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Report of the interlaboratory comparison organised by the Community Reference Laboratory for Food Contact Material

Plasticisers in Gaskets and Oil

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The mission of the IHCP is to provide scientific support to the development and implementation of EU policies related to health and consumer protection.

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Food Contact Materials

Report of the first interlaboratory comparison

Plasticisers in Gasket and Oil

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1. Summary

The Institute for Health and Consumer Protection (IHCP) of the European Commission's Directorate-General Joint Research Centre hosts the Community Reference Laboratory for Food Contact Materials (CRL-FCM). One of its core tasks is to organize interlaboratory comparisons (ILCs) among appointed National Reference Laboratories (NRLs). This report presents the results of the first ILC of the CRL-FCM which focused on the determination of Plasticisers content in PVC Gasket and in Oil matrix.

The test materials used in this exercise were virgin gasket lids coming from industrial sources for the proficiency exercise part A. For the second part of the exercise an industrial source of sunflower oil was used and spiked with several plasticisers by the CRL-FCM.

There were 41 participants to whom samples were dispatched 34 of which submitted results for at least 1 analyte-material. 21 laboratories reported results for more than 10 analyte-material combination out of 14 required.

The homogeneity studies were performed by the CRL-FCM laboratory.

The assigned value and its uncertainty for part A, virgin gaskets, were obtained after applying the robust statistics to the results obtained from the participants.

The assigned values for part B, oil samples, were those obtained based on formulation, from the gravimetric measurements used to spike the material.

The uncertainty of the assigned values for oil samples was calculated combining the uncertainty of the spiking procedure with a contribution for the between-bottle homogeneity.

Participants were invited to report four replicates measurements. This was done by most of the participants.

Laboratory results were rated with z and z' scores in accordance with ISO 13528 [1] Standard deviations for proficiency assessment (also called target standard deviations) were set based on Horwitz equation for substances in the two oil samples. For the plasticisers in the two gasket samples the target standard deviation was set by the organizers to 15% in order to fulfil the required criteria for sufficient homogeneity of the sample within the lots.

2. Introduction

Laboratory proficiency testing is an essential and very important 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.

It is one of the core duties of the Community Reference Laboratories to organize interlaboratory comparisons, as is stated in Regulation (EC) No 882/2004 of the European Parliament and of the Council [6]

In accordance with the above requirements the Community Reference Laboratory for Food Contact Material (CRL-FCM) organized in 2008 the first interlaboratory comparison test for the network of appointed NRLs.

The scope of the interlaboratory comparison test was discussed and agreed on the plenary meeting with all NRL's held in December 2007 at JRC, Ispra, Italy. On that meeting the decision was made in consensus that First interlaboratory comparison organized by CRL-FCM would be on determination of plasticizer's content in PVC Gasket and oil as food matrix.

3. Scope

The scope of this comparison was to test the competence of the appointed NRLs to analyze plasticizer's content in virgin gasket and oil as a food matrix, mainly ESBO, DINCH, phthalates as DIDP, BBP etc.. The compounds to be analyzed and their concentration levels in oil matrix were chosen in accordance with the legislation [3, 4]

The assessment of the measurement results was undertaken on the basis of requirements laid down in international standards and guidelines [1, 2, 9, 10]

4. Time frame

The interlaboratory comparison was first announced to the NRL network at the CRL-FCM workshop on 21/22 November 2007.

A questionnaire (Annex 1) was sent to all NRL's with questions for assessing the present situation in each laboratory on determination of

plasticisers, especially ESBO and phthalates in gaskets, oil and any other food material. The questionnaire was sent to 27 NRL's (incl. Switzerland and 2 laboratories from France). 16 NRLs sent back their answers which are summarized in Annex 2.

Invitation letters were sent to the laboratories on 10 June 2008 for PT001/A (Annex 3a) and on 8 July 2008 for PT001/B (Annex 3b). Laboratories are invited to fill a letter of confirmation of their participation (Annex 6 a, b)

The gasket samples (PT001/A) were dispatched to participants on 11 June 2008 together with letter accompanying the samples (Annex 4). The oil samples (PT001/B) were dispatched to participants on 08 July 2008. Letters were sent to the participant with detailed instructions for compilation of the results (Annex 5a) as well as instructions for compilation of the results in electronic format (Annex 5b) together with electronic files where the result should be inserted.

The participant are asked to fill a letter of conformation of the receipt of the samples (Annex 7 a, b)

Reporting deadline was 12 September 2008 either for PT001/A then for PT001/B. It was extended to 12 October and the ILC was closed at end of October.

5. Test material

Virgin gasket lids came from industrial source from Thailand Industry.

An industrial source of sunflower oil was used and spiked with several plasticisers by the CRL-FCM

Exercise	Sample	Source
PT001/A	Virgin Gasket type 1	Thailand industrial association
	Virgin Gasket type 2	Thailand industrial association
PT001/B	Oil 1+ spike	Italian oil producer + spike with substances from different brand (see table 1)
	Oil 2 + spike	Italian oil producer + spike with substances from different brand (see table 1)

Gaskets:

- Virgin Gasket type 1 (n=1 specimen) phthalate based;
- Virgin Gasket type 2 (n=5 specimens) ESBO based;

Oil:

- 2 bottle of blank oil (100 ml) (n=2 specimens);
- 1 bottle of oil (100 ml) (n=1 specimen) phthalate based;
- 1 bottle of oil (100 ml) (n=1 specimen) ESBO based.

Each participant received additional to the samples one standard substances kit containing:

Standard substances kit

Substance type	Brand	Trade name	abbrev	name	CAS No	volume (mL)
Phthalates	BASF	PALATINOL C	DBP	dibutyl phthalate	84-74-2	10
Phthalates	BASF	PALATINOL IC	DiBP	Diisobutyl phthalate	84-69-5	10
Phthalates	Ferro	SANTICIZER 160	BBP	benzylbutyl phthalate (2001)*	85-68-7	10
Phthalates	BASF	PALATINOL N	DINP	diisononyl phthalate	28553-12-0	10
Phthalates	ExxonMobil	JAYFLEX DINP	DINP	diisononyl phthalate	68515-48-0	10
Phthalates	ExxonMobil	JAYFLEX DIDP	DIDP	diisodecyl phthalate	68515-49-1	10
ESBO	Akcros	Lankroflex E2307	ESBO	epoxidised soybean oil	008013-07-8	10
ELO	Akcros	Lankroflex L	ELO	epoxidised linseed oil	008016-11-3	10
DINCH	BASF	HEXAMOLL	DINCH	1,2-Cyclohexanedicarboxylic acid diisononyl ester	66412-78-8	10
Sebacate	Sigma	-	DBS	Dibutyl-Sebacate	109-43-3	1
AMG	Danisco	GRINDSTED® SOFT-N-SAFE	AMG	acetylated monoglyceride	736150-63-3	1
Note: BASF could no longer provide DEHP or DIDP as they do not produce these substances any longer in Europe.						
Note: The specification for residual DINP content for DINCH is currently at max. 100 ppm i.e. = 0,01 %						
* BBP was no longer available, so we provided from the stock we had from our work on toys in 2001.						

5.1 Preparation

The gaskets were supplies from the Thai industrial food processing association.

The sunflower oil was purchased from an Italian oil producer and checked

for purity.

Preparation and homogenization of the test material was done by the CRL-FCM laboratory according to the procedure described in Annex 9.

After spiking and homogenization the oil was dispensed in glass bottles of approximately 100 mL capacity.

5.2 Homogeneity assessment

The samples were tested for homogeneity by the CRL Laboratory.

Ten randomly selected test specimens for each sample (gasket 1, gasket 2, oil 1 and oil 2) were analyzed in duplicate for all the required measurands.

Additionally in order to assess the homogeneity of the virgin gasket coating within the same gasket, samples were taken in duplicate from 10 evenly distributed points of the lids on two randomly chosen gaskets per batches 1 and 2 and analyzed.

Homogeneity was evaluated by the Prolab Software according to IUPAC International Harmonized Protocol [10] and to the method proposed in the ISO 13528 [1]. The results together with their statistical evaluation are given in Annex 10 (1-26)

All test materials has shown sufficient homogeneity for all the measurands for the target pre-defined standard deviation of the PT, except for DIDP, gasket type 2 (Annex 10-1d). The data were distributed around 2 mean values, showing the probability that the gasket came from 2 different lots. 100 gaskets from lot "gasket type 2" is analyzed in duplicate for DIDP (Annex 11). A sub-lot of 50 gaskets covering the requirement for sufficient homogeneity (Annex 12) is chosen as a sample for dispatch to the participant.

5.3 Distribution

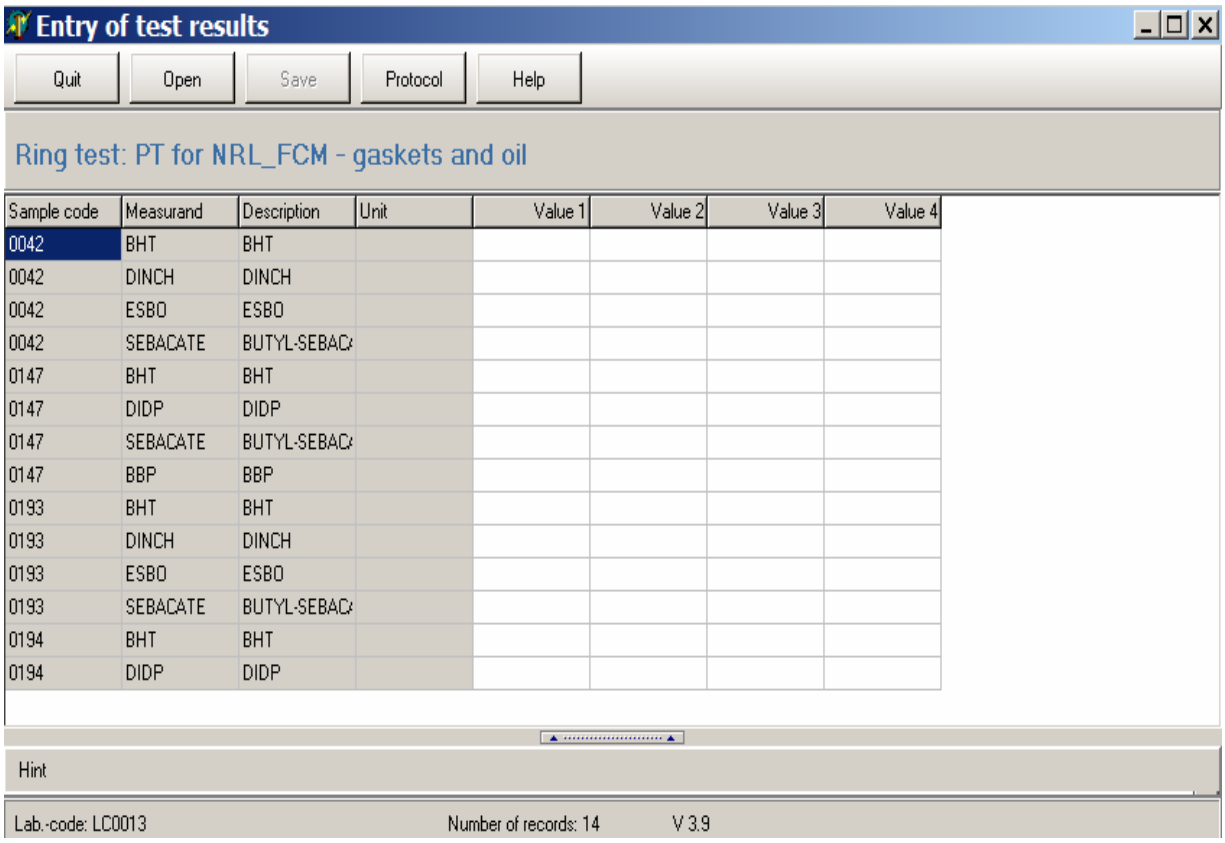
The samples were dispatched to the participants by the CRL-FCM on 10 June 2007 (PT001/A) and 08 July 2007 (PT001/B). Each participant received: a) two boxes containing the test materials, b) an accompanying letter with instructions on sample handling and reporting (cf. Annex 2) and c) a form that had to be sent back after receipt of the sample to confirm its arrival (cf. Annex 3).

6 Instructions to participants

Details of this interlaboratory comparison exercise were presented to the NRLs at the workshop held in Ispra on 4 July 2008. Concrete instructions were given to all participants in a letter that accompanied the samples (Annex 5a, 5b). The measurands and matrix were clearly defined.

Laboratories were asked to perform four independent measurements and report them. Participants were asked to follow their own procedures. The results were to be reported using the unit of measure indicated in the instruction letter.

The results were to be reported in a special ProLab [5] software form as shown below:



Sample code	Measurand	Description	Unit	Value 1	Value 2	Value 3	Value 4
0042	BHT	BHT					
0042	DINCH	DINCH					
0042	ESBO	ESBO					
0042	SEBACATE	BUTYL-SEBAC					
0147	BHT	BHT					
0147	DIDP	DIDP					
0147	SEBACATE	BUTYL-SEBAC					
0147	BBP	BBP					
0193	BHT	BHT					
0193	DINCH	DINCH					
0193	ESBO	ESBO					
0193	SEBACATE	BUTYL-SEBAC					
0194	BHT	BHT					
0194	DIDP	DIDP					

7 Assigned values and their uncertainties

As described earlier, the test materials used for part A in this exercise were virgin gasket lids coming from industrial source for the proficiency exercise. For the second part of the exercise an industrial source of sun flower oil was used and fortified with several plasticisers by the CRL-FCM.

The assigned value and its uncertainty for part A, virgin gaskets, were obtained after applying the robust statistics to the results obtained from the participants.

The assigned values for part B, oil samples, were calculated based on the formulation, from the gravimetric measurements of the oil and the solid substances (plasticisers), used to fortify the material (see Annex 9).

The uncertainty of the assigned values (u_{ref}) for oil samples was calculated by combining the uncertainty of formulation ($u_{formulation}$) and the contribution for between-bottle homogeneity (u^*_{bb}), as follows:

$$\mathbf{u}_{ref} = SQRT\left(\mathbf{u}_{formulation}^{2} + \mathbf{u}_{bb}^{2}\right)$$

where:

$\mathbf{u}_{ref}$	uncertainty associated to the assigned value
$\mathbf{u}_{formulation}$	standard uncertainty of the formulation;
$\mathbf{u}_{bb}$	contribution for the between-bottle homogeneity;

The reference value (X_{ref}) for this ILC was calculated using the following equation:

$$\mathbf{X}_{ref} = \mathbf{C}_{oil} = \left(\mathbf{m}_{plasticizer} / \mathbf{m}_{oil}\right) * \mathbf{P}_{plasticizer}$$

where:

$\mathbf{C}_{oil}$	final concentration of the plasticisers in the oil test material, respectively;
$\mathbf{m}_{plsticizer}$	mass of the plasticizer, in mg respectively;
$\mathbf{m}_{oil}$	final mass of oil test material after fortification with the plasticisers;
$\mathbf{P}_{plsticizer}$	purity of the plasticizer substance used for fortification

Hence, the standard uncertainty estimated based on formulation ($u_{formulation}$) for any substance-plasticiser is derived from the formula:

$$\mathbf{u}_{formulation} = SQRT\left(\mathbf{u}^2_{weight\ oil} + \mathbf{u}^2_{weight\ plasticizer} + \mathbf{u}^2_{purity}\right)$$

where

$\mathbf{u}_{weigth\ oil}$,	uncertainty from the weight of the oil material (balance)
$\mathbf{u}_{weight\ plasticizer}$,	uncertainty from the weight of the oil material (balance)
$\mathbf{u}_{purity\ of\ plasticizer}$	uncertainty from the purity of the plasticizer from its certificate of production

The maximum heterogeneity that could be hidden by method repeatability (u*bb) is determined:

$$\mathbf{u}^*_{bb} = (\mathbf{s}_{homo} / \sqrt{n}) * (2/\nu_{shomo})^{1/4}$$

where

s_{homo} is the within-bottle standard deviation obtained by the homogeneity study;

n is the number of replicate measurements per bottle (n=1)

ν_{shomo} is the degrees of freedom for the determination of this standard deviation (ν_{shomo} = 19 , as 20 bottles were analysed).

The values of $\mathbf{X}_{ref}$, $\mathbf{u}_{ref}$ and the expanded uncertainty (U_{ref}) are summarised in Table 1.

Table 1: Assigned values and their uncertainties for the parameters of this ILC.

Oil 1	Concentration, X_{ref}, mg/kg	$u_{formul.}$ %	u_{bb} %	u_{ref} %	U_{ref} mg/kg
ESBO	60.53	0.58	1.0	1.2	1.44
DINCH	14.86	0.30	0.8	0.9	0.27
BHT	2.96	0.60	2.0	2.1	0.13
b-sebacate	3.11	1.75	1.7	2.4	0.15
Oil 2	**Concentration, X_{ref}, mg/kg**	**$u_{formul.}$ %**	**u_{bb} %**	**u_{ref} %**	**U_{ref} mg/kg**
DIDP	8.00	0.24	1.1	1.1	0.17
BBP	14.97	0.69	0.7	1.0	0.29
BHT	1.07	0.69	0.7	1.0	0.02
b-sebacate	1.44	1.75	1.2	2.1	0.06

X_{ref} is the certified reference value and u_{ref} the corresponding standard uncertainty; U_{ref} is the estimated expanded uncertainty, with a coverage factor k = 2, corresponding to a level of confidence of about 95 %, as defined in the Guide to the Expression of Uncertainty in Measurement (GUM), ISO, 1995.

8. Target standard deviation of the inter-laboratory comparison (ILC)

The value σ_p determines the limits of satisfactory performance in ILC test. It should be set as a value that reflects best practice for the analysis in question. The standard deviation of the reproducibility found in the collaborative trials is generally considered an appropriate indicator of the best agreement that can be obtained between laboratories. However it is not applicable to all cases. In the absence of appropriate collaborative trial data, σ_p could be derived from the appropriate form of the modified Horwitz equation [2].

For all analytes/matrix combination in oil samples of this ILC test the target standard deviation was set to the calculated by Horwitz formula.

For the plasticisers in gaskets however, due to the very high concentration of the plasticisers in the gaskets, the calculated Horwitz standard deviation resulted to very low values in the range of 1-3%, unreasonable to be accepted as target standard deviation for ILC of plasticisers in gaskets. A target standard deviation of 15% was chosen in order to be in compliance with the requirements for sufficient homogeneity of the test materials – Annex 10.

9. Evaluation of results

9.1 General observations

There were forty-one participants from twenty-seven countries to whom samples are dispatched. They all received the samples. The ILC was closed permanently in November for statistical interpretation.

Thirty-four laboratories submitted results for at least 1 analyte-material. Twenty one laboratories reported results for more than 10 analyte-material combination out of 14 required. As requested, most of the laboratories reported four measurement replicate results for any analyte-material combination.

From the CRL-NRL network 20 laboratories out of 25 reported results for at least 1 analyte-material combination. 5 NRL laboratories did not send any results. 3 NRL laboratories reported results only for 2 measurand-matrix combination out of 14; 1 laboratory – for 3 combinations and 1 laboratory – for 4 combinations. Majority of participants reported results for about 10 and more measurand-matrix combination.

For plasticisers in gaskets 30 laboratories reported their results for DIDP, 26 for DINCH and butyl-sebacate and 23 for ESBO, and 6 for BHT, of which only 3> LOD. This gave rise to re-evaluation of the available data and repetition by the organisers of the test procedure for determination of BHT in gaskets. Final results showed that the preliminary determined content could be originating from a cross contamination during the analytical stage. Therefore no statistical evaluation was further performed on BHT in gaskets and this measurand-matrix combination was not taken into account for final evaluation of the laboratories.

For oil samples the number of reported results varied between 14 (for BHT) and 20 (or DINCH, ESBO, sebacate).

9.2. Statistical evaluation of results

9.2.1. Determination of the consensus value and standard deviation of the interlaboratory comparison.

Statistical evaluation of the results was performed using the ProLab software [5] applying different algorithms for the determination of the consensus value and its standard deviation according to ISO 13528 [1], DIN 38402 A 45 [7] and ISO/TS 20612 [8]. The three mainly used algorithms for robust statistic determination of the consensus value - Q-median, Q/Huber-estimator and Q/Hampel estimator - gave very close results. The choice was made on Hampel estimator as one of the most robust.

9.2.2. Identification of modes using kernel density plotting

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 [12, 13]. Kernel density plots are computed by the ProLab software [5] from the analytical results by representing the individual numeric values each as a normalised Gaussian distribution centred on the respective analytical value. The sum of these normal distributions forms then the Kernel density distribution.

9.2.3. Scores and evaluation criteria

Individual laboratory performance was expressed in terms of z and z'-scores in accordance with ISO 13528[1] and the International Harmonised Protocol[10]

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

$$z' = \frac{(x_{lab} - X)}{\sqrt{\sigma_p{}^2 + u^2_{assigned}}}$$

where

x_{lab}	is the measurement result reported by a participant
$X_{assigned}$	is the assigned value
σ_p	is the target standard deviation for proficiency assessment
$u_{assigned}$	is the standard uncertainty of the assigned value

The z- and z'-scores can be interpreted as follow:

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

The z-scores compared the participant's deviation from the assigned value with target standard deviation accepted for the interlaboratory comparison σ_p

z'-scores could be used when the assigned value is not calculated using the results reported by the participants. z'-score takes in consideration the uncertainty of the assigned values. In case the guidelines for limiting the uncertainty of the assigned value $u_{assigned} < 0.3\ \sigma_p$ [1] are met, then z'-scores will be similar to z'-scores

When the guideline was not met, the difference in magnitude of the *z'*-scores and *z*-scores may be such that some *z*-scores exceed the critical values of 2,0 or 3,0 and so give "warning signals" by an "action signals", whereas the corresponding *z'*-scores do not exceed these critical values and so do not give signals.

For results reported as "smaller than" (<-values), the reported value was not used in any calculations and no evaluation of the measurement results was made. No scores were given.

9.2.4. Mandel's h- and k-statistics

Mandel's h-statistic and Mandel's k-statistic [11] present measures for graphically surveying the consistency of the data. They are helpful for laboratory assessment. For answering the questions if there are differences between the mean values of the laboratories, Mandel's h-statistic can be considered. In order to assess the variance of each laboratory compared to the variances of the other laboratories, Mandel's

k-statistic is useful. Mandel's h- and k- values are calculated by ProLab software following ISO 5275.

The examination of the plots of Mandel's h- and k-statistics may indicate that specific laboratories exhibit patterns of results that are markedly different from the others. This is indicated by (compared to the other laboratories) consistently high or low variation and/or extreme (high or low) mean values.

Various patterns can appear in the plot of Mandel's h-statistic. All laboratories can have both positive and negative values. Individual laboratories may tend to give either all positive or all negative values. This is no unusual pattern, but it may suggest that a common source of laboratory bias exists.

If one laboratory stands out on the k-statistic as having many large values, the respective laboratory has a poorer repeatability precision than the other laboratories. A laboratory could give rise to consistently small k-values because of such factors as excessive rounding of its data or an insensitive measurement scale.

9.3 Laboratory results and scores

The results as reported by the participants, were summarised in Table 2 (1-12) together with the mean values and z- or z' -scores. For the gasket z-scores were calculated as z and z' scores coincided. For oil z'-scores were used as more in favour for the assessment of the laboratory performance.

Three sets of figures were provided for each analyte/matrix combination in Fig 1 (1-12). Each set included (a) individual laboratories values and their mean and standard deviation, (b) the Kernel Density plot, (c) the z'-scores.

In Fig. 2 Mandel's h- and Mandel's k-statistics are shown for each laboratory-sample-combination grouped by measurand. Values differing statistically significant from values of the other laboratories are marked in a different color: a red bar indicates a value significant to the significance level of 1% while a yellow bar indicates a value significant to the level of 5%.

10 Comments on results and conclusions

The participation of the laboratories was satisfactory with regards the numbers of received results.

In table 2, a summary of number of participants and test results are shown

Sample	Measurand	Number of test results	Number of laboratories
GASKET1	SEBACATE	98	25
OIL2	BHT	56	14
GASKET2	DIDP	114	30
GASKET1	ESBO	88	23
OIL1	DINCH	80	20
OIL2	DIDP	68	17
GASKET1	DINCH	102	26
OIL1	BHT	56	14
OIL1	ESBO	73	20
OIL1	SEBACATE	80	20
OIL2	SEBACATE	80	20
OIL2	BBP	80	20

Summary of the of robust mean and robust reproducibility standard deviation calculated according to Hampel algorithm (ISO 20612:2007 and DIN 38402 A45) by ProLab software as well as assigned values and target standard deviation of the ILC, reference values and its uncertainty for oil samples are given in Table 3.

It should be mentioned that the robust mean derived from the results coincides very well with the reference values taken as assigned values for all measurand-matrix combination in oil samples. The difference between $x_{mean} - X_{ref}$ was less then twice its standard uncertainty for all the measurand-matrix combination in oil samples.

$$\left(\frac{(1{,}23s^{*})^2}{p} + u_X{}^2\right)^{1/2}$$

Where

u_x is the uncertainty of the reference values;

s* is the robust standard deviation;

p is the number of participating laboratories

Z and z'-sores assessed the laboratory performance against the target standard deviation, preliminary determined for the ILC. As mentioned before target standard deviation for gaskets was set to 15% and those for oil – to the calculated by Horvitz equation (see table 3).

Table 4 represents the laboratories' raw test results, their mean values and corresponding z'-score

Figure 1 represents graphs of the laboratory's test results with their repeatability SD (a), Kernel Density plot (b) and z'-scores (c)

Considering the z' scores (for gaskets z=z'-scores), the overall participation was evaluated as satisfactory. Summary of the results as z'-scores reported in table 5 and figure 3 showed good performance with more then 75% successful results by the laboratories for plasticisers in gaskets – DINCH (85%), DIDP (93%), butyl-sebacate (85%) and ESBO (74%). For oil samples the overall performance of the laboratories is slightly lower with 60-65% successful results for DINCH, ESBO and DIDP. The difference in laboratory performance came from the different target SD accepted for laboratory assessment. According to Horwitz formulae target SD for ESBO in oil should be 8.6 % and 10.5-11.6 for DINCH, DIDP and BBP, which is less than the defined target SD of 15% for gaskets in oil.

Mandel statistics grouped by measurand were represented in Figure 2. As mentioned before it evaluated the performance of each laboratory against the overall performance of all laboratories not only regarding mean values, but repeatability as well. "Outliers" considering mean values and repeatability SD were assessed for 1% significance level (in yellow on figure 2) and for 5% significance level (in red in Fig.2) Summary of the number of "outliers" for each measurand-matrix combination are given in Table 7.

The Youden plot displayed a combined graphic of the results of two measurand-matrix combinations. Such a presentation allowed identifying systematic effects in the laboratory-specific deviations. It gave an immediate idea of the dominating sources of error in the results. Laboratories having results in the upper left or lower right hand corner of the diagram had analyses dominated by random error. Alternatively, laboratories having results close to the 45° line shown in the plot but far away from the assigned value had results dominated by systematic error.

An example of a Youden plots for BHT and butyl sebacate which were analysed in both oil samples with different concentration levels are presented in Figure 5. It could be easily concluded that for butyl sebacate in oil Lab 55 and lab 28 suffer from systematic error; lab 17 and 2 – from random error whereas lab 21 and lab 3 from both type of errors.

Figure 6 represents the overall z'-score distribution for all the 249 measurand-matrix-laboratory combinations for 6 measurands, 4 samples and 34 laboratories'. Figure 7 represents them in histogram like Kernel density plot and normal distribution plot - showing its real normal distribution.

Additional information was gathered from the questionnaire on analysis ESBO and phthalates completed by the NRL part of the participants. The questionnaire and a summary of the answers from NRLs are given in Annexes 1 and 2. 10 laboratories did not reply. From the other 15 - for ESBO only 6 laboratories have performed analysis of ESBO in any of the

matrix – gasket, oil, food and only 2 of the laboratories have validated the method before. For phthalates –only 5 laboratories did not perform any phthalate analysis before. 10 of the laboratories use validated method or participated in another PT schemes as FAPAS, or Dutch – ILS

Table 3
Table of robust mean and robust SD calculated according to Hampel algorithm (ISO 20612:2007 and DIN 38402 A45)

Sample	Measurand	Unit	Mean	MU	Ref. value	MU (Ref.)	Reprod. S.D.	Repeat. S.D.	Assigned value	Assigned SD	Assigned SD
											%
Gasket type 1	DINCH	%	4.969	0.279			0.731	0.194	4.969	0.749	15.00
Gasket type 1	ESBO-epoxydised	%	22.088	2.754			6.640	0.804	22.088	3.301	15.00
Gasket type 1	butyl sebacate	%	3.363	0.198			0.505	0.121	3.363	0.508	15.00
Gasket type 2	DIDP-diisodecyl	%	32.753	1.796			5.038	1.254	32.753	4.926	15.00
oil ESBO based	BHT-butylated	mg/kg	2.618	0.384	2.960	0.126	0.733	0.169	2.960	0.402	13.50
oil ESBO based	DINCH	mg/kg	14.560	1.859	14.860	0.268	4.230	0.896	14.860	1.584	10.66
oil ESBO based	ESBO-epoxydised	mg/kg	59.974	5.856	60.530	1.444	3.244	2.298	60.530	5.222	8.63
oil ESBO based	butyl sebacate	mg/kg	3.230	0.364	3.110	0.150	0.821	0.118	3.110	0.419	13.48
oil phthalate	BHT-butylated	mg/kg	1.011	0.109	1.070	0.022	0.214	0.078	1.070	0.169	15.83
oil phthalate	DIDP-diisodecyl	mg/kg	7.705	1.410	8.000	0.174	2.914	0.512	8.000	0.936	11.70
oil phthalate	butyl sebacate	mg/kg	1.501	0.187	1.440	0.062	0.426	0.090	1.440	0.218	15.14
oil phthalate	BBP-benzyl butyl	mg/kg	14.980	1.218	14.9700	0.288	2.751	0.438	14.970	1.594	10.64

Note: the results are reported as they come out from the software because exactly those figures are taken afterwards for z and z' score calculations. Any rounding made in the table of that report may result in lack of traceability for the further calculations

Table 4. Summary of the test results and corresponding z'-score

Sample:	**Gasket type 1 ESBO base**	**Assigned value:**	**4.97 % (Empirical value)**
Measurand:	**DINCH**	**Rel. target s.d.:**	**15.08%**
No. of laboratories:	**26**	**Rel. reproducibility s.d.:**	**14.72%**
Limits of tolerance:	**3.47 - 6.47 %**	**(|Z'-Score| < 2.00)**	

Laboratory code	M	M 1	M 2	M 3	M 4	S.d.	Z score
LC0000	6.525	6.500	7.100	6.400	6.100	0.419	2.041
LC0002	4.900	5.000	5.000	4.800	4.800	0.115	-0.091
LC0003	5.805	5.890	5.930	5.750	5.650	0.129	1.096
LC0004							
LC0005	5.021	5.015	5.101	4.971	4.997	0.056	0.068
LC0006	5.255	5.200	5.280	5.310	5.230	0.049	0.375
LC0009							
LC0010							
LC0011							
LC0012							
LC0013	2.703	2.740	2.790	2.680	2.600	0.082	-2.974
LC0014							
LC0016							
LC0017	4.450	4.500	4.200	4.500	4.600	0.173	-0.681
LC0018	5.000	5.100	5.300	4.900	4.700	0.258	0.040
LC0020	0.443	0.422	0.446	0.444	0.461	0.016	-5.938
LC0021	5.175	4.900	5.000	5.400	5.400	0.263	0.270
LC0025							
LC0026							
LC0028	4.808	4.720	4.790	4.740	4.980	0.119	-0.212
LC0029							
LC0031	5.375	5.300	5.300	5.400	5.500	0.096	0.532
LC0033	4.950	4.800	5.000	5.000	5.000	0.100	-0.025
LC0035							
LC0037	5.625	5.300	5.800	6.000	5.400	0.330	0.860
LC0038	4.050	3.900	4.200	3.700	4.400	0.311	-1.206
LC0040	5.098	5.060	5.030	5.030	5.270	0.116	0.168
LC0041	3.600	3.800	3.600	3.600	3.400	0.163	-1.796
LC0042							
LC0043	5.550	4.300	6.800			1.768	0.762
LC0044	4.900	4.700	4.700	5.000	5.200	0.245	-0.091
LC0046	4.933	4.950	4.990	4.830	4.960	0.070	-0.048
LC0047							
LC0048	4.403	4.670	4.730	5.140	3.070	0.913	-0.744
LC0049	5.160	4.940	5.020	5.180	5.500	0.248	0.250
LC0050							
LC0051							
LC0052	5.350	5.800	5.800	4.700	5.100	0.545	0.499
LC0054	5.125	4.700	5.400	5.700	4.700	0.506	0.204
LC0055	2.872	2.980	3.050	2.770	2.690	0.170	-2.751
LC0056	5.545	5.320	5.430	5.620	5.810	0.216	0.755

Table 2. Summary of the test results and corresponding z'-score

Sample:	**Gasket type 1 ESBO base**	**Assigned value:**	**22.01 % (Empirical value)**
Measurand:	**ESBO-epoxydised soyabeen oil**	**Rel. target s.d.:**	**15.00%**
No. of laboratories:	**23**	**Rel. reproducibility s.d.:**	**30.17%**
Limits of tolerance:	**15.41 - 28.61 %**	**(|Z'-Score| < 2.00)**	

Laboratory code	M	M 1	M 2	M 3	M 4	S.d.	Z score
LC0000							
LC0002	25.350	25.800	25.900	24.500	25.200	0.645	0.934
LC0003	30.025	30.900	30.200	30.300	28.700	0.936	2.241
LC0004							
LC0005	26.803	25.756	27.697	25.941	27.818	1.106	1.341
LC0006	20.293	22.100	20.230	20.020	18.820	1.356	-0.480
LC0009							
LC0010							
LC0011							
LC0012							
LC0013	27.900	27.300	28.400	29.400	26.500	1.268	1.647
LC0014							
LC0016							
LC0017	29.525	28.300	29.600	30.300	29.900	0.866	2.102
LC0018	5.675	5.800	5.900	5.800	5.200	0.320	-4.566
LC0020	27.575	29.600	28.200	28.800	23.700		
LC0021	22.525	22.800	22.800	22.000	22.500	0.377	0.145
LC0025	20.875	21.200	20.800	21.500	20.000	0.650	-0.317
LC0026							
LC0028							
LC0029							
LC0031	23.400	23.700	23.200	23.600	23.100	0.294	0.389
LC0033	20.225	20.500	19.700	20.300	20.400	0.359	-0.498
LC0035							
LC0037	25.225	25.400	25.000	25.100	25.400	0.206	0.899
LC0038	22.275	22.300	22.500	21.100	23.200	0.873	0.075
LC0040	20.925	20.430	20.540	21.740	20.990	0.595	-0.303
LC0041	45.567	46.400	42.800	47.500		2.458	6.586
LC0042							
LC0043	7.400	6.700	8.100			0.990	-4.084
LC0044	11.700	10.900	12.300	11.700	11.900	0.589	-2.882
LC0046	26.175	25.500	26.000	26.600	26.600	0.532	1.165
LC0047							
LC0048							
LC0049	16.385	15.530	16.350	16.640	17.020	0.633	-1.572
LC0050							
LC0051							
LC0052	26.875	27.000	27.400	26.600	26.500	0.411	1.361
LC0054	8.025	7.200	8.300	9.700	6.900	1.269	-3.909
LC0055	24.050	24.000	26.100	22.100	24.000	1.634	0.571
LC0056	20.833	21.900	19.300	21.300		1.361	-0.328

Table 2. Summary of the test results and corresponding z'-score

Sample:	**Gasket type 1 ESBO base**	**Assigned value:**	**3.36 % (Empirical value)**
Measurand:	**butyl sebacate**	**Rel. target s.d.:**	**15.11%**
No. of laboratories:	**25**	**Rel. reproducibility s.d.:**	**15.02%**
Limits of tolerance:	**2.35 - 4.38 %**	**(|Z'-Score| < 2.00)**	

Laboratory code	M	M 1	M 2	M 3	M 4	S.d.	Z score
LC0000	3.825	3.900	4.000	3.700	3.700	0.150	0.892
LC0002	3.675	3.700	3.700	3.600	3.700	0.050	0.603
LC0003	3.875	4.000	3.960	3.820	3.720	0.129	0.989
LC0004							
LC0005	3.505	3.452	3.503	3.481	3.585	0.057	0.275
LC0006	3.495	3.560	3.440	3.530	3.450	0.059	0.255
LC0009							
LC0010							
LC0011							
LC0012							
LC0013	1.573	1.650	1.620	1.520	1.500	0.074	-3.458
LC0014							
LC0016							
LC0017	3.025	3.000	2.900	3.100	3.100	0.096	-0.653
LC0018	5.750	5.800	5.400	5.700	6.100	0.289	4.610
LC0020	0.396	0.390	0.399	0.398	0.396	0.004	-5.731
LC0021	3.525	3.800	3.200	3.500	3.600	0.250	0.313
LC0025							
LC0026							
LC0028	3.338	3.270	3.350	3.290	3.440	0.076	-0.049
LC0029							
LC0031	3.775	3.800	3.700	3.800	3.800	0.050	0.796
LC0033	3.175	2.900	3.300	3.500	3.000	0.275	-0.363
LC0035							
LC0037	3.400	3.400	3.400	3.400	3.400	0.000	0.072
LC0038	3.475	3.500	3.400	3.600	3.400	0.096	0.216
LC0040	3.575	3.640	3.540	3.520	3.600	0.055	0.410
LC0041	2.625	2.900	2.400	3.000	2.200	0.386	-1.425
LC0042							
LC0043	2.050	1.900	2.200			0.212	-2.536
LC0044	3.475	3.400	3.300	3.600	3.600	0.150	0.216
LC0046	3.343	3.020	3.090	3.420	3.840	0.375	-0.040
LC0047							
LC0048							
LC0049	2.873	2.750	2.820	2.880	3.040	0.124	-0.947
LC0050							
LC0051							
LC0052	3.425	3.800	3.700	3.500	2.700	0.499	0.120
LC0054	3.000	2.900	3.000	3.300	2.800	0.216	-0.701
LC0055	3.853	3.820	3.830	3.930	3.830	0.052	0.945
LC0056	3.608	3.610	3.810	3.510	3.500	0.144	0.472

Table 2. Summary of the test results and corresponding z'-score

Sample:	**Gasket type 2 phthalate based**	**Assigned value:**	**32.75 % (Empirical value)**
Measurand:	**DIDP-diisodecyl phthalate**	**Rel. target s.d.:**	**15.04%**
No. of laboratories:	**30**	**Rel. reproducibility s.d.:**	**15.38%**
Limits of tolerance:	**22.90 - 42.60 %**	**(|Z'-Score| < 2.00)**	

Laboratory code	M	M 1	M 2	M 3	M 4	S.d.	Z score
LC0000							
LC0002							
LC0003	34.400	34.600	35.000	34.400	33.600	0.589	0.329
LC0004	37.160	37.250	37.330	36.780	37.280	0.255	0.880
LC0005	28.422	29.644	28.369	27.869	27.805	0.853	-0.865
LC0006	30.348	30.440	30.510	29.890	30.550	0.308	-0.480
LC0009							
LC0010	36.180	39.470	32.750	42.910	29.590	6.091	0.684
LC0011							
LC0012	39.160	36.300	40.730	39.720	39.890	1.957	1.280
LC0013	35.815	36.600	36.120	33.490	37.050	1.596	0.612
LC0014							
LC0016							
LC0017	32.625	33.600	30.100	34.700	32.100	1.992	-0.025
LC0018	35.400	36.400	34.700	34.400	36.100	0.997	0.529
LC0020	3.390	3.350	3.450	3.300	3.460	0.078	-5.864
LC0021	35.625	35.200	35.700	35.900	35.700	0.299	0.574
LC0025	35.025	33.400	35.400	36.100	35.200	1.150	0.454
LC0026	30.045	29.770	29.870	30.060	30.480	0.314	-0.541
LC0028	34.950	33.400	36.500	34.000	35.900	1.484	0.439
LC0029							
LC0031	37.125	36.900	36.600	37.300	37.700	0.479	0.873
LC0033	30.150	26.200	30.500	31.900	32.000	2.721	-0.520
LC0035							
LC0037	25.875	27.600	25.400	25.100	25.400	1.159	-1.374
LC0038	33.600	34.000	34.500	33.700	32.200	0.990	0.169
LC0040	36.778	36.630	34.890	41.160	34.430	3.071	0.804
LC0041	35.525	34.700	38.000	40.000	29.400	4.631	0.554
LC0042	14.600	14.600					-3.625
LC0043	26.600	27.900	25.300			1.838	-1.229
LC0044	29.875	28.700	27.200	31.700	31.900	2.307	-0.575
LC0046	31.775	34.400	31.900	30.000	30.800	1.916	-0.195
LC0047							
LC0048	31.300	30.600	30.500	31.900	32.200	0.876	-0.290
LC0049	34.020	34.690	33.920	33.270	34.200	0.593	0.253
LC0050							
LC0051							
LC0052	35.975	37.800	37.800	34.300	34.000	2.111	0.644
LC0054	30.100	34.300	22.600	33.400		6.511	-0.530
LC0055	24.523	25.090	24.600	23.410	24.990	0.771	-1.644
LC0056	32.543	31.570	32.530	33.280	32.790	0.719	-0.042

Table 2. Summary of the test results and corresponding z'-score

Sample:	**oil ESBO based**	**Assigned value:**	**2.96 mg/kg (Reference value)**
Measurand:	**BHT-butylated hyrdotoluene**	**Rel. target s.d.:**	**13.59% (Horwitz function)**
No. of laboratories:	**14**	**Rel. reproducibility s.d.:**	**24.75%**
Limits of tolerance:	**2.16 - 3.76 mg/kg (|Z'-Score| < 2.00)**		

Laboratory code	M	M 1	M 2	M 3	M 4	S.d.	Z score
LC0000							
LC0002							
LC0003	2.878	2.860	2.950	2.830	2.870	0.051	-0.196
LC0004							
LC0005	3.351	3.380	3.270	3.492	3.263	0.108	0.928
LC0006							
LC0009							
LC0010							
LC0011							
LC0012							
LC0013	2.043	1.940	2.010	2.160	2.060	0.093	-2.177
LC0014							
LC0016							
LC0017							
LC0018	2.715	2.210	3.040	2.930	2.680	0.369	-0.581
LC0020	1.658	1.720	1.500	1.480	1.930	0.212	-3.091
LC0021							
LC0025							
LC0026							
LC0028	1.595	1.630	1.530	1.500	1.720	0.100	-3.239
LC0029							
LC0031	2.725	2.700	2.700	2.600	2.900	0.126	-0.558
LC0033							
LC0035							
LC0037	2.625	2.620	2.630	2.670	2.580	0.037	-0.795
LC0038	2.415	2.450	2.760	2.300	2.150	0.261	-1.293
LC0040	3.308	3.250	3.310	3.320	3.350	0.042	0.825
LC0041							
LC0042							
LC0043							
LC0044	2.750	2.700	2.700	2.700	2.900	0.100	-0.498
LC0046	2.440	2.430	2.460	2.090	2.780	0.282	-1.234
LC0047							
LC0048							
LC0049	4.700	4.100	5.000	5.200	4.500	0.497	4.129
LC0050							
LC0051							
LC0052							
LC0054							
LC0055	2.435	2.520	2.660	2.190	2.370	0.202	-1.246
LC0056							

Table 2. Summary of the test results and corresponding z'-score

Sample:	**oil ESBO based**	**Assigned value:**	**14.86 mg/kg (Reference value)**
Measurand:	**DINCH**	**Rel. target s.d.:**	**10.66% (Horwitz function)**
No. of laboratories:	**20**	**Rel. reproducibility s.d.:**	**28.46%**
Limits of tolerance:	**11.69 - 18.03 mg/kg (|Z'-Score| < 2.00)**		

Laboratory code	M	M 1	M 2	M 3	M 4	S.d.	Z score
LC0000							
LC0002	16.250	17.000	16.000	16.000	16.000	0.500	0.865
LC0003	12.275	11.700	12.900	12.400	12.100	0.506	-1.609
LC0004							
LC0005	10.091	11.251	9.811	9.656	9.647	0.777	-2.969
LC0006							
LC0009							
LC0010							
LC0011							
LC0012							
LC0013	11.983	12.140	11.860	11.620	12.310	0.305	-1.791
LC0014							
LC0016							
LC0017	18.350	19.300	17.000	16.900	20.200	1.658	2.173
LC0018	0.000	0.000	0.000	0.000	0.000	0.000	-9.252
LC0020	8.028	8.700	9.680	7.570	6.160	1.514	-4.254
LC0021	20.450	20.200	20.400	19.700	21.500	0.759	3.480
LC0025							
LC0026							
LC0028	17.775	17.600	17.800	18.400	17.300	0.465	1.815
LC0029							
LC0031	15.475	15.200	15.400	15.900	15.400	0.299	0.383
LC0033	17.500	18.000	18.000	17.000	17.000	0.577	1.644
LC0035							
LC0037	11.477	10.430	11.410	11.450	12.620	0.896	-2.106
LC0038	15.700	14.500	15.300	16.000	17.000	1.061	0.523
LC0040	18.275	19.800	18.700	17.300	17.300	1.212	2.126
LC0041							
LC0042							
LC0043							
LC0044	16.550	16.100	17.100	16.200	16.800	0.480	1.052
LC0046	13.450	13.200	13.300	13.600	13.700	0.238	-0.878
LC0047							
LC0048							
LC0049	16.350	12.900	20.100	16.500	15.900	2.955	0.928
LC0050							
LC0051							
LC0052	14.000	16.000	13.000	14.000	13.000	1.414	-0.535
LC0054	16.750	16.000	16.000	18.000	17.000	0.957	1.177
LC0055	10.188	10.200	10.150	10.060	10.340	0.117	-2.909
LC0056							

Table 2. Summary of the test results and corresponding z'-score

Sample:	**oil ESBO based**	**Assigned value:**	**60.53 mg/kg (Reference value)**
Measurand:	**ESBO-epoxydised soyabeen oil**	**Rel. target s.d.:**	**8.63% (Horwitz function)**
No. of laboratories:	**20**	**Rel. reproducibility s.d.:**	**21.88%**
Limits of tolerance:	**50.09 - 70.97 mg/kg (|Z'-Score| < 2.00)**		

Laboratory code	M	M 1	M 2	M 3	M 4	S.d.	Z score
LC0000							
LC0002	59.000	59.000	60.000	55.000	62.000	2.944	-0.282
LC0003	72.725	72.500	70.700	73.900	73.800	1.493	2.251
LC0004							
LC0005	65.197	63.276	65.260	63.216	69.036	2.730	0.861
LC0006	68.650	70.600	68.700	74.600	60.700	5.843	1.499
LC0009							
LC0010							
LC0011							
LC0012							
LC0013	75.500	77.000	70.000	72.000	83.000	5.802	2.763
LC0014							
LC0016							
LC0017	78.525	77.900	72.400	77.800	86.000	5.607	3.321
LC0018	52.025	53.100	45.000	54.000	56.000	4.838	-1.570
LC0020	46.275	48.700	50.700	41.100	44.600	4.284	-2.631
LC0021	56.000	56.000	55.000	56.000	57.000	0.816	-0.836
LC0025							
LC0026							
LC0028							
LC0029							
LC0031	36.750	37.000	34.000	39.000	37.000	2.062	-4.389
LC0033	51.500	56.000	47.000	51.000	52.000	3.697	-1.667
LC0035							
LC0037	57.100	55.100	60.000	56.200	57.100	2.099	-0.633
LC0038	55.725	55.600	54.300	58.100	54.900	1.670	-0.887
LC0040	60.150	58.370	60.410	60.110	61.710	1.375	-0.070
LC0041							
LC0042							
LC0043							
LC0044	64.100	63.800	64.400			0.424	0.659
LC0046	61.575	60.600	62.000	59.400	64.300	2.105	0.193
LC0047							
LC0048							
LC0049							
LC0050							
LC0051							
LC0052	66.500	68.000	65.000			2.121	1.102
LC0054	147.667	148.000	148.000	147.000		0.577	16.083
LC0055	68.750	69.000	68.000	68.000	70.000	0.957	1.517
LC0056	39.005	38.950	39.060			0.078	-3.973

Table 2. Summary of the test results and corresponding z'-score

Sample:	**oil ESBO based**	**Assigned value:**	**3.11 mg/kg (Reference value)**
Measurand:	**butyl sebacate**	**Rel. target s.d.:**	**13.49% (Horwitz function)**
Method:	**DIN 38402 A45**	**Rel. repeatability s.d.:**	**3.78%**
No. of laboratories:	**20**	**Limits of tolerance:**	**2.27 - 3.95 mg/kg (|Z'-Score| < 2.**

Laboratory code	M	M 1	M 2	M 3	M 4	S.d.	Z score
LC0038	3.200	3.200	3.000	3.400	3.200	0.163	0.202
LC0021	3.775	3.800	3.700	3.800	3.800	0.050	1.493
LC0054	2.750	2.000	3.000	3.000	3.000	0.500	-0.808
LC0042							
LC0014							
LC0026							
LC0000							
LC0012							
LC0043							
LC0002	4.000	4.000	4.000	4.000	4.000	0.000	1.998
LC0047							
LC0046	3.203	3.200	3.270	3.290	3.050	0.109	0.208
LC0033	3.975	3.900	4.500	3.800	3.700	0.359	1.942
LC0051							
LC0035							
LC0040	3.525	3.500	3.600	3.500	3.500	0.050	0.932
LC0018	2.950	2.180	3.550	3.350	2.720	0.623	-0.359
LC0052	2.750	3.000	4.000	2.000	2.000	0.957	-0.808
LC0004							
LC0055	1.868	1.840	1.890	1.870	1.870	0.021	-2.789
LC0017	3.850	3.900	3.700	3.500	4.300	0.342	1.661
LC0028	4.555	4.520	4.530	4.630	4.540	0.051	3.244
LC0056							
LC0013	2.973	2.900	3.010	3.020	2.960	0.055	-0.309
LC0025							
LC0003	3.763	3.730	3.860	3.680	3.780	0.077	1.465
LC0041							
LC0037	2.775	2.700	2.800	2.950	2.650	0.132	-0.752
LC0020	1.135	0.900	1.280	1.180	1.180	0.164	-4.434
LC0016							
LC0031	3.025	3.000	3.100	3.000	3.000	0.050	-0.191
LC0011							
LC0050							
LC0029							
LC0009							
LC0048							
LC0005	3.685	3.647	3.604	3.902	3.586	0.147	1.290
LC0049	2.900	2.600	3.400	2.500	3.100	0.424	-0.471
LC0010							
LC0006							
LC0044	3.050	3.000	3.100	3.000	3.100	0.058	-0.135

Table 2. Summary of the test results and corresponding z'-score

Sample:	**oil phthalate based**	**Assigned value:**	**14.970 mg/kg (Reference value)**
Measurand:	**BBP-benzyl butyl phthalate**	**Rel. target s.d.:**	**10.65% (Horwitz function)**
Method:	**DIN 38402 A45**	**Rel. repeatability s.d.:**	**3.46%**
No. of laboratories:	**19**	**Limits of tolerance:**	**11.783 - 18.157 mg/kg (|Z'-Score|**

Laboratory code	M	M 1	M 2	M 3	M 4	S.d.	Z score
LC0000							
LC0002							
LC0003	14.075	14.100	14.200	14.100	13.900	0.126	-0.553
LC0004	23.350	23.100	23.300	23.300	23.700	0.252	5.175
LC0005	15.792	15.691	15.909	15.928	15.638	0.148	0.507
LC0006							
LC0009							
LC0010	10.332	8.640	10.210	12.450	10.030	1.576	-2.864
LC0011							
LC0012							
LC0013	16.175	15.310	16.140	17.990	15.260	1.276	0.744
LC0014							
LC0016							
LC0017	16.775	17.700	15.700	17.000	16.700	0.830	1.115
LC0018	15.563	18.200	18.700	13.950	11.400	3.499	0.366
LC0020							
LC0021	15.775	16.000	15.400	15.800	15.900	0.263	0.497
LC0025							
LC0026							
LC0028	16.925	17.200	17.000	16.900	16.600	0.250	1.207
LC0029							
LC0031	15.150	15.200	15.200	15.400	14.800	0.252	0.111
LC0033	13.750	13.000	13.000	14.000	15.000	0.957	-0.753
LC0035							
LC0037	14.260	14.410	13.640	15.000	13.990	0.585	-0.438
LC0038	15.150	15.000	15.500	15.300	14.800	0.311	0.111
LC0040	14.275	14.800	14.600	14.000	13.700	0.512	-0.429
LC0041							
LC0042							
LC0043							
LC0044	15.850	15.800	15.800	15.900	15.900	0.058	0.543
LC0046	14.450	14.300	15.000	14.700	13.800	0.520	-0.321
LC0047							
LC0048							
LC0049	21.750	15.300	22.900	26.700	22.100	4.745	4.187
LC0050							
LC0051							
LC0052							
LC0054	10.750	11.000	9.000	11.000	12.000	1.258	-2.606
LC0055	8.338	8.390	8.180	8.380	8.400	0.105	-4.096
LC0056							

Table 2. Summary of the test results and corresponding z'-score

Sample:	**oil phthalate based**	**Assigned value:**	**1.07 mg/kg (Reference value)**
Measurand:	**BHT-butylated hyrdotoluene**	**Rel. target s.d.:**	**15.83% (Horwitz function)**
No. of laboratories:	**14**	**Rel. reproducibility s.d.:**	**20.01%**
Limits of tolerance:	**0.73 - 1.41 mg/kg (|Z'-Score| < 2.00)**		

Laboratory code	M	M 1	M 2	M 3	M 4	S.d.	Z score
LC0000							
LC0002							
LC0003	0.997	1.020	1.010	0.986	0.970	0.023	-0.430
LC0004							
LC0005	0.870	0.885	0.892	0.850	0.854	0.021	-1.169
LC0006							
LC0009							
LC0010							
LC0011							
LC0012							
LC0013	0.970	0.800	0.940	1.090	1.050	0.130	-0.585
LC0014							
LC0016							
LC0017							
LC0018	1.150	1.060	1.060	0.980	1.500	0.236	0.468
LC0020	0.665	0.620	0.660	0.680	0.700	0.034	-2.370
LC0021							
LC0025							
LC0026							
LC0028	0.950	1.010	0.940	0.880	0.970	0.055	-0.702
LC0029							
LC0031	1.075	1.000	1.200	1.100	1.000	0.096	0.029
LC0033							
LC0035							
LC0037	0.928	0.780	1.040	1.070	0.820	0.149	-0.834
LC0038	1.100	1.070	1.090	1.130	1.110	0.026	0.176
LC0040	1.275	1.290	1.280	1.260	1.270	0.013	1.200
LC0041							
LC0042							
LC0043							
LC0044	1.025	1.100	1.000	1.000	1.000	0.050	-0.263
LC0046	1.053	1.260	0.950	1.030	0.970	0.142	-0.102
LC0047							
LC0048							
LC0049	1.675	1.600	1.400	2.200	1.500	0.359	3.541
LC0050							
LC0051							
LC0052							
LC0054							
LC0055	0.765	0.780	0.630	0.900	0.750	0.111	-1.785
LC0056							

Table 2. Summary of the test results and corresponding z'-score

Sample:	**oil phthalate based**	**Assigned value:**	**8.00 mg/kg (Reference value)**
Measurand:	**DIDP-diisodecyl phthalate**	**Rel. target s.d.:**	**11.70% (Horwitz function)**
No. of laboratories:	**17**	**Rel. reproducibility s.d.:**	**36.76%**
Limits of tolerance:	**6.13 - 9.87 mg/kg (|Z'-Score| < 2.00)**		

Laboratory code	M	M 1	M 2	M 3	M 4	S.d.	Z score
LC0000							
LC0002							
LC0003	6.785	7.050	6.440	7.200	6.450	0.397	-1.276
LC0004							
LC0005	6.975	6.975	6.501	6.991	7.431	0.380	-1.077
LC0006							
LC0009							
LC0010	30.608	28.700	29.640	30.250	33.840	2.247	23.750
LC0011							
LC0012							
LC0013	6.605	7.050	6.310	6.640	6.420	0.327	-1.465
LC0014							
LC0016							
LC0017	8.175	7.900	7.600	8.700	8.500	0.512	0.184
LC0018	35.475	46.500	26.300	33.600	35.500	8.352	28.863
LC0020	5.743	5.640	5.910	5.850	5.570	0.163	-2.372
LC0021	8.725	8.100	8.700	8.500	9.600	0.634	0.762
LC0025							
LC0026							
LC0028	10.475	10.800	10.300	10.100	10.700	0.330	2.600
LC0029							
LC0031	8.050	8.100	8.200	7.800	8.100	0.173	0.053
LC0033		<5.00	<5.00	<5.00	<5.00		
LC0035							
LC0037	5.818	6.030	5.680	5.960	5.600	0.210	-2.293
LC0038	8.875	9.000	8.800	9.200	8.500	0.299	0.919
LC0040		<20.00	<20.00	<20.00	<20.00		
LC0041							
LC0042							
LC0043							
LC0044	7.450	8.400	7.300	7.000	7.100	0.645	-0.578
LC0046	9.000	8.700	10.200	8.700	8.400	0.812	1.051
LC0047							
LC0048							
LC0049	10.350	10.100	12.400	10.400	8.500	1.601	2.469
LC0050							
LC0051							
LC0052	9.250	8.000	6.000	13.000	10.000	2.986	1.313
LC0054		<10.00	<10.00	<10.00	<10.00		
LC0055	2.963	2.680	2.810	3.410	2.950	0.318	-5.292
LC0056							

Table 2. Summary of the test results and corresponding z'-score

Sample:	**oil phthalate based**	**Assigned value:**	**1.44 mg/kg (Reference value)**
Measurand:	**butyl sebacate**	**Rel. target s.d.:**	**15.14% (Horwitz function)**
No. of laboratories:	**20**	**Rel. reproducibility s.d.:**	**29.59%**
Limits of tolerance:	**1.00 - 1.88 mg/kg**	**(|Z'-Score| < 2.00)**	

Laboratory code	M	M 1	M 2	M 3	M 4	S.d.	Z score
LC0000							
LC0002	1.250	1.000	2.000	1.000	1.000	0.500	-0.838
LC0003	2.230	2.270	2.230	2.200	2.220	0.029	3.485
LC0004							
LC0005	1.559	1.644	1.579	1.481	1.530	0.070	0.523
LC0006							
LC0009							
LC0010							
LC0011							
LC0012							
LC0013	1.500	1.470	1.310	1.790	1.430	0.205	0.265
LC0014							
LC0016							
LC0017	0.975	0.900	0.900	1.100	1.000	0.096	-2.051
LC0018	1.568	1.560	1.390	1.680	1.640	0.128	0.562
LC0020	1.300	1.150	1.450	1.280	1.320	0.124	-0.618
LC0021	2.125	2.100	2.200	2.100	2.100	0.050	3.022
LC0025							
LC0026							
LC0028	2.143	2.290	2.080	2.160	2.040	0.110	3.099
LC0029							
LC0031	1.325	1.400	1.300	1.300	1.300	0.050	-0.507
LC0033	1.475	1.500	1.500	1.600	1.300	0.126	0.154
LC0035							
LC0037	1.350	1.300	1.320	1.530	1.250	0.124	-0.397
LC0038	1.600	1.580	1.640	1.520	1.660	0.063	0.706
LC0040	1.925	1.900	2.000	1.900	1.900	0.050	2.139
LC0041							
LC0042							
LC0043							
LC0044	1.525	1.600	1.500	1.500	1.500	0.050	0.375
LC0046	1.470	1.490	1.530	1.460	1.400	0.055	0.132
LC0047							
LC0048							
LC0049	1.425	1.300	1.300	1.900	1.200	0.320	-0.066
LC0050							
LC0051							
LC0052	1.250	1.000	2.000	1.000	1.000	0.500	-0.838
LC0054	1.250	0.000	2.000	1.000	2.000	0.957	-0.838
LC0055	0.863	0.880	0.820	0.880	0.870	0.029	-2.547
LC0056							

Figure 1. Summary graphs of the laboratory's test results with their repeatability SD (a), Kernel Density plot (b) and z'-scores (c)

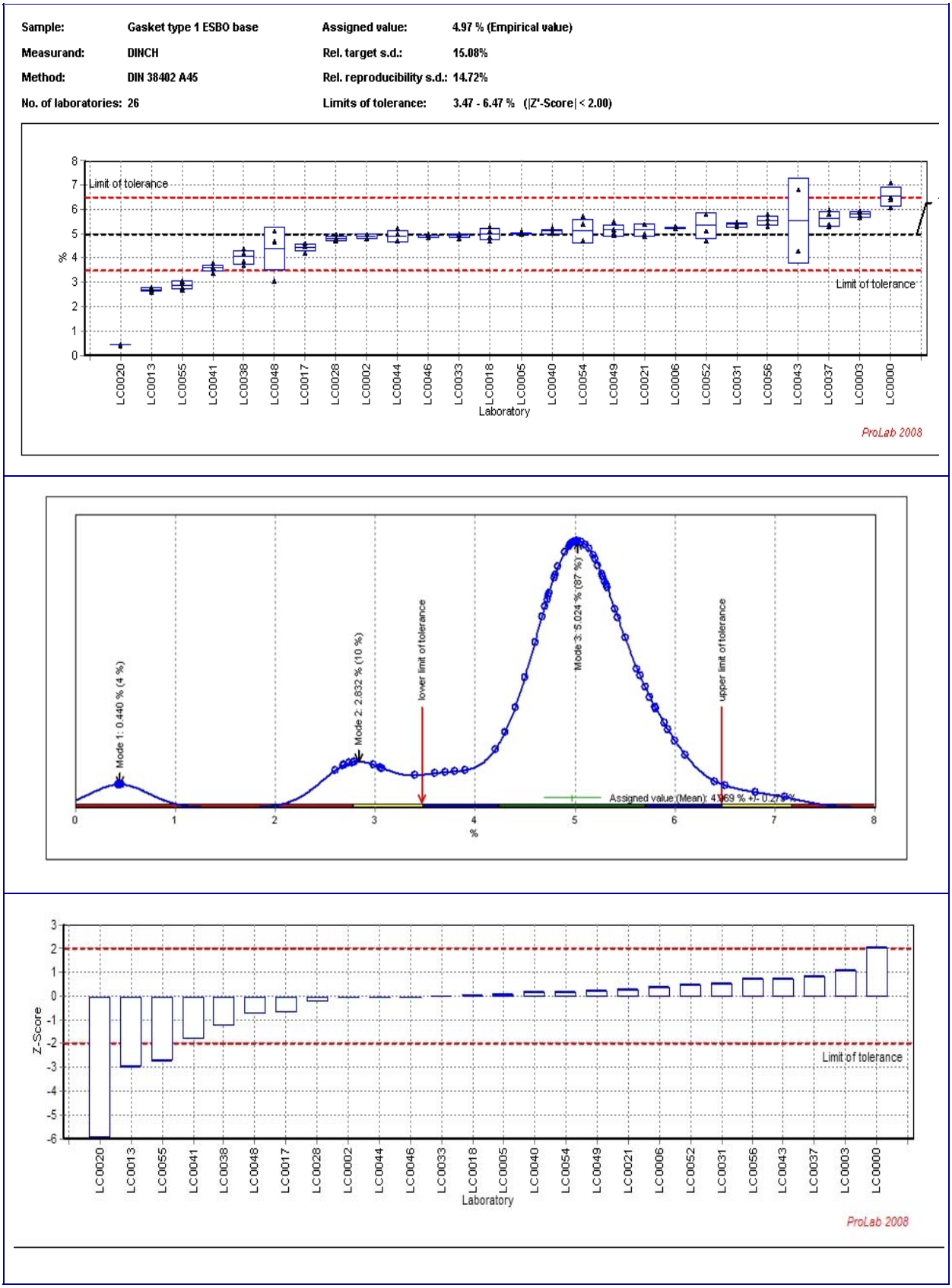
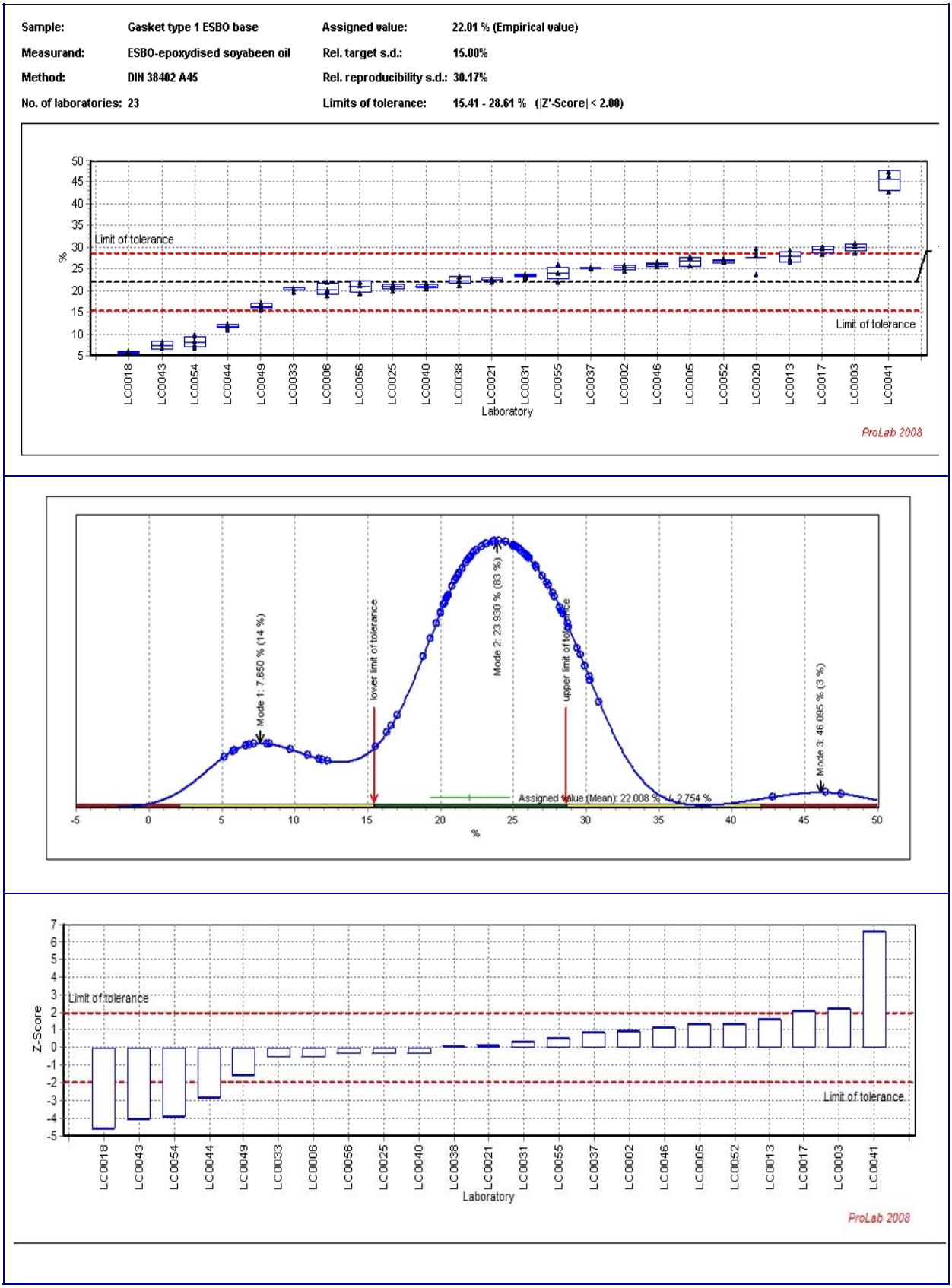


Figure 1. Summary graphs of the laboratory's test results with their repeatability SD (a), Kernel Density plot (b) and z'-scores (c)



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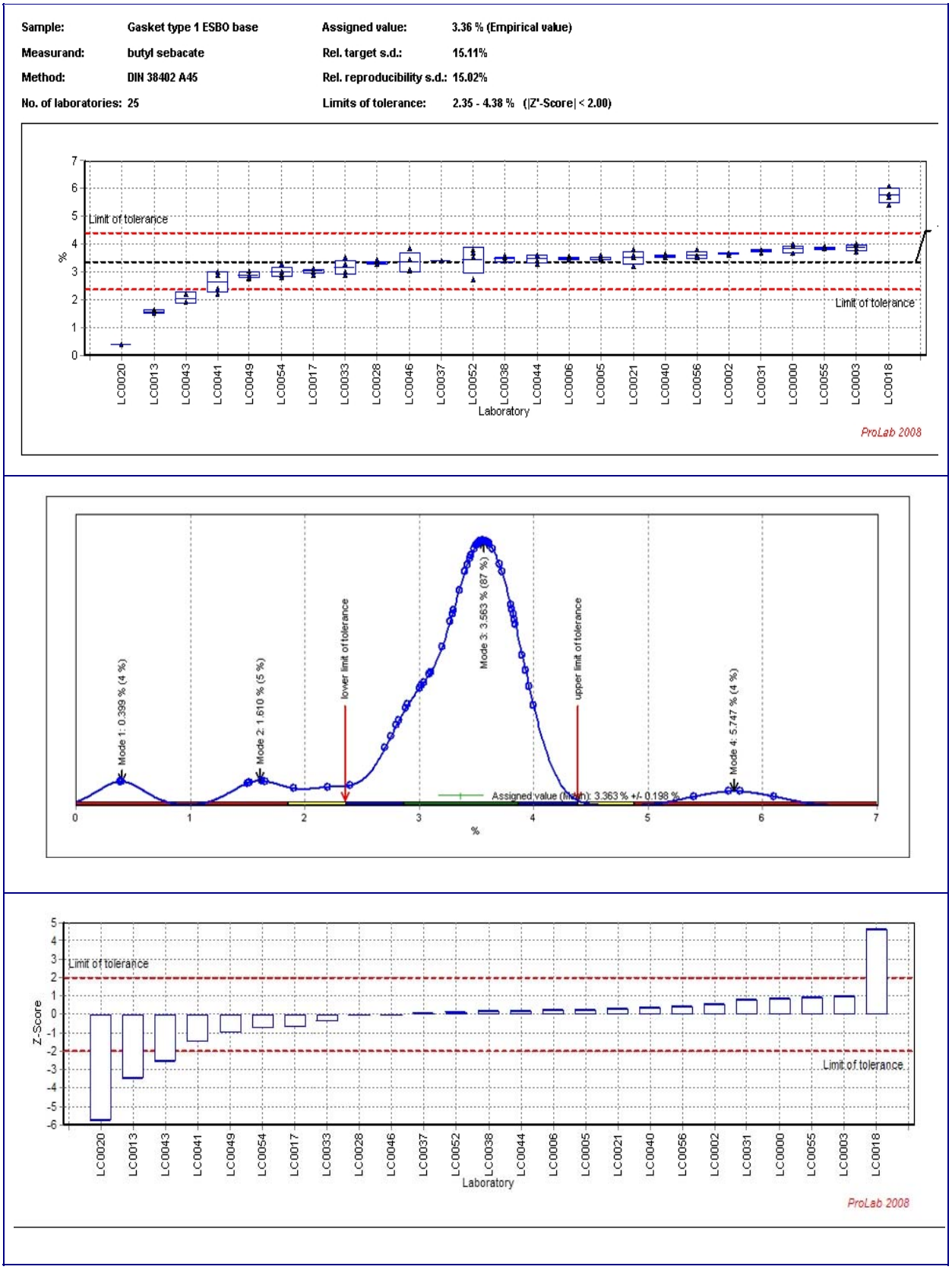

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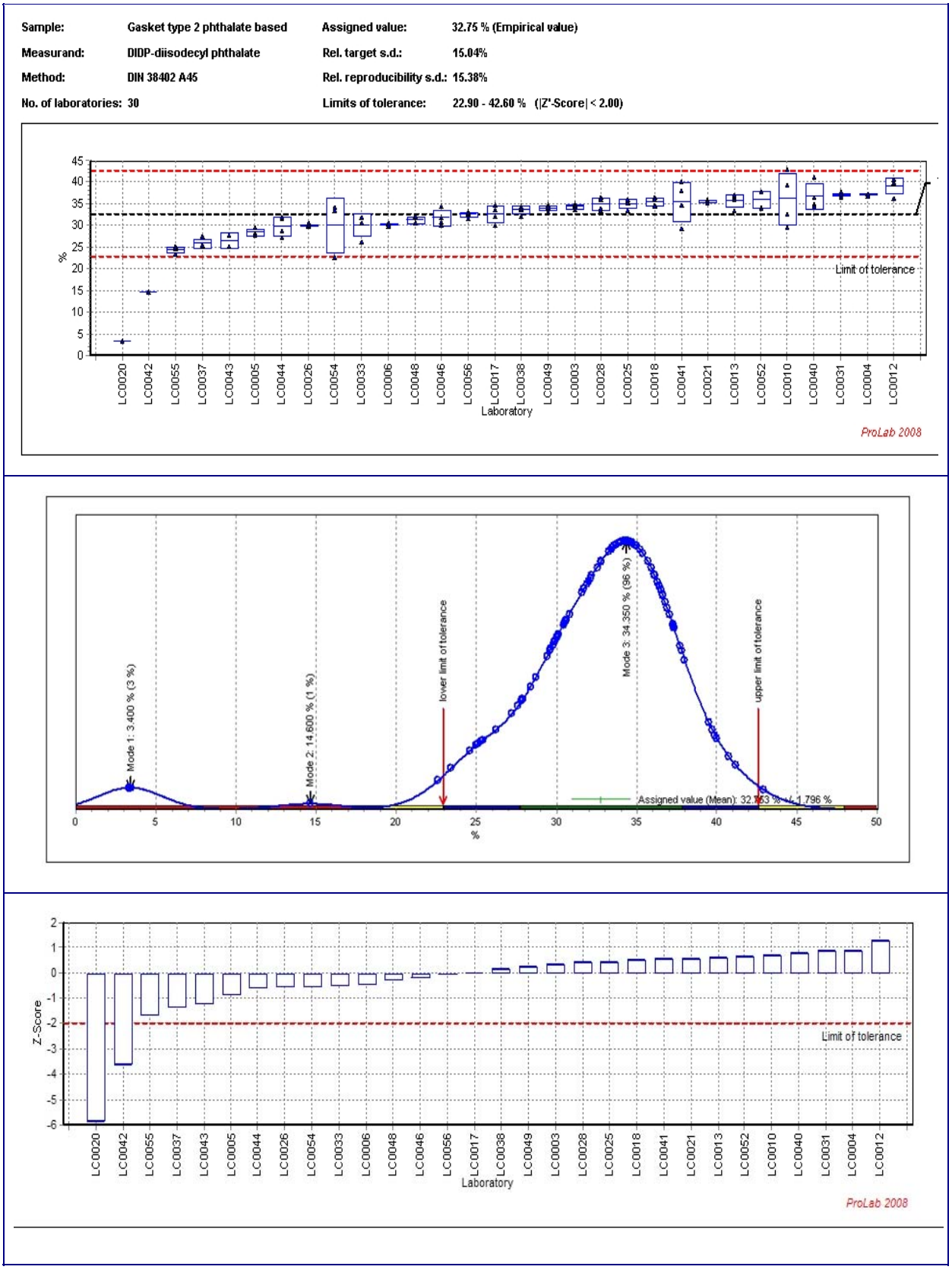

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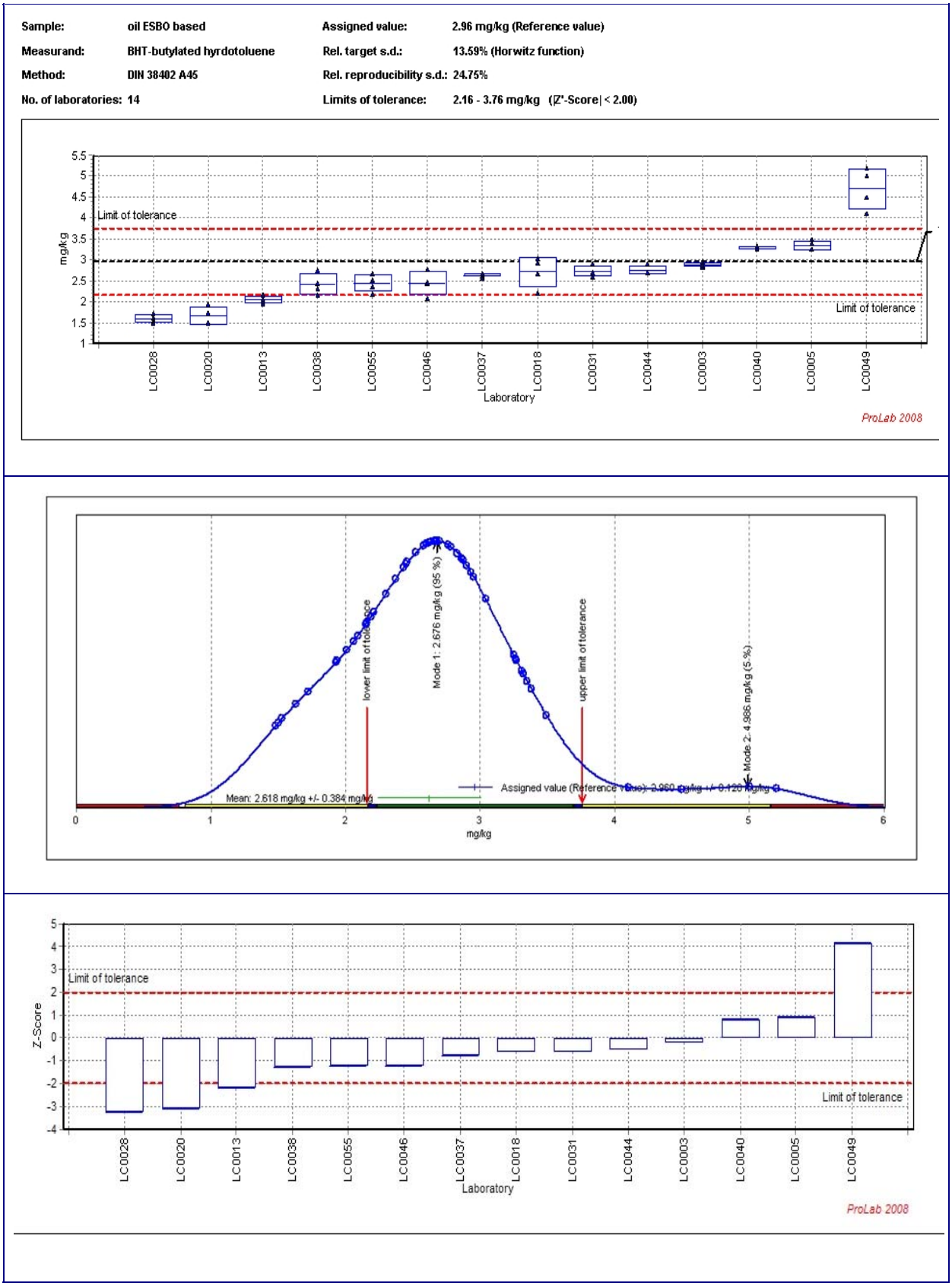


Figure 1. Summary graphs of the laboratory's test results with their repeatability SD (a), Kernel Density plot (b) and z'-scores (c)

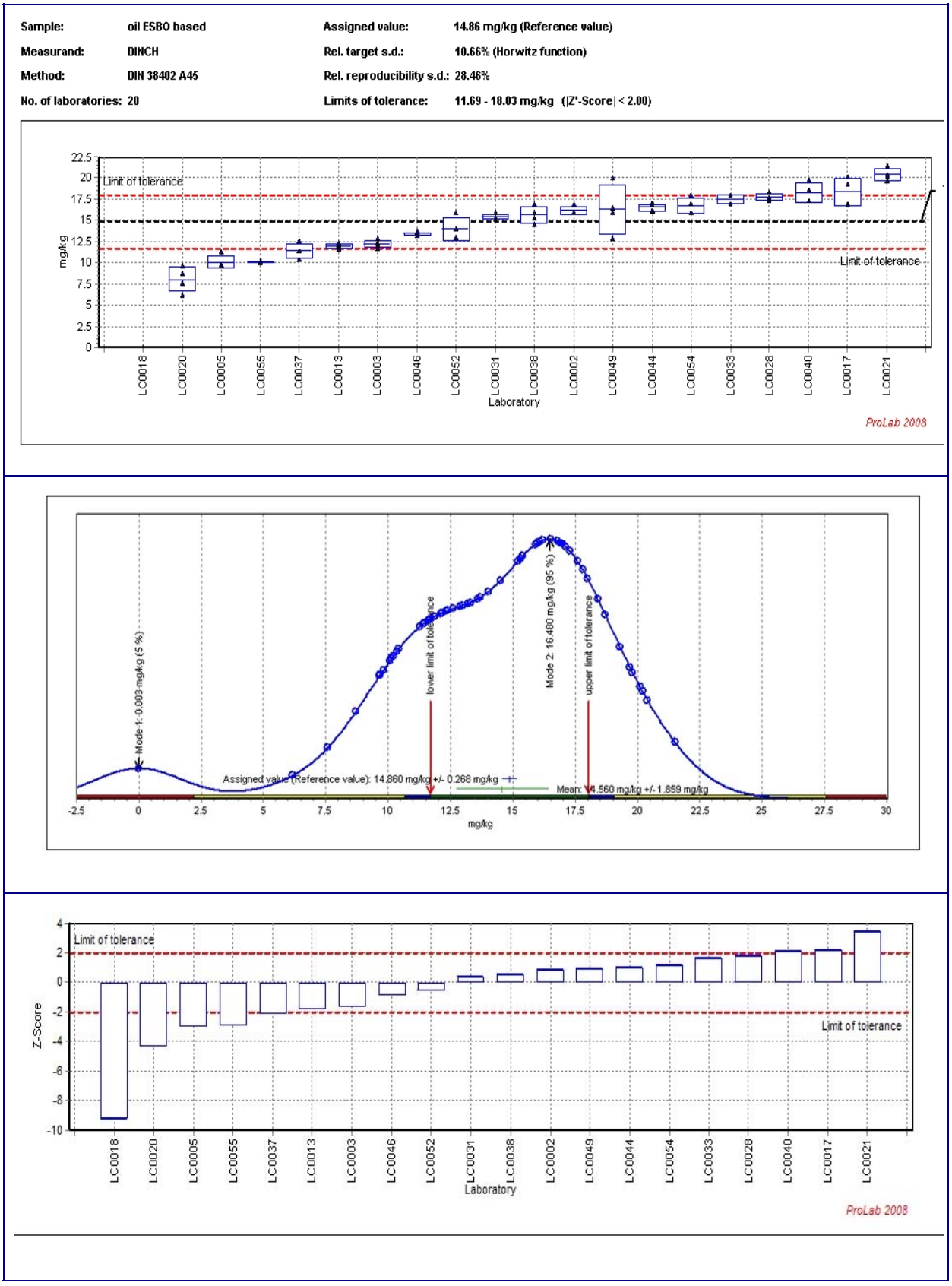


Figure 1. Summary graphs of the laboratory's test results with their repeatability SD (a), Kernel Density plot (b) and z'-scores (c)

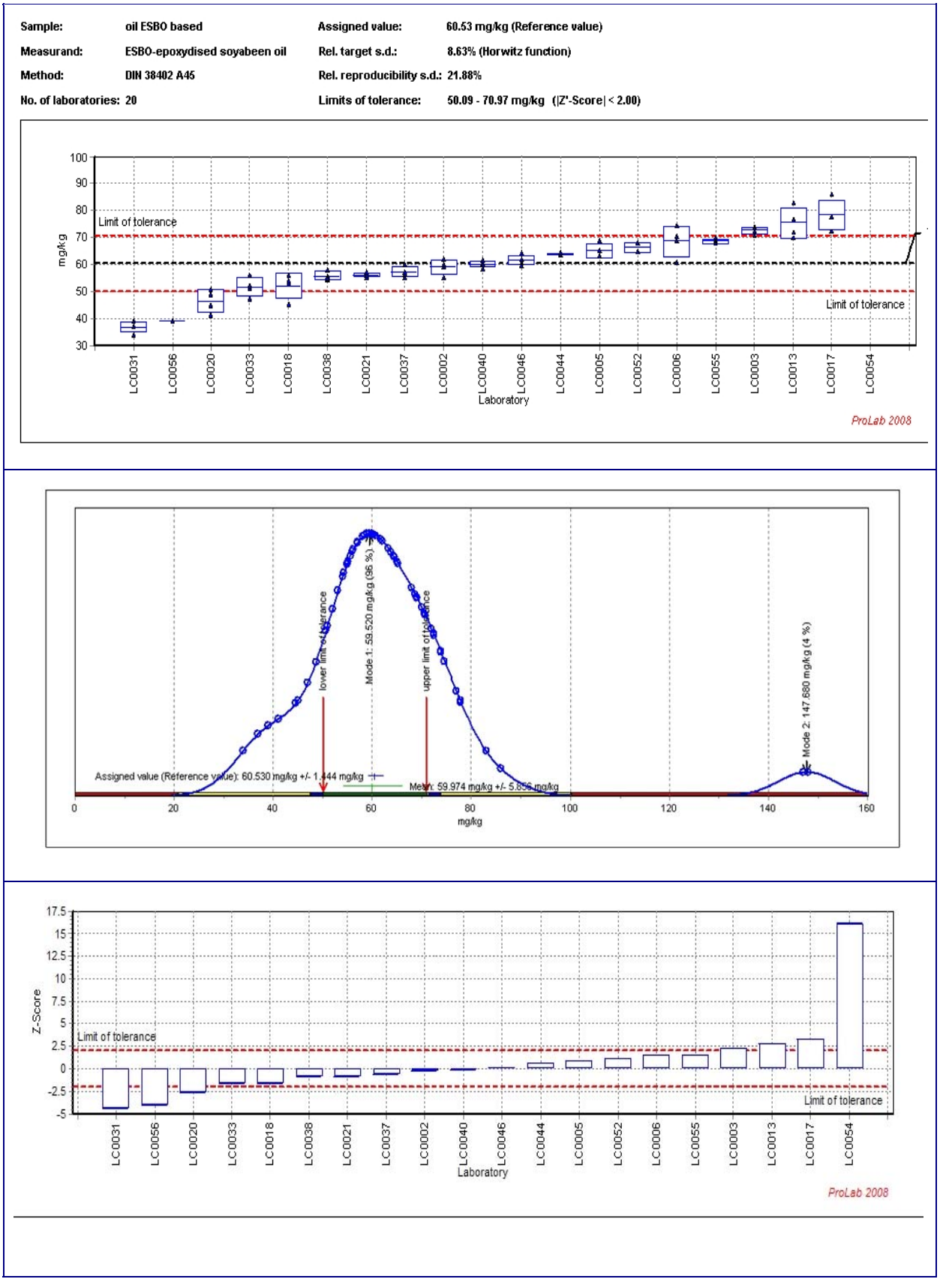


Figure 1. Summary graphs of the laboratory's test results with their repeatability SD (a), Kernel Density plot (b) and z'-scores (c)

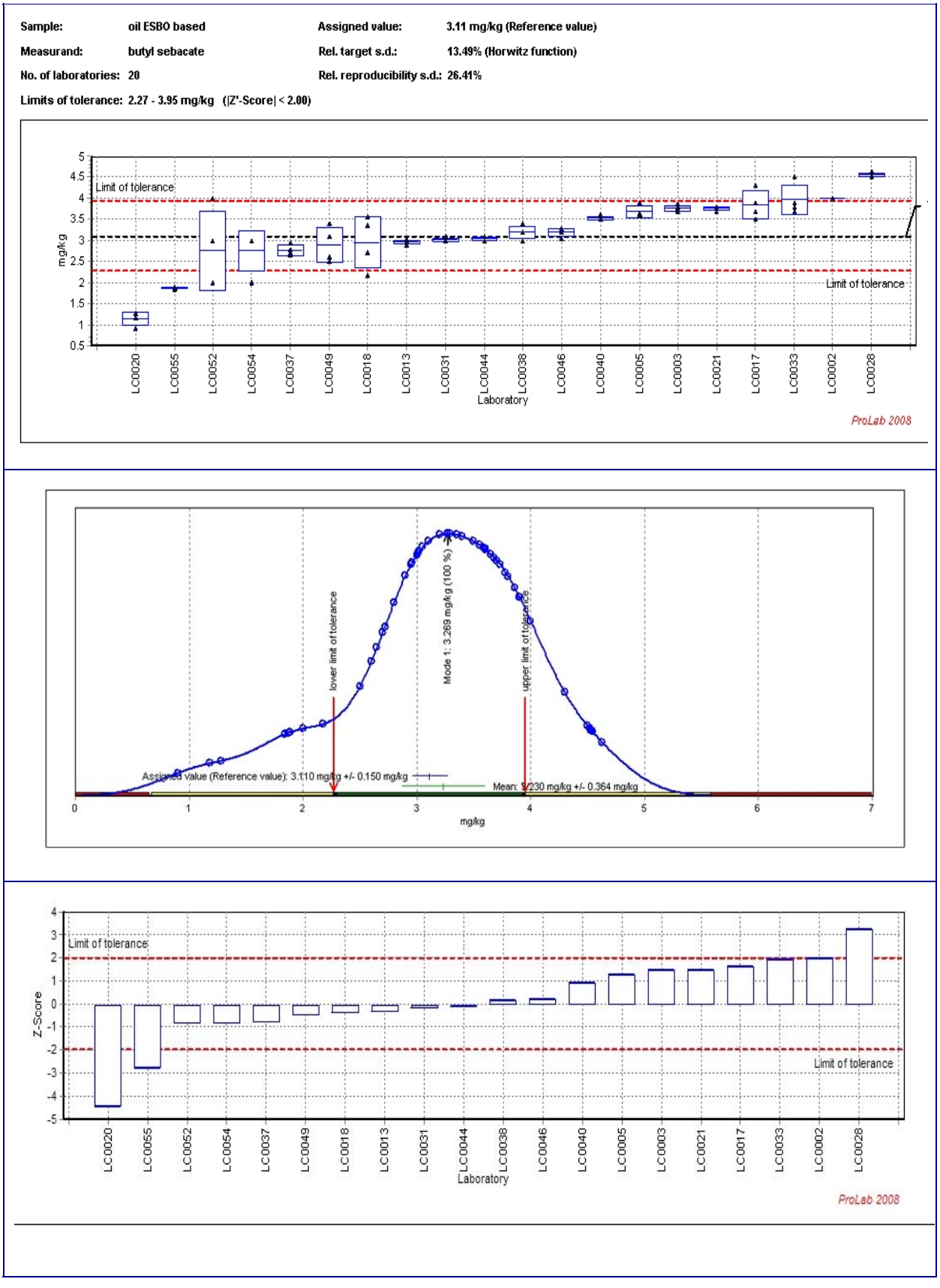


Figure 1. Summary graphs of the laboratory's test results with their repeatability SD (a), Kernel Density plot (b) and z'-scores (c)

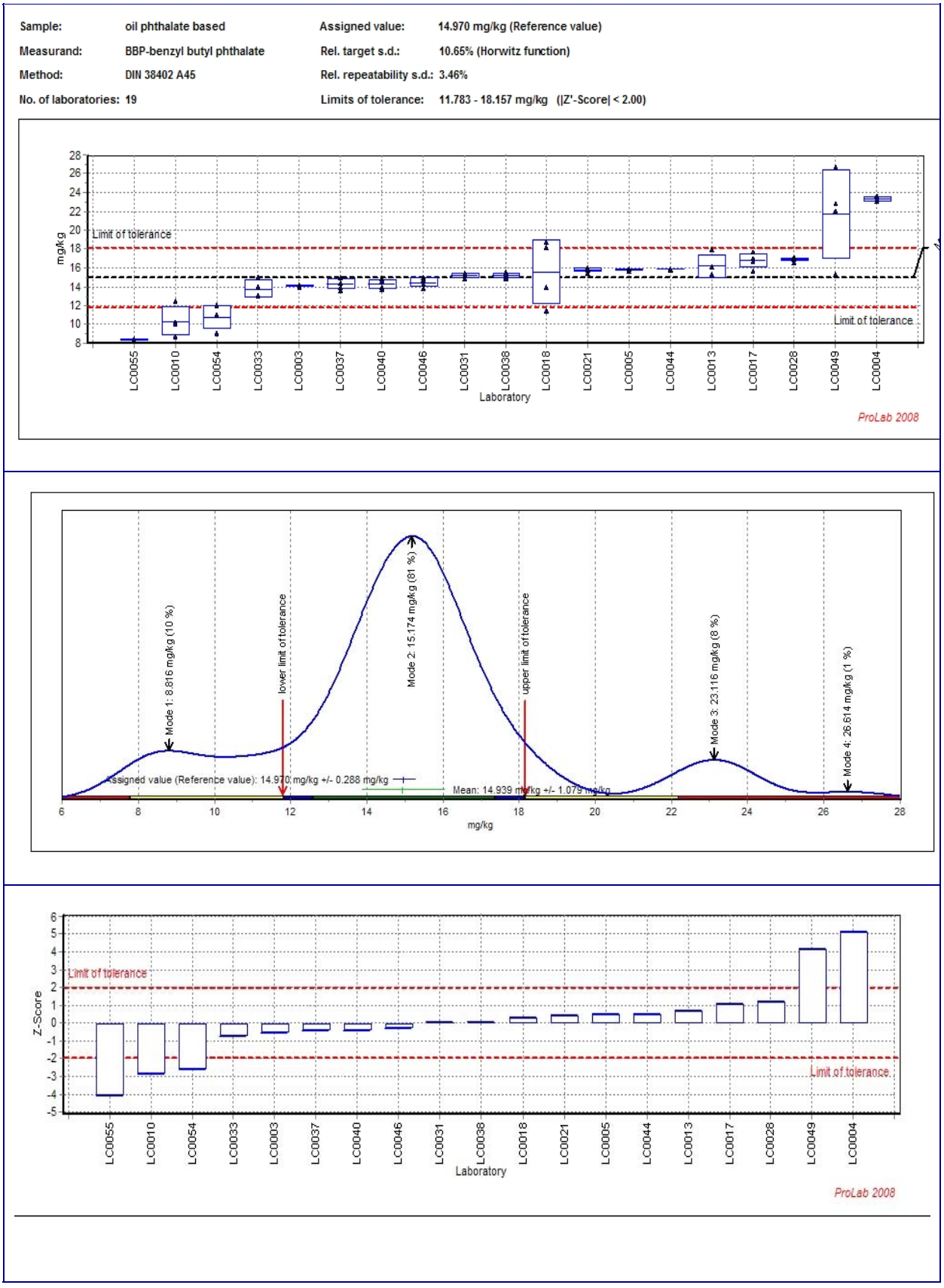


Figure 1. Summary graphs of the laboratory's test results with their repeatability SD (a), Kernel Density plot (b) and z'-scores (c)

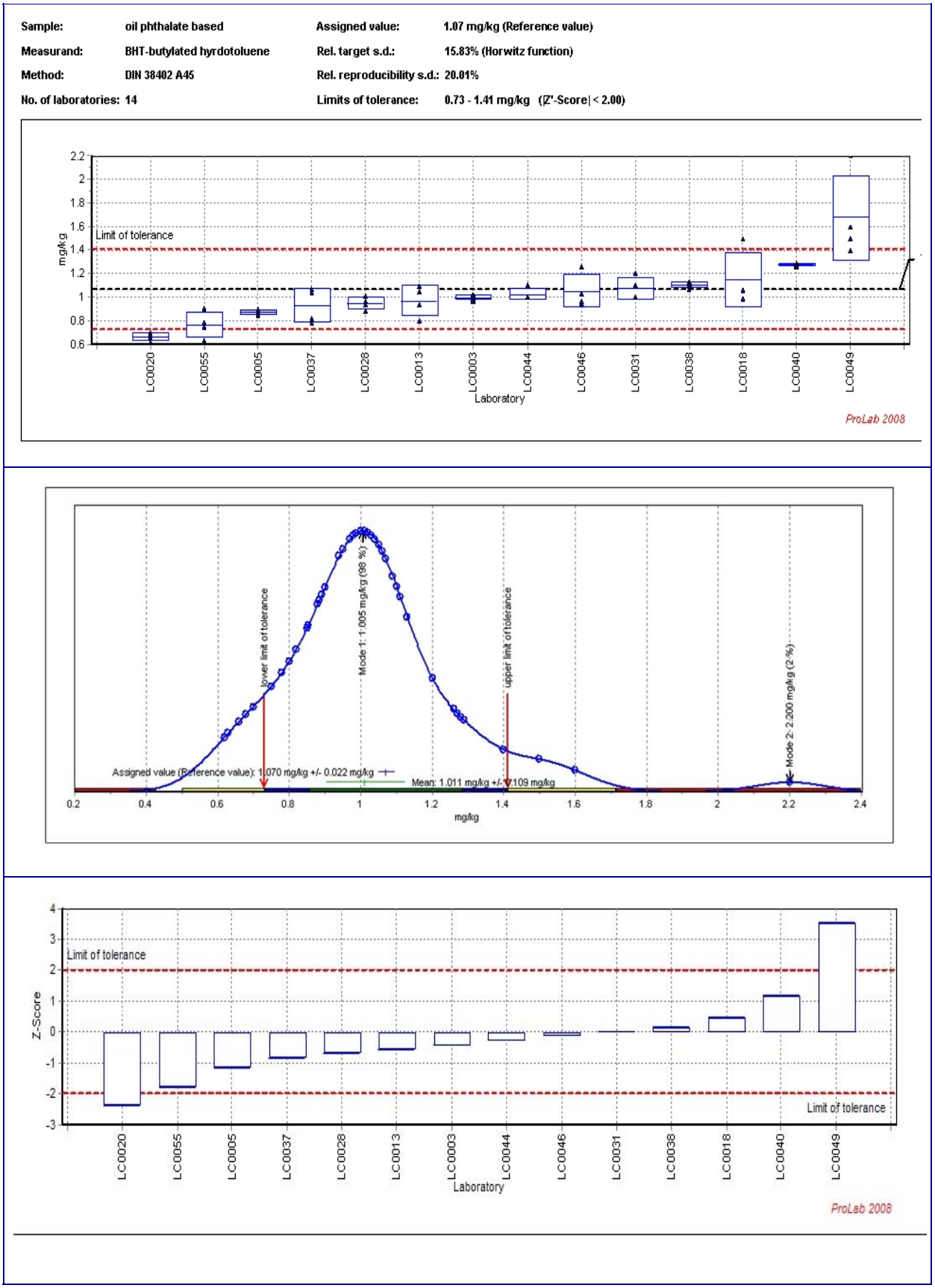


Figure 1. Summary graphs of the laboratory's test results with their repeatability SD (a), Kernel Density plot (b) and z'-scores (c)

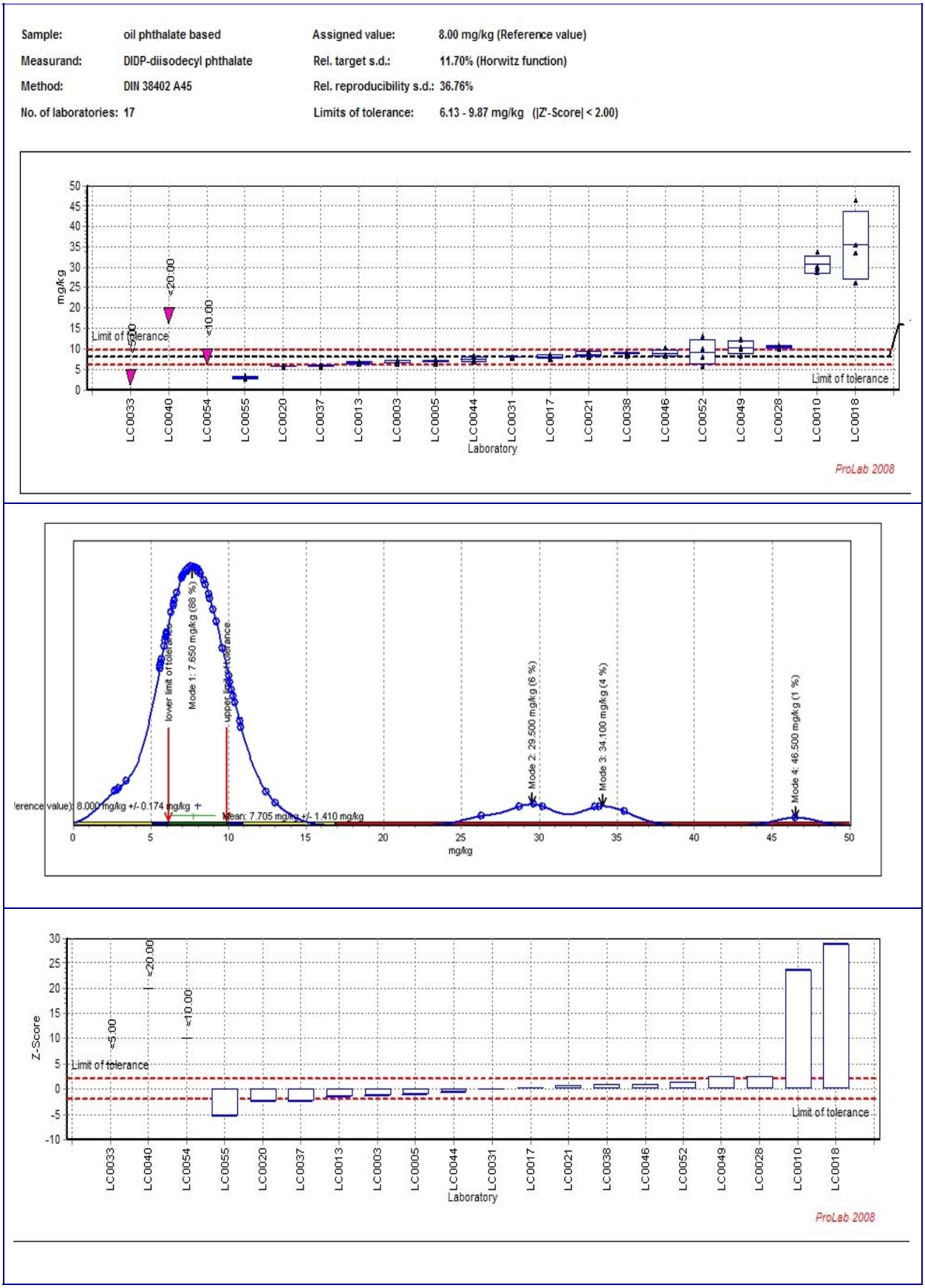


Figure 1. Summary graphs of the laboratory's test results with their repeatability SD (a), Kernel Density plot (b) and z'-scores (c)

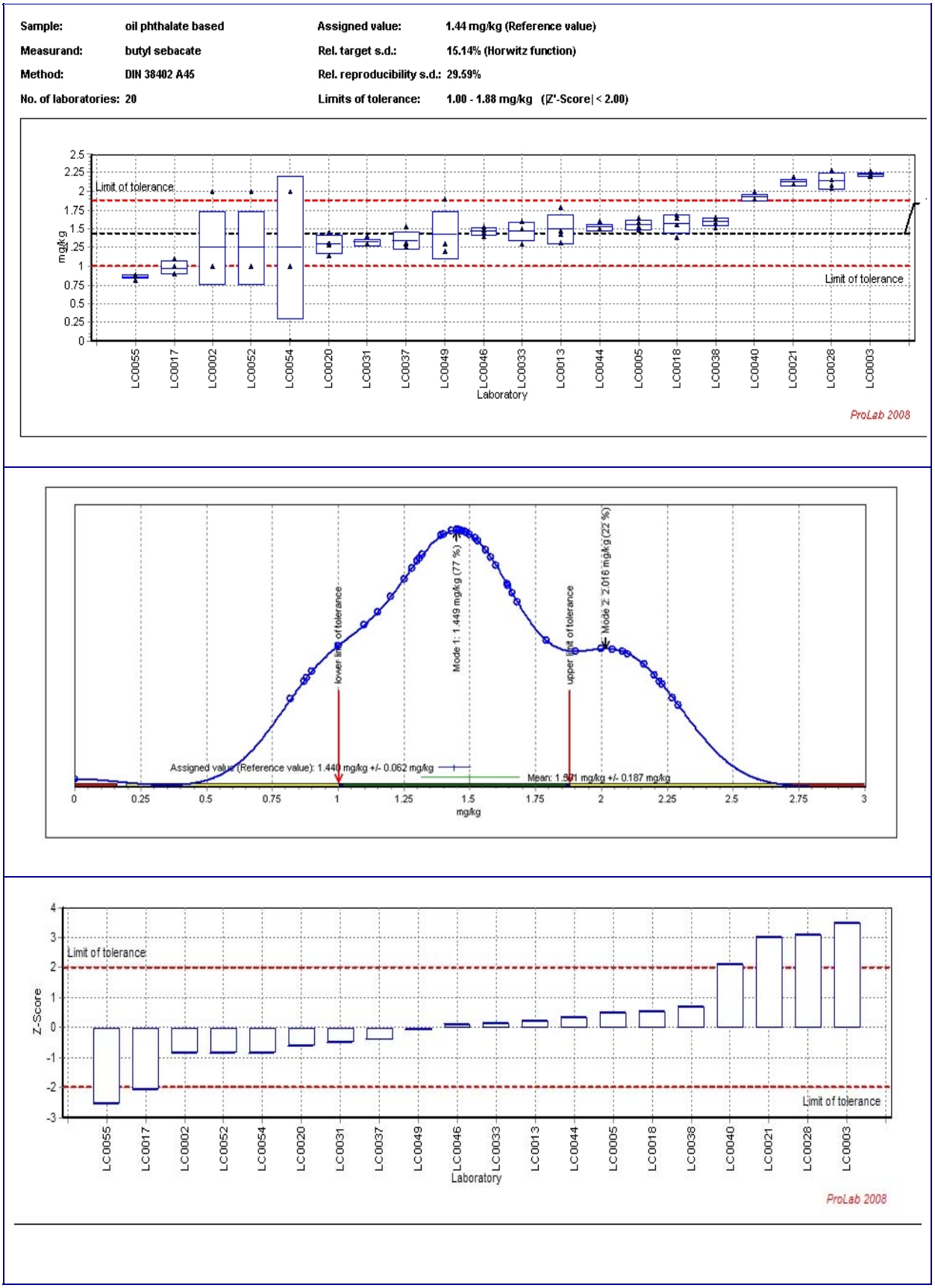
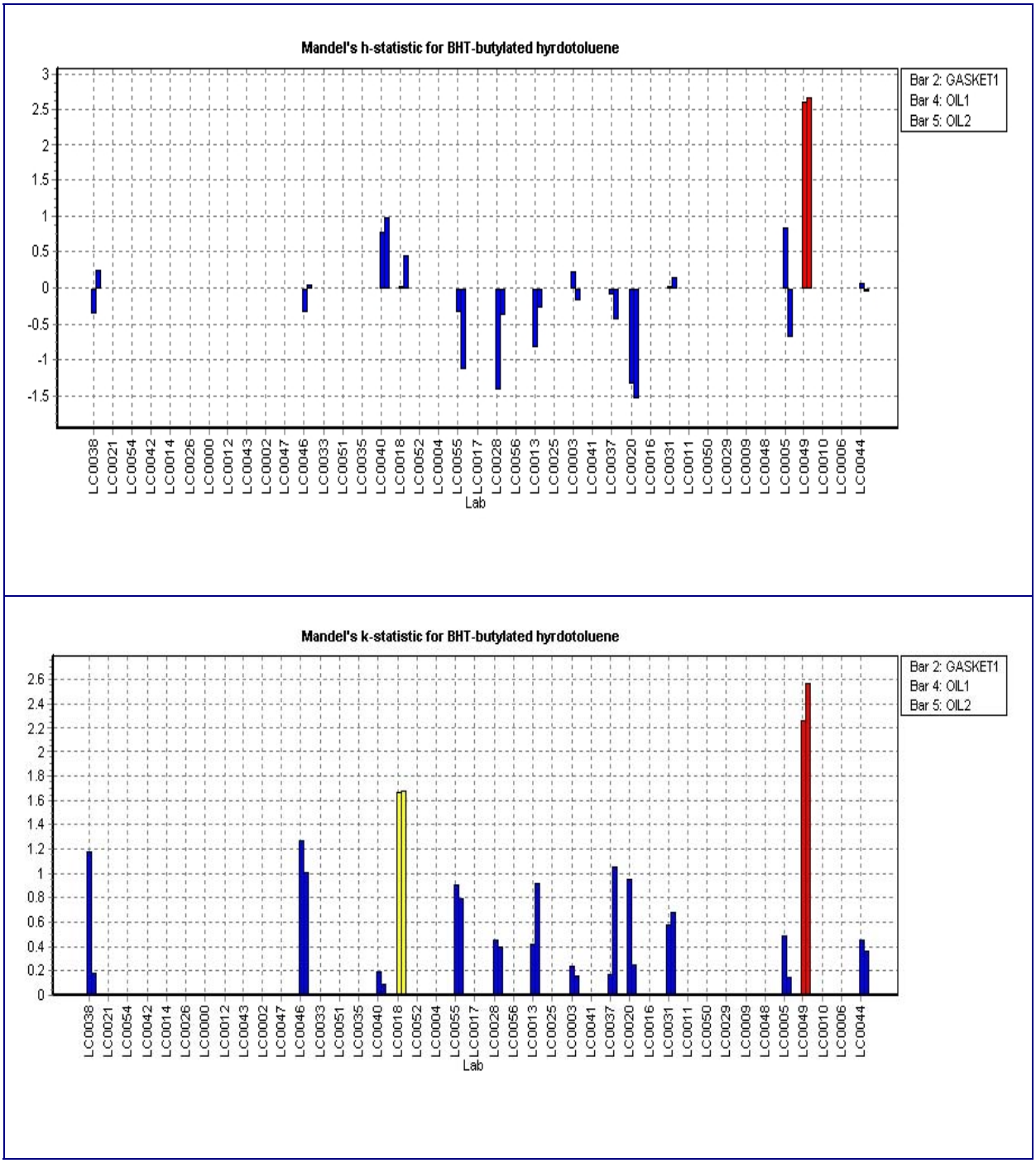
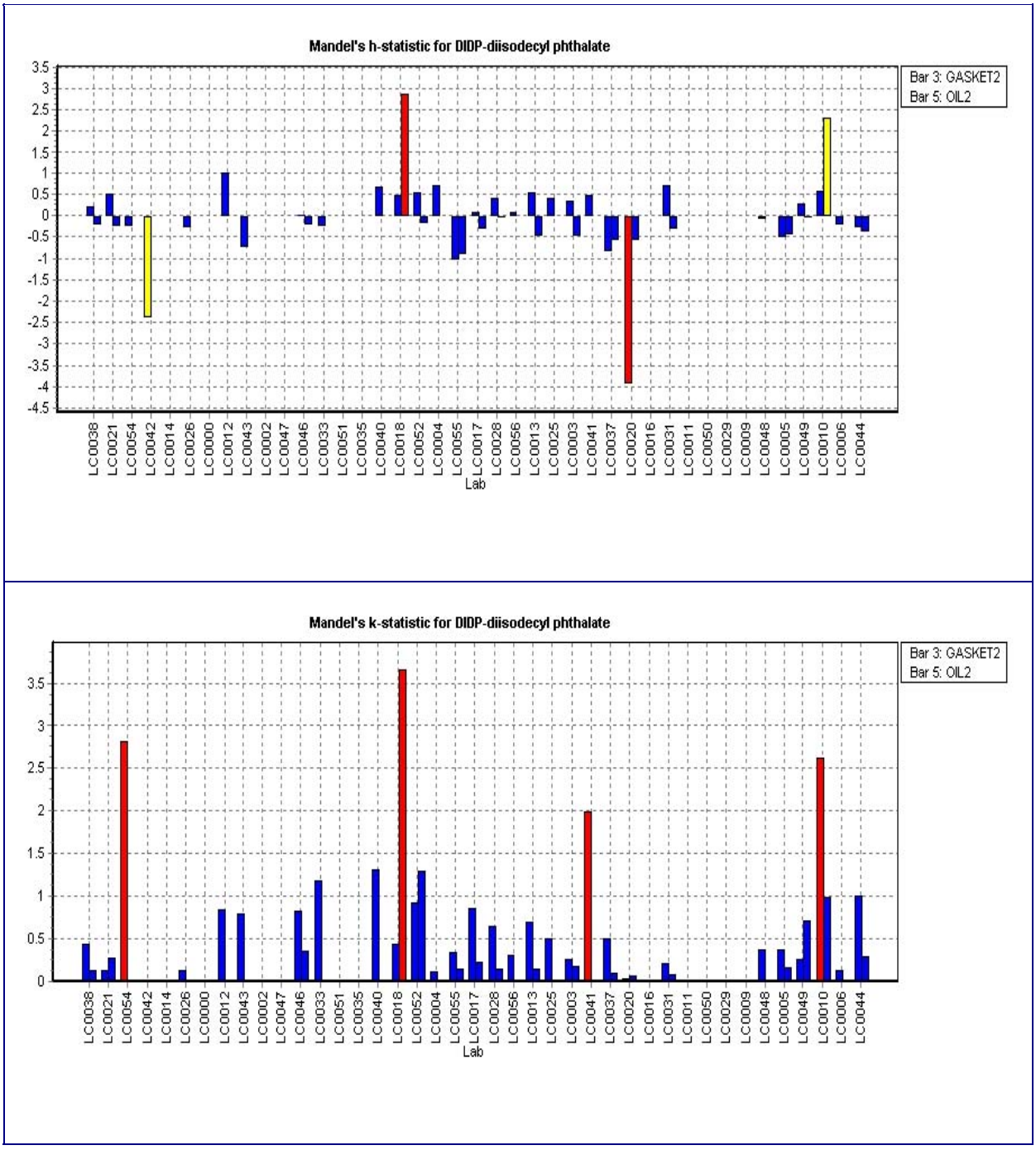


Figure 2. Mandel h- and k-statistics for BHT



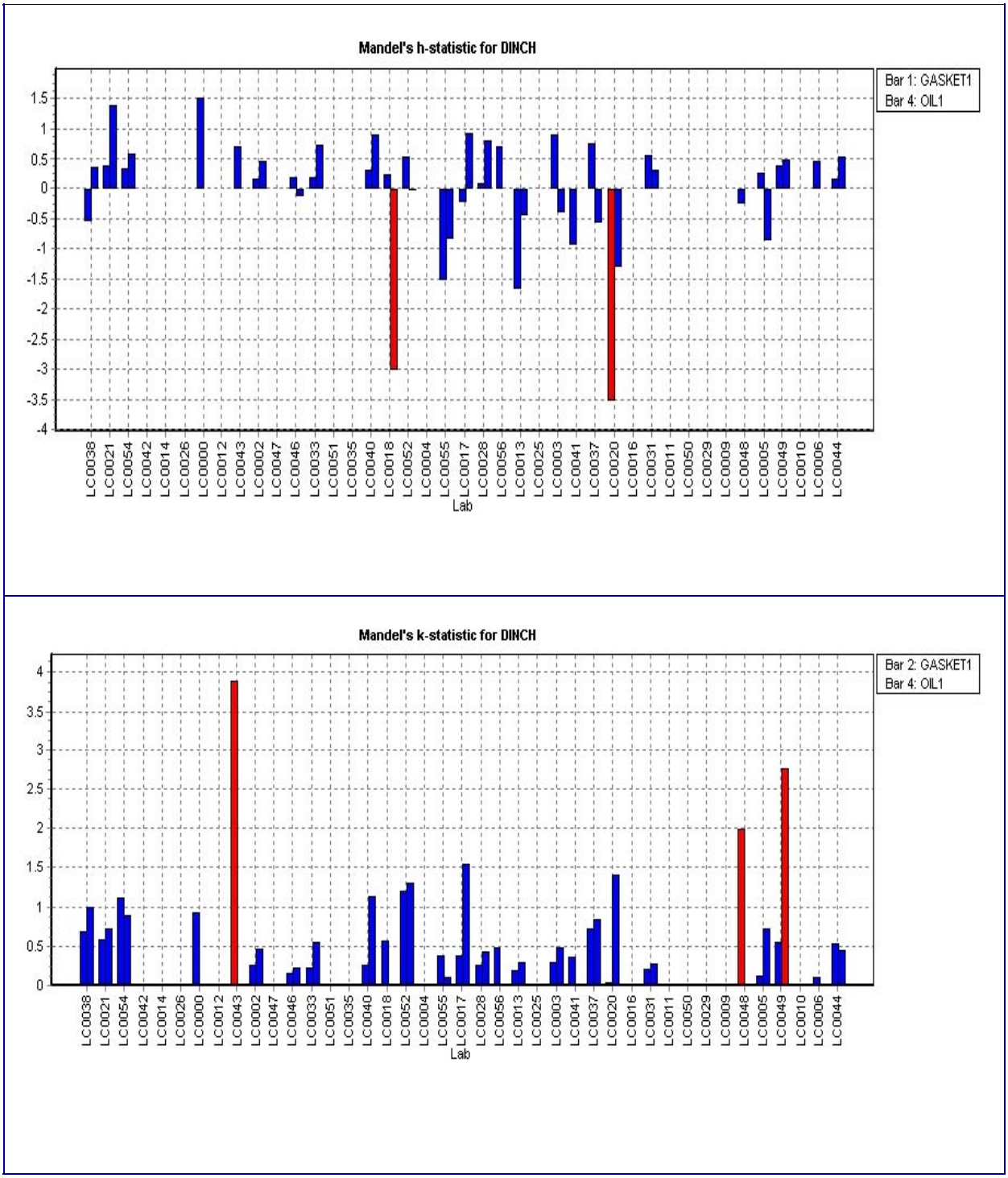
* for 1% significant level the indicative Mandel's h value is 2.43 and k-value (for n=4 replicates) is 1.90 Laboratories with higher values are marked in red
* for 5% significant level the indicative Mandel's h value is 1.90 and k-value (for n=4 replicates) is 1.60 Laboratories with higher values are marked in yellow
** The legend next to the figure explains the sequence of the bars for each laboratory, i.e. the first entry in the legend coincides with the bar at the farthest-left (for one laboratory), while the last legend entry coincides with the bar on the farthest-right (for one laboratory).

Figure 2. Mandel h- and k-statistics for DIDP



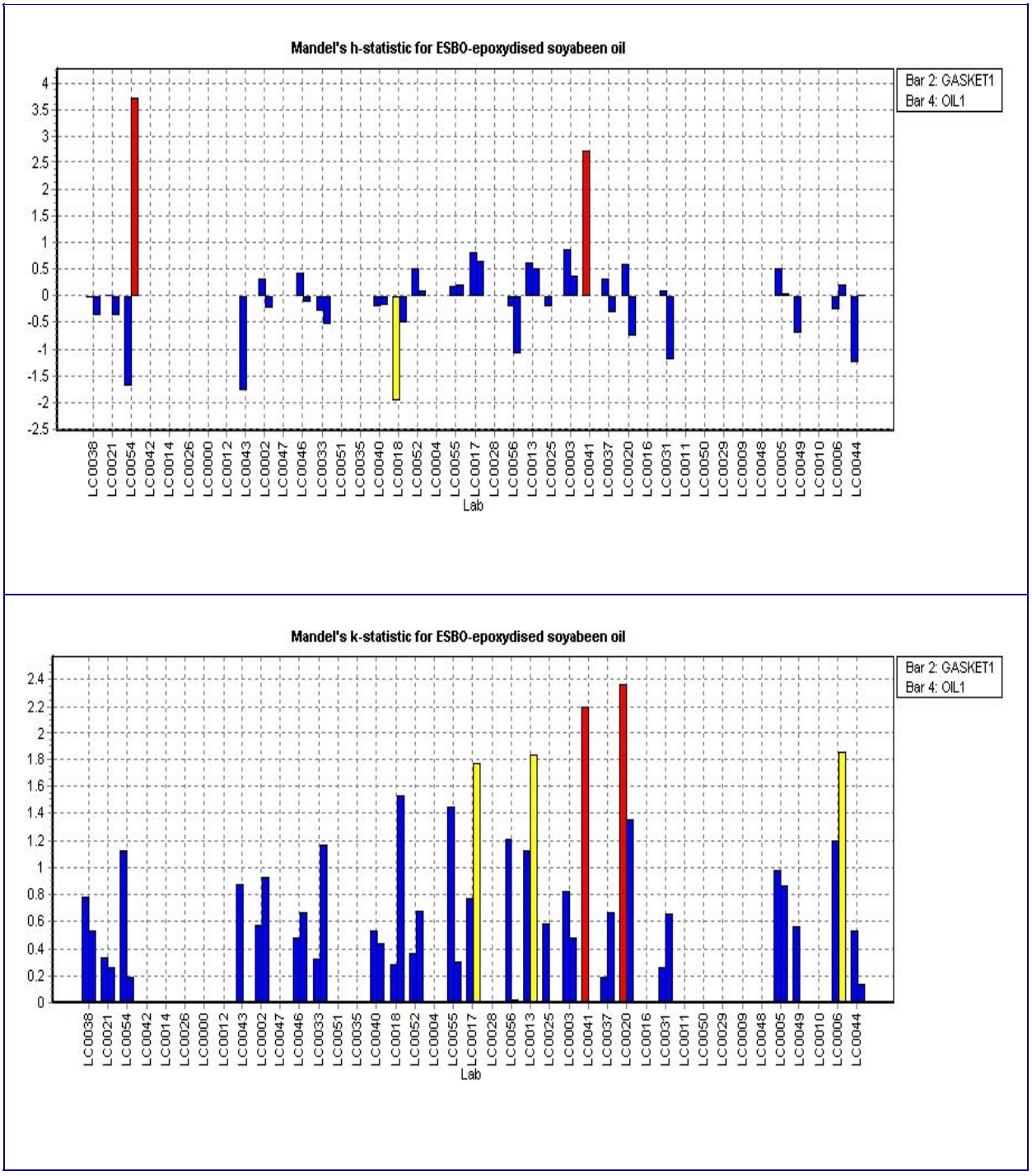
* for 1% significant level the indicative Mandel's h value is 2.43 and k-value (for n=4 replicates) is 1.90 Laboratories with higher values are marked in red
* for 5% significant level the indicative Mandel's h value is 1.90 and k-value (for n=4 replicates) is 1.60 Laboratories with higher values are marked in yellow
** The legend next to the figure explains the sequence of the bars for each laboratory, i.e. the first entry in the legend coincides with the bar at the farthest-left (for one laboratory), while the last legend entry coincides with the bar on the farthest-right (for one laboratory).

Figure 2. Mandel h- and k-statistics for DINCH



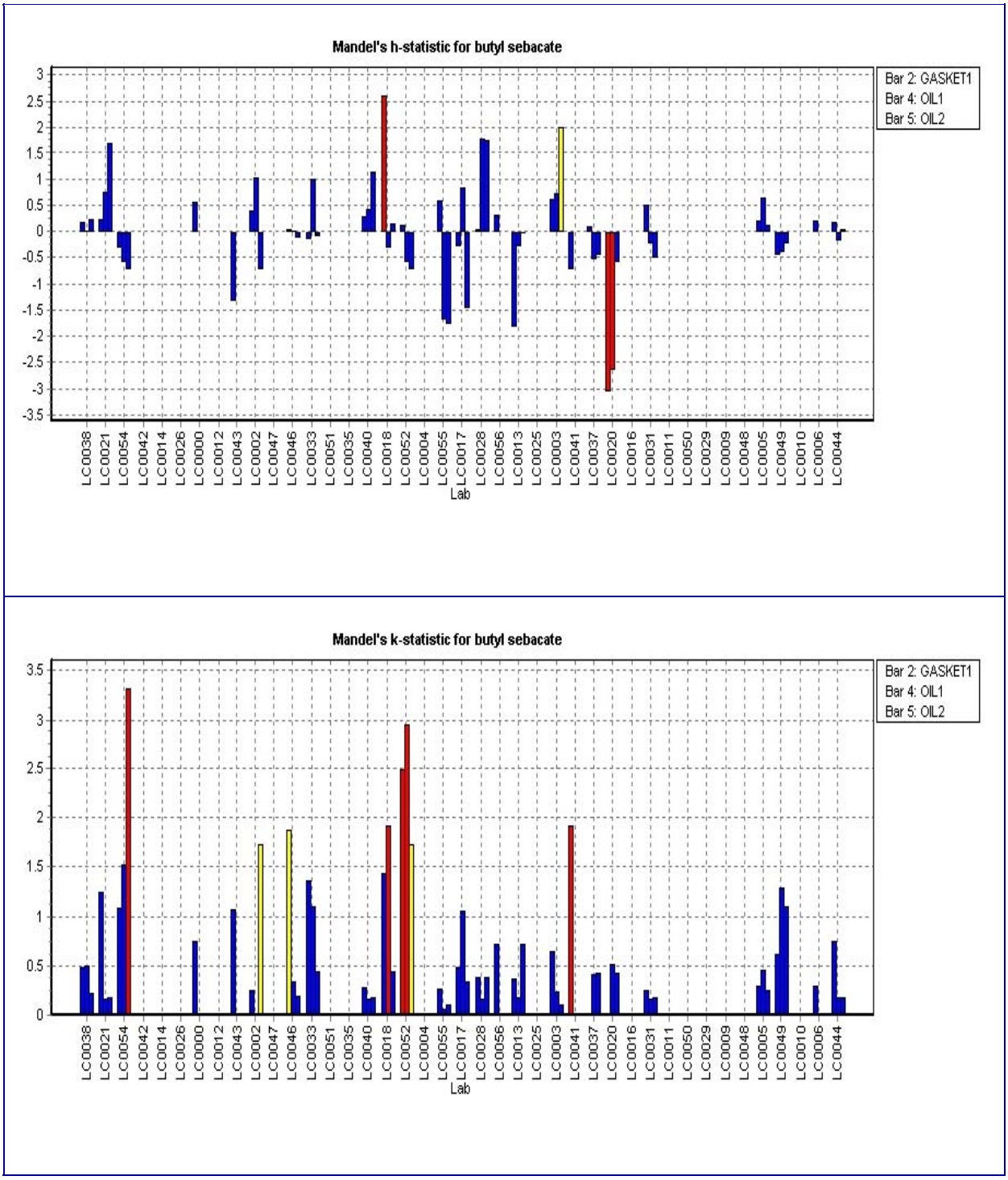
* for 1% significant level the indicative Mandel's h value is 2.43 and k-value (for n=4 replicates) is 1.90 Laboratories with higher values are marked in red
* for 5% significant level the indicative Mandel's h value is 1.90 and k-value (for n=4 replicates) is 1.60 Laboratories with higher values are marked in yellow
** The legend next to the figure explains the sequence of the bars for each laboratory, i.e. the first entry in the legend coincides with the bar at the farthest-left (for one laboratory), while the last legend entry coincides with the bar on the farthest-right (for one laboratory).

Figure 2. Mandel h- and k-statistics for ESBO



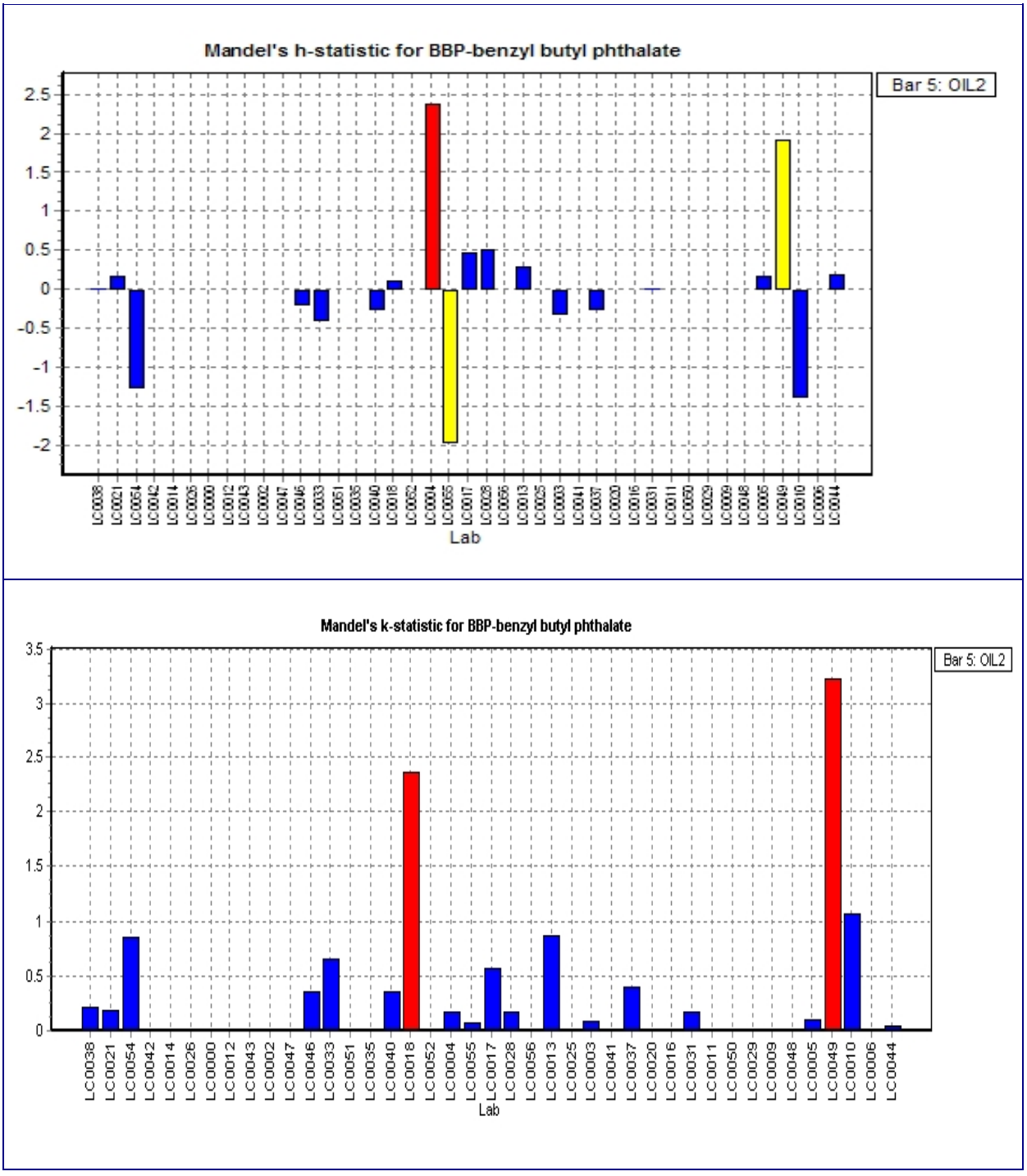
* for 1% significant level the indicative Mandel's h value is 2.43 and k-value (for n=4 replicates) is 1.90 Laboratories with higher values are marked in red
* for 5% significant level the indicative Mandel's h value is 1.90 and k-value (for n=4 replicates) is 1.60 Laboratories with higher values are marked in yellow
** The legend next to the figure explains the sequence of the bars for each laboratory, i.e. the first entry in the legend coincides with the bar at the farthest-left (for one laboratory), while the last legend entry coincides with the bar on the farthest-right (for one laboratory).

Figure 2. Mandel h- and k-statistics for butyl sebacate



* for 1% significant level the indicative Mandel's h value is 2.43 and k-value (for n=4 replicates) is 1.90 Laboratories with higher values are marked in red
* for 5% significant level the indicative Mandel's h value is 1.90 and k-value (for n=4 replicates) is 1.60 Laboratories with higher values are marked in yellow
** The legend next to the figure explains the sequence of the bars for each laboratory, i.e. the first entry in the legend coincides with the bar at the farthest-left (for one laboratory), while the last legend entry coincides with the bar on the farthest-right (for one laboratory).

Figure 2. Mandel h- and k-statistics for BBP



* for 1% significant level the indicative Mandel's h value is 2.43 and k-value (for n=4 replicates) is 1.90. Laboratories with higher values are marked in red
* for 5% significant level the indicative Mandel's h value is 1.90 and k-value (for n=4 replicates) is 1.60. Laboratories with higher values are marked in yellow
** The legend next to the figure explains the sequence of the bars for each laboratory, i.e. the first entry in the legend coincides with the bar at the farthest-left (for one laboratory), while the last legend entry coincides with the bar on the farthest-right (for one laboratory).

Table 5. Summary of z (z')-scores against target SD of 15% for gaskets and Horwitz – for oil

Laboratory	GASKET1/ DINCH	GASKET1/ ESBO	GASKET1/ SEBACATE	GASKET2 /DIDP	OIL1/ BHT	OIL1/ DINCH	OIL1/ ESBO	OIL1/ SEBACATE	OIL2/ BHT	OIL2/ DIDP	OIL2/ SEBACATE	OIL2/ BBP
LC0000	2.04		0.89									
LC0002	-0.09	0.93	0.6			0.87	-0.28	2			-0.84	
LC0003	1.1	2.24	0.99	0.33	-0.2	-1.61	2.25	1.46	-0.43	-1.28	3.48	-0.55
LC0004				0.88								5.17
LC0005	0.07	1.34	0.27	-0.86	0.93	-2.97	0.86	1.29	-1.17	-1.08	0.52	0.51
LC0006	0.37	-0.48	0.26	-0.48			1.5					
LC0010				0.68						23.75		-2.86
LC0012				1.28								
LC0013	-2.97	1.65	-3.46	0.61	-2.18	-1.79	2.76	-0.31	-0.59	-1.47	0.26	0.74
LC0017	-0.68	2.1	-0.65	-0.03		2.17	3.32	1.66		0.18	-2.05	1.11
LC0018	0.04	-4.57	4.61	0.53	-0.58	-9.25	-1.57	-0.36	0.47	28.86	0.56	0.37
LC0020	-5.94		-5.73	-5.86	-3.09	-4.25	-2.63	-4.43	-2.37	-2.37	-0.62	
LC0021	0.27	0.14	0.31	0.57		3.48	-0.84	1.49		0.76	3.02	0.5
LC0025		-0.32		0.45								
LC0026				-0.54								
LC0028	-0.21		-0.05	0.44	-3.24	1.81		3.24	-0.7	2.6	3.1	1.21
LC0031	0.53	0.39	0.8	0.87	-0.56	0.38	-4.39	-0.19	0.03	0.05	-0.51	0.11
LC0033	-0.03	-0.5	-0.36	-0.52		1.64	-1.67	1.94			0.15	-0.75
LC0037	0.86	0.9	0.07	-1.37	-0.79	-2.11	-0.63	-0.75	-0.83	-2.29	-0.4	-0.44
LC0038	-1.21	0.07	0.22	0.17	-1.29	0.52	-0.89	0.2	0.18	0.92	0.71	0.11
LC0040	0.17	-0.3	0.41	0.8	0.82	2.13	-0.07	0.93	1.2		2.14	-0.43
LC0041	-1.8	6.59	-1.43	0.55								
LC0042				-3.63								
LC0043	0.76	-4.08	-2.54	-1.23								
LC0044	-0.09	-2.88	0.22	-0.57	-0.5	1.05	0.66	-0.13	-0.26	-0.58	0.37	0.54
LC0046	-0.05	1.16	-0.04	-0.2	-1.23	-0.88	0.19	0.21	-0.1	1.05	0.13	-0.32
LC0048	-0.74			-0.29								
LC0049	0.25	-1.57	-0.95	0.25	4.13	0.93		-0.47	3.54	2.47	-0.07	4.19
LC0052	0.5	1.36	0.12	0.64		-0.54	1.1	-0.81		1.31	-0.84	
LC0054	0.2	-3.91	-0.7	-0.53		1.18	16.08	-0.81			-0.84	-2.61
LC0055	-2.75	0.57	0.95	-1.64	-1.25	-2.91	1.52	-2.79	-1.79	-5.29	-2.55	-4.1
LC0056	0.76	-0.33	0.47	-0.04			-3.97					

Table 5 (continue). Summary of z (z')-scores against target SD of 15% for gaskets and Horwitz – for oil

Number of laboratories	*GASKET1/ DINCH*	*GASKET1/ ESBO*	*GASKET1/ SEBACATE*	*GASKET2 /DIDP*	*OIL1/ BHT*	*OIL1/ DINCH*	*OIL1/ ESBO*	*OIL1/ SEBACATE*	*OIL2/ BHT*	*OIL2/ DIDP*	*OIL2/ SEBACATE*	*OIL2/ BBP*
with z>2	3	2	1		1	5	3	1	1	4	3	2
with z>3	1	4	3	2	3	3	4	2	1	3	3	3
total N	26	23	26	30	14	20	20	20	14	17	20	19
% successful	**85**	**74**	**85**	**93**	**71**	**60**	**65**	**85**	**86**	**59**	**70**	**70**

Figure 3 Summary of z (z') – scores

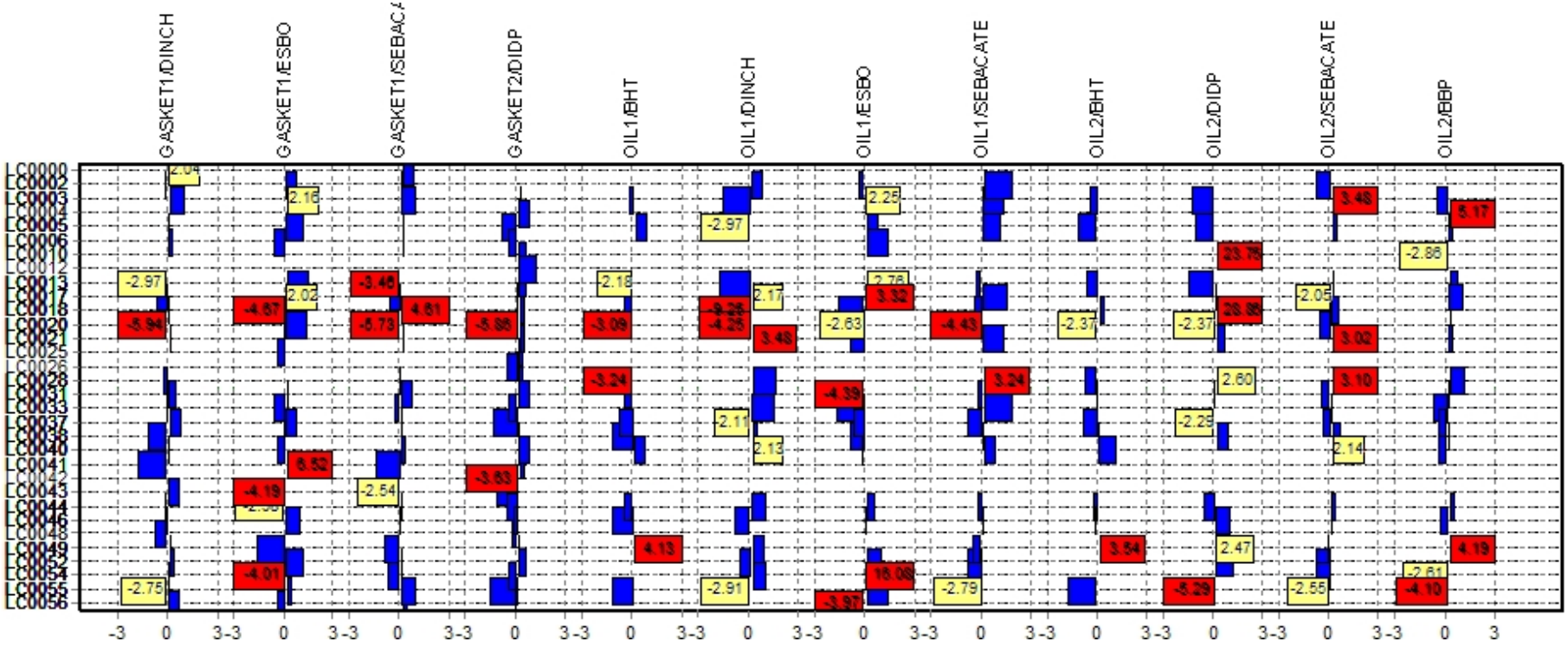


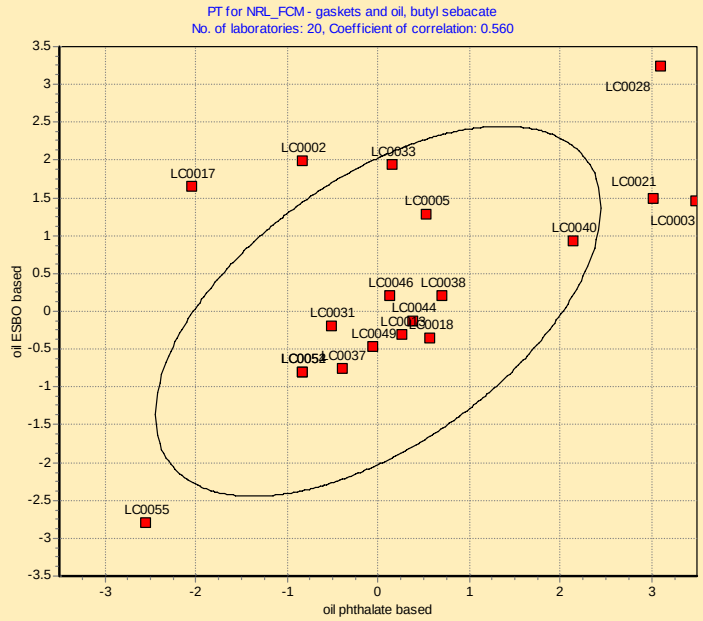
Table 7. Summary of the number of laboratories outliers according to the Mandel tests

Evaluation of the consistency of the laboratory data amongst them - Mandel statistics

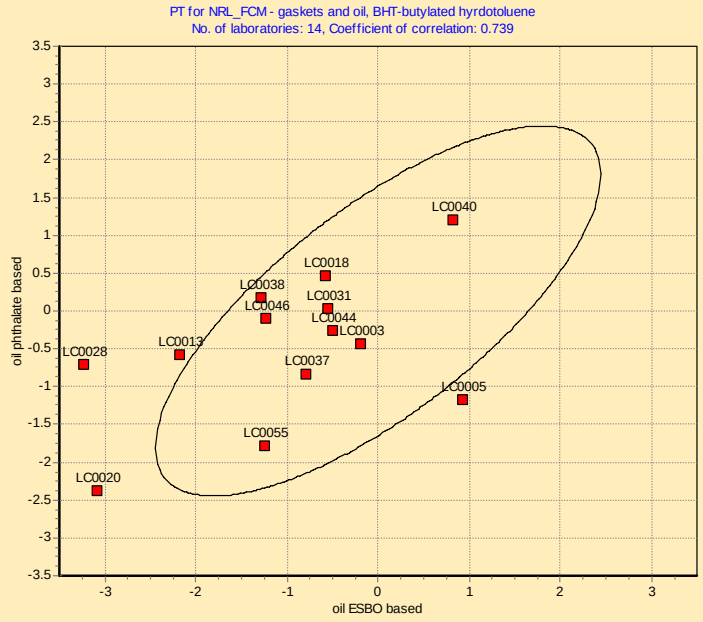
Number of laboratories with Mandel h statistics non consistent with 5% and 1% significance level												
	DINCH		ESBO		DIDP		BHT		SEBACATE		BBP	
	5%	1%	5%	1%	5%	1%	5%	1%	5%	1%	5%	1%
G1		1	1	1						2		
G2					1	1						
O1		1		1				1		1		
O2					1	1		1		1	1	1
Mandel k- statistics non consistent with 5% and 1% significance level												
	DINCH		ESBO		DIDP		BHT		SEBACATE		BBP	
	5%	1%	5%	1%	5%	1%	5%	1%	5%	1%	5%	1%
G1		2		2					1	2		
G2						2						
O1		1	3				1	1		2		
O2						2	1	1	2	1		2

Fig 5. Youden plot

butyl sebacate in oil 1 and oil 2



BHT in oil 1 and oil 2



Results in the upper left or lower right hand corner of the diagram are dominated by random error. Results close to the 45° line shown in the plot, but far away from the assigned value have results dominated by systematic error

Figure 6. Distribution of all z-score - scatter

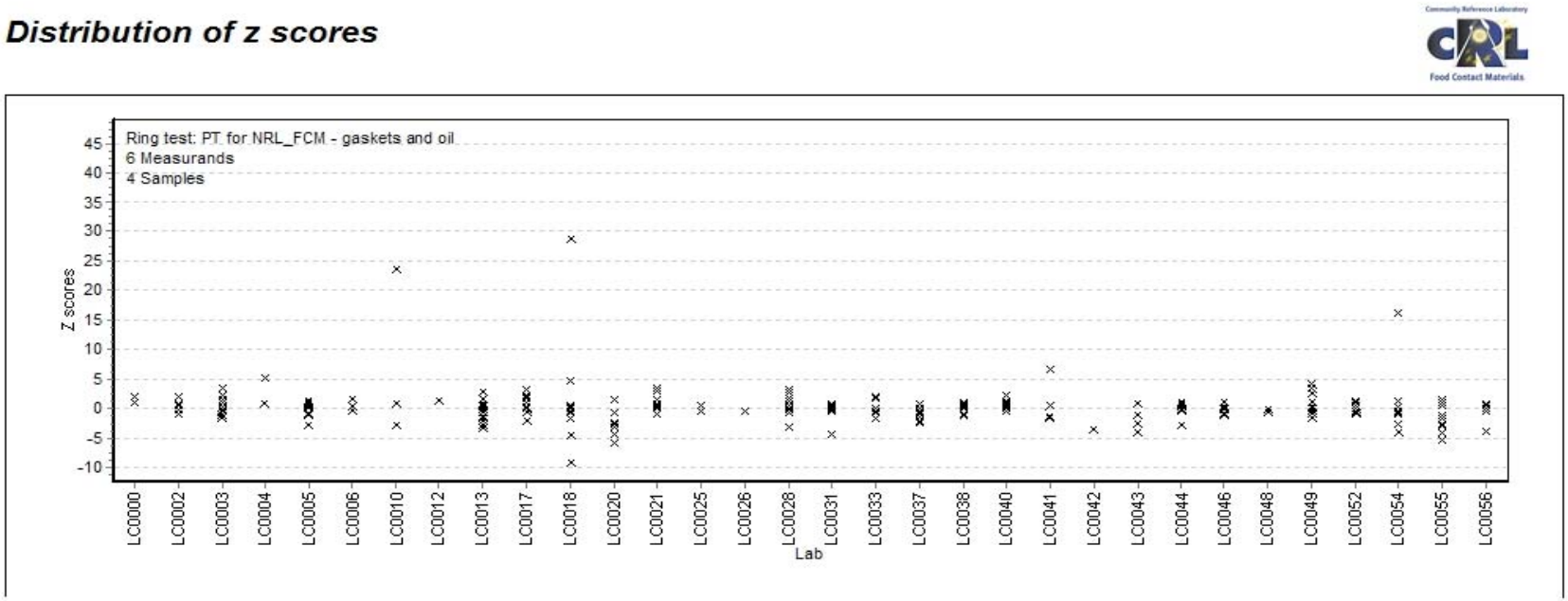
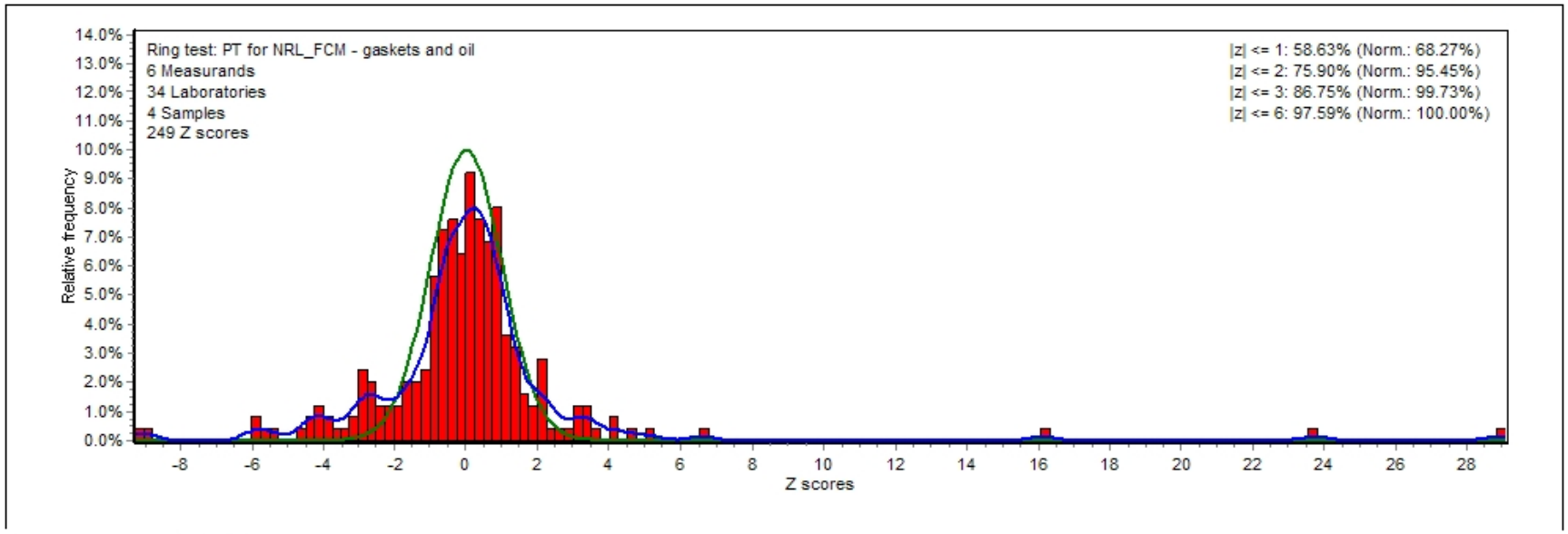


Figure 7. Distribution of all z score histogram (red bars), Kernel density plot (blue line) and normal distribution plot (green line)



11. Acknowledgements

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

AUSTRIA	Austrian Agency for Health and Food Safety (AGES),
BELGIUM	Institute of Public Health, ISSP-LP
REPUBLIC OF CYPRUS	Laboratory for Control of Food Contact Materials and Control of Toys Ministry of Health, State General Laboratory (SGL)
CZECH REPUBLIC	NIPH- NRL for Food Contact Materials and for Articles for children under 3 years old, National Institute of Public Health (SZU')
DENMARK	Department of Food Chemistry, National Food Institute Technical University of Denmark
ESTONIA	Health Protection Inspectorate - Central Laboratory of Chemistry
FINLAND	Finnish Customs Laboratory
FRANCE	Center for Energy Material and Packaging - Laboratoire National d'Essais
SCL	Laboratoire de Bordeaux-Pessac
GERMANY	Bundesinstitut für Risikobewertung (BFR) (Federal Institute for Risk Assessment)
GREECE	General Chemical State Laboratory, D' Chemical Service of Athens, Section, Laboratory of Articles and Materials in Contact with Foodstuffs
HUNGARY	National Institute of Food Hygiene and Nutrition – Dept of Food additives and contaminants, Section Food Additives and Contact Materials
IRELAND	Public Analyst Laboratory - Sir Patrick Duns Hospital
POLAND	Laboratory of Department of Food and Consumer Articles Research , National Institute of Hygiene,
PORTUGAL	ESB-SE (Portuguese Catholic University - Biotechnology College – Packaging Department)
SLOVENIA	National Institute of Public Health of Republic of Slovenia , Dept of Sanitary Chemistry,
SLOVAK REPUBLIC	National Reference Centre and Laboratory for material and articles intended to come into contact with food, Regional Public Health Authority
SPAIN	Centro Nacional de Alimentación, Agencia Espanola de Seguridad Alimentaria y Nutrición
SWEDEN	National Food Administration, Food Standards Division
THE NETHERLANDS	Food and Consumer Product Safety Authority (VWA), Inspectorate for Health Protection region North
UNITED KINGDOM	Central Science Laboratory
ITALY	Neotron.S.p.a.
ITALY	SSICA
THAILAND	Food Packaging Laboratory Biological science program , Bangkok, THAILAND
Germany	Landesuntersuchungsanstalt fr das Gesundheits- und Veterin rwesen Sachsen FG 6.3 - Bedarfsgegenstände
Germany	Bayerisches Landesamt für Gesundheit und Lebensmittelsicherheit Bavarian Health and Food Safety Authority
Germany	Hessisches Landeslabor LHL Standort Wiesbaden
Germany	Landesamt für Landwirtschaft, Lebensmittelsicherheit und Fischerei Mecklenburg Vorpommern Dezernat kosmetische Mittel und Bedarfsgegenstände
Germany	Landesuntersuchungsamt Institut für Lebensmittelchemie Koblenz
Germany	Chemisches und Veterinäruntersuchungsamt Rhein-Ruhr-Wupper (CVUA-RRW)

12 References

[1] ISO 13528:2005; Statistical Methods for Use in Proficiency Testing by Interlaboratory Comparisons

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

[3] (EC) No 372/2007 Commission Regulation of 2 April 2007 laying down transitional migration limits for plasticisers in gaskets in lids intended to come into contact with foods.

[4] 2002/72/EC Commission Directive of 6 August 2002 relating to plastic materials and articles intended to come into contact with foodstuffs. (Plastics: Unofficial consolidated version including 2002/72/EC, 2004/1/EC, 2004/19/EC, 2005/79/EC, 2007/19/EC, 2008/39/EC)

[5] ProLab Software – QuoData, Drezden – www.quodata.de

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

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

[8] ISO/TS 20612 Water quality – Interlaboratory comparison for proficiency testing of analytical chemistry laboratories

[9] T. Linsinger *et al.*, *Accreditation and Quality Assurance in Analytical Chemistry* (2001), 6, 20-25

[10] The International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories by M. Thompson *et al.*, *Pure and Applied Chemistry* (2006), 78, 145–196

[11] ISO 5725-2:1994 (E) Accuracy of measurement methods and results – Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method

[12] AMC, *Representing data distributions with kernel density estimates*. AMC Technical Brief, 2006, http://www.rsc.org/images/brief4_tcm18-25925.pdf.

[13] Lowthian, P.J. and M. Thompson, *Bump-Hunting for the proficiency tester - searching for multimodality.* The Analyst, 2002. 127: p. 1359, https://www.swetswise.com/eAccess/viewAbstract.do?articleID=14625081.

13. Annexes

Annex 1 Questionnaire sent in March 2008 to NRLs on the state of use of the methods for analysis of plasticisers' in gasket/oil/food samples

Annex 2 Summary of the answers from the questionnaire sent to NRLs before the dispatch of samples on the state of use of the methods for analysis of plasticisers' in gasket/oil/food samples

Annex 3a: Invitation letter to laboratories PT001/A

Annex 3b: Invitation letter to laboratories PT001/B

Annex 4: Letter accompanying the sample PT001/A

Annex 5a: Instruction for the compilation of the results

Annex 5b: Instruction for the compilation of the results in electronic format

Annex 6a: Letters of confirmation of participation PT001/A

Annex 6b: Letters of confirmation of participation PT001/B

Annex 7a: Letters of confirmation of receipt PT001/A

Annex 7b: Letters of confirmation of receipt PT001/B

Annex 8 Summary of laboratories participation in interlaboratory comparison exercises

Annex 9 Procedure for the preparation of the spike of plasticisers in sunflower oil for PT test

Annex 10: Results of the homogeneity study

Annex 1
Questionnaire sent in March 2008 to NRLs on the state of use of the methods for analysis of plasticisers' in gasket / oil / food samples

Dear Madame/Sir

Being CRL-Food Contact Material, our duty is to provide NRLs with the possibility to participate in interlaboratory comparative exercise. In order to organize the forthcoming PT for 2008 in the best way CRL need to gather information about the competence of NRLs in performing analysis for plasticizers as ESBO and phthalates in gaskets/plastics and oily food simulants.

Therefore we kindly ask you to fill in this spreadsheet and to send it by ……. latest to:

catherine.simoneau@jrc.it

General information

Laboratory/ Company detail ……..
Contact person ……..
Telephone: ……..
E-mail: ……..

Do you have experience in determination of ESBO in

- food (what kind) Y/N
- oil Y/N
- gaskets Y/N

Do you use the method routinely? How often you analyze ESBO in any of the above mentioned matrix sample/year?

- food (what kind) n/year
- oil n/year
- gaskets n/year

What type of analytical procedure you use (Castle, Grob, modified)?

- extraction: ……
- derivatisation: ……
- calibration (source of reference substance, working range)
- determination:

Is the method that you use validated by the laboratory? Y/N

Do you perform any quality control and quality assurance scheme for that method? Y/N

Do you have experience in determination of phthalates in

- food (what kind) Y/N
- oil Y/N
- gaskets Y/N

Which phthalates do you determine?

BBP	Butyl-benzyl phthalate	DIBP	Di-isobutylphthalate
DBP	Di-Butylphthalate	DIDP	Di-isodecylphthalate
DEHP	Bis(2-ethylhexyl)phthalate	DINP	Di-isononylphthalate
DEP	Di-ethylphthalate	DMP	Di-methylphthalate
DHP	Di-hexylphthalate	DNOP	Di-n-octylphthalate

Do you use the method routinely? How often you analyze phthalates in any of the above mentioned matrix sample/year?

- food (what kind) n/year
- oil n/year
- gaskets n/year

What type of analytical procedure you use?

- extraction: ……..
- clean up: ……
- calibration (working range and IS used) ……
- determination: GS/MS
  LC/MS

Is the method that you use validated by the laboratory? Y/N

Do you perform any quality control and quality assurance scheme for that method?
……
……

Have you participated up to know in a PT exercise for phthalates

- oil (FAPAS 2007 and 2008) Y/N
- PVC material :

  IIS (www.iisnl.com) Y/N

  Dutch PT scheme Y/N

Annex 2
Summary of the answers from the questionnaire sent to NRLs before the dispatch of samples on the state of use of the methods for analysis of plasticisers' in gasket / oil / food samples

QUESTIONNAIRE ESBO PHTHALATES ANALYSIS															
	ESBO							PHTHALATES							
	FOOD	OIL	GASKETS	FREQ	METHOD	VALIDATED	QA	FOOD	OIL	GASKETS	FREQ USE	METHOD	VALIDATED	QA	PT
LC0040	N	N	N	0	N	N	N	Y	Y	Y	>100 2007	GC-MS	Y	Y	N
LC0018	Y	N	N	100	GROB	N		Y	N	N	80	GC-MS	N	N	N
LC0004	N	N	N	-	-	-	-	N	N	N	-	-	-	-	N
LC0055															
LC0017	N	N	Y	150/year	GROB	N	N	N	N	Y	150/year	GS/MS and LC-MS/MS	N	N	FAPAS 2007, 2008
LC0028	N	N	N	-	-	-	-	Y	Y	Y	-	GS/MS	Y	-	FAPAS, Dutch
LC0056	N	N	N	-	-	-	-	N	N	N	-		-	-	N
LC0013	N	Y	N	SET UP	CASTLE	-	-	NN	Y	N	SET UP	LC-MS	-	-	FAPAS 2008
LC0025	N	N	N	-	-	-	-	N	N	N	-	-	-	-	N
LC0003	N	N	N	-	-	-	-	N	N	N	-	-	-	-	N
LC0041	N	N	N	-	-	-	-	Y	Y	Y	FEW	GC-MS	N	N	FAPAS 2008
LC0037															
LC0020	N	N	N	-	-	-	-	N	N	Y	5-6/YEAR	GC-MS	in progess		N
LC0031	Y	Y	N	50-100/year	CASTLE	-	-	Y	Y	Y	SET UP	GC-MS	N	N	FAPAS, Dutch, Check
LC0016															
LC0029															
LC0050															
LC0011															
LC0009															
LC0048															
LC0049															
LC0005	N	N	N	-	-	-	-	N	N	N	PAPER - 110/year	GC-MS	N	-	IIS
LC0006															
LC0010	N	N	N	-	-	-	-	N	N	Y	45/year	GC-MS	Y	Y	Dutch
LC0043															
LC0044	Y	N	N	50/year	Suman	Y	Y	Y	Y	Y	20/year	GC-MS	Y	Y	FAPAS
LC0052	Y	Y	Y	1000	GROB	Y	Y	Y	Y	Y	1000	GC-MS	Y	Y	N

Annex 3a: Invitation letter to laboratories PT001/A

Ispra June 10, 2008
I05-PCE/CS/sm(2008)D/14582

Dear Madam, Sir

Comparative trial 2008 PT001-A (part A) for CRL FOOD CONTACT MATERIALS
Analysis of plasticisers in gaskets

On behalf of the CRL on food contact materials, I would like to invite you to participate in a comparative test/proficiency test exercise for the determination of plasticisers in gaskets which is due to take place in the next months.

I would like to remind you that it is a duty for you as an NRL-FCM to participate in the PTs organised by the CRL-FCM since the work programme is decided on a network consensus basis. For this reason we encourage all of you to actively participate in this exercise. There is no charge for participation. Feel free to involve your local controls.

We have pre-registered everyone, which means we will send test kits to all of you. We however need to receive the **proformat of your participation** for our own administrative purposes. Kindly send back the proformat **by June 13** to: Catherine Simoneau (catherine.simoneau@jrc.it)**.** If you need more test kits to involve more labs at the national level we have another 20 kits test materials for ESBO gaskets and about 10 for phthalate based gaskets. In this case please let me know immediately by e-mail so we can pack accordingly.

The samples will be sent to you this coming week. You will find additional information in the kit sent and on the form "shipping- gaskets". You will also receive more detailed instructions for the compilation of the results. The deadline for submission of results is **12 September 2008**

If you have any question, please contact Catherine Simoneau (catherine.simoneau@jrc.it), ph. +39.0332.785889

Sincerely yours,

Catherine Simoneau

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

Annex 3b: Invitation letter to laboratories PT001/B

Ispra July 08, 2008
I05-PCE/CS/sm(2008)D/17207

Dear Madam, Sir

Comparative trial 2008 PT001-B (part B) for CRL FOOD CONTACT MATERIALS
Analysis of plasticisers in oil

On behalf of the CRL on food contact materials, I would like to invite you to participate in a comparative test/proficiency test exercise for the determination of plasticisers in oil which is due to take place in the next months.

I would like to remind you that it is a duty for you as an NRL-FCM to participate in the PTs organised by the CRL-FCM since the work programme is decided on a network consensus basis. For this reason we encourage all of you to actively participate in this exercise. There is no charge for participation. Feel free to involve your local controls.

The samples will be sent to you this coming week. You will find additional information in the kit sent". You will also receive more detailed instructions for the compilation of the results. The deadline for submission of results is **12 September 2008**

If you have any question, please contact Catherine Simoneau (catherine.simoneau@jrc.it), ph. +39.0332.785889

Sincerely yours,

Catherine Simoneau

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

Annex 4: Letter accompanying the sample PT001/A

Ispra June 10, 2008
Annex to I05-PCE/CS/sm(2008)D/14582

Shipping kit for comparative/proficiency testing CRL-FCM PT001- gaskets
12 June: Shipping of standards + Shipping of gaskets

Standard substances kit:

substance type	brand	trade name	abbrev	name	CAS No	volume (mL)
Phthalates	BASF	PALATINOL C	DBP	dibutyl phthalate	84-74-2	10
Phthalates	BASF	PALATINOL IC	DiBP	Diisobutyl phthalate	84-69-5	10
Phthalates	Ferro	SANTICIZER 160	BBP	benzylbutyl phthalate (2001)*	85-68-7	10
Phthalates	BASF	PALATINOL N	DINP	diisononyl phthalate	28553-12-0	10
Phthalates	ExxonMobil	JAYFLEX DINP	DINP	diisononyl phthalate	68515-48-0	10
Phthalates	ExxonMobil	JAYFLEX DIDP	DIDP	diisodecyl phthalate	68515-49-1	10
ESBO	Akcros	Lankroflex E2307	ESBO	epoxidised soybean oi;	008013-07-8	10
ELO	Akcros	Lankroflex L	ELO	epoxidised linseed oil	008016-11-3	10
DINCH	BASF	HEXAMOLL	DINCH	1,2-Cyclohexanedicarboxylic acid diisononyl ester	66412-78-8	10
Sebacate	Sigma	-	DBS	Dibutyl-Sebacate	109-43-3	1
AMG	Danisco	GRINDSTED® SOFT-N-SAFE	AMG	acetylated monoglyceride	736150-63-3	1

Notes: BASF could no longer provide DEHP or DIDP as they do not produce these substances any longer in Europe.
Note 2: The specification for residual DINP content for DINCH is currently at max. 100 ppm i.e. = 0,01 %
* BBP was no longer available, so we provide from the stock we had from our work on toys in 2001.

Note: you may not need all the substances, and you will need a standard of BHT available commercially.

Gaskets:

- 1 gasket (n=1 specimen) phthalate based – expect to find also present to quantify. As the lot was not homogeneous, we had to pre-analyse a portion of the lot to generate a sub-lot of homogenous specimens, which is why each gasket has an aliquot missing.
- 1 gasket (n=2 specimens) ESBO based; expect to find 2 other relevant monomeric plasticisers as well as BHT present. Quantify all of them.

Method of analysis:
free choice

Results requested:
Perform 4 replicates
Result format: Use the forms that will be provided with some instructions

Next shipment: early July : part B
2 oils:
One set with a spiked oil with phthalates and other plasticisers
One set with a spiked oil with ESBO and some additional plasticisers.

Annex 5a: Instruction for the compilation of the results

Ispra June 23, 2008
I05-PCE/CS/sm(2008)D/17212

Instructions for the compilation of the results for comparative/proficiency testing CRL-FCM PT001-A (part A) and PT001-B (part B)

Data generated by the laboratories for the comparative test CRL-FCM PT001 will be processsed by the CRL-FCM using a software package for statistical analyses and professional data handling of interlaboratory tests.

For that purpose:

– a simple data entry program (RingDat3.exe) is provided to each participating laboratory;
– two additional lab files with the extension "participant.LAB" and "participant.LA2", generated by the ProLab software are provided to each laboratory individually (personal files);
– the name of each laboratory and the samples are codified by the software, so that each participant will receive a sample with unique codified numbers (i.e., 0586);
– The "*.LA2" file contains information about the participant – laboratory name and laboratory code;
– The "*.LAB" file is unique to each laboratory (personal) and contains information about the samples (samples code) and measurands that have to be analysed and reported.

Each laboratory has to start the RingDat3.exe program and to open "name.LAB" file for reporting the results. A table will appear with cells for every measurand/sample combination.

One lab file (i.e.,Nrl_it.lab) per participant is generated for both parts of the Proficiency test – Part A (the 2 gaskets, e.g. ESBO and phthalate based) as well as for the Part B (the 2 oils, e.g. ESBO and phthalate based). That is why 4 sample codes will appear for each participant/file. As the deadline for reporting the results for part A and part B is the same you have the possibility to fill all your results together and send it by e-mail to Catherine Simoneau (catherine.simoneau@jrc.it) by 12 September.

Results requested:
Perform four replicates for each sample and report all the four replicate data using the unit of measure specified below:
PT001-A (part A), the two gasket in mg/100 mg (%) using only gasket (PVC);
PT001-B (part B), the two oil in mg/kg.

If you have any question, please contact Catherine Simoneau (catherine.simoneau@jrc.it), ph. +39.0332.785889

Annex 5b: Instruction for the compilation of the results in electronic format

Ispra June 23, 2008
Annex to I05-PCE/CS/sm(2008)D/17212

Further explanation for the compilation of results in electronic format [CRL-FCM PT001-A (part A) and PT001-B (part B)]

Procedure to be followed by laboratories for the compilation of the results:

- Save all the files received in a folder
- Rename the file "**RINGDAT3.txt**" with the extension **.exe** ("**RINGDAT3.exe**")
- Open the file "**RINGDAT3.exe**",the folder "**Translations**" will appear.
- Put the "**German.xml**" and "**English.xml**" files inside the folder "**Translations**" (in this way the content of the file will be show in English)
- Open the file "**RINGDAT3.exe**"
- Click on "**Open**" command
- Select the "**NRL_X.LAB**" file (where X is the member state abbreviation) and click on "**Open**" command using the button on the top menu of the window
- Fill the table with your data
- Save the file using the button on the top menu of the window
- Send only the "**NRL_X.LAB**" file by e-mail to catherine.simoneau@jrc.it by 12 September

Annex 6a: letters of confirmation of participation PT001/A

Ispra June 10, 2008
Annex to I05-PCE/CS/sm(2008)D/14582

Participation to CRL-FCM PT001-A (part A)
Proficiency test exercise for the determination of plasticisers in gaskets.

CONFIRMATION OF PARTICIPATION

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

item	YES	NO
I will participate the collaborative trial on analysis of an **ESBO-based** gasket and will deliver results and abide by the schedule in exchange for keeping the calibrants		
I will participate the collaborative trial on analysis of a **phthalate-based** gasket and will deliver results and abide by the schedule in exchange for keeping the calibrants		

Kindly send back this proformat to: Catherine Simoneau (catherine.simoneau@jrc.it) **by June 13.**
The samples will be sent to you this coming week. You will find additional information in the kit sent.
The deadline for submission of results is **12 September 2008**

If you have any question, please contact Catherine Simoneau (catherine.simoneau@jrc.it), ph. +39.0332.785889

Sincerely yours,

Catherine Simoneau

Annex 6b: letters of confirmation of participation PT001/B

Ispra June 10, 2008
Annex to I05-PCE/CS/sm(2008)D/14582

Participation to CRL-FCM PT001-B (part B)
Proficiency test exercise for the determination of plasticisers in oil.

CONFIRMATION OF PARTICIPATION

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

item	YES	NO
I will participate the collaborative trial on analysis of an **ESBO-based** oil and will deliver results and abide by the schedule in exchange for keeping the calibrants		
I will participate the collaborative trial on analysis of a **phthalate-based** oil and will deliver results and abide by the schedule in exchange for keeping the calibrants		

Kindly send back this proformat to: Catherine Simoneau (catherine.simoneau@jrc.it) **by June 13.**
The samples will be sent to you this coming week. You will find additional information in the kit sent.
The deadline for submission of results is **12 September 2008**

If you have any question, please contact Catherine Simoneau (catherine.simoneau@jrc.it), ph. +39.0332.785889

Sincerely yours,

Catherine Simoneau

Annex 7a: letters of confirmation of receipt PT001/A

Ispra June 10, 2008
Annex to I05-PCE/CS/sm(2008)D/14582

Participation to CRL-FCM PT001-A (part A)
Proficiency test exercise for the determination of plasticisers in gaskets.

CONFIRMATION OF RECEIPT OF THE SAMPLES

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

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

Any remarks ……

Date arrival package ……

Signature ……

Kindly send back this form to: Catherine Simoneau (catherine.simoneau@jrc.it).

Sincerely yours,

Catherine Simoneau

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

Annex 7b: letters of confirmation of receipt PT001/B

Ispra July 08, 2008
Annex to I05-PCE/CS/sm(2008)D/17207

Participation to CRL-FCM PT001-B (part B)
Proficiency test exercise for the determination of plasticisers in Oil.

CONFIRMATION OF RECEIPT OF THE SAMPLES

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

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

Any remarks ……

Date arrival package ……

Signature ……

Kindly send back this form to: Catherine Simoneau (catherine.simoneau@jrc.it).

Sincerely yours,

Catherine Simoneau

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

Annex 8 Summary of laboratories participation in interlaboratory comparison exercises

Member State	Name of NRL/PARTICIPANT
AUSTRIA	Austrian Agency for Health and Food Safety (AGES),
BELGIUM	Institute of Public Health, ISSP-LP
REPUBLIC OF CYPRUS	Laboratory for Control of Food Contact Materials and Control of Toys Ministry of Health, State General Laboratory (SGL)
CZECH REPUBLIC	NIPH- NRL for Food Contact Materials and for Articles for children under 3 years old, National Institute of Public Health (SZU')
DENMARK	Department of Food Chemistry, National Food Institute Technical University of Denmark
ESTONIA	Health Protection Inspectorate - Central Laboratory of Chemistry
FINLAND	Finnish Customs Laboratory
FRANCE	Center for Energy Material and Packaging - Laboratoire National d'Essais SCL Laboratoire de Bordeaux-Pessac
GERMANY	Bundesinstitut für Risikobewertung (BFR) (Federal Institute for Risk Assessment) + 7 laboratories
GREECE	General Chemical State Laboratory, D' Chemical Service of Athens, Section, Laboratory of Articles and Materials in Contact with Foodstuffs
HUNGARY	National Institute of Food Hygiene and Nutrition – Dept of Food additives and contaminants, Section Food Additives and Contact Materials
IRELAND	Public Analyst Laboratory - Sir Patrick Duns Hospital
POLAND	Laboratory of Department of Food and Consumer Articles Research , National Institute of Hygiene,
PORTUGAL	ESB-SE (Portuguese Catholic University - Biotechnology College – Packaging Department)
SLOVENIA	National Institute of Public Health of Republic of Slovenia , Dept of Sanitary Chemistry,
SLOVAK REPUBLIC	National Reference Centre and Laboratory for material and articles intended to come into contact with food, Regional Public Health Authority In
SPAIN	Centro Nacional de Alimentación, Agencia Espanola de Seguridad Alimentaria y Nutrición (AESAN)
SWEDEN	National Food Administration, Food Standards Division
THE NETHERLANDS	Food and Consumer Product Safety Authority (VWA), Inspectorate for Health Protection region North
UNITED KINGDOM	Central Science Laboratory
ITALY	Neotron.S.p.a.
ITALY	SSICA
THAILAND	Food Packaging Laboratory Biological science program , Bangkok, THAILAND
Germany	Landesuntersuchungsanstalt fr das Gesundheits- und Veterin rwesen Sachsen FG 6.3 - Bedarfsgegenstände
Germany	Bayerisches Landesamt für Gesundheit und Lebensmittelsicherheit Bavarian Health and Food Safety Authority
Germany	Hessisches Landeslabor LHL Standort Wiesbaden
Germany	Landesamt für Landwirtschaft, Lebensmittelsicherheit und Fischerei Mecklenburg Vorpommern Dezernat kosmetische Mittel und Bedarfsgegenstände
Germany	Landesuntersuchungsamt Institut für Lebensmittelchemie Koblenz
Germany	Chemisches und Veterinäruntersuchungsamt Rhein-Ruhr-Wupper (CVUA-RRW)

Annex 9

Procedure for the preparation of the spike of plasticisers in sunflower oil for PT test

1. Washing of the mixing tank:

- The mixing tank was filled with 20 L of deionized water (resistivity 18.0 MΩ.cm @ 25 °C) and mixed overnight
- The water was removed
- The tank was filled with 20 mL of ethanol (Fluka HPLC grade) and mixed for 4 hours
- Ethanol was removed. The walls of the tank and the mixing device were rinsed with ethanol
- The tank was filled with 20 L of n-hexane (Sigma-Aldrich HPLC grade) and mixed for 2 hours
- n-hexane was removed. The walls of the tank and the mixing device were rinsed with n-hexane
- the walls of the tank and the mixing device was dried with a stream of pure nitrogen

Note: all the solvents used were previously distilled and the glassware used during the procedure rinsed with n-hexane and pre-heated at 450ºC to prevent phtalates contamination.

2. Preparation of BLANK OIL:

- A 20 L can of sunflower oil (A) was weighted.
- The oil was transferred into the mixing tank and it was mixed for 5 hours
- The empty can (A) was weighted.
- The procedure was repeated for a second can of oil (B)
- The weight of the oil inside the tank (P1) was calculated by subtracting the weights of the empty cans to the weights of the full cans
- An empty can (C) was weighted
- Part of the oil inside the tank was transferred into the empty can (C)
- The can (C) was weighted
- The difference between the weights of the can (C) before and after the transfer of the oil was the weight of the oil transferred (blank oil) (P2)
- The weight of the residual oil (P3) inside the mixing tank was obtained: P3= P1-P2
- The blank oil was distributed in 100 mL bottles

Note: all the weights were repeated for 3 times and the average value was calculated.

3. Spiking procedure

- Each compound was weighted on a glass support
- Each glass support was immersed into the oil using stainless steel rods
- The oil was mixed for 18 hours
- The oil was distributed in 100 mL bottles

4. Target and final concentrations for plasticisers, spiked in oil 1 and oil 2

	Units	Target concentr.	Weighted amount	Purity, %	Concentr. obtained	Uncertainty
ESBO	mg/kg	60	656.66	> 99	60.53	1.44
DINCH	mg/kg	15	161.22	> 99.5	14.86	0.27
BHT	mg/kg	2.9	32.25	> 99	2.96	0.13
b-sebacate	mg/kg	3.1	34.65	> 97	3.11	0.15

	Units	Target concentr.	Weighted amount	Purity, %	Concentr. obtained	Uncertainty
DIDP	mg/kg	8	115.21	> 99.6	8.00	0.17
BHT	mg/kg	1.1	15.44	> 99.0	1.07	0.02
b-sebacate	mg/kg	1.5	21.22	> 97	1.44	0.06
BBP	mg/kg	15	214.87	>	14.97	0.29

Annex 10: Results of the homogeneity study

1a. Homogeneity data for DINCH in gasket.

homogeneity gasket total between

Test on homogeneity

Sample: **ESBO based**
Measurand: **DINCH**

Date: 11/7/2008

Mean:	4.004 %
Analytical standard deviation:	1.506%
Heterogeneity standard deviation s(samples):	4.465%
Target standard deviation:	15.00% (Manual)

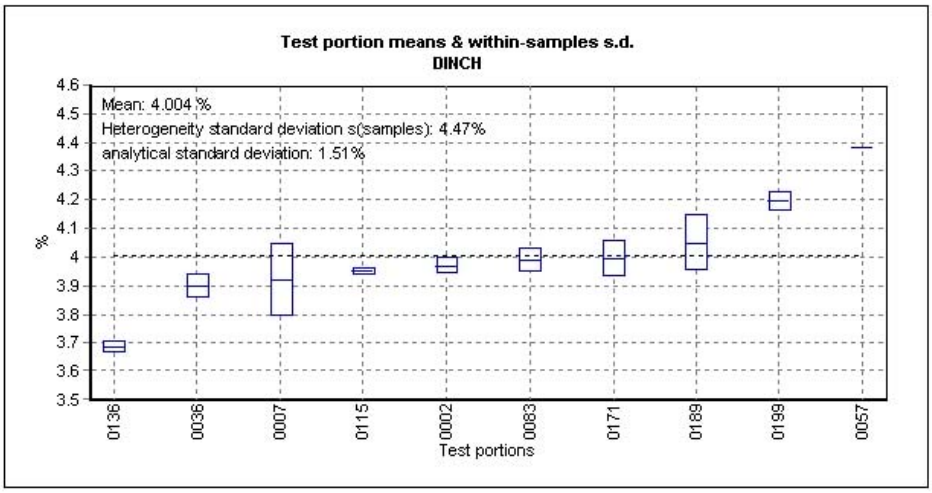
Results of homogeneity analysis (with statistical background)

According to ISO 13528, the heterogeneity standard deviation s(samples) between the samples of the test material should be no more than one third of the target standard deviation. For a heterogeneity check, 10 of the test portions of ESBO based gasket were selected at random, and DINCH was determined 2 times at each test portion.
The average content over all samples is 4.004 %, the relative standard deviation of the sample averages is 4.59%, the relative within-samples deviation s(analytical) is 1.51% and hence the relative heterogeneity standard deviation s(samples) equals 4.47%. For the tests on homogeneity a significance level of 5% is assumed.

According to the F-test, the relative heterogeneity standard deviation differs statistically significant from zero. Therefore it can be concluded that the samples are heterogeneous.

For the specified target standard deviation of 15.00% (Manual) the analytical precision of the method fulfils the requirements of the Harmonized Protocol. Also according to the Harmonized Protocol, no statistically significant heterogeneity of the samples can be detected.
It should be noted that the absence of significant heterogeneity is no proof of homogeneity of samples.

According to ISO 13528, the samples are suitable for the interlaboratory study only for a relative target standard deviation above 15%.



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quo data

Annex 10: Results of the homogeneity study

1b. Homogeneity data for ESBO in gasket.

homogeneity gasket total between

Test on homogeneity

Sample: **ESBO based** Date: 11/7/2008
Measurand: **ESBO-**

Mean:	22.391 %
Analytical standard deviation:	2.758%
Heterogeneity standard deviation s(samples):	4.324%
Target standard deviation:	15.00% (Manual)

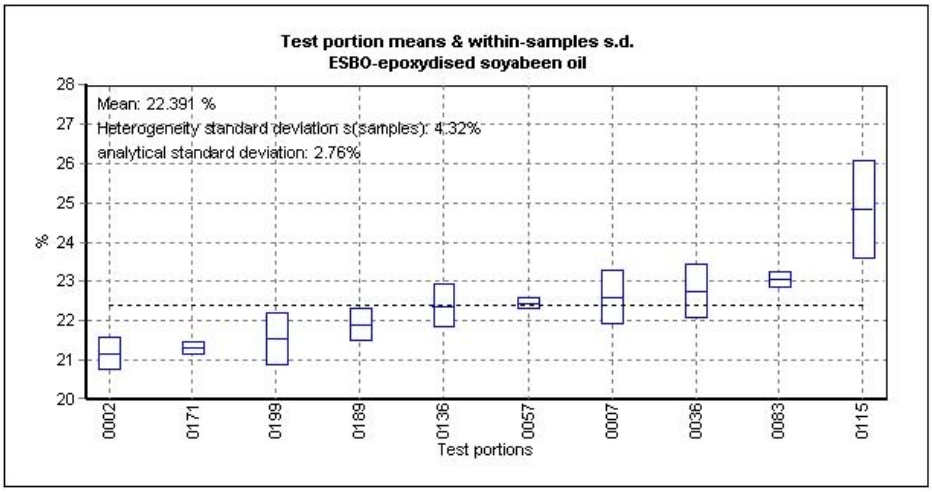
Results of homogeneity analysis (with statistical background)

According to ISO 13528, the heterogeneity standard deviation s(samples) between the samples of the test material should be no more than one third of the target standard deviation. For a heterogeneity check, 10 of the test portions of ESBO based gasket were selected at random, and ESBO-epoxydised soyabeen oil was determined 2 times at each test portion.
The average content over all samples is 22.391 %, the relative standard deviation of the sample averages is 4.74%, the relative within-samples deviation s(analytical) is 2.76% and hence the relative heterogeneity standard deviation s(samples) equals 4.32%. For the tests on homogeneity a significance level of 5% is assumed.

According to the F-test, the relative heterogeneity standard deviation differs statistically significant from zero. Therefore it can be concluded that the samples are heterogeneous.

For the specified target standard deviation of 15.00% (Manual) the analytical precision of the method fulfils the requirements of the Harmonized Protocol. Also according to the Harmonized Protocol, no statistically significant heterogeneity of the samples can be detected.
It should be noted that the absence of significant heterogeneity is no proof of homogeneity of samples.

According to ISO 13528, the samples are suitable for the interlaboratory study only for a relative target standard deviation above 14%.



Annex 10: Results of the homogeneity study

1c. Homogeneity data for butyl-sebacate in gasket.

homogeneity gasket total between

Test on homogeneity

Sample: **ESBO based**
Measurand: **butyl sebacate**

Date: 11/7/2008

Mean:	3.480 %
Analytical standard deviation:	2.980%
Heterogeneity standard deviation s(samples):	0.000%
Target standard deviation:	15.00% (Manual)

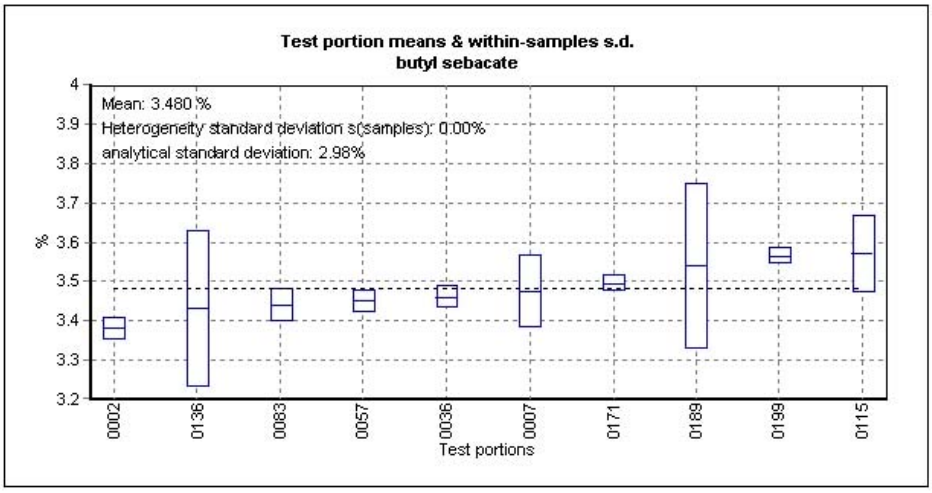
Results of homogeneity analysis (with statistical background)

According to ISO 13528, the heterogeneity standard deviation s(samples) between the samples of the test material should be no more than one third of the target standard deviation. For a heterogeneity check, 10 of the test portions of ESBO based gasket were selected at random, and butyl sebacate was determined 2 times at each test portion.
The average content over all samples is 3.480 %, the relative standard deviation of the sample averages is 2.11%, the relative within-samples deviation s(analytical) is 2.98% and hence the relative heterogeneity standard deviation s(samples) equals 0.00%. For the tests on homogeneity a significance level of 5% is assumed.

The relative heterogeneity standard deviation s(samples) equals 0.00% and therefore also no statistically significant difference to zero can be detected by the F-test.

For the specified target standard deviation of 15.00% (Manual) the analytical precision of the method fulfils the requirements of the Harmonized Protocol. Also according to the Harmonized Protocol, no statistically significant heterogeneity of the samples can be detected.
It should be noted that the absence of significant heterogeneity is no proof of homogeneity of samples.

According to ISO 13528, the samples are suitable for the interlaboratory study only for a relative target standard deviation above 0.00%.



Annex 10: Results of the homogeneity study

1d. Homogeneity data for DIDP in gasket.

homogeneity gasket total between

Test on homogeneity

Sample: **DIDP based**
Measurand: **DIDP-**

Date: 11/7/2008

Mean:	32.350 %
Analytical standard deviation:	4.166%
Heterogeneity standard deviation s(samples):	10.580%
Target standard deviation:	15.00% (Manual)

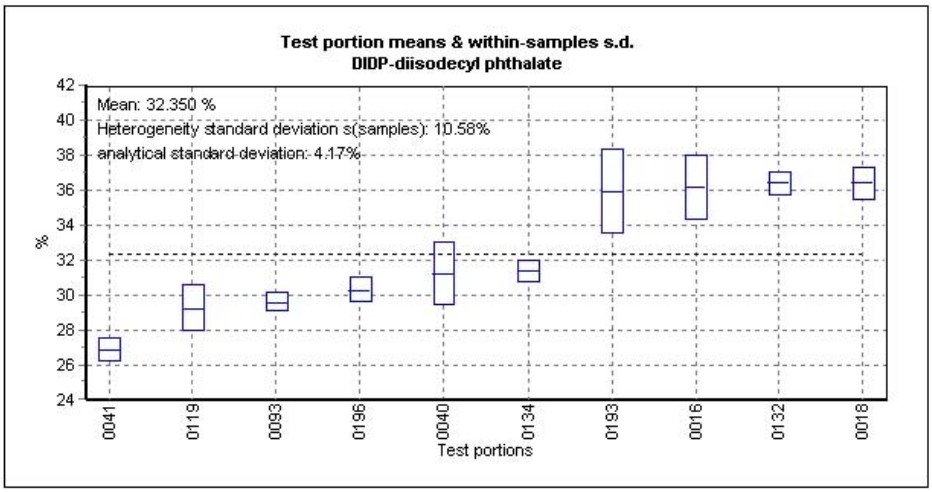
Results of homogeneity analysis (with statistical background)

According to ISO 13528, the heterogeneity standard deviation s(samples) between the samples of the test material should be no more than one third of the target standard deviation. For a heterogeneity check, 10 of the test portions of DIDP based gasket were selected at random, and DIDP-diisodecyl phthalate was determined 2 times at each test portion.
The average content over all samples is 32.350 %, the relative standard deviation of the sample averages is 10.98%, the relative within-samples deviation s(analytical) is 4.17% and hence the relative heterogeneity standard deviation s(samples) equals 10.58%. For the tests on homogeneity a significance level of 5% is assumed.

According to the F-test, the relative heterogeneity standard deviation differs statistically significant from zero. Therefore it can be concluded that the samples are heterogeneous.

For the specified target standard deviation of 15.00% (Manual) the analytical precision of the method fulfils the requirements of the Harmonized Protocol. However, also according to the Harmonized Protocol a statistically significant heterogeneity of the samples can be detected.

According to ISO 13528, the heterogeneity of the samples is acceptable if the relative target standard deviation is greater than 35%.



Annex 10: Results of the homogeneity study

1e. Homogeneity data for BHT in oil 1.

homogeneity oil 1

Test on homogeneity

Sample: **oil1 ESBO** Date: 11/7/2008
Measurand: **BHT-butylated**

Mean:	3.030 mg/kg
Analytical standard deviation:	5.039%
Heterogeneity standard deviation s(samples):	0.000%
Target standard deviation:	13.54% (Horwitz)

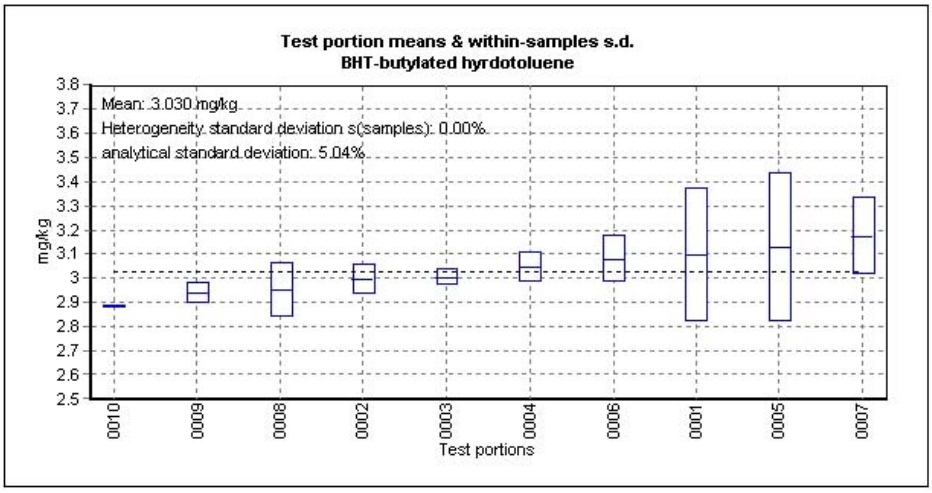
Results of homogeneity analysis (with statistical background)

According to ISO 13528, the heterogeneity standard deviation s(samples) between the samples of the test material should be no more than one third of the target standard deviation. For a heterogeneity check, 10 of the test portions of oil1 ESBO based were selected at random, and BHT-butylated hyrdotoluene was determined 2 times at each test portion.
The average content over all samples is 3.030 mg/kg, the relative standard deviation of the sample averages is 3.56%, the relative within-samples deviation s(analytical) is 5.04% and hence the relative heterogeneity standard deviation s(samples) equals 0.00%. For the tests on homogeneity a significance level of 5% is assumed.

The relative heterogeneity standard deviation s(samples) equals 0.00% and therefore also no statistically significant difference to zero can be detected by the F-test.

For the specified target standard deviation of 13.54% (Horwitz) the analytical precision of the method fulfils the requirements of the Harmonized Protocol. Also according to the Harmonized Protocol, no statistically significant heterogeneity of the samples can be detected.
It should be noted that the absence of significant heterogeneity is no proof of homogeneity of samples.

According to ISO 13528, the samples are suitable for the interlaboratory study only for a relative target standard deviation above 0.00%.



Annex 10: Results of the homogeneity study

1f. Homogeneity data for DINCH in oil 1.

homogeneity oil 1

Test on homogeneity

Sample: **oil1 ESBO** Date: 11/7/2008
Measurand: **DINCH**

Mean:	15.548 mg/kg
Analytical standard deviation:	2.103%
Heterogeneity standard deviation s(samples):	0.000%
Target standard deviation:	10.59% (Horwitz)

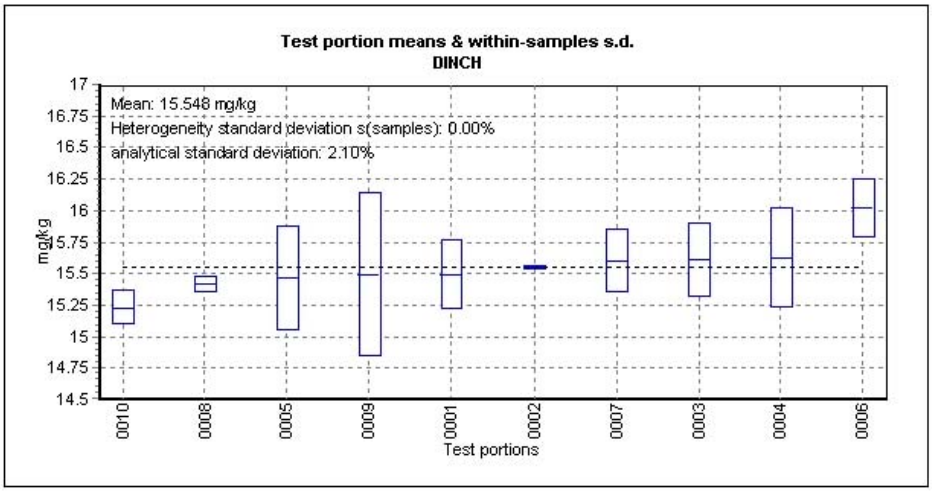
Results of homogeneity analysis (with statistical background)

According to ISO 13528, the heterogeneity standard deviation s(samples) between the samples of the test material should be no more than one third of the target standard deviation. For a heterogeneity check, 10 of the test portions of oil1 ESBO based were selected at random, and DINCH was determined 2 times at each test portion.
The average content over all samples is 15.548 mg/kg, the relative standard deviation of the sample averages is 1.49%, the relative within-samples deviation s(analytical) is 2.10% and hence the relative heterogeneity standard deviation s(samples) equals 0.00%. For the tests on homogeneity a significance level of 5% is assumed.

The relative heterogeneity standard deviation s(samples) equals 0.00% and therefore also no statistically significant difference to zero can be detected by the F-test.

For the specified target standard deviation of 10.59% (Horwitz) the analytical precision of the method fulfils the requirements of the Harmonized Protocol. Also according to the Harmonized Protocol, no statistically significant heterogeneity of the samples can be detected.
It should be noted that the absence of significant heterogeneity is no proof of homogeneity of samples.

According to ISO 13528, the samples are suitable for the interlaboratory study only for a relative target standard deviation above 0.00%.



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Demo version Page 1

Annex 10: Results of the homogeneity study

1g. Homogeneity data for ESBO in oil 1.

homogeneity oil 1

Test on homogeneity

Sample: **oil1 ESBO** Date: 11/7/2008
Measurand: **ESBO-**

Mean:	56.252 mg/kg
Analytical standard deviation:	2.488%
Heterogeneity standard deviation s(samples):	2.579%
Target standard deviation:	8.72% (Horwitz)

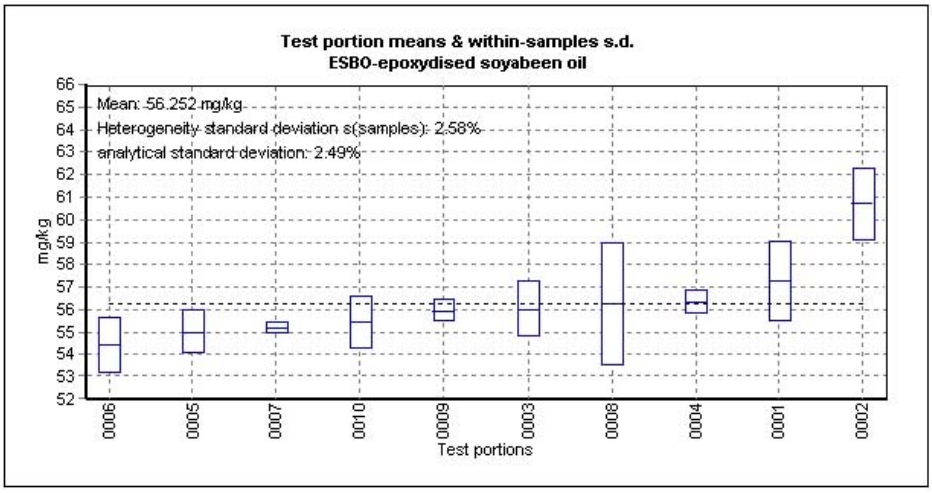
Results of homogeneity analysis (with statistical background)

According to ISO 13528, the heterogeneity standard deviation s(samples) between the samples of the test material should be no more than one third of the target standard deviation. For a heterogeneity check, 10 of the test portions of oil1 ESBO based were selected at random, and ESBO-epoxydised soyabeen oil was determined 2 times at each test portion.
The average content over all samples is 56.252 mg/kg, the relative standard deviation of the sample averages is 3.12%, the relative within-samples deviation s(analytical) is 2.49% and hence the relative heterogeneity standard deviation s(samples) equals 2.58%. For the tests on homogeneity a significance level of 5% is assumed.

According to the F-test, the relative heterogeneity standard deviation differs statistically significant from zero. Therefore it can be concluded that the samples are heterogeneous.

For the specified target standard deviation of 8.72% (Horwitz) the analytical precision of the method fulfils the requirements of the Harmonized Protocol. Also according to the Harmonized Protocol, no statistically significant heterogeneity of the samples can be detected.
It should be noted that the absence of significant heterogeneity is no proof of homogeneity of samples.

According to ISO 13528, the samples are suitable for the interlaboratory study only for a relative target standard deviation above 9%.



Annex 10: Results of the homogeneity study

1h. Homogeneity data for b-sebacate in oil 1.

homogeneity oil 1

Test on homogeneity

Sample: **oil1 ESBO** Date: 11/7/2008
Measurand: **butyl sebacate**

Mean:	3.285 mg/kg
Analytical standard deviation:	4.106%
Heterogeneity standard deviation s(samples):	0.000%
Target standard deviation:	13.38% (Horwitz)

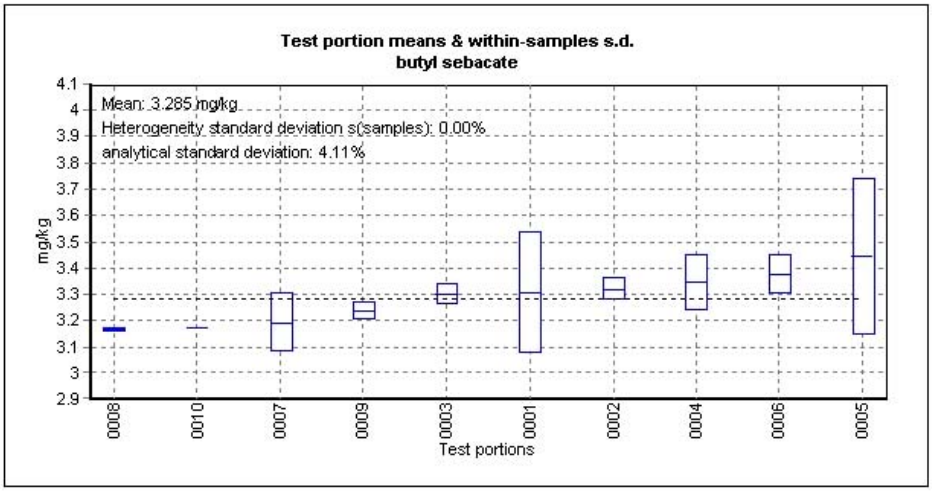
Results of homogeneity analysis (with statistical background)

According to ISO 13528, the heterogeneity standard deviation s(samples) between the samples of the test material should be no more than one third of the target standard deviation. For a heterogeneity check, 10 of the test portions of oil1 ESBO based were selected at random, and butyl sebacate was determined 2 times at each test portion.
The average content over all samples is 3.285 mg/kg, the relative standard deviation of the sample averages is 2.90%, the relative within-samples deviation s(analytical) is 4.11% and hence the relative heterogeneity standard deviation s(samples) equals 0.00%. For the tests on homogeneity a significance level of 5% is assumed.

The relative heterogeneity standard deviation s(samples) equals 0.00% and therefore also no statistically significant difference to zero can be detected by the F-test.

For the specified target standard deviation of 13.38% (Horwitz) the analytical precision of the method fulfils the requirements of the Harmonized Protocol. Also according to the Harmonized Protocol, no statistically significant heterogeneity of the samples can be detected.
It should be noted that the absence of significant heterogeneity is no proof of homogeneity of samples.

According to ISO 13528, the samples are suitable for the interlaboratory study only for a relative target standard deviation above 0.00%.



CRL-FCM 11/7/2008 ProLab
Demo version Page 1

Annex 10: Results of the homogeneity study

1i. Homogeneity data for DIDP in oil 2.

homogeneity oil 2

Test on homogeneity

Sample: **oil 2 phtalate** Date: 11/7/2008
Measurand: **DIDP-**

Mean:	9.058 mg/kg
Analytical standard deviation:	2.417%
Heterogeneity standard deviation s(samples):	2.627%
Target standard deviation:	11.48% (Horwitz)

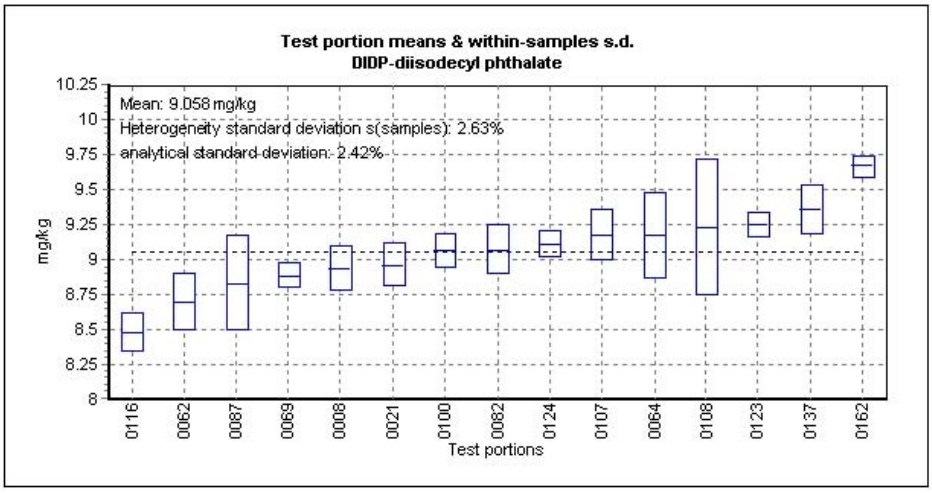
Results of homogeneity analysis (with statistical background)

According to ISO 13528, the heterogeneity standard deviation s(samples) between the samples of the test material should be no more than one third of the target standard deviation. For a heterogeneity check, 15 of the test portions of oil 2 phtalate based were selected at random, and DIDP-diisodecyl phthalate was determined 2 times at each test portion.
The average content over all samples is 9.058 mg/kg, the relative standard deviation of the sample averages is 3.13%, the relative within-samples deviation s(analytical) is 2.42% and hence the relative heterogeneity standard deviation s(samples) equals 2.63%. For the tests on homogeneity a significance level of 5% is assumed.

According to the F-test, the relative heterogeneity standard deviation differs statistically significant from zero. Therefore it can be concluded that the samples are heterogeneous.

For the specified target standard deviation of 11.48% (Horwitz) the analytical precision of the method fulfils the requirements of the Harmonized Protocol. Also according to the Harmonized Protocol, no statistically significant heterogeneity of the samples can be detected.
It should be noted that the absence of significant heterogeneity is no proof of homogeneity of samples.

According to ISO 13528, the samples are suitable for the interlaboratory study only for a relative target standard deviation above 9%.



Annex 10: Results of the homogeneity study

1k. Homogeneity data for BBP in oil 2.

homogeneity oil 2

Test on homogeneity

Sample: **oil 2 phtalate** Date: 11/7/2008
Measurand: **BBP-benzyl**

Mean:	15.042 mg/kg
Analytical standard deviation:	1.667%
Heterogeneity standard deviation s(samples):	0.469%
Target standard deviation:	10.64% (Horwitz)

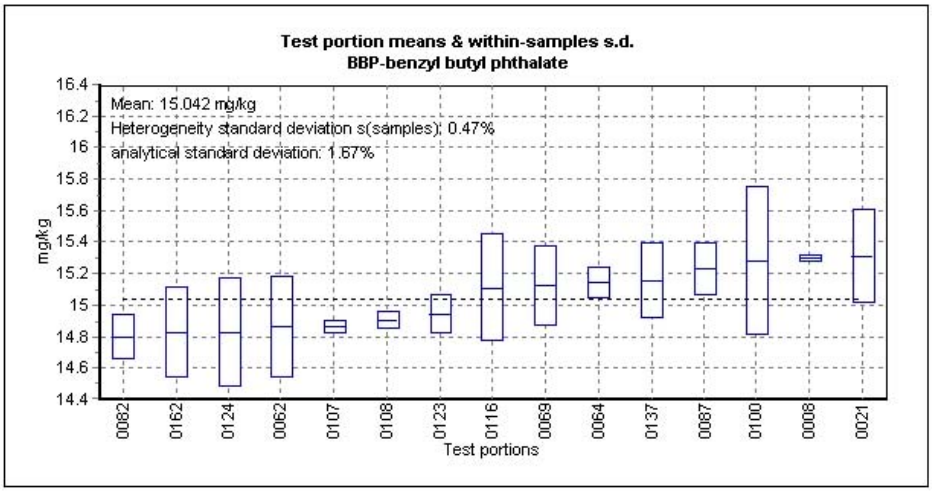
Results of homogeneity analysis (with statistical background)

According to ISO 13528, the heterogeneity standard deviation s(samples) between the samples of the test material should be no more than one third of the target standard deviation. For a heterogeneity check, 15 of the test portions of oil 2 phtalate based were selected at random, and BBP-benzyl butyl phthalate was determined 2 times at each test portion.
The average content over all samples is 15.042 mg/kg, the relative standard deviation of the sample averages is 1.27%, the relative within-samples deviation s(analytical) is 1.67% and hence the relative heterogeneity standard deviation s(samples) equals 0.47%. For the tests on homogeneity a significance level of 5% is assumed.

Although the relative heterogeneity standard deviation s(samples) differs from zero, this deviation is not statistically significant according to the F-test.

For the specified target standard deviation of 10.64% (Horwitz) the analytical precision of the method fulfils the requirements of the Harmonized Protocol. Also according to the Harmonized Protocol, no statistically significant heterogeneity of the samples can be detected.
It should be noted that the absence of significant heterogeneity is no proof of homogeneity of samples.

According to ISO 13528, the samples are suitable for the interlaboratory study only for a relative target standard deviation above 1.6%.



Annex 10: Results of the homogeneity study

1l. Homogeneity data for b-sebacate in oil 2

homogeneity oil 2

Test on homogeneity

Sample: **oil 2 phtalate**
Measurand: **butyl sebacate**

Date: 11/7/2008

Mean:	1.616 mg/kg
Analytical standard deviation:	2.308%
Heterogeneity standard deviation s(samples):	2.960%
Target standard deviation:	14.88% (Horwitz)

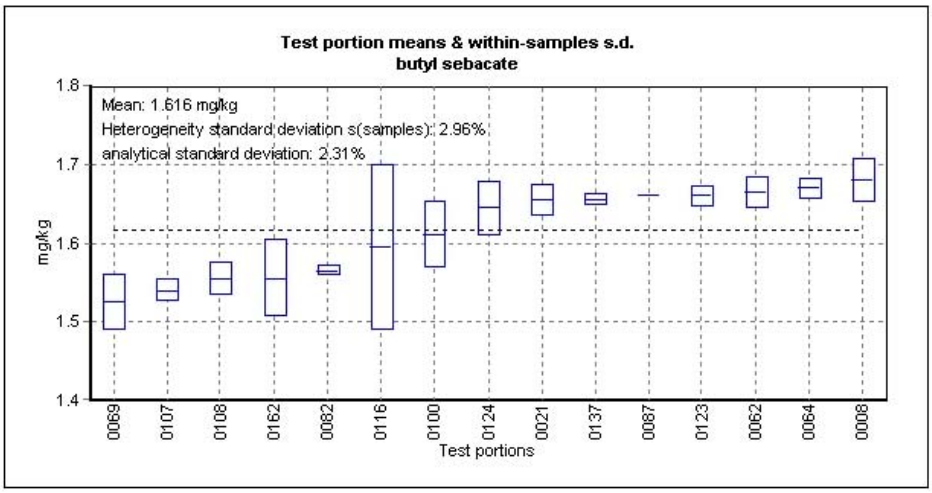
Results of homogeneity analysis (with statistical background)

According to ISO 13528, the heterogeneity standard deviation s(samples) between the samples of the test material should be no more than one third of the target standard deviation. For a heterogeneity check, 15 of the test portions of oil 2 phtalate based were selected at random, and butyl sebacate was determined 2 times at each test portion.
The average content over all samples is 1.616 mg/kg, the relative standard deviation of the sample averages is 3.38%, the relative within-samples deviation s(analytical) is 2.31% and hence the relative heterogeneity standard deviation s(samples) equals 2.96%. For the tests on homogeneity a significance level of 5% is assumed.

According to the F-test, the relative heterogeneity standard deviation differs statistically significant from zero. Therefore it can be concluded that the samples are heterogeneous.

For the specified target standard deviation of 14.88% (Horwitz) the analytical precision of the method fulfils the requirements of the Harmonized Protocol. Also according to the Harmonized Protocol, no statistically significant heterogeneity of the samples can be detected.
It should be noted that the absence of significant heterogeneity is no proof of homogeneity of samples.

According to ISO 13528, the samples are suitable for the interlaboratory study only for a relative target standard deviation above 10%.



Annex 10: Results of the homogeneity study

1m. Homogeneity data for BHT in oil 2.

homogeneity oil 2

Test on homogeneity

Sample: **oil 2 phtalate**
Measurand: **BHT-butylated**

Date: 11/7/2008

Mean:	1.082 mg/kg
Analytical standard deviation:	1.848%
Heterogeneity standard deviation s(samples):	0.904%
Target standard deviation:	15.81% (Horwitz)

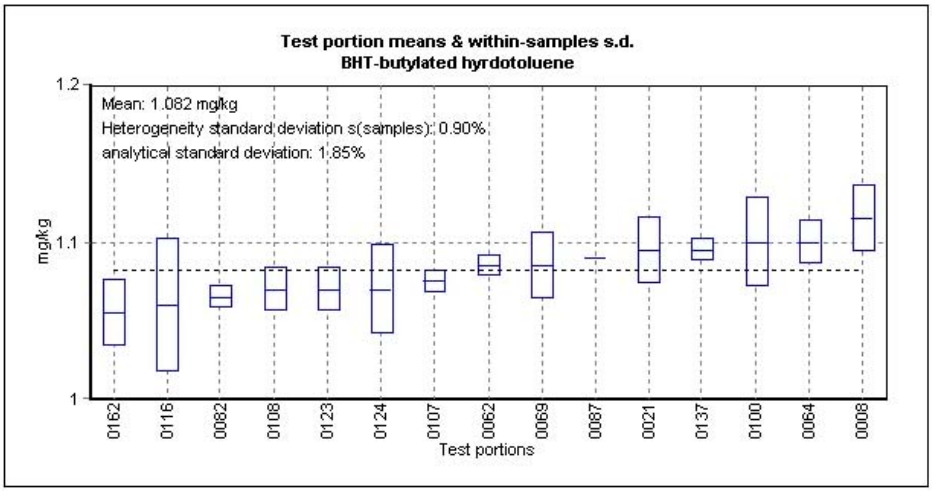
Results of homogeneity analysis (with statistical background)

According to ISO 13528, the heterogeneity standard deviation s(samples) between the samples of the test material should be no more than one third of the target standard deviation. For a heterogeneity check, 15 of the test portions of oil 2 phtalate based were selected at random, and BHT-butylated hyrdotoluene was determined 2 times at each test portion.
The average content over all samples is 1.082 mg/kg, the relative standard deviation of the sample averages is 1.59%, the relative within-samples deviation s(analytical) is 1.85% and hence the relative heterogeneity standard deviation s(samples) equals 0.90%. For the tests on homogeneity a significance level of 5% is assumed.

Although the relative heterogeneity standard deviation s(samples) differs from zero, this deviation is not statistically significant according to the F-test.

For the specified target standard deviation of 15.81% (Horwitz) the analytical precision of the method fulfils the requirements of the Harmonized Protocol. Also according to the Harmonized Protocol, no statistically significant heterogeneity of the samples can be detected.
It should be noted that the absence of significant heterogeneity is no proof of homogeneity of samples.

According to ISO 13528, the samples are suitable for the interlaboratory study only for a relative target standard deviation above 3%.



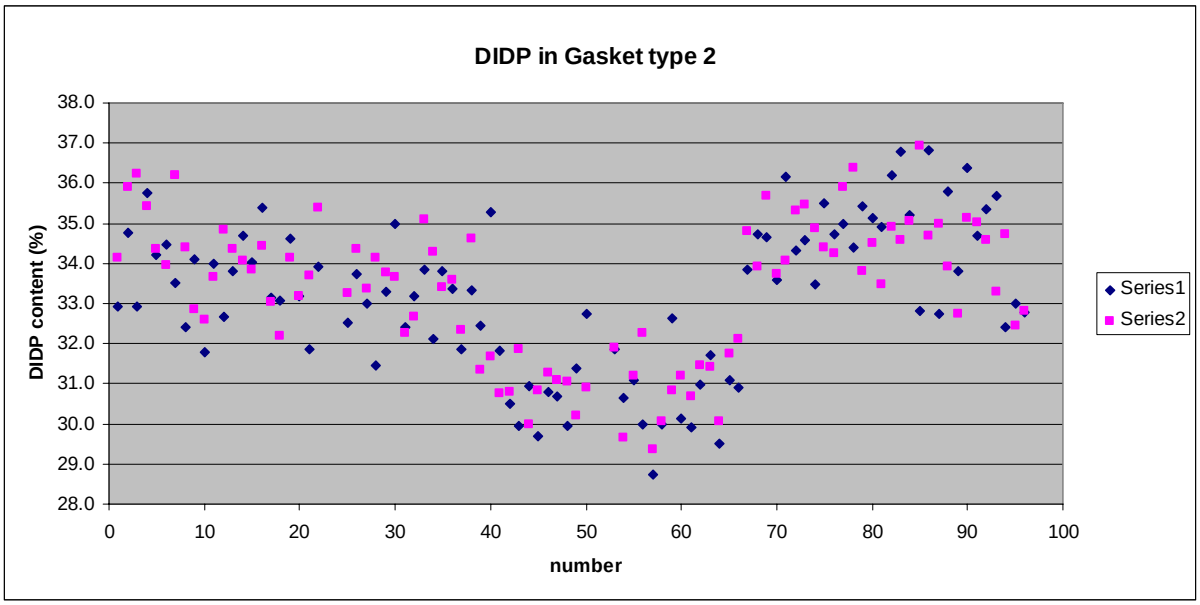
Annex 10: Results of the homogeneity study

2. Summary of the homogeneity test between the samples in the lots

Sample	Measurand	Unit	Mean	s(analytical) %	s(samples) %	Mode s(target)	s(target) %	ISO 13528 Check for sufficient homogeneity	Harmonized Protocol - test on significant heterogeneity
GASKET1	DINCH	%	4.0	1.5	4.5	Manual	15	OK	OK
GASKET1	ESBO	%	22.4	2.8	4.3	Manual	15	OK	OK
GASKET1	SEBACATE	%	3.5	3.0	0.0	Manual	15	OK	OK
GASKET2	DIDP	%	32.4	4.2	10.6	Manual	15	Not OK	Not OK
GASKET2/50	DIDP		33.9	3.0	1.9	Manual	15	OK	OK
OIL1	BHT	mg/kg	3.0	5.0	0.0	Horwitz	13.5	OK	OK
OIL1	DINCH	mg/kg	15.5	2.1	0.0	Horwitz	10.6	OK	OK
OIL1	ESBO	mg/kg	56.3	2.5	2.6	Horwitz	8.7	OK	OK
OIL1	SEBACATE	mg/kg	3.3	4.1	0.0	Horwitz	13.4	OK	OK
OIL2	BBP	mg/kg	15.0	1.7	0.5	Horwitz	10.6	OK	OK
OIL2	BHT	mg/kg	1.1	1.9	0.9	Horwitz	15.8	OK	OK
OIL2	DIDP	mg/kg	9.1	2.4	2.6	Horwitz	11.5	OK	OK
OIL2	SEBACATE	mg/kg	1.6	2.3	3.0	Horwitz	14.9	OK	OK

Annex 10: Results of the homogeneity study

3. Homogeneity test for lot “gasket type2”



Annex 10: Results of the homogeneity study

4. Homogeneity data for DIDP in 50 gasket samples.

homogeneity DIDP gasket

Test on homogeneity

Sample: **GASKET**
Measurand: **DIDP-**

Date: 11/7/2008

Mean:	33.947
Analytical standard deviation:	2.964%
Heterogeneity standard deviation s(samples):	1.916%
Target standard deviation:	15.00% (Manual)

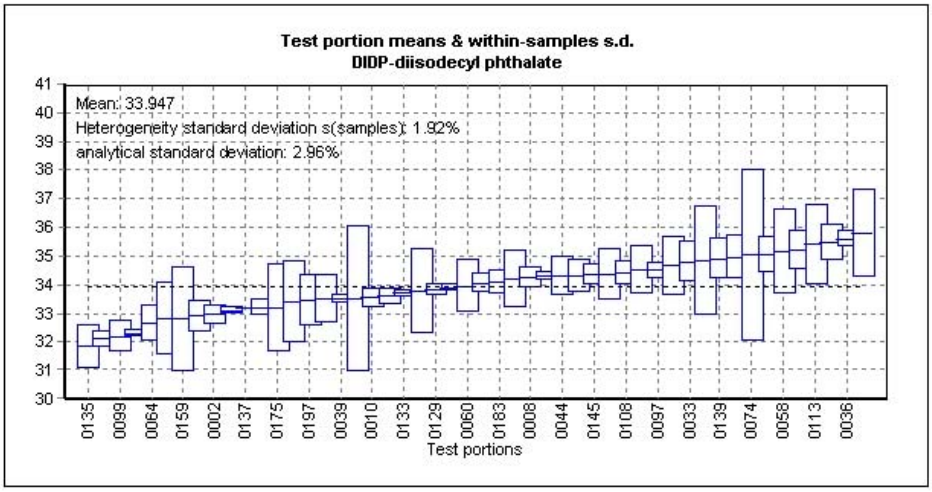
Results of homogeneity analysis (with statistical background)

According to ISO 13528, the heterogeneity standard deviation s(samples) between the samples of the test material should be no more than one third of the target standard deviation. For a heterogeneity check, 50 of the test portions of GASKET were selected at random, and DIDP-diisodecyl phthalate was determined 2 times at each test portion.
The average content over all samples is 33.947 , the relative standard deviation of the sample averages is 2.84%, the relative within-samples deviation s(analytical) is 2.96% and hence the relative heterogeneity standard deviation s(samples) equals 1.92%. For the tests on homogeneity a significance level of 5% is assumed.

According to the F-test, the relative heterogeneity standard deviation differs statistically significant from zero. Therefore it can be concluded that the samples are heterogeneous.

For the specified target standard deviation of 15.00% (Manual) the analytical precision of the method fulfils the requirements of the Harmonized Protocol. Also according to the Harmonized Protocol, no statistically significant heterogeneity of the samples can be detected.
It should be noted that the absence of significant heterogeneity is no proof of homogeneity of samples.

According to ISO 13528, the samples are suitable for the interlaboratory study only for a relative target standard deviation above 6%.



Annex 10: Results of the homogeneity study

5. Summary of the results for the homogeneity test within gaskets

Sample	Measurand	Unit	Mean	S (analytical) %	S (samples) %	Mode s (target)	S (target) %	ISO 13528 Check for sufficient homogeneity	Harmonized Protocol - test on significant heterogeneity
GASKET1/1	DINCH	%	4.38	2.19	2.37	Manual	15	OK	OK
GASKET1/1	ESBO	%	22.36	4.3	0.0	Manual	15	OK	OK
GASKET1/1	SEBACATE	%	3.45	3.53	0.0	Manual	15	OK	OK
GASKET2/1	DIDP	%	36.41	2.53	2.61	Manual	15	OK	OK
GASKET1/2	DINCH	%	4.19	2.62	1.92	Manual	15	OK	OK
GASKET1/2	ESBO	%	26.33	6.7	3.11	Manual	15	OK	OK
GASKET1/2	SEBACATE	%	3.56	3.0	0.44	Manual	15	OK	OK
GASKET2/2	DIDP	%	36.39	4.00	0.00	Manual	15	OK	OK

European Commission

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Abstract
The Institute for Health and Consumer Protection (IHCP) of the European Commission's Directorate-General Joint Research Centre hosts the Community Reference Laboratory for Food Contact Materials (CRL-FCM). One of its core tasks is to organize interlaboratory comparisons (ILCs) among appointed National Reference Laboratories (NRLs). This report presents the results of the first ILC of the CRL-FCM which focused on the determination of Plasticisers content in PVC Gasket and in Oil matrix.
The test materials used in this exercise were virgin gasket lids coming from industrial sources for the proficiency exercise part A. For the second part of the exercise an industrial source of sunflower oil was used and spiked with several plasticisers by the CRL-FCM.
There were 41 participants to whom samples were dispatched 34 of which submitted results for at least 1 analyte-material. 21 laboratories reported results for more than 10 analyte-material combination out of 14 required.
The homogeneity studies were performed by the CRL-FCM laboratory.
The assigned value and its uncertainty for part A, virgin gaskets, were obtained after applying the robust statistics to the results obtained from the participants. The assigned values for part B, oil samples, were those obtained based on formulation, from the gravimetric measurements used to spike the material.
The uncertainty of the assigned values for oil samples was calculated combining the uncertainty of the spiking procedure with a contribution for the between-bottle homogeneity.
Participants were invited to report four replicates measurements. This was done by most of the participants.
Laboratory results were rated with z and z' scores in accordance with ISO 13528 [1] Standard deviations for proficiency assessment (also called target standard deviations) were set based on Horwitz equation for substances in the two oil samples. For the plasticisers in the two gasket samples the target standard deviation was set by the organizers to 15% in order to fulfil the required criteria for sufficient homogeneity of the sample within the lots.

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