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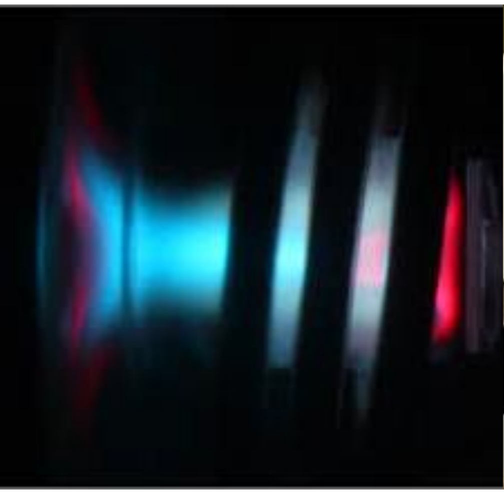

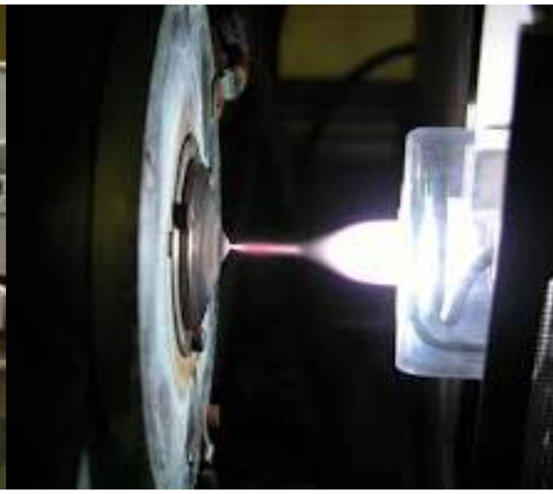

Precision criteria of methods for the quantification of metals migrated from Food Contact Materials

Pre-validation data derived from ILCs on elements representative for plastics and ceramics

Giorgia Beldì, Natalia Jakubowska and Catherine Simoneau

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Abstract

Precision criteria for the quantification of metals in acetic acid 3% and acetic acid 4% migration solutions for different analytical techniques were calculated and are presented in this report. They were derived from two interlaboratory comparisons (ILC03/04 2014) data, including repeatability and reproducibility standard deviations. Three groups of analytical techniques were identified from the ILCs: Inductively coupled plasma mass spectrometry (ICP-MS), inductively coupled plasma Optical emission spectrometry (ICP-OES) and atomic absorption spectrometry (AAS). AAS included graphite furnace (GF-AAS) and flame (F-AAS). Precision criteria were calculated applying robust statistic methods with algorithms A and S (ISO 5725-5 and ISO 13528) and DIN 38402 A45 (Q-method/Hampel-estimator) The robust means calculated according to both algorithms were checked for significant difference (student's t-test) and equivalence test. Relative repeatability standard deviations were very low generally less than 2% and reached 4.4% only for Pb in acetic acid 4%. Relative reproducibility standard deviations for Pb were around 10% for ICP-MS and ICP-OES techniques and 16% for AAS. Relative reproducibility standard deviations for Cd were around 5%, 10% and 20% using respectively ICP-MS, AAS or ICP-OES techniques. For other elements the relative reproducibility standard deviations obtained were for most of the cases less than 10%. From the comparison study it was possible to establish with the exception of Fe, for all elements of the two ILCs in both solutions (corresponding to elements from ceramics and elements from plastics) the analytical techniques were generally equivalent and not significantly different at the concentrations investigated.

Precision criteria on the quantification of metals migrated from Food Contact Materials:

Pre-validation data derived from ILCs on elements representative for plastics and ceramics

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Index

1. Summary

The Institute for Health and Consumer Protection (IHCP) of the European Commission's Directorate-General Joint Research Centre hosts the EU Reference Laboratory for Food Contact Materials (EURL-FCM). One of its core tasks is to organise inter-laboratory comparisons (ILCs) amongst appointed National Reference Laboratories (NRLs). This report presents precision criteria of analytical techniques derived from the results of a proficiency test (PT) organized by the EURL-FCM, which focused on the determination of elements migrated from food contact materials.

The PT was designed to combine two exercises into one unique inter-laboratory comparison (ILC03 2014, ILC04 2014). The first exercise aimed at testing the ability to determine concentrations of metals with migration limits assigned for plastics in the European Regulation (EU) No 10/2011 [1]. It focused on the quantification of Barium (Ba), Cobalt (Co), Copper (Cu), Iron (Fe), Manganese (Mn), Zinc (Zn), Lithium (Li) and Antimony (Sb) in a solution of 3% acetic acid as simulant for acidic foods for contact with plastics. The Sb was undisclosed. The second exercise aimed at testing the ability to determine the migration of Ba, Co, Mn, Lead (Pb), Cadmium (Cd), Nickel (Ni), Arsenic (As), and Aluminium (Al) from ceramics.

In this proficiency test the participants were free to use their own analytical methods. Participants were asked to analyse the analytes in the migration solutions and to report four replicate results for each element and the analytical technique used for quantification. Fifty five laboratories (28 NRLs, 26 Official Control Laboratories (OCLs)) from 27 countries registered for the exercise and fifty four reported results. All results were statistically treated together and the assigned values were derived from the consensus of the results submitted by participants. Laboratories were rated with z-scores in accordance with ISO 13528 [2]. The rate of success was higher than 90% for all elements in both food simulants and the results obtained showed a satisfactory laboratory performance.

The outcome of this exercise was satisfactory. The rate of success was higher than 90% for all elements in both food simulants. The results were then grouped according to the analytical technique used for the quantification with the aim to derive precision criteria for each of them. Three groups were identified: Inductively coupled plasma mass spectrometry (ICP-MS), inductively coupled plasma Optical emission spectrometry (ICP-OES) and Atomic absorption spectrometry (AAS). AAS includes graphite furnace (GF-AAS) and flame (F-AAS). Two laboratories provided results obtained using two different techniques. Laboratories that did not provide details of analytical technique used were not taken into account. Precision criteria were calculated applying robust statistic methods with boths algorithms A and S (ISO 5725-5 and ISO 13528)[2,3] and DIN 38402 A45 (Q-method/Hampel-estimator)[4]. The robust means calculated according to both algorithms were checked for significant difference (student's t-test) and equivalence test. Relative repeatability standard deviations were very low, generally less than 2%, and reached 4.4% only for Pb in 4% acetic acid solution. Relative reproducibility standard deviations for Pb were around 10% for ICP-MS and ICP-OES techniques and 16% for AAS. Relative reproducibility standard deviations for Cd were around 5%, 10% and 20% using respectively ICP-MS, AAS or ICP-OES techniques. For most of the cases the relative reproducibility standard deviations of other elements were lower than 10%. From the comparison study it was possible to establish that the analytical techniques were generally equivalent and not significantly different for all elements and both acetic acid solutions. The only

exception was for Fe in 3% acetic acid solution where results between ICP-MS and ICP-OES were significantly different.

2. Introduction

Inter-laboratory comparisons (ILC) studies are an essential element of laboratory quality assurance, which allow individual laboratories to compare their analytical results against objective standards and with those from other laboratories. In this context, it is one of the core duties of the European Reference Laboratories to organise inter-laboratory comparisons, as is stated in Regulation (EC) No 882/2004 of the European Parliament and of the Council [5].

In accordance with the above requirements the European Reference Laboratory for Food Contact Materials (EURL-FCM) organised ILCs for the network of appointed National Reference Laboratories (NRLs) in 2014. The ILC was a double proficiency test (PT) and focused on the determination of elements that can potentially migrate from plastics and ceramics, respectively. The first ILC03 2014 exercise consisted in the quantification of the 7 metals subjected to specific migration limits (SMLs) assigned for plastics materials and articles in Annex II of Regulation (EU) 10/2011 [1]. Ba, Co, Cu, Fe, Mn, Zn, Li plus Sb as undisclosed element. The second ILC04 2014 exercise focused on the quantification of 8 elements relevant to ceramics, i.e. Ba, Co, Mn, Pb, Cd, Ni, As, and Al. The concentration values were much lower than those laid down in the ceramic Directive 84/500/EEC and were chosen as a compromise between the values adopted by the Council of Europe (CoE) in the context of metals and alloys and the discussion starting values (DSVs) proposed by DG-SANTE.

In this follow up work the inetion was to group the results based on the analytical technique with the aim to derive precision criteria for each of them. Three groups were identified: Inductively coupled plasma mass spectrometry (ICP-MS), inductively coupled plasma optical emission spectrometry (ICP-OES) and atomic absorption spectrometry (AAS). AAS included both GF-AAS and F-AAS to have sufficient number of results to be treated statistically.

3. Scope

The objectives of this report were the assessment of precision criteria from the ILC03/04 2014 data, including reproducibility and repeatability standard deviations for the quantification of metals in acidic migration solutions using different analytical techniques.
The data can be used to contribute to the development of international standards and can also serve as evaluate the enforceability for elements under the scope of the plastics Regulation (EU) 10/2011 [1] as well as towards a potential revision of the ceramics Directive 84/500/EEC [6].

4. Test materials

A solution of acetic acid 3 % (w/v) spiked with Ba, Co, Cu, Fe, Mn, Zn, Li and Sb (PL1) and a solution of acetic acid 4 % (v/v) spiked with Ba, Co, Mn, Pb, Cd, Ni, As, and Al (C2).

5. ILC03/04 2014 overall outcome

Fifty five laboratories (28 NRLs, 26 OCLs and the EURL-FCM) from 27 countries registered for the exercise and fifty four reported results (98%). The percentage of participation considering individual elements is reported in table 1.

Table 1. Percentage of participation for individual elements.

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

As requested, most of the laboratories reported four replicate results under repeatability conditions. The techniques used for the determination of elements by the laboratories for this exercise were ICP-MS, ICP-OES, F-AAS, GF-AAS and combinations of them, such as ICP-MS+F-AAS.

In the ILC03/04 2014 the true values for the concentration of the elements in the samples, were treated as unknown. As there were no other reference values available, the robust mean values obtained from the reported results of the participants were used as assigned values. The robust mean, the repeatability and reproducibility standard deviations were obtained using robust method DIN 38402 A45 (Q-method/Hampel-estimator). All results were analysed together without any grouping based on analytical method. The statistical evaluation of the results was performed using the ProLab software [7]. The final results are reported in table 2.

The overall outcome for both exercises was satisfactory. The majority of laboratories obtained good results for all elements in both samples. The rate of success reported in table 3 was higher than 90% for almost all elements in both food simulants, apart from the 80% for As due to a background amount of arsenic in the blank. The results of the robust statistics indicated a good reproducibility for the determination of most of the elements. The relative reproducibility standard deviation ranged between 5% and 16% for metals in both solutions. The only exception was the relative reproducibility standard deviation of As of 34% for As.

Table 2. ILC03/04 2014 Summary results for Al, As, Ba, Cd, Co, Pb, Mn and Ni in sample C2 and Sb, Ba, Co, Cu, Fe, Li, Mn and Zn in sample PL1 calculated according to DIN 38402 A45.

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

Table 3. Percentage of successfully results for the ILC03/04-2014

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

6. Precision criteria by analytical techniques

Taking into account the high rate of success of the ILC03/04 2014 the raw results provided (annex 1) were retreated statistically after a grouping them according to the analytical technique applied. The overall aim was to compare different analytical techniques (e.g. with regard to the mean values, sample-specific differences, differences across samples, differences across analytes) and derive precision criteria for each technique.

It was not possible to align the minimum number of participants to the various statistical methods for all situations. The minimum number depended on a variety of situations:

- The statistical method used (particular robust method or outlier removal);
- The experience of the participants and PT provider;
- Whether the intent is to determine the assigned value or the standard deviation (or both).

For example, some outlier techniques require at least 20 results; others can be used with as few as 7 results. A median can be determined with as few as 2 values, but this would not be recommended; however with experienced participants and the ability to compare a small data set with prior expectations, a median could be used with as few as 5 results. Estimates of the standard deviation using robust procedures suggest at least 12 results for DIN 38402 A45 and 6 or better 8 results for ISO 5725.

The analytical techniques used for the quantification of elements in acidic solutions for this exercise were ICP-MS, ICP-OES, F-AAS, GF-AAS and combinations of them, such as ICP-MS + F-AAS. Three different groups were identified: ICP-MS, ICP-OES and AAS. The F-AAS and GF-AAS techniques were grouped together to reach an adequate number of results to be treated statistically. Few laboratories provided results obtained with two different analytical techniques, in this case the results were divided and treated separately according the groups identified. Tables 4 and 5 show an overview of the analytical techniques used for the quantification of metals in PL1 and C2 samples. The ICP-MS was the most applied technique for both samples with a percentage greater than 50%. ICP-OES was used from 12% to 33% of the participants depending on the element and sample. AAS was the least used analytical technique except for Pb and Cd in sample C2 (26%).

The three groups (ICP-MS, ICP-OES and AAS) were statistically treated independently to derive the assigned value, repeatability and reproducibility standard deviations as well as repeatability and reproducibility limits for each analytical technique. As there were no other reference values available, the robust mean values obtained from the results reported by the participants were used as assigned values. The robust mean and the precision parameters were obtained using robust methods DIN 38402 A45 (Q-method/Hampel-estimator) and Algorithms A and S (ISO 5725-5 and ISO 13528). These methods are designed primarily for the analysis of proficiency testing schemes, but they can also be used for method validation ring tests. Robust statistical methods provide procedures that can be thought of as describing a central part of the distribution of results from competent participants, but without requiring the identification of specific values as outliers and excluding them from subsequent analyses. If a normal distribution for method validation ring test results is assumed, DIN 38402 A45 give results that are very similar to ISO 5725-5. DIN 38402 A45 remains viable even with more than 40% outlier laboratories [7]. It should be noted that no tests for outliers are carried out when the Hampel estimator is used. The DIN 38402 A45 algorithm works in a way that values which differ more than 3 times the standard deviation from the mean value are truncated more than using a Huber estimator. The algorithm also excludes values which differ more than 4.5 times the standard deviation and do not affect the calculated results [7]. In Algorithms A and S (ISO 5725-5 and ISO 13528) the extreme outliers are taken into account in the calculation of the mean values (Huber). The results reported as "smaller than" (<values) were not used in any calculation.

Tables 6-11 summarise the results for Al, Ba, Cd, Co, Pb, Mn and Ni in sample C2 and Sb, Ba, Co, Cu, Fe, Li, Mn and Zn in sample PL1 obtained according to ISO 5725-5 Algorithm A+S and DIN 38402 A45 for each analytical technique group are reported. The tables report the number of laboratories, robust means, repeatability and reproducibility standard deviations, relative repeatability and reproducibility standard deviations, as well as repeatability and reproducibility limits. Values marked in red were obtained with less than 6 laboratory results. No precision criteria were calculated for As in sample C2 due to a possible contamination of the blank.

Table 4. Overview of analytical techniques used to quantify sample PL1

SAMPLE PL1								
Lab. Code	Ba	Co	Cu	Fe	Mn	Zn	Li	Sb
LC0003								
LC0004								
LC0005								
LC0006								
LC0007								
LC0008								
LC0010								
LC0011								
LC0013								
LC0016								
LC0017								
LC0018								
LC0020								
LC0022								
LC0024								
LC0025								
LC0028								
LC0029								
LC0031								
LC0032								
LC0033								
LC0034								
LC0037								
LC0037b								
LC0038								
LC0039								
LC0040								
LC0041								
LC0042								
LC0043								
LC0044								
LC0046								
LC0049								
LC0050								
LC0055								
LC0056								
LC0056b								
LC0061								
LC0062								
LC0063								
LC0064								
LC0065								
LC0067								
LC0068								
LC0089								
LC0097								
LC0101								
LC0106								
LC0113								
LC0114								
LC0115								
LC0116								
LC0117								
LC0118								
LC0119								
LC0120								
Sum ICP-MS	26 61.9%	28 59.6%	27 54.0%	25 53.2%	26 53.1%	24 50.0%	22 57.9%	25 80.6%
Sum ICP-OES	14 33.3%	11 23.4%	13 26.0%	12 25.5%	13 26.5%	14 29.2%	13 34.2%	5 16.1%
Sum AAS	2 4.8%	8 17.0%	10 20.0%	10 21.3%	10 20.4%	10 20.8%	3 7.9%	1 3.2%
Total	42	47	50	47	49	48	38	31

Table 5. Overview of analytical techniques used to quantify sample C2

	SAMPLE C2							
Lab. Code	Ba	Co	Mn	Pb	Cd	Ni	As	Al
LC0003								
LC0004								
LC0005								
LC0006								
LC0007								
LC0008								
LC0010								
LC0011								
LC0013								
LC0016								
LC0017								
LC0018								
LC0020								
LC0022								
LC0024								
LC0025								
LC0028								
LC0029								
LC0031								
LC0032								
LC0033								
LC0034								
LC0037								
LC0037b								
LC0038								
LC0039								
LC0040								
LC0041								
LC0042								
LC0043								
LC0044								
LC0046								
LC0049								
LC0050								
LC0055								
LC0056								
LC0056b								
LC0061								
LC0062								
LC0063								
LC0064								
LC0065								
LC0067								
LC0068								
LC0089								
LC0097								
LC0101								
LC0106								
LC0113								
LC0114								
LC0115								
LC0116								
LC0117								
LC0118								
LC0119								
LC0120								
Sum ICP-MS	26 65.0%	28 62.2%	26 54.2%	30 61.2%	29 59.2%	28 59.6%	30 71.4%	28 68.3%
Sum ICP-OES	13 32.5%	11 24.4%	12 25.0%	6 12.2%	8 16.3%	10 21.3%	8 19.0%	12 29.3%
Sum AAS	1 2.5%	6 13.3%	10 20.8%	13 26.5%	12 24.5%	9 19.1%	4 9.5%	1 2.4%
Total	40	45	48	49	49	47	42	41

Table 6. (ICP-MS) summary of results for Ba, Co, Cu, Fe, Mn, Zn, Li and Sb in sample PL1 and Ba, Co, Mn, Pb, Cd, Ni and Al in sample C2 calculated according to ISO 5725-5 Algorithm A+S

Sample	Element	N° Lab	Robust Mean [µg/kg]	Reprod. S.D. [µg/kg]	Rel. Reprod. S.D. [%]	Repeat.S.D. [µg/kg]	Rel.repeat. S.D. [%]	Repeatability Limit (r) [µg/kg]	Reproducibility Limit (R) [µg/kg]
PL1	Ba	26	693.31	31.90	4.60	10.09	1.46	28.25	89.32
	Co	28	50.91	2.86	5.61	0.87	1.71	2.43	8.00
	Cu	27	3062.18	146.77	4.79	36.04	1.18	100.92	410.97
	Fe	25	19151.54	1675.18	8.75	337.13	1.76	943.97	4690.51
	Mn	26	494.65	24.50	4.95	7.42	1.50	20.76	68.59
	Zn	24	20127.54	906.02	4.50	245.08	1.22	686.24	2536.86
	Li	22	505.01	26.92	5.33	9.80	1.94	27.43	75.37
	Sb	25	34.49	3.92	11.38	0.57	1.65	1.60	10.99
C2	Ba	26	785.23	38.17	4.86	8.71	1.11	24.37	106.86
	Co	28	51.27	1.85	3.62	0.88	1.72	2.48	5.19
	Mn	26	397.82	17.67	4.44	5.54	1.39	15.50	49.48
	Pb	30	9.58	0.99	10.32	0.20	2.13	0.57	2.77
	Cd	29	4.89	0.24	4.89	0.09	1.77	0.24	0.67
	Ni	28	69.92	3.62	5.17	1.25	1.79	3.50	10.13
	Al	28	728.78	66.06	9.06	17.29	2.37	48.40	184.95

Table 7. (ICP-MS) summary of results for Ba, Co, Cu, Fe, Mn, Zn, Li and Sb in sample PL1 and Ba, Co, Mn, Pb, Cd, Ni and Al in sample C2 calculated according to DIN 38402 A45

Sample	Element	N° Lab	Robust Mean [µg/kg]	Reprod. S.D. [µg/kg]	Rel. Reprod. S.D. [%]	Repeat.S.D. [µg/kg]	Rel.repeat. S.D. [%]	Repeatability Limit (r) [µg/kg]	Reproducibility Limit (R) [µg/kg]
PL1	Ba	26	694.55	37.57	5.41	8.15	1.17	22.83	105.18
	Co	28	50.64	3.22	6.36	0.87	1.72	2.44	9.02
	Cu	27	3048.73	147.36	4.83	30.49	1.00	85.36	412.61
	Fe	25	19493.66	1226.29	6.29	273.63	1.40	766.15	3433.61
	Mn	26	493.55	22.02	4.46	6.92	1.40	19.38	61.66
	Zn	24	20212.01	865.10	4.28	208.61	1.03	584.10	2422.28
	Li	22	502.95	28.31	5.63	8.14	1.62	22.78	79.26
	Sb	25	34.16	3.94	11.53	0.52	1.53	1.46	11.03
C2	Ba	26	786.62	42.22	5.37	8.03	1.02	22.49	118.21
	Co	28	51.29	2.09	4.07	0.85	1.65	2.37	5.84
	Mn	26	398.46	19.65	4.93	4.22	1.06	11.82	55.02
	Pb	30	9.62	1.06	10.97	0.18	1.91	0.52	2.96
	Cd	29	4.89	0.27	5.53	0.08	1.62	0.22	0.76
	Ni	28	69.85	3.46	4.95	0.98	1.40	2.73	9.68
	Al	28	728.92	78.06	10.71	13.88	1.90	38.87	218.56

Table 8. (ICP-OES) summary of results for Ba, Co, Cu, Fe, Mn, Zn, Li and Sb in sample PL1 and Ba, Co, Mn, Pb, Cd, Ni and Al in sample C2 calculated according to ISO 5725-5 Algorithm A+S

Sample	Element	N° Lab	Robust Mean [µg/kg]	Reprod. S.D. [µg/kg]	Rel. Reprod. S.D. [%]	Repeat.S.D. [µg/kg]	Rel.repeat. S.D. [%]	Repeatability Limit (r) [µg/kg]	Reproducibility Limit (R) [µg/kg]
PL1	Ba	14	719.62	73.79	10.25	5.61	0.78	15.71	206.61
	Co	11	51.20	2.74	5.35	0.70	1.37	1.96	7.67
	Cu	13	3148.60	266.15	8.45	26.83	0.85	75.12	745.22
	Fe	12	20416.13	835.78	4.09	193.37	0.95	541.43	2340.19
	Mn	13	499.98	24.63	4.93	3.63	0.73	10.17	68.97
	Zn	14	19703.50	1380.38	7.01	241.07	1.22	674.99	3865.06
	Li	13	507.27	42.45	8.37	6.23	1.23	17.43	118.85
	Sb	5	44.01	8.46	19.22	0.94	2.13	2.62	23.68
C2	Ba	13	841.59	150.07	17.83	5.76	0.68	16.12	420.19
	Co	11	53.23	3.34	6.27	0.54	1.01	1.51	9.34
	Mn	12	399.67	20.92	5.23	2.94	0.74	8.23	58.57
	Pb	6	9.12	1.24	13.62	0.29	3.23	0.82	3.48
	Cd	8	5.09	0.98	19.20	0.05	0.91	0.13	2.73
	Ni	10	73.95	5.37	7.26	0.73	0.99	2.04	15.04
	Al	12	722.55	101.01	13.98	10.39	1.44	29.10	282.82

Table 9. (ICP-OES) summary of results for Ba, Co, Cu, Fe, Mn, Zn, Li and Sb in sample PL1 and Ba, Co, Mn, Pb, Cd, Ni and Al in sample C2 calculated according to DIN 38402 A45

Sample	Element	N° Lab	Robust Mean [µg/kg]	Reprod. S.D. [µg/kg]	Rel. Reprod. S.D. [%]	Repeat.S.D. [µg/kg]	Rel.repeat. S.D. [%]	Repeatability Limit (r) [µg/kg]	Reproducibility Limit (R) [µg/kg]
PL1	Ba	14	692.28	45.65	6.59	5.21	0.75	14.58	127.82
	Co	11	50.79	2.59	5.10	0.75	1.47	2.09	7.25
	Cu	13	3147.73	260.07	8.26	29.85	0.95	83.57	728.20
	Fe	12	20449.78	924.00	4.52	157.95	0.77	442.25	2587.21
	Mn	13	496.91	27.25	5.48	2.88	0.58	8.08	76.29
	Zn	14	19513.39	1635.35	8.38	266.77	1.37	746.96	4578.99
	Li	13	502.59	42.70	8.50	6.57	1.31	18.40	119.55
	Sb	5	42.95	5.61	13.07	0.95	2.21	2.65	15.71
C2	Ba	13	837.67	84.95	10.14	7.51	0.9	21.03	237.86
	Co	11	52.73	3.07	5.82	0.66	1.25	1.85	8.59
	Mn	12	403.14	19.20	4.76	2.42	0.6	6.77	53.75
	Pb	6	9.11	0.91	9.99	0.41	4.45	1.14	2.55
	Cd	8	5.09	0.97	19.16	0.07	1.37	0.20	2.73
	Ni	10	72.85	4.84	6.64	0.84	1.16	2.36	13.55
	Al	12	740.45	104.11	14.06	9.74	1.32	27.28	291.49

Table 10. (AAS) summary of results for Ba, Co, Cu, Fe, Mn, Zn, Li and Sb in sample PL1 and Ba, Co, Mn, Pb, Cd, Ni and Al in sample C2 calculated according to ISO 5725-5 Algorithm A+S

Sample	Element	N° Lab	Robust Mean [µg/kg]	Reprod. S.D. [µg/kg]	Rel. Reprod. S.D. [%]	Repeat.S.D. [µg/kg]	Rel.repeat. S.D. [%]	Repeatability Limit (r) [µg/kg]	Reproducibility Limit (R) [µg/kg]
PL1	Ba	2	560.43	102.86	18.35%	51.73	9.23	144.85	288.00
	Co	8	47.96	9.10	18.97%	0.78	1.63	2.19	25.47
	Cu	10	3109.85	195.19	6.28%	40.92	1.32	114.59	546.54
	Fe	10	19365.19	1678.35	8.67%	511.86	2.64	1433.19	4699.39
	Mn	10	503.50	41.41	8.22%	13.03	2.59	36.48	115.95
	Zn	10	19344.58	1426.53	7.37%	159.67	0.83	447.07	3994.29
	Li	3	491.14	19.01	3.87%	5.58	1.14	15.62	53.23
	Sb	1	-	-	-	-	-	-	-
C2	Ba	1	-	-	-	-	-	-	-
	Co	6	49.82	4.60	9.23%	1.30	2.6	3.63	12.87
	Mn	10	398.93	42.45	10.64%	5.71	1.43	15.99	118.87
	Pb	13	9.23	1.43	15.51%	0.24	2.59	0.67	4.01
	Cd	12	4.78	0.38	8.03%	0.10	1.98	0.27	1.07
	Ni	9	73.09	5.71	7.81%	1.99	2.72	5.58	15.99
	Al	1	-	-	-	-	-	-	-

Table 11. (AAS) summary of results for Ba, Co, Cu, Fe, Mn, Zn, Li and Sb in sample PL1 and Ba, Co, Mn, Pb, Cd, Ni and Al in sample C2 calculated according to DIN 38402 A45.

Sample	Element	N° Lab	Robust Mean [µg/kg]	Reprod. S.D. [µg/kg]	Rel. Reprod. S.D. [%]	Repeat.S.D. [µg/kg]	Rel.repeat. S.D. [%]	Repeatability Limit (r) [µg/kg]	Reproducibility Limit (R) [µg/kg]
PL1	Ba	2	560.43	173.76	31.01	13.92	2.48	38.98	486.52
	Co	8	47.96	9.01	18.79	0.66	1.38	1.85	25.23
	Cu	10	3075.36	204.16	6.64	30.85	1.00	86.38	571.65
	Fe	10	19365.19	1656.91	8.56	428.10	2.21	1198.68	4639.36
	Mn	10	503.50	52.14	10.36	7.41	1.47	20.74	146.00
	Zn	10	19344.58	1593.14	8.24	157.53	0.81	441.09	4460.80
	Li	3	491.14	36.99	7.53	4.98	1.01	13.94	103.56
	Sb	1	-	-	-	-	-	-	-
C2	Ba	1	-	-	-	-	-	-	-
	Co	6	48.45	4.91	10.14	1.52	3.14	4.26	13.76
	Mn	10	399.54	39.16	9.80	5.85	1.46	16.38	109.66
	Pb	13	9.18	1.47	16.04	0.23	2.54	0.65	4.12
	Cd	12	4.77	0.43	9.04	0.09	1.94	0.26	1.21
	Ni	9	73.20	6.36	8.69	1.85	2.52	5.17	17.80
	Al	1	-	-	-	-	-	-	-

7. Comparison of the analytical techniques

All results reported in the ILC03/04 2014 were graphically analysed by analytical technique used to check if any visual difference could be detected. From those graphs it was however not possible to underline any clear difference among them (e.g. for Pb and Cd annex 2). Thus robust means obtained for the three selected groups were compared using the classical t-test approach and the test on equivalence of systematic differences.

A t-test is any statistical hypothesis test in which the test statistic follows a Student's t distribution if the null hypothesis is assumed. The t-test looks at the t-statistic, t-distribution and degrees of freedom to determine a p-value (probability) that can be used to determine whether the population of means differ.

To obtain the t value, the difference between the mean values is divided by the related standard errors and can be calculated as follows:

$$t = \frac{|\overline{X_1} - \overline{X_2}|}{\sqrt{\frac{s_1^2}{n_1} + \frac{s_2^2}{n_2}}}$$

$$\frac{{s_1}^2}{n_1} = {s_{er1}}^2$$

$$\frac{{s_2}^2}{n_2} = {s_{er2}}^2$$

where:

$\overline{X_1}$ and $\overline{X_2}$ are the robust mean values of two techniques compared;
s_1 and s_2 are the respective standard deviations;
n_1 and n_2 are the number of laboratories which applied each analytical technique;
s_{er1} and s_{er2} are the respective standard error.

T_{crit} are taken from the Student's t distribution table and depends on α= 5% and degree of freedom df. df are obtained based on Satterthwaite formula and can be calculated as follow :

$$df = \frac{\left(\frac{s_1^2}{n_1} + \frac{s_2^2}{n_2}\right)^2}{\frac{1}{n_1 - 1}\left(\frac{s_1^2}{n_1}\right)^2 + \frac{1}{n_2 - 1}\left(\frac{s_2^2}{n_2}\right)^2}$$

If the *t* is smaller than T_{crit} analytical techniques do not differ significantly.

The principle of equivalence states that two analytical methods have to be considered as equivalent if their deviation falls below a certain value with a certain level of statistical significance. The proof of equivalence always includes several aspects. Thus in the context of a ring test it has to be ensured that besides the deviation of the general mean, the standard deviation under repeatability and reproducibility conditions do not exceed a given value.

For assessing if two analytical techniques are equivalent for a particular sample (PL1 or C2) the following steps were applied:

1. Assigning the significance level α = 5%;
2. Assigning the maximal tolerated theoretical deviation Δ_{WFR} = 20% taking into consideration low concentration levels of elements in acetic acid solutions and analytical requirements;
3. Calculation of the empirical relative deviation (Bias [%]):

$$\frac{\overline{X_1} - \overline{X_2}}{\overline{X_2}}$$

If this value already exceeds the predetermined value Δ_{WFR}, there is no need for further calculations, as the equivalence criterion $\frac{|\overline{X_1} - \overline{X_2}|}{\overline{X_2}} < \Delta_{WFR}$ is not satisfied;

4. Calculation of the tolerances $Var[\overline{X_1}] = \frac{s_1^2}{n_1}$ and $Var[\overline{X_2}] = \frac{s_2^2}{n_2}$ with robust statistics (Q method);
5. Calculation of degrees of freedom df_1 and of the non-centrality parameter δ_1 according to:

$$df_1 = min\{n_1, n_2\} - 1$$

and

$$\delta_1 = \frac{\Delta_{WFR}}{\frac{\sqrt{Var[\overline{X_1}] + Var[\overline{X_2}]}}{\overline{X_2}}}$$

6. Calculation of the critical value k from the condition $F_{t(df_1,\delta_1)}(k) - F_{t(df_1,\delta_1)}(-k) = \alpha$, for this purpose, an iterative approach is required. F_t is the distribution function of the non-central t distribution;

7. Calculation of the maximum tolerated empirical limit

$$\Delta_{emp} = \frac{\sqrt{Var[\overline{X_1}] + Var[\overline{X_2}]}}{\overline{X_2}} \; k(\alpha, df_1, \Delta_{WFR}, \delta_1)$$

8. Verification of the criterion:

$$\frac{\overline{X_1} - \overline{X_2}}{\overline{X_2}} < \frac{\sqrt{Var[\overline{X_1}] + Var[\overline{X_2}]}}{\overline{X_2}} k(\alpha, df_1, \Delta_{WFR}, \delta_1)$$

Equivalence is verified if the absolute value of the empirical relative deviation is not larger than Δ_{emp}
All those calculations explained above were performed using ProLab software [7], which takes only results obtained according DIN 38402 A45.
Results from the t-test and equivalence test were combined to build a decision matrix as is reported in table 12.

Table 12. the decision matrix for t test and equivalence test is presented

Decision t-test	Decision test for equivalence of systematic differences		
	Equivalent	**Not Equivalent**	**No decision possible**
Not Different	Equivalent and no significant differences	Although not significant the deviation between method exceed tolerated limits	No significant differences and no decision with regards to equivalence
Different	Significant differences but equivalent with regard to tolerated limits	Not equivalent	Significantly different but not decision with regard to equivalence

All results obtained after comparison of the analytical techniques used are presented in tables 13 and 14.

Table 13. Summary of t- test and equivalence test results for PL1 according to DIN 38402 A45

DIN 38402 A45									
Sample		PL1							
Element		**Ba**	**Co**	**Cu**	**Fe**	**Mn**	**Zn**	**Li**	**Sb**
Level of significance		5.00%	5.00%	5.00%	5.00%	5.00%	5.00%	5.00%	5.00%
Δ_{WFR}		+/- 20.0 %	+/- 20.0 %	+/- 20.0 %	+/- 20.0 %	+/- 20.0 %	+/- 20.0 %	+/- 20.0 %	+/- 20.0 %
ICP-MS	No. of laboratories	26	28	27	25	26	24	22	25
	Mean [µg/kg]	694.55	50.64	3048.73	19493.66	493.55	20212.01	502.95	34.16
	Standard error	7.56	0.62	29.10	251.63	4.43	181.18	6.19	0.81
ICP-OES	No. of laboratories	14	11	13	12	13	14	13	5
	Mean [µg/kg]	692.28	50.79	3147.73	20449.78	496.91	19513.39	502.59	42.95
	Standard error	12.52	0.80	74.01	273.67	7.75	448.42	12.15	2.58
t test	t value	0.16	0.15	1.25	2.57	0.38	1.45	0.03	3.26
	Critical value	2.07	2.07	2.12	2.05	2.09	2.11	2.10	2.57
Test on equivalence	Relative difference	-0.33%	0.29%	3.25%	4.90%	0.68%	-3.46%	-0.07%	25.73%
	Δ_{emp}	+/- 14.41%	+/- 14.11%	+/- 13.89%	+/- 14.31%	+/- 14.51%	+/- 14.18%	+/- 13.80%	+/- 6.88%
Test decision		equivalent and no significant differences	equivalent and no significant differences	equivalent and no significant differences	significant differences but equivalent with regard to tolerated limits	equivalent and no significant differences	equivalent and no significant differences	equivalent and no significant differences	not equivalent
ICP-MS	No. of laboratories	26	28	27	25	26	24	22	-
	Mean [µg/kg]	694.55	50.64	3048.73	19493.66	493.55	20212.01	502.95	-
	Standard error	7.56	0.62	29.10	251.63	4.43	181.18	6.19	-
AAS	No. of laboratories	2	8	10	10	10	10	3	-
	Mean [µg/kg]	560.43	47.96	3075.36	19365.19	503.50	19344.58	491.14	-
	Standard error	126.06	3.27	66.24	537.57	16.92	516.88	21.91	-
t test	t value	1.06	0.81	0.37	0.22	0.57	1.58	0.52	-
	Critical value	12.71	2.31	2.16	2.16	2.23	2.20	4.30	-
Test on equivalence	Relative difference	-19.31%	-5.30%	0.87%	-0.66%	2.02%	-4.29%	-2.35%	-
	Δ_{emp}	+/- 2.61%	+/- 8.83%	+/- 13.70%	+/- 13.15%	+/- 12.68%	+/- 13.44%	+/- 10.03%	-
Test decision		no significant differences and no decision with regard to equivalence	equivalent and no significant differences	equivalent and no significant differences	equivalent and no significant differences	equivalent and no significant differences	equivalent and no significant differences	equivalent and no significant differences	-
ICP-OES	No. of laboratories	14	11	13	12	13	14	13	-
	Mean [µg/kg]	692.28	50.79	3147.73	20449.78	496.91	19513.39	502.59	-
	Standard error	12.52	0.80	74.01	273.67	7.75	448.42	12.15	-
AAS	No. of laboratories	2	8	10	10	10	10	3	-
	Mean [µg/kg]	560.43	47.96	3075.36	19365.19	503.50	19344.58	491.14	-
	Standard error	126.06	3.27	66.24	537.57	16.92	516.88	21.91	-
t test	t value	1.04	0.84	0.73	1.80	0.35	0.25	0.46	-
	Critical value	12.71	2.31	2.08	2.15	2.16	2.09	3.18	-
Test on equivalence	Relative difference	-19.05%	-5.58%	-2.30%	-5.30%	1.33%	-0.87%	-2.28%	-
	Δ_{emp}	+/- 2.61%	+/- 8.75%	+/- 13.05%	+/- 13.24%	+/- 12.48%	+/- 12.72%	+/- 9.70%	-
Test decision		no significant differences and no decision with regard to equivalence	equivalent and no significant differences	equivalent and no significant differences	equivalent and no significant differences	equivalent and no significant differences	equivalent and no significant differences	equivalent and no significant differences	-

Table 14. Summary of t- test and equivalence test results for C2 according to DIN 38402 A45

DIN 38402 A45								
Sample		C2						
Element		**Ba**	**Co**	**Mn**	**Pb**	**Cd**	**Ni**	**Al**
Level of significance		5.00%	5.00%	5.00%	5.00%	5.00%	5.00%	5.00%
Δ_{WFR}		+/- 20.0 %	+/- 20.0 %	+/- 20.0 %	+/- 20.0 %	+/- 20.0 %	+/- 20.0 %	+/- 20.0 %
ICP-MS	No. of laboratories	26	28	26	30	29	28	28
	Mean [µg/kg]	786.62	51.29	398.46	9.62	4.89	69.85	728.92
	Standard error	8.49	0.41	3.95	0.20	0.05	0.67	15.13
ICP-OES	No. of laboratories	13	11	12	6	8	10	12
	Mean [µg/kg]	837.67	52.73	403.14	9.11	5.09	72.85	740.45
	Standard error	24.17	0.95	5.69	0.38	0.35	1.57	30.83
t test	t value	1.99	1.39	0.68	1.21	0.55	1.76	0.34
	Critical value	2.13	2.15	2.07	2.31	2.37	2.18	2.11
Test on equivalence	Relative difference	6.49%	2.80%	1.18%	-5.37%	3.98%	4.29%	1.58%
	Δ_{emp}	+/- 13.27%	+/- 14.10%	+/- 14.42%	+/- 11.15%	+/- 7.80%	+/- 13.65%	+/- 11.51%
Test decision		equivalent and no significant differences	equivalent and no significant differences	equivalent and no significant differences	equivalent and no significant differences	equivalent and no significant differences	equivalent and no significant differences	equivalent and no significant differences
ICP-MS	No. of laboratories	-	28	26	30	29	28	-
	Mean [µg/kg]	-	51.29	398.46	9.62	4.89	69.85	-
	Standard error	-	0.41	3.95	0.20	0.05	0.67	-
AAS	No. of laboratories	-	6	10	13	12	9	-
	Mean [µg/kg]	-	48.45	399.54	9.18	4.77	73.20	-
	Standard error	-	2.06	12.71	0.42	0.13	2.17	-
t test	t value	-	1.36	0.08	0.96	0.86	1.48	-
	Critical value	-	2.57	2.20	2.10	2.13	2.23	-
Test on equivalence	Relative difference	-	-5.54%	0.27%	-4.61%	-2.43%	4.80%	-
	Δ_{emp}	-	+/- 11.51%	+/- 12.88%	+/- 11.43%	+/- 13.60%	+/- 12.82%	-
Test decision		-	equivalent and no significant differences	equivalent and no significant differences	equivalent and no significant differences	equivalent and no significant differences	equivalent and no significant differences	-
ICP-OES	No. of laboratories	-	11	12	6	8	10	-
	Mean [µg/kg]	-	52.73	403.14	9.11	5.09	72.85	-
	Standard error	-	0.95	5.69	0.38	0.35	1.57	-
AAS	No. of laboratories	-	6	10	13	12	9	-
	Mean [µg/kg]	-	48.45	399.54	9.18	4.77	73.20	-
	Standard error	-	2.06	12.71	0.42	0.13	2.17	-
t test	t value	-	1.89	0.26	0.13	0.84	0.13	-
	Critical value	-	2.37	2.16	2.13	2.26	2.13	-
Test on equivalence	Relative difference	-	-8.12%	-0.89%	0.81%	-6.17%	0.49%	-
	Δ_{emp}	-	+/- 11.31%	+/- 12.77%	+/- 9.16%	+/- 7.67%	+/- 12.42%	-
Test decision		-	equivalent and no significant differences	equivalent and no significant differences	equivalent and no significant differences	equivalent and no significant differences	equivalent and no significant differences	-

8. Conclusion

Precision criteria from the ILC03/04 2014 data, including the repeatability and reproducibility standard deviations for the quantification of metals in acetic acid 3% and acetic acid 4% migration solutions for different analytical techniques were calculated using robust approaches. Relative repeatability standard deviations were very low generally less than 2% and reached 4.4% only for Pb in sample C2.

Relative reproducibility standard deviations for Pb were around 10% for ICP-MS and ICP-OES techniques and 16% for AAS. Relative reproducibility standard deviations for Cd were around 5%, 10% and 20% using respectively ICP-MS, AAS or ICP-OES techniques. For other elements the relative reproducibility standard deviations obtained were for most of the cases less than 10%.

From the comparison study it was concluded that the analytical techniques were equivalent and not significantly different for most elements in both samples. The only expection was Fe in acetic acid 3% for which ICP-MS were ICP-OES significantly different. Results for Ba and Sb were obtained with less than 6 results and were therefore considered not reliable.

The results of the participating laboratories can be used to assess the uncertainty obtained during in-house validation studies. An essential condition to enable the use of circuit data is the requirement of z-score < 2 by the laboratories. This study can be also used to evaluate the performance of various instrumental methods for the quantification of elements migrated from ceramics (under and beyond the scope of the ceramics Directive 84/500/EEC) and from plastics (under he scope of Regulation (EU) 10/2011). The relative repeatability and reproducibility standard deviations can be used to contribute to the development of updated or expanded international standards under ISO or CEN.

Table 15. Table Relative Repeatability standard deviation [%]

Sample	Element	All results DIN 38402 A45	ICP-MS DIN 38402 A45	ICP-MS ISO 5725-5	ICP-OES DIN 38402 A45	ICP-OES ISO 5725-5	AAS DIN 38402 A45	AAS ISO 5725-5
PL1	Ba	1.03	1.17	1.46	0.75	0.78	2.48	9.23
	Co	1.66	1.72	1.71	1.47	1.37	1.38	1.63
	Cu	0.97	1.00	1.18	0.95	0.85	1.00	1.32
	Fe	1.42	1.40	1.76	0.77	0.95	2.21	2.64
	Mn	1.08	1.40	1.50	0.58	0.73	1.47	2.59
	Zn	1.03	1.03	1.22	1.37	1.22	0.81	0.83
	Li	1.43	1.62	1.94	1.31	1.23	1.01	1.14
	Sb	1.80	1.53	1.65	2.21	2.13	-	-
C2	Ba	0.99	1.02	1.11	0.90	0.68	-	-
	Co	1.47	1.65	1.72	1.25	1.01	3.14	2.6
	Mn	1.01	1.06	1.39	0.60	0.74	1.46	1.43
	Pb	2.38	1.91	2.13	4.45	3.23	2.54	2.59
	Cd	1.65	1.62	1.77	1.37	0.91	1.94	1.98
	Ni	1.38	1.40	1.79	1.16	0.99	2.52	2.72
	Al	1.68	1.90	2.37	1.32	1.44	-	-

Table 16. Relative Reproducibility standard deviation [%]

Sample	Element	All results DIN 38402 A45	ICP-MS DIN 38402 A45	ICP-MS ISO 5725-5	ICP-OES DIN 38402 A45	ICP-OES ISO 5725-5	AAS DIN 38402 A45	AAS ISO 5725-5
PL1	Ba	6.40	5.41	4.60	6.59	10.25	31.01	18.35
	Co	7.29	6.36	5.61	5.10	5.35	18.79	18.97
	Cu	5.52	4.83	4.79	8.26	8.45	6.64	6.28
	Fe	6.15	6.29	8.75	4.52	4.09	8.56	8.67
	Mn	5.49	4.46	4.95	5.48	4.93	10.36	8.22
	Zn	6.11	4.28	4.50	8.38	7.01	8.24	7.37
	Li	6.96	5.63	5.33	8.50	8.37	7.53	3.87
	Sb	15.68	11.53	11.38	13.07	19.22	-	-
C2	Ba	5.87	5.37	4.86	10.14	17.83	-	-
	Co	4.61	4.07	3.62	5.82	6.27	10.14	9.23
	Mn	5.42	4.93	4.44	4.76	5.23	9.80	10.64
	Pb	12.66	10.97	10.32	9.99	13.62	16.04	15.51
	Cd	8.54	5.53	4.89	19.16	19.20	9.04	8.03
	Ni	5.62	4.95	5.17	6.64	7.26	8.69	7.81
	Al	10.02	10.71	9.06	14.06	13.98	-	-

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

Official Controls Laboratories (OCLs)

Germany	Chemisches und Veterinäruntersuchungsamt Münsterland, Münster
Germany	Bayerisches Landesamt für Gesundheit und Lebensmittelsicherheit, Erlangen
Germany	Chemisches und Veterinäruntersuchungsamt Stuttgart, Fellbach
Germany	Zentrales Institut des Sanitätsdienstes der Bundeswehr KOBLENZ, Laborabteilung III, Lebensmittelchemie und Ökochemie, Koblenz
Germany	Chemisches und Veterinäruntersuchungsamt Rheinland (AöR), Leverkusen
Germany	Niedersächsisches Amt für Verbraucherschutz und Lebensmittelsicherheit (LAVES), Institut für Bedarfsgegenstände, Lüneburg
Germany	LANDESUNTERSUCHUNGSAMT RLP, Koblenz
Germany	Landesamt für Verbraucherschutz (LAV) Abt. B3B, Saarbrücken
Germany	Landesbetrieb Hessisches Landeslabor, Wiesbaden
Germany	Landeshauptstadt Düsseldorf Amt für Verbraucherschutz Chemische und Lebensmitteluntersuchung, Düsseldorf
Italy	IZS Lombardia Emilia Romagna- reparto chimico degli alimenti Bologna, Bologna
Italy	Laboratorio di Sanità Pubblica Area Vasta Toscana Centro - Azienda Sanitaria di Firenze, Firenze
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	ARPA Umbria, Terni
Italy	ASL Varese, Varese
Italy	Stazione Sperimentale del Vetro SSV
Spain	Laboratorio de Salud Pública de Alicante, Alicante
Spain	Centro Analítico de Inspección y control de Calidad de Comercio Exterior, Madrid
Spain	Laboratorio de Salud Pública de Burgos (Junta de Castilla y León), Paseo Burgos
Spain	CNTA, San Adrián - Navarra
Portugal	Centro de Formação Profissional para a Indústria Caldas da Rainha
Belgium	FAVV-lab FLVGG, Braemkasteelstraat 59 9050 Gentbrugge, Belgium

10. References

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

[2] ISO 13528:2015(E). Statistical Methods for Use in Proficiency Testing by Inter-laboratory Comparisons

[3] ISO 5725-5:1998(E). Accuracy (trueness and precision) of measurements methods and results

[4] DIN 38402 A45 Ringversuche zur externen Qualitätskontrolle von Laboratorien.

[5] 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

[6] 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

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

Annex 1a. Results received from participants for Sb and Ba in sample PL1

SAMPLE PL1												
	Lab code	M1 [µg/Kg]	M2 [µg/Kg]	M3 [µg/Kg]	M4 [µg/Kg]	Anal. Tech.		M1 [µg/Kg]	M2 [µg/Kg]	M3 [µg/Kg]	M4 [µg/Kg]	Anal. Tech.
ANTIMONY	LC0003	57.26	57.14	57.7	57.45	-	BARIUM	607.49	618.49	612.49	617.49	-
	LC0004							634.33	631.83	633.83	622.83	ICP-OES
	LC0005							687	688	685	687	ICP-OES
	LC0006	21.14	20.37	22.38	20.15	GF-AAS		929	917	918	936	ICP-OES
	LC0007											
	LC0008	39	39	39	38	ICP-MS		735	713	709	733	ICP-MS
	LC0010	35	36	36	35	ICP-MS		720	730	740	745	ICP-MS
	LC0011							620	622	618	630	GF-AAS
	LC0013											
	LC0016							734	734	706	732	ICP-OES
	LC0017	31.1	31.6	32.9	33.5	ICP-MS		642.2	657.3	666.3	672.5	ICP-MS
	LC0018	34.18	34.23	34.54	34.26	ICP-MS		680.2	681.9	675.3	679	ICP-MS
	LC0020	38.2	38.1	38.7	39.1	ICP-MS		673	667	666	682	ICP-MS
	LC0022							693	689	685	689	ICP-OES
	LC0024	44.7				ICP-OES		673				ICP-OES
	LC0025	45.2	46.5	36	36.9	ICP-MS		674	680	678	679	ICP-OES
	LC0028							717	713	718	701	ICP-MS
	LC0029	47.23	47.37	47.17	47.42	ICP-MS		851.9	865.08	884.72	888.46	ICP-MS
	LC0031	35.46	35.25	35.46	35.47	ICP-MS		707.47	711.69	711.74	708.99	ICP-MS
	LC0032	39	38	39	40	ICP-OES		708	697	701	699	ICP-OES
	LC0033							678	656	678	656	ICP-MS
	LC0034											
	LC0037							682.3	675.1	685	680.2	ICP-OES
	LC0038											
	LC0039	37	35.9	35.7	37.2	ICP-MS		704	699	677	708	ICP-MS
	LC0040	37.4	36.5	36.6	36.5	ICP-MS		676	683	658	661	ICP-MS
	LC0041	34	35	35	35	ICP-MS		531	531	530	531	ICP-MS
	LC0042											
	LC0043	31.9	28.6	30.9	28.3	ICP-MS		698	712	703	708	ICP-MS
	LC0044	32.5	31.9	32.4	31.9	ICP-MS		760	765	770	757	ICP-MS
	LC0046	35.1	35.4	35.8	36.2	-		710.3	704.9	711.5	690.4	-
	LC0049							397.58	538.42	504.42	552.98	GF-AAS
	LC0050	34	33	34	34	ICP-MS		690	691	698	703	ICP-MS
	LC0055	34.8	35.1	34.9	35.5	ICP-MS		692	696	690	702	ICP-MS
	LC0056	31.5	31	30.9	30.9	ICP-MS		674	662	670	675	ICP-MS
	LC0061	23.35	23.81	36.39	30.59	ICP-MS		475.06	499.34	609.42	613.14	ICP-MS
	LC0062	34.53	33.21	32.26	33.3	ICP-MS		710	700	700	700	ICP-OES
	LC0063	36.1	30.5	29.8	29.8	ICP-MS		650	700	650	650	ICP-MS
	LC0064	53	53	52.7	51.9	ICP-MS		708	702.8	704.4	709.5	ICP-MS
	LC0065	33.7	35.1	35.5	35.2	ICP-MS		686	705	710	688	ICP-MS
	LC0067	33	33	32.9	32.9	ICP-MS		669.2	677.3	671.4	675.3	ICP-MS
	LC0068	35.64	35.49	35.42	35.48	ICP-MS		689.67	687.17	688.1	683.45	ICP-MS
	LC0089											
	LC0097											
	LC0101	29.3	29	29.1	29.1	ICP-MS		731	738	734	725	ICP-MS
	LC0106							684	700			ICP-MS
	LC0113	59.5	60.6	58.3	59.7	ICP-OES		957	960	951	956	ICP-OES
	LC0114											
	LC0115	30.8	30.5	30.3	30	ICP-MS		867.7	875.4	862.7	862.2	ICP-OES
	LC0116											
	LC0117							696.25	731.25			ICP-MS
	LC0118	39	40	38	39	ICP-OES		730	750	740	740	ICP-OES
	LC0119	38	42	42		ICP-OES		676	667	683	679	ICP-OES
	LC0120											

Annex 1b. Results received from participants for Co and Cu in sample PL1

	SAMPLE PL1											
	Lab code	M1 [µg/Kg]	M2 [µg/Kg]	M3 [µg/Kg]	M4 [µg/Kg]	Anal. Tech.		M1 [µg/Kg]	M2 [µg/Kg]	M3 [µg/Kg]	M4 [µg/Kg]	Anal. Tech.
COBALT	LC0003	52.47	53.62	54.2	53.93	-	COPPER	3050	3050	3030	3050	-
	LC0004	50.52	50.76	50.75	50.72	ICP-OES		3095	3111	3096	3055	ICP-OES
	LC0005	51.00	50	50	51	ICP-OES		3289	3287	3288	3286	ICP-OES
	LC0006	43.62	43.97	43.65	43.3	GF-AAS		3678.1	3309.7	3376.9	3406	GF-AAS
	LC0007	43.6	43.8	43.1	44.7	GF-AAS		3045	3040	3016	3005	F-AAS
	LC0008	47	47	48	47	ICP-MS		3181	3115	3056	3137	ICP-MS
	LC0010	53	51	53	51	ICP-MS		3039	3046	2950	2963	ICP-MS
	LC0011	52.5	46.8	59.5	58.2	GF-AAS		3230	3200	2910	3000	F-AAS
	LC0013	49.8	48.8	49.1	48.4	GF-AAS		3132	3127	3108	3124	F-AAS
	LC0016							2950	2910	2910	2960	ICP-OES
	LC0017	48.6	49.7	52.2	52.4	ICP-MS		2958.2	2976.7	3133.1	3315.6	ICP-MS
	LC0018	46.24	46.39	46.76	46.25	ICP-MS		2925	2924	2937	2927	ICP-MS
	LC0020	48.7	49.7	51.6	50.7	ICP-MS		2720	2940	2910	2920	ICP-MS
	LC0022	49.9	50.4	50.5	50.1	ICP-OES		3208	3198	3170	3178	ICP-OES
	LC0024	54.7				ICP-OES		3928				ICP-OES
	LC0025	49.8	51.5	51.1	51.1	ICP-OES		3120	3070	3040	3070	ICP-OES
	LC0028	55	55	54	53	ICP-MS		3156	3195	3153	3107	ICP-MS
	LC0029	70.01	70.07	70.44	69.19	ICP-MS		3770.94	3829.94	3825.64	3837.37	ICP-MS
	LC0031	50.72	50.5	50.38	50.78	ICP-MS		3058.39	3043.06	3056.5	3045.57	ICP-MS
	LC0032	51	53	52	55	ICP-OES		3106	3066	3073	3090	ICP-OES
	LC0033	53.5	50.5	49.2	52.7	ICP-MS		2935	2913	2935	2923	F-AAS
	LC0034											
	LC0037	46.8	48.2	49.3	48.9	ICP-OES+F-AAS		2920	2980	2950	2960	F-AAS
	LC0038	70.9	65.8	69.8	65.6	GF-AAF						
	LC0039	52.4	50.3	48.7	51.3	ICP-MS		2980	3050	3000	3000	ICP-MS
	LC0040	50.6	51.5	51.7	51.1	ICP-MS		3012	3063	2988	3003	ICP-MS
	LC0041	50	51	53	51	ICP-MS		1417	1476	1517	1494	ICP-MS
	LC0042							3104	3125.2	3125.1	3135.6	ICP-MS
	LC0043	49.8	49.1	47.8	47.4	ICP-MS		3050	3014	3019	3029	ICP-MS
	LC0044	48.2	48.5	49.4	47.1	ICP-MS		3750	3780	3800	3700	ICP-MS
	LC0046	52.8	52.6	52.8	51	-		3167.6	3200.2	3220.1	3042.4	-
	LC0049							4475	4625	3675	3825	F-AAS
	LC0050	46	47	47	47	ICP-MS		3129	3092	3121	3133	ICP-MS
	LC0055	52.5	52	52.2	52.9	ICP-MS		3055	3043	3042	3085	ICP-MS
	LC0056	51.1	50.7	51	52.2	ICP-MS		3080	3100	3110	3110	ICPMS+F-AAS
	LC0061	36.24	36	40.37	40.47	ICP-MS		2135.9	2164.69	2751.03	2802.64	ICP-MS
	LC0062	53.08	54.38	54.37	52.96	ICP-MS		3050.57	3032.35	3057.54	3046.55	ICP-MS
	LC0063	49.5	51.6	51	47.6	ICP-MS		3000	3250	3050	3050	ICP-MS
	LC0064	55	55	54.8	55	ICP-MS		3658	3706	3708	3730	ICP-MS
	LC0065	52.7	53.6	52.8	53.6	ICP-MS		3170	3070	3150	3110	ICP-MS
	LC0067	49.2	49.52	49.36	49.53	ICP-MS		3305	3320.5	3346.5	3296.5	ICP-MS
	LC0068	50.94	50.38	50.32	50.54	ICP-MS		3064.9	3056.2	3075.2	3079.1	ICP-MS
	LC0089	47	47.67	47.65	49.37	GF-AAS		3115.6	3138.6	3125.6	3157.6	GF-AAS
	LC0097	33	32.9	32.9	32.9	GF-AAS		2932.2	2799.5	2988	3056.3	F-AAS
	LC0101	52.5	51.5	52.5	52.5	ICP-MS		2993	2984	3011	2966	ICP-MS
	LC0106	51.6	51.2			ICP-MS		3049	3030			ICP-MS
	LC0113	81.6	81.5	83.6	82.1	ICP-OES		3420	3260	3400	3360	ICP-OES
	LC0114											
	LC0115	49.8	49.5	49.2	49.6	ICP-MS		3579.5	3593.4	3578.2	3598.2	ICP-OES
	LC0116							2657.75	2685.02	2723.96	2770.75	ICP-OES
	LC0117	135.9	137.38			ICP-MS		2797.5	2895			ICP-MS
	LC0118	51	54	53	52	ICP-OES		3130	3070	3060	3160	ICP-OES
	LC0119	49	49	49	49	ICP-OES		2992	2936	2986	2979	ICP-OES
	LC0120	49.01	49.34			ICP-OES		2978	3007			ICP-OES

Annex 1c. Results received from participants for Fe and Li in sample PL1

					SAMPLE PL1							
	Lab code	M1 [µg/Kg]	M2 [µg/Kg]	M3 [µg/Kg]	M4 [µg/Kg]	Anal. Tech.		M1 [µg/Kg]	M2 [µg/Kg]	M3 [µg/Kg]	M4 [µg/Kg]	Anal. Tech.
	LC0003	20285.17	20255.17	19925.17	20315.17	-		427	427	429	431	-
	LC0004	20030	19870	19890	19590	ICP-OES		574.4	544	552.5	541.7	ICP-OES
	LC0005	19970	19968	19969	19968	ICP-OES		521	523	521	522	ICP-OES
	LC0006	17757	14593.9	17271.7	17552.6	GF-AAS		500	496	497	497	ICP-OES
	LC0007	19800	18670	18590	19570	F-AAS						
	LC0008	19994	18758	19179	19455	ICP-MS		450	424	424	431	ICP-MS
	LC0010	18150	18660	19240	19180	ICP-MS		485	494	500	529	ICP-MS
	LC0011	20200	18800	19100	20000	F-AAS		503	510	505	506	F-AAS
	LC0013	22807	23121	22986	22268	F-AAS		474	471	478	470	F-AAS
	LC0016	20680	20620	20600	21160	ICP-OES		510	510	480	500	ICP-OES
	LC0017	19430.7	19658.1	20063.7	20079.4	ICP-MS		473.3	477.9	502.9	523	ICP-MS
	LC0018	19457	19401	19423	19450	ICP-MS		486.6	486.1	487.5	485.8	ICP-MS
	LC0020	16940	17780	17870	17970	ICP-MS		478	488	476	477	ICP-MS
	LC0022	19830	19550	19810	19710	ICP-OES						
	LC0024	20430				ICP-OES		511.4				ICP-OES
	LC0025	18900	15200	17200	17400	ICP-OES		480	472	459	473	ICP-OES
	LC0028	20973	20633	20740	20404	ICP-MS		487	489	522	496	ICP-MS
	LC0029	20068.2	19645.8	20365.7	20662.4	ICP-MS		668.47	673.87	687.82	680.44	ICP-MS
	LC0031	19857.31	19816.61	19892.39	19889.72	ICP-MS		520.56	524.04	520.21	518.79	ICP-MS
	LC0032	19940	20050	20140	20310	ICP-OES		483	480	483	490	ICP-OES
	LC0033	20250	20340	20460	20380	F-AAS						
	LC0034											
	LC0037	16800	17300	17000	17400	F-AAS		462.5	459	468	460	ICP-OES
	LC0038											
	LC0039	15800	16900	17600	17900	ICP-MS		502	509	501	498	ICP-MS
	LC0040	18650	18860	18360	18680	ICP-MS		505	507	496	487	ICP-MS
IRON	LC0041	8152	8094	8237	8465	ICP-MS	LITHIUM	490	505	507	526	ICP-MS
	LC0042	2116.7	2092.3	2152	2146.4	ICP-MS						
	LC0043	19713	19476	19442	19564	ICP-MS		482	470	451	534	ICP-MS
	LC0044	19200	19100	19300	19000	ICP-MS		497	502	508	514	ICP-MS
	LC0046	19399	19291	19405	20542	-		568	560	574.8	569.1	-
	LC0049	23426	18287	19243	18506	GF-AAS		476.32	487	528.92	484.4	F-AAS
	LC0050	20433	20554	50556	20805	ICP-MS		481	488	488	490	ICP-MS
	LC0055	20153	20154	20036	20313	ICP-MS		535	532	527	527	ICP-MS
	LC0056	20100	19600	19600	20000	ICP-MS+F-AAS						
	LC0061	14064.12	14402.28	16259.11	16279.9	ICP-MS						
	LC0062	21340	21350	21370	21330	ICP-OES		546.45	554.76	559.19	550.59	ICP-MS
	LC0063	19225	19515	19570	20065	ICP-MS		577	501	486	493	ICP-MS
	LC0064	19100	18800	19300	19500	F-AAS		561.7	547.7	548.4	548.4	ICP-MS
	LC0065	20600	22800	21300	20600	ICP-MS		488	488	485	484	ICP-MS
	LC0067	19661.1	19661.1	19661.1	19661.1	ICP-MS						
	LC0068	19658.2	19885.1	19150.7	19895.6	ICP-MS		489.28	487.41	490.76	489.58	ICP-MS
	LC0089											
	LC0097	19488.1	20192.8	20272	19977.7	F-AAS						
	LC0101	20271	20299	20520	20148	ICP-MS		480	489	481	489	ICP-MS
	LC0106	19472	19672			ICP-MS		519.7	559.3			ICP-MS
	LC0113	20500	20900	20800	20600	ICP-OES		675	666	661	665	ICP-OES
	LC0114											
	LC0115	21660.8	21719.3	21600	21698.3	ICP-OES		642	637.2	643.9	665.4	ICP-OES
	LC0116							475.2	475.3	477.3	472.3	ICP-OES
	LC0117	12706.25	14776.25			ICP-MS						
	LC0118	22700	20800	20600	20500	ICP-OES		510	500	510	500	ICP-OES
	LC0119	20400	19850	19880	20350	ICP-OES		467	466	477	477	ICP-OES
	LC0120											

Annex 1d. Results received from participants for Mn and Zn in sample PL1

SAMPLE PL1												
	Lab code	M1 [µg/Kg]	M2 [µg/Kg]	M3 [µg/Kg]	M4 [µg/Kg]	Anal. Tech.		M1 [µg/Kg]	M2 [µg/Kg]	M3 [µg/Kg]	M4 [µg/Kg]	Anal. Tech.
	LC0003	501	500	494	503	-		20099.3	20149.3	20179.3	19899.3	-
	LC0004	468.63	468.73	469.33	470.93	ICP-OES		18600	18190	17840	17290	ICP-OES
	LC0005	498	499	497	498	ICP-OES		17231	17230	17234	17233	ICP-OES
	LC0006	518.84	540.31	461.31	537.08	GF-AAS		29310	29410	29320	30080	ICP-OES
	LC0007	495	488	493	498	F-AAS		19750	19500	20104	19286	F-AAS
	LC0008	476	454	458	469	ICP-MS		21421	20515	20555	20980	ICP-MS
	LC0010	490	496	479	500	ICP-MS		19994	20160	19650	20100	ICP-MS
	LC0011	520	451	536	508	GF-AAS		20200	18900	20700	18900	F-AAS
	LC0013	508	513	501	512	F-AAS		20417	20327	20808	20357	F-AAS
	LC0016	530	520	520	520	ICP-OES		20050	20050	19730	21520	ICP-OES
	LC0017	451	476.3	497	506.6	ICP-MS		19287.4	19730.8	19804	19916.6	ICP-MS
	LC0018	488.3	487.4	488.8	487.4	ICP-MS		19353	19357	19336	19346	ICP-MS
	LC0020	493	506	528	532	ICP-MS		19360	19700	19570	19870	ICP-MS
	LC0022	504	499	498	502	ICP-OES		19555	19605	19565	19475	ICP-OES
	LC0024	522.9				ICP-OES		19300				ICP-OES
	LC0025	473	482	489	480	ICP-OES		20300	20800	20800	20700	ICP-OES
	LC0028	513	519	520	508	ICP-MS		20420	20655	20656	20290	ICP-MS
	LC0029	717.93	703.49	716.16	704.89	ICP-MS		23264.15	24046.01	24064.28	23195.14	ICP-MS
	LC0031	502.44	502.67	501.75	503.11	ICP-MS		20204.28	20164.4	20142.11	20166.11	ICP-MS
	LC0032	515	516	528	524	ICP-OES		20080	20050	20420	20390	ICP-OES
	LC0033	508.4	507.6	506.6	508	GF-AAS		20500	20460	20440	20480	F-AAS
	LC0034											
	LC0037	549	553.3	546.2	548.8	F-AAS		17300	17800	17600	17600	F-AAS
	LC0038											
	LC0039	508	516	510	512	ICP-MS		20700	22700	20400	21000	ICP-MS
	LC0040	500	495	485	489	ICP-MS		20080	20520	19510	19640	ICP-MS
	LC0041	488	474	495	494	ICP-MS		15232	15174	15148	15143	ICP-MS
MANGANESE	LC0042	500.4	508.4	506.9	505	ICP-MS	ZINC					
	LC0043	464	485	480	496	ICP-MS		19872	19671	19450	19463	ICP-MS
	LC0044	455	457	457	439	ICP-MS		19900	20000	20100	19700	ICP-MS
	LC0046	517	513.7	516.4	517.6	-		23032	23022	25510	25600	-
	LC0049	623.2	629.6	499.7	607.8	GF-AAS		21330	21160	21160	21160	F-AAS
	LC0050	499	498	501	506	ICP-MS		19574	19397	19558	19730	ICP-MS
	LC0055	518	508	510	514	ICP-MS		20140	20204	19974	19680	ICP-MS
	LC0056	494	487	490	483	ICP-MS+F-AAS		20000	19500	19900	19300	ICP-MS+F-AAS
	LC0061	350.29	355.62	415	422.04	ICP-MS		12149.49	11227.65	18278.16	18468.3	ICP-MS
	LC0062	495	492	495	494	ICP-OES		18360	18340	18320	18330	ICP-OES
	LC0063	483	430	420	455	ICP-MS		20450	22300	20650	20850	ICP-MS
	LC0064	582.1	586.9	584.9	590.1	ICP-MS		17600	17600	17700	17500	F-AAS
	LC0065	540	511	510	501	ICP-MS		20400	20400	20700	20300	ICP-MS
	LC0067	507.7	504	508.1	504	ICP-MS		22886	22436	22736	22526	ICP-MS
	LC0068	495.36	497.12	495.05	498.59	ICP-MS		19747.8	19866.8	19803	19941.9	ICP-MS
	LC0089	397	371.5	380.5	374	GF-AAS		18000	18000	18000	18000	GF-AAS
	LC0097	445.4	479.8	454.2	478.2	GF-AAS		19329.7	19443.8	19413.5	19357.2	F-AAS
	LC0101	505	496	501	503	ICP-MS		19395	19338	19447	19103	ICP-MS
	LC0106	501.2	504.9			ICP-MS		20047	20027			ICP-MS
	LC0113	690	691	682	685	ICP-OES		20200	19800	19600	19750	ICP-OES
	LC0114											
	LC0115	480.6	477.6	478.7	475.2	ICP-OES		21630	21332	21090	21256	ICP-OES
	LC0116	513.2	513.63	514.45	513.26	ICP-OES		20761	20781	20432	20166	ICP-OES
	LC0117	457.5	465			ICP-MS		22383.75	23443.75			ICP-MS
	LC0118	480	490	490	490	ICP-OES		19300	18800	18900	18800	ICP-OES
	LC0119	473	469	478	478	ICP-OES		19220	19280	19540	19570	ICP-OES
	LC0120											

Annex 1e. Results received from participants for Al and As in sample C2

SAMPLE C2										
	ALUMINIUM					ARSENIC				
Lab code	M1 [µg/Kg]	M2 [µg/Kg]	M3 [µg/Kg]	M4 [µg/Kg]	Anal. Tech.	M1 [µg/Kg]	M2 [µg/Kg]	M3 [µg/Kg]	M4 [µg/Kg]	Anal. Tech.
LC0003	720.72	714.98	718.46	720.32	-	28.46	28.88	28.96	28.82	-
LC0004	752.55	750.05	734.45	724.25	ICP-OES	10.87	10.81	10.78	11.5	ICP-OES
LC0005	900.00	890	899	900	ICP-OES	20	19	20	20	ICP-MS
LC0006	696.40	718.96	657.36	708.34	GF-AAS	10.8	10.69	9.78	9.45	GF-AAS
LC0007										
LC0008	761	771	757	810	ICP-MS	10	11	10	11	ICP-MS
LC0010	724	800	895	721	ICP-MS	11	11.2	11.3	11.1	ICP-MS
LC0011										
LC0013										
LC0016										
LC0017	666.8	675.5	681.1	706.7	ICP-MS	7.9	10.5	11	12.8	ICP-MS
LC0018	765.6	766.9	772.4	776.3	ICP-MS	14.35	14.27	13.98	13.97	ICP-MS
LC0020	730	720	716	738	ICP-MS	9.8	13.4	13	14.3	ICP-MS
LC0022	688	688	690	707	ICP-OES	11.4	10.3	10	9.6	ICP-OES
LC0024	981.1				ICP-OES	127.5				ICP-OES
LC0025	637	640	622	619	ICP-OES	10.1	14.1	11.5	9.5	ICP-OES
LC0028	770	709	705.00	716	ICP-MS	16	13	16	13	ICP-MS
LC0029	768.43	764.26	773.53	760.74	ICP-MS	38.51	40.23	40.59	39.41	ICP-MS
LC0031	777.72	765.48	765.43	779.72	ICP-MS	12.65	12.47	12.45	12.38	ICP-MS
LC0032	759	739	750	748	ICP-OES	13	14	13	13	ICP-OES
LC0033	699	701	706		ICP-MS	7.9	8.36	8.2		ICP-MS
LC0034										
LC0037	162.3	163.5	161.2	160.9	ICP-OES	17.9	17.7	18.4	18.1	GF-AAS
LC0038						12.1	12.7	15.4	13.9	GF-AAS
LC0039	760	752	766	767	ICP-MS	19	16.4	17.1	17.2	ICP-MS
LC0040	664	695	646	644	ICP-MS	6.7	3.7	3.9	5.7	ICP-MS
LC0041	506	518	475	526	ICP-MS	13	15	15	18	ICP-MS
LC0042	661.20	657.90	660.40	645.70	ICP-MS	25.1	24.4	24.1	23.7	ICP-MS
LC0043	725	690	706	655	ICP-MS	7	6.7	6.9	6.9	ICP-MS
LC0044	848	848	817	809	ICP-MS	16.1	15.5	15.5	15.7	ICP-MS
LC0046	719.7	719.1	725	720.9	-	11.8	12.25	11.4	12.2	-
LC0049										
LC0050	702	720	707	715	ICP-MS	11	11	11	12	ICP-MS
LC0055	707	699	706	694	ICP-MS	14.1	13.8	13.7	13.6	ICP-MS
LC0056	739	747			ICP-MS	15	14.3	13	15.6	ICP-MS
LC0061	462.53	418.39	610.66	620.24	ICP-MS	10.48	9.39	12.68	13.37	ICP-MS
LC0062	722	713	716	711	ICP-MS	12.01	11.86	11.8	11.56	ICP-MS
LC0063	743	677	666	704	ICP-MS	16.8	16.3	16.4	16.5	ICP-MS
LC0064	761.6	774.5	786.2	778.9	ICP-MS	9.9	9.8	9.6	9.6	ICP-MS
LC0065	757	745	730	777	ICP-MS	11.5	15.9	14.3	14.4	ICP-MS
LC0067	965.9	965.1	957.6	988.1	ICP-MS	47.02	47.23	47.35	48.57	ICP-MS
LC0068	833.77	812.44	812.25	827.06	ICP-MS	13.225	12.097	11.841	11.919	ICP-MS
LC0089										
LC0097										
LC0101	702	701	682	691	ICP-MS	8.5	9.1	8.2	8.1	ICP-MS
LC0106	716.6	837.5	757.50		ICP-MS	16.8	17.1			ICP-MS
LC0113	684	693	684	682	ICP-OES	11.95	35.1	34.8	34.5	ICP-OES
LC0114										
LC0115	677.4	677.4	670.5	712.6	ICP-OES	9.84	9.27	9.58	9.8	ICP-MS
LC0116	678.65	725.51	734.24	721.32	ICP-OES					
LC0117	439	463			ICP-MS	13	14			ICP-MS
LC0118	680	670	690	670	ICP-OES	11	11	11	11	ICP-OES
LC0119	786	774	760	788	ICP-OES	16.3	17.1	18	15.8	GF-AAS
LC0120						13.49	14.04			ICP-OES

Annex 1f. Results received from participants for Ba and Cd in sample C2

SAMPLE C2												
	Lab code	M1 [µg/Kg]	M2 [µg/Kg]	M3 [µg/Kg]	M4 [µg/Kg]	Anal. Tech.		M1 [µg/Kg]	M2 [µg/Kg]	M3 [µg/Kg]	M4 [µg/Kg]	Anal. Tech.
BARIUM	LC0003	786.78	780.58	773.39	780.65	-	CADMIUM	6.21	6.29	6.11	6.02	-
	LC0004	748.03	748.43	739	745.83	ICP-OES		4.37	4.27	4.29	4.3	ICP-OES
	LC0005	790.00	791	790	791	ICP-OES		4	4	4	4	ICP-OES
	LC0006	1111.0	1108	1112	1126	ICP-OES		4.28	4.58	4.34	4.34	GF-AAS
	LC0007							4.92	4.88	4.79	4.81	GF-AAS
	LC0008	806	792	801	803	ICP-MS		5.1	5.3	5.2	5.2	ICP-MS
	LC0010	815	808	810	807	ICP-MS		5.1	5.1	5	5	ICP-MS
	LC0011	760	770	775	769	GF-AAS		3.97	4.3	3.9	3.74	GF-AAS
	LC0013							5	5.1	5.1	5.2	GF-AAS
	LC0016							4.94	4.94	4.88	4.87	GF-AAS
	LC0017	729.8	736.9	765.7	769.3	ICP-MS		4.5	4.5	4.7	4.7	ICP-MS
	LC0018	783	789.4	782.5	787.7	ICP-MS		4.789	4.784	4.719	4.73	ICP-MS
	LC0020	764	757	756	747	ICP-MS		4.6	4.7	4.5	4.6	ICP-MS
	LC0022	806	809	801	798	ICP-OES		5.2	5.2	5.2	5.1	ICP-OES
	LC0024	757.7				ICP-OES		4.1				ICP-OES
	LC0025	772	777	780	782	ICP-OES		4.61	4.66	4.89	4.91	ICP-MS
	LC0028	754	755	766.00	759	ICP-MS		4.7	4.8	4.7	4.8	ICP-MS
	LC0029	1132.31	1153.9	1143.85	1135.57	ICP-MS		7.89	7.92	7.91	7.8	ICP-MS
	LC0031	825.88	820.75	821.02	820.76	ICP-MS		5	4.96	5.06	4.99	ICP-MS
	LC0032	821	814	817	805	ICP-OES		5.8	5.7	5.5	5.5	ICP-OES
	LC0033	756	756	764		ICP-MS		5.18	5.27	5.21	5.18	GF-AAS
	LC0034							4.78	4.78	4.71	4.8	GF-AAS
	LC0037	1020	1035	1058	1069	ICP-OES		4.4	4.2	4.3	4.5	GF-AAS
	LC0038							4.7	4.43	4.52	4.65	GF-AAS
	LC0039	809	782	789	787	ICP-MS		4.83	4.69	4.79	4.93	ICP-MS
	LC0040	769	794	748	751	ICP-MS		4.8	4.7	4.8	4.7	ICP-MS
	LC0041	431	450	447	460	ICP-MS		3.1	3.4	3.5	3.3	ICP-MS
	LC0042							5.9	5.8	5.7	5.7	ICP-MS
	LC0043	843	846	859	855	ICP-MS		5	5.1	5.1	5	ICP-MS
	LC0044	759	739	752	749	ICP-MS		4.78	4.25	4.75	4.53	ICP-MS
	LC0046	785.5	792.6	797	686.6	-		5.4	5.5	5.4	5.4	-
	LC0049											
	LC0050	803	811	796	807	ICP-MS		5	5	5	5	ICP-MS
	LC0055	804	801	806	808	ICP-MS		4.9	4.76	4.8	4.82	ICP-MS
	LC0056	791	790	796		ICP-MS+GF-AAS		4.74	4.87	4.87		ICP-MS
	LC0061	546.22	554.27	702.74	715.58	ICP-MS		3.46	3.33	4.52	4.51	ICP-MS
	LC0062	820	810	810	810	ICP-OES		5.08	5.13	5.17	5.13	ICP-MS
	LC0063	760	750	770	740	ICP-MS		4.9	4.8	5.2	4.9	ICP-MS
	LC0064	821.1	820.7	823.7	820.4	ICP-MS		5	5	5	5	ICP-MS
	LC0065	815	795	799	805	ICP-MS		5.4	4.4	5	5.4	ICP-MS
	LC0067	775.5	773.6	768.1	776.9	ICP-MS		5	4.9	4.9	5	ICP-MS
	LC0068	780.7	782.7	778	796	ICP-MS		4.91	4.74	4.72	4.64	ICP-MS
	LC0089							4.73	4.83	4.72	4.78	GF-AAS
	LC0097							5.15	4.98	4.98	5.16	GF-AAS
	LC0101	839	849	844	838	ICP-MS		4.86	4.9	4.87	4.86	ICP-MS
	LC0106	782	802	784.00		ICP-MS		5.1	5.1			ICP-MS
	LC0113	40.6	42.8	43.1	42.1	ICP-OES		5.3	5.3	5.2	5.3	ICP-OES
	LC0114											
	LC0115	1000.9	1036.6	1066	1030.3	ICP-OES		5	4.92	4.64	4.78	ICP-MS
	LC0116											
	LC0117	740	771			ICP-MS		5	5			ICP-MS
	LC0118	880	880	890	890	ICP-OES		6	6	6	7	ICP-OES
	LC0119	790	798	788		ICP-OES		5.2	5.2	5.1	5.1	GF-AAS
	LC0120							5.95	5.94			ICP-OES

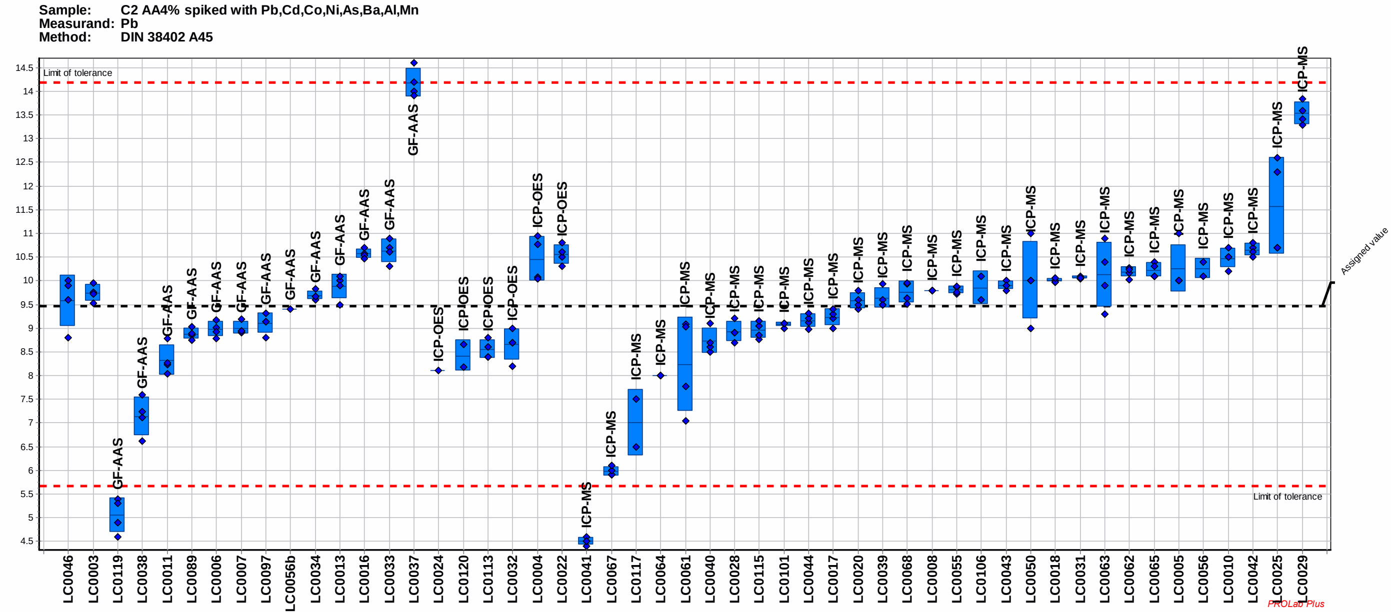
Annex 1g. Results received from participants for Mn and Ni in sample C2

SAMPLE C2										
	MANGANESE					NICKEL				
Lab code	M1 [µg/Kg]	M2 [µg/Kg]	M3 [µg/Kg]	M4 [µg/Kg]	Anal. Tech.	M1 [µg/Kg]	M2 [µg/Kg]	M3 [µg/Kg]	M4 [µg/Kg]	Anal. Tech.
LC0003	385.96	390.39	390.43	386.24	-	69.75	68.7	70.87	70.8	-
LC0004	370.47	367.87	365.67	367.07	ICP-OES	68.79	69.07	68.62	68.74	ICP-OES
LC0005	400.00	401	400	401	ICP-OES	69	70	70	68	ICP-OES
LC0006	456.88	468.66	453.54	464.19	GF-AAS	74.95	75.93	75.72	75.67	GF-AAS
LC0007						90.95	90.85	97.85	90.35	GF-AAS
LC0008	388	385	382	382	ICP-MS	68	68	68	68	ICP-MS
LC0010	394	400	391	393	ICP-MS	70	72	71	71	ICP-MS
LC0011	393	453	425	448	GF-AAS	68.1	73.2	73.2	70	GF-AAS
LC0013	393	393	395	402	F-AAS	71.3	72	73.8	73.6	GF-AAS
LC0016	388.20	386.60	383.20	389.20	GF-AAS	68.46	70.82	71.81	71.98	GF-AAS
LC0017	372.3	397.6	400.5	401.4	ICP-MS	69.4	71.7	72	72.2	ICP-MS
LC0018	394.4	391.8	394.4	395.8	ICP-MS	68.86	68.9	67.89	67.1	ICP-MS
LC0020	405	425	428	424	ICP-MS	69.8	70.4	69.5	71.6	ICP-MS
LC0022	416	409	405	407	ICP-OES	72.8	72.7	71.9	73.4	ICP-OES
LC0024	431.9				ICP-OES	83.98				ICP-OES
LC0025	385	387	391	387	ICP-OES	69.7	69.7	70.2	70.7	ICP-OES
LC0028	415	406	400	393	ICP-MS	72	71	70	69	ICP-MS
LC0029	649.96	654.08	656.59	651.23	ICP-MS	108.55	108.47	108.23	108.52	ICP-MS
LC0031	410.25	409.84	410.05	413.45	ICP-MS	72.32	72.89	72.34	72.73	ICP-MS
LC0032	404	401	401	396	ICP-OES	74	74	73	73	ICP-OES
LC0033	406	408	405	405	GF-AAS	70.3	71	72.4	72.6	GF-AAS
LC0034										
LC0037	442	442.3	440.5	450.1	F-AAS	112.5	113.1	114.4	114	ICP-OES
LC0038						78.6	80.6	79.8	79.5	GF-AAS
LC0039	382	371	379	379	ICP-MS	70.3	66.3	67.1	66	ICP-MS
LC0040	402	411	397	392	ICP-MS	71.5	69.6	67.8	67.1	ICP-MS
LC0041	381	386	381	396	ICP-MS	62	59	61	62	ICP-MS
LC0042	73.30	72.90	71.60	70.60	ICP-MS	64	63	65.5	65	ICP-MS
LC0043	413	414	412	410	ICP-MS	70.7	71.3	70.9	70.6	ICP-MS
LC0044	409	398	398	401	ICP-MS	63.9	64.8	67.4	65.9	ICP-MS
LC0046	408.2	408.4	407.9	505.3	-	80.8	72.5	72.5	75.1	-
LC0049	373.00	341.60	401.90	379.40	GFAAS					
LC0050	403	407	401	406	ICP-MS	71	72	70	71	ICP-MS
LC0055	412	410	410	415	ICP-MS	71	71.1	71.3	71.5	ICP-MS
LC0056	385	398	375	370	ICP-MS+F-AAS	70.8	69.1	70	70.5	ICP-MS
LC0061	287.84	276.48	366.13	361.05	ICP-MS	48.68	45.34	65.7	65.63	ICP-MS
LC0062	406	404	405	404	ICP-OES	73.38	76.8	75.97	73.31	ICP-MS
LC0063	387	363	349	364	ICP-MS	69.6	64.6	62.1	66.3	ICP-MS
LC0064	430.8	438.3	444.8	443.6	ICP-MS	71.3	71.8	72.3	71.4	ICP-MS
LC0065	406	409	411	404	ICP-MS	71.8	77.2	73.9	77.2	ICP-MS
LC0067	406.7	403.2	400.9	413.4	ICP-MS	74.1	73.4	72.6	74.7	ICP-MS
LC0068	394.34	395.98	395.16	397.36	ICP-MS	72.21	72.93	71.29	73.08	ICP-MS
LC0089	275.5	282	285	273	GF-AAS	64.57	68.76	65.03	67.45	GF-AAS
LC0097	381	385.6	380	391.9	GF-AAS	63.5	66.7	70.8	73.1	GF-AAS
LC0101	406	409	407	405	ICP-MS	72.4	72.3	72.1	72.3	ICP-MS
LC0106	395.6	397.7	395.50		ICP-MS	70.4	69.4			ICP-MS
LC0113	15.9	17	17.2	16.7	ICP-OES	71.9	71.9	71.4	71.5	ICP-OES
LC0114										
LC0115	397	393.5	407.6	399.1	ICP-OES	66.6	66	69.5	68.9	ICP-MS
LC0116	422.97	421.36	422.35	420.70	ICP-OES					
LC0117	377	387			ICP-MS	67	70			ICP-MS
LC0118	400	410	410	400	ICP-OES	70	70	80	80	ICP-OES
LC0119	407	394	398		ICP-OES	75	73	76	74	ICP-OES
LC0120										

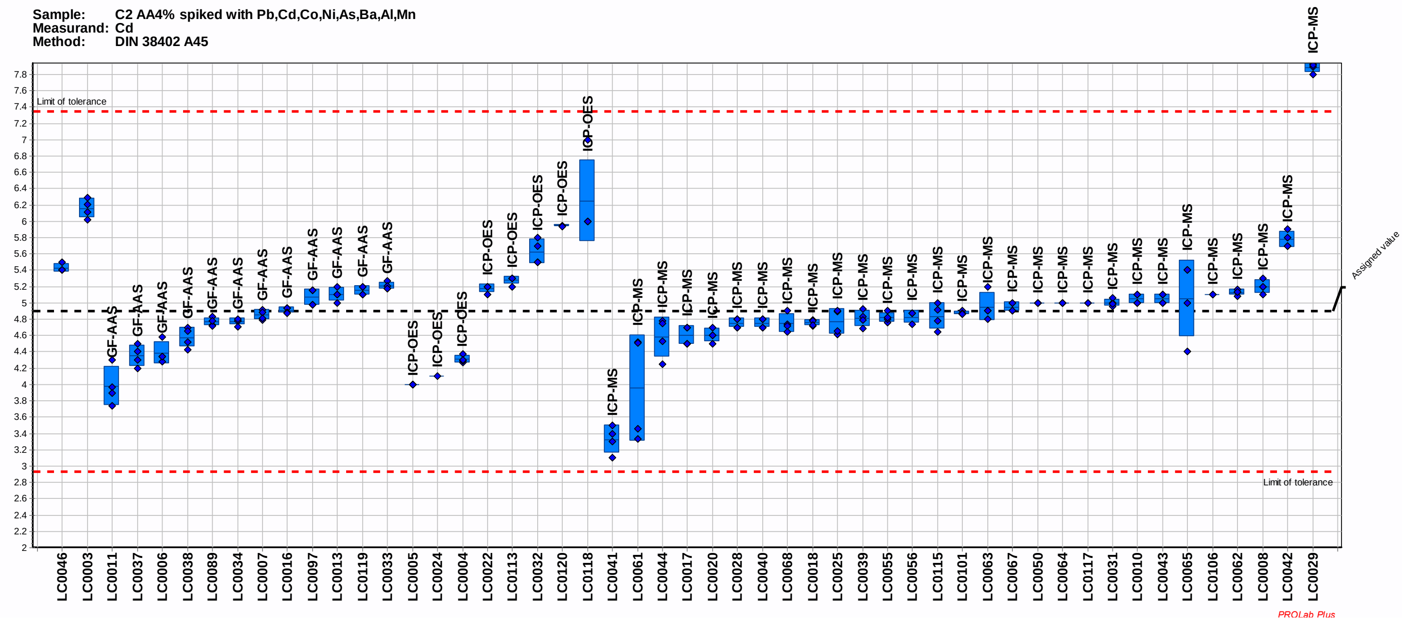
Annex 1h. Results received from participants for Co and Pb in sample C2

				SAMPLE C2								
	Lab code	**M1 [µg/Kg]**	**M2 [µg/Kg]**	**M3 [µg/Kg]**	**M4 [µg/Kg]**	Anal. Tech.		**M1 [µg/Kg]**	**M2 [µg/Kg]**	**M3 [µg/Kg]**	**M4 [µg/Kg]**	**Anal. Tech.**
	LC0003	53.43	53.21	53.84	52.69	-		9.95	9.75	9.72	9.53	-
	LC0004	50.20	50.01	50.31	50.34	ICP-OES		10.07	10.04	10.77	10.94	ICP-OES
	LC0005	50.00	51	51	49	ICP-OES		10	11	10	10	ICP-MS
	LC0006	45.72	45.66	45.86	45.96	GF-AAS		9.02	9.18	8.93	8.79	GF-AAS
	LC0007							8.95	8.91	9.19	8.95	GF-AAS
	LC0008	51	52	51	51	ICP-MS		9.8	9.8	9.8	9.8	ICP-MS
	LC0010	53	52	51	52	ICP-MS		10.5	10.7	10.5	10.2	ICP-MS
	LC0011	46.8	53.2	50.6	47.9	GF-AAS		8.24	8.03	8.26	8.78	GF-AAS
	LC0013	51.7	51.7	51.4	51.6	GF-AAS		10	10.1	9.5	9.9	GF-AAS
	LC0016							10.58	10.53	10.69	10.46	GF-AAS
	LC0017	49.2	50.6	51.4	52	ICP-MS		9	9.2	9.3	9.4	ICP-MS
	LC0018	50.48	50.41	49.81	50.03	ICP-MS		9.975	9.997	9.975	10.06	ICP-MS
	LC0020	50.8	49.8	50.9	51.6	ICP-MS		9.8	9.4	9.5	9.6	ICP-MS
	LC0022	54.5	54.5	54.1	54.9	ICP-OES		10.8	10.5	10.6	10.3	ICP-OES
	LC0024	56.96				ICP-OES		8.1				ICP-OES
	LC0025	49.5	50.8	50.7	50.7	ICP-OES		12.6	12.3	10.7	10.7	ICP-MS
	LC0028	53	52	51	51	ICP-MS		8.9	9.2	8.7	8.9	ICP-MS
	LC0029	80.86	79.74	80.46	79.14	ICP-MS		13.84	13.29	13.41	13.59	ICP-MS
	LC0031	52.59	52.49	52.8	52.8	ICP-MS		10.04	10.1	10.06	10.05	ICP-MS
	LC0032	55	55	54	54	ICP-OES		8.7	8.7	8.2	9	ICP-OES
	LC0033	49.8	50.3	50		ICP-MS		10.9	10.3	10.7	10.6	GF-AAS
	LC0034							9.6	9.67	9.66	9.82	GF-AAS
	LC0037	71.2	72.8	71	72.8	ICP-OES+F-AAS		14.2	14	14.6	13.9	GF-AAS
	LC0038	51.7	51.2	49.3	44.5	GF-AAS		7.6	7.11	6.61	7.23	GF-AAS
	LC0039	52.3	49.8	50.1	50	ICP-MS		9.94	9.49	9.59	9.5	ICP-MS
	LC0040	51.5	53	50.2	51.4	ICP-MS		8.5	8.6	9.1	8.7	ICP-MS
COBALT	LC0041	51	49	50	51	ICP-MS	LEAD	4.4	4.5	4.6	4.5	ICP-MS
	LC0042							10.7	10.6	10.8	10.5	ICP-MS
	LC0043	51.3	51.1	51.2	51.4	ICP-MS		9.8	10	9.9	9.9	ICP-MS
	LC0044	49	48	46.8	48.7	ICP-MS		8.97	9.12	9.21	9.31	ICP-MS
	LC0046	52.3	51.8	51.9	50.1	-		9.9	9.6	10	8.8	-
	LC0049											
	LC0050	48	48	47	48	ICP-MS		9	10	10	11	ICP-MS
	LC0055	52.4	52.9	52.3	52.6	ICP-MS		9.86	9.72	9.88	9.75	ICP-MS
	LC0056	52.9	50.6	51.2	51	ICP-MS		10.1	10.4	9.4		ICP-MS+GF-AAS
	LC0061	37.18	34.99	45.76	47.67	ICP-MS		7.77	7.05	9.08	9.03	ICP-MS
	LC0062	56.71	57.1	54.87	54.94	ICP-MS		10.27	10.16	10.24	10.03	ICP-MS
	LC0063	50.4	50.3	51.4	48	ICP-MS		9.3	10.4	9.9	10.9	ICP-MS
	LC0064	51.5	51.9	52.3	52	ICP-MS		8	8	8	8	ICP-MS
	LC0065	55.8	53.9	54	55.7	ICP-MS		10.4	10.1	10.1	10.3	ICP-MS
	LC0067	50.12	50.39	50.32	50.69	ICP-MS		5.9	5.9	6	6.1	ICP-MS
	LC0068	49.81	49.8	49.2	50.31	ICP-MS		9.63	9.93	9.51	9.96	ICP-MS
	LC0089							8.85	9.03	8.88	8.74	GF-AAS
	LC0097	46.7	46.8	45.2	45.5	GF-AAS		8.8	9.13	9.31	9.13	GF-AAS
	LC0101	52.8	53.3	53	52.4	ICP-MS		9.1	9	9.1	9.1	ICP-MS
	LC0106	51.2	50.4			ICP-MS		9.6	10.1			ICP-MS
	LC0113	51.5	52.1	51.4	51.5	ICP-OES		8.4	8.8	8.6	8.4	ICP-OES
	LC0114											
	LC0115	50.1	50.4	51.7	52.8	ICP-MS		9.05	8.76	9.16	8.86	ICP-MS
	LC0116											
	LC0117	52	57			ICP-MS		6.5	7.5			ICP-MS
	LC0118	56	56	56	56	ICP-OES						ICP-OES
	LC0119	54	52	52	51	ICP-OES		4.9	4.6	5.4	5.3	GF-AAS
	LC0120	50.91	50.28			ICP-OES		8.65	8.18			ICP-OES

Annex 2a. Summary graph for Pb analysed in sample C2 sorted by analytical technique applied



Annex 2b. Summary graph for Cd analysed in sample C2 sorted by analytical technique applied



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