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Report of an Inter-laboratory Comparison from the European Union Reference Laboratory for Food Contact Materials

*ILC01-2014 – Follow-up ILC01-2013
on Proficiency Testing on Food
Simulant E containing a cocktail of
potential migrant substances*

Juliana Silva Félix and Catherine Simoneau

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Joint Research Centre
Institute for Health and Consumer Protection

Contact information

Catherine Simoneau

Address: Joint Research Centre, Via Enrico Fermi 2749, TP 260, 21027 Ispra (VA) Italy

E-mail: JRC-FCM@ec.europa.eu

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Abstract

This report shows the results of the Interlaboratory Comparison ILC01-2014, a follow-up of the previous ILC01-2013. It is a Proficiency Testing (PT) on food simulant E spiked with potential migrant substances carried out by the European Union Reference Laboratory (EURL) for Food Contact Materials (FCM). Regulation (EU) No 10/2011 establishes food simulant E for testing migration compliance of plastics in contact with dry foodstuffs. The simulant E (Tenax®) was spiked with 7 substances at given concentrations tailored for this exercise, of which 3 were replaced from the previous year. A list containing 10 substances in which 7 were effectively present in the simulant was distributed to the participants. Homogeneity and stability studies were conducted. The main scope of this ILC01-2014 was to evaluate the improvement on the performance of the National Reference Laboratories (NRLs) for specific analysis of potential migrants in Tenax®. The participants should identify and quantify the substances spiked in the food simulant by using the analytical method of their choice. The z-score values, for each substance/laboratory, were obtained by the assigned value calculated with the results reported by the participants (robust mean). The participation of the laboratories in this follow-up on identification and quantification of the substances in food simulant E was very satisfactory regarding the number of received results as well as the significant improvement in the performance of the NRLs.

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1. Summary

Regulation (EU) No 10/2011 [1] established the food simulant E (Tenax[®]) for testing specific migration into dry foodstuffs.

This report shows the results of the follow-up Interlaboratory Comparison Proficiency Testing (PT) ILC01-2014 on the food simulant E containing potential migrant substances carried out by the European Union Reference Laboratory (EURL) for Food Contact Materials (FCM).

In 2013 the EURL-FCM performed a first-time PT on identification and quantification of unknown substances spiked in Tenax[®]. The participation of the National Reference Laboratories (NRLs) in 2013 was satisfactory (79%), in which 23 participants out of 29 invited laboratories, reported results. Regarding the identification of the unknown substances, 48% of the NRLs (11 out of 23) could identify correctly all the 7 substances. In relation to the quantification, 32% of the NRLs (7 out of 22) could quantify all the substances within tolerance limits (z-scores ≤ 2). Consequently, a follow-up for the ILC01-2013 was established with the NRLs.

The outcome was discussed and a follow-up was established in consensus with all NRLs. The main scope of this ILC01-2014 was to evaluate the improvement on the performance of the NRLs for specific analysis of potential migrants in Tenax[®]. It was agreed that 3 substances from the previous exercise ILC01-2013 would be replaced. The participants had to identify and quantify 7 substances spiked in the food simulant, and using the analytical method of their choice, including general instructions provided by the EURL-FCM for the extraction of the substances spiked in Tenax[®] [2].

The test material was prepared by the EURL-FCM and consisted of Tenax[®] spiked with 7 potential migrant substances at one concentration level. For control purposes blank Tenax[®] from the same batch as the spiked one was also sent to the participants. A list containing the name of 10 chemical substances was distributed to the participants, from which 7 would be present in the food simulant. The choice of the substances was based on relevant scientific literature [2-9].

The substances chosen for the follow-up ILC01-2014 were benzophenone (BP), caprolactam (CAP), dibutyl sebacate (DBS), bis(2-ethylhexyl)adipate (DEHA), bis(2-ethylhexyl) phthalate (DEHP), 2,6-diisopropylnaphthalene (DIPN) and 2-ethyl-1-hexanol (ETHX). The concentration of each substance in the food simulant was tailored for the exercise.

The homogeneity and stability studies for the substances in Tenax[®] were performed by the EURL-FCM. There were 36 participants from 28 countries to whom samples were dispatched. Twenty nine laboratories submitted results, of which 23 were NRLs, 5 were Official Control Laboratories (OCLs) and one was the EURL-FCM itself. The participants were invited to report four replicate measurements under repeatability conditions.

The assigned values were obtained as consensus values after applying robust statistics to the results reported by the participants. Laboratory results were rated with z-scores in accordance with ISO 13528 [10]. Reproducibility standard deviations (%) calculated by DIN 38402 A45 [11] for the different substances in Tenax[®] are summarised below. The values obtained in the ILC01-2013 for the substances evaluated in this ILC01-2014 are also shown.

SUBSTANCES	SML (mg/kg) [1]	Assigned value (mg/kg) 2013	Reprod. SD % at the assigned value 2013	Assigned value (mg/kg) 2014	Reprod. SD % at the assigned value 2014
<i>Benzophenone</i>	0.6	0.70	30.0	0.53	36.4
<i>Bis(2-ethylhexyl) adipate</i>	18	8.37	32.5	4.09	22.6
<i>Bis(2-ethylhexyl) phthalate</i>	1.5	not performed	not performed	1.42	30.7
<i>Caprolactam</i>	15 [T]	11.89	37.6	9.59	20.7
<i>Dibutyl sebacate</i>	60 [T]	not performed	not performed	4.64	23.2
<i>2,6-Diisopropylnaphthalene</i>	No SML*	not performed	not performed	1.42	20.1
<i>2-Ethyl-1-hexanol</i>	30	9.24	23.5	7.81	29.6

* Substance not reported in the Positive List [1].

The participation of the laboratories in this follow-up on identification and quantification of BP, CAP, DBS, DEHA, DEHP, DIPN and ETHX in food simulant E was very satisfactory regarding the number of received results as well as the significant improvement in the performance of the NRLs.

2. Introduction

Interlaboratory Comparisons (ILC) are widely used to evaluate the performance of an analytical method, as well as the ability of official control laboratories to deliver results with accuracy to their customers. ILC studies are also an essential element of laboratory quality assurance, which allows individual laboratories to compare their analytical results with those from other laboratories while providing them objective standards to perform against.

Regulation (EC) No 882/2004 of the European Parliament and of the Council [12] stipulated the organization of ILC as one of the principal responsibilities of the European Reference Laboratories (EURLs).

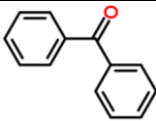
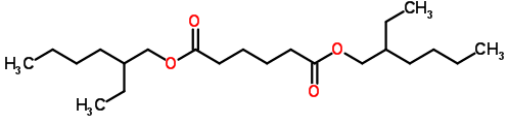
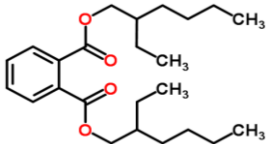
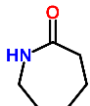
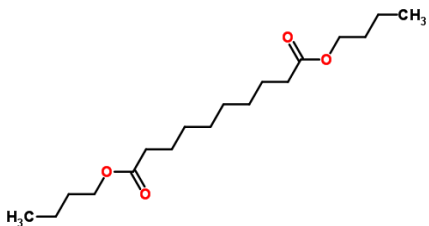
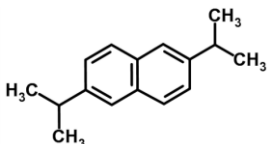
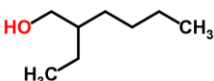
In accordance with the above requirements the EURL-FCM organised in 2014 ILCs for the network of appointed NRLs.

The scope of the ILCs for 2014 were discussed and established on plenaries with all NRLs. It was agreed that the objective of this ILC01-2014 would be a follow-up of the ILC01-2013 to evaluate the improvement of the NRLs performance on identification and quantification of 7 potential migrant substances in food simulant E, of which would be given in a list containing 10 chemical substances (Annex 5).

These substances were chosen because they were reported in the literature as potential migrants from food packaging materials into different food simulants, including food simulant E [2-9]. In addition, these substances were suitable for this ILC01-2014 due to their adequate stability in that particular simulant. Of those, 7 were chosen for spiking as follows: benzophenone (BP), a light stabilizer (UV absorber), caprolactam (CAP) is the monomer of polyamide-6, dibutyl sebacate (DBS), a plasticizer recently identified as migrant coming from set-off [13], bis(2-ethylhexyl) adipate (DEHA) and bis(2-ethylhexyl) phthalate (DEHP) which are also used as plasticizers in plastic food packaging, 2,6-diisopropylnaphthalene (DIPN) used in carbonless copy paper and 2-ethyl-1-hexanol (ETHX) which is used as

monomer or starting substance. Additional characteristics of each substance evaluated in the ILC01-2014 are shown in Table 1.

Table 1. Substances used in the ILC01-2104.

Substance	CAS	SML (mg/kg) [1]	Exercise	Molecular structure
BP	119-61-9	0.6	ILC01-2013 ILC01-2014	
DEHA	103-23-1	18	ILC01-2013 ILC01-2014	
DEHP	117-81-7	1.5	ILC01-2014	
CAP	105-60-2	15 (Total)	ILC01-2013 ILC01-2014	
DBS	109-43-3	60 (Total)	ILC01-2014	
DIPN	24157-81-1	Not in Positive List	ILC01-2014	
ETHX	104-76-7	30	ILC01-2013 ILC01-2014	

The participants were asked to identify and to quantify the potential migrants spiked in simulant E at one concentration level. The laboratories were free to use their own analytical methods, since this ILC was a Proficiency Testing. They were invited to report four replicate measurements in repeatability conditions.

3. Scope

This ILC was a follow-up from the previous exercise ILC01-2013. The scope of this ILC was a PT with the following objectives:

- to evaluate the improvement on the performance of the NRLs for specific analysis of potential migrants in Tenax;

- to assess the NRLs performance on identification of the substances in Tenax;
- to control the NRLs performance on quantification of the substances in Tenax.

The assessment of the measurement results was undertaken on the basis of requirements laid down in international standards and guidelines [10, 14-16].

4. Time frame

The ILC01-2014 was officially launched on the 8th of May 2014. Invitation letters and instructions (Annexes 1-8) were sent to the participants by mail.

The laboratories were invited to fill in a letter of confirmation of their participation (Annex 2).

The final batch of Tenax[®], spiked with the seven substances at one concentration level was prepared by the EURL-FCM in the beginning of May 2014.

The homogeneity of the sample was checked and the sample kit was dispatched to the participants on 12th of May 2014, together with the shipping kit (Annex 3). The participants were asked to fill in the confirmation of receipt of the sample (Annex 4).

The deadline for reporting the results was set to 18th of July 2014.

5. Test material

The sample kit sent to the participants is shown in Table 2. The non-spiked (blank) Tenax[®], from the same batch as the spiked one, was sent to the participants for control purposes.

Table 2. Sample kit for the ILC01-2014

Name	Sample
TNX	Glass bottle containing Tenax [®] spiked with seven substances
Blank TNX	Glass vial containing non-spiked Tenax [®] , from the same batch of the spiked one

5.1. Sample preparation

Preparation and homogenization of the test materials were done by the EURL-FCM according to the procedure described in Annex 9. After spiking and homogenization, circa 20 g portions of the spiked Tenax[®] were filled into glass amber bottles; which were placed in a thermostatic oven at 20°C.

5.2. Homogeneity assessment

The sample batch (TNX) was tested for homogeneity by the EURL-FCM. Ten randomly selected test specimens for the sample TNX were analysed in duplicate to quantify BP, CAP, DBS, DEHA, DEHP, DIPN and ETHX.

Homogeneity was evaluated according to ISO 13528 [10] and IUPAC International Harmonized Protocol [16] using the ProLab Software [17]. The homogeneity results and their statistical evaluation were obtained using Horwitz as target standard deviation (Annex 10).

All test materials showed sufficient homogeneity.

5.3. Stability test

The test samples were monitored for stability by the EURL-FCM for all substances from the homogeneity assessment to the closing of the ILC01-2014. The stability was evaluated as described in ISO GUIDE 35:2006 [18]. Randomly selected specimens for sample TNX were stored at 3 different temperature conditions (4 °C, 20 °C and 40 °C).

The evaluation of data was carried out by performing a linear regression on the experimentally determined concentrations of BP, CAP, DBS, DEHA, DEHP, DIPN and ETHX (mean values) versus time (days). For a stable material, it is expected that the intercept is (within uncertainty) equal to the assigned value, whereas the slope does not differ significantly from zero.

Using the linear regression equation:

$$Y_{(\text{BP, CAP, DBS, DEHA, DEHP, DIPN and ETHX mg/kg})} = b_0 + b_1 X_{(\text{time, days})}$$

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

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

where

- b1 is the slope obtained from the linear regression,
- $t_{0,95,n-2}$ is the Student's t-factor for n-2 degrees of freedom and p = 0.95 (95% level of confidence) and
- s(b₁) is the uncertainty associated with the slope.

This can be calculated as follows:

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

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

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

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

The stability results and their statistical evaluation are given in Annex 11. No significant trend was observed for the test sample at all temperature conditions (4 °C, 20 °C and 40 °C) for the time of the ILC01-2014. It was concluded that the substances were stable for at least fifty days following their preparation.

6. Instructions to the participants

Instructions were given to all participants in the letters that accompanied the sample (Annex 5). Laboratories were asked to report 4 quantitative results for each substance using the unit of measure indicated in the electronic excel file (Annex 8). Participants were free to use their own analytical methods, or the general instructions sent to them (Annex 6) and the SOP [2] validated in the previous exercise on Tenax[®].

7. Evaluation of the results

7.1. General observations

There were 36 participants to whom samples were dispatched (EURL-FCM, 30 NRLs + 5 OCLs from Germany and Spain). The results were submitted by 23 NRLs, 5 OCLs and 1 EURL-FCM.

7.2. Statistical evaluation of results

The statistical evaluation of the results was performed using the ProLab software [16].

7.2.1. Assigned value

A robust mean of the results reported by the participants was chosen as a consensus for the assigned value in the ILC01-2014. The assigned value was evaluated according to Q-method/Hampel estimator (DIN 38402 A45) [11].

7.2.2. Target standard deviation

The value of target standard deviation (σ_p) determines the limits of satisfactory performance in an ILC test. In most cases Horwitz SD [10], a general model commonly used for chemical application, is a good compromise but does not reflect different levels of complexity of a given analytical method. If the target standard deviation is not chosen realistically the interpretation “satisfactory”, “questionable”

and “unsatisfactory” would not be valid. On the other hand, the standard deviation of the reproducibility in collaborative trials can be considered as an appropriate indicator of the best agreement between laboratories.

The σ_p of each substance evaluated in this ILC01-2014 was its reproducibility SD.

7.2.3. Kernel density

Kernel density plots were additionally used to identify multi-modality in the reported values' distributions. Frequently analytical results from a collaborative study are not normally distributed or contain values from different populations giving rise to multiple distribution modes. These modes can be visualised by using Kernel density plots [19, 20]. Kernel density plots were computed by the ProLab software [17] from the analytical results by representing the individual numeric values each as a normalized Gaussian distribution centered on the respective analytical value. The sum of these normal distributions formed then the Kernel density distribution.

7.2.4. z-score

Individual laboratory performance was expressed in terms of z-score in accordance with ISO 13528 [10] and the International Harmonised Protocol [16]. The z-scores compared the participant's deviation from the assigned value with the target standard deviation accepted for the PT:

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

Where:

x_{lab}	is the measurement result (mean value) reported by a participant;
$X_{assigned}$	is the assigned value;
σ_p	is the target standard deviation for proficiency assessment.

The z-score can be interpreted as follow:

$ z \leq 2$	satisfactory result;
$2 < z \leq 3$	questionable result;
$ z > 3$	unsatisfactory result.

In this exercise the z-scores calculated with the reproducibility SD as σ_p were used to assess the performance of the laboratories.

8. Results

8.1. Preliminary considerations

The results were submitted by 29 laboratories, of which 23 were from NRL network, 5 were OCLs (4 from Germany and 1 from Spain) and one was the EURL-FCM itself.

As requested, all the participants reported four replicate results under repeatability conditions.

The participation of the laboratories was regarded as satisfactory (81%) concerning the number of received results (29 out of 36). Regarding exclusively the NRLs, the participation was 77% (23 out of 30).

8.2. Laboratories performance and z-scores

The overall performance of the laboratories regarding the identification of the substances (qualitative analysis) is shown in Table 3. For individual substances, more than 96% of the qualitative results provided were correct. In this follow-up exercise the laboratories have their performance improved when comparing to the identification reported in the ILC01-2013.

Table 3. Qualitative analysis of the substances spiked in the food simulant E.

ILC01-2013	BP	CAP	DEHA	ETHX	ACPH	DTBP	OCT
Total results	30	30	30	30	30	30	30
Correct	27	24	30	26	22	23	25
% Correct results	90	80	100	87	73	77	83
ILC01-2014	BP	CAP	DEHA	ETHX	DBS	DEHP	DIPN
Total results	29	28	29	29	29	29	29
Correct	28	27	28	27	28	28	28
% Correct results	97	96	97	96	97	97	97

Relating to the identification of all the 7 substances, the global performance was of 76%, i.e. 22 laboratories out of 29 could identify correctly all the substances spiked in the food simulant. Regarding only the NRLs, 78% (18 out of 23 NRLs) identified correctly all the substances, which represented an improvement of 30% compared to the previous ILC01-2013.

Concerning the quantitative analysis, summaries of the results obtained by using ProLab [17] for all the substances with their robust repeatability SD and robust reproducibility SD calculated according to Q-method/Hampel estimator (DIN 38402 A45) [11] as well as assigned values, target standard deviations and z-scores, are given in Tables 4 and 5 and in Figures 1-7.

Additionally, the results sent by each participant for each of the evaluated substances, as well as the characteristics of the analytical method used, can be seen in the Annexes 12-18.

Table 4. Summary of results for the substances spiked in Tenax[®] calculated according to DIN 38402 A45.

ProLab Results	SUBSTANCES						
	<i>BP</i>	<i>CAP</i>	<i>DBS</i>	<i>DEHA</i>	<i>DEHP</i>	<i>DIPN</i>	<i>ETHX</i>
Assigned value = Robust Mean, mg/kg	0.533	9.593	4.641	4.086	1.418	1.418	7.811
Robust Repeatability s.d., mg/kg	0.028	0.448	0.203	0.187	0.104	0.066	0.404
Robust Reproducibility s.d., mg/kg	0.194	1.982	1.075	0.924	0.435	0.287	2.311
Target SD (Reproducibility s.d.), mg/kg	0.194	1.982	1.075	0.924	0.435	0.287	2.311
Target SD (Horwitz), mg/kg	0.094	1.092	0.589	0.529	0.215	0.215	0.917
Rel. target SD (Reproducibility s.d.), %	36.35	20.66	23.17	22.63	30.67	20.21	29.59
Rel. target SD (Horwitz), %	17.58	11.38	12.70	12.94	15.18	15.18	11.74
Rel. Repeatability s.d., %	5.18	4.67	4.37	4.57	7.33	4.64	5.17
Lower limit of tolerance, mg/kg	0.146	5.630	2.490	2.237	0.548	0.845	3.189
Upper limit of tolerance, mg/kg	0.921	13.556	6.792	5.935	2.288	1.992	12.433
Laboratories	28	27	28	28	28	28	27
Test results	112	108	112	112	112	112	108

Table 5. z-Scores obtained using the reproducibility SD as target SD.

Laboratory	ETHX	DIPN	BP	DEHP	CAP	DBS	DEHA
LC0002	1.250	0.791	0.757	-0.237	0.016	0.008	-0.023
LC0004	-0.700	-0.247	3.505	0.464	2.930	0.094	-2.151
LC0005	-0.473	0.416	0.435	0.062	-0.727	0.401	0.659
LC0006	-2.861	-0.456	-1.259	2.045	-0.158	-1.126	-0.574
LC0007	1.883	1.663	-*	17.078	0.161	1.703	2.863
LC0008	-*	-*	0.269	-0.455	-*	1.056	-0.433
LC0010	0.057	-0.116	-0.945	0.085	-0.446	2.278	-0.023
LC0013	0.858	-0.369	0.805	-0.271	0.063	-0.478	-0.307
LC0016	-0.005	0.721	-0.042	-0.329	-*	-1.038	-0.255
LC0017	0.600	-0.229	0.203	0.028	0.282	0.555	0.483
LC0018	0.748	1.838	0.499	-0.639	-0.439	-0.059	0.843
LC0020	0.983	8.893	31.547	19.510	2.128	-*	4.988
LC0025	0.718	-0.142	-0.068	-0.565	-0.113	-0.322	-0.428
LC0031	-0.223	-0.299	0.577	-0.024	-0.413	0.522	0.643
LC0032	-0.978	0.111	-0.107	-0.329	-1.227	-0.224	-*
LC0037	-0.513	0.416	-0.133	0.390	-0.161	0.185	0.288
LC0038	-0.399	-0.081	0.525	0.269	-0.108	1.029	0.713
LC0040	-0.453	-0.779	0.190	0.246	-0.489	-0.319	-1.334
LC0041	-0.009	-0.421	0.074	-0.588	-0.499	-0.510	-0.169
LC0044	-0.143	0.172	0.296	1.172	0.987	-0.089	0.305
LC0047	0.071	-0.064	1.067	0.131	0.798	0.311	0.583
LC0048	1.325	3.380	4.386	1.781	0.667	1.096	1.866
LC0049	-1.556	-3.221	-1.671	-2.028	-2.935	-1.813	-2.230
LC0050	-*	-2.310	-1.217	1.139	-0.727	0.718	0.520
LC0055	-0.464	-0.674	-0.113	-0.559	-0.516	-0.361	-0.150
LC0056	-2.706	-3.988	-1.887	-2.588	-4.232	-3.421	-3.611
LC0059	0.682	-0.003	-0.107	-0.392	3.598	-0.089	-0.342
LC0064	-0.208	-0.822	-1.139	0.401	0.707	-0.650	-0.396
LC0113	0.276	1.245	1.325	-*	0.205	-1.131	0.286

* Values not reported by the participants.

Regarding the quantification for all the seven substances within tolerance limits ($|z| \leq 2$), 19 results out of 29 were satisfactory, which represented a success rate of 66%. This percentage increases to 76% if the z-score values higher than 2 and lower or equal 3 are also taken into account. Concerning only the NRLs, 52% (12 out of 23) could quantify all the substances within tolerance limits. This value is 61% if also the laboratories that reported results for 5-6 substances within tolerance limits are included. If z-scores values $2 < |z| \leq 3$ are used for the calculation, so the % rises to 74%.

An overview about the number of z-score values within the tolerance limits ($|z| \leq 2$) and also z-scores between $2 < |z| \leq 3$, obtained in both PT on Tenax[®], can be seen in Table 6. The delivery of results to a larger number of substances evaluated by each

participant was higher in 2014, as well as an increase in the number of z-scores within the limits of tolerance was observed.

Table 6. Comparison of the number of z-scores obtained in both PTs on Tenax®.

z-Score ILC01-2013	BP	CAP	DEHA	ETHX	ACPH	DTBP	OCT
Total results	25	21	28	23	18	17	23
Number of scores, $2 < z \leq 3$	2	3	1	0	0	1	1
% $2 < z \leq 3$	8	14	4	0	0	6	4
Number of scores, $z \leq 2$	20	18	23	19	17	15	20
% $z \leq 2$	80	86	82	83	94	88	87
Total%	88	100	86	83	94	94	91
z-Score ICL01-2014	BP	CAP	DEHA	ETHX	DBS	DEHP	DIPN
Total results	28	27	28	27	28	28	28
Number of scores, $2 < z \leq 3$	0	3	3	2	1	3	1
% $2 < z \leq 3$	0	11	11	7	4	11	4
Number of scores, $z \leq 2$	25	22	23	25	26	23	23
% $z \leq 2$	89	82	82	93	93	82	82
Total %	89	93	93	100	97	93	86

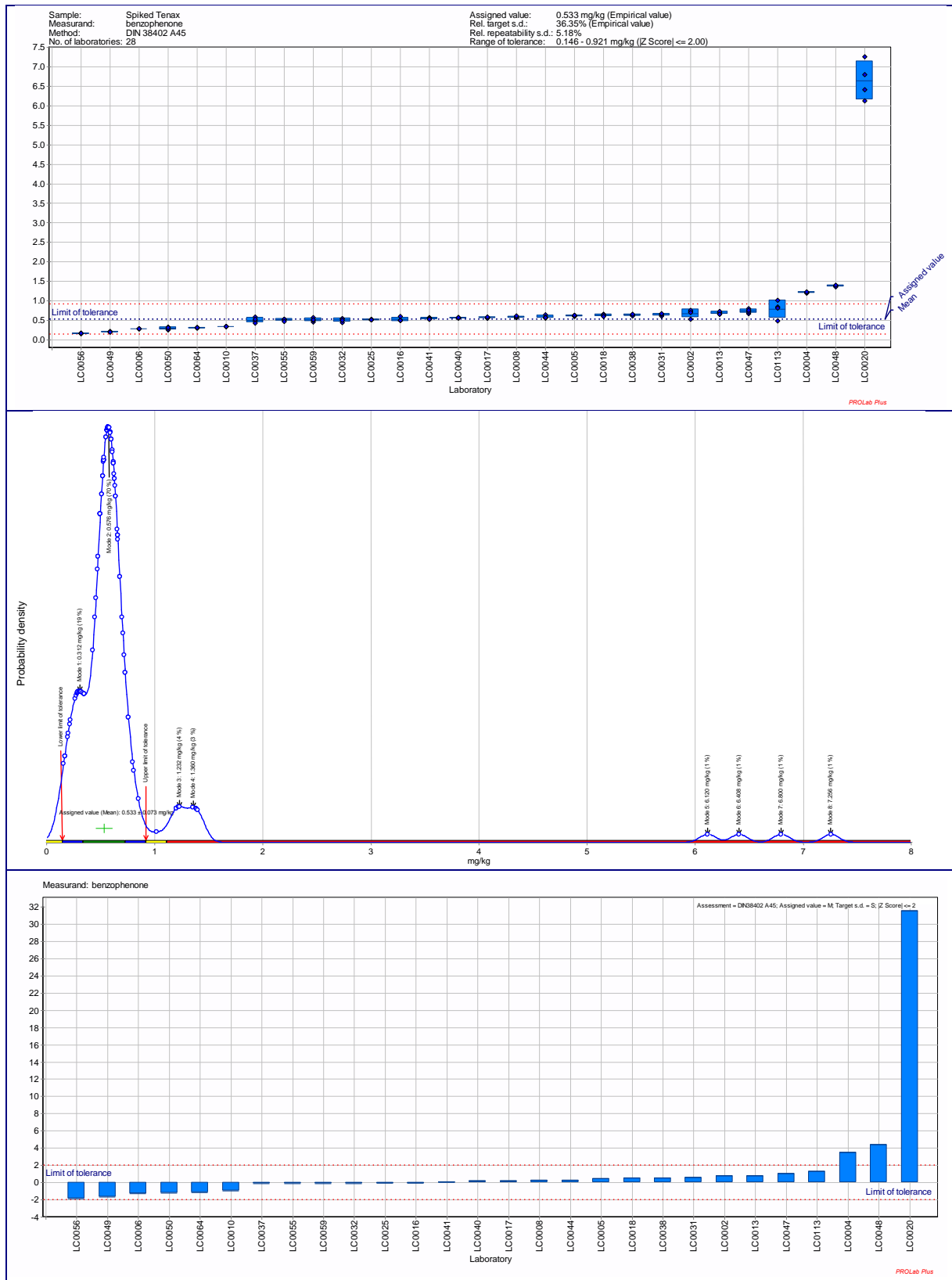


Figure 1. Summary of the laboratories test results for BP with their repeatability SD (1st graph), Kernel density plot (2nd graph) and z-scores (3rd graph).

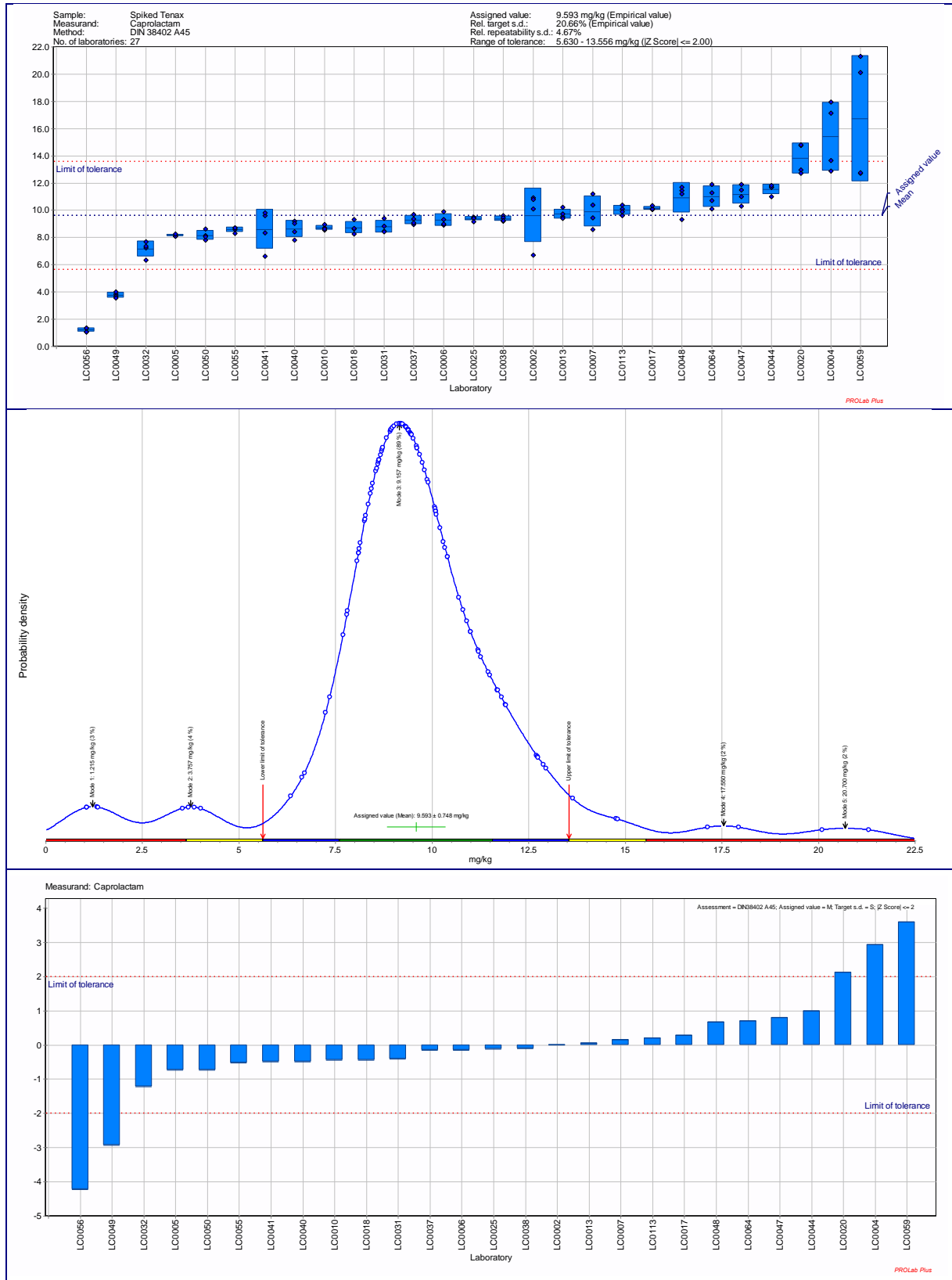


Figure 2. Summary of the laboratories test results for CAP with their repeatability SD (1st graph), Kernel density plot (2nd graph) and z-scores (3rd graph).

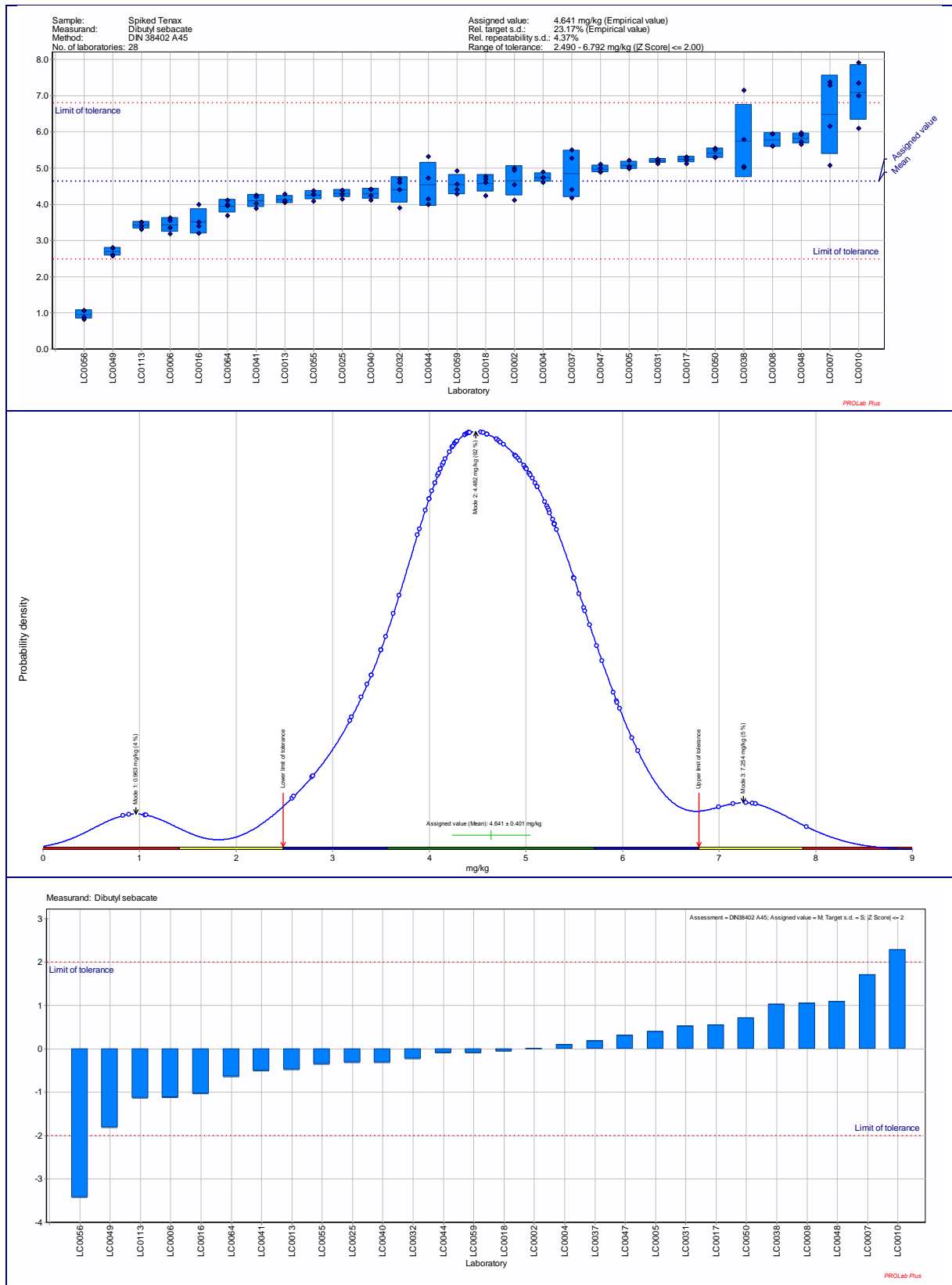


Figure 3. Summary of the laboratories test results for DBS with their repeatability SD (1st graph), Kernel density plot (2nd graph) and z-scores (3rd graph)

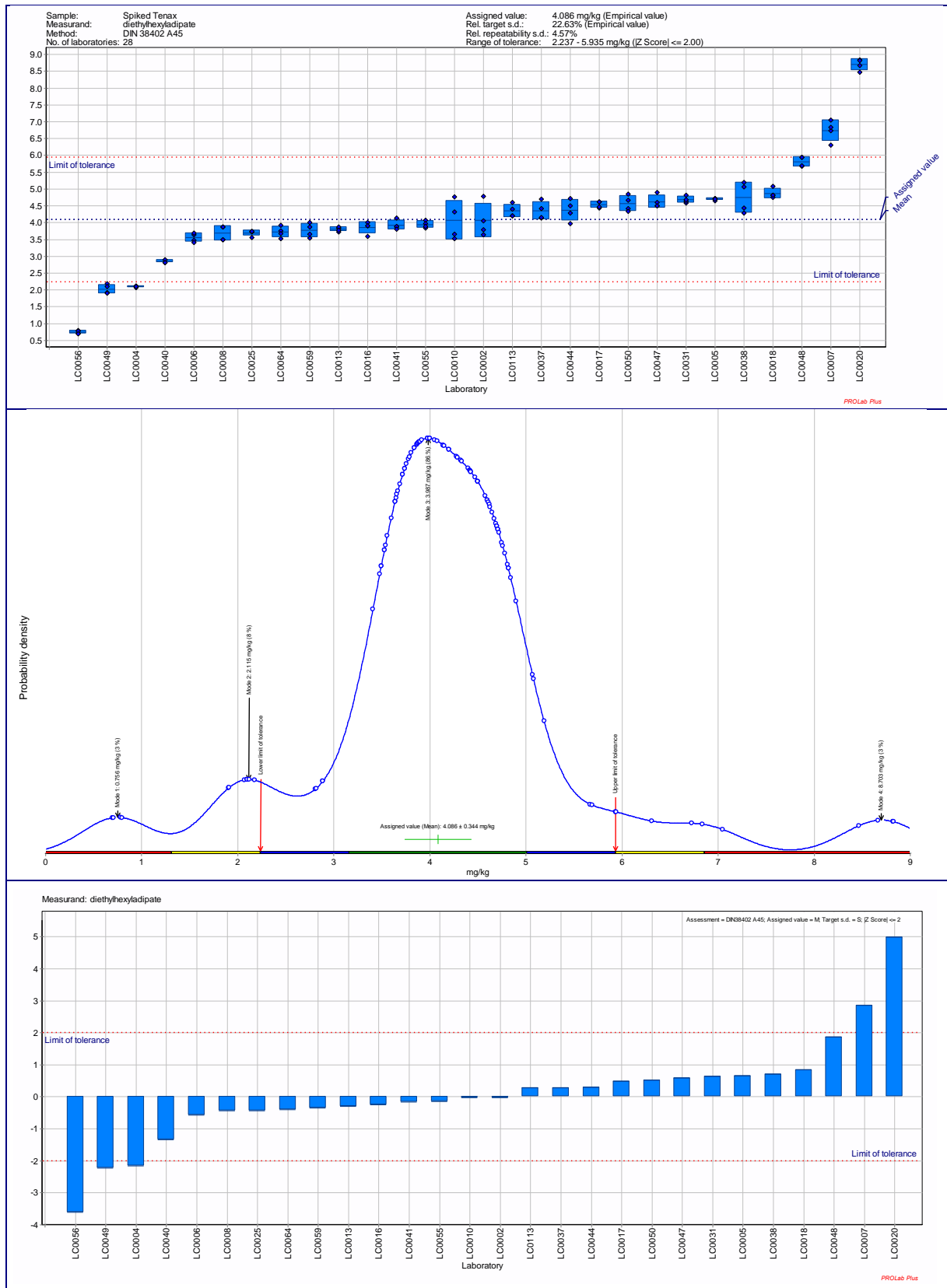


Figure 4. Summary of the laboratories test results for DEHA with their repeatability SD (1st graph), Kernel density plot (2nd graph) and z-scores (3rd graph)

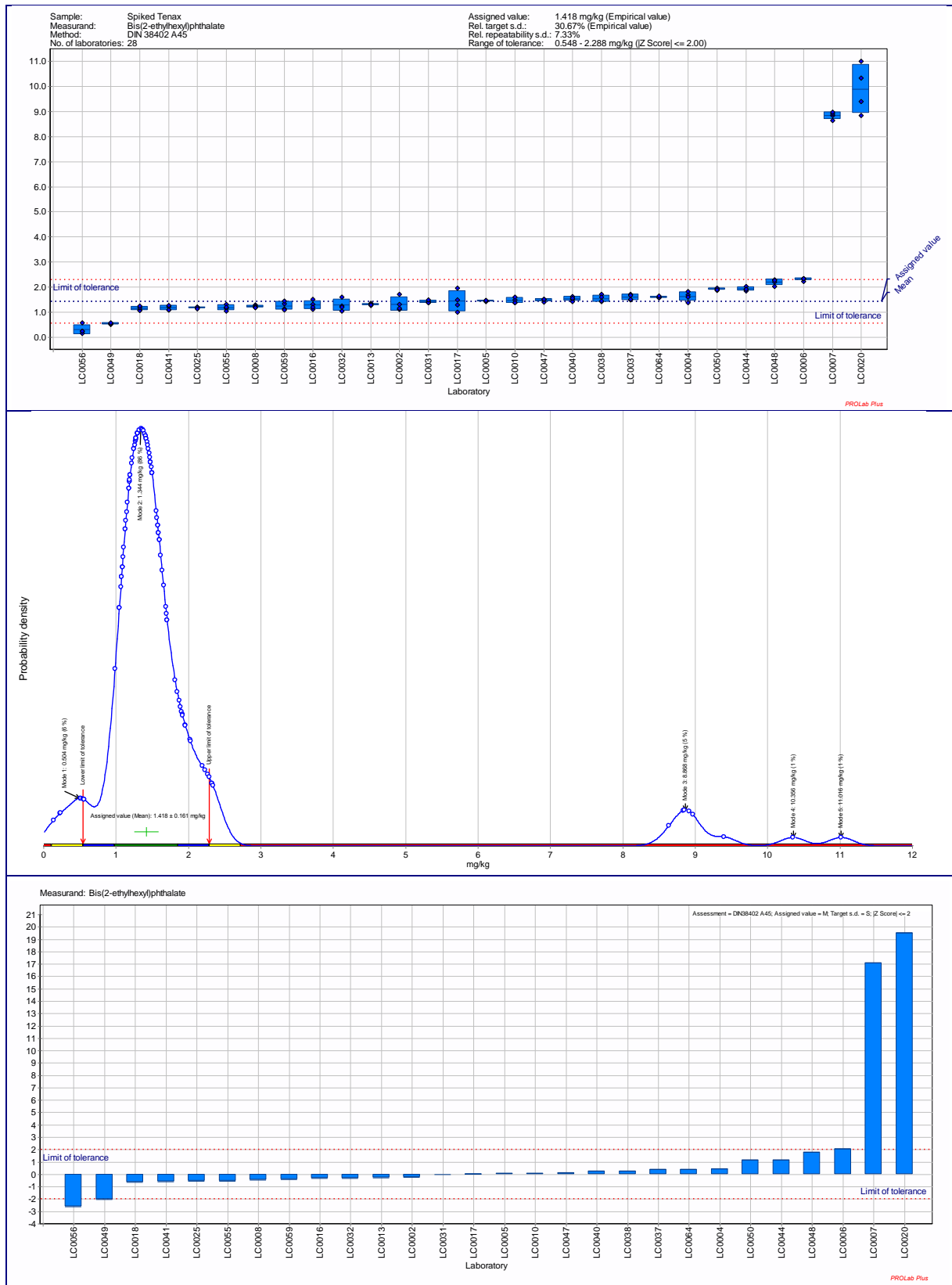


Figure 5. Summary of the laboratories test results for DEHP with their repeatability SD (1st graph), Kernel density plot (2nd graph) and z-scores (3rd graph)

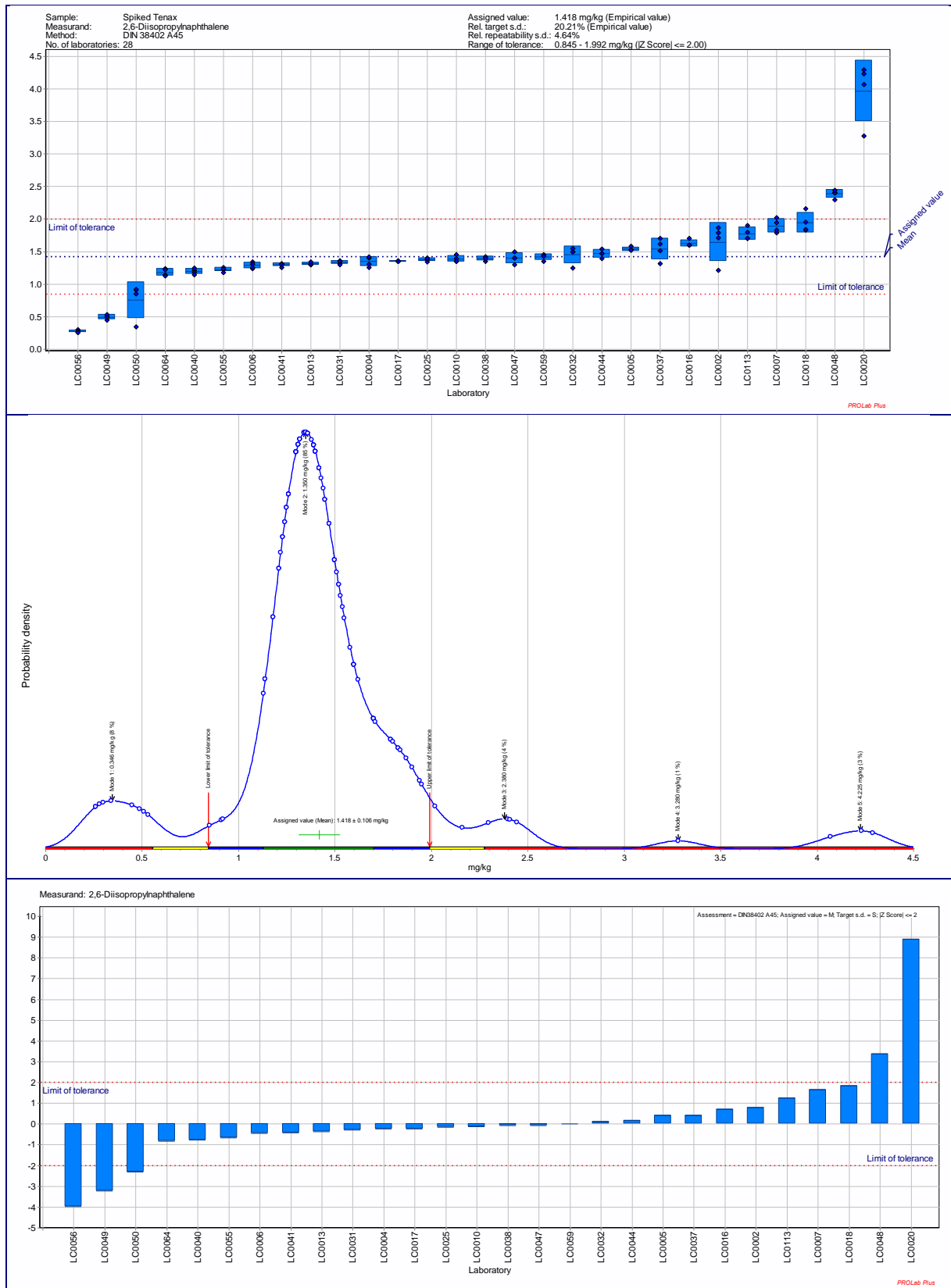


Figure 6. Summary of the laboratories test results for DPN with their repeatability SD (1st graph), Kernel density plot (2nd graph) and z-scores (3rd graph)

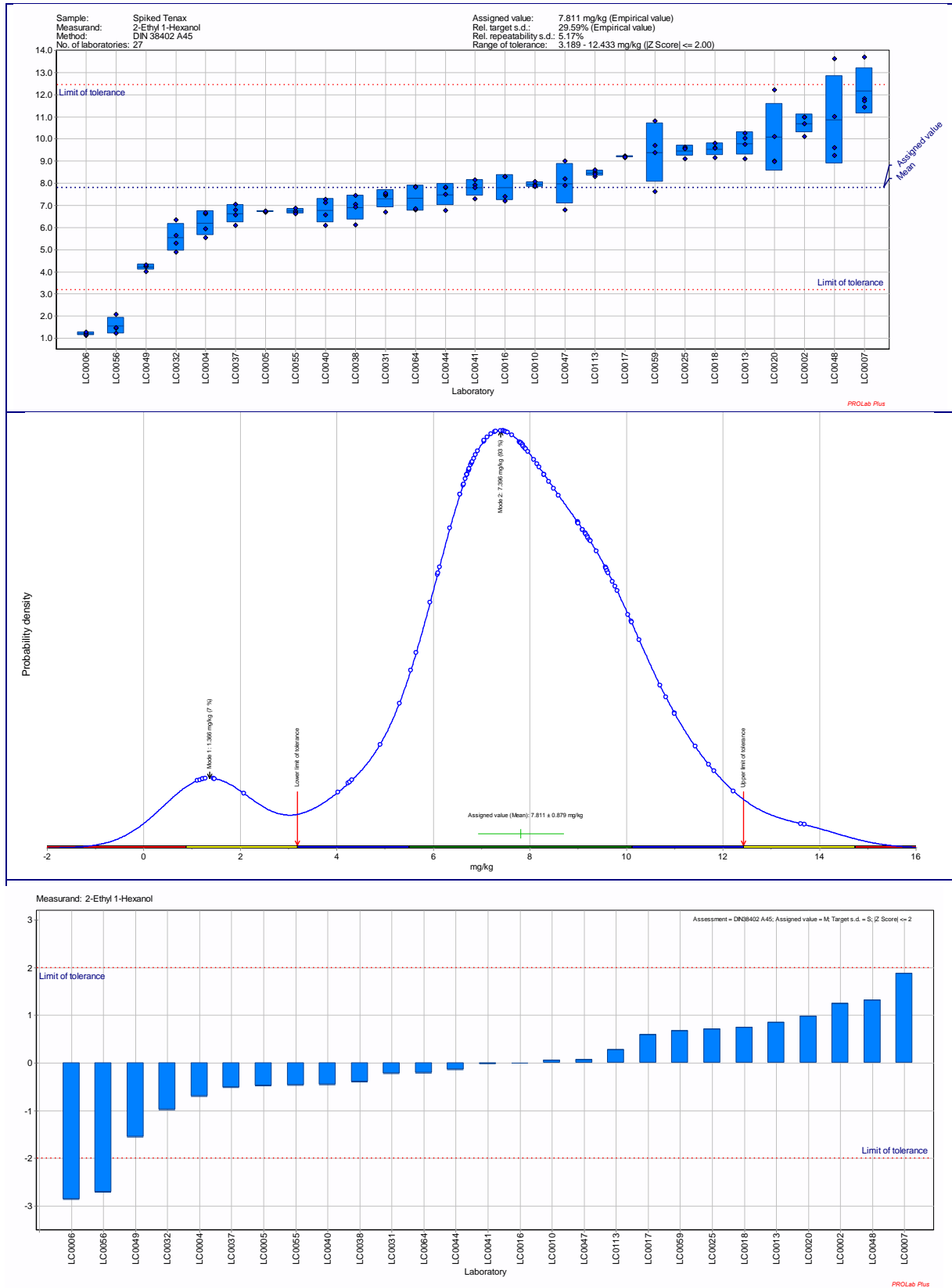


Figure 7. Summary of the laboratories test results for ETHX with their repeatability SD (1st graph), Kernel density plot (2nd graph) and z-scores (3rd graph)

9. Final conclusion

The overall participation in the ILC01-2014 was satisfactory, with 29 laboratories (out of 36 invited laboratories) which reported results (81% of participation). Concerning NRLs, the participation was 77% (23 out of 30 laboratories).

The laboratories' performance concerning the qualitative analysis of the potential migrant substances in the simulant was satisfactory; with more than 76% of correct identification for all the evaluated substances. Regarding the NRLs, 78% of the laboratories (18 out of 23 NRLs) identified correctly all the substances spiked in the Tenax[®], which represented an improvement of 30% compared to the previous ILC01-2013. Taken each individual substance into account, more than 96% of correct qualitative results were provided.

Regarding the quantification for all the seven substances within tolerance limits ($|z| \leq 2$), 19 results out of 29 were satisfactory, which represented a success rate of 66%. This percentage increases to 76% if the z-score values higher than 2 and lower or equal 3 are also taken into account. Concerning only the NRLs, 52% (12 out of 23) could quantify all the substances within tolerance limits. This value is 61% if also the laboratories that reported results for 5-6 substances within tolerance limits are included. If z-scores values $2 < |z| \leq 3$ are used for the calculation, so the % rises to 74%.

This exercise was the follow-up of the first Proficiency Testing carried out at EU level on food simulant E with the scope to evaluate the improvement of the performance of the National Reference Laboratories across the Member States.

The realization of this follow-up was very effective since most laboratories showed significant improvement in the identification and the quantification of the potential migrant substances in food simulant E, which are commonly found in food contact materials.

Acknowledgements

The NRLs and OCLs participating in this exercise - listed below - are kindly acknowledged.

NRLs

AUSTRIA	Austrian Agency for Health and Food Safety (AGES), Institut für Lebensmittelsicherheit, Wien
BELGIUM	Institute of Public Health, ISSP-LP, Bruxelles
BULGARIA	National Centre for Public Health Protection, Sofia
CROATIA	Croatian Institute of Public Health, Zagreb, Croatia
CYPRUS	Laboratory for Control of Food Contact Materials and Control of Toys Ministry of Health, State General Laboratory (SGL), Nicosia
CZECH REPUBLIC	NIPH - NRL for Food Contact Materials and for Articles for children under 3 years old, National Institute of Public Health (SZU), Praha
DENMARK	Department of Food Chemistry, Danish Veterinary and Food Administration, Søborg
ESTONIA	Health Protection Inspectorate - Central Laboratory of Chemistry, Tallinn
FINLAND	Finnish Customs Laboratory, Espoo
FRANCE	SCL Laboratoire de Bordeaux-Pessac, Pessac
GERMANY	Bundesinstitut für Risikobewertung (BfR) (Federal Institute for Risk Assessment), Berlin
GREECE	General Chemical State Laboratory, D' Chemical Service of Athens, Section Laboratory of Articles and Materials in Contact with Foodstuffs, Athens
HUNGARY	Central Agricultural Office, Food and Feed Safety Directorate, Food Toxicology National Reference Laboratory, Budapest
IRELAND	Public Analyst Laboratory - Sir Patrick Duns Hospital, Dublin
ITALY	Istituto Superiore di Sanità, Laboratorio Esposizione e rischio da materiali, c/o Dipartimento ambiente e connessa prevenzione primaria, Roma
LUXEMBOURG	Laboratoire National de Santé, Division du Contrôle des Denrées Alimentaires, Luxembourg
POLAND	Laboratory of Department of Food and Consumer Articles Research, National Institute of Hygiene, Warsaw
PORTUGAL	ESB Escola Superior de Biotecnologia, Universidade Católica Portuguesa, Packaging Department, Porto
SLOVAKIA	National Reference Centre and Laboratory for material and articles intended to come into contact with food, Regional Public Health Authority In Poprad (RUVZ)
SLOVENIA	National Institute of Public Health of Republic of Slovenia, Department of Sanitary Chemistry, Ljubljana
SPAIN	Centro Nacional de Alimentación, Agencia Española de Seguridad Alimentaria y Nutrición (AESAN), Madrid
THE NETHERLANDS	Food and Consumer Product Safety Authority (VWA), Ministry of Economic Affairs, Agriculture and Innovation, Groningen
UNITED KINGDOM	The Food and Environment Research Agency, York

OCLs

GERMANY	Landesamt für Verbraucherschutz Sachsen-Anhalt, Halle
GERMANY	Landesuntersuchungsanstalt für das Gesundheits- und Veterinärwesen Sachsen, Dresden
GERMANY	Chemisches und Veterinäruntersuchungsamt, Fellbach
GERMANY	Bayerisches Landesamt für Gesundheit und Lebensmittelsicherheit, Erlangen
SPAIN	Laboratorio de Salud Pública, Alicante

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11. Annexes

- Annex 1.** Invitation letter to the laboratories to ILC01-2014
- Annex 2.** Letter for confirmation of participation to ILC01-2014
- Annex 3.** Shipping kit letter accompanying the sample TNX1 to ILC01-2014
- Annex 4.** Letter of confirmation of receipt of the sample to ILC01-2014
- Annex 5.** Letter Instructions for the compilation of the results from ILC01-2014
- Annex 6.** General instructions for ILC01-2014
- Annex 7.** Method information used for the participant during the ILC01-2014
- Annex 8.** Form for the compilation of the results in non-electronic format
- Annex 9.** Procedure for the preparation of the spiked food simulant E
- Annex 10.** Results of the homogeneity study
- Annex 11.** Results of the stability study and the statistical evaluation
- Annex 12.** Benzophenone values reported by the participants
- Annex 13.** Bis(2-ethylhexyl) adipate values reported by the participants
- Annex 14.** Bis(2-ethylhexyl) phthalate values reported by the participants
- Annex 15.** Caprolactam values reported by the participants
- Annex 16.** Dibutyl sebacate values reported by the participants
- Annex 17.** 2,6-Diisopropylnaphthalene values reported by the participants
- Annex 18.** 2-Ethyl-1-hexanol values reported by the participants

Annex 1. Invitation letter to the laboratories to ILC01-2014



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



Ispira, 07th May 2014

Dear Madam, Sir

Interlaboratory Comparison (ILC) exercise on food simulant E – ILC01-2014 Follow-up ILC001-2013

On behalf of the EURL for food contact materials, I would like to invite you to participate in the follow-up of ILC exercise for the determination of substances in food simulant E which is due to start by the middle of May. As agreed in the last EURL-NRL FCM plenary, the scope of this ILC exercise is a Proficiency Testing, laboratories are free to use their own methods.

This ILC aims at the extraction and identification/quantification of substances from food simulant E. There is no migration experiment in this exercise.

You will receive a list of ten substances. Seven substances from that list have been spiked into the food simulant E. You need to identify and quantify them. Regarding the quantification, we tried to maintain similar GC-MS response among all the substances (similar peak heights). So, consequently the exercise may not necessarily target only around SMLs. The concentration of the substances will range up to 10-15 mg/kg.

I would like to remind you that it is a duty for you as an NRL-FCM to participate in the ILCs organised by the EURL-FCM since the work programme is decided with your agreement. For this reason we require all of you to actively participate in this exercise. There is no charge for participation.

We have pre-registered everyone, which means we will send test kits to all of you. We however need to receive the **proformat of your participation** for our own administrative purposes. Kindly send back it **by 26th May** to: Juliana Silva Félix (juliana.silva-felix@ec.europa.eu).

The samples will be sent to you in the middle of May. You will find additional information on the form "Shipping kit ILC01-2014" that you will receive by e-mail. You will also receive more detailed instructions for the compilation of the results on the form "Instructions Excel ILC01-2014". The **deadline** for submission of results is **18th July 2014**.

If you have any question, please contact Juliana Silva Félix (juliana.silva-felix@ec.europa.eu).

Sincerely yours,

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

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

Annex 2. Letter for confirmation of participation to ILC01-2014

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



Ispra, 07th May 2014

**Participation to EURL-FCM ILC01-2014
 Interlaboratory comparison exercise on food simulant E
 Follow-up ILC001-2013**

CONFIRMATION OF PARTICIPATION

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

Kindly send back this proformat to: Juliana Silva Félix (juliana.silva-felix@ec.europa.eu) **by the 26th May 2014.**

The samples will be sent to you in the middle of May. You will find additional information in the kit sent. The **deadline** for submission of results is **18th July 2014.**

Sincerely yours,

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

Annex 3. Shipping kit letter accompanying the sample TNX1 to ILC01-2014

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



Ispra, 07th May 2014

**Shipping kit ILC01-2014
exercise on food simulant E - EURL-FCM ILC01-2014
Follow-up ILC001-2013**

Shipping kit - samples

- Sample TNX – One glass bottle containing food simulant E spiked with the unknown substances
- Blank TNX – For control purposes: one glass vial containing the food simulant E not spiked, from the same batch of the spiked one

Shipping kit – documentation (send by email)

- Letter of confirmation of receipt of the sample;
- General instructions to perform the calibration and the extraction
- Letter with instructions for compilation of the results;
- Electronic excel file " ILC01-2014 test results TNX.xls".

Storage

- Sample TNX – could be stored between 4 and 20°C.

Instructions

Use the Excel form "ILC01-2014 test results TNX.xls" to provide your results in 4 replicates.

Sincerely yours,

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

Annex 4. Letter of confirmation of receipt of the sample to ILC01-2014



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



Ispra, 7th May 2014

**Participation to ILC01-2014 on food simulant E
 Follow-up ILC001-2013**

CONFIRMATION OF RECEIPT OF THE SAMPLE

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

Your Name:	
Organization:	
E-mail:	
Phone:	

Any remarks
 Date arrival package
 Signature

Kindly send back this form to: Juliana Silva Félix (juliana.silva-felix@ec.europa.eu)

Sincerely yours,

Catherine Simoneau
 Operating Manager, EU Laboratory for Food Contact Materials
 European Commission, DG-Joint Research Centre
 Institute for Health and Consumer Protection
 Unit CAT, T.P. 260
 Ispra VA 21020 Italy

Annex 5. Letter Instructions for the compilation of the results from ILC01-2014

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



Ispra, 7th May 2014

Instructions Excel ILC01-2014

**Instructions for compilation of the results for ILC01-2014 on food simulant E
 Follow-up ILC001-2013**

DEADLINE: Wednesday, July 18th 2014

You can find in the table below a list of ten substances. **Seven substances** from that list are spiked in the food simulant E. You need to identify and quantify them.

SUBSTANCES	CAS number
1-Octene	111-66-0
Bis(2-ethylhexyl) adipate	103-23-1
Dibutyl sebacate	109-43-3
Benzophenone	119-61-9
Bis(2-ethylhexyl) phthalate	117-81-7
Erucamide	112-84-5
2-Ethyl-1-hexanol	104-76-7
2,6-Diisopropylnaphthalene	24157-81-1
Methyl stearate	112-61-8
Caprolactam	105-60-2

Perform four replicates for the sample TNX and fill your four results in the Excel file (ILC01-2014 test results TNX.xls). The laboratory code is reported in the sample bottle. Send the results back by e-mail to Juliana S. Félix (juliana.silva-felix@ec.europa.eu) **by July 18th 2014.**

Sincerely yours,

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

Annex 6. General instructions for ILC01-2014



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



GENERAL INSTRUCTIONS FOR ILC01-2014

Determination of Substances in Food Simulant E

1 Introduction

These general instructions describe the methods for the extraction procedure with subsequent quantification of the substances from the Tenax[®].

It is important to highlight that this Interlaboratory Comparison (ILC01-2014) is a follow-up of the ILC001-2013.

As this exercise is a Proficiency Test, the participants are free to use the analytical method of their choice.

2 Calibration

Calibration is done by spiking Tenax[®] with appropriate amounts of a multianalyte standard solution. After 1h the substances are extracted using the extraction procedure described in the Section 3.

Pay attention: Use your own Tenax[®] to do the calibration. The BLANK TNX should be used as control in order to have a background of the sample TNX.

2.1 Solutions

2.1.1 Stock solutions

Prepare stock solutions with each analytical standard and appropriate solvent. Calculate the exact concentration of the substances in µg/mL.

2.1.2 Multianalyte standard solution in acetone

This solution is used to spike your own Tenax[®] and carry out the calibration. Pipette appropriate amounts of each stock solution into a volumetric flask and fill the flask up to the mark with acetone. Calculate the exact concentration of the substances in µg/mL.

2.1.3 Acetone extraction solution containing the internal standard

Pipette an appropriate amount of the internal standard stock solution into a volumetric flask and fill the flask up to the mark with acetone. Calculate the exact concentration of the internal standard in µg/mL.

NOTE 1:

All the solutions shall be stored in refrigerator (app. 4°C) (till 2 months).

2.2 Preparation of the calibration curve

- Weight 1.0 g of clean Tenax[®] in 40 mL vials;
- Spike the Tenax[®] with appropriate amounts of the multianalyte standard solution using the digital pipette or syringe;
- Extract the substances from Tenax[®] as reported in Section 3;
- Use the Tenax[®] without spiking as blank and extract it as in Section 3.

3 Extraction of the substances from the spiked Tenax[®]

The substances are extracted twice from the Tenax[®] with an extraction solution containing an internal standard. The extractions carried out with the acetone extraction solution provided recoveries in the range of 80-120% for all the spiked substances.

Procedure steps:

- Transfer 1.0 g of the spiked Tenax[®] into a 40 mL glass vial;
- Add 20 mL of the acetone extract solution (containing the internal standard);
- 2 min in ultrasonic bath (20 °C, 40kHz, 100% power) (or alternatively, manually shaking for 1-2 min);
- Leave it for 5 min without shaking;
- Gently decant the first acetone extract from the Tenax[®] into a new 40 mL glass vial (it is also possible decant the extract through a Büchner funnel with a glass microfiber filter);
- Repeat this extraction procedure using once again 20 mL of the acetone extract solution;
- Collect the second extract in the same 40 mL glass vial;
- Evaporate the acetone extract (approx.. 40 mL) under the nitrogen stream to around 5 mL;
- Filter the extract using a PTFE filter (0.2 µm) into a 2 mL glass vial;
- Inject the concentrated extract into a gas chromatography-mass spectrometry (GC-MS). Quantification is done using an internal standard and in SIM mode.

4. Quantification

4.1 Analysis of calibration standard solutions

Inject the extracts from calibration (2.2) into a GC-MS. Integrate peaks and measure peak area for the substances of interest and also for the internal standard. Construct a calibration function by plotting the analytes concentration against the ratio of the peak area of the analytes and the peak area internal standard in the calibration solutions. Calculate the regression parameters and correlation coefficient.

The calibration curve shall be linear and the correlation coefficient shall be 0,99 or better. If either of the two requirements is not met, fresh standard solutions shall be prepared from the original standard solutions. Analysis of the solutions and construction of the calibration graph have to be repeated.

4.2 Analysis of samples

Inject the sample solutions, prepared in Section 3, under the same chromatographic conditions used for the calibration solutions. Measure the area of the peaks for calculation of the substance concentrations against established in 4.1 regression. Internal standard is used to compensate for the

losses of analytes caused by sample handling, adsorption effects etc.

4.3 Evaluation of data

Calculate of the substance concentration in the spiked Tenax[®] sample in mg/kg:

$$\text{Concentration in Tenax}^{\text{®}} [\text{mg/kg}] = \frac{C}{W}$$

Where:

C is the substance mass in μg derived from the calibration curve, and

W is the weight of sample taken in g.

5 Confirmation

For confirmation of peak identity the retention time and a ratio between target and qualifier ions shall be taken into account.

Annex 7. Method information used for the participant during the ILC01-2014

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

**ILC01-2014**

Dear Madame/Sir

In order to have a better understanding about the different analytical methods used to extract the studied substances from Tenax[®], EURL-FCM need to gather information about the analytical method used.

Therefore we kindly ask you to fill the following table and to send it by e-mail together with your results to: juliana.silva-felix@ec.europa.eu

Cleaning

Food Simulant E cleaning procedure applied:

Sample preparation

Solvents used for extraction:

Extraction procedure:

Internal standard:

Calibration

By spiking food simulant E or standard solution in solvents:

Analytical technique used

Gas chromatography	Liquid chromatography
Analytical Column:	Mobile phase:
Oven temperature programme:	Gradient:
Detector used:	Column:
SIM or SCAN:	Detector:

Other analytical techniques used:

Thank you for your collaboration.

Sincerely yours,

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

Annex 8. Form for the compilation of the results in non-electronic format



FCM EURL-NRL ILC01-2014 on Food Simulant E

TEST RESULTS

Lab Code - LC00.....



Identification and quantification of the substances in the spiked food simulant E

Concentration in the sample TNX (mg/kg)							
No.	Substance name	CAS	Replicate 1	Replicate 2	Replicate 3	Replicate 4	remark
1							
2							
3							
4							
5							
6							
7							

Place and date

.....
Laboratory Manager

.....
Signature

Annex 9. Procedure for the preparation of the spiked food simulant E

1. Washing of the 10 L round bottom flask:

- 10 L mixing round bottom flask was washed in a dish washer, then filled with the deionised water (resistivity 18 M Ω .cm @ 25 °C) and mixed;
- The water was removed;
- The mixing bottle was filled with 0.5 L of pentane (Aldrich, HPLC grade + 99%) and shaken;
- Pentane was removed.

2. Preparation of the spiking solutions:

a) Stock solution of BP, DBS, DEHA, DEHP, DIPN and ETHX prepared in acetone (approx. 1000 mg/L)

- 50 mg of each compound was weighted separately into a 50 mL class A volumetric flask and dissolved in acetone;
- the flask was filled up to the mark with acetone.

b) Stock solution of CAP in ethanol (approx. 1000 mg/L)

- 50 mg of CAP was weighted into a 50 mL class A volumetric flask and dissolved in ethanol;
- the flask was filled up to the mark with ethanol.

c) Spiking solution of BP, CAP, DBS, DEHA, DEHP, DIPN and ETHX in pentane

- 0.6 mL of BP, 10 mL of CAP, 5 mL of DBS, 4.5 mL of DEHA, 1.5 mL of DEHP, 1.4 mL of DIPN and 8 mL of ETHX (from the stock solutions) were pipetting into the 2 L volumetric flask;
- the flask was filled up to the mark with pentane.

3. Preparation of the spiking food simulant E:

- 1 kg of the cleaned simulant was weighted and transferred into the 10 L round bottom flask;
- 2 L of the spiking solution in pentane was add to the simulant;
- The volumetric flask was rinsed by other 2 L of pentane and all the solvent was collected into the round bottom flask;
- The flask was closed and the simulant was mixed continuously for 2.5 hours;
- The flask was opened and the simulant was mixed till the complete evaporation of the pentane (approximately 3 days).
- An amount of ca. 20 g were distributed into amber bottles, which were placed into a thermostatic oven at 20 °C.
- Ten randomly selected test specimens for the sample TNX were tested for homogeneity before being sent to participants.

Annex 10. Results of the homogeneity study.

Analyte	Mean (mg/kg)	Mode s(target)	s(samples) [%]	s(target) [%]	ISO 13528 Check for sufficient homogeneity	Harmonized Protocol Test on significant heterogeneity
BP	0.627	Horwitz	0.000	17.161	OK	OK
CAP	9.616	Horwitz	0.953	11.379	OK	OK
DBS	5.013	Horwitz	2.154	12.551	OK	OK
DEHA	4.287	Horwitz	0.919	12.850	OK	OK
DEHP	1.423	Horwitz	1.533	15.169	OK	OK
DIPN	1.424	Horwitz	0.132	15.169	OK	OK
ETHX	7.445	Horwitz	1.986	11.826	OK	OK

Annex 11. Results of the stability study and the statistical evaluation.

Up to 50 days						
Substance	Temperature	Intercept	Slope	s(b1)	$t(\alpha=0.95, n-2) \cdot s(b1)$	$ b1 < t(\alpha=0.95, n-2) \cdot s(b1)$
BP	4 °C	0.6252	0.0003	0.0003	0.0012	OK
	20 °C	0.6322	-0.0005	0.0002	0.0007	OK
	40 °C	0.6372	-0.0006	0.0003	0.0014	OK
CAP	4 °C	9.5574	-0.0049	0.0015	0.0063	OK
	20 °C	9.6787	-0.0135	0.0034	0.0145	OK
	40 °C	9.6716	-0.0189	0.0078	0.0334	OK
DEHA	4 °C	3.6252	-0.0080	0.0055	0.0176	OK
	20 °C	3.6483	-0.0085	0.0048	0.0152	OK
	40 °C	3.6038	-0.0060	0.0056	0.0179	OK
ETHX	4 °C	5.2199	-0.0093	0.0102	0.0324	OK
	20 °C	4.9503	-0.0030	0.0191	0.0609	OK
	40 °C	5.1936	-0.0282	0.0223	0.0709	OK
DBS	4 °C	5.1499	-0.0121	0.0061	0.0195	OK
	20 °C	5.1963	-0.0123	0.0065	0.0208	OK
	40 °C	5.1552	-0.0093	0.0064	0.0203	OK
DEHP	4 °C	1.7243	-0.0053	0.0020	0.0063	OK
	20 °C	1.7183	-0.0049	0.0035	0.0151	OK
	40 °C	1.6914	-0.0036	0.0024	0.0076	OK
DIPN	4 °C	1.6626	-0.0025	0.0017	0.0055	OK
	20 °C	1.6594	-0.0029	0.0018	0.0058	OK
	40 °C	1.6416	-0.0029	0.0019	0.0061	OK

Annex 12. Benzophenone values reported by the participants.

Lab Code	Concentration (µg/g)				Average	SD	RSD (%)	Extraction	Calibration	Instrumentation	Quantification
	Repetition 1	Repetition 2	Repetition 3	Repetition 4							
LC0002	0.730	0.760	0.700	0.530	0.680	0.103	15.1	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0004	1.200	1.220	1.200	1.230	1.213	0.015	1.2	Hexane	Spiking Tenax	GC-MS/GC-FID	Internal Standard
LC0005	0.600	0.630	0.610	0.630	0.618	0.015	2.4	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0006	0.292	0.293	0.285	0.287	0.289	0.004	1.3	not specified	not specified	HPLC-DAD	not specified
LC0008	0.566	0.571	0.592	0.612	0.585	0.021	3.6	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0010	0.350	0.350	0.350	0.350	0.350	0.000	0.0	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0013	0.662	0.708	0.730	0.657	0.689	0.036	5.1	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0016	0.500	0.500	0.500	0.600	0.525	0.050	9.5	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0017	0.580	0.560	0.560	0.590	0.573	0.015	2.6	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0018	0.660	0.620	0.640	0.600	0.630	0.026	4.1	Acetonitrile	Spiking Tenax	LC-MS/MS	Internal Standard
LC0020	7.260	6.800	6.120	6.410	6.648	0.494	7.4	Acetone	Standard Solution	GC-MS	External Standard
LC0025	0.530	0.520	0.520	0.510	0.520	0.008	1.6	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0031	0.620	0.620	0.660	0.680	0.645	0.030	4.7	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0032	0.450	0.550	0.550	0.500	0.513	0.048	9.3	Acetone	Standard Solution	GC-MS	External Standard
LC0037	0.590	0.480	0.430	0.530	0.508	0.068	13.5	Acetone	Spiking Tenax	LC-UV-VIS PDA	Internal Standard
LC0038	0.627	0.634	0.619	0.660	0.635	0.018	2.8	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0040	0.580	0.570	0.560	0.570	0.570	0.008	1.4	Acetonitrile	Standard Solution	GC-MS	Internal Standard
LC0041	0.530	0.570	0.560	0.530	0.548	0.021	3.8	Hexane	Spiking TNX	GC-MS	Internal Standard
LC0044	0.640	0.591	0.565	0.566	0.591	0.035	5.9	not specified	not specified	not specified	not specified
LC0047	0.680	0.800	0.720	0.760	0.740	0.052	7.0	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0048	1.390	1.386	1.356	1.401	1.383	0.019	1.4	Acetone	Standard Solution	GC-MS	Internal Standard
LC0049	0.222	0.202	0.216	0.197	0.209	0.012	5.6	Acetone	Spiking Tenax	GC-MS/GC-FID	Internal Standard
LC0050	0.334	0.265	0.276	0.314	0.297	0.032	10.9	not specified	not specified	not specified	not specified
LC0055	0.510	0.532	0.529	0.474	0.511	0.027	5.3	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0056	0.160	0.170	0.170	0.170	0.168	0.005	3.0	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0059	0.570	0.460	0.500	0.520	0.513	0.046	8.9	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0064	0.320	0.300	0.310	0.320	0.313	0.010	3.1	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0113	0.850	0.810	0.480	1.020	0.790	0.226	28.6	Acetone	Spiking Tenax	not specified	Internal Standard

Annex 13. Bis(2-ethylhexyl) adipate values reported by the participants.

Lab Code	Concentration (µg/g)				Average	SD	RSD (%)	Extraction	Calibration	Instrumentation	Quantification
	Repetition 1	Repetition 2	Repetition 3	Repetition 4							
LC0002	3.64	4.05	4.78	3.79	4.07	0.51	12.4	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0004	2.10	2.07	2.12	2.10	2.10	0.02	1.0	Hexane	Spiking Tenax	GC-MS/GC-FID	Internal Standard
LC0005	4.72	4.71	4.65	4.70	4.70	0.03	0.7	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0006	3.48	3.64	3.69	3.41	3.56	0.13	3.7	not specified	not specified	GC-FID	not specified
LC0007	6.73	7.05	6.84	6.31	6.73	0.31	4.6	Acetone	Spiking Tenax	GC-FID	Internal Standard
LC0008	3.50	3.50	3.87	3.88	3.69	0.22	5.9	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0010	3.53	4.32	3.65	4.76	4.07	0.58	14.2	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0013	3.84	3.87	3.72	3.78	3.80	0.07	1.8	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0016	4.00	3.90	3.60	3.90	3.85	0.17	4.5	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0017	4.43	4.61	4.62	4.47	4.53	0.10	2.1	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0018	4.82	5.08	4.75	4.81	4.87	0.15	3.0	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0020	8.83	8.82	8.47	8.67	8.70	0.17	1.9	Acetone	Standard Solution	GC-MS	External Standard
LC0025	3.72	3.74	3.74	3.56	3.69	0.09	2.4	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0031	4.58	4.63	4.82	4.69	4.68	0.10	2.2	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0037	4.14	4.15	4.42	4.70	4.35	0.27	6.1	Acetone	Spiking Tenax	GC-MS/GC-FID	Internal Standard
LC0038	4.29	4.43	5.19	5.07	4.74	0.45	9.5	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0040	2.89	2.89	2.82	2.81	2.85	0.04	1.5	Acetonitrile	Standard Solution	GC-MS	Internal Standard
LC0041	3.88	3.89	4.14	3.81	3.93	0.14	3.7	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0044	4.71	3.98	4.28	4.50	4.37	0.31	7.2	not specified	not specified	not specified	not specified
LC0047	4.50	4.60	4.50	4.90	4.63	0.19	4.1	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0048	5.67	5.94	5.69	5.94	5.81	0.15	2.5	Acetone	Standard Solution	GC-MS	Internal Standard
LC0049	1.91	2.18	1.91	2.10	2.02	0.14	6.7	Acetone	Spiking Tenax	GC-MS/GC-FID	Internal Standard
LC0050	4.84	4.42	4.67	4.33	4.57	0.23	5.1	not specified	not specified	not specified	not specified
LC0055	3.84	4.08	3.98	3.89	3.95	0.10	2.6	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0056	0.71	0.80	0.78	0.70	0.75	0.05	6.7	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0059	3.54	3.66	4.00	3.88	3.77	0.21	5.5	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0064	3.76	3.53	3.67	3.92	3.72	0.16	4.4	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0113	4.40	4.20	4.20	4.60	4.35	0.19	4.4	Acetone	Spiking Tenax	not specified	Internal Standard

Annex 14. Bis(2-ethylhexyl) phthalate values reported by the participants.

Lab Code	Concentration ($\mu\text{g/g}$)				Average	SD	RSD (%)	Extraction	Calibration	Instrumentation	Quantification
	Repetition 1	Repetition 2	Repetition 3	Repetition 4							
LC0002	1.71	1.11	1.14	1.30	1.32	0.28	21.0	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0004	1.69	1.82	1.38	1.59	1.62	0.19	11.5	Hexane	Spiking Tenax	GC-MS/GC-FID	Internal Standard
LC0005	1.46	1.44	1.41	1.47	1.45	0.03	1.8	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0006	2.23	2.33	2.33	2.34	2.31	0.05	2.2	not specified	not specified	HPLC-DAD	not specified
LC0007	8.85	8.92	8.97	8.64	8.85	0.15	1.6	Acetone	Spiking Tenax	GC-FID	Internal Standard
LC0008	1.275	1.25	1.193	1.162	1.22	0.05	4.2	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0010	1.37	1.48	1.37	1.60	1.46	0.11	7.5	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0013	1.35	1.27	1.30	1.28	1.30	0.03	2.7	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0016	1.30	1.50	1.10	1.20	1.28	0.17	13.4	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0017	1.96	0.99	1.28	1.49	1.43	0.41	28.6	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0018	1.08	1.07	1.18	1.23	1.14	0.08	6.8	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0020	10.35	11.02	9.40	8.84	9.90	0.97	9.8	Acetone	Standard Solution	GC-MS	External Standard
LC0025	1.19	1.18	1.19	1.13	1.17	0.03	2.4	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0031	1.41	1.37	1.37	1.48	1.41	0.05	3.7	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0032	1.05	1.25	1.20	1.60	1.28	0.23	18.3	Acetone	Standard Solution	GC-MS	External Standard
LC0037	1.48	1.71	1.50	1.66	1.59	0.11	7.2	Acetone	Spiking Tenax	GC-MS/GC-FID	Internal Standard
LC0038	1.42	1.43	1.70	1.59	1.54	0.13	8.5	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0040	1.62	1.50	1.42	1.56	1.53	0.09	5.6	Acetonitrile	Standard Solution	GC-MS	Internal Standard
LC0041	1.22	1.09	1.26	1.08	1.16	0.09	7.8	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0044	2.03	1.85	1.92	1.91	1.93	0.07	3.9	not specified	not specified	not specified	Standard Addition
LC0047	1.50	1.40	1.50	1.50	1.48	0.05	3.4	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0048	2.289	2.193	2.266	2.023	2.19	0.12	5.5	Acetone	Standard Solution	GC-MS	Internal Standard
LC0049	0.512	0.550	0.518	0.564	0.54	0.03	4.7	Acetone	Spiking Tenax	GC-MS/GC-FID	Internal Standard
LC0050	1.924	1.875	1.961	1.894	1.91	0.04	2.0	not specified	not specified	not specified	not specified
LC0055	1.15	1.30	1.20	1.05	1.18	0.10	8.6	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0056	0.23	0.14	0.24	0.56	0.29	0.18	62.9	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0059	1.13	1.32	1.45	1.09	1.25	0.17	13.5	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0064	1.58	1.57	1.58	1.64	1.59	0.03	2.0	Acetone	Spiking Tenax	GC-MS	Internal Standard

Annex 15. Caprolactam values reported by the participants.

Lab Code	Concentration (µg/g)				Average	SD	RSD (%)	Extraction	Calibration	Instrumentation	Quantification
	Repetition 1	Repetition 2	Repetition 3	Repetition 4							
LC0002	10.10	10.90	10.80	6.70	9.63	1.98	20.6	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0004	17.14	17.94	13.64	12.88	15.40	2.51	16.3	Hexane	Spiking Tenax	GC-MS/GC-FID	Internal Standard
LC0005	8.15	8.10	8.10	8.26	8.15	0.08	0.9	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0006	8.92	9.34	9.88	8.98	9.28	0.44	4.8	not specified	not specified	GC-FID	not specified
LC0007	11.21	8.60	10.40	9.44	9.91	1.14	11.5	Acetone	Spiking Tenax	GC-FID	Internal Standard
LC0010	8.73	8.55	8.62	8.94	8.71	0.17	2.0	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0013	9.52	10.21	9.39	9.75	9.72	0.36	3.7	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0017	10.08	10.07	10.12	10.34	10.15	0.13	1.2	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0018	9.32	8.63	8.67	8.27	8.72	0.44	5.0	Acetonitrile	Spiking Tenax	LC-MS/MS	Internal Standard
LC0020	14.77	14.81	12.95	12.71	13.81	1.14	8.2	Acetone	Standard Solution	GC-MS	External Standard
LC0025	9.45	9.47	9.40	9.16	9.37	0.14	1.5	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0031	8.47	9.39	8.83	8.41	8.78	0.45	5.1	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0032	6.35	7.70	7.25	7.35	7.16	0.58	8.0	Acetone	Standard Solution	GC-MS	External Standard
LC0037	8.95	9.38	9.09	9.68	9.28	0.32	3.5	Acetone	Spiking Tenax	GC-MS/GC-FID	Internal Standard
LC0038	9.62	9.46	9.20	9.24	9.38	0.19	2.1	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0040	9.22	9.02	8.44	7.82	8.63	0.63	7.3	Acetonitrile	Standard Solution	GC-MS	Internal Standard
LC0041	8.35	9.62	9.81	6.64	8.61	1.46	17.0	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0044	11.70	11.70	11.00	11.80	11.55	0.37	3.2	not specified	not specified	not specified	not specified
LC0047	10.30	11.00	11.90	11.50	11.18	0.69	6.2	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0048	11.45	11.70	11.19	9.31	10.91	1.09	10.0	Acetone	Standard Solution	GC-MS	Internal Standard
LC0049	3.85	4.02	3.70	3.54	3.78	0.20	5.4	Acetone	Spiking Tenax	GC-MS/GC-FID	Internal Standard
LC0050	8.63	7.80	8.12	8.06	8.15	0.35	4.3	not specified	not specified	not specified	not specified
LC0055	8.57	8.72	8.29	8.70	8.57	0.20	2.3	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0056	1.36	1.33	1.07	1.07	1.21	0.16	13.2	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0059	21.31	12.73	20.10	12.75	16.72	4.63	27.7	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0064	10.10	10.70	11.27	11.91	11.00	0.77	7.0	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0113	9.60	10.40	10.10	9.90	10.00	0.34	3.4	Acetone	Spiking Tenax	not specified	Internal Standard

Annex 16. Dibutyl sebacate values reported by the participants.

Lab Code	Concentration (µg/g)				Average	SD	RSD (%)	Extraction	Calibration	Instrumentation	Quantification
	Repetition 1	Repetition 2	Repetition 3	Repetition 4							
LC0002	5.00	4.54	4.94	4.12	4.65	0.41	8.8	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0004	4.60	4.74	4.89	4.74	4.74	0.12	2.5	Hexane	Spiking Tenax	GC-MS/GC-FID	Internal Standard
LC0005	5.04	5.05	5.22	4.98	5.07	0.10	2.0	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0006	3.36	3.63	3.55	3.18	3.43	0.20	5.9	not specified	not specified	GC-FID	not specified
LC0007	5.07	7.38	7.28	6.16	6.47	1.09	16.8	Acetone	Spiking Tenax	GC-FID	Internal Standard
LC0008	5.94	5.95	5.61	5.60	5.78	0.19	3.4	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0010	7.00	6.10	7.35	7.91	7.09	0.76	10.7	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0013	4.29	4.06	4.06	4.10	4.13	0.11	2.7	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0016	3.20	3.40	3.50	4.00	3.53	0.34	9.7	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0017	5.30	5.30	5.12	5.23	5.24	0.08	1.6	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0018	4.77	4.60	4.24	4.70	4.58	0.24	5.1	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0025	4.37	4.39	4.27	4.15	4.30	0.11	2.6	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0031	5.20	5.12	5.25	5.24	5.20	0.06	1.1	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0032	3.90	4.70	4.60	4.40	4.40	0.36	8.1	Acetone	Standard Solution	GC-MS	External Standard
LC0037	4.41	5.28	4.17	5.50	4.84	0.65	13.4	Acetone	Spiking Tenax	GC-MS/GC-FID	Internal Standard
LC0038	5.01	5.04	5.79	7.15	5.75	1.00	17.5	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0040	4.42	4.41	4.24	4.12	4.30	0.14	3.4	Acetonitrile	Standard Solution	GC-MS	Internal Standard
LC0041	4.25	4.21	4.03	3.88	4.09	0.17	4.2	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0044	5.32	4.00	4.14	4.72	4.55	0.60	13.3	not specified	not specified	not specified	not specified
LC0047	5.10	4.90	5.00	4.90	4.98	0.10	1.9	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0048	5.74	5.91	5.66	5.97	5.82	0.14	2.5	Acetone	Standard Solution	GC-MS	Internal Standard
LC0049	2.60	2.79	2.80	2.58	2.69	0.12	4.3	Acetone	Spiking Tenax	GC-MS/GC-FID	Internal Standard
LC0050	5.56	5.30	5.50	5.30	5.41	0.13	2.5	not specified	not specified	not specified	not specified
LC0055	4.09	4.37	4.27	4.28	4.25	0.12	2.8	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0056	0.83	1.07	1.06	0.89	0.96	0.12	12.6	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0059	4.29	4.41	4.92	4.56	4.55	0.27	6.0	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0064	4.12	3.96	3.69	4.00	3.94	0.18	4.6	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0113	3.30	3.50	3.50	3.40	3.43	0.10	2.8	Acetone	Spiking Tenax	not specified	Internal Standard

Annex 17. 2,6-Diisopropylnaphthalene values reported by the participants.

Lab Code	Concentration (µg/g)				Average	SD	RSD (%)	Extraction	Calibration	Instrumentation	Quantification
	Repetition 1	Repetition 2	Repetition 3	Repetition 4							
LC0002	1.71	1.79	1.87	1.21	1.65	0.30	18.1	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0004	1.26	1.40	1.31	1.42	1.35	0.08	5.6	Hexane	Spiking Tenax	GC-MS/GC-FID	Internal Standard
LC0005	1.53	1.58	1.52	1.52	1.54	0.03	1.9	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0006	1.24	1.34	1.31	1.26	1.29	0.05	3.6	not specified	not specified	GC-FID	not specified
LC0007	1.79	2.02	1.94	1.83	1.90	0.10	5.5	Acetone	Spiking Tenax	GC-FID	Internal Standard
LC0010	1.45	1.35	1.39	1.35	1.39	0.05	3.4	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0013	1.30	1.30	1.31	1.34	1.31	0.02	1.4	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0016	1.70	1.60	1.60	1.60	1.63	0.05	3.1	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0017	1.35	1.36	1.35	1.35	1.35	0.01	0.4	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0018	1.83	1.95	1.84	2.16	1.95	0.15	7.9	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0020	4.23	4.29	3.28	4.07	3.97	0.47	11.8	Acetone	Standard Solution	GC-MS	External Standard
LC0025	1.40	1.38	1.39	1.34	1.38	0.03	1.9	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0031	1.32	1.36	1.35	1.30	1.33	0.03	2.1	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0032	1.25	1.55	1.50	1.50	1.45	0.14	9.3	Acetone	Standard Solution	GC-MS	External Standard
LC0037	1.32	1.51	1.62	1.70	1.54	0.16	10.7	Acetone	Spiking Tenax	GC-MS/GC-FID	Internal Standard
LC0038	1.42	1.42	1.35	1.39	1.40	0.03	2.4	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0040	1.21	1.25	1.18	1.14	1.20	0.05	3.9	Acetonitrile	Standard Solution	GC-MS	Internal Standard
LC0041	1.30	1.32	1.31	1.26	1.30	0.03	2.0	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0044	1.54	1.39	1.47	1.47	1.47	0.06	4.2	not specified	not specified	not specified	not specified
LC0047	1.40	1.30	1.50	1.40	1.40	0.08	5.8	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0048	2.41	2.40	2.30	2.45	2.39	0.06	2.7	Acetone	Standard Solution	GC-MS	Internal Standard
LC0049	0.51	0.53	0.45	0.49	0.50	0.03	7.0	Acetone	Spiking Tenax	GC-MS/GC-FID	Internal Standard
LC0050	0.34	0.85	0.91	0.92	0.76	0.28	36.8	not specified	not specified	not specified	not specified
LC0055	1.23	1.26	1.23	1.18	1.22	0.03	2.8	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0056	0.30	0.26	0.28	0.26	0.28	0.02	7.0	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0059	1.44	1.35	1.45	1.43	1.42	0.05	3.2	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0064	1.24	1.22	1.14	1.13	1.18	0.06	4.7	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0113	1.70	1.90	1.80	1.70	1.78	0.10	5.4	Acetone	Spiking Tenax	not specified	Internal Standard

Annex 18. 2-Ethyl-1-hexanol values reported by the participants.

Lab Code	Concentration (µg/g)				Average	SD	RSD (%)	Extraction	Calibration	Instrumentation	Quantification
	Repetition 1	Repetition 2	Repetition 3	Repetition 4							
LC0002	11.00	10.10	11.00	10.70	10.70	0.42	4.0	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0004	5.54	6.62	5.94	6.67	6.19	0.55	8.8	Hexane	Spiking Tenax	GC-MS/GC-FID	Internal Standard
LC0005	6.70	6.73	6.70	6.74	6.72	0.02	0.3	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0006	1.23	1.12	1.17	1.28	1.20	0.07	5.8	not specified	not specified	GC-FID	not specified
LC0007	11.43	11.82	13.69	11.71	12.16	1.03	8.5	Acetone	Spiking Tenax	GC-FID	Internal Standard
LC0010	7.96	8.09	7.85	7.87	7.94	0.11	1.4	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0013	10.04	10.27	9.77	9.10	9.80	0.51	5.2	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0016	7.20	8.30	8.30	7.40	7.80	0.58	7.5	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0017	9.24	9.15	9.21	9.19	9.20	0.04	0.4	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0018	9.61	9.58	9.16	9.81	9.54	0.27	2.9	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0020	12.22	9.01	10.11	8.99	10.08	1.52	15.1	Acetone	Standard Solution	GC-MS	External Standard
LC0025	9.58	9.63	9.57	9.10	9.47	0.25	2.6	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0031	7.45	7.49	7.54	6.70	7.30	0.40	5.5	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0032	5.30	5.65	4.90	6.35	5.55	0.62	11.1	Acetone	Standard Solution	GC-MS	External Standard
LC0037	6.09	6.80	6.56	7.05	6.63	0.41	6.2	Acetone	Spiking Tenax	GC-MS/GC-FID	Internal Standard
LC0038	7.45	7.06	6.92	6.13	6.89	0.55	8.0	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0040	7.28	7.12	6.56	6.10	6.77	0.54	8.0	Acetonitrile	Standard Solution	GC-MS	Internal Standard
LC0041	7.30	7.92	7.79	8.15	7.79	0.36	4.6	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0044	7.51	7.81	7.82	6.78	7.48	0.49	6.5	not specified	not specified	not specified	not specified
LC0047	6.80	8.20	9.00	7.90	7.98	0.91	11.4	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0048	13.62	9.61	9.26	11.01	10.87	1.98	18.2	Acetone	Standard Solution	GC-MS	Internal Standard
LC0049	4.28	4.24	4.32	4.03	4.22	0.13	3.1	Acetone	Spiking Tenax	GC-MS/GC-FID	Internal Standard
LC0055	6.70	6.75	6.88	6.63	6.74	0.11	1.6	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0056	2.08	1.23	1.45	1.47	1.56	0.36	23.4	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0059	9.72	7.63	10.82	9.38	9.39	1.32	14.1	Hexane	Spiking Tenax	GC-MS	Internal Standard
LC0064	6.81	7.85	6.84	7.82	7.33	0.58	8.0	Acetone	Spiking Tenax	GC-MS	Internal Standard
LC0113	8.50	8.30	8.40	8.60	8.45	0.13	1.5	Acetone	Spiking Tenax	not specified	Internal Standard

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