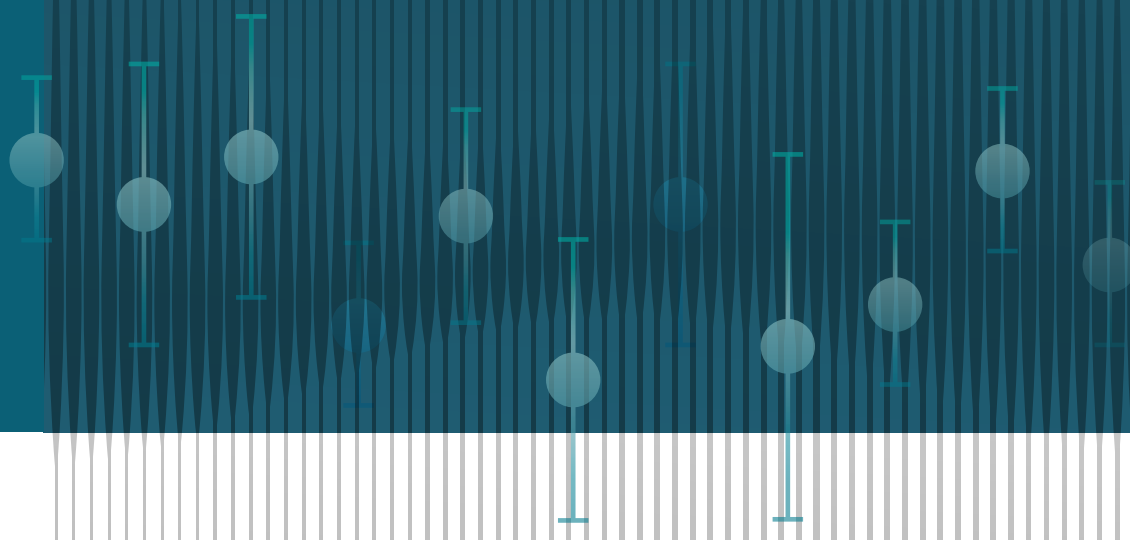


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No. 76

Interlaboratory Comparison on the Determination of Trace Elements and Rare Earth Element Mass Fractions in Marine Sediment Sample IAEA-MESL-2024-ILC-TE-SEDIMENT



INTERLABORATORY COMPARISON
ON THE DETERMINATION
OF TRACE ELEMENTS AND RARE EARTH
ELEMENT MASS FRACTIONS
IN MARINE SEDIMENT SAMPLE
IAEA-MESL-2024-ILC-TE-SEDIMENT

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MASS FRACTIONS IN MARINE SEDIMENT SAMPLE IAEA-MESL-2024-ILC-TE-SEDIMENT

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FOREWORD

The identification of environmental pollution relies on monitoring campaigns that periodically assess the quality of seawater, marine sediment and biota samples. Ensuring the reliability and comparability of analytical results in this context is crucial for effective marine environment management, supporting informed decision making and meaningful actions related to remediation policies.

The IAEA provides support to its Member States in the area of data quality and quality assurance by organizing interlaboratory comparisons and producing marine certified reference materials (biota and sediments) characterized for mass fractions of trace elements and, more recently, rare earth elements.

Periodic external assessments of the measurement performance of monitoring laboratories, conducted through interlaboratory comparisons and targeted proficiency tests, are of particular importance for laboratories because they provide clear information on the laboratories' measurement capabilities. These exercises are designed not only to monitor and demonstrate the performance and analytical capabilities of participating laboratories, but also to identify gaps and problem areas where further development is needed.

This publication summarizes the results of the interlaboratory comparison on the determination of trace and rare earth elements in a sediment sample organized in 2024.

The IAEA is grateful to the Government of Monaco for the support provided to its Marine Environment Laboratories and to the individuals and laboratories who participated in this intercomparison exercise. The IAEA officer responsible for this publication was S. Azemard of the Division of IAEA 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 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 certified reference materials (CRMs), validated analytical procedures, training, and through the evaluation of measurement performance via interlaboratory comparisons (ILC).

1.2. OBJECTIVE

Periodic external assessments of measurement performances via ILC and targeted proficiency tests are important for laboratories. These assessments provide clear information on their measurement capabilities, allowing them to monitor and demonstrate their analytical capabilities, identify gaps, and highlight areas where further development is needed.

The ILC (IAEA-MESL-2024-ILC-TE-SEDIMENT) presented in this report was designed to offer the possibility of laboratories routinely involved in the measurements of trace elements and rare earth elements (REE) in marine sediment to evaluate their performance.

1.3. SCOPE

The scope of this publication is to describe obtained results of the ILC.

1.4. STRUCTURE

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

2. STUDY SETUP

In February 2024, invitation letters were sent to 344 laboratories from 107 Member States, which previously participated in the IAEA ILC or have expressed interest in participating in ILC. Positive responses with the intent to participate were received from 138 laboratories in 73 Member States.

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 trace element and REE analytes (listed in Table 1) using the analytical procedures routinely applied in their laboratories.

Participating laboratories were requested to report their results for the ILC sample together with standard and expanded uncertainties, results of internal quality control samples (e.g. CRMs or other reference materials), detection and quantification limits.

TABLE 1. CLASSIFICATION OF ANALYTES IN THE REPORT

Analyte	Group
Ag, Al, As, Ba, Cd, Co, Cr, Cs, Cu, Fe, Hg, Li, Mn, Ni, Pb, Sr, Th, U, V, Zn	Trace element
Ce, Dy, Er, Eu, Gd, Ho, La, Nd, Pr, Sc, Sm, Tb, Tm, Y, Yb	REE

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, quality procedures and accreditation.

In total, 101 laboratories from 60 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 and REE, are included in this report. All results were treated confidentially, and each laboratory was identified with a unique confidential code number.

3. ILC TEST MATERIAL

3.1. PREPARATION

The ILC test sample was a sediment collected from the Irish Sea. After drying, the sediment was ground into powder, passed through a 250 μm sieve and homogenized under a nitrogen atmosphere. The samples were then bottled in polyethylene containers.

3.2. HOMOGENEITY AND STABILITY

The homogeneity of the material was evaluated by determining mass fraction of analytes listed in Table 1 in duplicate in five units.

About 0.25g of sample was digested using microwave oven with nitric and hydrofluoric acid followed by treatment with boric acid to remove excess of hydrofluoric acid and facilitate dissolution of fluoride precipitates. Measurements were performed at MESL by triple quadrupole inductively coupled plasma mass spectrometry and inductively coupled plasma optical emission. For Hg determination were performed with a solid mercury analyser using about 100mg. All methods used have been preliminary validated, IAEA 158A and IAEA 458 were used as certified reference material.

Uncertainty associated with the inhomogeneity of the material was estimated using analysis of variance (ANOVA) as recommended in ISO/IEC 33405:2024 [1].

Except for Hg, all analytes demonstrate good homogeneity with u_{hom} (uncertainty arising from inhomogeneity) being less than 5%. For Hg, some outliers were observed and the u_{hom}

was estimated using a rectangular distribution, the obtained 7% u_{hom} was still considered to be acceptable for the purpose of the exercise.

As the exercise was conducted in less than six months and based on experience with similar samples, no uncertainty was associated with the stability of the material over the time of the ILC.

4. ASSIGNED VALUES AND UNCERTAINTIES

The assigned values directly influence conclusions regarding the measurement capabilities of participating laboratories. Consequently, it is essential to seek the most metrologically credible values. Assigned values of the ILC samples were calculated using reported data from laboratories that have demonstrated good analytical performance in previous ILC's and data from IAEA laboratories.

Data sets from selected laboratories underwent rigorous scrutiny to ensure technical validity. This process included reviewing quality control measures, analytical strategies (e.g., total digestion), and assessing uncertainties to confirm the data's suitability for its intended purpose. Results with expanded uncertainties exceeding 20% were deemed unfit for the study and were excluded. After excluding data considered not technically valid, the number of results per analyte was at least four, except for Ag, for which only one result was available. Consequently, no assigned value could be estimated for Ag.

Selected data were tested for outliers using the Grubbs and Dixon tests and verified for normal distribution with the Shapiro–Wilk test. For data sets with more than eight results, a Kernel density plot was used to visually check for unimodal distribution. If any bimodality was observed, it was investigated for potential method bias by plotting the reported mean grouped by instrumental method.

Data sets for all evaluated analytes were normally distributed (at least at the 99% confidence level), and no outliers were detected. Although a few bimodalities were observed, they were not significant. However, the distribution of Cr values was slightly affected by the sample preparation approach, with data obtained using the neutron activation method (nondestructive) being slightly higher and significantly different (t test) than data obtained after digestion. In some cases, it has been shown that incomplete dissolution of natural Cr can occur, and only very strong digestion procedures might achieve 100% recovery in the solution [2]. To account for this potential effect, an uncertainty component for between-method bias (u_{MB}) was added. This increased the final expanded uncertainty associated with the assigned value (from 6.5% to 21.1%), ensuring potential bias is accounted for.

Consensus values were estimated as the means of the mean of retain data sets. The uncertainty associated with the assigned values were calculated according to ISO/IEC 33405:2024 [1]. The combined uncertainty of the assigned values consists of uncertainty contributors related to its characterization (u_{char}), sample heterogeneity (u_{hom}), and between method bias (u_{MB}) for Cr only as previously explained. These contributions were combined and multiplied with a coverage factor k , to estimate the expanded uncertainty (U) as shown in Eq. 1.

$$U = k \times \sqrt{u_{\text{char}}^2 + u_{\text{MB}}^2 + u_{\text{hom}}^2} \quad (1)$$

Where k is a coverage factor equaling 2, representing a level of confidence of about 95%.

u_{char} was estimated as described in ISO/IEC 33405:2024 [1] using Eq. 2.

$$u_{\text{char}} = \frac{s}{\sqrt{p}} \quad (2)$$

Where s is the standard deviation of the mean; p is the number of datasets.

The means of the mean values, their relative expanded uncertainties ($k=2$), and uncertainty contributions from characterization and homogeneity are presented in Table 2.

With a few exceptions (Cd, Er, Gd, Ho, Pr, and Tm), the assigned values are derived from at least five datasets and two independent analytical methods, and the associated expanded uncertainty is less than 25%, which is considered fit for purpose to evaluate the results reported in this ILC.

For Cd despite being obtained from only four results, the assigned values were deemed sufficiently credible based on comparisons with IAEA values and robust estimate of consensus value using all reported results (excluding obviously non-technically valid data, such as those lacking quality control or non-total results).

For Er, Gd, Ho, Pr, and Tm, assigned values were derived from datasets obtained via inductively coupled plasma mass spectrometry exclusively, with exception of one result for Pr reported by Xray Fluorescence. Therefore, it was considered that the assigned value is suitable for evaluating analytical performance as no method bias component should be considered in this case.

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

Analyte	Assigned (mg kg ⁻¹)	U (mg kg ⁻¹) ($k=2$)	u_{char} , relative	u_{hom} , relative	p	Number of Methods	U_{relative} ($k=2$)
Al	58.6×10^3	2.8×10^3	1.3%	2.0%	10	4	4.8%
As	11.03	0.76	2.2%	2.7%	10	2	6.8%
Ba	292	14	1.6%	1.9%	9	3	4.8%
Cd	90×10^{-3}	14×10^{-3}	7.2%	2.7%	4	2	15%
Ce	58.1	1.3	0.9%	0.7%	8	2	2.2%
Co	11.81	0.48	1.7%	1.1%	11	3	4.1%
Cr ¹	80	17	2.9%	1.5%	13	4	21%
Cs	6.57	0.28	1.9%	0.8%	10	2	4.2%
Cu	17.5	1.7	4.2%	2.5%	10	4	9.8%
Dy	4.15	0.34	3.1%	2.7%	9	2	8.3%
Er	2.34	0.17	3.0%	1.8%	5	1	7.1%
Eu	1.024	0.057	2.7%	0.7%	8	2	5.5%
Fe	30.3×10^3	1.8×10^3	2.1%	2.0%	12	4	5.8%
Gd	4.35	0.32	3.2%	2.0%	6	1	7.5%
Hg	0.0477	0.0078	4.3%	7.0%	5	2	16%
Ho	0.812	0.075	4.2%	1.8%	5	1	9.2%
La	28.5	1.8	2.4%	2.0%	11	2	6.2%
Li	59.2	3.2	1.8%	2.0%	8	2	5.4%

¹Uncertainty component from between method bias of 10% was included

TABLE 2. ASSIGNED VALUES AND UNCERTAINTY FOR THE ILC TEST SAMPLE
cont.

Analyte	Assigned (mg kg ⁻¹)	U (mg kg ⁻¹) ($k=2$)	$u_{\text{char,}}$ relative	$u_{\text{hom,}}$ relative	P	Number of Methods	U_{rel} ($k=2$)
Mn	540	24	1.0%	2.0%	12	3	4.5%
Nd	26.2	1.2	1.0%	2.0%	7	2	4.5%
Ni	31.2	1.7	1.9%	2.0%	11	4	5.6%
Pb	32.4	3.0	2.1%	4.1%	11	4	9.2%
Pr	6.76	0.44	2.6%	2.0%	6	1	6.6%
Sc	10.81	0.98	4.1%	2.0%	7	2	9.1%
Sm	4.89	0.29	2.2%	2.0%	11	2	5.9%
Sr	259	13	1.4%	2.0%	10	3	5.0%
Tb	0.656	0.039	2.2%	2.0%	6	2	5.9%
Th	8.30	0.97	5.5%	2.0%	8	2	12%
Tm	0.349	0.024	2.8%	2.0%	5	1	6.9%
U	2.49	0.16	2.6%	2.0%	9	2	6.4%
V	91.1	5.1	2.0%	2.0%	13	3	5.6%
Y	21.0	2.2	4.8%	2.0%	5	2	10%
Yb	2.32	0.14	2.2%	2.0%	8	2	6.0%
Zn	101.2	6.5	1.8%	2.7%	11	4	6.4%

¹Uncertainty component from between method bias of 10% was included.

5. EVALUATION OF ANALYTICAL PERFORMANCE

The individual laboratory performance was expressed in terms of z -scores and *Zeta*-scores, in accordance with ISO/IEC 17043:2023 [3].

The determination of target standard deviation σ_t , for the proficiency assessment was based on the outcome of previous ILCs organized by MESL for the same population of laboratories and similar sample matrices and was set as 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 assigned values for the respective measurands.

The z -score, which is calculated as shown in Eq. (3), defines the difference between the mean value provided by the laboratory and the reference value, expressed in the units of the target standard deviation.

$$Z = \frac{x_{\text{lab}} - x_{\text{ass}}}{\sigma_t} \quad (3)$$

where:

x_{lab} is the result reported by the participating laboratory

x_{ass} is the assigned value

σ_t is the target standard deviation

The *Zeta*-score, which is calculated as shown in Eq. (4), demonstrates the agreement of the results reported by participating laboratories with the reference value within the respective uncertainties. The denominator in the Eq. (4) is calculated from the combined uncertainty of the assigned value and the combined uncertainty reported by the respective participant.

$$Zeta = \frac{x_{lab} - x_{ass}}{\sqrt{u_{x_{lab}}^2 + u_{x_{ass}}^2}} \quad (4)$$

where:

x_{lab} is the result reported by the participating laboratory

x_{ass} is the assigned value

$u_{x_{lab}}$ is the combined uncertainty reported by the participating laboratory

$u_{x_{ass}}$ is the combined uncertainty of the assigned value.

The interpretation of a laboratory's performance was evaluated according to the following internationally accepted limits [4]:

$ z \text{ or } Zeta \leq 2$	Satisfactory
$2 < z \text{ or } Zeta < 3$	Questionable
$ z \text{ or } Zeta \geq 3$	Unsatisfactory

6. RESULTS AND DISCUSSION

Hundred and one sets of data for 38 analytes were submitted by participating laboratories, comprising 1357 numerical results and 16 non-numerical (i.e., <quantification or detection limit). *z*-scores and *Zeta*-scores were only calculated for 37 analytes (1341 numerical results).

The reported results (mean $\pm$ expanded uncertainty) per analyte are shown in the Appendix using a scatter plot along assigned value, assigned expanded uncertainty and two target standard deviation.

Figures 1 and 2 summarize the overall performance, by participating laboratories as defined by *z* and *Zeta* scores respectively.

Figures 3 and 4 summarize the overall performance by analytes as defined by *z* and *Zeta* scores respectively.

Table 3 and Table 4 show the overall performance as defined by *z*-scores for trace element and REE (as define in Table 1) respectively.

Table 5 and Table 6 show the overall performance as defined by *Zeta*-scores for trace element and REE (as define in Table 1) respectively..

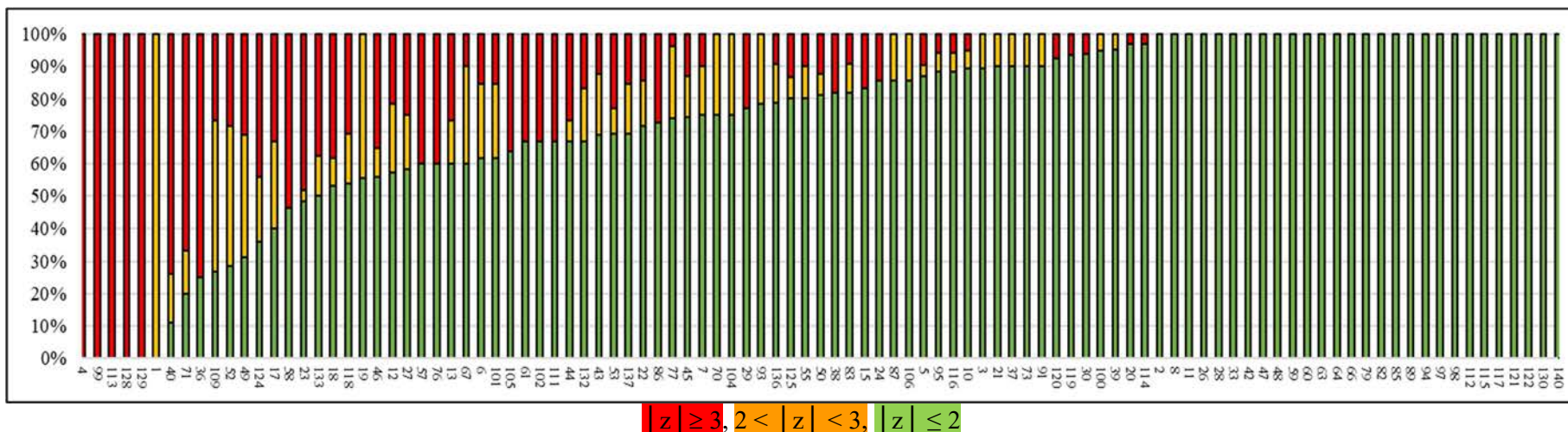


FIG. 1. z-scores calculated from the results reported by the participating laboratories per laboratory

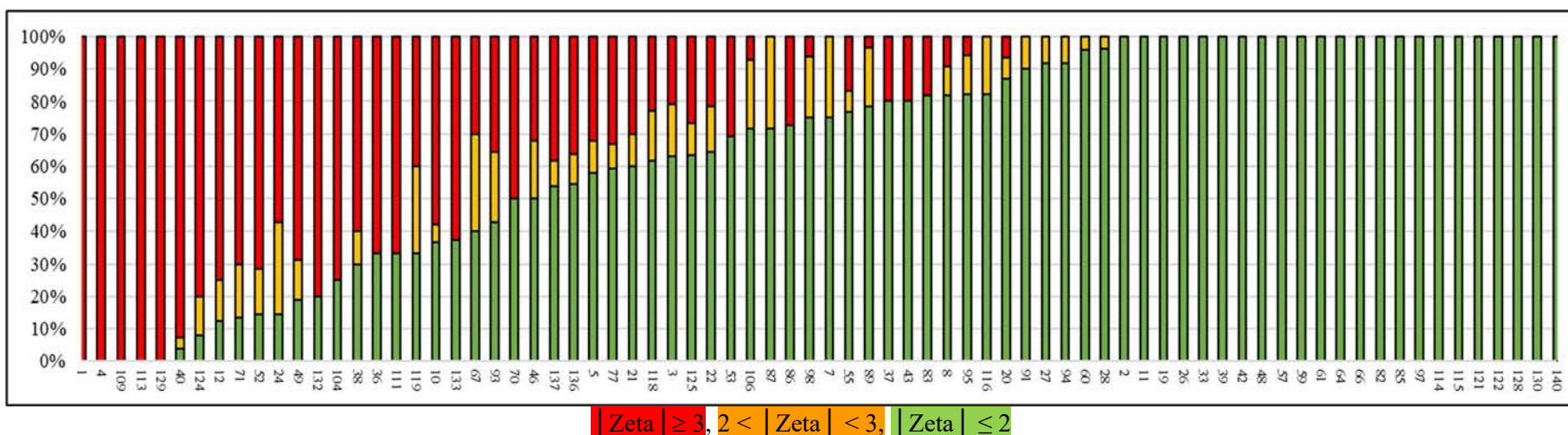


FIG. 2. Zeta-scores calculated from the results reported by the participating laboratories per laboratory

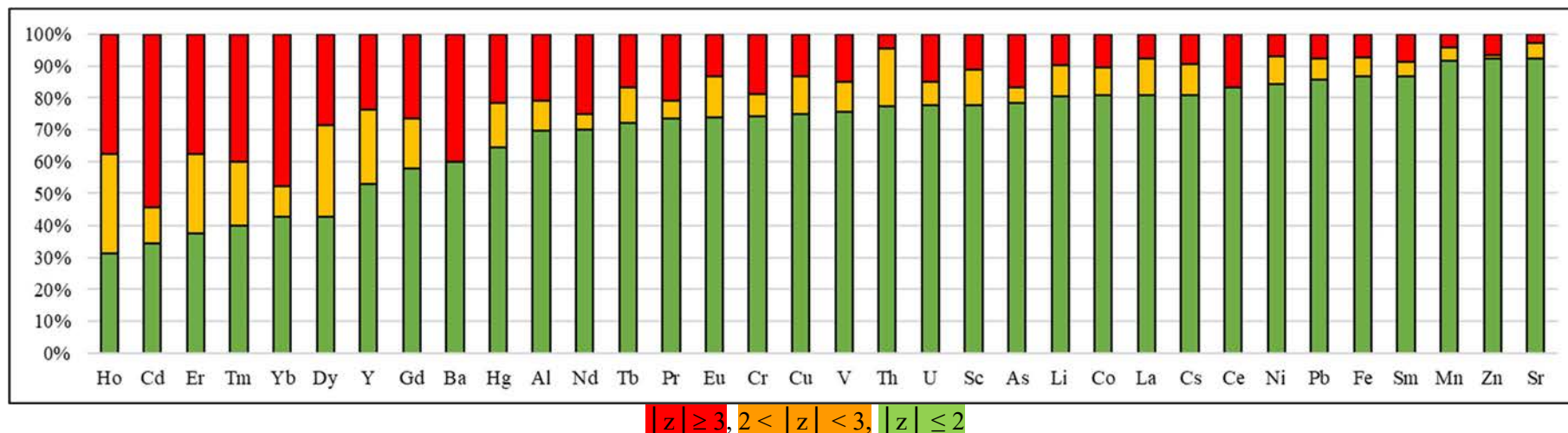


FIG. 3. z-scores calculated from the results reported by the participating laboratories for each analyte.

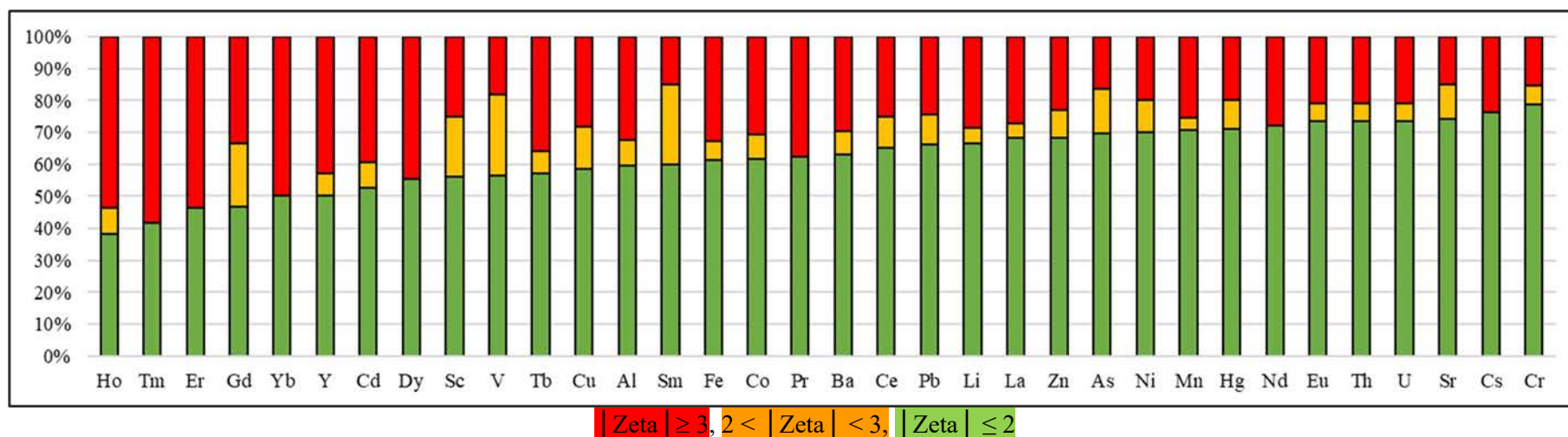


FIG. 4. Zeta-scores calculated from the results reported by the participating laboratories for each analyte.

TABLE 3. OVERALL ASSESSMENT OF LABORATORIES PERFORMANCE (z-SCORE) BY TRACE ELEMENT ($|z| \geq 3$, $2 < |z| < 3$, $|z| \leq 2$)

Lab. Code	Al	As	Ba	Cd	Co	Cr	Cs	Cu	Fe	Hg	Li	Mn	Ni	Pb	Sr	Th	U	V	Zn
1	-	-	-	-	-	-	-	-	-	-2.6	-	-	-	-	-	-	-	-	-
2	-	-	-	-	-	-	-	-	-	0	-	-	-	-	-	-	-	-	-
3	0.6	-0.3	1.6	-	-	2.3	-	0.1	1.6	-	-	1.7	0.5	0.3	0	2.1	-	1.2	0
4	-	-	-	-	-	-	-	-	-	7546.	-	-	-	-	-	-	-	-	-
5	-7.1	0.1	0	29.9	-1.1	-0.5	0.2	-1.6	-7.1	-	-0.6	0	-0.9	0.3	0.1	-	0.1	-0.7	-0.8
6	-2.3	-1.8	-3.7	-	-1	-2.3	-	-3.3	-0.8	0.6	-	-0.7	-1.1	-2	-	-	-	-2.3	-0.5
7	-	-0.7	18.6	0.3	-0.3	-0.8	-	-1.4	0.3	-2.6	-2.2	1	-0.2	-0.7	1.2	-	-1.7	-0.2	-0.4
8	0.1	-1.8	-	-	-	-1.5	-	-0.7	-1.2	-1.2	0.3	0.5	-1.8	-0.7	-	-	-	-	-0.6
10	1.3	0.6	-	-	0.1	-0.7	-0.9	-3.6	0.9	-	-	-	0	0.2	-	-2.3	-	0.5	-0.5
11	-	-	-	-	-	-	-	-	-	-0.2	-	-	-	-	-	-	-	-	-
12	-6.2	-2	-6.7	-	-0.8	-2.9	-	-1	-1.4	-2.1	-0.6	-2.1	1.5	-1.8	-	-	-	-4.2	0.3
13	-2.9	0.2	9.2	8.1	1.1	0.1	-	0.7	-0.4	7243	-	0.1	0.8	7.3	2.4	-	-	0.3	0
15	-	0.5	-	-3.5	-	-	-	-1	-1.5	-	-	-	-0.2	0.6	-	-	-	-	-
17	-6.5	-2.3	-7.2	-1.8	-2	-5.4	-	-2.2	-1.7	1.1	-3.7	-1.5	-2.4	-1.1	-	-	-5.3	-	-1.7
18	-0.2	-0.6	-0.3	-0.9	-0.2	-0.5	-4.6	-0.7	-0.5	78.1	-0.7	0.3	-0.3	0	-0.4	-3.3	-1.5	0	-0.9
19	-	-	1.8	2.4	-	-0.2	-	2.1	-	-2.3	-	-0.1	-0.2	2.1	-	-	-	-	0.5
20	-0.9	-0.3	-0.2	4.1	0.1	0	0.3	0.6	-0.5	-	-0.3	-0.7	0	-0.3	-0.6	-	-0.4	-0.7	0.4
21	2.2	0.6	-0.8	1.8	-0.3	-0.1	-1.1	0.1	0.4	-0.1	0.5	0	-0.1	-0.6	-0.7	-2.2	-1.1	1	0.6
22	-5.5	-	-	-	1.8	-	2.4	-	0	-	-	-1	-	-	-	0.9	-	-1.9	-1.9
23	-1.7	-	7765	13.6	0.3	-0.7	-	1.6	-0.7	-0.7	2.8	0.2	-	1.5	-0.6	-	-	-1	0.6
24	-	-	-	244	1.9	-2	-	-	-	-	-	0.2	2	-1.1	-	-	-	-	1.2

“-“ No data available

TABLE 3. OVERALL ASSESSMENT OF LABORATORIES PERFORMANCE (z-SCORE) BY TRACE ELEMENT ($|z| \geq 3$, $2 < |z| < 3$, $|z| \leq 2$)
cont.

Lab. Code	Al	As	Ba	Cd	Co	Cr	Cs	Cu	Fe	Hg	Li	Mn	Ni	Pb	Sr	Th	U	V	Zn
26	-	-	-	-	-	-	-	-	-	-0.5	-	-	-	-	-	-	-	-	-
27	0.8	-	-	214	-	-0.3	-	3	-0.1	-	-	-0.6	2.5	16.9	0.2	-	-	1.1	0.1
28	-0.1	1.1	-	0.9	-0.7	-0.2	-	-0.5	0	-	0.3	0	-0.2	0.8	-	-	1	-0.3	0.4
29	-5.2	-	-0.1	13.7	3.6	0.1	-	0.1	0.4	-	-	0.6	0.8	0.2	0.3	-	-	0.6	0.4
30	0.5	-	-0.8	16	-1.4	0.2	-1.5	-1.8	0.7	-1.2	-	0.6	0.2	0.1	-0.2	-0.3	-1.5	-	0.4
33	-	-	-	-	-	-	-	-	-	0.6	-	-	-	-	-	-	-	-	-
36	-	-	5926	-	-	0.7	-	4.3	-	-	-	-4.3	-	-	-	-	-	-	-
37	0.4	-0.2	-	-1.1	-	-1.2	-	-2.3	1	1.1	-	-	-0.2	1.5	-	-	-	-	0
38	-	0.1	-	122	8.8	0.5	-	1.2	-1.1	-	-1	-1.4	-1.2	1.6	-	-	-	-	0.4
39	0.4	0.2	0.2	-	0.9	1.5	0.5	-	0.9	-	-	0.4	-	-	0.2	0.3	0.6	1.4	1.7
40	-	-3.6	-6.2	-2.6	-3.6	-5.5	-2.9	-4.2	-5.5	3.4	-5.6	-5.6	-5.3	-1.6	-5.7	-	-	-5.2	-4.8
42	-	-0.2	-	0.2	-	-0.5	-	-0.3	-	-0.9	-	-0.2	0.1	0.1	-	-	-	-	-0.2
43	-2.2	-1.7	3307	-1.2	-1.4	-1.5	-	-2.5	-0.9	0.9	-1.3	-4.5	-1	-2	-	-	-2.5	-1.6	-1.9
44	-5	-0.5	-6.1	2.4	0.3	-3.7	-	0.5	-0.9	-0.8	-	-0.6	-0.2	0	-1	-	-	-3.7	0.6
45	0.2	1.3	-0.2	12	0.1	0.5	0.2	-0.7	-0.3	-	-1.3	-0.3	3.2	1.3	-0.1	0.5	-0.4	-0.1	0.1
46	-1.1	36.2	7.4	129	5.4	0.1	1.1	15.9	0.1	13.1	0.7	0.9	3.1	21.3	1	1	0.6	0.7	15.5
47	1	-	-0.8	-	1	-0.4	-	0.4	0.1	-	-0.2	-1	-0.7	-0.3	-0.6	-	-	-	-0.1
48	-	-0.2	-	1.5	-0.3	-	-	-1.1	-	-	-	-	-0.4	-0.5	-	-	-	0	-0.2
49	-3	-0.9	-4.3	-0.6	-2.5	-4	-	-2.7	-2.7	0.7	-1.6	-2.3	-2.2	-1.1	-	-	-3.9	-3.4	-2.1
50	-	-0.4	8199	28.4	-0.2	0.1	-	-1.6	-0.1	2.9	-0.8	-0.8	-0.6	-0.5	0.7	-	-0.8	-0.6	-0.3
52	-	-	-	-	-	-4.9	-	-1.4	-2.2	-	-	-2.8	-1.2	2.6	-	-	-	-	-3.1

“-“ No data available

TABLE 3. OVERALL ASSESSMENT OF LABORATORIES PERFORMANCE (z-SCORE) BY TRACE ELEMENT ($|z| \geq 3$, $2 < |z| < 3$, $|z| \leq 2$)
cont.

Lab. Code	Al	As	Ba	Cd	Co	Cr	Cs	Cu	Fe	Hg	Li	Mn	Ni	Pb	Sr	Th	U	V	Zn
53	0.5	56.1	-	9E+3	-	2.3	-	0.7	0.4	-1.2	-	1.1	1.4	0.7	0.1	-	-	63.1	0.3
55	-0.3	-0.3	-0.7	1.2	0	-1.4	-	-1.1	-0.4	-0.2	0.7	-0.2	0.1	-0.6	-0.4	-	-2.1	-0.4	0.1
57	-6.8	-6.7	-	223	-	-7.1	-	-0.5	-	0.3	-	-0.7	-1.2	2	-	-	-	-	-0.8
58	-	3.8	-5.7	37.4	-1.2	-4	-	-0.9	-	4.9	-	-1.7	-1.2	-0.2	-	-	-3	-4.4	-0.8
59	-	-	-	-	-	-	-	-	-	-0.6	-	-	-	-	-	-	-	-	-
60	0.3	-0.5	0.2	-	-0.4	-	-0.3	-0.6	-0.2	-	-	-0.1	-0.2	-0.2	-0.2	0.5	-1.1	0.5	-0.3
61	-	-4.9	-	-0.2	-	-	-	-	-	-	-	-	-	-1.2	-	-	-	-	-
63	-	-	-	-	-	-	-	-	-	1.3	-	-	-	-	-	-	-	-	-
64	-0.1	-0.6	-	-0.9	-	-1.1	-	0.8	-0.2	-1.1	-	-0.1	-0.5	-0.8	-	-	-	-	0.2
66	-1.3	0.7	0	-	-1	-1	-	-1	-0.6	-	-	-1	0	-0.1	-0.1	-	-	-0.9	-0.3
67	-1.3	-	-	-	8.9	0.6	-	-0.2	-1	-2.1	-	0.1	-2.9	2.9	-	-	-	-	0.1
70	-	-	-	-	-	-	-	2.2	-0.4	-	-	-0.4	-	-	-	-	-	-	1.7
71	-6.7	-2.1	-7.5	-3.6	-1.9	-5.7	-7.4	-2.7	-2.5	-1.3	-3.9	-1.1	-2.3	-1.1	-1.8	-	-5.2	-6	-1.4
73	-	-1.7	-	-	2.9	-1.1	-	-1.2	-	0.7	-	-0.8	-0.7	-1	-	-	-	-1.2	0.9
76	0	-	-	-6	-	-0.4	-	-7.9	0.2	1382	-	-0.9	-1.8	-0.8	-	-	-	-4.4	-
77	-	0	-0.2	5.3	0.1	-1.1	-	-0.4	-0.4	-0.3	-	-	0.2	-0.4	-	-0.3	-	-0.8	-1.1
79	-	-	-	-	-	-	-	-	-	-1.1	-	-	-	-	-	-	-	-	-
82	-1	0.2	-0.4	1.5	-0.3	-0.6	-	-0.8	-0.2	-0.3	-	-0.1	-0.1	-0.9	-	-	-	-0.3	-0.2
83	-	-	-	15.2	0.1	0.9	-	0.1	-	2.3	0.4	0.1	0.4	-0.1	-	-	-	0	0.2
85	-	0.5	0.5	-	0.4	0.3	0.2	-	-	-	0.2	0.2	0.3	1	0.4	0.3	0.1	0.1	-
86	-	1	-	126.	-0.4	-4.9	-	-0.5	-0.5	416.	-	-0.3	-0.6	-1	-	-	-	-	0.2
87	-	-0.7	-	-	-	-2.9	-	0	-0.3	-	-	-	-0.2	-0.7	-	-	-	-	1.7

“-“ No data available

TABLE 3. OVERALL ASSESSMENT OF LABORATORIES PERFORMANCE (z-SCORE) BY TRACE ELEMENT ($|z| \geq 3$, $2 < |z| < 3$, $|z| \leq 2$)
cont.

Lab. Code	Al	As	Ba	Cd	Co	Cr	Cs	Cu	Fe	Hg	Li	Mn	Ni	Pb	Sr	Th	U	V	Zn
89	-	-	0.2	-	-0.2	0.6	0.2	1.3	-	-	-	0.2	-0.2	-0.1	0.3	-1.3	-0.1	0.2	0.9
91	-0.7	-	-	-2.7	-	-0.6	-	-0.8	-1	0.3	-	-0.3	-0.5	-1.3	-	-	-	-	-0.1
93	-2.1	-0.5	-	-	-1.2	1.1	-	-1.8	-1.1	-	-	-0.5	-0.3	-	-2.2	-1	-	-2.7	0.1
94	-0.6	-	-	-	0	-0.1	-	1.5	-0.5	-	0	0	1.1	-0.8	0.1	-	-	-0.2	-0.1
95	0.1	-0.1	-	-	-	1.2	0.1	-	0.1	-	-	0.3	-	-	-	1.8	-0.1	-0.3	4.4
97	-	-	-	-	-	0	-	-0.1	-	0.6	-	-0.9	-	-0.2	-	-	-	-	0.3
98	-0.4	-0.6	-0.3	-1.1	-1	-0.1	-	-1.6	0	-1.9	-0.7	0	-0.2	-0.6	-0.2	-	-	-1	-0.6
99	-	-	-	-	-	-	-	-	-	621.5	-	-	-	-8	-	-	-	-	-
100	0.3	-0.8	0.6	2.2	0.1	-0.4	-0.2	-0.3	0.1	-1.4	0.9	0.4	-0.2	1.3	0.4	-	0	-1	0.1
101	-	-1.2	1E+4	-	-2.1	-1.5	-	-0.3	-	-2.1	-	-	-0.5	0.2	-0.9	-	-	-2.4	-0.8
102	-	-	-	-	-	-	-	8.3	5.4	-1.3	-	0.5	-	1.8	-	-	-	-	0.2
104	-	-	-	-	-	-	-	-0.7	-2.1	-	-	-1.9	-	-	-	-	-	-	-1.8
105	-	-7.9	-	18.7	-0.1	-3.8	-	-0.2	-1.9	2932	-	0.3	-0.9	0.6	-	-	-	-	-2
106	-0.6	-1.4	-	2.1	-0.3	-0.9	-	2.3	0	1.3	0.2	0	-0.1	0.4	-	-	-	-0.2	1
111	-	-	-	4.3	-	-	-	-	-	1.1	-	-	-	-1.8	-	-	-	-	-
112	-0.8	-	-	-1.6	-	-0.4	-	-0.8	-0.2	-	-	0.5	0.1	-0.7	-	-	-	-	-0.9
113	-	-7.8	-7.7	-3.3	-	-7.9	-	-7.9	-7.9	-	-	-	-7.8	-5.4	-	-	-	-	-7.9
114	-0.7	-1.5	-0.2	-0.7	-0.3	-0.6	-0.5	-0.4	-0.8	3.7	-0.5	-0.4	0.4	0.5	-0.4	-0.5	-1.2	-1	-0.5
115	0	0.3	0.3	-	-0.1	0.6	0.5	-	-0.1	1.3	-	0.1	-	-	0.5	0.4	-0.1	0.2	0
116	-0.7	-0.4	-1.5	3.8	-0.8	-1	-	-0.1	-0.3	1.8	-	-0.2	0.2	2.2	-0.7	-0.5	1.4	-0.4	-0.2
117	-	-1.3	-	-	0.1	0	-	-1	-0.6	-	-	0.1	0.6	-0.4	-	-	-	-	-0.2

“-“ No data available

TABLE 3. OVERALL ASSESSMENT OF LABORATORIES PERFORMANCE (z-SCORE) BY TRACE ELEMENT ($|z| \geq 3$, $2 < |z| < 3$, $|z| \leq 2$)
cont.

Lab. Code	Al	As	Ba	Cd	Co	Cr	Cs	Cu	Fe	Hg	Li	Mn	Ni	Pb	Sr	Th	U	V	Zn
118	-	3.7	0.4	-	-	-4	-	2.6	-1.4	-	-	-2	-3.2	-1.2	-0.2	-2.8	-	-0.9	0.2
119	-0.2	1.1	0.1	19.6	-1.3	-1.1	-	-1.8	-0.2	-1.7	-	-1	0.6	-1	0.3	-	-	-0.6	-1.1
120	0.7	1.8	-	3.6	0.1	0.1	-	-0.1	0.5	-1.9	0.4	0.1	0.5	0.5	-	-	-	-	-0.2
121	-	-	-	-	-	-	-	-	-	-0.6	-	-	-	-	-	-	-	-	-
122	-	-	-	-	-	-	-	-	-	-0.7	-	-	-	-	-	-	-	-	-
124	1.1	5.1	-0.3	-3.8	2.2	1.3	-	1.6	0.9	-	2.7	-1.7	-2.8	2.5	-1.2	-	1.4	2.1	-1
125	6.E ⁺⁶	-1.3	5129	-1.5	-0.7	-1.5	-0.6	-0.5	7E ⁺⁶	-0.7	-0.1	-0.4	-0.4	-0.2	-0.5	-1.9	-	-1.6	-0.1
128	-	-	-	-	-	-	-	-	-	8.5	-	-	-	-	-	-	-	-	-
129	-	-	-	-5.8	-	-	-	-	-	21	-	-	-	-3.7	-	-	-	-	-
130	-	-	-	-	-	-	-	-	-	1.3	-	-	-	-	-	-	-	-	-
132	-	-	-	-	-	-4.1	-	-1.3	-1.8	2.3	-	-0.1	-	-	-	-	-	-	-1
133	-5.2	-2.7	-	-1.5	-	-3.6	-	-3.1	-	-	-	-	-0.3	-1.8	-	-	-	-	-0.4
136	0.2	0.3	1.1	9.2	-0.1	-0.9	0.1	-0.2	0.4	-	1.3	0.4	-0.1	1.5	0.7	-0.3	-1.6	0	0.5
137	-0.7	-	-	-	-3.4	-	-	0.2	-0.2	-4.9	-	-0.7	-0.9	-0.5	-0.1	1.6	-	-2.1	-0.1
140	-	-	-	-	-	-	-	-	-	-1.5	-	-	-	-	-	-	-	-	-

“-“ No data available

TABLE 4. OVERALL ASSESSMENT OF LABORATORIES PERFORMANCE (z-SCORE) BY REE ($|z| \geq 3$, $2 < |z| < 3$, $|z| \leq 2$)

Lab. Code	Ce	Dy	Er	Eu	Gd	Ho	La	Nd	Pr	Sc	Sm	Tb	Tm	Y	Yb
3	0.6	-	-	-	-	-	1.6	-0.1	0.9	-	1.8	-	-	1	-
5	-0.1	0.6	0.6	1	2.7	0.7	0.4	0.2	0.3	-	0.7	1.4	0.5	0.7	0.4
7	-	-	-	-1.5	-0.3	-	-2.1	-	-	-	-	-3.1	-	-	-
10	-1.5	-	-	-	-0.9	-	-1.1	-2	-1.9	-1.3	-1	-	-	-	-
18	-3.1	-3.6	-3.6	-2.7	-2.9	-3.6	-1.3	-3.4	-3.3	-1.7	-3	-3	-3.6	-3.9	-3.7
20	0.1	-0.2	0	0.4	-0.3	-0.1	0.2	-0.2	0	-	0.2	-0.2	-0.1	0.1	-0.3
21	-1.8	-	-	-	-	-	-	-	-	-	-	-	-	-	-
22	-0.2	-	-	2.9	-	-	-1.1	-	-	0.3	-0.8	-	-	-	-3.5
23	14.7	5.7	3.3	13.7	17.2	3.3	13.8	12.7	13.2	-	13.9	8.7	1.6	-	1.1
27	-	-	-	-	-	-	-2.1	-	-	-	-	-	-	-	-
28	0.3	-0.4	-0.4	0.3	0.6	-0.3	0.1	0.1	0.4	-	0.4	-0.1	-0.4	-	-0.7
39	-0.3	0	-	2.8	-	-	1.3	-	-	1.3	-0.4	0.6	-	-	-0.6
40	-	-3.8	-4.4	-0.2	5	-4.1	-2.6	-7.9	-2.9	-	-1.5	-	-4.8	-	-5.4
45	0.3	-2.1	-2.6	0.6	0.7	-2.1	0.2	-	0.1	-3.1	0.2	-0.5	-2.3	-	41.8
46	0.9	-2.1	-2	0.9	3.2	-2.5	0.6	0.6	0.8	0.2	0.6	0	-3.2	-2.5	-3.3
55	-0.7	-2.8	-3.1	-0.7	-0.4	-2.9	-0.7	-1.2	-0.8	-	-0.5	-1.9	-	-3.2	-3.4
60	0.3	0.2	-	-0.9	-	-	-0.2	-0.2	-	-0.2	-0.5	-0.7	-	-0.6	0.6
66	-	-	-	-	-	-	-	-	-	-0.6	-	-	-	0.4	-
71	-5	-5.3	-5.8	-4.4	-4.5	-	-6.1	-5.5	-5.6	-5.1	-	-4.7	-5.2	-	-5.6
77	-1.3	-2.4	-2.1	-0.6	-0.5	-2.3	-1.6	0.8	-1.5	0	-1.2	-	-2.2	-2.1	-2.3
89	-0.1	0.4	0.5	0	0.7	0.8	0.3	0.3	0.5	0.9	0.6	-0.1	0.6	1	0.4
93	-	-	-	-	-	-	-1.2	-	-	-	0.8	-	-	-	-

“-“ No data available

TABLE 4. OVERALL ASSESSMENT OF LABORATORIES PERFORMANCE (z-SCORE) BY REE ($|z| \geq 3$, $2 < |z| < 3$, $|z| \leq 2$) cont..

Lab. Code	Ce	Dy	Er	Eu	Gd	Ho	La	Nd	Pr	Sc	Sm	Tb	Tm	Y	Yb
95	0.2	0.4	-	-0.2	-	-	0.2	-	-	-0.1	0.6	-	-	-	2.9
100	-	-	-	-	-	-	-	-	-	-	-	-	-	-1.7	-
101	3.6	-	-	-	-	-	-	-	-	-	-	-	-	0	-
109	-1.5	-3.1	-2.9	-1.4	-2.8	-3.1	-1.4	-2.3	-1.2	-2.1	-2.1	-2.8	-3.1	-2.8	-3.1
114	-0.4	0.1	0.3	0.2	0.7	0.2	-0.1	-0.1	-0.1	-1.6	0.4	0.5	-0.1	1.1	-0.2
115	-0.1	0.9	-	0	-	-	0	0	-	0	0.1	0.3	-	-	0.3
118	-	-	-	-	-	-	-	-	-	-	-	-	-	15	-
124	-	-6.8	-6.8	-6.5	-6.6	-6.8	-	-6.4	-5.9	-	-	-	-6.9	-	-6.9
125	-1.4	-2.8	-	-1.2	-1.1	-3.04	-1.5	-1.3	-1.2	-1.5	-0.9	-1.8	-	-2.9	-
136	-0.5	-2.4	-2.8	0.6	-0.5	-2.9	0.1	0.1	0.3	-1.1	0.1	-1.3	-2.8	-3.3	-3
137	-	-	-	-	-	-	-	-	-	2.3	-	-	-	-	-

“-“ No data available

TABLE 5. OVERALL ASSESSMENT OF LABORATORIES PERFORMANCE (*ZETA*-SCORE) BY TRACE ELEMENT (|Zeta| ≥ 3, 2 < |Zeta| < 3, |Zeta| ≤ 2)

Lab. Code	Al	As	Ba	Cd	Co	Cr	Cs	Cu	Fe	Hg	Li	Mn	Ni	Pb	Sr	Th	U	V	Zn
1	-	-	-	-	-	-	-	-	-	-3.9	-	-	-	-	-	-	-	-	-
2	-	-	-	-	-	-	-	-	-	0	-	-	-	-	-	-	-	-	-
3	0.8	-0.7	3.5	-	-	2.4	-	0.1	3.5	-	-	3.6	1.1	0.5	0	1.9	-	2.1	-0.1
4	-	-	-	-	-	-	-	-	-	13.3	-	-	-	-	-	-	-	-	-
5	-37	0.3	-0.2	27.6	-6.4	-0.6	0.8	-3.6	-30	-	-2.8	-0.2	-4	0.8	0.5	-	0.3	-2.7	-3
7	-	-2.1	-	0.4	-0.7	-0.8	-	-2.4	0.5	-2.2	-	1.5	-0.5	-1.4	-	-	-	-0.4	-1
8	0.1	-2.8	-	-	-	-1.5	-	-1.2	-1.3	-1.5	0.3	0.9	-3.7	-1.1	-	-	-	-	-0.9
10	5.6	0.3	-	-	0.3	-0.7	-4.8	-3.6	3.1	-	-	-	0.1	0.4	-	-4.6	-	1.2	-1.3
11	-	-	-	-	-	-	-	-	-	-0.2	-	-	-	-	-	-	-	-	-
12	-	-	-	-	-4.1	-3.5	-	-2.5	-5.5	-	-	-11	5.9	-4.7	-	-	-	-	1.1
19	-	-	1.8	1.1	-	-0.2	-	0.6	-	-1.3	-	-0.1	-0.1	0.7	-	-	-	-	0.4
20	-3.8	-0.9	-0.8	1.7	0.6	0	1.5	1.4	-2	-	-1.1	-3.4	0	-0.7	-2.8	-	-1.4	-2.9	1
21	4.7	0.8	-3.3	1.8	-1.3	-0.1	-3.5	0.3	1.6	0	1.4	0	-0.6	-1	-3.2	-4.6	-2	2.4	2.2
22	-10	-	-	-	3.1	-	1.9	-	0	-	-	-0.7	-	-	-	0.7	-	-2.1	-2.2
24	-	-	-	5.2	5.5	-2.3	-	-	-	-	-	1.2	7.4	-2.8	-	-	-	-	3.4
26	-	-	-	-	-	-	-	-	-	-0.5	-	-	-	-	-	-	-	-	-
27	0.3	-	-	0.8	-	-0.1	-	0.8	-0.2	-	-	-0.5	1.1	2.8	0.6	-	-	1.3	0.1
28	-0.3	2.1	-	1.2	-1.6	-0.2	-	-1	0	-	0.6	0	-0.3	1.5	-	-	2	-0.6	0.8
33	-	-	-	-	-	-	-	-	-	0.1	-	-	-	-	-	-	-	-	-
36	-	-	-	-	-	0.6	-	11	-	-	-	-24	-	-	-	-	-	-	-
37	1.4	-0.8	-	-1	-	-1.4	-	-5.6	1.8	1.5	-	-	-0.5	3.3	-	-	-	-	0.1

“-“ No data available

TABLE 5. OVERALL ASSESSMENT OF LABORATORIES PERFORMANCE (ZETA-SCORE) BY TRACE ELEMENT ($|Zeta| \geq 3$, $2 < |Zeta| < 3$, $|Zeta| \leq 2$) cont.

Lab. Code	Al	As	Ba	Cd	Co	Cr	Cs	Cu	Fe	Hg	Li	Mn	Ni	Pb	Sr	Th	U	V	Zn
38	-	0.2	-	22.8	20.2	0.6	-	2.7	-3.8	-	-3.8	-	-4.7	3.5	-	-	-	-	1.1
39	0.3	0.2	0.1	-	0.7	1	0.4	-	0.9	-	-	0.5	-	-	0.1	0.3	0.4	1.3	1
40	-	-12	-32	-2.9	-19	-6.4	-16	-11	-23	5.2	-24	-31	-24	-4.2	-28	-	-	-22	-19
42	-	-0.2	-	0.2	-	-0.6	-	-0.4	-	-1	-	-0.5	0.3	0.2	-	-	-	-	-0.3
43	-6.3	-0.5	-	-0.2	-1.3	-0.9	-	-0.4	-3.8	0.1	-0.9	-9.6	-0.9	-0.8	-	-	-1	-0.8	-1.1
46	-2.5	15.8	2.6	4.3	3.2	0.1	0.9	7.1	0.1	7.5	1.3	2.2	4.9	18.4	1.7	0.4	0.5	2.5	24.3
48	-	-0.4	-	1.4	-0.7	-	-	-1.8	-	-	-	-	-0.9	-0.7	-	-	-	0.1	-0.4
49	-9	-1.1	-16	-0.6	-6.5	-4.4	-	-6.2	-8.4	1	-3.4	-5.9	-2.8	-2.5	-	-	-11	-12	-6.1
52	-	-	-	-	-	-5.5	-	-2.4	-4.1	-	-	-3.3	-1.3	3.6	-	-	-	-	-5.7
53	0.4	7.7	-	25.5	-	2	-	0.3	1.4	-0.7	-	5.9	0.9	0.7	0.1	-	-	4.4	0.1
55	-0.5	-0.4	-1.2	1.3	0.1	-1.4	-	-1.3	-0.8	-0.2	0.6	-0.3	0.2	-0.4	-0.6	-	-2.2	-0.7	0.2
57	-	-	-	-	-	-	-	-	-	0.5	-	-	-	-	-	-	-	-	-
59	-	-	-	-	-	-	-	-	-	-0.4	-	-	-	-	-	-	-	-	-
60	0.5	-1	0.4	-	-1	-	-0.7	-1.3	-0.4	-	-	-0.2	-1	-0.3	-0.3	0.9	-1.9	1	-0.6
61	-	-0.4	-	0	-	-	-	-	-	-	-	-	-	-0.4	-	-	-	-	-
64	-0.1	-0.4	-	-1	-	-1.1	-	0.9	-0.3	-0.8	-	-0.1	-0.6	-1	-	-	-	-	0.3
66	-1.2	0.7	-0.1	-	-0.8	-0.9	-	-1.8	-1.1	-	-	-1.4	-0.1	-0.1	-0.2	-	-	-1.2	-0.7
67	-2.9	-	-	-	7.5	0.6	-	-0.4	-2.1	-2.6	-	0.1	-8.5	3.6	-	-	-	-	0.1
70	-	-	-	-	-	-	-	3.8	-1.3	-	-	-1.4	-	-	-	-	-	-	3.9
71	-27	-2.6	-36	-4.4	-2.3	-6.4	-39	-2.7	-3.1	-1.6	-6.8	-1.2	-2.8	-1.3	-2.2	-	-12	-17	-1.5
77	-	0.1	-0.7	5.5	0.4	-1.2	-	-0.9	-1.1	-0.3	-	-	0.3	-0.7	-	-0.5	-	-2.9	-1.5

-- No data available

TABLE 5. OVERALL ASSESSMENT OF LABORATORIES PERFORMANCE (ZETA-SCORE) BY TRACE ELEMENT ($|Zeta| \geq 3$, $2 < |Zeta| < 3$, $|Zeta| \leq 2$) cont.

Lab. Code	Al	As	Ba	Cd	Co	Cr	Cs	Cu	Fe	Hg	Li	Mn	Ni	Pb	Sr	Th	U	V	Zn
82	-1.4	0.3	-0.6	1.6	-0.4	-0.5	-	-1.2	-0.3	-0.3	-	-0.1	-0.2	-1.5	-	-	-	-0.4	-0.4
83	-	-	-	4.4	0.2	1	-	0.2	-	3.3	0.9	0.5	0.9	-0.1	-	-	-	0.1	0.2
85	-	0.9	1.5	-	1	0.3	0.5	-	-	-	0.6	0.9	0.5	1.3	1.3	0.5	0.3	0.2	-
86	-	0.8	-	9.5	-0.4	-5.2	-	-0.5	-0.5	6.5	-	-0.3	-0.6	-0.9	-	-	-	-	0.2
87	-	-1.4	-	-	-	-2.9	-	0.1	-0.6	-	-	-	-0.5	-1.5	-	-	-	-	2.4
89	-	-	0.8	-	-1.1	0.7	1.4	2.3	-	-	-	0.7	-0.7	-0.3	1.3	-2.8	-0.3	0.8	3.4
91	-1.9	-	-	0	-	-0.7	-	-0.5	-2.2	0	-	-1.9	-0.4	-1	-	-	-	-	-0.4
93	-8.4	-1.3	-	-	-2.2	0.9	-	-4.3	-3.5	-	-	-1.3	-1	-	-3.6	-1.2	-	-9.3	0.3
94	-1.4	-	-	-	0.1	-0.1	-	1.4	-1.3	-	0.1	0.1	2.4	-1.4	0.2	-	-	-0.5	-0.2
95	0.7	-0.2	-	-	-	1.2	0.2	-	0.3	-	-	1	-	-	-	3.5	0	-0.4	2.3
97	-	-	-	-	-	0	-	0	-	0.6	-	-1.7	-	-0.1	-	-	-	-	0.3
98	-0.7	-1.3	-0.7	-1.5	-2.6	-0.1	-	-3.4	0	-2.6	-1.6	-0.1	-0.5	-1	-0.5	-	-	-2.8	-1.8
104	-	-	-	-	-	-	-	-1.3	-6.1	-	-	-9.8	-	-	-	-	-	-	-5.1
106	-1.5	-2.8	-	2.3	-0.6	-1	-	3.4	0.1	1.7	0.4	0	-0.1	0.7	-	-	-	-0.7	2.1
111	-	-	-	3.3	-	-	-	-	-	1	-	-	-	-3.7	-	-	-	-	-
113	-	-28	-40	-5.3	-	-9.3	-	-20	-33	-	-	-	-36	-14	-	-	-	-	-31
114	-0.4	-0.1	0	0	0	-0.4	0	-0.1	-0.2	0	-0.2	-1.6	0.1	0.1	-0.7	0	0	-0.8	-0.4
115	0	0.6	0.8	-	-0.3	0.6	1.3	-	-0.3	1.6	-	0.3	-	-	1.3	0.7	-0.2	0.6	0.1
116	-1.5	-0.4	-2.2	2.3	-1.1	-0.8	-	-0.1	-0.3	1.5	-	-0.2	0.2	2.1	-0.9	-0.6	1.1	-0.4	-0.1
118	-	2.7	0.2	-	-	-1.6	-	0.8	-6	-	-	-4.7	-2.7	-0.8	0	-1.8	-	-0.9	0.3
119	-0.7	3.7	0.1	6.3	-8	-1.3	-	-4.6	-0.5	-2.3	-	-5.5	2.7	-2.6	1.7	-	-	-2.5	-4.3

-- No data available

TABLE 5. OVERALL ASSESSMENT OF LABORATORIES PERFORMANCE (ZETA-SCORE) BY TRACE ELEMENT ($|Zeta| \geq 3$, $2 < |Zeta| < 3$, $|Zeta| \leq 2$) cont.

Lab. Code	Al	As	Ba	Cd	Co	Cr	Cs	Cu	Fe	Hg	Li	Mn	Ni	Pb	Sr	Th	U	V	Zn
121	-	-	-	-	-	-	-	-	-	-0.8	-	-	-	-	-	-	-	-	-
122	-	-	-	-	-	-	-	-	-	-1.1	-	-	-	-	-	-	-	-	-
124	2.9	6.4	-1.4	-6	5.1	1.4	-	2.6	2.1	-	9.4	-9.5	-9.6	4.9	-5.2	-	4.2	6.6	-3.6
125	14.1	-1.5	13.3	-1.7	-1.1	-1.3	-1.2	-0.8	15.7	-0.9	-0.1	-0.7	-0.6	-0.3	-0.9	-4.1	-	-2.3	-0.2
128	-	-	-	-	-	-	-	-	-	0.9	-	-	-	-	-	-	-	-	-
129	-	-	-	-8.9	-	-	-	-	-	6.6	-	-	-	-6.6	-	-	-	-	-
130	-	-	-	-	-	-	-	-	-	1.6	-	-	-	-	-	-	-	-	-
132	-	-	-	-	-	-	-	-3.4	-7.3	3.5	-	-0.3	-	-	-	-	-	-	-3.2
133	-16	-5.9	-	-1.7	-	-3.9	-	-7	-	-	-	-	-0.6	-4.1	-	-	-	-	-0.6
136	0.7	0.9	4	4.9	-0.8	-1.1	0.2	-0.6	1.7	-	4.2	1.9	-0.2	3.6	2.6	-0.6	-5.8	0.2	2
137	-3.1	-	-	-	-10	-	-	0.1	-0.9	-6.7	-	-2.4	-1.5	-0.8	-0.6	1.4	-	-7.6	-0.3
140	-	-	-	-	-	-	-	-	-	-1.5	-	-	-	-	-	-	-	-	-

“-“ No data available

TABLE 6. OVERALL ASSESSMENT OF LABORATORIES PERFORMANCE (ZETA-SCORE) BY REE (|Zeta| ≥ 3, 2 < |Zeta| < 3, |Zeta| ≤ 2)

Lab. Code	Ce	Dy	Er	Eu	Gd	Ho	La	Nd	Pr	Sc	Sm	Tb	Tm	Y	Yb
3	1.8	-	-	-	-	-	3.3	-0.1	1.4	-	2.6	-	-	1.8	-
5	-0.6	1.7	1.9	3.6	8.9	1.8	1.2	0.8	1.1	-	2.9	5.4	1.3	1.4	1.6
10	-6.5	-	-	-	-2.1	-	-3.3	-8	-5.4	-3.4	-3.3	-	-	-	-
20	0.4	-0.4	-0.1	1.3	-1	-0.3	0.7	-1.1	0	-	0.5	-0.1	-0.2	0.1	-1.2
21	-12.4	-	-	-	-	-	-	-	-	-	-	-	-	-	-
22	-0.2	-	-	1.9	-	-	-0.9	-	-	0.6	-1.1	-	-	-	-3.3
27	-	-	-	-	-	-	-0.6	-	-	-	-	-	-	-	-
28	0.7	-0.8	-0.9	0.6	1.1	-0.6	0.1	0.2	0.8	-	0.9	-0.2	-0.9	-	-1.6
39	-0.2	0	-	0.6	-	-	1.2	-	-	1.1	-0.3	0.4	-	-	-0.5
40	-	-11.6	-15.3	-0.8	16.8	-11	-10.1	-43.4	-11.3	-	-6.1	-	-17.4	-	-22.2
46	1.5	-1.8	-1.4	0.4	2.1	-2.3	1.5	1.1	0.7	0.3	0.3	0	-6.3	-3.7	-3.2
55	-1	-3.9	-4.5	-0.9	-0.6	-4	-1.1	-1.4	-1	-	-0.5	-2.9	-	-3.8	-4.6
60	0.9	0.2	-	-2.5	-	-	-0.6	-0.4	-	-0.5	-1.3	-1.3	-	-1.3	1.4
66	-	-	-	-	-	-	-	-	-	-0.9	-	-	-	0.6	-
71	-11.9	-10.5	-14.4	-8.5	-9.6	-	-17.1	-14.3	-12.8	-9.9	-	-10.7	-9.8	-	-14.1
77	-4.3	-3.8	-3.3	-0.9	-0.7	-3.8	-3.3	1.9	-3.3	0	-1.9	-	-3.8	-2.5	-4
89	-1.1	1	1.1	-0.1	2.1	1.6	1.1	1.3	1.6	2.4	2.5	-0.5	1.7	2	1.4
93	-	-	-	-	-	-	-2.7	-	-	-	2.4	-	-	-	-
95	0.3	0.5	-	-0.2	-	-	0.8	-	-	-0.4	2.3	-	-	-	0.9
109	-4.7	-9.2	-10.1	-6.4	-9.4	-8.3	-5.6	-12.3	-4.7	-5.9	-8.8	-10.6	-10.9	-6.7	-12.2
114	-0.2	0	0	0	0	0	0	0	0	-0.1	0	0	0	0.2	0
115	-0.3	1.8	-	-0.1	-	-	0.1	0.1	-	0.1	0.3	0.9	-	-	0.8

“-“ No data available

TABLE 6. OVERALL ASSESSMENT OF LABORATORIES PERFORMANCE (ZETA-SCORE) BY REE (|Zeta| ≥ 3, 2 < |Zeta| < 3, |Zeta| ≤ 2)
cont.

Lab. Code	Ce	Dy	Er	Eu	Gd	Ho	La	Nd	Pr	Sc	Sm	Tb	Tm	Y	Yb
118	-	-	-	-	-	-	-	-	-	-	-	-	-	9.9	-
124	-	-20.8	-23.5	-29.3	-22.2	-18.5	-	-33	-20.8	-	-	-	-24.9	-	-28.3
125	-2.1	-5.3	-	-2	-2	-5.9	-1.8	-1.9	-1.6	-2.1	-1.5	-3.1	-	-4.4	-
136	-2.6	-6.6	-7.8	1.6	-1.3	-7.1	0.3	0.2	1	-2.9	0.4	-5	-7.8	-7.7	-4.4
137	-	-	-	-	-	-	-	-	-	6.3	-	-	-	-	-

“-“ No data available

6.1. z-SCORES

The z -scores compare each participating laboratory's deviation from the assigned value against the target standard deviation (σ_t) for proficiency assessment. The σ_t was set by the ILC organizer at 12.5 %, meaning the maximum acceptable deviation ($|z| \leq 2$) corresponds to 25% of the assigned value.

From the 1341 calculated z -scores, 74% were satisfactory with $|z| \leq 2$, while 17% were unsatisfactory with $|z| \geq 3$. Among the 101 participating laboratories, 28 achieved satisfactory z -scores $|z| \leq 2$ for all their reported values. Of these, 11 laboratories only reported results for one analyte.

As shown in Figure 3, some REE were satisfactorily measured by fewer than half of the participants reporting these analytes, likely due to unresolved analytical issues or inadequate sample preparation (i.e., partial decomposition). In addition to being emerging contaminants, REEs are widely used in the study of geochemical processes. For this purpose, REE concentrations in sediment are typically normalized to a reference material, such as Post-Archean Australian Shale, to analyze trends or detect anomalies. Therefore, inaccurate results of REE concentrations can lead to incorrect interpretations, especially if the inaccuracies affect specific elements within the lanthanide series.

It was also observed that some results receiving negative unsatisfactory z -scores (i.e., $z\text{-score} \leq -3$), indicating a significant low bias, were associated with the partial decomposition of the ILC sediment sample (i.e., not using hydrofluoric acid) during digestion. This issue was particularly evident for Al, Cr and V. Due to the high level of silicates in marine sediment samples, complete dissolution is not trivial analytically. The addition of hydrofluoric acid is essential for the total decomposition of the silicate lattice. Without hydrofluoric acid, the dissolution of a sediment sample remains incomplete, resulting in negatively biased mass fractions for refractory elements such as Al or Cr. For analytes like Al, which are commonly used to normalize trace elements in marine sediment, it is recommended to use the total concentration rather than the leachable portion as it accounts for all forms of the selected analyte present in the sample.

Conversely, for Cd and Hg, a high proportion of z -scores exceeded 3 (80% and 93%, respectively), suggesting analytical difficulties in accurately determining these analytes at low levels. These challenges may stem from issues such as uncontrolled contamination, unresolved interferences, or incorrect estimation of the quantification limit.

Participating laboratories with results assessed as questionable and/or unsatisfactory are encouraged to carefully review their laboratory procedures and working instructions.

6.2. ZETA-SCORES

The *Zeta*-score assesses the agreement between a laboratory's result and the assigned value while accounting for the associated uncertainties. The denominator in Eq. (2) includes the combined uncertainties of both the reference values and the reported values by the participating laboratories.

For the 25 laboratories that did not report uncertainties associated with their results, *Zeta*-scores could not be calculated. A significant number of participating laboratories did not report uncertainty estimations, despite it being a fundamental component of method validation. Laboratories are encouraged to establish routine procedures for uncertainty estimation to improve the reliability and validity of their results.

As shown in Figures 1 to 4, the comparison between measurement performances evaluated using *z* - score and *Zeta*-score indicates that the number of unsatisfactory *Zeta*-scores is higher than that of unsatisfactory *z*-score (17% of calculated *z*-scores and 27% of calculated *Zeta*-scores). Only 17 participating laboratories reported results that were evaluated as 100% satisfactory with both $|z|$ and $|Zeta| \leq 2$; of these, nine participating laboratories reported results for only one analyte.

Relative expanded uncertainties ranged from 0.01% to nearly 40000%, indicating inconsistencies in the approaches used for uncertainty estimation. A few laboratories (i.e., laboratory codes 61, 91 and 114) evidently reported their expanded uncertainties in incorrect units, resulting in extreme *U* values, although many of the high values observed could not be readily explained.

Since *Zeta*-scores incorporate the estimation of uncertainties, results receiving $|Zeta| \geq 3$ while $|z| < 3$ could indicate an underestimation of uncertainties. Figure 5 shows the distribution of results per range of reported relative expanded uncertainties along with the number of unsatisfactory scores. More than 40% of the results receiving unsatisfactory *Zeta*-scores were reported with expanded uncertainties below 5%, likely estimated only as the standard deviation of replicate analyses. In general, laboratories should be aware that uncertainties based solely on the precision of measurements (measurement standard deviation) are frequently underestimated. Such estimates typically just reflect variability coming from the measurement step and usually exclude the contribution of uncertainty from other major contributors such as recovery, procedural blank *etc.*

It should also be noted that an unsatisfactory *Zeta*-score can also result from an inappropriate estimation of the mass fraction of the respective trace element. Participating laboratories reporting results receiving $|z|$ and $|Zeta| \geq 3$ are encouraged to review their analytical procedures, as noted in Section 6.1. In fact, 90% of reported results with unsatisfactory *z*-score ($|z| \geq 3$) also received an unsatisfactory *Zeta*-score.

Participating laboratories that received $|z| < 3$ and $|Zeta| \geq 3$ are specifically encouraged to review their approach to the estimation of measurement uncertainty.

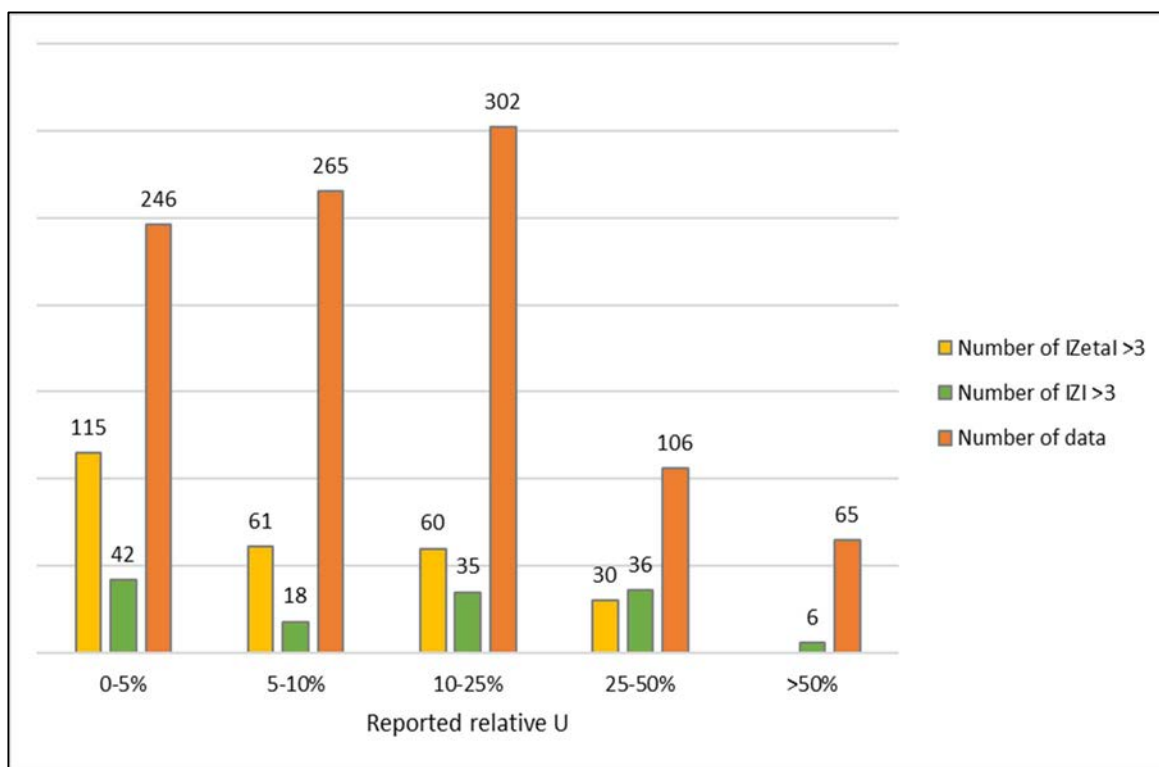


FIG. 5. Unsatisfactory results versus reported relative expanded uncertainties.

6.3. ANALYTICAL METHODS

Table 7 presents the distribution of values reported by different techniques, along with the number of participating laboratories being equipped with each type of instrumentation. The analytical methods used by participating laboratories in this ILC can be categorized into three groups: nondestructive techniques (neutron activation analysis, X-ray fluorescence spectroscopy); plasma spectrometric methods (inductively coupled plasma mass spectrometry and inductively coupled plasma optical emission spectrometry) and atomic absorption spectroscopy methods. The most frequently used technique was inductively coupled plasma mass spectrometry, which accounted for more than half of all reported values and was utilized by 43% of the participating laboratories. For mercury determination, solid mercury analyzers were prevalent, with nearly 40% of laboratories applying this technique, contributing to almost 60% of the reported mercury results.

TABLE 7. DISTRIBUTION OF THE INSTRUMENTAL METHODS APPLIED IN THE ILC

Instrumental Method	Reported values	Number of laboratories
Inductively coupled plasma mass spectrometry	54.7%	43
Inductively coupled plasma optical emission spectrometry	12.3%	23
Flame atomic absorption spectroscopy	8.1%	20
X-ray fluorescence spectroscopy	7.9%	10
Neutron activation analysis	7.7%	6
Graphite furnace atomic absorption spectroscopy	4.0%	15
Solid atomic absorption spectroscopy (mercury analyzer)	2.8%	37
Cold vapor/hydride atomic absorption spectroscopy	1.4%	19
Microwave Plasma-Atomic Emission Spectroscopy	0.7%	1
Cold Vapor Atomic Fluorescence Spectroscopy	0.2%	3
Cold vapor/hydride Inductively coupled plasma mass spectrometry	0.1%	2
Not reported	0.1%	3

6.4. REVIEW OF QUESTIONNAIRES AND OTHER VALIDATION PARAMETERS

Participants were asked to complete a questionnaire; however a small number (eight out of the 101 participating laboratories) did not respond, and several others provided incomplete answers. This made the evaluation of their answer's incomplete.

Approximately 70% of participating laboratories reported having quality control procedure in place, while only 55% indicated the existence of a quality system. About 30% of participating laboratories stated to be accredited, though not all of them for the determination of trace elements in marine sediments.

Participating laboratories were also requested to report the detection limits and quantification limits of their analytical procedures. With few exceptions, these parameters were appropriately evaluated, and no numerical results were reported when the quantification limit exceeded the assigned values.

Thirteen participants (12.8%) did not report results for a CRM in the reporting form. An important criterion in reference material selection is the principle of matrix and concentration range matching with the test sample. CRMs used in this interlaboratory comparison were generally appropriately selected: most laboratories were using sediment-based CRM when analyzing the ILC sediment sample. The two most used CRMs were IAEA-158A and MESS 4 used by 20% and 14% of the participating laboratories respectively.

More than 30% of results reported without the use of CRMs were deemed unsatisfactory, compared to only 13% of those reported with CRM use. The regular inclusion of a CRM in internal quality control is a key practice for ensuring the reliability of results produced in a laboratory, as recommended by ISO/IEC 17025:2017 [5]. Participating laboratories that do not routinely analyze CRMs alongside their test samples are therefore encouraged to do so.

7. CONCLUSIONS

This ILC attracted a significant number of participants from 60 Members States reflecting strong worldwide interest from laboratories for the external evaluation of their analytical performance for the determination of trace elements in marine sediment.

The overall performance of the laboratories taking part in this ILC was generally satisfactory, with 74% of z - scores and 64% of *Zeta*-scores assessed as satisfactory. However, fewer than 20% of participating laboratories were able to report results that were evaluated as 100% satisfactory.

Notably, nearly 20% of participating laboratories did not report their results with respective associated uncertainties, despite the importance of this method performance criteria.

Approximately 20% of participating laboratories reported results for REEs, demonstrating a growing interest in monitoring these emerging contaminants. However, the evaluation of reported REE results revealed ongoing analytical challenges, demonstrating the need for continued effort in helping Member States' laboratories in maintaining and improving the reliability of analytical measurement results for REE in marine samples.

All participating laboratories are encouraged to thoroughly investigate the cause of any unsatisfactory scores (i.e., $|z|$ and/or $|Zeta| \geq 3$) and implement appropriate corrective actions to prevent recurrence of the problem. This practice aligns with the requirements of ISO/IEC 17025:2017 [5].

Some participating laboratories still need to strengthen their quality assurance and quality control procedures, particularly through the regular use of CRMs and the implementation of quality control charts in their routine laboratory practice. This process provides ongoing feedback to analysts and is an essential tool for monitoring data quality, ultimately contributing to the generation of reliable results.

A complete catalogue of IAEA reference materials is published regularly and can be consulted on the IAEA website: <https://analytical-reference-materials.iaea.org/>.

As a follow-up to their participation in this ILC, participating laboratories are encouraged to contact the organizers for further information on any of the points discussed above if necessary.

APPENDIX

REPORTED RESULTS BY ELEMENTS

Figures 6 – 39 provide graphical representation of the individual results and their expanded uncertainties (as dot and error bar) as well as the assigned value $\pm$ expanded uncertainty ($k=2$) as black line $\pm$ dash black line. The red dashed lines represent assigned value ± 2 target standard deviation, and the black arrows represents results out of scale.

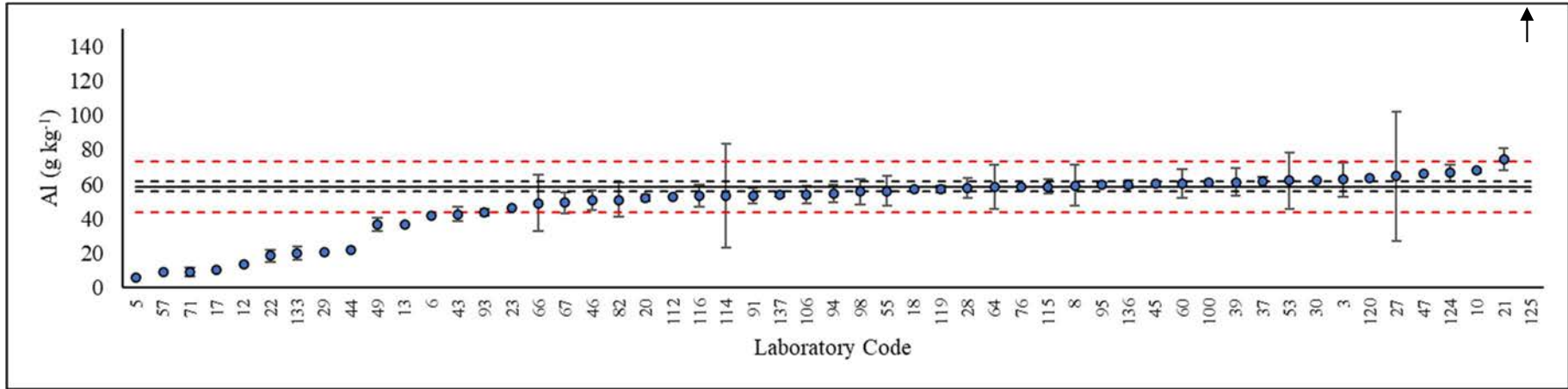


FIG. 6. Laboratory results for Al mass fraction (g kg^{-1}) in ILC sample.

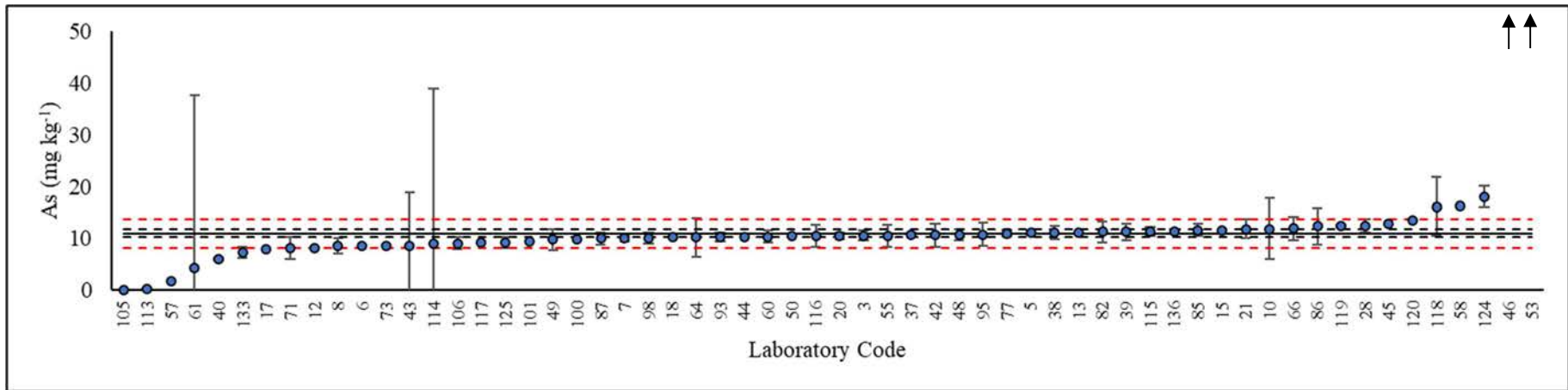


FIG. 7. Laboratory results for As mass fraction (mg kg^{-1}) in ILC sample.

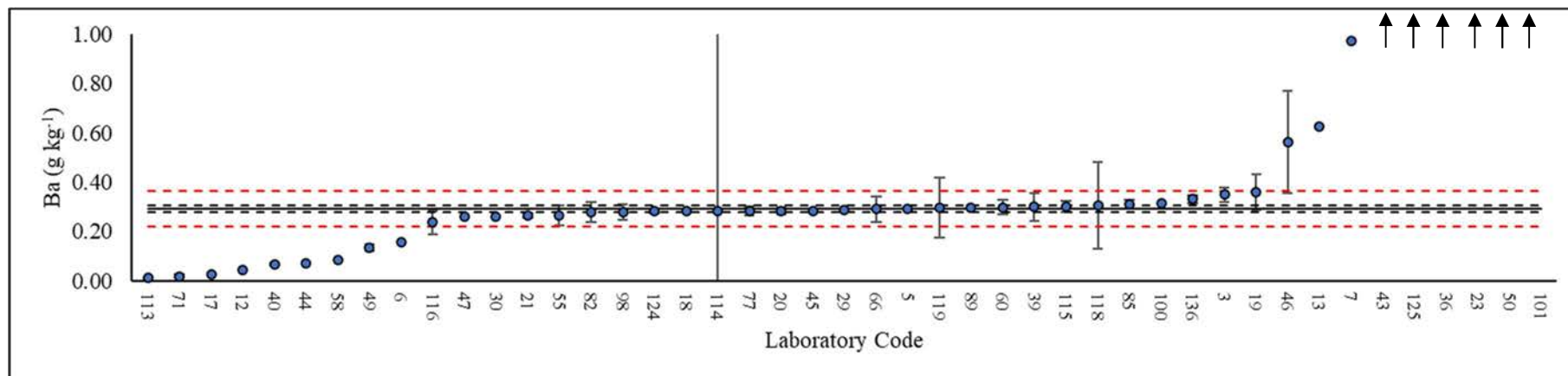


FIG. 8. Laboratory results for Ba mass fraction (g kg^{-1}) in ILC sample.

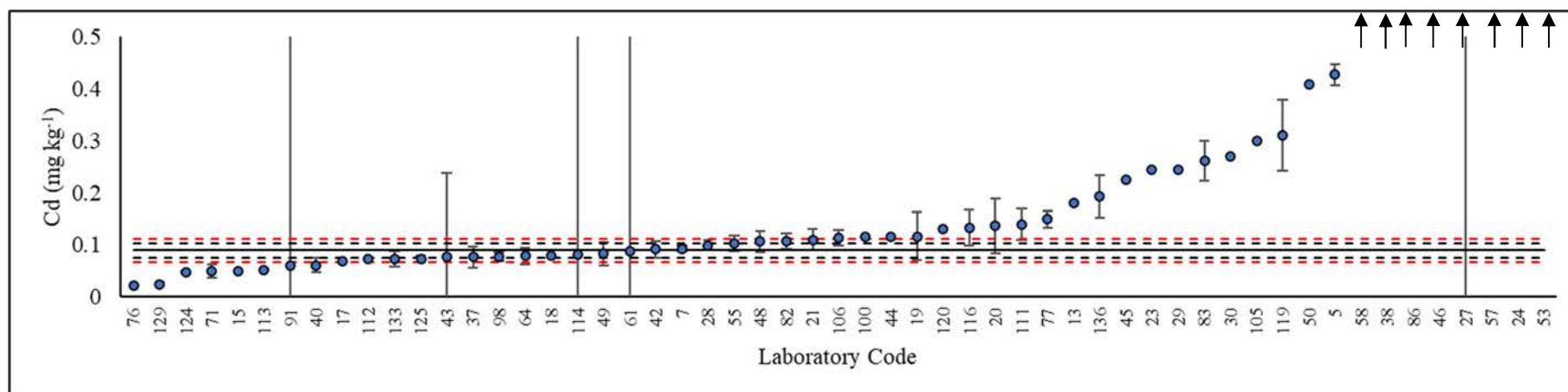


FIG. 9. Laboratory results for Cd mass fraction (mg kg^{-1}) in ILC sample.

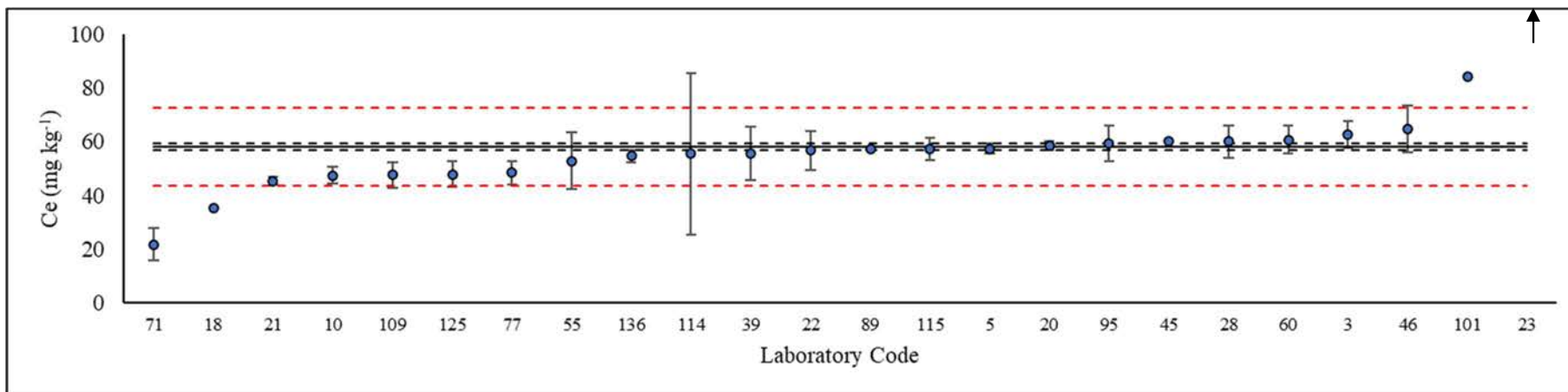


FIG. 10. Laboratory results for Ce mass fraction (mg kg^{-1}) in ILC sample.

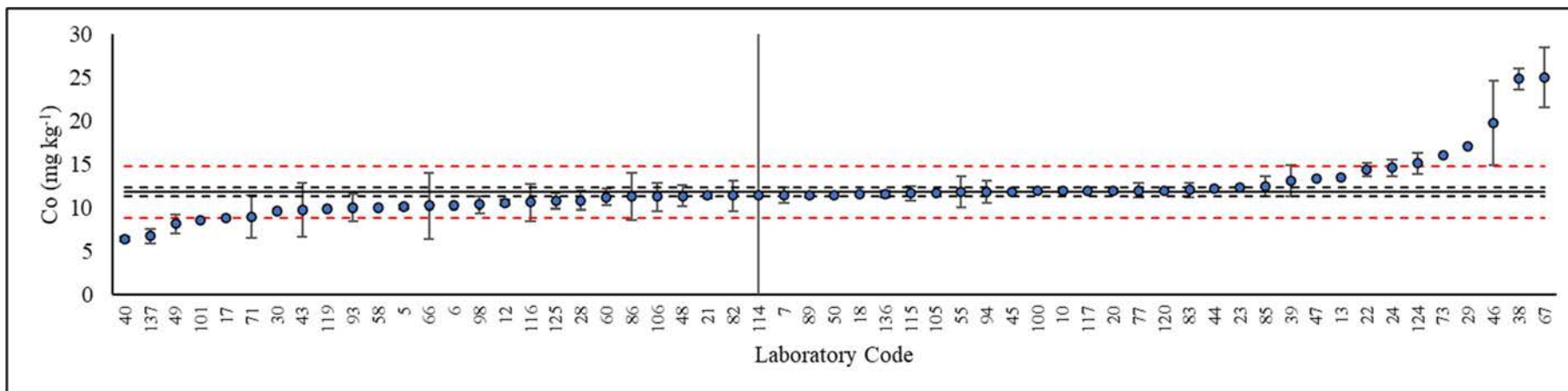


FIG. 11. Laboratory results for Co mass fraction (mg kg^{-1}) in ILC sample.

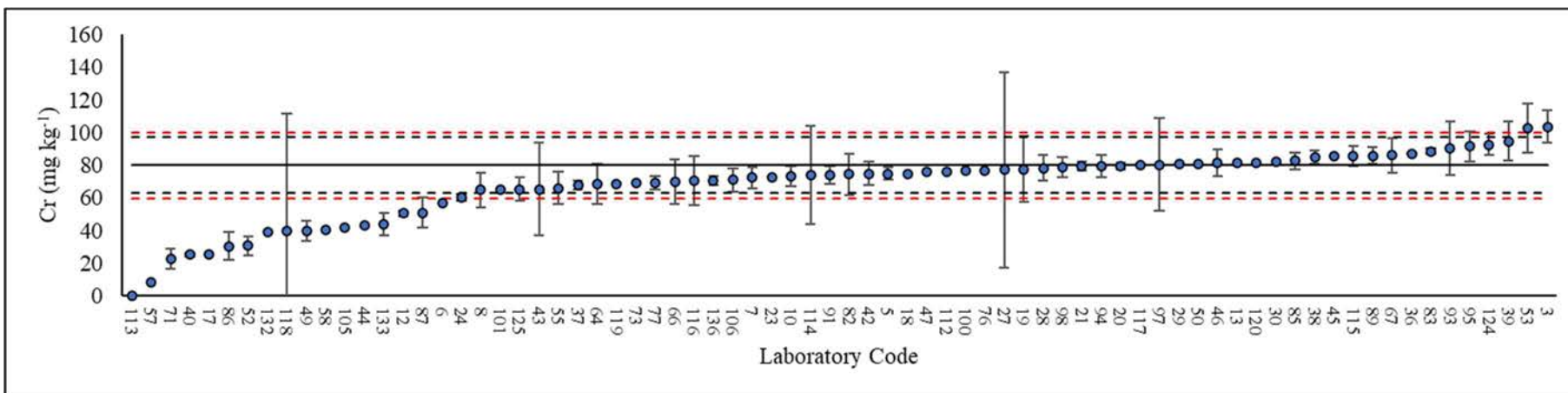


FIG. 12. Laboratory results for Cr mass fraction (mg kg^{-1}) in ILC sample.

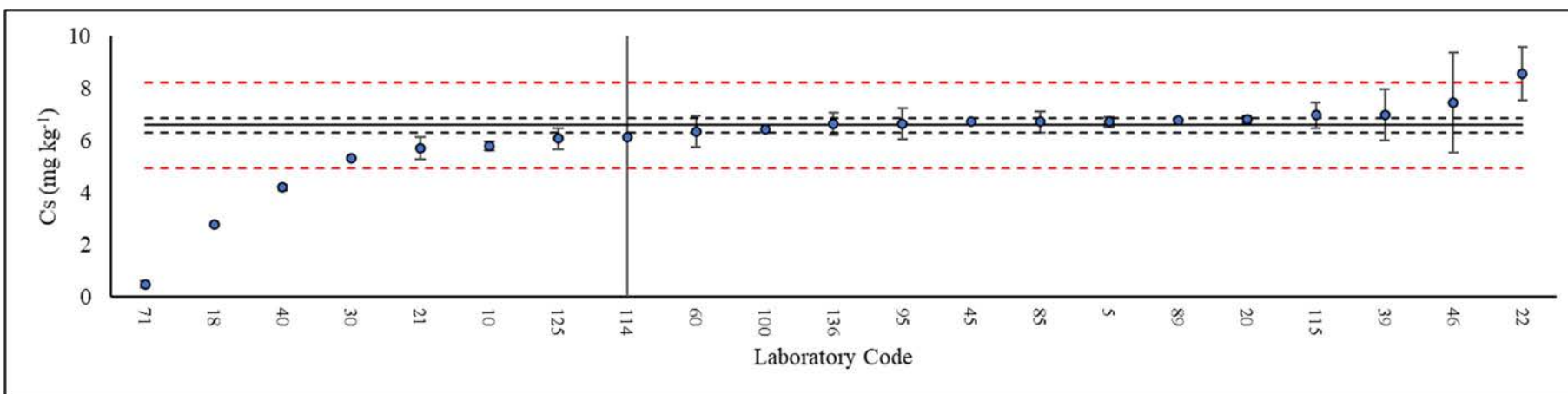


FIG. 13. Laboratory results for Cs mass fraction (mg kg^{-1}) in ILC sample.

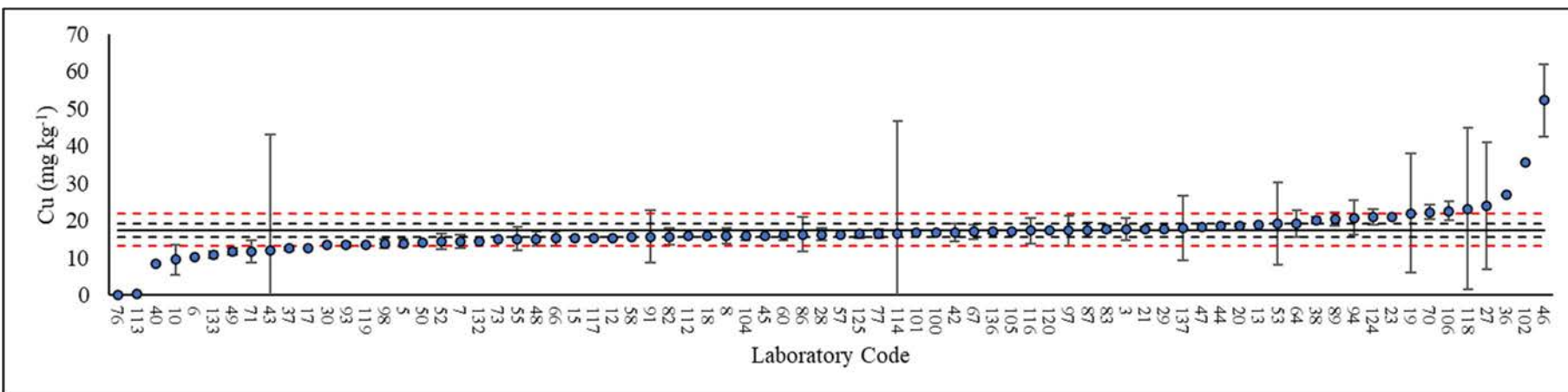


FIG. 14. Laboratory results for Cu mass fraction (mg kg^{-1}) in ILC sample.

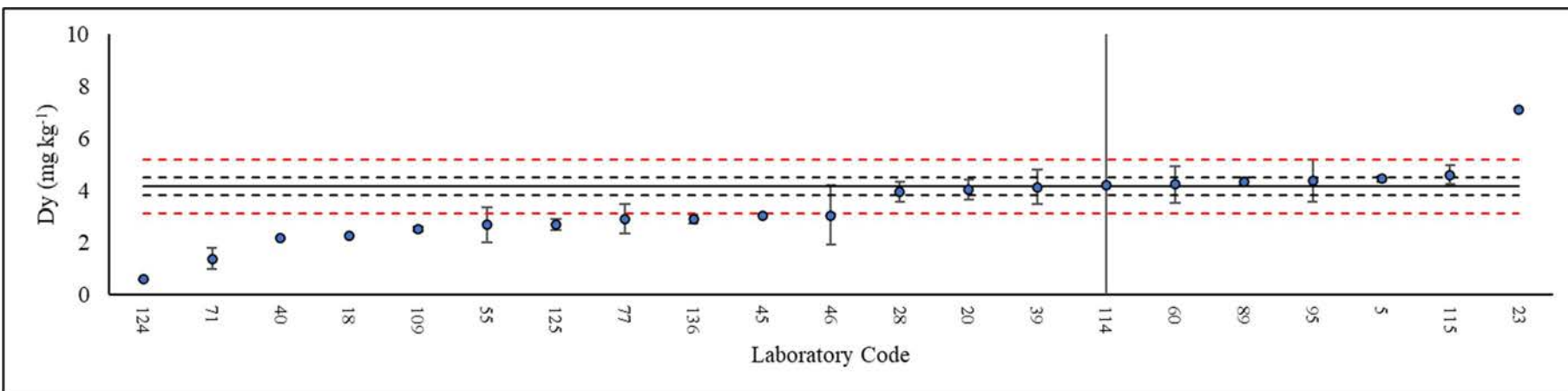


FIG. 15. Laboratory results for Dy mass fraction (mg kg^{-1}) in ILC sample.

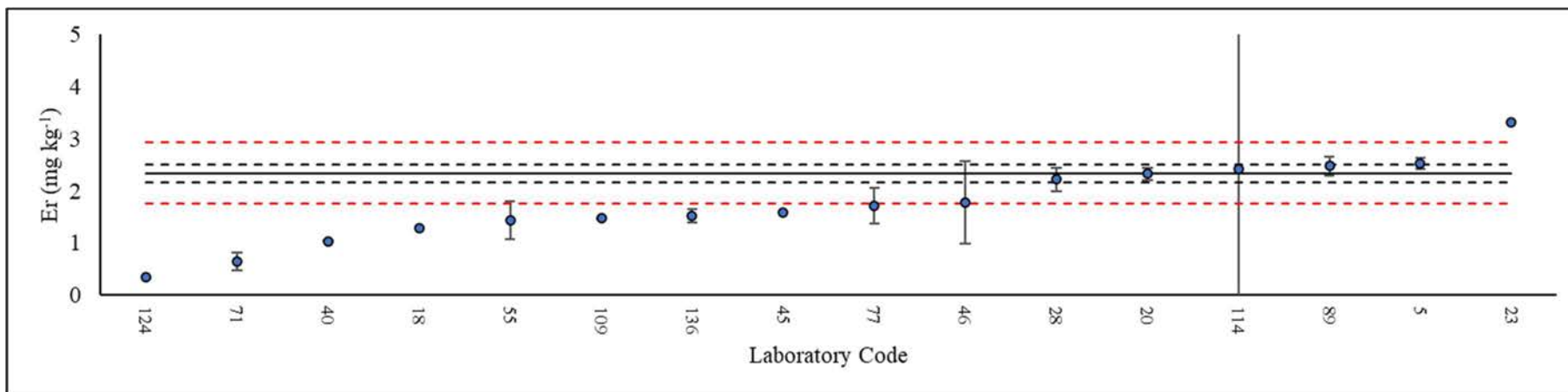


FIG. 16 Laboratory results for Er mass fraction (mg kg^{-1}) in ILC sample.

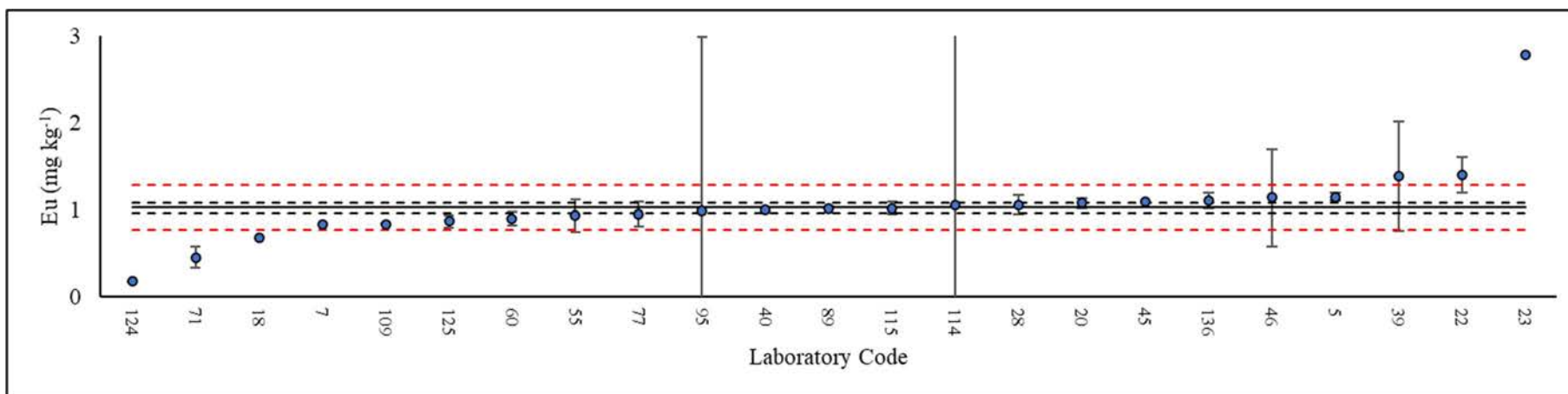


FIG. 17. Laboratory results for Eu mass fraction (mg kg^{-1}) in ILC sample.

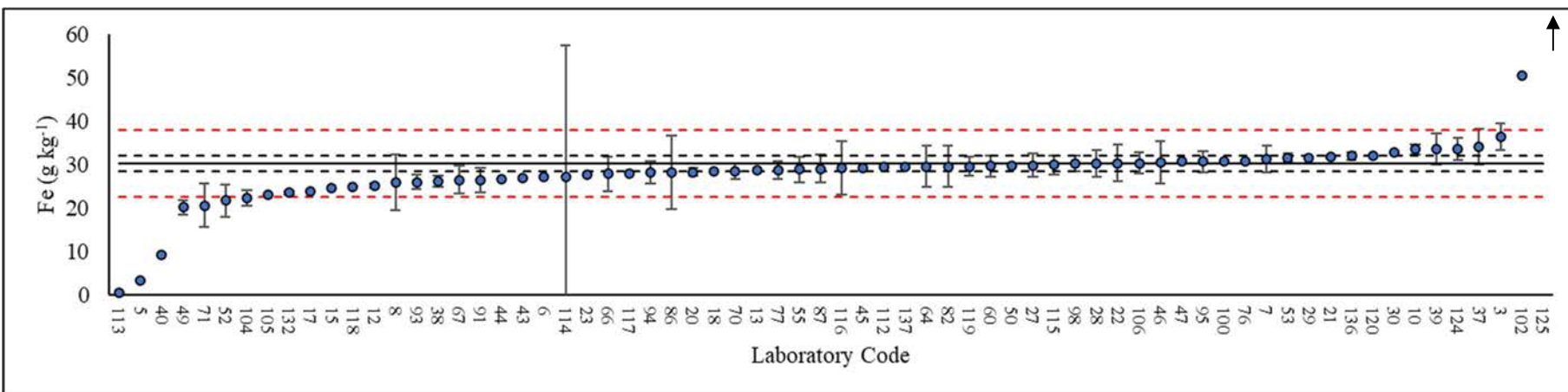


FIG. 18. Laboratory results for Fe mass fraction (g kg^{-1}) in ILC sample.

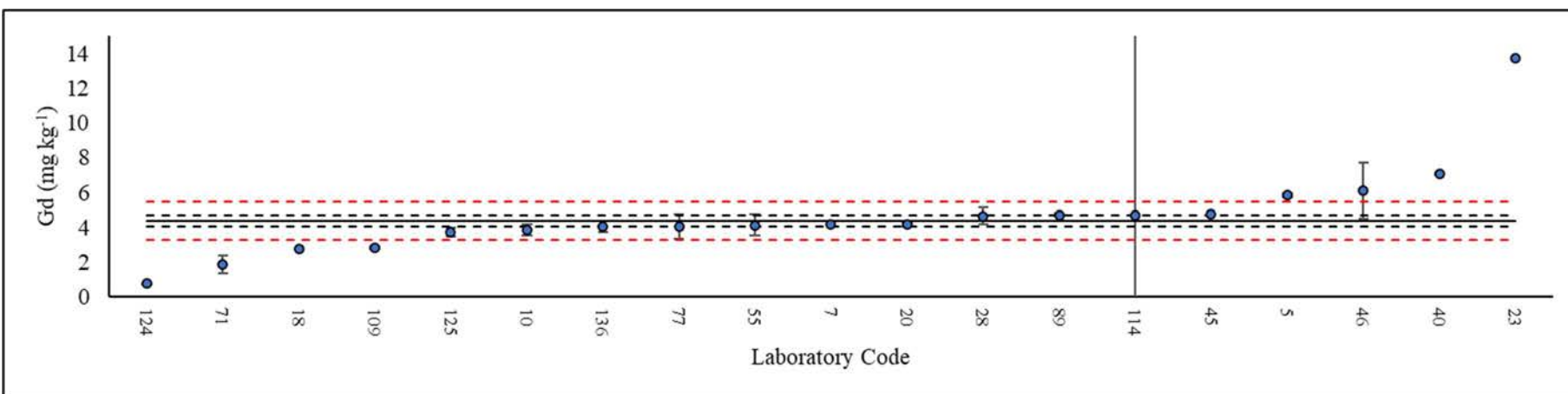


FIG. 19. Laboratory results for Gd mass fraction (mg kg^{-1}) in ILC sample.

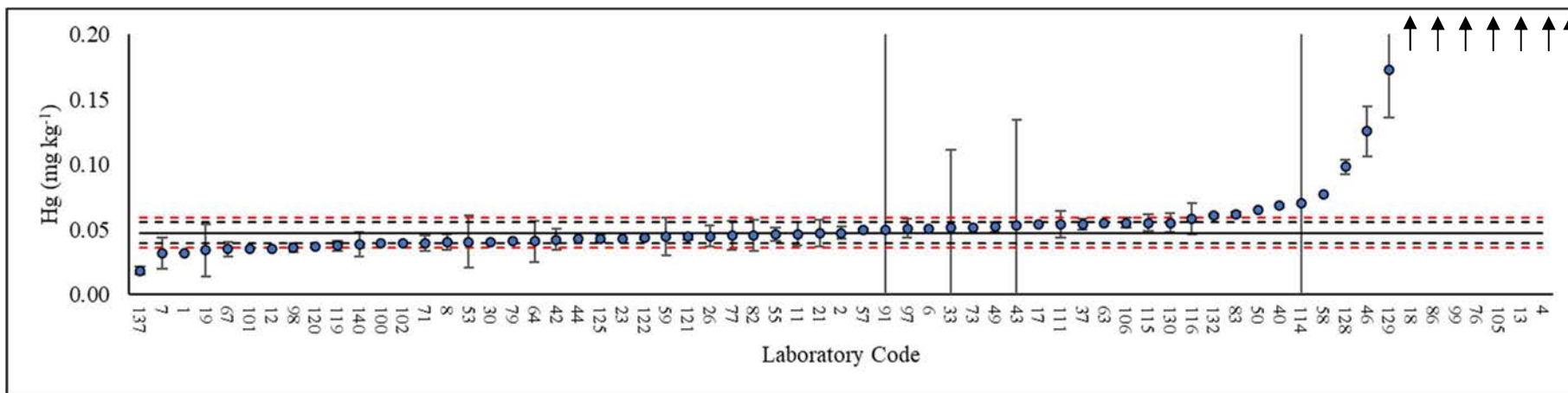


FIG. 20. Laboratory results for Hg mass fraction (mg kg⁻¹) in ILC sample.

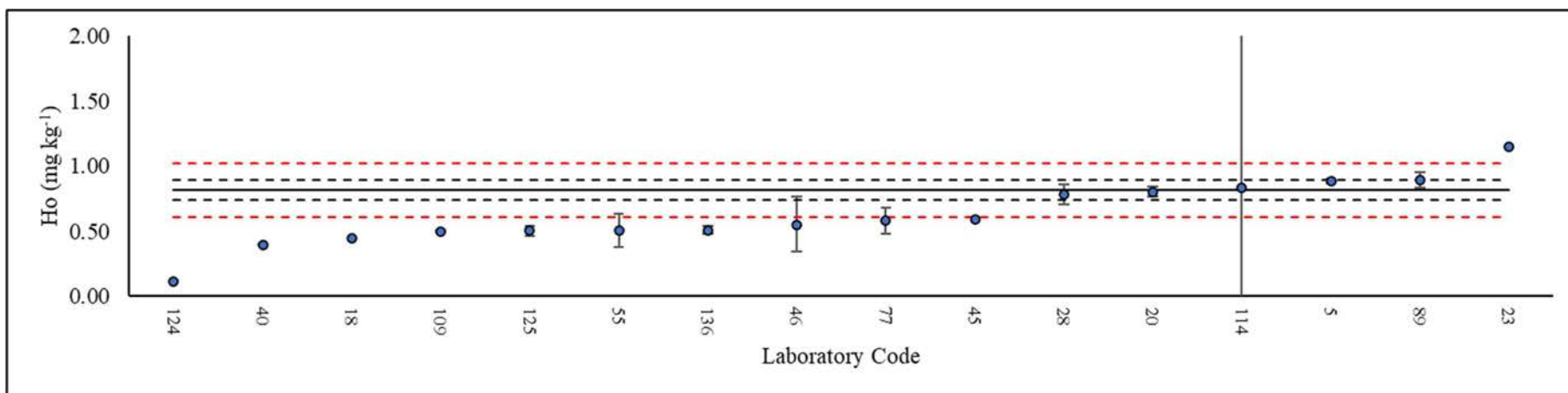


FIG. 21. Laboratory results for Ho mass fraction (mg kg⁻¹) in ILC sample.

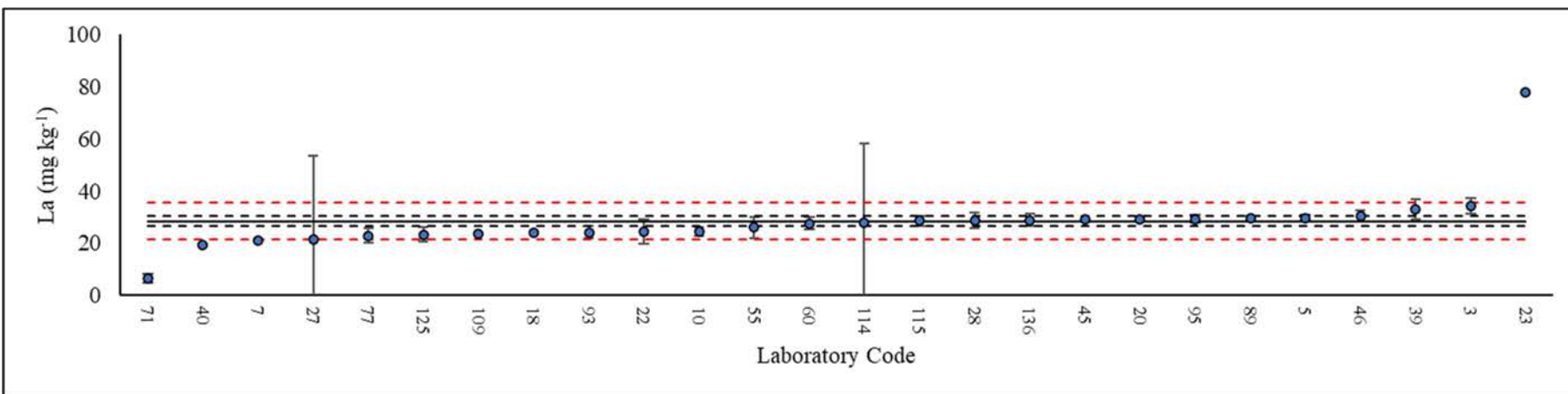


FIG. 22. Laboratory results for La mass fraction (mg kg^{-1}) in ILC sample.

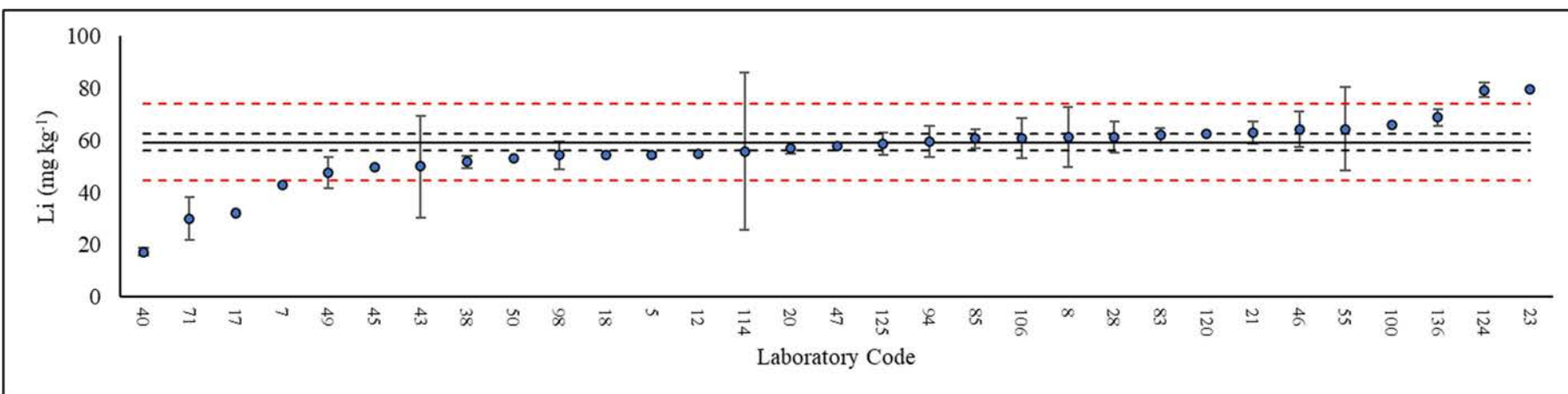


FIG. 23. Laboratory results for Li mass fraction (mg kg^{-1}) in ILC sample.

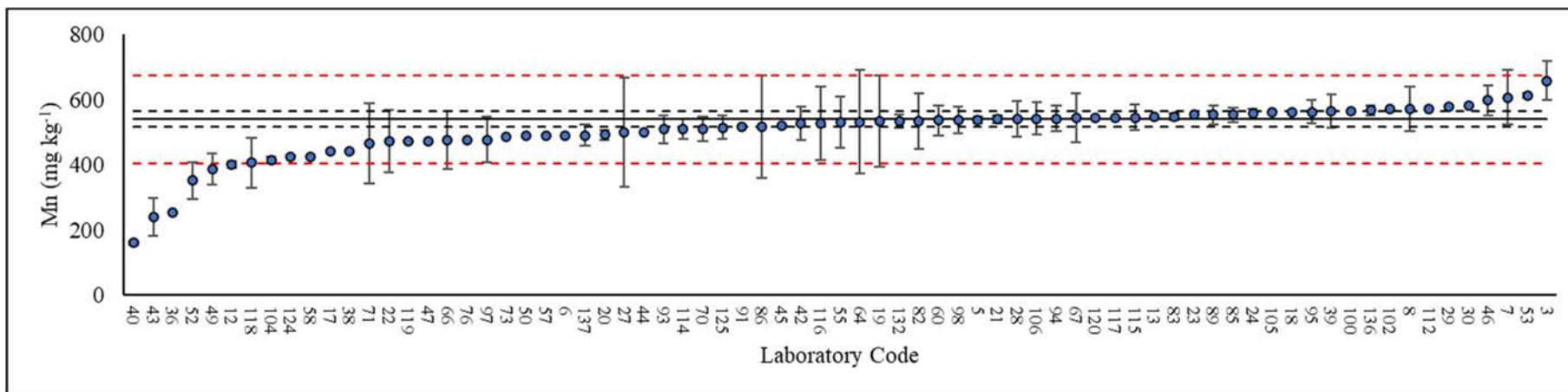


FIG. 24. Laboratory results for Mn mass fraction (mg kg^{-1}) in ILC sample.

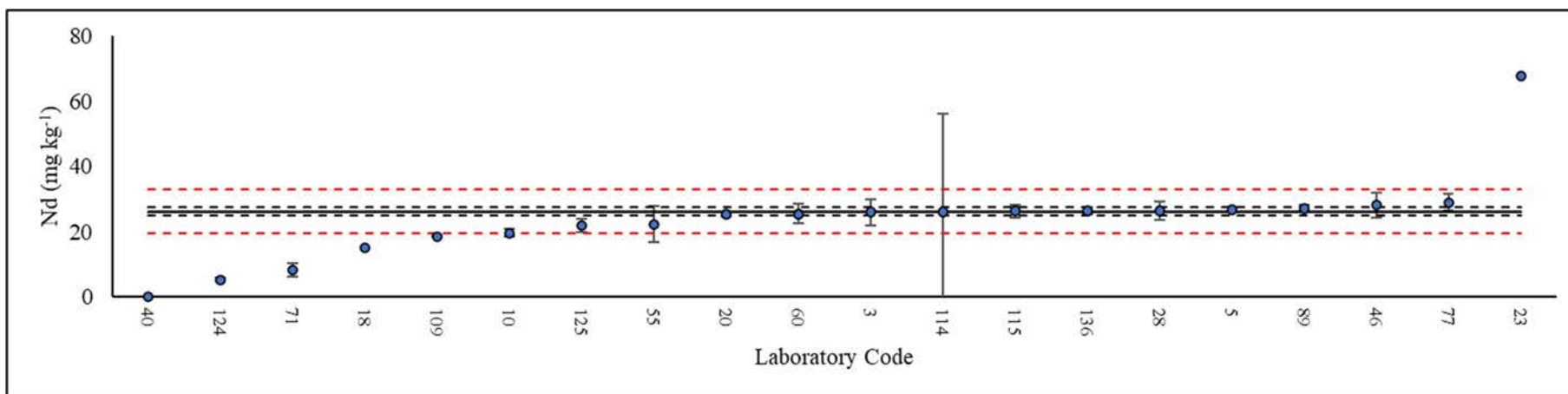


FIG. 25. Laboratory results for Nd mass fraction (mg kg^{-1}) in ILC sample.

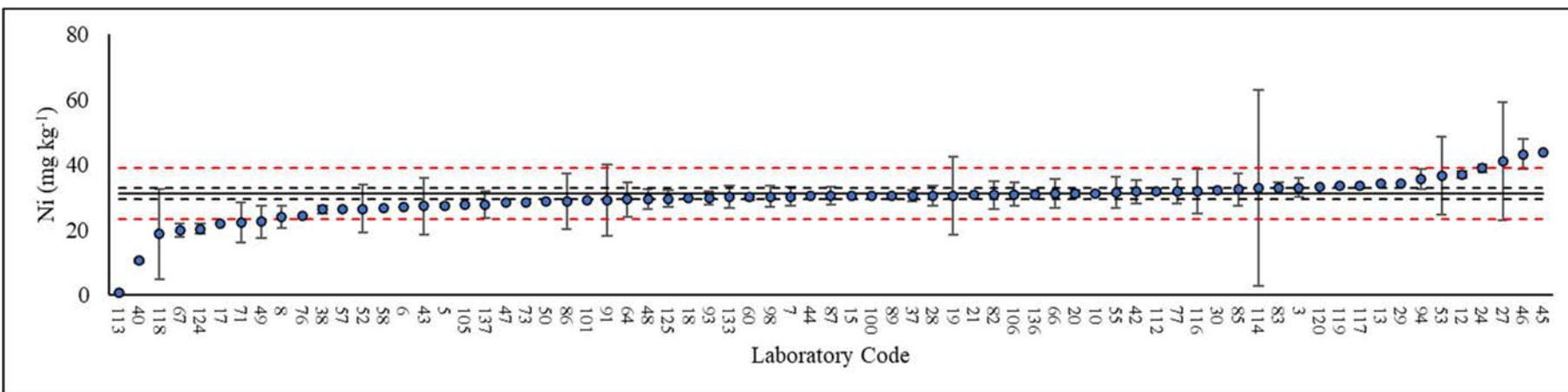


FIG. 26. Laboratory results for Ni mass fraction (mg kg^{-1}) in ILC sample.

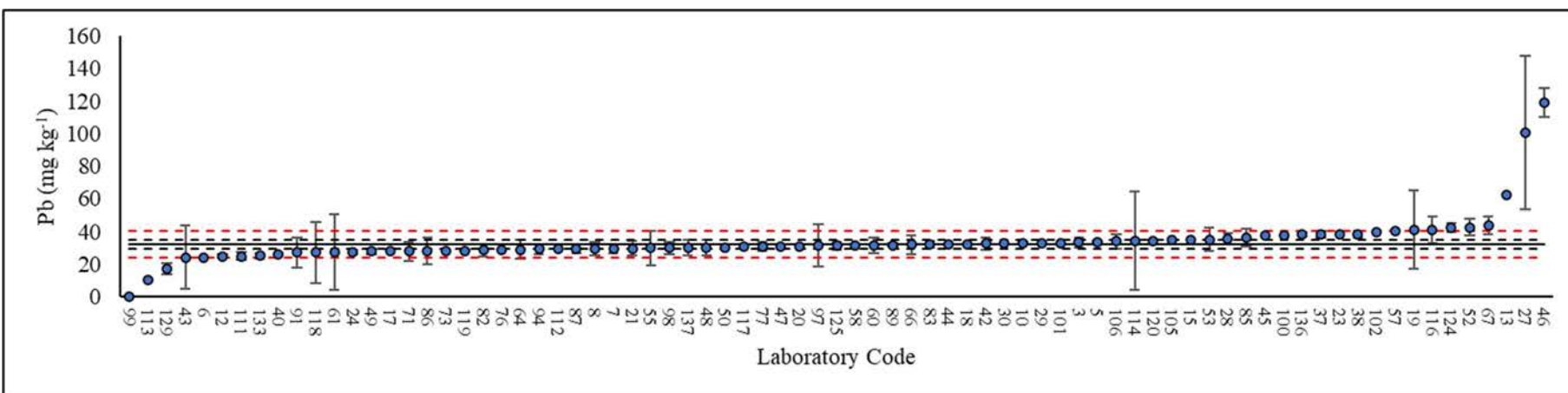


FIG. 27. Laboratory results for Pb mass fraction (mg kg^{-1}) in ILC sample.

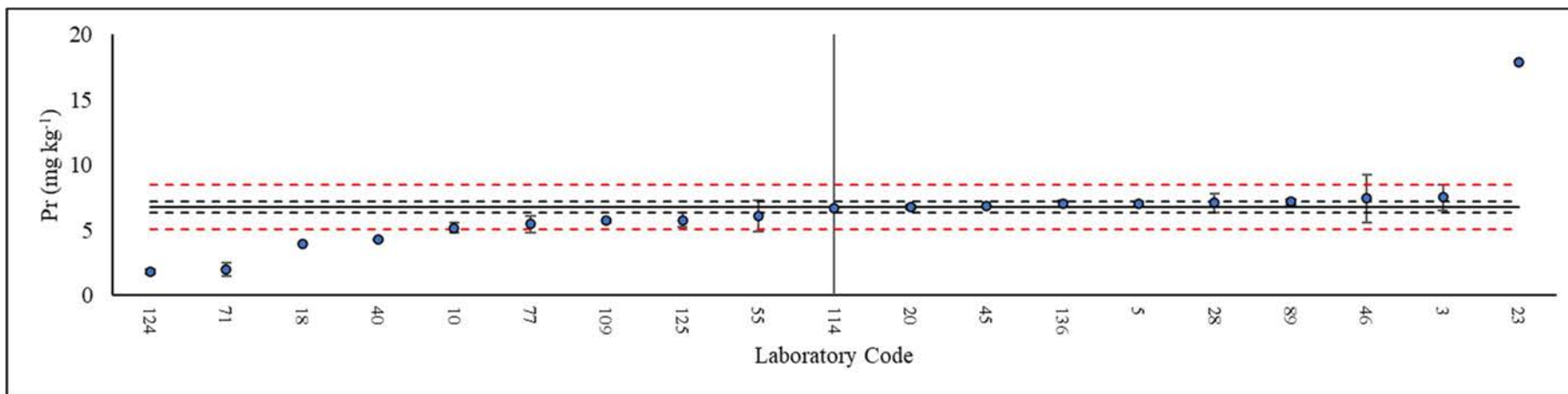


FIG. 28. Laboratory results for praseodymium mass fraction (mg kg^{-1}) in ILC sample.

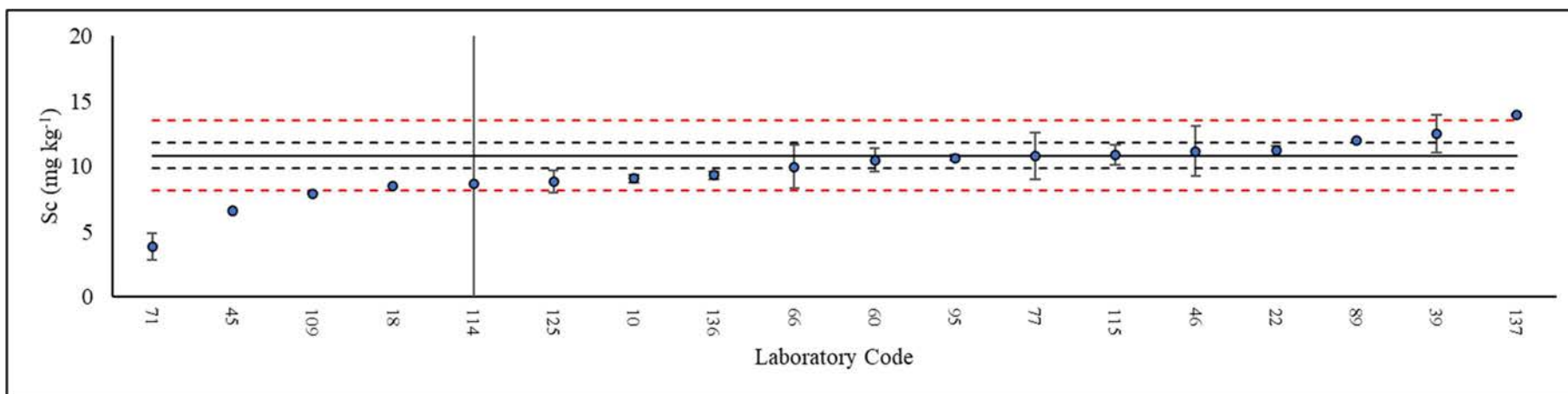


FIG. 29. Laboratory results for Sc mass fraction (mg kg^{-1}) in ILC sample.

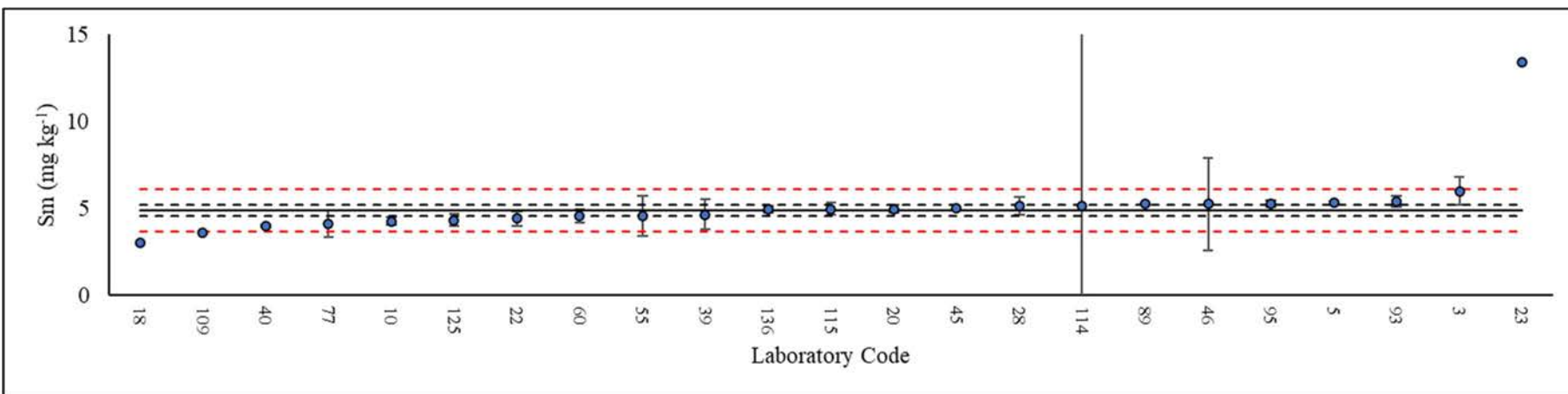


FIG. 30. Laboratory results for Sm mass fraction (mg kg^{-1}) in ILC sample.

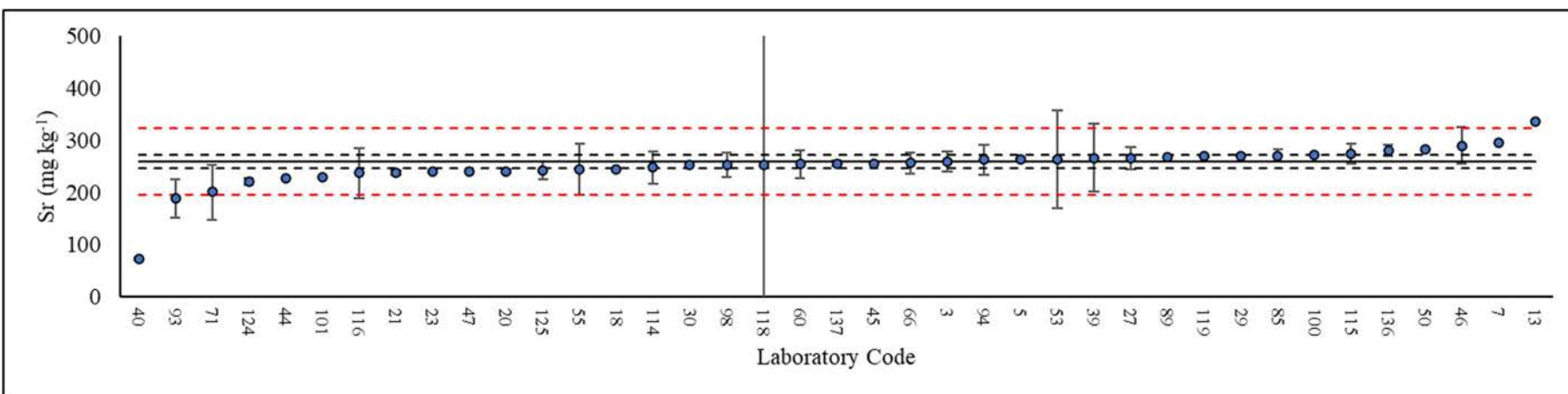


FIG. 31. Laboratory results for Sr mass fraction (mg kg^{-1}) in ILC sample.

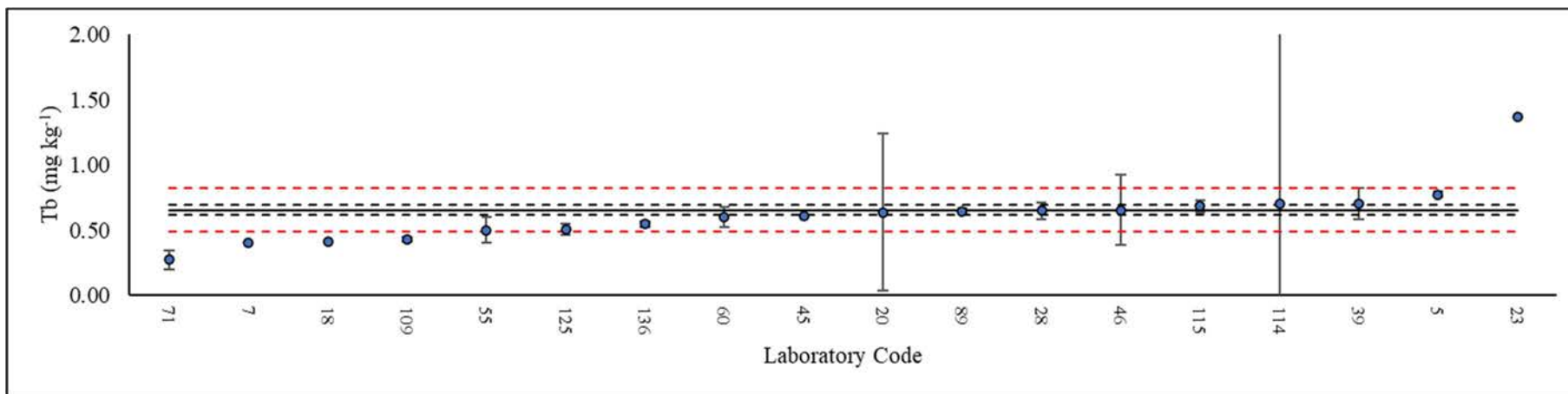


FIG. 32. Laboratory results for Tb mass fraction (mg kg⁻¹) in ILC sample.

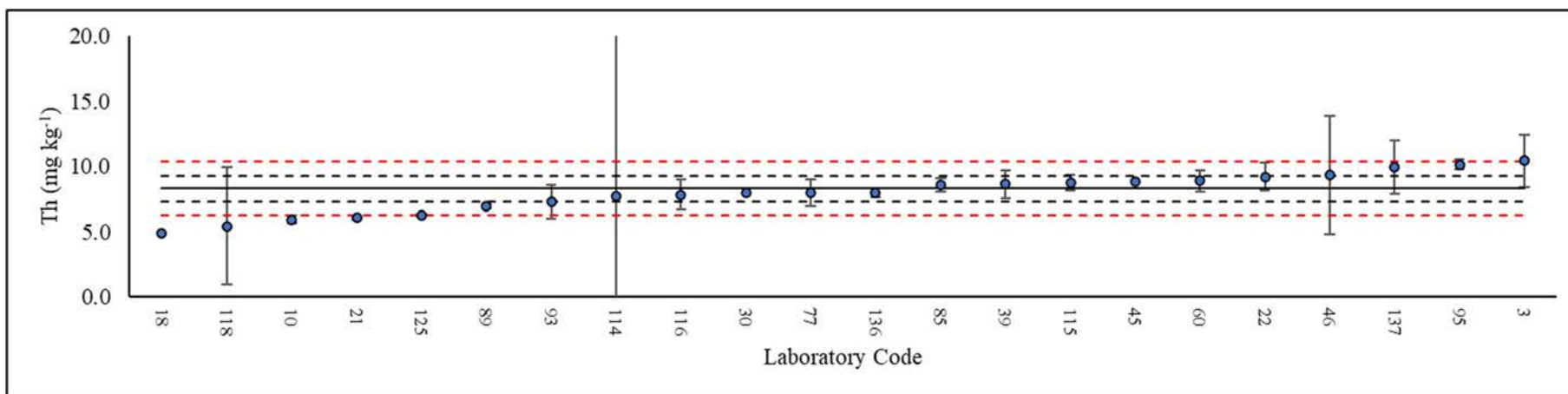


FIG. 33. Laboratory results for Th mass fraction (mg kg⁻¹) in ILC sample.

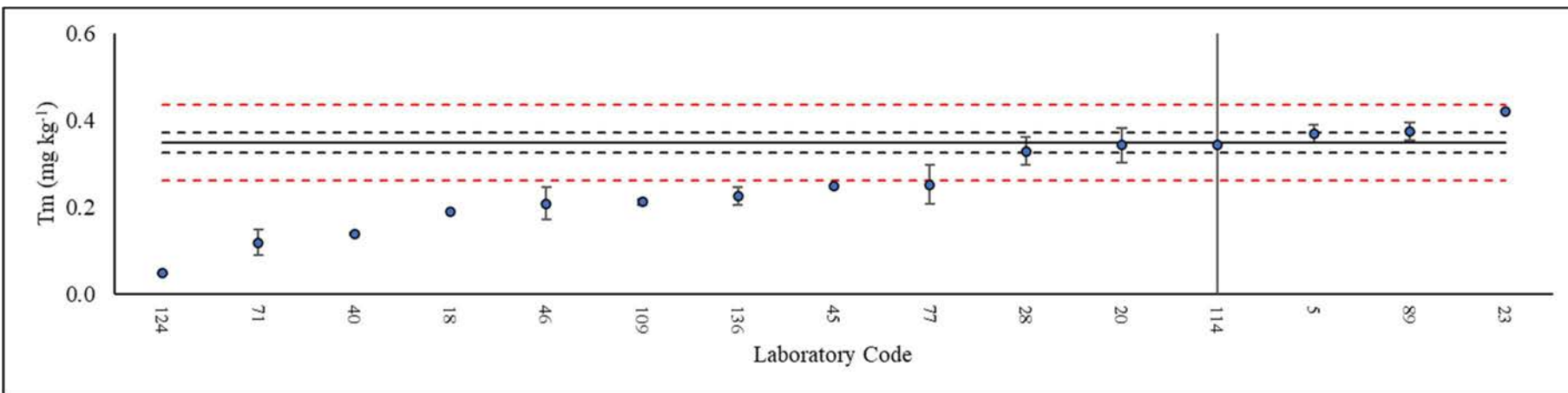


FIG. 34. Laboratory results for Tm mass fraction (mg kg^{-1}) in ILC sample.

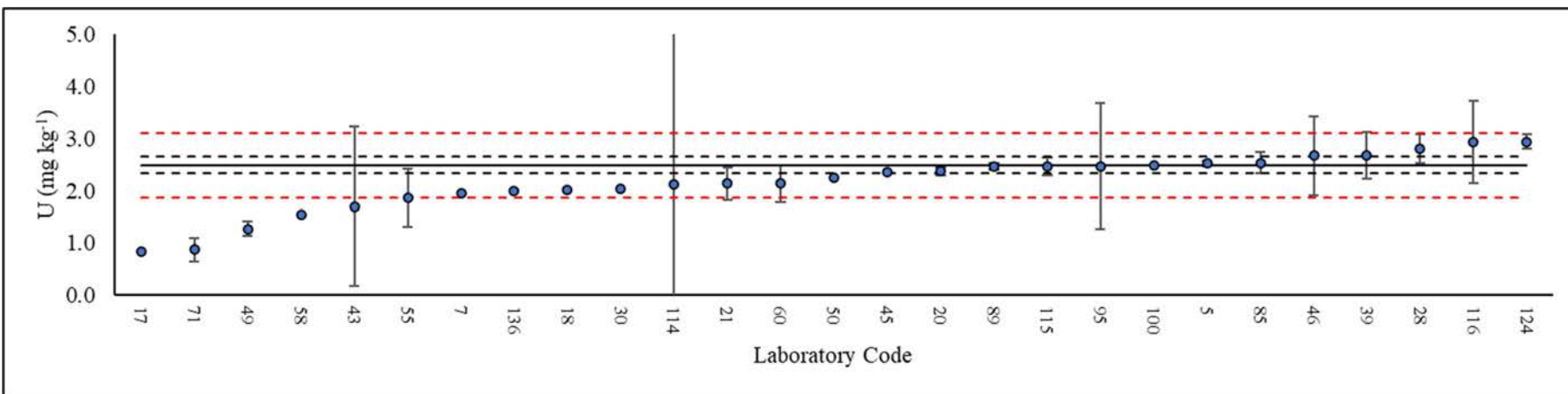


FIG. 35. Laboratory results for U mass fraction (mg kg^{-1}) in ILC sample.

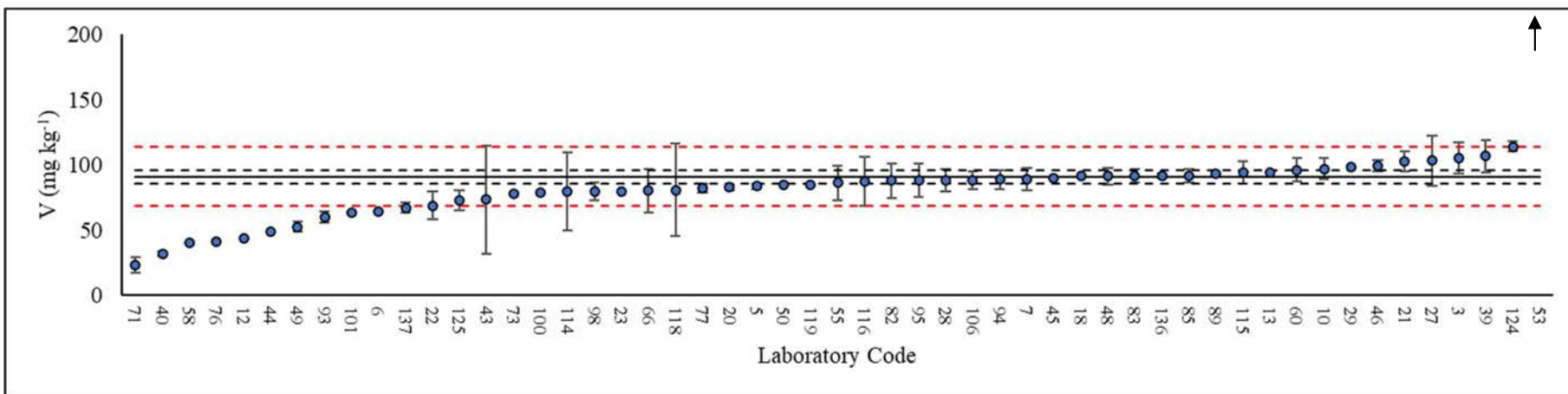


FIG. 36. Laboratory results for V mass fraction (mg kg^{-1}) in ILC sample.

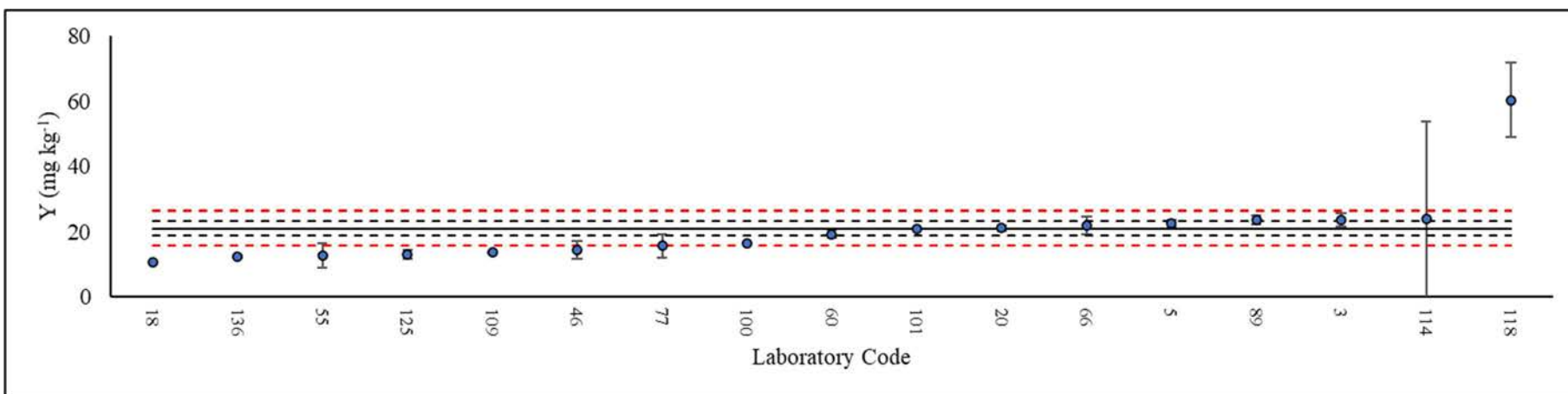


FIG. 37. Laboratory results for Y mass fraction (mg kg^{-1}) in ILC sample.

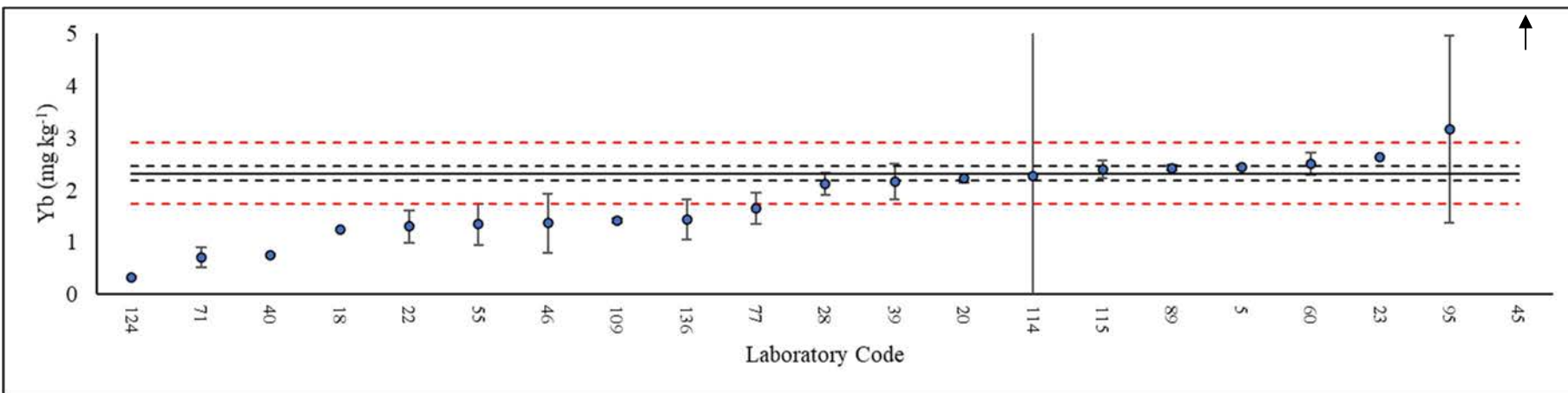


FIG. 38. Laboratory results for Yb mass fraction (mg kg^{-1}) in ILC sample.

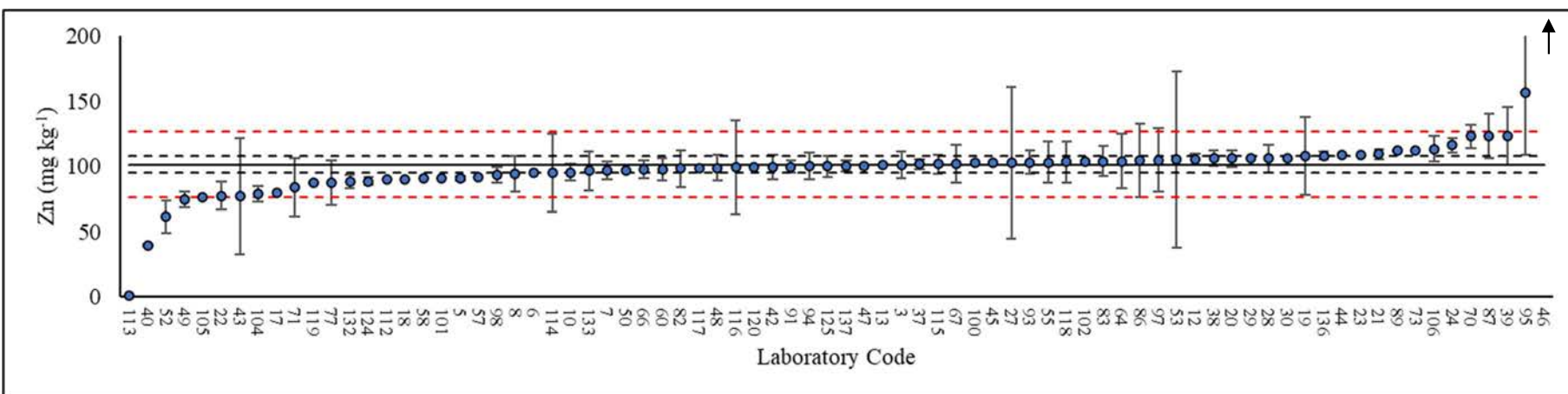


FIG. 39. Laboratory results for Zn mass fraction (mg kg^{-1}) in ILC sample.

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