xpt} 0.02 mg/kg*											
59	**0.46**	0.01	**-5.31**	0.50	Hg: 1	Dry Ashing	-	AAS, Shimadzu	Hg 253.7	Y	As: IK A2-LM10 (AAS), Pb+Cd: SNI 3751:2009 point A.14.1, Hg: SNI 01-2354.6-2006
69	**0.32**	-	-1.66	-	-	-	-	-	-	-	-
72	**0.66**	0.06	**2.63**	**5.82**	4 (Hg 0.5)	Ashing (Hg Acid Digestion)	HNO3 (Hg Aqua regia)	ICP-OES, JY Ultima (Hg FIMS 400, Perkin Elmer)	Hg 253.7	N	AOAC 999.11 (Hg EPA 7471)
75	**0.51**	0.01	0.74	**2.86**	1	Wet digestion (hot block)	HNO3 + H2O2	ICP-OES Agilent 5100, ICP-MS Agilent 7700x	Hg 253.7	N	In House Method ICP-MS & ICP-OES
76	**0.44**	0.02	-0.13	-0.45	-	-	-	-	-	-	-
82B	**0.55**	0.08	1.29	**2.37**	1.00	Microwave	Nitric Acid	Agilent, Hg, Cu: AAS GBC	-	Y	Hg: Hydride Generation SSA
84	**0.48**	0.05	0.33	0.83	0.5	Microwave Digestion	HNO3 / H2O2	ICP-MS	-	N	AOAC 999.10:2005
88	**0.56**	0.00	1.39	**5.52**	0.3 (Cu, Zn 3)	Microwave	H2O2 2 ml + HNO3 8 mL	AAS GBC Hydride vapour	Hg 253.70	N	In house method (AAS)
95	**0.23**	0.06	**-2.70**	**-6.05**	-	-	-	-	-	-	-
99	**0.02**	-	**-5.44**	-	0.3 ± 0.001	Microwave	Nitric Acid & Hydrogen Peroxide	Digestion (MULTI GO Anton Paar) Determination (ICP/MS, PE)	Refer Mass each element	N	EPA 3015A

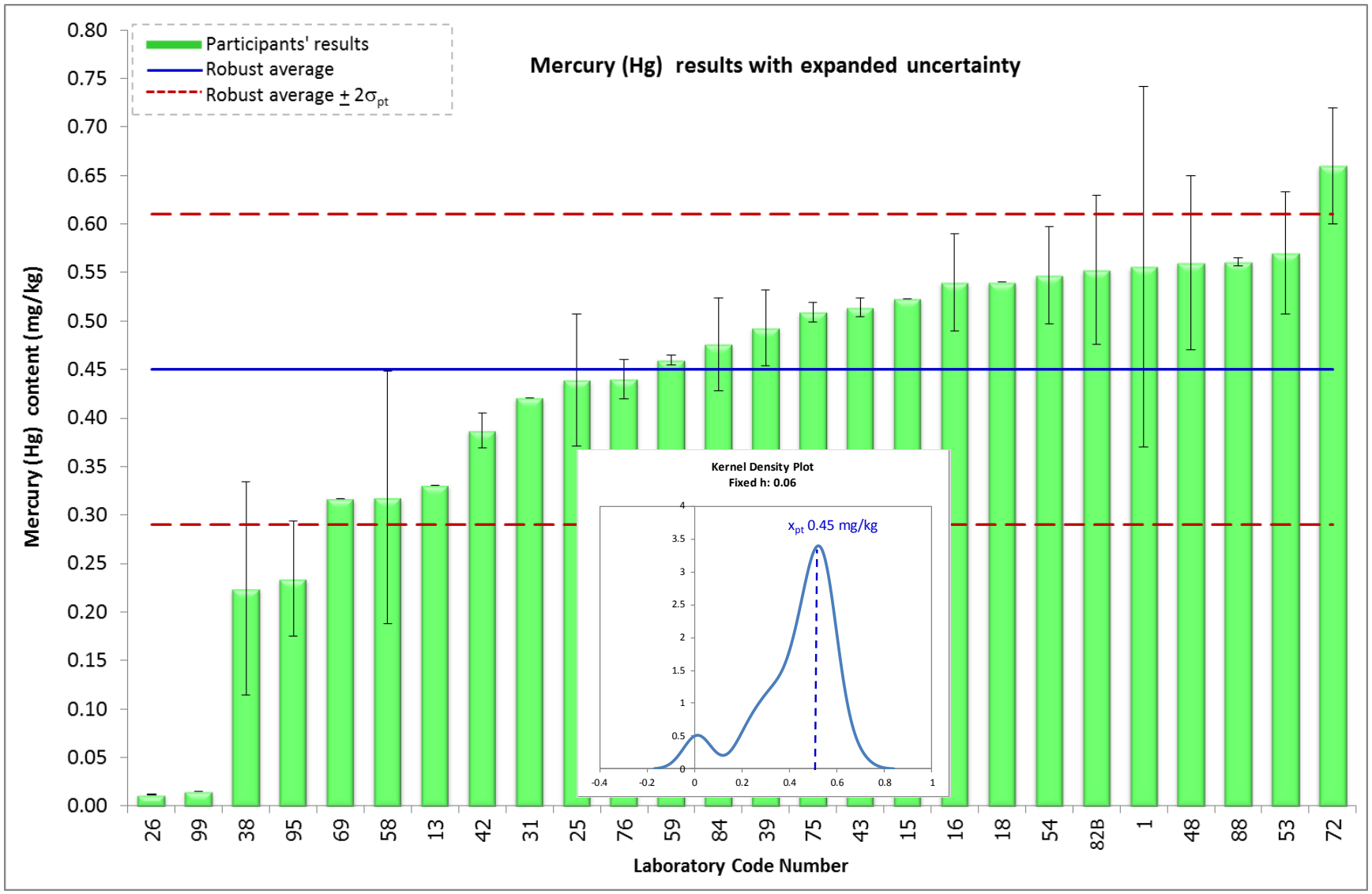

Figure 151. Distribution of **mercury** results (ascending order) in spiked rice flour with expanded uncertainty

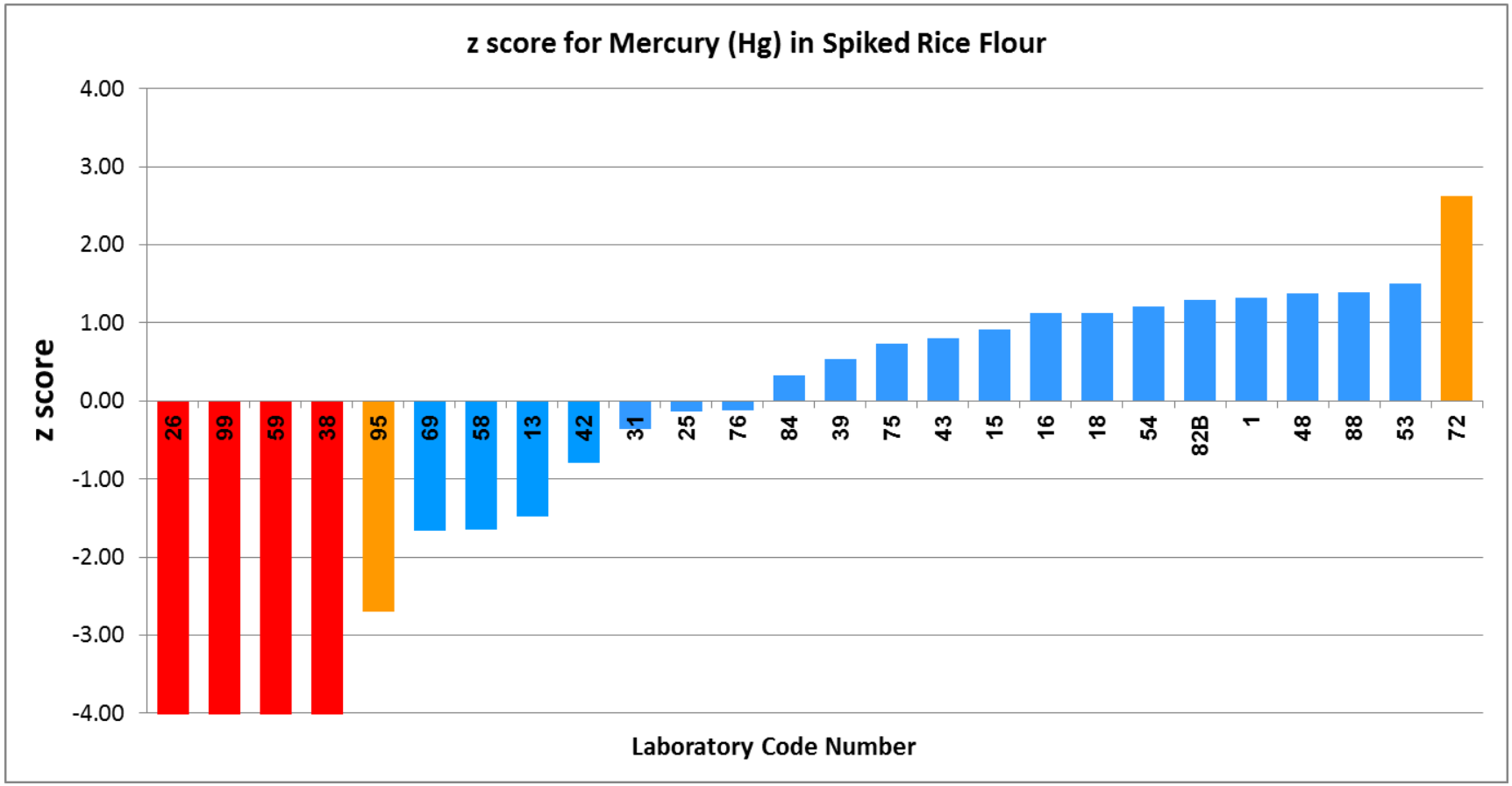

Figure 152. Plot of ordered *z* scores for **mercury** results in spiked rice flour

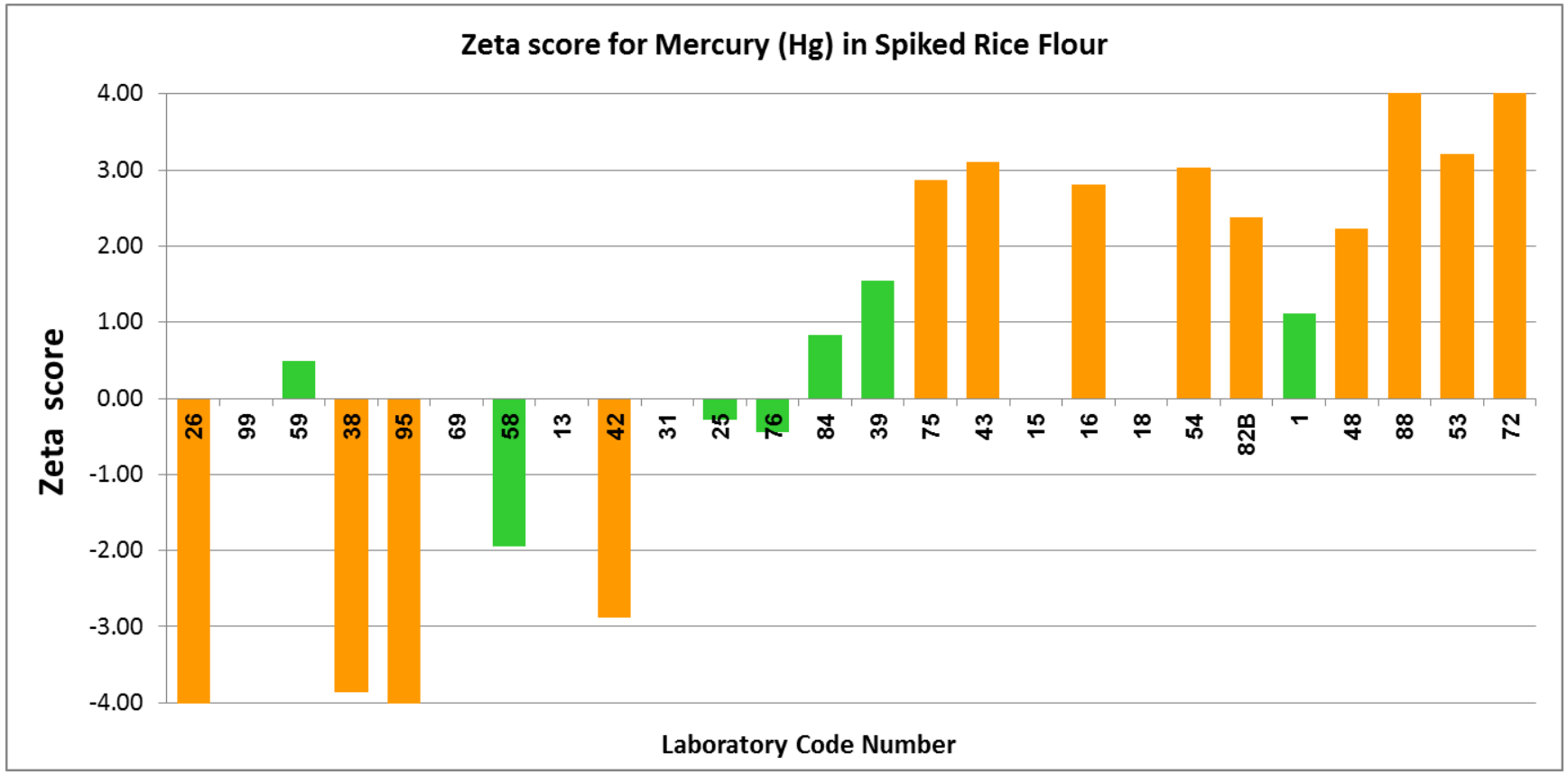

Figure 153. Plot of *Zeta* score for **mercury** spiked rice flour, following the ordered z scores in the above Figure 152.

Table 56. Evaluation of laboratory performance on **tin** analysis (mg/kg) in spiked rice flour

Lab Number	Tin (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight g	Digestion Technique	Digestion Medium	Instrument	Wave-length (nm or mass)	Recovery correction	Method Reference
Assigned value obtained from robust average (x) ± 2SDp from Horwitz' s equation = 10.28 ± 2.31 g/100 g (CV 22.5%, n= 19) with u_{xpt} 0.64 mg/kg*											
Acceptance criteria			|z score| ≤ 2.00	|ζ score| ≤ 2.00							
13	12.90	-	1.13	-	0.5	Microwave	HNO3 10 mL + HCl 2 mL	ICP-MS Thermo Scientific	M/z: Sn 118	N	Internal Method
14	2.62	-	-3.32	-	0.5	Wet Digestion	HNO3, H2O2	ICP Horiba Jobin Yvon	Sn 189.989	Y	AOAC 999.10
15	10.10	-	-0.08	-	0.5	Ultrawave Digestion	5% HNO3 + 0.5% HCl	ICP-MS (7900 Agilent)	Sn 118	N	Based on USFDA 4.7 v 1.1
16	2.56	0.13	-3.34	-12.00	2	Microwave	HNO3+H2O2	ICP-MS 7700X Agilent	-	N	In-house Method
18	12.50	-	0.96	-	2.0	Wet Digestion for Hg, Sn	HCl (HNO3 for Hg, Sn)	ICP-OES Agilent (AAS-VGA Varian)	Sn 283.998	N	SNI 19-2896-1998
25	8.66	0.07	-0.70	-2.53	5.0160 / 5.0123 (Hg 0.2244 / 0.2245	HNO3-HCl (Hg Cold Vapour)	Water	ICP-OES (Hg Hydra IIAA)	Sn 189.925		USEPA Method 3050B (Hg EPA-SW 846, Method 7470A
31	< 4.5	-	-3.48	-	5 (1 for Hg)	Dry Ashing (Microwave for Hg)	-	AAS (Cold Vapour for Hg)	-	N	AOAC 999.11, (Hg: SNI 01-3751, As: AOAC 986.15, Sn: SNI 3551:2012)
38	100.00	0.96	38.84	112.23	1.000	Dry Ashing	0.1 M HNO3	Flame AAS, Shimadzu AA6300	, Sn 286.30		Modified AOAC 999.11
42	10.50	0.79	0.10	0.29	10 (0.5 for Hg, Sn)	Dry Ashing (Microwave Digestion for Hg, Sn)	HNO3-HCl (HNO3 for Hg, Sn)	Pb, Cd, Sn: GFAAS Agilent 240 FS	Sn 286.3	N	AOAC 999.11.2005 (Sn: BS EN 15764:2009)
43	8.44	0.95	-0.80	-2.31	0.5	Microwave	HNO3	ICP-OES, ICP-MS	Sn 189.925	N	AOAC

Lab Number	Tin (mg/kg)	MU (mg/kg)	z score	Zeta score	Sample Weight g	Digestion Technique	Digestion Medium	Instrument	Wave-length (nm or mass)	Recovery correction	Method Reference
Assigned value obtained from robust average (x*) ± 2SDp from Horwitz' s equation = 10.28 ± 2.31 g/100 g (CV 22.5%, n= 19) with u_{xpt} 0.64 mg/kg											
48	**< 0.05**	-	**-4.44**	-	5	Dry Digestion	-	AA800 Perkin Elmer	Sn 286.3	N	MU-03/20 (AAS)
49	**3.94**	0.20	**-2.74**	**-9.79**	2	Dry Ashing	6 M HCl	ICP-OES 5110 Agilent Technologies	Sn 283.998	N	AOAC 20th Ed 2016
53	**9.61**	0.21	-0.29	**-1.03**	0.3	Microwave	4 mL HNO3, 1 mL HCl, 1 mL H2O2	ICPMS Thermo	-	-	In house method
54	**9.26**	0.73	-0.44	**-1.38**	1	Microwave digestion	HNO3 / H2O2	ICP / Shimadzu	Sn 189.989	N	AOAC 984.27
58	**9.62**	0.50	-0.29	-0.96	1.0	Acid Digestion	HNO3, H2O2	ICP-OES	Sn 189.925	-	Acid Digestion and Quantitation by ICP-OES
59	**< 0.10**		**-4.43**	-	Sn: 2.5,	Dry Ashing	-	AAS, Shimadzu	Sn 286.3	Y	Sn: SNI 01-2896-1995 point 5
69	**1.49**	-	**-3.81**	-	-	-	-	-	-	-	-
72	**11.80**	1.00	0.66	1.87	4 (Hg 0.5)	Ashing	HNO3 (Hg Aqua regia)	ICP-OES, JY Ultima	Sn 286.333	N	AOAC 999.11 (Hg EPA 7471)
75	**7.73**	0.75	-1.10	**-3.43**	1	Wet digestion (hot block)	HNO3 + H2O2	ICP-OES Agilent 5100, ICP-MS Agilent 7700x	Sn 189.925	N	In House Method ICP-MS & ICP-OES
76	**1.39**	0.21	**-3.85**	**-13.71**	-	-	-	-	-	-	-
84	**9.01**	0.90	-0.55	-1.63	0.5	Microwave Digestion	HNO3 / H2O2	ICP-MS	-	N	AOAC 999.10:2005
88	**0.31**	0.00	**-4.31**	**-15.57**	0.3	Microwave	H2O2 2 ml + HNO3 8 mL	AAS GBC Hydride vapour	Sn 235.50,	N	In house method (AAS)
99	**13.90**	-	1.57	-	0.3 ± 0.001	Microwave	Nitric Acid & Hydrogen Peroxide	Digestion (MULTI GO Anton Paar) Determination (ICP/MS, PE)	Refer Mass each element	N	EPA 3015A

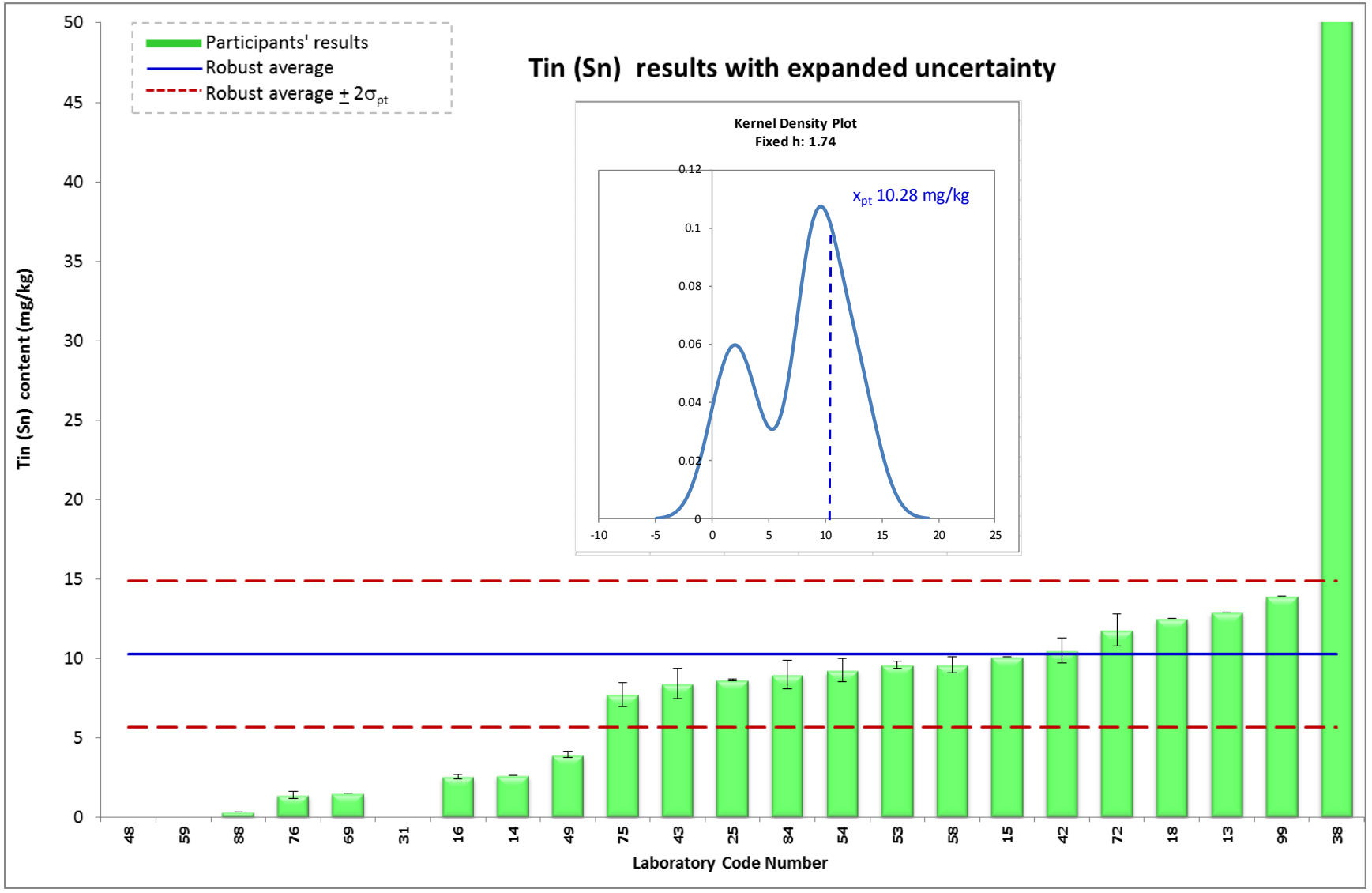

Figure 154. Distribution of **tin** results (ascending order) in spiked rice flour with expanded uncertainty

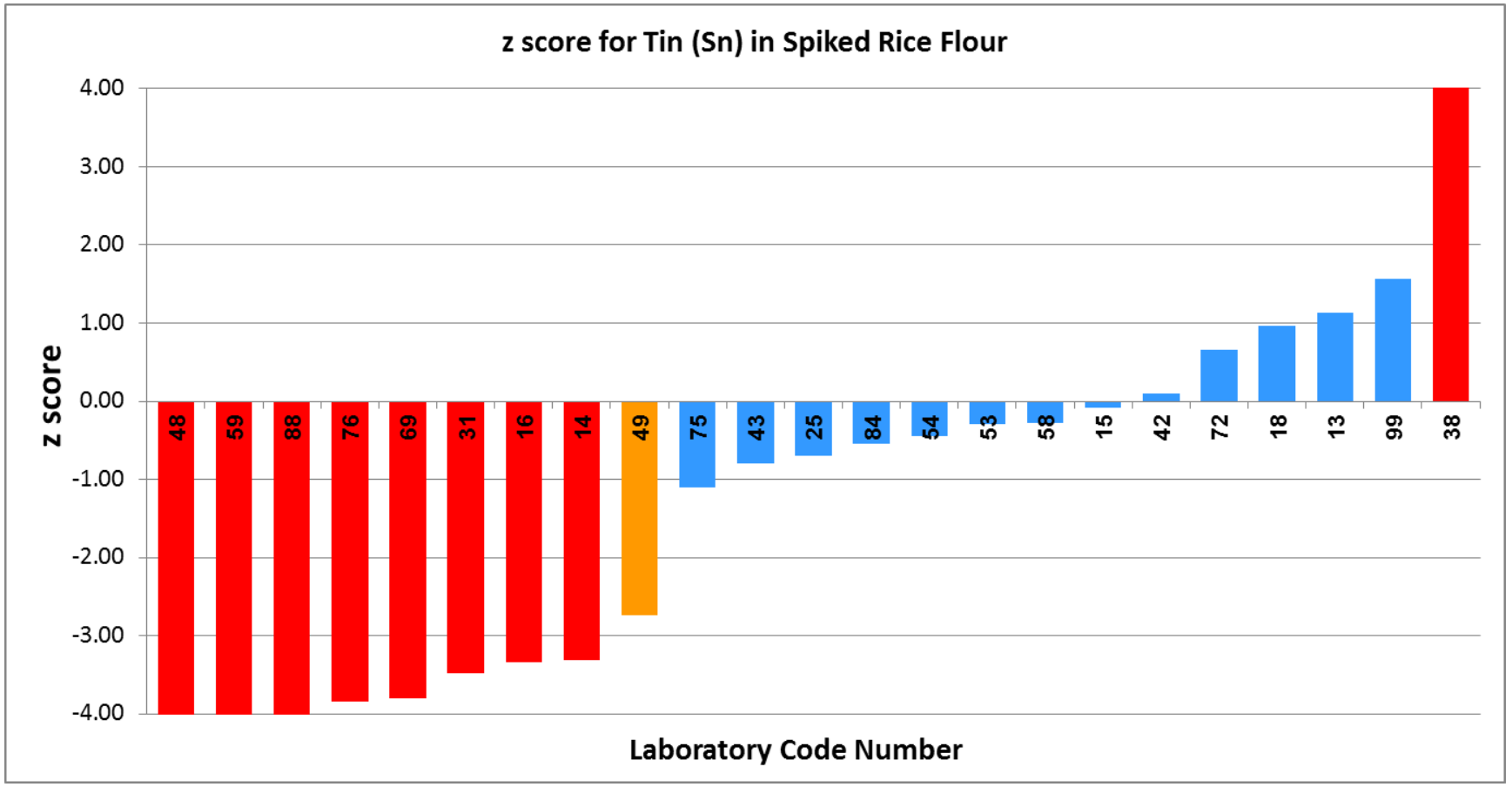

Figure 155. Plot of ordered *z* scores for **tin** results in spiked rice flour

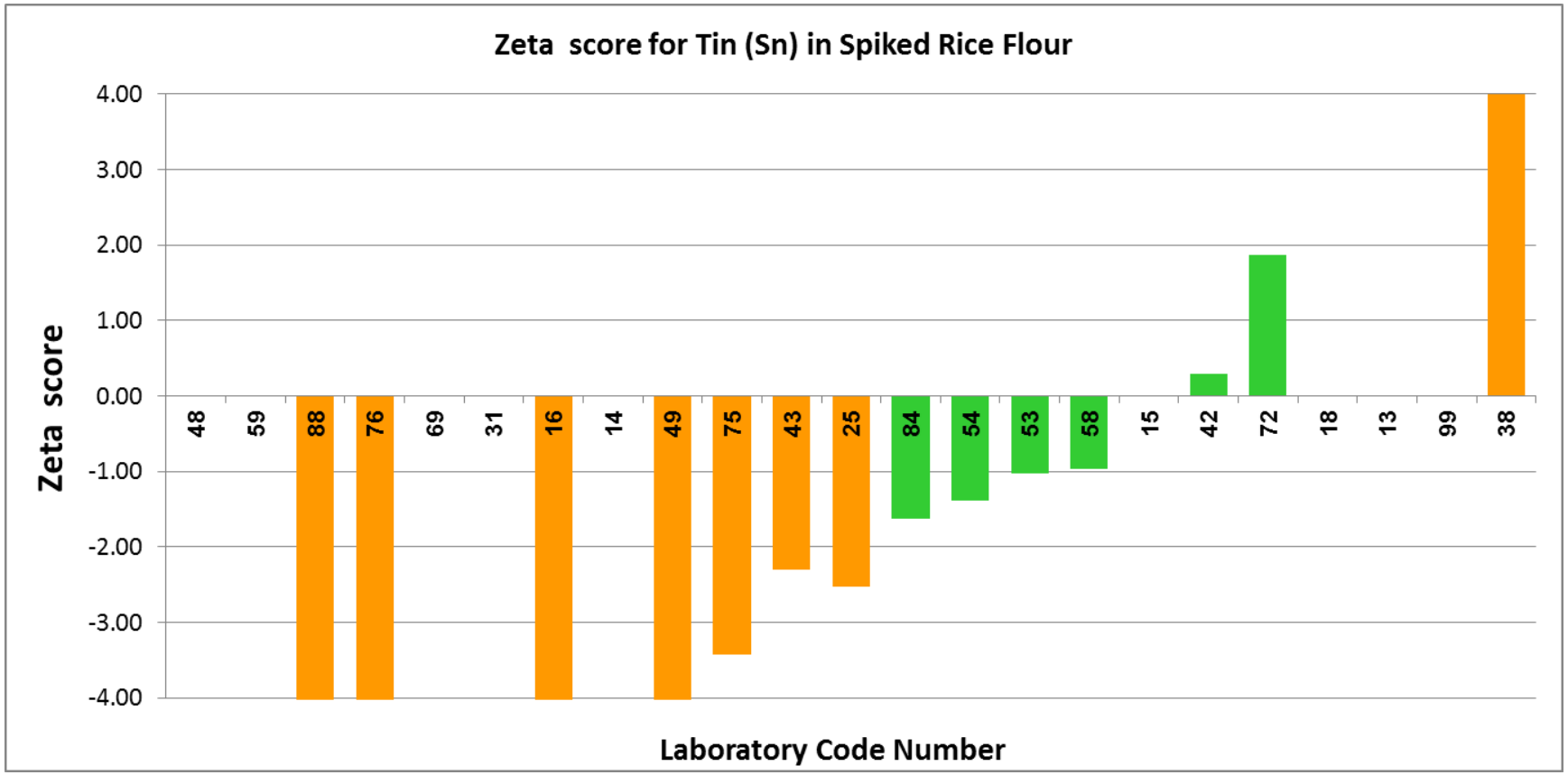

Figure 156. Plot of *Zeta* score for **tin** spiked rice flour, following the ordered z scores in the above Figure 155.

Table 57. Summary: evaluation of laboratory performance in spiked rice flour

Parameters	Total participating laboratory	Evaluation results (number of laboratory, percentage in bracket)		
		Satisfactory	Questionable	Unsatisfactory
Moisture (g/100g)	23	20 (87.0%)	1 (4.3%)	2 (8.7%)
Arsenic (mg/kg)	27	18 (66.7%)	2 (7.4%)	7 (25.9%)
Cadmium (mg/kg)	30	22 (73.3%)	3 (10.0%)	5 (16.7%)
Lead (mg/kg)	31	17 (54.8%)	5 (16.1%)	9 (29.0%)
Mercury (mg/kg)	26	20 (76.9%)	2 (7.7%)	4 (15.4%)
Tin (mg/kg)	23	13 (56.5%)	1 (4.3%)	9 (39.1%)