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JRC VALIDATED METHODS, REFERENCE METHODS AND MEASUREMENTS REPORT

Report of two Inter-laboratory comparisons from the European Reference Laboratory for Food Contact Materials

Elements from Food Contact Materials
ILC 03 2014 – Plastics
ILC 04 2014 – Ceramics

Giorgia Beldì, Mercedes Peltzer and Catherine Simoneau

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Joint Research Centre
Institute for Health and Consumer Protection

Contact information
Catherine Simoneau
Address: Joint Research Centre, Via Enrico Fermi 2749, TP 260, 21027 Ispra (VA) Italy
E-mail: JRC-FCM@ec.europa.eu

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Abstract

This report presents the results of two proficiency tests (PT) organized by the EURL-FCM, which focused on the determination of elements migrated from food contact materials .Two exercises of Interlaboratory Comparisons (ILC) were conducted. One to determine the concentrations of the metals with migration limits assigned for plastics in European Regulation 10/2011 [1] and one to determine the migration of metals relevant to ceramics. The first ILC consisted on the quantification of Ba, Co, Cu, Fe, Mn, Zn, Li and Sb (Sb was undisclosed for participants) in acetic acid solution 3%. The second ILC consisted on the quantification of Ba, Co, Mn, Pb, Cd, Ni, As, and Al in acetic acid solution 4%. The general aim of the exercise was to assess the competence of official control laboratories to measure elements that can potentially migrate from food contact materials. The exercise was a proficiency test and so participants were free to use their own analytical methods. The assigned values were derived from the consensus of the resulted submitted by participants. The robust mean was chosen as the assigned value. The standard deviation for proficiency assessment was set at a value that reflected best practices for the analysis in question, at a maximum of 22 % of the assigned value based on the modified Horwitz equation. Fifty five laboratories (28 NRLs, 26 Official Control Laboratories OCLs and the EURL-FCM) from 27 countries registered to the exercise and fifty four reported results. The outcome of this exercise was satisfactory. The rate of success was always higher than 90% for all elements in both simulants (and 80% for Arsenic as extra element).

Report of two Interlaboratory Comparisons

Elements from Food Contact Materials

EC-JRC-IHCP, CAT Unit

2015

No SANCO/2014/FOOD SAFETY083-Food Contact Materials

Giorgia Beldì, Mercedes Peltzer and Catherine Simoneau

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1. Summary

The Institute for Health and Consumer Protection (IHCP) of the European Commission's Directorate-General Joint Research Centre hosts the EU Reference Laboratory for Food Contact Materials (EURL-FCM). One of its core tasks is to organise inter-laboratory comparisons (ILCs) among appointed National Reference Laboratories (NRLs). This report presents the results of two proficiency tests (PTs) organized by the EURL-FCM, which focused on the determination of elements migrated from food contact materials. The general aim of these two PTs ILC03 2014 and ILC04 2014 was to assess the competence of official control laboratories to measure elements that can potentially migrate from food contact materials.

The first ILC aimed at testing the ability to determine concentrations of metals with migration limits assigned for plastics in the European Regulation (EU) No 10/2011 [1]. It focused on the quantification of Barium (Ba), Cobalt (Co), Copper (Cu), Iron (Fe), Manganese (Mn), Zinc (Zn), Lithium (Li) and Antimony (Sb) from a solution of 3% acetic acid solution as simulant for acidic foods for plastics. The latter one (Sb) was undisclosed.

The second ILC aimed at testing the ability to determine the migration of metals that are relevant to ceramics. The original work programme of this ILC included only Cd and Pb at two levels of concentration in a solution of 4% acetic acid solution which is the conventional simulant for ceramics. However, the progress made in the scoping work on migration carried out by JRC on migration from tableware made possible to consider other elements as being relevant from a release standpoint for ceramics. These elements were amongst others those under discussion for inclusion in future revisions of the ceramics legislation and were in also metals considered in the Resolution of the Council of Europe (CoE) on metals and alloys [2]. The scope of the exercise was thus extended to the quantification of Barium (Ba), Cobalt (Co), Manganese (Mn), Lead (Pb), Cadmium (Cd), Nickel (Ni), Arsenic (As) and Aluminium (Al) in a 4% acetic acid solution at one level of concentration. The concentration values were chosen in agreement with DG SANTE as a compromise between the values as discussion starting values (DSVs) proposed by DG-SANTE and the values adopted by the Council of Europe (CoE) in the context of metals and alloys.

The test materials were prepared by the EURL-FCM. They consisted in a spiked 3% acetic acid solution as well as an unspiked one (for ILC 03 on plastics), and a spiked 4% acetic acid solution as well as an unspiked one (for ILC04 on ceramics). The homogeneity and stability studies were performed by the EURL-FCM. Each participant received four samples of approximately 40 mL. The exercise was a proficiency test and so participants were free to use their own analytical methods. Participants were asked to analyse the analytes in the migration solutions and report four replicate results for each element. Fifty five laboratories (28 NRLs, 26 Official Control Laboratories (OCLs) and the EURL-FCM) from 27 countries registered for the exercise and fifty four reported results. The assigned values were derived from the consensus of the results submitted by participants. The robust mean was chosen as the assigned value. The standard deviation for proficiency assessment σ_p was set at a value that reflected best practices for the analysis in question. It was set at a maximum of 22% of the assigned value based on the modified Horwitz equation. Laboratories were rated with z-scores in accordance with ISO 13528 [3].

The outcome of this exercise was satisfactory. The rate of success was higher than 90% for all elements in both simulants. For Arsenic the satisfactory scores were of 80% due to the presence of few overestimated values very likely due to a blank contamination. Results overall showed a satisfactory laboratory performance.

2. Introduction

Inter-laboratory comparisons (ILCs) are an essential element of laboratory quality assurance, which allow individual laboratories to compare their analytical results with those from other laboratories while providing them objective standards to perform against. In this context, it is one of the core duties of the European Reference Laboratories to organise inter-laboratory comparisons, as is stated in Regulation (EC) No 882/2004 of the European Parliament and of the Council [4]. In accordance with the above requirements the European Reference Laboratory for Food Contact Materials (EURL-FCM) organised inter-laboratory comparison tests for the network of appointed National Reference Laboratories (NRLs) in 2014. Two ILC exercises focused on the determination of elements that can potentially migrate from food contact materials.

The first exercise consisted in the quantification of the 7 metals subjected to specific migration limits (SMLs) assigned for **plastics** materials and articles in Regulation (EU) 10/2011 [1], Annex II, as reported in table 1.

Table 1. Specific Migration Limits laid down in Regulation 10/2011

Metal	SML [mg/Kg]
Ba	1
Co	0.05
Cu	5
Li	48
Mn	0.6
Zn	25

The migration solution consisted of the simulant for acidic foods which is the worst case for migration of elements. Laboratories received a solution of acetic acid 3% solution (w/v) fortified with the metals as well as a blank sample (solution alone).
The exercise also included as additional task the identification and quantification of one undisclosed element. The undisclosed element was antimony (Sb).

For **ceramics** limits have been in place only for lead (Pb) and cadmium (Cd), in Directive 84/500/EEC [5]. Ceramic articles are recognised as satisfying the requirements of this Directive if the quantities of lead and/or cadmium extracted during the test carried out in a 4% acetic acid solution do not exceed the limits reported in table 2.

Table 2. Limits laid down in directive 84/500/EEC for Lead and Cadmium

Category	Category Description	Limits Pb	Limits Cd
1	Articles which cannot be filled and articles which can be filled, the Internal depth of which, measured from the lowest point to the horizontal plane passing through the upper rim, does not exceed 25 mm	0,8 mg/dm^2	0,07 mg/dm^2
2	All other articles which can be filled	4,0 mg/L	0,3 mg/L
3	Cooking ware; packaging and storage vessels having a capacity of more than three litres.	1,5 mg/L	0,1 mg/L

Investigations have also shown other metals can migrate into foods, which could pose concerns and should be considered by EU legislation. Scientific data have also shown the need to lower the current limits. Consequently the European Commission (EC) is considering to revise the Directive 84/500/EEC [5] related to ceramic articles intended to come into contact with foodstuffs. In this context, the EURL-FCM organised a dedicated ILC to evaluate the laboratory performance of the appointed NRLs and OCLs laboratories to determine the concentration Pb, Cd at level much lower than those laid down in the ceramic directive 84/500/EEC as well as the concentration of other metals that can possibly migrate from ceramics to estimate the enforceability of potential future measures.

This exercise focused on the quantification of 8 elements relevant to ceramics, i.e. Ba, Co, Mn, Pb, Cd, Ni, As, and Al. The concentration values were chosen as a compromise between the values as discussion starting values (DSVs*) proposed by DG-SANTE and the values adopted by the Council of Europe (CoE) in the context of metals and alloys [2]. The values are reported in table 3.

Table 3. Metals Limits

Metal	CoE Metals june 2011//(2013) [µg/Kg]	DSV* [µg/Kg]
Cd	5	5
Pb	10	10
Al	900//(5000)	1000
As	2	18
Ba	1200	1000
Co	10//(20)	84
Mn	900//(1800)	400
Ni	70//(140)	72

* European Commission Discussion Starting Values: These values are meant to facilitate discussion, and are not intended as a proposal for SML. None of these values should be taken up in legislation without an EFSA opinion fit for this purpose as is set out in the Regulation (EU) No1935/2004. It is not necessarily the intention to add all these metals to the future legislation.

3. Scope

The objectives of these ILCs were:

1. To test the competence of the appointed NRLs and OCLs to determine the concentration of Ba, Co, Cu, Fe, Mn, Zn, Li and Sb with migration limits assigned for plastics in Regulation 10/2011.

2. To assess the laboratory performance of the appointed NRLs and OCLs laboratories to determine the concentration of Ba, Co, Mn, Pb, Cd, Ni, As, and Al potentially migrating from ceramics. The concentration values were chosen considering DSVs proposed for evaluation by DG SANTE and limits estimated for metals and alloys under the Council of Europe (CoE).

A follow up work is foreseen to derive precision criteria for ceramics towards support to the work of ISO TC 166.

4. Time frame

The ILCs were launched in May 2014. Invitation letters with instructions were sent by e-mail to NRLs and OCLs (Annex 1). Laboratories were invited to fill in a letter of confirmation of their participation (Annex 2). The samples were prepared and tested for homogeneity in June 2014 by EURL-FCM. The samples were dispatched to the participants on the 16th of June 2014, together with the shipping kit (Annex 3). The participants were asked to confirm the sample receipt and fill in the respective letter of confirmation (Annex 4) and use the electronic excel file for reporting the results (Annex 5). The deadline to report the results was set to the 21st of July 2014.

5. Test materials

A solution of acetic acid 3 % (w/v) spiked with Ba, Co, Cu, Fe, Mn, Zn, Li and Sb (undisclosed) and solution of acetic acid 4 % (v/v) spiked with Ba, Co, Mn, Pb, Cd, Ni, As, and Al were prepared by the EURL-FCM. After spiking and homogenization, 40 mL portions of the spiked simulants were subsampled. The sample kit sent to the participants is shown in Table 4. Unfortified food simulants (acetic acid 3% and 4%), from the same batch as the fortified one, were also sent to the participants as blanks. The participants were asked to correct their results for the blanks.

Table 4. Sample kit

Name	Sample
PL1	Approximately 40 mL of acetic acid 3 % (w/v) spiked with Barium, Cobalt, Copper, Iron, Manganese, Zinc, Lithium plus Antimony as unknown element.
PL1- Blank	Approximately 40 mL of acetic acid 3 % (w/v) of the same batch used to prepare the spiked solution PL1.
C2	Approximately 40 mL of acetic acid 4 % (v/v) spiked with Barium, Cobalt, Manganese, Lead, Cadmium, Nickel, Arsenic and Aluminum.
C2-Blank	Approximately 40 mL of acetic acid 4 % (v/v) of the same batch used to prepare the spiked solution C2.

5.1 Homogeneity assessment

The samples were tested for homogeneity by the EURL-FCM. Ten randomly selected test specimens for the sample PL1 and C2 were analysed in duplicate. Homogeneity was evaluated according to 13528:2005(E) Annex B [3] and IUPAC International

Harmonized Protocol [7] using the ProLab Software [8]. The homogeneity results and their statistical evaluation were obtained using truncated (modified) Horwitz as target standard deviation (Annex 6a, 6b). All test materials showed sufficient homogeneity and thus were suitable for the exercise.

5.2. Stability test and pH control

Randomly selected specimens for sample PL1 and C2 were stored at 3 different temperature conditions (4ºC, 20°C and 40ºC). The test samples were monitored for stability by the EURL-FCM for approximately 60 days to cover the period allowed for the exercise. The stability was evaluated as described in ISO GUIDE 35:2006 [9].

The evaluation of data was carried out by performing a linear regression on the experimentally measured concentrations of the elements (mean values) versus time (days). For a stable material, the intercept is (within uncertainty) should be equal to the assigned value, whereas the slope should not differ significantly from zero.

Using the linear regression equation:
$Y_{(\text{metal } \mu g/Kg)} = b_0 + b_1 X_{(\text{time, days})}$

The slope is not significantly different from zero if the following requirement is respected:

$$|b_1| < t_{0,95,n-2} \cdot s(b_1)$$

Where

b1	is the slope obtained from the linear regression,
$t_{0.95,n-2}$	is the Student's t-factor for n-2 degrees of freedom and p = 0.95 (95% level of confidence) and
$s(b_1)$	is the uncertainty associated with the slope.

This can be calculated as follows:

$$s(b_1) = \frac{s}{\sqrt{\sum_{i=1}^{n}\left(X_i - \bar{X}\right)^2}}$$

The value of s (standard deviation of the time-points) can be obtained from:

$$s^2 = \frac{\sum_{i=1}^{n}\left(Y_i - b_0 - b_1 X_i\right)^2}{n-2}$$

Where n is the number of points of the linear regression.

The stability results and their statistical evaluation are given in Annex 7a, 7b. No significant trend was observed for the test sample at any temperature conditions (4ºC, 20°C and 40ºC) for 55 days. It was concluded that the substances were stable for at least two months following their preparation.

In addition, randomly selected specimens for PL1 and C2 were stored at room temperature and monitored for the pH values across the time period of the exercise. This control was performed to assess the pH stability and possible correlation between pH values and relative stability in the concentrations of metals in the solution over time. T The pH values for samples PL1 and C2 are reported in Annex 8. The pH values for both solutions PL1 and C2 were constant and no significant trend was observed for the test sample over approximately 60 days.

6. Instructions to participants and requested measured parameters

Instructions were provided to all participants in letters accompanying the samples (Annex 3). Laboratories were asked to analyse samples PL1 and C2 in four replicates, correct the results for the blank value and report 4 results for each element using the unit of measure indicated in the electronic excel file for reporting the results (Annex 5). Participants were free to use their own analytical methods.

7. Statistical evaluation of results

7.1. General observations

Fifty five laboratories (28 NRLs, 26 OCLs and the EURL-FCM) from 27 countries registered to the exercise and fifty four reported results. The assessment of the measurement results was undertaken on the basis of requirements laid down in international standards and guidelines [3,6].

7.2. Statistical evaluation of results

The statistical evaluation of the results was performed using the ProLab software [8].

7.2.1. Assigned value and its uncertainty

The true values for the concentration of the elements in the samples were unknown. As there were no other reference values available, the robust mean values obtained from the reported results of the participants were used as assigned values.

The robust mean values were obtained using the Hampel estimator, as described in ISO/TS 20612 [6]. The Hampel estimator is a tool of robust statistics to obtain reference values from the results of the participants of an inter-laboratory comparison test [6]. It remains viable even with more than 40% outlier laboratories [8]. It should be noted that no tests for outliers are carried out when the Hampel estimator is used. The algorithm works in a way that values, which differ from the mean value by more than 4.5 times the standard deviation, do not affect the calculated results [6]. The results reported as "smaller than" (<values) were not used in any calculation. No

evaluation of these measurement results was done and no scores were given.

When the assigned value is derived as a robust average, the standard uncertainty of the assigned value is estimated as:

$$u_X = \frac{1.25 \cdot \sigma_p}{\sqrt{p}}$$

where σ_p is the robust standard deviation and p is the number of laboratories. The factor 1.25 represents the ratio of the standard deviation of the median to the standard deviation of the arithmetic mean, for large samples p>10.

If

$$u_X \leq 0.3 \cdot \sigma_p$$

then the uncertainty of assigned value is negligible and need not be included into interpretation of the results of the proficiency test.

7.2.2. Target standard deviation

The value of target standard deviation (σ_p) determines the limits of satisfactory performance in an ILC test. It should be set as a value that reflects best practice for the analysis in question. The standard deviation of the reproducibility found in collaborative trials is generally considered as an appropriate indicator of the best agreement that can be obtained between laboratories. The 2014 ILCs on elements from migration solutions were the first exercises of this kind for food contact materials. Hence, there were no comparative test data available in the specific sector. In the absence of appropriate collaborative trial data, σ_p could be derived from the appropriate form of the Horwitz equation [10].

The target standard deviations for proficiency assessment σ_p were calculated applying the modified Horwitz equation for all elements and was set at a maximum of 22 % of the respective assigned value.

7.2.3. Kernel density

Kernel density plots were used additionally to identify multi-modality in the reported values' distributions. Frequently analytical results from a collaborative study are not normally distributed or contain values from different populations giving rise to multiple distribution modes. These modes can be visualised by using Kernel density plots [11, 12]. Kernel density plots were computed by the ProLab software from the analytical results by representing the individual numerical values as a normalised Gaussian distribution centred on the respective analytical value. The sum of these normal distributions forms then the Kernel density distribution.

7.2.4. z-score

Individual laboratory performance was expressed in terms of z-score in accordance with ISO 13528 [3] and the International Harmonised Protocol [7]. The z-scores

describe the deviation of the individual laboratory result to the assigned value, standardised by the target standard deviation accepted for the PT:
The calculation of z-scores (z) is done as follows:

$$z = \frac{(x_{lab} - X_{assigned})}{\sigma_p},$$

where:

x_{lab} is the measurement result reported by a participant;

$X_{assigned}$ is the assigned value;

σ_p is the target standard deviation for proficiency assessment .

The z-score can be interpreted as follow:

|z|≤2 satisfactory result;
2<|z|≤3 questionable result;
|z|>3 unsatisfactory result.

8. Results and Conclusions

8.1. Participation

Samples were dispatched to 55 laboratories (28 NRLs and 26 OCLs and EURL), 54 of them submitted results, corresponding to a percentage of participation of 98%.

The percentage of participation considering individual elements is reported in table 5.

Table 5. Percentage of participation for individual elements.

% Participation : Elements to be quantified in Acetic acid 3 % (w/v) - PL1			
Barium	44 results / 55 participant = 80.0%	Manganese	50 results / 55 participant = 90.9%
Cobalt	48 results / 55 participant = 87.3%	Zinc	49 results / 55 participant = 89.1%
Copper	51 results / 55 participant = 92.7%	Lithium	40 results / 55 participant = 72.7%
Iron	48 results / 55 participant = 87.3%	Antimony	33 results / 55 participant = 60.0%
% Participation : Elements to be quantified in Acetic acid 4 % (v/v) - C2			
Barium	42 results / 55 participant = 76.4%	Cadmium	51 results / 55 participant = 92.7%
Cobalt	46 results / 55 participant = 83.6%	Nickel	49 results / 55 participant = 89.1%
Manganese	49 results / 55 participant = 89.1%	Arsenic	44 results / 55 participant = 80%
Lead	50 results / 55 participant = 90.9%	Aluminium	43 results / 55 participant = 78.2%

As requested, most of the laboratories reported four replicate results under repeatability conditions.

The techniques used for the determination of elements by the laboratories for this exercise were varied (e.g. ICP-MS, ICP-OES, AAS), yet with an overwhelming majority of the results obtained by ICP-MS.

8.2. Laboratories performance and z-scores

A summary of the statistical data obtained for samples PL1 and C2 is given in table 6. The summary results reported by the laboratories, the Kernel density plots and the obtained z-scores are shown in figures (1-16).

The outcome of this exercise was satisfactory. The rate of success was always higher than 90% for all elements in both samples apart from 80% for Aresenic. The rate of success for individual elements/samples is reported in table 7.

The tabulated values for the z-score are reported in the tables 8, 9.

The Kernel density plots for the samples PL1 and C2 showed one major mode for each element with 76-94% probability, indicating homogeneous data populations. Using the Hampel estimator, a robust mean value was used as assigned value. The tolerance limits for each element are reported in table 6. Consequently, all those laboratories that reported a value below ($|z_U| \leq 2$) did not receive satisfactory results.

The results of the robust statistics indicated a good reproducibility for the determination of most of the elements. The relative reproducibility standard deviation ranged between 5% and 16% for metals in both solutions apart from a 33.6% for Arsenic probably due to the presence of few overestimated values very likely due to a blank contamination. For this reason the EURL-FCM did not allocate z-score for Arsenic in C2 even if most of the laboratories 35/44 performed an adequate blank correction and got satisfactory results ($|z| \leq 2$). Results showed a satisfactory laboratory performance. No difference between the performance of NRLs and OCLs was observed, so they were treated as one data population.

9. Conclusions

These ILCs were the first exercise of its kind with a focus on the quantification of elements potentially released from food contact materials. The involvement of the NRLs and the OCLs demonstrated a very satisfactory participation in the ILC.

Two separate ILC exercises were organized and completed: one exercise aimed at testing the ability to determine concentrations of metals with migration limits assigned for plastics in the European Regulation (EU) No 10/2011 [1]. It focused on the quantification of 7 metals from a solution of 3% acetic acid solution as simulant for acidic foods for plastics, as well as one undisclosed additional metal. The second exercise aimed at testing the ability to determine the migration of metals that are relevant to ceramics in a solution of 4% acetic acid solution.

The outcome of both ILCs was satisfactory. The majority of laboratories obtained good results for all elements in both samples. The rate of success was higher than 90% for almost all elements in both simulants, apart from the 80% for Arsenic due to a trace of arsenic in the blank.

The first exercise of the ILC allowed to underline the competence of the appointed NRLs and OCLs to determine the concentration of Ba, Co, Cu, Fe, Mn, Zn, Li and Sb, metals with SMLs laid down in regulation 10/2011 potentially released from plastic materials.

In addition, the second exercise of the ILC demonstrated that it was possible to show satisfactory laboratory performance of the appointed NRLs and OCLs laboratories to determine Pb and Cd at concentration levels much lower than current levels from directive 84/500/EEC. In addition it demonstrated and also their ability to determine successfully other metals such as Ba, Co, Mn, Ni, As and Al, which are potentially released from ceramic articles. The exercise on ceramics was a key work item to evaluate the enforceability of future measures since for ceramics the elements tested are amongst others under discussion for the inclusion in future revision(s) of the ceramics legislation. In this context the work served better support preparedness and improved performance of the NRLs and OCLs in anticipation of future legislative developments.

Table 6. Summary of results for Al, As, Ba, Cd, Co, Pb, Mn and Ni in sample C2 and Sb, Ba, Co, Cu, Fe, Li, Mn and Zn in sample PL1 calculated according to DIN 38402 A45.

Sample	Element	Assigned Value = Robust Mean, [ug/Kg]	Rel. target s.d. = Truncated Horwitz	Uncertainty of assigned value, [ug/Kg]	Tolerance limits |z| ≤ 2 [ug/Kg]	Rel. Reproducibility s.d.	Rel. Repeatability s.d.
PL1	Ba	688.26	16.92%	8.29	455.32-921.20	6.40%	1.03%
	Co	50.46	22.00%	0.66	28.26-72.66	7.29%	1.66%
	Cu	3072.30	13.51%	29.67	2242.12-3902.47	5.52%	0.97%
	Fe	19755.75	10.21%	219.35	15721.51-23789.99	6.15%	1.42%
	Mn	497.55	17.77%	4.83	320.73-674.36	5.49%	1.08%
	Zn	19897.15	10.20%	217.14	15838.40-23955.90	6.11%	1.03%
	Li	499.91	17.76%	6.88	322.37-677.44	6.96%	1.43%
	Sb	35.42	22.00%	1.21	19.84-51.01	15.68%	1.80%
C2	Ba	786.84	16.58%	8.92	525.84-1047.82	5.87%	0.99%
	Co	51.36	22.00%	0.44	28.76-73.96	4.61%	1.47%
	Mn	401.07	18.36%	3.88	253.84–548.30	5.42%	1.01%
	Pb	9.44	22.00%	0.21	5.29-13.60	12.66%	2.38%
	Cd	4.89	22.00%	0.07	2.74-7.04	8.54%	1.65%
	Ni	70.77	22.00%	0.71	39.63-101.91	5.62%	1.38%
	As	13.01	22.00%	0.83	7.29-18.74	33.65%	4.37%
	Al	727.18	16.78%	13.89	483.10-971.27	10.02%	1.68%

Table 7. Percentage of successfully results for the ILC03-2014 and ILC04 2014

Sample	Element	N° Results	Results within tolerance limits |z| ≤ 2 Satisfactory Results	Results between limits 2 ≤ |z| ≤ 3 Questionable Results	Results above |z| > 3 Unsatisfactory Results
PL1	Ba	44	42/44 = 95.5%	2/44 = 4.5%	0/44 = 0.0%
	Co	48	46/48 = 95.8%	1/48 = 2.1%	1/48 = 2.1%
	Cu	51	48/51 = 94.1%	2/51 = 3.9%	1/51 = 2.0%
	Fe	48	43/48 = 89.6%	2/48 = 4.2%	3/48 = 6.2%
	Mn	50	48/50 = 96.0%	2/50 = 4.0%	0/50 = 0.0%
	Zn	49	45/49 = 91.8%	3/49 = 6.1%	1/49 = 2.1%
	Li	40	40/40 = 100%	0/40 = 0.0%	0/40 = 0.0%
	Sb	33	30/33 = 90.9%	2/33 = 6.1%	1/33 = 3.0%
C2	Ba	42	38/42 = 90.5%	3/42 = 7.1%	1/42 = 2.4%
	Co	46	45/46 = 97.8%	1/46 = 2.2%	0/46 = 0.0%
	Mn	49	46/49 = 93.9%	0/49 = 0.0%	3/49 = 6.1%
	Pb	50	47/50 = 94%	3/50 = 6.0%	0/50 = 0.0%
	Cd	51	50/51 = 98.0%	1/51 = 2.0%	0/51 = 0.0%
	Ni	49	47/49 = 95.9%	2/49 = 4.1%	0/49 = 0.0%
	As	44	35/44 = 79.6%	3/44 = 6.8%	6/44 = 13.6%
	Al	43	40/43 = 93.0%	2/43 = 4.7%	1/43 = 2.3%

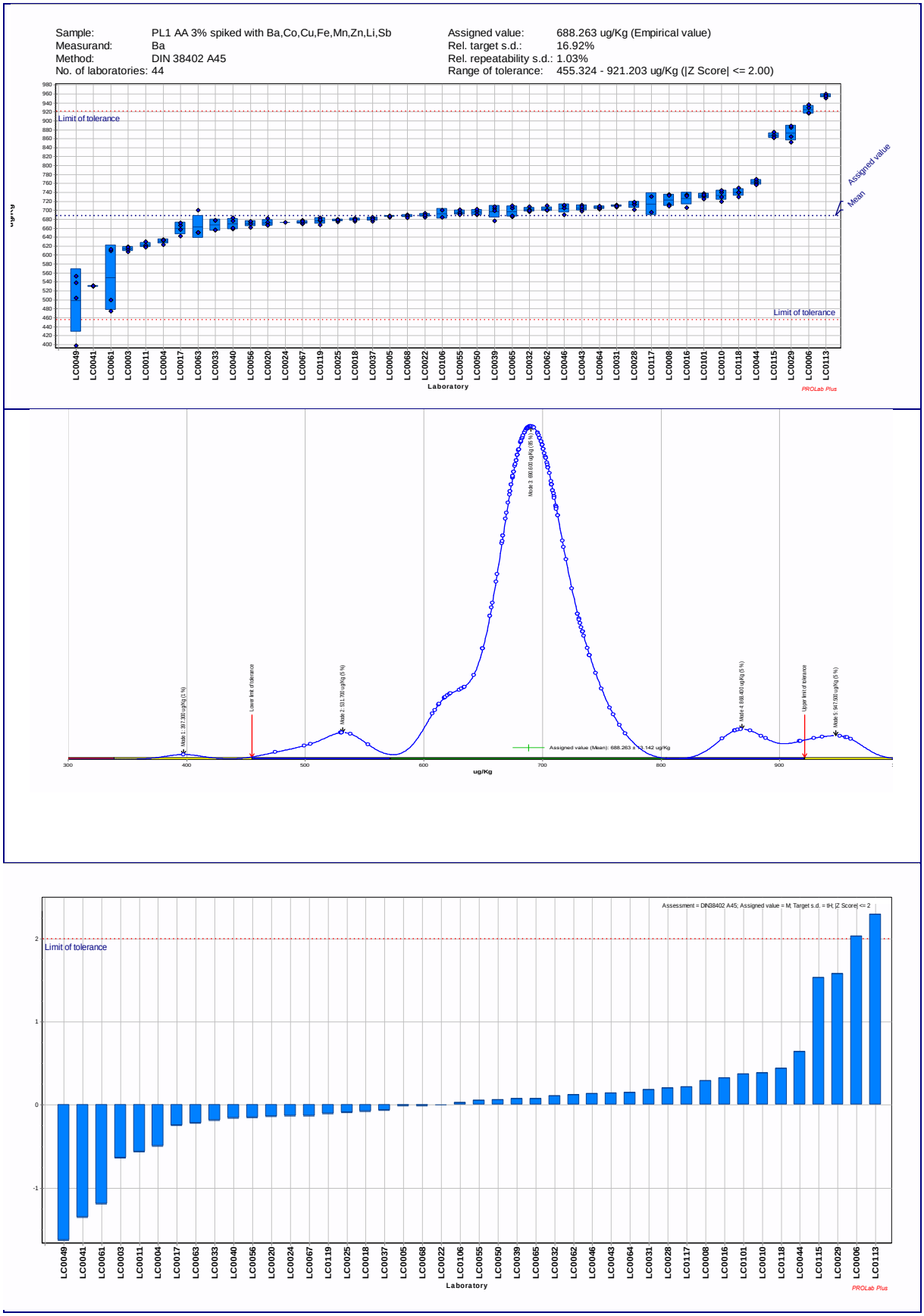

Figure 1. Summary of the laboratories test results for **Barium** in sample PL1 with their repeatability SD (1st graph), Kernel density plot (2nd graph) and z-scores (3rd graph)

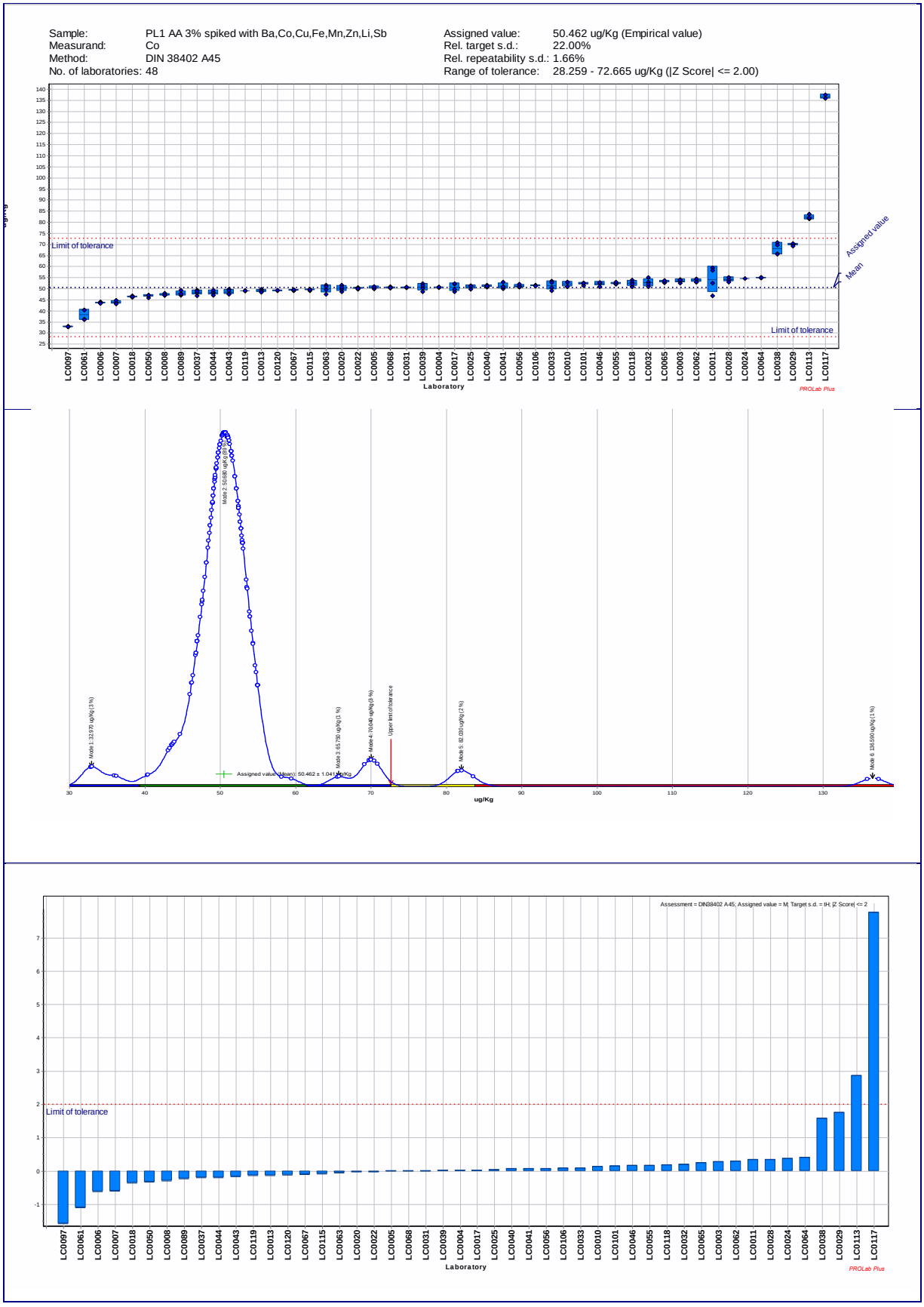

Figure 2. Summary of the laboratories test results for **Cobalt** in sample PL1 with their repeatability SD (1st graph), Kernel density plot (2nd graph) and z-scores (3rd graph)

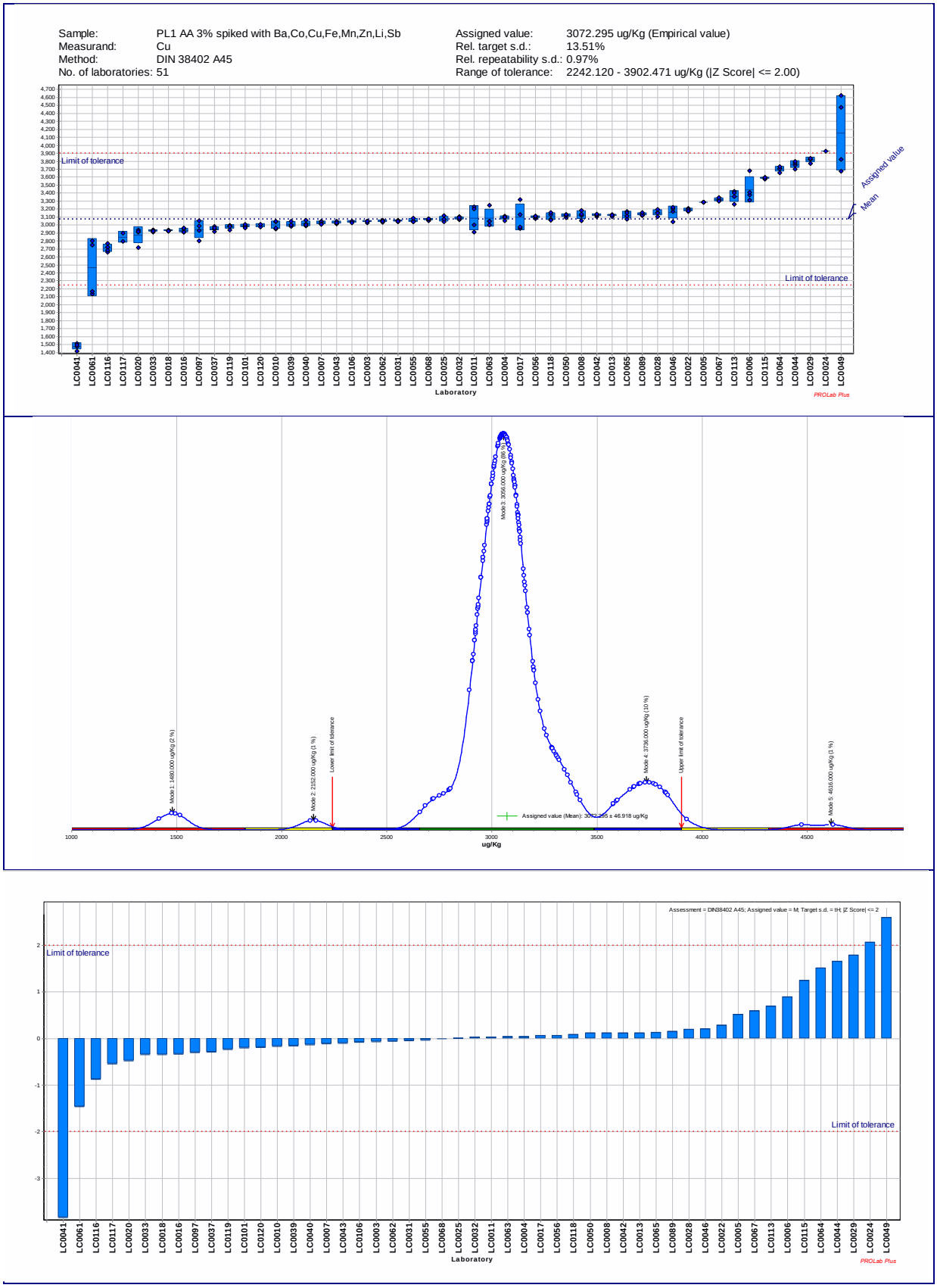

Figure 3. Summary of the laboratories test results for **Copper** in sample PL1 with their repeatability SD (1st graph), Kernel density plot (2nd graph) and z-scores (3rd graph)

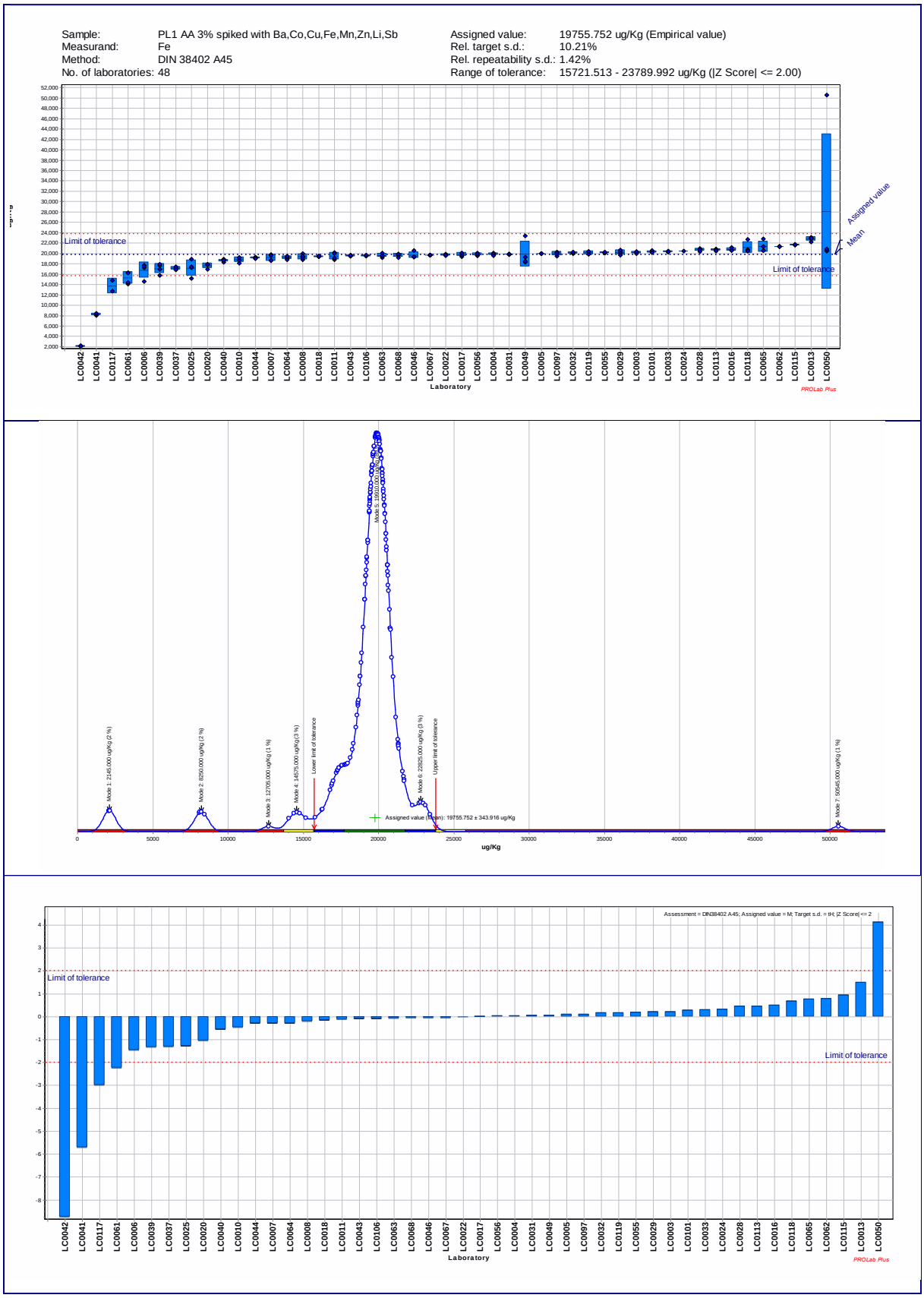

Figure 4. Summary of the laboratories test results for **Iron** in sample PL1 with their repeatability SD (1st graph), Kernel density plot (2nd graph) and z-scores (3rd graph)

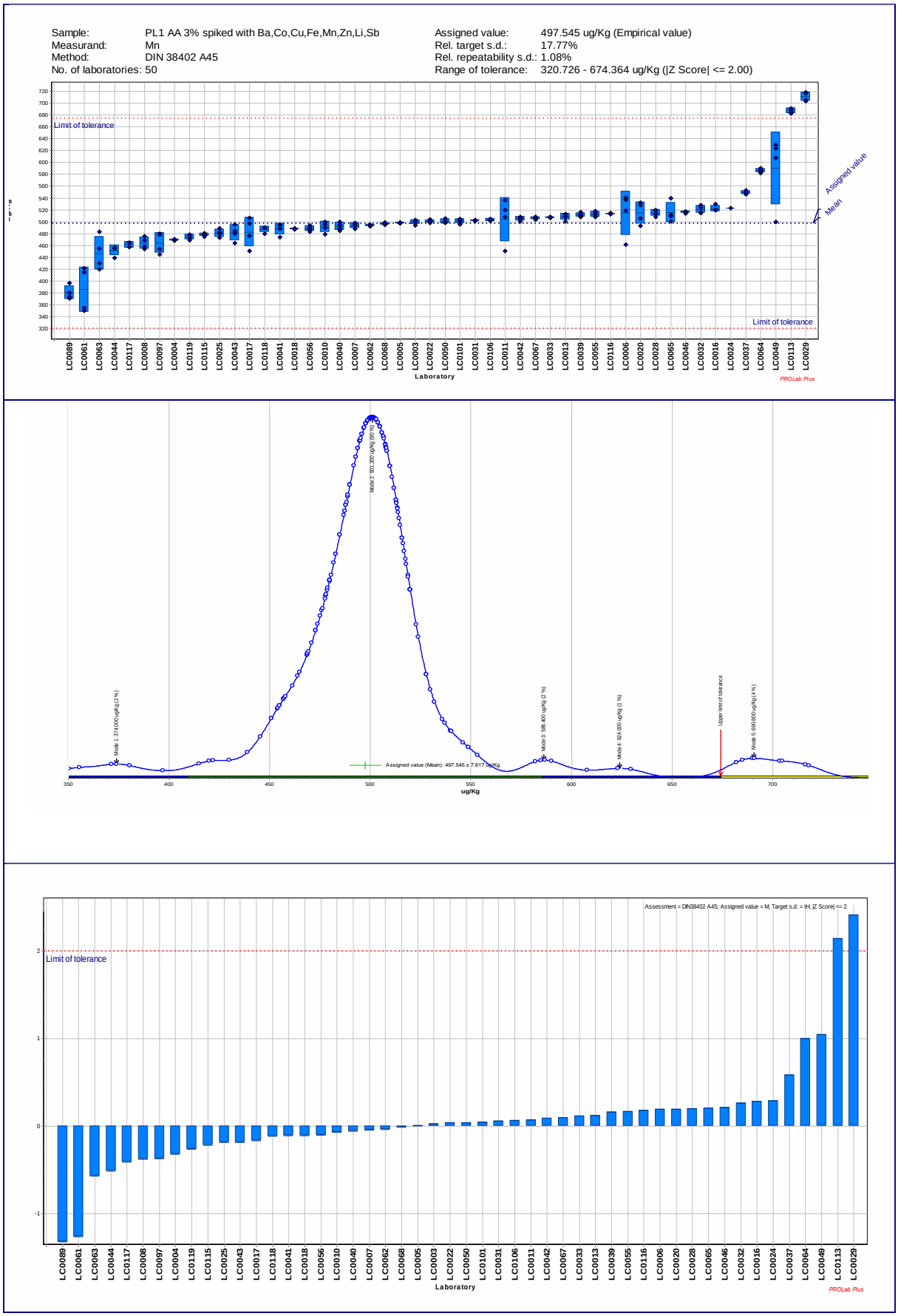

Figure 5. Summary of the laboratories test results for **Manganese** in sample PL1 with their repeatability SD (1st graph), Kernel density plot (2nd graph) and z-scores (3rd graph)

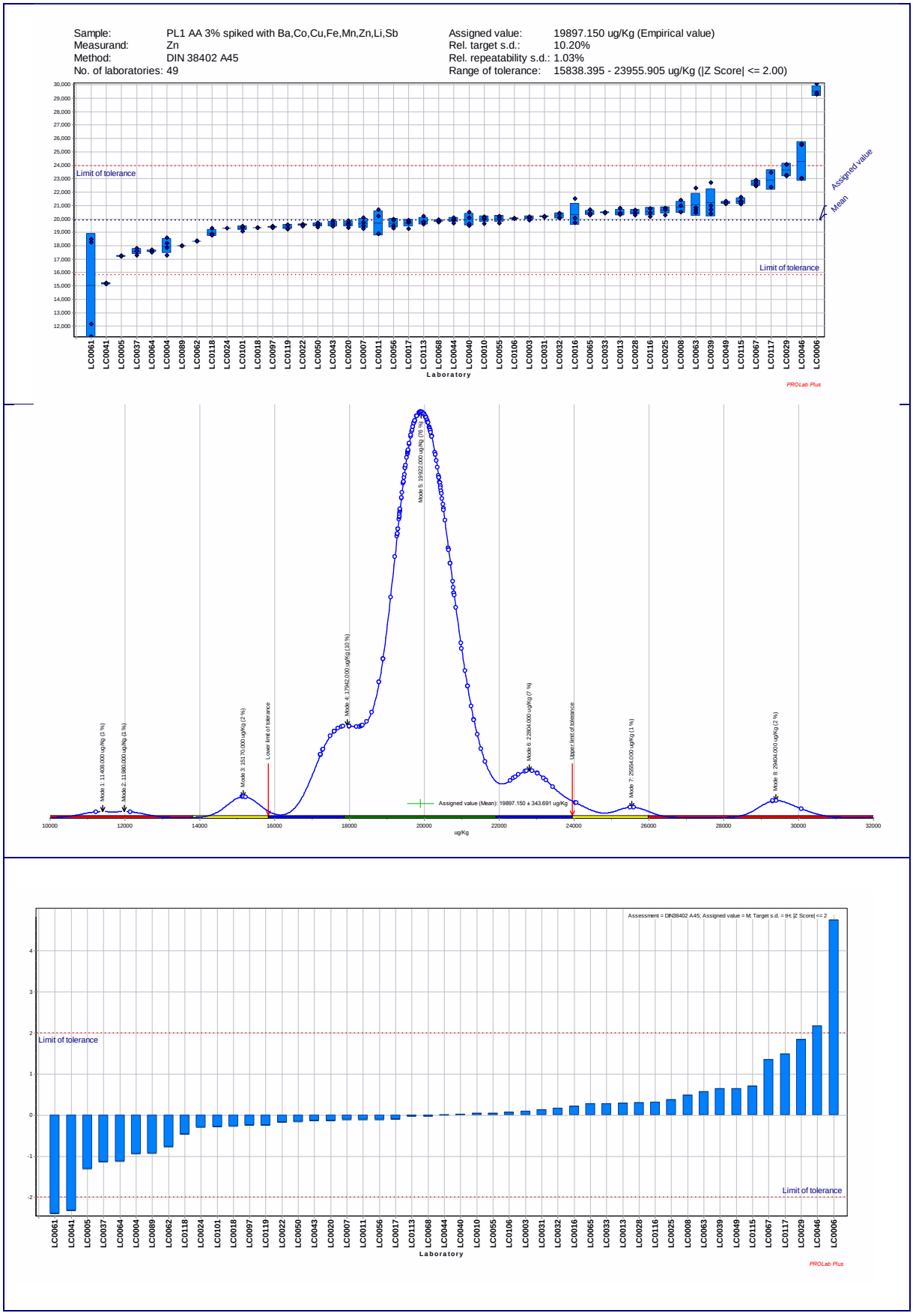

Figure 6. Summary of the laboratories test results for **Zinc** in sample PL1 with their repeatability SD (1st graph), Kernel density plot (2nd graph) and z-scores (3rd graph)

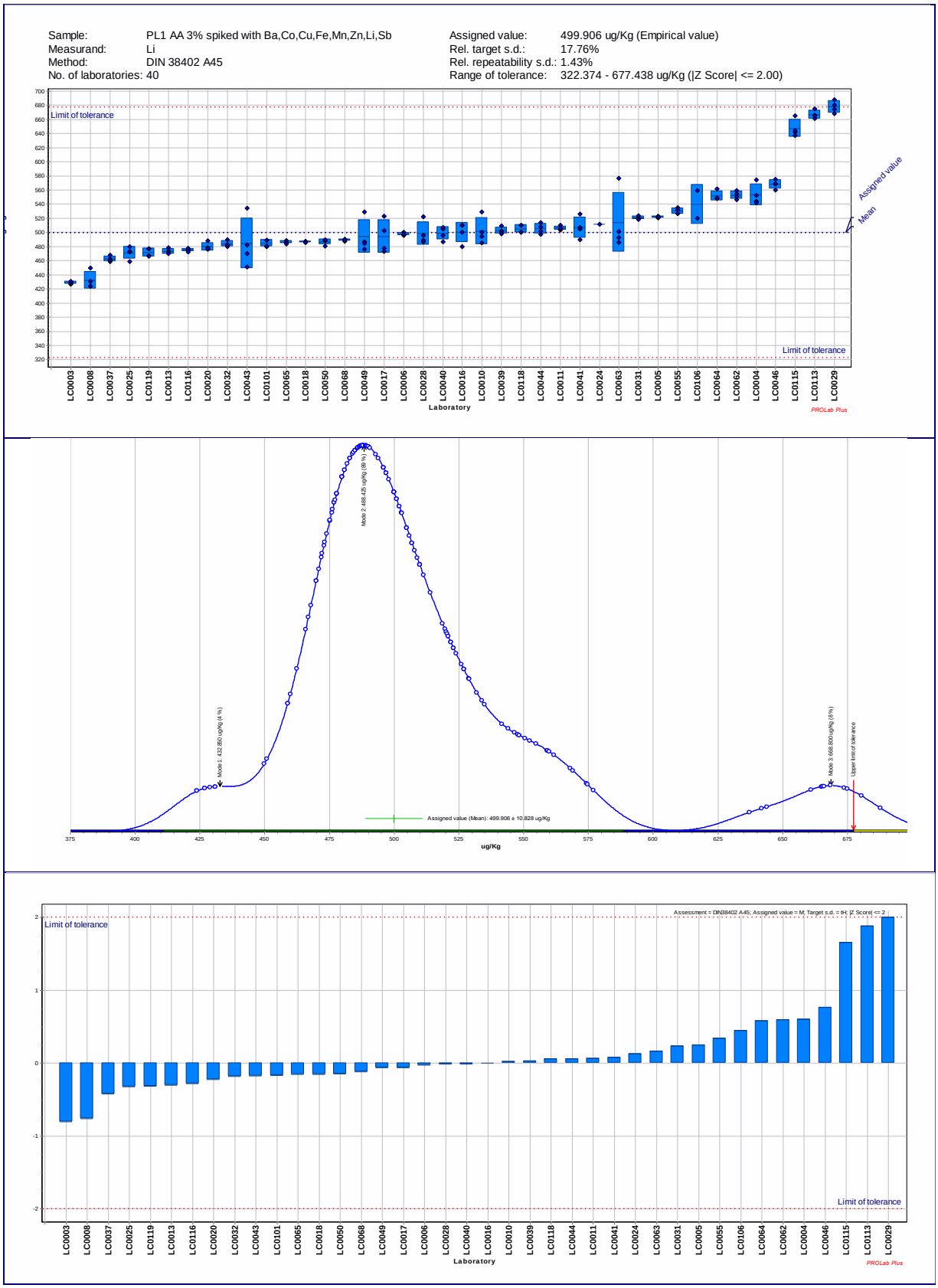

Figure 7. Summary of the laboratories test results for **Lithium** in sample PL1 with their repeatability SD (1st graph), Kernel density plot (2nd graph) and z-scores (3rd graph)

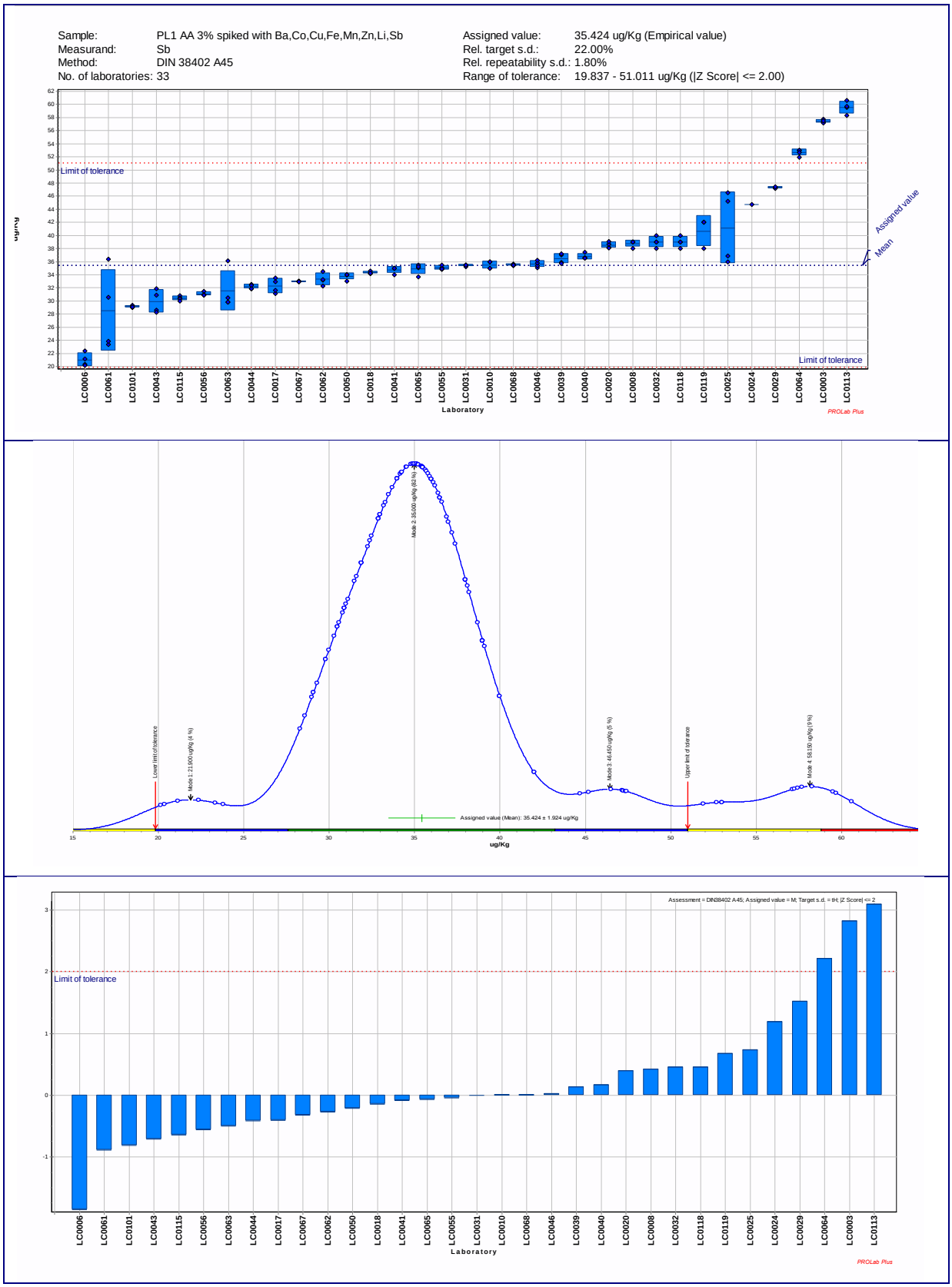

Figure 8. Summary of the laboratories test results for **Antimony** in sample PL1 with their repeatability SD (1st graph), Kernel density plot (2nd graph) and z-scores (3rd graph)

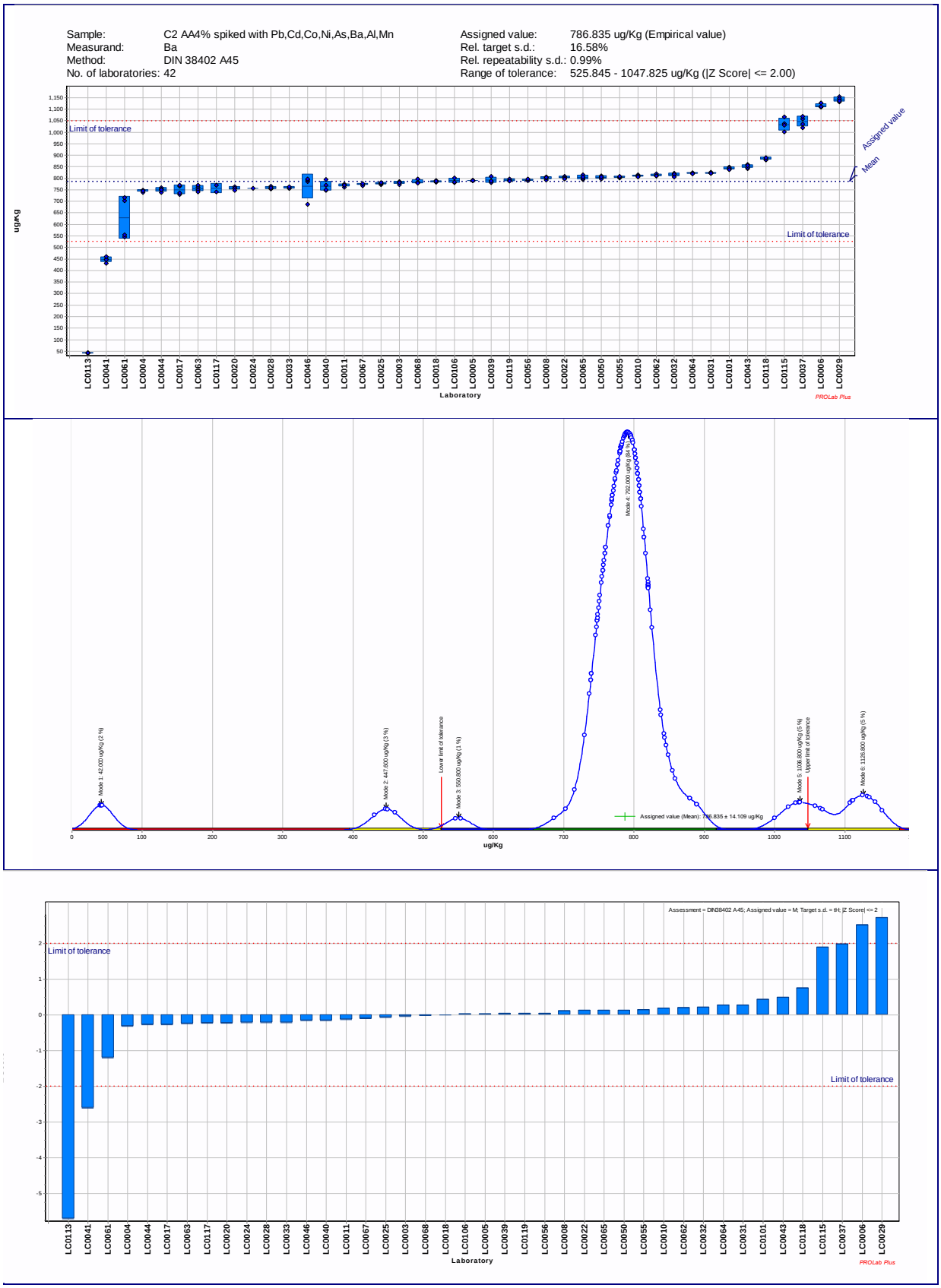

Figure 9. Summary of the laboratories test results for **Barium** in sample C2 with their repeatability SD (1st graph), Kernel density plot (2nd graph) and z-scores (3rd graph)

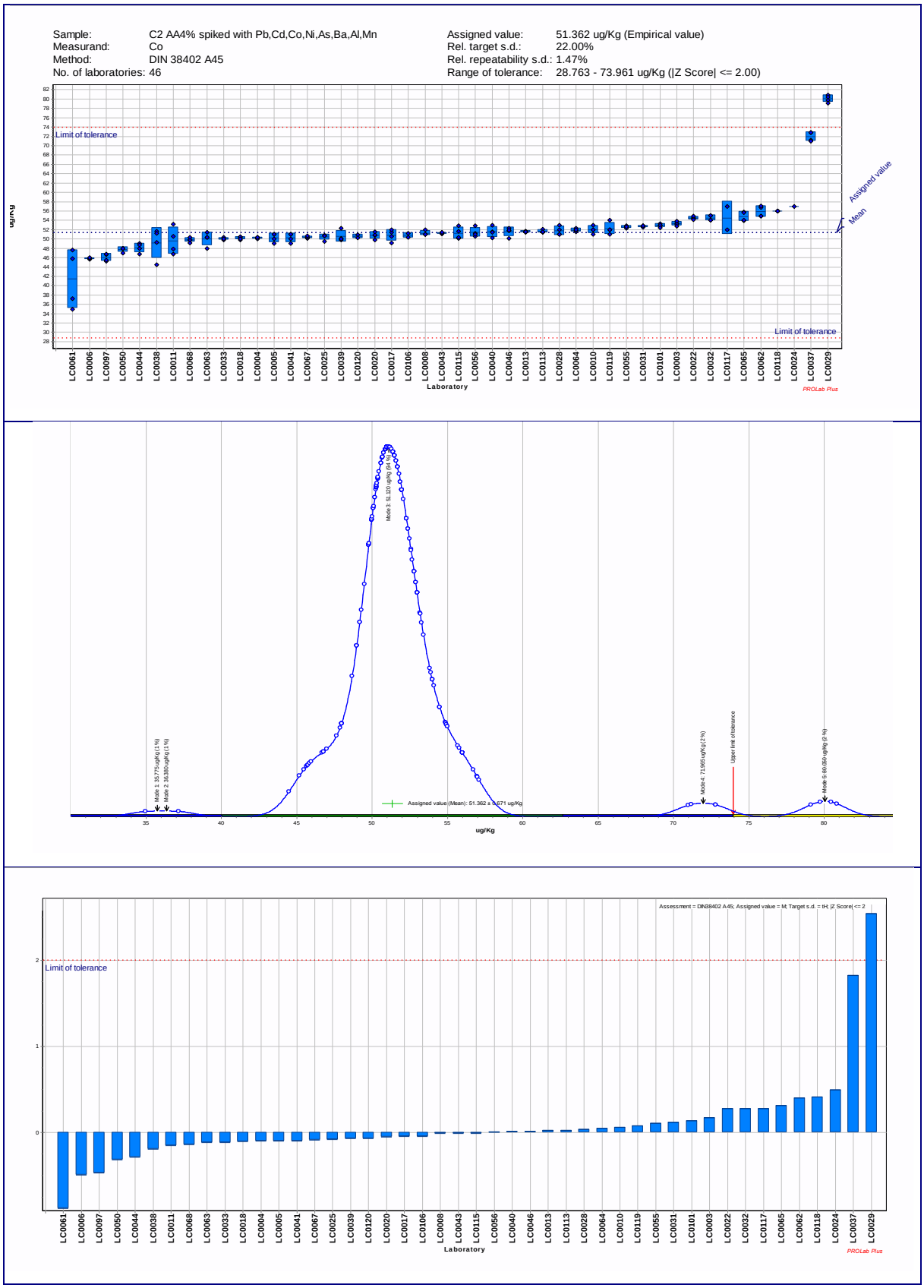

Figure 10. Summary of the laboratories test results for **Cobalt** in sample C2 with their repeatability SD (1st graph), Kernel density plot (2nd graph) and z-scores (3rd graph)

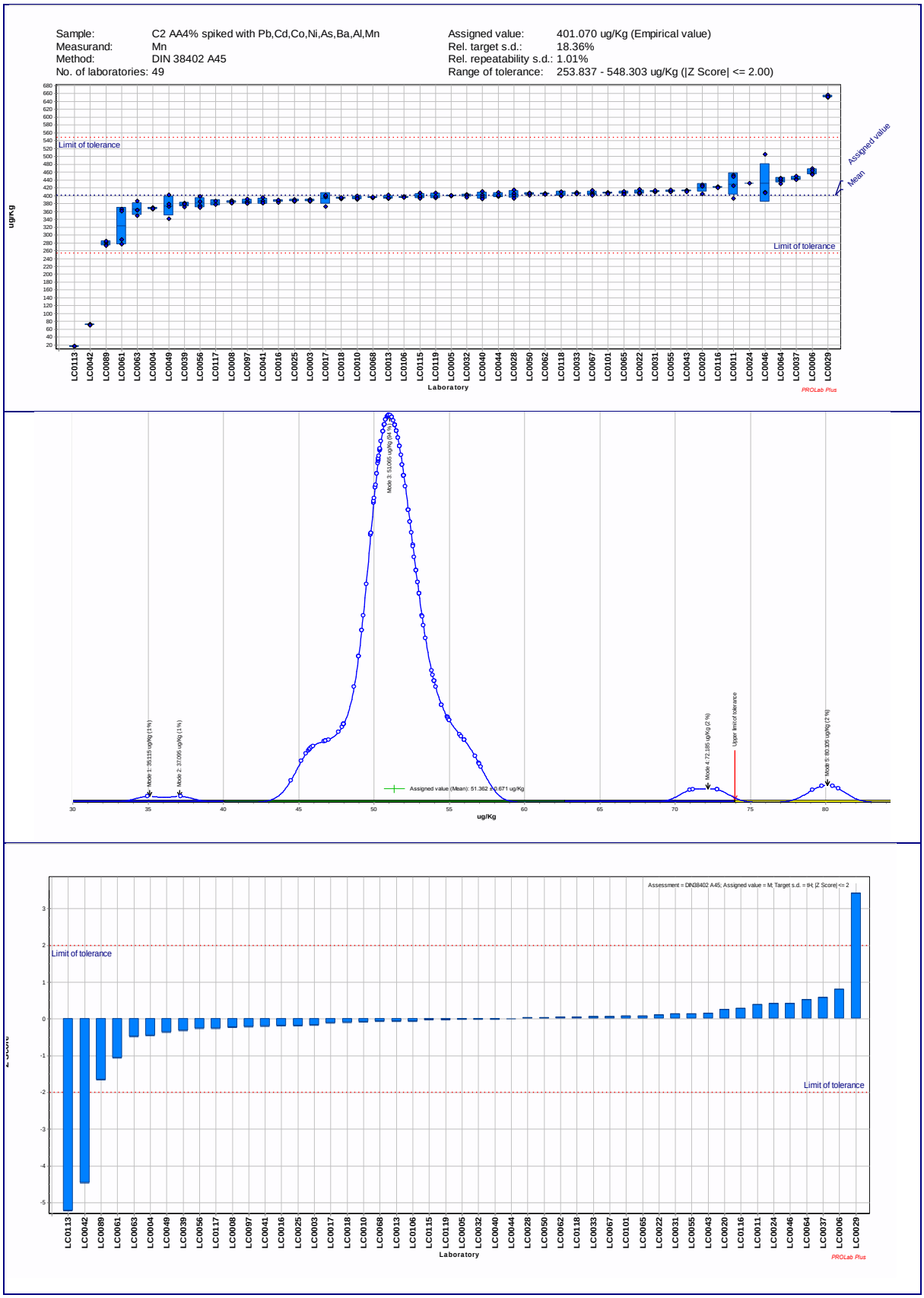

Figure 11. Summary of the laboratories test results for **Manganese** in sample C2 with their repeatability SD (1st graph), Kernel density plot (2nd graph) and z-scores (3rd graph)

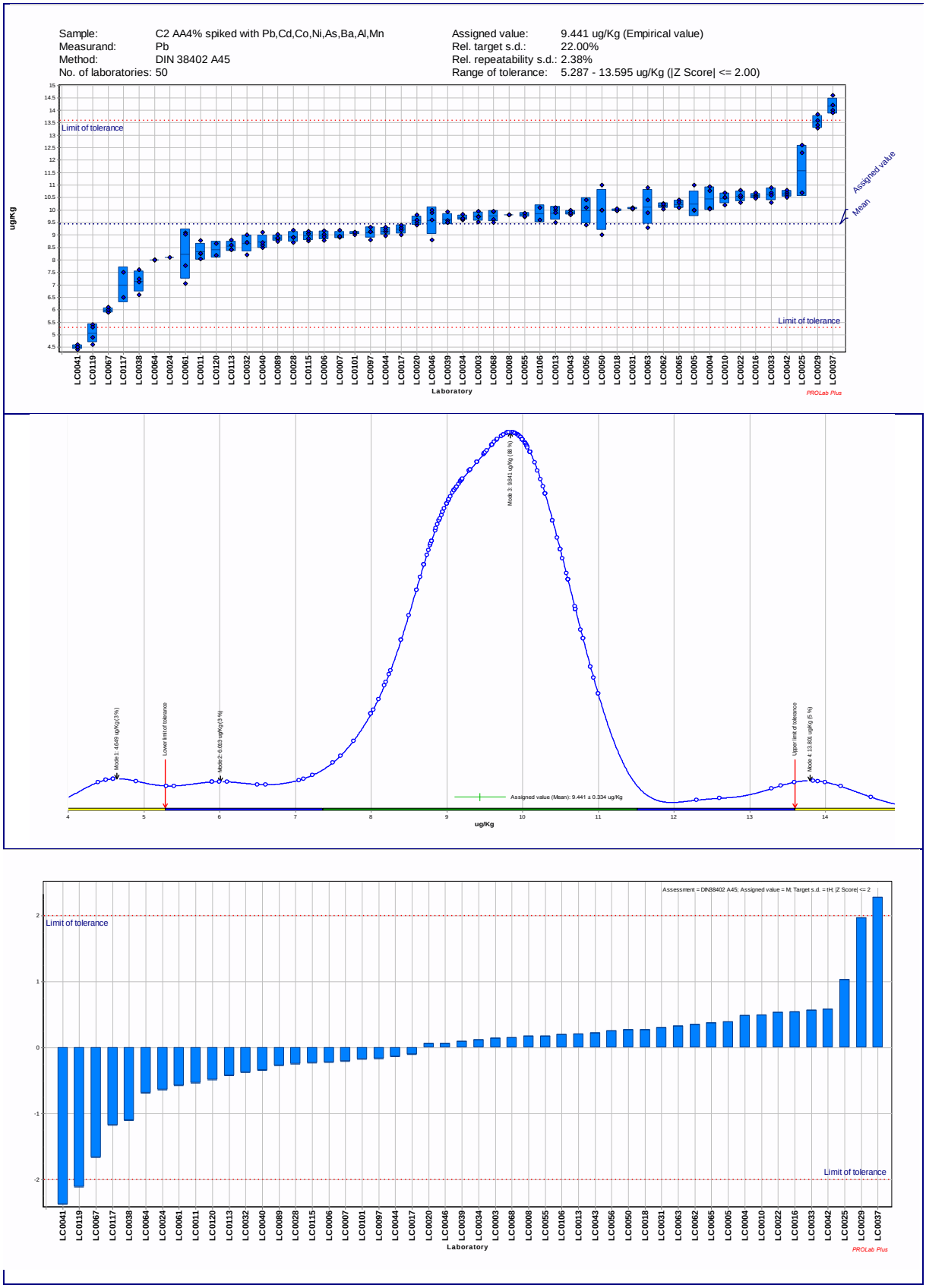

Figure 12. Summary of the laboratories test results for **Lead** in sample C2 with theirs repeatability SD (1st graph), Kernel density plot (2nd graph) and z-scores (3rd graph)

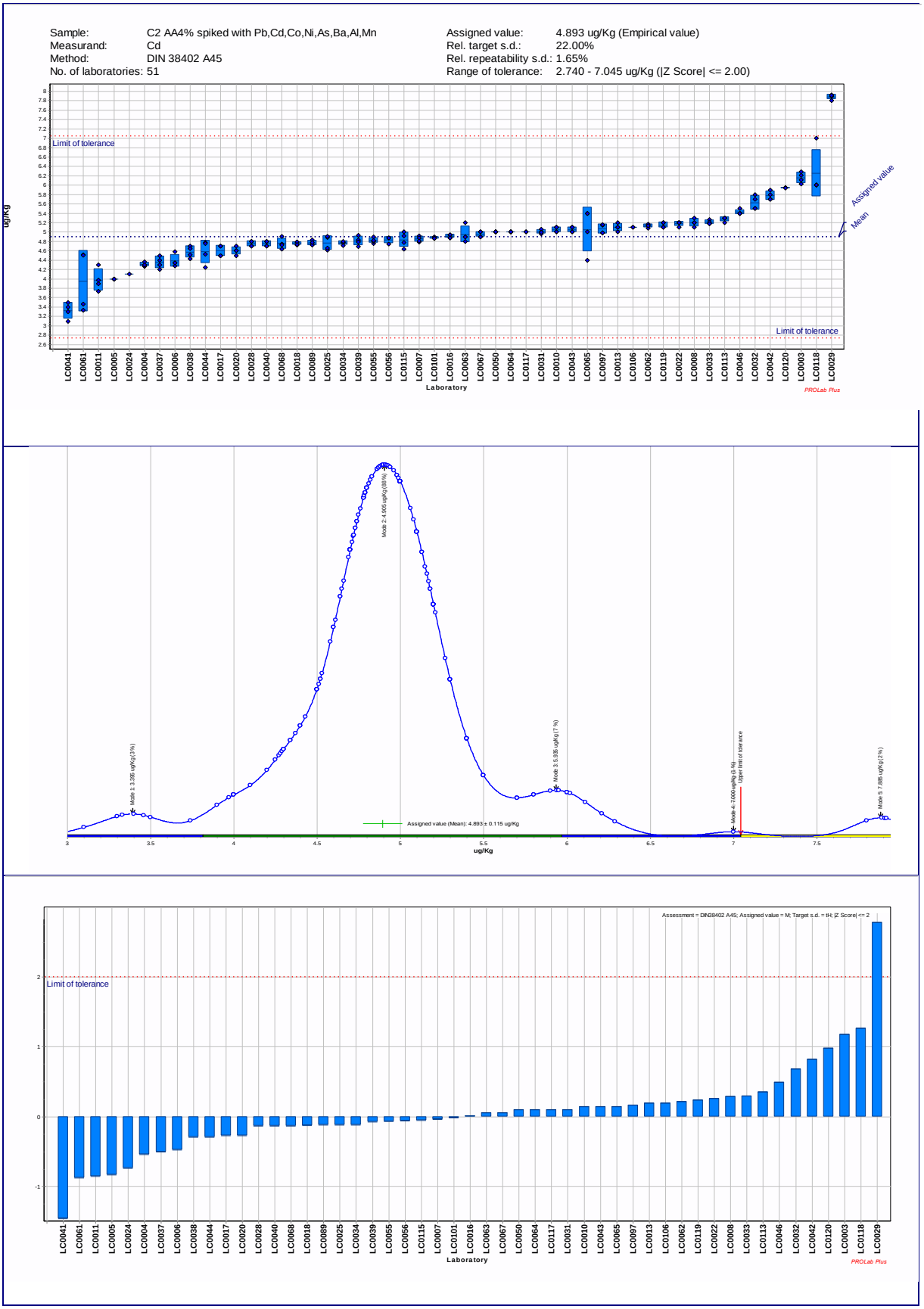

Figure 13. Summary of the laboratories test results for **Cadmium** in sample C2 with their repeatability SD (1st graph), Kernel density plot (2nd graph) and z-scores (3rd graph)

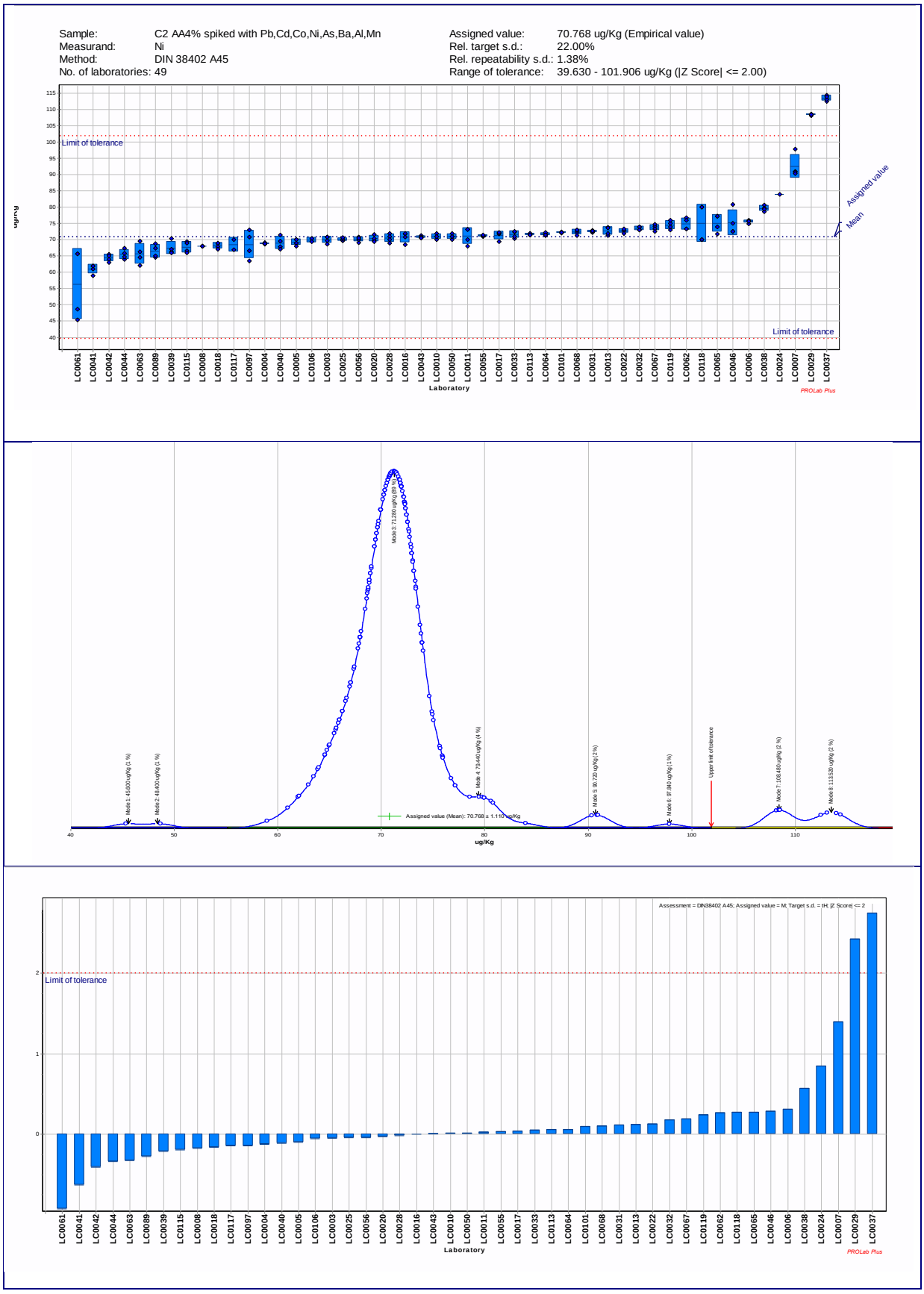

Figure 14. Summary of the laboratories test results for **Nickel** in sample C2 with theirs repeatability SD (1st graph), Kernel density plot (2nd graph) and z-scores (3rd graph)

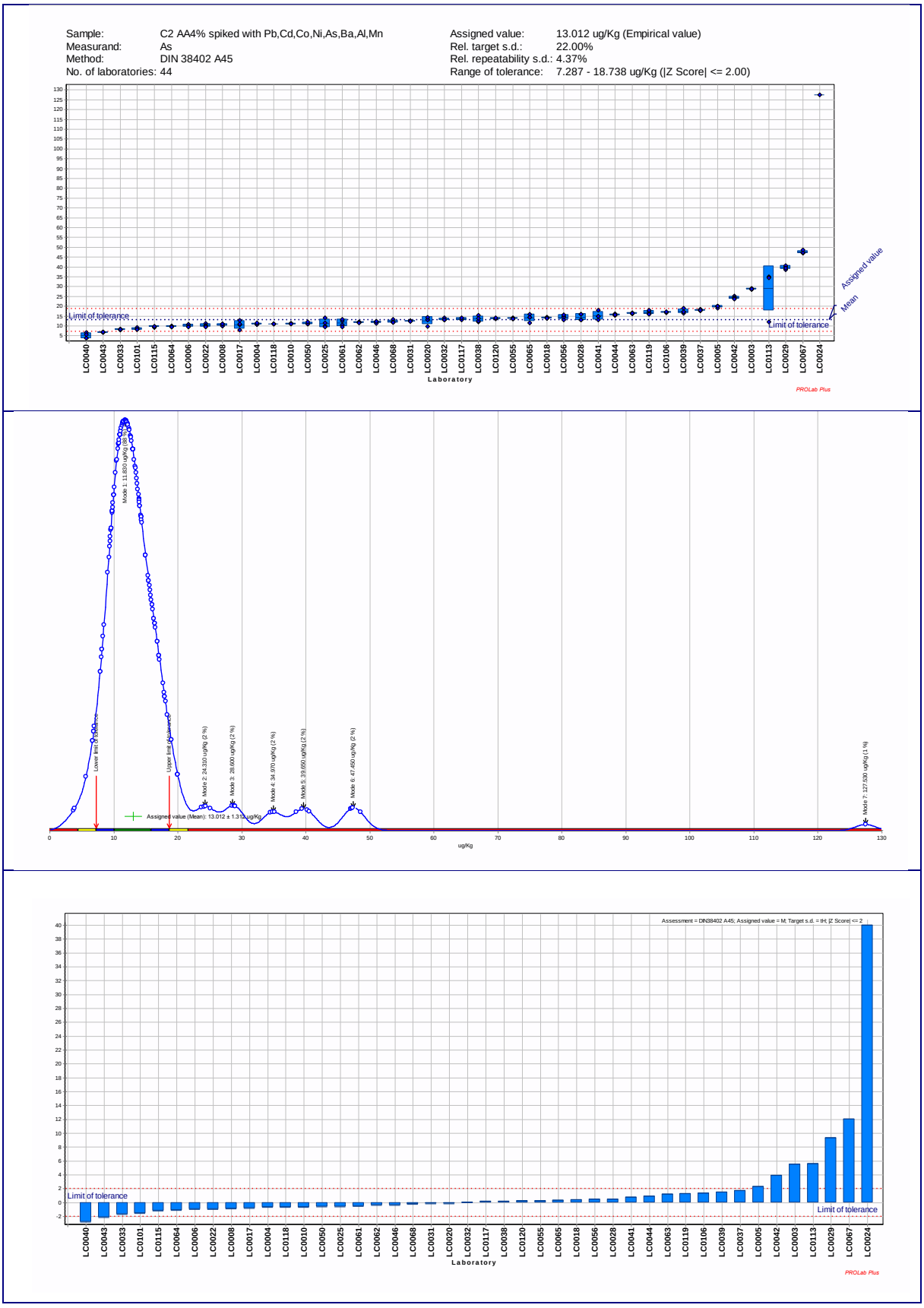

Figure 15. Summary of the laboratories test results for **Arsenic** in sample C2 with their repeatability SD (1st graph), Kernel density plot (2nd graph) and z-scores (3rd graph)

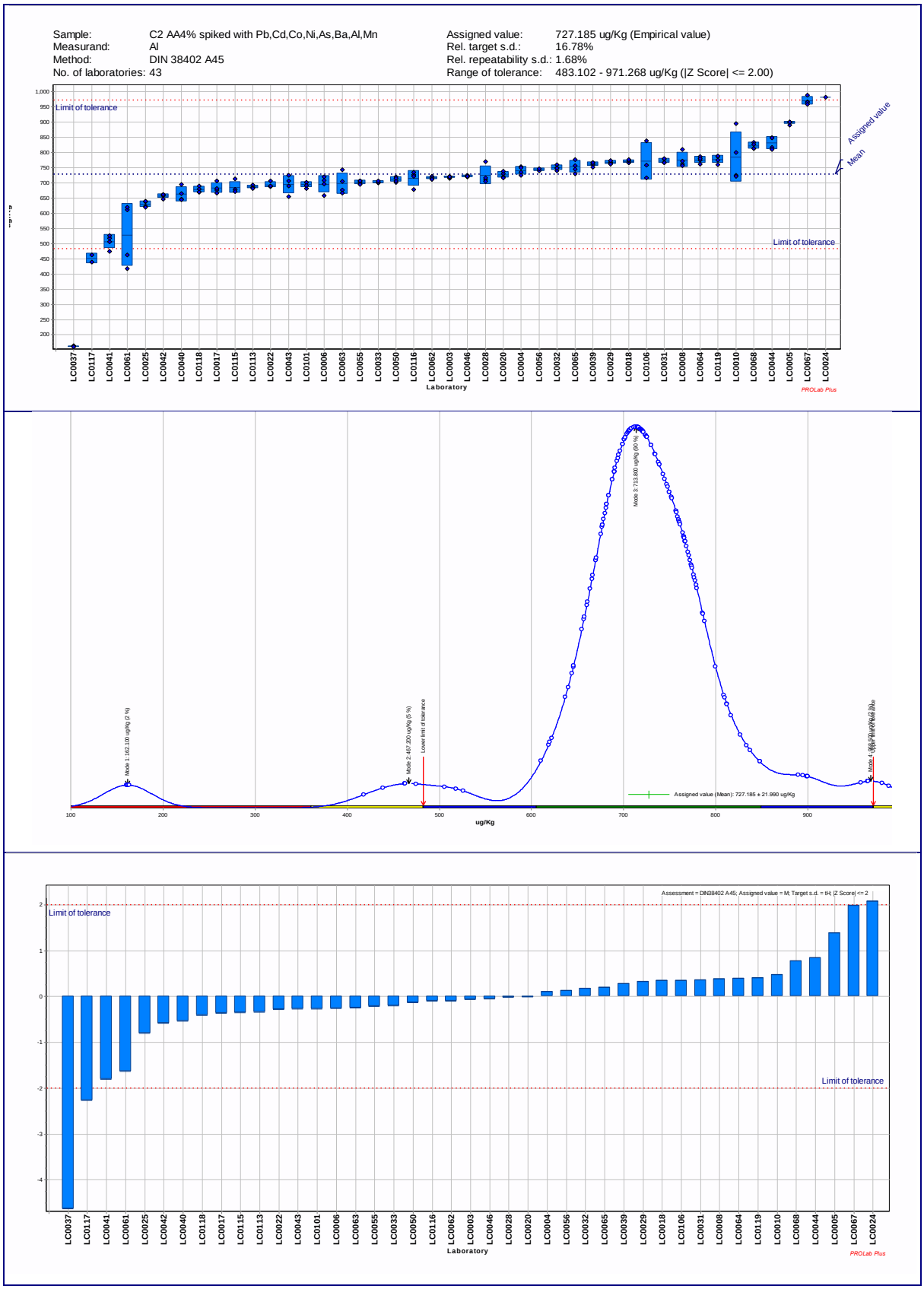

Figure 16. Summary of the laboratories test results for **Aluminium** in the sample C2 with their repeatability SD (1st graph), Kernel density plot (2nd graph) and z-scores (3rd graph)

Table 8. Summary of the z-scores for sample PL1

Z-SCORES SAMPLE PL1								
LAB CODE	BARIUM	COBALT	COPPER	IRON	MANGANESE	ZINC	LITHIUM	ANTIMONY
LC0003	-0.64	0.28	-0.07	0.22	0.02	0.09	-0.80	2.82
LC0004	-0.49	0.02	0.04	0.04	-0.32	-0.94	0.60	
LC0005	-0.01	0.00	0.52	0.11	0.01	-1.31	0.25	
LC0006	2.03	-0.62	0.89	-1.47	0.19	4.75	-0.03	-1.85
LC0007		-0.60	-0.11	-0.30	-0.05	-0.12		
LC0008	0.29	-0.29	0.12	-0.20	-0.38	0.48	-0.76	0.43
LC0010	0.39	0.14	-0.18	-0.47	-0.07	0.04	0.02	0.01
LC0011	-0.56	0.34	0.03	-0.11	0.07	-0.11	0.07	
LC0013		-0.13	0.12	1.51	0.12	0.29	-0.30	
LC0016	0.33		-0.34	0.50	0.28	0.22	0.00	
LC0017	-0.25	0.02	0.06	0.03	-0.17	-0.10	-0.06	-0.40
LC0018	-0.08	-0.36	-0.35	-0.16	-0.11	-0.27	-0.15	-0.14
LC0020	-0.14	-0.03	-0.48	-1.05	0.20	-0.13	-0.23	0.40
LC0022	0.01	-0.02	0.28	-0.02	0.04	-0.17		
LC0024	-0.13	0.38	2.06	0.33	0.29	-0.29	0.13	1.19
LC0025	-0.09	0.04	0.01	-1.28	-0.19	0.37	-0.33	0.74
LC0028	0.21	0.34	0.19	0.46	0.20	0.30	-0.02	
LC0029	1.58	1.75	1.79	0.21	2.41	1.85	2.00	1.52
LC0031	0.19	0.01	-0.05	0.05	0.06	0.13	0.24	0.00
LC0032	0.11	0.21	0.03	0.18	0.26	0.17	-0.18	0.46
LC0033	-0.18	0.09	-0.35	0.30	0.11	0.28		
LC0034								
LC0037	-0.07	-0.20	-0.29	-1.30	0.59	-1.14	-0.42	
LC0038		1.58						
LC0039	0.08	0.02	-0.16	-1.34	0.16	0.64	0.03	0.13
LC0040	-0.16	0.07	-0.13	-0.55	-0.06	0.02	-0.01	0.17
LC0041	-1.35	0.07	-3.85	-5.71	-0.11	-2.33	0.08	-0.09
LC0042			0.12	-8.74	0.09			
LC0043	0.15	-0.17	-0.11	-0.10	-0.18	-0.14	-0.18	-0.71
LC0044	0.64	-0.20	1.65	-0.30	-0.52	0.01	0.06	-0.42
LC0046	0.14	0.17	0.20	-0.05	0.21	2.16	0.77	0.03
LC0049	-1.63		2.60	0.05	1.05	0.64	-0.06	
LC0050	0.06	-0.33	0.11	4.13	0.04	-0.16	-0.15	-0.22
LC0055	0.06	0.18	-0.04	0.20	0.17	0.05	0.34	-0.04
LC0056	-0.16	0.07	0.07	0.03	-0.10	-0.11		-0.56
LC0061	-1.19	-1.10	-1.47	-2.23	-1.26	-2.40		-0.88
LC0062	0.12	0.29	-0.06	0.79	-0.04	-0.77	0.60	-0.27
LC0063	-0.22	-0.05	0.04	-0.08	-0.57	0.57	0.16	-0.50
LC0064	0.15	0.40	1.51	-0.29	1.00	-1.13	0.58	2.21
LC0065	0.08	0.24	0.13	0.78	0.20	0.27	-0.15	-0.07
LC0067	-0.13	-0.10	0.59	-0.05	0.10	1.36		-0.32
LC0068	-0.01	0.01	-0.01	-0.05	-0.01	-0.03	-0.12	0.01
LC0089		-0.23	0.15		-1.32	-0.94		
LC0097		-1.58	-0.31	0.11	-0.38	-0.25		
LC0101	0.38	0.16	-0.20	0.28	0.04	-0.28	-0.17	-0.81
LC0106	0.03	0.08	-0.08	-0.09	0.06	0.07	0.45	
LC0113	2.30	2.86	0.69	0.47	2.14	-0.03	1.88	3.09
LC0114								
LC0115	1.54	-0.08	1.24	0.95	-0.22	0.70	1.66	-0.64
LC0116			-0.87		0.18	0.31	-0.28	
LC0117	0.22	7.76	-0.54	-2.98	-0.41	1.49		
LC0118	0.44	0.18	0.08	0.69	-0.11	-0.47	0.06	0.46
LC0119	-0.10	-0.13	-0.24	0.18	-0.26	-0.24	-0.32	0.67
LC0120		-0.12	-0.19					

Table 9. Summary of the z-scores for sample C2

Z-SCORES SAMPLE C2								
LAB CODE	BARIUM	COBALT	MANGANESE	LEAD	CADMIUM	NICKEL	ARSENIC	ALUMINIUM
LC0003	-0.05	0.17	-0.17	0.14	1.18	-0.05	-	-0.07
LC0004	-0.32	-0.10	-0.45	0.49	-0.54	-0.13	-	0.11
LC0005	0.03	-0.10	-0.01	0.39	-0.83	-0.10	-	1.39
LC0006	2.51	-0.49	0.81	-0.22	-0.47	0.31	-	-0.26
LC0007				-0.21	-0.04	1.40	-	
LC0008	0.10	-0.01	-0.23	0.17	0.29	-0.18	-	0.39
LC0010	0.18	0.06	-0.09	0.50	0.15	0.02	-	0.47
LC0011	-0.14	-0.15	0.39	-0.54	-0.85	0.02	-	
LC0013		0.02	-0.07	0.21	0.19	0.12	-	
LC0016			-0.19	0.54	0.01	0.00	-	
LC0017	-0.28	-0.05	-0.11	-0.10	-0.27	0.04	-	-0.37
LC0018	-0.01	-0.10	-0.10	0.27	-0.13	-0.17	-	0.35
LC0020	-0.24	-0.05	0.26	0.06	-0.27	-0.03	-	-0.01
LC0022	0.13	0.28	0.11	0.53	0.26	0.12	-	-0.28
LC0024	-0.22	0.50	0.42	-0.65	-0.74	0.85	-	2.08
LC0025	-0.07	-0.08	-0.18	1.03	-0.12	-0.04	-	-0.80
LC0028	-0.22	0.03	0.03	-0.25	-0.13	-0.02	-	-0.02
LC0029	2.72	2.54	3.42	1.97	2.78	2.42	-	0.32
LC0031	0.27	0.12	0.13	0.30	0.10	0.12	-	0.37
LC0032	0.21	0.28	-0.01	-0.38	0.68	0.18	-	0.18
LC0033	-0.22	-0.12	0.07	0.57	0.30	0.05	-	-0.21
LC0034				0.12	-0.12		-	
LC0037	1.98	1.82	0.58	2.28	-0.50	2.74	-	-4.63
LC0038		-0.19		-1.11	-0.30	0.57	-	
LC0039	0.04	-0.07	-0.32	0.09	-0.08	-0.21	-	0.28
LC0040	-0.16	0.01	-0.01	-0.34	-0.13	-0.11	-	-0.53
LC0041	-2.60	-0.10	-0.20	-2.38	-1.46	-0.63	-	-1.81
LC0042			-4.47	0.58	0.82	-0.41	-	-0.58
LC0043	0.49	-0.01	0.15	0.22	0.15	0.01	-	-0.27
LC0044	-0.28	-0.29	0.01	-0.14	-0.29	-0.34	-	0.85
LC0046	-0.16	0.01	0.43	0.06	0.50	0.29	-	-0.05
LC0049			-0.37				-	
LC0050	0.13	-0.32	0.04	0.27	0.10	0.02	-	-0.13
LC0055	0.14	0.11	0.14	0.17	-0.07	0.03	-	-0.21
LC0056	0.04	0.01	-0.26	0.25	-0.06	-0.04	-	0.13
LC0061	-1.20	-0.88	-1.06	-0.58	-0.87	-0.93	-	-1.63
LC0062	0.20	0.40	0.05	0.35	0.22	0.26	-	-0.10
LC0063	-0.24	-0.12	-0.48	0.33	0.05	-0.33	-	-0.24
LC0064	0.27	0.05	0.52	-0.69	0.10	0.06	-	0.39
LC0065	0.13	0.31	0.09	0.38	0.15	0.27	-	0.20
LC0067	-0.10	-0.09	0.07	-1.67	0.05	0.19	-	1.98
LC0068	-0.02	-0.14	-0.07	0.15	-0.13	0.10	-	0.77
LC0089			-1.66	-0.27	-0.12	-0.28	-	
LC0097		-0.47	-0.22	-0.17	0.16	-0.14	-	
LC0101	0.43	0.13	0.08	-0.18	-0.02	0.10	-	-0.27
LC0106	0.02	-0.05	-0.07	0.20	0.19	-0.06	-	0.36
LC0113	-5.71	0.02	-5.22	-0.43	0.36	0.06	-	-0.34
LC0114							-	
LC0115	1.89	-0.01	-0.02	-0.23	-0.05	-0.19	-	-0.35
LC0116			0.28				-	-0.10
LC0117	-0.24	0.28	-0.26	-1.18	0.10	-0.15	-	-2.26
LC0118	0.75	0.41	0.05		1.26	0.27	-	-0.41
LC0119	0.04	0.08	-0.02	-2.11	0.24	0.24	-	0.41
LC0120		-0.07		-0.49	0.98		-	

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Switzerland	Official Food Control Authority, Canton of Zurich, Zürich
United Kingdom	The Food and Environment Research Agency, York

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Portugal	Centro de Formação Profissional para a Indústria Caldas da Rainha
Belgium	FAVV-lab FLVGG, Braemkasteelstraat 59 9050 Gentbrugge, Belgium

11. References

[1] Regulation EC10/2011 of the European Parliament and the Council of 14 January 2011 on plastic materials and articles intended to come into contact with food

[2] Metals and alloys used in food contact materials and articles. A practical guide for manufactures and regulators, Council of Europe Resolution, 1st Edition, 1-216 (2013)

[3] ISO 13528:2005(E). Statistical Methods for Use in Proficiency Testing by Inter-laboratory Comparisons

[4] Regulation (EC) No 882/2004 of the European Parliament and of the Council of 29 April 2004 on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules

[5] Directive 84/500/EEC, Council Directive of 15 Oct. 1984 on the approximation of the laws of the Member States relating to ceramic articles intended to come into contact with foodstuffs

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12. Annexes

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Annex 1. Invitation letter to the laboratories

EUROPEAN COMMISSION
GENERAL DIRECTORATE JRC
JOINT RESEARCH CENTRE
Institute for Health and Consumer Protection – IHCP
Unit Chemical Assessment and Testing

Ispra May 12th, 2014

Dear Madam, Sir

Interlaboratory Comparison Exercise (ILC03 2014-ILC04 2014) on Elements from Food Contact Materials

On behalf of the EURL for food contact materials, I would like to invite you to participate in an Interlaboratory Comparison (ILC) Exercise for the determination of known and unknown Elements released from Food Contact Materials which is due to start by the beginning of June 2014. As agreed in the last EURL-NRL FCM plenary, the scope of this ILC exercise is a Proficiency testing, laboratories are free to use their own methods.

This ILC will include two exercises:

First part: Quantification of Elements released from Plastic Materials
(COMMISSION REGULATION (EU) No 10/2011)

The aim of the first exercise will be the quantification of the 8 substances potentially released from plastic materials and articles into **Acetic acid 3 % (w/v)** reported in table 1. Please be careful an unknown substance will be present in the migration solution Acetic acid 3 % (w/v).

Table 1

First part: Elements to be quantified in Acetic acid 3 % (w/v)	
Barium	Manganese
Cobalt	Zinc
Copper	Lithium
Iron	**Unknown Element**

Note: The concentration range will not exceed 25 mg/Kg with respect to Commission Regulation (EU) No 10/2011.

Second part: Quantification of Elements released from Ceramics

The aim of the second exercise will be the quantification of the 8 substances potentially released from Ceramics into **Acetic acid 4 % (w/v)** reported in table 2.

Table 2

Second part: Elements to be quantified in Acetic acid 4 % (w/v)	
Lead	Arsenic
Cadmium	Barium
Cobalt	Aluminium
Nickel	Manganese

Note: the concentration range is based on table 3.

Table 3

Metal	Basis	Organoleptic concerns (µg/kg)	Plastics (10/2011) (µg/kg)	WHO, GL drinking water (µg/kg)	NL (RIVM 2000) (µg/kg)	CoE Metals june 2011 (µg/kg)	**DSV* (µg/kg)**
Al	EFSA 2008	yes			-	900	**1000**
As	EFSA 2009			10	1	2	**18**
Ba	EFSA 2009	yes	1000	700	1200	1200	**1000**
Co	RIVM		50		84	10	**50**
Cu	SCF		5000	2000	8400	1000	**1000**
Fe	WHO 2008	yes	48000	2000	-	2000	**2500**
Mn	WHO 2008		600	400	-	900	**400**
Ni	WHO 2008			70	3000	70	**72**
Sb	CoE 2011				20	40	**40**
Zn	EFSA/SCF	yes	25000	3000	30000	5000	**1500**

* Discussion starting values.

We have pre-registered everyone, which means we will send test kits to all of you. We however need to receive the **proformat of your participation** for our own administrative purposes. Kindly send back the proformat **by 30th May** to: Giorgia Beldì (giorgia.beldi@ec.europa.eu)**.**

The samples will be sent to you in the beginning of June. You will receive by email the form "ILC03-04 2014 Elements test results.xls" for the compilation of the results. The deadline for submission of results is **21st July 2014.**

If you have any question, please contact Giorgia Beldì (giorgia.beldi@ec.europa.eu).

Sincerely yours,

Dr. Catherine Simoneau
Operating Manager, Community Reference Laboratory for Food Contact Materials
European Commission, DG-Joint Research Centre
Institute for Health and Consumer Protection
Unit Chemical Assessment and Testing, T.P. 260
Ispra Va 21020 Italy

Cc: P. Aguar (JRC), D. Rembges (JRC)
B. Schupp (SANCO)

Annex 2. Letter for confirmation of participation

EUROPEAN COMMISSION
GENERAL DIRECTORATE JRC
JOINT RESEARCH CENTRE
Institute for Health and Consumer Protection – IHCP
Unit Chemical Assessment and Testing

Ispra May 12th 2014

Participation to EURL-FCM ILC03-04 2014
Interlaboratory Comparison (ILC) Exercise
Elements from Food Contact Materials

CONFIRMATION OF PARTICIPATION

Your Name:	
Organization:	
Address:	
E-mail:	
Phone:	

Item	YES	NO
I have already the package with the files for filling the results and especially RingDat3.exe file from last year and I need only lab files for this year's ILC		
I don't have the package with the files for filling the results from last year		

Kindly send back this format to: Giorgia Beldì (giorgia.beldi@ec.europa.eu) **by the 30th of May 2014.**
The samples will be sent to you in the beginning of June. You will find additional information in the kit sent. The deadline for submission of results is **21st July 2014.**

Sincerely yours,

Catherine Simoneau

Annex 3. Shipping kit letter accompanying the sample TNX1 to ILC001 2013

EUROPEAN COMMISSION
GENERAL DIRECTORATE JRC
JOINT RESEARCH CENTRE
Institute for Health and Consumer Protection – IHCP
Unit Chemical Assessment and Testing

Ispra 12th June 2014

Shipping Kit ILC03-04 2014-Elements from Food Contact Materials

Samples

- **PL1** – 60 mL glass vials containing Acetic acid 3 % (w/v) spiked with the substances reported in table1
- **PL1- Blank of Acetic acid 3 % (w/v)**

- **C2** – 60 mL glass vials containing Acetic acid 4 % (w/v) spiked with the substances reported in table 2.
- **C2- Blank of Acetic acid 4 %** (v/v)

Table 1

First part: Quantification of Elements released from Food Plastic Materials	
Elements to be quantified	
Barium	Manganese
Cobalt	Zinc
Copper	Lithium
Iron	**Unknown Element**

Table 2

Second part: Quantification of Elements released from Ceramic	
Elements to be quantified	
Lead	Arsenic
Cadmium	Barium
Cobalt	Aluminium
Nickel	Manganese

Documents (sent by email)

- Letter of confirmation of receipt (Sample Receipt ILC03-04 2014.doc) ;
- Electronic excel file" ILC03-04 2014 Elements test results.xls".

Storage

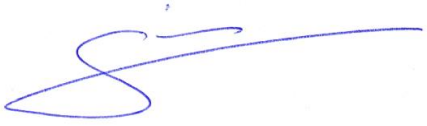
Samples PL1 and C2 should be kept in the fridge at + 4°C.

Instructions

Perform four replicates for the sample PL1 and C2 and report their concentration and the name of the unknown element using the unit of measure specified in the Excel form "ILC03-04 2014 Elements test results.xls".
Please send the form "ILC03-04 2014 Elements test results.xls" filled with your results by e-mail to Giorgia Beldì (giorgia.beldi@ec.europa.eu) **by July 21st 2014.**

If you have any question, please contact Giorgia Beldì (giorgia.beldi@ec.europa.eu) ph. +39.0332.789903

Sincerely yours,



Annex 4. Letter of confirmation of receipt of the sample

EUROPEAN COMMISSION
GENERAL DIRECTORATE JRC
JOINT RESEARCH CENTRE
Institute for Health and Consumer Protection – IHCP
Unit Chemical Assessment and Testing

Ispra May 12th 2014

Participation to Interlaboratory Comparison Exercise
- EURL-FCM ILC03-04 2014 on Elements from Food Contact Materials -

CONFIRMATION OF RECEIPT OF THE SAMPLES

Please return this form to confirm that the sample package has arrived. In case the package is damaged, please state this on the form and contact us immediately.

Your Name:	
Organization:	
E-mail:	
Phone:	

Any remarks …….

Date arrival package …….

Signature …….

Kindly send back this form to: Giorgia Beldì (giorgia.beldi@ec.europa.eu)

Sincerely yours,

Catherine Simoneau

Annex 5. Form for the compilation of the results

European Commission

FCM EURL ILC 03 2014 Elements from FCM

TEST RESULTS

Lab Code - LC00.....

EURL European Union Reference Laboratory for Food Contact Materials

First part: Quantification of Elements released from Plastic Materials into Acetic acid 3 % (w/v).

Concentration in the sample PL1 (µg/Kg)						
Sample code	Element name	Replicate 1	Replicate 2	Replicate 3	Replicate 4	Remark
	Barium					
	Cobalt					
	Copper					
	Iron					
	Manganese					
	Zinc					
	Lithium					
	.....(to be filled)					

Second part: Quantification of Elements released from Ceramic Materials into Acetic acid 4 % (w/v).

Concentration in the sample C2 (µg/Kg)						
Sample code	Element name	Replicate 1	Replicate 2	Replicate 3	Replicate 4	Remark
	Lead					
	Cadmium					
	Cobalt					
	Nickel					
	Arsenic					
	Barium					
	Aluminium					
	Manganese					

..... Place and date

..... Laboratory Manager

..... Signature

Annex 6a. Results of the Homogeneity study for sample PL1

Element	Mean [µg/Kg]	Mode s(target)	s(target) [%]	ISO 13528	Harmonized Protocol	Result of homogeneity analysis
BARIUM	692.65	HORWITZ corrected	16.91	OK	OK	According to ISO 13528, the heterogeneity standard deviation s(samples) between the samples of the test material is no more than one third of the target standard deviation hence the samples are suitable for the ring test. For the specified target standard deviation of 22.00% (Corrected Horwitz) the analytical precision of the method fulfils the requirements of the Harmonized Protocol. Also according to the Harmonized Protocol, no statistically significant heterogeneity of the samples can be detected.
COBALT	50.93		22.00	OK	OK	
COPPER	3069.67		13.51	OK	OK	
IRON	20083.35		10.18	OK	OK	
MANGANESE	492.15		17.80	OK	OK	
ZINC	19927.49		10.20	OK	OK	
LITHIUM	480.15		17.86	OK	OK	
ANTIMONY	35.46		22.00	OK	OK	

Annex 6b. Results of the Homogeneity study for sample C2

Element	Mean [µg/Kg]	Mode s(target)	s(target) [%]	ISO 13528	Harmonized Protocol	Result of homogeneity analysis
BARIUM	765.78	HORWITZ corrected	16.65	OK	OK	According to ISO 13528, the heterogeneity standard deviation s(samples) between the samples of the test material is no more than one third of the target standard deviation hence the samples are suitable for the ring test. For the specified target standard deviation (Corrected Horwitz) the analytical precision of the method fulfils the requirements of the Harmonized Protocol. Also according to the Harmonized Protocol, no statistically significant heterogeneity of the samples can be detected.
COBALT	48.90		22.00	OK	OK	
MANGANESE	387.32		18.45	OK	OK	
LEAD	10.00		22.00	OK	OK	
CADMIUM	4.58		22.00	OK	OK	
NICKEL	74.20		22.00	OK	OK	
ARSENIC	11.36		22.00	OK	OK	
ALUMINIUM	848.66		16.40	OK	OK	

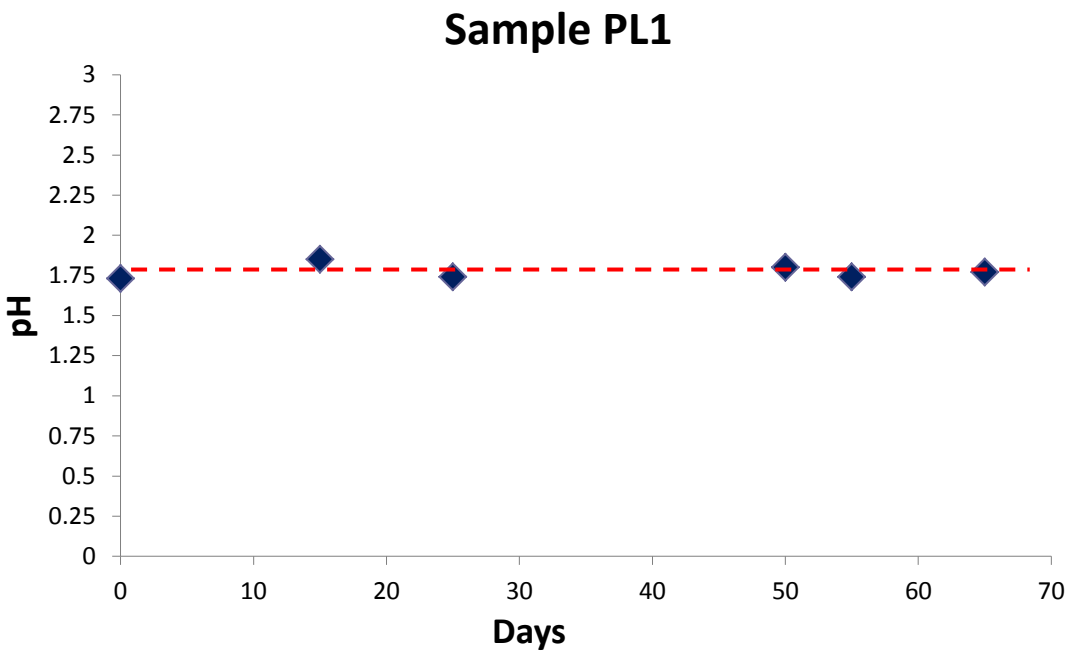
Annex 7a. Results of the stability study for sample PL1

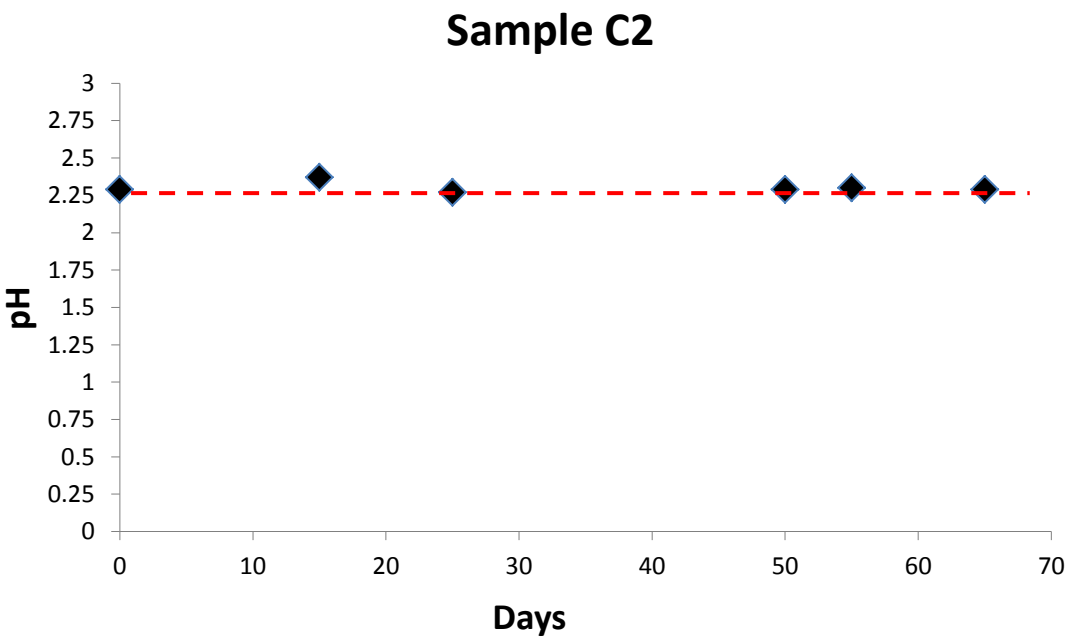
Sample PL1	Storage condition	Intercept (b0)	Slope (b1)	s(b1)	t(α=0.95,n-2)·s(b1)	|b1|< t(α=0.95,n-2)·s(b1)
Ba	40ºC	699.9152	-0.0940	0.5463	1.7387	OK
	20ºC	689.6054	0.0597	0.4490	1.4290	OK
	4ºC	682.2285	0.0610	0.3857	1.2274	OK
Co	40ºC	51.0246	-0.0265	0.0233	0.0741	OK
	20ºC	50.3069	-0.0095	0.0183	0.0581	OK
	4ºC	50.1654	-0.0170	0.0181	0.0577	OK
Cu	40ºC	3487.8424	-11.1536	6.3371	20.1675	OK
	20ºC	3501.3136	-11.3970	5.9919	19.0690	OK
	4ºC	3523.2042	-11.4868	5.4630	17.3857	OK
Fe	40ºC	20550.6695	-6.8468	10.8101	34.4024	OK
	20ºC	20568.5784	-7.2971	11.6557	37.0935	OK
	4ºC	20437.2128	-2.8195	11.4422	36.4143	OK
Mn	40ºC	445.0504	1.1915	0.9764	3.1073	OK
	20ºC	441.8793	1.2588	0.8882	2.8266	OK
	4ºC	443.6689	1.1148	0.8992	2.8616	OK
Zn	40ºC	20138.6200	-1.2229	4.8476	20.8577	OK
	20ºC	20141.6029	-0.9176	3.3145	14.2612	OK
	4ºC	19949.7171	4.9661	4.9018	21.0907	OK
Li	40ºC	487.8829	-0.1591	1.1887	3.7830	OK
	20ºC	477.3769	-0.0130	0.9111	2.8996	OK
	4ºC	472.7338	0.0950	0.7749	2.4662	OK
Sb	40ºC	121.1674	0.1486	0.1486	0.4730	OK
	20ºC	117.9146	0.0971	0.0736	0.2341	OK
	4ºC	116.8713	0.1071	0.0550	0.1749	OK

Annex 7b. Results of the stability study for sample C2

Sample C2	Storage condition	Intercept (b0)	Slope (b1)	s(b1)	t(α=0.95,n-2)·s(b1)	|b1|< t(α=0.95,n-2)·s(b1)
Ba	40ºC	841.7445	-1.0593	1.4653	4.6631	OK
	20ºC	840.2084	-1.0489	1.2164	3.8711	OK
	4ºC	838.2293	-0.9581	1.1970	3.8093	OK
Co	40ºC	49.6413	-0.0254	0.0404	0.1287	OK
	20ºC	49.2048	-0.0135	0.0298	0.0950	OK
	4ºC	49.7823	-0.0247	0.0334	0.1063	OK
Mn	40ºC	362.5431	0.6696	0.8604	2.7381	OK
	20ºC	358.6474	0.7490	0.7219	2.2974	OK
	4ºC	360.7886	0.6905	0.7871	2.5048	OK
Pb	40ºC	10.6792	-0.0162	0.0190	0.0605	OK
	20ºC	10.4894	-0.0116	0.0112	0.0356	OK
	4ºC	10.6018	-0.0134	0.0166	0.0529	OK
Cd	40ºC	5.2524	-0.0068	0.0078	0.0247	OK
	20ºC	5.1782	-0.0049	0.0039	0.0124	OK
	4ºC	5.2727	-0.0061	0.0069	0.0218	OK
Ni	40ºC	64.6393	0.0888	0.0518	0.1648	OK
	20ºC	63.7273	0.0976	0.0951	0.3026	OK
	4ºC	65.1545	0.0706	0.0510	0.1622	OK
As	40ºC	15.2049	-0.0474	0.0210	0.0667	
	20ºC	15.4281	-0.0045	0.0248	0.0789	
	4ºC	15.3823	-0.0416	0.0183	0.0582	
Al	40ºC	858.1493	-1.5313	0.6143	1.9551	OK
	20ºC	847.4966	-0.5654	0.5085	1.6182	OK
	4ºC	844.0634	-1.1212	0.6522	2.0757	OK

Annex 8. Results of the pH monitoring for sample PL1 and C2





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