Bundesinstitut für Risikobewertung

Inter-laboratory comparison exercise on the release of Aluminium and Cobalt from enamel cups into 3 % and 4 % acetic acid solutions by article filling

Report on the Inter-laboratory comparison exercise NRL-DE-FCM-01/2019 of the German National Reference Laboratory (NRL) for Food Contact Materials

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Table of contents

1 Summary

The German National Reference Laboratory (NRL) for Food Contact Materials has organized an Inter-laboratory comparison exercise (ILC) on the release of Aluminium (Al) and Cobalt (Co) from commercially available enamel cups into 3 % and 4 % acetic acid solutions by article filling (NRL-DE-FCM-01/2019). This ILC was open to National Reference Laboratories (NRLs), Official Control Laboratories (OCLs) and the European Union Reference Laboratory for Food Contact Materials (EURL-FCM).

The participating laboratories were asked to carry out release tests (1st, 2nd, and 3rd releases) by filling of the provided industry made enameled cups (volume 0.3L) with 3 % acetic acid for 2h at 70°C (according to ISO 4531:2018 [1]) and with 4 % acetic acid for 24h at 22°C (according to European Directive 84/500/EEC [2]) to determine the concentrations of Al and Co in the food simulants. Additionally, the concentrations of Al and Co should have been determined in provided solutions.

Early studies within the EURL-NRL network for Food Contact Materials indicated that the required contact temperature when performing migration or release tests by article filling is often reached only after an (unacceptably) long period of time or even not at all [3]. In the following ILCs performed by the EURL for FCM the performance of the labs has been significantly increased and most of the participants were able to keep the required temperature to a satisfying degree [4]. As the contact temperature affects results, it may also affect the decision about compliance or non-compliance of samples. Therefore, we again requested to monitor and report the temperature of the food simulants during the release at 70°C.

In total, fourteen laboratories from six Member States as well as the EURL have taken part in this ILC. Fifteen laboratories have reported results for Al and fourteen for Co.

Each participant received six enameled cups (3 cups for each simulant solution) and three already prepared release solutions. The homogeneity and stability of all test items were evaluated and the assigned values were derived as a robust average of the single results reported by the participants.

Laboratory results were rated using *z* and zeta (ζ) scores in accordance with ISO 13528:2015 [5]. Based on expert judgment, a relative standard deviation for proficiency assessment (σ_{pt}) was set to 20 % of the assigned value for first release, to 30 % for second and third releases, and to 15 % for analysis of solutions.

All of the participating laboratories performed satisfactory (according to the *z* score) in the analysis of both Al and Co for the solutions and for releases at 22°C. These results confirm that all of the participating laboratories are able to perform experiments for the determination of Al and Co release from enameled articles according to European Directive 84/500/EEC [2].

For the releases at 70°C a satisfactory performance was achieved by more than 79 % of the participating laboratories (according to the *z* score). These results demonstrate that most of the participants are able to perform experiments for the determination of Al and Co release from enameled articles according to ISO 4531:2018 [1]. No participant reported results with unsatisfactory z scores clearly confirming that comparable results can be achieved with a high reliability from the participating labs even after three release tests.

However further studies for the harmonization of release tests are essential to further lower the interlaboratory scatter of the results for the second and third release tests at 70°C.

2 Introduction

The inter-laboratory comparison (ILC) exercise on release of Aluminium and Cobalt from enamel cups into 3 % and 4 % acetic acid solutions by article filling was organized by the National Reference Laboratory (NRL) for Food Contact Materials (FCM) established within the German Federal Institute for Risk Assessment (BfR). Enamelled articles are currently not regulated on a European level. Historically lead and cadmium testing was performed uniformly for all silicate matrices (e.g. ceramics, glass, enamels) (DIN 51031:1986 [6], ISO 6486-1:1981 [7], ISO 7086-1:1982 [8]). Testing conditions and limits were the same as in European Directive 84/500/EEC (Ceramic Directive) [2]. In 1995 the European Committee for Standardization (CEN) decided to publish different standards for ceramic ware (EN 1388-1:1995 [9]) and silicate surfaces other than ceramic ware (EN 1388-2:1995 [10]), preserving the conditions laid down previously. Especially for enameled articles ISO developed ISO 4531-1:1998 [11] in closely following EN 1388-2:1995 [10]. For a decade, a revision of the Ceramics Directive has been announced, covering more elements than lead and cadmium and possibly extending its scope to other silicate matrices such as enamelware [12]. Since the Council of Europe published Resolution CM/Res(2013)9 [13] on metals and alloys used in food contact materials and articles and the accompanying technical guide in 2013, this guidance was often applied to enamelware. On the basis of these limits, ISO and CEN issued in 2018 a revision of the enamel specific standard (EN ISO 4531:2018 [1]) specifying adapted test conditions (e.g. 3 % acetic acid for acidic food and temperature conditions according to the intended use of the article). It is yet not clear, what conditions will be mandatory in the future; this ILC therefore implies two different experiments:

1) Release testing of commercial enamelled cups according to European Directive 84/500/EEC [2] (Ceramics)
2) Release testing of commercial enamelled cups according to ISO 4531:2018 [1]

This proficiency test was open to National Reference Laboratories (NRLs), Official Control Laboratories (OCLs), and the European Union Reference Laboratory for Food Contact Materials (EURL-FCM). The fifteen laboratories listed hereafter are kindly acknowledged for their participation in the ILC exercise.

Table 1: Participating laboratories

Organization	Country
European Commission, Joint Research Centre (JRC), Directorate F – Health, Consumer and Reference Materials, Food and Feed Compliance	
Health Board, Central Chemistry Laboratory	Estonia
Bayerisches Landesamt für Gesundheit und Lebensmittelsicherheit (LGL)	Germany
Chemisches und Veterinäruntersuchungsamt Ostwestfalen-Lippe (CVUA-OWL)	Germany
Chemisches und Veterinäruntersuchungsamt Stuttgart (CVUA Stuttgart)	Germany
Landesamt für Verbraucherschutz Saarland (LAV Saarland)	Germany
Landesamt für Verbraucherschutz Sachsen-Anhalt	Germany
Landesportal Schleswig-Holstein	Germany
Landesuntersuchungsanstalt für das Gesundheits- und Veterinärwesen (LUA) Sachsen	Germany
Thüringer Landesamt für Verbraucherschutz	Germany

Continuation Table 1: Participating laboratories

Organization	Country
German Federal Institute for Risk Assessment (BfR)	Germany
National public health surveillance laboratory (NPHSL)	Lithuania
Escola Superior de Biotecnologia, Universidade Católica Portuguesa	Portugal
National Laboratory of Health, Environment and Food	Slovenia
Centro Nacional Alimentación - Agencia Española de Seguridad Alimentaria y Nutrición (AESAN)	Spain

This report summarizes the outcome of the ILC exercise.

3 Scope

As stated in Regulation (EU) 2017/625 [14] one of the core duties of NRLs is to organise inter-laboratory comparative testing or proficiency tests between official laboratories. The present ILC aims to assess the analytical capabilities of NRLs, OCLs and EURL-FCM on release tests performed by article filling. The participants were asked to carry out release tests (1st, 2nd, and 3rd releases) by filling the provided commercially available enameled cups with 3 % acetic acid solution for 2h at 70°C and 4 % acetic acid solution for 24h at 22°C to determine the mass fractions of Al and Co in the food simulant.

This ILC is identified as “NRL-DE-FCM-01/2019”.

4 Set up of the exercise

4.1 Time frame of the ILC

The organization of the NRL-DE-FCM-01/2019 was announced on 06.09.2019. Registration was opened till 09.10.2019. Samples were sent to the participants on 10.10.2019 and the deadline for reporting of results was set to 16.11.2019. This deadline was extended until 6.12.2019 for individual laboratories triggered upon request with well justified reasoning.

4.2 Quality assurance

The NRL-DE-FCM is accredited according to: ISO/IEC 17025:2005 [15] (certificate number: D-PL-18583-02). The reported results were evaluated following the relevant administrative and logistic procedures.

4.3 Confidentiality

The procedures used for the organization of this inter-laboratory comparison exercise (ILC) guarantee that the identity of the participants and the information provided by them is treated as confidential. The participants in this ILC received a unique laboratory code used throughout this report.

4.4 Distribution

Each participant received:

- enameled cups
- 3 solutions (solutions No 1, No 2, and No 3)
- NRL_DE_FCM_01_2019_Confirmation of receipt.pdf
- NRL_DE_FCM_01_2019_Instructions.pdf
- NRL_DE_FCM_01_2019_Questionnaire_Results.xlsx

4.5 Instructions to participants

Detailed instructions to the participants were given in the "NRL_DE_FCM_01_2019_Instructions.pdf" (see Annex 12.1 “Instructions”)

Participants were asked to check whether the test items were undamaged after transport and to report by using the "NRL_DE_FCM_01_2019_Confirmation of receipt.pdf" form.

Participants were asked to carry out release tests (1st, 2nd, and 3rd releases) by filling of the provided cups (volume 0.3 L) with 3 % acetic acid (v/v) for 2 h at 70°C and with 4 % acetic acid (v/v) for 24 h at 22°C to determine the concentrations of Al and Co in the food simulant (3 cups for each simulant solution). EN ISO 4531:2018 [1] defines 3 % acetic acid (m/v) as simulant for acidic food. However in the last draft of that standard (ISO/DIS 4531.2:2017 [16]) 3 % acetic acid (v/v) was defined and the organizers of this ILC erroneously used this simulant. As the difference between 3 % m/v and 3 % v/v acetic acid solutions is negligible, no corrective actions were taken. In addition, participants were asked to determine the concentrations of Al and Co in provided solutions.

Besides the determined concentrations, participants were asked to report the associated expanded measurement uncertainty, the coverage factor and the approach followed for uncertainty calculations.

Participants were informed that the analytical method used for the analysis should resemble as closely as possible their analytical methods used in routine analysis.

Participants received an individual code to report their measurement results and to complete the related questionnaire. The questionnaire was used to gather additional information related to measurements and laboratories.

The laboratory codes were given randomly and communicated to the participants via e-mail.

5 Test Items

5.1 Preparation

5.1.1 Enameled cups samples

Commercially available enameled cups, packed in cardboard and covered with packaging paper, were stored at room temperature (22°C) in the dark. These cups were used for the release experiments.

5.1.2 Solutions

The release solutions were a mixture of the release solutions prepared earlier for homogeneity testing from these kinds of enamel cups (solutions No 1, No 2 in 4 % and No 3 in 3 % acetic acid solutions).

Portions of approximately 100 mL were manually filled into 125 mL screw capped HDPE Bottles, and then stored at +4°C until shipment.

5.2 Homogeneity and stability

Investigations for the homogeneity and stability studies and the statistical treatment of data were performed by the NRL for Food Contact Materials (NRL-FCM, Germany). The homogeneity assessment of solutions was performed after the preparation of the test items and before distribution to participants. The homogeneity assessment of cups was performed before distribution to participants. Cups for the homogeneity study were selected from the same charge as analyzed in the comparison exercise (analyzed in duplicate, 1st release in 3 % acetic acid at 70°C for 2 h). Results were evaluated according to ISO 13528:2015 [5]. The test items (cups) and solutions were proved to be adequately homogeneous (see Annex 12.2 “Homogeneity and stability results”).

The stability study of distributed solutions was performed over a time period of 15 weeks. For this reason, two additional samples of each solution were prepared and stored at +4°C for 15 weeks in similar flasks as used for the distribution of the solutions. Results of the stability study (see Annex 12.2 “Homogeneity and stability results”) confirm that all three solutions are adequately stable at +4 °C over the entire period of time of the ILC exercise (from date of shipment up to extended deadline).

6 Assigned values and standard uncertainties

6.1 Assigned values

No reference values were available for the measurands of concern. The assigned values x_{pt} for Al and Co concentrations [mg L^{-1}] were derived as a robust average (Q/Hampel method [17]; according to ISO 13528:2015 (C.5.4) [5] and DIN 38402 A 45 [18]) of the single results reported by the participants.

6.2 Standard uncertainties of the assigned values

Because the assigned values were derived as robust averages of the single results reported by the participants the standard uncertainties $u(x_{pt})$ of the assigned values were estimated according to ISO 13528:2015 (7.7.3) [5]:

$$u(x_{pt}) = 1.25 \frac{s^*}{\sqrt{p}} \qquad \text{(Eq.1)}$$

Where s^* is the robust standard deviation (Q/Hampel method [17]; according to ISO 13528:2015 (C.5.4) [5] and DIN 38402 A 45 [18]) of the single results reported by the participants and p is the number of values used for the calculation.

In this model, where the assigned value x_{pt} and the robust standard deviation s^* are determined from participants results, the uncertainty $u(x_{pt})$ of the assigned values can be assumed to include the effects of uncertainty due to inhomogeneity, transport, and instability (ISO 13528:2015 [5]).

6.3 Standard deviations for proficiency assessment, σ_{pt}

Relative standard deviations for proficiency assessment σ_{pt} were set for all test items based on expert judgment:

- 20 % of the assigned value for first release (both release tests)
- 30 % of the assigned value for second and third releases (both release tests)
- 15 % of the assigned value for analysis of all solutions

Table 2 and Table 3 present the relevant parameters needed for scoring, namely, the assigned values (x_{pt}) of Al and Co concentrations (expressed in mg L^{-1}) determined by the NRL-FCM (Germany), its associated expanded uncertainty ($U(x_{pt})$ calculated with a coverage factor k=2), and the standard deviation for the PT assessment (σ_{pt}).

Table 2: Relevant parameters related to the determination of Al

Test	x_{pt} ± $U(x_{pt})$* [mg L^{-1}]	σ_{pt} [mg L^{-1}]	[% of x_{pt}]	$u(x_{pt})/\sigma_{pt}$§
Release in 3 % acetic acid at 70°C for 2 h				
1st Release	6.603 ± 0.418	1.321	20	0.2
2nd Release	1.639 ± 0.164	0.492	30	0.2
3rd Release	1.442 ± 0.227	0.433	30	0.3
Release in 4 % acetic acid at 22°C for 24 h				
1st Release	14.084 ± 0.798	2.817	20	0.1
2nd Release	1.053 ± 0.079	0.316	30	0.1
3rd Release	0.639 ± 0.038	0.192	30	0.1
Solutions				
Solution No 1	14.447 ± 1.067	2.167	15	0.2
Solution No 2	1.522 ± 0.113	0.228	15	0.2
Solution No 3	0.973 ± 0.052	0.146	15	0.2

x_{pt} and $U(x_{pt})$ values were estimated using single results reported by the participants (n=15)
* $U(x_{pt})$ is the expanded uncertainty given at a given coverage factor (k=2)
§ The uncertainty of the standard value may be considered to be negligible if $u(x_{pt})/\sigma_{pt} < 0.3$ ([5])

Table 3: Relevant parameters related to the determination of Co

Test	x_{pt} ± $U(x_{pt})$ [mg L^{-1}]	σ_{pt} [mg L^{-1}]	[% of x_{pt}]	$u(x_{pt})/\sigma_{pt}$§
Release in 3 % acetic acid at 70°C for 2 h				
1st Release	0.454 ± 0.025	0.091	20	0.1
2nd Release	0.081 ± 0.011	0.024	30	0.2
3rd Release	0.076 ± 0.015	0.023	30	0.3
Release in 4 % acetic acid at 22°C for 24 h				
1st Release	0.796 ± 0.058	0.159	20	0.2
2nd Release	0.045 ± 0.004	0.014	30	0.2
3rd Release	0.031 ± 0.002	0.009	30	0.1
Solutions				
Solution No 1	0.845 ± 0.044	0.127	15	0.2
Solution No 2	0.068 ± 0.003	0.010	15	0.1
Solution No 3	0.037 ± 0.002	0.006	15	0.2

x_{pt} and $U(x_{pt})$ values were estimated using single results reported by the participants (n=14)
* $U(x_{pt})$ is the expanded uncertainty given at a given coverage factor (k = 2)
§ The uncertainty of the standard value may be considered to be negligible if $u(x_{pt})/\sigma_{pt} < 0.3$ ([5])

7 Evaluation of results

7.1 Scores and evaluation criteria

The individual laboratory performance was expressed in terms of *z* and *ζ* scores according to ISO 13528:2015 [5].

The *z* and *ζ* scores for the proficiency test results x_i were calculated as:

$$z_i = \frac{x_i - x_{pt}}{\sigma_{pt}} \qquad \text{(Eq. 2)}$$

$$\zeta_i = \frac{x_i - x_{pt}}{\sqrt{u^2(x_i) + u^2(x_{pt})}} \qquad \text{(Eq. 3)}$$

Where:

x_i	is the mean value, calculated from single values reported by the participant "i"
x_{pt}	is the assigned value
σ_{pt}	is the standard deviation for proficiency test assessment
$u(x_i)$	calculated standard uncertainty of mean value from participant "i"
$u(x_{pt})$	standard uncertainty of the assigned value

The interpretation of the z and ζ performance scores is done according to ISO 13528:2015 [5]:

$\lvert z_i \rvert \leq 2.00$	satisfactory performance	(green in Annex 13 "Results of the ILC")
$2.00 < \lvert z_i \rvert < 3.00$	questionable performance	(yellow in Annex 13 "Results of the ILC")
$\lvert z_i \rvert \geq 3.00$	unsatisfactory performance	(red in Annex 13 "Results of the ILC")

Both performance scores (z and ζ scores) were rounded to the nearest hundredth.
The *z* score demonstrates the deviation between participant mean and assigned values in terms of the standard deviation for proficiency test assessment (σ_{pt}).

The ζ score is a modified *z* score that includes uncertainties for the participant result and the assigned value. It can be used in addition to the *z* score in order to evaluate whether participant's results are close to the assigned value within their reported uncertainty.

Standard measurement uncertainty of the laboratory $u(x_i)$ was calculated by dividing the reported expanded measurement uncertainty $U(x_i)$ by the reported coverage factor *k*.

In order to verify how reasonable the measurement uncertainty of the laboratory is an additional assessment was performed for each of $u(x_i)$. For this reason, a relative standard uncertainty of the mean value from participant "i" was calculated. When no uncertainty was reported, it was set to zero ($u(x_i)_{\%} = 0$) by the PT coordinator.

$$u(x_i)_{\%} = 100\% \left(\frac{u(x_i)}{x_i}\right) \qquad \text{(Eq. 4)}$$

The values of $u(x_i)_{\%}$ were divided into three groups:

a:	$u_{\min\%} \leq u(x_i)_{\%} \leq u_{\max\%}$	reasonable estimation of $u(x_i)_{\%}$
b:	$u(x_i)_{\%} < u_{min\%}$	underestimation of $u(x_i)_{\%}$
c:	$u(x_i)_{\%} > u_{\max\%}$	overestimation of $u(x_i)_{\%}$

Where:

$u_{\min\,\%} = u(x_{pt})_{\%} = 100\%\left(\frac{u(x_{pt})}{x_{pt}}\right)$ is the minimum of the accepted relative standard uncertainty

$u_{\max\,\%} = \sigma_{pt\,\%} = 100\%\left(\frac{\sigma_{pt}}{x_{pt}}\right)$ is the maximum of the accepted relative standard uncertainty

If $u(x_i)_{\%}$ is in the range between a minimum and a maximum of allowed uncertainty (case “a”) the laboratory standard uncertainty may be reasonably estimated.

If $u(x_i)_{\%}$ is smaller than $u_{\min\,\%} = u(x_{pt})_{\%}$ (case "b") the laboratory standard uncertainty may be underestimated. However, the following should be taken into account. Because the values of $u(x_{pt})$ were derived from the robust standard deviation of the single results reported by the participants, these values include contributions for homogeneity, transport, and instability. Therefore, a relative standard uncertainty $u(x_i)_{\%}$ smaller than $u(x_{pt})_{\%}$ is possible and plausible if contributions from homogeneity, transport, and instability are significant.

If $u(x_i)_{\%}$ is larger than $u_{\max\,\%} = \sigma_{pt\,\%}$ (case “c”) the laboratory standard uncertainty may be overestimated. However, if $u(x_i)_{\%} > \sigma_{pt\,\%}$ but x_i agrees with x_{pt} within their respective expanded measurement uncertainties, then the measurement uncertainty is properly assessed. In this case, however, the corresponding performance expressed as a z score may be questionable or unsatisfactory.

7.2 General observations

Fourteen laboratories from six Member States as well as the EURL-FCM have taken part in this ILC. Fifteen laboratories have reported results for Al and fourteen for Co. One of the laboratories has not reported measurement uncertainties for the release at 70°C for 2h.

Most of the participants have used inductively coupled plasma (ICP) in combination with mass spectrometry (MS) or with optical emission spectrometry (OES) for analysis. Some laboratories used atomic absorption spectrometry (AAS) in combination with flame (FAAS) or with graphite furnace (GF-AAS) atomizers. The overview of analytical techniques used for the analysis of Al and Co in this ILC is presented in Table 4.

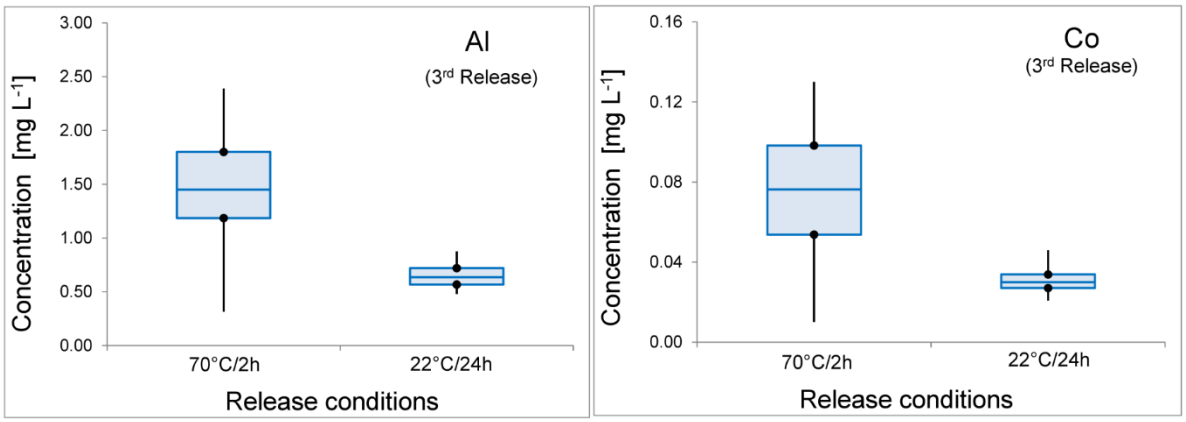
Table 4: Analytical techniques used in this ILC for the analysis of Al and Co

Technique	Al		Co	
	No. of Labs	[%]	No. of Labs	[%]
ICP-MS	9	60	10	71
ICP-OES	4	27	3	21
GFAAS	1	7	1	7
FAAS	1	7	0	0
Total	15	100	14	100

It should be noted that the deviations in the results (for both elements) reported for the second and (especially) for the third releases at 70°C for 2h are significantly higher as compared to those for the releases at 22°C for 24h.

Figure 1 (top) demonstrates the results reported for third releases at 70°C for 2h and at 22°C for 24h by using a box-plot graph. The interquartile ranges (IQR; blue box in Figure 1) for the third release at 22°C for 24h are equal to 24 % (Al) and 22 % (Co) of corresponding assigned values. For release at 70°C for 2h these values are 2 to 3 times higher, namely, 43 % (Al) and 59 % (Co).

Interestingly, the mean values of the intralaboratory variation coefficients (calculated for each participant) are similar for the releases at both conditions and for both elements [see Figure 1, bottom, intralaboratory]. This indicates that most of the participants can perform releases at both conditions and for both elements with comparable precision. However, the difference in the interlaboratory variation coefficients (calculated for all the reported values) between releases at 70°C for 2h and at 22°C for 24h is significant [see Figure 1, bottom, interlaboratory]. For the release at 22°C for 24h these variation coefficients are only 1.3 to 1.5 times higher as compared to these calculated within one lab (intralaboratory, see above), whereas for the release at 70°C for 2h this proportion is much higher (3.0 to 3.4). This indicates much higher interlaboratory spread for release at 70°C for 2h as compared to that for release at 22°C for 24h. These results show that homogeneity of the commercial cups is given/sufficient and the reasons for the interlaboratory scatter have to be found in differences between the methods used in the labs. First hints to this can be found in chapter 7.5 “Information obtained from a subsequent participant survey”.



	70°C/2h		22°C/24h	
Variation Coeff. [%]	Al	Co	Al	Co
Intralaboratory	12	15	12	14
Interlaboratory	41	45	16	20

Figure 1: Top: box-plots for the reported values of Al (left) and Co (right) for the 3rd releases at 70°C for 2h and at 22°C for 24h. Bottom: table with the variation coefficients calculated for all the reported values (interlaboratory) and the mean values of variation coefficients calculated for each participant (intralaboratory)

The experimental details, method parameters, and performance characteristics provided by the participants can be found in Annex 13.4 “Results of the Questionnaire”.

7.3 Laboratory results and scorings

7.3.1 Performances

The reported results for each measurand in form of tables and graphs can be found in Annex 13 “Results of the ILC”. The corresponding graphs have been obtained by using the PROLab™ software ([17], QuoData GmbH, Dresden).

The laboratory performance for the determination of Al and Co was assessed using z and ζ scores [5].

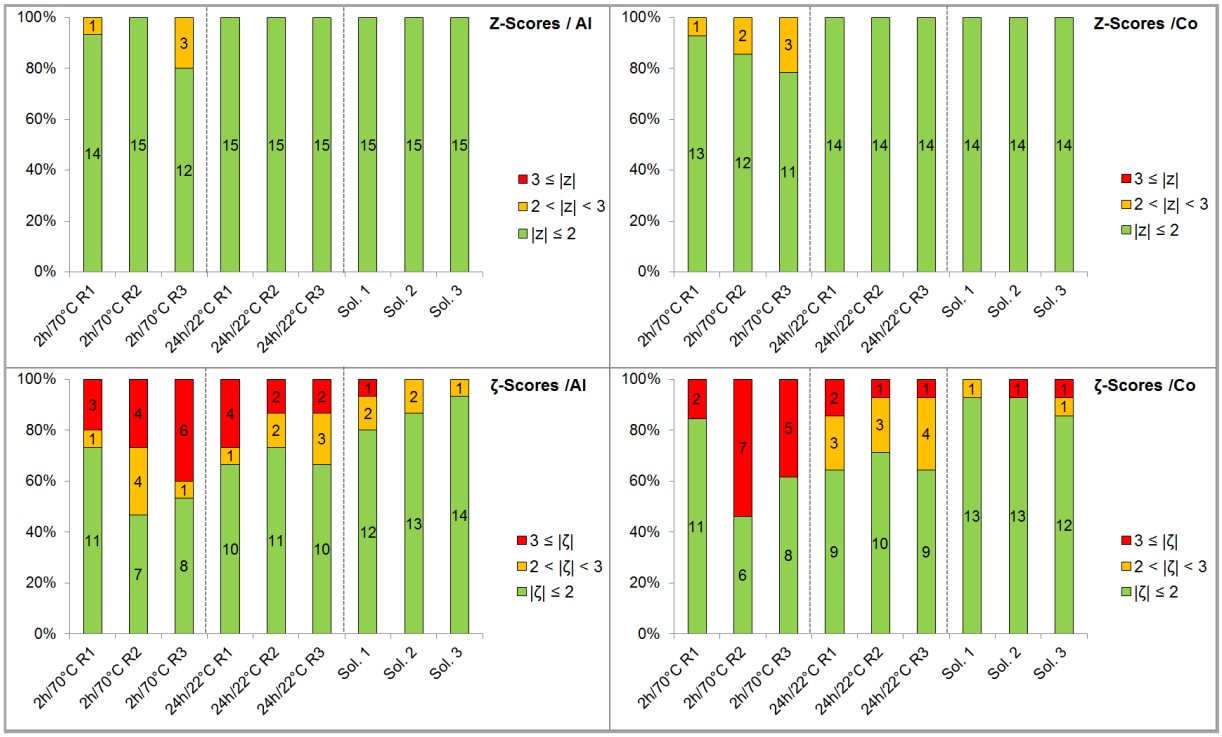


Figure 2: Overview of laboratory performance according to z and ζ scores. Corresponding number of laboratories are included in the graphs. Satisfactory, questionable and unsatisfactory performances are indicated in green, yellow and red. Releases 1, 2, and 3, are indicated with R1, R2, and R3

All of the participants have reported results with satisfactory z scores for both elements for all three releases at 22°C for 24h and for the analysis of Solutions No 1-3, while the fraction of satisfactory z scores for releases at 70°C for 2h was above 79 %. No participants have reported results with unsatisfactory z scores confirming that comparable results can be achieved with a high reliability from the participating labs.

The ζ scores were significantly worse as compared to the corresponding z scores. Fractions of satisfactory ζ scores were above 80 % for Solutions No 1-3, 64 % for releases at 22°C for 24h, and 53 % for releases at 70°C for 2h. It is notable that for releases at 70°C for 2h a very high fraction of unsatisfactory ζ scores was observed (15 – 54 %).

7.3.2 Measurement uncertainties (MU)

According to the questionnaire, only some of the participating laboratories (4 of 15) routinely report uncertainties for this type of analysis to their customers. Most of the laboratories (11 of 15) report uncertainties sometimes to their customers.

The great majority of participants have reported quantitative results provided with expanded MUs and coverage factors.

It is notable that most of the participating laboratories report comparable relative MUs $[U(x_i)_{\%} = 100\% \left(\frac{U(x_i)}{x_i}\right)]$ for the release experiments and for the analysis of solutions (see Table 5). However, due to an additional uncertainty from the release testing $U(x_i)_{\%}$ for the release experiments should be higher than that for the analysis of a solution.

Table 5: Calculated relative MUs. The values of $U(x_i)_{\%}$ were rounded to the nearest hundredth

$U(x_i)_{\%}$*									
Al									
Lab. code	at 70°C for 2h			at 22°C for 24h			Solutions		
	Rel. 1	Rel. 2	Rel. 3	Rel. 1	Rel. 2	Rel. 3	Sol. 1	Sol 2	Sol. 3
LC-001	20	20	20	20	20	20	20	20	20
LC-002	8	8	8	7	10	8	7	7	11
LC-003	21	21	21	21	21	21	21	21	30
LC-004	40	40	40	40	40	41	10	10	10
LC-005	15	15	15	15	15	15	15	15	15
LC-006	10	11	11	10	11	11	10	10	10
LC-007	12	15	15	11	16	17	11	16	16
LC-008	18	18	18	18	18	18	18	18	18
LC-009				8	10	16	8	7	
LC-010	4	7	4	7	16	7	8	8	7
LC-011	6	6	7	6	6	6	7	7	6
LC-012	20	8	17	10	15	15	10	15	8
LC-014	13	13	13	13	13	13	13	13	13
LC-015	6	5	7	6	6	6	6	6	6
LC-016	15	15	15	15	15	15	15	15	15
Co									
LC-001	15	15	15	15	15	15	15	15	15
LC-002	2	2	3	2	2	3	2	2	3
LC-003	21	21	21	21	21	21	21	21	39
LC-004	40	40	40	40	40	40	10	10	10
LC-005	15	15	15	15	16	15	15	15	16
LC-006	10	13	16	10	19	21	11	14	19
LC-007	18	24	23	17	25	28	17	24	26
LC-008	16	16	25	16	17	20	16	16	16
LC-009	0	0	0	5	5	4	5	5	0
LC-010	12	5	5	8	8	7	8	5	9
LC-011	6	6	8	9	6	6	4	4	4
LC-012									
LC-014	9	9	9	9	9	9	9	8	8
LC-015	13	13	17	12	13	13	12	12	14
LC-016	15	15	15	15	15	15	15	15	15

* $U(x_i)_{\%} = 100\% \left(\frac{U(x_i)}{x_i}\right)$

Figure 3 presents the measurement uncertainty (MU) evaluation. Most of the participants have estimated their MUs reasonably (case “a”; green) for all three releases at 22°C for 24h [87-100 % (Al) and 64-86 % (Co)] and for the analysis of Solutions 1-3 [73-93 % (Al) and 71-86% (Co)]. However, a significant part of the laboratories [up to 27 % (Al) and 36 % (Co)] has underestimated their measurement uncertainty (case “b”; yellow).

A very high fraction of underestimated MUs [27-60 % (Al) and 14-64 % (Co)] was reported for releases at 70°C for 2h. These results are in good agreement with the high fraction of unsatisfactory results [21-43 % (Al) and 15-54 % (Co)] assessed according to the ζ score. Thus, the underestimation of MUs explains the reason for the comparably bad ζ score.

Only in three cases the reported MUs were classified as overestimated (case “c”; violet). In these cases, the reported $u(x_i)_{\%}$ values are higher than the corresponding $\sigma_{pt\,\%}$ values, but the reported concentrations agree with corresponding assigned values within their respective expanded MUs. Therefore, these MUs can be classified as properly assessed. Despite the high MUs of these three values, the corresponding z scores (-0.29 [Al] and -0.18 [Co] for 1st release at 70°C for 2h; 0.40 [Co] for Solution No 3) are not questionable because the $u(x_i)_{\%}$ values are not substantially higher than the corresponding $\sigma_{pt\,\%}$ values [the proportion $u(x_i)_{\%}/\,\sigma_{pt\,\%}$ = 1.001 for the release at 70°C for 2h (for both Al and Co) and 1.286 for analysis of Co in Solution No 3].

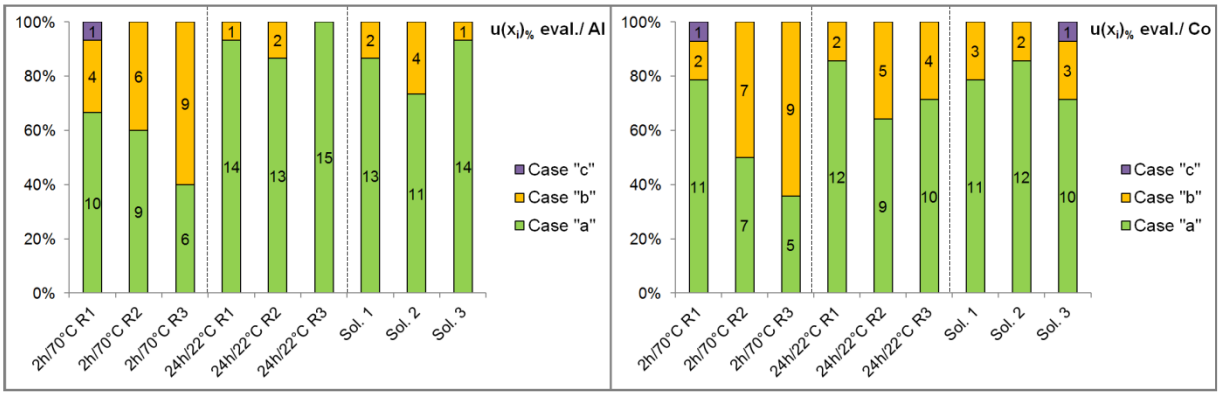


Figure 3: Evaluation of the participant's uncertainties. Corresponding numbers of the laboratories are included in the graphs. Case "a": reasonable estimation of $u(x_i)_{\%}$ (green); Case "b": underestimation of $u(x_i)_{\%}$ (yellow); Case "c": overestimation of $u(x_i)_{\%}$ (violet). Releases 1, 2, and 3, are indicated with R1, R2, and R3

7.4 Additional information extracted from the questionnaire

The questionnaire was answered by the majority of the participants providing valuable information on the laboratories, their way of working and used analytical methods. *Nevertheless, some participants have not answered all the questions. In such cases they were not taken into account and therefore the total number of entries can differ from the total number of participants in this ILC*.

General
The general information about laboratories and the used analytical methods is summarized in Annex 13.4.1 "General Information".

All participants have a quality management system based on ISO 17025 [15]. Both analytical methods used in this ILC are accredited or validated in most the participating laboratories [11 of 15 for release at 22°C for 24h; 8 of 15 for release at 70°C for 2h]. A majority of the participating laboratories (9 of 15) didn't use certified reference materials for this ILC.

For each of the investigated release methods, two participants have reported that this method is never used in their laboratories (2 of 15). However, these methods are used for more than 5 years in 73 % (11 of 15; for release at 22°C for 24h) and 47 % (7 of 15; for release at 70°C for 2h) of the participating laboratories. Nevertheless, these methods are not frequently performed and only 47 % [7 of 15; for release at 22°C for 24h] and 27 % [4 of 15; for release at 70°C for 2h] of the participants use them more than 50 times per year.

In general, most laboratories are more experienced with the method for release at 22°C for 24h as compared to that for release at 70°C for 2h.

Blanks:
Most participants (9 of 15) reported results after substraction of blank values.

Uncertainties:
Most of the laboratories (11 of 15) reported that they sometimes provide an uncertainty statement for this type of analysis to their customers. Only some (4 of 15) participants reported that uncertainties are routinely included into their report.

A majority of the laboratories estimated their uncertainty by in house validation (9 of 15) and by NORDTEST (3 of 15) [19]. Several other approaches [GUM, DIN ISO 11352 [20], and Horwitz [21],[22] (1 of 15 each)] were also used to estimate MUs.

Release testing at 70°C for 2h

Details about the single steps of the release procedure at 70°C for 2h can be found in Annex 13.4.3 “Release at 70°C for 2h”. This includes the details about preheating of simulant, thermostatic oven and enameled cups as well as on the filling procedure and on the contact phase.

Preheating of simulant, thermostatic oven, and test specimen:

Most of the participants (12 of 15) used a calibrated thermometer or a calibrated data logger for verification of the desired food simulant temperature. In most of the laboratories the temperature was measured inside a separate food simulant portion (9 of 15) or directly inside the food simulant portion (4 of 15); the food simulant portion which contained the probe was covered (11 of 15) during the preheating step.

All participants (13 of 13) preheated the thermostatic oven at temperatures between 70 and 75 °C for a time period of 15 to 960 min.

The test specimen was preheated by all participants (14 of 14) at temperatures between 70 and 75 °C for a time period of 10 to 60 min.

Filling procedure:

Half of the participants (7 of 14) did the test specimen filing inside and the other half (7 of 14) outside the oven at distances from 0.5 to 3 m. In case of filling the specimen inside the oven, the maximal time for that the oven’s door was opened was 60 second. Filling time per test specimen was within the range of 3 to 117 seconds.

All participants (14 of 14) covered the test specimen. For this reason different items were used: watch glass (9 of 14), silicon lid (3 of 14), plastic lid (1 of 14), and petri dish (1 of 14). This resulted in only a minor loss of the simulant during the release experiments (0 to 10 mL).

Temperature control of the food simulant during release testing:

Only some participants (3 of 14) performed the temperature control of the food simulant during the release testing directly “in the filled specimen”. In one laboratory the temperature was not controlled and in one it was controlled only in the beginning and in the end of release experiments. Most participants (10 of 14) performed the temperature control “on a separate mode (e.g. glass bottle)”. However, in this case the recorded temperature and the actual temperature of the food simulant in the test specimen can differ due to possible differences in the heat capacities. Most of the participants (11 of 14) used a calibrated thermometer or a calibrated data logger for verification of the desired food simulant temperature.

Most of the reported temperatures are within the acceptable range of 70 ± 2 °C. Nevertheless, a few participants have reported temperature values that are above 72 °C or below 68 °C over the main portion of the release time. These deviations, however, are not high. The lowest and the highest mean temperatures were found to be 67.0 and 73.0 °C, respectively. In some cases the reported temperature values are above 72 °C in the beginning and below 68 °C in the end of the release experiment. However, no clear correlation between the reported temperatures and the performance could be found.

The reported temperature profile of the food simulant during the release at 70°C for 2h can be found in Figure 4 and in Annex 13.4.5 “Temperature of the food simulant during the release at 70°C for 2h”.

Annex 13.4.4 “Release at 22°C for 24 h” provide details about single steps of release procedure at 24°C (22°C). This includes the details about preheating of simulant, thermostatic oven and enameled cups as well as on contact phase.

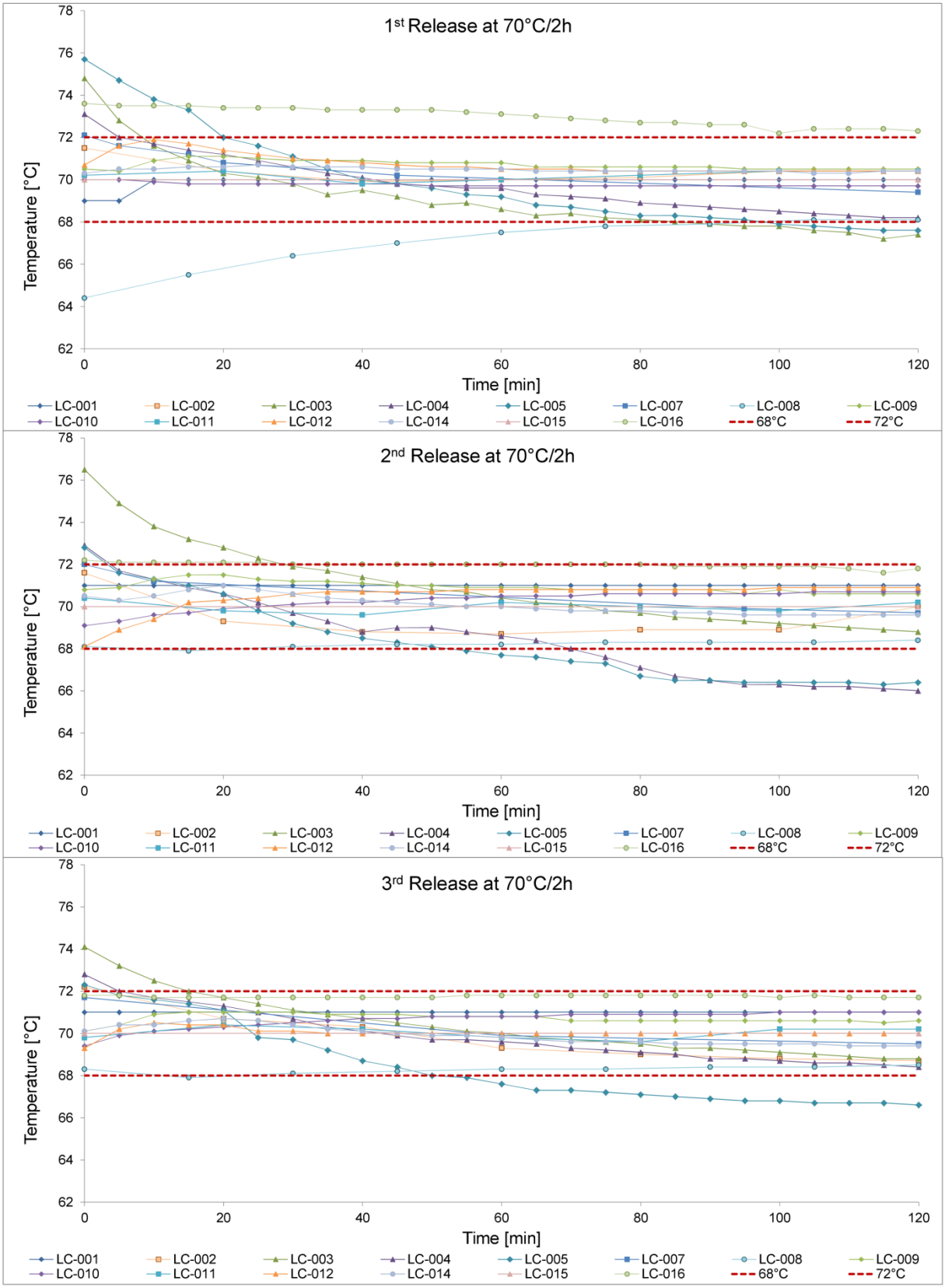


Figure 4: Reported temperatures of the food simulant during the releases at 70°C for 2h

7.5 Information obtained from a subsequent participant survey

In order to clarify the possible reasons for the high deviation in the concentrations reported for 2nd and 3rd releases at 70°C for 2h, participants were asked after the exercise to specify some details on the utilized release method.

The following questions were answered by the majority (13 of 15) of the participating laboratories:

1) Did you perform all three migrations on one day?
2) How much time was in between the single migration steps?
3) Did you flush the cups between the release tests? If so: How did you do the cleaning and which solvent did you use?

The reported information is summarized in Table 6. This table demonstrates that there is a huge diversity in implementation of release method between the participating laboratories, giving first hints to an explanation for the comparably high interlaboratory differences for the third migration at 70° C.

36 % (5 of 14) of the participants performed all releases on the same day; 29 % (4 of 14) performed two releases (1st and 2nd) on the first day and the third release on the next day; 14 % (2 of 14) performed first release on the first day and two releases (2nd and 3rd) on the next day; 21 % (3 of 14) performed the releases on three successive days.

Most of the participants (11 of 14) flushed the cups between migrations. However, three participants didn't flush the cups. This may result in additional corrosion of the cups between the releases.

Table 6: Additional information obtained from the participant survey for the release at 70°C for 2h

Lab. code	Release No			Time between Releases [h]		Did you flush the cups between the migrations?		z-scores (for release at 70°C for 2h)					
								Al			Co		
	Day 1	Day 2	Day 3	R1/R2	R2/R3		Solvent:	R1	R2	R3	R1	R2	R3
LC-001	1	2	3	19	15	Yes	Dest. W.	-0.40	-0.96	-1.60	-0.12	-1.25	-1.65
LC-002	1,2,3			0.5	0.5	Yes	Bidest. W.	0.17	1.75	-0.25	0.21	2.13	-0.47
LC-003	1,2,3			0.25	0.25	Yes	Ultrapure W.	-0.49	-0.32	-0.85	-0.31	-0.05	-0.03
LC-004	1,2	3		2	17	No		-0.29	0.23	1.16	-0.18	0.29	1.58
LC-005	1	2,3		22	2	No		0.29	1.74	1.53	0.20	2.83	2.06
LC-006	1	2	3	22	22	Yes	Purified W.	2.02	-1.41	-2.32	2.45	-1.56	-2.42
LC-007	1,2	3		0.2	~24	Yes	Deion. W.	-0.48	-0.21	0.59	-0.44	-0.12	1.01
LC-008	1,2,3			0.5-0.75	0.5-0.75	Yes	Dest. W.	-0.88	-0.80	-2.18	0.13	0.36	-2.46
LC-009	1	2,3		18	2	Yes	Ultrapure W.	-0.63	-0.22	0.60	-0.78	-0.33	1.14
LC-010	1,2	3		1	19	Yes	Ultrapure W.	-0.07	-0.09	-0.20	-0.37	-0.95	-0.61
LC-011								1.39	0.05	-0.26	1.27	0.04	0.03
LC-012	1,2,3			0	0	No		0.12	0.58	2.10			
LC-014	1,2	3		0.4	20	Yes	Ultrapure W.	-0.45	0.72	0.43	-0.41	1.40	0.63
LC-015	1	2	3	24(±1)	24(±1)	Yes	Milli-Q W.	1.06	-0.48	1.44	0.73	-0.93	1.94
LC-016	1,2,3			0.8	0.8	Yes	Milli-Q W.	-0.41	0.04	-0.47	-0.41	-0.66	-0.90

8 Conclusion

The inter-laboratory comparison exercise NRL-DE-FCM-01/2019 was organized to assess the analytical capabilities of NRLs, OCLs and EURL-FCM on release tests performed by article filling. The participants were asked to carry out release tests (1st, 2nd, and 3rd releases) by filling the provided commercially available enameled cups with 3 % acetic acid solution for 2h at 70°C and 4 % acetic acid solution for 24h at 22°C to determine the concentrations of Al and Co in the food simulant. As the temperature affects results, the participants were requested to monitor and report the temperature of the food simulants during the release test at 70°C.

The overall performance of the participants, expressed as a z score, was satisfactory and the temperature monitoring clearly showed that all labs are able to perform the release tests at 70°C in the correct temperature range.

All participants have reported results with satisfactory z scores for both elements for all three releases at 22°C for 24h and for the analysis of Solutions No 1-3. No participants have reported results with questionable or unsatisfactory z scores. These results confirm that all participating laboratories are able to perform experiments for the determination of Al and Co release from enameled articles according to European Directive 84/500/EEC [2].

For the releases at 70°C a satisfactory performance was achieved by more than 79 % of the participating laboratories. No participants have reported results with unsatisfactory z scores. These results demonstrate that most participating laboratories are able to perform experiments for the determination of Al and Co release from enameled articles according to ISO 4531:2018 [1]. The subsequent survey of the particapants revealed significant differences in the performance of the release test itself, and thus further studies for the harmonization are essential to further lower the interlaboratorial scatter of results for the second and third release tests at 70° C.

The ζ scores were significantly worse as compared to the corresponding z scores. Fractions of satisfactory ζ scores were above 80 % for Solutions No 1-3, 64 % for releases at 22°C for 24h, and 53 % for releases at 70°C for 2h. It is notable that for releases at 70°C for 2h a very high fraction of unsatisfactory ζ scores was observed (15 – 54 %).

Most laboratories report similar relative MUs [$U(x_i)_{\%}$] for the release experiments and for the analysis of the solutions. However, due to additional uncertainty from migration the $U(x_i)_{\%}$ for the release experiments should be higher than that for the analysis of a solution

Most of the participants have estimated their MUs reasonably for all three releases at 22°C for 24h [87-100 % (Al) and 64-86 % (Co)] and for the analysis of Solutions 1-3 [73-93 % (Al) and 71-86 % (Co)]. However, a significant part of the laboratories [up to 27 % (Al) and 36 % (Co)] have underestimated their measurement uncertainty.

A very high fraction of underestimated MUs [27-60 % (Al) and 14-64 % (Co)] was reported for releases at 70°C for 2h. These results are in good agreement with high fraction of unsatisfactory results [21-43 % (Al) and 15-54 % (Co)] assessed according to the ζ score.

The reported temperatures are within the acceptable range of 70 ± 2 °C for most of the labs. Nevertheless, a few participants have reported temperature values that are slightly above 72 °C or below 68 °C over the main portion of the release time. These deviations, however, are not high, the lowest and the highest mean temperatures were found to be 67.0 and 73.0 °C, respectively. However, no clear correlation between the reported temperatures and the performance could be found.

9 References

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10 List of Figures

11 List of Tables

12 Annex

12.1 Instructions

Please perform release testing as specified below. Analyze the three additionally provided solutions together with the release test solution.

For added value of this study, we would appreciate if you could send us an aliquot (~ 15 mL) of each of the respective release solutions. In consequence, we would examine all incoming release solutions in a sequence with our ICP-MS. We expect to improve the data basis for the estimation of the measurement uncertainty.

1. Pre-treatment of all commercial enamelled cups prior release testing

Each commercial enamelled cup must be cleaned prior release testing with non-acidic diluted detergent at a temperature of about 40 °C followed by ultrapure water before first release test. Dry them carefully before use without direct contact to the surface.

2. Release testing of commercial enamelled cups according to European Directive 84/500/EEC (Ceramics)

- Three enamelled cups shall be used for the release test.
- Each enamelled cup shall be consecutively tested three times (1st to 3rd release test) within three days (Please ensure, that between release testing of the samples is no weekend).
- 24 hours (±0.5 h) at 22 °C (±2 °C)
- 300 mL of simulant (4 % acetic acid (v/v))
- Use a suited lid (e.g. silicone) to cover each cup during the entire release test.
- Please ensure homogeneity of the release solution prior final determination.
- Prior to the final analytical determination store the solutions in pre-cleaned adequate containers and additionally in the provided BfR-vials.
- Rinse each enamelled cup in between tests with ultrapure water.

3. Release testing of commercial enamelled cups according to EN ISO 4531:2018

- Three enamelled cups shall be used for the release test.
- Each enamelled cup shall be consecutively tested three times (1st to 3rd release test) within two days (please ensure, that between release testing of the samples is no week-end).
- 2 hours (±5 minutes) at 70 °C (±2 °C)
- Heat 300 mL of food simulant (3 % (v/v) acetic acid) in a closed 500 mL glass bottle at a temperature (e.g. 75 °C) that ensures the temperature stability of the release test in a drying cabinet during the entire release test.
- Use a suited lid (e.g. silicone) to cover each cup during the entire release test.
- Preheat the enamelled cups at (e.g. 72 °C) in the drying cabinet as short as possible (e.g. 15 minutes) that ensures the temperature stability of the release test in a drying cabinet during the entire release test.

- Fill the preheated food simulant as completely as possible into the preheated enamelled cups.
- Implement a temperature control (in solution) while testing (if possible in an equal proceeded additional cup, or, if not possible, in one of the test specimens).
- Use a suitable lid (e.g. silicone) to cover each single cup.
- Rinse in between tests with ultrapure water.
- Please ensure homogeneity of the release solution prior final determination.
- Prior to the final analytical determination store the solutions in pre-cleaned adequate containers and additionally in the provided BfR-vials.

12.2 Homogeneity and stability results

The homogeneity assessment of the solutions was performed after preparation of the test items and before distribution to the participants. Results are evaluated according to ISO 13528:2015.

12.2.1 Homogeneity study (Solution No 1)

Table 7: Results of the homogeneity study for solution No 1

	Al		Co	
	Rep. 1	Rep. 2	Rep. 1	Rep. 2
1	13.156	13.246	0.685	0.696
2	12.966	12.992	0.679	0.700
3	13.182	13.115	0.688	0.688
4	13.191	12.866	0.685	0.686
5	13.034	12.942	0.677	0.692
6	13.025	13.095	0.683	0.684
7	13.196	13.091	0.683	0.696
8	13.311	12.958	0.686	0.688
9	13.104	13.103	0.681	0.681
10	12.927	12.924	0.681	0.691
11	13.112	13.274	0.677	0.679
12	13.074	13.004	0.684	0.681
13	13.056	12.897	0.675	0.679
14	13.192	13.009	0.684	0.685
15	12.877	12.870	0.668	0.682
16	13.381	12.979	0.687	0.672
17	13.158	12.740	0.681	0.689
18	12.950	12.866	0.672	0.683
19	12.962	12.954	0.666	0.690
20	13.144	12.963	0.674	0.676
$Mean$	13.047		0.683	
$s_{\bar{x}}$	0.100		0.005	
s_w	0.136		0.007	
s_s	0.030		0.000	
σ_{pt}(15% of Mean)	1.957		0.102	
σ_{allow}	0.587		0.031	
$s_s \leq \sigma_{allow}$	Passed		Passed	
Assessment	Homogenous		Homogenous	

All values expressed in mg L^{-1}

Where:
- $s_{\bar{x}}$ Standard deviation of sample averages
- s_w Within-sample standard deviation
- s_s Estimate of between-sample standard deviation
- σ_{pt} Standard deviation for proficiency assessment
- σ_{allow} $\sigma_{allow} = 0.3\ \sigma_{pt}$; criterion of sufficient homogeneity

12.2.2 Homogeneity study (Solution No 2)

Table 8: Results of the homogeneity study for solution No 2

	Al		Co	
	Rep. 1	Rep. 2	Rep. 1	Rep. 2
1	1.654	1.722	0.066	0.069
2	1.664	1.700	0.066	0.067
3	1.647	1.662	0.066	0.066
4	1.597	1.629	0.065	0.066
5	1.617	1.670	0.065	0.066
6	1.641	1.648	0.066	0.066
7	1.605	1.690	0.065	0.067
8	1.653	1.675	0.066	0.067
9	1.660	1.695	0.066	0.067
10	1.658	1.704	0.066	0.068
11	1.608	1.664	0.065	0.067
12	1.645	1.710	0.065	0.068
13	1.623	1.671	0.065	0.067
14	1.660	1.717	0.067	0.069
15	1.632	1.688	0.065	0.067
16	1.634	1.673	0.065	0.068
17	1.664	1.669	0.067	0.068
18	1.610	1.707	0.064	0.068
19	1.648	1.683	0.065	0.067
20	1.629	1.668	0.065	0.067
$Mean$	1.660		0.066	
$s_{\overline{x}}$	0.019		0.001	
s_w	0.036		0.001	
s_s	0.000		0.000	
σ_{pt}(15% of Mean)	0.249		0.010	
σ_{allow}	0.075		0.003	
$s_s \leq \sigma_{allow}$	Passed		Passed	
Assessment	Homogenous		Homogenous	

All values expressed in mg L^{-1}

Where:
- $s_{\overline{x}}$ Standard deviation of sample averages
- s_w Within-sample standard deviation
- s_s Estimate of between-sample standard deviation
- σ_{pt} Standard deviation for proficiency assessment
- σ_{allow} $\sigma_{allow} = 0.3\ \sigma_{pt}$; criterion of sufficient homogeneity

12.2.3 Homogeneity study (Solution No 3)

Table 9: Results of the homogeneity study for solution No 3

	Al		Co	
	Rep. 1	Rep. 2	Rep. 1	Rep. 2
1	1.004	1.101	0.038	0.035
2	1.024	1.107	0.038	0.035
3	1.027	1.083	0.037	0.035
4	1.021	1.107	0.038	0.035
5	1.023	1.115	0.038	0.036
6	1.029	1.132	0.038	0.035
7	1.062	1.121	0.038	0.035
8	1.051	1.098	0.037	0.035
9	1.038	1.131	0.038	0.036
10	1.073	1.146	0.039	0.035
11	1.045	1.106	0.038	0.035
12	1.052	1.144	0.039	0.035
13	1.053	1.129	0.038	0.035
14	1.035	1.091	0.037	0.035
15	1.042	1.152	0.039	0.034
16	1.035	1.110	0.038	0.035
17	1.030	1.131	0.039	0.035
18	1.056	1.108	0.038	0.035
19	1.065	1.114	0.038	0.035
20	1.051	1.119	0.039	0.035
$Mean$	1.079		0.037	
$s_{\overline{x}}$	0.015		0.000	
s_w	0.056		0.002	
s_s	0.000		0.000	
σ_{pt}(15% of Mean)	0.162		0.005	
σ_{allow}	0.049		0.002	
$s_s \leq \sigma_{allow}$	Passed		Passed	
Assessment	Homogenous		Homogenous	

All values expressed in mg L^{-1}

Where:
- $s_{\overline{x}}$ Standard deviation of sample averages
- s_w Within-sample standard deviation
- s_s Estimate of between-sample standard deviation
- σ_{pt} Standard deviation for proficiency assessment
- σ_{allow} $\sigma_{allow} = 0.3\ \sigma_{pt}$; criterion of sufficient homogeneity

12.2.4 Homogeneity study (Enameled Cups)

The homogeneity assessment of the enameled cups was performed before distribution to the participants. Cups for the homogeneity study were selected from the same charge as analyzed in the comparison exercise (analyzed in duplicate, 1st release in 3 % acetic acid at 70°C for 2h). Results are evaluated according to ISO 13528:2015 (B.2.3).

Table 10: Results of the homogeneity study for enameled cups

	Al		Co	
	Rep. 1	Rep. 2	Rep. 1	Rep. 2
1	7.136	6.758	0.448	0.461
2	5.792	5.617	0.382	0.390
3	5.869	5.753	0.405	0.420
4	7.026	6.856	0.438	0.460
5	6.728	6.600	0.409	0.409
6	6.781	6.642	0.370	0.393
7	6.653	6.517	0.423	0.433
8	6.876	6.625	0.459	0.483
9	6.092	5.949	0.379	0.394
10	6.156	5.926	0.400	0.413
11	6.580	6.324	0.429	0.441
12	6.708	6.424	0.421	0.427
$Mean$	6.433		0.420	
$s_{\bar{x}}$	0.430		0.029	
s_w	0.152		0.011	
s_w^2	0.023		0.0001	
s_s	0.417		0.028	
σ_{pt}(20% of Mean)	1.287		0.084	
σ_{allow}	0.386		0.025	
F_1	1.79		1.79	
F_2	0.86		0.86	
$(\sigma_{allow})^2$	0.149		0.001	
c	0.286		0.001	
$\sqrt{c}$	0.535		0.035	
$s_s \leq \sqrt{c}$	Passed		Passed	
Assessment	Homogenous		Homogenous	

All values expressed in mg L^{-1}

Where:
- $s_{\bar{x}}$ Standard deviation of sample averages
- s_w Within-sample standard deviation
- s_s Estimate of between-sample standard deviation
- σ_{pt} Standard deviation for proficiency assessment
- σ_{allow} $\sigma_{allow} = 0.3\,\sigma_{pt}$; criterion of sufficient homogeneity
- F_1, F_2 Factors for use in testing for sufficient homogeneity
- c $c = F_1\sigma_{allow}^2 + F_2 s_w^2$; is used to expand the criterion to allow for the actual sampling error and repeatability

12.2.5 Stability study

(Solutions stored at 4 °C for 15 weeks)

Table 11: Results of the stability study. Results are evaluated according to ISO 13528:2015 (B.5.1)

	Solution No 1			Solution No 2			Solution No 3		
Bottle ID	#1	#11	Mean	#40	#50	Mean	#10	#29	Mean
Al									
w_0	13.309	13.436	13.373	1.598	1.585	1.591	1.031	1.034	1.032
w_{15}	13.071	13.199	13.135	1.555	1.521	1.538	0.989	1.006	0.998
$\lvert w_0-w_{15}\rvert$			0.237			0.053			0.035
σ_{pt}			2.167			0.228			0.146
$0.3\sigma_{pt}$			0.650			0.069			0.044
$\lvert w_0-w_{15}\rvert \leq 0.3\sigma_{pt}$			Passed			Passed			Passed
Assess-ment			Stable			Stable			Stable
Co									
w_0	0.708	0.710	0.709	0.0653	0.0647	0.0650	0.0349	0.0353	0.0351
w_{15}	0.689	0.694	0.692	0.0626	0.0622	0.0624	0.0338	0.0338	0.0338
$\lvert w_0-w_{15}\rvert$			0.017			0.0026			0.0012
σ_{pt}			0.127			0.0103			0.0056
$0.3\sigma_{pt}$			0.038			0.0031			0.0017
$\lvert w_0-w_{15}\rvert \leq 0.3\sigma_{pt}$			Passed			Passed			Passed
Assess-ment			Stable			Stable			Stable

All values expressed in mg L^{-1}

Where:
- w_0 Analysis in the beginning of stability study
- w_{15} Analysis in the end
- σ_{pt} Standard deviation for proficiency assessment

13 Results of the ILC

13.1 Release into 3 % acetic acid solution (2h at 70°C)

13.1.1 First release test of Al into 3 % acetic acid solution (2h at 70°C)

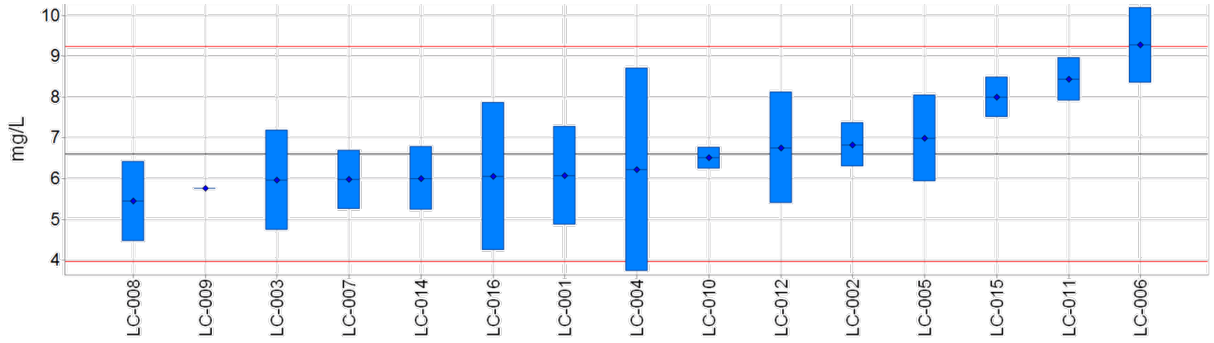

Figure 5: Measurement result range reported by the participants for the first release test of Al into 3 % acetic acid (2h at 70°C). Red lines demonstrate z score = 2 (x_{pt}± 2 σ_{pt}), black line is x_{pt}, points and bars show x_i and U_i

Table 12: Results for the first release test of Al into 3 % acetic acid solution (2h at 70°C). Assigned range: x_{pt} = 6.603 ± 0.418 [mg L^{-1}]; σ_{pt} = 1.321 [mg L^{-1}]; x_i, U_i, and u_i are in [mg L^{-1}]

Lab code	x_i	U_i	k	u_i	z score	ζ score	u_i $est.$
LC-001	6.070	1.214	2	0.607	-0.40	-0.83	a
LC-002	6.833	0.533	2	0.267	0.17	0.68	a
LC-003	5.956	1.227	2	0.613	-0.49	-1.00	a
LC-004	6.217	2.490	2	1.245	-0.29	-0.31	c
LC-005	6.990	1.063	2	0.532	0.29	0.68	a
LC-006	9.273	0.933	3	0.311	2.02	7.12	a
LC-007	5.973	0.730	2	0.365	-0.48	-1.50	a
LC-008	5.444	0.981	2	0.490	-0.88	-2.17	a
LC-009	5.767				-0.63		b
LC-010	6.506	0.259	2	0.129	-0.07	-0.39	b
LC-011	8.443	0.533	2	0.267	1.39	5.43	b
LC-012	6.757	1.363	2	0.682	0.12	0.22	a
LC-014	6.007	0.780	2	0.390	-0.45	-1.35	a
LC-015	8.000	0.500	2	0.250	1.06	4.29	b
LC-016	6.059	1.818	2	0.909	-0.41	-0.58	a

13.1.2 First release test of Co into 3 % acetic acid solution (2h at 70°C)

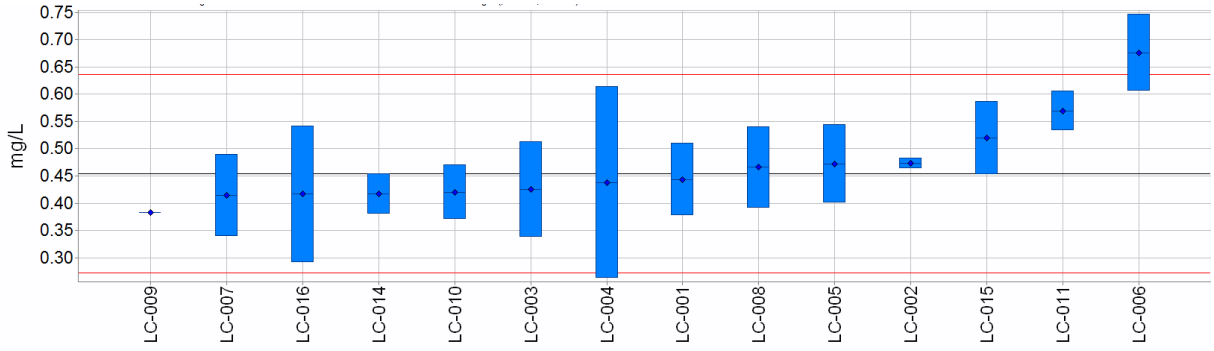

Figure 6: Measurement result range reported by the participants for the first release test of Co into 3 % acetic acid (2h at 70°C). Red lines demonstrate z score = 2 (x_{pt}± 2 σ_{pt}), black line is x_{pt}, points and bars show x_i and U_i

Table 13: Results for the first release test of Co into 3 % acetic acid solution (2h at 70°C). Assigned range: x_{pt} = 0.454 ± 0.025 [mg L^{-1}]; σ_{pt} = 0.091 [mg L^{-1}]; x_i, U_i, and u_i are in [mg L^{-1}]

Lab code	x_i	U_i	k	u_i	z score	ζ score	u_i $est.$
LC-001	0.443	0.067	2	0.033	-0.12	-0.30	a
LC-002	0.473	0.010	2	0.005	0.21	1.43	b
LC-003	0.426	0.087	2	0.044	-0.31	-0.62	a
LC-004	0.438	0.175	2	0.088	-0.18	-0.18	c
LC-005	0.472	0.072	2	0.036	0.20	0.48	a
LC-006	0.676	0.070	3	0.023	2.45	8.37	a
LC-007	0.414	0.076	2	0.038	-0.44	-0.99	a
LC-008	0.311	0.075	2	0.037	0.13	0.31	a
LC-009	0.383				-0.78		b
LC-010	0.420	0.050	2	0.025	-0.37	-1.21	a
LC-011	0.569	0.036	2	0.018	1.27	5.21	a
LC-012							
LC-014	0.417	0.037	2	0.019	-0.41	-1.64	a
LC-015	0.520	0.067	2	0.033	0.73	1.85	a
LC-016	0.417	0.125	2	0.063	-0.41	-0.59	a

13.1.3 Second release test of Al into 3 % acetic acid solution (2h at 70°C)

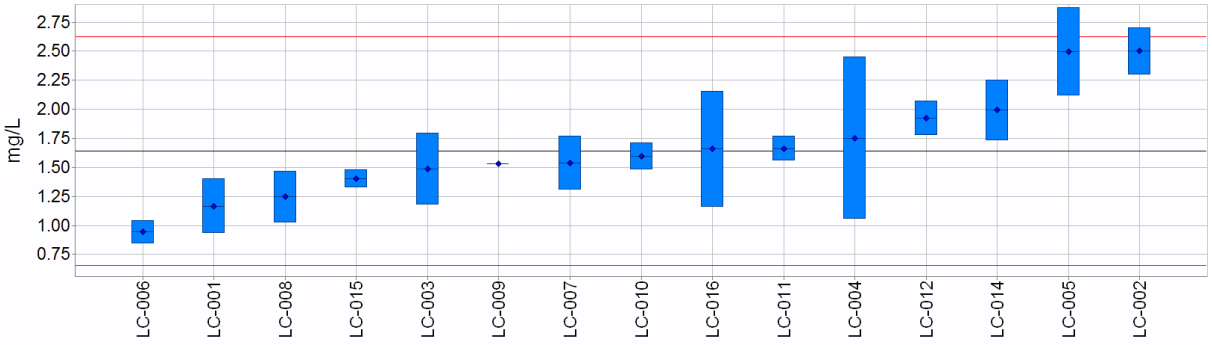

Figure 7: Measurement result range reported by the participants for the second release test of Al into 3 % acetic acid (2h at 70°C). Red lines demonstrate z score = 2 (x_{pt}± 2 σ_{pt}), black line is x_{pt}, points and bars show x_i and U_i

Table 14: Results for the second release test of Al into 3 % acetic acid solution (2h at 70°C). Assigned range: x_{pt} = 1.639 ± 0.164 [mg L^{-1}]; σ_{pt} = 0.492 [mg L^{-1}]; x_i, U_i, and u_i are in [mg L^{-1}]

Lab code	x_i	U_i	k	u_i	z score	ζ score	u_i $est.$
LC-001	1.167	0.233	2	0.117	-0.96	-3.32	a
LC-002	2.500	0.200	2	0.100	1.75	6.66	b
LC-003	1.484	0.308	2	0.154	-0.32	-0.89	a
LC-004	1.753	0.700	2	0.350	0.23	0.32	a
LC-005	2.497	0.377	2	0.188	1.74	4.18	a
LC-006	0.945	0.100	3	0.033	-1.41	-7.85	a
LC-007	1.537	0.230	2	0.115	-0.21	-0.73	a
LC-008	1.246	0.224	2	0.112	-0.80	-2.83	a
LC-009	1.533				-0.22		b
LC-010	1.597	0.115	2	0.058	-0.09	-0.43	b
LC-011	1.663	0.107	2	0.053	0.05	0.25	b
LC-012	1.923	0.150	2	0.075	0.58	2.56	b
LC-014	1.993	0.260	2	0.130	0.72	2.30	a
LC-015	1.403	0.077	2	0.038	-0.48	-2.61	b
LC-016	1.659	0.498	2	0.249	0.04	0.08	a

13.1.4 Second release test of Co into 3 % acetic acid solution (2h at 70°C)

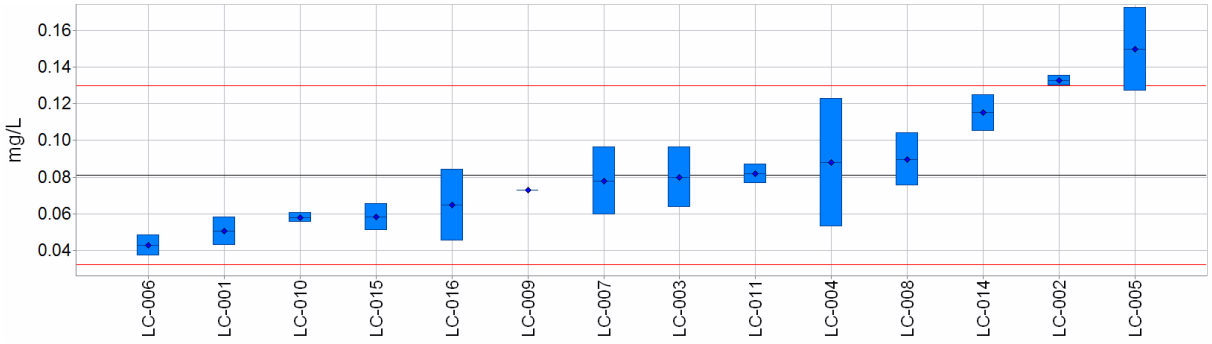

Figure 8: Measurement result range reported by the participants for the second release test of Co into 3 % acetic acid (2h at 70°C). Red lines demonstrate z score = 2 (x_{pt}± 2 σ_{pt}), black line is x_{pt}, points and bars show x_i and U_i

Table 15: Results for the second release test of Co into 3 % acetic acid solution (2h at 70°C). Assigned range: x_{pt} = 0.081 ± 0.011 [mg L^{-1}]; σ_{pt} = 0.024 [mg L^{-1}]; x_i, U_i, and u_i are in [mg L^{-1}]

Lab code	x_i	U_i	k	u_i	z score	ζ score	u_i $est.$
LC-001	0.051	0.008	2	0.004	-1.25	-4.55	a
LC-002	0.133	0.003	2	0.002	2.13	9.10	b
LC-003	0.080	0.016	2	0.008	-0.05	-0.11	a
LC-004	0.088	0.035	2	0.018	0.29	0.38	a
LC-005	0.150	0.023	2	0.011	2.83	5.45	a
LC-006	0.043	0.006	3	0.002	-1.56	-6.56	b
LC-007	0.078	0.019	2	0.009	-0.12	-0.28	a
LC-008	0.090	0.014	2	0.007	0.36	0.96	a
LC-009	0.073				-0.33		b
LC-010	0.058	0.003	2	0.001	-0.95	-4.08	b
LC-011	0.082	0.005	2	0.003	0.04	0.15	b
LC-012							
LC-014	0.115	0.010	2	0.005	1.40	4.58	b
LC-015	0.058	0.007	2	0.004	-0.93	-3.44	b
LC-016	0.065	0.020	2	0.010	-0.66	-1.44	a

13.1.5 Third release test of Al into 3 % acetic acid solution (2h at 70°C)

Figure 9: Measurement result range reported by the participants for the third release test of Al into 3 % acetic acid (2h at 70°C). Red lines demonstrate z score = 2 (x_{pt}± 2 σ_{pt}), black line is x_{pt}, points and bars show x_i and U_i

Table 16: Results for the third release test of Al into 3 % acetic acid solution (2h at 70°C). Assigned range: x_{pt} = 1.442 ± 0.227 [mg L^{-1}]; σ_{pt} = 0.433 [mg L^{-1}]; x_i, U_i, and u_i are in [mg L^{-1}]

Lab code	x_i	U_i	k	u_i	z score	ζ score	u_i $est.$
LC-001	0.750	0.150	2	0.075	-1.60	-5.08	a
LC-002	1.333	0.100	2	0.050	-0.25	-0.88	b
LC-003	1.074	0.221	2	0.110	-0.85	-2.32	a
LC-004	1.943	0.777	2	0.388	1.16	1.24	a
LC-005	2.103	0.320	2	0.160	1.53	3.37	b
LC-006	0.436	0.047	3	0.016	-2.32	-8.77	b
LC-007	1.697	0.250	2	0.125	0.59	1.51	b
LC-008	0.500	0.090	2	0.045	-2.18	-7.71	a
LC-009	1.700				0.60		b
LC-010	1.356	0.060	2	0.030	-0.20	-0.73	b
LC-011	1.330	0.090	2	0.045	-0.26	-0.92	b
LC-012	2.350	0.400	2	0.200	2.10	3.95	a
LC-014	1.630	0.210	2	0.105	0.43	1.21	b
LC-015	2.063	0.143	2	0.072	1.44	4.62	b
LC-016	1.240	0.372	2	0.186	-0.47	-0.93	a

13.1.6 Third release test of Co into 3 % acetic acid solution (2h at 70°C)

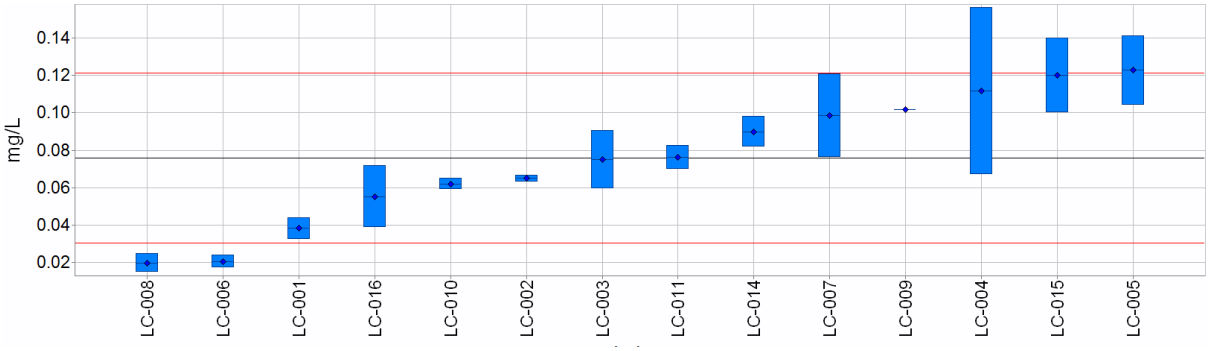

Figure 10: Measurement result range reported by the participants for the third release test of Co into 3 % acetic acid (2h at 70°C). Red lines demonstrate z score = 2 (x_{pt}± 2 σ_{pt}), black line is x_{pt}, points and bars show x_i and U_i

Table 17: Results for the third release test of Co into 3 % acetic acid solution (2h at 70°C). Assigned range: x_{pt} = 0.076 ± 0.015 [mg L^{-1}]; σ_{pt} = 0.023 [mg L^{-1}]; x_i, U_i, and u_i are in [mg L^{-1}]

Lab code	x_i	U_i	k	u_i	z score	ζ score	u_i $est.$
LC-001	0.038	0.006	2	0.003	-1.65	-4.74	b
LC-002	0.065	0.002	2	0.001	-0.47	-1.46	b
LC-003	0.075	0.015	2	0.008	-0.03	-0.07	a
LC-004	0.112	0.045	2	0.022	1.58	1.53	a
LC-005	0.123	0.019	2	0.009	2.06	3.94	b
LC-006	0.021	0.003	3	0.001	-2.42	-7.41	b
LC-007	0.099	0.022	2	0.011	1.01	1.71	a
LC-008	0.020	0.005	2	0.002	-2.46	-7.21	a
LC-009	0.102				1.14		b
LC-010	0.062	0.003	2	0.001	-0.61	-1.84	b
LC-011	0.076	0.006	2	0.003	0.03	0.08	b
LC-012							
LC-014	0.090	0.008	2	0.004	0.63	1.68	b
LC-015	0.120	0.020	2	0.010	1.94	3.56	b
LC-016	0.055	0.017	2	0.008	-0.90	-1.84	a

13.2 Release into 4 % acetic acid solution (24h at 22°C)

13.2.1 First release test of Al into 4 % acetic acid solution (24h at 22°C)

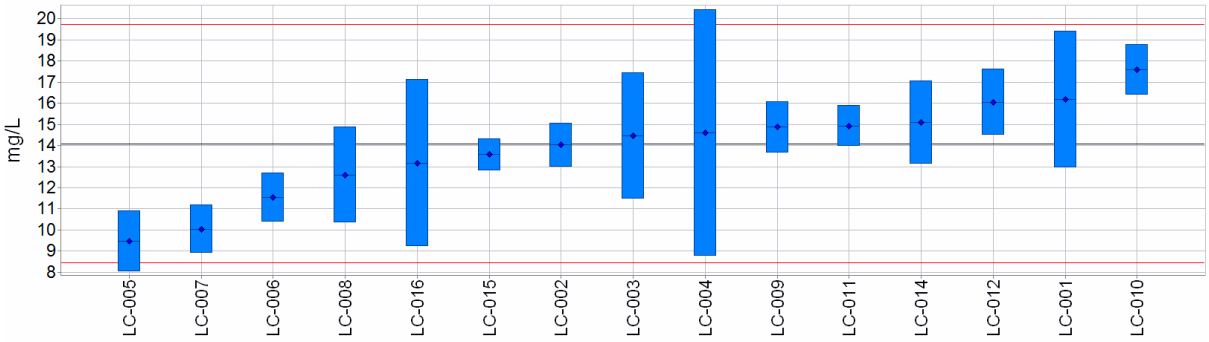

Figure 11: Measurement result range reported by the participants for the first release test of Al into 4 % acetic acid (24h at 22°C). Red lines demonstrate z score = 2 (x_{pt}± 2 σ_{pt}), black line is x_{pt}, points and bars show x_i and U_i

Table 18: Results for the first release test of Al into 4 % acetic acid solution (24h at RT). Assigned range: x_{pt} = 14.084 ± 0.798 [mg L^{-1}]; σ_{pt} = 2.817 [mg L^{-1}]; x_i, U_i, and u_i are in [mg L^{-1}]

Lab code	x_i	U_i	k	u_i	z score	ζ score	u_i $est.$
LC-001	16.167	3.233	2	1.617	0.74	1.25	a
LC-002	14.033	1.033	2	0.517	-0.02	-0.08	a
LC-003	14.462	2.972	2	1.486	0.13	0.25	a
LC-004	14.600	5.840	2	2.920	0.18	0.18	a
LC-005	9.470	1.440	2	0.720	-1.64	-5.61	a
LC-006	11.547	1.150	3	0.383	-0.90	-4.59	a
LC-007	10.050	1.137	2	0.568	-1.43	-5.81	a
LC-008	12.610	2.270	2	1.135	-0.52	-1.23	a
LC-009	14.867	1.200	2	0.600	0.28	1.09	a
LC-010	17.592	1.207	2	0.603	1.25	4.85	a
LC-011	14.933	0.967	2	0.483	0.30	1.36	a
LC-012	16.053	1.570	2	0.785	0.70	2.24	a
LC-014	15.100	1.967	2	0.983	0.36	0.96	a
LC-015	13.567	0.767	2	0.383	-0.18	-0.94	b
LC-016	13.176	3.953	2	1.976	-0.32	-0.45	a

13.2.2 First release test of Co into 4 % acetic acid solution (24h at 22°C)

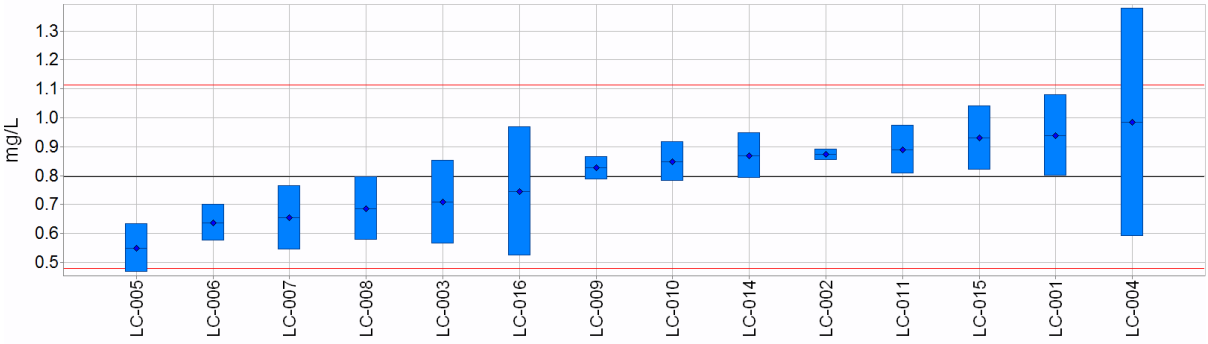

Figure 12: Measurement result range reported by the participants for the first release test of Co into 4 % acetic acid (24h at 22°C). Red lines demonstrate z score = 2 (x_{pt}± 2 σ_{pt}), black line is x_{pt}, points and bars show x_i and U_i

Table 19: Results for the first release test of Co into 4 % acetic acid solution (24h at 22°C). Assigned range: x_{pt} = 0.796 ± 0.058 [mg L^{-1}]; σ_{pt} = 0.159 [mg L^{-1}]; x_i, U_i, and u_i are in [mg L^{-1}]

Lab code	x_i	U_i	k	u_i	z score	ζ score	u_i $est.$
LC-001	0.939	0.141	2	0.070	0.90	1.87	a
LC-002	0.872	0.019	2	0.010	0.48	2.49	b
LC-003	0.709	0.146	2	0.073	-0.55	-1.12	a
LC-004	0.985	0.394	2	0.197	1.18	0.95	a
LC-005	0.550	0.084	2	0.042	-1.55	-4.84	a
LC-006	0.637	0.063	3	0.021	-1.00	-4.44	a
LC-007	0.655	0.112	2	0.056	-0.89	-2.25	a
LC-008	0.686	0.110	2	0.055	-0.69	-1.78	a
LC-009	0.827	0.040	2	0.020	0.19	0.86	b
LC-010	0.849	0.068	2	0.034	0.33	1.18	a
LC-011	0.890	0.084	2	0.042	0.59	1.83	a
LC-012							
LC-014	0.870	0.078	2	0.039	0.46	1.51	a
LC-015	0.930	0.110	2	0.055	0.84	2.15	a
LC-016	0.745	0.224	2	0.112	-0.32	-0.44	a

13.2.3 Second release test of Al into 4 % acetic acid solution (24h at 22°C)

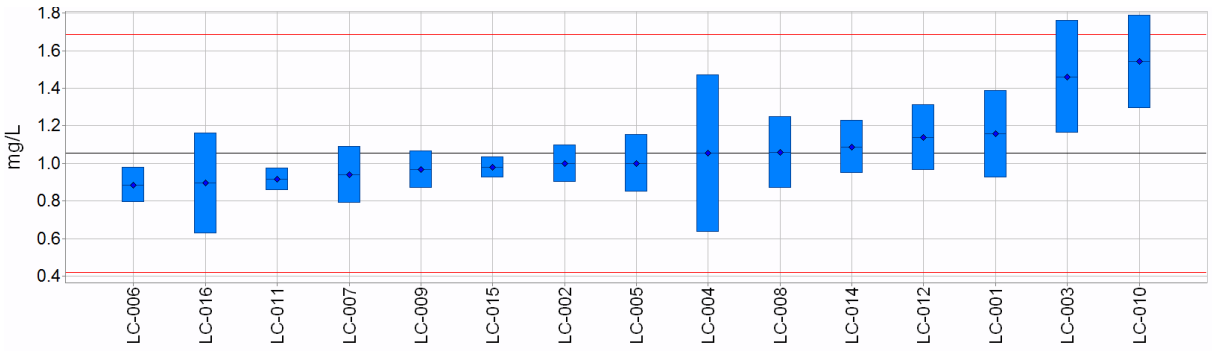

Figure 13: Measurement result range reported by the participants for the second release test of Al into 4 % acetic acid (24h at 22°C). Red lines demonstrate z score = 2 (x_{pt}± 2 σ_{pt}), black line is x_{pt}, points and bars show x_i and U_i

Table 20: Results for the second release test of Al into 4 % acetic acid solution (24h at 22°C). Assigned range: x_{pt} = 1.053 ± 0.079 [mg L^{-1}]; σ_{pt} = 0.316 [mg L^{-1}]; x_i, U_i, and u_i are in [mg L^{-1}]

Lab code	x_i	U_i	k	u_i	z score	ζ score	u_i $est.$
LC-001	1.157	0.231	2	0.116	0.33	0.85	a
LC-002	1.000	0.100	2	0.050	-0.17	-0.83	a
LC-003	1.461	0.301	2	0.150	1.29	2.63	a
LC-004	1.053	0.420	2	0.210	0.00	0.00	a
LC-005	1.001	0.152	2	0.076	-0.17	-0.61	a
LC-006	0.886	0.093	3	0.031	-0.53	-3.32	a
LC-007	0.939	0.152	2	0.076	-0.36	-1.33	a
LC-008	1.057	0.190	2	0.095	0.01	0.04	a
LC-009	0.967	0.100	2	0.050	-0.27	-1.36	a
LC-010	1.543	0.249	2	0.124	1.55	3.75	a
LC-011	0.916	0.058	2	0.029	-0.43	-2.80	b
LC-012	1.138	0.173	2	0.087	0.27	0.89	a
LC-014	1.088	0.141	2	0.071	0.11	0.43	a
LC-015	0.980	0.057	2	0.028	-0.23	-1.50	b
LC-016	0.894	0.268	2	0.134	-0.50	-1.14	a

13.2.4 Second release test of Co into 4 % acetic acid solution (24h at 22°C)

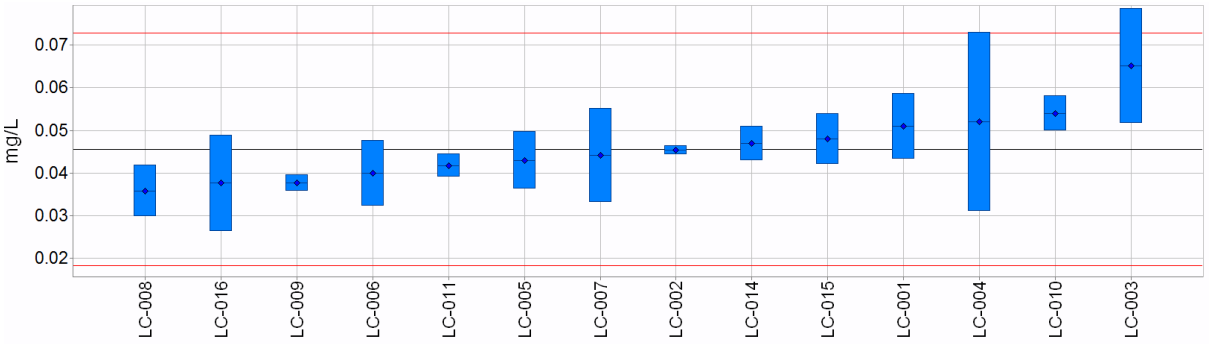

Figure 14: Measurement result range reported by the participants for the second release test of Co into 4 % acetic acid (24h at 22°C). Red lines demonstrate z score = 2 (x_{pt}± 2 σ_{pt}), black line is x_{pt}, points and bars show x_i and U_i

Table 21: Results for the second release test of Co into 4 % acetic acid solution (24h at 22°C). Assigned range: x_{pt} = 0.045 ± 0.004 [mg L^{-1}]; σ_{pt} = 0.014 [mg L^{-1}]; x_i, U_i, and u_i are in [mg L^{-1}]

Lab code	x_i	U_i	k	u_i	z score	ζ score	u_i $est.$
LC-001	0.051	0.008	2	0.004	0.40	1.27	a
LC-002	0.045	0.001	2	0.001	-0.01	-0.07	b
LC-003	0.065	0.013	2	0.007	1.44	2.80	a
LC-004	0.052	0.021	2	0.011	0.48	0.61	a
LC-005	0.043	0.007	2	0.003	-0.18	-0.63	a
LC-006	0.040	0.008	3	0.003	-0.40	-1.66	a
LC-007	0.044	0.011	2	0.006	-0.10	-0.22	a
LC-008	0.036	0.006	2	0.003	-0.71	-2.61	a
LC-009	0.038	0.002	2	0.001	-0.57	-3.39	b
LC-010	0.054	0.004	2	0.002	0.62	2.93	b
LC-011	0.042	0.003	2	0.001	-0.27	-1.50	b
LC-012							
LC-014	0.047	0.004	2	0.002	0.11	0.53	b
LC-015	0.048	0.006	2	0.003	0.18	0.69	a
LC-016	0.038	0.011	2	0.006	-0.57	-1.30	a

13.2.5 Third release test of Al into 4 % acetic acid solution (24h at 22°C)

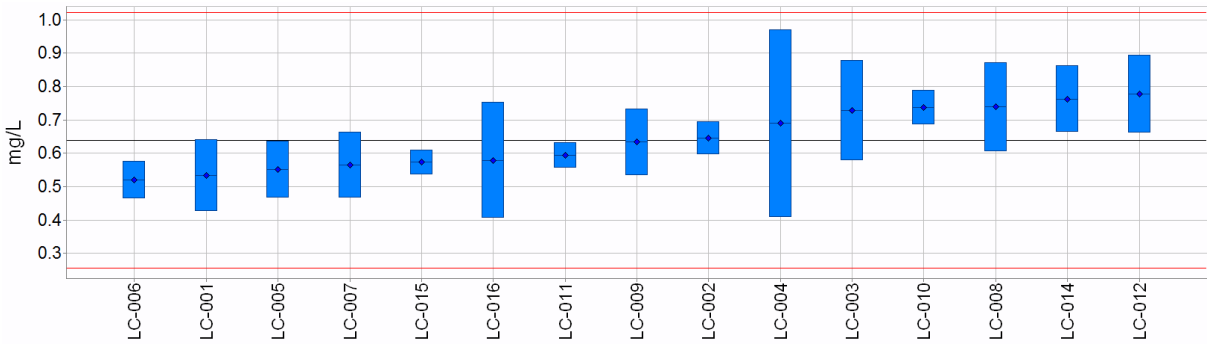

Figure 15: Measurement result range reported by the participants for the third release test of Al into 4 % acetic acid (24h at 22°C). Red lines demonstrate z score = 2 (x_{pt}± 2 σ_{pt}), black line is x_{pt}, points and bars show x_i and U_i

Table 22: Results for the third release test of Al into 4 % acetic acid solution (24h at 22°C). Assigned range: x_{pt} = 0.639 ± 0.038 [mg L^{-1}]; σ_{pt} = 0.192 [mg L^{-1}]; x_i, U_i, and u_i are in [mg L^{-1}]

Lab code	x_i	U_i	k	u_i	z score	ζ score	u_i $est.$
LC-001	0.533	0.107	2	0.053	-0.55	-1.86	a
LC-002	0.645	0.049	2	0.025	0.04	0.22	a
LC-003	0.729	0.150	2	0.075	0.47	1.16	a
LC-004	0.689	0.280	2	0.140	0.26	0.36	a
LC-005	0.552	0.085	2	0.042	-0.45	-1.87	a
LC-006	0.520	0.057	3	0.019	-0.62	-4.43	a
LC-007	0.564	0.098	2	0.049	-0.39	-1.41	a
LC-008	0.739	0.133	2	0.067	0.52	1.45	a
LC-009	0.633	0.100	2	0.050	-0.03	-0.10	a
LC-010	0.738	0.051	2	0.026	0.52	3.11	a
LC-011	0.594	0.038	2	0.019	-0.23	-1.68	a
LC-012	0.777	0.117	2	0.058	0.72	2.26	a
LC-014	0.763	0.099	2	0.050	0.65	2.35	a
LC-015	0.573	0.037	2	0.018	-0.34	-2.47	a
LC-016	0.579	0.174	2	0.087	-0.31	-0.67	a

13.2.6 Third release test of Co into 4 % acetic acid solution (24h at 22°C)

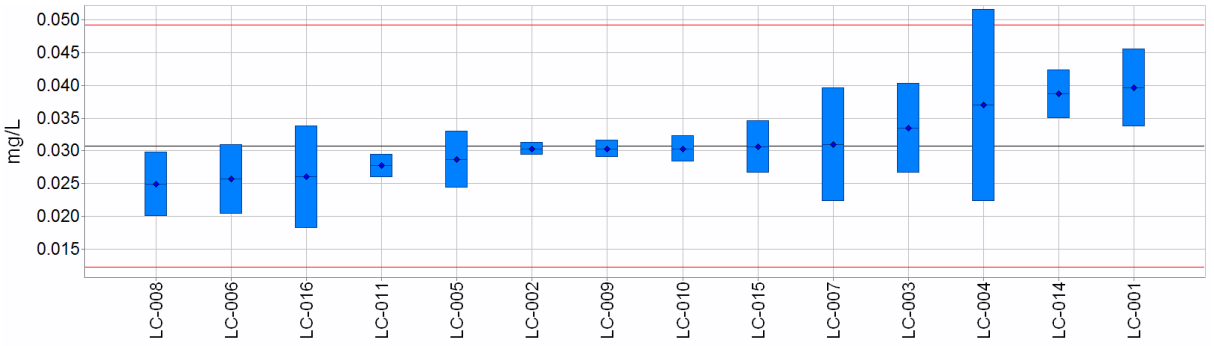

Figure 16: Measurement result range reported by the participants for the third release test of Co into 4 % acetic acid (24h at 22°C). Red lines demonstrate z score = 2 (x_{pt}± 2 σ_{pt}), black line is x_{pt}, points and bars show x_i and U_i

Table 23: Results for the third release test of Co into 4 % acetic acid solution (24h at 22°C). Assigned range: x_{pt} = 0.031 ± 0.002 [mg L^{-1}]; σ_{pt} = 0.009 [mg L^{-1}]; x_i, U_i, and u_i are in [mg L^{-1}]

Lab code	x_i	U_i	k	u_i	z score	ζ score	u_i $est.$
LC-001	0.040	0.006	2	0.003	0.96	2.77	a
LC-002	0.030	0.001	2	0.001	-0.05	-0.34	b
LC-003	0.033	0.007	2	0.003	0.29	0.75	a
LC-004	0.037	0.015	2	0.007	0.68	0.84	a
LC-005	0.029	0.004	2	0.002	-0.23	-0.85	a
LC-006	0.026	0.005	3	0.002	-0.55	-2.39	a
LC-007	0.031	0.009	2	0.004	0.03	0.05	a
LC-008	0.025	0.005	2	0.002	-0.64	-2.16	a
LC-009	0.030	0.001	2	0.001	-0.05	-0.32	b
LC-010	0.030	0.002	2	0.001	-0.05	-0.28	b
LC-011	0.028	0.002	2	0.001	-0.33	-2.07	b
LC-012							
LC-014	0.039	0.004	2	0.002	0.86	3.63	a
LC-015	0.031	0.004	2	0.002	-0.01	-0.04	a
LC-016	0.026	0.008	2	0.004	-0.51	-1.16	a

13.3 Solutions

13.3.1 Reported concentrations of Al in Solution No 1

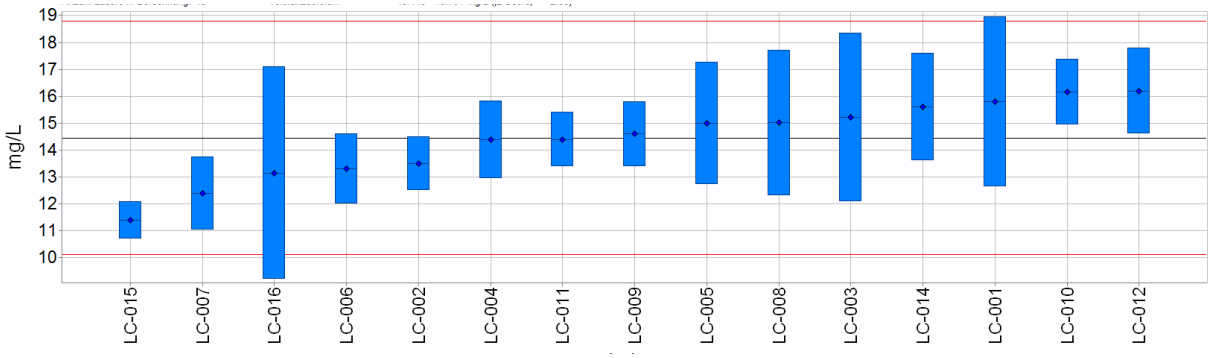

Figure 17: Measurement result range reported by the participants for the concentration of Al in Solution No 1. Red lines demonstrate z score = 2 (x_{pt}± 2 σ_{pt}), black line is x_{pt}, points and bars show x_i and U_i

Table 24: Results reported by the participants for the concentrations of Al in Solution No 1. Assigned range: x_{pt} = 14.447 ± 1.067 [mg L^{-1}]; σ_{pt} = 2.167 [mg L^{-1}]; x_i, U_i, and u_i are in [mg L^{-1}]

Lab code	x_i	U_i	k	u_i	z score	ζ score	u_i $est.$
LC-001	15.800	3.160	2	1.580	0.62	0.81	a
LC-002	13.500	1.000	2	0.500	-0.44	-1.30	a
LC-003	15.210	3.126	2	1.563	0.35	0.46	a
LC-004	14.400	1.440	2	0.720	-0.02	-0.05	a
LC-005	15.000	2.280	2	1.140	0.26	0.44	a
LC-006	13.300	1.300	3	0.433	-0.53	-1.67	a
LC-007	12.400	1.360	2	0.680	-0.94	-2.37	a
LC-008	15.020	2.700	2	1.350	0.26	0.39	a
LC-009	14.600	1.200	2	0.600	0.07	0.19	a
LC-010	16.155	1.220	2	0.610	0.79	2.11	a
LC-011	14.400	1.000	2	0.500	-0.02	-0.06	b
LC-012	16.200	1.590	2	0.795	0.81	1.83	a
LC-014	15.600	2.000	2	1.000	0.53	1.02	a
LC-015	11.400	0.700	2	0.350	-1.41	-4.78	b
LC-016	13.154	3.946	2	1.973	-0.60	-0.63	a

13.3.2 Reported concentrations of Co in Solution No 1

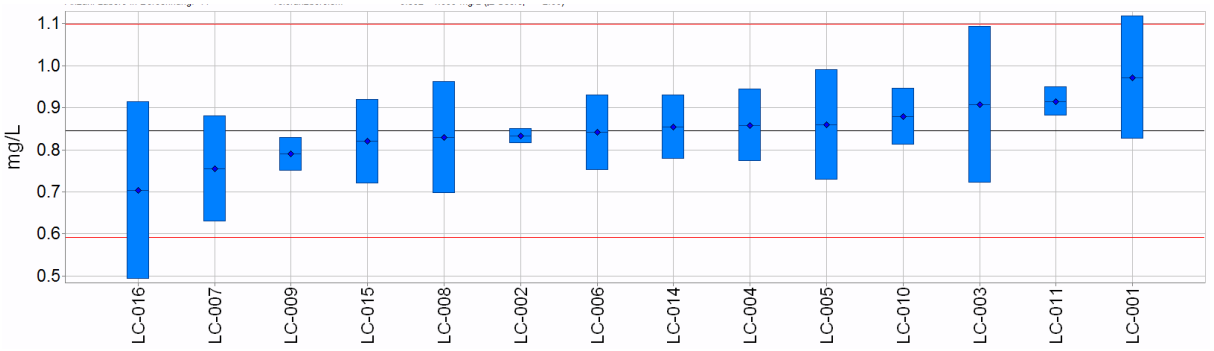

Figure 18: Measurement result range reported by the participants for the concentration of Co in Solution No 1. Red lines demonstrate z score = 2 (x_{pt}± 2 σ_{pt}), black line is x_{pt}, points and bars show x_i and U_i

Table 25: Results reported by the participants for the concentrations of Co in Solution No 1. Assigned range: x_{pt} = 0.845 ± 0.044 [mg L^{-1}]; σ_{pt} = 0.127 [mg L^{-1}]; x_i, U_i, and u_i are in [mg L^{-1}]

Lab code	x_i	U_i	k	u_i	z score	ζ score	u_i $est.$
LC-001	0.972	0.146	2	0.073	1.00	1.67	a
LC-002	0.833	0.018	2	0.009	-0.09	-0.50	b
LC-003	0.907	0.187	2	0.093	0.49	0.65	a
LC-004	0.858	0.086	2	0.043	0.10	0.27	a
LC-005	0.859	0.131	2	0.066	0.11	0.20	a
LC-006	0.841	0.090	3	0.030	-0.03	-0.11	a
LC-007	0.755	0.126	2	0.063	-0.71	-1.35	a
LC-008	0.829	0.133	2	0.067	-0.13	-0.23	a
LC-009	0.790	0.040	2	0.020	-0.43	-1.85	b
LC-010	0.879	0.067	2	0.033	0.27	0.85	a
LC-011	0.915	0.035	2	0.018	0.55	2.48	b
LC-012							
LC-014	0.854	0.077	2	0.039	0.07	0.20	a
LC-015	0.820	0.100	2	0.050	-0.20	-0.46	a
LC-016	0.703	0.211	2	0.105	-1.12	-1.31	a

13.3.3 Reported concentrations of Al in Solution No 2

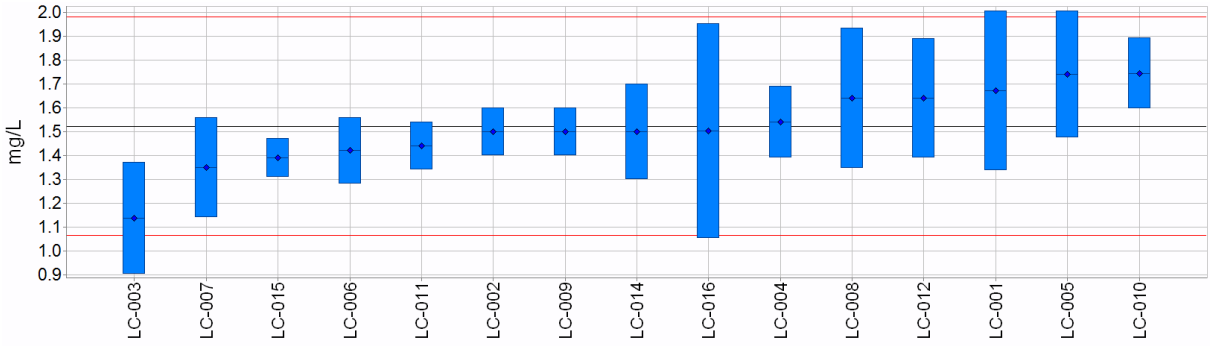

Figure 19: Measurement result range reported by the participants for the concentration of Al in Solution No 2. Red lines demonstrate z score = 2 (x_{pt}± 2 σ_{pt}), black line is x_{pt}, points and bars show x_i and U_i

Table 26: Results reported by the participants for the concentrations of Al in Solution No 2. Assigned range: x_{pt} = 1.522 ± 0.113 [mg L^{-1}]; σ_{pt} = 0.228 [mg L^{-1}]; x_i, U_i, and u_i are in [mg L^{-1}]

Lab code	x_i	U_i	k	u_i	z score	ζ score	u_i $est.$
LC-001	1.670	0.334	2	0.167	0.65	0.84	a
LC-002	1.500	0.100	2	0.050	-0.10	-0.30	b
LC-003	1.137	0.234	2	0.117	-1.69	-2.97	a
LC-004	1.540	0.150	2	0.075	0.08	0.19	a
LC-005	1.740	0.264	2	0.132	0.95	1.52	a
LC-006	1.420	0.140	3	0.047	-0.45	-1.40	a
LC-007	1.350	0.210	2	0.105	-0.75	-1.45	a
LC-008	1.640	0.295	2	0.148	0.51	0.74	a
LC-009	1.500	0.100	2	0.050	-0.10	-0.30	b
LC-010	1.744	0.148	2	0.074	0.97	2.38	a
LC-011	1.440	0.100	2	0.050	-0.36	-1.09	b
LC-012	1.640	0.250	2	0.125	0.51	0.86	a
LC-014	1.500	0.200	2	0.100	-0.10	-0.20	a
LC-015	1.390	0.080	2	0.040	-0.58	-1.92	b
LC-016	1.502	0.451	2	0.225	-0.09	-0.09	a

13.3.4 Reported concentrations of Co in Solution No 2

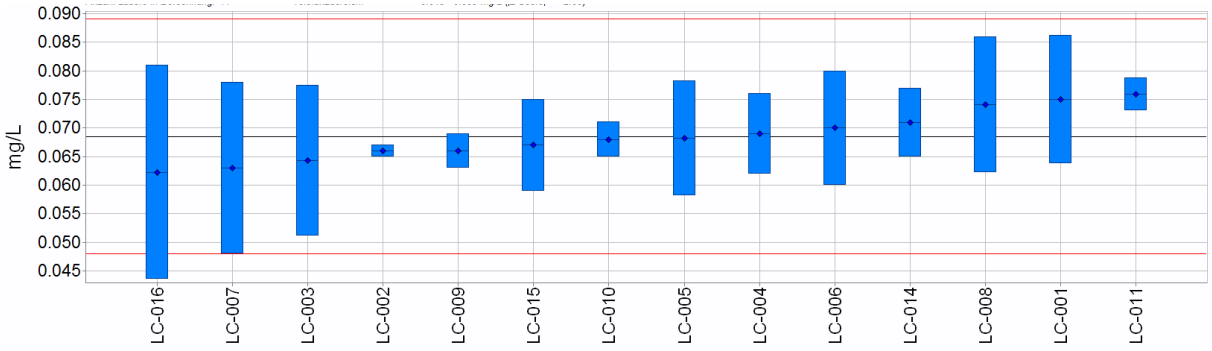

Figure 20: Measurement result range reported by the participants for the concentration of Co in Solution No 2. Red lines demonstrate z score = 2 (x_{pt}± 2 σ_{pt}), black line is x_{pt}, points and bars show x_i and U_i

Table 27: Results reported by the participants for the concentrations of Co in Solution No 2. Assigned range: x_{pt} = 0.068 ± 0.003 [mg L^{-1}]; σ_{pt} = 0.010 [mg L^{-1}]; x_i, U_i, and u_i are in [mg L^{-1}]

Lab code	x_i	U_i	k	u_i	z score	ζ score	u_i $est.$
LC-001	0.075	0.011	2	0.006	0.63	1.12	a
LC-002	0.066	0.001	2	0.001	-0.24	-1.61	b
LC-003	0.064	0.013	2	0.007	-0.41	-0.62	a
LC-004	0.069	0.007	2	0.004	0.05	0.13	a
LC-005	0.068	0.010	2	0.005	-0.03	-0.06	a
LC-006	0.070	0.010	3	0.003	0.15	0.41	a
LC-007	0.063	0.015	2	0.008	-0.53	-0.72	a
LC-008	0.074	0.012	2	0.006	0.55	0.91	a
LC-009	0.066	0.003	2	0.002	-0.24	-1.19	a
LC-010	0.068	0.003	2	0.002	-0.05	-0.23	a
LC-011	0.076	0.003	2	0.001	0.72	3.59	b
LC-012							
LC-014	0.071	0.006	2	0.003	0.24	0.75	a
LC-015	0.067	0.008	2	0.004	-0.15	-0.35	a
LC-016	0.062	0.019	2	0.009	-0.61	-0.66	a

13.3.5 Reported concentrations of Al in Solution No 3

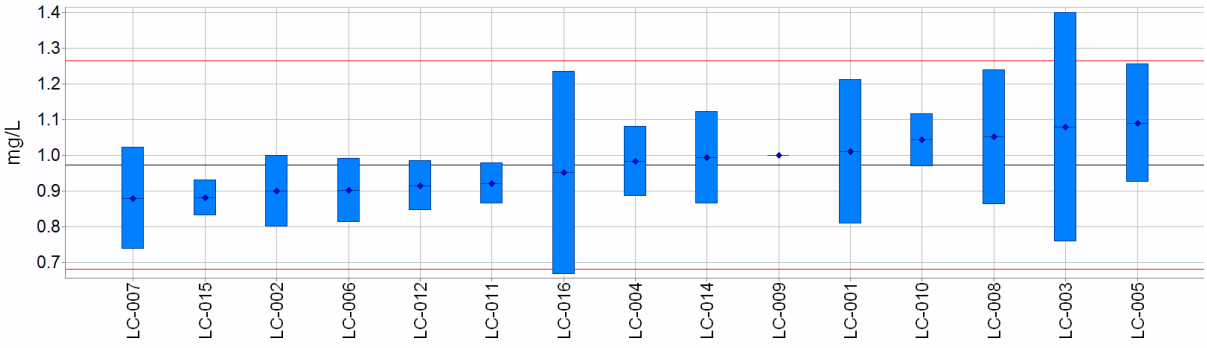

Figure 21: Measurement result range reported by the participants for the concentration of Al in Solution No 3. Red lines demonstrate z score = 2 (x_{pt}± 2 σ_{pt}), black line is x_{pt}, points and bars show x_i and U_i

Table 28: Results reported by the participants for the concentrations of Al in Solution No 3. Assigned range: x_{pt} = 0.973 ± 0.052 [mg L^{-1}]; σ_{pt} = 0.146 [mg L^{-1}]; x_i, U_i, and u_i are in [mg L^{-1}]

Lab code	x_i	U_i	k	u_i	z score	ζ score	u_i $est.$
LC-001	1.010	0.202	2	0.101	0.25	0.35	a
LC-002	0.900	0.100	2	0.050	-0.50	-1.30	a
LC-003	1.079	0.322	2	0.161	0.73	0.65	a
LC-004	0.982	0.098	2	0.049	0.06	0.16	a
LC-005	1.090	0.166	2	0.083	0.80	1.35	a
LC-006	0.902	0.090	3	0.030	-0.49	-1.80	a
LC-007	0.879	0.143	2	0.072	-0.64	-1.24	a
LC-008	1.051	0.189	2	0.095	0.53	0.80	a
LC-009	1.000				0.18		b
LC-010	1.043	0.074	2	0.037	0.48	1.55	a
LC-011	0.921	0.058	2	0.029	-0.36	-1.34	a
LC-012	0.915	0.070	2	0.035	-0.40	-1.34	a
LC-014	0.993	0.129	2	0.065	0.14	0.29	a
LC-015	0.880	0.050	2	0.025	-0.64	-2.59	a
LC-016	0.951	0.285	2	0.143	-0.15	-0.15	a

13.3.6 Reported concentrations of Co in Solution No 3

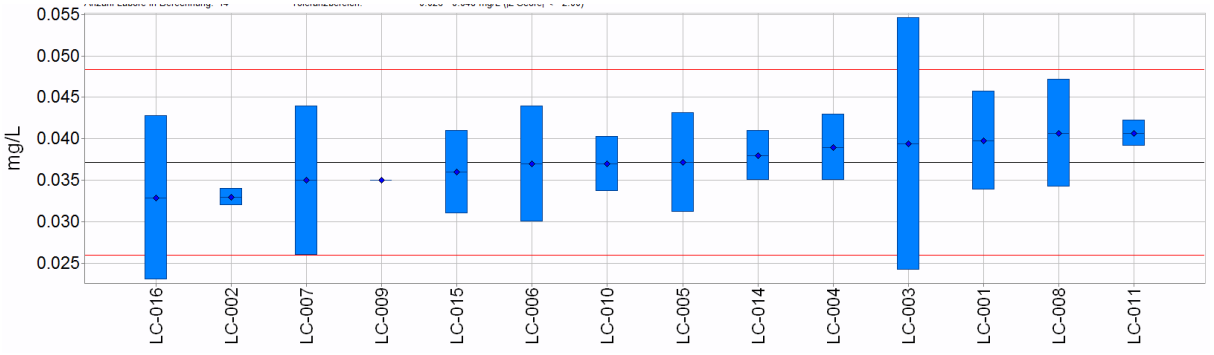

Figure 22: Measurement result range reported by the participants for the concentration of Co in Solution No 3. Red lines demonstrate z score = 2 (x_{pt}± 2 σ_{pt}), black line is x_{pt}, points and bars show x_i and U_i

Table 29: Results reported by the participants for the concentrations of Co in Solution No 3. Assigned range: x_{pt} = 0.037 ± 0.002 [mg L^{-1}]; σ_{pt} = 0.006 [mg L^{-1}]; x_i, U_i, and u_i are in [mg L^{-1}]

Lab code	x_i	U_i	k	u_i	z score	ζ score	u_i $est.$
LC-001	0.040	0.006	2	0.003	0.47	0.81	a
LC-002	0.033	0.001	2	0.001	-0.75	-3.28	b
LC-003	0.039	0.015	2	0.008	0.40	0.29	c
LC-004	0.039	0.004	2	0.002	0.32	0.78	a
LC-005	0.037	0.006	2	0.003	0.00	0.00	a
LC-006	0.037	0.007	3	0.002	-0.03	-0.07	a
LC-007	0.035	0.009	2	0.005	-0.39	-0.47	a
LC-008	0.041	0.007	2	0.003	0.63	1.01	a
LC-009	0.035				-0.39		b
LC-010	0.037	0.003	2	0.002	-0.03	-0.10	a
LC-011	0.041	0.002	2	0.001	0.63	2.46	b
LC-012							
LC-014	0.038	0.003	2	0.002	0.14	0.42	a
LC-015	0.036	0.005	2	0.003	-0.21	-0.43	a
LC-016	0.033	0.010	2	0.005	-0.77	-0.84	a

13.4 Results of the Questionnaire

13.4.1 General Information

Table 30: General Information

Labora-tory code	1. Please identify yourself. You are....	2. Does your laboratory have a quality management system?	if Yes, based on which standards?	if other specify here:	3. Do you provide an uncertainty statement to your customer?
LC-001	OCL	Yes	ISO 17025		sometimes
LC-002	OCL	Yes	ISO 17025		sometimes
LC-003	OCL	Yes	ISO 17025		sometimes
LC-004	OCL	Yes	ISO 17025		always
LC-005	OCL	Yes	ISO 17025		sometimes
LC-006	OCL	Yes	ISO 17025		sometimes
LC-007	OCL	Yes	ISO 17025		always
LC-008	OCL	Yes	ISO 17025		sometimes
LC-009	NRL / EURL	Yes	ISO 17025		sometimes
LC-010	NRL / EURL	Yes	ISO 17025		sometimes
LC-011	OCL	Yes	ISO 17025	ISO 9001, ISO 15189	sometimes
LC-012	NRL / EURL	Yes	ISO 17025		sometimes
LC-014	NRL / EURL	Yes	ISO 17025		always
LC-015	NRL / EURL	Yes	ISO 17025		sometimes
LC-016	NRL / EURL	Yes	ISO 17025		always

OCL: Official Control Laboratory; NRL: National Reference Laboratory

13.4.2 Analytical Method

Table 31: Information on the used analytical methods (Part 1)

Laboratory code	1. Which analytical technique was used for analysis?	if other specify here:	2. Is your method validated/accredited for following conditions?	
			3 % AcOH (2h at 70°C)	4 % AcOH (24h at 22°C)
LC-001	ICP-MS		Accredited method	Accredited method
LC-002	ICP-OES (axial)		Accredited method	Accredited method
LC-003	ICP-MS		Accredited method	Accredited method
LC-004	Other	for aluminium: ICP-OES for cobalt: ICP-MS	Accredited method	Accredited method
LC-005	ICP-MS		Accredited method	Accredited method
LC-006	ICP-OES (axial)		Not validated/accredited	Accredited method
LC-007	ICP-MS		Validated Method	Validated Method
LC-008	GFAAS	and FAAS (Al)	Accredited method	Accredited method
LC-009	ICP-MS		Not validated/accredited	Validated Method
LC-010	ICP-MS		Not validated/accredited	Not validated/accredited
LC-011	ICP-OES (axial)		Not validated/accredited	Not validated/accredited
LC-012	GFAAS		Not validated/accredited	Not validated/accredited
LC-014	ICP-MS		Not validated/accredited	Validated Method
LC-015	ICP-MS		Not validated/accredited	Not validated/accredited
LC-016	ICP-MS		Accredited method	Accredited method

Table 32: Information on the used analytical methods (Part 2)

Labora-tory code	3. How long and how frequently this method is used in your laboratory?			
	3 % AcOH (2h at 70°C)		4 % AcOH (24h at 22°C)	
	year(s)	/ year	year(s)	/ year
LC-001	2-5	1-50	>5	51-250
LC-002	>5	51-250	>5	51-250
LC-003	>5	1-50	>5	1-50
LC-004	1-2	1-50	>5	51-250
LC-005	>5	51-250	>5	51-250
LC-006	<1	Never	>5	1-50
LC-007	>5	1-50	>5	1-50
LC-008	2-5	1-50	>5	51-250
LC-009	<1	1-50	<1	1-50
LC-010	<1	Never	<1	Never
LC-011	>5	51-250	>5	51-250
LC-012	1-2	1-50	Please Select	Never
LC-014	>5	1-50	>5	1-50
LC-015	1-2	51-250	1-2	251-1000
LC-016	>5	1-50	>5	1-50

Table 33: Information on the used analytical methods (Part 3)

Laboratory code	4. Were certified reference materials used for quality control?	if Yes specify here:	5. Please provide LOQs [mg L^{-1}]			
			3 % AcOH (2h at 70°C)		4 % AcOH (24h at 22°C)	
			Al	Co	Al	Co
LC-001	No	but, we participate in interlaboratory comparison tests	0.2	0.002	0.2	0.002
LC-002	No		0.1	0.005	0.1	0.005
LC-003	Yes		0.000065	0.000033	0.00014	0.000079
LC-004	No		0.1	0.0002	0.1	0.0002
LC-005	Yes	Wasser NIST 1640a; Algenpulver (Eignungsprüfung NRL SM0317; 2017)	0.11	0.0008	0.11	0.0008
LC-006	No		0.05	0.005	0.05	0.005
LC-007	Yes	SPS-SW2 (Oberflächenwasser), TMDA 64.3 (Water Lake Ontario)	0.0008	0.00001	0.0008	0.00001
LC-008	No		0.011	0.006	0.011	0.006
LC-009	Yes	High Purity Standard	0.01	0.0005	0.01	0.0005
LC-010	No		0.01	0.0002	0.01	0.0002
LC-011	No		0.004	0.005	0.004	0.005
LC-012	No		0.008	-	0.017	-
LC-014	Yes	SRM 1640a Trace Elements in Natural Water	0.01	0.001	0.01	0.001
LC-015	No		0.033	0.013	0.033	0.013
LC-016	Yes		0.02	0.04	0.02	0.04

Table 34: Information on the used analytical methods (Part 4)

Laboratory code	6. Please enter the method for estimation of measurements uncertainty	if other specify here:	Is the uncertainty of release-step included into the estimation of measurements uncertainty?	7. Were Blank values subtracted?
LC-001	In house validation		Yes	Yes
LC-002	In house validation		No	No
LC-003	Other	DIN ISO 11352	No	Yes
LC-004	GUM		Yes	No
LC-005	In house validation		No	No
LC-006	In house validation		No	No
LC-007	Other	Horwitz	Yes	Yes
LC-008	In house validation		No	No
LC-009	In house validation		No	No
LC-010	NORDTEST		No	Yes
LC-011	NORDTEST		No	Yes
LC-012	In house validation	By the measurement of replicates (precision)	No	Yes
LC-014	In house validation		No	Yes
LC-015	In house validation		No	Yes
LC-016	NORDTEST		Yes	Yes

Table 35: Information on the used analytical methods (Part 5)

Labora-tory code	8. Did you apply any special treatment to the samples pro-vided?	if YES specify here:	9. Did you encounter any problems with sample analy-sis?	if YES specify here:
LC-001	No		No	
LC-002	Yes	Household cleaning	No	
LC-003	Yes	Cleaning with non-acidic diluted detergent at a temperature of about 40°C followed by ultrapure water	No	
LC-004	No		No	
LC-005	No		No	
LC-006	No		No	
LC-007	Yes	see instructions no. 1.	No	
LC-008	No		No	
LC-009	No		No	
LC-010	No		No	
LC-011	Yes	Household cleaning	No	
LC-012	No		No	
LC-014	No		No	
LC-015	Yes	1 in 10 dilution with 2 % HNO3-solution (for Co analysis)	No	
LC-016	No		Yes	Signal drift

13.4.3 Release at 70°C for 2h

13.4.3.1 Preheating of simulant, thermostatic oven and enameled cups

Table 36: Information on the preheating of the food simulant (Part 1)

Laboratory code	1. Food simulant preheated portions	2. Please include the volume [mL]	3. Material type of lab ware used for the preheating of the food simulant		4. Is the lab ware used for pre-heating blank value controlled in terms of element analysis?
				if other specify here:	
LC-001	Single Portion	300	Glas ware		Yes
LC-002	Single Portion	300	Glas ware		No
LC-003	Single Portion	300	Glas ware		Yes
LC-004	Single Portion	300	Glas ware		No
LC-005	Single Portion	300	Glas ware		No
LC-006	Single Portion	300	Glas ware		Yes
LC-007	Multiple Portion	300	Glas ware		Yes
LC-008	Single Portion	2500	Glas ware		Yes
LC-009	Multiple Portion	300	Other (Specify)	PFA flask	Yes
LC-010	Multiple Portion	300	Glas ware		Yes
LC-011	Single Portion	300	Glas ware		No
LC-012	Multiple Portion	300	Glas ware	This preheating phase was performed using a hot plate.	No
LC-014	Single Portion	300	Glas ware		Yes
LC-015	Single Portion	300	Glas ware		Yes
LC-016	Multiple Portion	300	Glas ware	500ml Schott flask with covers	Yes

Table 37: Information on the preheating of the food simulant (Part 2)

Laboratory code	5. Verification of the desired food simulant temperature by:		6. Way of measuring the desired food simulant temperature	
		if non-calibrated or other specify here:		if other specify here:
LC-001	Calibrated thermometer		Inside a separate food simulant portion	
LC-002	Calibrated thermometer	Thermometer of the water bath	Other (please specify)	inside the water bath
LC-003	Calibrated thermometer		Inside a separate food simulant portion	
LC-004	Non-calibrated thermometer*	lab thermometer IP65 LT-101	Inside a separate food simulant portion	
LC-005	Calibrated thermometer		Inside the food simulant portion	
LC-006	Calibrated data logger		Inside a separate food simulant portion	
LC-007	Calibrated thermometer		Inside a separate food simulant portion	
LC-008	Other (please specify)	temperature in preheated food simulant not controlled, portion over night in Oven at 72 °C	Other (please specify)	temperature in preheated food simulant not controlled
LC-009	Calibrated data logger		Inside a separate food simulant portion	
LC-010	Calibrated data logger		Inside the food simulant portion	
LC-011	Calibrated thermometer		Inside the food simulant portion	
LC-012	Calibrated thermometer		Inside a separate food simulant portion	
LC-014	Calibrated data logger		Inside the food simulant portion	
LC-015	Non-calibrated thermometer*	Fluke 50S K/J thermometer	Other (please specify)	Inside a separate food simulant portion at the beginning and at the end of the 2 hours period.
LC-016	Calibrated data logger		Inside a separate food simulant portion	

Table 38: Information on the preheating of the food simulant (Part 3)

Labora-tory code	7. Did you immerse the temperature probe in the food simulant portion during the entire preheating phase OR at the end of the preheating phase?	8. Did you cover the food simulant portion which contained the probe and, if so, how did you do it?	
			specify here:
LC-001	Entire phase	Yes (Please specify)	with clock glass
LC-002	Entire phase	Yes (Please specify)	plastic cap of the glass bottle
LC-003	Entire phase	Yes (Please specify)	clock glas
LC-004	End of preheating	Yes (Please specify)	with glas ware
LC-005	Entire phase	No	
LC-006	Entire phase	Yes (Please specify)	PE-Film
LC-007	Entire phase	Yes (Please specify)	clock glass
LC-008	Please Select	No	temperature in preheated food simulant not controlled
LC-009	Entire phase	Yes (Please specify)	film PVC
LC-010	Entire phase	Yes (Please specify)	Clock glass
LC-011	End of preheating	Yes (Please specify)	glass cap of the glass bottle
LC-012	End of preheating	Yes (Please specify)	Petri dish
LC-014	Entire phase	No	
LC-015	End of preheating	No	
LC-016	End of preheating	Yes (Please specify)	silicone lid

Table 39: Information on the used thermostatic ovens/incubators (Part 1)

Laboratory code	9. Please provide details on the used thermostatic ovens/incubators				
	Model:	Dimensions:	Volume:	Electric power:	Temperature range:
LC-001	Binder FD 53	400x400x330 (mm)	53 l	1200 W	0-300
LC-002	WTC Binder 15115300002020/930364 (oven for the migration! Not the water bath for preheating)	48x60x40 cm (h x w x d)	115 L	1,6 kW	0 - 300 °C
LC-003	VWR DL 56 Prime	B: 395mm, H: 395mm, T: 360mm	56L	1700W	plus 50 bis 300°C
LC-004	Universalschrank UE 400, Memmert	ca. 50 cm x 53 cm x 40 cm	53 L	1400 W	20°C - 220°C
LC-005	memmert UN 110	560 x 480 x 400 mm	108 l	250 V	20 °C - 300 °C
LC-006					
LC-007	Memmert UNP 500	inside: 560 mm x 480 mm x 400 mm	108 l	2000 W	Nenntemp. 220 °C
LC-008	circulating air drying oven 115	55cm/ 55cm/ 42cm	127L	230V	20-199°C
LC-009	BINDER FD 240	Width- Height- Depth internall (800x600x510) mm	240 L	2.7 kW	RT + 5 ºC - 300 ºC
LC-010	Froilabo	interior dimensions: length-400mm, height-390mm, depth-370mm	60L	5A	amb +10C - 250C
LC-011	Memmert ULM 400 (oven for the migration)	46x55x34 cm (h x w x d)	53 L	1,4 kW	30 - 220 °C
LC-012	UFE 500	Chamber - width 560mm; height 480mm; depth 400mm	108 L	2000 W	from 10C above ambient temperatura up to máx temperature 250C
LC-014	MEMMERT	40x30x43cm	53L	1400W	20-220°C
LC-015	Elbanton stainless steel drying cabinet	(1.5 * 1.2 * 0.55)m per compartiment, two compar-timents	2 m^3	380V	50-200°C
LC-016	Binder Trockenschrank FED 240	B*H*T 103cm*82.2cm*74.5cm	240L 80cm*60cm*50cm	2,7kW	5°C-300°C

Table 40: Information on the used thermostatic ovens/incubators (Part 2)

Labora-tory code	9. Please provide details on the used thermostatic ovens/incubators				
	Fan available	Fan operation:	Level of fan operation:	Air exchange:	Intervallic air exchange
LC-001	-	-	-	yes	59 x/h
LC-002	-	-	-	-	-
LC-003	no	no	no	no	no
LC-004	-	-	-	-	-
LC-005	yes	no	n/a	no	n/a
LC-006					
LC-007					
LC-008	yes	yes	/	/	/
LC-009	YES	YES	medium	YES	NO
LC-010	Yes	ventilation valve adjustable	-	-	-
LC-011	Yes	Constant	Not adjustable	-	-
LC-012	yes	yes	High	closed	
LC-014	yes	yes	middle	yes	no
LC-015	yes	speed controlable	at lowest setting	No	no
LC-016		100 %	100 %	11x/h	

Table 41: Information on the used thermostatic ovens/incubators (Part 3)

Laboratory code	10. Temperature and time of thermostatic oven/incubator preheating			11. Verification/control of preheated oven/incubator temperature?	
	Temperature [°C]	Time [min]		If yes:	if non-calibrated or other specify here:
LC-001	75	120	Yes	Calibrated thermometer	
LC-002	75	120	Yes	Calibrated thermometer	
LC-003	74	960	Yes	Calibrated data logger	
LC-004	73	60	Yes	Other (please specify)	not each time; two times a year with a calibrated thermometer
LC-005	70	15	Yes	Other (please specify)	display on oven
LC-006			Please Select	Please Select	
LC-007	72	45	No	Please Select	
LC-008	72	600	Yes	Calibrated data logger	
LC-009	70	120	Yes	Calibrated data logger	
LC-010	72	30	Yes	Calibrated data logger	
LC-011	75	120	Yes	Calibrated thermometer	
LC-012			No	Please Select	
LC-014	72	120	Yes	Other (please specify)	Calibrated temperature probe installed in incubator.
LC-015	70	60	No	Please Select	
LC-016	75	180	Yes	Calibrated data logger	

Table 42: Information on the used thermostatic ovens/incubators (Part 4)

Laboratory code	12. Control of preheated oven/incubator temperature homogeneity			How did you do it (e.g. several probes placed in different spots inside the thermostatic oven/incubator - close to the bottom/ceiling/side walls, in the centre,…)?
		If yes:	if non-calibrated or other specify here:	
LC-001	Yes	Calibrated thermometer		close to the botton, side walls (rigth - left) and centre (-2°C)
LC-002	No	Please Select		
LC-003	Yes	Calibrated thermometer		in the center
LC-004	No	Please Select		in the middle, more at the back side
LC-005	No	Please Select		in the centre
LC-006	Please Select	Please Select		
LC-007	No	Please Select		
LC-008	No	Please Select		
LC-009	No	Please Select	Homogeneity is controled in the calibratrion of the oven/incubator	Several probes placed in different spots and at different heights
LC-010	No	Please Select		-
LC-011	Yes	Calibrated data logger		5 different calibrated thermologers at 5 different places in the oven. One place close to the center in the lower part of the fan-equiped incubator wall. And 4 places close to the walls.
LC-012	No	Please Select		
LC-014	No	Other (please specify)	Homogeneity was not specificaly controlled during this ILC. However, incubator is calibrated annually by accredited calibration laboratory and calibration includes homogeneity testing. 10 measuring probes are placed in the incubator: 8 in the corners and 2 in the "middle part".	
LC-015	No	Please Select		
LC-016	No	Please Select		close in the centre

Table 43: Information on the used thermostatic ovens/incubators (Part 5)

Laboratory code	13. In case your thermostatic oven/incubator is equipped with a fan to enable air circulation inside, did you turn it on during the preheating phase?			14. In case your thermostatic oven/incubator provides the possibility for an exchange of air, did you enable it during the preheating phase?		
		If so, at which level (low/medium/high)?	Did you notice effects on the temperature homogeneity inside the thermostatic oven/incubator?		If so, at which frequency?	Did you notice effects on the temperature constancy inside the oven/incubator?
LC-001	Please Select	Please Select	Please Select	No		Yes
LC-002	Please Select	Please Select	Please Select	Please Select		Please Select
LC-003	Yes	Please Select	Please Select	No		No
LC-004	Please Select	Please Select	Please Select	Please Select		Please Select
LC-005	Please Select	Please Select	Please Select	No		No
LC-006	Please Select	Please Select	Please Select	Please Select		Please Select
LC-007	Yes	Medium	No	Please Select		Please Select
LC-008	Yes	Please Select	No	No		Please Select
LC-009	Yes	Medium	No	Yes		No
LC-010	Yes	Medium	No	Please Select		Please Select
LC-011	Yes	Medium	No	No		Please Select
LC-012	Yes	High	No	No		Please Select
LC-014	Yes	Medium	No	No		Please Select
LC-015	Yes	Low	No	No		Please Select
LC-016	Yes	High	No	Yes	11x/h	No

13.4.3.2 Filling procedure

Table 44: Information on the filling procedure (Part 1)

Laboratory code	15. Temperature and time of test specimen preheating before filling			16. Place of test specimen filling	
	Was the specimen preheated before filling?	Temperature [°C]	Time [min]		If outside, please specify the distance to the oven [m]
LC-001	Yes	72	15	Outside the oven (lab/work bench)	0.5
LC-002	Yes	72	15	Inside the oven	
LC-003	Yes	72	15	Outside the oven (lab/work bench)	3
LC-004	Yes	73	15	Outside the oven (lab/work bench)	0.5
LC-005	Yes	70	10	Inside the oven	
LC-006	Please Select			Please Select	
LC-007	Yes	75	15	Inside the oven	
LC-008	Yes	72	15	Outside the oven (lab/work bench)	0.5
LC-009	Yes	70	15	Inside the oven	
LC-010	Yes	72	15	Outside the oven (lab/work bench)	ca 1.2
LC-011	Yes	72	15	Inside the oven	
LC-012	Yes	72	15	Outside the oven (lab/work bench)	1
LC-014	Yes	72	10	Outside the oven (lab/work bench)	2
LC-015	Yes	70	60	Inside the oven	
LC-016	Yes	75	30	Inside the oven	

Table 45: Information on the filling procedure (Part 2)

Laboratory code	17. Placing of test specimens into the oven		18. Filling time per test specimen (approximately, either inside or outside the oven)	19. In case of filling the specimen inside the oven, please provide the time that the ovens' door was opened [seconds]
		Comment		
LC-001	All at one		45	
LC-002	All at one		5	20
LC-003	All at one		R1: 97, R2: 106, R3: 117	-
LC-004	All at one		90	
LC-005	All at one		10	30
LC-006	Please Select			
LC-007	Sequential with time intervalls (specify)	1.: all first releases with blank and temperature control, 2.: all second releases with blank and temperature control, 3. all third releases with blank and temperature control	3	30
LC-008	All at one		3	/
LC-009	All at one		10	60
LC-010	Sequential with time intervalls (specify)	All cups were filled separately and right after filling sequently placed straight into the oven. Different times were documented and taken out from the oven accordingly.	ca 30-40 depending on the cup	-
LC-011	All at one		5	20
LC-012	All at one		45	
LC-014	Sequential with time intervalls (specify)	10 minute time intervals	10	
LC-015	All at one		10	60
LC-016	All at one		30	60

Table 46: Information on the filling procedure (Part 3)

Laboratory code	20. Covering of test specimens		21. External insulation of the test specimen?	22. Number of persons involved in filling process	23. Please add any other comments regarding the filling procedure
		Comment			
LC-001	Clock glass		Yes	1	
LC-002	Clock glass		No	1	
LC-003	Clock glass		No	2	
LC-004	Clock glass		No	1	
LC-005	Clock glass		No	1	
LC-006	Please Select		Please Select		
LC-007	Clock glass		No	1	
LC-008	Clock glass		No	1	/
LC-009	Silicon lid		No	1	
LC-010	Clock glass	For the cup that contained the probe aluminium foil was used	No	1 person	-
LC-011	Clock glass		No	1	-
LC-012	Petri dish		No	1	
LC-014	Silicon lid		No	One	/
LC-015	Plastic lid		No	one	
LC-016	Silicon lid		No	1	

13.4.3.3 Contact phase

Table 47: Information on the contact phase (Part 1)

Laboratory code	24. Temperature control of the food simulant during release testing			25. Simulant losses during release testing [mL]
		Type of thermometer/data logger used	if non-calibrated or other specify here:	
LC-001	On a separate mode (e.g. glass bottle)	Calibrated thermometer		approx. 5
LC-002	On a separate mode (e.g. glass bottle)	Calibrated thermometer		0
LC-003	On a separate mode (e.g. glass bottle)	Calibrated data logger		5
LC-004	On a separate mode (e.g. glass bottle)	Non-calibrated thermometer*	lab thermometer IP65 LT-101	5
LC-005	In the filled specimen	Non-calibrated thermometer*	digital probe thermometer provided in former ILC by EURL	0
LC-006	Please Select	Please Select		
LC-007	On a separate mode (e.g. glass bottle)	Calibrated thermometer		not observed
LC-008	On a separate mode (e.g. glass bottle)	Calibrated data logger	temperature measured every 15 min	10
LC-009	On a separate mode (e.g. glass bottle)	Calibrated data logger		Between 2 and 5
LC-010	In the filled specimen	Calibrated data logger		10
LC-011	On a separate mode (e.g. glass bottle)	Calibrated thermometer		0
LC-012	On a separate mode (e.g. glass bottle)	Calibrated thermometer		less than 10
LC-014	In the filled specimen	Calibrated data logger		0
LC-015	No control	Please Select		How can you test this?
LC-016	On a separate mode (e.g. glass bottle)	Calibrated data logger		3-5

Table 48: Information on the contact phase (Part 2)

Laboratory code	26. Thermostatic oven program (please specify)		27. Oven temperature verification during release testing	
		specify temperature or gradient here:		if non-calibrated or other specify here:
LC-001	Constant oven temperature		Calibrated thermometer	
LC-002	Constant oven temperature		Calibrated thermometer	Oven-mounted thermometer
LC-003	Constant oven temperature		Calibrated thermometer	
LC-004	Constant oven temperature	73°C	Other (please specify)	not each time; two times a year with a calibrated thermometer
LC-005	Constant oven temperature	oven set to 70 °C	Other (please specify)	display on oven
LC-006	Please Select		Please Select	
LC-007	Constant oven temperature		Calibrated thermometer	
LC-008	Constant oven temperature		Calibrated data logger	temperature measured every 15 min
LC-009	Constant oven temperature	70ºC	Calibrated data logger	
LC-010	Constant oven temperature	72C	Calibrated data logger	
LC-011	Constant oven temperature		Calibrated thermometer	Oven-mounted thermometer
LC-012	Constant oven temperature		Other (please specify)	Checking oven temperature indicator
LC-014	Constant oven temperature	The temperature was lowered from 72°C to 71°C after 15 minutes from the beginning of release test.	Other (please specify)	Calibrated temperature probe. The temperature of the probe is continuously monitored with laboratory temperature control system.
LC-015	Constant oven temperature	70°C	Non-calibrated thermometer*	internal oven sensor
LC-016	Constant oven temperature	72°C	Calibrated data logger	

13.4.4 Release at 22°C for 24 h

13.4.4.1 Preconditioning of simulant, thermostatic oven and enameled cups

Table 49: Information on the preconditioning of the food simulant (Part 1)

Laboratory code	1. Food simulant preconditioning		2. Material type of lab ware used for the preconditioning of the food simulant	
		specify here:		specify here:
LC-001	Yes	production of the simulant (V/V) at room temperature	Glas ware	
LC-002	No	manufacturing at room temperature	Glas ware	
LC-003	Yes		Glas ware	
LC-004	No	it's prepared and stored in a room with about 22 +/- 2 degree	Other (Specify)	PE
LC-005	No		Please Select	
LC-006	Please Select		Please Select	
LC-007	No		Glas ware	
LC-008	No		Glas ware	
LC-009	No		Please Select	
LC-010	No		Glas ware	
LC-011	No	manufacturing at room temperature	Glas ware	
LC-012	Yes	24 hours in a room with temperature control of 23 +/- 2C	Glas ware	
LC-014	No		Please Select	
LC-015	No		PTFE flask	
LC-016	Yes	3-4h 22°C strorage in cooled incubators	Glas ware	500ml Schott flask with covers

Table 50: Information on the preconditioning of the food simulant (Part 2)

Laboratory code	3. Is the lab ware used for preconditioning blank value controlled in terms of element analysis?	4. Did you cover the food simulant portion which contained the probe and, if so, how did you do it?	
			specify here:
LC-001	Yes	No	
LC-002	Yes	Yes (Please specify)	plastic cap of the glass bottle
LC-003	Yes	Yes (Please specify)	With clock glas
LC-004	Yes	Yes (Please specify)	with glas ware
LC-005	No	Please Select	
LC-006	Please Select	Please Select	
LC-007	Yes	Yes (Please specify)	clock glass
LC-008	Yes	No	
LC-009	No	No	
LC-010	Yes	No	
LC-011	Yes	Yes (Please specify)	plastic cap of the glass bottle
LC-012	No	Yes (Please specify)	Petri dish
LC-014	Please Select	Please Select	
LC-015	Yes	No	
LC-016	Yes	Yes (Please specify)	silicone lid

13.4.4.2 Contact phase

Table 51: Information on the contact phase (Part 1)

Laboratory code	5. Did you perform the release test in an oven or on the bench?		6. Temperature control of the food simulant during release testing		
		if other specify here:		Type of thermometer/data logger used	if non-calibrated or other specify here:
LC-001	Bench		On a separate mode (e.g. glass bottle)	Calibrated thermometer	
LC-002	Bench		No control	Please Select	
LC-003	Bench		On a separate mode (e.g. glass bottle)	Calibrated thermometer	
LC-004	Bench		No control	Please Select	
LC-005	Bench		No control	Please Select	
LC-006	Please Select		Please Select	Please Select	
LC-007	Oven		No control	Please Select	
LC-008	Other	in a cupboard	No control	Please Select	room temperature in the cupboard between 22 and 24 °C
LC-009	Oven		On a separate mode (e.g. glass bottle)	Calibrated data logger	
LC-010	Bench		No control	Please Select	
LC-011	Oven		On a separate mode (e.g. glass bottle)	Calibrated thermometer	
LC-012	Bench		No control	Please Select	
LC-014	Oven		In the filled specimen	Calibrated data logger	
LC-015	Oven		On a separate mode (e.g. glass bottle)	Non-calibrated thermometer*	Fluke 1502A thermometer
LC-016	Other	cooled incubators with compressor	On a separate mode (e.g. glass bottle)	Calibrated data logger	

Table 52: Information on the contact phase (Part 2)

Laboratory code	7. Simulant losses during release testing	8. Temperature verification during release testing	if non-calibrated or other specify here:
LC-001	0.1	Calibrated thermometer	
LC-002	0	Please Select	
LC-003	Cup1R1: 5ml, Cup3R3: 2mL, sonst keine Verluste	Calibrated data logger	
LC-004	<5	Calibrated thermometer	
LC-005	0	Non-calibrated thermometer*	
LC-006		Please Select	
LC-007	not observed	Please Select	
LC-008	no control	Other (please specify)	room temperature in the cupboard between 22 and 24 °C
LC-009	0	Calibrated data logger	
LC-010	-	Non-calibrated data logger*	Ventilation data logger for room temperature measuring was monitored during migration process.
LC-011	0	Calibrated thermometer	
LC-012	0	Other (please specify)	The test was performed in a room with temperature control of 23 +/- 2C; the temperature is check once a week with calibrated thermometers.
LC-014	0	Other (please specify)	Calibrated temperature probe. The temperature of the probe is continuously monitored with laboratory temperature control system.
LC-015	not measured	Non-calibrated thermometer*	Fluke 1502A thermometer
LC-016	1-2	Calibrated data logger	

13.4.5 Temperature of the food simulant during the release at 70°C for 2h

Table 53: Temperature of the food simulant during the 1st release at 70°C for 2h

Time [min]:	0	5	10	15	20	25	30	35	40	45	50	55	60	65	70	75	80	85	90	95	100	105	110	115	120
Lab code	Temperature [°C]																								
LC-001	69	69	70	70	70	70	70	70	70	70	70	70	70	70	70	70	70	70	70	70	70	70	70	70	70
LC-002	71.5				70.4				69.9				70				70.1				70.4				70.4
LC-003	74.8	72.8	71.6	70.9	70.3	70.1	69.8	69.3	69.5	69.2	68.8	68.9	68.6	68.3	68.4	68.2	68.1	68	67.9	67.8	67.8	67.6	67.5	67.2	67.4
LC-004	73.1	72	71.7	71.4	71.2	70.9	70.6	70.3	70.1	69.8	69.7	69.6	69.6	69.3	69.2	69.1	68.9	68.8	68.7	68.6	68.5	68.4	68.3	68.2	68.2
LC-005	75.7	74.7	73.8	73.3	72	71.6	71.1	70.5	70	69.8	69.6	69.3	69.2	68.8	68.7	68.5	68.3	68.3	68.2	68.1	67.9	67.8	67.7	67.6	67.6
LC-006																									
LC-007	72.1	71.6		71.2	70.8					70.2															69.4
LC-008	64.4			65.5			66.4			67			67.5			67.8			67.9			68.1			68.1
LC-009	70.5	70.4	70.9	71.1	71.1	71	70.9	70.9	70.9	70.8	70.8	70.8	70.8	70.6	70.6	70.6	70.6	70.6	70.6	70.5	70.5	70.5	70.5	70.5	70.5
LC-010	70	70	69.9	69.8	69.8	69.8	69.8	69.8	69.8	69.8	69.7	69.7	69.7	69.7	69.7	69.7	69.7	69.7	69.7	69.7	69.7	69.7	69.7	69.7	69.7
LC-011	70.2				70.4				69.8				70				70.2				70.4				70.4
LC-012	70.7	71.6	71.9	71.7	71.4	71.2	71	70.9	70.8	70.7	70.6	70.6	70.5	70.5	70.5	70.4	70.4	70.4	70.4	70.4	70.4	70.4	70.4	70.4	70.4
LC-014	70.3	70.5	70.5	70.6	70.6	70.7	70.6	70.6	70.6	70.5	70.5	70.5	70.5	70.4	70.4	70.4	70.4	70.4	70.4	70.4	70.4	70.3	70.3	70.4	70.4
LC-015	70																								70
LC-016	73.6	73.5	73.5	73.5	73.4	73.4	73.4	73.3	73.3	73.3	73.3	73.2	73.1	73	72.9	72.8	72.7	72.7	72.6	72.6	72.2	72.4	72.4	72.4	72.3

Table 54: Temperature of the food simulant during the 2nd release at 70°C for 2h

Time [min]:	0	5	10	15	20	25	30	35	40	45	50	55	60	65	70	75	80	85	90	95	100	105	110	115	120
Lab code	Temperature [°C]																								
LC-001	71	71	71	71	71	71	71	71	71	71	71	71	71	71	71	71	71	71	71	71	71	71	71	71	71
LC-002	71.6				69.3				68.8				68.7				68.9				68.9				70
LC-003	76.5	74.9	73.8	73.2	72.8	72.3	71.9	71.7	71.4	71.1	70.8	70.7	70.4	70.2	70.1	69.8	69.7	69.5	69.4	69.3	69.2	69.1	69	68.9	68.8
LC-004	72.9	71.7	71.3	70.9	70.6	70.2	69.7	69.3	68.8	69	69	68.8	68.6	68.4	68	67.6	67.1	66.7	66.5	66.3	66.3	66.2	66.2	66.1	66
LC-005	72.8	71.6	71.3	71	70.6	69.8	69.2	68.8	68.5	68.3	68.1	67.9	67.7	67.6	67.4	67.3	66.7	66.5	66.5	66.4	66.4	66.4	66.4	66.3	66.4
LC-006																									
LC-007	72		71.2																	69.9					69.7
LC-008	68.1			67.9			68.1			68.2			68.2			68.3			68.3			68.3			68.4
LC-009	70.8	70.9	71.3	71.5	71.5	71.3	71.2	71.2	71.1	71	71	70.9	70.9	70.9	70.8	70.8	70.8	70.8	70.8	70.6	70.8	70.6	70.6	70.6	70.6
LC-010	69.1	69.3	69.6	69.7	69.9	70	70.1	70.2	70.2	70.3	70.4	70.4	70.5	70.5	70.5	70.6	70.6	70.6	70.6	70.6	70.6	70.7	70.7	70.7	70.7
LC-011	70.4				69.8				69.6				70.2				70				69.8				70.2
LC-012	68.1	68.9	69.4	70.2	70.3	70.4	70.6	70.7	70.7	70.7	70.7	70.8	70.8	70.8	70.8	70.8	70.8	70.8	70.8	70.8	70.9	70.9	70.9	70.9	70.9
LC-014	70.5	70.3	70.5	70.8	71	70.8	70.6	70.4	70.3	70.2	70.1	70	70	69.9	69.8	69.8	69.8	69.7	69.7	69.6	69.6	69.6	69.6	69.6	69.6
LC-015	70																								70
LC-016	72.2	72.1	72.1	72.1	72.1	72.1	72	72	72	72	72	72	72	72	72	72	72	71.9	71.9	71.9	71.9	71.9	71.8	71.6	71.8

Table 55: Temperature of the food simulant during the 3rd release at 70°C for 2h

Time [min]:	0	5	10	15	20	25	30	35	40	45	50	55	60	65	70	75	80	85	90	95	100	105	110	115	120
Lab code	Temperature [°C]																								
LC-001	71	71	71	71	71	71	71	71	71	71	71	71	71	71	71	71	71	71	71	71	71	71	71	71	71
LC-002	72.2				70.7				70.3				69.3				69				68.8				68.7
LC-003	74.1	73.2	72.5	72	71.7	71.4	71.1	70.9	70.7	70.5	70.3	70.1	70	69.8	69.7	69.6	69.5	69.3	69.3	69.2	69.1	69	68.9	68.8	68.8
LC-004	72.8	72	71.7	71.5	71.3	71	70.7	70.3	70.1	69.9	69.7	69.7	69.6	69.5	69.3	69.2	69.1	69	68.8	68.8	68.7	68.6	68.6	68.5	68.4
LC-005	72.3	71.8	71.6	71.4	71.1	69.8	69.7	69.2	68.7	68.4	68	67.9	67.6	67.3	67.3	67.2	67.1	67	66.9	66.8	66.8	66.7	66.7	66.7	66.6
LC-006																									
LC-007	71.7												69.9												69.5
LC-008	68.3			67.9			68.1			68.2			68.3			68.3			68.4			68.4			68.5
LC-009	70.1	70.4	70.9	71	71	71	71	70.9	70.9	70.9	70.8	70.8	70.8	70.8	70.6	70.6	70.6	70.6	70.6	70.6	70.6	70.6	70.6	70.5	70.6
LC-010	69.4	69.9	70.1	70.2	70.3	70.4	70.5	70.6	70.7	70.7	70.8	70.8	70.8	70.8	70.9	70.9	70.9	70.9	70.9	70.9	71	71	71	71	71
LC-011	69.8				70.4				70.2				69.8				69.6				70.2				70.2
LC-012	69.3	70.2	70.5	70.4	70.4	70.1	70.1	70	70	70	70	70	70	70	70	70	70	70	70	70	70	70	70	70	70
LC-014	70.1	70.4	70.4	70.6	70.7	70.6	70.4	70.2	70.1	70	69.9	69.9	69.8	69.7	69.6	69.6	69.6	69.5	69.5	69.5	69.5	69.5	69.4	69.4	69.4
LC-015	70																								70
LC-016	71.8	71.8	71.7	71.7	71.7	71.7	71.7	71.7	71.7	71.7	71.7	71.8	71.8	71.8	71.8	71.8	71.8	71.8	71.8	71.8	71.7	71.8	71.7	71.7	71.7