IAEA/AQ/72

IAEA Analytical Quality in Nuclear Applications Series No. 72

Interlaboratory Comparison on the Determination of Trace Elements and Methyl Mercury in Fish Sample IAEA 407A

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INTERLABORATORY COMPARISON ON THE DETERMINATION OF TRACE ELEMENTS AND METHYL MERCURY IN FISH SAMPLE IAEA 407A

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INTERNATIONAL ATOMIC ENERGY AGENCY
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FOREWORD

The protection of the marine environment is among one of the highest priorities worldwide. The identification of environmental pollution is based on monitoring campaigns that periodically assess the quality of seawater, marine sediments and biota samples. The reliability and comparability of analytical results produced in this context are crucial for the management of the marine environment in general, such as when taking decisions and meaningful actions in relation to remediation policies.

The IAEA provides support to Member States in the field of data quality and quality assurance by organizing interlaboratory comparisons and producing marine certified reference materials (biota and sediments) which are characterized for trace elements and methylmercury mass fractions.

To ensure compliance with the international standard ISO/IEC 17034:2016, certified reference materials produced by the IAEA are characterized with the participation of analytical laboratories with demonstrated measurement competence. This is ensured by regularly organizing targeted interlaboratory comparisons involving these laboratories. Interlaboratory comparisons involve comparing participants' respective results to an assigned value, which is usually derived as a consensus value from the overall population of obtained results. The design of interlaboratory comparisons allows for the monitoring and demonstration of the measurement performance and analytical capabilities of the participating laboratories and, at the same time, the identification of existing gaps and problem areas where further improvement is needed.

This publication summarizes the results of the interlaboratory comparisons on the determination of trace elements and methylmercury in a fish sample organized in 2021.

The IAEA is grateful to the Government of Monaco for its support and wishes to thank the participants and laboratories involved in this comparison exercise. The IAEA officer responsible for this publication was S. Azemard of the Marine Environment Laboratories.

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1 INTRODUCTION

1.1 BACKGROUND

The Marine Environmental Studies Laboratory (MESL) of the International Atomic Energy Agency's Marine Environment Laboratories (NAML) has the programmatic responsibility to provide assistance to Member States laboratories in maintaining and improving the reliability of analytical measurement results, both for trace elements and organic contaminants. This is accomplished through the provision of reference materials of marine origin, validated analytical procedures, training on the implementation of internal quality control, and through the evaluation of measurement performance via the organized worldwide and regional interlaboratory comparisons (ILC).

The results of ILC or Proficiency Tests (PT) are of crucial importance for the participating laboratories as they provide clear information on their measurement capabilities.

1.2 OBJECTIVE

This exercise was designed to evaluate the measurement performance and analytical capabilities of participating laboratories with respect to the determination of trace elements and methyl mercury in fish sample IAEA-407A. The objectives of this ILC was to evaluate the competence of selected laboratories regularly involved in the characterization of certified reference materials (CRMs) produced by the IAEA.

1.3 SCOPE

The present ILC study was designed to evaluate the measurement performance of selected laboratories for trace elements and methyl mercury (MeHg) in fish. The scope of this publication is to describe obtained results of that ILC.

1.4 STRUCTURE

This publication is structured in five sections: Section 1 being the introduction. Section 2 describes the test sample. The individual performances assessment with *z* and Zeta-scores is explained in Section 3 and the results obtained are reviewed in Section 4. Section 5 provides some conclusions on the results obtained in this ILC.

2 STUDY SET UP

In July 2021, invitation letters were sent to 16 laboratories from 13 Member States, which previously participated to the IAEA characterization exercises. Positive responses with intent to participate were received from 12 laboratories in 10 Member States.

At the beginning of September 2021 each laboratory received one bottle of the test sample, accompanied by an information sheet. Participants were requested to determine as many elements as possible from the following list: Ag, Al, As, Ca, Cd, Co, Cr, Cu, Hg, K, Li, MeHg, Mg, Mn, Ni, Pb, Rb, Sb, Se, Sn, Sr, V, and Zn, using the analytical procedures routinely applied in their laboratories. The deadline for reporting results was set at the end of November 2021.

Participating laboratories were requested to report their results for the ILC sample accompanied with standard and expanded uncertainties, description and results of internal quality control

samples (e.g CRMs or other reference materials (RMs)), analyte recovery, detection and quantification limits, digestion and instrumental technique used.

In addition, participating laboratories were requested to answer some questions on their analytical procedure, calibration, recovery correction, uncertainties estimation, moisture determination, validation of analytical method, CRM or RM used, quality procedures and accreditation.

In total, 11 laboratories from 9 Member States reported results back to MESL. The data submitted by the laboratories, together with the technical and statistical evaluations of the results for the requested trace elements, are included in this report. All results were treated confidentially, and each laboratory was identified with a unique confidential code number.

3 DESCRIPTION OF ILC TEST MATERIAL

The ILC test sample (IAEA-407A) is a fish flesh homogenate sample from the North Sea, which was characterized in a separate exercise. All details on sample preparation, homogeneity, stability, and assignment of values can be found in the certification report [1]. The assigned values were determined according to the requirements of ISO/IEC 17034:2016 [2] and ISO/IEC guide 35:2017 [3] and are presented in Table 1. For some analytes as shown in Table 2, associated uncertainty of assigned values are above 20% and therefore assigned mass fractions were given only as information.

The assigned value has a direct impact on conclusions about the 'measurement capability' of the participating laboratories, and therefore, the most metrologically credible value should be sought. Therefore, only analytes with certified values were evaluated while results reported for Ag, Al, Co, Cr, Li, Ni, Pb, Sb and Sn are given for information only.

TABLE 1. ASSIGNED VALUES AND UNCERTAINTY FOR THE ILC TEST SAMPLE

Analyte	Unit	Certified value	Expanded uncertainty (k=2)
As	mg kg^{-1}	13.33	0.62
Ca	mg kg^{-1}	24.5×10^3	3.0×10^3
Cd	mg kg^{-1}	0.184	0.021
Cu	mg kg^{-1}	3.09	0.21
Hg	mg kg^{-1}	0.219	0.011
K	mg kg^{-1}	12.83×10^3	0.95×10^3
MeHg	mg kg^{-1}as Hg	0.193	0.012
Mg	mg kg^{-1}	2.39×10^3	0.22×10^3
Mn	mg kg^{-1}	3.49	0.39
Na	mg kg^{-1}	14.2×10^3	1.2×10^3
Rb	mg kg^{-1}	2.46-	0.22
Se	mg kg^{-1}	2.6	0.2
Sr	mg kg^{-1}	128	12
V	mg kg^{-1}	1.46	0.18
Zn	mg kg^{-1}	66.4	2.7

TABLE 2. INFORMATION VALUES AND UNCERTAINTY FOR THE ILC TEST SAMPLE

Analyte	Unit	Information value	Expanded uncertainty (k=2)
Ag	mg kg^{-1}	0.035	0.007
Al	mg kg^{-1}	11.1	4.6
Co	mg kg^{-1}	0.089	0.018
Cr	mg kg^{-1}	0.67	0.20
Li	mg kg^{-1}	0.697	0.130
Ni	mg kg^{-1}	0.456	0.098
Pb	mg kg^{-1}	0.102	0.033
Sb	mg kg^{-1}	0.010	0.003
Sn	mg kg^{-1}	0.082	0.034

4 EVALUATION OF ANALYTICAL PERFORMANCE

The individual laboratory performance was expressed in terms of *z*-scores and Zeta-scores, in accordance with ISO/IEC 17043:2010 [4]. Scores were calculated only for analytes with a certified value.

The determination of target standard deviation σ_p, for the proficiency assessment was based on the outcome of previous ILCs organized by the MESL for the same population of laboratories and similar sample matrices and was fixed to 12.5% of the assigned values. The appropriateness of this level of tolerated variability of results was confirmed by calculation of the robust standard deviation of the participant's results and the uncertainty of the reference values for the respective measurands.

The *z*-score, which is calculated as shown in Eq. (1), defines the difference between the mean value provided by the laboratory and the reference value, expressed in the units of the target standard deviation (σ_p).

$$z = \frac{x_{lab} - x_{ref}}{\sigma_p} \quad (1)$$

The Zeta-score, which is calculated as shown in Eq. (2), demonstrates the agreement of the results reported by participating laboratories with the reference value within the respective uncertainties. The denominator in Eq. (2) is calculated from the combined uncertainty of the assigned value and the combined uncertainty reported by the respective participant (k=1).

$$\text{Zeta} = \frac{x_{lab} - x_{ref}}{\sqrt{u_{x_{lab}}^2 + u_{x_{ref}}^2}} \quad (2)$$

where:

x_{lab} is the result reported by the participating laboratory (expressed as the mean of multiple determinations)

x_{ref} is the reference value

σ_p is the target standard deviation

u_{xlab} is the combined uncertainty (k=1) reported by the participating laboratory

u_{xref} is the combined uncertainty (k=1) of the assigned value

The interpretation of a laboratory's performance was evaluated according to the following internationally accepted limits [2]:

	$\lvert z$ or Zeta$\rvert \leq 2$	Satisfactory
$2 <$	$\lvert z$ or Zeta$\rvert < 3$	Questionable
	$\lvert z$ or Zeta$\rvert \geq 3$	Unsatisfactory

5 RESULTS AND DISCUSSION

Eleven sets of data for 24 analytes were submitted by participating laboratories, comprising 162 numerical results. As explained above, z-scores and Zeta-scores were only calculated for 15 analytes. Almost all studied analytes were reported by at least about half of participating laboratories. The only exception was MeHg and Sn, which were only reported by three and two participating laboratories, respectively.

Reported results by analyte are shown in the Appendix along Kernel density plots [5] (if at least 8 results are available), z and Zeta scores (if calculated, see Section 3), a summary of statistical evaluation and PomPlots.

The PomPlot graphical method [6] displays the relative deviation of individual results x_{lab} from the reference (or information) value on the horizontal axis and relative uncertainties on the vertical axis. Distances (D) and uncertainties (u) are calculated as described in Eq. (3) and Eq. (4) and are expressed as multiple of median absolute deviation (MAD) calculated using Eq. (5).

$$D_i = x_{lab,i} - x_{ref}, \quad (i=1,\ldots n) \tag{3}$$

With n being the number of reported values per analytes

$$u = \sqrt{u_{xlab}^2 + u_{xref}^2} \tag{4}$$

$$\mathrm{MAD} = \mathrm{Median}\,|D_i|\,, \quad (\mathrm{i} = 1, \ldots \mathrm{n}) \tag{5}$$

With n being the number of reported values per analyte.

As shown in Fig. 1, the points on the right and left side of the graph correspond to biased results, results reported with small uncertainties are shown on the top of the graph, while point at the bottom of the graph represent results reported with large uncertainties. The reference value and the value(s) from organizer are also shown on those graphs for comparison purpose.

Figures 2 and 4 summarize the overall performance as defined by *z*-scores, by analyte and by participating laboratory respectively. Figure 3 and 5 summarizes the overall performance as defined by Zeta-scores by trace element and by participating laboratory respectively.

Table 3 and Table 4 show the overall performance as defined by *z*-scores and Zeta-scores respectively by trace element.

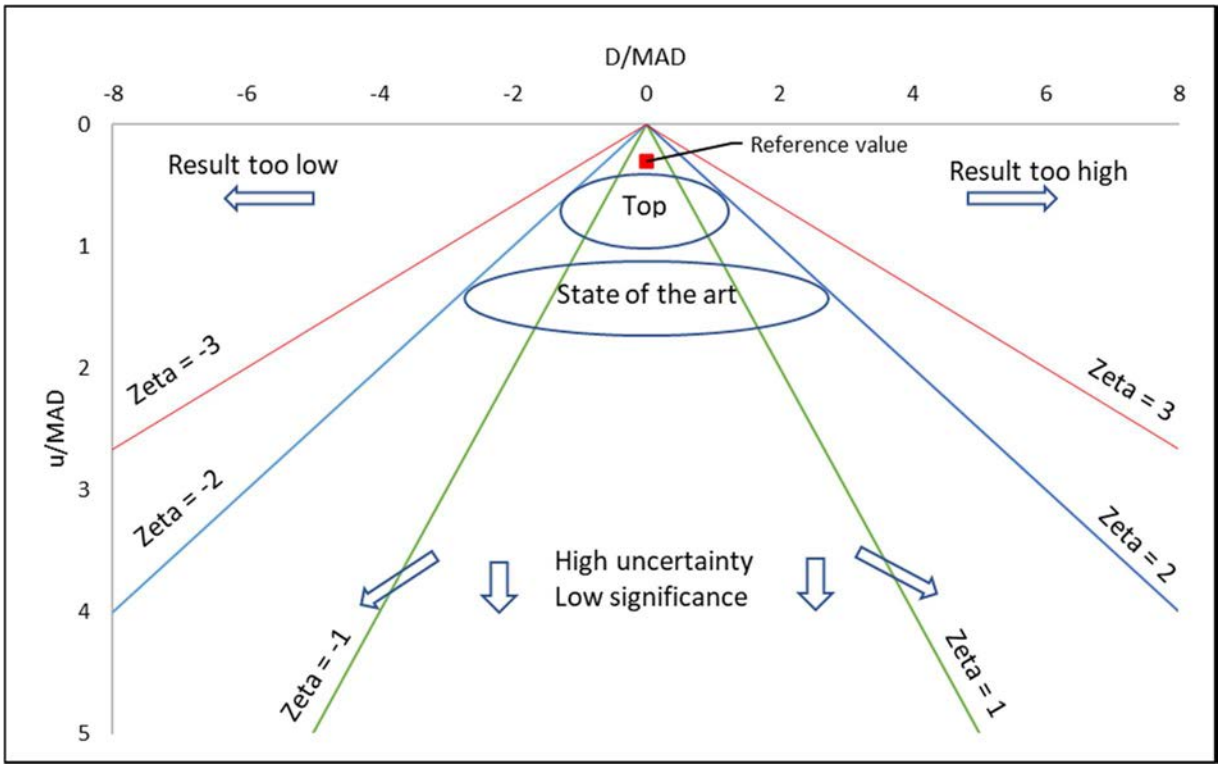
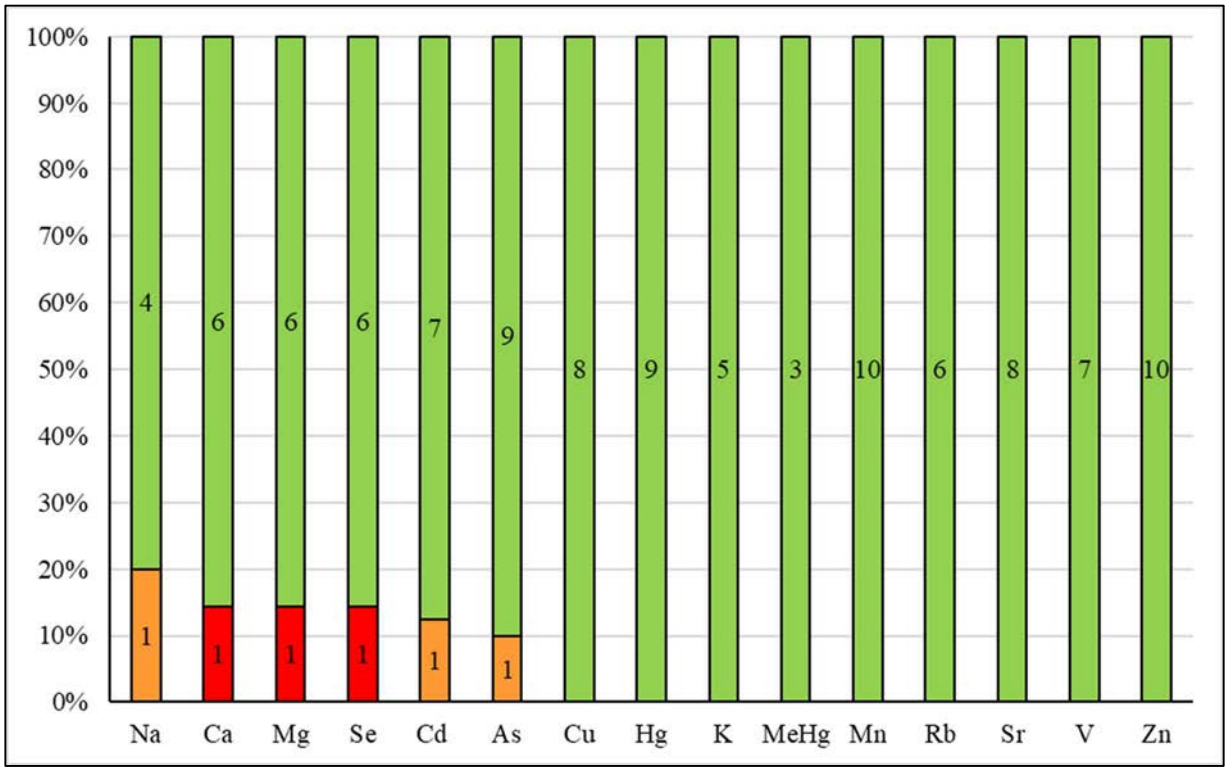
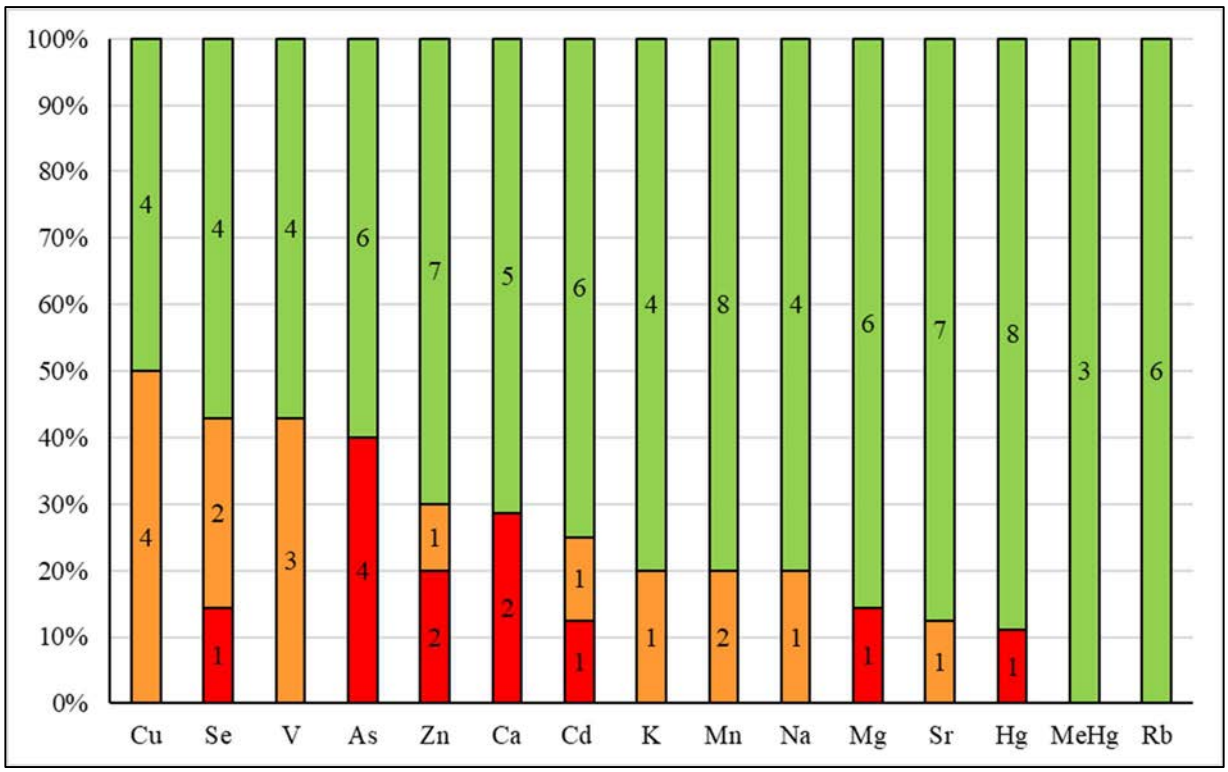


FIG. 1. Interpretation of a PomPlot (adapted from [6]).



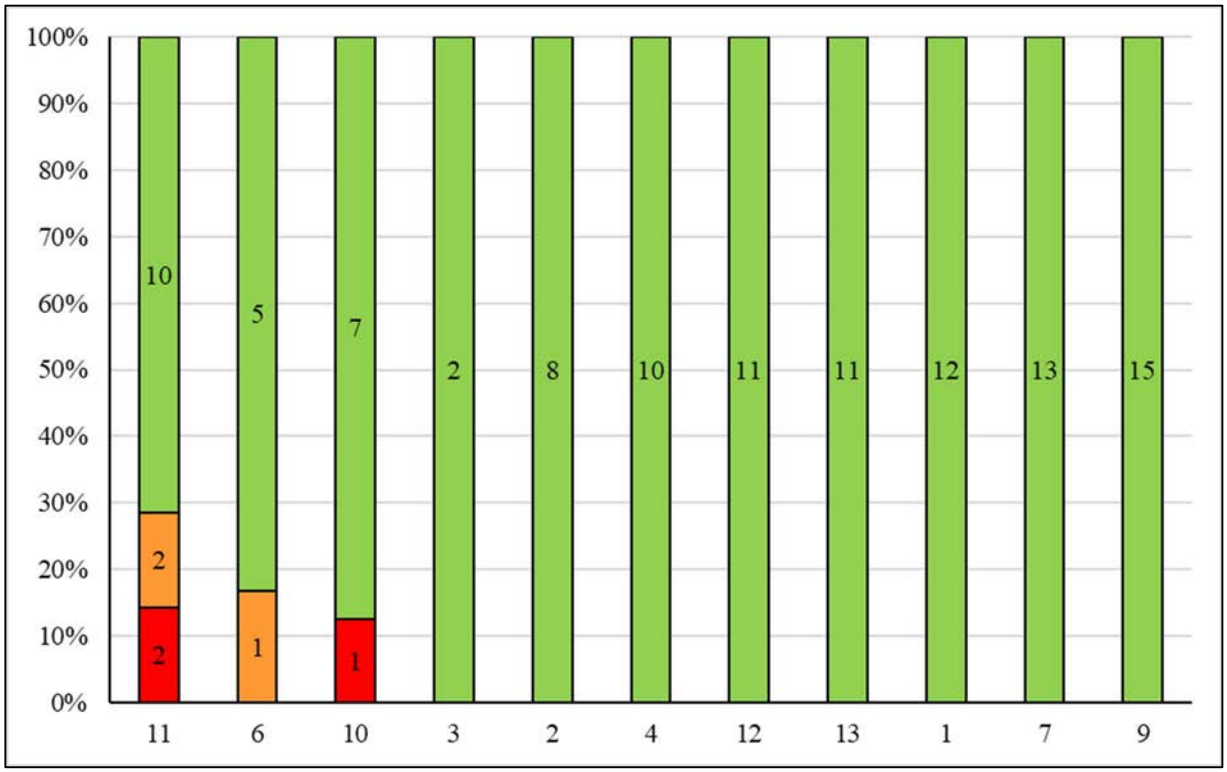
$|z| \geq 3$, $2 < |z| < 3$, $|z| \leq 2$

FIG. 2. z-scores calculated from the results reported by the participating laboratories for each trace element. Numbers provided in the bars are for participating laboratories.



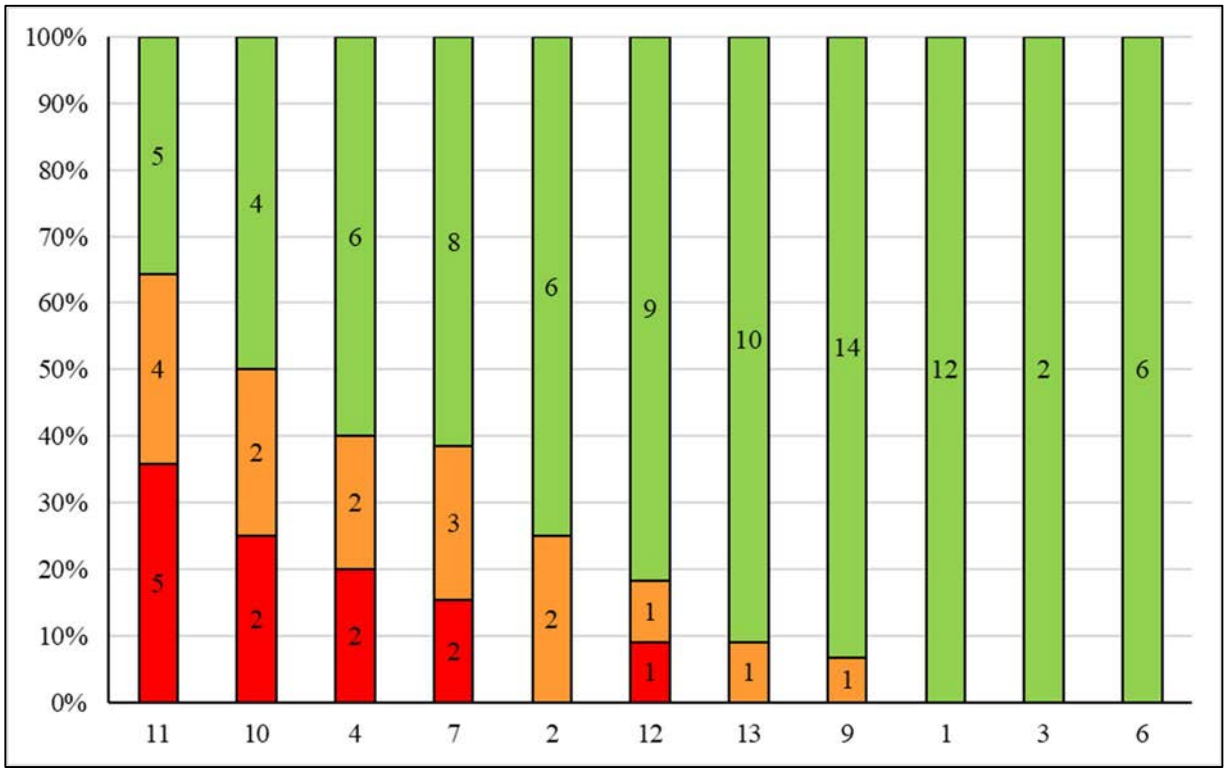
$|\text{Zeta}| \geq 3$, $2 < |\text{Zeta}| < 3$, $|\text{Zeta}| \leq 2$

FIG. 3. Zeta-scores calculated from the results reported by the participating laboratories for each trace element. Numbers provided in the bars are for participating laboratories.



$|z| \geq 3$, $2 < |z| < 3$, $|z| \leq 2$

FIG. 4. z-scores calculated from the results reported by the participating laboratories per laboratory.



$|\text{Zeta}| \geq 3$, $2 < |\text{Zeta}| < 3$, $|\text{Zeta}| \leq 2$

FIG. 5. Zeta-scores calculated from the results reported by the participating laboratories for each trace per laboratory.

TABLE 3. OVERALL ASSESSMENT OF LABORATORIES PERFORMANCE (*Z*-SCORE) BY TRACE ELEMENT*

Lab. Code	As	Ca	Cd	Cu	Hg	K	MeHg	Mg	Mn	Na	Rb	Se	Sr	V	Zn
1	-0.35	-0.38			0.80	-0.29	0.11	0.07	-0.57	-0.24	-0.24	-0.41	-0.48		-0.21
2	0.32		0.38	0.98	-0.12				0.63		0.32		0.77		0.58
3					-0.17		-0.22								
4	-1.83	0.08	-1.29	-1.79				-0.53	0.69	-0.20			-0.39	0.08	-0.92
6	-2.12		0.11	-0.55	0.00				-0.04						-0.29
7	-1.05	-0.61	-0.63	-0.82	1.23	-0.65		-0.74	-0.33		-0.28	-0.88	-1.06	-0.96	-0.07
9	0.16	0.88	-0.59	-0.40	-0.86	0.77	-0.66	0.26	0.48	0.56	0.15	0.10	0.77	0.35	-0.35
10	1.40		-0.22	-0.40	-0.37				1.38			3.01		1.23	0.64
11	-1.28	7.49	-2.33	-1.12	0.19	0.98		8.03	-1.31	2.70	-0.90	-0.79	-0.90	-1.40	-1.06
12	-0.71	-1.58				-0.11		0.35	0.02	-0.62	-0.25	1.29	-0.46	-0.33	-0.52
13	0.37	-0.08	-0.01	-0.31	0.24			-0.21	-0.91			-0.16	-0.29	-1.48	-0.39

TABLE 4. OVERALL ASSESSMENT OF LABORATORIES PERFORMANCE (ZETA-SCORE) BY TRACE ELEMENT*

Lab. Code	As	Ca	Cd	Cu	Hg	K	MeHg	Mg	Mn	Na	Rb	Se	Sr	V	Zn
1	-0.99	-0.68			1.93	-0.73	0.29	0.12	-0.99	-0.55	-0.51	-0.98	-0.99		-0.65
2	1.12		0.67	2.75	-0.23				0.94		0.78		1.83		2.01
3					-0.25		-0.20								
4	-4.58	0.16	-2.71	-2.74				-1.33	1.07	-0.55			-0.85	0.15	-5.04
6	-1.78		0.12	-0.85	0.00				-0.05						-0.45
7	-4.64	-1.13	-1.25	-2.32	5.86	-1.61		-1.62	-0.62		-0.64	-2.58	-2.71	-1.89	-0.12
9	0.34	1.63	-0.90	-0.83	-1.48	2.34	-0.56	0.59	0.65	1.21	0.26	0.17	1.19	0.50	-0.65
10	3.85		-0.43	-1.08	-1.35				2.54			6.28		2.11	1.94
11	-4.04	4.28	-3.97	-2.31	0.32	1.36		4.43	-2.50	2.89	-1.96	-1.60	-1.89	-2.26	-3.18
12	-1.75	-3.09				-0.16		0.43	0.03	-1.10	-0.43	2.04	-0.52	-0.27	-1.08
13	0.56	-0.11	-0.01	-0.49	0.04			-0.31	-1.32			-0.32	-0.43	-2.13	-0.66

* $|z| \geq 3$, $2 < |z| < 3$

5.1 *z*-SCORES

The *z*-scores compare the results of the participating laboratories deviation from the reference value with the target standard deviation σ_p, for proficiency assessment. The target standard deviation, σ_p was set up by the ILC organizer to 12.5 %, so the maximum acceptable deviation ($|z| \leq 2$) was 25% of the reference value.

As indicated in Section 2, *z*-scores could only be calculated for 15 out of 24 analytes reported: for Ag, Al, Co, Cr, Li, Ni, Pb, Sb and Sn assigned values are only given as information. As a result, out of the 11 datasets received from participating laboratories, 110 *z*-scores were calculated. From these 110 calculated *z*-scores, 94.5% were satisfactory with $|z| < 2$, and 2.7% were considered to be unsatisfactory with $|z| > 3$. Among the 11 participating laboratories, 8 (73% of participating laboratories) achieved satisfactory *z*- scores $|z| \leq 2$ for all their reported values. As the participants are considered expert laboratories based on previous performance, obtained score results were (not surprisingly) better than those typically observed for worldwide ILC's which usually indicate 75% satisfactory results.

It should be noted that 4 out 6 reported results that were evaluated as unsatisfactory and/or questionable based on *z*-scores were reported by the same participating laboratory, and two of them were evaluated as extreme outlier (|z| >7). Extreme outliers are generally indicating a transcription or calculation error.

Participating laboratories with results assessed as questionable and/or unsatisfactory with *z*-scores are encouraged to carefully check laboratory procedures and applied working instructions.

5.2 ZETA-SCORES

The Zeta-score shows the agreement of laboratory result with the reference value considering the respective uncertainties. The denominator in Eq. (4) includes combined uncertainties of the reference values and the reported values by the participating laboratories.

As shown in Figs 2–5, the comparison of measurement performances evaluated by *z*-score and Zeta-score clearly indicate that the number of unsatisfactory Zeta-scores is higher than the number of unsatisfactory *z*-scores (2.7% of calculated *z*-scores and 11% of calculated Zeta-scores). Only two participating laboratories (18% of participating laboratories) reported values which were evaluated as 100% satisfactory with both $|z|$ and $|\text{Zeta}| \leq 2$.

As Zeta-scores include the estimation of uncertainties, values receiving $|\text{Zeta}| > 3$ while $|z| < 3$ could indicate an underestimation of uncertainties. In Fig. 5, absolute Zeta-scores are plotted against expanded uncertainties of results reported by participants. More than half of the results receiving questionable or unsatisfactory Zeta-scores have been reported with expended uncertainties less than 10%, generally estimated as standard deviation of replicate analyses.

In general, laboratories should keep in mind that uncertainties based on the precision of measurement results (standard deviation) are frequently underestimated. In many cases, they reflect variations coming from the measurement step and usually do not include the contribution of uncertainty coming from other major contributors, such as recovery, procedural blank, moisture content, etc.

An unsatisfactory Zeta-score can be explained as for *z*-score by an inaccurate result and participating laboratories reporting values receiving $|z|$ and $|\text{Zeta}| > 3$ are encouraged to review their analytical procedures, as already mentioned in 4.1. Indeed the three reported results receiving an unsatisfactory z-score ($|z| > 3$) also received unsatisfactory Zeta-score.

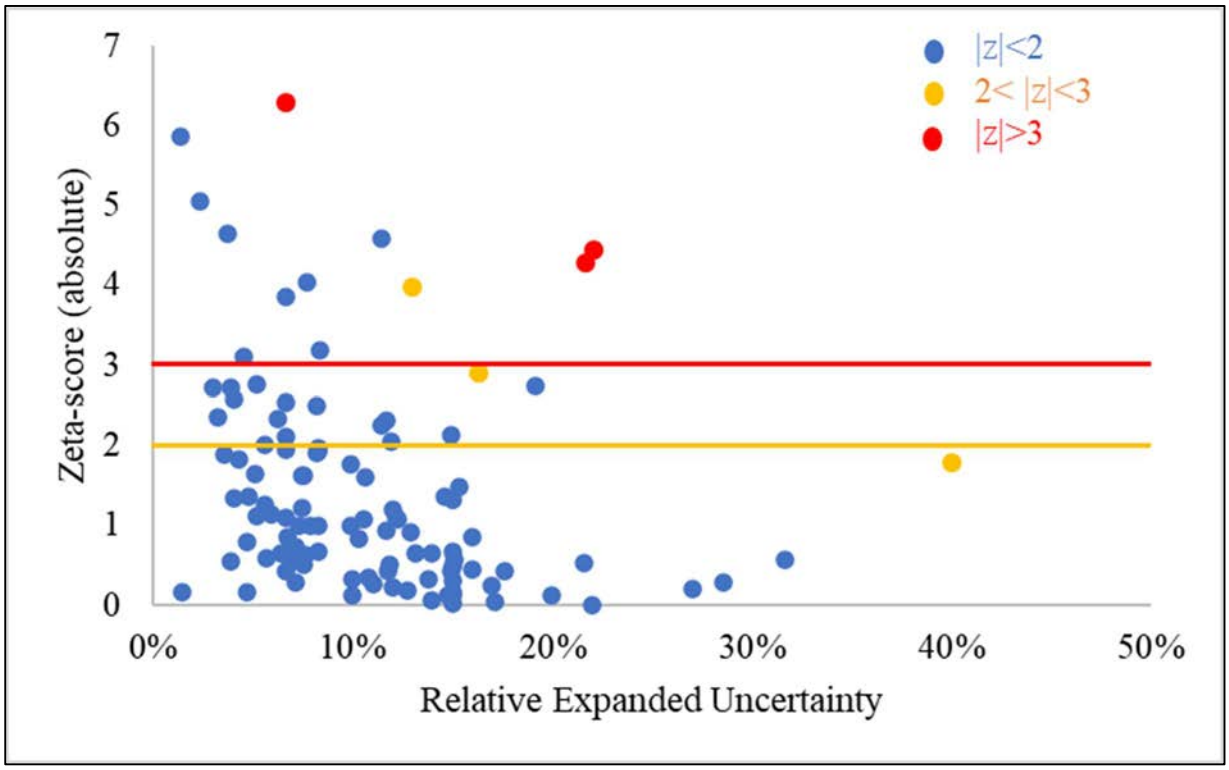


FIG. 5. Zeta-scores versus reported expanded uncertainties.

5.3 ANALYTICAL METHODS

Table 5 shows the distribution of values reported by different techniques as well as the number of participating laboratories being equipped with each type of instrumentation. Analytical methods used by participating laboratories in this ILC can be divided to three groups: nondestructive techniques (NAA, XRF); plasma spectrometric methods (ICP-MS and ICP-OES) and atomic absorption spectroscopy methods. The most used method was ICP-MS, which accounted for almost 70% of reported values and was used by 64% of the participating laboratories.

TABLE 5. DISTRIBUTION OF THE INSTRUMENTATION METHODS APPLIED IN THE ILC

Instrumental Method	Reported values	Number of laboratories
Inductively coupled plasma mass spectrometry	67.3%	7
Neutron activation	15.4%	2
Inductively coupled plasma optical emission spectrometry	5.6%	2
Cold vapor atomic fluorescence spectroscopy	1.9%	3
Flame atomic absorption spectroscopy	1.9%	1
Gaz chromatography atomic fluorescence	1.9%	3
Graphite furnace atomic absorption spectroscopy	1.9%	1
Solid atomic absorption spectroscopy	1.9%	3
X-ray fluorescence spectroscopy	1.2%	1
Cold vapor atomic absorption spectroscopy	0.6%	1
Cold vapor inductively coupled plasma mass spectrometry	0.6%	1

5.4 REVIEW OF QUESTIONNAIRES

All participating laboratories reported results for a CRM along with their results for the ILC sample. The regular use of a CRM as part of the internal quality control process is one way to ensure the quality of results produced in a laboratory, as recommended in ISO/IEC 17025:2017 [7]. An important principle for the selection of appropriate CRM by laboratories is the principle of matrix and concentration range matching. The CRMs used in this interlaboratory comparison were generally appropriately selected. Most of laboratories used biota of a marine origin (e.g. IAEA-407 Fish and IAEA 436-A Tuna fish, DORM-4 fish protein from NRCC), when analyzing trace elements in the ILC fish sample.

All participating laboratories have a quality system in place and about half of the participating laboratories declared to be accredited to ISO/IEC 17025:2017 [7], and the determination of trace elements in biota samples was within their scope of accreditation.

The fish sample used as a test sample in the present ILC was freeze-dried as part of its preparation procedure. At the time of bottling, the moisture content of the material was around 4%. Depending on local storage conditions and humidity levels, the ILC sample might absorb moisture from the environment. Consequently, users were advised to determine the moisture content of the test sample. As the moisture is an operationally dependent parameter, a detailed procedure on moisture content determination in the test sample was provided in the accompanying letter. All participating laboratories reported results that were corrected for moisture. Of these, seven reported the use of the recommended protocol (85°C) while others applied in-house methods using another temperature for the oven drying. The moisture content reported by the participating laboratories was in the range of 3–5%, and deviation from the recommended protocol did not impact the accuracy of reported results, most probably as the difference in obtained moisture results at different temperature was negligible.

ILC participating laboratories were requested to report the detection and quantification limits of their analytical procedures used in this ILC. Those method parameters are an element of method validation and are especially important for the determination of analytes at sub ng g^{-1} levels. All results were reported with the associated detection and quantification limit of the

applied analytical procedure. In general, quantification limits were correctly estimated when compared with reported values and/or with reference values.

All information reported by participating laboratories underlie their proper application of quality control procedures and traceability.

6 CONCLUSIONS

The current ILC was designated to evaluate the analytical capabilities of selected laboratories regularly involved in the characterization campaign of IAEA CRMs.

Almost 95% of reported values were assessed as satisfactory based on *z*-scores which demonstrate the accuracy of results produced by the selected laboratories. On the other hand, a non-negligible part of results was not considered satisfactory based on Zeta-scores (11%), indicating a tendency of under estimating uncertainties associated with the reported results.

Participating laboratories are encouraged to carefully investigate the cause of any unsatisfactory scores (i.e. $|z|$ or $|\text{Zeta}| > 3$) and put in place the necessary corrective actions to prevent reoccurrence of the problem. This is a requirement for accreditation to ISO/IEC 17025:2017 [7].

The implementation of the Minamata convention [8], especially article 19, should lead to an increasing number of laboratories involved in the monitoring of mercury (Hg), including mercury species. There are many participating laboratories performing Hg measurement (81%) and all the results produced are assessed as satisfactory which is encouraging. The proportion of participating laboratories performing mercury speciation is still low (3 laboratories, 27% of participating laboratories), but reported values were all assessed as satisfactory.

As a follow-up action of their participation in this ILC, participating laboratories are encouraged to contact the organizers to get more information on the above points, as necessary.

APPENDIX

REPORTED RESULTS BY ELEMENTS

A.1. EVALUATION OF REPORTED DATA FOR Ag

Kernel density Plot

< 8 data

Summary of results:

X_{info}:	13.33 mg kg^{-1}
U_{info} (k=2):	0.62 mg kg^{-1}
$2\sigma_p$:	3.3 mg kg^{-1}
Number of results:	10
Number of method:	4

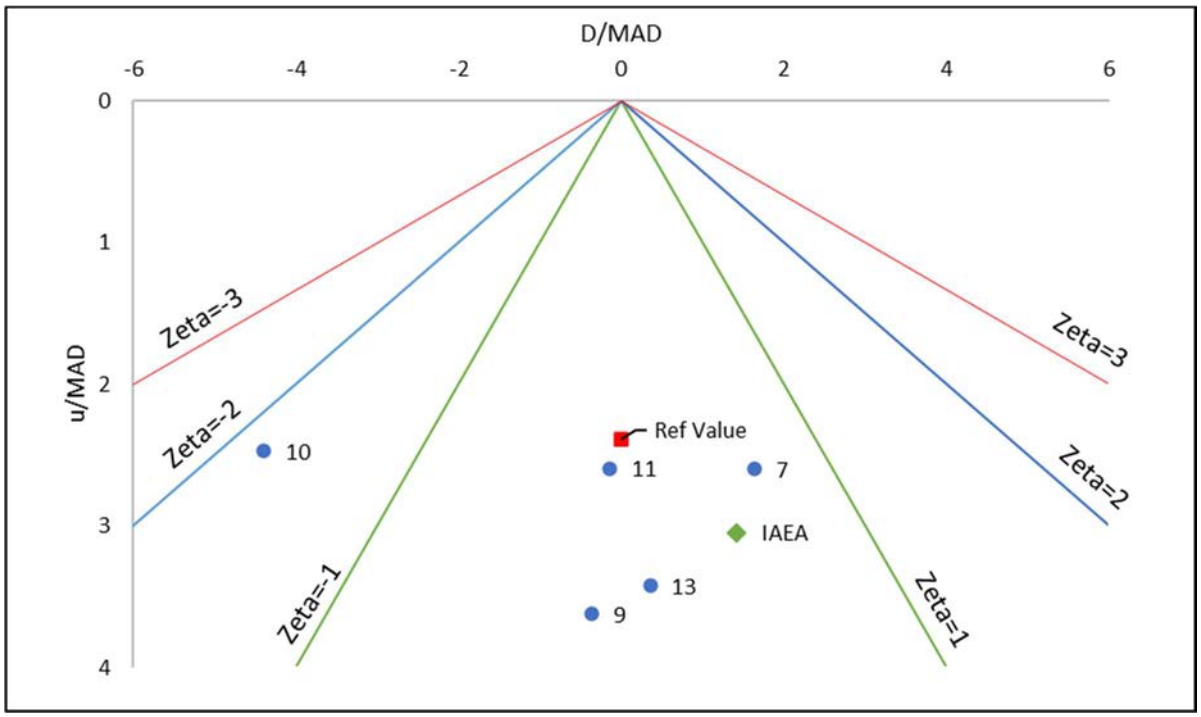
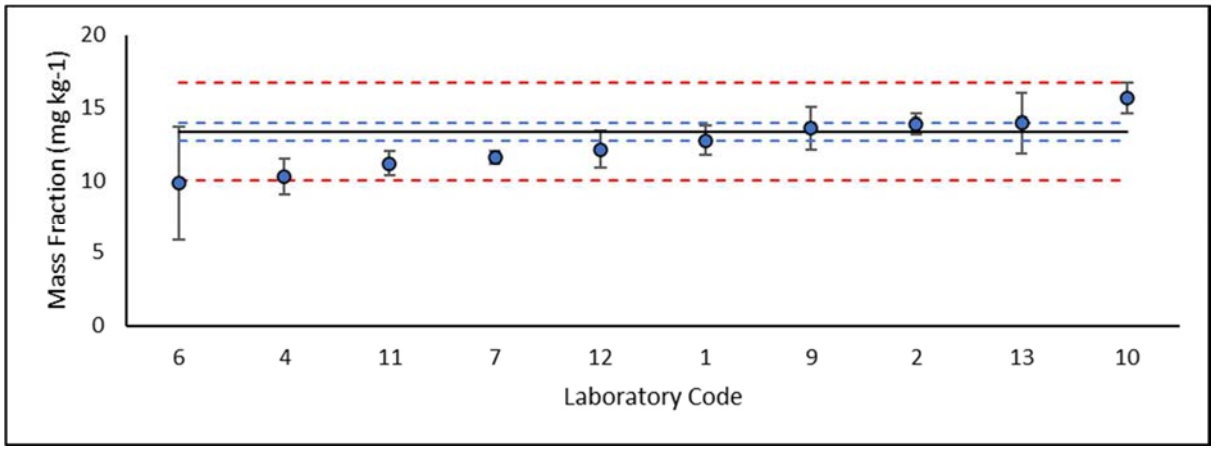


FIG. 6. PomPlot: Number are laboratory codes. (See Section 4. for more details).



— X_{ass} ; $X_{lab} \pm U_{lab}$; ---- $X_{ass} \pm 2\sigma_p$; ---- $X_{ass} \pm U_{ass}(k=2)$

FIG. 7. Reported results and expanded uncertainties.

A.2. EVALUATION OF REPORTED DATA FOR Al

Kernel density Plot

< 8 data

Summary of results:

X_{info}:	11.1 mg kg^{-1}
U_{info} (k=2):	4.6 mg kg^{-1}
$2\sigma_p$:	2.8 mg kg^{-1}
Number of results:	6
Number of method:	2

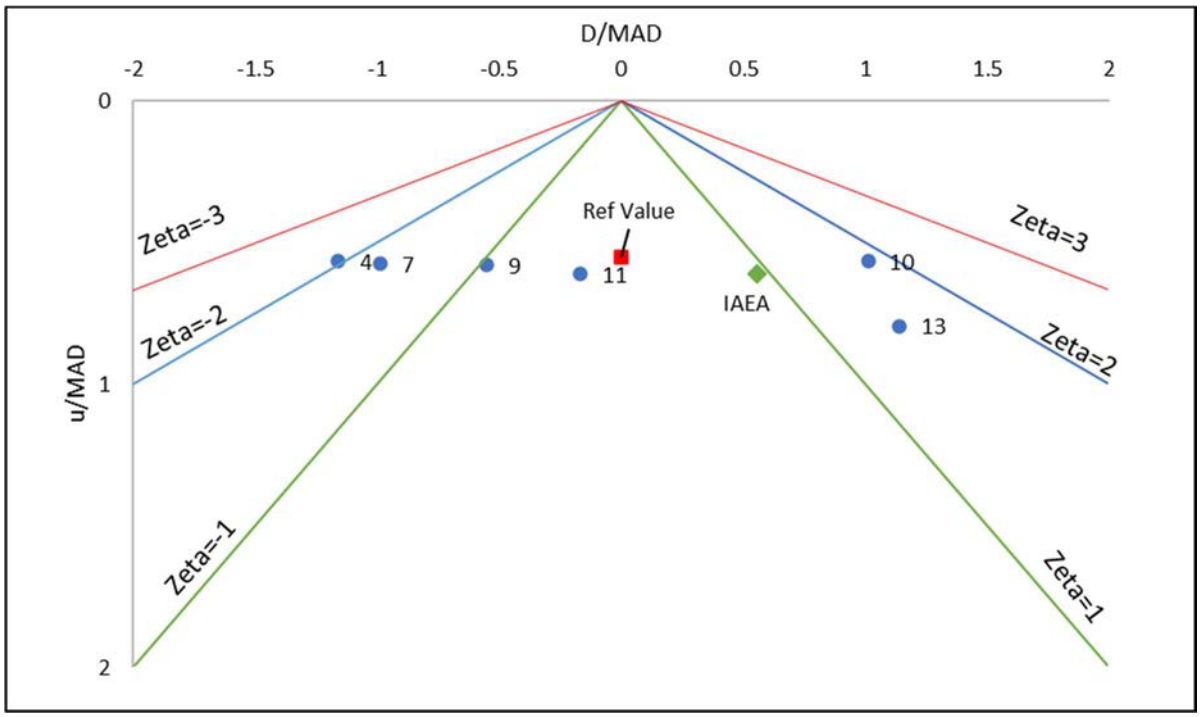
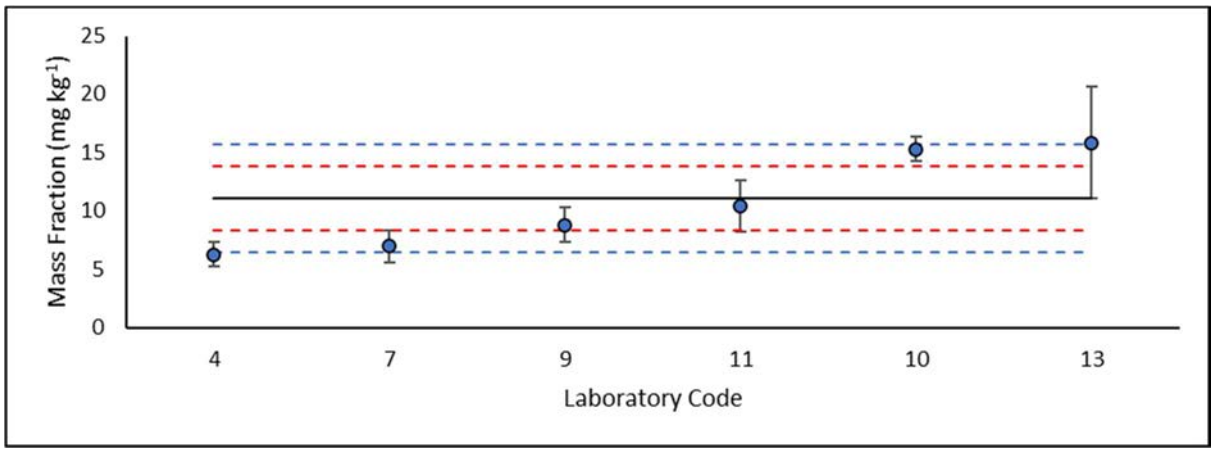


FIG. 8. PomPlot: Number are laboratory codes. (See Section 4. for more details).



— X_{ass} ; ⊥ $X_{lab} \pm U_{lab}$; ---- $X_{ass} \pm 2\sigma_p$; ---- $X_{ass} \pm U_{ass}(k=2)$

FIG. 9. Reported results and expanded uncertainties.

A.3. EVALUATION OF REPORTED DATA FOR As

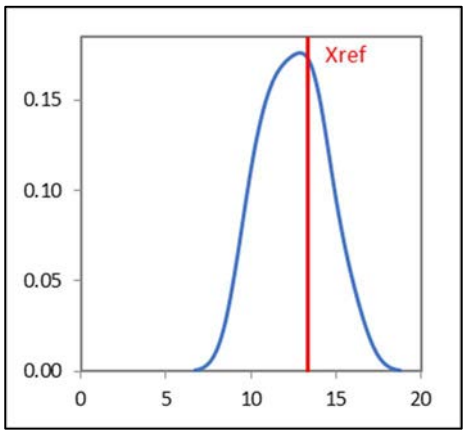


FIG. 10 Kernel density Plot.

Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	90%	10%	0%
Zeta-score	60%	0%	40%

X_{ref}:	13.33 mg kg^{-1}
U_{ref} (k=2):	0.62 mg kg^{-1}
$2\sigma_p$:	3.3 mg kg^{-1}
Number of results:	10
Number of method:	4

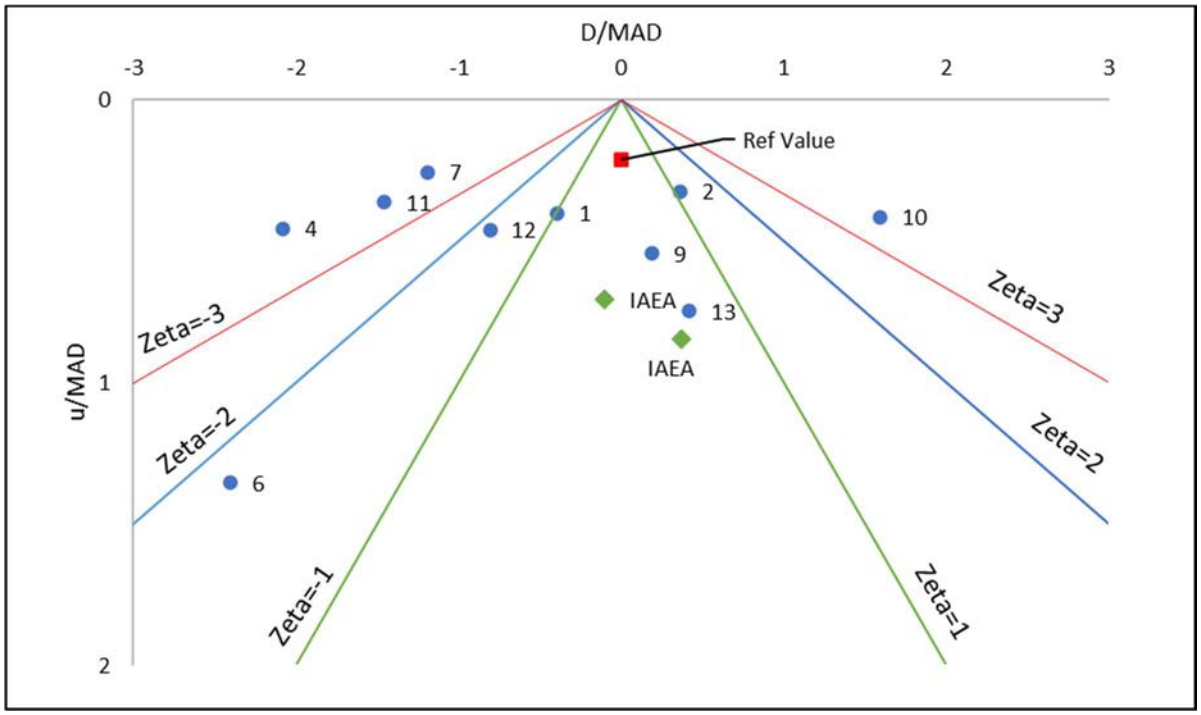
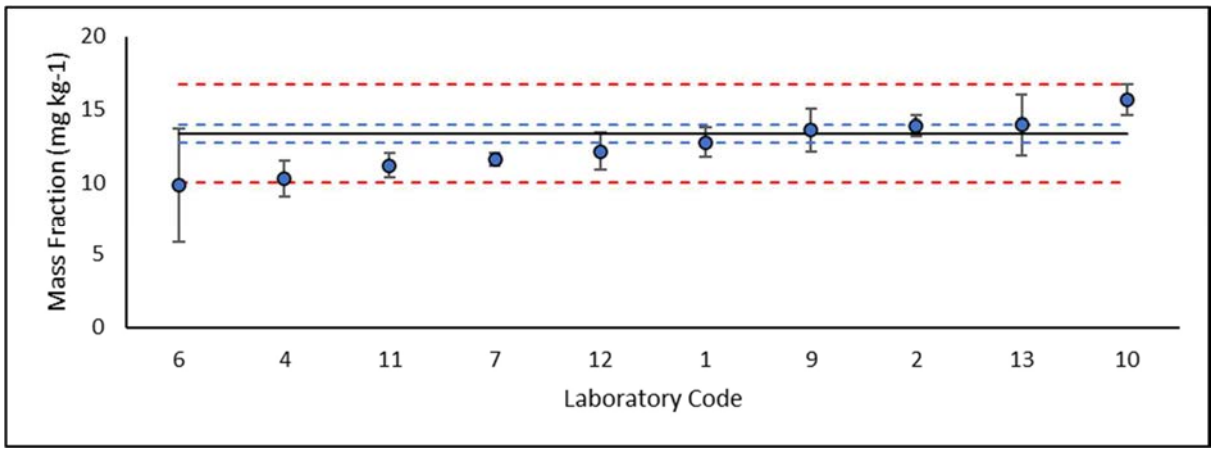


FIG. 11. PomPlot: Number are laboratory codes. (See Section 4. for more details).



— X_{ass} ; $X_{lab} \pm U_{lab}$; ---- $X_{ass} \pm 2\sigma_p$; ---- $X_{ass} \pm U_{ass}(k=2)$

FIG. 12. Reported results and expanded uncertainties.

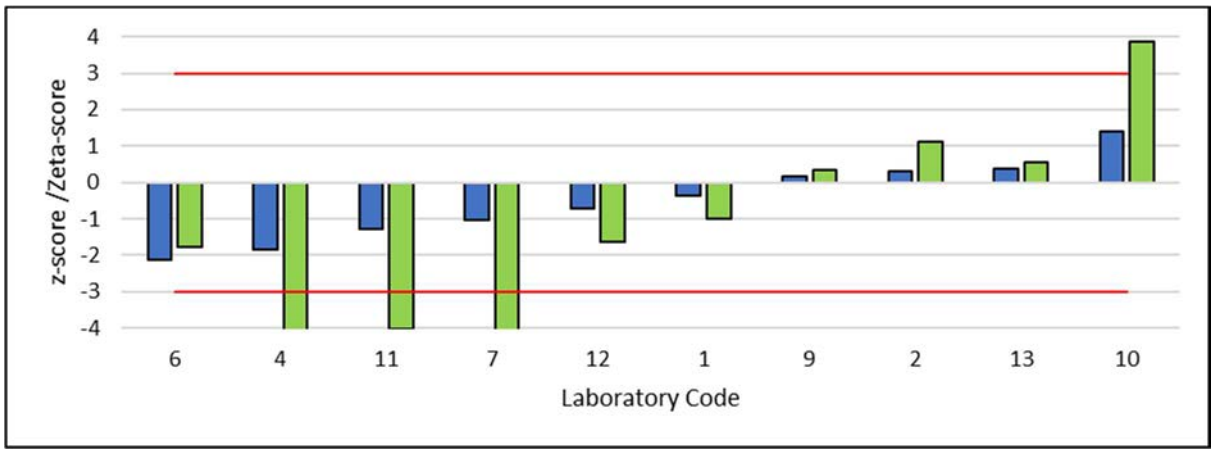


FIG. 13. Performance evaluation: *z*-score Zeta-score.

A.4. EVALUATION OF REPORTED DATA FOR Ca

Kernel density Plot

< 8 data

Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	86%	0%	14%
Zeta-score	71%	0%	29%

X_{ref}:	24.5 g kg^{-1}
U_{ref} (k=2):	3.0 g kg^{-1}
$2\sigma_p$:	6.1 mg kg^{-1}
Number of results:	7
Number of method:	4

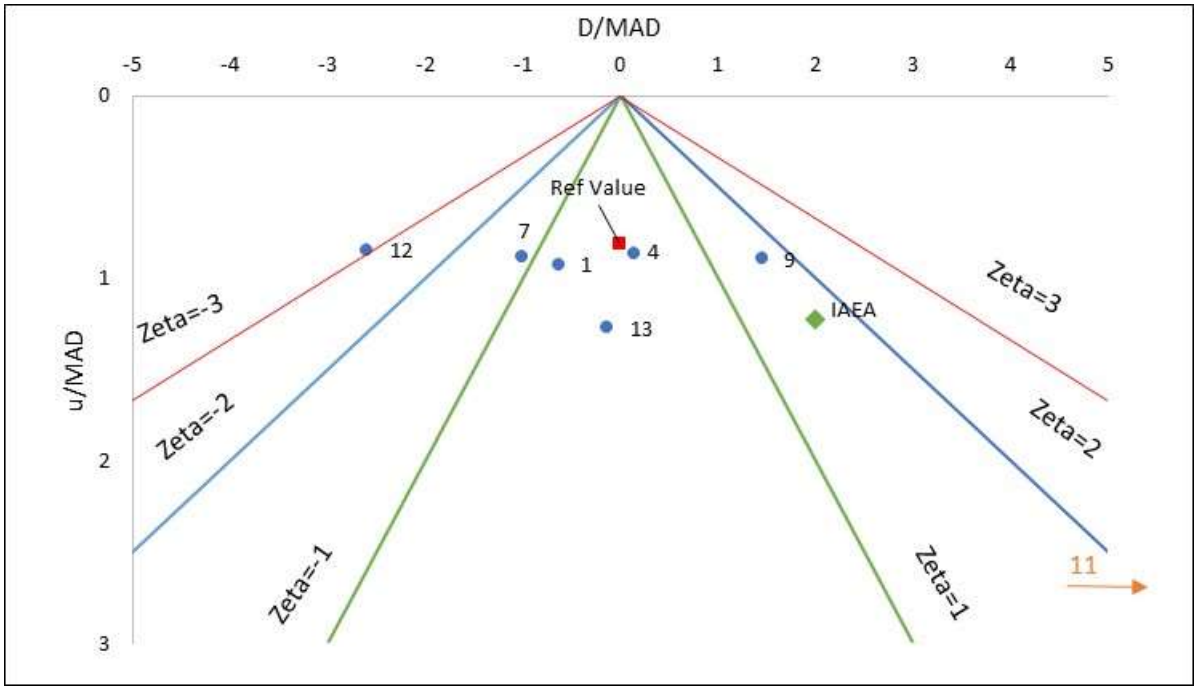
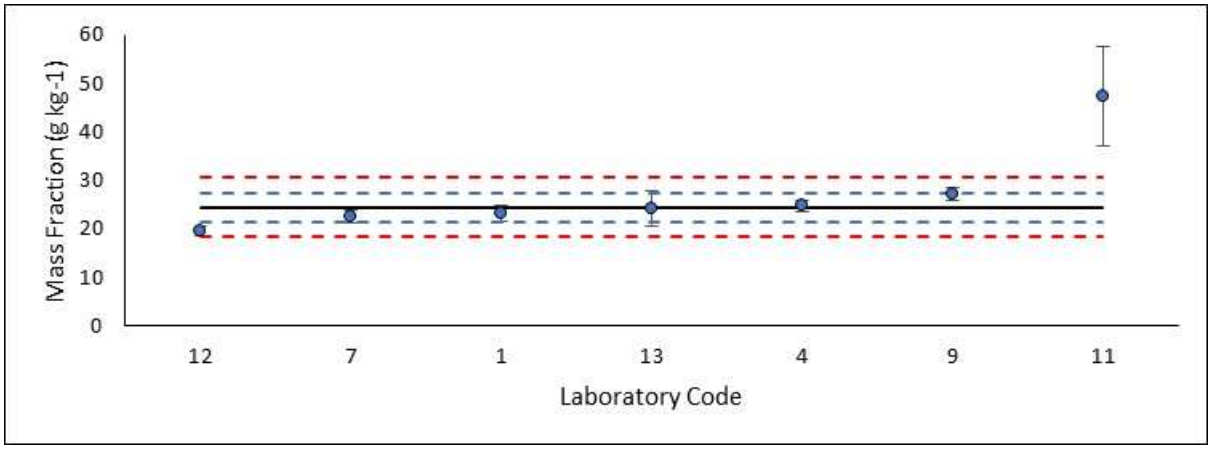


FIG. 14. PomPlot: Number are laboratory codes. (See Section 4. for more details).



— X_{ass} ; $\pm X_{lab} \pm U_{lab}$; ---- $X_{ass} \pm 2\sigma_p$; ---- $X_{ass} \pm U_{ass}(k=2)$

FIG. 15. Reported results and expanded uncertainties.

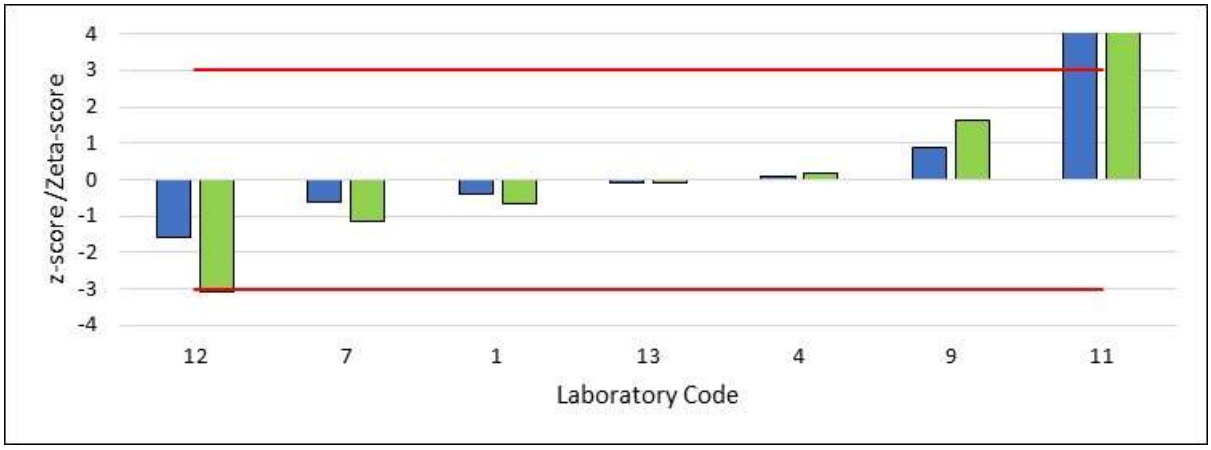


FIG. 16. Performance evaluation: *z*-score Zeta-score.

A.5. EVALUATION OF REPORTED DATA FOR Cd

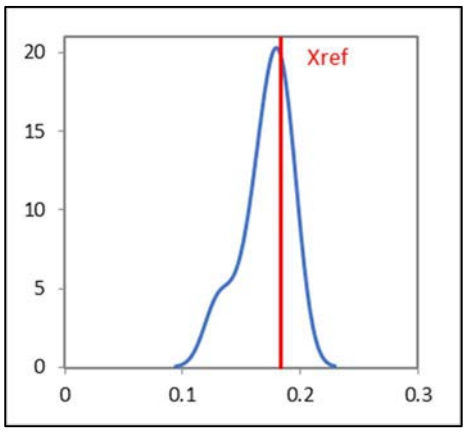


FIG. 17 Kernel density Plot.

Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	88%	13%	0%
Zeta-score	75%	13%	13%

X_{ref}:	0.184 mg kg^{-1}
U_{ref} (k=2):	0.021 mg kg^{-1}
$2\sigma_p$:	6.1 mg kg^{-1}
Number of results:	8
Number of method:	2

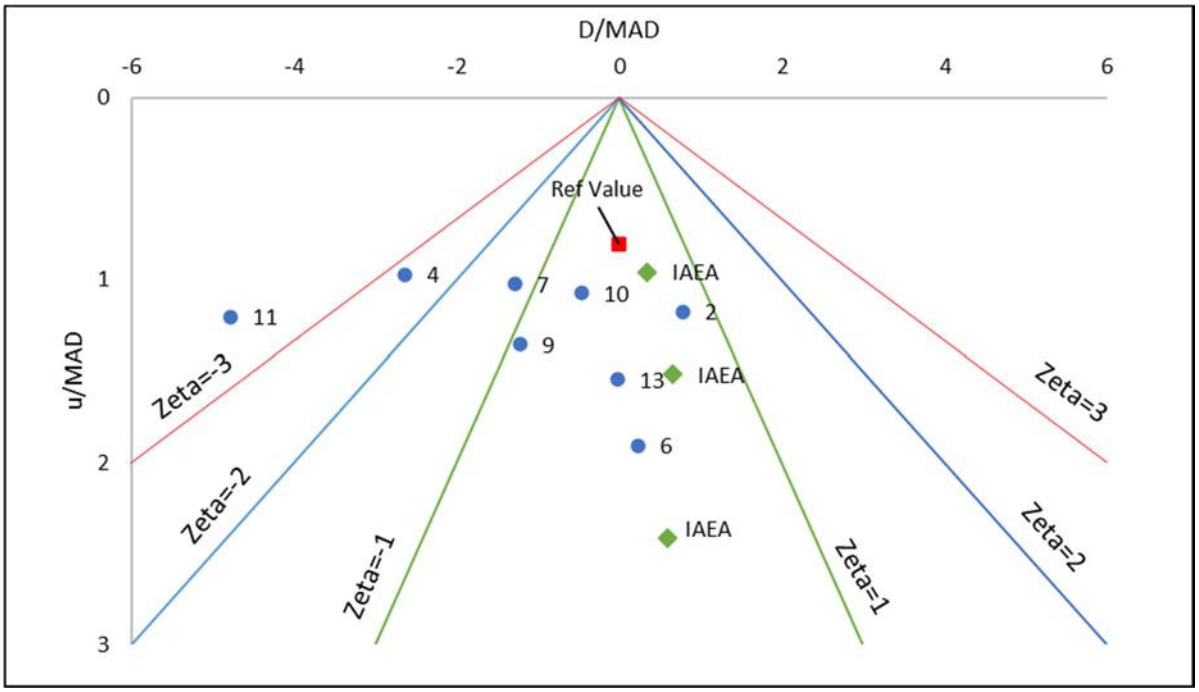
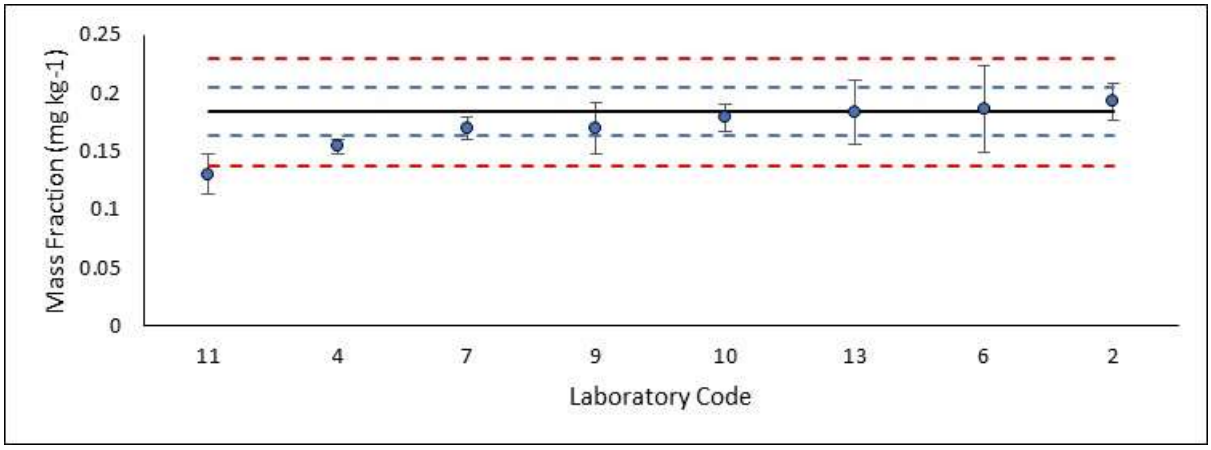


FIG. 18. PomPlot: Number are laboratory codes. (See Section 4. for more details).



— X_{ass} ; $X_{lab} \pm U_{lab}$; ---- $X_{ass} \pm 2\sigma_p$; ---- $X_{ass} \pm U_{ass}(k=2)$

FIG. 19. Reported results and expanded uncertainties.

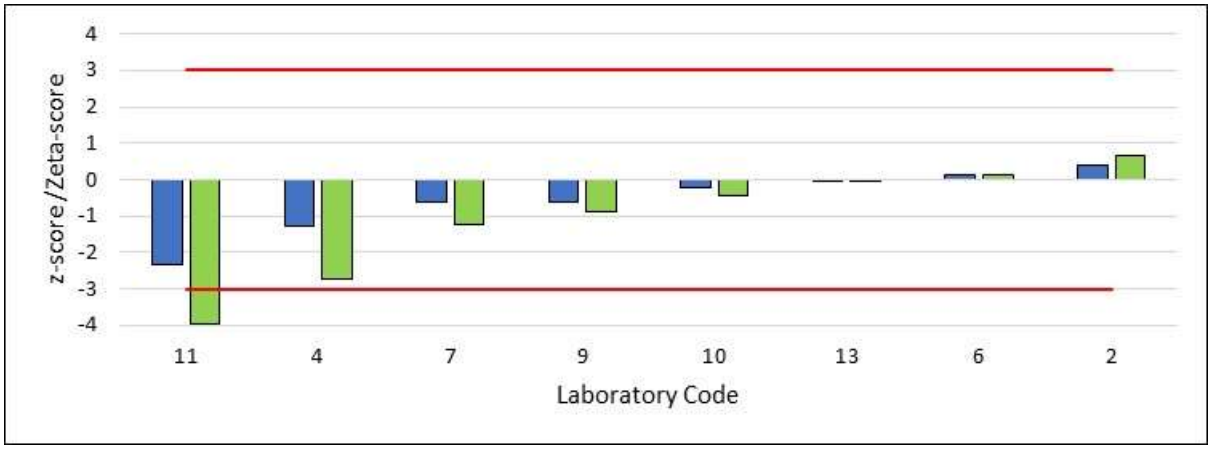


FIG. 20. Performance evaluation: *z*-score Zeta-score.

A.6. EVALUATION OF REPORTED DATA FOR Co

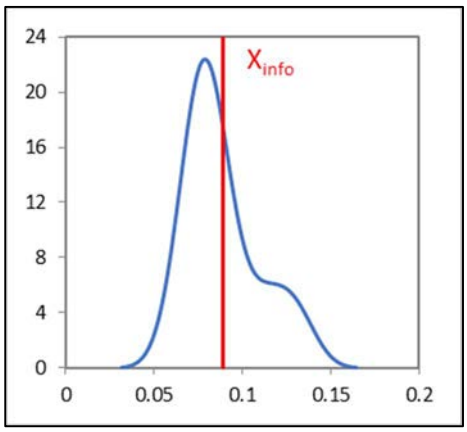


FIG. 21 Kernel density Plot.

Summary of results:

X_{info}:	0.089 mg kg^{-1}
U_{info} (k=2):	0.018 mg kg^{-1}
$2\sigma_p$:	0.022 mg kg^{-1}
Number of results:	9
Number of method:	2

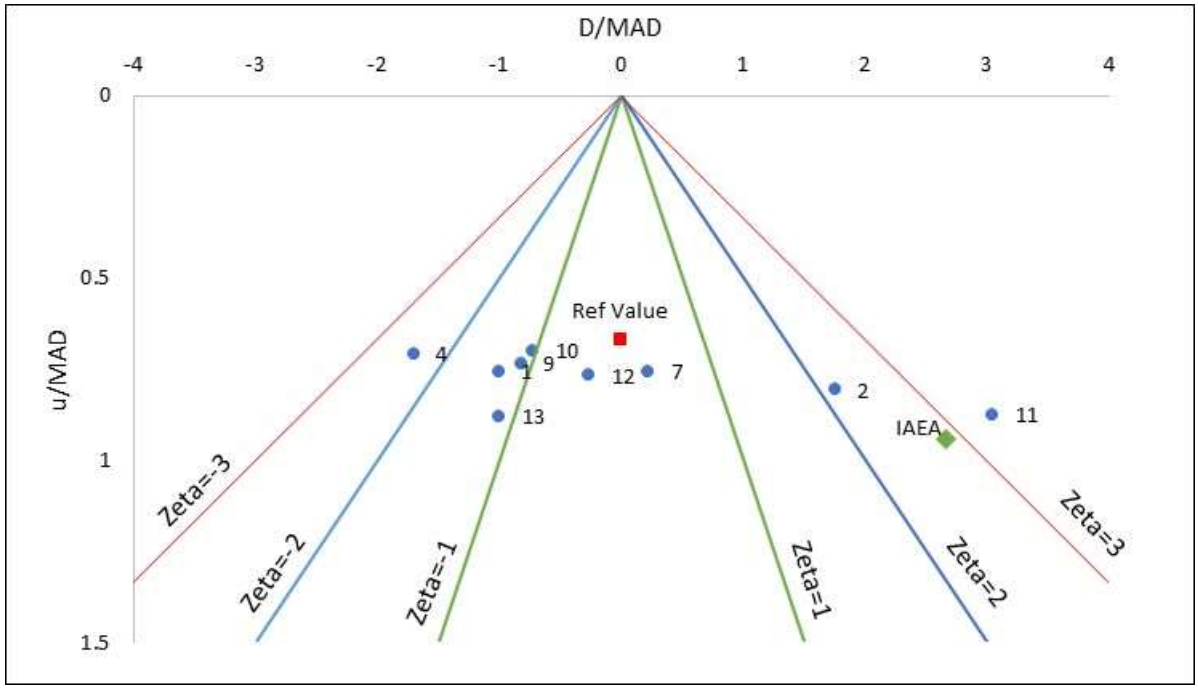
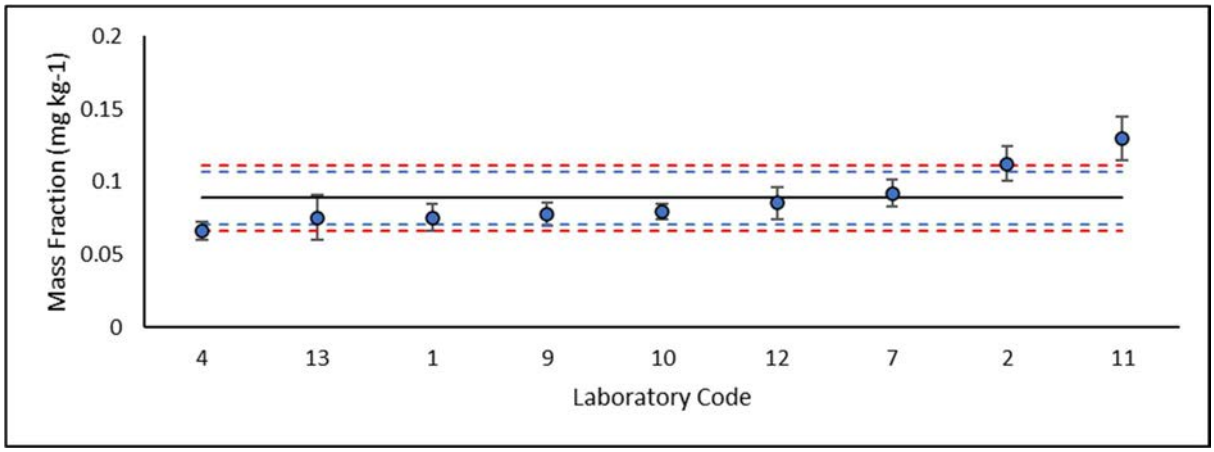


FIG. 22. PomPlot: Number are laboratory codes. (See Section 4. for more details).



— X_{ass} ; $\pm X_{lab} \pm U_{lab}$; ---- $X_{ass} \pm 2\sigma_p$; ---- $X_{ass} \pm U_{ass}(k=2)$

FIG. 23. Reported results and expanded uncertainties.

A.7. EVALUATION OF REPORTED DATA FOR Cr

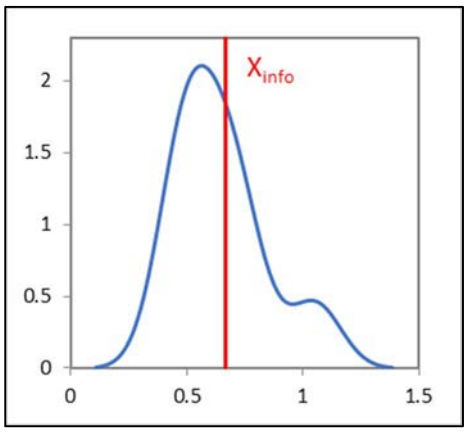


FIG. 24 Kernel density Plot.

Summary of results:

X_{info}:	0.089 mg kg^{-1}
U_{info} (k=2):	0.018 mg kg^{-1}
$2\sigma_p$:	0.022 mg kg^{-1}
Number of results:	9
Number of method:	2

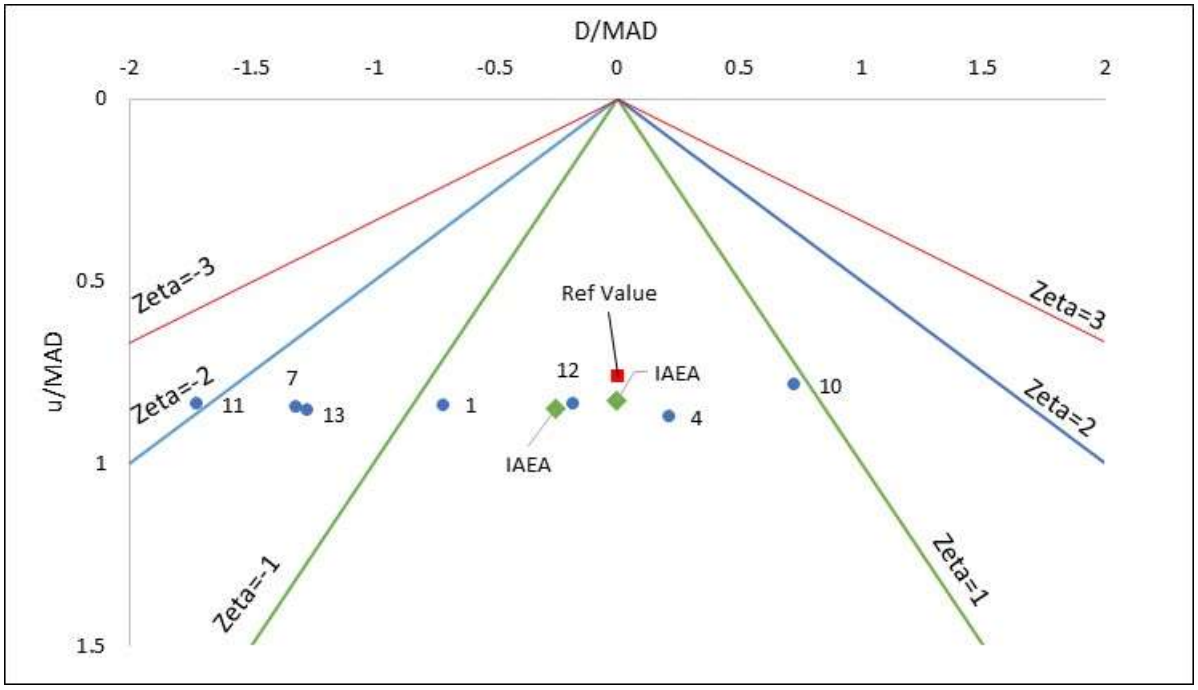
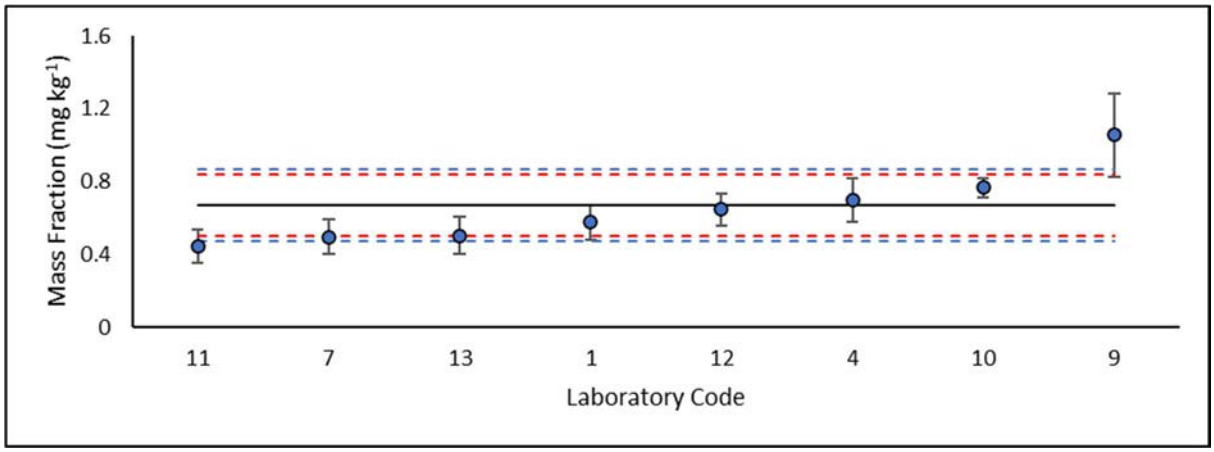


FIG. 25. PomPlot: Number are laboratory codes. (See Section 4. for more details).



— X_{ass} ; $\pm X_{lab} \pm U_{lab}$; ---- $X_{ass} \pm 2\sigma_p$; ---- $X_{ass} \pm U_{ass}(k=2)$

FIG. 26. Reported results and expanded uncertainties.

A.8. EVALUATION OF REPORTED DATA FOR Cu

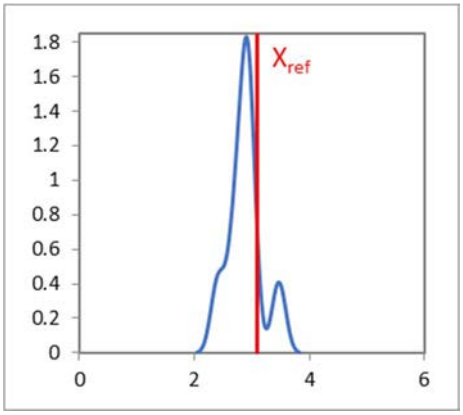


FIG. 27 Kernel density Plot.

Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	100%	0%	0%
Zeta-score	50%	50%	11%

X_{ref}:	3.09 mg kg^{-1}
U_{ref} (k=2):	0.21 mg kg^{-1}
$2\sigma_p$:	0.39 mg kg^{-1}
Number of results:	8
Number of method:	2

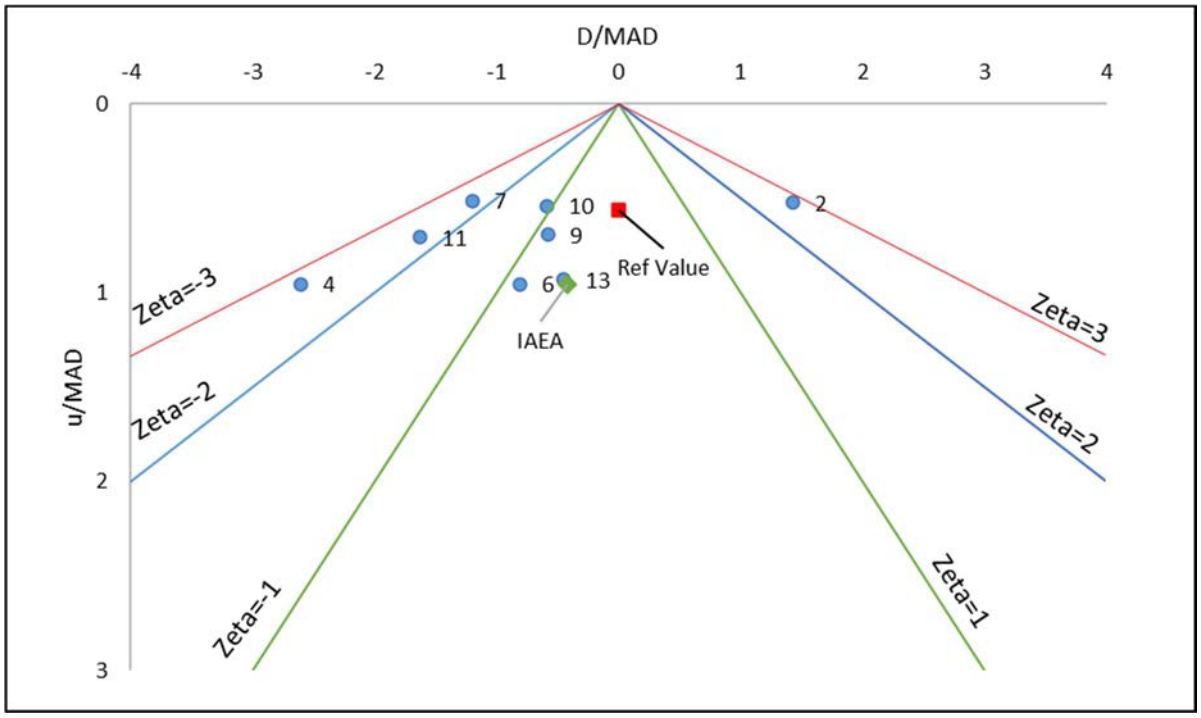
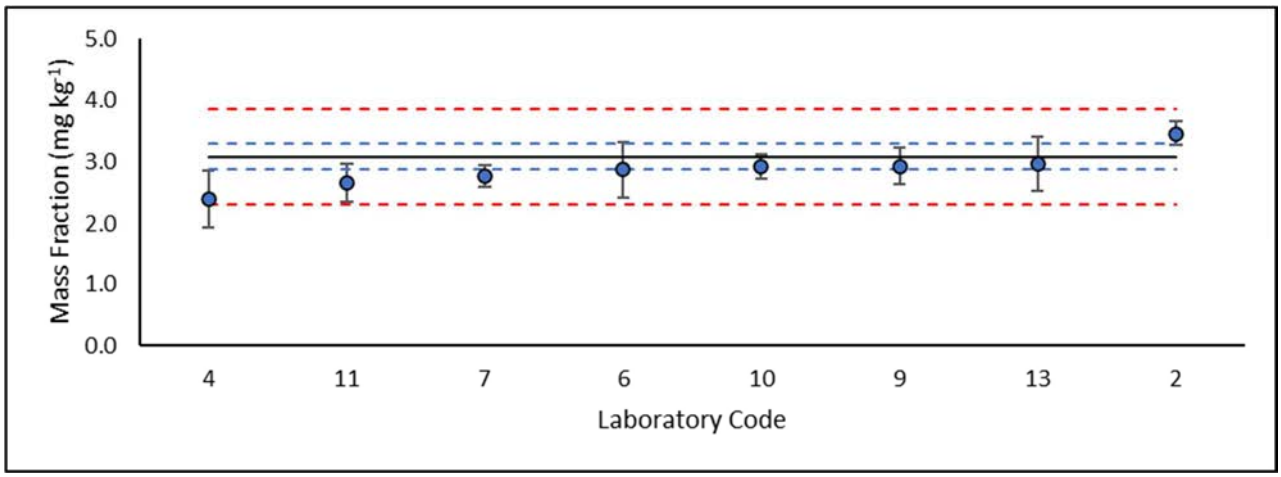


FIG. 28. PomPlot: Number are laboratory codes. (See Section 4. for more details).



— X_{ass} ; $\pm X_{lab} \pm U_{lab}$; ---- $X_{ass} \pm 2\sigma_p$; ---- $X_{ass} \pm U_{ass}(k=2)$

FIG. 29. Reported results and expanded uncertainties.

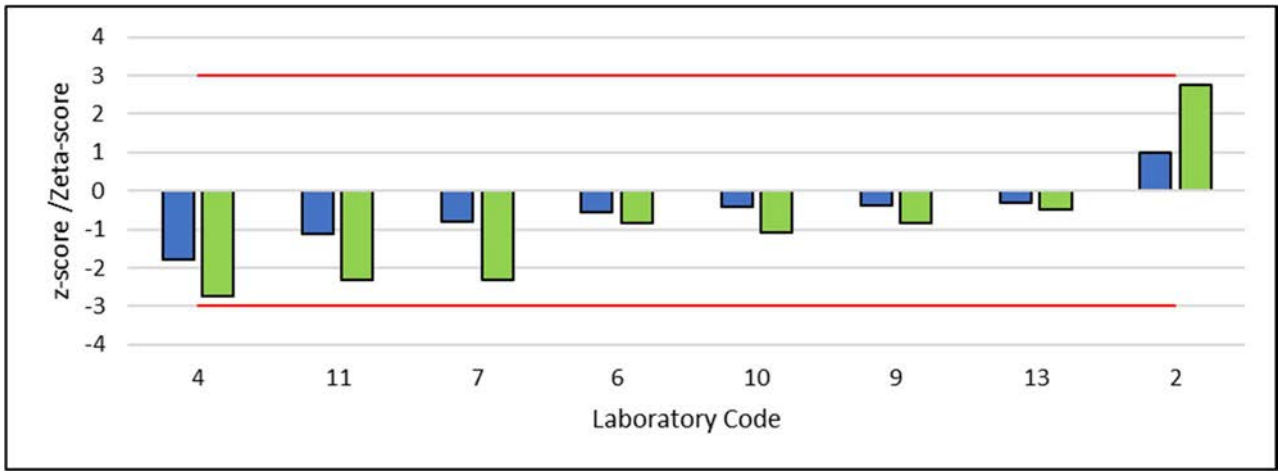


FIG. 30. Performance evaluation: ■ *z*-score ■ Zeta-score.

A.8. EVALUATION OF REPORTED DATA FOR Hg

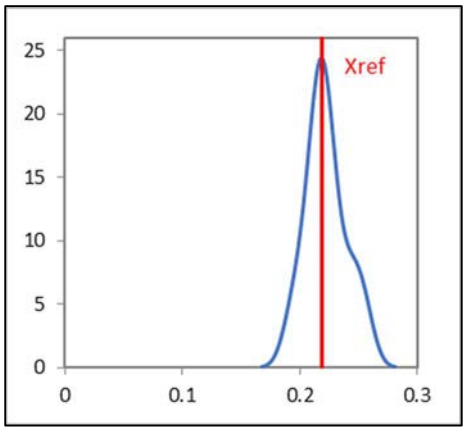


FIG. 31 Kernel density Plot.

Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	100%	0%	0%
Zeta-score	89%	0%	11%

X_{ref}:	0.219 mg kg^{-1}
U_{ref} (k=2):	0.011 mg kg^{-1}
$2\sigma_p$:	0.055 mg kg^{-1}
Number of results:	9
Number of method:	5

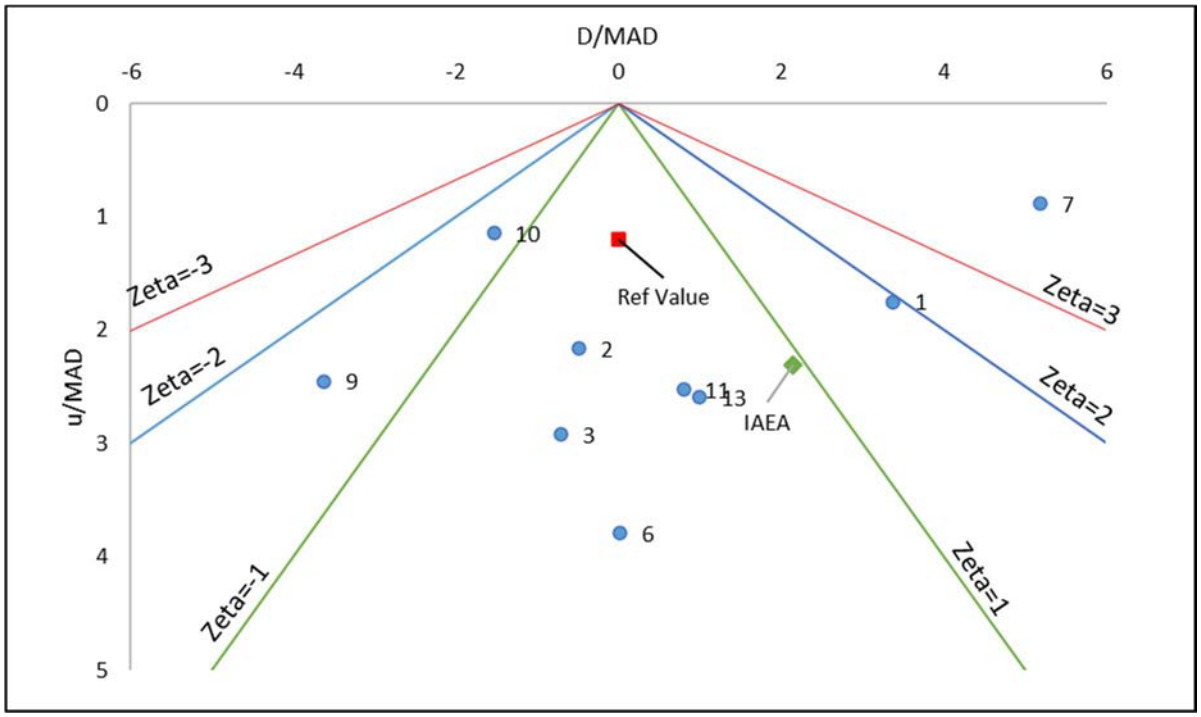
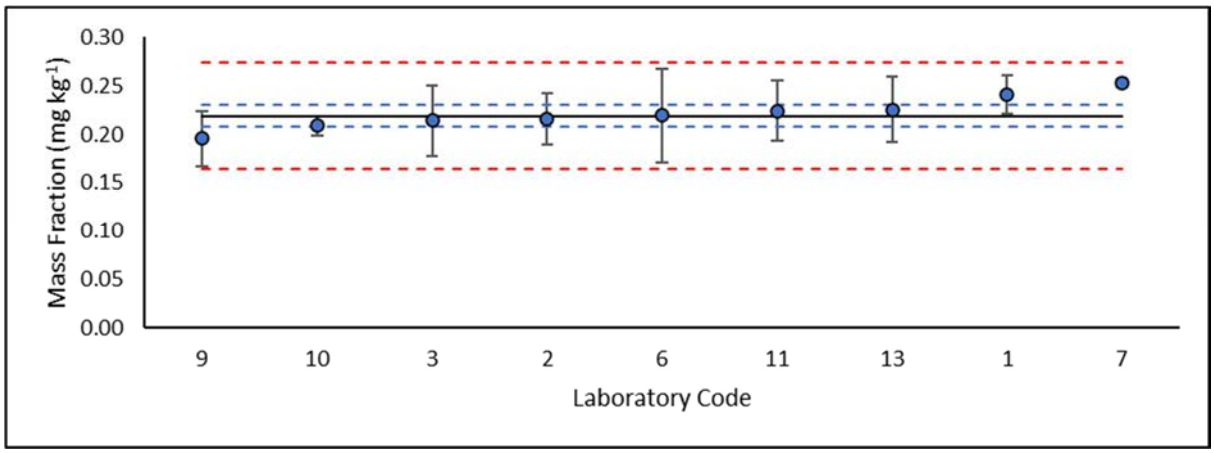


FIG. 31. PomPlot: Number are laboratory codes. (See Section 4. for more details).



— X_{ass} ; $X_{lab} \pm U_{lab}$; ---- $X_{ass} \pm 2\sigma_p$; ---- $X_{ass} \pm U_{ass}(k=2)$

FIG. 32. Reported results and expanded uncertainties.

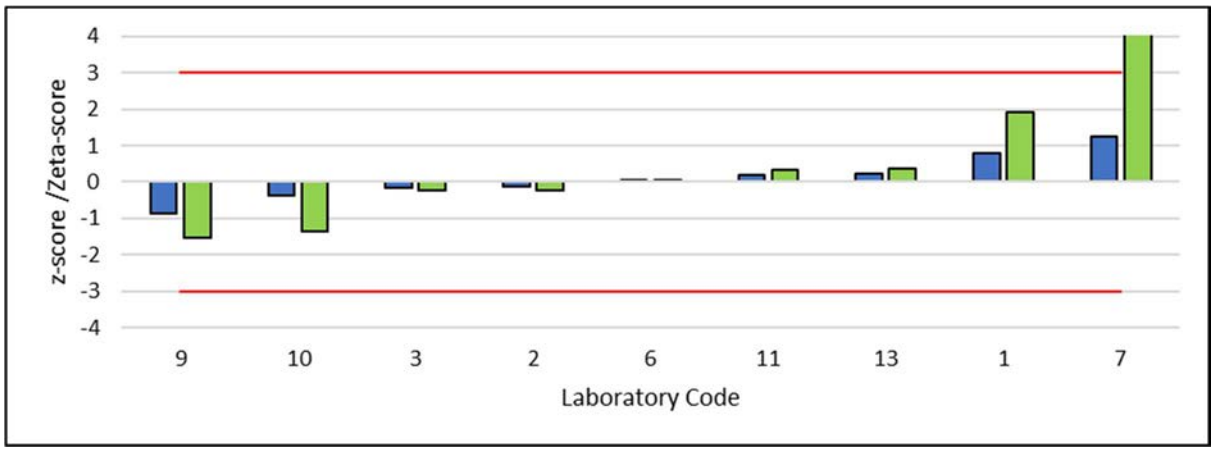


FIG. 33. Performance evaluation: *z*-score Zeta-score.

A.9. EVALUATION OF REPORTED DATA FOR K

Kernel density Plot

<8 data

Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	100%	0%	0%
Zeta-score	80%	20%	0%

X_{ref}:	12.83 g kg^{-1}
U_{ref} (k=2):	0.95 g kg^{-1}
$2\sigma_p$:	3.21 g kg^{-1}
Number of results:	5
Number of method:	3

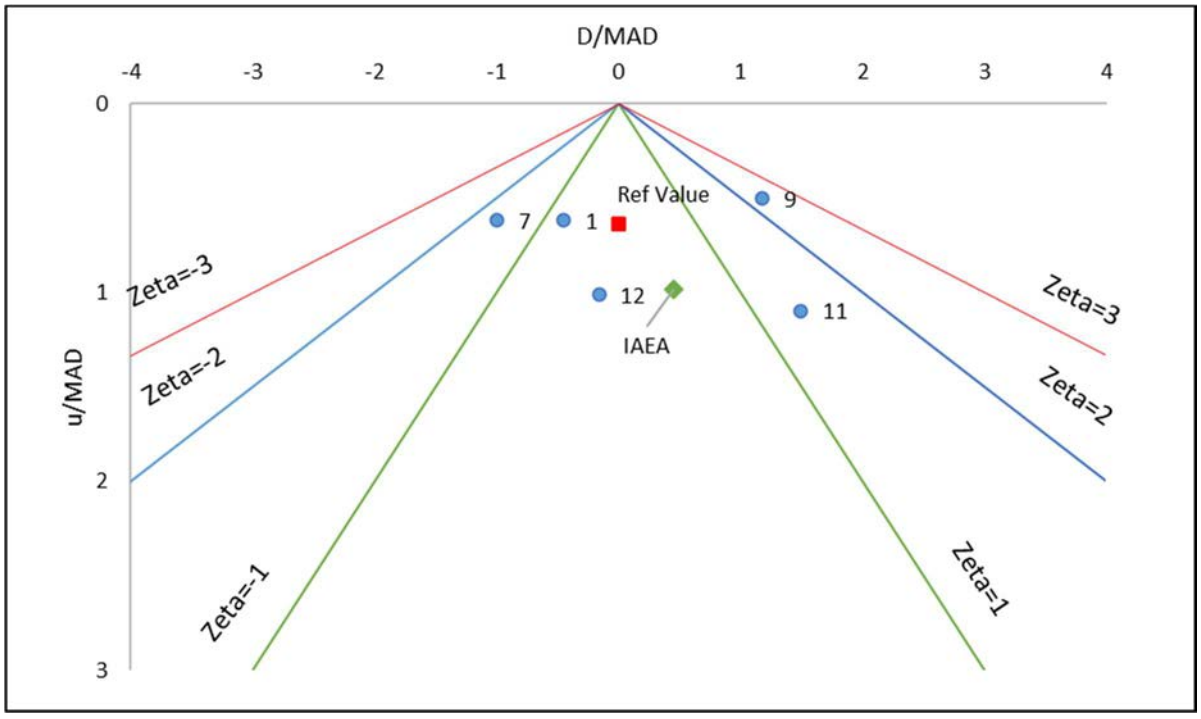
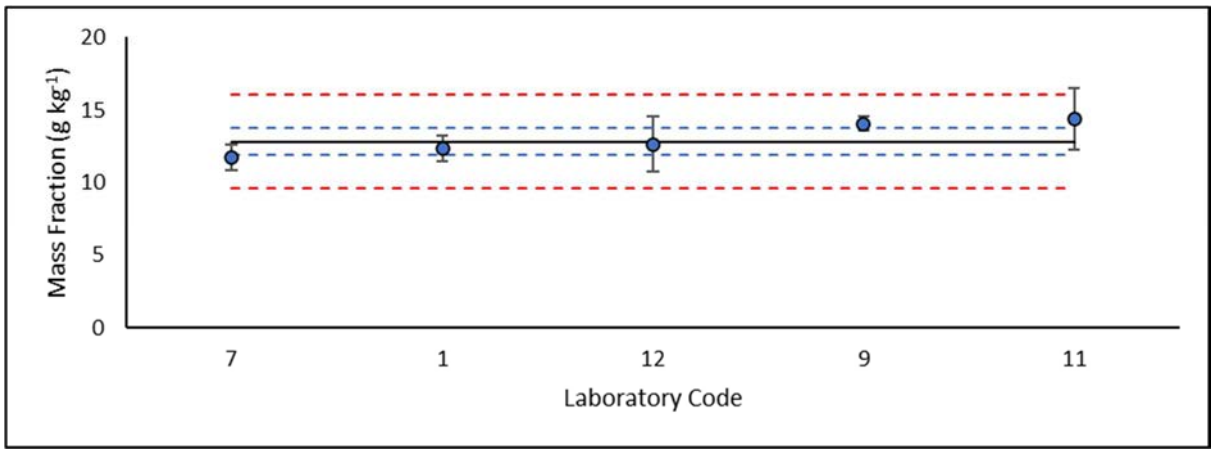


FIG. 34. PomPlot: Number are laboratory codes. (See Section 4. for more details).



— X_{ass} ; $X_{lab} \pm U_{lab}$; ---- $X_{ass} \pm 2\sigma_p$; ---- $X_{ass} \pm U_{ass}(k=2)$

FIG. 35. Reported results and expanded uncertainties.

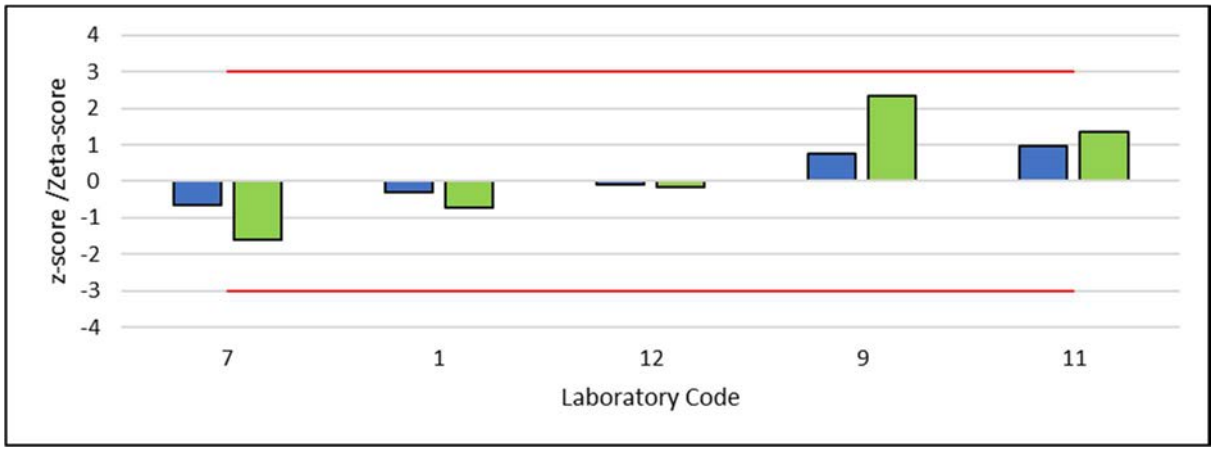


FIG. 36. Performance evaluation: *z*-score Zeta-score.

A.10. EVALUATION OF REPORTED DATA FOR Li

Kernel density Plot

< 8 data

Summary of results:

X_{info}:	0.70 mg kg^{-1}
U_{info} (k=2):	0.13 mg kg^{-1}
$2\sigma_p$:	0.17 mg kg^{-1}
Number of results:	4
Number of method:	1

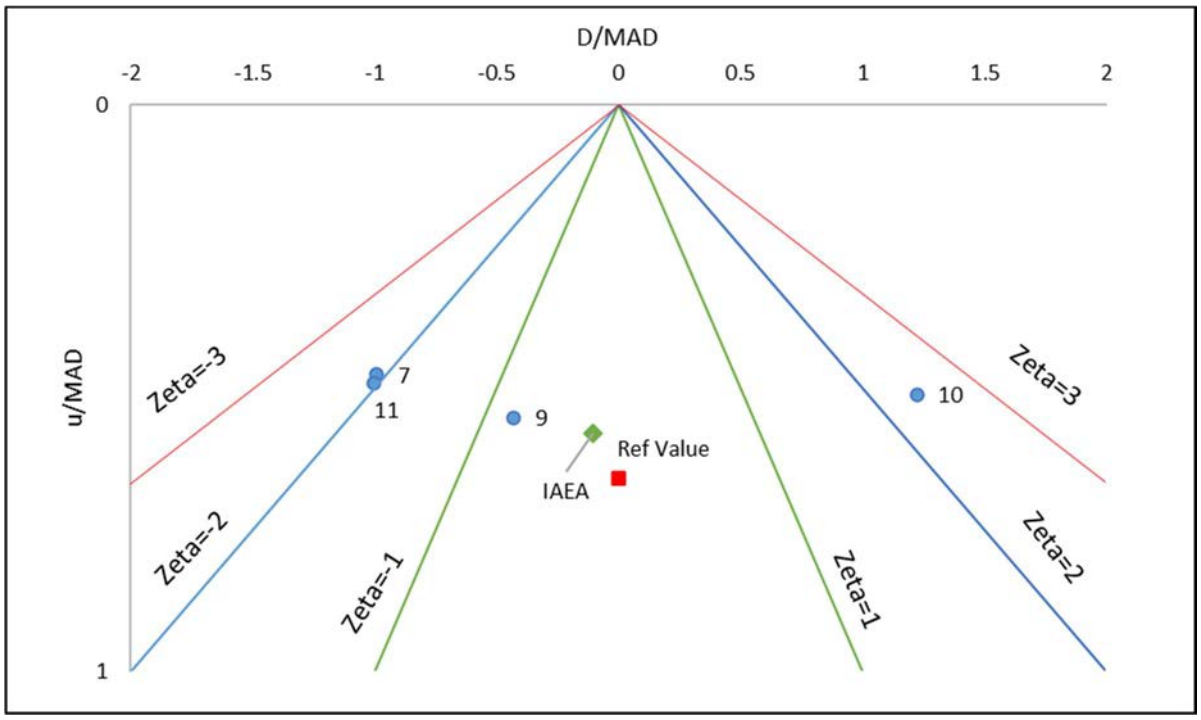
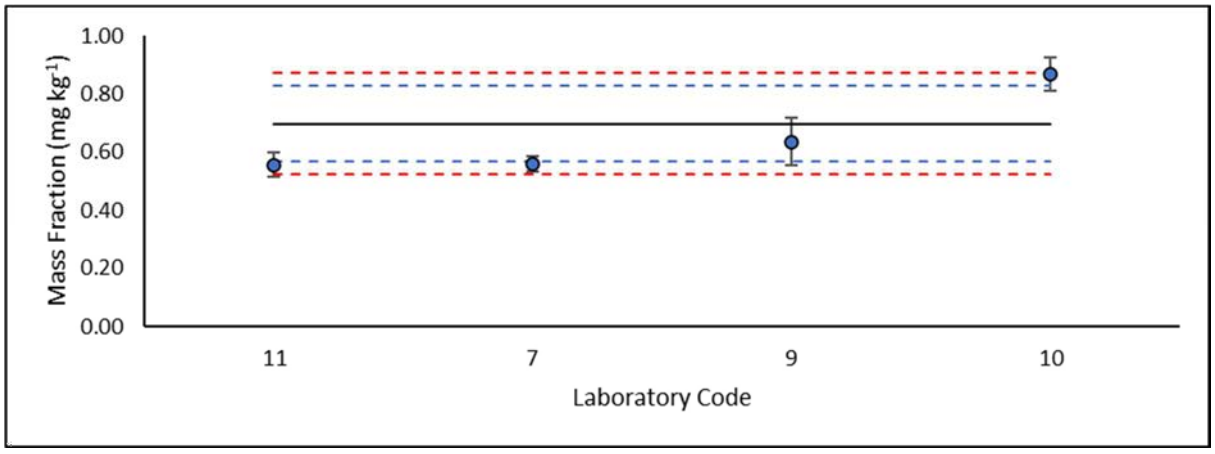


FIG. 37. PomPlot: Number are laboratory codes. (See Section 4. for more details).



— X_{ass} ; $\pm X_{lab} \pm U_{lab}$; ---- $X_{ass} \pm 2\sigma_p$; ---- $X_{ass} \pm U_{ass}(k=2)$

FIG. 38. Reported results and expanded uncertainties.

A.11. EVALUATION OF REPORTED DATA FOR MeHg

Kernel density Plot

<8 data

Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	100%	0%	0%
Zeta-score	100%	0%	0%

X_{ref}:	0.193 mg kg^{-1} as Hg
U_{ref} (k=2):	0.012 mg kg^{-1} as Hg
$2\sigma_p$:	0.048 mg kg^{-1} as Hg
Number of results:	3
Number of method:	1

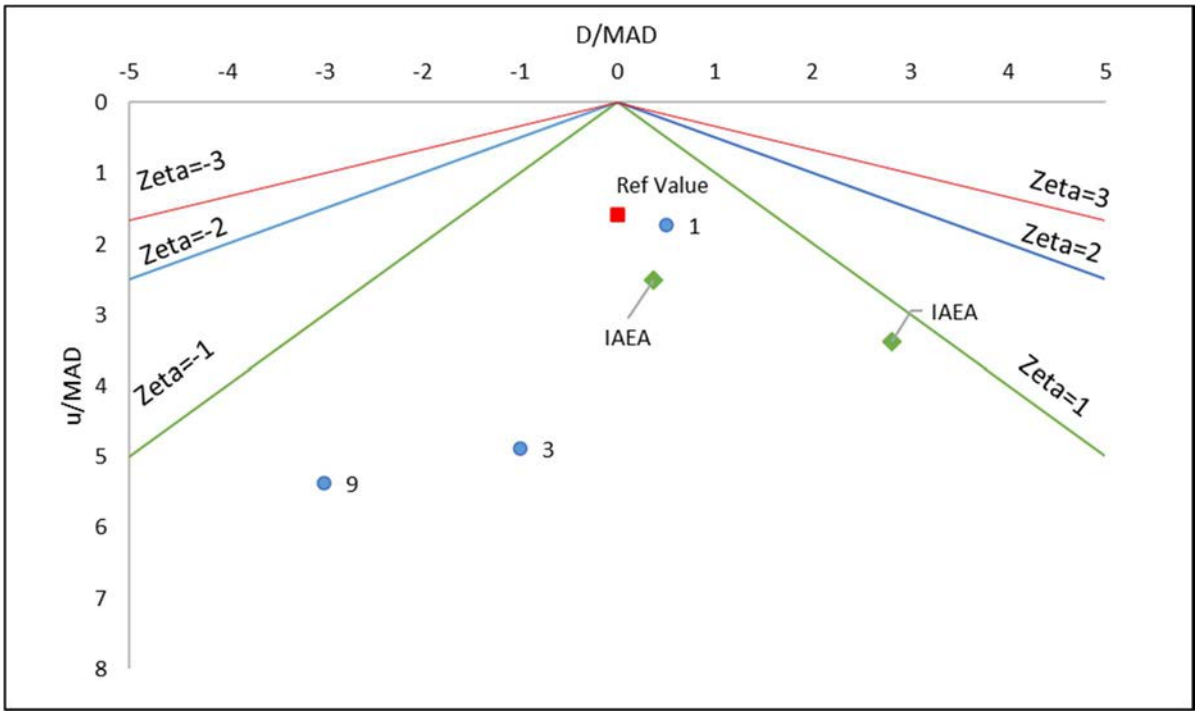
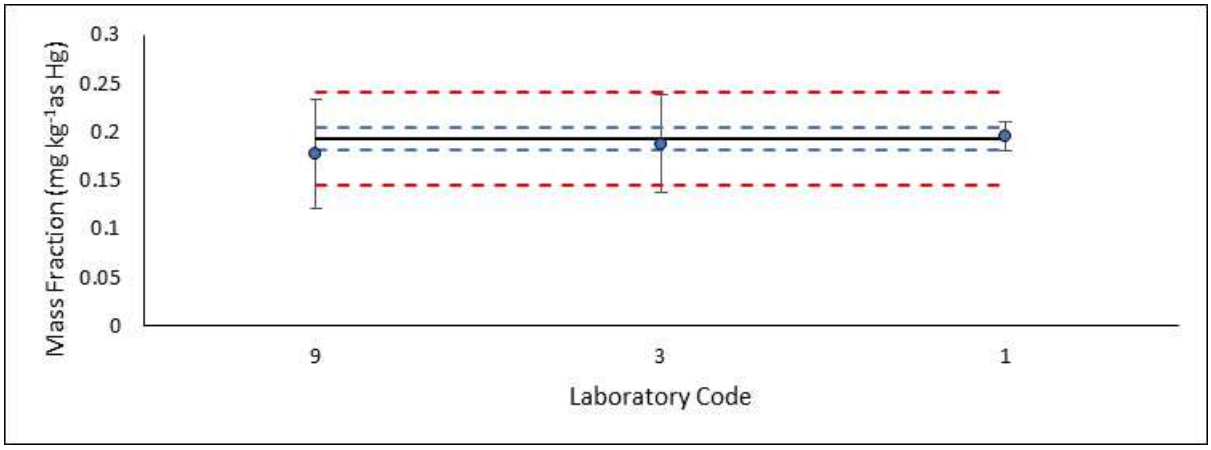


FIG. 39. PomPlot: Number are laboratory codes. (See Section 4. for more details).



— X_{ass} ; $\pm X_{lab} \pm U_{lab}$; ---- $X_{ass} \pm 2\sigma_p$; ---- $X_{ass} \pm U_{ass}(k=2)$

FIG. 40. Reported results and expanded uncertainties.

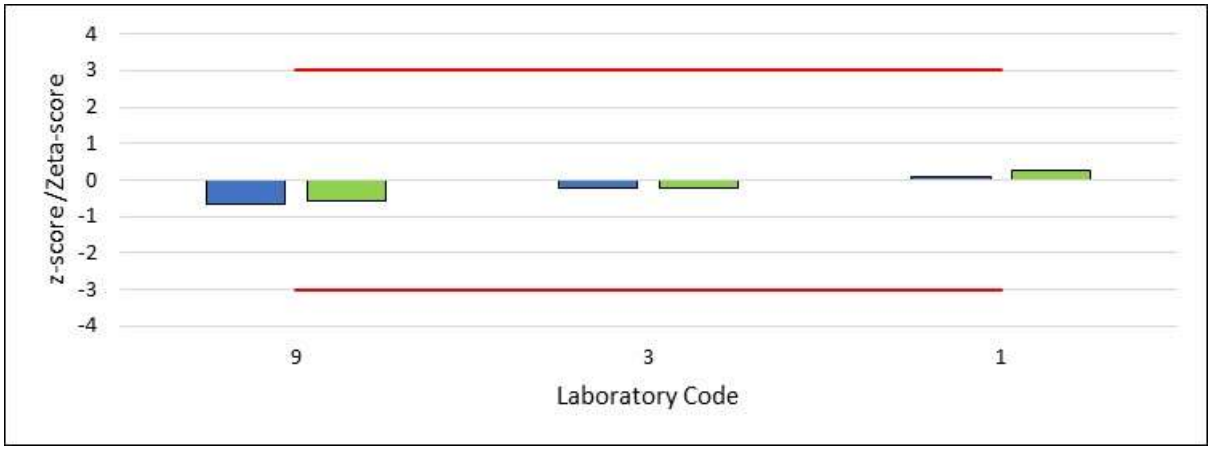


FIG. 41. Performance evaluation: *z*-score Zeta-score.

A.12. EVALUATION OF REPORTED DATA FOR Mg

Kernel density Plot

<8 data

Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	86%	0%	14%
Zeta-score	86%	0%	14%

X_{ref}:	2.39 g kg^{-1}
U_{ref} (k=2):	0.22 g kg^{-1}
$2\sigma_p$:	0.60 g kg^{-1}
Number of results:	7
Number of method:	3

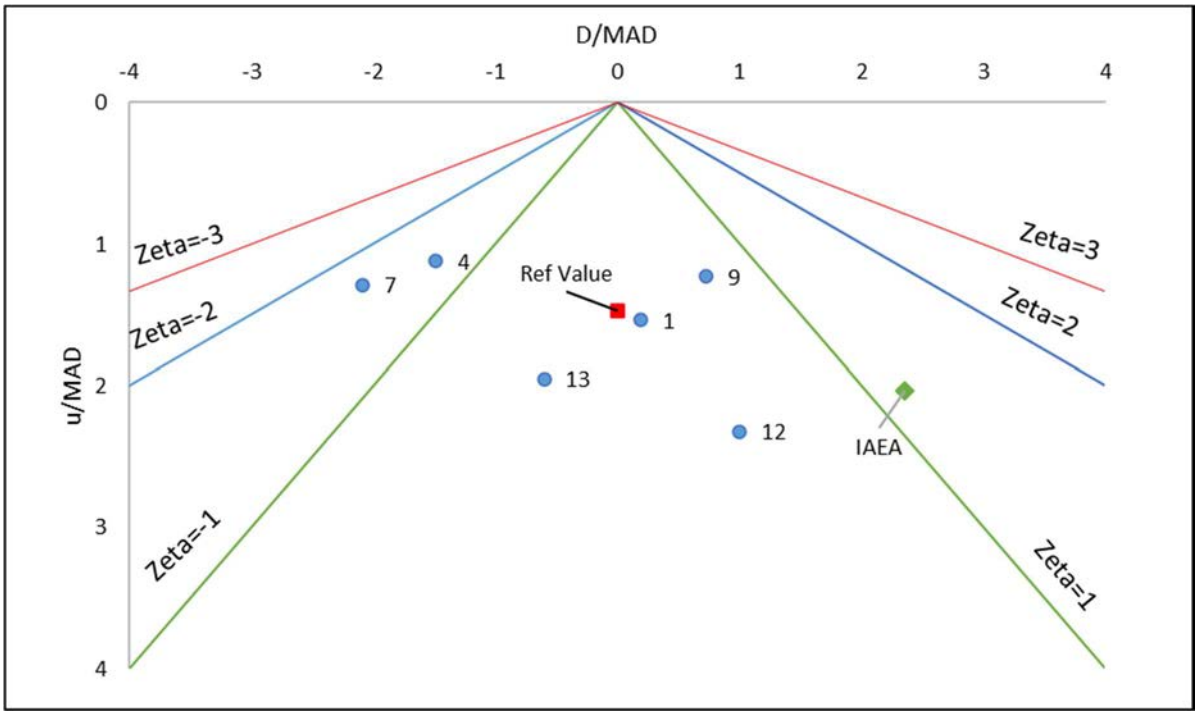
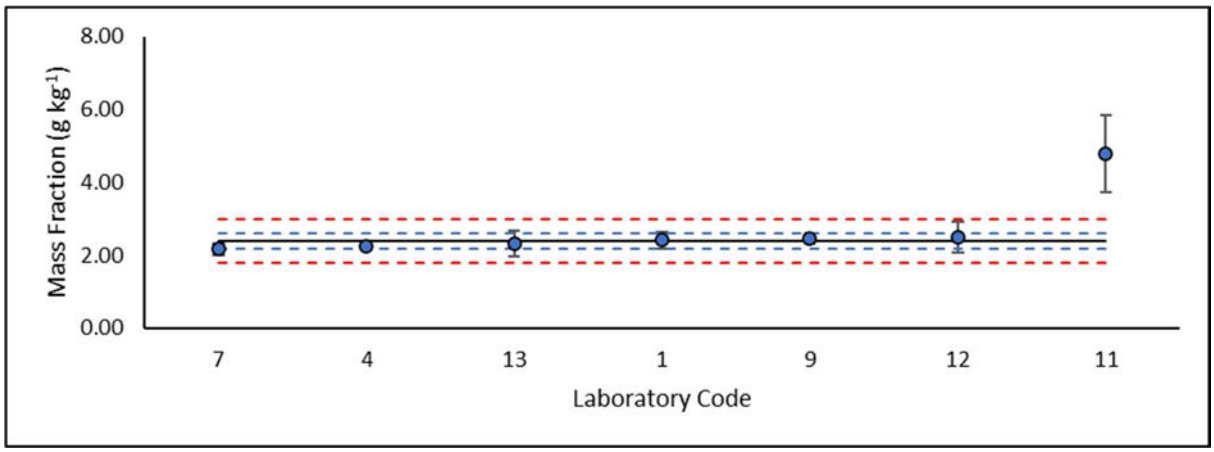


FIG. 42. PomPlot: Number are laboratory codes. (See Section 4. for more details).



— X_{ass} ; $X_{lab} \pm U_{lab}$; ---- $X_{ass} \pm 2\sigma_p$; ---- $X_{ass} \pm U_{ass}(k=2)$

FIG. 43. Reported results and expanded uncertainties.

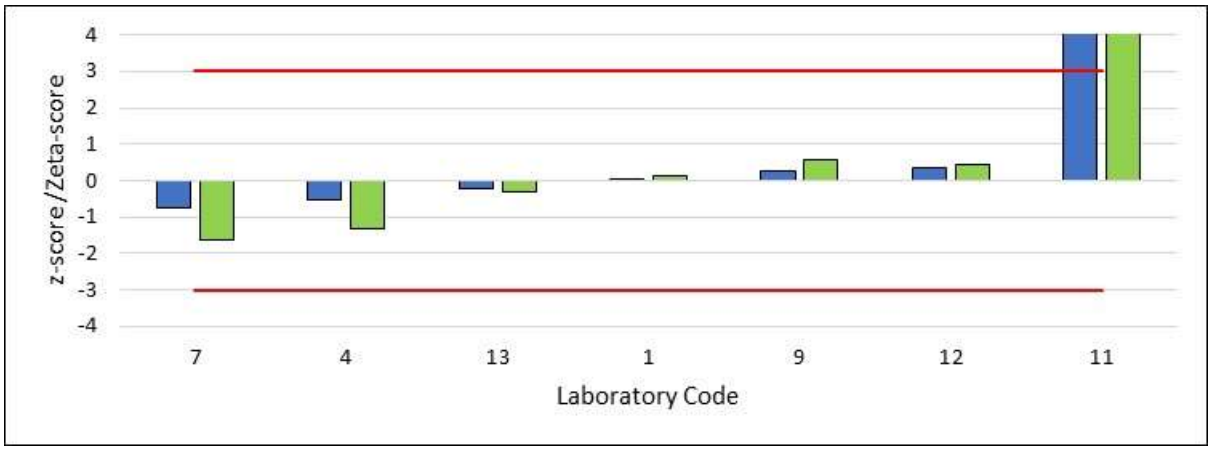


FIG. 44. Performance evaluation: *z*-score Zeta-score.

A.13. EVALUATION OF REPORTED DATA FOR Mn

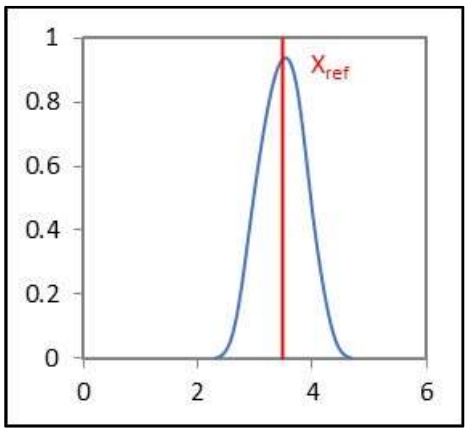


FIG. 45 Kernel density Plot.

Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	100%	0%	0%
Zeta-score	80%	20%	0%

X_{ref}:	3.49 mg kg^{-1}
U_{ref} (k=2):	0.39 mg kg^{-1}
$2\sigma_p$:	0.89 mg kg^{-1}
Number of results:	10
Number of method:	3

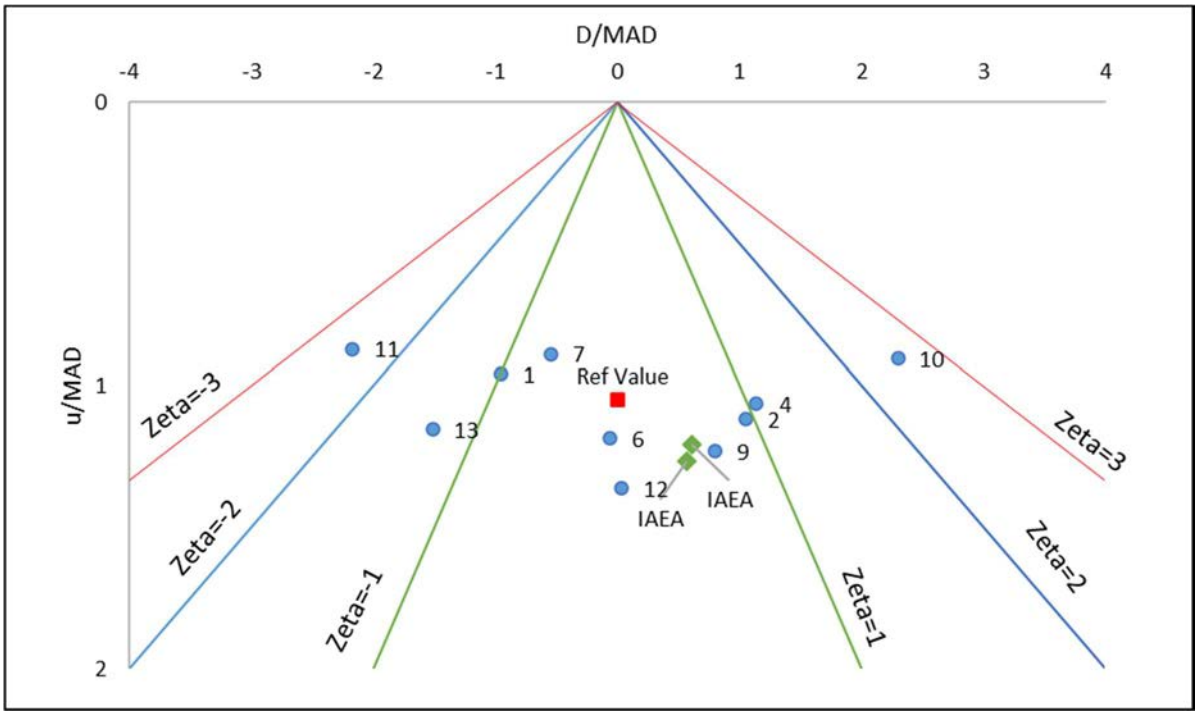
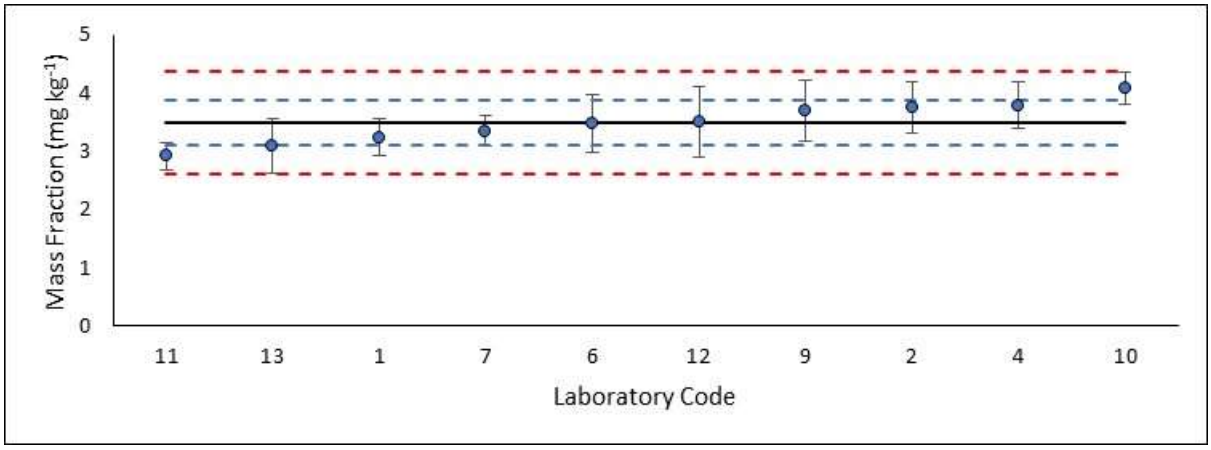


FIG. 46. PomPlot: Number are laboratory codes. (See Section 4. for more details).



— X_{ass} ; $\pm X_{lab} \pm U_{lab}$; ---- $X_{ass} \pm 2\sigma_p$; ---- $X_{ass} \pm U_{ass}(k=2)$

FIG. 47. Reported results and expanded uncertainties.

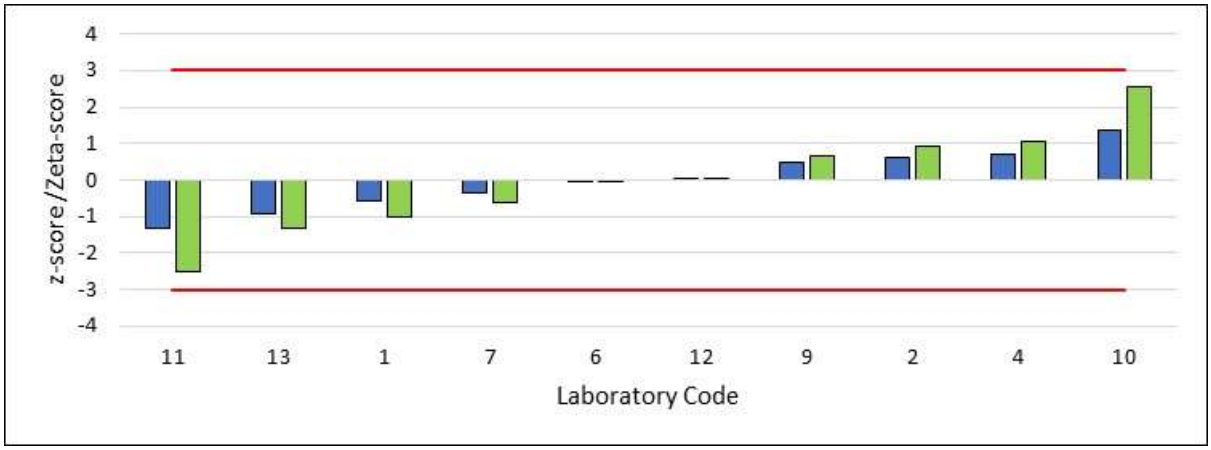


FIG. 48. Performance evaluation: *z*-score Zeta-score.

A.14. EVALUATION OF REPORTED DATA FOR Na

Kernel density Plot

<8 data

Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	80%	20%	0%
Zeta-score	80%	20%	0%

X_{ref}:	14.2 g kg^{-1}
U_{ref} (k=2):	1.2 g kg^{-1}
$2\sigma_p$:	3.6 g kg^{-1}
Number of results:	5
Number of method:	3

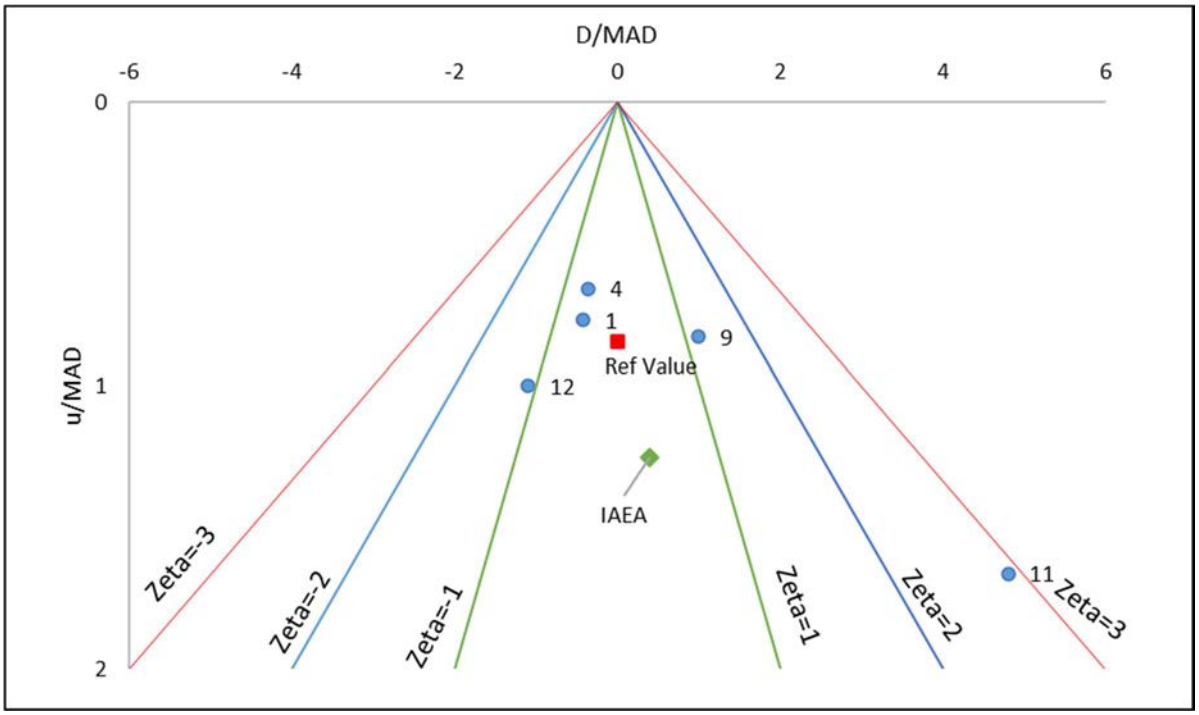
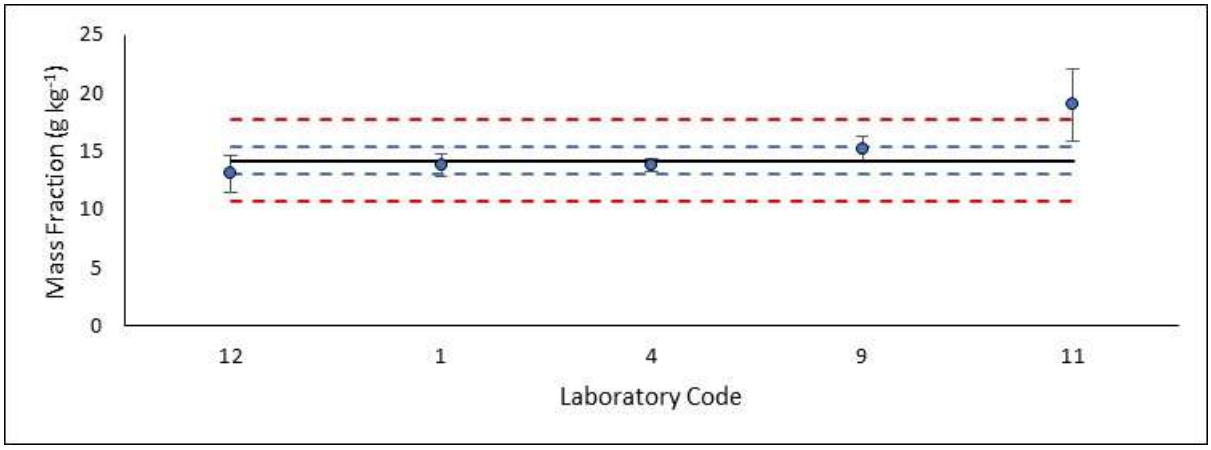


FIG. 49. PomPlot: Number are laboratory codes. (See Section 4. for more details).



— X_{ass} ; $X_{lab} \pm U_{lab}$; ---- $X_{ass} \pm 2\sigma_p$; ---- $X_{ass} \pm U_{ass}(k=2)$

FIG. 50. Reported results and expanded uncertainties.

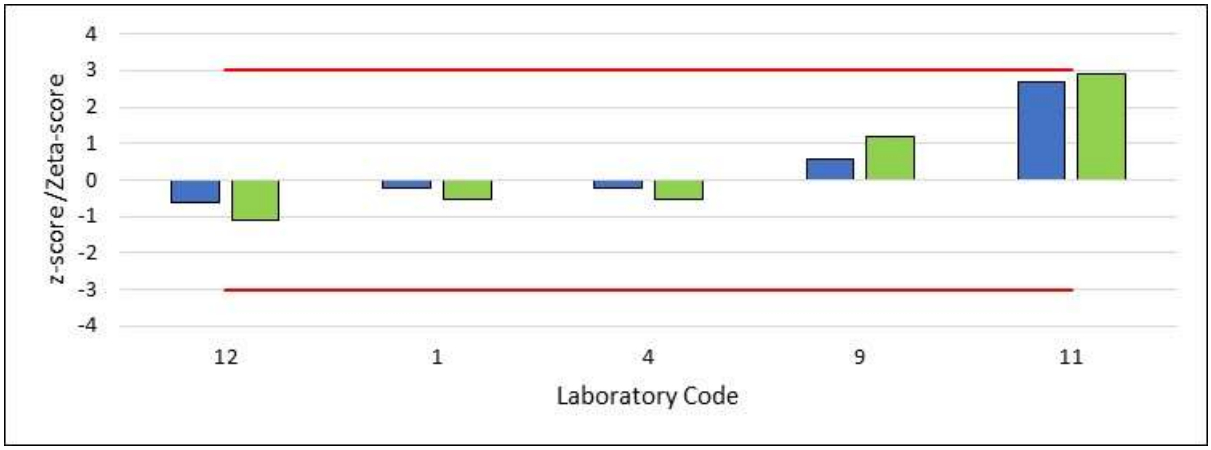


FIG. 51. Performance evaluation: *z*-score Zeta-score.

A.15. EVALUATION OF REPORTED DATA FOR Ni

Kernel density Plot

< 8 data

Summary of results:

X_{info}:	0.456 mg kg^{-1}
U_{info} (k=2):	0.098 mg kg^{-1}
$2\sigma_p$:	0.11 mg kg^{-1}
Number of results:	5
Number of method:	1

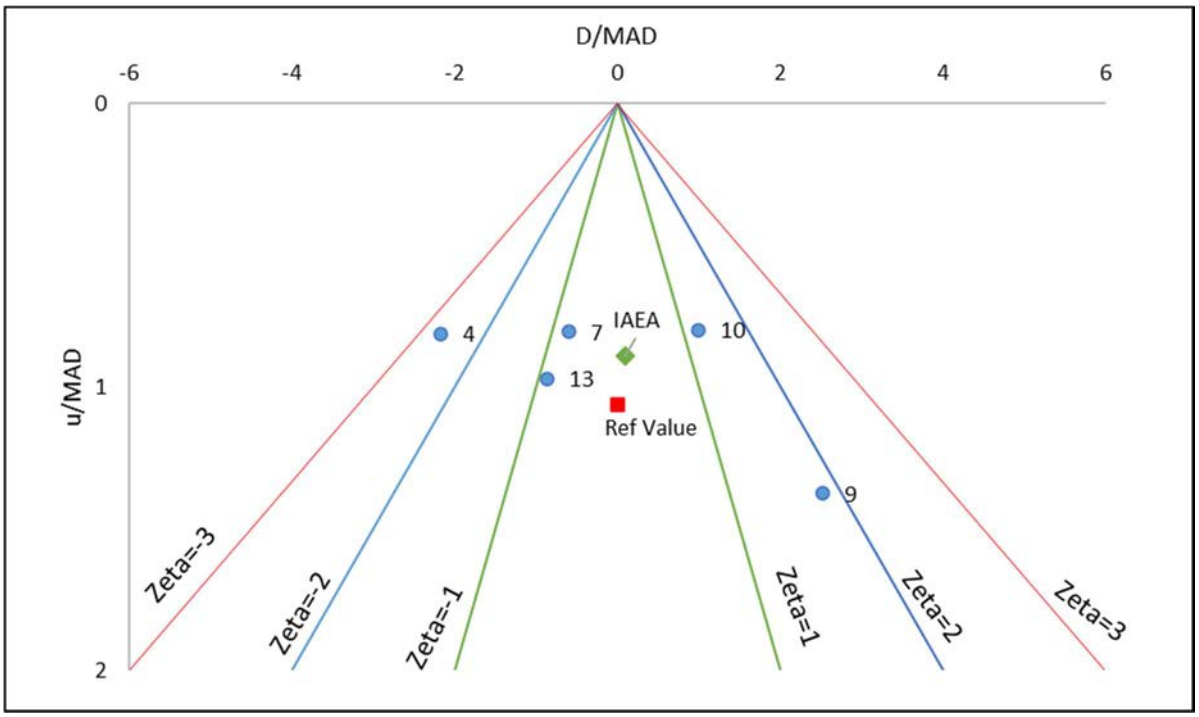
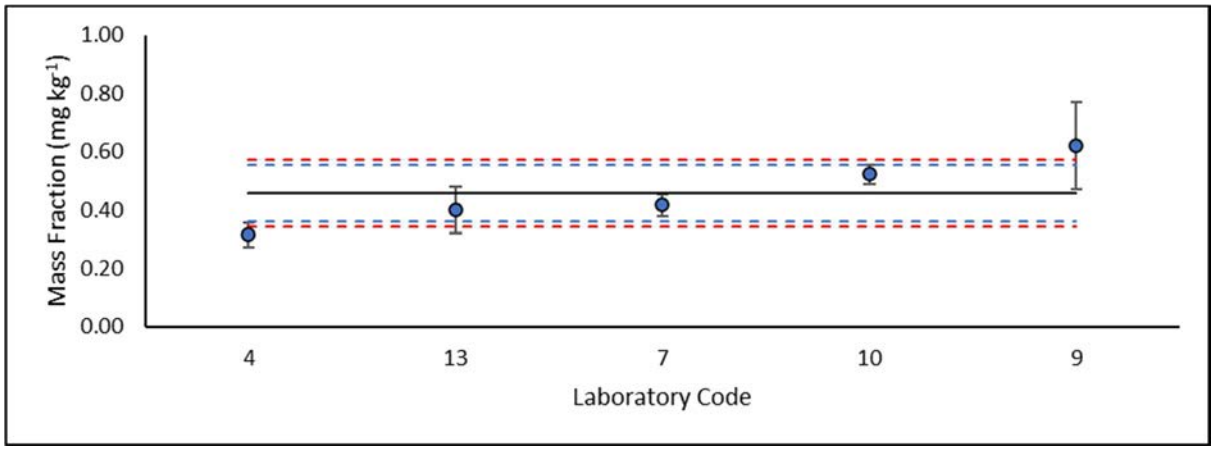


FIG. 52. PomPlot: Number are laboratory codes. (See Section 4. for more details).



— X_{ass} ; $\overline{X}_{lab} \pm U_{lab}$; ---- $X_{ass} \pm 2\sigma_p$; ---- $X_{ass} \pm U_{ass}(k=2)$

FIG. 53. Reported results and expanded uncertainties.

A.16. EVALUATION OF REPORTED DATA FOR Pb

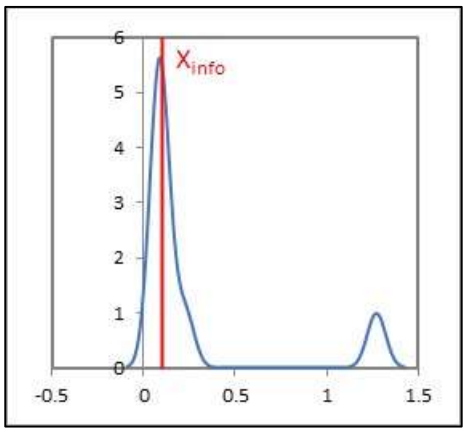


FIG. 54 Kernel density Plot.

Summary of results:

X_{info}:	0.102 mg kg^{-1}
U_{info} (k=2):	0.033 mg kg^{-1}
$2\sigma_p$:	0.025 mg kg^{-1}
Number of results:	8
Number of method:	1

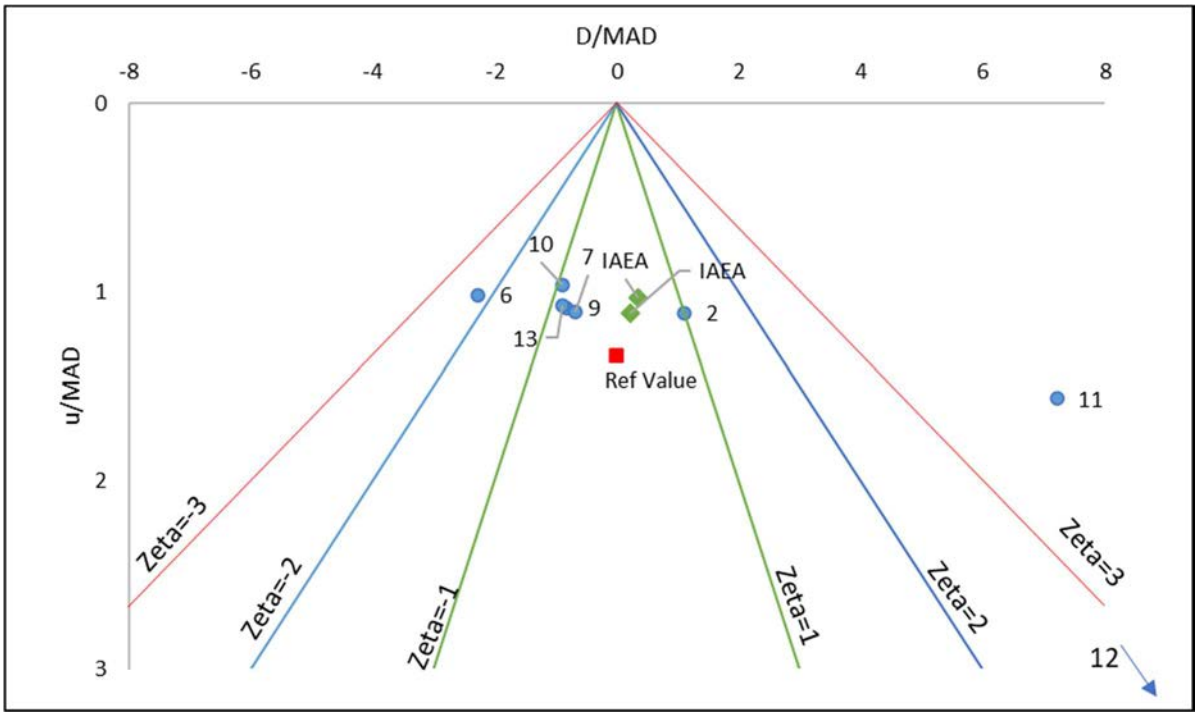
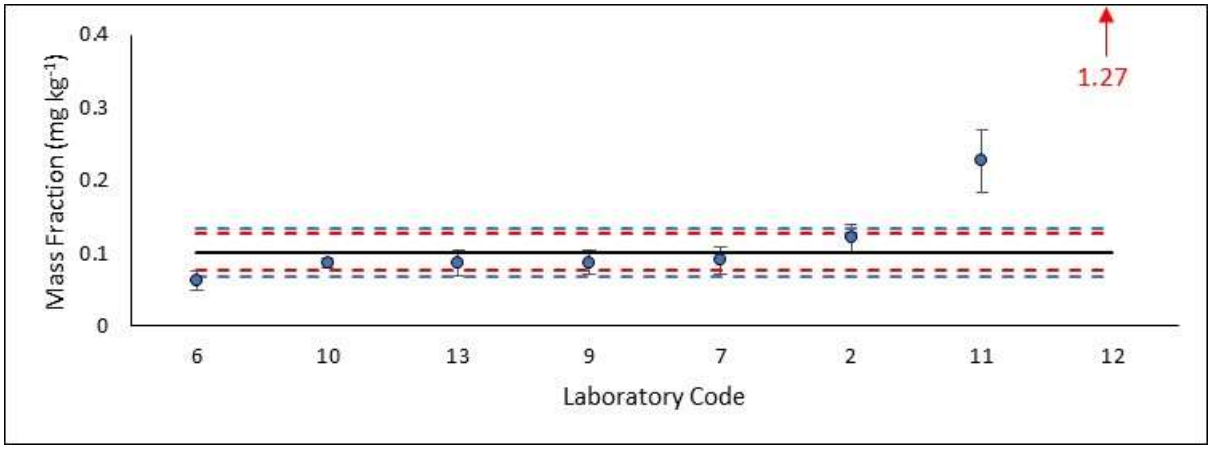


FIG. 55. PomPlot: Number are laboratory codes. (See Section 4. for more details).



— X_{ass} ; $X_{lab} \pm U_{lab}$; ---- $X_{ass} \pm 2\sigma_p$; ---- $X_{ass} \pm U_{ass}(k=2)$

FIG. 56. Reported results and expanded uncertainties.

A.17. EVALUATION OF REPORTED DATA FOR Rb

Kernel density Plot

<8 data

Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	100%	0%	0%
Zeta-score	100%	0%	0%

X_{ref}:	2.46 mg kg^{-1}
U_{ref} (k=2):	0.22 mg kg^{-1}
$2\sigma_p$:	0.61 mg kg^{-1}
Number of results:	6
Number of method:	2

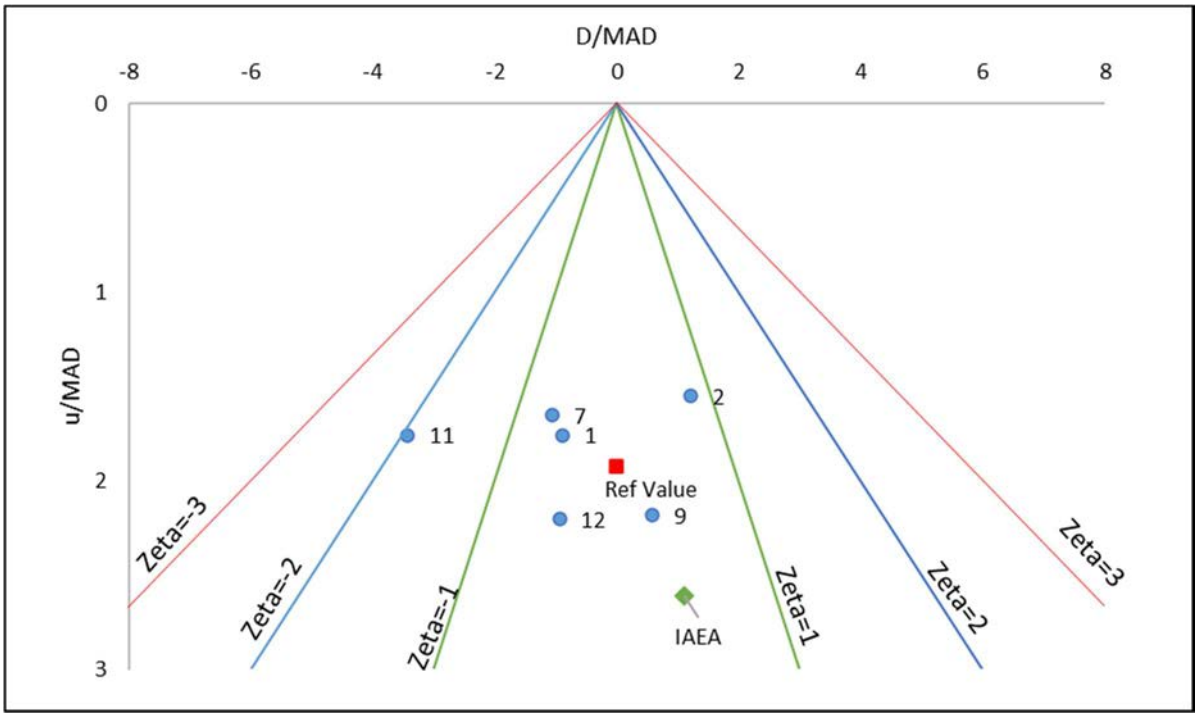
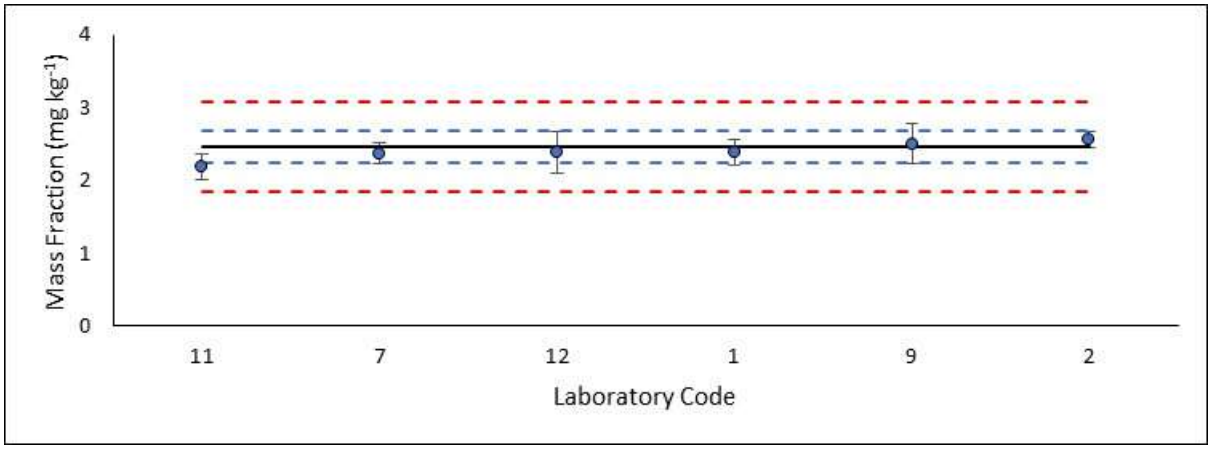


FIG. 57. PomPlot: Number are laboratory codes. (See Section 4. for more details).



— X_{ass} ; $\pm X_{lab} \pm U_{lab}$; ---- $X_{ass} \pm 2\sigma_p$; ---- $X_{ass} \pm U_{ass}(k=2)$

FIG. 58. Reported results and expanded uncertainties.

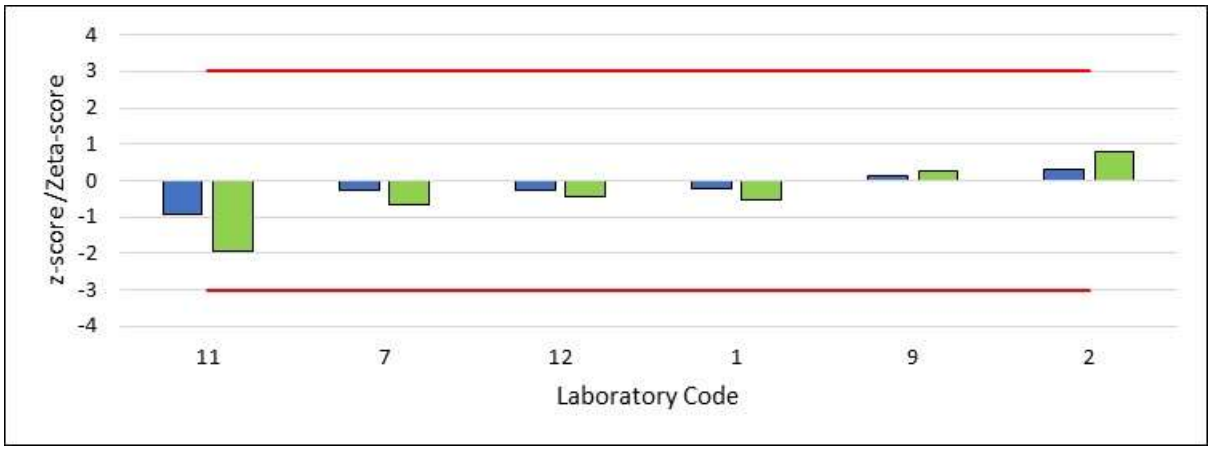


FIG. 59. Performance evaluation: *z*-score Zeta-score.

A.18. EVALUATION OF REPORTED DATA FOR Sb

Kernel density Plot

< 8 data

Summary of results:

X_{info}:	0.010 mg kg^{-1}
U_{info} (k=2):	0.003 mg kg^{-1}
$2\sigma_p$:	0.003 mg kg^{-1}
Number of results:	8
Number of method:	1

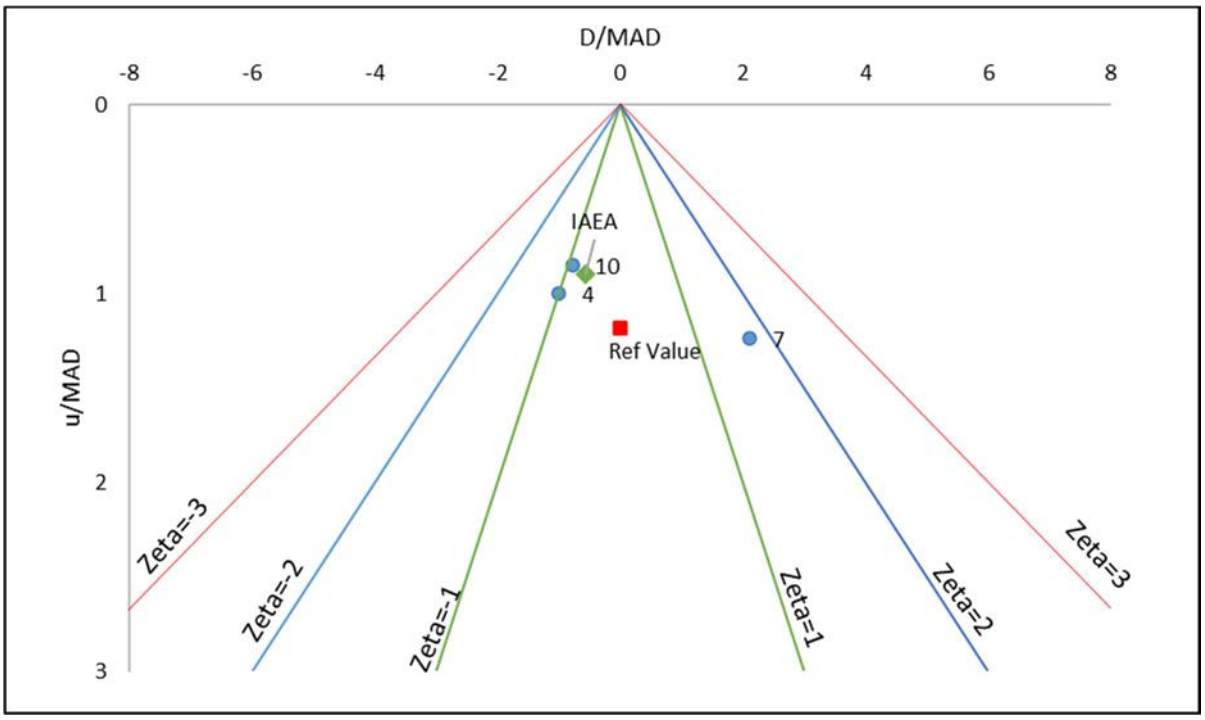
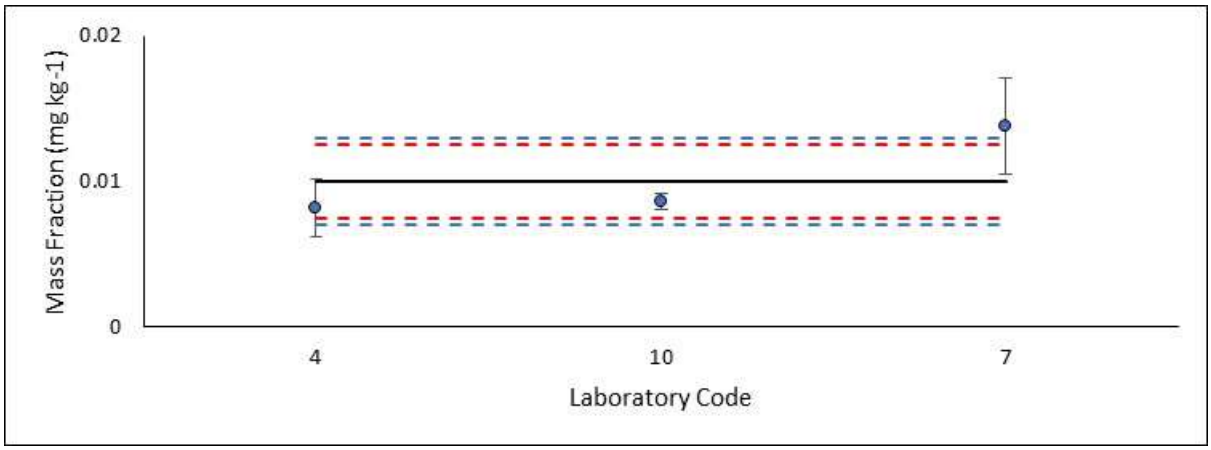


FIG. 60. PomPlot: Number are laboratory codes. (See Section 4. for more details).



— X_{ass} ; $X_{lab} \pm U_{lab}$; ---- $X_{ass} \pm 2\sigma_p$; ---- $X_{ass} \pm U_{ass}(k=2)$

FIG. 61. Reported results and expanded uncertainties.

A.18. EVALUATION OF REPORTED DATA FOR Se

Kernel density Plot

<8 data

Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	86%	0%	14%
Zeta-score	57%	29%	14%

X_{ref}:	2.60 mg kg^{-1}
U_{ref} (k=2):	0.20 mg kg^{-1}
$2\sigma_p$:	0.65 mg kg^{-1}
Number of results:	7
Number of method:	2

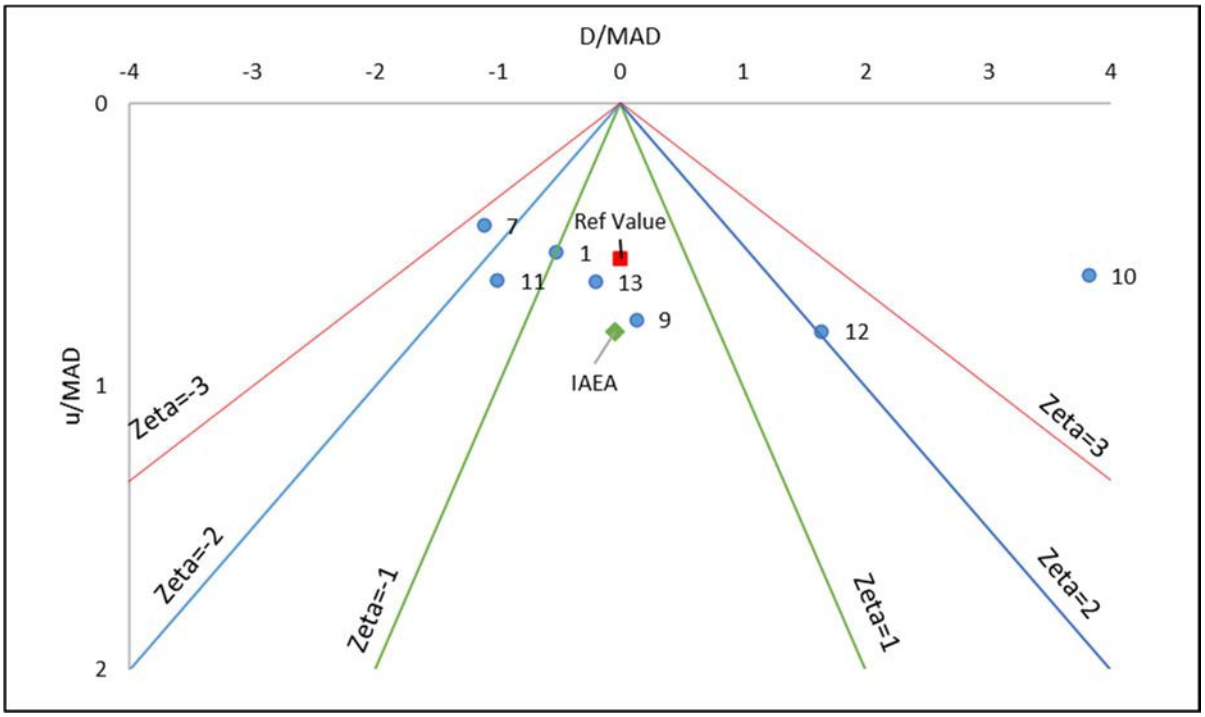
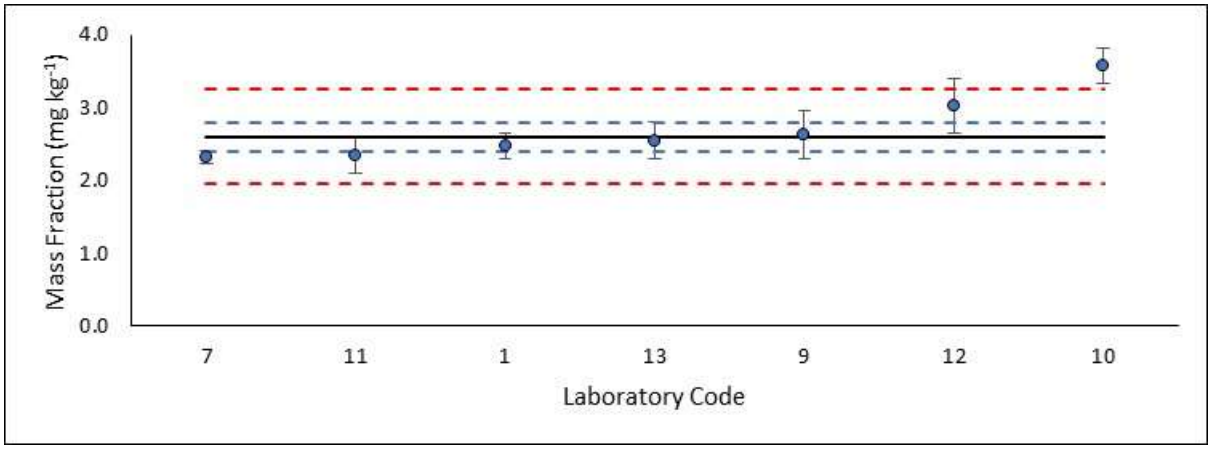


FIG. 62. PomPlot: Number are laboratory codes. (See Section 4. for more details).



— X_{ass} ; $\pm X_{lab} \pm U_{lab}$; ---- $X_{ass} \pm 2\sigma_p$; ---- $X_{ass} \pm U_{ass}(k=2)$

FIG. 61. Reported results and expanded uncertainties.

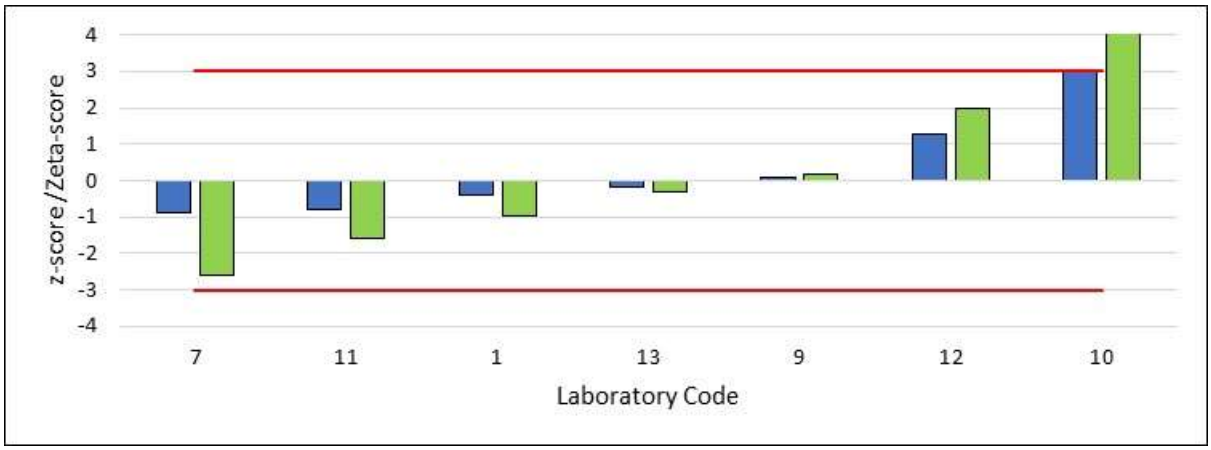


FIG. 62. Performance evaluation: *z*-score Zeta-score.

A.19. EVALUATION OF REPORTED DATA FOR Sr

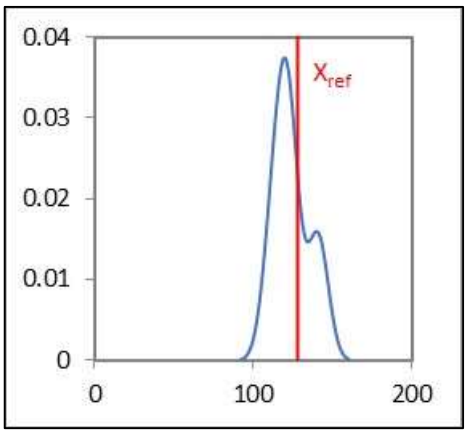


FIG. 63 Kernel density Plot.

Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	100%	0%	0%
Zeta-score	88%	13%	0%

X_{ref}:	128 mg kg^{-1}
U_{ref} (k=2):	12 mg kg^{-1}
$2\sigma_p$:	32 mg kg^{-1}
Number of results:	8
Number of method:	2

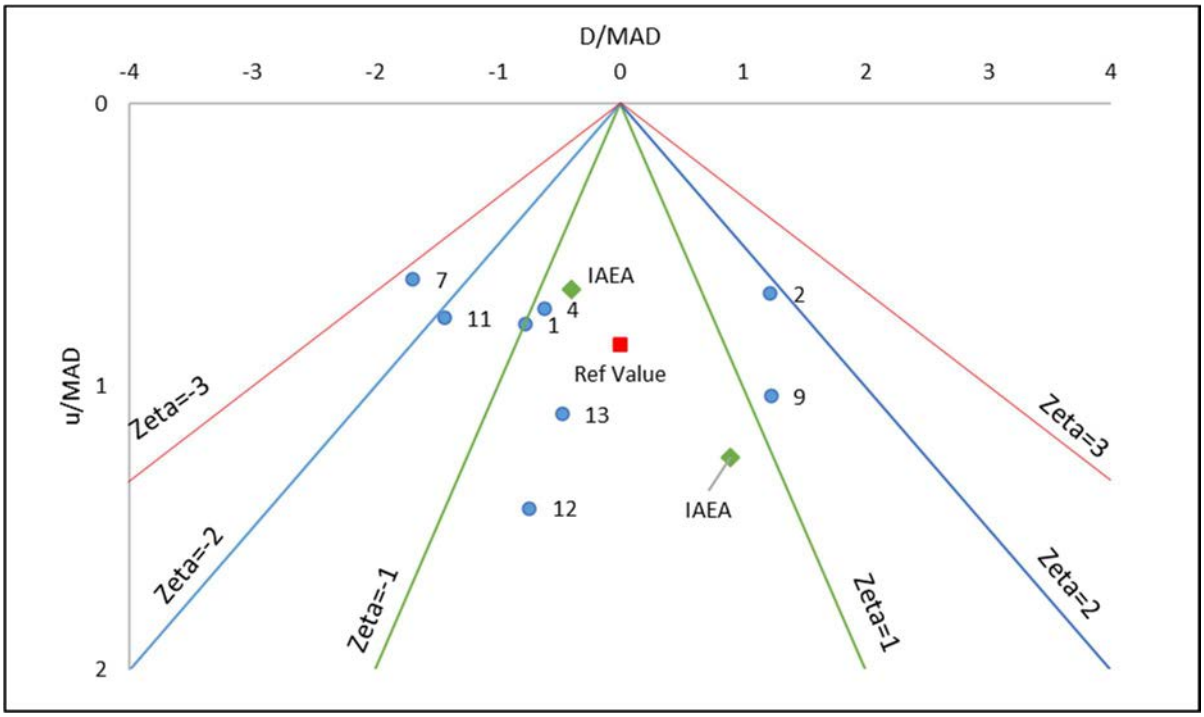
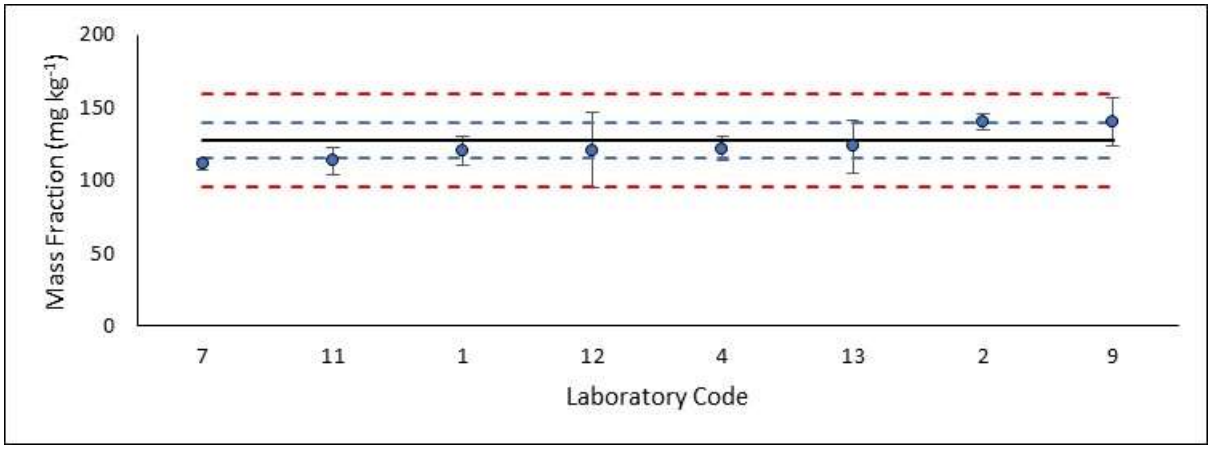


FIG. 64. PomPlot: Number are laboratory codes. (See Section 4. for more details).



— X_{ass} ; $\pm X_{lab} \pm U_{lab}$; ---- $X_{ass} \pm 2\sigma_p$; ---- $X_{ass} \pm U_{ass}(k=2)$

FIG. 65. Reported results and expanded uncertainties.

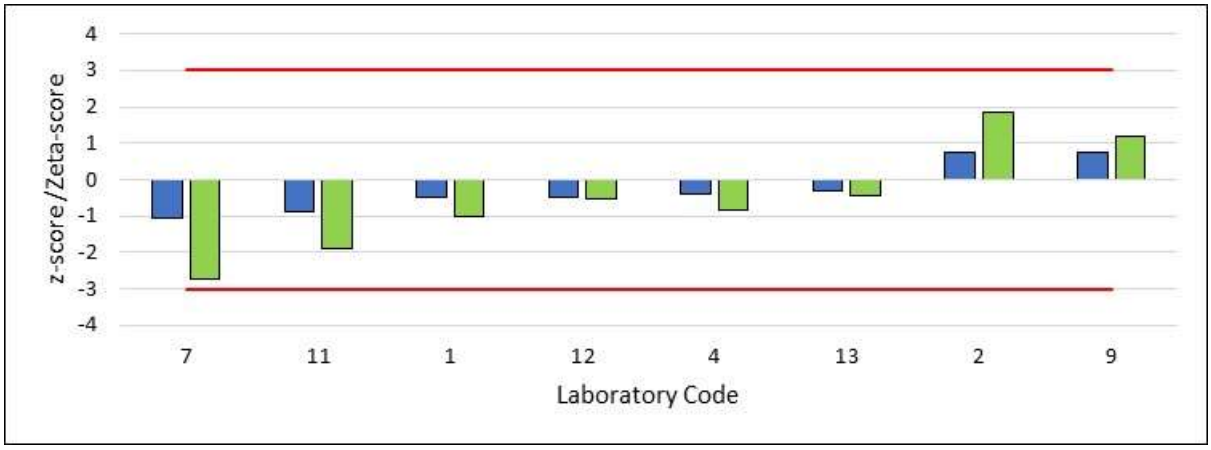


FIG. 66. Performance evaluation: *z*-score Zeta-score.

A.20. EVALUATION OF REPORTED DATA FOR V

Kernel density Plot

<8 data

Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	100%	0%	0%
Zeta-score	57%	43%	0%

X_{ref}:	1.46 mg kg^{-1}
U_{ref} (k=2):	0.18 mg kg^{-1}
$2\sigma_p$:	0.36 mg kg^{-1}
Number of results:	7
Number of method:	2

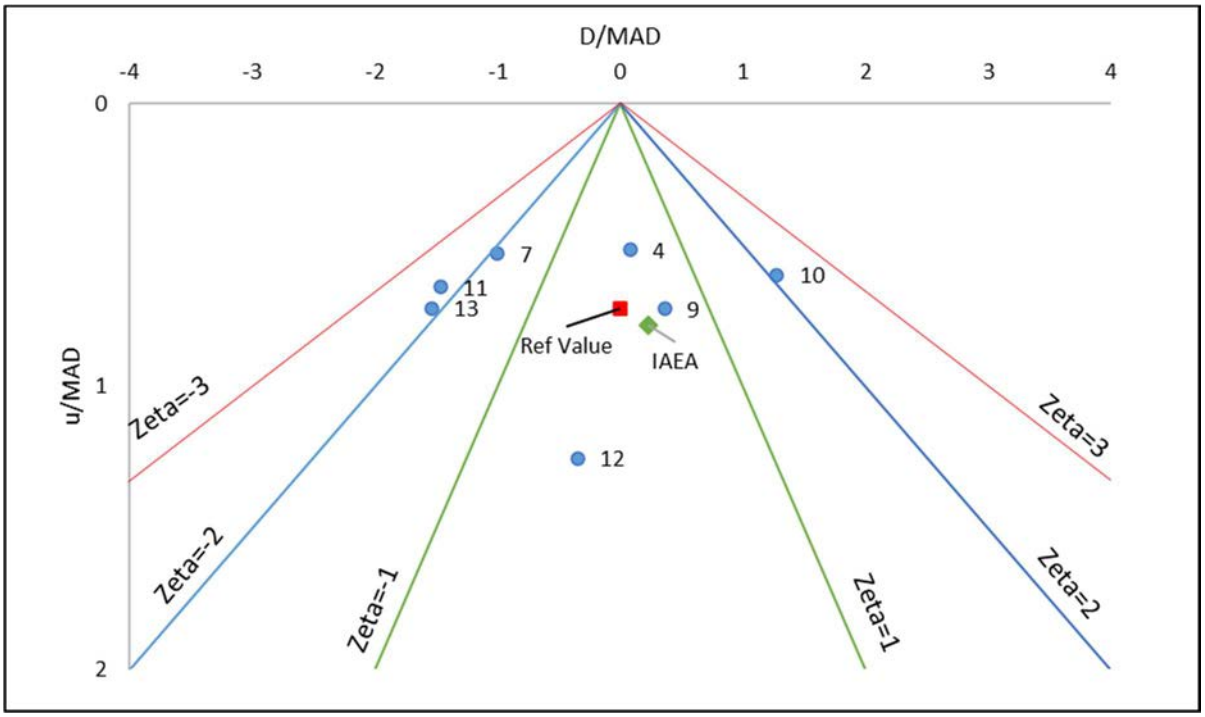
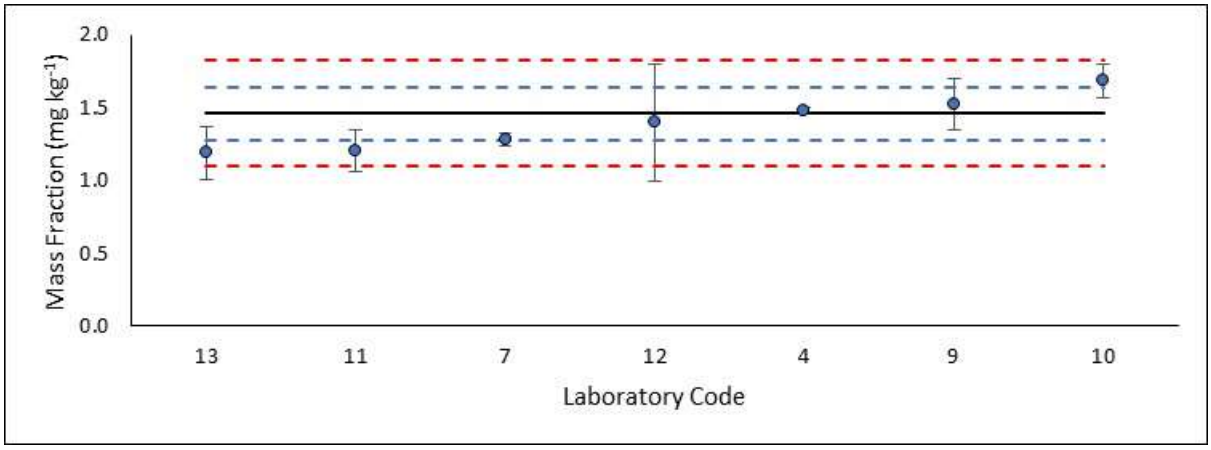


FIG. 67. PomPlot: Number are laboratory codes. (See Section 4. for more details).



— X_{ass} ; $\pm X_{lab} \pm U_{lab}$; ---- $X_{ass} \pm 2\sigma_p$; ---- $X_{ass} \pm U_{ass}(k=2)$

FIG. 68. Reported results and expanded uncertainties.

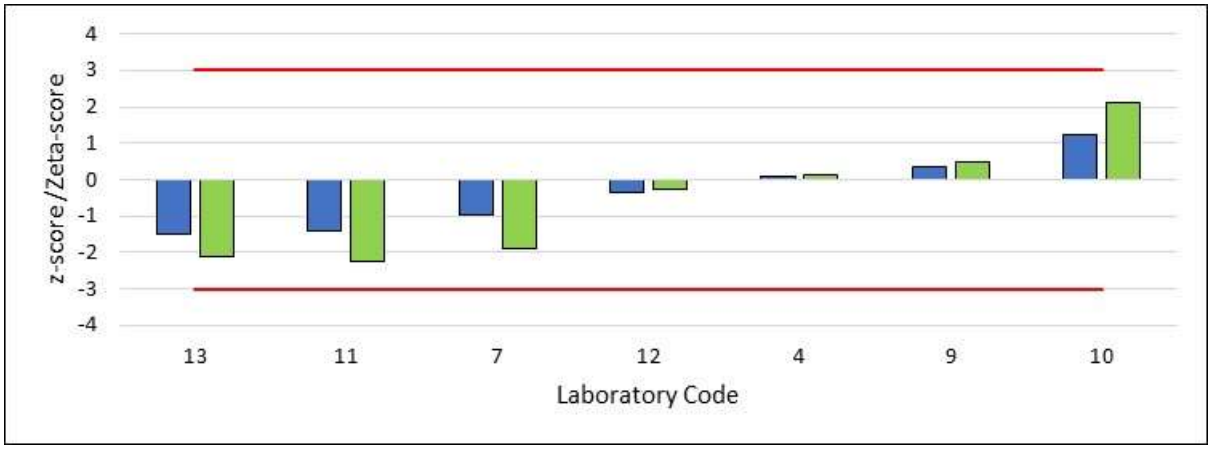


FIG. 69. Performance evaluation: *z*-score Zeta-score.

A.21. EVALUATION OF REPORTED DATA FOR Zn

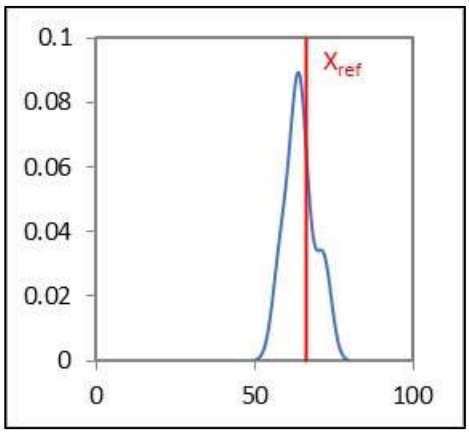


FIG. 70 Kernel density Plot.

Summary of results:

	Satisfactory	Questionable	Unsatisfactory
z-score	100%	0%	0%
Zeta-score	57%	43%	0%

X_{ref}:	66.4 mg kg^{-1}
U_{ref} (k=2):	2.7 mg kg^{-1}
$2\sigma_p$:	16.6 mg kg^{-1}
Number of results:	10
Number of method:	3

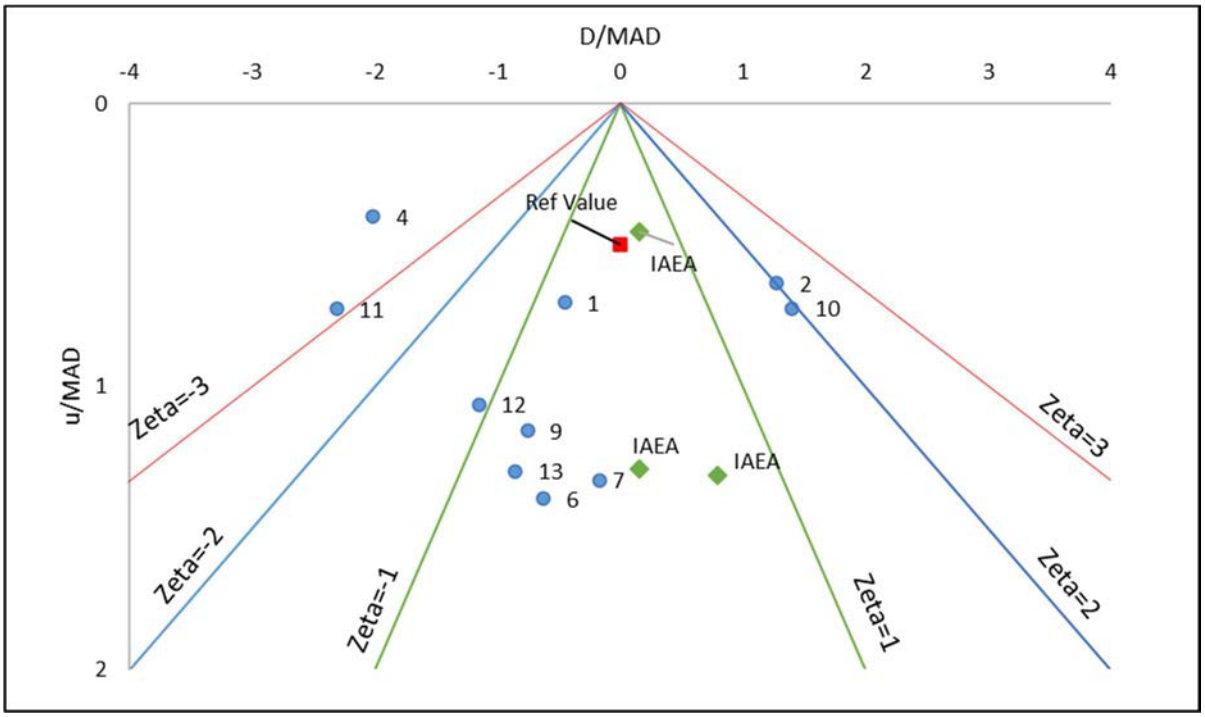
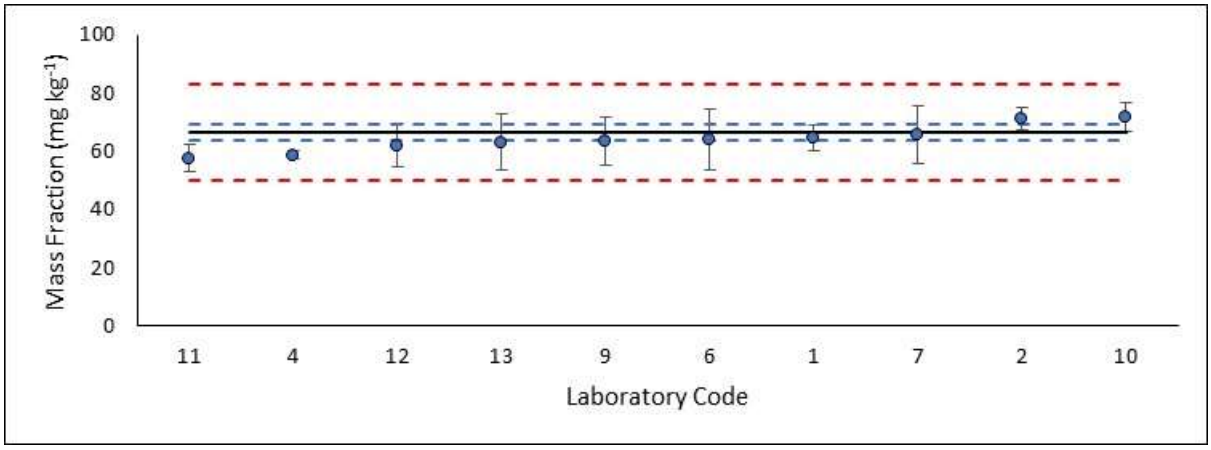


FIG. 71. PomPlot: Number are laboratory codes. (See Section 4. for more details).



— X_{ass} ; $\pm X_{lab} \pm U_{lab}$; ---- $X_{ass} \pm 2\sigma_p$; ---- $X_{ass} \pm U_{ass}(k=2)$

FIG. 72. Reported results and expanded uncertainties.

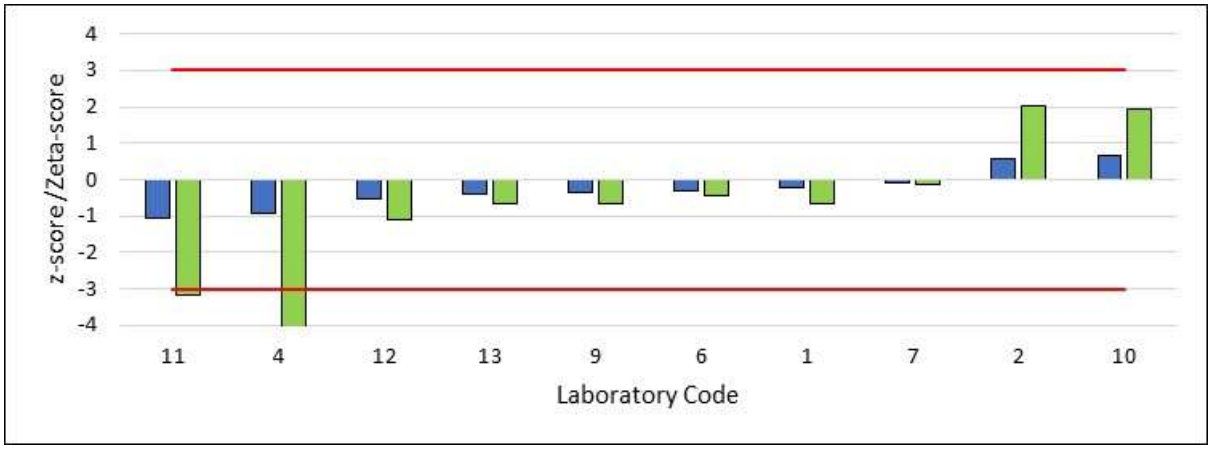


FIG. 73. Performance evaluation: *z*-score Zeta-score.

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