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Report of an interlaboratory comparison organised by the European Union Reference Laboratory for Food Contact Materials

ILC01 2010 - Photoinitiators in solvent and in paperboard

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Report of the interlaboratory comparison

Photoinitiators in solvent and in paperboard

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1. Summary

Commission's Directorate-General Joint Research Centre hosts the EU Reference Laboratory for Food Contact Materials (EURL-FCM). One of its core tasks is to organize interlaboratory comparisons (ILCs) among appointed National Reference Laboratories (NRLs).

This report presents the results of the ILC of the EURL-FCM which focused on the screening for photoinitiators (PI) content in paperboard as well as in a solvent. The test materials used were two types of paperboard supplied by the industry, containing benzophenone (BP) and 4-methylbenzophenone (4MBP) and mixtures of five PIs in a solvent (acetonitrile) prepared in-house.

The general aim of the exercise was to assess the proficiency of the official control laboratories and consequently the participants were free to use an analytical method of their choice.

There were 36 participants to whom samples were dispatched (26 NRLs + 10 official national control laboratories from Germany and Spain) 32 of which submitted results. The homogeneity and stability studies were performed by the EURL-FCM laboratory. The assigned values were obtained after applying robust statistics to the results of the participants. 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 solvent. For the PI paperboard 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.

As a conclusion of the ILC exercise on the quantification of photoinitiators in the paperboard and the solvent, this ILC showed that:

- The participation in the ILC was satisfactory regarding the number of the participating labs. Thirty-two laboratories submitted their results. From the EURL-NRL network 22 laboratories out of 25 reported results. 3 NRLs laboratories did not send any results.

- For the network of NRL-FCM the outcome from the participation could be regarded as satisfactory as there are about 84% satisfactory results (262 out of 312) and only 3 NRLs did not participated out of 25.The Institute for Health and Consumer Protection (IHCP) of the European

2. Introduction

ILC studies is an essential and very important element of laboratory quality assurance, which allow 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 EU 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 EU Reference Laboratory for Food Contact Materials (EURL-FCM) organized in 2010 for a third year interlaboratory comparison tests for the network of appointed National Reference Laboratories (NRLs).

The objectives of the interlaboratory comparison (ILC) tests for 2010 were discussed and agreed in the plenary meeting with all NRLs held in July 2009 in Varese, Italy In that meeting the NRLs were asked to suggest possible tests for inclusion in the 2010 work program and one of the preferences was expressed for photoinitiators in paper and board.

Photo-initiators are used in inks hardened with ultraviolet (UV) light. Their benefits include reduced drying times compared to water-based coatings, allowing a continuous production process. UV inks are used to produce an appealing glossy cover to the printed packaging, especially for paperboard, and high amounts of photo-initiators are used for this purpose in the ink preparation. One of the most known photo-initiators is benzophenone (BP). It has been determined to migrate from printed paperboard into the food as it might be neither totally used up during the printing process, nor removed afterwards, nor irreversibly bound into the print film layer. The migration of photo-initiators from the packaging to the food through the vapor phase is possible even from the secondary packaging, i.e. when the ink is not in direct contact with the food. The commonly used BP is known to migrate easily through the vapor phase and it has been substituted with other derivatives over the last few years.

4-Methylbenzophenone (4MBP) is an example of a photo-initiator used either instead or, more rarely, together with BP. At the present time, printing inks and/or paperboard boxes are not covered by specific European legislation on food contact materials.

The substances were a cause of Europe-wide issue last year after Germany and Belgium reported the chemicals had leached into breakfast cereals. In May 2009, EFSA concluded that for adults the estimated exposure from the chemicals in the cereal was unlikely to be a health hazard but that this could not be ruled out for children.

In the wake of the 2009 alert both the European Printing Ink Association and the European Carton Board Manufacturers recommended to their members that printing inks containing 4MBP and BP were not suitable for printing of food packaging unless a functional barrier is present that blocks the transfer into food also via the gasphase.

The European Commission's Standing Committee on the Food Chain and Animal Health recommended that food contact materials printed with inks containing benzophenone or 4MBP should not be brought in contact with foods unless it is demonstrated that the transfer into food of the sum of 4MBP and BP is below 0.6mg per kg of food .

The initial aim of the ILC01 2010 exercise was to quantify the concentration of a number (n = 6-8) of photoinitiators (incl. BP and 4MBP) in a paperboard sample that has been spiked with these substances by the EURL and/or to obtain retail samples of unknown (to the NRLs) composition to test that the screening procedures in use in the laboratories are capable of detecting the photoinitiators.

At the stage of preliminary investigations on feasibility of homogeneous spiking of the paperboard with PI and on finding supplier from the industry of the paperboard containing several PI, the EURL arrived at the following conclusions:

- the in-house spiked virgin paperboard were not homogeneous enough for an ILC exercise;
- the industry could provide two types of paperboard materials containing only a single PI;

A decision was then taken by EURL that the ILC01 2010 would consist of determination of BP and 4MBP in two types of paperboard samples and that two mixtures of 5 PI fortified at different concentration levels in solvent would be included for quantitative determination of PI. The additional 3 PI - 4HBP, PBZ and ITX - were chosen after consultation with NRLs via mails.

Each NRL was free to use its own methodology. Therefore this exercise was a 'proficiency test' and z-scores were assigned for each substance.

3. Scope

The scope of this ILC was to test the competence of the appointed NRLs to analyze photoinitiators in paperboard and solvent

MIX1 and MIX2 were spiked at two concentration levels of PI corresponding to the lowest and highest concentrations normally found in solvent after analysis of commercial samples. For BP and 4MBP the lower concentrations are chosen in line with the recent recommendations of the EFSA for limitation to 0.6 mg kg^{-1} for the sum of BP and 4MBP in paperboard as in plastics.

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

ILC01 2010 was launched in April 2010. Invitation letters were sent to the laboratories on 26th April 2010 (Annex 1). Laboratories were invited to fill in a letter of confirmation of their participation (Annex 2).

The samples were prepared in the end of April 2010. Homogeneity tests for solvent and paperboard were run in the beginning of May. The results from homogeneity

test were accepted as time zero for stability testing

The samples were dispatched to participants on 10th May 2010, together with a letter accompanying the samples (Annex 3), detailed instructions for compilation of the results in electronic format (Annex 5), a format for the compilation of results to be eventually sent in non-electronic format (Annex 6) and electronic files where the result should be inserted.

The participants were asked to fill in a letter of confirmation of the receipt of the samples (Annex 4)

The deadline for reporting has been set to 1st July 2010.

5. Test material

5.1 Preparation

Preparation of the test material was done by the EURL-FCM.

After spiking and homogenization the solvent was dispensed in screw cap glass vials of approximately 40 ml capacity.

Paperboard samples were A4 format but containing glossy and non-glossy parts. Thorough homogeneity study showed that only glossy part contained PI at homogeneous levels, while the non-glossy part was not homogeneous at all. Therefore we cut the samples with dimensions 5 cm × 5 cm or 5 cm x 10 cm only from the glossy parts of the A4 foils and re-ran the homogeneity test again at the level of a portion of that surface (5 cm x 1 cm)

Exercise	Name	Sample
ILC 01 2010	**MIX1**	**1 screw cap vial of ACN (30ml) spiked at level 1**
	MIX2	**1 screw cap vial of ACN (30ml) spiked at level 2**
	P&B1	**10 pieces of 5 cm × 5 cm from paperboard 1 (for BP)**
	P&B2	**10 pieces of 10 cm × 5 cm from paperboard 2 (for 4MBP)**

Spiked concentration levels in MIX1 and MIX2 shown in the following table:

	MIX1 spiked concentrations, mg/kg	MIX2 spiked concentrations, mg/kg
BP	93.6	0.546
4MBP	0.669	87.5
ITX	32.3	1.22
PBZ	54.9	7.27
4HBP	3.29	68.4

Each participant received additionally to the samples one kit of standard substances (Table 1). First four substances were delivered as powder whereas ITX was delivered as a solution (c (ITX in ACN) = 399.6 mg/L).

Table1: Sample kit for ILC01 2010

	Standard	CAS	Provider	Purity
BP	benzophenone	119-61-9	Sigma-Aldrich	99.9%
4MBP	4-methylbenzophenone	134-84-9	Aldrich	99.9%
4HBP	4-hydroxybenzophenone	1137-42-4	Aldrich	99.2%
PBZ	4-benzoylbiphenyl	2128-93-0	Aldrich	99.8%
ITX	2-isopropylthioxanthone	5495-84-1	Sigma-Aldrich	99.8%

5.2 Homogeneity assessment

The samples were tested for homogeneity by the EURL laboratory.

Ten randomly selected test specimens for each sample (MIX1, MIX2, P&B1 and P&B2 at level 0.05 and 0.25 dm^2) were analyzed in duplicate for all 5 PI.

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 7.

All test materials has shown sufficient homogeneity for the target standard deviation shown in Annex 7.

5.3 Stability test

Randomly selected specimens for each sample (MIX1, MIX2, P&B1 and P&B2) were stored at 3 different temperature conditions (+4ºC, room temperature, +40ºC). The test samples were monitored for stability by the EURL laboratory, by mean of PI determination, from the date of preparation over the period of 4 months long after the data of closing the ILC02. The samples were analysed in duplicate altogether seven times over the given time frame.

Stability was evaluated as described in ISO GUIDE 35:2006 [16].

The evaluation of data was carried out by performing a linear regression on the determined concentrations of the measurand (mean values) vs time. For a stable material it is expected that the intercept is (within uncertainty) equal to the assigned value, whereas the slope does not differ significantly from zero.

Being the linear regression equation:

$$Y_{(\text{BPA conc, mg/kg})} = b_0 + b_1 X_{(\text{time, weeks})}$$

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

respected;

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

where

b1	is the slope obtained from the linear regression,
$t_{0.95,n-2}$	is the Student's t-factor for n-2 degrees of freedom and p = 0.95 (95% level of confidence) and
$s(b_1)$	is the uncertainty associated with the slope.

This can be calculated as follows:

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

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

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

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

The results together with their statistical evaluation are given in Annex 8.

It should be pointed out that no significant trend was observed for the test samples at all temperature conditions (4°C, RT and +40°C) for the time of the ILC. It could be concluded as well that the environment conditions of sample storage didn't influence the stability of the samples within two months after their preparation.

5.4 Distribution

The sample kits were dispatched to the participants by the EURL-FCM on 10 May 2010. Each participant received a box containing:

a) the test materials – 2 solvent mix and 2 paperboards;

b) the reference substances - according to table 1;

c) an accompanying letter with instructions on sample handling (Annex 3);

d) instructions to the participant for reporting (Annex 5);

e) a form that had to be sent back after receipt of the sample to confirm its arrival (cf. Annex 4) and

f) a form for reporting the result in non-electronic format (Annex 6)

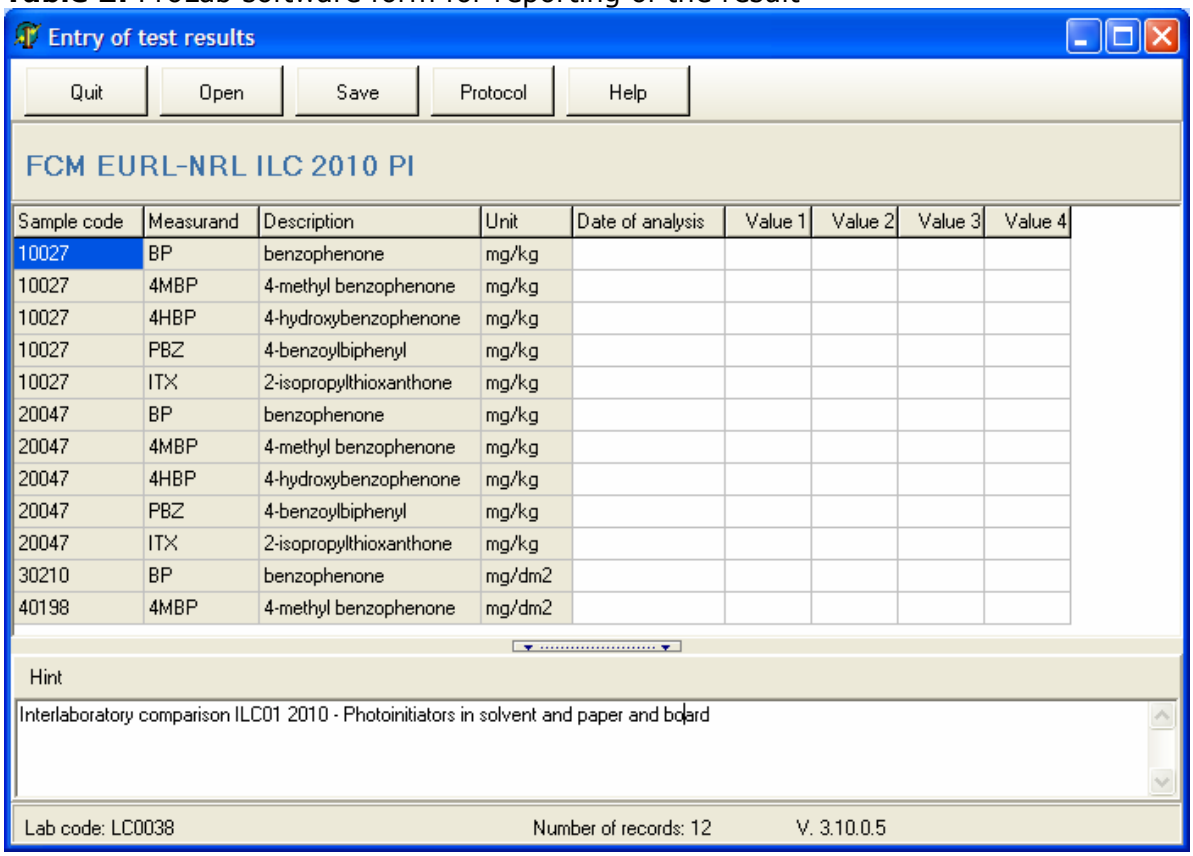
6. Instructions to participants

Concrete instructions were given to all participants in a letter that accompanied the samples (Annex 5).

Laboratories were asked to perform four replicate 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 (Table 2).

Table 2: ProLab software form for reporting of the result



7. Assigned values and their uncertainties

As described earlier, the test materials used for this exercise were two solvent mixtures prepared in the laboratory and two types of paperboards coming from industrial source for the proficiency exercise.

The assigned value and its uncertainty were obtained after applying the robust statistics to the participant's test results.

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 solvent samples of this ILC test the target standard deviation was set to the calculated by Horwitz formula.

However, for the photo-initiators in paperboard, 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 7).

9. Evaluation of results

9.1 General observations

There were thirty six participants from twenty-six countries to whom samples are dispatched. Participants were the 25 NRL laboratories (two NRLs for France, NRL Malta represented by NRL UK, Sweden and Romania without NRL represented) from the network for FCM and some national control laboratories from Member States especially Germany, Spain, France, Switzerland as a guests. They all received the samples. The ILC was closed permanently at 8th of July for statistical interpretation.

Thirty-two laboratories submitted their results. As requested, most of the laboratories reported four measurement replicate results for any analyte-material combination.

From the EURL-NRL network 22 laboratories out of 25 reported results. 3 NRL laboratories did not send any results.

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 (DIN 38402 A 45) as

one of the most robust, necessary especially when there is presence of many outliers.

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 in accordance with ISO 13528[1] and the International Harmonised Protocol[10]

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

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

The 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

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 3 (1-12) together with the mean values and z-scores.

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.

Z -scores assessed the laboratory performance against the target standard deviation, preliminary determined for the ILC. As mentioned before target standard deviation for paperboard was set to 15% and those for solvent mix – to the calculated by Horwitz equation.

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.

Table 3.a: Laboratories' raw test results, their mean values and corresponding z-score

FCM EURL-NRL ILC 2010 PI

Test results

Sample:	**solvent mix 1**	**Rel. target s.d.:**	**8.12% (Horwitz function)**
Measurand:	**benzophenone**	**Rel. reproducibility s.d.:**	**7.29%**
Method:	**DIN 38402 A45**	**Rel. repeatability s.d.:**	**1.11%**
Assigned value:	**90.3 mg/kg (Empirical value)**	**Limits of tolerance:**	**75.6 - 105.0 mg/kg (|Z-Score| < 2.**

Laboratory code	M 1	M 2	M 3	M 4	M	Z score
LC0002	95.7	95.3	94.3	94.9	95.1	0.6
LC0003	69.9	71.7	69.9	69.4	70.2	-2.7
LC0005	85.4	89.6	88.0	87.5	87.6	-0.4
LC0006	75.8	80.4	77.1	77.8	77.8	-1.7
LC0010	86.8	87.2	87.7	88.4	87.5	-0.4
LC0011	98.4	93.3	92.3	90.4	93.6	0.4
LC0012	93.3	92.3	95.4	95.4	94.1	0.5
LC0013	82.0	82.1	83.0	86.9	83.5	-0.9
LC0017	90.7	87.4	97.0	89.1	91.0	0.1
LC0018	88.8	88.9	88.9	88.6	88.8	-0.2
LC0020	96.1	96.1	96.2	96.1	96.1	0.8
LC0021	91.9	92.6	92.5	92.7	92.4	0.3
LC0023	79.8	74.1	81.7	73.8	77.4	-1.8
LC0025	92.0	90.7	94.8	92.5	92.5	0.3
LC0027	94.3	94.8	96.3	96.0	95.4	0.7
LC0028	93.0	94.0	97.0	97.0	95.3	0.7
LC0029	94.4	94.1	94.6	94.3	94.4	0.6
LC0031	93.7	91.8	93.2	92.3	92.7	0.3
LC0033	70.1	67.1	68.7	68.6	68.6	-3.0
LC0034						
LC0035	87.2	87.0	87.0	86.8	87.0	-0.4
LC0036	80.8	82.0	80.6		81.1	-1.2
LC0037	100.4	99.4	101.4	98.8	100.0	1.3
LC0038	94.1	94.0	94.0		94.1	0.5
LC0040	87.2	87.1	86.6	86.4	86.8	-0.5
LC0041	93.5	93.6	93.6	93.6	93.6	0.4
LC0044	91.7	90.8	92.4	90.3	91.3	0.1
LC0045	6.3	6.0	6.2	6.0	6.1	-11.5
LC0047	58.9	58.7	60.9	61.1	59.9	-4.1
LC0049	94.1	93.9	94.3	94.1	94.1	0.5
LC0050	95.8	96.1			95.9	0.8
LC0051	98.0	96.9	95.0	95.8	96.4	0.8
LC0053	72.3	65.7	73.6	72.0	70.9	-2.6
LC0055	94.0	94.8	93.6	91.6	93.5	0.4
LC0056	91.9	89.1	90.2	93.3	91.1	0.1

Table 3.b: Laboratories' raw test results, their mean values and corresponding z-score

FCM EURL-NRL ILC 2010 PI

Test results

Sample:	**solvent mix 1**	**Rel. target s.d.:**	**16.87% (Horwitz function)**
Measurand:	**4-methyl benzophenone**	**Rel. reproducibility s.d.:**	**19.21%**
Method:	**DIN 38402 A45**	**Rel. repeatability s.d.:**	**2.29%**
Assigned value:	**0.702 mg/kg (Empirical value)**	**Limits of tolerance:**	**0.460 - 0.940 mg/kg (|Z-Score| <**

Laboratory code	M 1	M 2	M 3	M 4	M	Z score
LC0002	0.760	0.770	0.770	0.780	0.770	0.577
LC0003	0.570	0.540	0.590	0.500	0.550	-1.281
LC0005	0.709	0.684	0.742	0.709	0.711	0.079
LC0006	0.660	0.560	0.610	0.610	0.610	-0.774
LC0010	0.601	0.625	0.633	0.636	0.624	-0.659
LC0011	0.910	0.809	0.784	0.909	0.853	1.278
LC0012	0.700	0.690	0.690	0.690	0.692	-0.078
LC0013	0.740	0.750	0.790	0.760	0.760	0.492
LC0017	0.780	0.750	0.880	0.960	0.843	1.189
LC0018	0.580	0.580	0.580	0.590	0.583	-1.007
LC0020	0.818	0.844	0.834	0.874	0.843	1.189
LC0021	0.700	0.500	0.700	0.700	0.650	-0.437
LC0023	0.550	0.540	0.530	0.550	0.543	-1.345
LC0025	0.741	0.732	0.764	0.743	0.745	0.366
LC0027	0.850	0.910	0.880	0.950	0.898	1.654
LC0028	<0.500	<0.500	<0.500	<0.500		
LC0029	0.610	0.610	0.610	0.610	0.610	-0.774
LC0031	0.890	0.890	0.870	0.870	0.880	1.506
LC0033	0.000	0.000	0.000	0.000	0.000	-5.927
LC0034						
LC0035	0.700	0.690	0.700	0.700	0.698	-0.035
LC0036	0.687	0.678	0.720		0.695	-0.057
LC0037	2.080	2.080	1.760	2.090	2.003	10.987
LC0038	0.691	0.691	0.688		0.690	-0.099
LC0040	0.490	0.520	0.490	0.490	0.498	-1.725
LC0041	0.673	0.673	0.673	0.672	0.673	-0.245
LC0044						
LC0045	0.670	0.750	0.790	0.760	0.743	0.345
LC0047	0.220	0.200	0.220	0.220	0.215	-4.111
LC0049	0.750	0.750	0.750	0.750	0.750	0.408
LC0050	0.700	0.710			0.705	0.028
LC0051	0.780	0.770	0.780	0.780	0.778	0.640
LC0053						
LC0055	0.670	0.690	0.670	0.670	0.675	-0.225
LC0056	0.693	0.671	0.708	0.729	0.700	-0.015

Table 3.c: Laboratories' raw test results, their mean values and corresponding z-score

FCM EURL-NRL ILC 2010 PI

Test results

Sample:	**solvent mix 1**	**Rel. target s.d.:**	**13.47% (Horwitz function)**
Measurand:	**4-hydroxybenzophenone**	**Rel. reproducibility s.d.:**	**19.17%**
Method:	**DIN 38402 A45**	**Rel. repeatability s.d.:**	**1.45%**
Assigned value:	**3.13 mg/kg (Empirical value)**	**Limits of tolerance:**	**2.29 - 3.97 mg/kg (|Z-Score| < 2.**

Laboratory code	M 1	M 2	M 3	M 4	M	Z score
LC0002	0.00	0.00	0.00	0.00	0.00	-7.42
LC0003	2.20	2.23	2.24	2.28	2.24	-2.12
LC0005	2.82	2.72	2.86	2.86	2.81	-0.75
LC0006	3.47	2.85	3.17	3.16	3.16	0.08
LC0010	2.92	2.94	2.90	2.98	2.94	-0.46
LC0011	2.96	2.97	2.87	3.20	3.00	-0.32
LC0012	3.25	3.26	3.37	3.32	3.30	0.40
LC0013	3.29	3.19	3.16	2.99	3.16	0.06
LC0017	3.35	3.30	3.39	3.35	3.35	0.51
LC0018	2.27	2.27	2.27	2.28	2.27	-2.03
LC0020	3.47	3.48	3.50	3.50	3.49	0.85
LC0021	3.00	3.00	3.10	3.10	3.05	-0.19
LC0023	2.23	2.23	2.31	2.24	2.25	-2.08
LC0025	3.23	3.12	3.19	3.19	3.18	0.11
LC0027	2.94	3.59	3.50	3.52	3.39	0.61
LC0028	<0.50	<0.50	<0.50	<0.50		
LC0029	3.63	3.63	3.61	3.61	3.62	1.16
LC0031	2.88	2.73	3.01	2.89	2.88	-0.60
LC0033	0.00	0.00	0.00	0.00	0.00	-7.42
LC0034						
LC0035	3.26	3.27	3.28	3.27	3.27	0.33
LC0036	2.75	2.70	2.78		2.74	-0.92
LC0037	8.68	8.69	8.74	8.68	8.70	13.20
LC0038	3.15	3.14	3.13		3.14	0.02
LC0040	3.05	3.10	3.06	3.03	3.06	-0.17
LC0041	3.60	3.60	3.60	3.60	3.60	1.11
LC0044						
LC0045	3.83	4.14	4.17	3.93	4.02	2.10
LC0047	4.88	4.89	5.25	5.03	5.01	4.46
LC0049	3.61	3.62	3.60	3.64	3.62	1.16
LC0050	3.25	3.32			3.29	0.37
LC0051	3.14	3.21	3.19	3.22	3.19	0.14
LC0053						
LC0055	1.08	1.26	1.29	1.31	1.24	-4.49
LC0056	3.42	3.31	3.34	3.45	3.38	0.59

Table 3.d: Laboratories' raw test results, their mean values and corresponding z-score

FCM EURL-NRL ILC 2010 PI

Test results

Sample:	**solvent mix 1**	**Rel. target s.d.:**	**8.85% (Horwitz function)**
Measurand:	**4-benzoylbiphenyl**	**Rel. reproducibility s.d.:**	**10.97%**
Method:	**DIN 38402 A45**	**Rel. repeatability s.d.:**	**1.96%**
Assigned value:	**51.0 mg/kg (Empirical value)**	**Limits of tolerance:**	**42.0 - 60.1 mg/kg (|Z-Score| < 2.**

Laboratory code	M 1	M 2	M 3	M 4	M	Z score
LC0002	41.3	42.2	41.2	41.1	41.5	-2.1
LC0003	41.6	41.8	41.3	41.9	41.7	-2.1
LC0005	53.8	54.9	53.1	52.9	53.7	0.6
LC0006	43.6	42.1	45.2	43.6	43.6	-1.6
LC0010	51.9	52.0	52.4	52.7	52.2	0.3
LC0011	55.8	51.8	52.3	51.1	52.8	0.4
LC0012	54.5	53.9	56.0	56.0	55.1	0.9
LC0013	53.3	50.0	47.0	44.0	48.5	-0.6
LC0017	55.6	54.6	53.6	52.9	54.2	0.7
LC0018	51.6	51.6	51.6	51.6	51.6	0.1
LC0020	44.5	44.6	44.5	44.5	44.5	-1.4
LC0021	52.5	52.8	52.6	53.1	52.8	0.4
LC0023	45.2	47.2	43.8	44.2	45.1	-1.3
LC0025	54.0	52.8	55.6	53.9	54.1	0.7
LC0027	56.0	56.3	57.5	57.6	56.9	1.3
LC0028	39.0	38.0	39.0	41.0	39.3	-2.6
LC0029	55.5	55.6	55.6	55.7	55.6	1.0
LC0031	56.8	55.3	54.9	56.5	55.9	1.1
LC0033	34.9	36.5	33.5	34.6	34.9	-3.6
LC0034						
LC0035	56.5	56.3	56.4	56.3	56.4	1.2
LC0036	45.7	45.5	44.2		45.1	-1.3
LC0037	46.8	46.1	52.5	50.9	49.1	-0.4
LC0038	52.9	52.7	52.7		52.8	0.4
LC0040	57.3	59.0	59.6	58.9	58.7	1.7
LC0041	55.5	55.4	55.3	55.4	55.4	1.0
LC0044	56.4	52.5	52.5	54.3	53.9	0.6
LC0045	40.4	39.0	37.2	36.0	38.2	-2.9
LC0047	50.3	52.2	48.8	51.9	50.8	-0.1
LC0049	59.6	59.5	59.6	59.6	59.6	1.9
LC0050	55.8	56.4			56.1	1.1
LC0051	51.8	53.8	53.2	54.0	53.2	0.5
LC0053	21.1	22.9	17.1	18.4	19.9	-6.9
LC0055	48.9	54.6	51.1	50.9	51.4	0.1
LC0056	53.0	51.5	52.1	53.9	52.6	0.3

Table 3.e: Laboratories' raw test results, their mean values and corresponding z-score

FCM EURL-NRL ILC 2010 PI

Test results

Sample: solvent mix 1 — **Rel. target s.d.:** 9.55% (Horwitz function)

Measurand: 2-isopropylthioxanthone — **Rel. reproducibility s.d.:** 11.38%

Method: DIN 38402 A45 — **Rel. repeatability s.d.:** 1.59%

Assigned value: 30.9 mg/kg (Empirical value) — **Limits of tolerance:** 25.0 - 36.8 mg/kg (|Z-Score| < 2.

Laboratory code	M 1	M 2	M 3	M 4	M	Z score
LC0002	24.4	24.0	23.3	23.2	23.7	-2.4
LC0003	31.2	30.4	30.2	30.5	30.6	-0.1
LC0005	30.8	29.8	29.7	29.8	30.0	-0.3
LC0006	34.1	29.4	26.3	29.9	29.9	-0.3
LC0010	29.1	29.2	29.3	29.5	29.3	-0.6
LC0011	31.5	29.7	30.1	29.8	30.3	-0.2
LC0012	32.0	31.7	32.9	33.0	32.4	0.5
LC0013	39.4	34.8	35.5	32.6	35.6	1.6
LC0017	32.8	32.5	31.7	31.4	32.1	0.4
LC0018	32.8	32.8	32.8	32.7	32.8	0.6
LC0020	34.0	33.7	34.0	33.7	33.8	1.0
LC0021	34.6	34.8	34.6	35.0	34.8	1.3
LC0023	26.0	26.6	25.3	25.5	25.9	-1.7
LC0025	31.5	31.0	31.8	31.6	31.5	0.2
LC0027	31.9	32.1	32.7	32.7	32.4	0.5
LC0028	24.0	24.0	24.0	25.0	24.3	-2.3
LC0029	30.8	30.8	31.0	31.0	30.9	0.0
LC0031	32.7	33.0	32.7	33.3	32.9	0.7
LC0033	17.5	17.9	16.0	18.5	17.5	-4.6
LC0034						
LC0035	32.4	32.4	32.3	32.4	32.4	0.5
LC0036	26.9	27.5	27.6		27.3	-1.2
LC0037	25.5	25.4	28.5	28.6	27.0	-1.3
LC0038	32.0	32.0	32.0		32.0	0.4
LC0040	21.8	20.9	21.9	21.2	21.5	-3.2
LC0041	32.2	32.2	21.2	32.2	29.5	-0.5
LC0044	30.5	28.9	27.6	28.5	28.9	-0.7
LC0045	11.1	10.8	10.6	10.2	10.7	-6.9
LC0047	32.9	34.2	33.4	34.6	33.8	1.0
LC0049	31.2	31.2	31.5	31.3	31.3	0.1
LC0050	32.0	32.5			32.3	0.5
LC0051	31.6	32.6	33.5	33.2	32.7	0.6
LC0053	39.5	36.8	38.2	38.5	38.3	2.5
LC0055	35.1	37.7	34.8	34.4	35.5	1.6
LC0056	31.2	30.3	30.6	31.7	30.9	0.0

Table 3.f: Laboratories' raw test results, their mean values and corresponding z-score

FCM EURL-NRL ILC 2010 PI

Test results

Sample: solvent mix 2
Measurand: benzophenone
Method: DIN 38402 A45
Assigned value: 0.526 mg/kg (Empirical value)
Rel. target s.d.: 17.62% (Horwitz function)
Rel. reproducibility s.d.: 18.50%
Rel. repeatability s.d.: 3.74%
Limits of tolerance: 0.340 - 0.710 mg/kg (|Z-Score| <

Laboratory code	M 1	M 2	M 3	M 4	M	Z score
LC0002	0.640	0.660	0.670	0.660	0.658	1.418
LC0003	0.460	0.410	0.440	0.450	0.440	-0.928
LC0005	0.464	0.488	0.515	0.501	0.492	-0.367
LC0006	0.500	0.560	0.530	0.530	0.530	0.043
LC0010	0.505	0.509	0.507	0.508	0.507	-0.204
LC0011	0.755	0.594	0.660	0.706	0.679	1.648
LC0012	0.540	0.540	0.550	0.570	0.550	0.259
LC0013	0.480	0.470	0.490	0.480	0.480	-0.497
LC0017	0.650	0.630	0.690	0.800	0.693	1.796
LC0018	0.550	0.540	0.550	0.550	0.548	0.232
LC0020	0.635	0.606	0.596	0.601	0.609	0.901
LC0021	0.400	0.500	0.400	0.400	0.425	-1.090
LC0023	0.420	0.420	0.410	0.420	0.418	-1.171
LC0025	0.547	0.528	0.544	0.548	0.542	0.170
LC0027	0.410	0.410	0.460	0.480	0.440	-0.928
LC0028	<0.500	<0.500	<0.500	<0.500		
LC0029	0.450	0.450	0.450	0.450	0.450	-0.820
LC0031	0.530	0.540	0.540	0.530	0.535	0.097
LC0033	0.000	0.000	0.000	0.000	0.000	-5.675
LC0034						
LC0035	0.530	0.530	0.530	0.530	0.530	0.043
LC0036	0.506	0.546	0.541		0.531	0.054
LC0037	19.380	19.230	19.050	20.340	19.500	204.701
LC0038	0.537	0.537	0.538		0.537	0.122
LC0040	0.400	0.390	0.360	0.360	0.378	-1.602
LC0041	0.507	0.507	0.506	0.507	0.507	-0.210
LC0044						
LC0045	0.460	0.400	0.370	0.370	0.400	-1.360
LC0047	1.470	1.590	1.530	1.530	1.530	10.831
LC0049	0.570	0.570	0.570	0.570	0.570	0.474
LC0050	0.570	0.550			0.560	0.367
LC0051	0.580	0.560	0.560	0.560	0.565	0.420
LC0053	0.790	0.780	0.750	0.720	0.760	2.524
LC0055	0.480	0.500	0.500	0.500	0.495	-0.335
LC0056	0.503	0.530	0.558	0.573	0.541	0.160

Table 3.g: Laboratories' raw test results, their mean values and corresponding z-score

FCM EURL-NRL ILC 2010 PI

Test results

Sample:	**solvent mix 2**	**Rel. target s.d.:**	**8.18% (Horwitz function)**
Measurand:	**4-methyl benzophenone**	**Rel. reproducibility s.d.:**	**7.84%**
Method:	**DIN 38402 A45**	**Rel. repeatability s.d.:**	**1.02%**
Assigned value:	**85.9 mg/kg (Empirical value)**	**Limits of tolerance:**	**71.8 - 99.9 mg/kg (|Z-Score| < 2.**

Laboratory code	M 1	M 2	M 3	M 4	M	Z score
LC0002	83.6	85.8	83.2	83.5	84.0	-0.3
LC0003	67.6	70.0	68.2	68.2	68.5	-2.5
LC0005	83.7	92.2	86.6	88.1	87.6	0.3
LC0006	99.6	106.1	102.0	102.6	102.6	2.4
LC0010	79.9	80.3	80.7	81.2	80.5	-0.8
LC0011	86.7	83.8	82.2	82.5	83.8	-0.3
LC0012	85.5	85.6	87.3	87.5	86.5	0.1
LC0013	84.0	96.1	85.8	88.3	88.6	0.4
LC0017	89.3	86.1	85.7	85.9	86.8	0.1
LC0018	105.3	105.2	105.2	105.2	105.2	2.8
LC0020	88.8	88.7	88.7	88.6	88.7	0.4
LC0021	82.0	82.7	82.6	82.7	82.5	-0.5
LC0023	58.5	58.6	57.6	60.8	58.9	-3.8
LC0025	85.7	84.9	86.9	85.2	85.7	0.0
LC0027	88.3	87.7	88.0	87.8	88.0	0.3
LC0028	101.0	102.0	100.0	99.0	100.5	2.1
LC0029	88.6	88.6	88.7	88.8	88.7	0.4
LC0031	82.9	87.8	81.9	82.2	83.7	-0.3
LC0033	63.4	64.5	67.6	68.0	65.9	-2.8
LC0034						
LC0035	83.0	83.2	83.6	83.4	83.3	-0.4
LC0036	78.8	79.6	70.5		76.3	-1.4
LC0037	89.9	91.4	93.3	95.9	92.6	1.0
LC0038	85.1	84.9	85.3		85.1	-0.1
LC0040	89.3	90.0	88.0	88.6	89.0	0.4
LC0041	84.6	84.6	84.7	84.7	84.7	-0.2
LC0044	87.4	79.9	91.4	78.2	84.2	-0.2
LC0045	31.2	33.5	32.8	33.1	32.7	-7.6
LC0047	30.3	30.0	28.9	29.1	29.6	-8.0
LC0049	87.6	87.6	87.4	87.5	87.5	0.2
LC0050	88.6	88.2			88.4	0.4
LC0051	86.5	87.1	86.5	87.3	86.8	0.1
LC0053	138.1	131.2	130.3	131.5	132.8	6.7
LC0055	84.0	85.3	84.9	85.2	84.9	-0.1
LC0056	78.7	82.5	82.9	85.6	82.4	-0.5

Table 3.h: Laboratories' raw test results, their mean values and corresponding z-score

FCM EURL-NRL ILC 2010 PI

Test results

Sample: solvent mix 2 — **Rel. target s.d.:** 8.51% (Horwitz function)

Measurand: 4-hydroxybenzophenone — **Rel. reproducibility s.d.:** 9.80%

Method: DIN 38402 A45 — **Rel. repeatability s.d.:** 1.60%

Assigned value: 66.3 mg/kg (Empirical value) — **Limits of tolerance:** 55.0 - 77.6 mg/kg (|Z-Score| < 2.

Laboratory code	M 1	M 2	M 3	M 4	M	Z score
LC0002	0.0	0.0	0.0	0.0	0.0	-11.8
LC0003	53.9	61.4	64.0	64.1	60.9	-1.0
LC0005	67.9	55.5	70.9	69.5	65.9	-0.1
LC0006	86.6	93.9	95.4	92.0	92.0	4.6
LC0010	62.8	63.2	63.6	64.0	63.4	-0.5
LC0011	70.3	62.9	67.6	64.1	66.2	0.0
LC0012	66.9	67.0	68.5	68.6	67.7	0.3
LC0013	61.0	67.0	62.3	62.9	63.3	-0.5
LC0017	68.5	67.4	67.9	68.6	68.1	0.3
LC0018	62.7	62.6	62.7	62.7	62.7	-0.6
LC0020	69.9	69.8	69.9	69.6	69.8	0.6
LC0021	65.5	66.4	66.4	65.9	66.1	0.0
LC0023	52.5	52.7	50.4	52.3	52.0	-2.5
LC0025	66.9	65.8	66.3	66.7	66.4	0.0
LC0027	69.8	69.1	69.6	69.5	69.5	0.6
LC0028	64.0	75.0	79.0	72.0	72.5	1.1
LC0029	69.7	69.9	69.9	70.1	69.9	0.6
LC0031	66.4	64.7	61.9	62.6	63.9	-0.4
LC0033	54.6	53.8	59.2	59.8	56.9	-1.7
LC0034						
LC0035	67.3	67.6	68.0	67.7	67.6	0.2
LC0036	55.9	58.8	54.2		56.3	-1.8
LC0037	49.0	36.6	43.6	41.1	42.5	-4.2
LC0038	64.6	64.3	64.5		64.5	-0.3
LC0040	68.3	71.1	69.3	70.4	69.8	0.6
LC0041	65.2	65.2	65.2	65.2	65.2	-0.2
LC0044	67.3	63.7	69.3	60.1	65.1	-0.2
LC0045	3.9	3.9	6.1	5.1	4.8	-10.9
LC0047	77.5	76.3	78.6	76.5	77.2	1.9
LC0049	73.1	73.2	73.4	73.3	73.3	1.2
LC0050	69.8	70.5			70.1	0.7
LC0051	64.9	65.1	65.5	65.1	65.1	-0.2
LC0053	72.4	72.3	69.8	72.4	71.7	1.0
LC0055	64.7	66.3	68.0	66.4	66.3	0.0
LC0056	66.1	69.5	70.1	72.4	69.5	0.6

Table 3.i: Laboratories' raw test results, their mean values and corresponding z-score

FCM EURL-NRL ILC 2010 PI

Test results

Sample:	**solvent mix 2**	**Rel. target s.d.:**	**11.98% (Horwitz function)**
Measurand:	**4-benzoylbiphenyl**	**Rel. reproducibility s.d.:**	**16.63%**
Method:	**DIN 38402 A45**	**Rel. repeatability s.d.:**	**1.54%**
Assigned value:	**6.82 mg/kg (Empirical value)**	**Limits of tolerance:**	**5.18 - 8.45 mg/kg (|Z-Score| < 2.**

Laboratory code	M 1	M 2	M 3	M 4	M	Z score
LC0002	8.40	8.20	8.10	7.30	8.00	1.45
LC0003	5.38	5.84	5.42	5.73	5.59	-1.50
LC0005	6.46	6.19	6.75	6.52	6.48	-0.41
LC0006	8.16	7.67	7.44	7.76	7.76	1.15
LC0010	7.05	6.93	7.24	7.15	7.09	0.34
LC0011	7.01	6.89	6.74	6.63	6.82	0.00
LC0012	7.19	7.18	7.32	7.33	7.26	0.53
LC0013	6.09	6.54	6.06	6.22	6.23	-0.72
LC0017	7.44	7.68	7.27	7.41	7.45	0.77
LC0018	6.18	6.17	6.18	6.18	6.18	-0.78
LC0020	5.56	5.58	5.48	5.48	5.53	-1.58
LC0021	7.00	7.10	7.00	7.10	7.05	0.28
LC0023	5.55	5.58	5.61	5.57	5.58	-1.52
LC0025	7.09	6.85	7.22	7.07	7.06	0.29
LC0027	7.34	7.34	7.39	7.49	7.39	0.70
LC0028	3.80	3.70	3.80	3.80	3.78	-3.72
LC0029	7.38	7.39	7.39	7.39	7.39	0.70
LC0031	7.32	7.17	7.13	7.22	7.21	0.48
LC0033	6.45	4.55	6.36	6.46	5.96	-1.06
LC0034						
LC0035	7.91	7.91	7.94	7.92	7.92	1.35
LC0036	4.71	4.93	4.74		4.79	-2.48
LC0037	8.28	8.10	7.60	8.10	8.02	1.47
LC0038	7.08	7.07	7.06		7.07	0.31
LC0040	6.60	6.90	6.70	6.70	6.73	-0.11
LC0041	7.14	7.14	7.14	7.15	7.14	0.40
LC0044	6.30	5.60	7.00	5.60	6.13	-0.85
LC0045	4.47	4.38	4.48	4.87	4.55	-2.78
LC0047	22.56	22.49	22.06	21.91	22.26	18.89
LC0049	7.91	7.92	7.93	7.92	7.92	1.35
LC0050	7.52	7.55			7.54	0.88
LC0051	6.92	7.05	7.05	7.13	7.04	0.27
LC0053	1.97	1.58	1.84	1.97	1.84	-6.09
LC0055	9.72	9.67	9.71	9.67	9.69	3.52
LC0056	6.62	6.94	6.98	7.21	6.94	0.15

Table 3.j: Laboratories' raw test results, their mean values and corresponding z-score

FCM EURL-NRL ILC 2010 PI

Test results

Sample:	**solvent mix 2**	**Rel. target s.d.:**	**15.67% (Horwitz function)**
Measurand:	**2-isopropylthioxanthone**	**Rel. reproducibility s.d.:**	**16.20%**
Method:	**DIN 38402 A45**	**Rel. repeatability s.d.:**	**2.00%**
Assigned value:	**1.15 mg/kg (Empirical value)**	**Limits of tolerance:**	**0.79 - 1.51 mg/kg (|Z-Score| < 2.**

Laboratory code	M 1	M 2	M 3	M 4	M	Z score
LC0002	1.80	1.80	1.90	1.80	1.83	3.75
LC0003	1.08	1.11	1.15	1.11	1.11	-0.20
LC0005	1.13	1.18	1.20	1.16	1.17	0.09
LC0006	1.60	1.79	1.70	1.70	1.70	3.05
LC0010	1.12	1.11	1.12	1.13	1.12	-0.17
LC0011	1.47	1.29	1.27	1.33	1.34	1.05
LC0012	1.21	1.22	1.02	1.24	1.17	0.13
LC0013	1.21	1.15	1.21	1.21	1.19	0.25
LC0017	1.24	1.23	1.24	1.24	1.24	0.49
LC0018	0.91	0.92	0.91	0.92	0.92	-1.30
LC0020	1.28	1.29	1.27	1.27	1.28	0.72
LC0021	1.50	1.30	1.20	1.40	1.35	1.12
LC0023	0.90	0.89	0.90	0.90	0.90	-1.40
LC0025	1.17	1.17	1.19	1.18	1.18	0.15
LC0027	1.14	1.14	1.17	1.19	1.16	0.06
LC0028	0.60	0.60	0.60	0.60	0.60	-3.05
LC0029	1.17	1.17	1.17	1.17	1.17	0.12
LC0031	1.22	1.21	1.21	1.21	1.21	0.35
LC0033	0.00	0.00	0.00	0.00	0.00	-6.38
LC0034						
LC0035	1.26	1.26	1.26	1.26	1.26	0.62
LC0036	0.93	0.94	0.91		0.93	-1.23
LC0037	3.38	3.37	3.35	3.41	3.38	12.38
LC0038	1.21	1.21	1.20		1.21	0.32
LC0040	1.26	1.23	1.23	1.21	1.23	0.46
LC0041	1.17	1.17	1.17	1.17	1.17	0.12
LC0044	0.90	0.80	0.50	0.70	0.73	-2.36
LC0045	0.85	0.83	0.79	0.78	0.81	-1.87
LC0047	2.90	3.05	2.90	3.02	2.97	10.10
LC0049	1.23	1.23	1.23	1.23	1.23	0.45
LC0050	1.27	1.22			1.25	0.53
LC0051	1.23	1.26	1.23	1.26	1.25	0.53
LC0053	0.79	0.71	0.75	0.75	0.75	-2.22
LC0055	1.92	1.87	1.92	1.93	1.91	4.23
LC0056	1.11	1.15	1.17	1.21	1.16	0.06

Table 3.k: Laboratories' raw test results, their mean values and corresponding z-score

FCM EURL-NRL ILC 2010 PI

Test results

Sample:	**BP board**	**Rel. target s.d.:**	**12.87% (Horwitz function)**
Measurand:	**benzophenone**	**Rel. reproducibility s.d.:**	**24.48%**
Method:	**DIN 38402 A45**	**Rel. repeatability s.d.:**	**4.08%**
Assigned value:	**4.23 mg/dm2 (Empirical value)**	**Limits of tolerance:**	**3.14 - 5.32 mg/dm2 (|Z-Score| <**

Laboratory code	M 1	M 2	M 3	M 4	M	Z score
LC0002	1.72	1.65	1.66	1.72	1.69	-4.67
LC0003	3.97	3.54	3.88	3.83	3.81	-0.78
LC0005	4.37	4.37	4.30	4.62	4.41	0.33
LC0006	3.36	3.51	3.52	3.46	3.46	-1.41
LC0010	4.09	4.11	4.40	4.42	4.25	0.04
LC0011	4.36		4.46	4.41	4.41	0.33
LC0012	4.74	4.43	4.55	4.33	4.51	0.51
LC0013	3.18	3.61	3.61	3.40	3.45	-1.44
LC0017	4.49	4.51	4.77	4.61	4.60	0.67
LC0018	4.96	4.56	5.39	4.80	4.93	1.28
LC0020	4.41	4.39	4.37	4.30	4.37	0.25
LC0021	4.10	4.10	3.80	3.90	3.97	-0.47
LC0023	141.00	147.00	149.00	150.00	146.75	261.55
LC0025	4.84	4.80	4.95	4.86	4.86	1.16
LC0027	3.99	4.13			4.06	-0.32
LC0028	3.80	4.00	4.00	4.10	3.97	-0.47
LC0029	5.00	4.82	4.99	5.05	4.97	1.34
LC0031	8.30	7.90	6.20	6.40	7.20	5.45
LC0033	0.51	0.55	0.50	0.55	0.53	-6.80
LC0034						
LC0035	4.06	4.17	3.97	4.16	4.09	-0.26
LC0036	4.39	4.70	4.43	4.56	4.52	0.53
LC0037	581.52	506.96	635.44	552.24	569.04	1036.53
LC0038	4.33	4.21	4.31		4.28	0.09
LC0040	4.00	4.24	4.09	4.19	4.13	-0.19
LC0041	4.56	4.75	4.73	4.40	4.61	0.69
LC0044	4.10	4.80	4.60	4.60	4.52	0.54
LC0045	63.20	56.40	50.80	55.20	56.40	95.74
LC0047	2.36	2.29	2.06	2.30	2.25	-3.63
LC0049	5.44	5.41	5.27	5.28	5.35	2.05
LC0050	2.18	2.23			2.21	-3.72
LC0051	4.54	4.33	4.74	4.34	4.49	0.47
LC0053	7.68	7.28	6.90	7.68	7.39	5.79
LC0055	4.17	3.96	4.11	4.15	4.10	-0.25
LC0056	218.15	220.98	202.40	219.11	215.16	387.09

Table 3.I: Laboratories' raw test results, their mean values and corresponding z-score

FCM EURL-NRL ILC 2010 PI

Test results

Sample:	**4MBP board**	**Rel. target s.d.:**	**12.29% (Horwitz function)**
Measurand:	**4-methyl benzophenone**	**Rel. reproducibility s.d.:**	**19.24%**
Method:	**DIN 38402 A45**	**Rel. repeatability s.d.:**	**3.68%**
Assigned value:	**5.78 mg/dm2 (Empirical value)**	**Limits of tolerance:**	**4.36 - 7.20 mg/dm2 (|Z-Score| <**

Laboratory code	M 1	M 2	M 3	M 4	M	Z score
LC0002	1.85	1.76	1.83	1.86	1.83	-5.57
LC0003	5.10	5.07	5.22	5.24	5.16	-0.88
LC0005	6.06	5.98	5.61	5.51	5.79	0.02
LC0006	5.92	6.49	6.78	6.39	6.40	0.87
LC0010	5.30	5.33	5.14	5.27	5.26	-0.73
LC0011	5.87	5.84	5.79	5.68	5.80	0.02
LC0012	5.53	5.93	5.85	5.74	5.76	-0.02
LC0013	6.75	6.65	6.32	6.13	6.46	0.96
LC0017	6.00	6.21	6.34	6.04	6.15	0.52
LC0018	6.58	6.83	6.38	6.82	6.65	1.23
LC0020	5.84	5.82	5.80	5.62	5.77	-0.01
LC0021	4.70	5.30	4.50	4.50	4.75	-1.45
LC0023	162.00	169.00	175.00	169.00	168.75	229.55
LC0025	6.39	6.60	6.29	6.60	6.47	0.97
LC0027	5.73	6.26			6.00	0.30
LC0028	5.20	6.40	6.60	6.10	6.08	0.42
LC0029	6.59	6.41	6.40	6.19	6.40	0.87
LC0031	5.50	5.30	6.30	6.40	5.88	0.14
LC0033	1.39	1.40	1.38	1.39	1.39	-6.18
LC0034						
LC0035	5.38	5.34	5.42	5.67	5.45	-0.46
LC0036	7.30	7.02	6.79	6.80	6.98	1.69
LC0037	624.80	594.20	676.02	617.39	628.10	876.56
LC0038	5.78	5.91	5.78		5.82	0.06
LC0040	6.10	6.47	6.18	6.28	6.26	0.67
LC0041	6.26	6.28	6.47	6.18	6.30	0.73
LC0044	6.60	7.00	6.20	6.20	6.50	1.02
LC0045	471.00	512.00	363.00	412.00	439.50	610.91
LC0047	5.31	5.12	5.10	4.61	5.04	-1.05
LC0049	5.52	5.38	5.35	5.42	5.42	-0.51
LC0050	2.60	2.70			2.65	-4.41
LC0051	5.17	6.07	5.61	5.70	5.64	-0.20
LC0053	10.80	11.70	10.50	11.40	11.10	7.49
LC0055	5.54	5.36	5.47	5.34	5.43	-0.50
LC0056	260.98	279.58	264.90	279.97	271.36	374.08

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

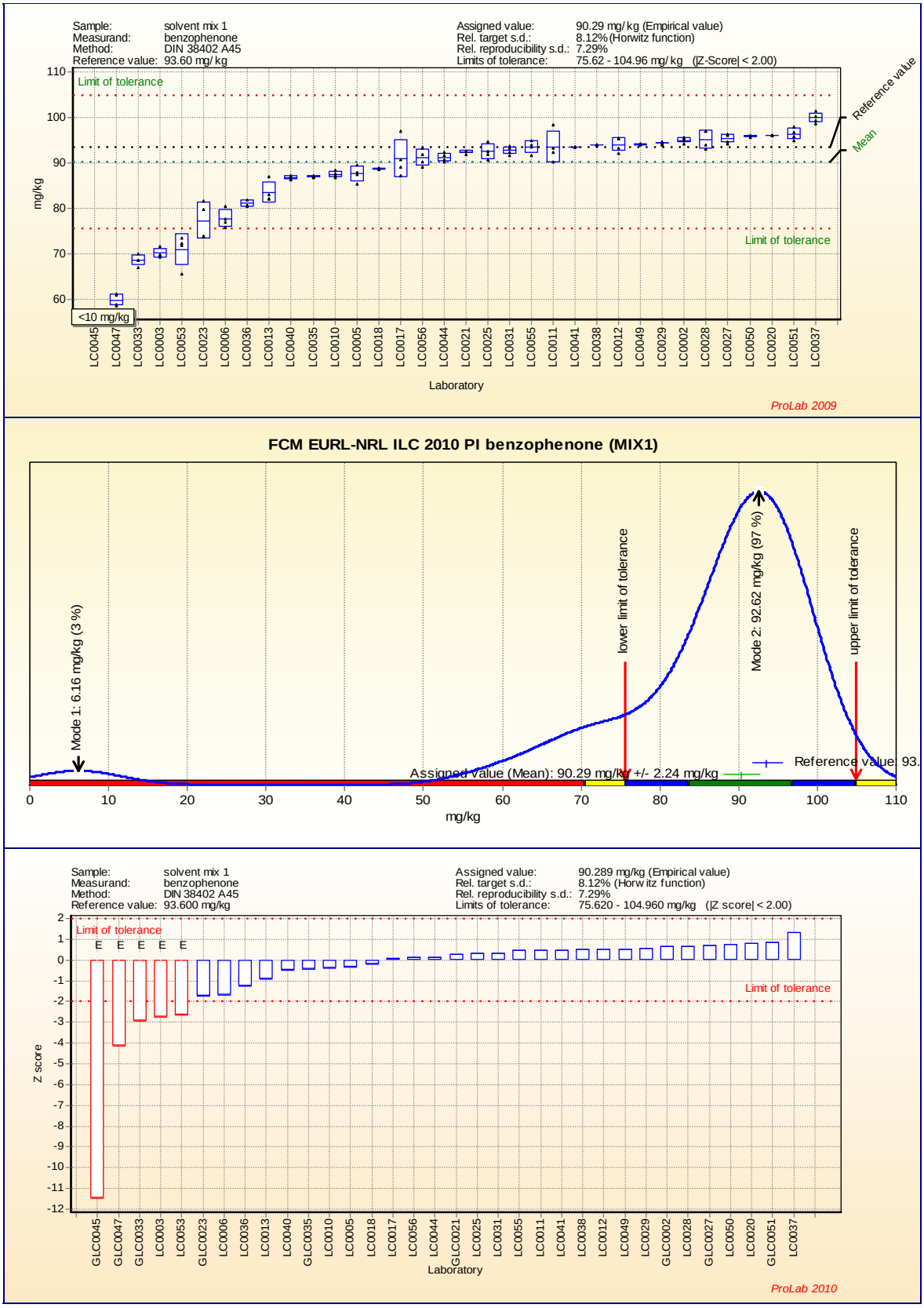


Figure 1-2: Summary graphs of the laboratory's test results with their repeatability SD (a), Kernel Density plot (b) and z'-scores (c)

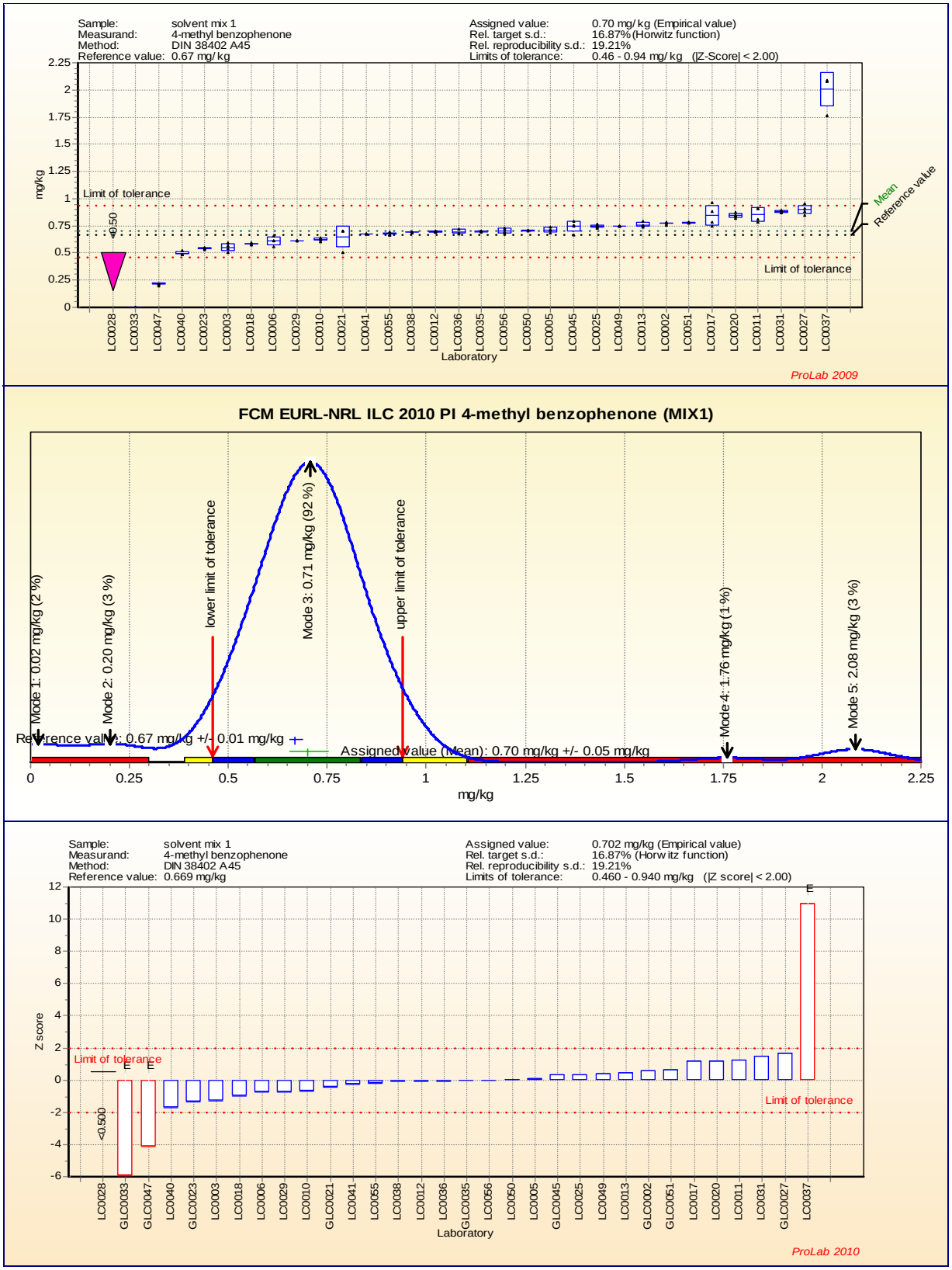


Figure 1-3: Summary graphs of the laboratory's test results with their repeatability SD (a), Kernel Density plot (b) and z'-scores (c)

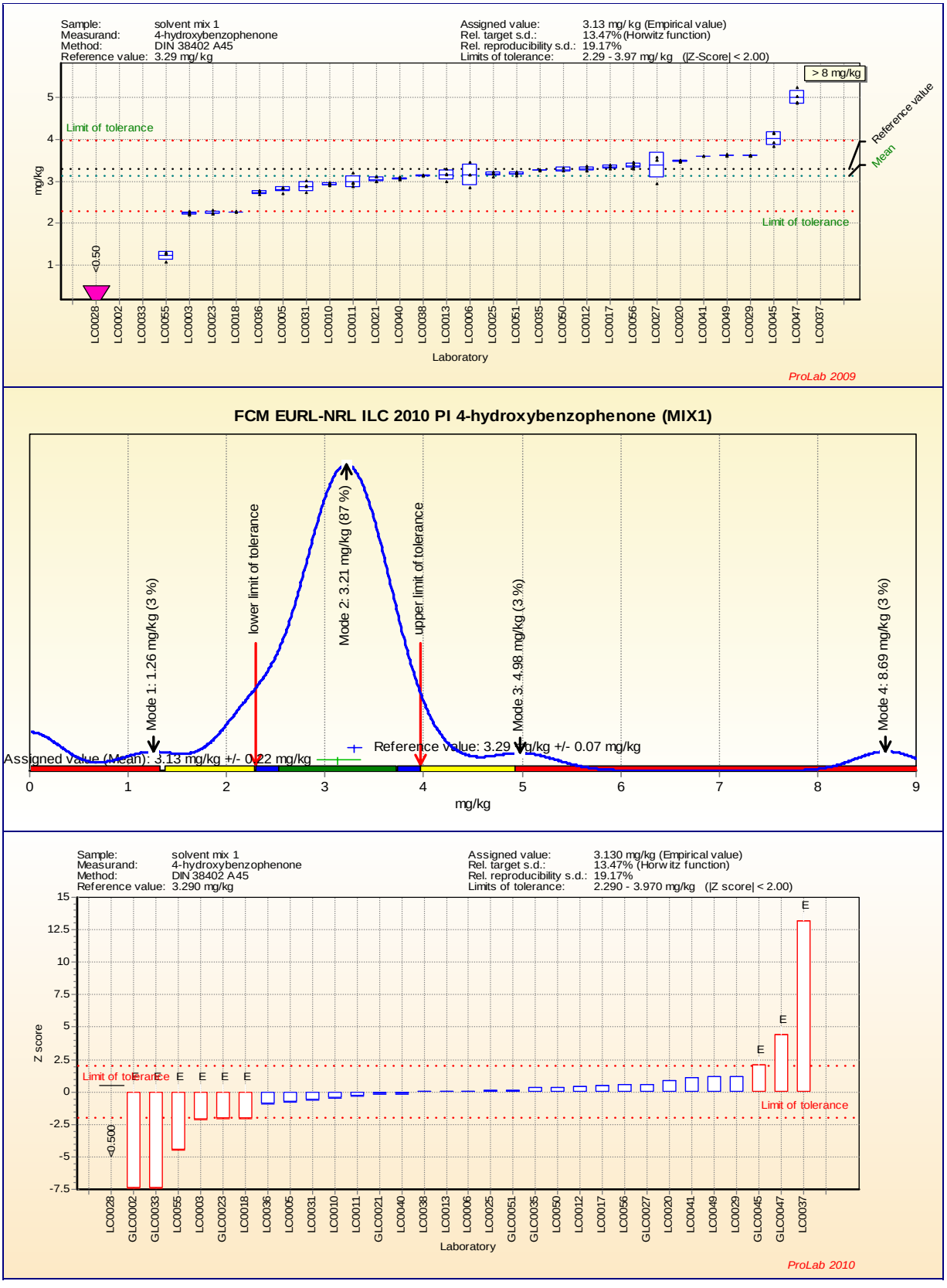


Figure 1-4: Summary graphs of the laboratory's test results with their repeatability SD (a), Kernel Density plot (b) and z-scores (c)

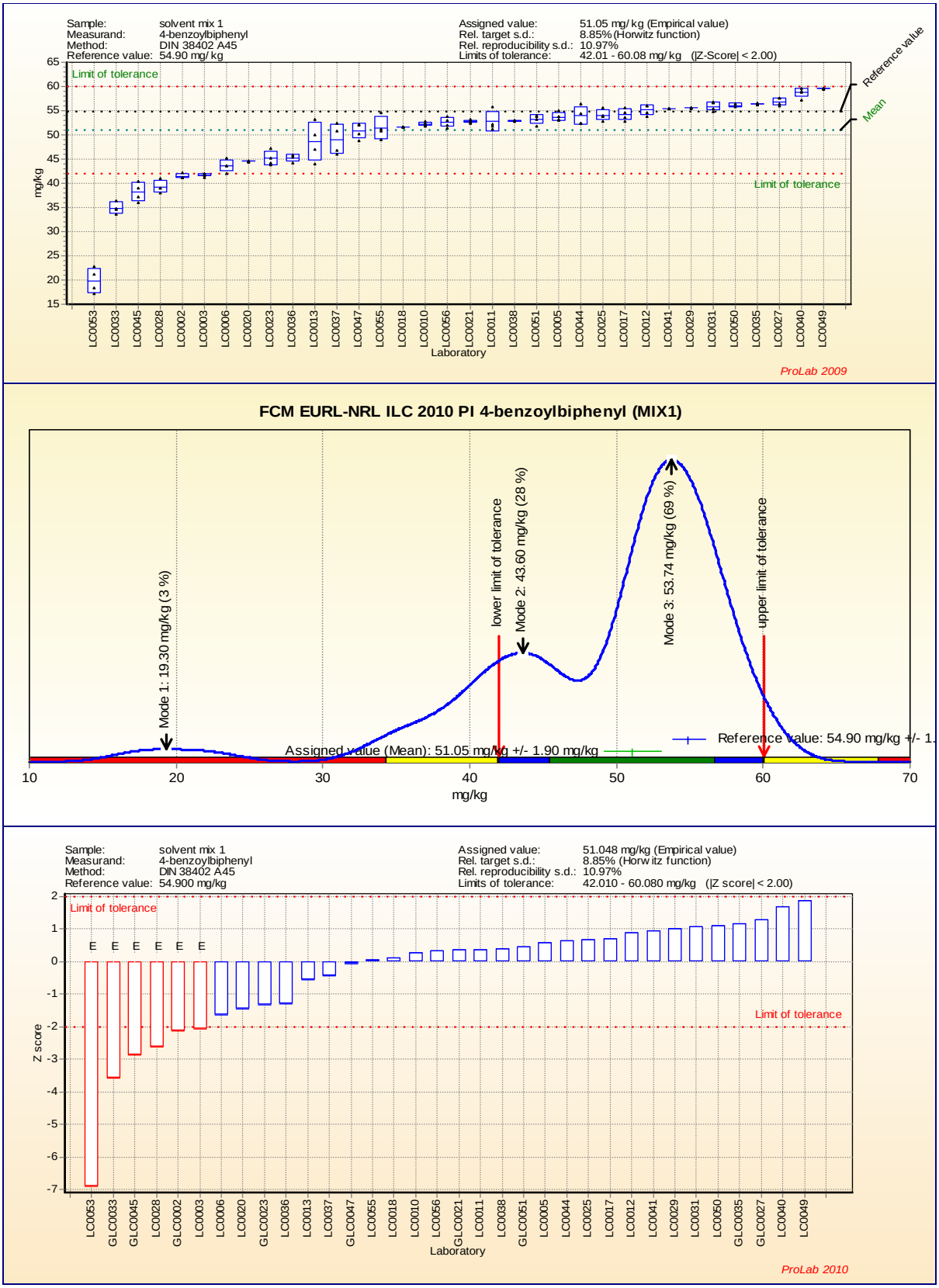


Figure 1-5: Summary graphs of the laboratory's test results with their repeatability SD (a), Kernel Density plot (b) and z-scores (c)

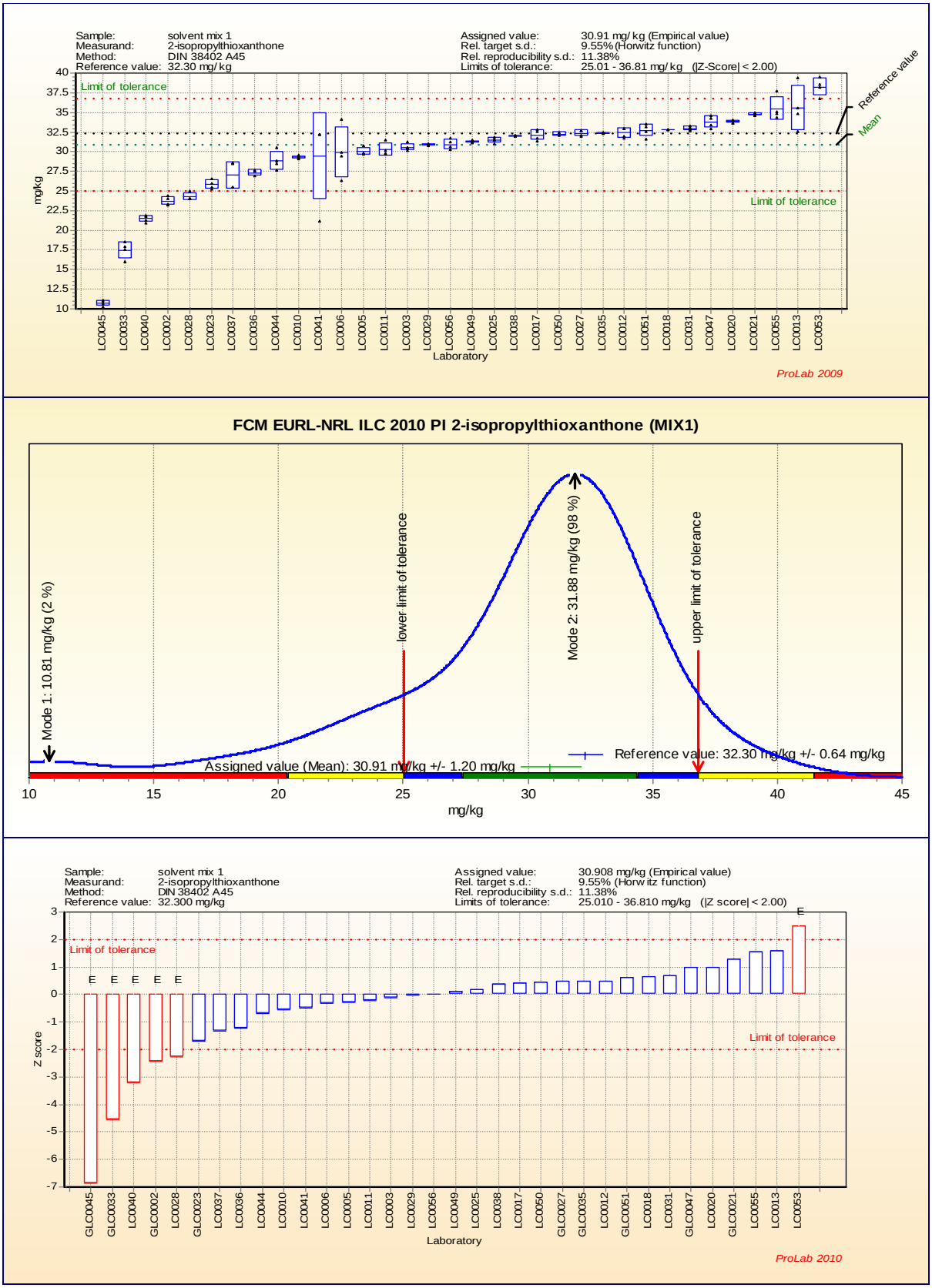


Figure 1-6: Summary graphs of the laboratory's test results with their repeatability SD (a), Kernel Density plot (b) and z-scores (c)

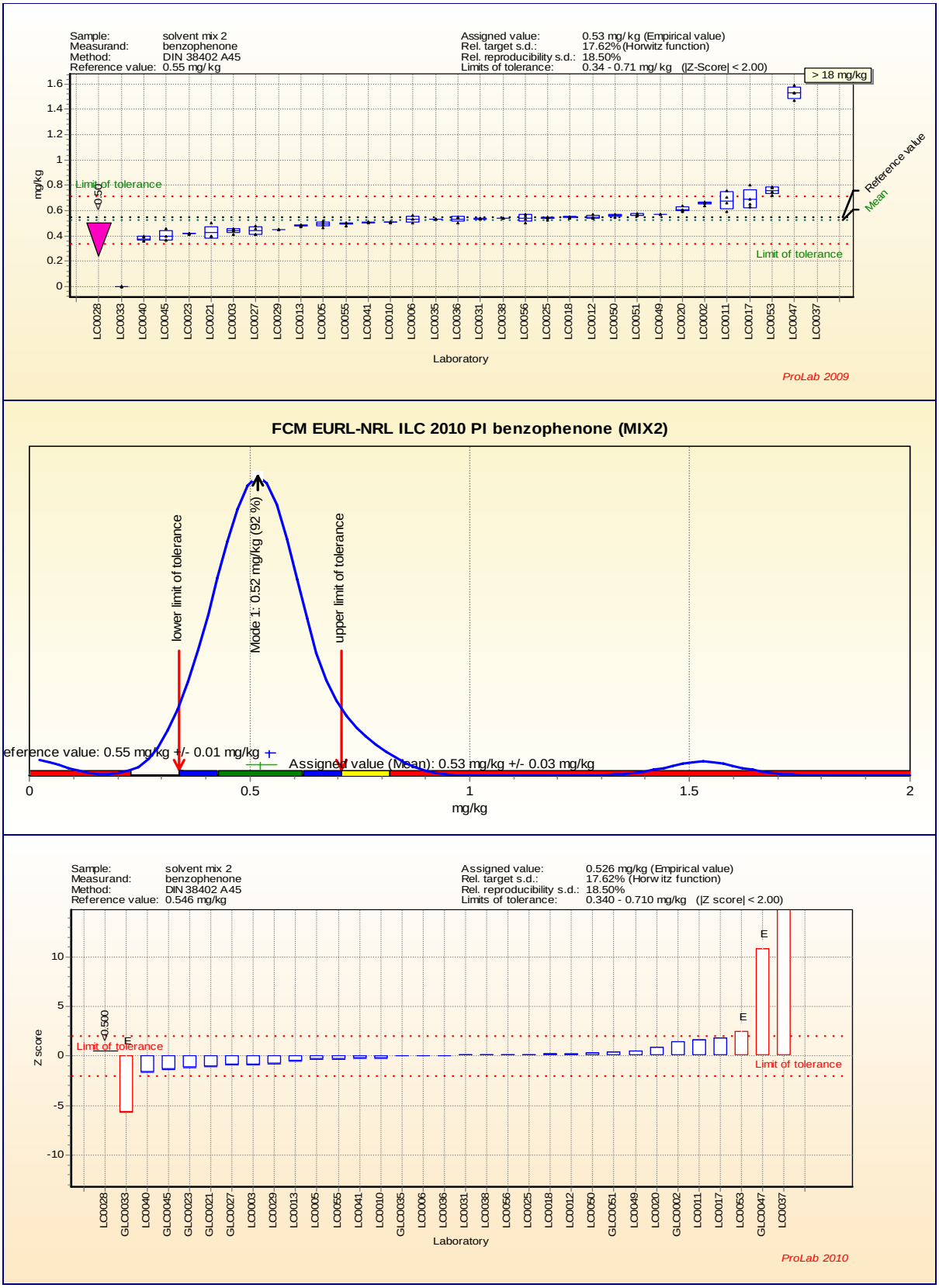


Figure 1-7: Summary graphs of the laboratory's test results with their repeatability SD (a), Kernel Density plot (b) and z-scores (c)

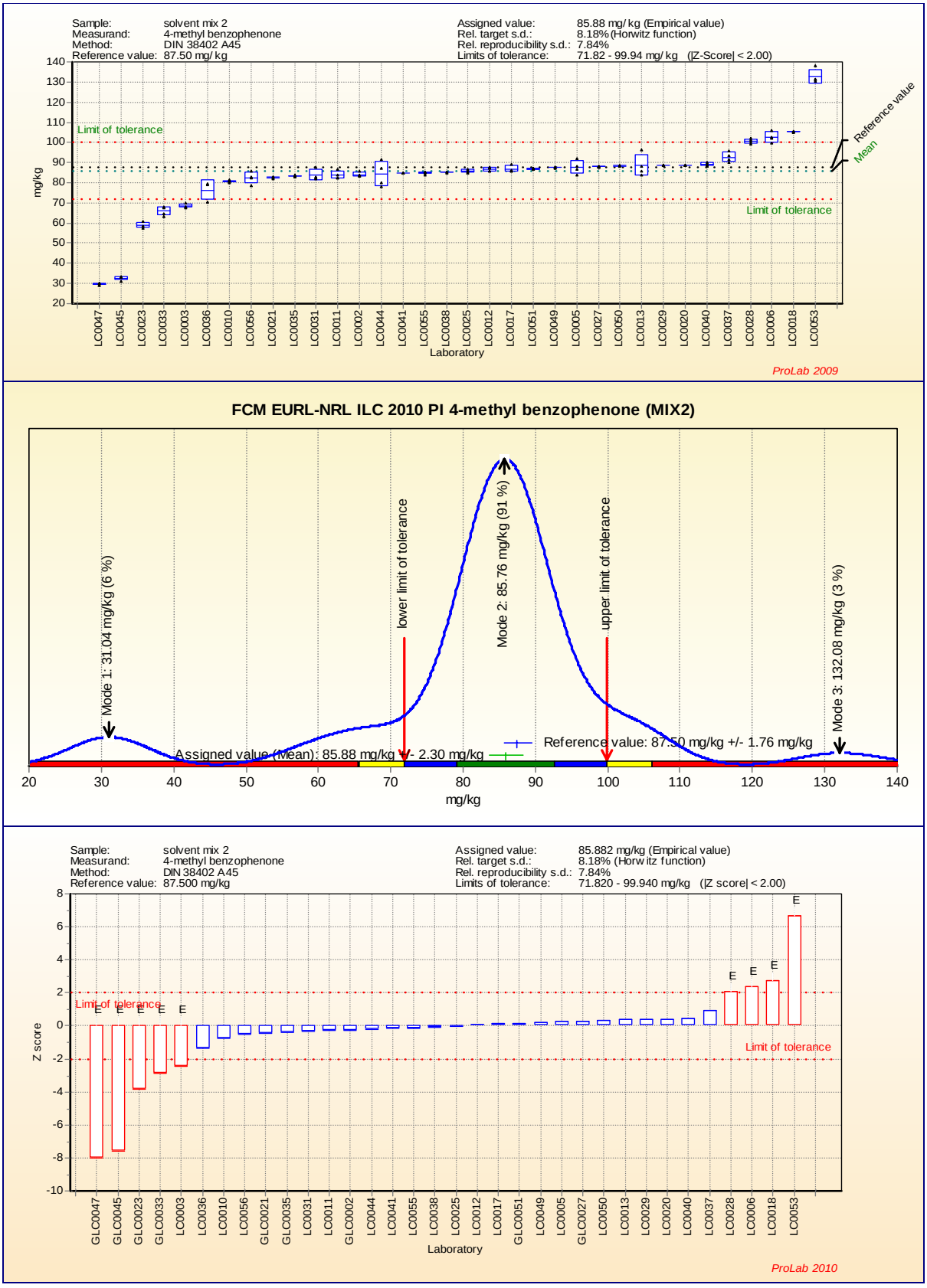


Figure 1-8: Summary graphs of the laboratory's test results with their repeatability SD (a), Kernel Density plot (b) and z-scores (c)

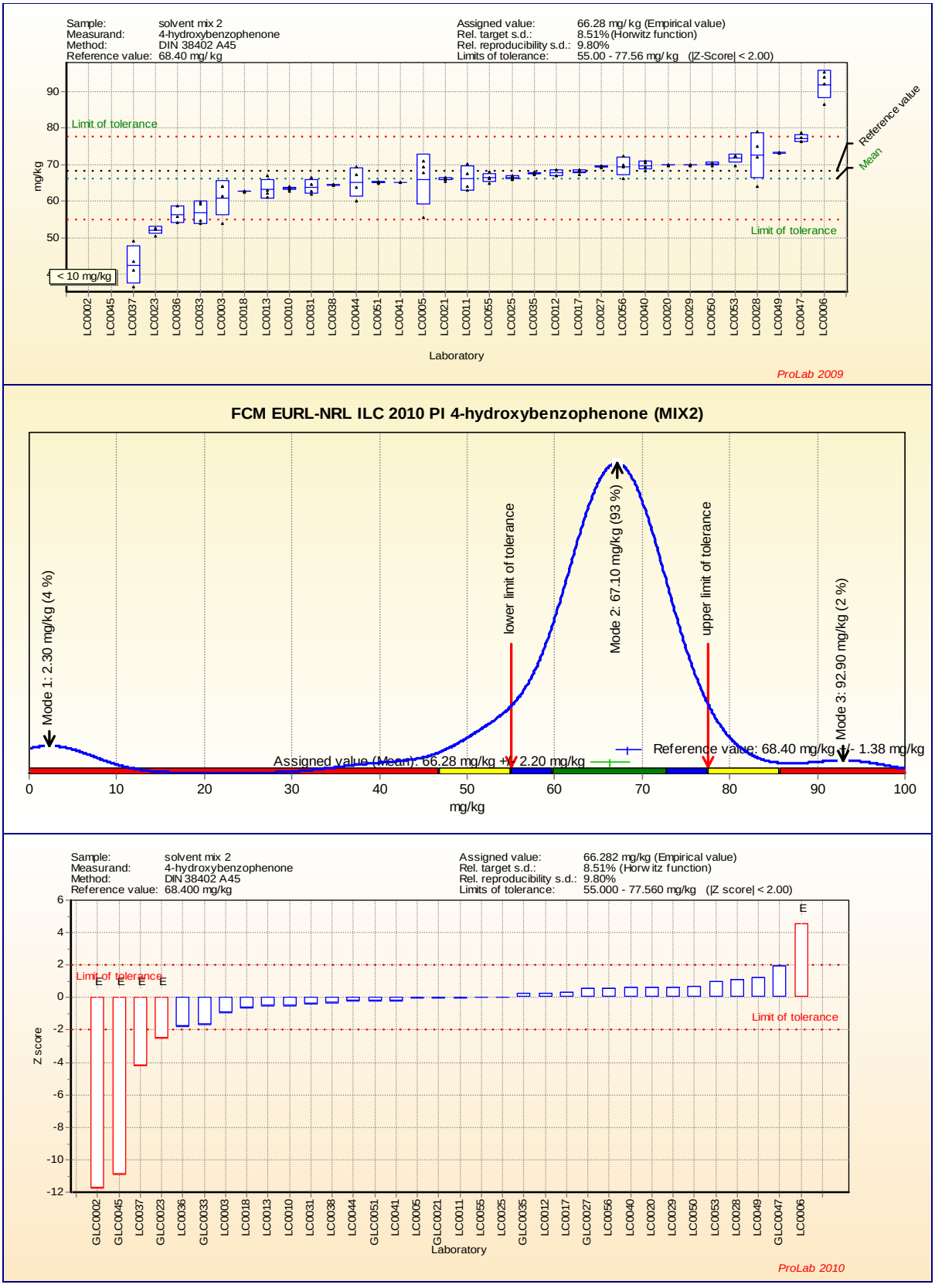


Figure 1-9: Summary graphs of the laboratory's test results with their repeatability SD (a), Kernel Density plot (b) and z-scores (c)

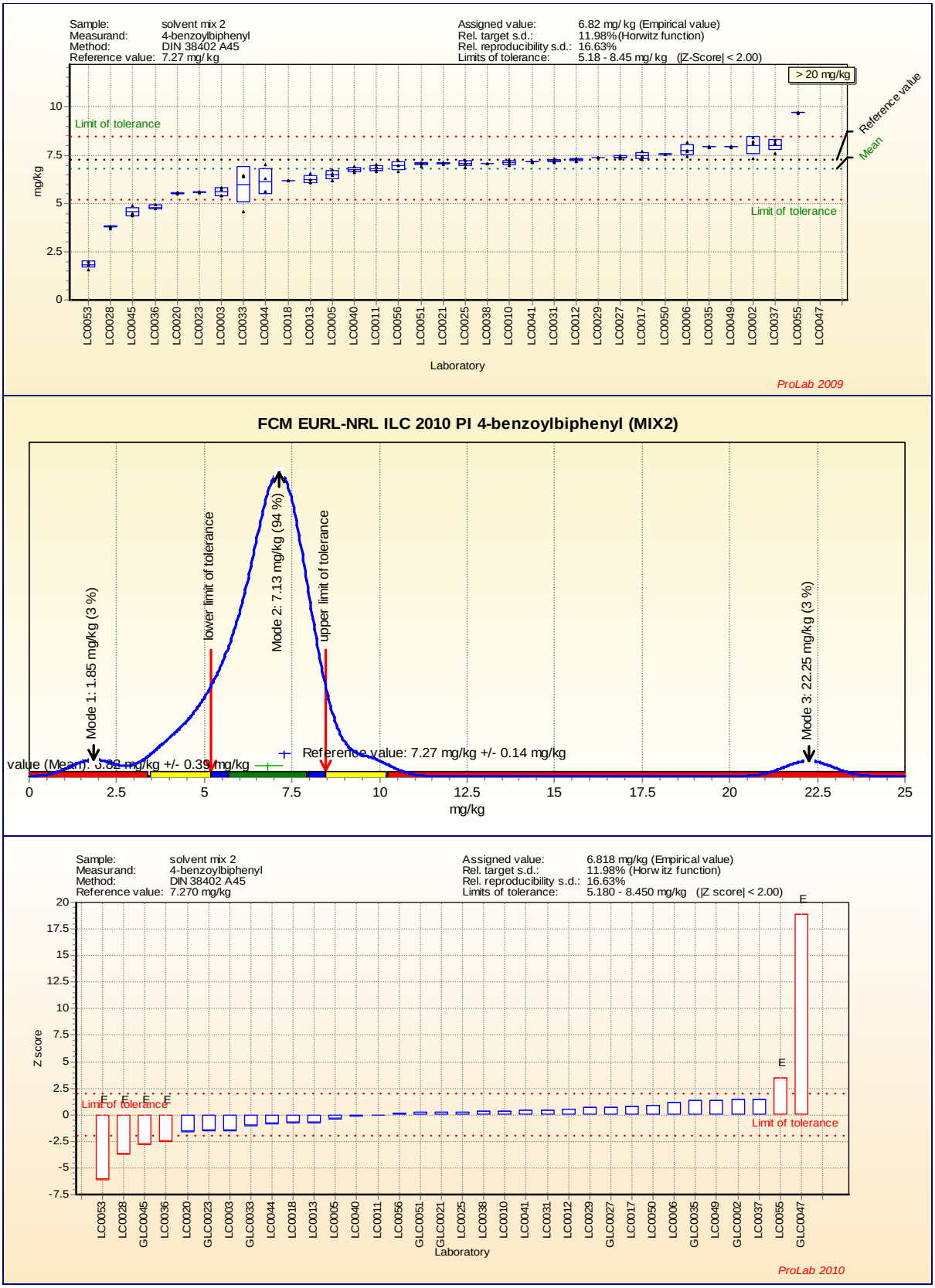


Figure 1-10: Summary graphs of the laboratory's test results with their repeatability SD (a), Kernel Density plot (b) and z-scores (c)

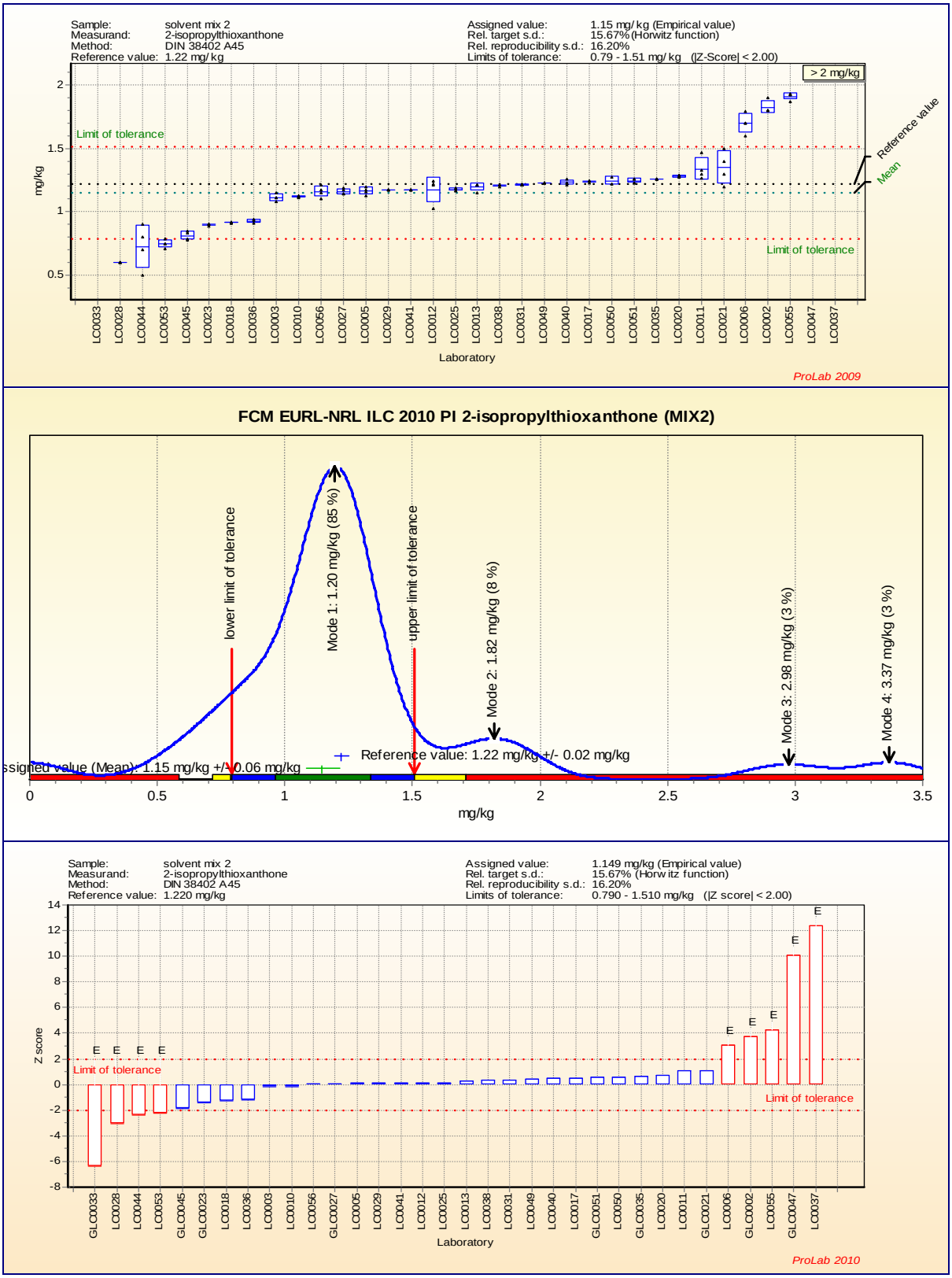


Figure 1-11: Summary graphs of the laboratory's test results with their repeatability SD (a), Kernel Density plot (b) and z-scores (c)

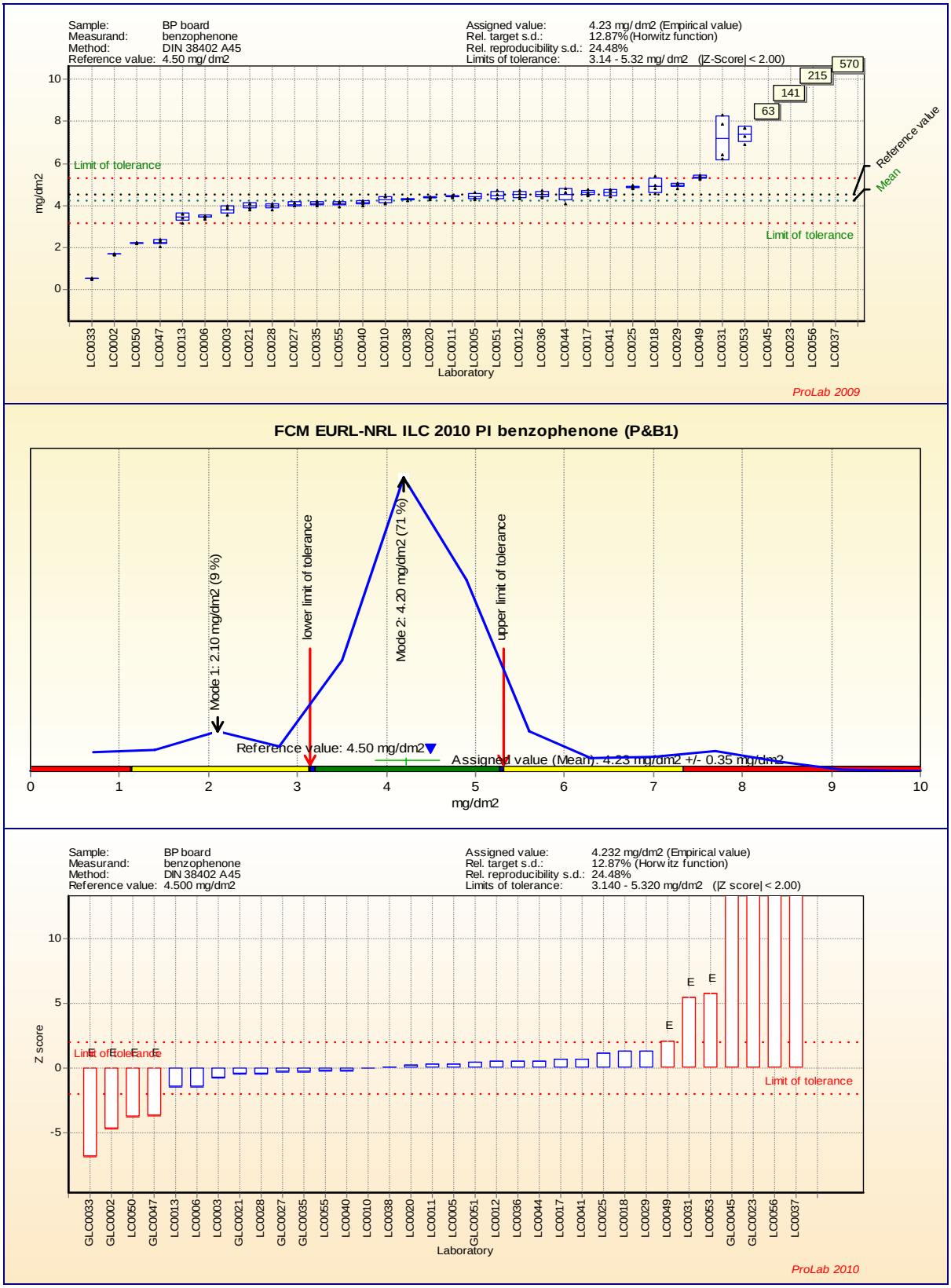


Figure 1-12: Summary graphs of the laboratory's test results with their repeatability SD (a), Kernel Density plot (b) and z-scores (c)

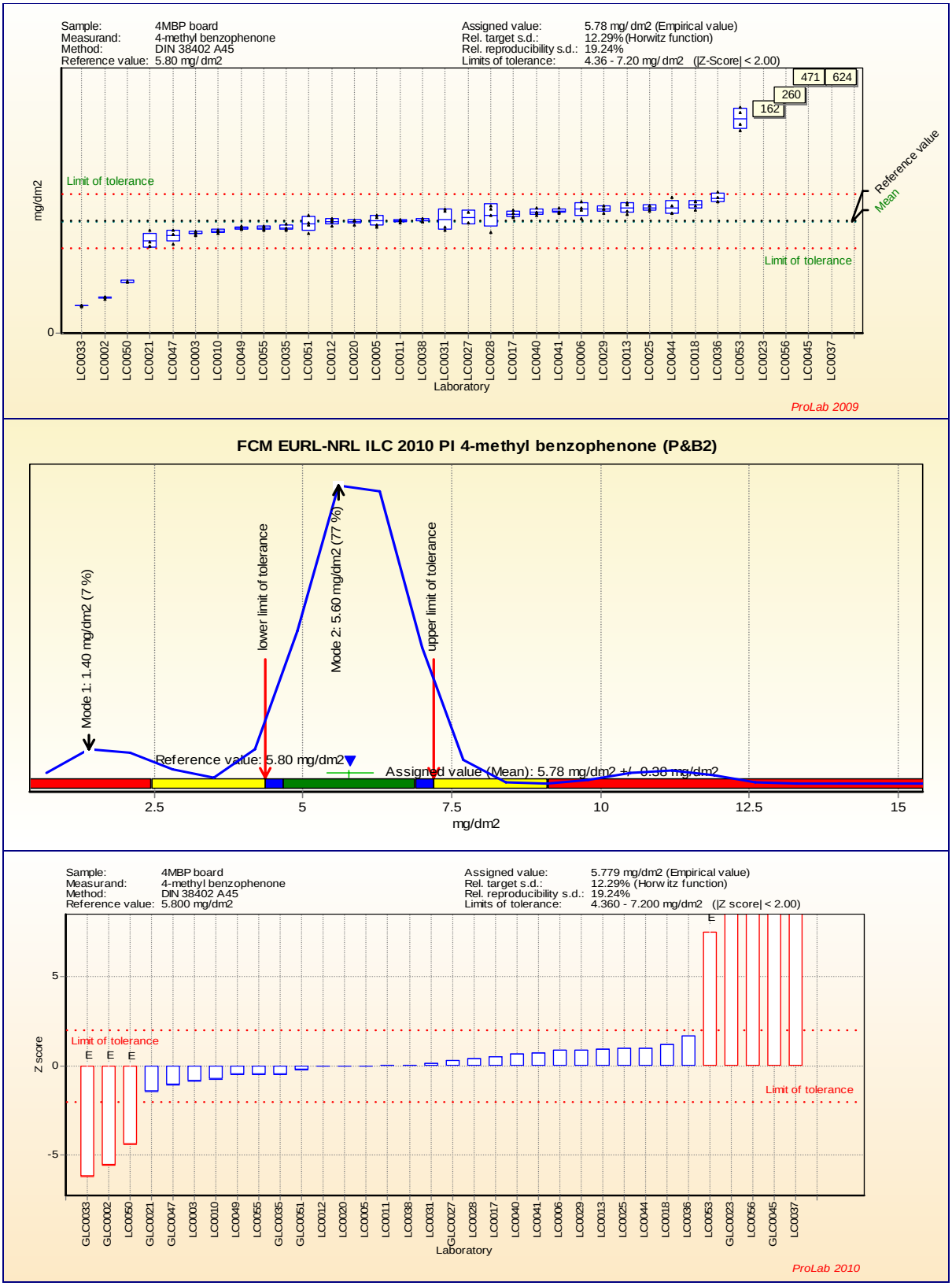


Figure 2-1: Mandel h-statistics for PI in solvent mix and paperboard

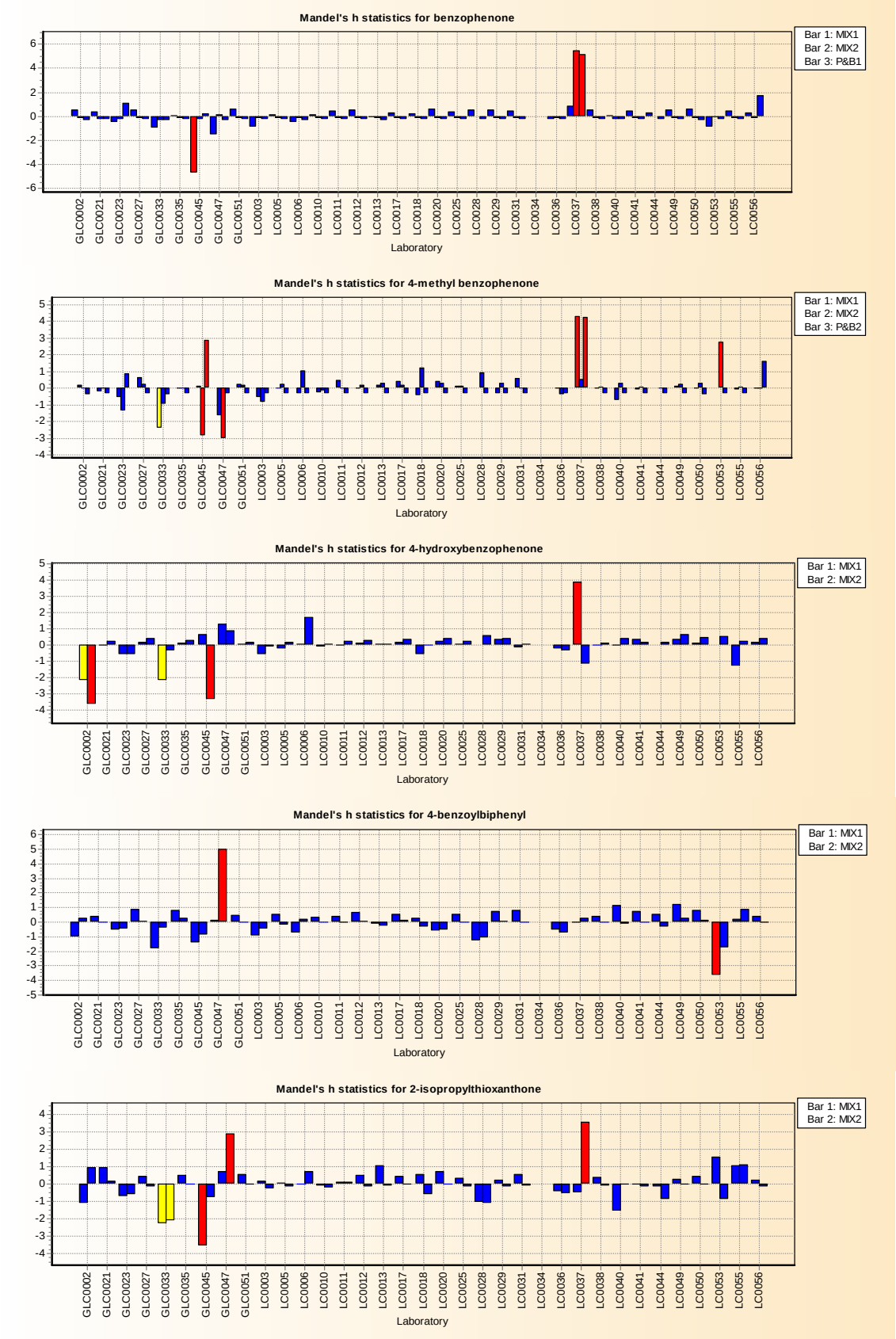
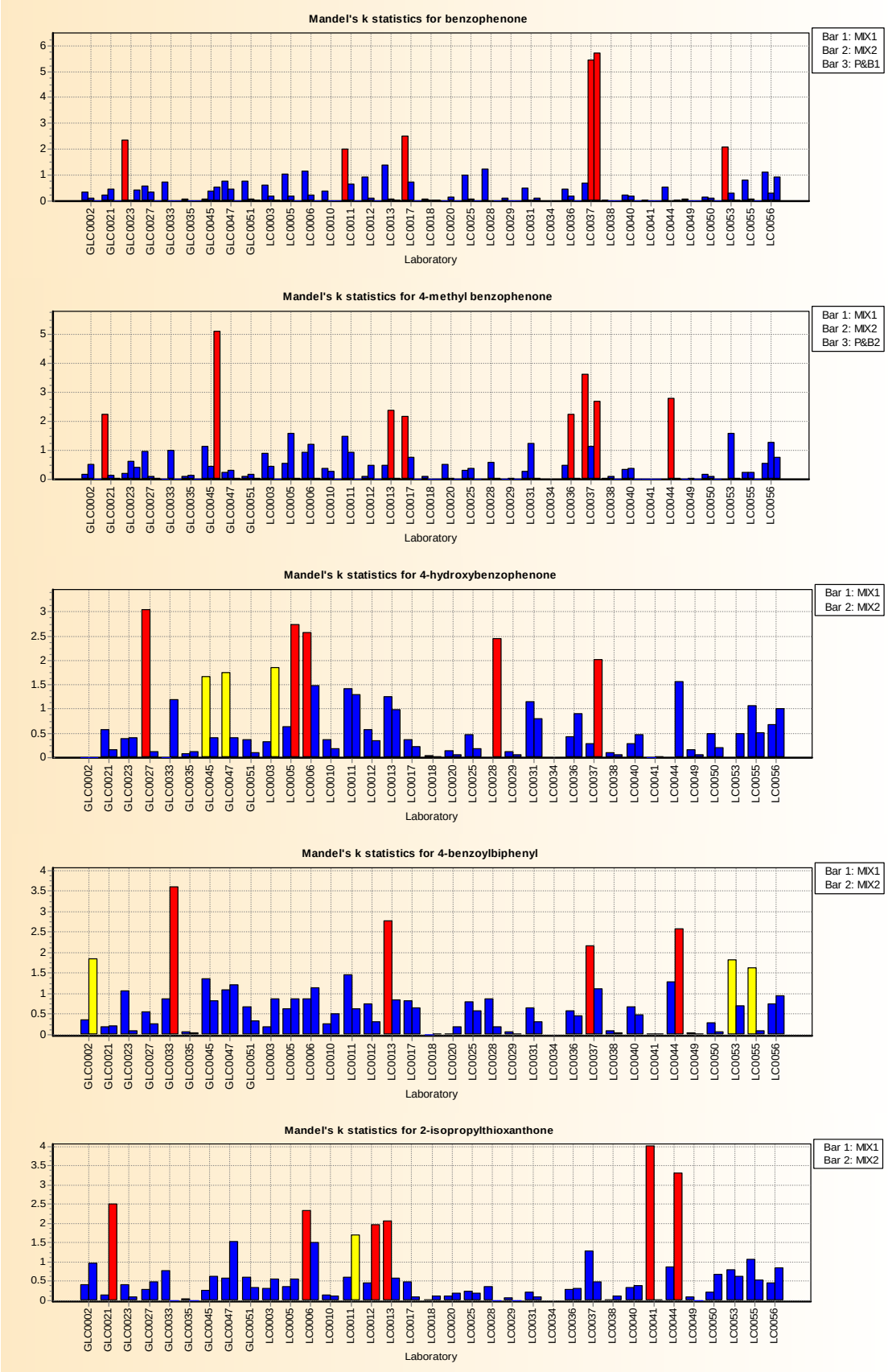


Figure 2-2: Mandel k-statistics for PI in solvent mix and paperboard



* 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).

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 are given in Table 3.

Table 4: Table of robust mean and robust SD calculated according to Hampel algorithm (ISO 20612:2007 and DIN 38402 A45)

DIN										
Sample	Measurand	Mean value mg/kg	Reference value mg/kg	Mode Kernel	Repeat. s.d. mg/kg	Rel. Repeat. SD %	Reprod. s.d. mg/kg	Reprod. s.d. %	Target s.d. %	Mode
MIX1	4HBP	3.13	3.29	3.21	0.05	1.51	0.63	19.2	13.4	kH
MIX1	4MBP	0.700	0.669	0.71	0.02	2.34	0.13	19.2	17.0	kH
MIX1	BP	90.3	93.6	92.62	1.06	1.18	6.82	7.3	8.1	kH
MIX1	ITX	30.9	32.3	31.9	0.52	1.68	3.68	11.4	9.5	kH
MIX1	PBZ	51.1	54.9	53.86	1.09	2.13	6.02	11.0	8.8	kH
MIX2	4HBP	66.3	68.4	67.1	1.15	1.73	6.70	9.8	8.5	kH
MIX2	4MBP	85.9	87.5	85.8	0.92	1.07	6.86	7.8	8.2	kH
MIX2	BP	0.524	0.546	0.52	0.02	3.77	0.10	18.5	17.5	kH
MIX2	ITX	1.15	1.22	1.2	0.02	1.99	0.20	16.2	15.5	kH
MIX2	PBZ	6.82	7.27	7.13	0.11	1.55	1.21	16.6	11.9	kH
		mg/dm2	mg/dm2							
P&B1	BP	4.23			0.17	3.92	0.00	24.5	15.0	M
P&B2	4MBP	5.78			0.21	3.64	0.00	19.2	15.0	M

Relative reproducibility SD complies with the Target
Relative reproducibility SD is slightly higher then the Target
Relative reproducibility SD is higher then the Target
Relative reproducibility SD is much above the Target

It should be mentioned that the robust mean derived from the results, taken as assigned values coincides well with the reference values for almost all measurand-matrix combination in solvent 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 value (e.g. uncertainty of the robust mean);

s* is the robust standard deviation;

p is the number of participating laboratories

In the cases where the difference between the robust mean values and reference values based on formulation is greatest, but still not significant, the Kernel density mode value, shown on the figure 1b (Kernel density plot) is closer to the reference one (e.g for BPZ and ITX in both samples)

More attention should be paid on the robust standard deviation for 4HBP and PBZ in the solvent mixes with lower concentration level, where the robust SD is up to 40-44%% higher then expected from Horwitz value, as well as for PI in paperboard where Horrat > 1.5 for BP. In the first case improvement could be reached after attempt for lowering the LOD for those compounds, whereas in the second case attention should be paid when calculating the PI content in mg/dm^2 units.

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).

Mandel h statistic should be considered carefully as Mandel h value < 1.9 resulting in blue bars not always means "good" results. When two or more labs have very different results from the "assigned value" but there is a third lab with n-fold much different result, then the Mandel h values of the first two would be < 1.9 (blue bars).

That is the case with BP paperboard where the bars for labs LC0023 and LC0056 are blue (Mandel h less then 1.9) nevertheless their z-scores are between 260 and 380. That is due to the fact that there is a lab with much higher value – z-score more than 1000, which truncate the higher results of labs LC0023 and LC0056.

Most frequent pattern for Mandel h-statistic for BP in the three samples is that the h-value for MIX1 (with the lowest BP concentration) is slightly positive, whether the other two h-values for MIX2 and P&B1 are slightly negative. That is a common pattern and does not mean a systematic error. Lab LC0037 has a systematic error as the three h-values are positive and very high.

Considering the 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 most of the measurand-matrix combination. For paperboard samples the overall performance of the laboratories is slightly lower with 71% successful results for BP as well as for 4MBP. The difference in laboratory performance came most probably from the error introduced by the calculation of the content from mg/kg to mg/dm^2.

15 laboratories had 100% successful results and for only 9 labs less than 75% of the results were satisfactory.

The Youden plot displays 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.

Figure 4a represents the Youden plots for each measurand in both solvent mixes - MIX1 and MIX2 in a scale of +/- z=13. Figure 4b shows the same plots however together for all PI in both scales z==/- 13 and cut up to z= =/- 5. The comparison is done for both similar acetonitrile solutions. Determination of PI in paperboard is not considered as there is strong error in some of the participant due eventually to the errors in recalculating of the PI content in mg/dm^2.

Youden plots presented on Figure 4a show no correlations between the results in both solvent MIX1 and MIX2 as the correlation coefficient is lower than 0.6.
It could be easily concluded that dominating systematic error could be found in the results of laboratories GLC0033 for BP, GLC0047 and GLC0032 for 4MBP, GLC0002 for 4HBP, LC0053 for PBZ and GLC0033 for ITX whereas from random errors suffered the results of lab GLC045 for almost all compounds, GLC0006 and GLC0033 for 4HPB, GLC047 for BP and ITX, GLC0047 for BP.

Figure 5 represents the overall z'-score distribution for all the 400 measurand-matrix-laboratory combinations for 4 measurands, 4 samples and 34 laboratories'. Figure 6 represents them in histogram like Kernel density plot and normal distribution plot - showing its real normal distribution. Only 92.5% of the results fall down in the z≤6 range, which is due to the large outliers for BP and 4MBP in paperboard.

As a conclusion of the ILC exercise on the quantification of photoinitiators in the paperboard and the solvent, this ILC showed that:

- The participation in the ILC was satisfactory regarding the number of the participating labs. Thirty-two laboratories submitted their results. From the EURL-NRL network 22 laboratories out of 25 reported results. 3 NRLs laboratories did not send any results.

- For the network of NRL-FCM the outcome from the participation could be regarded as satisfactory as there are about 84% satisfactory results (262 out of 312) and only 3 NRLs did not participated out of 25.

Figure 3: Summary of z -scores

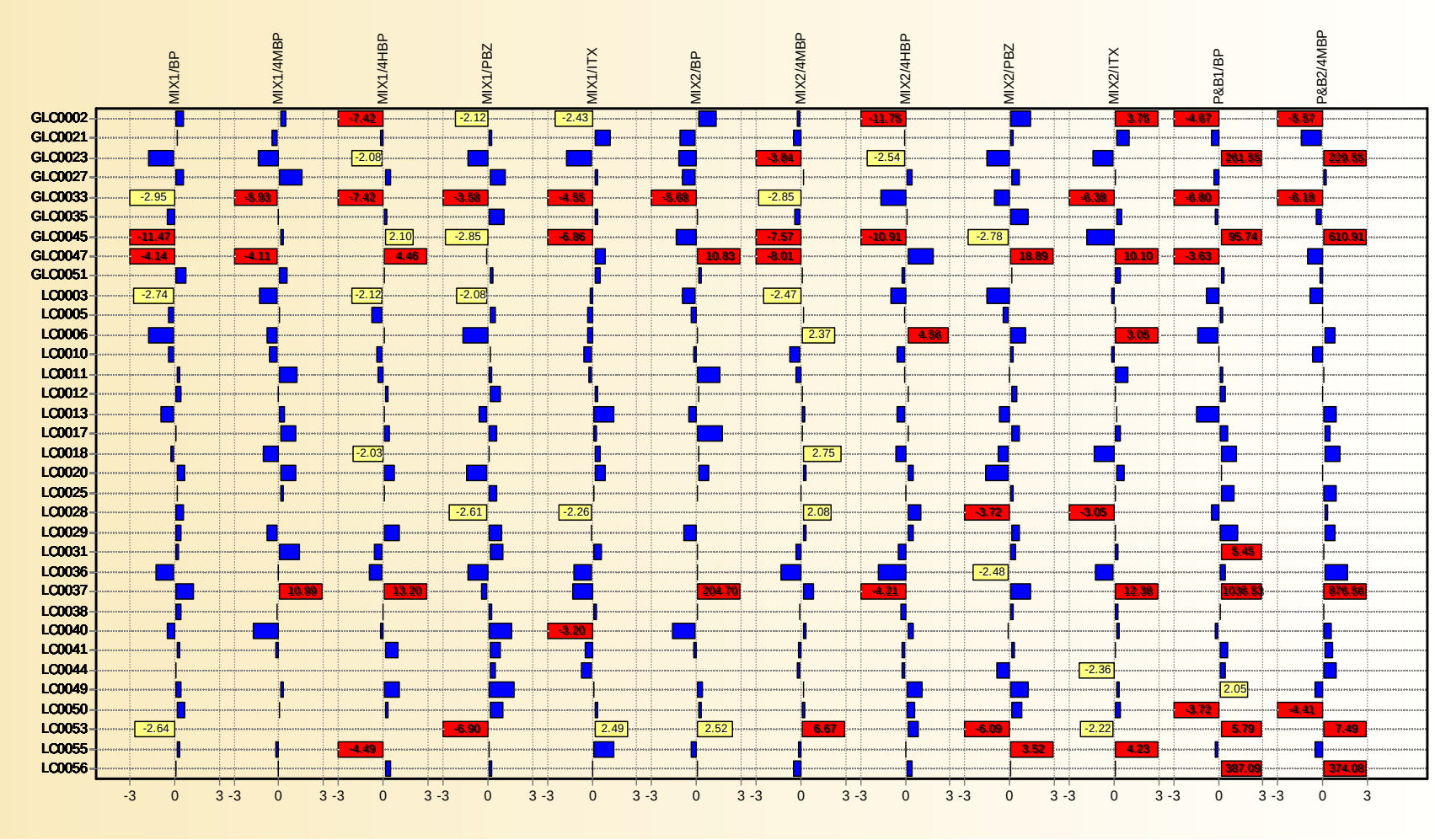


Table 5: Summary of z -scores

Laboratory	MIX1/ BP	MIX1/ 4MBP	MIX1/ 4HBP	MIX1/ PBZ	MIX1/ ITX	MIX2/ BP	MIX2/ 4MBP	MIX2/ 4HBP	MIX2/ PBZ	MIX2/ ITX	P&B1/ BP	P&B2/ 4MBP	Successful	%
GLC0002	0.65	0.58	-7.42	-2.12	-2.43	1.42	-0.26	-11.75	1.45	3.75	-4.67	-5.57	5	**42**
GLC0021	0.29	-0.44	-0.19	0.38	1.3	-1.09	-0.48	-0.04	0.28	1.12	-0.47	-1.45	12	**100**
GLC0023	-1.76	-1.34	-2.08	-1.32	-1.71	-1.17	-3.84	-2.54	-1.52	-1.4	261.5	229	7	**58**
GLC0027	0.69	1.65	0.61	1.28	0.49	-0.93	0.29	0.57	0.7	0.06	-0.32	0.3	12	**100**
GLC0033	-2.95	-5.93	-7.42	-3.58	-4.55	-5.68	-2.85	-1.67	-1.06	-6.38	-6.8	-6.18	2	**17**
GLC0035	-0.45	-0.04	0.33	1.18	0.49	0.04	-0.37	0.24	1.35	0.62	-0.26	-0.46	12	**100**
GLC0045	-11.47	0.34	2.1	-2.85	-6.86	-1.36	-7.57	-10.91	-2.78	-1.87	95.74	610	3	**25**
GLC0047	-4.14	-4.11	4.46	-0.06	0.97	10.83	-8.01	1.94	18.89	10.1	-3.63	-1.05	4	**33**
LC0003	-2.74	-1.28	-2.12	-2.08	-0.11	-0.93	-2.47	-0.96	-1.5	-0.2	-0.78	-0.88	8	**67**
LC0005	-0.36	0.08	-0.75	0.59	-0.3	-0.37	0.25	-0.06	-0.41	0.09	0.33	0.02	12	**100**
LC0006	-1.71	-0.77	0.08	-1.64	-0.33	0.04	2.37	4.56	1.15	3.05	-1.41	0.87	9	**75**
LC0010	-0.38	-0.66	-0.46	0.26	-0.55	-0.2	-0.76	-0.51	0.34	-0.17	0.04	-0.73	12	**100**
LC0011	0.45	1.28	-0.32	0.38	-0.22	1.65	-0.3	-0.01	0	1.05	0.33	0.02	12	**100**
LC0012	0.52	-0.08	0.4	0.9	0.5	0.26	0.09	0.26	0.53	0.13	0.51	-0.02	12	**100**
LC0013	-0.92	0.49	0.06	-0.55	1.58	-0.5	0.38	-0.53	-0.72	0.25	-1.44	0.96	12	**100**
LC0017	0.1	1.19	0.51	0.69	0.4	1.8	0.12	0.32	0.77	0.49	0.67	0.52	12	**100**
LC0018	-0.21	-1.01	-2.03	0.12	0.63	0.23	2.75	-0.64	-0.78	-1.3	1.28	1.23	10	**83**
LC0020	0.79	1.19	0.85	-1.44	0.99	0.9	0.4	0.63	-1.58	0.72	0.25	-0.01	12	**100**
LC0025	0.3	0.37	0.11	0.67	0.19	0.17	-0.03	0.03	0.29	0.15	1.16	0.97	12	**100**
LC0028	0.68			-2.61	-2.26		2.08	1.1	-3.72	-3.05	-0.47	0.42	4	**33**
LC0029	0.56	-0.77	1.16	1.01	-0.01	-0.82	0.4	0.64	0.7	0.12	1.34	0.87	12	**100**
LC0031	0.34	1.51	-0.6	1.07	0.69	0.1	-0.31	-0.42	0.48	0.35	5.45	0.14	11	**92**

Table 5: Summary of z -scores (continued)

Laboratory	MIX1/BP	MIX1/4MBP	MIX1/4HBP	MIX1/PBZ	MIX1/ITX	MIX2/BP	MIX2/4MBP	MIX2/4HBP	MIX2/PBZ	MIX2/ITX	P&B1/BP	P&B2/4MBP	Successful	%
LC0036	-1.25	-0.06	-0.92	-1.31	-1.22	0.05	-1.36	-1.77	-2.48	-1.23	0.53	1.69	11	**92**
LC0037	1.32	10.99	13.2	-0.44	-1.33	204.7	0.96	-4.21	1.47	12.38	1036	876	5	**42**
LC0038	0.51	-0.1	0.02	0.38	0.37	0.12	-0.11	-0.32	0.31	0.32	0.09	0.06	12	**100**
LC0040	-0.47	-1.72	-0.17	1.69	-3.2	-1.6	0.44	0.62	-0.11	0.46	-0.19	0.67	11	**92**
LC0041	0.45	-0.25	1.11	0.96	-0.49	-0.21	-0.17	-0.19	0.4	0.12	0.69	0.73	12	**100**
LC0044	0.14			0.64	-0.69		-0.24	-0.21	-0.85	-2.36	0.54	1.02	8	**67**
LC0049	0.52	0.41	1.16	1.89	0.12	0.47	0.23	1.24	1.35	0.45	2.05	-0.51	11	**92**
LC0050	0.77	0.03	0.37	1.12	0.45	0.37	0.35	0.68	0.88	0.53	-3.72	-4.41	10	**83**
LC0051	0.84	0.64	0.14	0.47	0.61	0.42	0.14	-0.2	0.27	0.53	0.47	-0.2	12	**100**
LC0053	-2.64			-6.9	2.49	2.52	6.67	0.96	-6.09	-2.22	5.79	7.49	1	**8**
LC0055	0.44	-0.23	-4.49	0.07	1.55	-0.33	-0.14	0.01	3.52	4.23	-0.25	-0.5	9	**75**
LC0056	0.12	-0.01	0.59	0.35	0.01	0.16	-0.49	0.57	0.15	0.06	387.0	374	10	**83**
Successful	27	27	24	28	27	29	28	29	28	26	24	24		
%	**79**	**79**	**71**	**82**	**79**	**85**	**82**	**85**	**82**	**76**	**71**	**71**		

Figure 4.a: Youden plot of z-scores of the five PI separately for solvent 1 against solvent 2

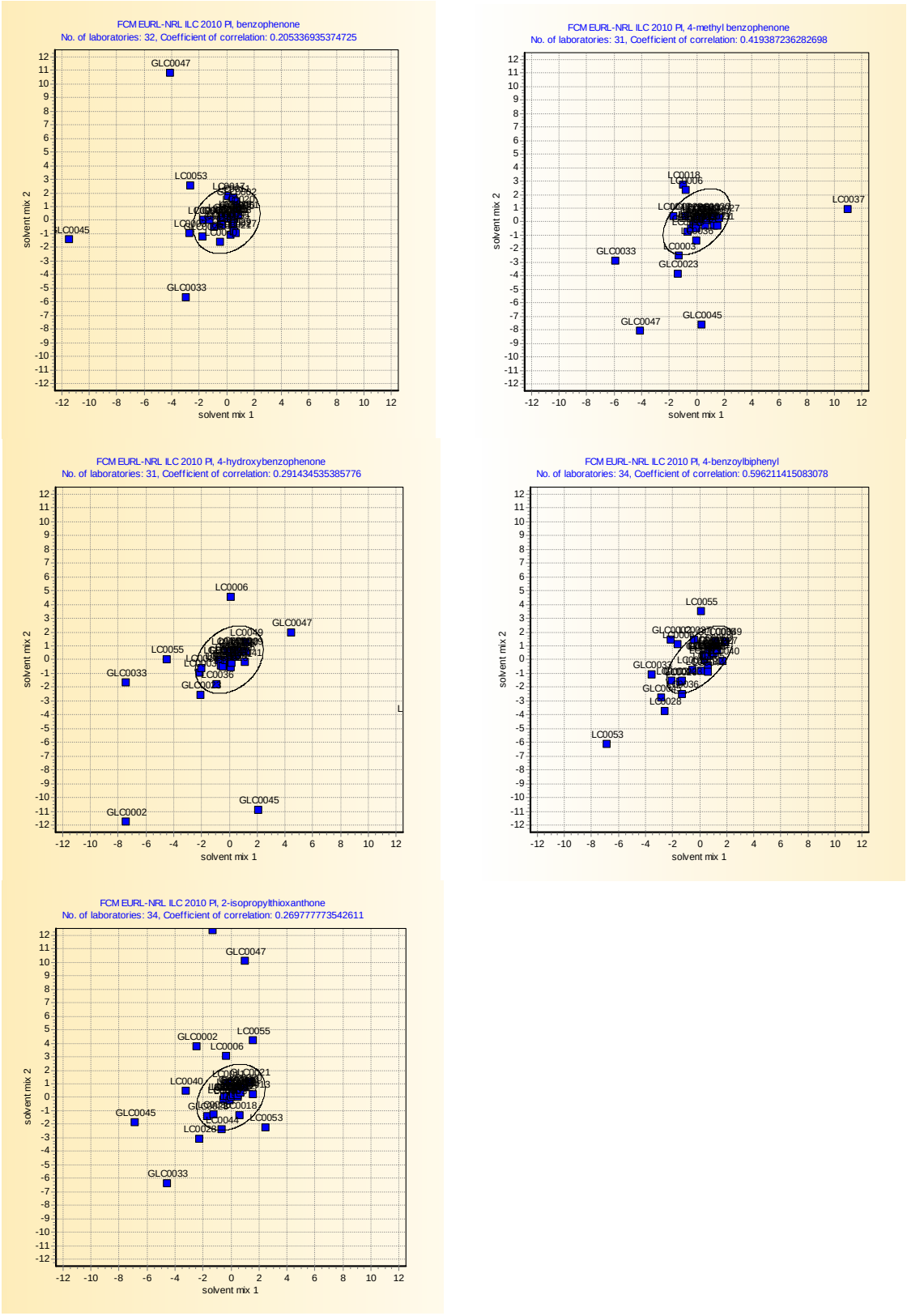
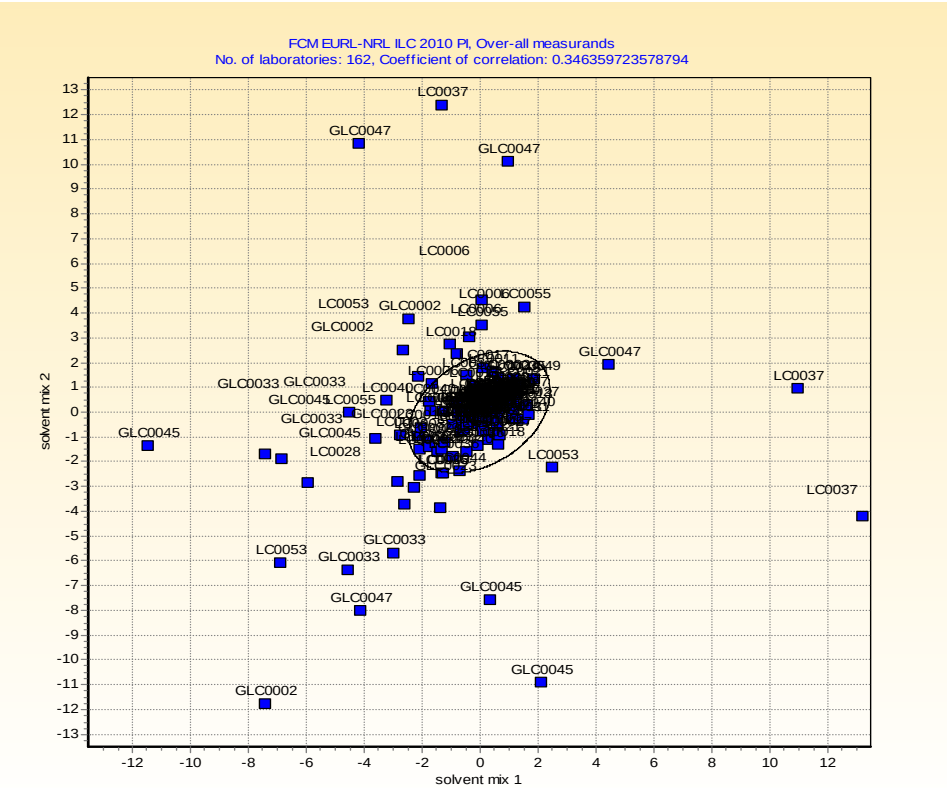


Figure 4.b: Youden plot of z-scores of the five PI together for solvent 1 against solvent 2



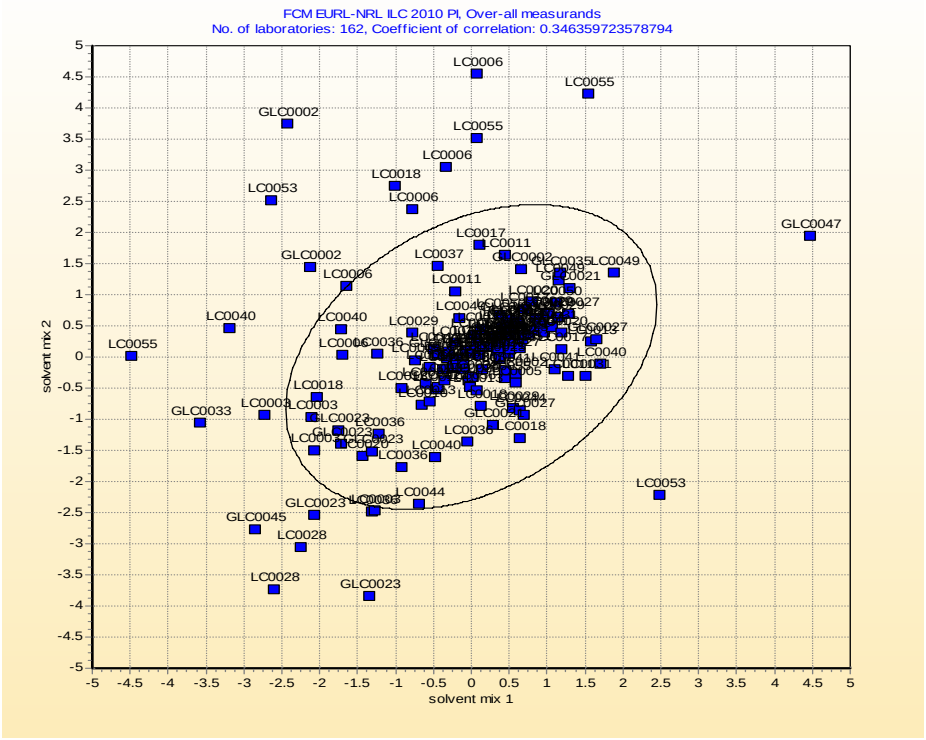
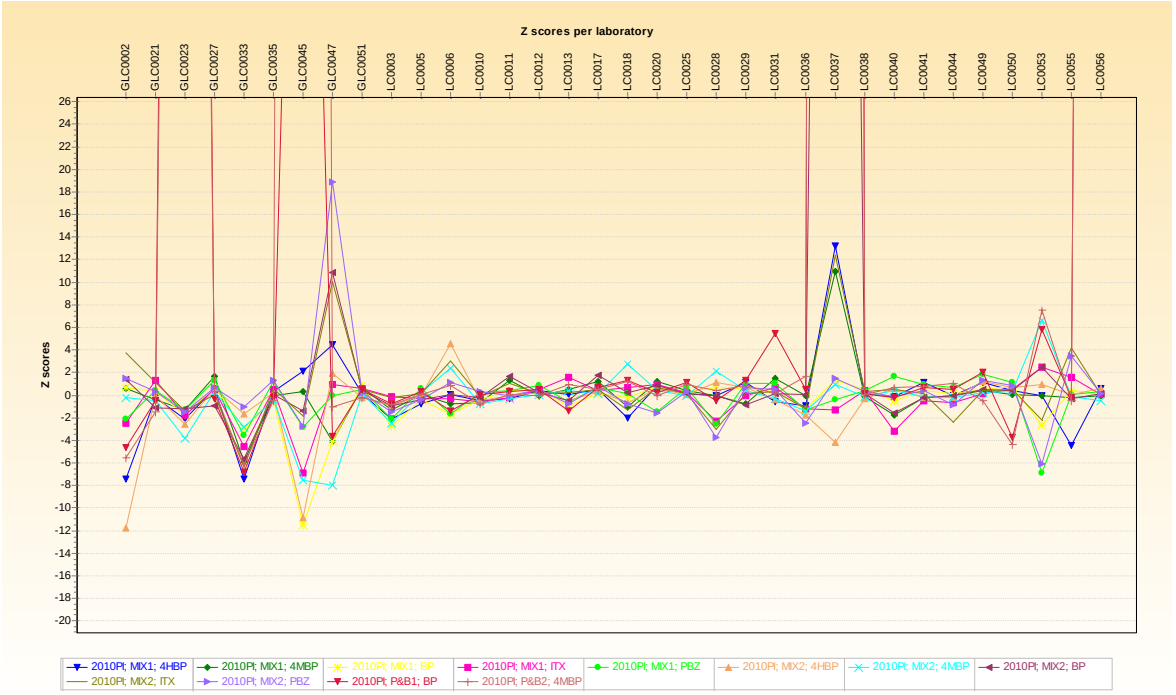


Figure 5: Distribution of all z-score – scatter

a) distribution by labs



b) distribution by samples (excl. BP and 4MBP from paperboard)

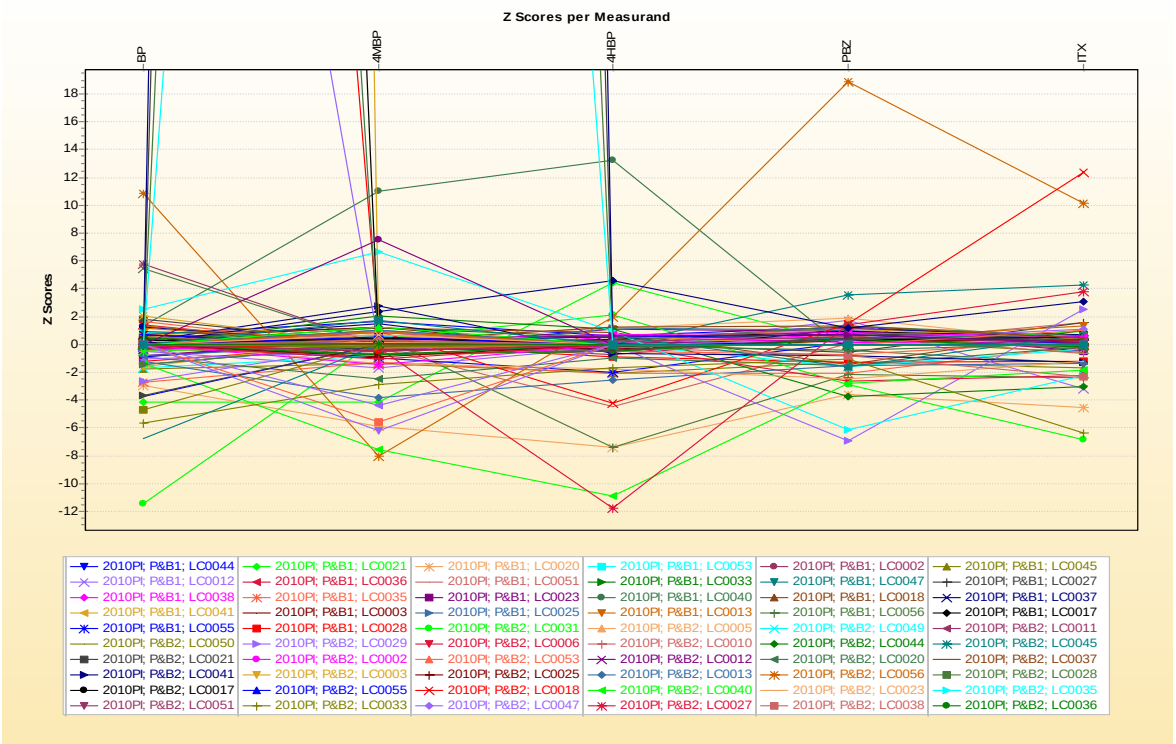
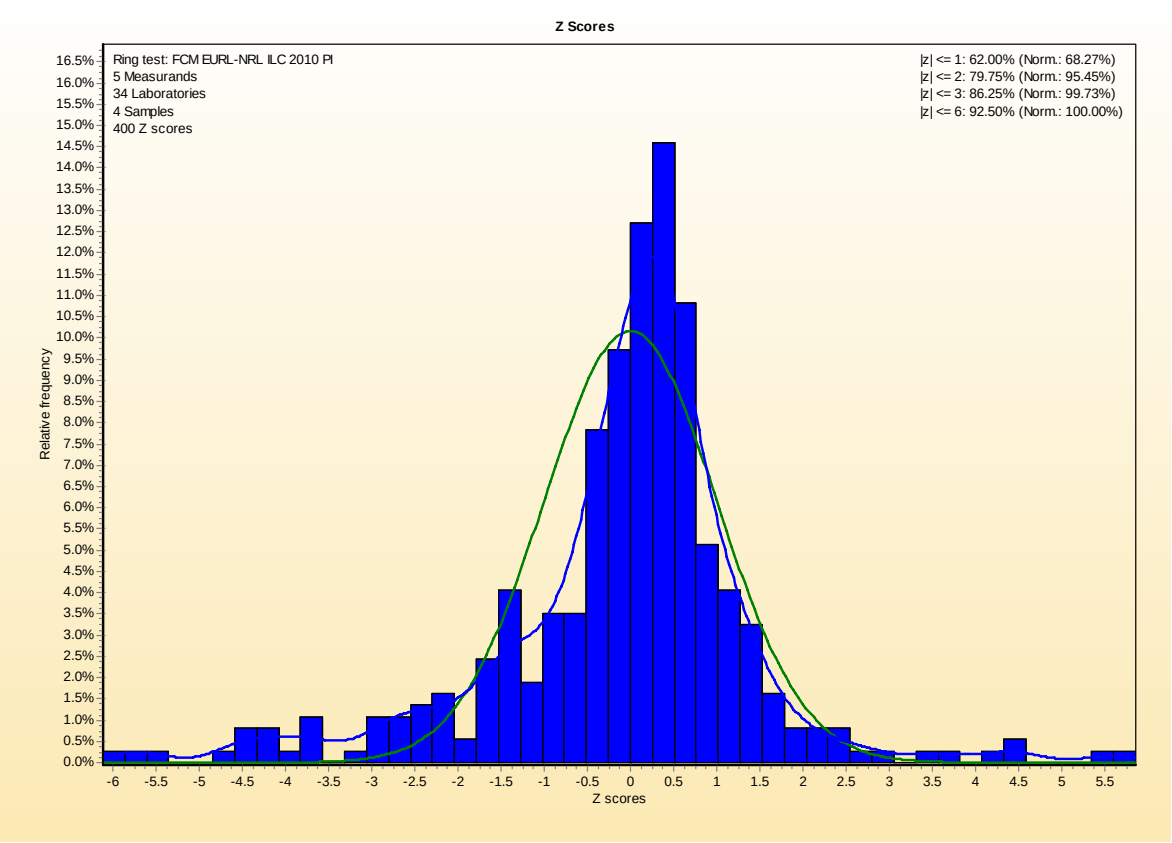


Figure 6: Distribution of all z score histogram (blue bars), Kernel density plot (blue line) and normal distribution plot (green line)



11. Acknowledgements

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

NRLs

AUSTRIA	Austrian Agency for Health and Food Safety (AGES)
BELGIUM	Institute of Public Health, ISSP-LP
BULGARIA	RIOKOZ, Veliko Turnovo
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
FRANCE	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
LUXEMBOURG	Laboratoire National de Sante', Division du Controle des denrées alimentaires
LATVIA	Head of Instrumental Analysis Division, Institute of Food Safety, Animal Health and Environment (BIOR)
LITUENIA	National Public Health Investigation Centre, Laboratory of Chemistry
POLAND	Laboratory of Department of Food and Consumer Articles Research , National Institute of Hygiene
PORTUGAL	ESB-SE (Portuguese Catholic University - Biotechnology College – Packaging Department)
SLOVAK Republic	National Reference Centre and Laboratory for material and articles intended to come into contact with food, Regional Public Health Authority In Slovak Republik
SLOVENIA	National Institute of Public Health of Republic of Slovenia, Dept of Sanitary Chemistry
SPAIN	Centro Nacional de Alimentación, Agencia Espanola de Seguridad Alimentaria y Nutrición (AESAN)
UK	Central Science Laboratory

GUESTS

Germany	Bayerisches Landesamt für Gesundheit und Lebensmittelsicherheit, Bavarian Health and Food Safety Authority
SWISS	Amt für Gesundheits- und Verbraucherschutz AfGVS. 9001 St. Gallen
SWISS	Service de la Consommation et des Affaires Vétérinaires (SCAV) Genève 4 Plainpalais,

FRANCE	Laboratoire SCL Oullins
GERMANY	Chemisches und Veterinäruntersuchungsamt OWL , 32717 Detmold
GERMANY	Landesuntersuchungsamt Rheinland-Pfalz, Institut für Lebensmittelchemie Koblenz
GERMANY	Niedersächsisches Landesamt für Verbraucherschutz, und Lebensmittelsicherheit (LAVES) Institut für Bedarfsgegenstände , Lüneburg
GERMANY	CVUA-MEL, Münster
GERMANY	Landesbetrieb Hessisches Landeslabor, Standort Wiesbaden
GERMANY	Landesuntersuchungsanstalt für das Gesundheits und Veterinärwesen Sachsen, Dresden

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 .

[14] ISO 5725 -2 Accuracy (trueness and precision) of measurement method

and results. Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.

[15] ISO 5725 -5 Accuracy (trueness and precision) of measurement method and results. Part 5: Alternative methods for the determination of the precision of a standard measurement method.

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

13. Annexes

Annex 1: Invitation letter to laboratories ILC 2010/01

Annex 2: Form for confirmation of participation to ILC 2010/01

Annex 3: Letter accompanying the samples ILC 2010/01

Annex 4: Letters of confirmation of receipt ILC 2010/01

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

Annex 6: Form for the compilation of results in non-electronic format

Annex 7: Results of the homogeneity study

Annex 8: Results of the stability study

Annex 1: Invitation letter to laboratories ILC 2010/01

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

Community Reference Laboratory
CRL
Food Contact Materials

Ispra April 26, 2010
Ref. Ares(2010)217332 - 26/04/2010

Dear Madam, Sir

Comparative trial ILC 2010 - 01 from EURL FOOD CONTACT MATERIALS "Determination of photoinitiators (PI) in paper and board"

On behalf of the EURL for food contact materials, I would like to invite you to participate in a comparative trial/interlaboratory comparison (ILC) exercise for the determination of photoinitiators (PI) in solvent (acetonitrile) and paper and board (P&B) which is due to start by the end of May. Please note that according to the agreement of the December's EURL-NRL FCM plenary, this year the ILC exercise is a Proficiency testing exercise. Each participant will be free to use the method of routine use in their laboratory. We will provide SOP for information only to those who don't have experience in the field, but the SOP will not be mandatory.

I would like to remind you that it is a duty for you as an NRL-FCM to participate in the ILCs organised by the EURL-FCM since the work programme is decided with your agreement. For this reason we 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 May 3rd** to: Stefanka Bratinova (stefanka-petkova.bratinova @jrc.ec.europa.eu)**.** If you need more test kits to involve more laboratories at the national level please let us know immediately by e-mail so we can prepare the test samples accordingly.

The samples will be sent to you by the **Middle of May**. You will find additional information in the kit sent and on the form "shipment test PI". You will also receive more detailed instructions for the compilation of the results. The deadline for submission of results is **1st July 2010.**

If you have any question, please contact Catherine Simoneau (catherine.simoneau@jrc.ec.europa.eu), 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 Chemical Assessment and Testing, T.P. 260
Ispra Va 21020 Italy

Cc: MM. D. Kotzias (JRC), D. Sarigiannis (JRC), B. Larsen (JRC), F. d'Atri (SANCO)
Mrs. A Schaefer (SANCO)

Annex 2: Form for confirmation of participation to ILC 2010/01

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

Community Reference Laboratory
Food Contact Materials

Ref. Ares(2010)217332 - 26/04/2010
Ispra April 26 2010
Annex

Participation to EURL-FCM ILC 2010 - 01
Interlaboratory comparison (ILC) exercise
for the determination of photoinitiators in solvent and paper and board

CONFIRMATION OF PARTICIPATION

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

item	Yes	NO
I will participate the collaborative trial on analysis of PI in solvent and paper and board and will deliver results on time		
I have already the package with the files for filling the results and especially RingDat3.exe file from last year and I need only lab files for this year's ILC		
I don't have the package with the files for filling the results from last year		

Kindly send back this proformat to: Stefanka Bratinova (stefanka-petkova.bratinova @jrc.ec.europa.eu) **by May 3rd.**
The samples will be sent to you by the Middle of May. You will find additional information in the kit sent. The deadline for submission of results is **1st July 2010**

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

Sincerely yours,

Catherine Simoneau

Annex 3: Letter accompanying the samples ILC 2010/01

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

Community Reference Laboratory
Food Contact Materials

Ispra May 10, 2010
Ref. Ares(2010)217332 - 26/04/2010

Shipping kit for interlaboratory comparative testing EURL-FCM ILC01 2010 – Photo-initiators in solvent and in paper and board

Shipping kit - PI in solvent:

- two 40 ml screw cap vials containing acetonitrile, spiked with five PI (BP, 4-MBP, 4-HBP, PBZ, ITX) at 2 concentration levels in the range 0.5-100 mg/kg

*NOTE: Report the results in **mg/kg** taking into account the real density of ACN !!!!!*

Shipping kit - PI in paper and board:

- ten 5x5 cm^2 pieces of board (for BP) and ten 10x5 pieces of board (for 4-MBP)

*NOTE: Extraction of PI from P&B should be performed with acetonitrile, results reported in **mg/dm²***

Shipping kit - reference substances:

- approx. 1 g BP
- approx. 1 g 4-MBP
- approx. 1 g 4-HBP;
- approx. 0.75 g PBZ
- approx. 10 ml 400 mg/l ITX

Documentation:

- instruction for the compilation of results;
- letters of confirmation of receipt ILC 01 2010;
- empty form “Test results” for filling the results;

Results requested:
result format: Use the personal “NAME.LAB” file that will be provided by e-mail; use the printed empty form “Test results” in case of difficulties with the files;

Annex 4: Letter of confirmation of receipt of ILC 2010/01

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

Community Reference Laboratory
Food Contact Materials

Ispra May 10, 2010
Ref. Ares(2010)217332 - 26/04/2010

PARTICIPATION TO CRL-FCM ILC02 2010
PHOTOINITIATORS IN SOLVENT AND PAPER AND BOARD

CONFIRMATION OF RECEIPT OF THE SAMPLES

.

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

Any remarks ……

Date arrival package ……

Signature ……

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

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 Chemical Assessment and Testing, T.P. 260
Ispra Va 21020 Italy

Direct access EURL: ph: +39.0332.785889 Fax: +39.0332.785707 e-mail: catherine.simoneau@jrc.it http://EURL-fcm.jrc.it/

Annex 5: Instructions for the compilation of the results in electronic format

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

Community Reference Laboratory
Food Contact Materials

Ispra May 10, 2010
Ref. Ares(2010)217332 - 26/04/2010

Instructions for the compilation of the results for interlaboratory comparative testing EURL-FCM ILC01 2010

DEADLINE: Thursday, July 1st

Results requested:

Perform four replicates for each sample and report all the four replicate data using the unit of measure specified in the lab file or printed paper "Test results":

Compilation of results

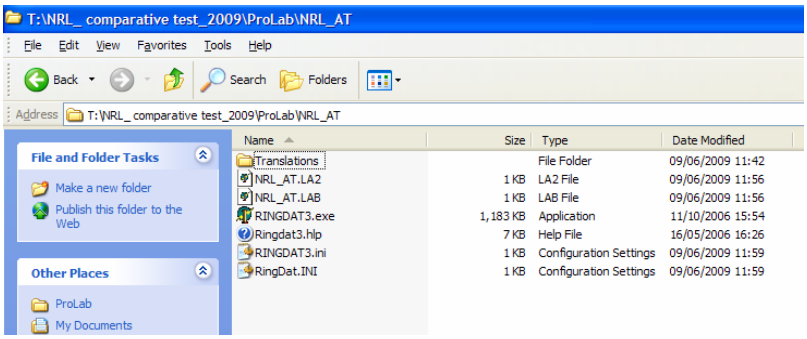
Data generated by the laboratories for the comparative test EURL-FCM PT001 will be processed by the EURL-FCM using a software package for statistical analyses and professional data handling of interlaboratory tests.

- a simple data entry program (RingDat3.exe) is provided to each participating laboratory (from last year PT 2008 or now on CD-ROM 2009) ;
- 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) by e-mail;
- 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 analyzed 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. That is why 6 sample codes will appear for each participant/file - 3 for acetonitrile and 3 for oil samples.

Procedure to be followed by laboratories for the opening the LAB files from the software.

First: Create a folder on your computer and transfer RingDat files and Translation directory (from last year PT2008 or from CD-ROM 2009) into it. Copy there as well the "NRL_X.LAB" and "NRL_X".LA2 files which you'll receive by mail. The folder should contain:



Direct access EURL: ph: +39.0332.785889 Fax: +39.0332.785707 e-mail: catherine.simoneau@jrc.it http://EURL-fcm.jrc.it/

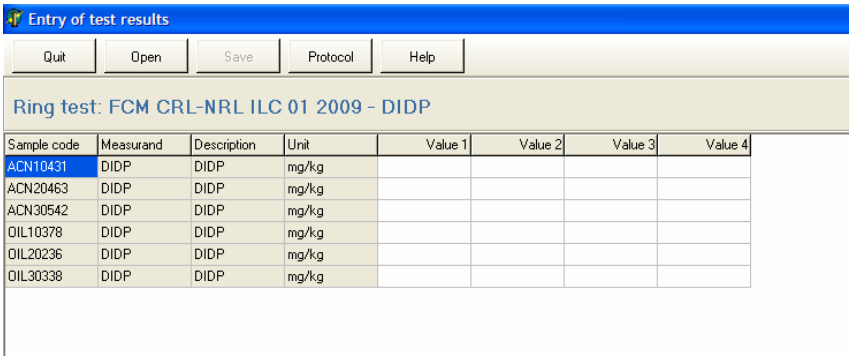
EUROPEAN COMMISSION
GENERAL DIRECTORATE JRC
JOINT RESEARCH CENTRE
Institute for Health and Consumer Protection – IHCP
Unit Chemical Assessment and Testing

Community Reference Laboratory
Food Contact Materials

Then:

- Open the file "RINGDAT3.exe"
- Click on "Open" command
- Select the "NRL_X.LAB" file (where X is the member state abbreviation- with one to three letters) and click on "Open" command using the button on the top menu of the window

- Windows you should see using the software is :



Sample code	Measurand	Description	Unit	Value 1	Value 2	Value 3	Value 4
ACN10431	DIDP	DIDP	mg/kg				
ACN20463	DIDP	DIDP	mg/kg				
ACN30542	DIDP	DIDP	mg/kg				
OIL10378	DIDP	DIDP	mg/kg				
OIL20236	DIDP	DIDP	mg/kg				
OIL30338	DIDP	DIDP	mg/kg				

- 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 Stefanka Bratinova (stefanka-petkova.bratinova @jrc.ec.europa.eu)

Please fill your results and send it back by e-mail to Stefanka Bratinova (stefanka-petkova.bratinova @jrc.ec.europa.eu) **by July 1st 2010.**

If you have any question, please contact Stefanka Bratinova (stefanka-petkova.bratinova @jrc.ec.europa.eu or Catherine Simoneau (catherine.simoneau@jrc.ec.europa.eu), ph. +39.0332.785889

Sincerely yours,

Dr. Catherine Simoneau
Operating Manager, Community Reference Laboratory for Food Contact Materials
European Commission, DG-Joint Research Centre
Institute for Health and Consumer Protection
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Annex 6: Form for the compilation of the results in non-electronic format

Test results

Lab code: LC0038

Sample code	Measurand	Description	Unit	Date of analysis	Value 1	Value 2	Value 3	Value 4
10027	BP	benzophenone	mg/kg					
10027	4MBP	4-methyl benzophenone	mg/kg					
10027	4HBP	4-hydroxybenzophenone	mg/kg					
10027	PBZ	4-benzoylbiphenyl	mg/kg					
10027	ITX	2-isopropylthioxanthone	mg/kg					
20047	BP	benzophenone	mg/kg					
20047	4MBP	4-methyl benzophenone	mg/kg					
20047	4HBP	4-hydroxybenzophenone	mg/kg					
20047	PBZ	4-benzoylbiphenyl	mg/kg					
20047	ITX	2-isopropylthioxanthone	mg/kg					
30210	BP	benzophenone	mg/dm2					
40198	4MBP	4-methyl benzophenone	mg/dm2					

..... Place and date

..... Manager of laboratory (in block letters)

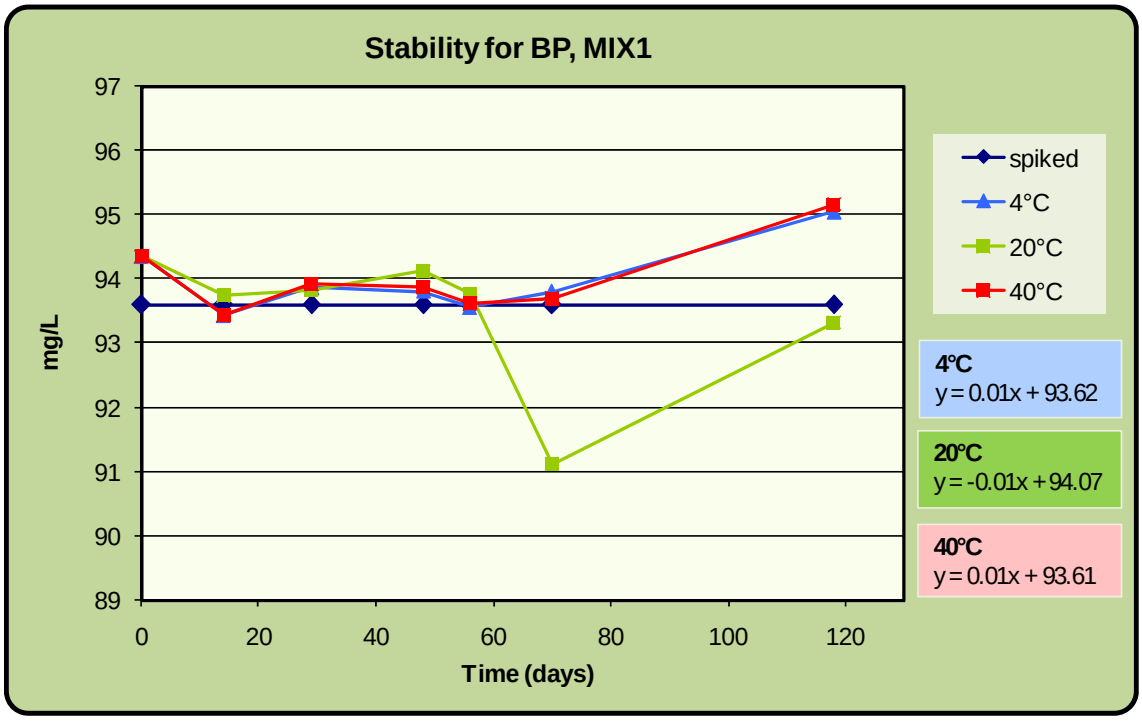
..... Signature

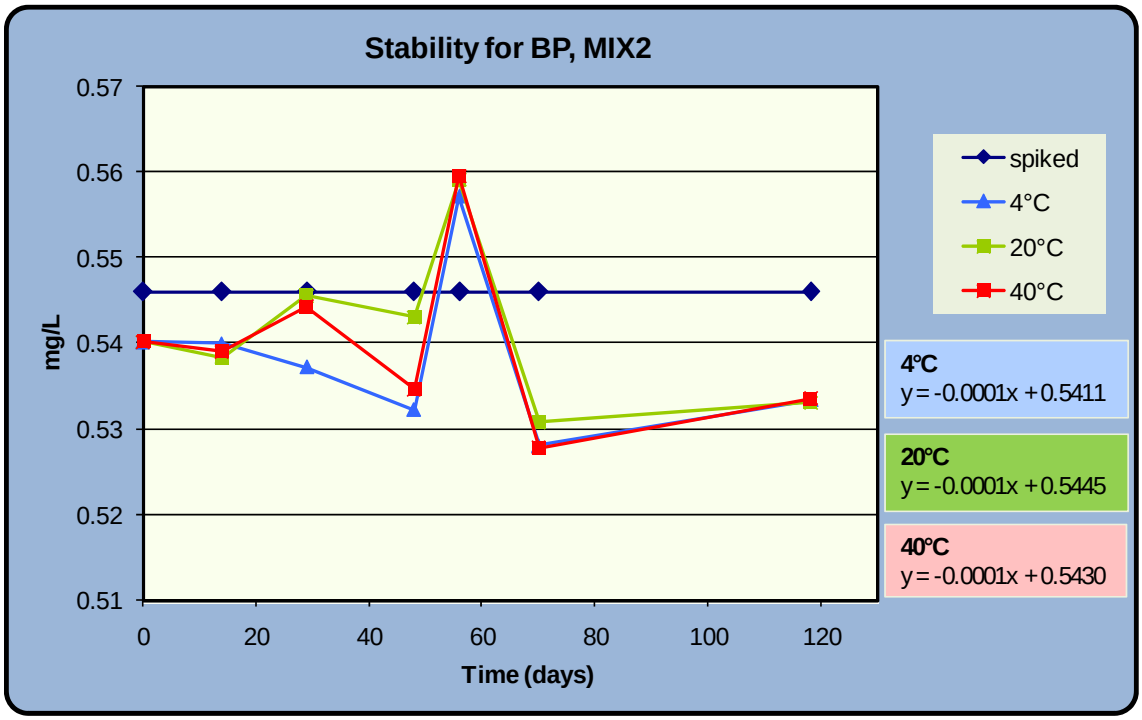
Annex 7: Results of the homogeneity study

Results from homogeneity test for BPA samples according to ISO 13528 and Harmonized protocol.

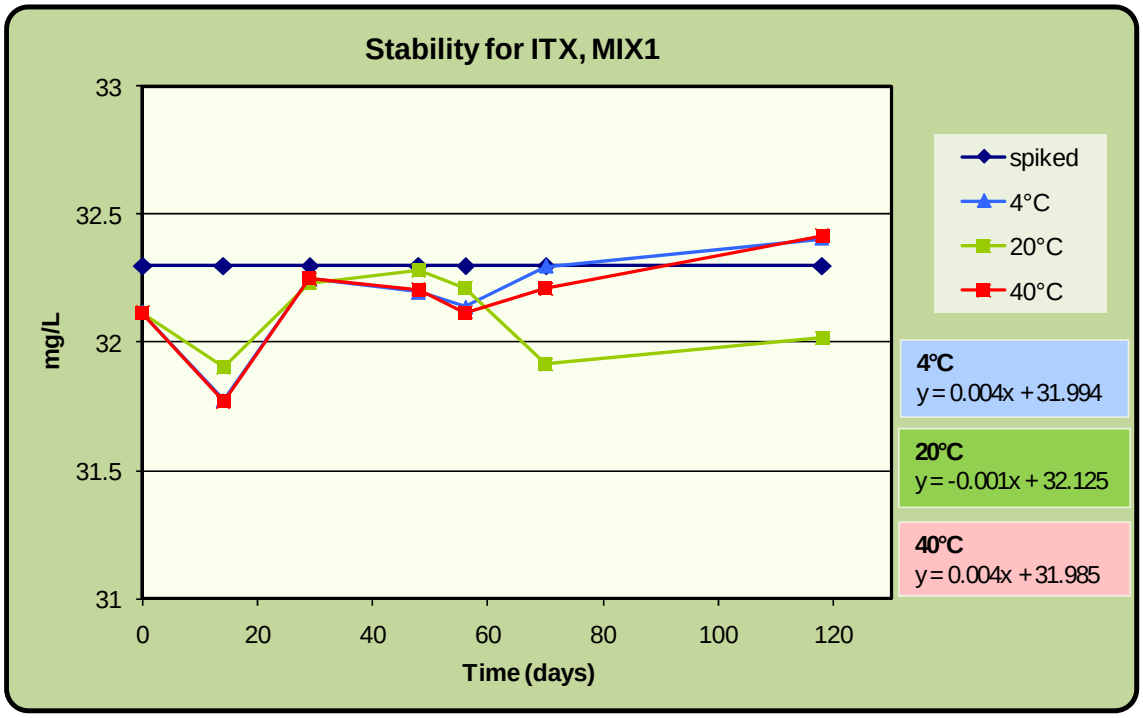
Sample	Measurand	Unit	Mean	s(analytical) %	s(samples) %	Mode s(target)	HORRAT	s(target) %	ISO 13528 Check for sufficient homogeneity	Harmonized Protocol - test on significant heterogeneity
MIX1	4HBP	mg/kg	3.04	2.7	0.0	Horwitz	1	13.5	OK	OK
MIX1	4MBP	mg/kg	0.691	0.5	0.6	Horwitz	1	16.9	OK	OK
MIX1	BP	mg/kg	94.5	0.1	0.4	Horwitz	1	8.1	OK	OK
MIX1	ITX	mg/kg	32.2	0.0	0.4	Horwitz	1	9.5	OK	OK
MIX1	PBZ	mg/kg	53.1	0.0	0.4	Horwitz	1	8.8	OK	OK
MIX2	4HBP	mg/kg	64.4	1.3	0.7	Horwitz	1	8.5	OK	OK
MIX2	4MBP	mg/kg	85.1	0.1	0.5	Horwitz	1	8.2	OK	OK
MIX2	BP	mg/kg	0.540	0.3	0.4	Horwitz	1	17.6	OK	OK
MIX2	ITX	mg/kg	1.216	0.1	0.7	Horwitz	1	15.5	OK	OK
MIX2	PBZ	mg/kg	7.092	0.1	0.7	Horwitz	1	11.9	OK	OK
P&B1	BP	mg/dm^2	4.27	1.6	3.5	Manual	1	15	OK	OK
P&B2	4MBP	mg/dm^2	5.81	0.1	0.7	Manual	1	15	OK	OK

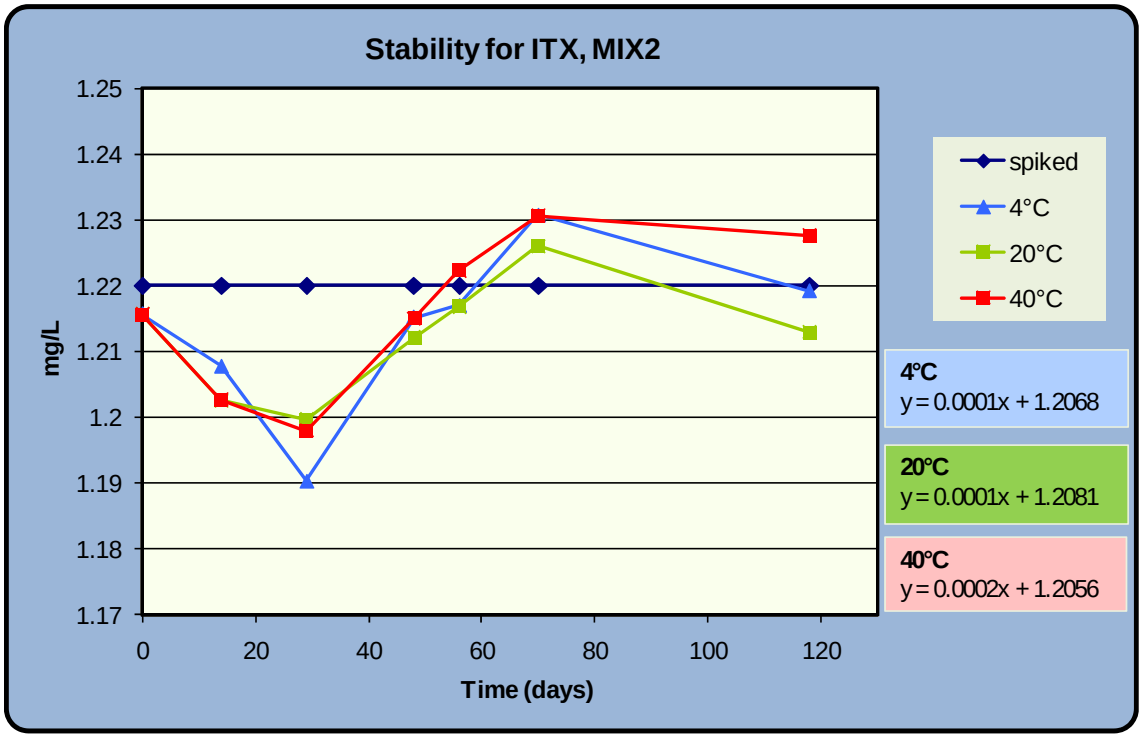
Annex 8: Results of the stability study



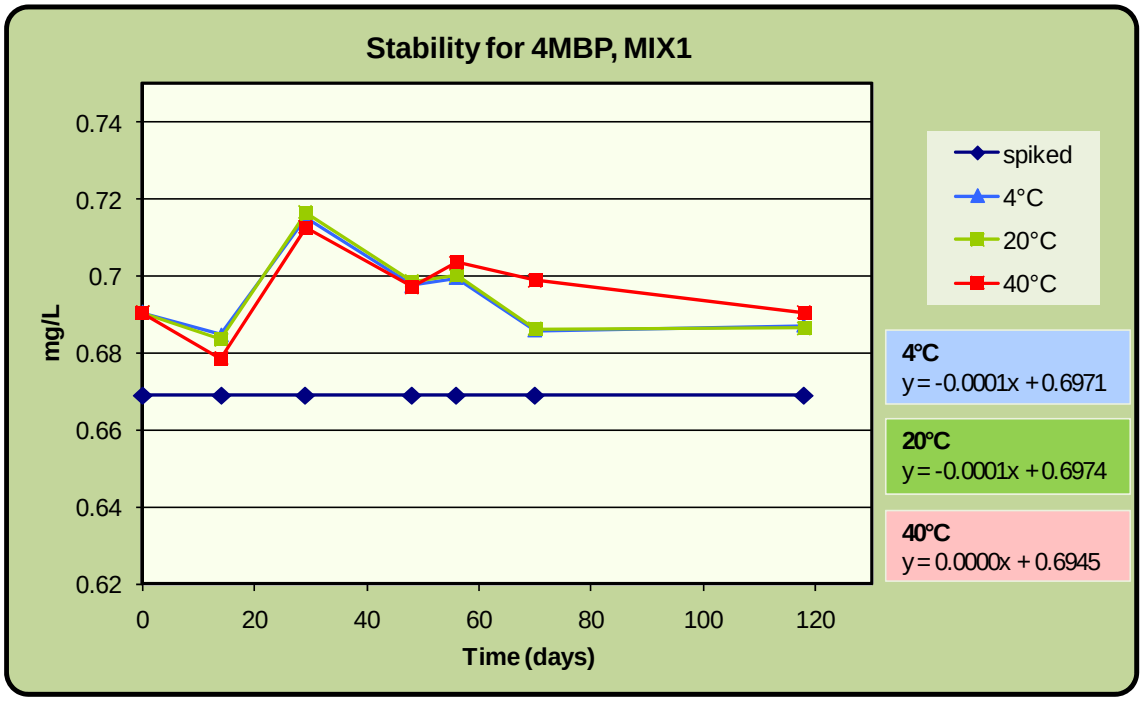


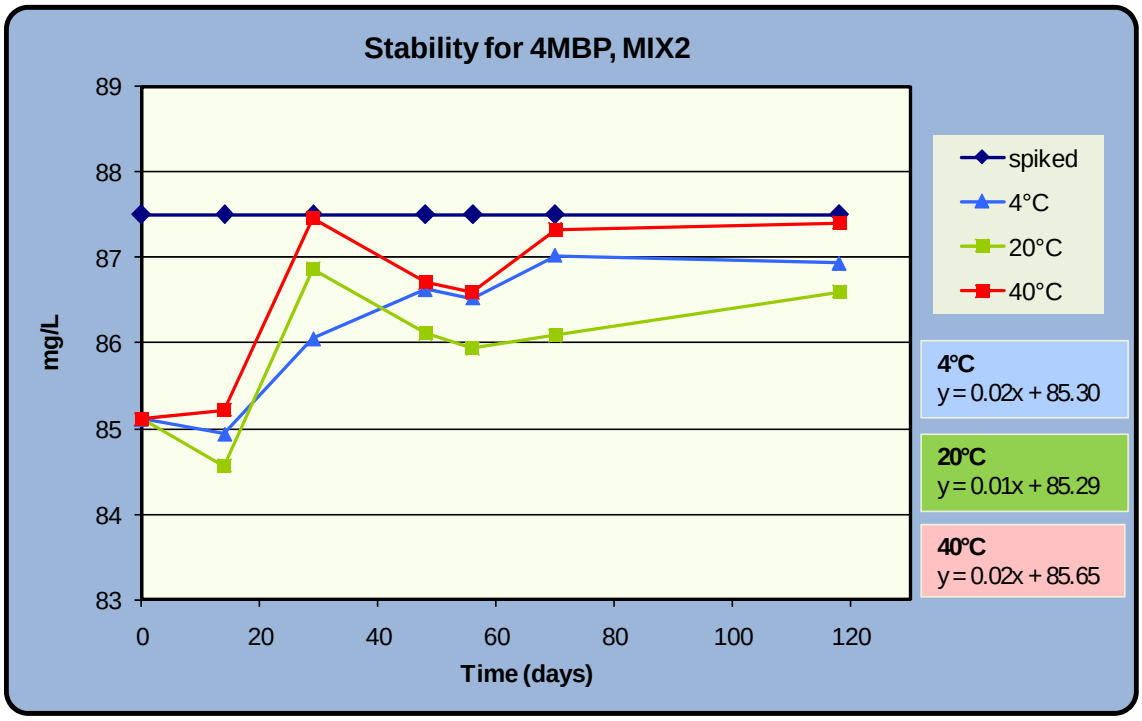
Annex 8 (continue): Results of the stability study



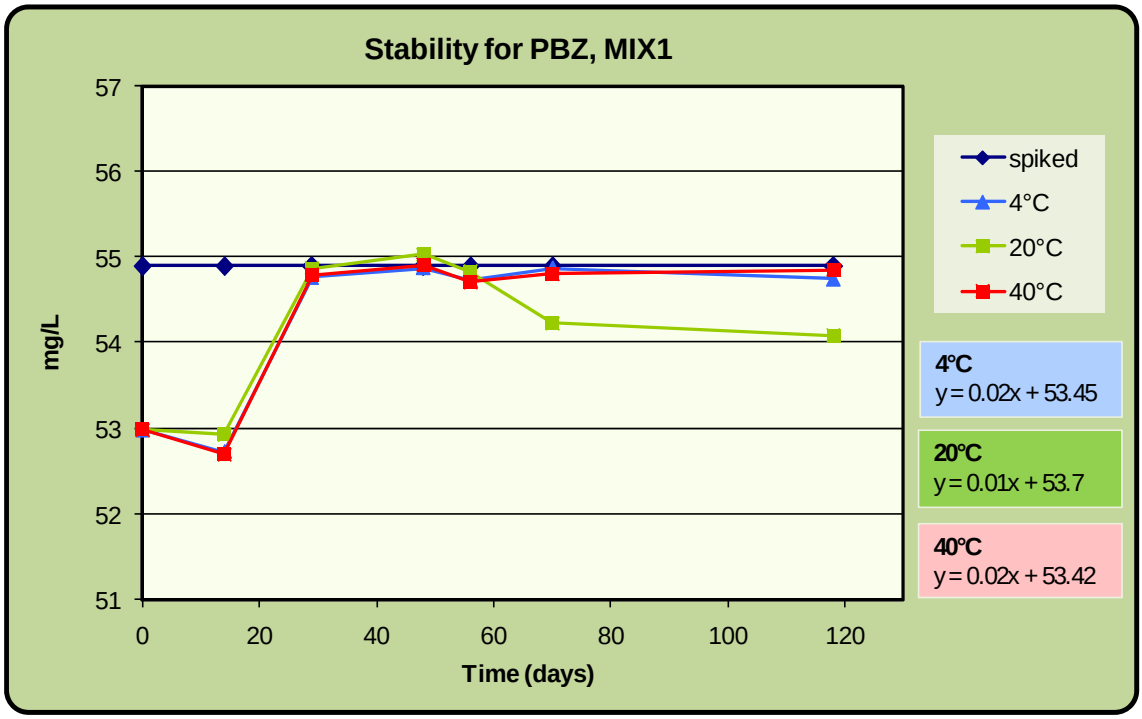


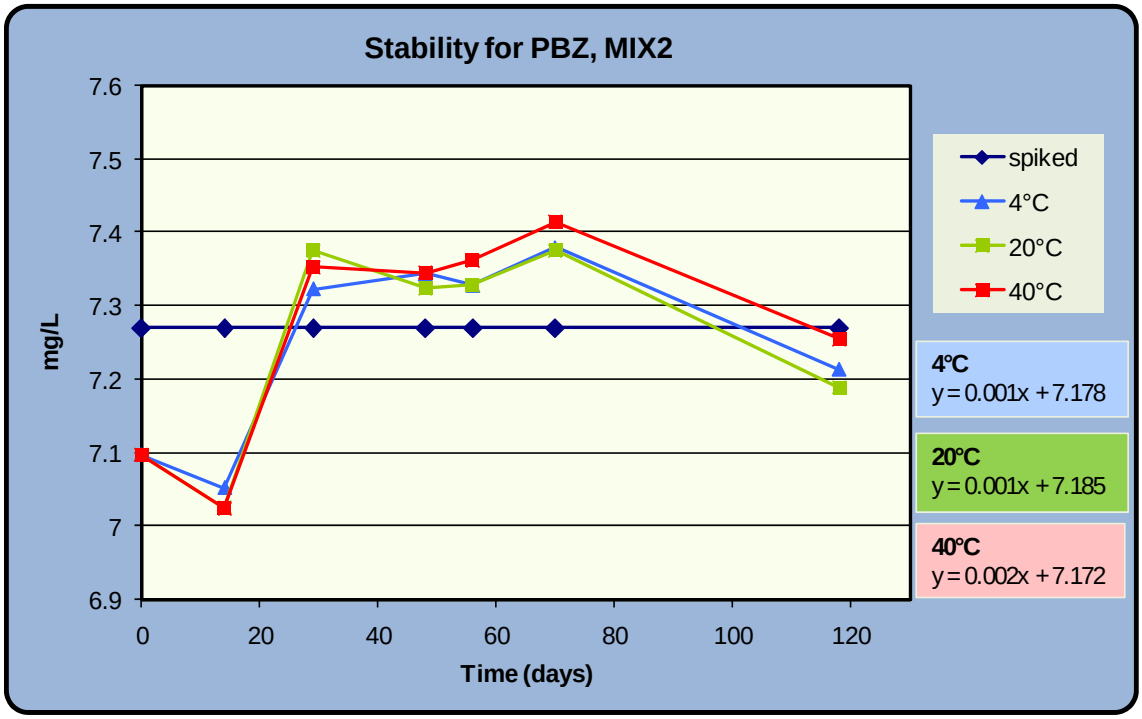
Annex 9 (continue): Results of the stability study



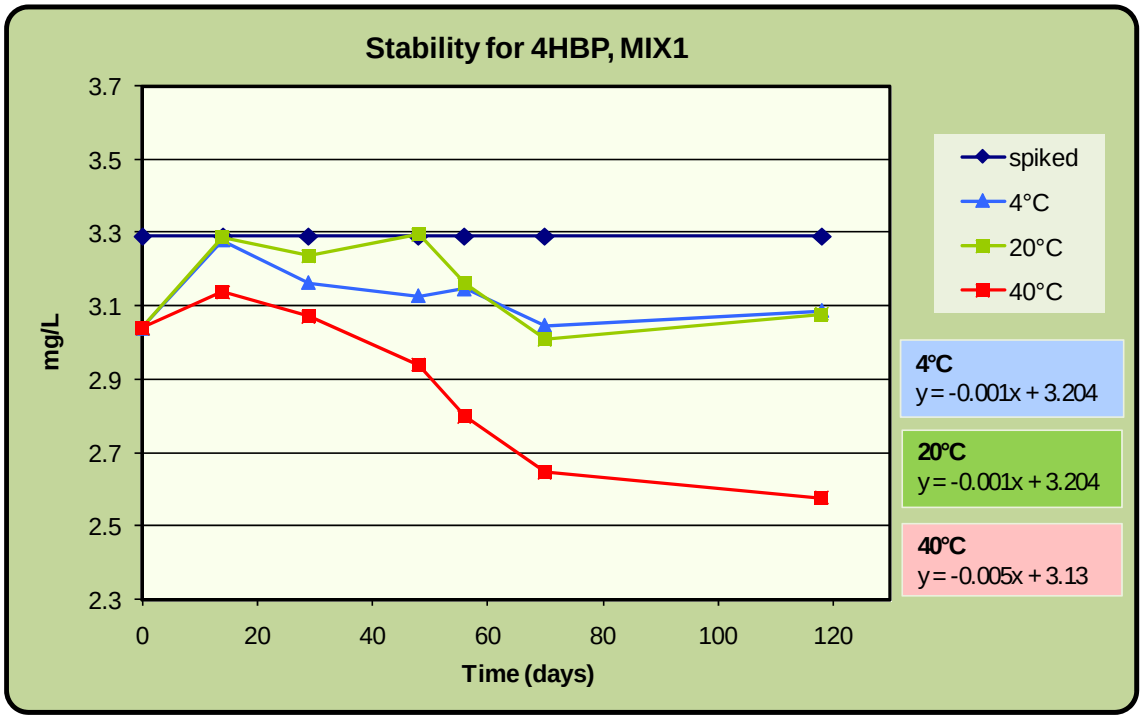


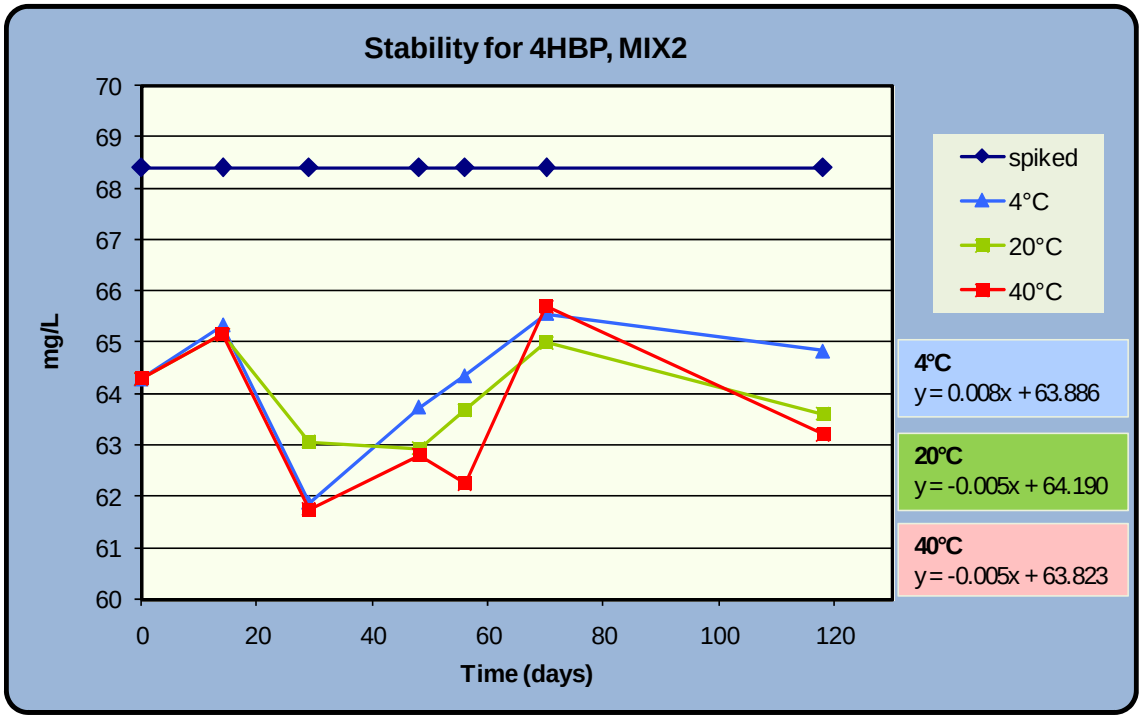
Annex 8 (continue): Results of the stability study



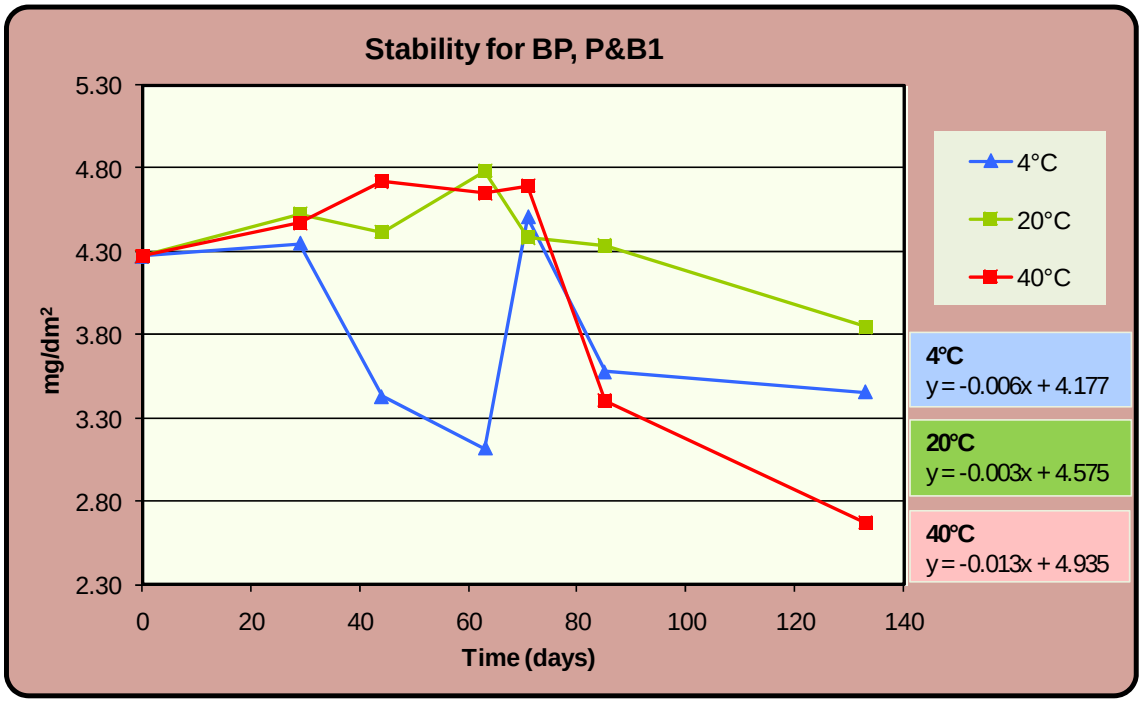


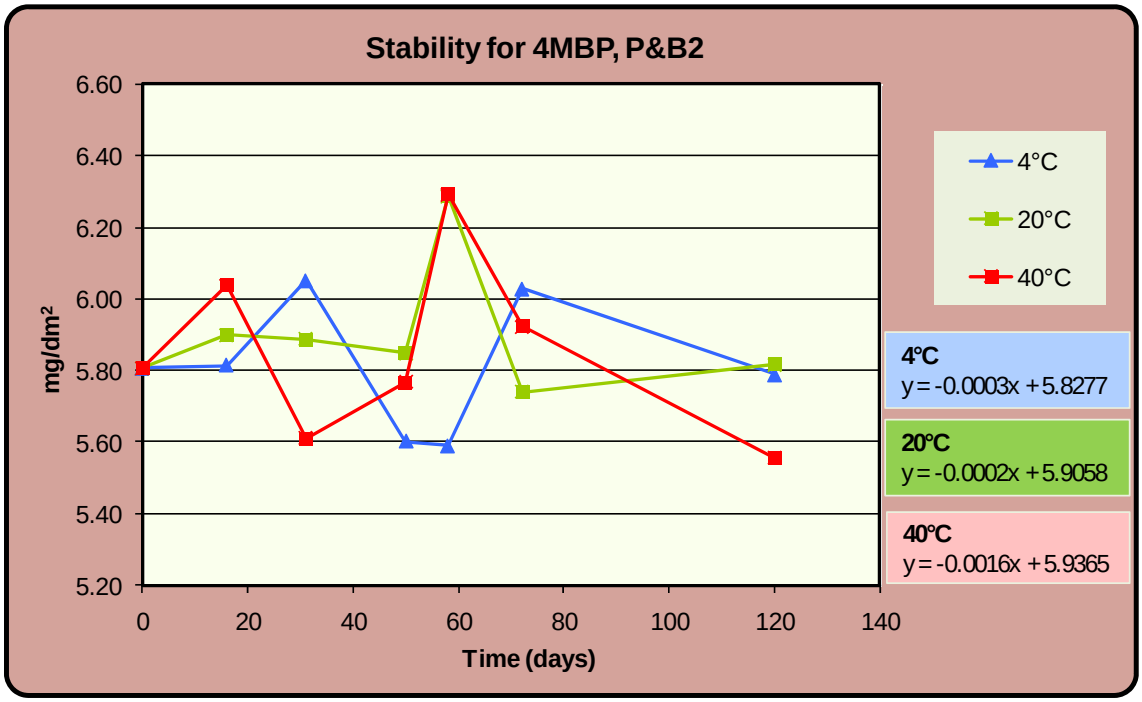
Annex 8 (continue): Results of the stability study





Annex 8 (continue): Results of the stability study





Annex 8 (continue): Results of the stability study

	reference value (mg/kg)	test condition	intercept (b0)	slope (b1)	s(b1)	t(α=0.95,g=5)·s(b1)
BP MIX1	93.6	**4°C**	93.618	**0.00750**	0.0053	**0.0136**
		20°C	94.072	**-0.01275**	0.0109	**0.0281**
		40°C	93.615	**0.00817**	0.0056	**0.0143**
BP MIX2	0.546	**4°C**	0.541	**-0.00006**	0.0001	**0.0003**
		20°C	0.544	**-0.00006**	0.0001	**0.0003**
		40°C	0.543	**-0.00007**	0.0001	**0.0003**
ITX MIX1	32.3	**4°C**	31.994	**0.00362**	0.0016	**0.0041**
		20°C	32.125	**-0.00060**	0.0017	**0.0045**
		40°C	31.985	**0.00353**	0.0016	**0.0042**
ITX MIX2	1.22	**4°C**	1.207	**0.00014**	0.0001	**0.0003**
		20°C	1.208	**0.00009**	0.0001	**0.0002**
		40°C	1.206	**0.00022**	0.0001	**0.0003**
4MBP MIX1	0.669	**4°C**	0.697	**-0.00006**	0.0001	**0.0003**
		20°C	0.697	**-0.00006**	0.0001	**0.0003**
		40°C	0.695	**0.00003**	0.0001	**0.0003**
4MBP MIX2	87.5	**4°C**	85.296	**0.01830**	0.0050	**0.0130**
		20°C	85.287	**0.01277**	0.0063	**0.0162**
		40°C	85.646	**0.01878**	0.0077	**0.0198**
PBZ MIX1	54.9	**4°C**	53.447	**0.01673**	0.0078	**0.0202**
		20°C	53.696	**0.00931**	0.0091	**0.0233**
		40°C	53.421	**0.01741**	0.0078	**0.0201**
PBZ MIX2	7.27	**4°C**	7.178	**0.00147**	0.0013	**0.0034**
		20°C	7.185	**0.00126**	0.0015	**0.0039**
		40°C	7.172	**0.00193**	0.0015	**0.0037**
4HBP MIX1	3.29	**4°C**	3.159	**-0.00066**	0.0009	**0.0023**
		20°C	3.204	**-0.00098**	0.0013	**0.0033**
		40°C	3.130	**-0.00508**	0.0010	**0.0026**
4HBP MIX2	68.4	**4°C**	63.886	**0.00826**	0.0135	**0.0348**
		20°C	64.190	**-0.00475**	0.0098	**0.0252**
		40°C	63.823	**-0.00476**	0.0168	**0.0432**

Annex 8 (continue): Results of the stability study

	reference value (mg/dm^2)	test condition	intercept (b0)	slope (b1)	s(b1)	t(α=0.95,g=5)·s(b1)
BP P&B1	4.27	**4°C**	4.177	**-0.00594**	0.0056	**0.0143**
		20°C	4.575	**-0.00342**	0.0026	**0.0067**
		40°C	4.935	**-0.01332**	0.0066	**0.0170**
4MBP P&B2	5.81	**4°C**	5.828	**-0.00032**	0.0021	**0.0053**
		20°C	5.906	**-0.00015**	0.0020	**0.0053**
		40°C	5.936	**-0.00161**	0.0028	**0.0072**

European Commission

EUR 24550 EN – Joint Research Centre – Institute for Health and Consumer Protection

Title: Report of the interlaboratory comparison organised by the EU Reference for Laboratory Food Contact Material: ILC02 2009- Bisphenol A in 50% aqueous ethanol (milk simulant) - Laboratory performance and precision criteria of a harmonised method

Author(s): S. Bratinova, S. Valzacchi, M. Multari, S. Spalla and C. Simoneau

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Abstract

This report presents the results of the ILC of the EURL-FCM which focused on the screening for photoinitiators (PI) content in paperboard as well as in a solvent. The test materials used were two types of paperboard supplied by the industry, containing benzophenone (BP) and 4-methylbenzophenone (4MBP) and mixtures of five PIs in a solvent (acetonitrile) prepared in-house. The general aim of the exercise was to assess the proficiency of the official control laboratories and consequently the participants were free to use an analytical method of their choice. There were 36 participants to whom samples were dispatched (26 NRLs + 10 official national control laboratories from Germany and Spain) 32 of which submitted results. The homogeneity and stability studies were performed by the EURL-FCM laboratory. The assigned values were obtained after applying robust statistics to the results of the participants. 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 solvent. For the PI paperboard 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.

As a conclusion of the ILC exercise on the quantification of photoinitiators in the paperboard and the solvent, this ILC showed that: 1) The participation in the ILC was satisfactory regarding the number of the participating labs. Thirty-two laboratories submitted their results. From the EURL-NRL network 22 laboratories out of 25 reported results. 3 NRLs laboratories did not send any results. 2) For the network of NRL-FCM the outcome from the participation could be regarded as satisfactory as there are about 84% satisfactory results (262 out of 312) and only 3 NRLs did not participated out of 25

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