European Commission

JRC TECHNICAL REPORTS

Report on the inter-laboratory comparison exercise organised by the European Union Reference Laboratory for Food Contact Materials

Determination of elements in acetic acid solutions and in migration from ceramic and glass tableware

Jakubowska N., Beldì G., Simoneau C. and Hoekstra E.

2017

EUR 28690 EN

Contact information
Name: Catherine Simoneau
Email: JRC-FCM@ec.europa.eu

JRC Science Hub
https://ec.europa.eu/jrc

JRC106715

EUR 28690 EN

PDF ISBN 978-92-79-70531-1 ISSN 1831-9424 doi:10.2760/573036

Luxembourg: Publications Office of the European Union, 2017

How to cite this report: Jakubowska N., Beldì G., Simoneau C., Hoekstra E., Report on the inter-laboratory comparison exercise organised by the European Union Reference Laboratory for Food Contact Materials - Determination of elements in acetic acid solutions and in migration from ceramic and glass tableware, EUR 28690 EN, doi:10.2760/573036

Table of contents

Acknowledgements

The authors would like to thank Industria Porcellane S.p.A (IPA) and the ARC INTERNATIONAL Group for providing the samples used in this inter-laboratory comparison exercise. Furthermore, the laboratories having participated in this exercise are kindly acknowledged (list below). Finally the authors thank Piotr Robouch for his critical review of this report.

National Reference Laboratories

Austria	Austrian Agency for Health and Food Safety (AGES) Institut für Lebensmittelsicherheit, Wien
Belgium	Institute of Public Health, ISSP-LP, Bruxelles
Bulgaria	National Centre of Public Health & Analysis , Sofia
Cyprus	State General Laboratory, Nicosia
Czech Republic	National Institute of Public Health, Praha 10
Croatia	Croatian Institute of Public Health, Zagreb, Croatia
Denmark	Danish Veterinary & Food Administration, Laboratory Århus, Lystrup
Estonia	Central Laboratory of Chemistry, Tallinn
Finland	Finnish Customs Laboratory, Espoo
France	Testing Department, Laboratoire National d'Essais, Trappes Cedex
France	SCL Laboratoire de Bordeaux-Pessac, Pessac
Germany	Bundesinstitut für Risikobewertung (BfR) (Federal Institute for Risk Assessment), Berlin
Greece	General Chemical State Laboratory, Laboratory of Articles and Materials in Contact with Foodstuffs, Athens
Hungary	National Food Chain Safety Office, Food and Feed Safety Directorate, Food Toxicological NRL, Budapest
Ireland	Public Analyst's Laboratory, Dublin
Italy	Istituto Superiore di Sanità, Labor Esposizione e rischio da materiali, Roma
Latvia	Institute of Food Safety, Animal Health and Environment (BIOR), Riga
Lithuania	National Public Health Surveillance Laboratory, Laboratory of Chemistry, Vilnius
Luxembourg	Laboratoire National de Santé, Division de Contrôle Alimentaires, Luxembourg (G.D. of Luxembourg)
Poland	National Institute of Public Health - National Institute of Hygiene, Warsaw
Portugal	ESB Escola Superior de Biotecnologia, Universidade Católica Portuguesa, Packaging Department, Porto
Romania	NRL for Food Contact Materials, National Institute of Public Health, Bucharest
Slovakia	Regional Public Health Authority Poprad (RUVZ), Poprad
Slovenia	Institute of Public Health Maribor, Laboratory in Ljubljana, Ljubljana
Spain	Centro Nacional de Alimentación, Agencia Espanola de Seguridad, Alimentaria y Nutrición (AESAN), Majadahonda-Madrid
Sweden	National Food Agency, Uppsala
United Kingdom	The Food and Environment Research Agency, York

Official Control Laboratories

Germany	Landeslabor Schleswig-Holstein, Neumünster
Germany	Bayerisches Landesamt für Gesundheit und Lebensmittelsicherheit, Erlangen
Germany	Chemisches- und Veterinäruntersuchungsamt Ostwestfalen-Lippe (CVUA-OWL) AöR, Detmold
Germany	LANDESUNTERSUCHUNGSAMT RLP, Koblenz
Germany	Landesamt für Verbraucherschutz (LAV) Abt. B3B, Saarbrücken
Germany	Landesuntersuchungsanstalt für das Gesundheit und Veterinär wesen , Sachsen
Germany	Dezernatsleiter Bedarfsgegenstände, kosmetische Mittel,Rückstände, Kontaminanten Landesamt für Verbraucherschutz Sachsen-Anhalt, Halle
Italy	IZS Lombardia Emilia Romagna- reparto chimico degli alimenti Bologna, Bologna
Italy	ARPAL dipartimento di Genova, Genova
Italy	Polo Alimenti - Arpa Trento, TN
Italy	ASL Provincia di Milano 1, Parabiago MI
Italy	ARPA Lazio di Roma, UO Alimenti, Droghe e Cosmetici, Roma
Italy	ARPA Friuli Venezia Giulia, Pordenone
Italy	ASL Varese
Italy	ARPA Puglia
Italy	ARPA Piemonte
Italy	Stazione Sperimentale del Vetro SSV, Murano
Spain	Laboratorio de Salud Pública de Alicante, Alicante
Spain	Laboratorio de Salud Pública de Burgos (Junta de Castilla y León), Paseo Burgos
Spain	Comunidad autonoma de la Rioja laboratorio regional
Spain	Instituto de Ciencias de la Salud, Junta Comunidades Castilla – La Mancha
Belgium	FAVV-lab FLVGG, Braemkasteelstraat 59 9050 Gentbrugge, Belgium
Belgium	IEM Liege
Belgium	Eurofins

Abstract

This report presents the outcome of an inter-laboratory comparison exercise (ILC) on the determination of selected metals in acetic acid solutions and the determination of migration of elements from ceramic and glass tableware. The exercise was organised by the European Union Reference Laboratory for Food Contact Materials (EURL-FCM) in 2016 to evaluate the enforceability of measures under discussion for the future revision(s) of ceramics Directive 84/500/EEC [1] and the implementation of provisions laid down in the Commission Regulation 10/2011 [2] for plastics.

In 2015 National Reference Laboratories (NRLs) requested the EURL-FCM to organise an ILC with the aim (i) to check the analytical abilities of participating laboratories to quantify Cu, Fe, Zn and Sb in a solution of acetic acid 3 % and Ba, Co, Mn, Pb, Cd and Al in a solution of acetic acid 4 %; (ii) to test the laboratories performance to carry out the migration test on ceramic and glass articles and (iii) to derive precision criteria, including repeatability and reproducibility for the release of elements from tableware.

Participation in this ILC was mandatory for the nominated NRLs, and open to Official Control Laboratories (OCLs) and other invited laboratories. A total of 53 participants from 27 countries registered to the exercise. Twenty-nine NRLs from 27 countries participated in this ILC and all of them reported results.

Laboratory results were rated using z-score in accordance with ISO 13528:2015 [3]. The target standard deviation for the ILC assessment (σ_{pt}), for all measurands was calculated based on previous ILC03/04 2014 exercises [4] for spiked solutions and using the robust reproducibility standard deviation for the migration exercise on tableware. Repeatability and reproducibility standard deviations for the quantification of elements in acetic acid 3 % and acetic acid 4 % (spiked solutions and leachates) were calculated using robust approaches [3, 5].

The outcome of this exercise was satisfactory. The rate of success was higher than 80% for the elements in all samples except for Sb in acetic acid 3 % (79%). No difference between the performance of NRLs and OCLs was observed. Since the exercise required the uncertainty estimation, an additional assessment was provided to each laboratory, indicating how reasonable their measurement uncertainty estimation was. Zeta-scores were systematically higher than z-scores, indicating that there is room for improvement in estimation of the measurement uncertainties.

1 Introduction

Millions of tests checking the quality of food contact materials are performed in Europe every year. The results of these tests influence important decisions, i.e. compliance or non-compliance with legislation, for industry and food safety control. Testing results shall therefore be reliable and comparable throughout the European Union. This can only be realised through harmonisation, validation and standardisation of test methods.

In 2004 Regulation (EC) No 882/2004 [6] on official feed and food controls established the JRC as European Union Reference Laboratory for Food Contact Materials (EURL-FCM). The prime role of our EURL-FCM is building confidence in the comparability of measurements of official control laboratories that test compliance of food contact materials in the European Union.

As stated in Regulation (EC) No 882/2004 one of the core duties of the European Union Reference Laboratories (EURLs) is to organise inter-laboratory comparison exercises (ILCs) for the benefit of staff of National Reference Laboratories (NRLs).

Elements can occur in food as results of a release from food contact materials and articles. Materials and articles in contact with foods fall under a specific legislation at the EU level to ensure the safety of the consumer while facilitating trade. For plastic Commission Regulation (EU) 10/2011 [2] in annex II sets restrictions for several elements while for ceramic Directive 84/500/EEC describes specific migration limits and basic rules for determining the migration of lead (Pb) and cadmium (Cd) [1]. In this context, the EURL-FCM organised the ILC01 2016.

The ILC01 2016 contained two exercises. The first one aimed at testing the ability to determine concentrations of elements in acetic acid solutions with reference to the plastic Regulation (EU) No 10/2011 [2]. The second one aimed at testing the ability to determine concentrations of elements in acetic acid solutions in support of the revision of the ceramic Directive 84/500/EEC [1] (lowering the release limits of Cd and Pb, extending the scope to include other elements and covering glass and crystal ware).

This report summarises the outcome of ILC01 2016.

2 Scope and aim

The organisation of the ILC01 2016 was requested by NRLs at the EURL-FCM Plenary held on June 30, 2015. The scope of the ILC was:

i. to assess the analytical capabilities of participating laboratories in determining Cu, Fe, Zn and Sb in a solution of acetic acid 3 %, as follow up to the ILC03 2014 of elements from plastics in Regulation 10/2011 [2];

ii. to evaluate the laboratory performance to determine the concentration of Ba, Co, Mn, Pb, Cd and Al in a solution of acetic acid 4 %, elements under discussion in future revisions of the ceramics legislation Directive 84/500/EEC [1];

iii. to test the laboratories' performance to carry out the migration test on decorated ceramic and glass articles and derive precision criteria, including repeatability and reproducibility for the migration of elements from tableware.

The assessment of the reported results was performed on the basis of requirements set in international standards and guidelines [3, 5 and 7].

3 Set up of the exercise

3.1 Time frame

ILC01 2016 was included in the EURL-FCM work program 2016 and was further approved by DG SANTE. Invitation letters were sent to NRLs (Annex 1) on June 9, 2016. Participating laboratories were invited to fill in a letter of Confirmation of their participation (Annex 1). Registration was opened till June 17, 2016. The samples were dispatched to the participants on June 17, 2016, together with the Shipping and Instructions form (Annex 2). The participants were asked to confirm the sample receipt and fill in the Sample receipt acknowledgment form (Annex 3). Participating laboratories were asked to report the results using the Results reporting form (Annex 4) and fill the Questionnaire (Annex 5). The deadline for reporting results was set to July 29, 2016.

3.2 Instructions to participants

Detailed instructions were given to participants in the shipping and instruction form mentioned above. Laboratories were asked to determine

(i) The concentrations of Pb, Cd, Co, Ba, Mn and Al in 4 % acetic acid spiked solution (S1);

(ii) The concentrations of Cu, Fe, Zn and Sb in 3 % acetic acid spiked solution (S2);

(iii) The release of Pb, Cd and Co in leachates from decorated glass bowls (GB) (after I, II and III migration); and

(iv) The release of Pb and Cd in leachates from decorated ceramic cups (CC) – for NRLs only (after I, II and III migration).

For S1 and S2 samples laboratories were asked to perform and report four independent measurement values for each element, the mean with the associated expanded measurement uncertainty, the coverage factor and the approach followed for uncertainty calculation.

Laboratories were asked to treat test materials (GB and CC) following the provided Standard Operating Procedure (Annex 6) and report results for samples with their respective suffix (A, B, C and D for Replicate 1, 2, 3 and 4, respectively). Participants were requested to report also the mean of the four replicates with the associated expanded measurement uncertainty, the coverage factor and to describe the approach used for the uncertainty calculation.

Participants were asked to follow their routine procedures for the analysis and to report results following the instruction given (e.g. number of significant figures).

Participants received an individual laboratory code to report their measurement results and to complete the related questionnaire. The questionnaire was used to extract relevant information related to sample preparation and analytical method used for quantification.

4 Test item

4.1 Preparation

A solution of acetic acid 4 % v/v (S1) was spiked with Ba, Co, Mn, Pb, Cd and Al, while a solution of acetic acid 3 % w/v (S2) was spiked with Cu, Fe, Zn and Sb. Both solutions were prepared by the EURL-FCM. After spiking and homogenisation, 100 mL portions of the spiked food simulants were subsampled.

As a first step a feasibility study was carried out on several ceramics and glass articles with the aim to identify a suitable material to conduct the ILC exercise. Based on this study, three hundred decorated ceramic cups (CC) were produced by IPA – Industria Porcellane S.p.A and three hundred glass bowls (GB) were provided by the ARC INTERNATIONAL Group.

The samples kit is described in Table 1.

Table 1. Sample kit for ILC01 2016

Name	Sample
S1	1 plastic bottle containing acetic acid 4 % (v/v) solution spiked with Pb, Cd, Co, Ba, Mn and Al
S2	1 plastic bottle containing acetic acid 3 % (w/v) solution spiked with Cu, Fe, Zn and Sb
GB	4 decorated glass bowls
CC	4 ceramics cups (for NRLs only)

4.2 Homogeneity and stability

The measurements for the homogeneity and stability studies were performed by EURL-FCM. The homogeneity was evaluated according to ISO 13528:2015 [3] using the PROlab Software [8].

Ten randomly selected test specimens of solutions S1 and S2 were analysed in duplicate using Inductively Coupled Plasma Mass Spectrometer (ICP-MS). As expected, the solutions proved to be adequately homogeneous for all the investigated measurands.

Ten sets of 2 glass bowls were randomly selected. Three consecutive migration tests with acetic acid 4 % v/v were performed and the concentrations of Pb, Cd and Co were determined using ICP-MS. The batch resulted adequately homogenous.

Ten sets of 2 ceramic cup articles were randomly selected. Three consecutive migration tests with acetic acid 4 % v/v were performed and the concentrations of Pb and Cd were determined for each leachate using ICP-MS. Since the homogeneity of the ceramic cups batch was not proven, the EURL-FCM run the preliminary migration for all 276 samples to group them into a homogeneous subpopulation with smaller relative standard deviation. The homogeneity test was performed on this subpopulation.

The results of the homogeneity studies are reported in Annex 7.

The stability study was conducted on randomly selected specimens for samples S1 and S2. Both samples were stored at 3 different temperature conditions (4 ºC, 20 °C and 40 ºC). The test samples were monitored for stability by the EURL-FCM for approximately 80 days to cover the period allowed for the exercise. The stability was evaluated as described in ISO GUIDE 35:2006 [7]. The test materials proved to be stable for all elements, at all the investigated temperatures and for 11 weeks.

The results of the stability study are reported in Annex 8.

5 Assigned values and their uncertainties

5.1 Assigned value (x_{pt}) and associated standard uncertainty ($u(x_{pt})$)

No reference values were available for the measurands of concern. The assigned values (x_{pt}) were then derived as the robust mean of the results reported by the participants, as described in ISO 13528:2015 [3]. The Hampel estimator was used and no outlier test was carried out.

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 "consensus" value, the corresponding standard uncertainty ($u(x_{pt})$) is estimated as:

$$u(x_{pt}) = \frac{1.25\ s^*}{\sqrt{p}}$$

where s* is the robust standard deviation of the results and p is the number of laboratories.

Such an $u(x_{pt})$ can be assumed to include the uncertainty contributions from inhomogeneity, transport and instability.

$u(x_{pt})$ may be considered as "negligible" when compared to the target standard deviation for the proficiency assessment (σ_{pt}) if:

$$u(x_{pt}) \leq 0.3\ \sigma_{pt}$$

The corresponding test item is then considered fit-for-purpose for the PT.

5.2 Standard deviation of the ILC assessment σ_{pt}

σ_{pt} determines the limits of satisfactory performance in an ILC. 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 may be an appropriate indicator of the best agreement that can be obtained between laboratories.

σ_{pt} for determination of elements in solutions S1 and S2 was set to 10 % or 15 % or 20 %, based on the experience of the previous round ILC03/04 2014 exercises carried out on the same measurands with comparable concentration levels.

In the absence of appropriate collaborative trial data on migration test and being aware of specific difficulties associated to the reproducibility of the release of elements from tableware, σ_{pt} was calculated using the robust reproducibility standard deviation for all measurands.

5.3 Kernel density plot

Kernel density plot (KDE-plot) is a way of presenting graphically the general distribution shape of a dataset. The KDE could be used additionally to identify possible multi-modality in the reported data set distribution. In certain cases, the results are not ''normally'' distributed or contain values giving rise to multiple distribution modes. Kernel density plots were computed by PROLab software based on the results reported by the participating laboratories [8].

6 Evaluation of results

6.1 Criteria for evaluation of laboratories performance

Individual laboratory performance was expressed in terms of z-scores in accordance with ISO 13528: 2015 [3]:

$$z_i = \frac{x_i - x_{pt}}{\sigma_{pt}}$$

where:

x_i is the average of measurement results reported by a participant (calculated by PROlab);
x_{pt} is the assigned value; and
σ_{pt} is the standard deviation for proficiency assessment.

The interpretation of the z-score is done according to ISO 17043:2010 [9]:

|z-score| ≤ 2	satisfactory performance;
2 < |z-score| < 3	questionable performance (warning signal);
|z-score| ≥ 3	unsatisfactory performance (action signal).

The z-score compares the participant's deviation from the assigned value with the target standard deviation for proficiency assessment σ_{pt} used as common quality criterion. σ_{pt} is defined by the ILC organiser as the maximum acceptable standard deviation.

6.2 Uncertainty evaluation

Laboratories that have to comply with ISO 17025:2005 [10], the standard for the competence of testing and calibration laboratories, are obliged to provide measurement results with uncertainties. The use of laboratory evaluations of uncertainties in performance evaluation has been common in proficiency testing schemes; The EURL-FCM has recognised the usefulness of asking laboratories to provide uncertainty estimates for their reported results. This can be useful even when the uncertainties are not used in scoring.

Uncertainty estimation is not trivial, therefore an additional assessment was provided to each laboratory reporting uncertainty, indicating how reasonable their measurement uncertainty estimation was. The uncertainty assessment was carried out according ISO 13528:2015 [3] based on a/b/c evaluation approach introduced by the IMEP® project [11]. The standard measurement uncertainty from the laboratory (u_i) is most likely to fall in a range between a minimum uncertainty (u_{min}), and a maximum allowed (u_{max}, case "a"). u_{min} is set to the standard uncertainty of the assigned value ($u(x_{pt})$) and u_{max} is set to the standard deviation accepted for the ILC assessment (σ_{pt}). If u_i is smaller than u_{min}, (case "b") the laboratory may have underestimated its measurement uncertainty. Such a statement has to be taken with care as each laboratory reported only measurement uncertainty, whereas the uncertainty associated with the assigned value

also includes contributions of homogeneity and stability of the test item. If those components are large, measurement uncertainties smaller than u_{min} are possible and plausible. If $u_i > u_{max}$, (case "c") the laboratory may have overestimated the measurement uncertainty [11].

The ζ-score can be useful to evaluate a participant's ability to have results be close to the assigned value within their claimed uncertainty. The ζ-score were calculated according to ISO 13528:2015 [3] as:

$$\zeta = \frac{x_i - x_{pt}}{\sqrt{u(x_{pt})^2 + u_i^2}}$$

where:

x_i is the average from measurement results reported by a participant (calculated by PROlab);
u_i is the standard uncertainty reported by a participant;
x_{pt} is the assigned value; and
$u(x_{pt})$ is the standard uncertainty of the assigned value.

The denominator is the combined uncertainty of the assigned value and the measurement uncertainty as stated by the laboratory. The standard measurement uncertainty of the laboratory (u_i) was obtained by dividing the reported expanded uncertainty by the reported coverage factor, k. When no uncertainty was reported, it was set to zero ($u_i = 0$). When k was not specified, u_i was then calculated by dividing by √3, as recommended by Eurachem and CITAC [12].

The ζ-score includes all parts of a measurement result, namely the assigned value, its measurement uncertainty and the reported result as well as the uncertainty of the reported values. ζ-score may be interpreted using the same critical values of 2 and 3 as for z-scores. |ζ-score|>3 may indicate either a large deviation of participant results from assigned value an under-estimate of measurement uncertainty or combination of both. ζ-score used alone can be interpreted only as a test of whether the participant uncertainty is consistent with observed deviation and cannot be interpreted as an indication of fitness for purpose of a participant results. ζ-score is used in conjunction with z-scores. If a participant repeatedly obtains z-scores ≥ 3 but ζ-score < 2 this demonstrates that the participants may have assessed the uncertainty of their results accurately but that their results do not met the performance expected for the ILC. If a participant repeatedly obtains z-scores < 2 but ζ-score ≥ 3, it implies that the participant's uncertainty evaluation does not include all insignificant sources of uncertainty [3].

6.3 Laboratory results and scorings

In total 53 laboratories registered to ILC01 2016 Migration of elements from tableware of which 53 (27 countries) submitted results and 50 of them answered the associated questionnaire. Twenty-nine NRLs from 27 countries participated in this ILC and all of them reported results.

53 participating laboratories received spiked acetic acid solutions S1, S2 and decorated glass bowls. Due to the limited number of ceramic cups available, these articles were reserved for NRLs only. The number of reported results or "less than" values is presented in Table 2 for the various analytes and sample types.

As requested, most of the laboratories reported four replicate results under repeatability conditions. The following analytical techniques were used for the determination of elements by the laboratories for this exercise: Inductively Coupled Plasma Mass Spectrometer (ICP-MS), Inductively Coupled Plasma Optical Emission Spectrometry

(ICP-OES), Graphite Furnace Atomic Absorption Spectrometry (GF-AAS) and Flame Atomic Absorption Spectrometry (F-AAS). Most of the results were obtained by ICP-MS. No direct correlation could be found between the analytical methods used by the participants and the quality of their reported results.

A summary of the statistical data such as assigned value (x_{pt}), uncertainty of the assigned value $u(x_{pt})$, target standard deviation (σ_{pt}), relative repeatability standard deviation (RSD_r) and the relative reproducibility standard deviation (RSD_R) are given in Table 2 for samples S1, S2, GB and CC. Annexes 9 present the reported results as tables and graphs. The graphs include assigned value (x_{pt}), reference interval ($x_{pt} \pm U(x_{pt})$), target interval ($x_{pt} \pm 2\ \sigma_{pt}$), analytical technique used and the KDE-plot. The graphs were plotted using the PROLab software [6].

Table 2. Summary of results calculated according Q-Hampel (ISO 13528:2015). A total of 53 laboratories registered to analyse S1 & 2; GB I, II & III; while 30 NRLs registered for CC I, II & III.

Sample (tot. report. labs)	n° of report. lab	Reported "Less Than"	Element	x_{pt} [µg/kg]	$u(x_{pt})$ [%]	σ_{pt} [%]	RSD_r [%]	RSD_R [%]
S1 (AA 4 %)	43	0	Al	581	1.9	10	1.7	10
	41	0	Ba	497	1.2	10	1.0	5.8
	49	3	Cd	5.03	0.8	10	1.6	4.7
	45	1	Co	50.6	1.0	10	1.2	5.2
	47	3	Pb	9.64	1.8	15	2.7	11
	49	0	Mn	459	1.2	10	1.3	6.9
S2 (AA 3 %)	39	0	Sb	39.6	3.3	20	1.5	17
	47	0	Cu	2.51*	1.3	10	1.1	7.3
	46	0	Fe	14.7*	1.6	10	1.4	8.7
	46	0	Zn	18.0*	1.4	10	1.2	7.4
GB I	52	0	Cd	33.2	2.3	14	6.3	14
	43	1	Co	52.4	2.4	15	5.3	15
	52	0	Pb	569	2.7	16	6.2	16
GB II	46	5	Cd	3.83	5.5	29	5.8	29
	36	7	Co	3.23	5.9	29	5.3	29
	50	1	Pb	48.0	3.7	23	4.5	23
GB III	46	5	Cd	3.10	6.8	36	5.8	36
	36	7	Co	2.33	7.4	36	4.4	36
	49	2	Pb	35.5	5.5	30	5.7	30
CC I	26	4	Cd	2.98	8.5	43	19	43
	29	1	Pb	88.4	5.9	53	30	53
CC II	26	4	Cd	2.56	7.8	50	19	50
	29	1	Pb	78.9	9.1	54	26	54
CC III	26	4	Cd	2.31	8.5	51	24	51
	29	1	Pb	75.5	6.9	50	28	50

* mg/kg

For the samples S1 and S2 the robust mean derived from consensus value from participant results x_{pt} is in good agreement with the nominal value (gravimetric formulation, x_{form}):

$$\frac{x_{pt} - x_{form}}{\sqrt{u(x_{pt})^2 + u_{form}^2}} < 2$$

The overall performance of the participants according to z-scores is summarised in Table 3, Figure 1 and Annexes 10.

Table 3. Overview of laboratory performance for the different measurands (element & samples) for the NRLs and OCLs

		All	NRLs				OCLs			
Sample	Element	RR/LOQ	RR/LOQ	S	Q	U	RR/LOQ	S	Q	U
S1	Al	43/0	23	23 (100%)	0 (0%)	0 (0%)	20	16(80%)	2 (10%)	2 (10%)
	Ba	41/0	24	23 (96%)	0 (0%)	1 (4%)	17	17 (100%)	0 (0%)	0 (0%)
	Cd	49/3	26/3	26 (100%)	0 (0%)	0 (0%)	23	23 (100%)	0 (0%)	0 (0%)
	Co	45/1	25/1	25 (100%)	0 (0%)	0 (0%)	20	19 (95%)	0 (0%)	1 (5%)
	Pb	47/3	24/3	23 (96%)	0 (0%)	1 (4%)	23	23 (100%)	0 (0%)	0 (0%)
	Mn	49/0	27	27 (100%)	0 (0%)	0 (0%)	22	21 (95%)	1 (4%)	0 (0%)
S2	Sb	39/0	24	19 (79%)	0 (0%)	5 (21%)	15	14 (93%)	0 (0%)	1 (7%)
	Cu	47/0	27	26 (96%)	1 (4%)	0 (0%)	20	17 (85%)	2 (10%)	1 (5%)
	Fe	46/0	26	23 (88%)	3 (12%)	0 (0%)	20	18 (90%)	2 (10%)	0 (0%)
	Zn	46/0	27	26 (96%)	0 (0%)	1 (4%)	19	16 (84%)	1 (5%)	2 (11%)
GB I	Cd	52/0	29	25 (86%)	0 (0%)	4(14%)	23	22 (96%)	1 (4%)	0 (0%)
	Co	43/1	25/1	23 (92%)	0 (0%)	2 (8%)	18	17 (94%)	1 (6%)	0 (0%)
	Pb	52/0	29	25 (86%)	0 (0%)	4 (14%)	23	22 (96%)	1 (4%)	0 (0%)
GB II	Cd	46/5	25/4	21 (84%)	3 (12%)	1 (4%)	21/1	19 (90%)	1 (5%)	1 (5%)
	Co	36/7	21/4	20 (95%)	1 (5%)	0 (0%)	15/3	14 (93%)	1 (7%)	0 (0%)
	Pb	50/1	28/1	23 (82%)	5 (18%)	0 (0%)	22	20 (91%)	1 (5%)	1 (5%)
GB III	Cd	46/5	25/4	23 (92%)	1 (4%)	1 (4%)	21/1	17 (81%)	3 (14%)	1 (5%)
	Co	36/7	21/4	21 (100%)	0 (0%)	0 (0%)	15/3	13 (87%)	1 (7%)	1 (7%)
	Pb	49/2	27/2	25 (93%)	1 (4%)	1 (4%)	22	20 (91%)	1 (5%)	1 (5%)
CC I	Cd		26/4	22 (85%)	2 (8%)	2 (8%)				
	Pb		29/1	26 (90%)	1 (3%)	2 (7%)				
CC II	Cd		26/4	22 (88%)	3 (12%)	1 (4%)				
	Pb		29/1	26 (90%)	1 (3%)	2 (7%)				

- RR/LOQ for reported results/less than LOQ;
- S, Q and U for satisfactory, questionable and unsatisfactory results.

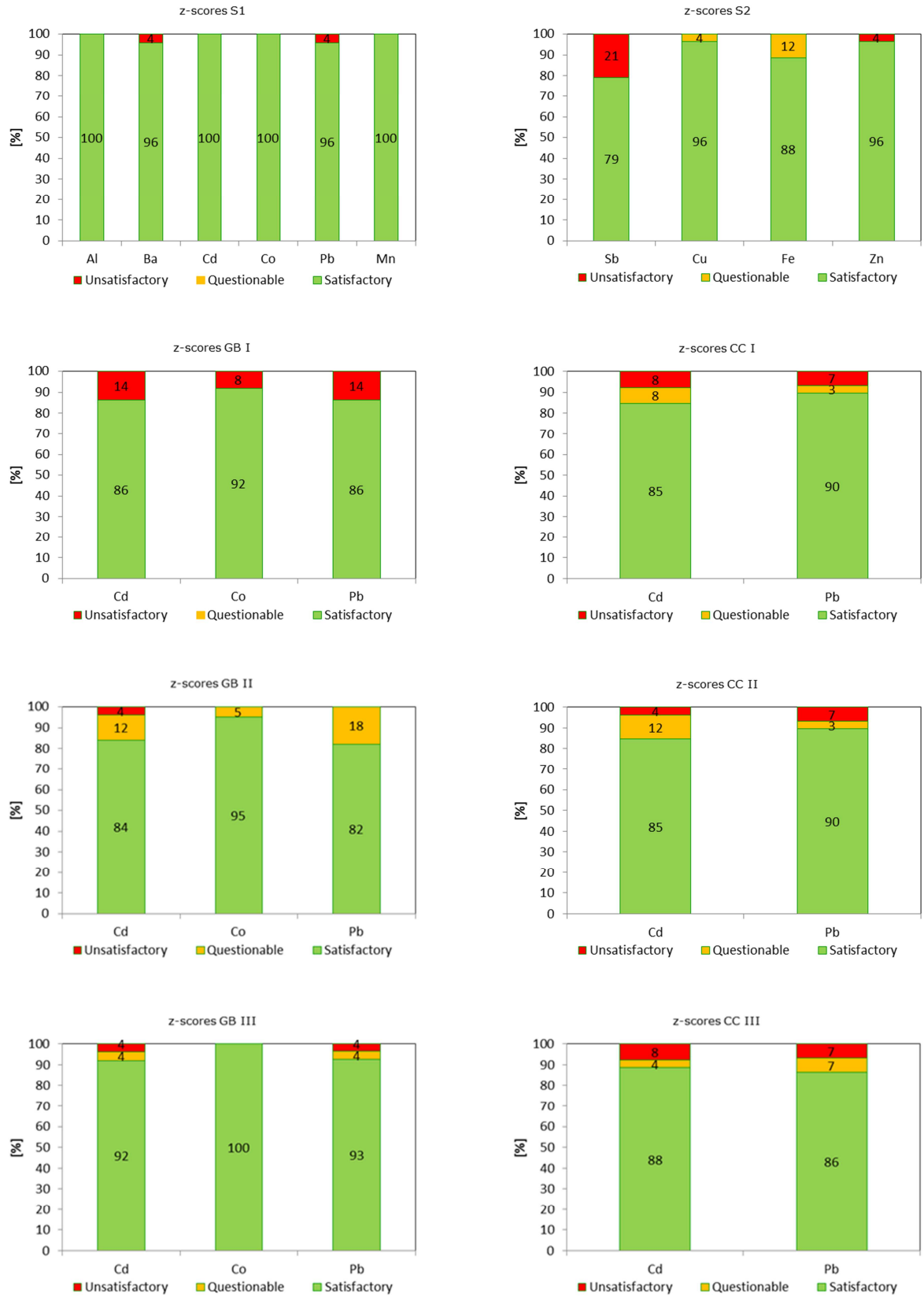

Figure 1. Percentages of NRLs with satisfactory, questionable and unsatisfactory performance

The outcome of this exercise was satisfactory. The rate of success was always higher than 79 % for all elements in all samples, even for the lower concentrations of Cd, Pb and Co of solution S1. None of NRLs got unsatisfactory results for solution S2 spiked with Cu and Fe. However Sb in S2 appeared to be more challenging and 5 NRLs and 1 OCL reported unsatisfactory results ($|z| > 3$).

ILC1 2016 introduced as a novelty the migration part with ceramic and glass items (CC and GB). Taking into account the difficulties of the migration exercise, for each mesurand the reproducibility standard deviation was set as the target standard deviation for the proficiency assessment ($\sigma_{pt} = S_R$), resulting in a satisfactory overall performance. No difference between the performance of NRLs and OCLs was observed.

The evaluation of the performance characteristics of a method is generally not an objective for a proficiency testing. However, it is possible to use the results of ILC to verify and establish the repeatability and reproducibility of a method considering that participants followed the same standard operating procedure (SOP) provided by the ILC organiser. The repeatability and reproducibility standard deviations (S_r and S_R) were calculated according ISO 5725-5 (algorithm A+S) and presented in Table 4. Test results were treated either together considering that analytical techniques used for the quantification are equivalent (based on previous exercise ILC03/04 2014) or using only the ICP-MS results (reported by the majority of laboratories). ICP-OES and AAS techniques were used by less than 12 participants (out of 53).

The results of the robust statistics indicated a good reproducibility for the determination of most of the elements in solutions S1 and S2 independently from the statistical approach used in the calculation. The relative reproducibility standard deviation (RSD_R) ranged between 4 % and 10 % for all elements in both solutions (up to 17 % for Sb).

RSD_R increased in the migration exercise. In the sample GB it started at 15 % in the first exposure to increase up to 40 % in the third migration, partly due to the decreasing of concentration levels through consecutive exposures. Similarly, for the ceramic cups the RSD_R of Pb was in the range of 30-50 % for a level of approximately 80 [µg/kg]. RSD_R for Cd were in the range of 40-70 % for approximately 3 [µg/kg].

Table 4. Repeatability and Reproducibility standard deviation calculated according ISO 5725-5 (algorithm A+S)

			All methods				ICP-MS only				
Sample	Element	N° lab	S_r [µg/kg]	RSD_r [%]	S_R [µg/kg]	RSD_R [%]	N° lab	S_r [µg/kg]	RSD_r [%]	S_R [µg/kg]	RSD_R [%]
AA4% (S1)	Al	43	10.4	1.8	54.5	9.4	29	10.3	1.8	45.5	7.8
	Ba	41	5.26	1.1	28.8	5.8	26	5.11	1.0	23.6	4.7
	Cd	49	0.08	1.7	0.22	4.3	31	0.08	1.6	0.18	3.6
	Co	45	0.65	1.3	2.45	4.8	31	0.62	1.2	2.06	4.0
	Pb	47	0.25	2.6	1.01	10	31	0.24	2.4	0.83	8.5
	Mn	49	5.81	1.3	28.7	6.2	30	6.06	1.3	24.1	5.2
AA3% (S2)	Sb	39	0.66	1.7	6.80	17	30	0.48	1.2	5.14	12
	Cu	47	0.03*	1.3	0.17*	6.6	29	0.03*	1.3	0.16*	6.4
	Fe	46	0.25*	1.7	1.26*	8.6	27	0.29*	1.9	0.98*	6.5
	Zn	46	0.24*	1.3	1.30*	7.2	28	0.26*	1.4	1.09*	6.0
GB I	Cd	52	2.15	6.5	4.50	14	32	2.05	6.1	5.15	15
	Co	43	3.23	6.2	7.24	14	29	3.51	6.5	6.26	12
	Pb	52	37.7	6.7	87.4	16	32	40.1	6.9	75.7	13
GB II	Cd	46	0.26	6.7	1.16	30	28	0.25	6.0	1.26	31
	Co	36	0.18	5.7	0.95	29	27	0.19	5.6	1.01	30
	Pb	50	2.84	5.9	9.89	20	31	2.63	5.3	8.58	17
GB III	Cd	46	0.20	6.2	1.29	41	28	0.18	5.7	1.27	41
	Co	36	0.13	5.5	0.88	38	27	0.12	5.1	0.84	37
	Pb	49	2.11	5.9	10.3	29	31	1.63	4.7	8.34	24
CC I	Cd	26	0.68	22	1.37	45	16	0.74	26	1.21	42
	Pb	29	28.9	32	35.9	39	18	30.1	33	30.1	33
CC II	Cd	26	0.57	22	1.36	52	16	0.60	25	1.58	67
	Pb	29	25.0	31	34.6	42	18	25.1	32	35.2	45
CC III	Cd	26	0.56	23	1.19	49	16	0.56	25	1.49	66
	Pb	29	22.8	29	27.8	36	18	22.8	32	24.6	34

* mg/kg

6.4 Uncertainty and ζ-scores

A total of 44 out of 53 participants (83 %) reported the measurement uncertainty as required in the instructions of ILC01 2016. The majority of the NRLs (23 out of 29; 79 %) and OCLs (21 out of 24; 87 %) reported measurement uncertainties.

The following approaches were used to evaluate measurement uncertainties:

- Based on replicates (precision);
- In house validation;
- Top down/ bottom up (In house validation);
- Horwitz/Thompson [13];
- Horwitz [14];
- Nordtest [15];
- GUM [16];
- EURACHEM / CITAC Guide CG4 [12];
- PT participation;
- ISO 11352:2012 [17].

The reported measurements uncertainties (MU) were evaluated as "realistic (when $u(x_{pt}) \leq u_i \leq \sigma_{pt}$); probably "underestimated" (when $u_i < u(x_{pt})$) or probably "overestimated" (when $u_i > \sigma_{pt}$).

Most of the participants reported realistic MUs for samples S1 and S2 (48 to 62 %). Underestimated MUs seem to be estimated from replicate measurements, while the overestimated MUs were derived from the Horwitz or Horwitz/Thompson equations.

Uncertainty estimation for the first migration of sample GB resulted similar to solution S1 and S2 with the majority of case "a". The uncertainties for samples GB (II & III migration) and for ceramic cups CC were more often underestimated compared to the solutions. Where contributions of homogeneity of the test item are large, as for GB and CC samples, the measurement uncertainty smaller than u_{min} case "b" are possible and plausible. Approaches used for uncertainty estimation and uncertainty assessment are reported in Annex 11.

In addition the uncertainty reported by participating laboratories was used together with the uncertainty of the assigned value to compute the ζ-scores. In all cases, the number of $|\zeta| < 2$ is lower that of $|z| < 2$. $|\zeta| > 3$ can either be caused by an inappropriate measurement of its estimation of measurement uncertainty or both. $|\zeta| > 3$ were often obtained when measurement uncertainty was not provided by participant because u_i was set to zero. All results are reported in Annex 9.

6.5 Outcome from questionnaire

Laboratories were asked to report details on analytical method, general experimental conditions, LODs of the methods that they used for the determination of the measurands and further details of the analytical technique applied such as isotopes or wavelength used for quantification (Annex 12). Fifty participants out of 53 answered the questions.

The majority of laboratories had a validated or accredited method for the quantification of Pb and Cd and eleven of which had extended the accreditation to other elements. All participants followed the instructions received to carry out the migration experiments. They applied different times between migration tests and between migration and analyses of leachates which did not have influence on their final results. Large discrepancies were observed in reported LODs even among laboratories using the same technique. No direct correlation could be found between the analytical methods used by the participants and the quality of their reported results. All details together with the isotopes or wavelength used for the measurands quantification are reported in the table 4 of Annex 12.

7 Conclusions

The ILC01 2016 was the first exercise of EURL-FCM which included the release of elements from decorated ceramic and glass articles. The aim of this ILC was to assess the ability of participating laboratories to quantify the elements in acetic acid solutions and to derive precision criteria, including repeatability and reproducibility for the release of elements from tableware. The involvement of the NRLs and the OCLs demonstrated a very satisfactory participation in the ILC.

The outcome of this exercise was satisfactory. The rate of success was almost always higher than 80 % for all elements in all samples. In solutions S1 even the lower concentrations of Cd, Pb and Co were satisfactorily quantified by all participants with success scores always being greater than 95 %. For solution S2 spiked with Cu and Fe none of the NRLs got unsatisfactory results. However the quantification of Sb in S2 was more difficult: five NRLs and one OCL had a lz-scorel higher than 3. For the migration exercise, the reproducibility standard deviation was set as target standard deviation to assess the z-scores and the overall performance was satisfactory. No difference between the performance of NRLs and OCLs was observed.

Repeatability and reproducibility standard deviations for the quantification of elements in acetic acid 3 % and acetic acid 4 % (spiked solutions and leachates) were calculated using robust approaches. For spiked solutions S1 and S2 the relative repeatability standard deviations were low generally less than 3 %. For the tableware the repeatability standard deviations which included the migration step were between 5 % for glass bowls and up to 30 % for ceramic cups.
Reproducibility standard deviations for spiked solutions were usually below 10 % apart from 17 % for Sb in sample S2 and can reach 35 % for glass bowls or 65 % for ceramic cups.

Since the exercise required the uncertainty estimation, an additional assessment was provided to each laboratory, indicating how reasonable their measurement uncertainty estimation was. For samples S1 and S2, the majority of the participating laboratories reported realistic measurement uncertainty which fall in a range between a minimum uncertainty $u(x_{pt})$, and a maximum allowed (ILC assessment σ_{pt}). The uncertainties for samples GB and for ceramic cups CC were more often underestimated compared to the solutions; this can be explained considering the contributions of homogeneity of the test item and the approach used for the estimation of uncertainty. Only few participants overestimated the uncertainty of the migration test. lζ-scoresl were systematically higher than z-scores. This underlines the need of an improvement in estimation of the measurement uncertainties.

This ILC confirmed the satisfactory performance of the NRLs and OCLs to determine elements in acetic acid solutions. The exercise also shows that NRLs and OCLs are prepared for lowering the release limits of Cd and Pb and adding additional elements and extending the Directive to glass and crystal ware.

References

[1] 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.

[2] 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.

[3] ISO 13528:2015, Statistical Methods for Use in Proficiency Testing by Inter-laboratory Comparisons.

[4] ILC03/04 2014, Elements from food contact materials. Report EUR 27167 EN.

[5] ISO 5725-5:1998/Cor.1:2005(en) Accuracy (trueness and precision) of measurement methods and results — Part 5: Alternative methods for the determination of the precision of a standard measurement method TECHNICAL CORRIGENDUM 1.

[6] 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.

[7] ISO GUIDE 35:2006; Reference materials–General and statistical principles for certification.

[8] PROLab Plus Version 2.14 – QuoData, Dresden (Germany) – www.quodata.de

[9] ISO 17043:2010, I., Conformity assessment - General requirements for proficiency testing. issued by ISO-Geneva (CH), International Organization for Standardization.

[10] ISO 17025:2005, General requirements for the competence of testing and calibration laboratories.

[11] The International Measurement Evaluation Programme (IMEP) https://ec.europa.eu/jrc/en/interlaboratory-comparisons/IMEP

[12] Eurachem/CITAC (2000), Quantifying Uncertainty in Analytical Measurement. http://www.eurachem.org

[13] M. Thompson, Analyst, (2000), 125, 385-386.

[14] Horwitz, W., Kamps, L.R., Boyer, K.W., J. Assoc. Off. Anal. Chem., (1980), 63; 1344.

[15] Nordtest TR 537, Handbook for calculation of measurement uncertainty in Environmental Laboratories, TR 537, (2012) Edition 3.1.

[16] JCGM 2008 (GUM), Evaluation of measurement data – Guide to the expression of uncertainty in measurement (2008).

[17] ISO 11352:2012, Water quality - Estimation of measurement uncertainty based on validation and quality control data.

List of abbreviations and definitions

AAS	Atomic Absorption Spectrometry
DG-SANTE	Directorate General for Health and Food Safety
F-AAS	Flame Atomic Absorption Spectrometry
GF-AAS	Graphite Furnace Atomic Absorption Spectrometry
CITAC	Co-operation for International Traceability in Analytical Chemistry
EU	European Union
EURACHEM	A focus for Analytical Chemistry in Europe
EURL-FCM	European Union Reference Laboratory for Food Contact Materials
GUM	Guide for the expression of Uncertainty in Measurement
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
ICP-OES	Inductively Coupled Plasma Optical Emission Spectroscopy
ILC	Inter Laboratory Comparison exercise
IMEP	International Measurement Evaluation Programme
JRC	Joint Research Centre
LOD	Limit of Detection
LOQ	Limit of Quantification
NRL	National Reference Laboratory
OCL	Official Control Laboratory
PT	Proficiency Test
SOP	Standard Operating Procedure
TR	Technical Report

List of figures

List of tables

Annex 1. Participation form

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

DETAILS OF THE INTERLABORATORY COMPARISON EXERCISE

ILC code	*ILC01 2016*
ILC Title	*Migration of elements from tableware*
Year	*2016*
Sample type	*- Decorated glass bowls (GB);* *- Ceramic cups (for NRLs) (CC);* *- Spiked solutions (S1, S2).*
Parameters for determination	*- Release of Pb, Cd and Co from decorated glass bowls (after I, II and III migration);* *- Release of Pb and Cd from decorated ceramic cups – for NRLs only (after I, II and III migration);* *- Concentrations of Pb, Cd, Co, Ba, Mn and Al from 4% acetic acid spiked solution;* *- Concentrations of Cu, Fe, Zn and Sb from 3% acetic acid spiked solution.*
Sample quantity	*- 4 decorated glass bowls (GB);* *- 4 ceramic cups (NRLs only) (CC);* *- 1 bottle of acetic acid 4% (v/v) solution spiked with Pb, Cd, Co, Ba, Mn and Al (S1);* *- 1 bottle of acetic acid 3%(v/v) solution spiked with Cu, Fe, Zn and Sb (S2).*
Packaging	*Cardboard box*
Shipment conditions	*Fragile items*
Sample dispatch	*20th June 2016*
Deadline for results	*29th July 2016*

PARTICIPATION DATA	
Organisation name:	
Laboratory:	
CONTACT INFORMATION	
Contact person:	
Address for sample dispatch:	
Telephone:	
Fax:	
e-mail:	

<JRC.I.1.Form.FIT-EURL.01 ver.1 _Participation Form >

Annex 2. Shipping and Instruction form

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

SHIPPING and INSTRUCTION FORM

ILC [*ILC01 2016* **]: [** *Migration of elements from tableware* **]**

Material/samples sent :
- 4 decorated glass bowls (GB)
- 4 ceramics cups (for NRLs only) (CC)
- 1 plastic bottle containing acetic acid 4% (v/v) solution spiked with Pb, Cd, Co, Ba, Mn and Al (S1)
- 1 plastic bottle containing acetic acid 3% (v/v) solution spiked with Cu, Fe, Zn and Sb (S2)
- Accompanying letter

Instructions :	
1)	*Sample S1 and S2 should be kept in the fridge at +4°C in the dark*
	Determine:
2)	*release of Pb, Cd and Co in leachates from decorated glass bowls GB (after I, II and III migration);*
3)	*release of Pb and Cd in leachates from decorated ceramic cups CC – for NRLs only (after I, II and III migration);*
4)	*concentrations of Pb, Cd, Co, Ba, Mn and Al in 4% acetic acid spiked solution (S1);*
5)	*concentrations of Cu, Fe, Zn and Sb in 3% acetic acid spiked solution (S2).*
6)	*Treat test materials (GB and CC) following the provided SOP (migration test and ICP-MS analysis). Other equivalent analytical method of analysis can be used. Both samples shall be consider as hollowware, fill them with acetic acid 4% (55mL for ceramic cups and 500 mL for decorated glass bowls).* *Perform three consecutive migration tests for all samples GB and CC as describe in provided SOP and report your results as specified in the Results Reporting Form (JRC.I.1.Form.FIT-EURL.04). Migration results for samples with suffix A in the code should be inserted in the form as Replicate 1, suffix B as Replicate 2, suffix C as Replicate 3 and suffix D as Replicate 4.*
7)	*Perform four replicates for the samples S1 and S2 and report concentrations of the elements as specified in the form (JRC.I.1.Form.FIT-EURL.04).*
8)	*Closing date: [29/07/2016]*
9)	*Please sent the JRC.I.1.Form.FIT-EURL.04 filled with your results by e-mail to Giorgia Beldì (giorgia.beldi@ec.europa.eu) or to Natalia Jakubowska (natalia.jakubowska@ec.europa.eu).*
10)	*If you have any question, please contact Giorgia Beldi (giorgia.beldi@ec.europa.eu), ph. +39 0332 78 9903 or Natalia Jakubowska (natalia.jakubowska@ec.europa.eu), ph. +39 0332 78 5507.*

<JRC.I.1.Form.FIT-EURL.02 ver.1_Shipping and Instruction Form>

Annex 3. Sample Receipt Acknowledgment form

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

Please complete the present form to acknowledge sample receipt and return it by fax or e-mail

TO

EUROPEAN UNION REFERENCE LABORATORY

***for* FOOD CONTACT MATERIALS**

FAX: + 39 0332 785707

e-mail: *giorgia.beldi@ec.europa.eu*

SAMPLE RECEIPT ACKNOWLEDGEMENT FORM

ILC [*ILC01 2016*]**:** [*Migration of elements from tableware*]

LABORATORY NAME:	[ ]
LABORATORY CODE:	[ ]
SAMPLE CODES:	GB[ ]
SAMPLE CODES:	CC[ ]
SAMPLE CODE:	S1[ ]
SAMPLE CODE:	S2[ ]
DATE OF RECEIPT:	[ ]
STATE OF SAMPLE:	[ ]

COMMENTS [ ]

Date [ ] Name/Signature [ ]

<JRC.I.1.Form.FIT-EURL.03 ver.1_Sample Receipt Acknowledgement Form>

Annex 4. Results reporting form

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

Use this form to submit your results by entering data in the space provided and return it by **[29TH JULY 2016]** by fax or e-mail

TO: EUROPEAN UNION REFERENCE LABORATORY *for* FOOD CONTACT MATERIALS

FAX: + 39 0332 785707 ; e-mail: giorgia.beldi@ec.europa.eu **or** natalia.jakubowska@ec.europa.eu

RESULTS REPORTING FORM - ILC [*ILC01 2016*]:

[*Migration of elements from tableware* **]**

LABORATORY CODE	

Reporting of results:

Perform three consecutive migration tests for all samples GB and CC.
Migration results for samples with suffix A in the code should be inserted in the form as Replicate 1, suffix B as Replicate 2, suffix C as Replicate 3 and suffix D as Replicate 4. Report also the mean of your four measurement results with associated expanded uncertainty, the coverage factor and the approach followed for uncertainty calculation.

Perform four replicates for the samples S1 and S2 and report:
- four independent concentrations values for each elements;
- the mean of your four measurement results with associated expanded uncertainty, the coverage factor and the approach followed for uncertainty calculation.

	CONCENTRATION IN THE GLASS BOWL - SAMPLE GB [µg / kg]						
ANALYTE	MIGRATION	REPLICATE 1*	REPLICATE 2*	REPLICATE 3*	REPLICATE 4*	AVERAGE	Expanded UNCERTAINTY
sample	GB	[sample code]A	[sample code]B	[sample code]C	[sample code]D	[µg / kg]	[µg / kg]
Pb	I						
	II						
	III						
Cd	I						
	II						
	III						
Co	I						
	II						
	III						
Coverage factor "k"							
Approach used for uncertainty calculation							

	CONCENTRATION IN THE SPIKED SOUTION - SAMPLE S1 [µg / kg]						
Sample	ANALYTE	REPLICATE 1*	REPLICATE 2*	REPLICATE 3*	REPLICATE 4*	AVERAGE	Expanded UNCERTAINTY
S1 [sample code]	Pb						
	Cd						
	Co						
	Ba						
	Mn						
	Al						
Coverage factor "k"							
Approach used for uncertainty calculation							

	CONCENTRATION IN THE SPIKED SOLUTION - SAMPLE S2 [µg / kg]						
Sample	ANALYTE	REPLICATE 1*	REPLICATE 2*	REPLICATE 3*	REPLICATE 4*	AVERAGE	Expanded UNCERTAINTY
S2 [sample code]	Cu						
	Fe						
	Zn						
	Sb						
Coverage factor "k"							
Approach used for uncertainty calculation							

		CONCENTRATION IN THE CERAMIC CUP - SAMPLE CC [µg / kg]					
ANALYTE	MIGRATION	REPLICATE 1*	REPLICATE 2*	REPLICATE 3*	REPLICATE 4*	AVERAGE	Expanded UNCERTAINTY
sample	CC	[sample code]A	[sample code]B	[sample code]C	[sample code]D	[µg / kg]	[µg / kg]
Pb	I						
	II						
	III						
Cd	I						
	II						
	III						
Coverage factor "k"							
Approach used for uncertainty calculation							

* [1] number of decimals e.g. 0.1

PLACE AND DATE	LABORATORY MANAGER	SIGNATURE

<JRC.I.1.Form.FIT-EURL.04.ver.1_Results reporting Form>

Annex 5. Questionnaire form

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

QUESTIONNAIRE FORM

ILC [*ILC01 2016* **]: [** *Migration of elements from tableware* **]**

Complete the form and return it by fax (+39 0332 785707) or e-mail

giorgia.beldi@ec.europa.eu **or** *natalia.jakubowska@ec.europa.eu*

LABORATORY CODE:	

METHOD DESCRIPTION

Did you follow provided SOP		YES [ ]	NO [ ]
Which analytical method did you use?			
Is the method validated? Please indicate the analytes it has been validated for			
Migration of Pb from ceramics/glass	Migration of Cd from ceramics/glass	Migration of other elements	
Is the method accredited? If YES, indicate the analytes it is accredited for			
Migration of Pb from ceramics/glass	Migration of Cd from ceramics/glass	Migration of other elements	

EXPERIMENTAL PART

Amount of sample (S1,S2) or leachates (GB, CC) used for analysis (mL) :	
Dilution factor used :	
Migration test applied (please specify all the conditions used):	
Time between the preparation of the sample and sample analysis (days)	
Time between migrations	
Did you apply any special treatment to the samples provided? This describe in the SOP or different?	

Please provide your analytical method details?			
	Technique followed	LOD [µg/kg]	Isotope used (ICP-MS) or Wavelenght [nm] (ICP-OES)
Pb			
Cd			
Co			
Ba			
Mn			
Al			
Cu			
Fe			
Zn			
Sb			
Did you encounter any problems with sample analysis? If YES, please specify			
Other Comments			

<JRC.I.1.Form.FIT-EURL.05 ver.1_Questionnaire Form>

Annex 6. Standard operating procedure

Standard Operating Procedure

Determination of Elements migrated from Tableware into acetic acid 4% (v/v)

8 SCOPE AND FIELD OF APPLICATION

This procedure describes a test method for the release of Al, Ba, Cd, Co, Cu, Fe, Mn, Pb, Sb and Zn from ceramic and glassware intended to be in contact with food.

9 NORMATIVE REFERENCES

ISO 17294-1:2005, Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) for the determination of elements — Part 1: General guidelines and basic principles

ISO 17294-2005, Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) for the determination of elements — Part 2: Determination of 62 elements

ISO 3696:1987, Water for analytical laboratory use - Specification and test methods

ISO 3585:1998, Borosilicate glass 3.3 — Properties

ISO 1042:1998, Laboratory glassware — One-mark volumetric flasks

ISO 648:1977, Laboratory glassware — One-mark pipettes

10 TERMS AND DEFINITIONS

3.1 Analytical blank
Value determined by a blank sample covering the complete analytical procedure including extraction, clean-up.

3.2 Blank calibration solution
Solution prepared in the same way as the calibration solution, but leaving out the analyte.

3.3 Calibration solution
Solution used to calibrate the instrument, prepared from stock solutions or from a certified standard.

3.4 Check calibration solution

Solution of known composition within the range of the calibration solutions.

3.5 Detection limit
The limit of detection is expressed as the mean analytical blank value plus three times the standard deviation of the analytical blank.

3.6 Extraction solution
Acetic acid 4% (v/v) used for migration test and analysed for Pb, Cd and other elements concentration.

3.6 GF-AAS
Atomic Absorption Spectrometry with Graphite Furnace.

3.7 ICP-MS
Inductively Coupled Plasma Mass Spectrometry.

3.7 ICP-OES
Inductively Coupled Plasma Optical Emission Spectrometry.

3.8 Instrument detection limit
Smallest concentration that can be detected with a defined statistical probability using a contaminant-free instrument and blank calibration solution.

3.9 Quantification limit
Limit above which a quantification of the measurands is possible, expressed as the mean analytical blank value plus, either, five to ten times the standard deviation of the analytical blank.

11 PRINCIPLE

Ceramic and glass articles are exposed to food simulant, 4 % (v/v) acetic acid solution for 24 hours (±0.5h) at 22 ± 2°C to extract present elements. The migration procedure is repeated three times. The amounts of extracted elements are determined by ICP-MS. Other equivalent analytical technique can be used (e.g. GF-AAS, ICP-OES).

12 REAGENTS AND MATERIALS

5.1 Reagents
All reagents shall be of recognised analytical grade.

5.1.1 Distilled water or water of equivalent purity (grade 3 water complying with the requirements of ISO 3696) shall be used throughout.

5.1.2 Acetic acid, (CH_3COOH), glacial, ρ = 1.05 g/ml, CAS 64-19-7.

5.1.3 Acetic acid test solution, 4 % (v/v) solution
Add 40 ml of acetic acid (5.1.2) to distilled water (5.1.1) and fill to 1 litre. This solution shall be freshly prepared for use. Proportionately greater quantities may be prepared.

5.1.4 Nitric acid, *r*(HNO_3) = 1.4 g/ml.
NOTE: Nitric acid is available both as:
r(HNO_3) = 1.40 g/ml equivalent to *w*(HNO_3) = 650 g/kg;
r(HNO_3) = 1.42 g/mlequivalent to *w*(HNO_3) = 690 g/kg.
Both are suitable for use in this method.

5.1.5 Elements stock solution
ρ (Al, Ba, Cd, Co, Cu, Fe, Mn, Pb, Sb and Zn) = 1000 mg/l

Single-element stock solutions and multi-element stock solutions with adequate specification stating the acid used and the preparation technique are commercially available. For example element stock solutions with concentrations of the analytes of 1000 mg/l are suitable. These solutions are considered to be stable for more than one year, but in reference to guaranteed stability, the recommendations of the manufacturer should be considered.

5.1.6 Standard solutions
ρ (Al, Ba, Cd, Co, Cu, Fe, Mn, Pb, Sb and Zn) = 10 mg/l

Pipette 10 ml of elements stock solutions of 1000 mg/l (5.1.5) separately or together, if suitable, into a 1000 ml glass volumetric flask. Add 10 ml of nitric acid (5.1.4). Bring to volume with water (5.1.1) and transfer to a suitable storage bottle. This solution can be prepared also gravimetrically.
Elements standard solutions are considered to be stable for several months, if stored in the dark. This does not apply to elements and multi-element standard solutions that are prone to hydrolysis, in particular solutions of Mo, Sn, Sb and Zr. In reference to guaranteed stability of all standard solutions, see the recommendations of the manufacturer.

5.1.7 Intermediate standard solutions
ρ (Al, Ba, Cd, Co, Cu, Fe, Mn, Pb, Sb and Zn) = 1 mg/l

Pipette 10 ml of elements standard solutions of 10 mg/l (5.1.6) separately or together, if suitable, into a 100 ml glass volumetric flask. Bring to volume with water (5.1.1) and transfer to a suitable bottle. Prepare the intermediate standard solutions freshly before each use.

5.1.8 Internal standard solution (reference element solution)
The choice of elements for the reference-element solution depends on the analytical problem. Solutions of these elements should cover the mass range of interest. Generally an internal standard should be no more than 50 amu removed from the analyte. The concentrations of these elements in the sample should be negligibly low.
The elements for example ^{45}Sc, ^{85}Y, ^{103}Rh, ^{165}Ho and ^{187}Re can be suitable for this purpose.
Internal standard solution, which contain one or more of proposed elements may be used: ρ (Sc, Y, Rh, Ho and Re) = 5 mg/l.
Pipette 5 ml of each element stock solution (1000 mg/L of each Sc, Y, Rh, Ho and/or Re) into a 1000 ml volumetric flask. Add 10 ml of nitric acid (6.1.4). Bring to volume with water and transfer to a suitable storage bottle.
A suitable concentration range of the internal standard in samples and calibration solutions is 10 - 100 µg/l.

5.1.9 Calibration solutions
Prepare the calibration solution(s) that cover the required working range by diluting the element standard solutions (5.1.6) or intermediate element standard solutions (5.1.7). Add an adequate volume of acetic acid 4 % (v/v) (6.1.3) to make the composition of the calibration solutions equal to

the composition of the test sample solutions to minimise the matrix effect. If necessary, add internal standard solution (5.1.8) to a concentration of for example 10 µg/l or 100 µg/l of the reference elements before bringing up to volume.

5.1.10 Calibration blank solution
The calibration blank solution is prepared in the same way as the calibration solutions, but leaving out the analytes. Prepare the calibration blank solution by adding an adequate volume of acetic acid 4 % (v/v) (5.1.3) to make the composition of the calibration blank solutions equal to the composition of the test sample. If necessary, add internal standard solution (5.1.8) to a concentration of for example 10 µg/l or 100 µg/l of the reference elements before bringing up to volume.

5.1.11 Initial calibration verification solution
The calibration verification solution is prepared by combining elements of interest from a standard source different from that of the calibration standard, and at concentration near the midpoint of the calibration curve. This standard may also be purchased. The solution should be prepared in the same acid composition (matrix) of the calibrations and the test samples.

5.1.12 Continuing calibration verification solution
The continuing calibration verification standard solution should be prepared combining metals of interest from the same standards used for calibration, at a concentration near the mid-point of the calibration curve. The solution should be prepared in the same acid composition (matrix) of the calibrations and the test samples.

5.1.13 Interference check solution
The interference check solution (ICS) is prepared to contain known concentrations of interfering elements (see paragraph 6 of ISO 17294-1) that will demonstrate the magnitude of interferences and provide an adequate test of any corrections, for example Molybdenum serves (if not presented in the samples) to indicate oxide effects on cadmium isotopes. The other components are present to evaluate the ability of the measurement system to correct for various molecular-ion isobaric interferences. The ICS is used to verify that the interference levels are corrected by the data system within quality control limits. These solutions must be prepared from ultrapure reagents or they can be obtained commercially.

5.1.14 Optimisation solution
The optimisation solution, commercially available, serves for mass calibration and for optimization of the ICP-MS apparatus conditions, for example adjustment of maximal sensitivity with respect to minimal oxide formation rate and minimal formation of doubly charged ions. It should contain elements covering the entire mass range, as well as elements prone to a high oxide formation rate or to the formation of doubly charged ions.

5.2 MATERIALS

5.2.1 Washing agent, commercially available non-acidic manual dishwashing detergent in dilution recommended by a manufacturer.

13 APPARATUS

6.1 Inductively coupled plasma mass spectrometer

ICP-MS system includes:

- Sample introduction system (pump, nebuliser, spray chamber);
- Inductively coupled plasma (radio-frequency generator, load coil, torch);
- Quadrupole or time-of-flight mass spectrometer, capable of scanning a mass range from 5 m/z (amu) to 240 m/z (AMU) with a resolution of at least 1 m/z peak width at 5 % of peak height, or sector field mass spectrometer;
- Collision/reaction cell that can be pressurised with helium and kinetic energy discrimination for polyatomic interference attenuation;
- Process control and data processing equipment;
- Argon gas supply - high purity grade, i.e. > 99.99 %;
- Hellium for collision cell – Ultra high purity grade, i.e > 99.999 %;
- Optional autosampler or additional (peristaltic) pump.

Principle

Determination of elements by inductively coupled plasma mass spectrometry (ICP-MS) consists of the following steps:

1. Introduction of a measuring solution into radiofrequency plasma to cause dissolution, atomization and ionization of elements;
2. Extraction of the ions from plasma through a differentially pumped vacuum interface and separation on the basis of their mass-to-charge ratio by a mass spectrometer;
3. Transmission of the ions through the mass separation unit and detection, usually by a continuous dynode electron multiplier assembly, and ion information processing by a data handling system;
4. Quantitative determination after calibration with suitable calibration solutions.

The relationship between signal intensity and mass concentration is usually a linear one over at least five orders of magnitude. For more details refer to ISO 17294-1:2005.

Interferences

It is important underline that when using ICP-MS, the presence of concomitant elements in the sample can cause interferences, for instance systematic errors in the measurement of the signal. Interferences are classified into spectral and non-spectral interferences.

The components that can cause spectral interferences are the following:

1. An isotope of another element having the same nominal mass-to charge-ratio as the analyte isotope, for example ^{58}Ni (analyte) and ^{58}Fe (interferant). Isobaric interferences may be corrected using the abundance of a different isotope of the interfering element. However correction options are often included in the instrument software. The isotope for measurements can usually be chosen free from isobaric interferences.

2. Polyatomic or molecular and doubly charged ion interferences. In many cases this ions contain argon (plasma gas) and/or oxygen originating from the water of the solution aspirated, for example ^{56}ArO(interferant) and ^{56}Fe (analyte).

3. Doubly charged ions for instance Ba^{2+} interference with ^{65}Cu, ^{66}Zn, ^{67}Zn and ^{68}Zn.

Significant molecular and doubly charged interferences shall be corrected using elemental equations (e.g. ^{58}Ni = - 0.04825 ^{54}Fe), a collision or a reaction cell or other possible elimination strategies given in ISO 17294-1 paragraph 6.

Non spectral physical interferences are associated with the sample nebulisation and transport processes as well as with ion-transmission efficiencies. Nebulisation and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause relevant signal suppression or enchantement. Dissolved solids can deposit on the nebuliser tip of a pneumatic nebuliser and on the interface skimmers. Total solid levels below 0.2 % (2.000 mg/l) are recommended to minimise solid deposition. An internal standard can be used to correct for physical interferences, if it is carefully matched to the measurement element so that the two elements are similarly affected by matrix changes. Dilution of the sample fivefold will usually eliminate the problem.

Detailed information on spectral and non-spectral interferences are given in ISO 17294-1 paragraph 6.

6.2 Accessories

6.2.1 Assorted glassware, as required, made of borosilicate glass as specified in ISO 3585. Immediately before use, all glassware used to prepare stock and standard solutions should be washed thoroughly with warm diluted nitric acid e.g. w(HNO_3) = 10 %, and then rinsed several times with distilled water (5.1.1).

6.2.2 Covers, for the articles under test, e.g. plates, watch glasses, Petri dishes of various sizes all of which shall be opaque if a darkroom is not available.

6.2.3 One-mark pipettes of capacities 10 ml and 100 ml, complying with ISO 648, class B or better plus other sizes as required.

6.2.4 One-mark volumetric flasks of capacities 100 ml and 1 000 ml, complying with ISO 1042, class B or better plus other sizes as required.

6.2.5 Precision piston pipettes, with a fixed stroke, typically 1 000 ml and 500 ml.

6.2.6 Plastic container, the stability of test samples and calibration solutions depends to a high degree on the container material. The material shall be checked according to the specific purpose. For the determination of metals in acetic acid 4 % (v/v) leachates, high density polyethene (HDPE) or polytetrafluoroethene (PTFE) containers (e.g. falcon tubes and storage bottles) are allowed.

14 SAMPLING

7.1 Sample size

In no case shall less than four ceramic or glass items be measured. Each of the articles shall be identical in size, shape, colour and decoration.

7.2 Preparation of test samples

Ceramics and glassware samples shall be cleaned with non-acidic diluted detergent (5.2.1) and tap water at approximately 40°C. Rinse it than with distilled or Milli-Q water. Dry the samples using filter paper or in a drying oven. Do not handle the surface to be tested after cleaning.

15 PROCEDURE

8.1 EXTRACTION

Conduct the migration test at a room temperature of 22 ± 2°C in the dark using an incubator.

Treat the ceramic and glass samples used in ILC01 2016 as hollowware articles. Fill each specimen with 4 % (v/v) acetic acid test solution. Use 55 ml of simulant for ceramic cup articles (CC) and 500 ml for decorated glass bowls (GB). Cover the specimen. Leach for 24 hours ± 30 minutes. After the first migration (I), wash the samples with distilled water and dry them. Refill all specimens with fresh simulant and incubate for the second migration (II). Follow the same procedure to conduct the third migration experiment (III).

Prior to sampling, mix the extraction solution by stirring or another appropriate method that avoids loss of the extraction solution or abrasion of the surface. Remove sufficient amount of the extraction solution with pipette and transfer it to a suitable storage container (6.2.7).

Analyse the extraction solution as soon as possible.

8.2 ICP-MS ANALYSIS

The amounts of extracted elements are determined by ICP-MS or by other equivalent analytical technique (e.g. GF-AAS, ICP-OES).

8.2.1 Instrument set up

Adjust the instrumental parameters of the ICP-MS system in accordance with the manufacturer's manual.
Wait at least 30 min to stabilise the plasma and adjust the instrument to working condition.
For guidance consult ISO 17294-1. For the selection of suitable isotopes refer to paragraph 6 of ISO 17294-1.
Use the recommended optimisation solution (6.1.14) to optimise or check the sensitivity and the stability of the system. Check the resolution and the mass calibration as often as required by the manufacturer. Define the relative atomic masses and the corresponding corrections. Define take-up and rinsing times to avoid memory effects.
Both standard mode or spectrum helium mode (KED - kinetic energy discrimination) are acceptable. KED mode should be used for elements with more interferences.

The use of an internal standard is recommended. Add the internal standard solution (6.1.8) to the interference check solution (6.1.13), to calibration solutions (6.1.9), to the blank calibration solutions (6.1.10) and to all test portions of samples before the analysis or add the internal standard solution

(6.1.8) on-line using two channel sample-introduction pump. The mass concentration of the reference elements shall be the same in all solutions.

8.2.2 LOD and LOQ determination

LOD can be calculated from the standard deviation of the blank. It is expressed as the mean analytical blank value (x_{bl}) plus three times the standard deviation of the analytical blank (sd_{bl}).
For the calculation of LOD, 10 determinations of the blank samples are analysed according to the same analytical method and their standard deviation is calculated. The LOD of the whole method is calculated as: LOD $= x_{bl} + 3\ sd_{bl}$, where x_{bl} is the means concentration calculates from the counts of the noise peak for the 10 determinations; sd_{bl} is the standard deviation of the analysis.

LOQ, the limit above which a quantification of the elements is possible, is expressed as the mean analytical blank value plus, either, five to ten times the standard deviation of the analytical blank.

$$LOQ = x_{bl} + F\ sd_{bl}$$

The factor F depends to the accepted measurement uncertainty.

8.2.3 Calibration
When the analytical system is first evaluated, establish a calibration curve for the elements of interest using at least five measuring points (for example, the blank calibration solution (5.1.10) and four calibration solutions (5.1.9) over a linear range. The calibration range should encompass the elements concentrations of the sample.
The working range in general may cover the range of 0.2 µg/l to 200 µg/L or a part of this.
For work on a daily basis, one blank solution (5.1.10) and one to two calibration solutions (6.1.9) are enough to set up a calibration graph, but check the validity of the calibration curve with a certified reference sample, a standard sample, or a suitable internal control sample.
For more details refer to ISO 17294-1 paragraph 9.
Linear regression correlation coefficient (r) must be ≥ 0.998. If the correlation coefficient is < 0.998, repeat calibration.

8.3 Determination of Al, Ba, Cd, Co, Cu, Fe, Mn, Pb, Sb and Zn
After establishing the calibration curves, measure the blanks and the interference check solution to establish interference correction or to check presence of interferences. Run the test samples and if the metals concentrations of the extraction solutions are found to be higher than the highest calibration point, dilute suitable aliquot portions to reduce concentrations within the working range with test solutions (5.1.3) or water (5.1.1) to have the same acidity composition of the calibration curve.
Within sufficient small intervals (for example, every 25 samples or less and at the beginning and end of the sample run) check the accuracy of at least one certified reference sample or one standard sample or a suitable internal control sample. If necessary, re-calibrate.

16 EXPRESSION OF RESULTS

State as many significant figures as are acceptable according to the precision of the measuring values.

17 QUALITY CONTROL

10.1 Blank

Result of the calibration blank check shall be within 3 times the instrumental detection limit.

10.2 Calibration verification and drift

Result of the initial and continuing calibration verification solutions shall not deviate more than 10 %.

10.3 Internal standard abundance

Internal standard shell not deviate more than 20 %.

10.4 Interference

The impact on the measured value of uncorrected isobaric, molecular and doubly charged interferences shall not be higher than 5 % or three times the instrumental detection limit. Successive values of a correction factor shall not differ more than 20 %.

18 SAFETY

General safety instructions should be followed at all times. All appropriate protective safety equipment should be worn and a fume cupboard must be used.

Annex 7. Results of the homogeneity study

Table 1. Results of the homogeneity study for samples S1 and S2

Element	Mean [µg/kg]	Mode for s.d. for proficiency assessment	s(target) [%]	s(analytical) [%]	s(sample) [%]	ISO 13528:2015	
						Test for adequate homogeneity	Test for significant heterogeneity
Solution S1							
Al	590	Manual (previous ILC)	10	0.7	0.6	OK	OK
Ba	517		10	0.7	0.5	OK	OK
Cd	4.85		10	1.4	1.3	OK	OK
Co	49.2		10	0.7	0.0	OK	OK
Pb	9.74		15	2.5	0.0	OK	OK
Mn	431		10	0.8	0.0	OK	OK
Solution S2							
Sb	44.6	Manual (previous ILC)	20	4.9	1.2	OK	OK
Cu	2.16*		10	2.3	0.0	OK	OK
Fe	14.3*		10	2.1	0.0	OK	OK
Zn	17.3*		10	2.3	0.0	OK	OK

*mg/kg

Table 2. Results of the homogeneity study for samples CC and GB

Element	Mean [µg/kg]	Mode for s.d. for proficiency assessment	s(target) [%]	s(analytical) [%]	s(sample) [%]	ISO 13528:2015	
						Test for adequate homogeneity	Test for significant heterogeneity
Ceramic cups (preliminary migration)							
Pb	214	Corrected Horwitz	20	17	0.0	OK	OK
Cd	14.2		22	25	0.0	OK	OK
Glass bowls I migration							
Pb	582	Corrected Horwitz	17	8.3	1.9	OK	OK
Cd	33.1		22	9.8	0.0	OK	OK
Co	51.6		22	11	0.0	OK	OK
Glass bowls II migration							
Pb	44.0	Corrected Horwitz	22	4.9	1.0	OK	OK
Cd	3.09		22	7.2	1.9	OK	OK
Co	2.72		22	5.2	3.6	OK	OK
Glass bowls III migration							
Pb	29.0	Corrected Horwitz	22	4.9	0.0	OK	OK
Cd	2.37		22	5.2	2.0	OK	OK
Co	1.95		22	6.5	2.5	OK	OK

Annex 8. Results of the stability study

Table 1. Results of the stability study

Sample S1	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)
Pb	40ºC	9.68	0.002	0.004	0.014	OK
	20ºC	9.68	0.002	0.005	0.016	OK
	4ºC	9.69	0.004	0.007	0.022	OK
Cd	40ºC	4.78	0.002	0.002	0.006	OK
	20ºC	4.80	0.001	0.001	0.004	OK
	4ºC	4.82	0.002	0.001	0.003	OK
Co	40ºC	48.0	-0.006	0.026	0.083	OK
	20ºC	48.1	-0.006	0.025	0.080	OK
	4ºC	48.2	0.005	0.023	0.075	OK
Ba	40ºC	511	-0.132	0.150	0.477	OK
	20ºC	510	-0.160	0.169	0.538	OK
	4ºC	509	-0.148	0.203	0.647	OK
Mn	40ºC	421	-0.394	0.210	0.667	OK
	20ºC	421	-0.376	0.222	0.707	OK
	4ºC	422	-0.327	0.227	0.722	OK
Al	40ºC	584	0.261	0.203	0.647	OK
	20ºC	588	0.199	0.269	0.857	OK
	4ºC	579	0.389	0.369	1.175	OK

Sample S2	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)
Fe	40ºC	14.1 $x10^3$	1.70	4.55	14.5	OK
	20ºC	14.2 $x10^3$	1.96	2.59	8.23	OK
	4ºC	14.0 $x10^3$	0.42	5.76	18.3	OK
Cu	40ºC	2.14 $x10^3$	-0.85	0.71	2.26	OK
	20ºC	2.16 $x10^3$	-0.79	0.51	1.64	OK
	4ºC	2.15 $x10^3$	-0.59	0.77	2.46	OK
Zn	40ºC	17.2 $x10^3$	-1.85	4.32	13.7	OK
	20ºC	17.3 $x10^3$	0.25	4.15	13.2	OK
	4ºC	17.2 $x10^3$	2.25	4.46	14.2	OK
Sb	40ºC	43.0	-0.06	0.03	0.11	OK
	20ºC	43.1	-0.06	0.03	0.12	OK
	4ºC	43.1	-0.06	0.03	0.10	OK

Annex 9. ILC01 2016 summary results

Table 1. **Pb in S1**, Assigned range: x_{pt} = 9.64, u(x_{pt}) = 0.17, σ_{pt} = 1.45 [all values are in µg/kg]

Lab code	x_i	U_i	k[a]	technique	u_i	z-score[b]	ζ-score	Uncert.[c]
LC0001	7.10	2.1	2	ICP-MS	1.05	-1.76	-2.39	a
LC0002	9.57	0.18	2	ICP-MS	0.09	-0.05	-0.40	b
LC0003	7.48	2.4	2	ICP-OES	1.20	-1.50	-1.79	a
LC0004	7.28			ICP-OES	0	-1.64	-13.54	b
LC0006	12.1	3.1	2	GF-AAS	1.55	1.66	1.54	c
LC0007	9.82	0.38	2	GF-AAS	0.19	0.12	0.69	a
LC0008	9.95	1.0	2	ICP-MS	0.50	0.21	0.58	a
LC0010	10.1	1.5	2	ICP-MS	0.75	0.32	0.59	a
LC0011	9.83			F-AAS	0	0.13	1.04	b
LC0012	9.18	0.04	2	ICP-MS	0.02	-0.32	-2.66	b
LC0013	10.2	1.2	2	GF-AAS	0.60	0.39	0.89	a
LC0014	10.8	4.6	2	ICP-MS	2.30	0.80	0.50	c
LC0016	10.3			GF-AAS	0	0.47	3.90	b
LC0017	9.60	0.12	2	ICP-MS	0.06	-0.03	-0.23	b
LC0020	10.2	1.3	2	ICP-MS	0.65	0.35	0.75	a
LC0022	10.3	4.4	2	ICP-OES	2.20	0.42	0.28	c
LC0025	16.2			ICP-MS	0	4.51	37.31	b
LC0028	10.4	0.7	2	ICP-MS	0.35	0.51	1.87	a
LC0029	10.8			ICP-MS	0	0.82	6.76	b
LC0031	10.1	0.5	2	ICP-MS	0.25	0.32	1.50	a
LC0032	10.1	6	√3	ICP-MS	3.47	0.34	0.14	c
LC0034	7.60	0.13	2	GF-AAS	0.06	-1.41	-10.95	b
LC0037	12.5	0.7	2	ICP-MS	0.35	1.94	7.18	a
LC0040	8.95	0.90	2	ICP-MS	0.45	-0.48	-1.44	a
LC0042	8.79	3.9	2	ICP-MS	1.95	-0.59	-0.43	c
LC0043	10.6	0.4	2	ICP-MS	0.20	0.65	3.51	a
LC0044	10.3	1.3	2	ICP-MS	0.65	0.42	0.90	a
LC0048	10.1	0.6	2	ICP-MS	0.30	0.30	1.25	a
LC0050	8.60	1.15	1	ICP-MS	1.15	-0.72	-0.90	a
LC0054	7.90			GF-AAS	0	-1.21	-9.97	b
LC0055	10.0	1.5	2	ICP-MS	0.75	0.25	0.46	a
LC0056	9.88	1.5	2	ICP-MS	0.75	0.16	0.30	a
LC0059	10.5	1.8	2	ICP-MS	0.90	0.56	0.88	a
LC0061	9.18	1.9	2	ICP-MS	0.95	-0.32	-0.48	a
LC0062	10.1			ICP-MS	0	0.29	2.39	b
LC0067	9.65				0	0.01	0.04	b
LC0068	9.79	1.36	2	ICP-MS	0.68	0.10	0.21	a
LC0097	8.48	3.4	2	GF-AAS	1.70	-0.81	-0.68	c
LC0101	9.72	1.26	2	ICP-MS	0.63	0.05	0.11	a
LC0115	8.35	1.2	3.18	ICP-MS	0.38	-0.89	-3.11	a
LC0116	8.38			ICP-MS	0	-0.87	-7.21	b
LC0118	11.4	5.0	2	ICP-OES	2.50	1.20	0.69	c
LC0120	9.27	3.0	2	ICP-OES	1.50	-0.26	-0.25	c
LC0121	9.10	4.0	2		2.00	-0.38	-0.27	c
LC0122	9.70	4.3	2	ICP-MS	2.15	0.04	0.03	c
LC0123	9.30	0.9	2	GF-AAS	0.45	-0.24	-0.71	a
LC0124	9.86	0.05	2	ICP-MS	0.03	0.15	1.24	b

[a] √3 is set by ILC coordinator when no coverage factor is reported. The reported uncertainty was assumed to have a rectangular distribution with k=√3

[b] Satisfactory, Questionable, Unsatisfactory

[c] "a": u(x_{pt}) ≤ u_{lab} ≤ σ_{pt}; "b": u_{lab} < u(x_{pt}); and "c": u_{lab} > σ_{pt}

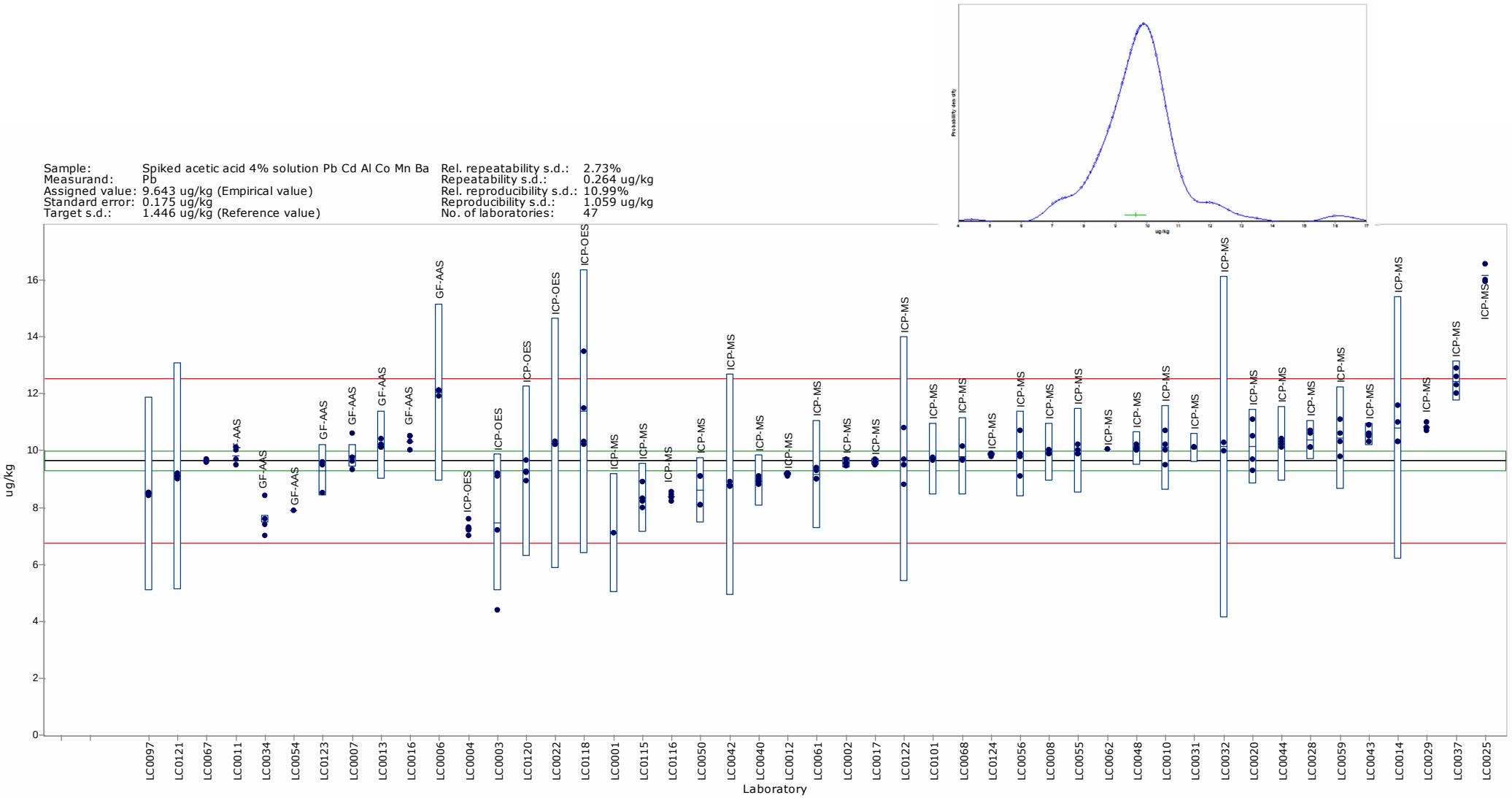

Figure 1. **Pb in S1**: Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (x_{pt}): solid black line; reference interval (x_{pt}±U(x_{pt})):dash green line; Target interval (x_{pt}±2σ_{pt}): solid red lines

Table 2. **Ba in S1**, Assigned range: x_{pt} = 497, u(x_{pt})= 5.75, σ_{pt} = 49.7 [all values are in µg/kg]

Lab code	x_i	U_i	k[a]	technique	u_i	z-score[b]	ζ-score	Uncert.[c]
LC0001	480	144	2	ICP-MS	72.00	-0.34	-0.23	c
LC0002	535	36.66	2	ICP-MS	18.33	0.76	1.98	a
LC0003	506	62.2	2	ICP-OES	31.10	0.19	0.29	a
LC0004	488			ICP-OES	0	-0.18	-1.54	b
LC0006	667	113.3	2	ICP-OES	56.65	3.42	2.98	c
LC0008	520	52	2	ICP-MS	26.00	0.46	0.86	a
LC0010	519	52	2	ICP-MS	26.00	0.44	0.82	a
LC0011	595			F-AAS	0	1.97	16.99	b
LC0012	492	8.21	2	ICP-MS	4.11	-0.09	-0.66	b
LC0014	528	158	2	ICP-MS	79.00	0.62	0.39	c
LC0016	510			ICP-OES	0	0.26	2.24	b
LC0017	453	5.00	2	ICP-MS	2.50	-0.88	-6.94	b
LC0018	459	65.3	√3	ICP-OES	37.75	-0.76	-0.99	a
LC0020	487	8.3	2	ICP-MS	4.15	-0.19	-1.36	b
LC0025	485			ICP-OES	0	-0.25	-2.14	b
LC0028	508	8	2	ICP-MS	4.00	0.21	1.52	b
LC0029	548			ICP-MS	0	1.02	8.82	b
LC0031	515	25.8	2	ICP-MS	12.90	0.37	1.29	a
LC0032	504	291	√3	ICP-OES	168.21	0.13	0.04	c
LC0037	457	55.1	2	GF-AAS	27.55	-0.80	-1.42	a
LC0040	492	49.2	2	ICP-MS	24.60	-0.09	-0.18	a
LC0041	478	170	2	ICP-MS	85.00	-0.39	-0.23	c
LC0043	505	3.4	2	ICP-MS	1.70	0.15	1.27	b
LC0044	501	90	2.	ICP-MS	45.00	0.08	0.09	a
LC0048	495			ICP-MS	0	-0.04	-0.36	b
LC0050	535	11.55	1	ICP-MS	11.55	0.77	2.95	a
LC0054	480			ICP-OES	0	-0.34	-2.93	b
LC0055	512	76.8	2	ICP-MS	38.40	0.30	0.38	a
LC0056	532	65.5	2	ICP-MS	32.75	0.71	1.07	a
LC0059	516	82	2	ICP-MS	41.00	0.38	0.45	a
LC0061	480			ICP-MS	0	-0.33	-2.89	b
LC0062	481			ICP-OES	0	-0.32	-2.76	b
LC0067	489				0	-0.15	-1.29	b
LC0068	514	64.26	2	ICP-MS	32.13	0.35	0.53	a
LC0101	500	75.0	2	ICP-MS	37.50	0.05	0.07	a
LC0113	440	41.3	2	ICP-OES	20.65	-1.15	-2.65	a
LC0115	503	4.4	3.18	ICP-MS	1.38	0.13	1.05	b
LC0118	443	160	2	ICP-OES	80.00	-1.09	-0.68	c
LC0120	503	75	2	ICP-OES	37.50	0.13	0.17	a
LC0122	486	173.3	2	ICP-MS	86.65	-0.22	-0.13	c
LC0124	447	11.89	2	ICP-MS	5.95	-1.01	-6.05	a

[a] √3 is set by ILC coordinator when no coverage factor is reported. The reported uncertainty was assumed to have a rectangular distribution with k=√3

[b] Satisfactory, Questionable, Unsatisfactory

[c] "a": u(x_{pt}) ≤ u_{lab} ≤ σ_{pt}; "b": u_{lab} < u(x_{pt}); and "c": u_{lab} > σ_{pt}

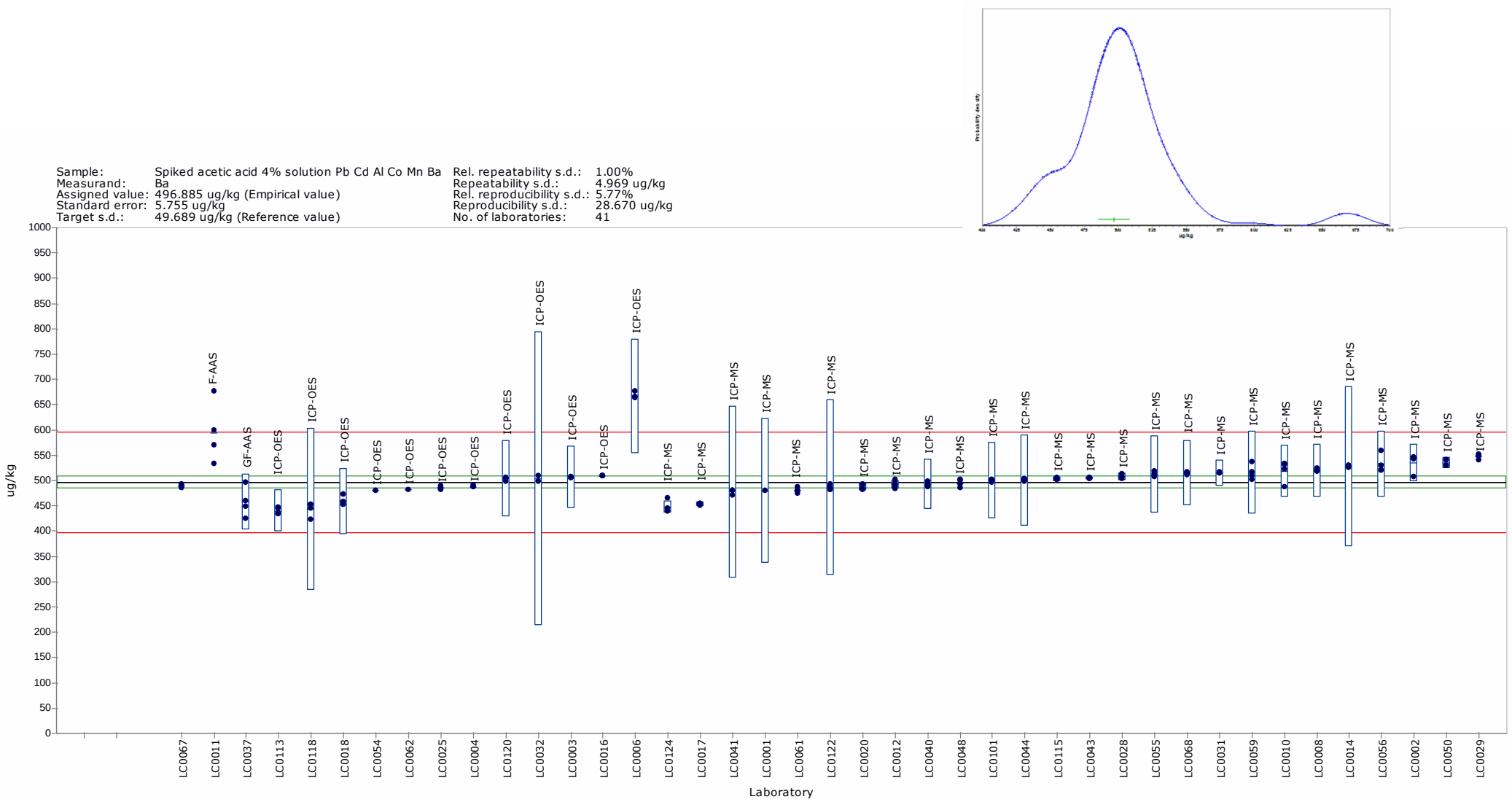

Figure 2. **Ba in S1**: Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (x_{pt}): solid black line; reference interval ($x_{pt}\pm U(x_{pt})$):dash green line; Target interval ($x_{pt}\pm 2\sigma_{pt}$): solid red lines

Table 3. **Co in S1**, Assigned range: x_{pt} = 50.6, u (x_{pt})= 0.48, σ_{pt} = 5.06 [all values are in µg/kg]

Lab code	x_i	U_i	k[a]	technique	u_i	z-score[b]	ζ-score	Uncert.[c]
LC0001	50.0	15	2	ICP-MS	7.50	-0.12	-0.08	c
LC0002	51.9	0.37	2	ICP-MS	0.18	0.25	2.44	b
LC0003	52.3	6.1	2	ICP-OES	3.05	0.34	0.55	a
LC0004	47.9			ICP-OES	0	-0.53	-5.56	b
LC0006	45.8	11.5	2	GF-AAS	5.75	-0.95	-0.83	c
LC0007	46.7	0.87	2	GF-AAS	0.44	-0.77	-5.94	b
LC0008	50.4	5.0	2	ICP-MS	2.50	-0.04	-0.09	a
LC0010	50.5	2.6	2	ICP-MS	1.30	-0.02	-0.09	a
LC0011	48.7			F-AAS	0	-0.38	-3.91	b
LC0012	46.0	1.31	2	ICP-MS	0.66	-0.91	-5.67	a
LC0013	47.7	2.8	2	GF-AAS	1.40	-0.58	-1.97	a
LC0014	53.4	17.8	2	ICP-MS	8.90	0.55	0.32	c
LC0016	52.0			ICP-MS	0	0.27	2.85	b
LC0017	49.6	0.71	2	ICP-MS	0.36	-0.19	-1.62	b
LC0018	48.6	4.7	√3	ICP-OES	2.72	-0.40	-0.73	a
LC0020	49.7	1.2	2	ICP-MS	0.60	-0.19	-1.23	a
LC0022	50.4	22	2	ICP-OES	11.00	-0.04	-0.02	c
LC0025	59.2			ICP-MS	0	1.70	17.78	b
LC0028	51.9	0.4	2	ICP-MS	0.20	0.25	2.39	b
LC0029	56.5			ICP-MS	0	1.16	12.08	b
LC0031	52.1	2.6	2	ICP-MS	1.30	0.30	1.10	a
LC0032	52.2	30	√3	ICP-MS	17.34	0.32	0.09	c
LC0037	56.0	5.4	2	ICP-MS	2.70	1.06	1.95	a
LC0040	50.1	5.01	2	ICP-MS	2.51	-0.11	-0.21	a
LC0043	51.1	0.6	2	ICP-MS	0.30	0.11	0.93	b
LC0044	50.5	9	2	ICP-MS	4.50	-0.02	-0.02	a
LC0048	47.8			ICP-MS	0	-0.56	-5.82	b
LC0050	50.7	1.00	1	ICP-MS	1.00	0.01	0.05	a
LC0054	56.8			GF-AAS	0	1.23	12.80	b
LC0055	52.9	7.9	2	ICP-MS	3.95	0.45	0.57	a
LC0056	47.8	6.7	2	ICP-MS	3.35	-0.56	-0.84	a
LC0059	53.6	8.6	2	ICP-MS	4.30	0.58	0.68	a
LC0061	48.9			ICP-MS	0	-0.35	-3.60	b
LC0062	51.2			ICP-MS	0	0.13	1.31	b
LC0067	69.8				0	3.79	39.51	b
LC0068	49.2	3.35	2	ICP-MS	1.68	-0.27	-0.78	a
LC0097	49.6	21.8	2	GF-AAS	10.90	-0.20	-0.09	c
LC0101	49.9	6.99	2	ICP-MS	3.50	-0.13	-0.19	a
LC0113	50.9	5.3	2	ICP-OES	2.65	0.05	0.10	a
LC0115	52.8	0.5	3.18	ICP-MS	0.16	0.43	4.28	b
LC0116	50.8			ICP-MS	0	0.04	0.42	b
LC0118	48.7	21.4	2	ICP-OES	10.70	-0.37	-0.18	c
LC0120	51.3	18	2	ICP-OES	9.00	0.15	0.08	c
LC0122	51.9	22.8	2	ICP-MS	11.40	0.26	0.12	c
LC0124	50.3	0.30	2	ICP-MS	0.15	-0.07	-0.67	b

[a] √3 is set by ILC coordinator when no coverage factor is reported. The reported uncertainty was assumed to have a rectangular distribution with k=√3

[b] Satisfactory, Questionable, Unsatisfactory

[c] "a": $u(x_{pt}) \leq u_{lab} \leq \sigma_{pt}$; "b": $u_{lab} < u(x_{pt})$; and "c": $u_{lab} > \sigma_{pt}$

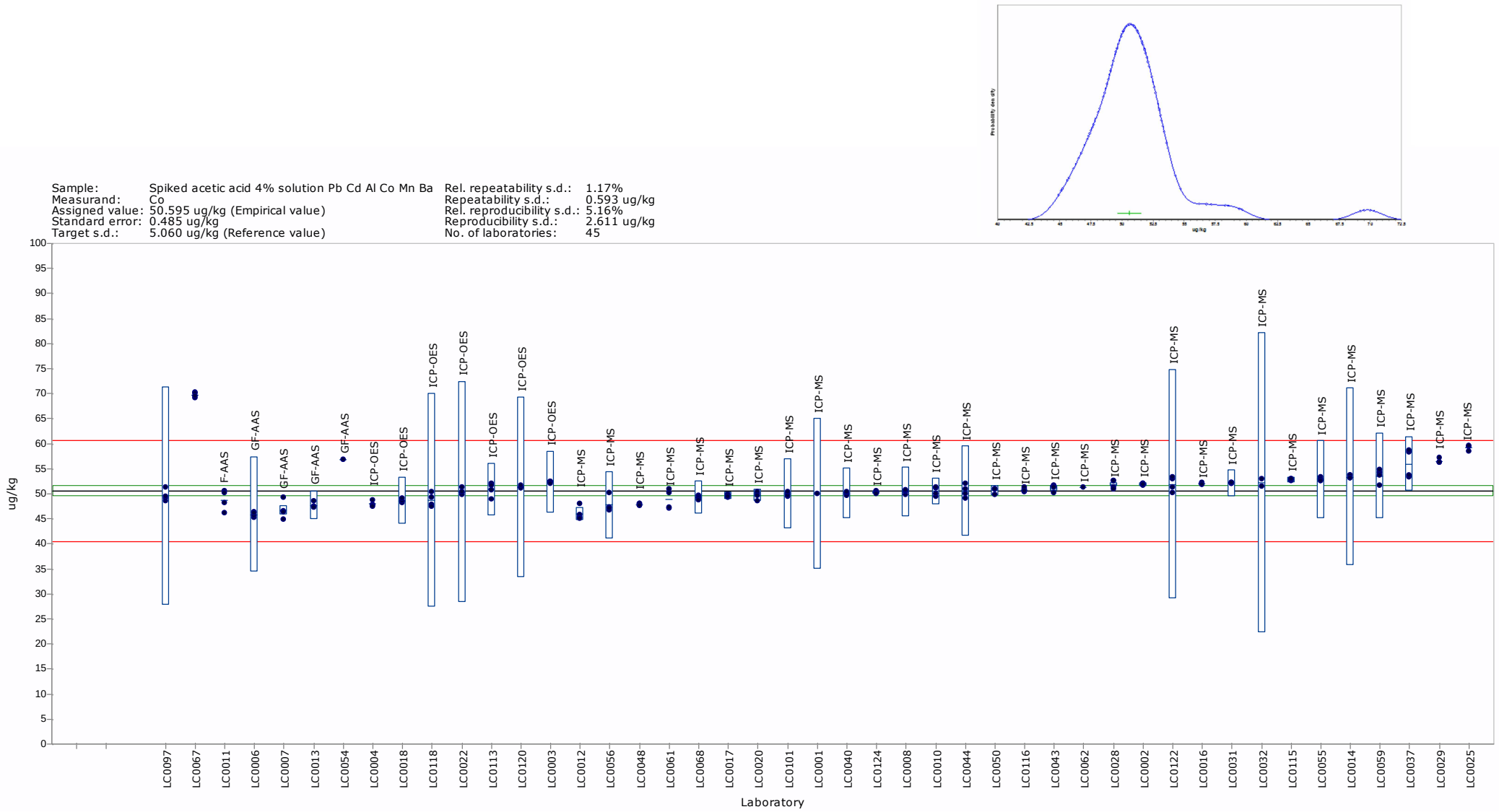

Figure 3. **Co in S1**: Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (x_{pt}): solid black line; reference interval (x_{pt}±U(x_{pt})):dash green line; Target interval (x_{pt}±2σ_{pt}): solid red line

Table 4. **Mn in S1**, Assigned range: x_{pt} = 459, u (x_{pt})= 5.27, σ_{pt} = 45.9 [all values are in µg/kg]

Lab code	x_i	U_i	k[a]	technique	u_i	z-score[b]	ζ-score	Uncert.[c]
LC0001	510	153	2	ICP-MS	76.50	1.11	0.67	c
LC0002	474	14.77	2	ICP-MS	7.39	0.33	1.66	a
LC0003	471	72.0	2	ICP-OES	36.00	0.26	0.33	a
LC0004	432			ICP-OES	0	-0.58	-5.07	b
LC0006	459	82.7	2	GF-AAS	41.35	0.01	0.01	a
LC0007	470	33.00	2	GF-AAS	16.50	0.24	0.63	a
LC0008	475	48	2	ICP-MS	24.00	0.35	0.66	a
LC0010	462	34	2	ICP-MS	17.00	0.05	0.14	a
LC0011	421			F-AAS	0	-0.83	-7.19	b
LC0012	445	4.09	2	ICP-MS	2.05	-0.30	-2.47	b
LC0013	476	22.4	2	F-AAS	11.20	0.37	1.37	a
LC0014	489	147	2	ICP-MS	73.50	0.66	0.41	c
LC0016	458			ICP-MS	0	-0.03	-0.27	b
LC0017	407	5.52	2	ICP-MS	2.76	-1.14	-8.76	b
LC0018	437	13.0	√3	ICP-OES	7.51	-0.48	-2.39	a
LC0020	444	13.9	2	ICP-MS	6.95	-0.32	-1.70	a
LC0022	433	160	2	ICP-OES	80.00	-0.58	-0.33	c
LC0025	490			ICP-OES	0	0.68	5.96	b
LC0028	469	10	2	ICP-MS	5.00	0.22	1.41	b
LC0029	497			ICP-MS	0	0.83	7.26	b
LC0031	462	23.1	2	ICP-MS	11.55	0.07	0.25	a
LC0032	480	277	√3	ICP-OES	160.12	0.46	0.13	c
LC0037	481	35.8	2	ICP-MS	17.90	0.48	1.19	a
LC0040	445	44.5	2	ICP-MS	22.25	-0.31	-0.63	a
LC0041	458	160	2	ICP-MS	80.00	-0.03	-0.02	c
LC0042	458	170	2	ICP-MS	85.00	-0.02	-0.01	c
LC0043	464	7.6	2	ICP-MS	3.80	0.11	0.80	b
LC0044	478	62	2	ICP-MS	31.00	0.40	0.59	a
LC0048	436			ICP-MS	0	-0.50	-4.33	b
LC0049	499			ICP-OES	0	0.88	7.65	b
LC0050	500	11.55	1	ICP-MS	11.55	0.89	3.20	a
LC0054	491	2	3.18	GF-AAS	0.63	0.69	5.98	b
LC0055	450	67.4	2	ICP-MS	33.70	-0.21	-0.28	a
LC0056	478	46.3	2	ICP-MS	23.15	0.40	0.78	a
LC0059	473	76	2	ICP-MS	38.00	0.31	0.37	a
LC0061	447	137.9	2	ICP-MS	68.95	-0.25	-0.17	c
LC0062	449			ICP-OES	0	-0.22	-1.90	b
LC0067	593				0	2.92	25.39	b
LC0068	432	40.20	2	ICP-MS	20.10	-0.58	-1.29	a
LC0097	380	167.4	2	GF-AAS	83.70	-1.72	-0.94	c
LC0101	453	72.0	2	ICP-MS	36.00	-0.14	-0.18	a
LC0113	401	37.6	2	ICP-OES	18.80	-1.26	-2.96	a
LC0115	490	2.1	3.18	ICP-MS	0.66	0.69	5.92	b
LC0116	445			ICP-MS	0	-0.32	-2.76	b
LC0118	407	149	2	ICP-OES	74.50	-1.14	-0.70	c
LC0120	463	69	2	ICP-OES	34.50	0.08	0.10	a
LC0122	480	171.6	2	ICP-MS	85.80	0.46	0.25	c
LC0123	447			GF-AAS	0	-0.26	-2.28	b
LC0124	408	6.21	2	ICP-MS	3.10	-1.12	-8.41	b

[a] √3 is set by ILC coordinator when no coverage factor is reported. The reported uncertainty was assumed to have a rectangular distribution with k=√3

[b] Satisfactory, Questionable, Unsatisfactory

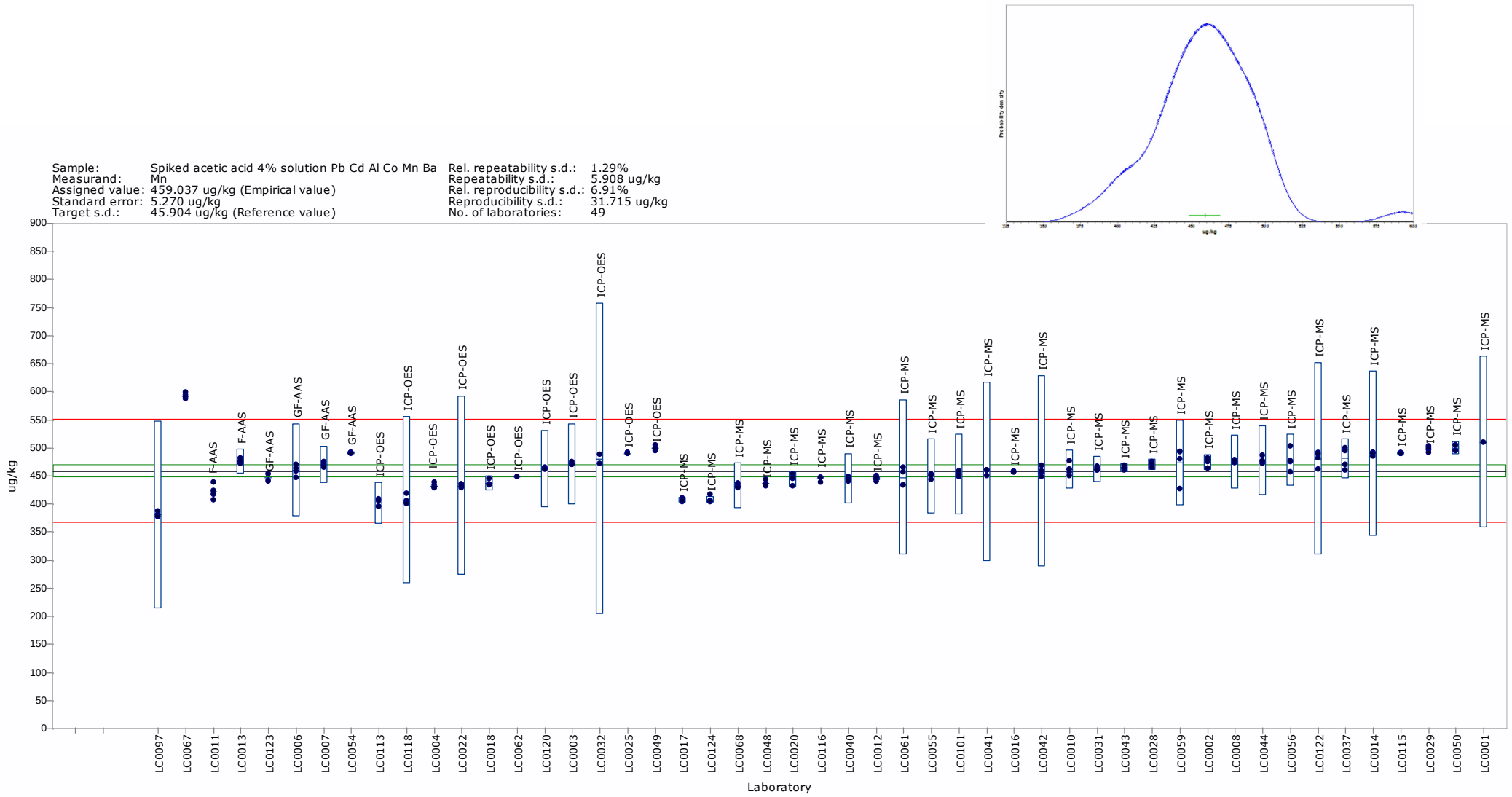

Figure 4. **Mn in S1**: Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (x_{pt}): solid black line; reference interval (x_{pt}±U(x_{pt})):dash green line; Target interval (x_{pt}±2σ_{pt}): solid red line

Table 5. **Cd in S1**, Assigned range: x_{pt} = 5.03, u (x_{pt})= 0.04, σ_{pt} = 0.50 [all values are in µg/kg]

Lab code	x_i	U_i	k[a]	technique	u_i	z-score[b]	ζ-score	Uncert.[c]
LC0001	4.70	1.4	2	ICP-MS	0.70	-0.65	-0.47	c
LC0002	4.89	0.07	2	ICP-MS	0.04	-0.27	-2.57	b
LC0003	5.43	0.6	2	ICP-OES	0.30	0.79	1.32	a
LC0004	4.23			ICP-OES	0	-1.59	-20.87	b
LC0006	4.90	1.2	2	GF-AAS	0.60	-0.25	-0.21	c
LC0007	5.04	0.08	2	GF-AAS	0.04	0.03	0.25	a
LC0008	5.08	0.5	2	ICP-MS	0.25	0.10	0.19	a
LC0010	4.98	0.3	2	ICP-MS	0.15	-0.10	-0.33	a
LC0011	5.25			F-AAS	0	0.45	5.84	b
LC0012	4.83	0.09	2	ICP-MS	0.04	-0.40	-3.40	a
LC0013	5.00	0.4	2	GF-AAS	0.20	-0.05	-0.13	a
LC0014	5.11	2.42	2	ICP-MS	1.21	0.16	0.07	c
LC0016	5.50			GF-AAS	0	0.94	12.35	b
LC0017	4.88	0.14	2	ICP-MS	0.07	-0.30	-1.89	a
LC0018	4.53	0.3	√3	ICP-OES	0.17	-1.00	-2.82	a
LC0020	4.93	0.6	2	ICP-MS	0.30	-0.20	-0.33	a
LC0022	4.90	2.2	2	ICP-OES	1.10	-0.25	-0.12	c
LC0025	5.58			ICP-MS	0	1.10	14.35	b
LC0028	5.05	0.2	2	ICP-MS	0.10	0.05	0.22	a
LC0029	5.23			ICP-MS	0	0.40	5.18	b
LC0031	5.08	0.3	2	ICP-MS	0.15	0.10	0.32	a
LC0032	5.08	3	√3	ICP-MS	1.73	0.10	0.03	c
LC0034	5.05	0.122	2	GF-AAS	0.06	0.05	0.33	a
LC0037	5.18	0.4	2	ICP-MS	0.20	0.30	0.73	a
LC0040	4.90	0.49	2	ICP-MS	0.25	-0.25	-0.51	a
LC0042	4.16	1.8	2	ICP-MS	0.90	-1.72	-0.96	c
LC0043	5.00	0.04	2	ICP-MS	0.02	-0.05	-0.60	b
LC0044	5.10	0.9	2	ICP-MS	0.45	0.15	0.16	a
LC0048	4.85	0.2	2	ICP-MS	0.10	-0.35	-1.64	a
LC0049	5.23			ICP-OES	0	0.40	5.26	b
LC0050	5.03	0.10	1	ICP-MS	0.10	0.00	-0.01	a
LC0054	4.90			GF-AAS	0	-0.25	-3.28	b
LC0055	5.23	0.8	2	ICP-MS	0.40	0.40	0.50	a
LC0056	5.75	0.9	2	ICP-MS	0.45	1.44	1.60	a
LC0059	5.19	0.83	2	ICP-MS	0.41	0.32	0.38	a
LC0061	4.88	1.3	2	ICP-MS	0.65	-0.30	-0.23	c
LC0062	4.70			ICP-MS	0	-0.65	-8.49	b
LC0067	4.95				0	-0.15	-1.98	b
LC0068	4.92	0.58	2	ICP-MS	0.29	-0.22	-0.37	a
LC0097	5.48	2.4	2	GF-AAS	1.20	0.89	0.37	c
LC0101	4.96	0.74	2	ICP-MS	0.37	-0.14	-0.18	a
LC0115	5.00	0.3	3.18	ICP-MS	0.09	-0.05	-0.26	a
LC0116	4.82			ICP-MS	0	-0.42	-5.43	b
LC0118	5.43	2.4	2	ICP-OES	1.20	0.79	0.33	c
LC0120	4.95	1.5	2	ICP-OES	0.75	-0.16	-0.11	c
LC0121	5.10	2.2	2		1.10	0.15	0.07	c
LC0122	5.08	2.2	2	ICP-MS	1.10	0.10	0.04	c
LC0123	4.80	0.5	2	GF-AAS	0.25	-0.45	-0.89	a
LC0124	5.09	0.04	2	ICP-MS	0.02	0.12	1.36	b

[a] √3 is set by ILC coordinator when no coverage factor is reported. The reported uncertainty was assumed to have a rectangular distribution with k=√3

[b] Satisfactory, Questionable, Unsatisfactory

[c] "a": $u(x_{pt}) \leq u_{lab} \leq \sigma_{pt}$; "b": $u_{lab} < u(x_{pt})$; and "c": $u_{lab} > \sigma_{pt}$

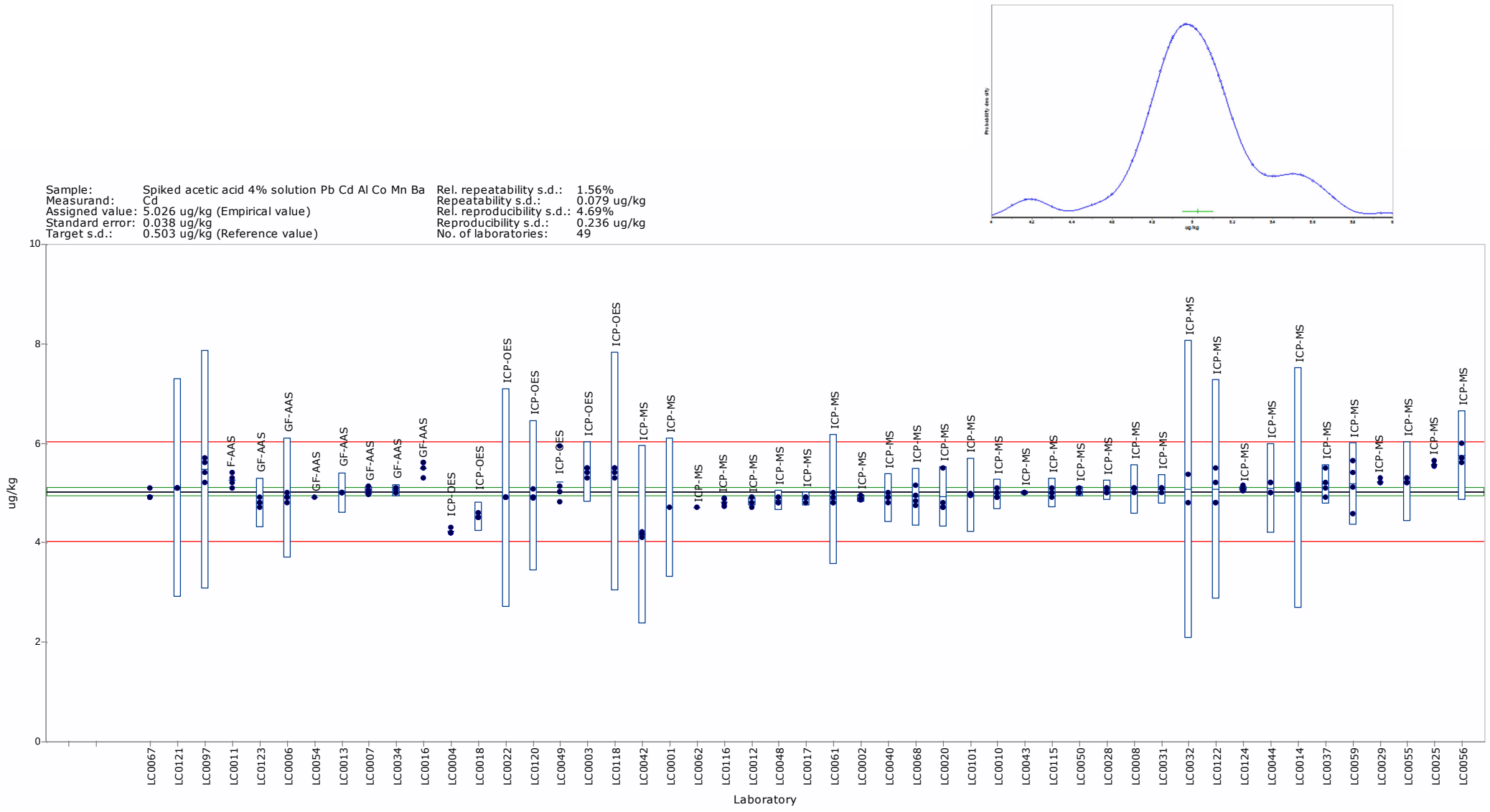

Figure 5. **Cd in S1**: Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (x_{pt}): solid black line; reference interval (x_{pt}±U(x_{pt})):dash green line; Target interval (x_{pt}±2σ_{pt}): solid red lines.

Table 6. **Al in S1**, Assigned range: x_{pt} = 581, u (x_{pt}) =11.2, σ_{pt} = 58.1 [all values are in µg/kg]

Lab code	x_i	U_i	k[a]	technique	u_i	z-score[b]	ζ-score	Uncert.[c]
LC0001	560	168	2	ICP-MS	84.00	-0.35	-0.24	c
LC0002	813	54.05	2	ICP-MS	27.03	4.01	7.95	a
LC0003	585	68.7	2	ICP-OES	34.35	0.08	0.14	a
LC0004	516			ICP-OES	0	-1.11	-5.72	b
LC0006	624	106.1	2	GF-AAS	53.05	0.75	0.80	a
LC0008	615	61	2	ICP-MS	30.50	0.58	1.04	a
LC0010	609	50	2	ICP-MS	25.00	0.49	1.05	a
LC0012	397	28.5	2	ICP-MS	14.25	-3.17	-10.14	a
LC0014	556	167	2	ICP-MS	83.50	-0.42	-0.29	c
LC0016	544			ICP-OES	0	-0.62	-3.23	b
LC0017	605	14.49	2	ICP-MS	7.24	0.43	1.86	b
LC0018	610	64.1	√3	ICP-OES	37.05	0.51	0.77	a
LC0020	598	22.8	2	ICP-MS	11.40	0.30	1.09	a
LC0025	514			ICP-OES	0	-1.16	-5.97	b
LC0028	566	19	2	ICP-MS	9.50	-0.25	-0.97	b
LC0029	646			ICP-MS	0	1.12	5.80	b
LC0031	590	29.5	2	ICP-MS	14.75	0.15	0.48	a
LC0032	630	364	√3	ICP-OES	210.16	0.84	0.23	c
LC0037	544	45.9	2	ICP-MS	22.95	-0.62	-1.42	a
LC0040	566	56.6	2	ICP-MS	28.30	-0.26	-0.49	a
LC0041	583	200	2	ICP-MS	100.00	0.03	0.02	c
LC0042	431	160	2	ICP-MS	80.00	-2.57	-1.85	c
LC0043	568	7.1	2	ICP-MS	3.55	-0.22	-1.11	b
LC0044	572	86	2	ICP-MS	43.00	-0.16	-0.20	a
LC0048	572			ICP-MS	0	-0.14	-0.74	b
LC0050	637	10.00	1	ICP-MS	10.00	0.98	3.77	b
LC0054	618			GF-AAS	0	0.64	3.33	b
LC0055	578	86.6	2	ICP-MS	43.30	-0.04	-0.05	a
LC0056	667	76.5	2	ICP-MS	38.25	1.49	2.17	a
LC0059	576	92	2	ICP-MS	46.00	-0.08	-0.10	a
LC0061	544			ICP-MS	0	-0.62	-3.22	b
LC0062	579			ICP-OES	0	-0.03	-0.14	b
LC0067	648				0	1.17	6.04	b
LC0068	588	89.92	2	ICP-MS	44.96	0.12	0.16	a
LC0101	593	77.0	2	ICP-MS	38.50	0.21	0.30	a
LC0113	479	45.0	2	ICP-OES	22.50	-1.74	-4.03	a
LC0115	610	35.5	3.18	ICP-MS	11.16	0.51	1.88	b
LC0116	525			ICP-MS	0	-0.96	-4.97	b
LC0118	510	180	2	ICP-OES	90.00	-1.22	-0.78	c
LC0120	628	126	2	ICP-OES	63.00	0.82	0.75	c
LC0122	633	216.9	2	ICP-MS	108.45	0.90	0.48	c
LC0123	738			GF-AAS	0	2.72	14.03	b
LC0124	482	5.15	2	ICP-MS	2.58	-1.69	-8.52	b

[a] √3 is set by ILC coordinator when no coverage factor is reported. The reported uncertainty was assumed to have a rectangular distribution with k=√3

[b] Satisfactory, Questionable, Unsatisfactory

[c] "a": $u(x_{pt}) \leq u_{lab} \leq \sigma_{pt}$; "b": $u_{lab} < u(x_{pt})$; and "c": $u_{lab} > \sigma_{pt}$

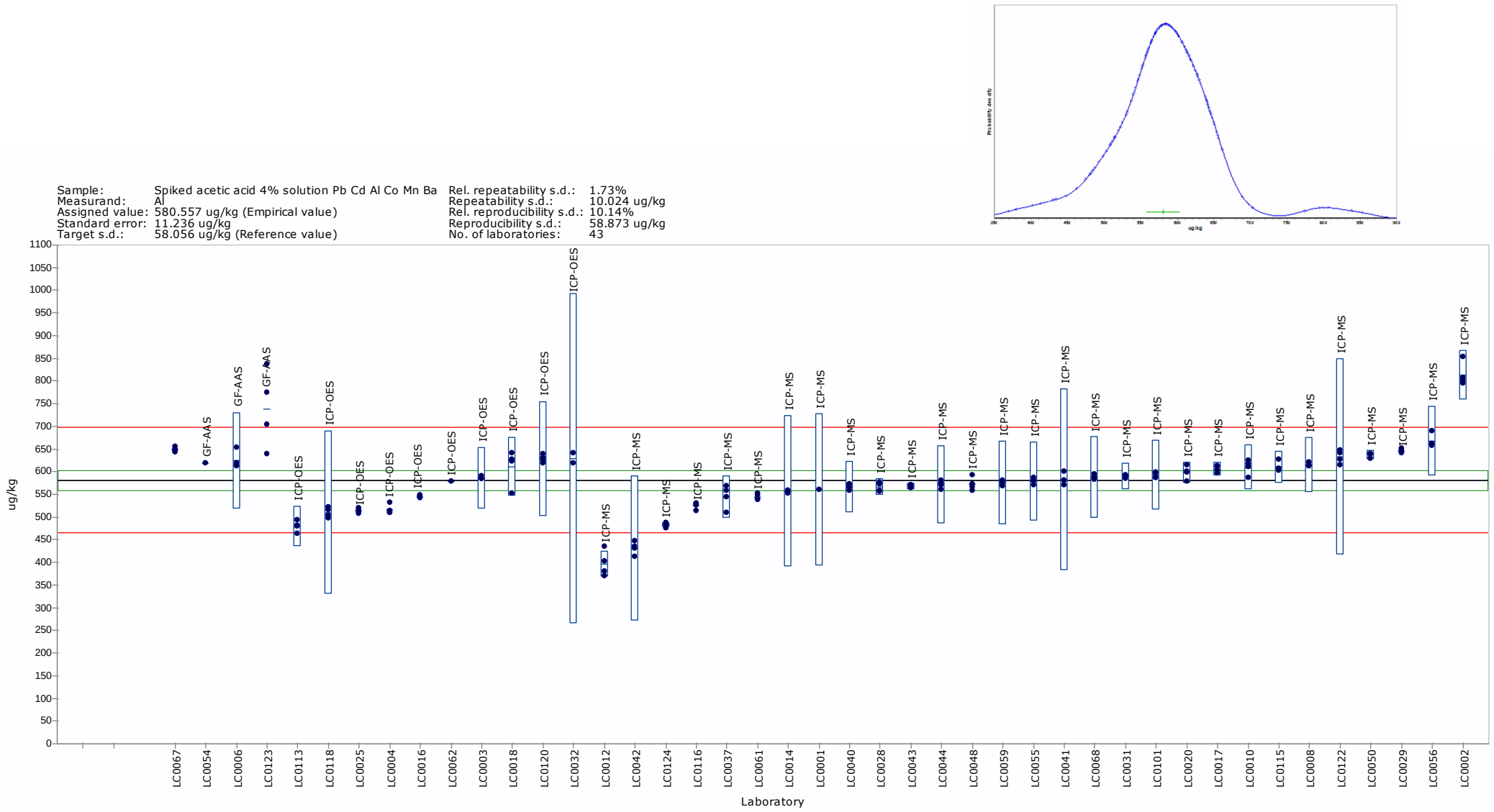

Figure 6. **Al in S1**: Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (x_{pt}): solid black line; reference interval (x_{pt}±U(x_{pt})):dash green line; Target interval (x_{pt}±2σ_{pt}): solid red lines.

Table 7. **Cu in S2**, Assigned range: x_{pt}= 2.51 x 10^3, u (x_{pt}) =31.9, σ_{pt} = 251 [all values are in µg/kg]

Lab code	x_i	U_i	k[a]	technique	u_i	z-score[b]	ζ-score	Uncert.[c]
LC0001	2300	690	2	ICP-MS	345.00	-0.85	-0.62	c
LC0002	2809	222.53	2	ICP-MS	111.27	1.18	2.56	a
LC0003	2628	315	2	ICP-OES	157.50	0.46	0.71	a
LC0004	2525			ICP-OES	0	0.05	0.36	b
LC0006	2480	347.7	2	GF-AAS	173.85	-0.13	-0.19	a
LC0007	2537	78.00	2	F-AAS	39.00	0.09	0.46	a
LC0008	2493	249	2	ICP-MS	124.50	-0.08	-0.16	a
LC0010	2566	250	2	ICP-MS	125.00	0.21	0.41	a
LC0011	2588			F-AAS	0	0.30	2.34	b
LC0012	1967	19.9	2	ICP-MS	9.95	-2.17	-16.35	b
LC0013	2432	153.2	2	F-AAS	76.60	-0.32	-0.98	a
LC0014	2680	804	2	ICP-MS	402.00	0.67	0.41	c
LC0016	2613			ICP-MS	0	0.40	3.14	b
LC0017	2416	106.8	2	ICP-MS	53.40	-0.39	-1.56	a
LC0018	2249	178.4	√3	ICP-OES	103.00	-1.05	-2.44	a
LC0020	2540	37.7	2	ICP-MS	18.85	0.11	0.72	b
LC0025	2358			ICP-OES	0	-0.62	-4.86	b
LC0028	2500	60	2	ICP-MS	30.00	-0.05	-0.30	b
LC0029	2620			ICP-MS	0	0.43	3.36	b
LC0031	2550	127.5	2	ICP-MS	63.75	0.15	0.52	a
LC0032	2585	1491	√3	ICP-OES	860.83	0.29	0.08	c
LC0037	3182	282.5	2	ICP-MS	141.25	2.66	4.62	a
LC0040	2470	246.98	2	ICP-MS	123.49	-0.17	-0.34	a
LC0041	2325	600	2	ICP-MS	300.00	-0.75	-0.62	c
LC0042	2610	720	2	ICP-MS	360.00	0.39	0.27	c
LC0043	2500	50	2	ICP-MS	25.00	-0.05	-0.32	b
LC0044	2409	313	2	ICP-MS	156.50	-0.42	-0.65	a
LC0048	2499			ICP-MS	0	-0.06	-0.45	b
LC0049	2405			ICP-OES	0	-0.43	-3.38	b
LC0050	2688	13.87	1	ICP-MS	13.87	0.70	5.03	b
LC0054	2112	35	3.18	GF-AAS	11.01	-1.60	-11.90	b
LC0055	2566	385	2	ICP-MS	192.50	0.21	0.27	a
LC0056	2407	284	2	ICP-MS	142.00	-0.42	-0.73	a
LC0059	2668	426	2	ICP-MS	213.00	0.61	0.72	a
LC0061	2705			ICP-MS	0	0.76	6.01	b
LC0062	2659			ICP-OES	0	0.58	4.57	b
LC0067	3356				0	3.35	26.41	b
LC0068	2124	438.48	2	ICP-MS	219.24	-1.55	-1.76	a
LC0097	2542	432.1	2	F-AAS	216.05	0.11	0.13	a
LC0101	2536	380.0	2	ICP-MS	190.00	0.09	0.12	a
LC0113	2475	232	2	ICP-OES	116.00	-0.15	-0.32	a
LC0115	2743	84.4	3.18	ICP-MS	26.52	0.92	5.54	b
LC0116	2429			ICP-MS	0	-0.34	-2.65	b
LC0118	2198	625	2	ICP-OES	312.50	-1.26	-1.01	c
LC0120	2486	373	2	ICP-OES	186.50	-0.11	-0.14	a
LC0122	2626	726.4	2	ICP-MS	363.20	0.45	0.31	c
LC0123	3050			GF-AAS	0	2.14	16.83	b

[a] √3 is set by ILC coordinator when no coverage factor is reported. The reported uncertainty was assumed to have a rectangular distribution with k=√3

[b] Satisfactory, Questionable, Unsatisfactory

[c] "a": $u(x_{pt}) \leq u_{lab} \leq \sigma_{pt}$; "b": $u_{lab} < u(x_{pt})$; and "c": $u_{lab} > \sigma_{pt}$

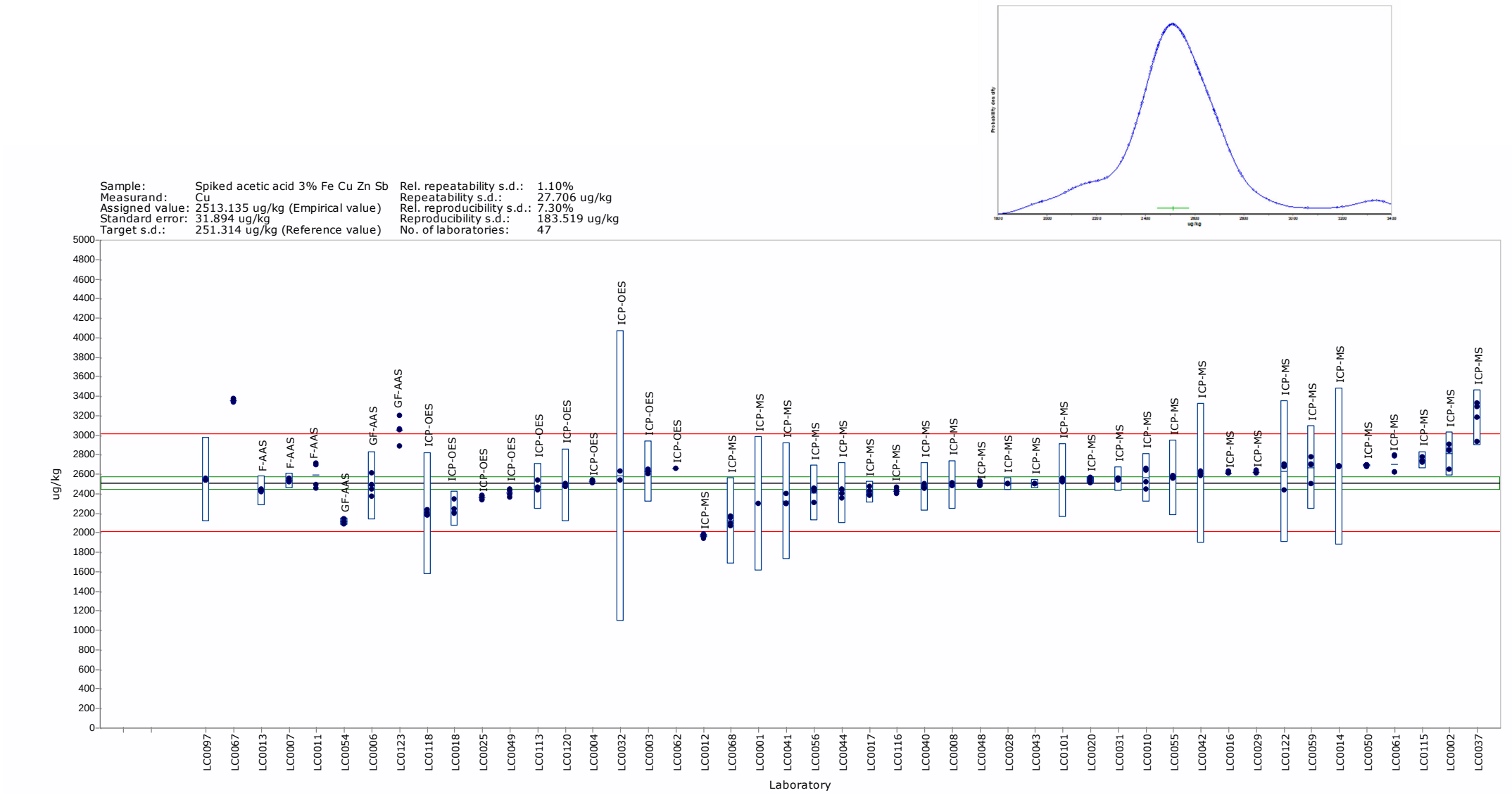

Figure 7. **Cu in S2**: Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (x_{pt}): solid black line; reference interval (x_{pt}±U(x_{pt})):dash green line; Target interval (x_{pt}±2σ_{pt}): solid red lines.

Table 8. **Fe in S2**, Assigned range: x_{pt} = 14.7 x 10^3, u (x_{pt}) =231, σ_{pt} = 1.47 x 10^3 [all values are in µg/kg]

Lab code	x_i	U_i	k[a]	technique	u_i	z-score[b]	ζ-score	Uncert.[c]
LC0001	16000	4800	2	ICP-MS	2400.00	0.88	0.54	c
LC0002	16273	734.17	2	ICP-MS	367.09	1.07	3.61	a
LC0003	14415	893	2	ICP-OES	446.50	-0.20	-0.58	a
LC0004	14630			ICP-OES	0	-0.05	-0.33	b
LC0006	15778	1735.6	2	GF-AAS	867.80	0.73	1.19	a
LC0007	14974	190.00	2	F-AAS	95.00	0.18	1.08	b
LC0008	15368	2305	2	ICP-MS	1152.50	0.45	0.56	a
LC0010	15783	1200	2	ICP-MS	600.00	0.73	1.68	a
LC0011	15618			F-AAS	0	0.62	3.95	b
LC0012	14682	334	2	ICP-MS	167.00	-0.02	-0.08	b
LC0013	13563	2454.8	2	F-AAS	1227.40	-0.78	-0.92	a
LC0014	15604	4681	2	ICP-MS	2340.50	0.61	0.38	c
LC0016	14740			ICP-OES	0	0.02	0.15	b
LC0017	12741	333.4	2	ICP-MS	166.70	-1.34	-6.90	b
LC0018	14567	391.8	√3	ICP-OES	226.21	-0.09	-0.43	b
LC0020	14725	496	2	ICP-MS	248.00	0.01	0.06	a
LC0025	13000			ICP-OES	0	-1.16	-7.39	b
LC0028	14675	400	2	ICP-MS	200.00	-0.02	-0.10	b
LC0029	15196			ICP-MS	0	0.33	2.13	b
LC0031	14410	720.5	2	ICP-MS	360.25	-0.20	-0.69	a
LC0032	14800	8544	√3	ICP-OES	4932.88	0.06	0.02	c
LC0037	19041	855.5	2	ICP-MS	427.75	2.95	8.92	a
LC0040	12668	1266.80	2	ICP-MS	633.40	-1.39	-3.02	a
LC0041	14075	3000	2	ICP-MS	1500.00	-0.43	-0.42	c
LC0042	13912	3000	2	ICP-MS	1500.00	-0.54	-0.52	c
LC0043	14825	600	2	ICP-MS	300.00	0.08	0.32	a
LC0044	14540	2617	2	ICP-MS	1308.50	-0.11	-0.13	a
LC0049	18501			ICP-OES	0	2.58	16.44	b
LC0050	16164	148.5	1	ICP-MS	148.50	0.99	5.31	b
LC0054	11228	495	3.18	GF-AAS	155.66	-2.37	-12.49	b
LC0055	14831	1780	2	ICP-MS	890.00	0.09	0.14	a
LC0056	14658	2463	2	ICP-MS	1231.50	-0.03	-0.04	a
LC0059	16050	2570	2	ICP-MS	1285.00	0.91	1.03	a
LC0061	14665			ICP-MS	0	-0.03	-0.18	b
LC0062	15900			ICP-OES	0	0.81	5.17	b
LC0067	15350				0	0.44	2.79	b
LC0068	13938	3085.98	2	ICP-MS	1542.99	-0.52	-0.49	c
LC0097	11296	4970.3	2	F-AAS	2485.15	-2.32	-1.37	c
LC0101	15006	2251.0	2	ICP-MS	1125.50	0.20	0.26	a
LC0113	10383	974	2	ICP-OES	487.00	-2.94	-8.02	a
LC0115	17307	1491.1	3.18	ICP-MS	468.60	1.77	4.98	a
LC0116	15032			ICP-MS	0	0.22	1.41	b
LC0118	13220	2868	2	ICP-OES	1434.00	-1.01	-1.02	a
LC0120	13945	2092	2	ICP-OES	1046.00	-0.52	-0.71	a
LC0122	14859	3167.2	2	ICP-MS	1583.60	0.10	0.10	c
LC0123	12900			GF-AAS	0	-1.23	-7.82	b

[a] √3 is set by ILC coordinator when no coverage factor is reported. The reported uncertainty was assumed to have a rectangular distribution with k=√3

[b] Satisfactory, Questionable, Unsatisfactory

[c] "a": $u(x_{pt}) \leq u_{lab} \leq \sigma_{pt}$; "b": $u_{lab} < u(x_{pt})$; and "c": $u_{lab} > \sigma_{pt}$

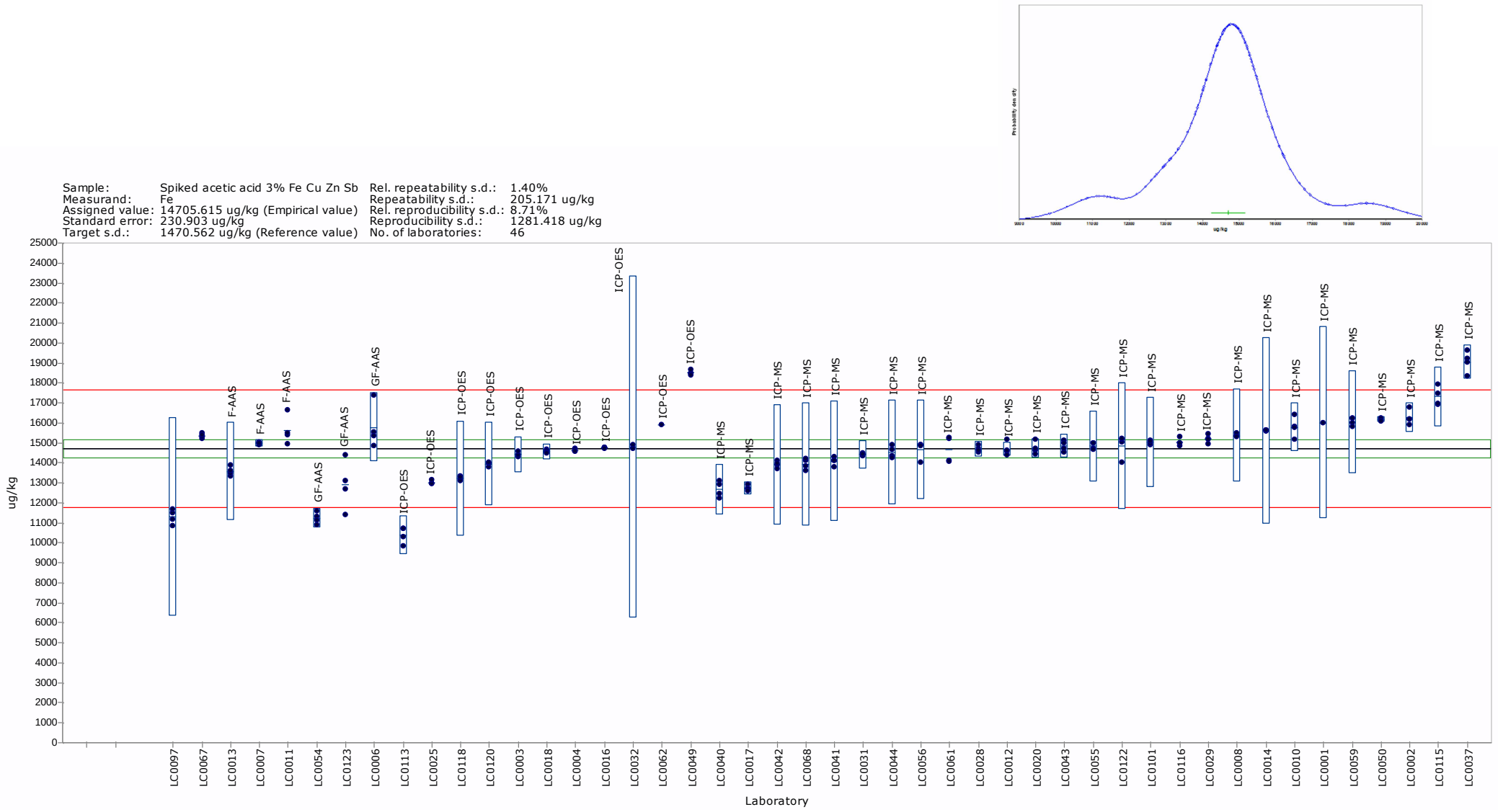

Figure 8. **Fe in S2**: Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (x_{pt}): solid black line; reference interval (x_{pt}±U(x_{pt})):dash green line; Target interval (x_{pt}±2σ_{pt}): solid red lines.

Table 9. **Zn in S2**, Assigned range: x_{pt} = 18.0 x10^3, u (x_{pt}) = 249, σ_{pt} = 1.80 x 10^3 [all values are in µg/kg]

Lab code	x_i	U_i	k[a]	technique	u_i	z-score[b]	ζ-score	Uncert.[c]
LC0001	17000	5100	2	ICP-MS	2550.00	-0.57	-0.40	c
LC0002	20478	972.9	2	ICP-MS	486.45	1.37	4.50	a
LC0003	18398	1139	2	ICP-OES	569.50	0.21	0.61	a
LC0004	16770			ICP-OES	0	-0.69	-5.01	b
LC0006	25221	2522.1	2	ICP-OES	1261.05	4.00	5.60	a
LC0007	17896	397.00	2	F-AAS	198.50	-0.07	-0.38	b
LC0008	19122	1912	2	ICP-MS	956.00	0.61	1.12	a
LC0010	18927	1100	2	ICP-MS	550.00	0.51	1.51	a
LC0011	18320			F-AAS	0	0.17	1.21	b
LC0012	17754	214	2	ICP-MS	107.00	-0.15	-0.97	b
LC0013	18413	1712.4	2	F-AAS	856.20	0.22	0.44	a
LC0014	18234	5470	2	ICP-MS	2735.00	0.12	0.08	c
LC0016	18063			ICP-MS	0	0.03	0.18	b
LC0017	17712	411.5	2	ICP-MS	205.75	-0.17	-0.95	b
LC0018	17038	906.4	√3	ICP-OES	523.31	-0.54	-1.69	a
LC0020	18235	246	2	ICP-MS	123.00	0.12	0.78	b
LC0025	16650			ICP-OES	0	-0.76	-5.49	b
LC0028	18075	300	2	ICP-MS	150.00	0.03	0.20	b
LC0029	18713			ICP-MS	0	0.39	2.79	b
LC0031	18027	901.3	2	ICP-MS	450.65	0.01	0.02	a
LC0032	17650	10182	√3	ICP-OES	5878.58	-0.20	-0.06	c
LC0037	19918	1179.4	2	ICP-MS	589.70	1.06	2.97	a
LC0040	17358	1735.80	2	ICP-MS	867.90	-0.37	-0.73	a
LC0041	14875	3100	2	ICP-MS	1550.00	-1.74	-2.00	a
LC0043	18100	500	2	ICP-MS	250.00	0.05	0.23	a
LC0044	18049	2166	2	ICP-MS	1083.00	0.02	0.03	a
LC0048	17139			ICP-MS	0	-0.49	-3.53	b
LC0049	18862			ICP-OES	0	0.47	3.39	b
LC0050	19087	100.4	1	ICP-MS	100.40	0.59	3.98	b
LC0054	17313	206	3.18	GF-AAS	64.78	-0.39	-2.74	b
LC0055	18473	2517	2	ICP-MS	1258.50	0.25	0.36	a
LC0056	20728	4146	2	ICP-MS	2073.00	1.50	1.30	c
LC0059	18375	2940	2	ICP-MS	1470.00	0.20	0.24	a
LC0061	17721			ICP-MS	0	-0.16	-1.19	b
LC0062	18720			ICP-OES	0	0.39	2.82	b
LC0067	26698				0	4.82	34.86	b
LC0068	17005	3380.61	2	ICP-MS	1690.31	-0.56	-0.59	a
LC0097	16174	7116.7	2	F-AAS	3558.35	-1.02	-0.52	c
LC0101	18176	3453.0	2	ICP-MS	1726.50	0.09	0.09	a
LC0113	14900	1397	2	ICP-OES	698.50	-1.73	-4.20	a
LC0115	22393	2320.6	3.18	ICP-MS	729.29	2.43	5.68	a
LC0116	17979			ICP-MS	0	-0.02	-0.15	b
LC0118	15615	3304	2	ICP-OES	1652.00	-1.33	-1.44	a
LC0120	17733	2660	2	ICP-OES	1330.00	-0.16	-0.21	a
LC0122	19855	4051.4	2	ICP-MS	2025.70	1.02	0.90	c
LC0123	4350			GF-AAS	0	-7.59	-54.89	b

[a] √3 is set by ILC coordinator when no coverage factor is reported. The reported uncertainty was assumed to have a rectangular distribution with k=√3

[b] Satisfactory, Questionable, Unsatisfactory

[c] "a": $u(x_{pt}) \leq u_{lab} \leq \sigma_{pt}$; "b": $u_{lab} < u(x_{pt})$; and "c": $u_{lab} > \sigma_{pt}$

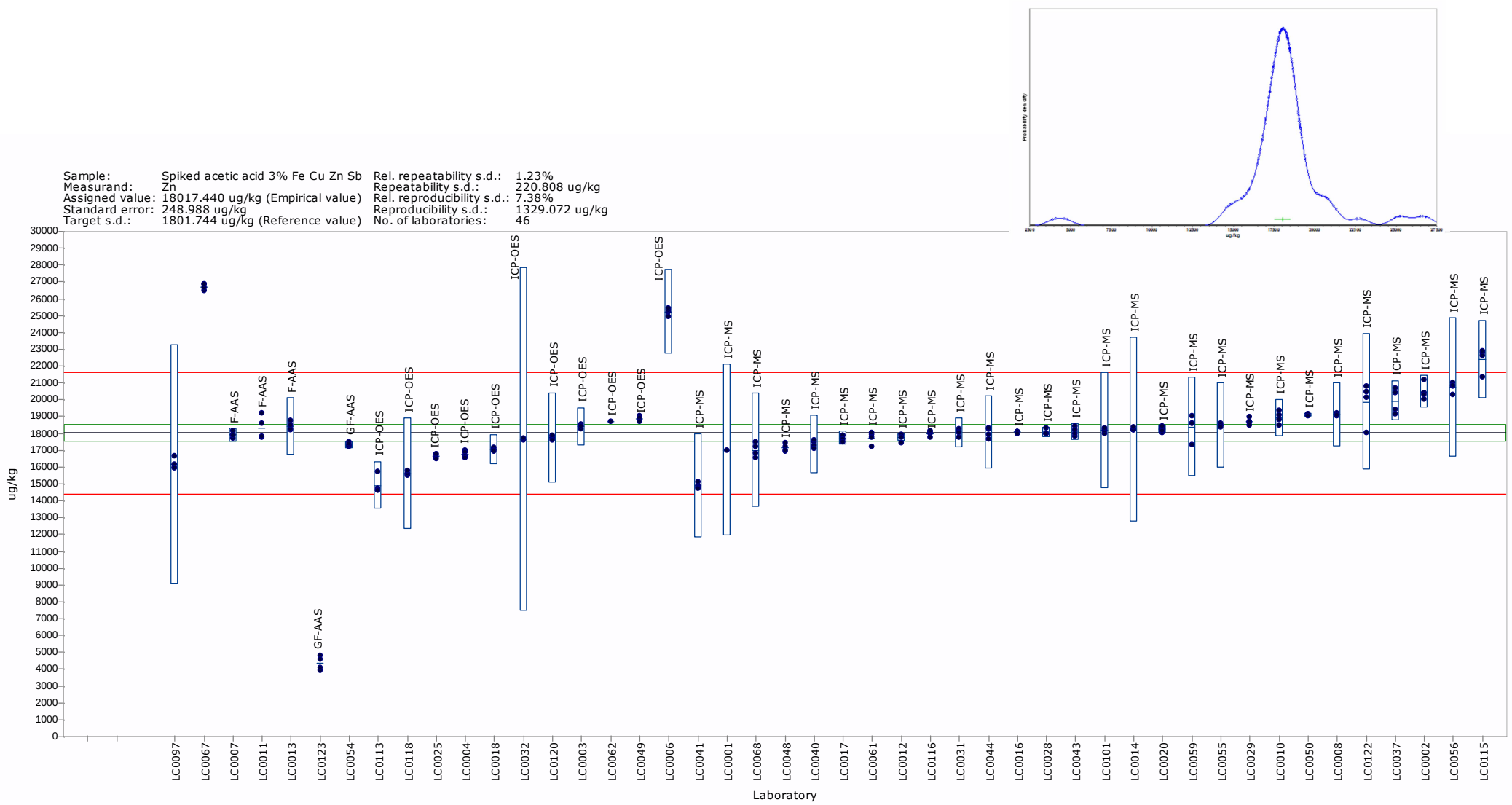

Figure 9. **Zn in S2**: Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (x_{pt}): solid black line; reference interval (x_{pt}±U(x_{pt})):dash green line; Target interval (x_{pt}±2σ_{pt}): solid red lines.

Table 10. **Sb in S2**, Assigned range: x_{pt} = 39.6, u (x_{pt}) =1.32, σ_{pt} = 7.92 [all values are in µg/kg]

Lab code	x_i	U_i	k[a]	technique	u_i	z-score[b]	ζ-score	Uncert.[c]
LC0001	40.0	12	2	ICP-MS	6.00	0.05	0.06	a
LC0002	44.5	0.53	2	ICP-MS	0.27	0.62	3.65	b
LC0003	36.6	4.7	2	ICP-OES	2.35	-0.39	-1.14	a
LC0004	47.9			ICP-OES	0	1.04	6.27	b
LC0006	29.0	7.8	2	GF-AAS	3.90	-1.34	-2.59	a
LC0008	44.3	4.4	2	ICP-MS	2.20	0.59	1.83	a
LC0010	40.8	3.5	2	ICP-MS	1.75	0.15	0.54	a
LC0011	24.2			F-AAS	0	-1.95	-11.74	b
LC0012	34.3	0.54	2	ICP-MS	0.27	-0.68	-4.00	b
LC0014	12.3	5.1	2	ICP-MS	2.55	-3.45	-9.52	a
LC0016	41.3			ICP-MS	0	0.21	1.24	b
LC0017	36.4	0.69	2	ICP-MS	0.35	-0.41	-2.39	b
LC0018	1.40	0.1	√3	ICP-OES	0.06	-4.82	-29.03	b
LC0020	39.6	0.5	2	ICP-MS	0.25	-0.01	-0.04	b
LC0025	104			ICP-MS	0	8.12	48.90	b
LC0028	70.0	0.9	2	ICP-MS	0.45	3.83	21.85	b
LC0029	40.0			ICP-MS	0	0.05	0.27	b
LC0031	44.2	2.2	2	ICP-MS	1.10	0.58	2.68	b
LC0032	40.2	133	√3	ICP-MS	76.79	0.07	0.01	c
LC0037	54.5	2.9	2	ICP-MS	1.45	1.87	7.57	a
LC0040	35.2	5.6	2	ICP-MS	2.80	-0.56	-1.44	a
LC0041	39.0	18	2	ICP-MS	9.00	-0.08	-0.07	c
LC0043	67.5	2	2	ICP-MS	1.00	3.52	16.87	b
LC0044	38.0	3	2	ICP-MS	1.50	-0.21	-0.83	a
LC0048	39.0			ICP-MS	0	-0.08	-0.49	b
LC0050	42.4	0.63	1	ICP-MS	0.63	0.35	1.91	b
LC0055	39.8	6.0	2	ICP-MS	3.00	0.03	0.06	a
LC0056	42.3	8.2	2	ICP-MS	4.10	0.34	0.62	a
LC0059	40.2	6.4	2	ICP-MS	3.20	0.08	0.17	a
LC0061	38.0			ICP-MS	0	-0.21	-1.25	b
LC0062	34.4			ICP-MS	0	-0.65	-3.94	b
LC0067	39.1				0	-0.07	-0.42	b
LC0068	43.2	4.58	2	ICP-MS	2.29	0.45	1.34	a
LC0101	41.8	8.6	2	ICP-MS	4.30	0.27	0.48	a
LC0113	14.3	3.4	2	ICP-OES	1.70	-3.19	-11.77	a
LC0115	37.5	1.2	3.18	ICP-MS	0.38	-0.27	-1.55	b
LC0120	32.8	5	2	ICP-OES	2.50	-0.86	-2.40	a
LC0122	50.2	22.1	2	ICP-MS	11.05	1.33	0.95	c
LC0123	41.3			GF-AAS	0	0.22	1.29	b

[a] √3 is set by ILC coordinator when no coverage factor is reported. The reported uncertainty was assumed to have a rectangular distribution with k=√3

[b] Satisfactory, Questionable, Unsatisfactory

[c] "a": $u(x_{pt}) \leq u_{lab} \leq \sigma_{pt}$; "b": $u_{lab} < u(x_{pt})$; and "c": $u_{lab} > \sigma_{pt}$

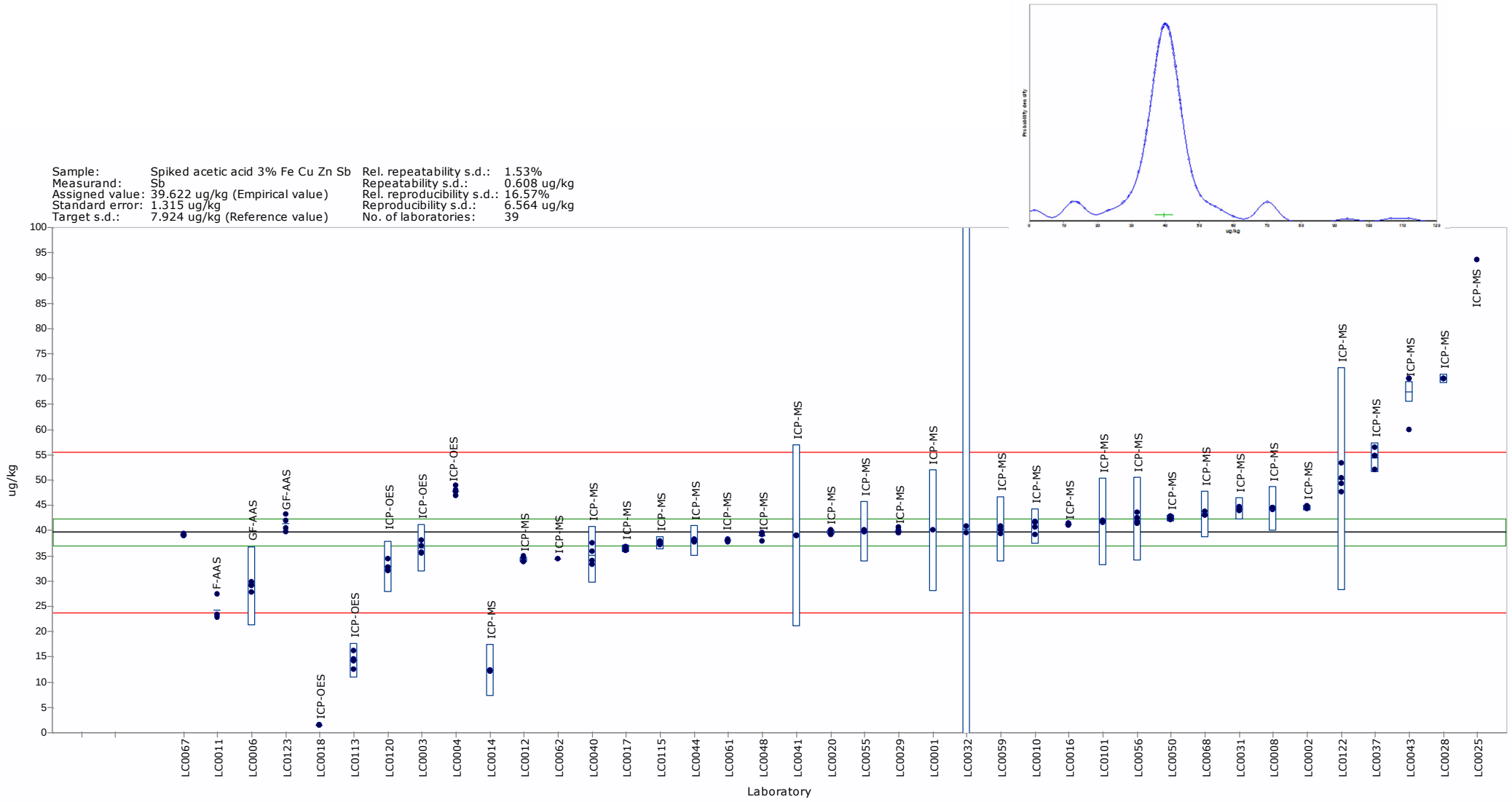

Figure 10. **Sb in S2**: Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (x_{pt}): solid black line; reference interval (x_{pt}±U(x_{pt})):dash green line; Target interval (x_{pt}±2σ_{pt}): solid red lines.

Table 11. **Pb in glass bowl (GB) I migration**,
Assigned range: x_{pt} = 569, u (x_{pt}) =15.2, σ_{pt} = 88.6 [all values are in µg/kg]

Lab code	x_i	U_i	k[a]	technique	u_i	z-score[b]	ζ-score	Uncert.[c]
LC0001	700	210	2	ICP-MS	105.00	1.48	1.24	c
LC0002	589	99.47	2	ICP-MS	49.74	0.24	0.40	a
LC0003	492	83.2	2	ICP-OES	41.60	-0.87	-1.74	a
LC0004	461			ICP-OES	0	-1.22	-7.12	b
LC0005	555	80	2	F-AAS	40.00	-0.15	-0.32	a
LC0006	693	83.1	2	GF-AAS	41.55	1.40	2.80	a
LC0007	559	19.22	2	F-AAS	9.61	-0.11	-0.55	b
LC0008	335	33.5	2	ICP-MS	16.75	-2.64	-10.34	a
LC0010	469	50.00	2	ICP-MS	25.00	-1.12	-3.40	a
LC0011	533			F-AAS	0	-0.40	-2.35	b
LC0012	581	22.6	2	ICP-MS	11.30	0.14	0.66	b
LC0013	567	77.7	2	GF-AAS	38.85	-0.02	-0.04	a
LC0014	575	173	2	ICP-MS	86.50	0.08	0.08	a
LC0016	664			GF-AAS	0	1.07	6.27	b
LC0017	610	43.81	2	ICP-MS	21.91	0.47	1.57	a
LC0018	521	51.8	1	ICP-OES	51.80	-0.53	-0.87	a
LC0020	624	38.6	3.18	ICP-MS	12.14	0.62	2.83	b
LC0022	512	180	2	ICP-OES	90.00	-0.64	-0.62	c
LC0025	651			ICP-MS	0	0.93	5.42	b
LC0028	270	4	2	ICP-MS	2.00	-3.37	-19.57	b
LC0029	687			ICP-MS	0	1.33	7.78	b
LC0031	536	22.5	2	ICP-MS	11.25	-0.37	-1.73	b
LC0032	608	43.83	√3	ICP-MS	25.31	0.45	1.34	a
LC0034	69	0.13	2	GF-AAS	0.06	-5.64	-32.96	b
LC0037	650	78.4	2	ICP-MS	39.20	0.92	1.93	a
LC0040	545	55.0	2	ICP-MS	27.50	-0.27	-0.76	a
LC0041	628	210	2	ICP-MS	105.00	0.67	0.56	c
LC0042	573	200	2	ICP-MS	100.00	0.05	0.04	c
LC0043	268	16	2	ICP-MS	8.00	-3.39	-17.54	b
LC0044	648	84	2	ICP-MS	42.00	0.89	1.77	a
LC0046	585			ICP-MS	0	0.18	1.07	b
LC0048	500	30.0	2	ICP-MS	15.00	-0.77	-3.20	b
LC0049	637			GF-AAS	0	0.77	4.51	b
LC0050	523	38.3	2	ICP-MS	19.15	-0.51	-1.87	a
LC0054	497	29	3.18	GF-AAS	9.12	-0.81	-4.08	b
LC0055	601	90.1	2	ICP-MS	45.05	0.36	0.68	a
LC0056	655	101.6	2	ICP-MS	50.80	0.98	1.64	a
LC0061	617	129.5	2	ICP-MS	64.75	0.55	0.73	a
LC0062	476	40.90	2	ICP-MS	20.45	-1.04	-3.63	a
LC0067	593				0	0.27	1.59	b
LC0068	582	36.37	3.18	ICP-MS	11.43	0.15	0.69	b
LC0097	445	178.1	2	GF-AAS	89.05	-1.39	-1.37	c
LC0101	649	84.4	2	ICP-MS	42.20	0.91	1.79	a
LC0113	186	17.5	2	ICP-OES	8.75	-4.32	-21.88	b
LC0115	575	114.4	3.18	ICP-MS	35.95	0.08	0.17	a
LC0116	582			ICP-MS	0	0.15	0.89	b
LC0118	586	203	2	ICP-OES	101.50	0.19	0.17	c
LC0120	539	173	2	ICP-OES	86.50	-0.33	-0.33	a
LC0121	423	154.0	2		77.00	-1.64	-1.86	a
LC0122	588	203.8	2	ICP-MS	101.90	0.22	0.19	c
LC0123	627	42	2	GF-AAS	21.00	0.66	2.26	a
LC0124	549	16.14	2	ICP-MS	8.07	-0.22	-1.12	b

[a] √3 is set by ILC coordinator when no coverage factor is reported. The reported uncertainty was assumed to have a rectangular distribution with k=√3
[b] Satisfactory, Questionable, Unsatisfactory [c] "a": $u(x_{pt}) \leq u_{lab} \leq \sigma_{pt}$; "b": $u_{lab} < u(x_{pt})$; and "c": $u_{lab} > \sigma_p$

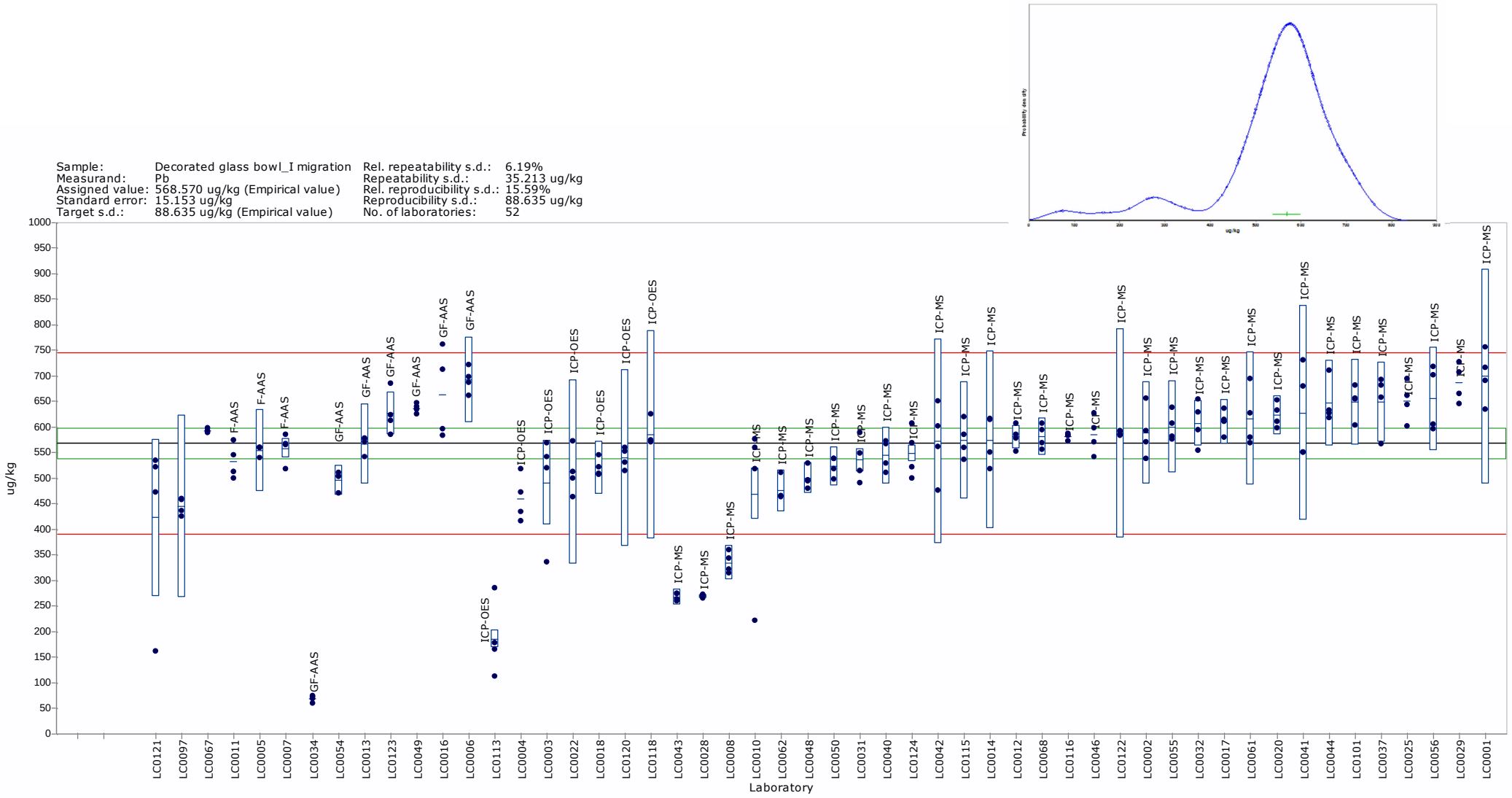

Figure 11. **Pb in GB (I migration)**: Measurement results and associated uncertainties (reported uncertainties shown). Reference value (x_{pt}): solid black line; reference interval ($x_{pt}\pm U(x_{pt})$):dash green line; Target interval ($x_{pt}\pm 2\sigma_{pt}$): solid red lines.

Table 12. **Co in glass bowl (GB) I migration**,
Assigned range: x_{pt}= 52.4, u (x_{pt}) =1.27, σ_{pt} = 7.97 [all values are in µg/kg]

Lab code	x_i	U_i	k[a]	technique	u_i	z-score[b]	ζ-score	Uncert.[c]
LC0001	56.4	17	2	ICP-MS	8.50	0.49	0.46	c
LC0002	56.5	11.71	2	ICP-MS	5.85	0.50	0.67	a
LC0003	45.3	7.6	2	ICP-OES	3.80	-0.89	-1.78	a
LC0004	47.4			ICP-OES	0	-0.63	-3.97	b
LC0006	47.1	11.8	2	GF-AAS	5.90	-0.67	-0.89	a
LC0007	44.5	0.85	2	GF-AAS	0.42	-0.99	-5.90	b
LC0008	29.5	2.9	2	ICP-MS	1.45	-2.88	-11.92	a
LC0010	46.1	5.00	2	ICP-MS	2.50	-0.80	-2.27	a
LC0011	42.5			F-AAS	0	-1.25	-7.83	b
LC0012	54.0	2.04	2	ICP-MS	1.02	0.20	0.96	b
LC0013	44.3	2.6	2	GF-AAS	1.30	-1.02	-4.47	a
LC0014	55.7	18.4	2	ICP-MS	9.20	0.40	0.35	c
LC0016	55.0			ICP-MS	0	0.32	1.98	b
LC0017	59.6	3.26	2	ICP-MS	1.63	0.90	3.45	a
LC0018	46.9	4.5	1	ICP-OES	4.50	-0.70	-1.19	a
LC0020	58.2	2.6	3.18	ICP-MS	0.82	0.72	3.81	b
LC0022	54.5	24	2	ICP-OES	12.00	0.26	0.17	c
LC0025	61.2			ICP-MS	0	1.10	6.89	b
LC0028	25.7	0.6	2	ICP-MS	0.30	-3.35	-20.47	b
LC0029	63.8			ICP-MS	0	1.42	8.91	b
LC0031	57.5	2.9	2	ICP-MS	1.45	0.63	2.61	a
LC0032	58.3	5.06	√3	ICP-MS	2.92	0.73	1.82	a
LC0037	58.6	6.9	2	ICP-MS	3.45	0.78	1.68	a
LC0040	50.2	5.0	2	ICP-MS	2.50	-0.28	-0.81	a
LC0043	25.3	1.4	2	ICP-MS	0.70	-3.41	-18.73	b
LC0044	58.0	10	2	ICP-MS	5.00	0.70	1.08	a
LC0048	49.7			ICP-MS	0	-0.35	-2.18	b
LC0050	49.7	3.4	2	ICP-MS	1.70	-0.34	-1.28	a
LC0054	57.5	4.7	3.18	GF-AAS	1.48	0.64	2.61	a
LC0055	55.9	8.4	2	ICP-MS	4.20	0.43	0.79	a
LC0056	49.7	7.00	2	ICP-MS	3.50	-0.35	-0.74	a
LC0061	59.5		2	ICP-MS	0.00	0.89	5.56	b
LC0062	43.6	4.26	2	ICP-MS	2.13	-1.11	-3.57	a
LC0067	62.6				0	1.27	7.98	b
LC0068	50.9	2.71	3.18	ICP-MS	0.85	-0.19	-0.99	b
LC0097	58.5	25.7	2	GF-AAS	12.85	0.75	0.47	c
LC0101	56.7	7.9	2	ICP-MS	3.97	0.53	1.02	a
LC0113	45.9	4.8	2	ICP-OES	2.40	-0.82	-2.40	a
LC0115	52.4	10.2	3.18	ICP-MS	3.21	0.00	-0.01	a
LC0116	54.2			ICP-MS	0	0.21	1.35	b
LC0118	56.7	24.9	2	ICP-OES	12.45	0.53	0.34	c
LC0120	49.1	18	2	ICP-OES	9.00	-0.42	-0.37	c
LC0122	53.7	23.6	2	ICP-MS	11.80	0.16	0.11	c

[a] √3 is set by ILC coordinator when no coverage factor is reported. The reported uncertainty was assumed to have a rectangular distribution with k=√3
[b] Satisfactory, Questionable, Unsatisfactory
[c] "a": $u(x_{pt}) \leq u_{lab} \leq \sigma_{pt}$; "b": $u_{lab} < u(x_{pt})$; and "c": $u_{lab} > \sigma_{pt}$

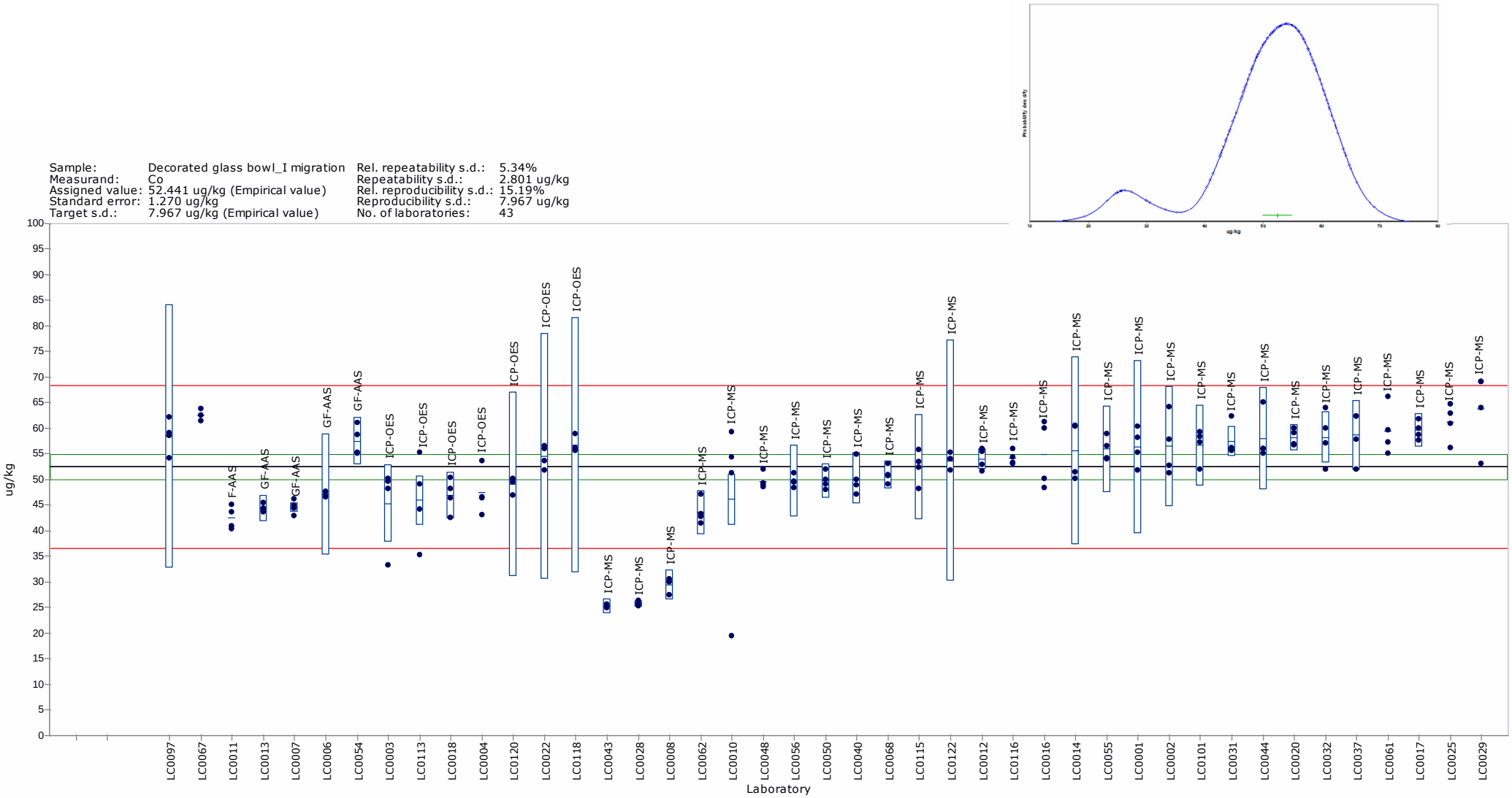

Figure 12. **Co in GB (I migration)**: Measurement results and associated uncertainties (reported uncertainties shown). Reference value (x_{pt}): solid black line; reference interval (x_{pt}±U(x_{pt})):dash green line; Target interval (x_{pt}±2σ_{pt}): solid red lines.

Table 13. **Cd in glass bowl (GB) I migration**,
Assigned range: x_{pt} = 33.2, u (x_{pt}) =0.77, σ_{pt} = 4.82 [all values are in µg/kg]

Lab code	x_i	U_i	k[a]	technique	u_i	z-score[b]	ζ-score	Uncert.[c]
LC0001	36.9	11	2	ICP-MS	5.50	0.76	0.66	c
LC0002	32.4	3.64	2	ICP-MS	1.82	-0.17	-0.41	a
LC0003	33.4	5.6	2	ICP-OES	2.80	0.02	0.04	a
LC0004	30.6			ICP-OES	0	-0.54	-3.41	b
LC0005	35.3	5	2	F-AAS	2.50	0.42	0.77	a
LC0006	32.1	6.1	2	GF-AAS	3.05	-0.24	-0.36	a
LC0007	38.0	1.81	2	GF-AAS	0.90	0.99	4.01	a
LC0008	20.5	2.1	2	ICP-MS	1.05	-2.64	-9.78	a
LC0010	26.7	3.00	2	ICP-MS	1.50	-1.37	-3.91	a
LC0011	29.6			F-AAS	0	-0.75	-4.72	b
LC0012	33.6	1.83	2	ICP-MS	0.92	0.08	0.33	a
LC0013	33.1	6.6	2	GF-AAS	3.30	-0.04	-0.05	a
LC0014	30.3	11.0	2	ICP-MS	5.50	-0.61	-0.53	c
LC0016	38.7			GF-AAS	0	1.14	7.14	b
LC0017	32.4	2.78	2	ICP-MS	1.39	-0.18	-0.54	a
LC0018	30.9	2.1	1	ICP-OES	2.10	-0.49	-1.04	a
LC0020	35.3	2	3.18	ICP-MS	0.63	0.42	2.04	b
LC0022	33.9	15	2	ICP-OES	7.50	0.14	0.09	c
LC0025	40.5			ICP-MS	0	1.51	9.53	b
LC0028	15.9	0.5	2	ICP-MS	0.25	-3.60	-21.57	b
LC0029	39.0			ICP-MS	0	1.19	7.47	b
LC0031	33.3	1.1	2	ICP-MS	0.55	0.01	0.07	b
LC0032	34.3	1.50	√3	ICP-MS	0.87	0.21	0.88	a
LC0034	7.28	0.122	2	GF-AAS	0.06	-5.39	-33.81	b
LC0037	37.9	5.0	2	ICP-MS	2.50	0.96	1.77	a
LC0040	35.6	3.6	2	ICP-MS	1.80	0.48	1.19	a
LC0041	30.0	16	2	ICP-MS	8.00	-0.67	-0.40	c
LC0042	30.2	13	2	ICP-MS	6.50	-0.62	-0.46	c
LC0043	15.6	1.5	2	ICP-MS	0.75	-3.66	-16.44	b
LC0044	37.0	6	2	ICP-MS	3.00	0.78	1.22	a
LC0046	32.7			ICP-MS	0	-0.11	-0.72	b
LC0048	31.8	1.6	2	ICP-MS	0.80	-0.29	-1.27	a
LC0049	36.7			GF-AAS	0	0.71	4.50	b
LC0050	31.6	1.9	2	ICP-MS	0.95	-0.33	-1.32	a
LC0054	33.4	3.0	3.18	GF-AAS	0.94	0.03	0.14	a
LC0055	36.9	5.5	2	ICP-MS	2.75	0.76	1.28	a
LC0056	50.5	7.90	2	ICP-MS	3.95	3.59	4.30	a
LC0061	37.1	10.0	2	ICP-MS	5.00	0.80	0.76	c
LC0062	24.4	2.19	2	ICP-MS	1.10	-1.84	-6.64	a
LC0067	29.8				0	-0.71	-4.47	b
LC0068	32.9	2.14	3.18	ICP-MS	0.67	-0.07	-0.33	b
LC0097	32.8	14.4	2	GF-AAS	7.20	-0.10	-0.07	c
LC0101	37.1	5.6	2	ICP-MS	2.80	0.80	1.32	a
LC0113	29.2	3.5	2	ICP-OES	1.75	-0.84	-2.11	a
LC0115	30.7	4.9	3.18	ICP-MS	1.54	-0.53	-1.47	a
LC0116	34.9			ICP-MS	0	0.34	2.14	b
LC0118	32.4	14.3	2	ICP-OES	7.15	-0.18	-0.12	c
LC0120	31.4	9	2	ICP-OES	4.50	-0.38	-0.40	a
LC0121	25.7	11.3	2		5.65	-1.56	-1.32	c
LC0122	40.9	18.0	2	ICP-MS	9.00	1.58	0.84	c
LC0123	36.9	0.9	2	GF-AAS	0.45	0.75	4.07	b
LC0124	32.0	2.02	2	ICP-MS	1.01	-0.26	-0.98	a

[a] √3 is set by ILC coordinator when no coverage factor is reported. The reported uncertainty was assumed to have a rectangular distribution with k=√3
[b] Satisfactory, Questionable, Unsatisfactory
[c] "a": $u(x_{pt}) \leq u_{lab} \leq \sigma_{pt}$; "b": $u_{lab} < u(x_{pt})$; and "c": $u_{lab} > \sigma_{pt}$

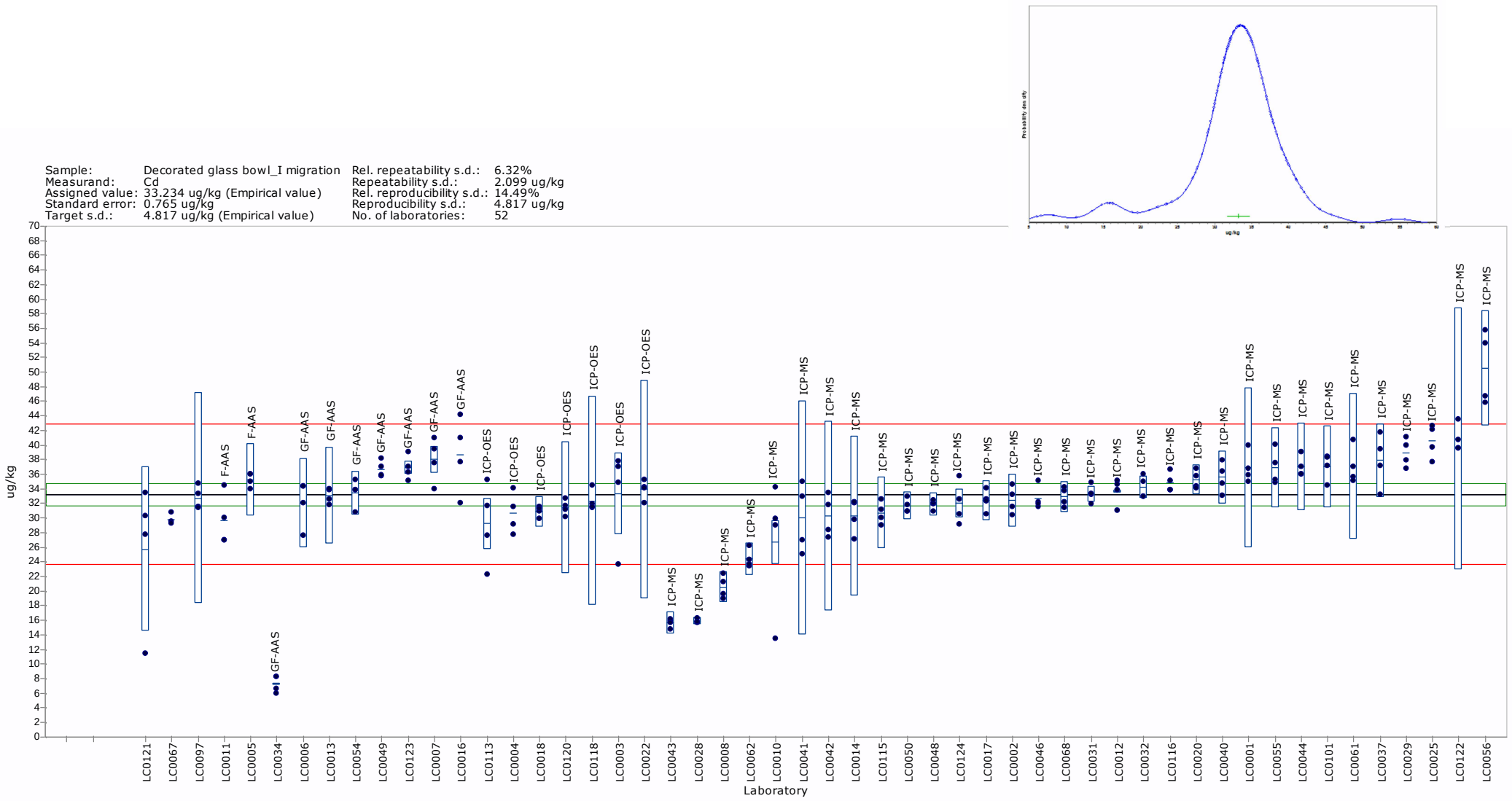

Figure 13. **Cd in GB (I migration)**: Measurement results and associated uncertainties (reported uncertainties shown). Reference value (x_{pt}): solid black line; reference interval (x_{pt}±U(x_{pt})):dash green line; Target interval (x_{pt}±2σ_{pt}): solid red lines.

Table 14. **Co in glass bowl (GB) II migration**,
Assigned range: x_{pt}= 3.23, u (x_{pt}) =0.19, σ_{pt} = 0.94 [all values are in µg/kg]

Lab code	x_i	U_i	k[a]	technique	u_i	z-score[b]	ζ-score	Uncert.[c]
LC0001	2.80	0.8	2	ICP-MS	0.40	-0.46	-0.97	a
LC0002	3.09	0.21	2	ICP-MS	0.11	-0.15	-0.66	b
LC0003	3.90	0.8	2	ICP-OES	0.40	0.71	1.51	a
LC0004	2.58			ICP-OES	0	-0.70	-3.41	b
LC0006	2.90	1.1	2	GF-AAS	0.55	-0.35	-0.57	a
LC0007	2.26	0.09	2	GF-AAS	0.05	-1.03	-4.92	b
LC0008	3.55	0.4	2	ICP-MS	0.20	0.34	1.16	a
LC0012	2.45	0.05	2	ICP-MS	0.03	-0.83	-4.03	b
LC0014	3.42	1.72	2	ICP-MS	0.86	0.20	0.22	a
LC0016	4.44			ICP-MS	0	1.28	6.28	b
LC0017	3.85	0.40	2	ICP-MS	0.20	0.66	2.24	a
LC0018	4.48	0.4	1	ICP-OES	0.40	1.33	2.81	a
LC0020	3.28	0.2	3.18	ICP-MS	0.06	0.05	0.22	b
LC0022	2.83	1.2	2	ICP-OES	0.60	-0.43	-0.64	a
LC0025	4.48			ICP-MS	0	1.33	6.49	b
LC0028	1.40	0.03	2	ICP-MS	0.02	-1.95	-9.50	b
LC0029	2.58			ICP-MS	0	-0.70	-3.41	b
LC0031	3.18	0.2	2	ICP-MS	0.10	-0.06	-0.25	b
LC0032	4.81	0.35	√3	ICP-MS	0.20	1.69	5.68	a
LC0037	3.70	0.2	2	ICP-MS	0.10	0.50	2.17	b
LC0040	2.90	0.29	2	ICP-MS	0.15	-0.36	-1.39	b
LC0043	1.35	0.08	2	ICP-MS	0.04	-2.00	-9.59	b
LC0044	3.50	0.6	2	ICP-MS	0.30	0.29	0.76	a
LC0048	2.85			ICP-MS	0	-0.40	-1.98	b
LC0050	3.43	0.2	2	ICP-MS	0.10	0.21	0.90	b
LC0055	3.00	0.5	2	ICP-MS	0.25	-0.25	-0.73	a
LC0056	1.65	0.20	2	ICP-MS	0.10	-1.68	-7.30	b
LC0061	3.28			ICP-MS	0.00	0.05	0.24	b
LC0062	5.10	0.59	2	ICP-MS	0.29	1.99	5.30	a
LC0067	1.98				0	-1.33	-6.49	b
LC0068	2.69	0.36	3.18	ICP-MS	0.11	-0.58	-2.45	b
LC0101	5.81	0.81	2	ICP-MS	0.40	2.74	5.75	a
LC0115	4.30	0.5	3.18	ICP-MS	0.16	1.14	4.31	b
LC0118	3.25	1.5	2	ICP-OES	0.75	0.02	0.03	a
LC0120	3.11	1	2	ICP-OES	0.50	-0.13	-0.23	a
LC0122	2.90	1.3	2	ICP-MS	0.65	-0.35	-0.49	a

[a] $\sqrt{3}$ is set by ILC coordinator when no coverage factor is reported. The reported uncertainty was assumed to have a rectangular distribution with k=$\sqrt{3}$
[b] Satisfactory, Questionable, Unsatisfactory
[c] "a": $u(x_{pt}) \leq u_{lab} \leq \sigma_{pt}$; "b": $u_{lab} < u(x_{pt})$; and "c": $u_{lab} > \sigma_{pt}$

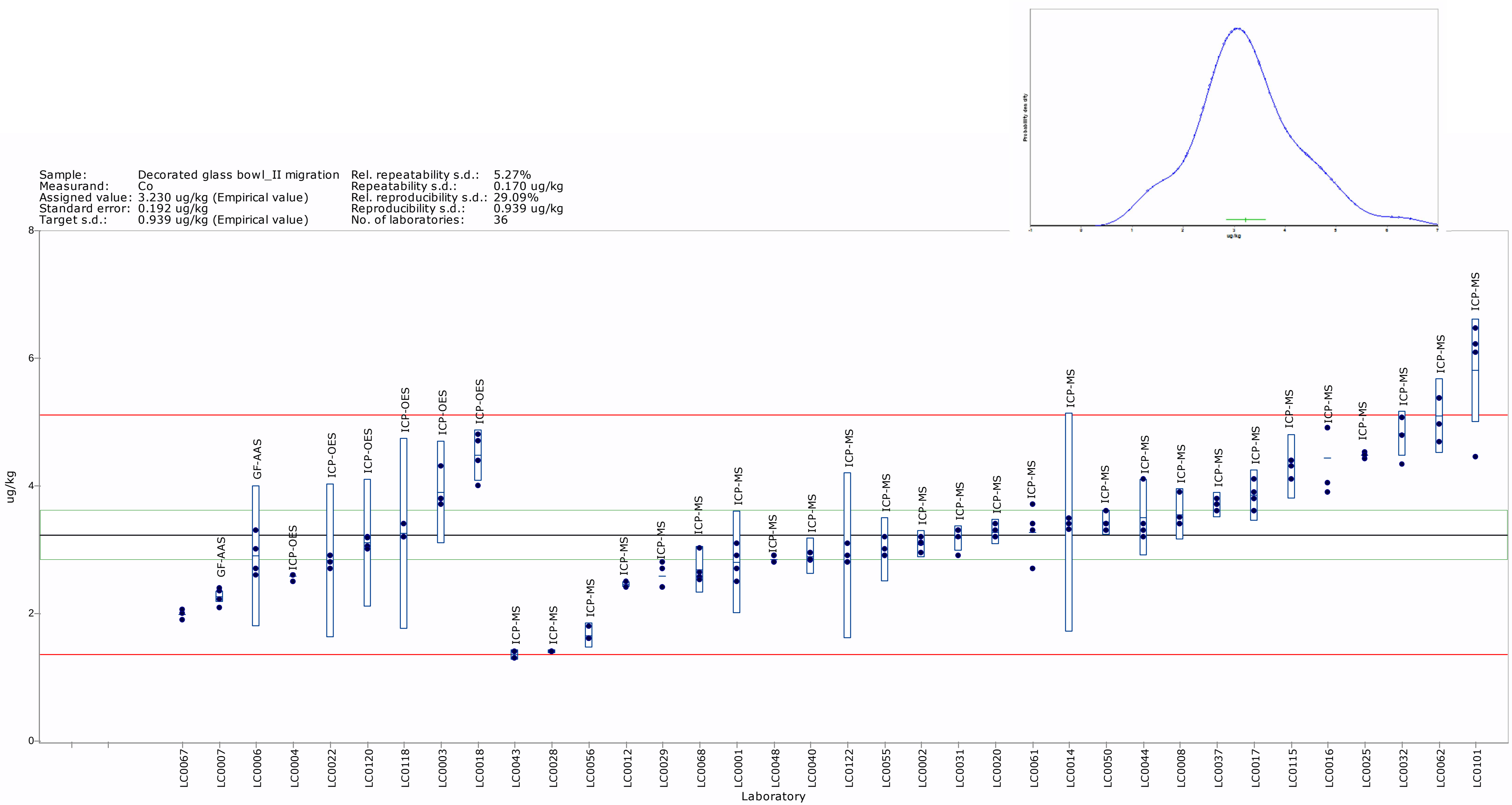

Figure 14. **Co in GB (II migration)**: Measurement results and associated uncertainties (reported uncertainties shown). Reference value (x_{pt}): solid black line; reference interval ($x_{pt} \pm U(x_{pt})$):dash green line; Target interval ($x_{pt} \pm 2\sigma_{pt}$): solid red lines.

Table 15. **Pb in glass bowl (GB) II migration**,
Assigned range: x_{pt} = 48.0, u (x_{pt}) =1.79, σ_{pt} = 10.8 [all values are in µg/kg]

Lab code	x_i	U_i	k[a]	technique	u_i	z-score[b]	ζ-score	Uncert.[c]
LC0001	39.1	12	2	ICP-MS	6.00	-0.83	-1.43	a
LC0002	50.9	6.28	2	ICP-MS	3.14	0.27	0.81	a
LC0003	47.5	10.2	2	ICP-OES	5.10	-0.05	-0.09	a
LC0004	42.2			ICP-OES	0	-0.54	-3.24	b
LC0006	45.7	7.3	2	GF-AAS	3.65	-0.21	-0.56	a
LC0007	48.5	1.49	2	GF-AAS	0.75	0.05	0.26	b
LC0008	55.4	5.5	2	ICP-MS	2.75	0.68	2.25	a
LC0010	55.0	5.00	2	ICP-MS	2.50	0.65	2.29	a
LC0011	59.0			F-AAS	0	1.02	6.16	b
LC0012	40.2	1.83	2	ICP-MS	0.92	-0.72	-3.87	b
LC0013	48.2	5.3	2	GF-AAS	2.65	0.02	0.06	a
LC0014	54.7	18.2	2	ICP-MS	9.10	0.62	0.72	a
LC0016	74.2			GF-AAS	0	2.43	14.66	b
LC0017	54.8	5.78	2	ICP-MS	2.89	0.63	2.01	a
LC0018	75.6	7.5	1	ICP-OES	7.50	2.55	3.58	a
LC0020	50.5	3.6	3.18	ICP-MS	1.13	0.23	1.18	b
LC0022	39.8	18	2	ICP-OES	9.00	-0.76	-0.89	a
LC0025	51.4			ICP-MS	0	0.31	1.90	b
LC0028	22.2	0.3	2	ICP-MS	0.15	-2.38	-14.33	b
LC0029	43.0			ICP-MS	0	-0.46	-2.76	b
LC0031	47.9	2.0	2	ICP-MS	1.00	-0.01	-0.06	b
LC0032	67.0	3.52	√3	ICP-MS	2.03	1.75	7.00	a
LC0034	29.6	0.13	2	GF-AAS	0.07	-1.70	-10.24	b
LC0037	55.8	2.0	2	ICP-MS	1.00	0.72	3.79	b
LC0040	46.8	4.7	2	ICP-MS	2.35	-0.11	-0.40	a
LC0041	44.8	23	2	ICP-MS	11.50	-0.30	-0.28	c
LC0042	45.3	20	2	ICP-MS	10.00	-0.25	-0.27	a
LC0043	22.3	1.5	2	ICP-MS	0.75	-2.37	-13.23	b
LC0044	56.8	7	2	ICP-MS	3.50	0.81	2.23	a
LC0048	43.5	2.6	2	ICP-MS	1.30	-0.42	-2.03	b
LC0049	58.2			GF-AAS	0	0.95	5.72	b
LC0050	52.3	3.0	2	ICP-MS	1.50	0.40	1.83	b
LC0054	38.8	4.3	3.18	GF-AAS	1.35	-0.85	-4.09	b
LC0055	49.3	7.4	2	ICP-MS	3.70	0.13	0.33	a
LC0056	45.4	7.0	2	ICP-MS	3.50	-0.24	-0.67	a
LC0061	54.7	11.4	2	ICP-MS	5.70	0.62	1.12	a
LC0062	74.6	4.98	2	ICP-MS	2.49	2.46	8.68	a
LC0067	31.3				0	-1.54	-9.32	b
LC0068	43.9	5.40	3.18	ICP-MS	1.70	-0.37	-1.64	b
LC0097	46.8	18.7	2	GF-AAS	9.35	-0.11	-0.12	a
LC0101	97.7	12.7	2	ICP-MS	6.35	4.59	7.53	a
LC0113	21.8	2.6	2	ICP-OES	1.30	-2.42	-11.82	b
LC0115	57.3	4.8	3.18	ICP-MS	1.51	0.86	3.99	b
LC0116	53.1			ICP-MS	0	0.47	2.85	b
LC0118	43.8	19.3	2	ICP-OES	9.65	-0.39	-0.43	a
LC0120	50.6	16	2	ICP-OES	8.00	0.24	0.32	a
LC0121	41.6	18.3	2		9.15	-0.59	-0.69	a
LC0122	46.7	20.3	2	ICP-MS	10.15	-0.12	-0.13	a
LC0123	54.9	9.0	2	GF-AAS	4.50	0.64	1.43	a
LC0124	28.3	0.66	2	ICP-MS	0.33	-1.81	-10.78	b

[a] √3 is set by ILC coordinator when no coverage factor is reported. The reported uncertainty was assumed to have a rectangular distribution with k=√3
[b] Satisfactory, Questionable, Unsatisfactory
[c] "a": $u(x_{pt}) \leq u_{lab} \leq \sigma_{pt}$; "b": $u_{lab} < u(x_{pt})$; and "c": $u_{lab} > \sigma_{pt}$

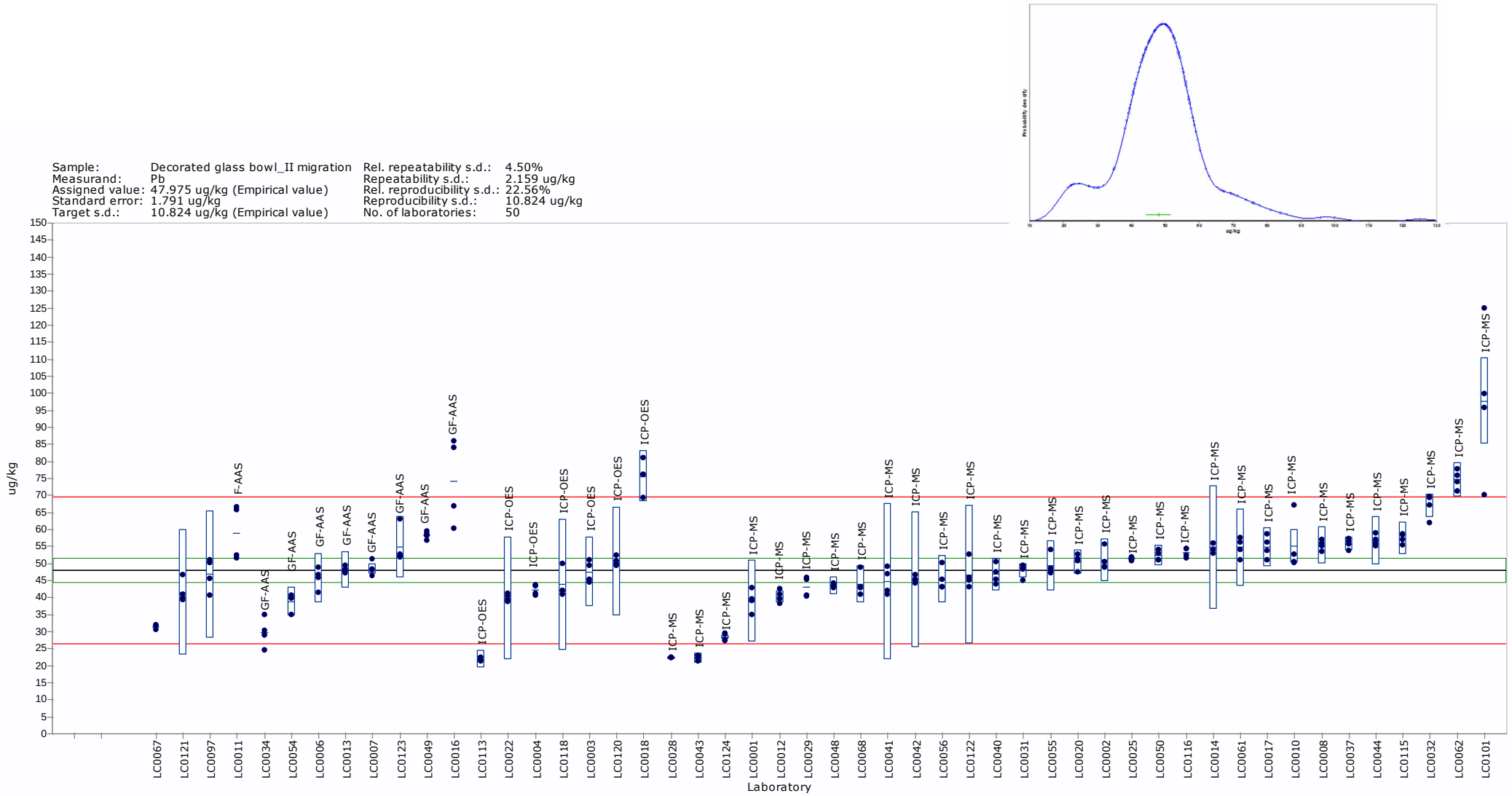

Figure 15. **Pb in GB (II migration)**: Measurement results and associated uncertainties (reported uncertainties shown). Reference value (x_{pt}): solid black line; reference interval (x_{pt}±U(x_{pt})):dash green line; Target interval (x_{pt}±2σ_{pt}): solid red lines.

Table 16. **Cd in glass bowl (GB) II migration**,
Assigned range: x_{pt}= 3.83, u (x_{pt}) =0.21, σ_{pt} = 1.11 [all values are in µg/kg]

Lab code	x_i	U_i	k[a]	technique	u_i	z-score[b]	ζ-score	Uncert.[c]
LC0001	2.88	0.9	2	ICP-MS	0.45	-0.86	-1.92	a
LC0002	3.41	0.16	2	ICP-MS	0.08	-0.38	-1.87	b
LC0003	6.45	1.1	2	ICP-OES	0.55	2.36	4.46	a
LC0004	2.58			ICP-OES	0	-1.13	-6.00	b
LC0006	3.10	0.7	2	GF-AAS	0.35	-0.66	-1.79	a
LC0007	3.44	0.11	2	GF-AAS	0.05	-0.35	-1.79	b
LC0008	3.78	0.4	2	ICP-MS	0.20	-0.05	-0.19	b
LC0011	4.75			F-AAS	0	0.83	4.41	b
LC0012	4.49	0.21	2	ICP-MS	0.11	0.60	2.83	b
LC0013	3.08	0.2	2	GF-AAS	0.10	-0.68	-3.25	b
LC0014	3.41	11.00	2	ICP-MS	5.50	-0.38	-0.08	c
LC0016	4.70			GF-AAS	0	0.79	4.17	b
LC0017	3.85	0.36	2	ICP-MS	0.18	0.02	0.08	b
LC0018	5.63	0.4	1	ICP-OES	0.40	1.62	3.98	a
LC0020	3.43	0.4	3.18	ICP-MS	0.13	-0.36	-1.65	b
LC0022	2.98	1.3	2	ICP-OES	0.65	-0.77	-1.25	a
LC0025	5.06			ICP-MS	0	1.11	5.90	b
LC0028	1.55	0.05	2	ICP-MS	0.03	-2.05	-10.83	b
LC0029	4.38			ICP-MS	0	0.49	2.62	b
LC0031	4.23	0.1	2	ICP-MS	0.05	0.36	1.85	b
LC0032	4.67	0.21	√3	ICP-MS	0.12	0.76	3.49	b
LC0034	3.23	0.122	2	GF-AAS	0.06	-0.54	-2.77	b
LC0037	3.78	0.2	2	ICP-MS	0.10	-0.05	-0.23	b
LC0040	5.50	0.55	2	ICP-MS	0.28	1.50	4.83	a
LC0042	2.81	1.2	2	ICP-MS	0.60	-0.92	-1.61	a
LC0043	1.50	0.1	2	ICP-MS	0.05	-2.10	-10.84	b
LC0044	3.55	0.6	2	ICP-MS	0.30	-0.25	-0.76	a
LC0048	3.18	0.2	2	ICP-MS	0.10	-0.59	-2.82	b
LC0049	3.99			GF-AAS	0	0.14	0.75	b
LC0050	3.75	0.3	2	ICP-MS	0.15	-0.07	-0.31	b
LC0054	3.40	0.5	3.18	GF-AAS	0.16	-0.39	-1.64	b
LC0055	3.78	0.6	2	ICP-MS	0.30	-0.05	-0.15	a
LC0056	11.4	1.80	2	ICP-MS	0.90	6.78	8.14	a
LC0061	5.88	1.5	2	ICP-MS	0.75	1.84	2.63	a
LC0062	5.27	0.40	2	ICP-MS	0.20	1.30	4.99	b
LC0067	2.23				0	-1.44	-7.67	b
LC0068	3.07	0.54	3.18	ICP-MS	0.17	-0.68	-2.81	b
LC0097	3.73	1.6	2	GF-AAS	0.80	-0.09	-0.13	a
LC0101	8.45	1.27	2	ICP-MS	0.63	4.16	6.91	a
LC0115	4.45	0.6	3.18	ICP-MS	0.19	0.56	2.21	b
LC0118	3.05	1.4	2	ICP-OES	0.70	-0.70	-1.07	a
LC0120	3.32	1	2	ICP-OES	0.50	-0.46	-0.94	a
LC0121	3.25	1.4	2		0.70	-0.52	-0.79	a
LC0122	7.03	3.1	2	ICP-MS	1.55	2.88	2.04	c
LC0123	3.90	0.5	2	GF-AAS	0.25	0.07	0.22	a
LC0124	4.39	0.20	2	ICP-MS	0.10	0.51	2.43	b

[a] √3 is set by ILC coordinator when no coverage factor is reported. The reported uncertainty was assumed to have a rectangular distribution with k=√3
[b] Satisfactory, Questionable, Unsatisfactory
[c] "a": $u(x_{pt}) \leq u_{lab} \leq \sigma_{pt}$; "b": $u_{lab} < u(x_{pt})$; and "c": $u_{lab} > \sigma_{pt}$

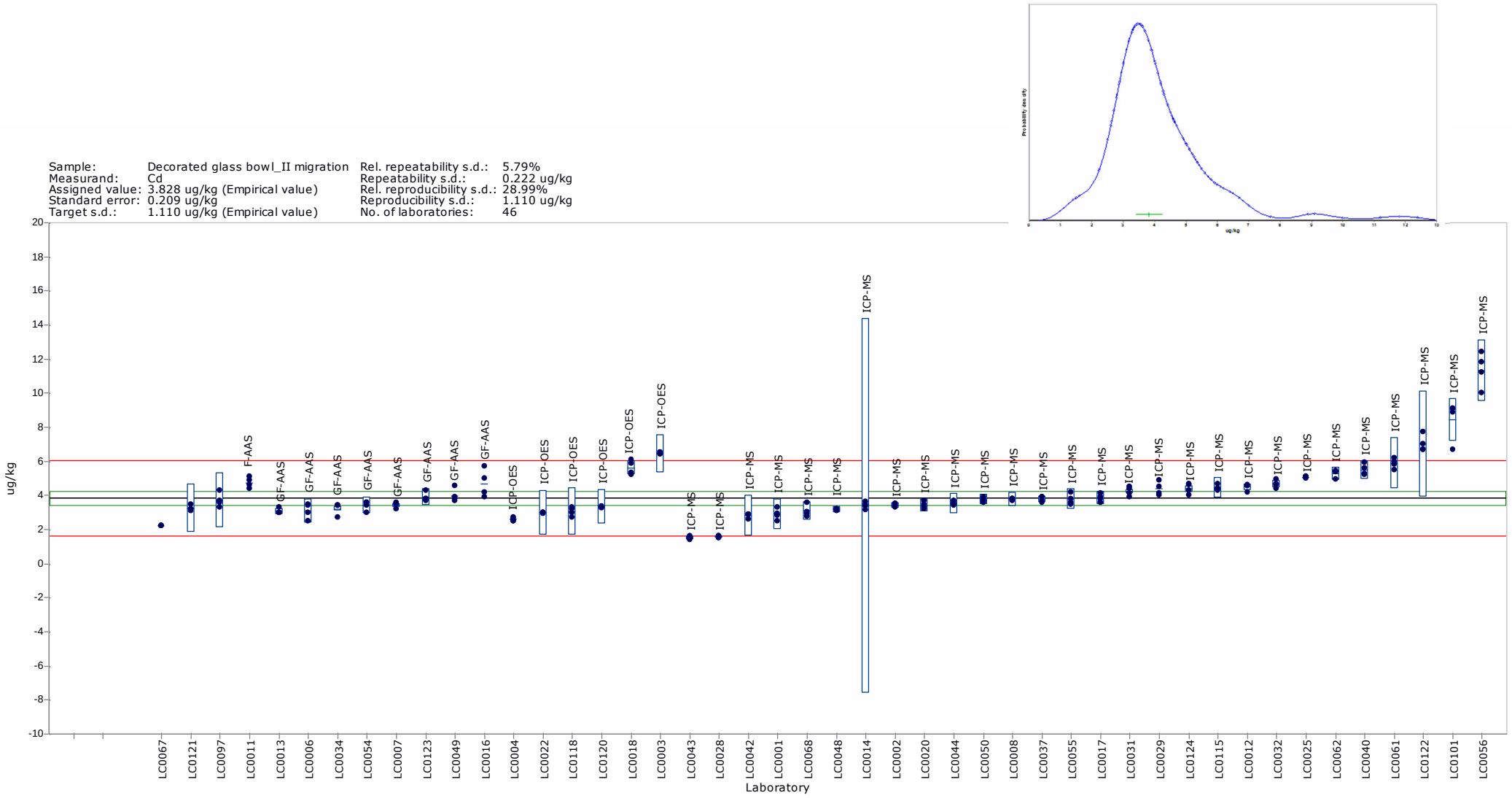

Figure 16. **Cd in GB (II migration)**: Measurement results and associated uncertainties (reported uncertainties shown). Reference value (x_{pt}): solid black line; reference interval (x_{pt}±U(x_{pt})):dash green line; Target interval (x_{pt}±2σ_{pt}): solid red lines.

Table 17. **Co in glass bowl (GB) III migration**,
Assigned range: x_{pt} = 2.33, u (x_{pt}) =0.17, σ_{pt} = 0.84 [all values are in µg/kg]

Lab code	x_i	U_i	k[a]	technique	u_i	z-score[b]	ζ-score	Uncert.[c]
LC0001	2.23	0.7	2	ICP-MS	0.35	-0.12	-0.27	a
LC0002	2.32	0.14	2	ICP-MS	0.07	-0.01	-0.03	b
LC0003	3.35	0.8	2	ICP-OES	0.40	1.22	2.34	a
LC0004	1.75			ICP-OES	0	-0.69	-3.34	b
LC0006	2.30	0.9	2	GF-AAS	0.45	-0.03	-0.06	a
LC0007	1.98	0.04	2	GF-AAS	0.02	-0.42	-2.03	b
LC0008	1.83	0.18	2	ICP-MS	0.09	-0.60	-2.58	b
LC0012	1.93	0.05	2	ICP-MS	0.03	-0.48	-2.31	b
LC0014	2.05	1.11	2	ICP-MS	0.55	-0.34	-0.48	a
LC0016	4.00			ICP-MS	0	2.00	9.64	b
LC0017	2.90	0.11	2	ICP-MS	0.06	0.68	3.14	b
LC0018	1.80	0.2	1	ICP-OES	0.20	-0.63	-2.00	a
LC0020	2.63	0.5	3.18	ICP-MS	0.16	0.35	1.27	b
LC0022	4.65	2	2	ICP-OES	1.00	2.78	2.29	c
LC0025	3.56			ICP-MS	0	1.47	7.07	b
LC0028	1.10	0.03	2	ICP-MS	0.02	-1.47	-7.06	b
LC0029	1.95			ICP-MS	0	-0.45	-2.18	b
LC0031	2.25	0.2	2	ICP-MS	0.10	-0.09	-0.39	b
LC0032	0.86	0.08	√3	ICP-MS	0.05	-1.76	-8.20	b
LC0037	3.15	0.4	2	ICP-MS	0.20	0.98	3.10	a
LC0040	2.18	0.22	2	ICP-MS	0.11	-0.18	-0.71	b
LC0043	1.08	0.06	2	ICP-MS	0.03	-1.50	-7.13	b
LC0044	2.88	0.5	2	ICP-MS	0.25	0.65	1.80	a
LC0048	2.18			ICP-MS	0	-0.18	-0.89	b
LC0050	2.55	0.3	2	ICP-MS	0.15	0.27	0.97	b
LC0055	2.20	0.3	2	ICP-MS	0.15	-0.15	-0.56	b
LC0056	1.03	0.10	2	ICP-MS	0.05	-1.55	-7.18	b
LC0061	2.58			ICP-MS	0.00	0.30	1.42	b
LC0062	1.47	0.29	2	ICP-MS	0.15	-1.03	-3.80	b
LC0067	1.80				0	-0.63	-3.03	b
LC0068	2.00	0.38	3.18	ICP-MS	0.12	-0.39	-1.55	b
LC0101	1.86	0.26	2	ICP-MS	0.13	-0.56	-2.16	b
LC0115	3.58	0.4	3.18	ICP-MS	0.13	1.49	5.82	b
LC0118	3.05	1.4	2	ICP-OES	0.70	0.86	1.00	a
LC0120	5.32	2	2	ICP-OES	1.00	3.58	2.95	c
LC0122	2.98	1.3	2	ICP-MS	0.65	0.77	0.96	a

[a] $\sqrt{3}$ is set by ILC coordinator when no coverage factor is reported. The reported uncertainty was assumed to have a rectangular distribution with k=$\sqrt{3}$
[b] Satisfactory, Questionable, Unsatisfactory
[c] "a": $u(x_{pt}) \leq u_{lab} \leq \sigma_{pt}$; "b": $u_{lab} < u(x_{pt})$; and "c": $u_{lab} > \sigma_{pt}$

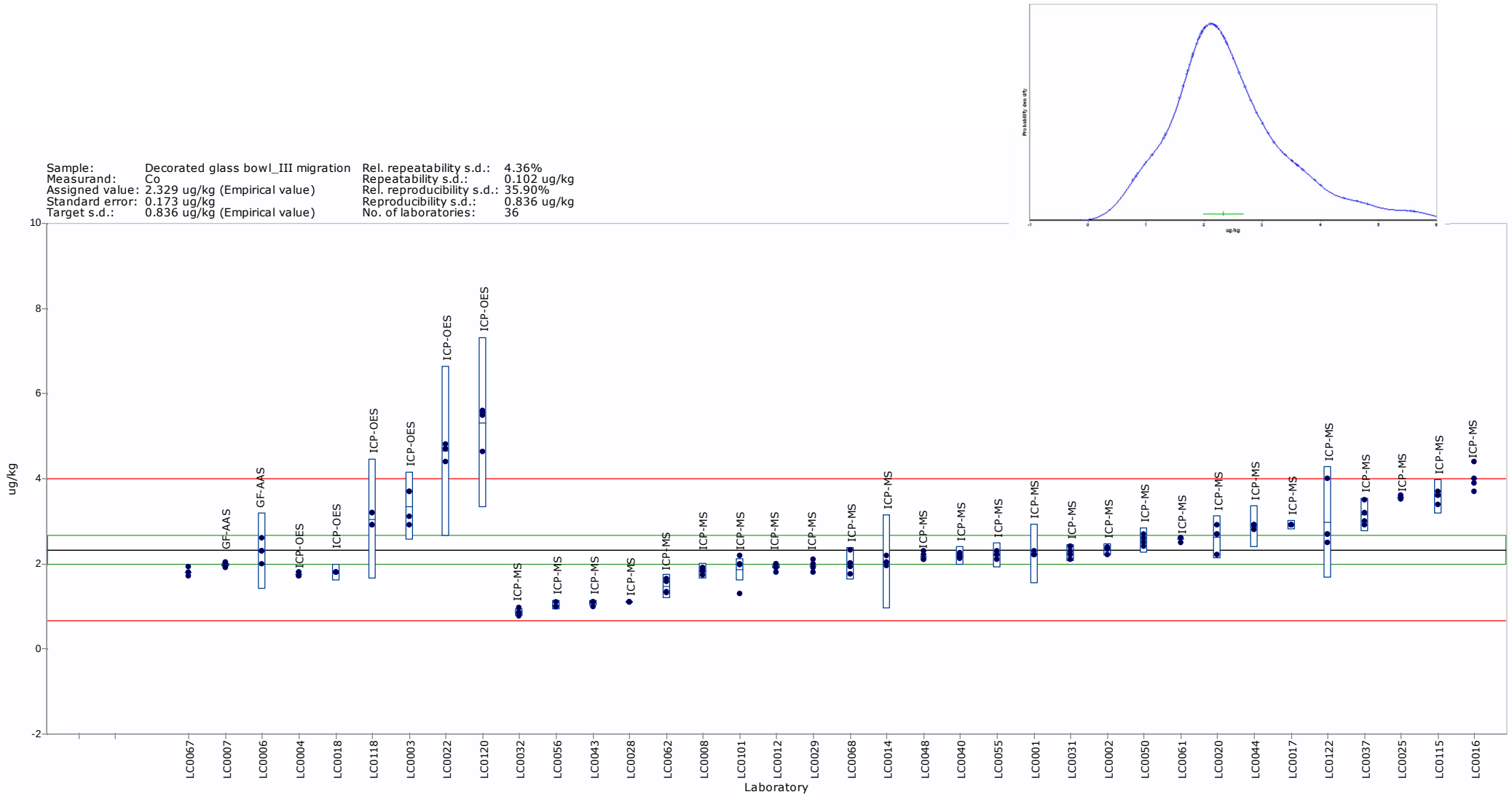

Figure 17. **Co in GB (III migration)**: Measurement results and associated uncertainties (reported uncertainties shown). Reference value (x_{pt}): solid black line; reference interval ($x_{pt}\pm U(x_{pt})$):dash green line; Target interval ($x_{pt}\pm 2\sigma_{pt}$): solid red lines.

Table 18. **Pb in glass bowl (GB) III migration**,
Assigned range: x_{pt} = 35.5, u (x_{pt}) =1.96, σ_{pt} = 10.6 [all values are in µg/kg]

Lab code	x_i	U_i	k[a]	technique	u_i	z-score[b]	ζ-score	Uncert.[c]
LC0001	29.5	9	2	ICP-MS	4.50	-0.57	-1.23	a
LC0002	37.4	1.72	2	ICP-MS	0.86	0.17	0.86	b
LC0003	49.0	10.5	2	ICP-OES	5.25	1.27	2.41	a
LC0004	26.8			ICP-OES	0	-0.83	-4.47	b
LC0006	41.2	6.6	2	GF-AAS	3.30	0.54	1.49	a
LC0007	36.9	1.20	2	GF-AAS	0.60	0.13	0.69	b
LC0008	35.2	3.52	2	ICP-MS	1.76	-0.03	-0.12	b
LC0010	45.6	5.00	2	ICP-MS	2.50	0.95	3.18	a
LC0011	42.8			F-AAS	0	0.69	3.72	b
LC0012	31.2	2.33	2	ICP-MS	1.17	-0.41	-1.89	b
LC0013	36.1	4	2	GF-AAS	2.00	0.06	0.22	a
LC0014	32.3	11.6	2	ICP-MS	5.80	-0.30	-0.53	a
LC0016	61.7			GF-AAS	0	2.47	13.38	b
LC0017	42.8	2.42	2	ICP-MS	1.21	0.68	3.15	b
LC0018	24.1	2.4	1	ICP-OES	2.40	-1.08	-3.70	a
LC0020	38.5	4.4	3.18	ICP-MS	1.38	0.28	1.25	b
LC0022	56.7	25	2	ICP-OES	12.50	2.00	1.68	c
LC0025	37.3			ICP-MS	0	0.17	0.91	b
LC0028	16.4	0.2	2	ICP-MS	0.10	-1.80	-9.75	b
LC0029	32.5			ICP-MS	0	-0.29	-1.56	b
LC0031	32.3	1.4	2	ICP-MS	0.70	-0.31	-1.56	b
LC0032	17.3	1.20	√3	ICP-MS	0.69	-1.72	-8.79	b
LC0034	22.0	0.13	2	GF-AAS	0.07	-1.28	-6.92	b
LC0037	42.9	6.4	2	ICP-MS	3.20	0.69	1.95	a
LC0040	33.7	3.4	2	ICP-MS	1.70	-0.17	-0.70	b
LC0041	32.0	17	2	ICP-MS	8.50	-0.33	-0.40	a
LC0042	30.4	13	2	ICP-MS	6.50	-0.48	-0.75	a
LC0043	16.1	1.1	2	ICP-MS	0.55	-1.84	-9.57	b
LC0044	45.3	6	2	ICP-MS	3.00	0.92	2.72	a
LC0048	32.2	0.2	2	ICP-MS	0.10	-0.31	-1.68	b
LC0049	91.0			GF-AAS	0	5.23	28.36	b
LC0050	36.8	3.4	2	ICP-MS	1.70	0.12	0.47	b
LC0054	54.0	4.6	3.18	GF-AAS	1.45	1.75	7.61	b
LC0055	34.1	5.1	2	ICP-MS	2.55	-0.13	-0.44	a
LC0056	28.4	6.80	2	ICP-MS	3.40	-0.67	-1.82	a
LC0061	38.8	8.1	2	ICP-MS	4.05	0.31	0.73	a
LC0062	27.4	2.62	2	ICP-MS	1.31	-0.77	-3.45	b
LC0067	27.3				0	-0.78	-4.21	b
LC0068	28.6	2.43	3.18	ICP-MS	0.76	-0.65	-3.28	b
LC0097	31.8	12.7	2	GF-AAS	6.35	-0.35	-0.56	a
LC0101	42.1	5.5	2	ICP-MS	2.75	0.62	1.96	a
LC0115	47.8	3.5	3.18	ICP-MS	1.10	1.16	5.48	b
LC0116	37.1			ICP-MS	0	0.15	0.81	b
LC0118	37.3	16.4	2	ICP-OES	8.20	0.16	0.21	a
LC0120	73.1	23	2	ICP-OES	11.50	3.55	3.22	c
LC0121	28.2	12.4	2		6.20	-0.69	-1.12	a
LC0122	51.3	22.6	2	ICP-MS	11.30	1.49	1.37	c
LC0123	36.4	9.0	2	GF-AAS	4.50	0.08	0.17	a
LC0124	23.1	0.89	2	ICP-MS	0.45	-1.18	-6.21	b

[a] √3 is set by ILC coordinator when no coverage factor is reported. The reported uncertainty was assumed to have a rectangular distribution with k=√3
[b] Satisfactory, Questionable, Unsatisfactory
[c] "a": $u(x_{pt}) \leq u_{lab} \leq \sigma_{pt}$; "b": $u_{lab} < u(x_{pt})$; and "c": $u_{lab} > \sigma_{pt}$

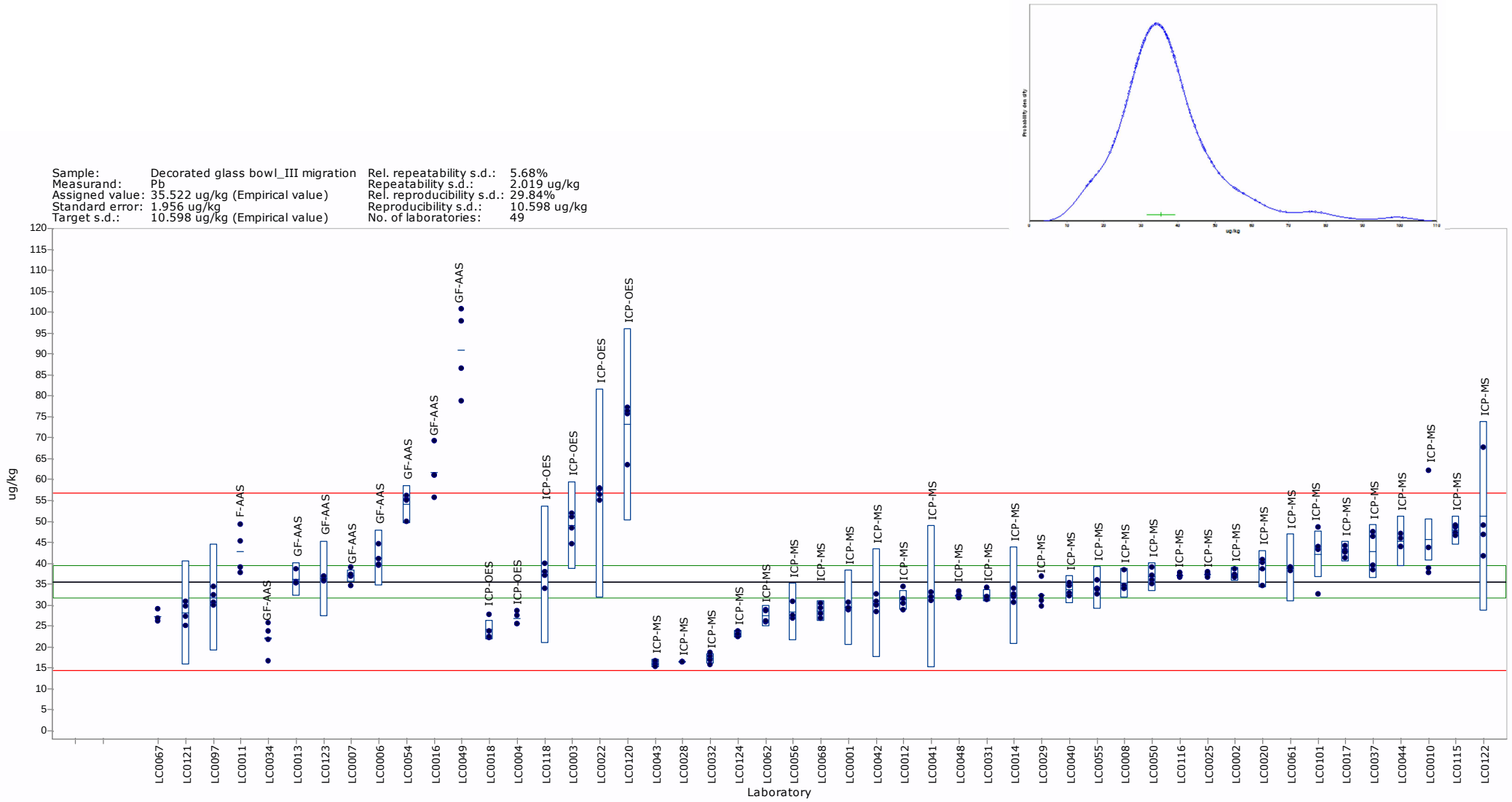

Figure 18. **Pb in GB (III migration)**: Measurement results and associated uncertainties (reported uncertainties shown). Reference value (x_{pt}): solid black line; reference interval (x_{pt}±U(x_{pt})):dash green line; Target interval (x_{pt}±2σ_{pt}): solid red lines.

Table 19. **Cd in glass bowl (GB) III migration**,
Assigned range: x_{pt} = 3.10, u (x_{pt}) =0.21, σ_{pt} = 1.13 [all values are in µg/kg]

Lab code	x_i	U_i	k[a]	technique	u_i	z-score[b]	ζ-score	Uncert.[c]
LC0001	2.75	0.8	2	ICP-MS	0.40	-0.31	-0.77	a
LC0002	2.51	0.2	2	ICP-MS	0.10	-0.53	-2.53	b
LC0003	3.40	0.6	2	ICP-OES	0.30	0.27	0.82	a
LC0004	1.80			ICP-OES	0	-1.15	-6.14	b
LC0006	2.58	0.9	2	GF-AAS	0.45	-0.47	-1.06	a
LC0007	2.73	0.11	2	GF-AAS	0.06	-0.33	-1.70	b
LC0008	2.55	0.3	2	ICP-MS	0.15	-0.49	-2.12	b
LC0011	3.95			F-AAS	0	0.76	4.02	b
LC0012	3.86	0.12	2	ICP-MS	0.06	0.68	3.46	b
LC0013	2.35	0.2	2	GF-AAS	0.10	-0.67	-3.20	b
LC0014	2.05	1.12	2	ICP-MS	0.56	-0.93	-1.75	a
LC0016	4.03			GF-AAS	0	0.82	4.38	b
LC0017	3.15	0.15	2	ICP-MS	0.08	0.05	0.23	b
LC0018	2.25	0.2	1	ICP-OES	0.20	-0.75	-2.92	b
LC0020	2.73	0.3	3.18	ICP-MS	0.09	-0.33	-1.62	b
LC0022	5.93	2.6	2	ICP-OES	1.30	2.51	2.15	c
LC0025	3.93			ICP-MS	0	0.73	3.90	b
LC0028	1.20	0.04	2	ICP-MS	0.02	-1.69	-8.94	b
LC0029	3.45			ICP-MS	0	0.31	1.66	b
LC0031	3.03	0.1	2	ICP-MS	0.05	-0.07	-0.34	b
LC0032	1.30	0.05	√3	ICP-MS	0.03	-1.60	-8.44	b
LC0034	2.60	0.122	2	GF-AAS	0.06	-0.44	-2.27	b
LC0037	3.13	0.4	2	ICP-MS	0.20	0.02	0.09	b
LC0040	4.51	0.45	2	ICP-MS	0.23	1.25	4.55	a
LC0042	2.09	0.92	2	ICP-MS	0.46	-0.90	-2.00	a
LC0043	1.18	0.1	2	ICP-MS	0.05	-1.71	-8.85	b
LC0044	2.95	0.5	2	ICP-MS	0.25	-0.13	-0.46	a
LC0048	2.60	0.1	2	ICP-MS	0.05	-0.44	-2.30	b
LC0049	6.12			GF-AAS	0.00	2.68	14.26	b
LC0050	2.75	0.1	2	ICP-MS	0.05	-0.31	-1.61	b
LC0054	5.60	1.1	3.18	GF-AAS	0.35	2.22	6.17	a
LC0055	2.83	0.4	2	ICP-MS	0.20	-0.24	-0.94	b
LC0056	6.60	1.00	2	ICP-MS	0.50	3.11	6.45	a
LC0061	5.00	1.3	2	ICP-MS	0.65	1.69	2.78	a
LC0062	1.95	0.16	2	ICP-MS	0.08	-1.02	-5.08	b
LC0067	2.13				0	-0.86	-4.57	b
LC0068	2.40	0.33	3.18	ICP-MS	0.10	-0.62	-2.98	b
LC0097	2.10	0.9	2	GF-AAS	0.45	-0.89	-2.01	a
LC0101	4.90	0.74	2	ICP-MS	0.37	1.60	4.23	a
LC0115	3.95	0.4	3.18	ICP-MS	0.13	0.76	3.46	b
LC0118	2.93	1.3	2	ICP-OES	0.65	-0.16	-0.26	a
LC0120	5.54	2	2	ICP-OES	1.00	2.17	2.39	a
LC0121	2.25	0.9	2		0.45	-0.75	-1.71	a
LC0122	6.68	2.9	2	ICP-MS	1.45	3.18	2.44	c
LC0123	2.75	0.5	2	GF-AAS	0.25	-0.31	-1.07	a
LC0124	3.38	0.15	2	ICP-MS	0.07	0.25	1.26	b

[a] √3 is set by ILC coordinator when no coverage factor is reported. The reported uncertainty was assumed to have a rectangular distribution with k=√3
[b] Satisfactory, Questionable, Unsatisfactory
[c] "a": $u(x_{pt}) \leq u_{lab} \leq \sigma_{pt}$; "b": $u_{lab} < u(x_{pt})$; and "c": $u_{lab} > \sigma_{pt}$

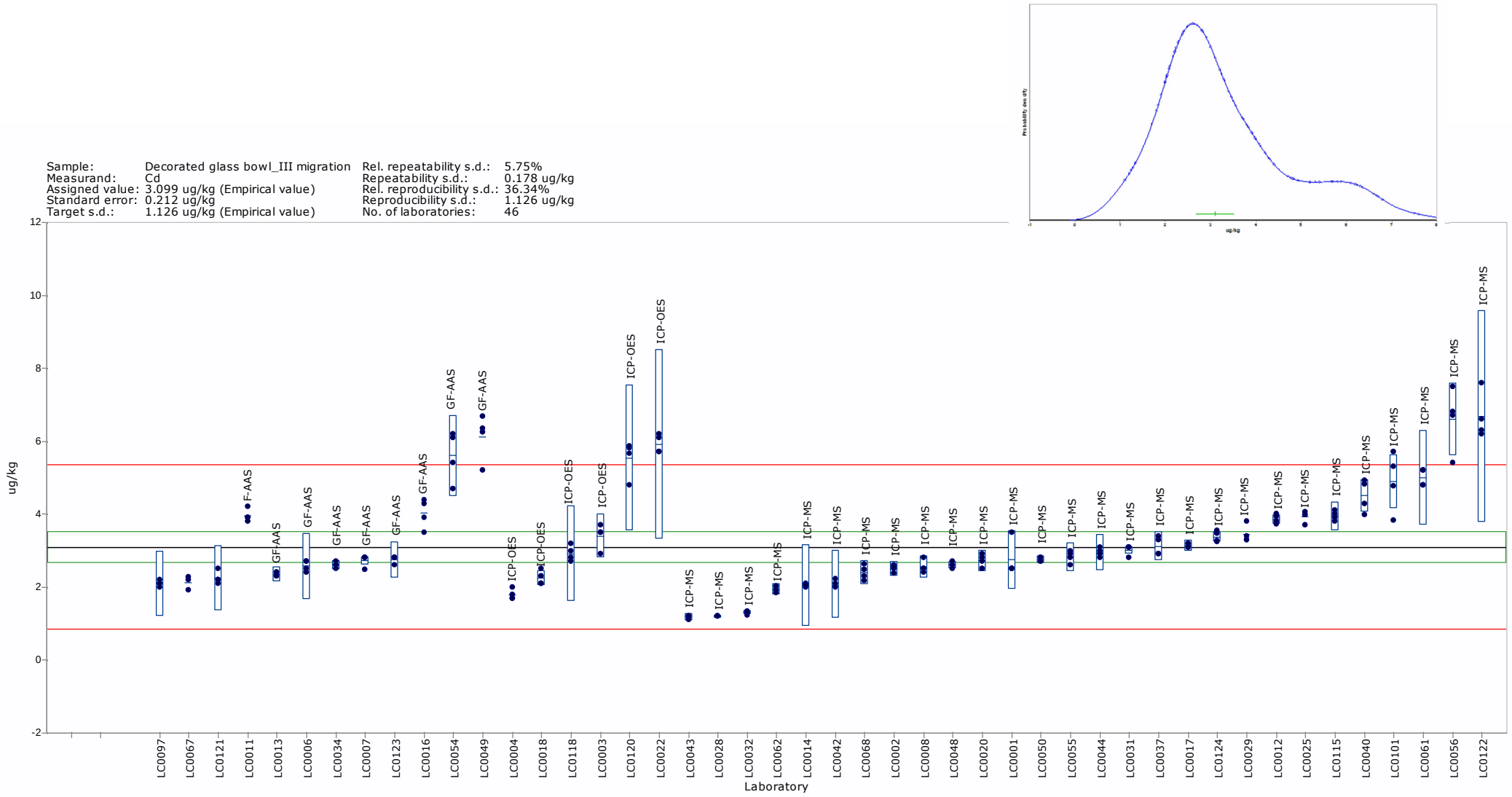

Figure 19. **Cd in GB (III migration)**: Measurement results and associated uncertainties (reported uncertainties shown). Reference value (x_{pt}): solid black line; reference interval (x_{pt}±U(x_{pt})):dash green line; Target interval (x_{pt}±2σ_{pt}): solid red lines.

Table 20. **(CC) I migration: Cd** Assigned range: x_{pt} = 2.98, u (x_{pt}) =0.25, σ_{pt} = 1.28; **Pb** Assigned range: x_{pt} = 88.4, u (x_{pt}) =5.25, σ_{pt} = 47.2 [values in µg/kg]

Element	Lab	x_i	U_i	k[a]	technique	u_i	z-	ζ-score	Uncert.[c]
Cd	LC0003	4.88	0.9	2	ICP-	0.45	1.48	3.66	a
	LC0004	2.40			ICP-	0	-0.46	-2.31	b
	LC0006	2.25	0.8	2	GF-	0.40	-0.58	-1.55	a
	LC0007	1.34	0.16	2	GF-	0.08	-1.29	-6.18	b
	LC0011	3.48			F-AAS	0	0.38	1.93	b
	LC0013	2.35	0.2	2	GF-	0.10	-0.50	-2.33	b
	LC0016	7.55			GF-	0	3.57	18.01	b
	LC0017	5.50	2.23	2	ICP-	1.12	1.97	2.20	a
	LC0018	2.87	0.2	1	ICP-	0.20	-0.09	-0.37	b
	LC0020	2.43	0.9	3.18	ICP-	0.28	-0.44	-1.47	a
	LC0025	3.23			ICP-	0	0.19	0.96	b
	LC0028	0.15	0.06	2	ICP-	0.03	-2.22	-	b
	LC0029	2.38			ICP-	0	-0.48	-2.41	b
	LC0031	2.49	0.1	2	ICP-	0.05	-0.39	-1.91	b
	LC0034	4.63	0.122	2	GF-	0.06	1.28	6.29	b
	LC0037	2.98	0.6	2	ICP-	0.30	-0.01	-0.03	a
	LC0040	2.33	0.74	2	ICP-	0.37	-0.51	-1.45	a
	LC0043	0.10	0.05	2	ICP-	0.03	-2.26	-	b
	LC0044	6.88	1.2	2	ICP-	0.60	3.05	5.97	a
	LC0048	2.55	0.1	2	ICP-	0.05	-0.34	-1.68	b
	LC0049	2.97			GF-	0	-0.01	-0.07	b
	LC0050	2.80	0.73	2	ICP-	0.37	-0.15	-0.42	a
	LC0055	2.68	0.4	2	ICP-	0.20	-0.24	-0.96	b
	LC0056	4.25	0.7	2	ICP-	0.35	0.99	2.93	a
	LC0068	3.63	1.46	3.18	ICP-	0.46	0.50	1.22	a
	LC0115	2.68	2.7	3.18	ICP-	0.85	-0.24	-0.35	a
Pb	LC0003	88.7	16.0	2	ICP-	8.00	0.01	0.04	a
	LC0004	80.5			ICP-	0	-0.17	-1.50	b
	LC0006	76.3	12.2	2	GF-	6.10	-0.26	-1.50	a
	LC0007	46.3	7.56	2	GF-	3.78	-0.89	-6.50	b
	LC0010	98.7	10	2	ICP-	5.00	0.22	1.42	b
	LC0011	148			F-AAS	0	1.26	11.31	b
	LC0013	79.1	8.7	2	GF-	4.35	-0.20	-1.35	b
	LC0016	286			GF-	0	4.19	37.62	b
	LC0017	185	83.59	2	ICP-	41.80	2.05	2.29	a
	LC0018	90.3	9.0	1	ICP-	9.00	0.04	0.19	a
	LC0020	86.8	41	3.18	ICP-	12.88	-0.03	-0.11	a
	LC0025	93.0			ICP-	0	0.10	0.89	b
	LC0028	5.20	2.2	2	ICP-	1.10	-1.76	-	b
	LC0029	83.1			ICP-	0	-0.11	-1.00	b
	LC0031	83.8	3.5	2	ICP-	1.75	-0.10	-0.82	b
	LC0034	93.9	0.13	2	GF-	0.07	0.12	1.05	b
	LC0037	95.0	31.8	2	ICP-	15.90	0.14	0.39	a
	LC0040	79.6	30.2	2	ICP-	15.10	-0.19	-0.55	a
	LC0041	100	45	2	ICP-	22.50	0.25	0.52	a
	LC0043	4.23	1.7	2	ICP-	0.85	-1.78	-	b
	LC0044	264	34	2	ICP-	17.00	3.72	9.86	a
	LC0048	75.8	4.5	2	ICP-	2.25	-0.27	-2.20	b
	LC0049	118			GF-	0	0.62	5.56	b
	LC0050	88.0	31.3	2	ICP-	15.65	-0.01	-0.02	a
	LC0055	79.6	11.9	2	ICP-	5.95	-0.19	-1.10	a
	LC0056	102	15.9	2	ICP-	7.95	0.30	1.46	a
	LC0068	139	56.26	3.18	ICP-	17.68	1.07	2.74	a
	LC0113	36.2	3.8	2	ICP-	1.90	-1.11	-9.34	b
	LC0115	78.5	115.2	3.18	ICP-	36.20	-0.21	-0.27	a

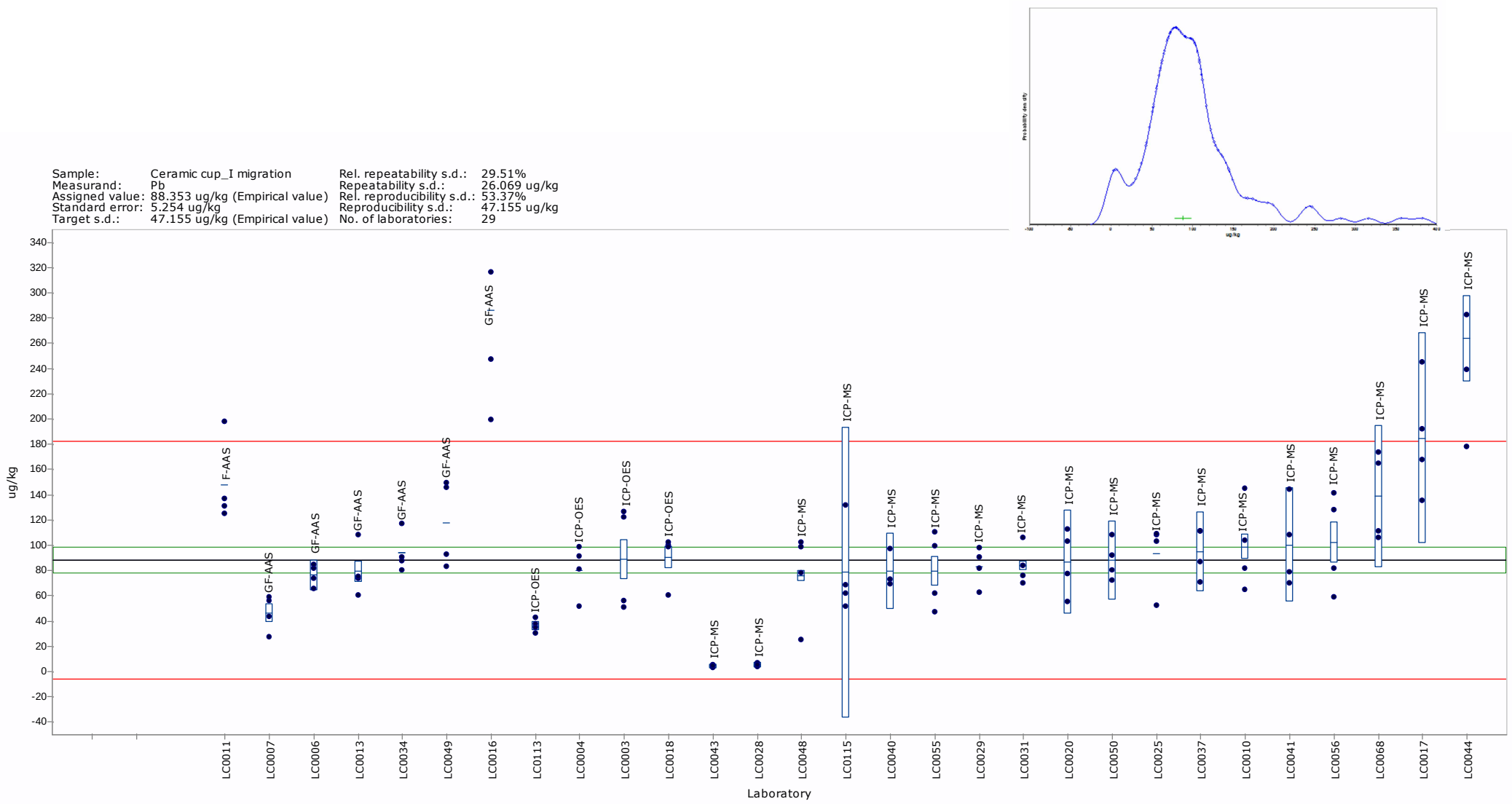

Figure 20. **Pb in CC (I migration)**: Measurement results and associated uncertainties (reported uncertainties shown). Reference value (x_{pt}): solid black line; reference interval (x_{pt}±U(x_{pt})):dash green line; Target interval (x_{pt}±2σ_{pt}): solid red lines.

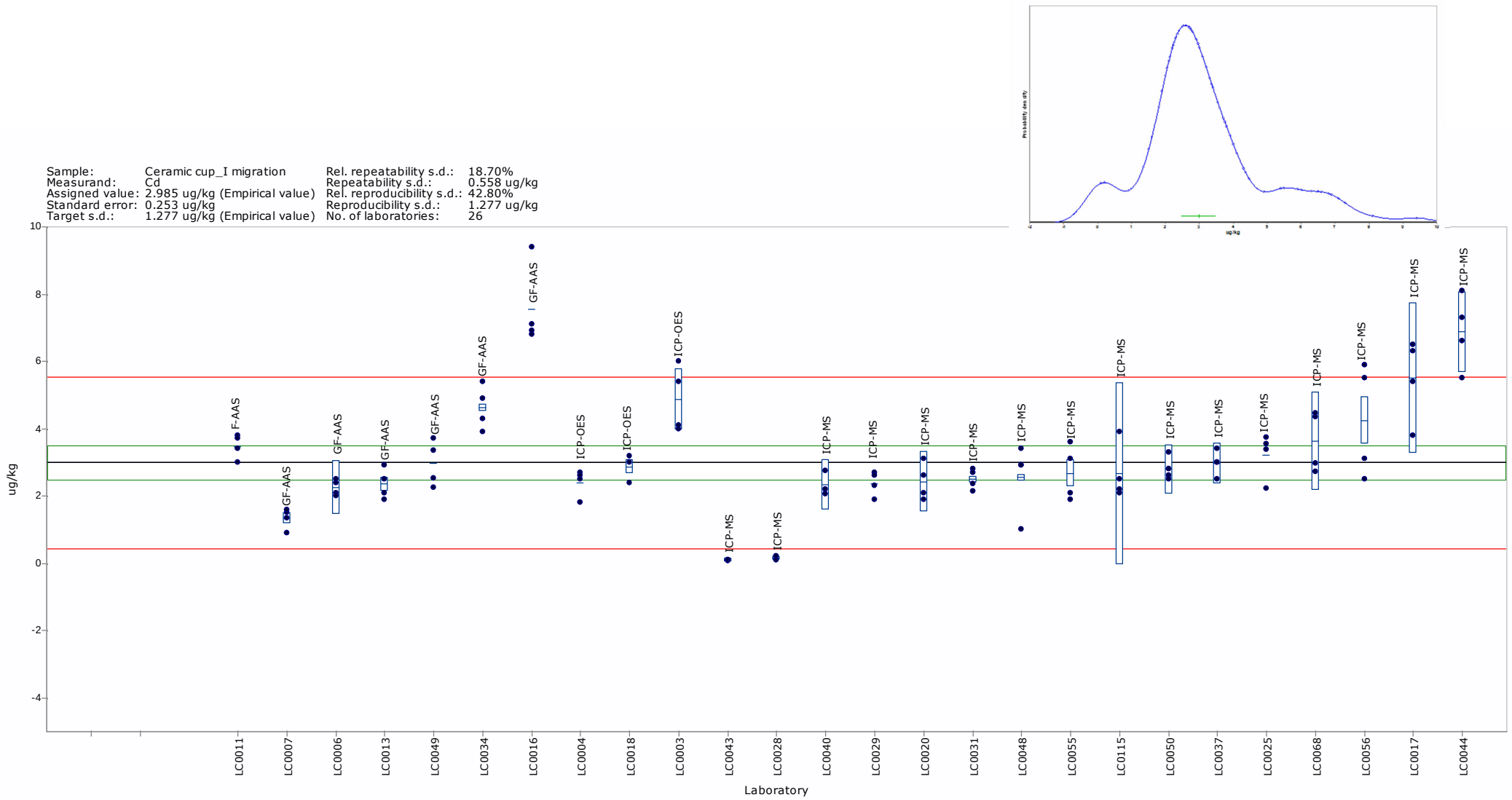

Figure 21. **Cd in CC (I migration)**: Measurement results and associated uncertainties (reported uncertainties shown). Reference value (x_{pt}): solid black line; reference interval ($x_{pt}\pm U(x_{pt})$):dash green line; Target interval ($x_{pt}\pm 2\sigma_{pt}$): solid red lines.

Table 21. **(CC) II migration: Cd** Assigned range: x_{pt} = 2.56, u (x_{pt}) =0.20, σ_{pt} = 1.28; **Pb** Assigned range: x_{pt} = 78.9, u (x_{pt}) =7.19, σ_{pt} = 42.6 [values in µg/kg]

Element	Lab code	x_i	U_i	k[a]	technique	u_i	z-score[b]	ζ-score	Uncert.[c]
Cd	LC0003	4.83	0.8	2	ICP-OES	0.40	1.77	5.08	a
	LC0004	2.05			ICP-OES	0	-0.40	-2.56	b
	LC0006	2.05	0.7	2	GF-AAS	0.35	-0.40	-1.26	a
	LC0007	2.18	0.33	2	GF-AAS	0.17	-0.30	-1.49	b
	LC0011	3.13			F-AAS	0	0.44	2.86	b
	LC0013	1.83	0.1	2	GF-AAS	0.05	-0.57	-3.59	b
	LC0016	7.03			GF-AAS	0	3.49	22.52	b
	LC0017	5.13	3.51	2	ICP-MS	1.76	2.01	1.45	c
	LC0018	2.60	0.2	1	ICP-OES	0.20	0.03	0.15	a
	LC0020	2.45	1.3	3.18	ICP-MS	0.41	-0.09	-0.24	a
	LC0025	2.31			ICP-MS	0	-0.19	-1.24	b
	LC0028	0.10	0.06	2	ICP-MS	0.03	-1.92	-12.27	b
	LC0029	1.53			ICP-MS	0	-0.81	-5.21	b
	LC0031	2.28	0.1	2	ICP-MS	0.05	-0.22	-1.39	b
	LC0034	3.80	0.122	2	GF-AAS	0.06	0.97	5.98	b
	LC0037	2.38	0.7	2	ICP-MS	0.35	-0.14	-0.46	a
	LC0040	1.53	0.26	2	ICP-MS	0.13	-0.80	-4.34	b
	LC0043	0.12	0.05	2	ICP-MS	0.02	-1.91	-12.19	b
	LC0044	6.18	1.1	2	ICP-MS	0.55	2.83	6.19	a
	LC0048	2.18	0.1	2	ICP-MS	0.05	-0.30	-1.88	b
	LC0049	2.42			GF-AAS	0	-0.11	-0.70	b
	LC0050	2.15	0.80	2	ICP-MS	0.40	-0.32	-0.92	a
	LC0055	2.08	0.3	2	ICP-MS	0.15	-0.38	-1.94	b
	LC0056	5.15	0.8	2	ICP-MS	0.40	2.03	5.81	a
	LC0068	2.65	1.05	3.18	ICP-MS	0.33	0.07	0.23	a
	LC0115	1.93	1.3	3.18	ICP-MS	0.41	-0.50	-1.40	a
Pb	LC0003	63.5	12.1	2	ICP-OES	6.05	-0.36	-1.64	b
	LC0004	71.6			ICP-OES	0	-0.17	-1.02	b
	LC0006	71.3	11.4	2	GF-AAS	5.70	-0.18	-0.83	b
	LC0007	85.5	14.88	2	GF-AAS	7.44	0.16	0.64	a
	LC0010	76.6	10	2	ICP-MS	5.00	-0.06	-0.27	b
	LC0011	146			F-AAS	0	1.57	9.33	b
	LC0013	67.3	7.4	2	GF-AAS	3.70	-0.27	-1.44	b
	LC0016	280			GF-AAS	0	4.71	27.93	b
	LC0017	169	130.08	2	ICP-MS	65.04	2.12	1.38	c
	LC0018	102	10.1	1	ICP-OES	10.10	0.54	1.84	a
	LC0020	93.9	55	3.18	ICP-MS	17.30	0.35	0.80	a
	LC0025	70.3			ICP-MS	0	-0.20	-1.20	b
	LC0028	4.88	2.1	2	ICP-MS	1.05	-1.74	-10.19	b
	LC0029	57.3			ICP-MS	0	-0.51	-3.01	b
	LC0031	81.4	3.4	2	ICP-MS	1.70	0.06	0.34	b
	LC0034	87.0	0.13	2	GF-AAS	0.07	0.19	1.13	b
	LC0037	84.0	43.7	2	ICP-MS	21.85	0.12	0.22	a
	LC0040	53.4	11.7	2	ICP-MS	5.85	-0.60	-2.76	b
	LC0041	72.8	34	2	ICP-MS	17.00	-0.14	-0.33	a
	LC0043	5.08	2.0	2	ICP-MS	1.00	-1.73	-10.17	b
	LC0044	242	31	2	ICP-MS	15.50	3.82	9.52	a
	LC0048	68.3	4.1	2	ICP-MS	2.05	-0.25	-1.42	b
	LC0049	101			GF-AAS	0	0.52	3.09	b
	LC0050	73.0	27.2	2	ICP-MS	13.60	-0.14	-0.38	a
	LC0055	70.9	10.6	2	ICP-MS	5.30	-0.19	-0.90	b
	LC0056	123	19.0	2	ICP-MS	9.50	1.03	3.69	a
	LC0068	109	42.02	3.18	ICP-MS	13.21	0.70	1.98	a
	LC0113	33.5	3.5	2	ICP-OES	1.75	-1.07	-6.13	b
	LC0115	70.1	78.4	3.18	ICP-MS	24.64	-0.21	-0.35	a

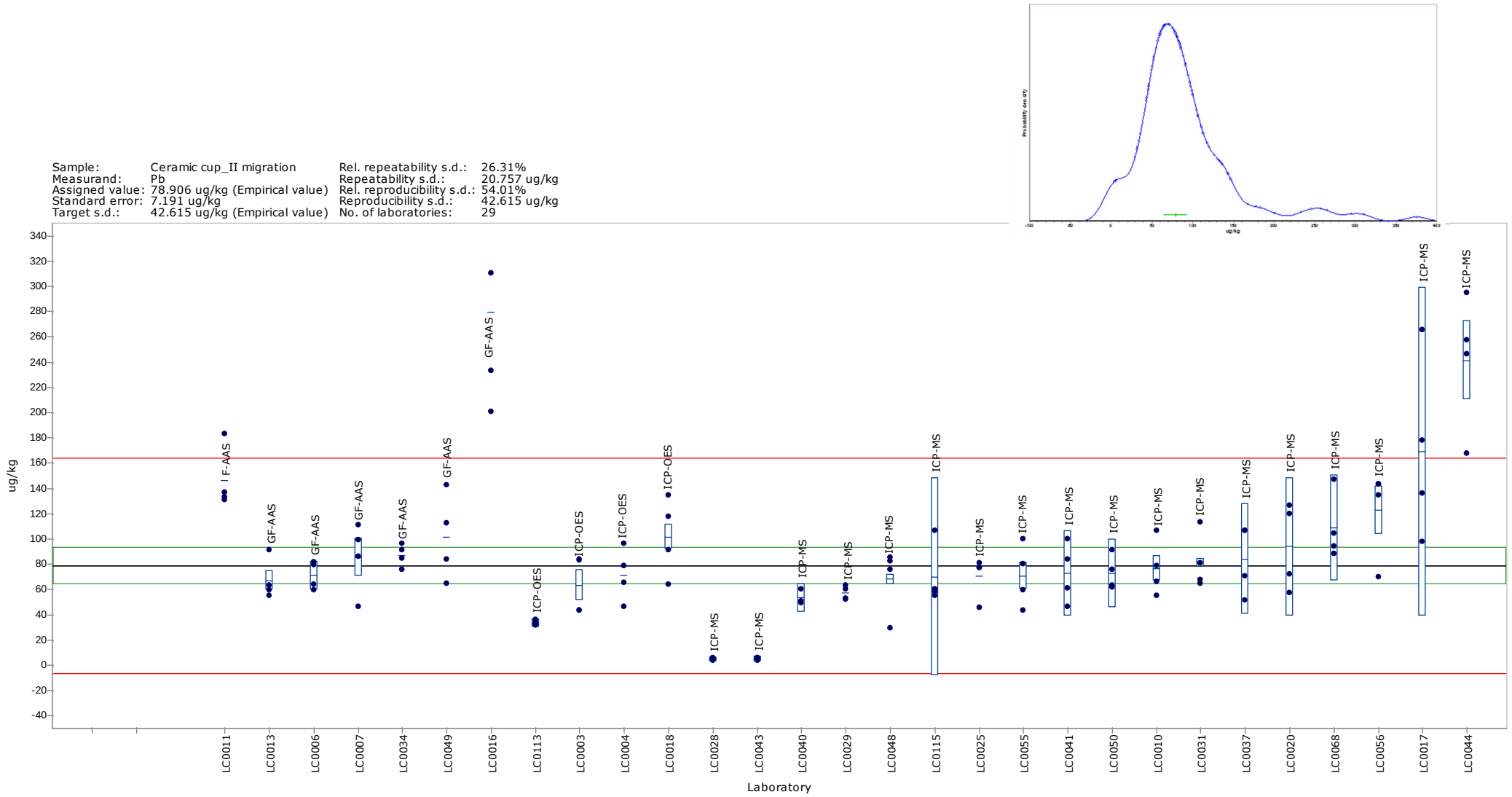

Figure 22. **Pb in CC (II migration)**: Measurement results and associated uncertainties (reported uncertainties shown). Reference value (x_{pt}): solid black line; reference interval ($x_{pt}\pm U(x_{pt})$):dash green line; Target interval ($x_{pt}\pm 2\sigma_{pt}$): solid red lines.

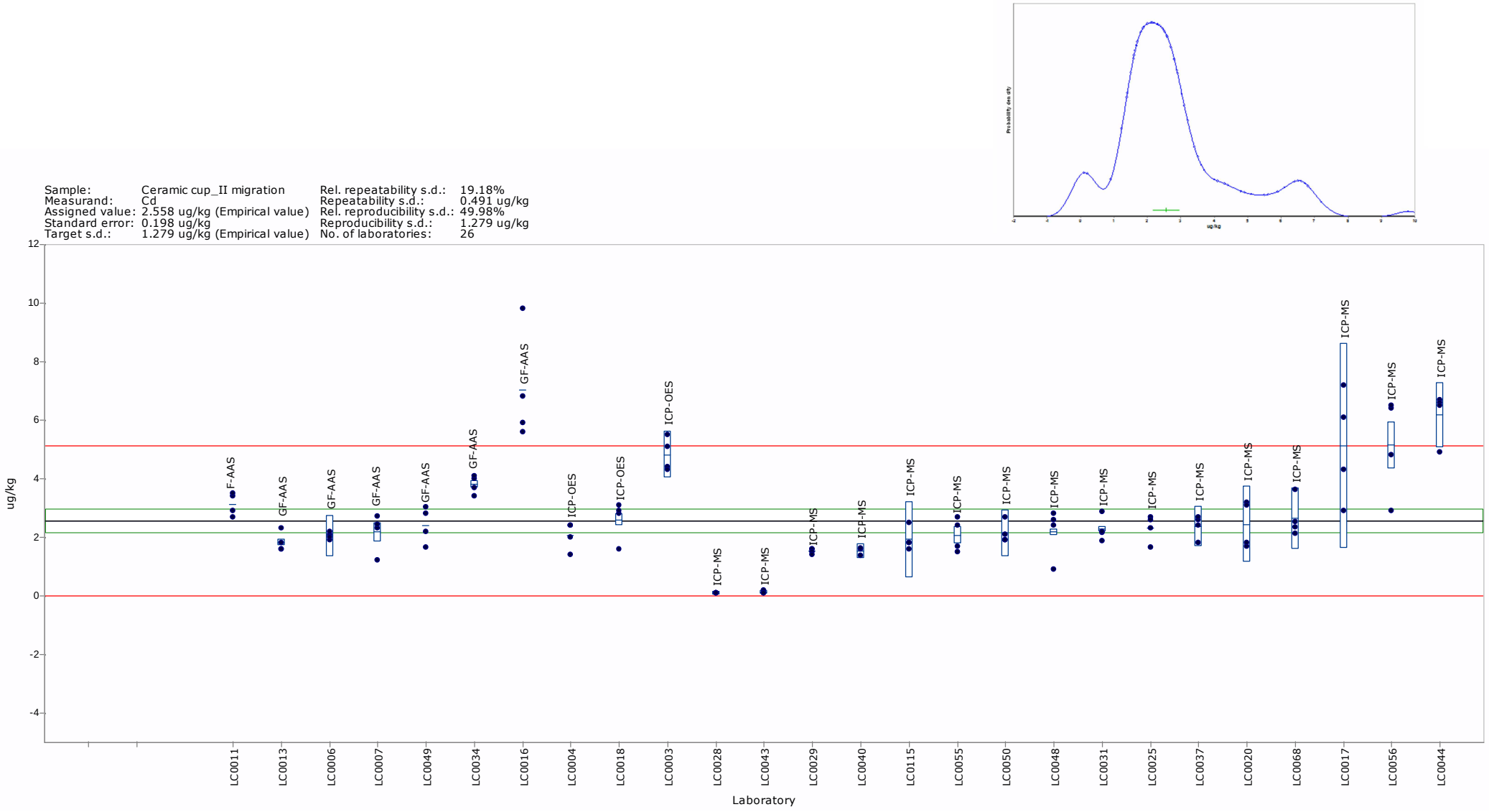

Figure 23. **Cd in CC (II migration)**: Measurement results and associated uncertainties (reported uncertainties shown). Reference value (x_{pt}): solid black line; reference interval ($x_{pt}\pm U(x_{pt})$):dash green line; Target interval ($x_{pt}\pm 2\sigma_{pt}$): solid red lines.

Table 22. **(CC) III migration: Cd** Assigned range: x_{pt} = 2.31, u (x_{pt}) =0.20, σ_{pt} = 1.18; **Pb** Assigned range: x_{pt} = 75.5, u (x_{pt}) =5.20, σ_{pt} = 37.9 [values are in µg/kg]

Element	Lab code	x_i	U_i	k[a]	technique	u_i	z-score[b]	ζ-score	Uncert.[c]
Cd	LC0003	1.80	0.4	2	ICP-OES	0.20	-0.43	-1.82	a
	LC0004	1.98			ICP-OES	0	-0.28	-1.71	b
	LC0006	2.38	0.8	2	GF-AAS	0.40	0.06	0.15	a
	LC0007	1.66	0.25	2	GF-AAS	0.13	-0.55	-2.80	b
	LC0011	2.88			F-AAS	0	0.48	2.90	b
	LC0013	2.03	0.1	2	GF-AAS	0.05	-0.24	-1.41	b
	LC0016	11.83			GF-AAS	0	8.09	48.76	b
	LC0017	5.18	2.73	2	ICP-MS	1.37	2.44	2.08	c
	LC0018	2.33	0.2	1	ICP-OES	0.20	0.02	0.09	a
	LC0020	2.28	0.8	3.18	ICP-MS	0.25	-0.03	-0.11	a
	LC0025	2.28			ICP-MS	0	-0.03	-0.16	b
	LC0028	0.10	0.05	2	ICP-MS	0.03	-1.88	-11.24	b
	LC0029	1.50			ICP-MS	0	-0.69	-4.15	b
	LC0031	2.05	0.1	2	ICP-MS	0.05	-0.22	-1.29	b
	LC0034	3.25	0.122	2	GF-AAS	0.06	0.80	4.60	b
	LC0037	1.98	1.1	2	ICP-MS	0.55	-0.28	-0.57	a
	LC0040	1.59	0.08	2	ICP-MS	0.04	-0.61	-3.59	b
	LC0043	0.13	0.06	2	ICP-MS	0.03	-1.86	-11.06	b
	LC0044	6.50	1.1	2	ICP-MS	0.55	3.56	7.18	a
	LC0048	1.98	0.10	2	ICP-MS	0.05	-0.28	-1.66	b
	LC0049	4.25			GF-AAS	0	1.65	9.92	b
	LC0050	1.98	0.75	2	ICP-MS	0.38	-0.28	-0.79	a
	LC0055	1.98	0.3	2	ICP-MS	0.15	-0.28	-1.36	b
	LC0056	4.33	0.7	2	ICP-MS	0.35	1.71	5.03	a
	LC0068	2.94	0.99	3.18	ICP-MS	0.31	0.54	1.73	a
	LC0115	2.18	1.8	3.18	ICP-MS	0.57	-0.11	-0.22	a
Pb	LC0003	82.25	14.3	2	ICP-OES	7.15	0.18	0.76	a
	LC0004	80.15			ICP-OES	0	0.12	0.89	b
	LC0006	82.23	13.1	2	GF-AAS	6.55	0.18	0.80	a
	LC0007	63.65	10.49	2	GF-AAS	5.25	-0.31	-1.61	a
	LC0010	72.75	10	2	ICP-MS	5.00	-0.07	-0.38	b
	LC0011	131.50			F-AAS	0	1.48	10.76	b
	LC0013	74.05	8.2	2	GF-AAS	4.10	-0.04	-0.22	b
	LC0016	464.25			GF-AAS	0	10.27	74.72	b
	LC0017	167.88	81.56	2	ICP-MS	40.78	2.44	2.25	c
	LC0018	76.30	7.6	1	ICP-OES	7.60	0.02	0.09	a
	LC0020	87.75	42	3.18	ICP-MS	13.21	0.32	0.86	a
	LC0025	69.13			ICP-MS	0	-0.17	-1.23	b
	LC0028	4.65	2.0	2	ICP-MS	1.00	-1.87	-13.38	b
	LC0029	57.80			ICP-MS	0	-0.47	-3.40	b
	LC0031	75.93	3.2	2	ICP-MS	1.60	0.01	0.08	b
	LC0034	89.40	0.13	2	GF-AAS	0.07	0.37	2.67	b
	LC0037	64.98	40.1	2	ICP-MS	20.05	-0.28	-0.51	a
	LC0040	56.97	11.6	2	ICP-MS	5.80	-0.49	-2.38	a
	LC0041	73.75	35	2	ICP-MS	17.50	-0.05	-0.10	a
	LC0043	5.83	2.3	2	ICP-MS	1.15	-1.84	-13.08	b
	LC0044	251.00	33	2	ICP-MS	16.50	4.64	10.14	a
	LC0048	63.78	3.8	2	ICP-MS	1.90	-0.31	-2.12	b
	LC0049	182.22			GF-AAS	0	2.82	20.51	b
	LC0050	67.25	24.0	2	ICP-MS	12.00	-0.22	-0.63	a
	LC0055	68.43	10.3	2	ICP-MS	5.15	-0.19	-0.97	b
	LC0056	62.25	9.6	2	ICP-MS	4.80	-0.35	-1.87	b
	LC0068	107.27	44.88	3.18	ICP-MS	14.10	0.84	2.11	a
	LC0113	50.68	5.0	2	ICP-OES	2.50	-0.66	-4.30	b
	LC0115	78.43	69.6	3.18	ICP-MS	21.87	0.08	0.13	a

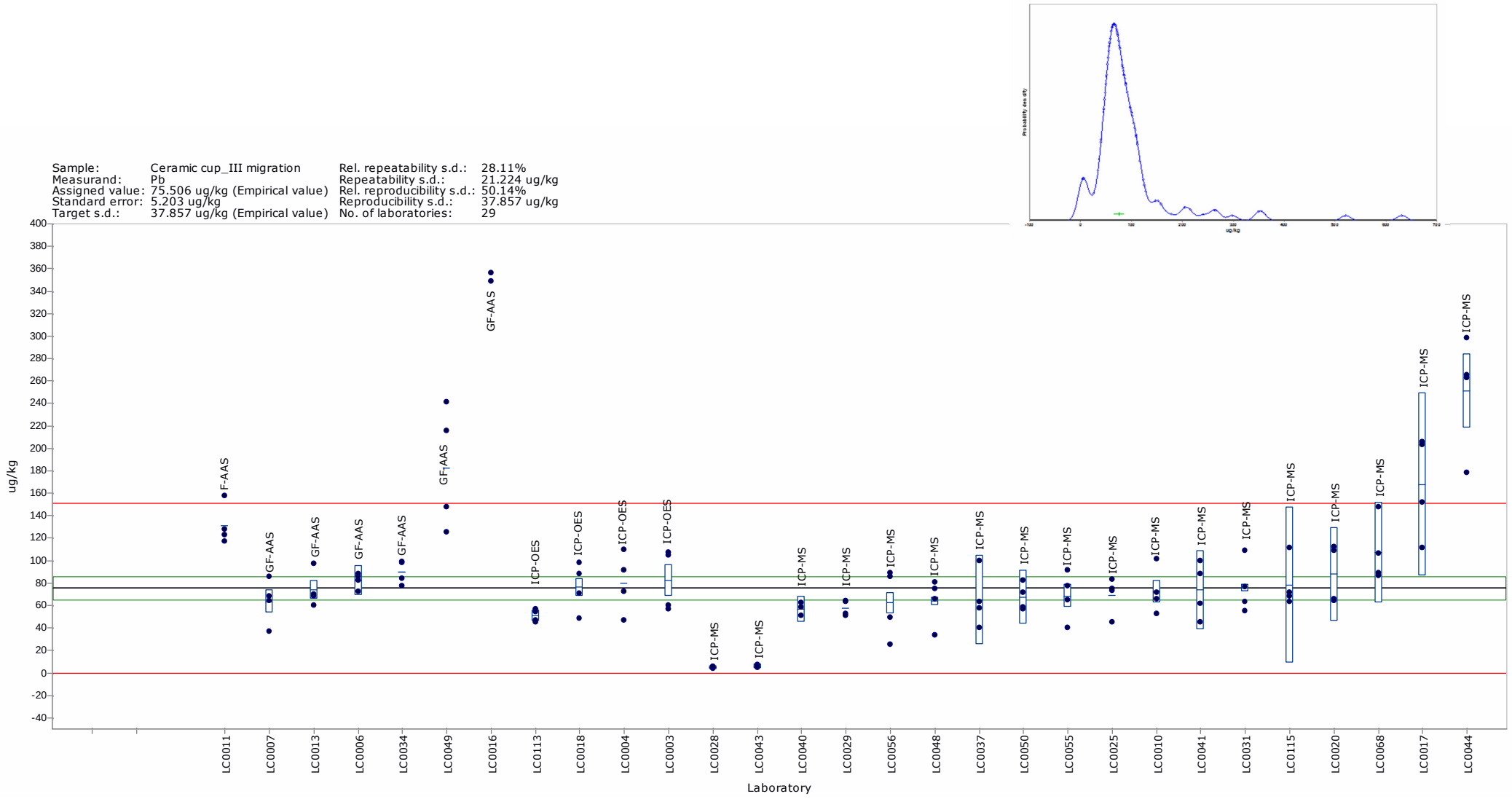

Figure 24. **Pb in CC (III migration)**: Measurement results and associated uncertainties (reported uncertainties shown). Reference value (x_{pt}): solid black line; reference interval (x_{pt}±U(x_{pt})):dash green line; Target interval (x_{pt}±2σ_{pt}): solid red lines.

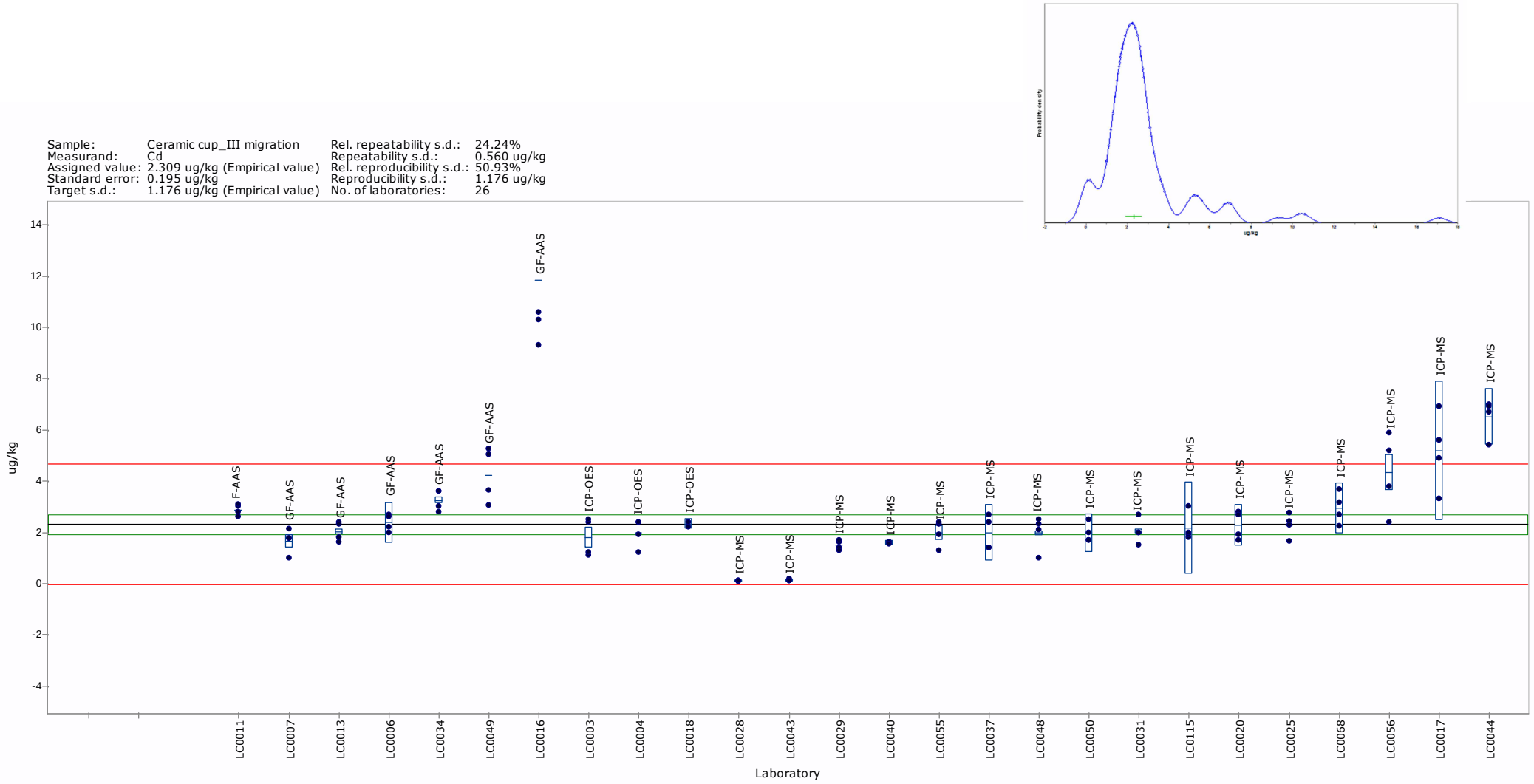

Figure 25. **Cd in CC (III migration)**: Measurement results and associated uncertainties (reported uncertainties shown). Reference value (x_{pt}): solid black line; reference interval ($x_{pt}\pm U(x_{pt})$):dash green line; Target interval ($x_{pt}\pm 2\sigma_{pt}$): solid red lines

Annex 10. Overview on z-scores

Table 1. z-scores matrix

z-score matrix																										Overall		
Sample	S1						S2				GB I			GB II			GB III			CC I		CC II		CC III		[%] reported results		
Lab code	Pb	Cd	Al	Co	Mn	Ba	Cu	Zn	Fe	Sb	Pb	Cd	Co	Pb	Cd	Co	Pb	Cd	Co	Pb	Cd	Pb	Cd	Pb	Cd	S	Q	U
LC0001																										100	0	0
LC0002																										95	0	5
LC0003																										96	4	0
LC0004																										100	0	0
LC0005																										100	0	0
LC0006																										92	0	8
LC0007																										100	0	0
LC0008																										84	16	0
LC0010																										100	0	0
LC0011																										100	0	0
LC0012																										89	5	5
LC0013																										100	0	0
LC0014																										95	0	5
LC0016																										72	8	24
LC0017																										80	20	0
LC0018																										92	4	4
LC0020																										100	0	0
LC0022																										77	23	0
LC0025																										92	0	8
LC0028																										72	12	16
LC0029																										100	0	0
LC0031																										100	0	0
LC0032																										100	0	0
LC0034																										88	0	13
LC0037																										92	8	0
LC0040																										100	0	0
LC0041																										100	0	0
LC0042																										92	8	0
LC0043																										68	16	16
LC0044																										76	4	20
LC0046																										100	0	0
LC0048																										100	0	0
LC0049																										76	18	6
LC0050																										100	0	0
LC0054																										88	13	0
LC0055																										100	0	0
LC0056																										84	4	12
LC0059																										100	0	0
LC0061																										100	0	0
LC0062																										95	5	0
LC0067																										79	5	16
LC0068																										100	0	0
LC0097																										93	7	0
LC0101																										84	5	11
LC0113																										73	13	13
LC0115																										96	4	0
LC0116																										100	0	0
LC0118																										100	0	0
LC0120																										84	5	11
LC0121																										100	0	0
LC0122																										89	5	5
LC0123																										79	14	7
LC0124																										100	0	0

Satisfactory, Questionable, Unsatisfactory; Results not reported or "less than"

Annex 11. Approaches used to estimate the uncertainty

Table 1. Approaches used to estimate the uncertainty by participants for sample S1

Solution S1							
Element	Aluminium		Barium			Cadmium	
Uncert.	[%]	N° lab Approach	[%]	N° lab Approach	[%]	N° lab Approach	
a	50	4 - Based on replicates (precision) 4 - In house validation 3 - Top down/ In house validation 2 - Nordtest 1 - GUM 1 - HORWITZ 1 - ISO 11352:2012 1 - PT participation 1 - EURACHEM / CITAC Guide CG4	51	5 - In house validation 4 - Based on replicates (precision) 3 - Top down/ In house validation 2 - Nordtest 1 - PT participation 1 - GUM 1 - Approach not specified 1 - ISO 11352:2012	61	7 - In house validation 7 - Based on replicates (precision) 3 - Top down/ In house validation 2 - Eurachem/CITAC Guide CG4 2 - Nordtest 1 - PT participation 1 - GUM 1 - ISO 11352:2012	
b	31	8 - Based on replicates (precision) 1 - Bottom up / in house validation 2 - In house validation	29	9 - Based on replicates (precision) 1 - Bottom up / in house validation	8	3 - Based on replicates (precision)	
c	19	3 - Horwitz Thompson 2 - Horwitz 1 - Based on replicates (precision) 1 - Nordtest TR 537	20	3 - Horwitz 2 - Horwitz Thompson 1 - Approach not specified 1 - Nordtest TR 537	31	5 - Horwitz Thompson 2 - Horwitz 1 - Bottom up / in house validation 1 - GUM 1 - Nordtest TR 537 1 - Based on replicates (precision) 1 - Approach not specified	
Element	Cobalt		Lead			Manganese	
Uncert.	[%]	N° lab Approach	[%]	N° lab Approach	[%]	N° lab Approach	
a	49	6 - In house validation 3 - Based on replicates (precision) 3 - Top down/ In house validation 2 - Nordtest 1 - GUM 1 - Approach not specified 1 - PT participation 1 - ISO 11352:2012	61	6 - Based on replicates (precision) 5 - In house validation 3 - Nordtest 3 - Top down/ In house validation 2 - Eurachem/CITAC Guide CG4 1 - ISO 11352:2012 1 - PT participation 1 - Bottom up / in house validation 1 - GUM	55	5 - In house validation 4 - Based on replicates (precision) 3 - Eurachem/CITAC Guide CG4 3 - Top down/ In house validation 2 - GUM 2 - Nordtest 1 - PT participation 1 - ISO 11352:2012	
b	27	8 - Based on replicates (precision) 1 - Eurachem/CITAC Guide CG4 1 - Bottom up / in house validation	13	4 - Based on replicates (precision) 1 - In house validation	18	7 - Based on replicates (precision)	
c	24	3 - Horwitz 3 - Horwitz Thompson 1 - Approach not specified 1 - Based on replicates (precision) 1 - Nordtest	26	5 - Horwitz Thompson 2 - Horwitz 1 - Approach not specified 1 - Based on replicates (precision) 1 - GUM	26	4 - Horwitz Thompson 3 - Horwitz 1 - Nordtest TR 537 1 - Approach not specified 1 - Bottom up / in house validation	

Table 2. Approaches used to estimate the uncertainty by participants for sample S2

Solution S2				
Element		Antymony		Copper
Uncert.	[%]	N° lab Approach	[%]	N° lab Approach
a	49	3 - In house validation 3 - Nordtest TR 537 2 - Based on replicates (precision) 2 - Horwitz 2 - Top down/ In house validation 1 - Eurachem/CITAC Guide CG4 1 - GUM 1 - ISO 11352:2012 1 - PT participation	58	5 - In house validation 4 - Based on replicates (precision) 3 - Top down/ In house validation 2 - Eurachem/CITAC Guide CG4 2 - Horwitz 2 - Nordtest TR 537 1 - GUM 1 - ISO 11352:2012 1 - PT participation
b	42	9 - Based on replicates (precision) 3 - In house validation 1 - Bottom up / in house validation 1 - Top down/ In house validation	22	6 - Based on replicates (precision) 2 - In house validation
c	9	1 - Horwitz 1 - Horwitz Thompson 1 - approach not specified	19	3 - Horwitz Thompson 2 - Horwitz 1 - Nordtest TR 537 1 - approach not specified
Element		Iron		Zinc
Uncert.	[%]	N° lab Approach	[%]	N° lab Approach
a	57	6 - Based on replicates (precision) 4 - In house validation 2 - Nordtest TR 537 2 - Top down/ In house validation 1 - Eurachem/CITAC Guide CG4 1 - GUM 1 - Horwitz 1 - Horwitz Thompson 1 - ISO 11352:2012 1 - PT participation	62	5 - Based on replicates (precision) 5 - In house validation 3 - Top down/ In house validation 2 - Horwitz 1 - Eurachem/CITAC Guide CG4 1 - GUM 1 - Horwitz Thompson 1 - ISO 11352:2012 1 - Nordtest TR 537 1 - PT participation
b	20	4 - Based on replicates (precision) 2 - In house validation 1 - Eurachem/CITAC Guide CG4	21	5 - Based on replicates (precision) 1 - Eurachem/CITAC Guide CG4 1 - In house validation
c	23	3 - Horwitz 2 - Horwitz Thompson 1 - Nordtest TR 537 1 - Top down/ In house validation 1 - approach not specified	18	2 - Horwitz 2 - Nordtest TR 537 1 - Horwitz Thompson 1 - approach not specified

Table 3. Approaches used to estimate the uncertainty by participants for sample GB

		GB (I migration)		GB (II migration)		GB (III migration)
Element		Cadmium		Cadmium		Cadmium
Uncert.	[%]	N° lab Approach	[%]	N° lab Approach	[%]	N° lab Approach
a	61	11 - Based on replicates (precision) 4 - In house validation 3 - GUM 2 - Eurachem/CITAC Guide CG4 2 - Nordtest TR 537 1 - ISO 11352:2012 1 - PT participation 1 - Top down/ In house validation 1 - approach not specified	44	4 - Horwitz Thompson 4 - In house validation 3 - Nordtest TR 537 2 - GUM 1 - Based on replicates (precision) 1 - Bottom up / in house validation 1 - Horwitz 1 - PT participation	42	3 - Horwitz Thompson 3 - Nordtest TR 537 2 - Based on replicates (precision) 2 - GUM 2 - In house validation 2 - Horwitz 1 - Bottom up / in house validation 1 - PT participation
b	16	4 - Based on replicates (precision) 2 - In house validation 1 - Top down/ In house validation	51	14 - Based on replicates (precision) 2 - In house validation 2 - Top down/ In house validation 1 - Eurachem/CITAC Guide CG4 1 - approach not specified	53	12 - Based on replicates (precision) 4 - In house validation 2 - Top down/ In house validation 1 - Eurachem/CITAC Guide CG4 1 - approach not specified
c	23	5 - Horwitz Thompson 3 - Horwitz 1 - Bottom up / in house validation 1 - Nordtest TR 537	5.1	1 - Horwitz 1 - Horwitz Thompson	5.3	2 - Horwitz Thompson
Element		Cobalt		Cobalt		Cobalt
Uncert.	[%]	N° lab Approach	[%]	N° lab Approach	[%]	N° lab Approach
a	62	6 - Based on replicates (precision) 4 - In house validation 2 - Nordtest TR 537 2 - Top down/ In house validation 1 - Based on replicates (precision) 1 - Eurachem/CITAC Guide CG4 1 - GUM 1 - Horwitz 1 - ISO 11352:2012 1 - PT participation 1 - approach not specified	55	4 - Based on replicates (precision) 3 - Horwitz Thompson 2 - Horwitz 2 - Nordtest TR 537 1 - GUM 1 - In house validation 1 - PT participation 1 - Top down/ In house validation 1 - approach not specified	31	2 - Horwitz 2 - Horwitz Thompson 1 - Based on replicates (precision) 1 - GUM 1 - In house validation 1 - Nordtest TR 537 1 - PT participation
b	18	5 - Based on replicates (precision) 1 - Eurachem/CITAC Guide CG4	45	9 - Based on replicates (precision) 1 - Eurachem/CITAC Guide CG4 1 - In house validation 1 - Nordtest TR 537 1 - Top down/ In house validation	62	11 - Based on replicates (precision) 2 - Nordtest TR 537 2 - Top down/ In house validation 1 - Eurachem/CITAC Guide CG4 1 - In house validation 1 - approach not specified
c	21	3 - Horwitz Thompson 2 - Horwitz 1 - Based on replicates (precision) 1 - Nordtest TR 537			6.9	1 - Based on replicates (precision) 1 - Horwitz Thompson
Element		Lead		Lead		Lead
Uncert.	[%]	N° lab Approach	[%]	N° lab Approach	[%]	N° lab Approach
a	56	7 - Based on replicates (precision) 5 - In house validation 3 - GUM 2 - Nordtest TR 537 1 - Bottom up / in house validation 1 - Horwitz 1 - Horwitz Thompson 1 - ISO 11352:2012 1 - PT participation 1 - Top down/ In house validation 1 - approach not specified	62	5 - Horwitz Thompson 5 - In house validation 4 - Based on replicates (precision) 3 - Nordtest TR 537 2 - GUM 2 - Horwitz 1 - Bottom up / in house validation 1 - ISO 11352:2012 1 - PT participation 1 - Top down/ In house validation 1 - approach not specified	46	4 - In house validation 3 - Horwitz 3 - Horwitz Thompson 3 - Nordtest TR 537 2 - GUM 1 - Based on replicates (precision) 1 - Bottom up / in house validation 1 - ISO 11352:2012 1 - PT participation
b	28	8 - Based on replicates (precision) 2 - Eurachem/CITAC Guide CG4 1 - In house validation 1 - Top down/ In house validation	36	11 - Based on replicates (precision) 2 - Eurachem/CITAC Guide CG4 1 - In house validation 1 - Top down/ In house validation	46	13 - Based on replicates (precision) 2 - In house validation 2 - Top down/ In house validation 1 - Eurachem/CITAC Guide CG4 1 - approach not specified
c	16	4 - Horwitz Thompson 2 - Horwitz 1 - Nordtest TR 537	2.4	1 - Horwitz	7.3	2 - Horwitz Thompson 1 - Based on replicates (precision)

Table 4. Approaches used to estimate the uncertainty by participants for sample CC

	CC (I migration)			CC (II migration)			CC (III migration)	
Element	Cadmium			Cadmium			Cadmium	
Uncert.	[%]	N° lab	Approach	[%]	N° lab	Approach	[%]	N° lab	Approach
a	55	7 2 1 1	Based on replicates (precision) GUM Nordtest PT participation	50	5 2 1 1 1	Based on replicates (precision) GUM In house validation Nordtest PT participation	50	5 2 1 1 1	Based on replicates (precision) GUM In house validation Nordtest PT participation
b	45	3 3 2 1	Based on replicates (precision) In house validation Eurachem/CITAC Guide CG4 top-down approach	45	4 2 2 1	Based on replicates (precision) Eurachem/CITAC Guide CG4 In house validation top-down approach	45	4 2 2 1	Based on replicates (precision) Eurachem/CITAC Guide CG4 In house validation top-down approach
c				5.0	1	Based on replicates (precision)	5.0	1	Based on replicates (precision)
Element	Lead			Lead			Lead	
Uncert.	[%]	N° lab	Approach	[%]	N° lab	Approach	[%]	N° lab	Approach
a	61	7 2 2 1 1 1	Based on replicates (precision) GUM In house validation Nordtest PT participation Horwitz	44	5 1 1 1 1 1	Based on replicates (precision) Eurachem/CITAC Guide CG4 Nordtest PT participation Horwitz In house validation	52	6 2 1 1 1 1	Based on replicates (precision) GUM Eurachem/CITAC Guide CG4 Horwitz In house validation PT participation
b	39	3 3 1 1 1	Based on replicates (precision) Eurachem/CITAC Guide CG4 In house validation ISO 11352:2012 top-down approach	52	4 2 2 2 1 1	Based on replicates (precision) Eurachem/CITAC Guide CG4 GUM In house validation ISO 11352:2012 top-down approach	44	3 2 2 1 1 1	Based on replicates (precision) Eurachem/CITAC Guide CG4 In house validation ISO 11352:2012 top-down approach Nordtest
c				4.3	1	Based on replicates (precision)	4.3	1	Based on replicates (precision)

Annex 12. Outcome from Questionnaire

Table 1. Questionnaire part 1 (**Method used**)

Lab code	Did you follow provided SOP?	Which analytical method did you use?	Is the method validated		Is the method accredited	
			Pb/Cd	other	Pb/Cd	other
LC0001	YES	ICP-MS	YES	YES	YES	YES
LC0002	YES	ICP-MS	YES	YES	YES	YES
LC0003	YES	ICP-OES	YES	-	YES	-
LC0004	-	84/500/EEC and SOP	-	-	YES	-
LC0005	YES	-	YES	Cr and Ni	YES	-
LC0006	NO	GF-AAS	YES	-	YES	-
LC0007	YES	GF-AAS and F-AAS	NO	NO	NO	NO
LC0008	YES	ICP-MS	YES	Ba, Co, Cu, Fe, Li, Mn, Zn	YES	Ba, Co, Cu, Fe, Li, Mn, Zn
LC0010	YES	EN 1388-1:1997 and ISO 17294: 2005	YES	YES	YES	NO
LC0011	YES	AAS	NO	-	NO	-
LC0012	YES	ICP-MS	YES	Co, Ni, Cr, Fe, As, Sb, Al, Li	YES	Co
LC0013	NO	GF-AAS and F-AAS	YES	Co, Mn, Cu, Fe, Zn, Ba	YES	-
LC0014	YES	ICP-MS and ICP-OES	YES	Al, Ba, Co, Cu, Fe, Mn, Sb, Zn	YES	YES
LC0016	YES		NOT FULLY	NOT FULLY	NO	NO
LC0017	NO	ICP-MS	YES	Li, Be, Al, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Mo, Sn,Sb, Ba, Hg,Tl	YES	Li, Be, Al, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, As, Mo, Sn,Sb, Ba, Hg,Tl
LC0018	YES	ICP-OES	YES	YES		
LC0020	YES	ISO 17294-2005	YES	YES	YES	YES
LC0022	YES	UNI EN 1388-1:1997	YES	-	YES	-
LC0025	YES	ICP-MS	ONLY ICP-OES	-	ONLY ICP-OES	-
LC0028	YES	ICP-MS	-	-	-	-
LC0029	YES	ICP-MS	NO	NO	NO	NO
LC0031	NO	ICP-MS	YES	Co, Ba, Al, Zn, Cu, Fe, Sb	NO	NO
LC0032	YES	ICP-MS and ICP-OES	YES	YES	YES	YES
LC0037	YES	HR-ICP-MS and GF-AAS	YES	YES	NO	NO
LC0040	-	ICP-MS	YES	YES	YES	-
LC0041	YES	ICP-MS	NO	NO	NO	NO
LC0042	YES	ISO 17294-1/ 2, ICP-MS	NO	NO	NO	NO
LC0043	YES	ICP-MS	-	-	-	-
LC0044	YES	ICP-MS	NO	NO	MIGR. - NO; ICP-MS - YES	MIGR. - NO; ICP-MS - YES
LC0046	YES	ICP-MS	YES	-	YES	-
LC0048	NO	ICP-MS	YES	-	YES	-
LC0049	YES	GF-AAS and ICP-OES	NO	NO	NO	NO
LC0050	YES	ICP-MS	NO	NO	NO	NO
LC0054	NO	AAS and ICPMS	YES	YES	YES	YES
LC0055	YES	ICP-MS	F-AAS	-	F-AAS	-
LC0056	YES	ICP-MS	YES	-	YES	-
LC0059	YES	ICP-MS				
LC0061	YES	ICP-MS	YES	-	YES	-
LC0062	NO	ICP-MS	YES		YES	
LC0068	YES	ICP-MS	YES	Co, Ba, Mn, Ni, Al	NO	NO
LC0097	YES	-	YES	NO	YES	NO
LC0101	YES	ICP-MS	NO	NO	NO	NO
LC0113	YES	ICP-OES	NO	NO	NO	NO
LC0115	YES	ISO 7086, ISO 6486	YES	-	YES	-
LC0116	YES	ICP-MS	NO	NO	NO	NO
LC0118	YES	DM21/3/73	YES	NO	YES	NO
LC0120	YES	ICP-OES	YES	As, Al, Co, Sb, Cr, Cu, Ni, Se, Cd and Pb	YES	As, Al, Co, Sb, Cr, Cu, Ni, Se, Cd and Pb
LC0122	YES	ISO 17294-2:2005	NO	NO	NO	NO
LC0123	NO	THGA AAS	YES	-	NO	-
LC0124	YES	ICP-MS	-	-	-	-

Table 2. Questionnaire part 2 (**Sample preparation**)

Lab code	Amount of sample used for analysis [ml]	Dilution factor used	Migration test applied	Time between migration and analysis	Time between migrations	Any special treatment to the samples
LC0001	10	01:10	as in SOP	1 day	0-2 hours	no
LC0002	-	max. 3000	as in SOP	1 day	max. 3 days	no
LC0003	5 to 10	no	as in SOP	max. 1 day	1 or 3 days	no
LC0004	all sample	no	as in SOP	1 day	immediately	no
LC0005	-	-	as in SOP	1 day	24 hours	no
LC0006	-	OES - no dilution; AAS - min. 1:10	as in SOP	7-14 days	immediately	no
LC0007	0.02 for GF-AAS; 0.5 for FAAS	1:2; 1:4;1:20	as in SOP	0 days	immediately or max. 3 days	no
LC0008	10	2-fold, 10-fold or 100-fold	as in SOP	10 days	from 1 day to 4 days	no
LC0010	10	1:10, 1:200	as in SOP	1 day	30 min.	no
LC0011	0.02 - 0.04	1:4; 1:5; 1:10; 1:25; 1:40	as in SOP	10 days	1 hour	as in SOP
LC0012	500	10only for Pb in 1st migration	as in SOP	1 day	1 hour	no
LC0013	6 or less	None or up to 1:100	as in SOP	7 days	immediately	as in SOP
LC0014	2*10	up to 3:1000	as in SOP	3 to 23 days	< 1 hour	as in SOP
LC0016			as in SOP	10 days	consecutive	
LC0017	1.5	1:10	24h, 22°C (±1°C), covered with PP plate in drying cabinet	4-6 days	2 hours	diluted with Ru and 3,5% HNO3 (with 200µg/L Au)
LC0018	5 and 40	1:5; 1:10; 1:20; 1:100 or 1:500;	as in SOP	few hours	1 day	no
LC0020	1	01:10	as in SOP	1 day	2 hours	no
LC0022	5	no	as in SOP	5 days	1 day	no
LC0025	3 - 5	1:5 or none	as in SOP	7-8 days	10-20 min	as in SOP
LC0028	5	1:10, 1:50, 1:100 and 1:200	as in SOP	2-3 hours	3 consecutive days	as in SOP
LC0029	7	None or 1:50	as in SOP	2 hours	1 hour	no
LC0031	GB: 525, CC: 60	extracts analysed directly	for 24h in an oven at 22±2°C	1 day	1 hour	no
LC0032	0.050	1:10; 1:20; 1:50 and 1:1000	as in SOP	up to one week	from 1 day to 1 week	no
LC0037	5	-	as in SOP	10 days	60-90 min	no
LC0040	3	01:10	as in SOP	3 days	10 min	as in SOP
LC0041	9	1.1	as in SOP	< 7 days	immediately	as in SOP
LC0042	1 to 50	between 1:20 and 1:500	as in SOP	from 1 to 3	5-10 min	as in SOP
LC0043	5	1:10; 1:50; 1:100 and 1:200	as in SOP	2-3 hours	immediately	as in SOP
LC0044	1	1:5	as in SOP	from 4 to 6 days	as in SOP	no
LC0046	500	1:100	as in SOP	1 day	-	no
LC0048	4	1:10; 1:100 and 1:200	as in SOP	1 day	2 hours	as in SOP
LC0049	0.020	from 1 till 25	as in SOP	13 days	from 1 day to 3 days	as in SOP
LC0050	1	2	as in SOP	from 5 till 7 days	immediately	as in SOP
LC0054		different for AAS	as in SOP	From 1 till 5 days	from 1 to 5	no
LC0055	1 till 5	1:5; 1:10 and 1:100	as in SOP	from 1 till 3 days	0	as in SOP
LC0056	10	none	as in SOP	same day	few hours	no
LC0059		5,10,20,50				
LC0061	1	3	as in SOP	< 1 day	24 hours	as in SOP
LC0062	5	01:01	as in SOP	7	Up to 3 days	as in SOP
LC0068	1	1:10, 1:100	as in SOP	till 3 days	as in SOP	as in SOP
LC0097	30	none, 1:2; 1:5; 1:10; 1:20; 1:50 and 1:100	as in SOP	5days	24 hours	no
LC0101	5	1:10 or 1:100	as in SOP	5 days	2-3 hours	sample + HNO3 (1% v/v)
LC0113	50	1:10 and 1:100	as in SOP	1-3 days	1 hour	as in SOP
LC0115		1:1; 1:10; 1:20	ISO 7086 & ISO 6486	till 4 days	some hours	as ISO 7086, ISO 6486
LC0116	30	1:1; 1:50	as in SOP	7 days	immediately	no
LC0118	10	1	as in SOP	10 days	30 minutes	as in SOP
LC0120	20 and 60	1:4; 1:10;and 1:50	as in SOP		from 1 to 5	no
LC0122	15 and 20	1:5; 1:10; 1:50 and 1:200	as in SOP	immediately or 1 day	immediately	as in SOP
LC0123	30	-	as in SOP	1 day	1 day	as in SOP
LC0124	40	1:10	as in SOP	9 days	±30minutes)	no

Table 3. Questionnaire part 3 **(LOD)**

Lab code	LOD [ug/kg]									
	Pb	Cd	Co	Ba	Mn	Al	Cu	Fe	Zn	Sb
LC0001	1	0.5	0.5	1	10	30	1	50	5	1
LC0002	0.0192	0.0091	0.0061	0.068	0.0195	1.0337	0.19	0.3069	0.3797	0.0057
LC0003	5	0.5	0.5	0.2	0.2	0.5	0.2	0.5	0.5	5.5
LC0005	21 mg/L	6 mg/L	-	-	-	-	-	-	-	-
LC0006	0.126	0.029	0.199	0.2	0.554	1.956	0.583	1.213	0.4	0.517
LC0007	F-AAS: 38	-	-	-	13	-	22	59	24	-
	GF-AAS: 0.6	0.04	1.0	-	-	-	-	-	-	-
LC0008	0.33	0.33	0.33	0.33	0.33	0.33	0.33	3.3	3.3	0.33
LC0010	2	0.1	0.5	5	1	5	1	10	5	1
LC0011	2	0.2	4	10	0.1	-	200	500	100	1
LC0012	1	0.5	1	1	1	5	1	5	1	1
LC0013	F-AAS: 50	5	-	-	25	-	100	100	25	-
	GF-AAS: 0.5	0.1	2	10	-	-	-	-	-	-
LC0014	ICP-MS: 2	1	2	2	10	30	100	100	100	1
	ICP-OES: 100	5	50	5	5	50	10	10	10	-
LC0017	0.05	0.0025	0.1	3.8	5.4	10.7	5	126	10.3	0.13
LC0018	4.14	0.512	0.589	0.508	0.111	2.466	0.524	0.735	0.862	5.229
LC0020	1	1	1	1	1	20	5	30	10	1
LC0022	1	1	1	-	1	-	-	-	-	-
LC0031	1	1	1	1	1	1	1	1	1	1
LC0032	0.5	0.5	0.2	0.01	0.01	0.1	0.05	0.05	0.05	0.1
LC0037	0.3	0.1	0.1	1.6	0.6	4.7	2.5	13.6	17.6	1.5
LC0040	0.02	0.01	0.01	0.1	0.12	2	0.1	3.5	0.5	0.03
LC0041	0.8	0.2	9	4	10	70	8	3000	300	0.8
LC0042	1	0.1	-	-	1	20	20	8	-	-
LC0044	0.04	0.04	0.06	0.03	0.29	3.6	0.5	1.3	1.6	0.1
LC0046	200	25	-	-	-	-	-	-	-	-
LC0048	0.007	0.007	0.001	0.003	0.003	0.02	0.001	-	0.013	0.001
LC0049	0.510-5.047	0.340-0.117	0.155	-	38.22	72.14	6.62	473	220	-
LC0050	0.5	0.2	1	1	1	1	1	1	1	1
LC0054	1.4	0.14	2.3	2.9	18	1.7	16.4	44.7	7	-
LC0055	0.05	0.05	0.05	0.5	1	3	2	40	10	0.2
LC0056	0.3	0.05	0.03	0.1	0.1	7	3	5	4	0.1
LC0059	2	0.35	0.4	1.1	1.5	20	8	20	15	3.8
LC0061	1	1	1	1	1	1	1	1	1	1
LC0062	0.09	0.13	0.11	3.4	2.2	10	2.2	5	2	0.13
LC0068	0.026	0.001	0.011	0.004	0.015	0.055	0.01	0.292	0.821	0.001
LC0097	100	10	2.5	-	0.5	-	100	5	25	-
LC0101	1	0.1	0.1	0.2	0.5	10	0.5	1	10	0.1
LC0113	20	10	10	1	10	20	20	100	100	10
LC0115	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
LC0116	5	5	5	-	5	5	5	5	5	-
LC0118	5	0.5	2	1	1	5	2	2	2	-
LC0120	2.5	1	0.5	25	25	25	2.5	25	25	15
LC0122	0.015	0.005	0.004	0.134	0.012	0.744	0.037	0.158	0.184	0.011
LC0123	1.5	0.25	-	-	2.5	0.25	2.5	2.5	0.13	2.5

Table 4. Questionnaire part 4 (**Analytical technique details**)

Lab code	Isotope used (ICP-MS) or wavelenght [nm] (ICP-OES)									
	Pb	Cd	Co	Ba	Mn	Al	Cu	Fe	Zn	Sb
LC0001	206+207 +208	114	59	137	55	27	63	56	66	121
LC0002	206+207 +208	111	59	138	55	27	65	56	66	121
LC0003	220.4nm	226.5nm	228.6nm	233.5nm	257.6nm	396.2nm	324.8nm	259.9nm	213.9nm	217.6nm
LC0004	220.4nm	228.8nm	228.6nm	455.4nm	257.6nm	167.1nm	324.7nm	238.2nm	202.5nm	206.8nm
LC0005	217nm	228.8nm	-	-	-	-	-	-	-	-
LC0006	283.3nm	228.8nm	240.7nm	-	403.1nm	309.3nm	324.8nm	248.3nm	-	217.6nm
LC0007	283.3nm	228.8nm	240.7nm	-	279.5	-	324.8nm	248.3nm	213.9nm	-
LC0008	206+207 +208	111	59	137	55	27	65	54	68	121
LC0010	206+207 +208	111	59	137	55	27	65	54+56	66	121
LC0011	-	-	-	-	-	-	-	-	-	-
LC0012	208	111	59	137	55	27	63	56	66	121
LC0013	-	-	-	-	-	-	-	-	-	-
LC0014	208; 217.0nm 220.4nm	111; 226.5nm 228.8nm	59; 228.6nm 238.9nm	138; 230.4nm 233.5nm	55; 257.61nm 259.4nm	27; 394.4nm 396.2nm	63/65; 324.8nm 327.4nm	56; 238.2nm 239.6nm	66; 206.2nm 213.9nm	121
LC0016	283.3nm	228.8nm	59	493.408	55	396.153	65	259.939	64	121
LC0017	206+207 +208	111	59	135	55	27	65	57	66	121
LC0018	220.4nm	228.8nm	228.6nm	233.5nm	257.6nm	396.2nm	327.4nm	238.2nm	206.2nm	206.8nm
LC0020	208	111	59	137	55	25	65	56	66	121
LC0022	220.4nm	214.4nm	228.6nm	-	257.6nm	-	-	-	-	-
LC0025	208	111	59	230.4nm	293.9nm	309.2nm	324.7nm	240.4nm	206.2nm	121
LC0028	208	111	59	137	55	27	65	57	66	121
LC0029	208	111	59	137	55	27	63	56	66	121
LC0031	208	111	59	138	55	27	63	54	66	121
LC0032	208	111	59	455nm	257nm	396nm	324nm	259nm	213nm	121
LC0037	208	111	59	553.6nm	55	27	63	56	66	121
LC0040	208	111	59	137	55	27	63	56	66	121
LC0041	206	111	59	135	55	27	63	56	64	121
LC0042	208	111	-	-	55	27	63	56	-	-
LC0043	208	111	59	137	55	27	65	57	66	121
LC0044	208	111	59	137	55	27	63	56	66	121
LC0046	208	111	-	-	-	-	-	-	-	-
LC0048	208 STD	111 KED	59 KED	138 STD	55 KED	27 KED	63 KED	-	66 KED	121 STD
LC0049	220.4nm	214nm	230nm	-	257.6nm	394.4nm	324.8nm	259.9nm	213.9nm	-
LC0050	206+207 +208	111	59	138	55	27	63	56	66	121
LC0054				455.4nm						
LC0055	206+207 +208	111	59	138	55	27	63	57	66	121
LC0056	208	111	59	137	55	27	63	56	66	121
LC0059	206+207 +208	111	59	135+137	55	27	65	56	66+67	121
LC0061	208	111	59	135	55	27	63	56	66	123
LC0062	208	114	59	233nm	257nm	396nm	324nm	238nm	213nm	121
LC0068	208	111	59	138	55	27	63	57 KED	66	121
LC0101	206+207 +208	111	59	137	55	27	63	56	66	121
LC0113	220.4nm	228.8nm	228.6nm	455.4nm	257.6nm	237.3nm	324.8nm	259.9nm	206.2mn	206.8nm
LC0115	208	111	59	137	55	27	63	56-57	66	121
LC0116	206+207 +208	114	59	-	55	27	63	56	68	-
LC0118	220.3nm	228.8nm	228.6nm	233.5nm	257.6nm	396.2nm	327.4nm	238.2nm	206.2nm	-
LC0120	220.4 nm	228.8 nm	228.6 nm	233.5 nm	257.6 nm	396.2 nm	327.4 nm	238.2 nm	213.9 nm	217.6 nm
LC0122	206+207 +208	111	59	138	55	27	63	56	66	121
LC0123	283.3nm	228.8nm	-	-	279.5nm	309.3nm	324.8nm	248.3nm	213.9nm	227.6nm
LC0124	208	111	59	138	55	27	-	-	-	-

KJ-NA-28690-EN-N

JRC Mission

As the science and knowledge service of the European Commission, the Joint Research Centre's mission is to support EU policies with independent evidence throughout the whole policy cycle.

EU Science Hub
ec.europa.eu/jrc

@EU_ScienceHub
EU Science Hub - Joint Research Centre
Joint Research Centre
EU Science Hub

Publications Office

doi:10.2760/573036
ISBN 978-92-79-70531-1