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Report of an inter-laboratory comparison from the European Reference Laboratory for Food Contact Materials:

ILC 003 2013 – Food Contact Surface Area of Kitchen Utensils

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Report of the inter-laboratory comparison

ILC03 2013 – Food Contact Surface Area of Kitchen Utensils

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Anja Mieth and Eddo Hoekstra

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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 EU Reference Laboratory for Food Contact Materials (EURL-FCM). One of its core tasks is to organise inter-laboratory comparisons (ILCs) among appointed National Reference Laboratories (NRLs).

This report presents the results of the ILC which focused on the determination of the food contact surface area of kitchen utensils.

The general aim of the exercise was to assess the capability of official control laboratories to measure the food contact surface area of kitchen utensils and to compare the most common approaches for the determination of the surface area in terms of reproducibility and trueness.

The determination of the surface area is an essential step in measurements for overall and specific migration. For plastic materials, the overall migration limit is set to 10 mg per dm^2 of food contact surface (see Regulation (EU) 10/2011 Art. 12 (1) [1]). Hence, migration results are expressed in mg/dm^2 food contact surface. Specific migration results shall be expressed in mg/kg food applying the real surface to volume ratio in actual or foreseen use. In case of kitchen utensils, it is difficult to estimate the quantity of food that will be in contact with the sample. Therefore, the value of migration shall be expressed in mg/kg applying a surface to volume ratio of 6 dm^2 per kg of food (Regulation (EU) 10/2011 Art. 17 (1+2b) [1]). In conclusion, specific migration values are first calculated in mg/dm^2 food contact surface and later transferred into a value in mg/kg food, so again the food contact surface area is needed for the expression of results. Therefore, an exact and reproducible determination of the food contact surface area is required.

In contrast to this need, the ILC01 2012 highlighted measurement in the determination of the surface area of kitchen utensils. The results submitted for the food contact surface area of a melamine spoon sample ranged from 0.73-1.99 dm^2 [2]. As the reasons for the broad distribution of results in the ILC01 2012 were unclear, it was decided to run an inter-laboratory comparison in order to figure out whether the approaches that were used for the determination of the surface area were unsuitable or whether the performance of the laboratories was unacceptable.

Standardised methods are not available for the determination of the food contact surface area. To find out which methods were in use by the laboratories, a survey was launched in February 2013 amongst the NRLs. 14 NRLs replied. The four approaches that were mentioned most often thereby were chosen for the ILC03 2013. These four methods were:

- calculation of the area using mathematical formulas for regular geometric shapes
- wrapping the sample in paper (cut and weigh the paper)
- wrapping the sample in aluminium foil (cut and weigh the foil)
- drawing the outline of the sample on paper (cut and weigh the paper)

As the exercise aimed on the validation of the four selected test methods, the participants were asked to follow the provided, detailed instructions to measure the surface area of the samples. To assess the feasibility of the test methods in terms of convenience, all participants were asked to fill a questionnaire.

As an additional voluntary exercise, the participants were asked to determine the envelope volume of the samples, using a 2-cm-scale and a 5-cm-scale. The "envelope volume" concept was proposed by the Council of Europe in a draft for a new resolution. It does not represent a way to determine the surface area of a kitchen article but it returns an estimated value for the amount of food that comes into contact with the article.

The test materials were five different types of plastic kitchen utensils obtained from a worldwide supplier. Homogeneity studies on width, depth, length and thickness of the samples were carried out by the EURL-FCM. They indicated sufficient sample homogeneity.

Samples were dispatched to 67 participants (30 NRLs + 37 national official control laboratories from Belgium, Germany, Italy, Spain and the United Kingdom), 63 of them submitted results for the surface area and 53 for the envelope volume.

Results showed a satisfactory laboratory performance.

Difficulties were observed for the determination of the sample height that will be foreseeably in food contact (H_f). They would not affect migration results if the migration is constant over the entire sample surface, i.e. unless the tested articles are multi-material products or have a printing on the handle.

For the determination of the surface area, the trueness and precision of the methods depended on the sample shape. "Calculation" generated accurate results for all sample types. "Drawing the shape" was most convenient and provided accurate results for flat samples that had a negligible thickness. For round-shaped samples, "wrapping in aluminium foil" was most convenient but it overestimated the surface area. The trueness might be improved if a thicker aluminium foil is used. "Wrapping in paper" generated accurate results for flat samples and simple geometric shapes. For round-shaped samples, the surface area was overestimated as well. In general, paper was less convenient for wrapping than aluminium foil.

With respect to the final migration result, the reproducibility standard deviations obtained for all four approaches were acceptable considering that the migration measurement itself can be affected by uncertainties of similar levels as those of the determination of the surface area.

The determination of the envelope volume was convenient. It required only the determination of H_f and the measurement of the depth and width of the sample. Despite this, some difficulties were observed regarding the measurement of the sample dimensions. The determination of the envelope volume is a new approach and most of the laboratories performed this determination for the first time. The laboratory performance is expected to improve with more training.

2. Introduction

ILC studies are an essential element of laboratory quality assurance and allow individual laboratories to check their analytical performance while providing them objective standards to perform against.

It is one of the core duties of the EU Reference Laboratories to organise inter-laboratory comparisons, as stated in Regulation (EC) No 882/2004 of the European Parliament and of the Council [3].

In accordance with the above requirements the European Reference Laboratory for Food Contact Materials (EURL-FCM) organised inter-laboratory comparison tests for the network of appointed National Reference Laboratories (NRLs) in 2013.

3. Scope

The objectives of this ILC were:

1. to assess the laboratory performance of the appointed NRLs and guest laboratories to determine the food contact surface area of kitchen utensils;

2. to gain data on reproducibility and trueness for the four most popular approaches to determine the surface area of kitchen utensils;

3. to assess the feasibility of these four most popular approaches and to find out which method is best suited for certain types of kitchen utensils.

In addition, the envelope volume of the samples was determined in an optional exercise. This is a new approach, developed by the Council of Europe and foreseen for the migration testing of metal kitchen utensils. NRLs and guest laboratories could voluntarily participate. The aims for this exercise were:

1. to assess the laboratory performance;

2. to gain data on the reproducibility of the method.

The assessment of all measurement results was undertaken on the basis of requirements laid down in international standards and guidelines ([4], [5], [6], [7]).

4. Time frame

The ILC03 2013 was launched in April 2013. Invitation letters were sent by e-mail to the NRLs and guest laboratories on the 5th and 10th of April 2013, respectively (0). Laboratories were invited to fill in a letter of confirmation of their participation (14.1.2).

Beforehand, a survey was launched among the NRLs in February 2013 to find out which methods for the determination of the surface area were currently in use by the laboratories.

The samples were purchased in March 2013 from a worldwide supplier. Homogeneity tests were then carried out in March and April 2013.

The samples were dispatched to the participants on the 9th and 12th of April, together with two letters (14.1.3, 14.1.5), instructions for the determination of the surface area and the envelope volume (14.1.6, 14.1.7), a print copy for the compilation of results (14.1.8) and a print copy of the questionnaire (14.1.9). An electronic Excel file, where the results should be inserted, and an electronic Word file with the questionnaire were sent by e-mail on the 12th and 15th of April 2013. The participants were asked to confirm the sample receipt and fill in the respective letter of confirmation (14.1.4).

The deadline to report the results was set to the 10th of May 2013.

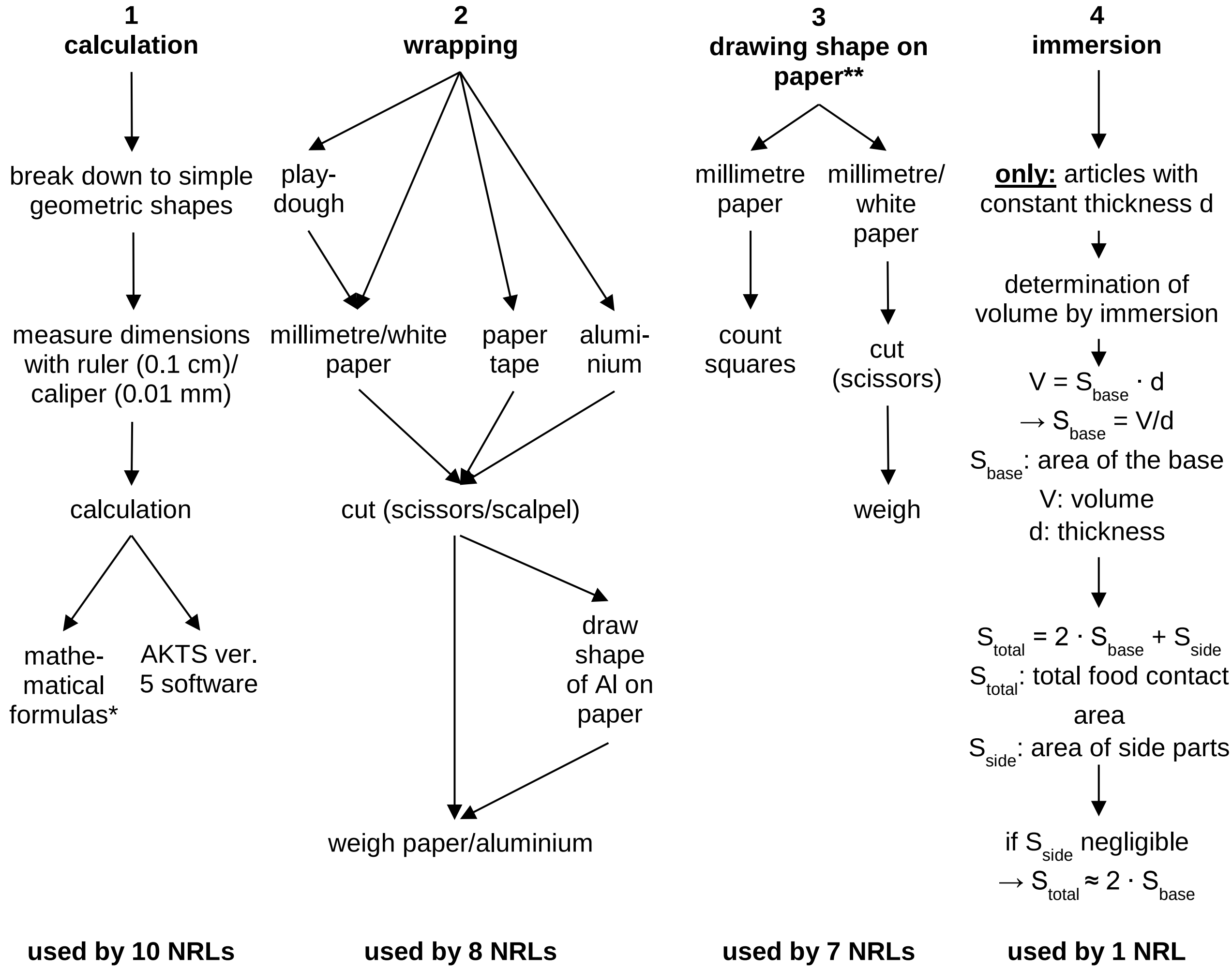
5. Selection of test methods included in the ILC

5.1. Results of the survey among all NRLs from February 2013

In the survey launched in February 2013, the NRLs were asked to provide protocols/descriptions of the methods they used to determine the food contact surface area of kitchen utensils. 14 NRLs replied. All but one declared to use two or more different analytical methods. Depending on the type of sample, they chose the most appropriate one.

5.1.1. Mentioned approaches to determine the surface area

Among all procedures provided, four different general concepts could be distinguished (see Figure 1). These were 1) calculation of the surface area via mathematical formulas for regular geometric shapes, 2) wrapping the article in paper, aluminium foil or tape, 3) drawing the outline of the article on paper, and 4) determination of the volume by immersion.



* +5% measurement uncertainty

** if necessary, the sample is cut into smaller pieces and each of the single pieces is outlined on paper to ensure that the outline is representative for the real surface.

Figure 1 Summary of survey from February 2013 on NRL methods for the determination of the surface area

Calculation of the surface area using mathematical formulas

Field of application:

This approach is mostly used for regular shaped samples like plates, cups, bowls and bottles, but also for whisks and spoons. 10 NRLs mentioned using this method. One of them exclusively used calculation, another one emphasized that calculation was always the preferred procedure and that it is used whenever it was possible.

Principle of the determination:

The sample is broken down to several regular geometric shapes (e.g. cylinders, rectangular solids, truncated cones). For each of this regular geometric shapes, the surface area is calculated. The total area is the sum of all these single parts. If the surface of an irregularly shaped sample shall be determined, it can be divided into different trapezoids.

To measure the dimensions of the sample that are needed for calculation, rulers or tape measures (0.1 cm accuracy) and/or calipers (0.01 mm accuracy) are used.

Calculations are done with mathematical formulas for the particular geometric shape. An overview of several formulas is given in the "Guidelines on testing conditions for articles in contact with foodstuffs (with a focus on kitchenware)" (EUR 23814, 1st Edition, 2009) 48 [8]. As additional tools, two websites (www.analyzemath.com [9], www.javascriptzoeker.nl/javascripts/javascripts.php?action=tel&id=240 [10]) as well as the migration modelling software AKTS-SML [11] were mentioned. They provide calculation programs for the surface area of some regular geometric shapes.

As the articles tested usually do not have a perfect geometric shape, the value obtained for the surface area will be afflicted with a certain measurement error. One laboratory estimated this measurement error to a default value of 5% that is taken into account for every sample.

Wrapping the sample in paper, aluminium foil or tape

Field of application:

8 NRLs declared to use any type of wrapping to determine the surface area. Most of them use it for irregularly and curved shaped samples (e.g. spoons, forks, ladles, spatulas).

Principle of the determination:

The sample is wrapped in white paper, millimetric paper, paper tape or aluminium foil. Wrapping is done as tight as possible. Excess wrapping material is removed using a scalpel or scissors. Afterwards, the sample is unwrapped and the wrapping material is weighed. Knowing the surface weight (grammage) of the paper, aluminium foil or paper tape, the surface area of the sample can be calculated.

As an alternative to direct weighing of the aluminium foil which was used for wrapping, one NRL declared to redraw the shape of the aluminium foil on paper and cut and weigh this piece of paper.

For difficult shapes, another NRL described how to create a negative or mould of the sample surface with a putty-like substance (play dough). This pliable mould is then lined with paper. The paper needed for lining is weighed. Knowing its grammage, the surface area of the sample can be determined.

One of the NRLs expressed to prefer wrapping in aluminium foil and directly weighing the aluminium foil for the following reasons:

a. Aluminium foil is more flexible than paper and so it is easier to cover the contour of the article;
b. The surface weight of the aluminium foil is very consistent;
c. No problems with humidity occur in comparison to paper.

Drawing the sample outline on paper

Field of application:

This method is used for flat samples, but also for curved samples with irregular shapes like spoons, ladles, spatulas, forks and tongs. It was mentioned by 7 NRLs but one them claimed to hardly use it and to prefer calculation or wrapping instead.

Principle of the determination:

The sample is placed on millimetre paper or white paper and its outline is drawn on the paper. To make sure that the outline is representative for the real surface, the sample can be cut into smaller pieces and the outline of each of the single pieces is drawn on paper. To determine the surface area, either the squares on the millimetre paper are counted or the drawings are cut and weighed. If the grammage of the paper is known, the surface area can be calculated.

Instead of drawing the shape of a sample, one laboratory described to photocopy items if appropriate. Then the photocopy is cut and weighed.

Immersion and determination of the volume (only applicable to samples with constant thickness)

Field of application:

A fourth approach was proposed by a single NRL. It refers only to samples with a constant thickness. For these samples, it is possible to calculate the surface area if the sample thickness and the volume of the sample are known.

Principle of the determination:

The general formula for the volume of a solid figure with <u>constant thickness</u> is:

$$V = S_{base} \cdot d$$

V: volume of the solid figure
S_{base}: area of the base of the solid figure
d: thickness of the solid figure

Hence, the area of the base can be calculated using:

$$S_{base} = \frac{V}{d}$$

The thickness d can be easily measured with a ruler or caliper. The volume V is determined by immersing the sample in water. The amount of water displaced thereby is equal to the volume of the sample. Before the immersion, the sample is cut into small pieces that fit into a 100 ml graduated cylinder with small diameter (2,5 cm). The graduated cylinder is filled with water up to a certain level (V_1, ml), then the sample pieces are inserted and completely submerged and the new water level (V_2, ml) is recorded. The difference V_2-V_1 is equal to the volume of the sample. To reduce the measurement error, a balance can be used instead of reading the meniscus. The balance is tared with the dry and empty cylinder. A certain amount of water is filled into the cylinder (level 1) and its weight is determined (mass 1). Then, the sample pieces are added and the new meniscus is marked (level 2). The cylinder is emptied, dried, refilled with water up to the mark of level 2 and weighed again (mass 2). In both fillings (m1 and m2), it is carefully ensured not to have water on the cylinder surface above the meniscus. The mass difference (m_2-m_1) refers to the amount of water displaced by the sample and hence to the sample volume.

It must be regarded that S_{base} does not need to be equivalent to the total food contact area. If top, bottom and side parts of the article come into contact with food (as it is the case for e.g. kitchen spatulas, spoons), the total food contact area arises from:

$$S_{total} = 2 \cdot S_{base} + S_{side}$$

S_{total}: total food contact area
S_{base}: area of the base of the sample
S_{side}: area of the sample side parts/walls

If the side parts do not significantly contribute to the total food contact area, the formula can be simplified as follows:

$$S_{total} \approx 2 \cdot S_{base}$$

5.1.2. Other possible approaches

Two more approaches for the determination of the surface area are presented here that were not mentioned by the NRLs. These are the use of a 3D scanner and creating a 3D-model of the sample using a computer aided-design (CAD) software.

3D scanners

3D scanners often create a 3D point cloud of the sample surface. For the scanning process itself, different techniques are used. Appropriate software (e.g. CAD software) joins all data points to create small triangles and calculates the area of each triangle. The sum of all these areas is approximately equal to the real surface area of the scanned sample. The result gets more accurate, the more data points are collected and the smaller the resulting triangles are as they then can fit the real surface area better.

Technical drawing (CAD software)

With the help of computer aided-design (CAD) software (e.g. AutoCAD), it is also possible to create a 3D-model of the sample starting from a technical drawing. As the model is created step by step from geometric figures, the software is able to calculate the surface area of the final model.

5.2. Selection of methods for the Inter-laboratory Comparison Exercise

As declared above, this Inter-laboratory Comparison Exercise aimed on a proficiency testing as well as a comparison of methods. It had been decided to select those approaches which were frequently used and well-established in the NRLs.

5.2.1. Selected methods for the determination of the surface area

From the methods that were presented in Figure 1, the following ones were chosen:

1) Calculation
2) Wrapping in paper and direct weighing
3) Wrapping in aluminium foil and direct weighing
4) Drawing the sample outline on paper

5.2.2. Reasons for selection

All variants of methods presented in section 5.1.1 were in use by the NRLs and all of them might be suitable for certain types of samples. Not all of them could be tested in this exercise but at least one method representative for each of the general concepts was chosen to allow a representative method comparison. That means, calculation using mathematical formulas for regular geometric shapes, wrapping, drawing the outline and determination of the volume (immersion) should be performed.

Despite this, the determination of the volume (immersion) was not included in this inter-laboratory comparison. The main reason for this decision was that it requires a constant sample thickness and only certain parts of the samples that were delivered to the NRLs fulfilled this condition. In addition, all samples would have to be cut into small pieces. As the samples were made of polyamide, their cutting would have required special efforts.

For calculation, the laboratories were allowed to choose themselves whether to use suitable software to ease the calculation or to do all calculations manually using appropriate mathematical formulas as listed for example in the "Guidelines on testing conditions for articles in contact with foodstuffs (with a focus on kitchenware)" (EUR 23814, 1st Edition, 2009) [8].

As presented in section 5.1.1, four different variants of wrapping were mentioned by the NRLs whereupon wrapping in paper and wrapping in aluminium were most commonly used. Therefore, one of these methods should have been selected for the inter-laboratory comparison.

As paper and aluminium differ in their flexibility and their characteristics of tearing and crinkling, it was decided to perform the test with both of them.
For quantification, direct weighing of the paper as well as the aluminium foil should have been applied. Direct weighing is easier to perform, needs less analytical steps and hence provides less sources of error compared to redrawing the shape of the aluminium foil on paper and weighing this piece of paper.

For drawing the outline on paper, the laboratories were also advised to cut the drawing along the outline and weigh it instead of counting the squares. The same reasons apply as mentioned above. Of course, this analytical approach requires a constant paper surface weight (grammage). If this cannot be ensured and problems concerning paper homogeneity appear, it is self-evident that direct weighing cannot be proceeded. In this case, the surface area of the drawing should be determined by counting squares on the millimetre paper.

As an additional approach, it was suggested to include 3D scanners in this inter-laboratory comparison. They may offer a quick, convenient and quite accurate determination of the surface area, even for irregular shaped samples. Unfortunately, these devices are quite expensive and the NRLs could not be provided with appropriate systems. In addition, the use of such scanners also needs training.

The EURL-FCM is doing some first experiments with such an instrument. It is foreseen to include a more intense study on the use of 3D laser scanners in the follow-up exercise in 2014. The purpose of this study will be to check whether 3D laser scanners are convenient and more precise or not.

For the ILC03 2013, the German Bundesinstitut für Risikobewertung entrusted an external company with the 3D laser scanning of the samples sent within this ILC and submitted the results to the EURL-FCM. These results were included in the data evaluation.

5.3. Determination of the foreseeable food contact part of a kitchenware article

Before the surface area can be determined, it must be clarified which part of the sample will foreseeably be in contact with food. Kitchen utensils normally consist of a part necessarily in contact with food (e.g. the elliptic part of a spoon), a part which might come in contact with food (usually the lower part of the handle) and a part which will not be in contact with foodstuffs because it serves as a handle. The difficulty is to define the part that might come into contact with food.

5.3.1. Recommendation expressed in the EURL-FCM Guideline on kitchenware

The "Guidelines on testing conditions for articles in contact with foodstuffs (with a focus on kitchenware)" (EUR 23814, 1st Edition, 2009) [8] state:

"If the article is a tableware or kitchenware (spatulas, spoons ladles, etc.) and can be immersed or is intended to be immersed partially or totally, then immersion should be used as a means for testing the migration. In such cases the volume of simulant should be proportional to the area required to cover the utensil. It should respond to the requirement of <u>covering the utensil entirely including 2 cm of the handle</u>." (see EUR 23814, 1st Edition, 2009, p. 39, 8.3.5) [8]

That means the food contact part of every kind of kitchen utensils always includes 2 cm of the handle. This is a default value. Therefore, it does not necessarily reflect the real use conditions.

5.3.2. Proposal by the Council of Europe

An advanced approach was proposed by the Council of Europe. Actually, it was not meant to be used for the determination of the food contact area itself. It is only part of another concept where an envelope volume of the sample is determined (see Instructions part II). The procedure is as follows.

At first, the total length of the sample (incl. the handle) (H_{total}) and the length of the handle (H_{handle}) are measured. If the handle is not clearly separated, a default length of 1/3 of H_{total} is assigned. Then, the part reasonably in contact with food (H_r) is determined. It results from: $H_r = H_{total} – H_{handle}$.

After this, the part which is necessarily in contact with food (H_n) is measured and the part that is probably in contact with food (H_p) is calculated. It arises from: $H_p = H_r – H_n$.

If $H_p \leq 0.5\ H_n$, the height (H_f) up to which the sample will foreseeably be in contact with food is considered to be equal to H_r. Otherwise, a value of 2/3 of H_r is assigned for H_f. H_f defines the sample part which should be regarded for migration testing.

Examples are given in Figure 2.

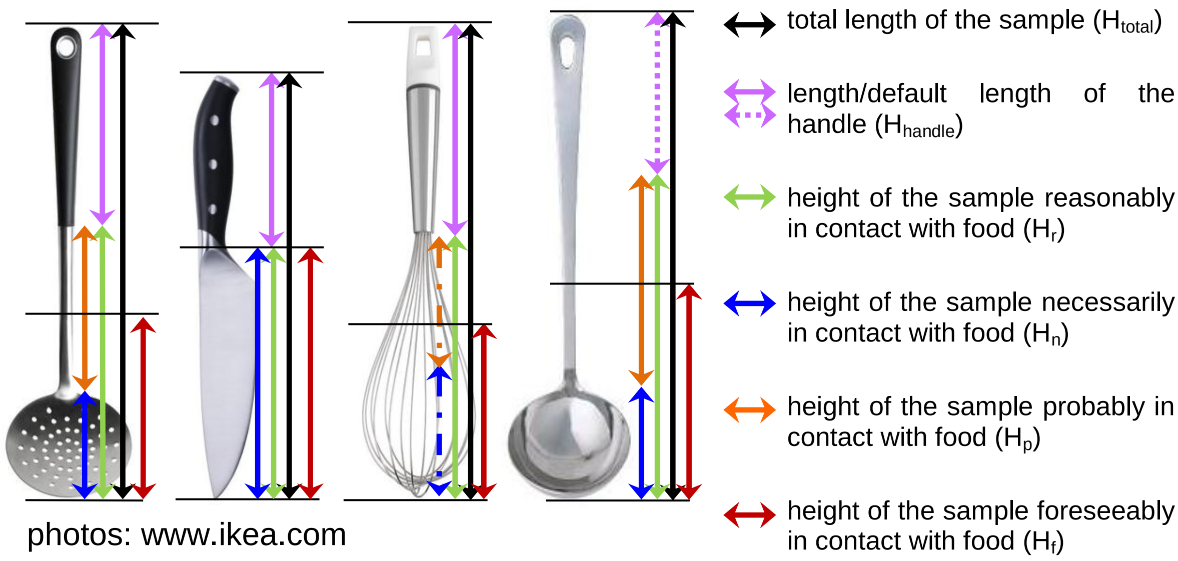


Figure 2 Determination of the sample height foreseeably in contact with food (H_f)

5.3.3. Method of choice

Compared to the recommendation laid down in the EURL-FCM Guideline on kitchenware (see 5.3.1, [8]), the CoE approach (see 5.3.2) seemed to be more flexible and applicable to all kind of kitchen utensils. The obtained results were supposed to better reflect real use conditions and be more reasonable. Therefore, the CoE approach was selected for this ILC.

5.4. Voluntary exercise – Determination of the Envelope Volume

The "envelope volume" concept was proposed by the Council of Europe in a draft for a new resolution. It does not represent a way to determine the surface area of a kitchen article but it is useful in assessing the specific migration of samples.

The envelope volume of a kitchen utensil is an estimated value for the amount of food that comes into contact with the article. Limits for the specific migration always refer to mg/kg foodstuff. According to Article 17 of the Regulation 10/2011, "*specific migration values shall be expressed in mg/kg applying the real surface to volume ratio in actual or foreseen use"*. This implies that the amount of food is known with which the article will be in contact. If this amount is unknown (e.g. in case of kitchen utensils), usually a value of 6 dm^2 per kg foodstuff is assumed. This is a default value and does not represent real use conditions for all types of samples. The envelope volume offers the possibility to obtain a more reasonable value for the amount of food in contact with the article.

According to the draft of the Council of Europe, the principle of the determination is as follows. The dimensions (depth x, width y, height z) of the sample part that will be in contact with food are determined on a 5-cm-scale. The envelope volume is the product of $x \cdot y \cdot z$ (in cm^3). Then, the reference weight W_{ref} (kg) results from: envelope volume (cm^3)/1000.

To determine the specific migration SM of a substance from the sample, the migrated mass M of this substance is divided by the reference weight: $SM = M/W_{ref}$

Reasons for selection

This is a new approach, so there are no data yet about the reproducibility and performance of laboratories. It is of the same importance as the determination of the surface area. Its implementation and use would allow the expression of results for the specific migration without the need to determine the surface area. It has a direct influence on migration results and therefore a high reproducibility of results is required.

For this ILC, it was decided to determine the envelope volume on a 5-cm-scale as described in the draft of the Council of Europe. In addition, also a 2-cm-scale was used to check which scale returns stricter but still reasonable results with respect to "worst case" conditions.

6. Test materials

6.1. Preparation

Samples of five different kitchen utensils were purchased from a worldwide supplier (see Table 1). All samples were labelled and then directly sent to the participants. No further sample preparation was done. Purchase and labelling were done by the EURL-FCM.

Table 1 Test materials

Exercise	Name	Sample
ILC 03 2013 Surface Area and Envelope Volume	A	slotted kitchen spatula
	B	fork
	C	oval spoon
	D	rectangular spoon
	E	cooking tweezers/tongs

6.2. Homogeneity assessment

The samples were tested for homogeneity by the EURL-FCM in accordance with ISO 13528:2005(E) Annex B [4]. As there was no opportunity to check the homogeneity of the surface area itself, other parameters like thickness, width, length and height of the samples or specific sample parts were measured to ensure homogeneous sample dimensions for all test items. Ten randomly selected test specimens of each sample A-E were analysed. For each test specimen, at least five dimensions were measured with a calliper. The results are given in 14.2.

The standard deviations for all measured dimensions were below 0.23 mm, corresponding to coefficients of variation in the range of 0.15-4.5 %. According to these results, the sample homogeneity can be regarded as sufficient.

6.3. Distribution

The sample kits were dispatched to the participants by the EURL-FCM in April 2013. Each participant received a padded envelope containing:

a) Five samples labelled with A-E;

b) The accompanying letters with instructions on sample handling, analysis and reporting of results (14.1.3, 14.1.5-14.1.7);

c) The form to confirm the sample receipt (14.1.4);

d) The forms for reporting the results and the questionnaire in non-electronic format (14.1.8, 14.1.9).

In addition, each participant received an e-mail sent by the EURL-FCM containing the respective laboratory code, the Excel file for reporting the results and the Word file for filling in the questionnaire.

7. Instructions to participants and requested measured parameters

Detailed instructions were given to all participants in the letters that accompanied the samples (14.1.5-14.1.7).

First of all, the laboratories were asked to determine and report the sample height (H_f) that would come in foreseeable contact with food for each of the five samples. Detailed instructions how to determine this value were provided (14.1.6). This height marked the foreseeable food contact part of the sample.

Then, the laboratories were asked to determine and report the surface area of the foreseeable food contact part (i.e. up to the height that they had determined in the first step) of all five samples. Determinations should be done with four different approaches, following the test protocols described in the provided instructions part I (14.1.6). The participants were asked to apply all four methods to all samples. These four methods were:

- calculation of the area using mathematical formulas for regular geometric shapes
- wrapping the sample in paper, cut and weigh the paper
- wrapping the sample in aluminium foil, cut and weigh the foil
- drawing the outline of the sample on paper, cut and weigh the paper

For the latter one (i.e. drawing the outline on paper), the laboratories were free to cut the samples into smaller pieces or to leave the samples uncut but they were asked to report which of the sample preparations they had chosen.

Each measurement had to be done as a single-fold determination. No replicates were asked.

For the voluntary exercise, the participants had to determine the envelope volume of all five samples on a 2-cm-scale and a 5-cm-scale, following part II of the provided instructions (14.1.7).

All results had to be reported using the unit of measure indicated in the provided Excel file for the compilation of results (and its print copy (14.1.8)).

8. Statistical evaluation of results

8.1. Assigned values

The true values for the surface area of the samples were unknown. As there were no other reference values available, the robust mean values obtained from the reported results of the participants were used as assigned values. The same applies for the sample height with foreseeable food contact. For the envelope volume, assigned values were set manually by the EURL-FCM. The chosen values based on the sample dimensions measured in the homogeneity testing carried out by the EURL-FCM.

The robust mean values were obtained using the Hampel estimator, as described in ISO/TS 20612 [5]. All calculations were done using the ProLab software [12].

The Hampel estimator is a tool of robust statistics to obtain reference values from the results of the participants of an inter-laboratory comparison test [5]. It remains viable even with more than 40% outlier laboratories [ProLab Manual]. It does not require replicates for the measured values and therefore could be applied in the present case.

It should be noted that no tests for outliers are carried out when the Hampel estimator is used. The algorithm works in a way that values which differ from the mean value by more than 4.5 times the standard deviation do not affect the calculated results [5].

8.2. Target standard deviation

The target standard deviation (σ_p) determines the limits for a satisfactory performance in an ILC test. It should be set to a value that reflects best practice for the analysis in question. The standard deviation of the reproducibility found in collaborative trials is generally considered as an appropriate indicator of the best agreement that can be obtained between laboratories. So far, the ILC03 2013 was the first inter-laboratory exercise that focused on the surface area and the envelope volume. Hence, there were no comparative test data available.

Therefore, the reproducibility standard deviation that was determined from the reported test results of the participants was set as the target standard deviation. The reproducibility standard deviation was calculated by the help of the Q-method described in ISO/TS 20612 [5].

Again, all calculations were done using the ProLab software [12].

8.3. Evaluation criteria for laboratory performance – z_U-scores

The individual laboratory performance was expressed in terms of z_U-scores (z_U) as described in ISO/TS 20612 [5].

Commonly, z-scores are used to describe the performance of laboratories. They describe the deviation of the individual laboratory result to the assigned value, standardised by the target standard deviation. A problem of z-scores is that laboratories which report values lower than the assigned value would generally obtain a better z-score than laboratories that submit values which are above the assigned value. To overcome this problem, z_U-scores were developed. They represent a modified form of z-scores. [5]

The calculation of z-scores (z) and z_U-scores (z_U) is done as follows [5]:

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

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 .

(2) $$z_U = \begin{cases} \frac{g}{k_1} \cdot z & if\ z < 0 \\ \frac{g}{k_2} \cdot z & if\ z \geq 0 \end{cases}$$

where:

z is the z-score;

g is the quality limit, here: g = 2

k_1 and k_2 are obtained by solving the following equations in an iterative procedure:

(3) $$\left(k_2 + \frac{1}{v}\right) \exp\left\{-\frac{1}{2}k_2^2\right\} = \left(-k_1 + \frac{1}{v}\right) \exp\left\{-\frac{1}{2}k_1^2\right\}$$

(4) $$\left(1 - \Phi\left(-\frac{1}{v}\right)\right)^{-1} \left(\Phi(k_2) - \Phi(-k_1)\right) = 1 - \alpha$$

The z_U-scores can be interpreted as follows:

$|z_U| \leq 2$ satisfactory result;

$2 < |z_U| \leq 3$ questionable result;

$|z_U| > 3$ unsatisfactory result.

For parameters that cannot reach negative values as it is the case in the present ILC, ISO/TS 20612 generally recommends the use of z_U-scores [5]. Therefore, they were chosen as criteria to describe the laboratory performance in the present study. For their calculation, again the ProLab software was used [12].

8.4. Test for normal distribution

All data were analysed for normal distribution by applying the Shapiro-Wilk test (α = 0.05). In addition, Kernel density plots were used to check graphically for normal distribution and to identify multi-modality in the data distributions.

The Shapiro-Wilk test and Kernel density plots were computed using the ProLab software [12].

8.5. Non-parametric tests to compare data populations

Most of the data sets were not normally distributed. Therefore, non-parametric rank tests, in particular the Mann-Whitney-Wilcoxon test (α = 0.05) and the Friedman's test (α = 0.05), were applied to compare two or more sets of depending data, respectively.

9. Results and Conclusions

9.1. Participation

Samples were dispatched to 67 laboratories (30 NRLs and 37 national official control laboratories from Belgium, Germany, Italy, Spain and the United Kingdom), 63 of them submitted results, corresponding to a percentage of participation of 94%.

46 laboratories had applied all four methods for the determination of the surface area to all five samples, as it was requested in the instructions. 17 laboratories reported values for the surface area of all samples, but they had not applied all four methods to each sample.

The percentage of participation in the voluntary exercise was also very high. 53 laboratories submitted results for the envelope volume, corresponding to a level of participation of 79%. 51 of them reported results for the envelope volume of all samples, determined on the 2-cm-scale and on the 5-cm-scale. One laboratory submitted results only for the 2-cm-scale, but for all samples. A second laboratory reported results for the 2-cm-scale and the 5-cm-scale but not for all samples, as two samples had been cut already before the envelope volume was determined.

9.2. Laboratory results and scores

The participants first had to determine the sample height H_f that will be foreseeably in food contact. This value defined the sample part that would be relevant for a migration testing. Then the surface area of this sample part should be determined using four different methods ("calculation", "wrap in paper", "wrap in Al foil" and "draw the outline"), following the test protocols provided in the sample kits (14.1.6). In the voluntary task, also the envelope volume of this sample part was determined (instructions see 14.1.7).

9.2.1. General problems that occurred

One laboratory explained that they performed the "wrapping" of the samples in paper and aluminium foil in the proper sense of the term. In conclusion, they did not remove the parts of the holes of the slotted samples A and B but this was not the intention of the provided instructions. Furthermore, one laboratory reported problems in understanding the instructions for the determination of the envelope volume. To avoid these misunderstandings, the instructions for the determination of the surface and the envelope volume should be adapted.

9.2.2. Sample height with foreseeable food contact (H_f)

A summary of the statistical data obtained for the sample height that will be foreseeably in food contact (H_f) is given in Table 2. The single results reported by the laboratories, the Kernel density plots and the obtained z_U-scores are shown in 14.3 Figure 15-Figure 19 and 14.4.1 Table 30. All values were calculated using the ProLab software, as described in the previous section.

Table 2 Summary of the statistical evaluation for the sample height with foreseeable food contact (H_f)

Method DIN 38402 A45	Sample A	Sample B	Sample C	Sample D	Sample E
Measurand	Hf	Hf	Hf	Hf	Hf
Robust Mean = Assigned Value [cm]	13.79	12.35	12.69	12.78	12.89
Robust Reproducibility = Target s.d. [cm]	0.35	0.26	0.27	0.25	0.23
Rel. Reproducibility s.d. [%]	2.50	2.11	2.14	1.92	1.74
Lower limit of tolerance [cm] ($z_U \geq -2$)	13.1	11.8	12.2	12.3	12.4
Upper limit of tolerance [cm] ($z_U \leq 2$)	14.5	12.9	13.2	13.3	13.3
Lower alarm limit [cm] ($z_U \geq -3$)	12.8	11.6	11.9	12.0	12.2
Upper alarm limit [cm] ($z_U \leq 3$)	14.8	13.1	13.5	13.5	13.6
Number of results	63	63	63	63	63
Lab performance					
$\lvert z_U\rvert \leq 2$	41 (65.1%)	49 (77.8%)	47 (74.6%)	54 (85.7%)	45 (71.4%)
$2<\lvert z_U\rvert \leq 3$	3 (4.8%)	0 (0%)	6 (9.5%)	2 (3.2%)	1 (1.6%)
$\lvert z_U\rvert >3$	19 (30.2%)	14 (22.2%)	10 (15.9%)	7 (11.1%)	17 (27.0%)

The results of the robust statistics indicate a very good reproducibility for the determination of H_f. The relative reproducibility standard deviation ranged between 1.13% and 2.14%. Between 71.4% and 86.7% of the laboratories reported satisfactory results ($|z_U| \leq 2$). The high amount (11.1-27.0%) of unsatisfactory results with $|z_U|>3$ displays difficulties in the determination of H_f. No difference between the performance of NRLs and OCLs was observed, so they were treated as one data population.

For sample A and E, the high amount of results outside the tolerance limits is due to different subpopulations of data.

The Kernel density plot for the H_f-value of sample A displayed two major modes, referring to two subpopulations of data (see 14.3 Figure 15). There was one group of laboratories (63% probability) that had obtained H_f-values between 12.5 and 16 cm, and a second group (21% probability) which had reported results in the range between 19 and 21 cm.

A possible explanation for the existence of the minor subpopulation is that the participants might have measured the total length of the sample in a different way (e.g. along the curved shape or diagonal - the provided instructions did not specify this, see 14.1.6) and/or assumed a different value for H_n. To define which sample part was considered as H_n, was a subjective decision and up to each laboratory. As, in case of sample A, H_p was about ½ H_n, already slight differences in the value measured for H_f and the one assumed for H_n made it necessary that H_f either had to be set to $2/3(H_n+H_p)$ or to H_n+H_p.

Additional investigations by the EURL-FCM showed, that the value obtained for the total length H_{total} may vary between approx. 31.5 cm and 31.0 cm, depending on whether the length is measured along the curved shape (e.g. with the help of a tape) or diagonal. This would lead to H_f-values of 14.0 cm and 20.7 cm, respectively, if a value of 13.9 cm is assumed for H_n. Assuming a lower, but still reasonable H_n-value, e.g. 13.6 cm, H_f-values of 14.0 cm and 13.8 cm, respectively, would result.

The robust mean value, obtained from all of the submitted data, was 13.79 cm and the calculated range of tolerance ($|z_U| \leq 2$) was between 13.1 and 14.5 cm. The upper alarm limit ($z_U \leq 3$) was 14.8 cm. Consequently, z_U-scores >3 arose for all H_f results in the range of 19 to 21 cm. It must be noted that these high z_U-scores misleadingly indicate an unacceptable laboratory performance if the high H_f-values were obtained by the way mentioned above. As the EURL-FCM does not know and cannot prove how the laboratories determined the H_f-values, it is not possible to specify which of the results are affected.

Sample E was a pair of tongs (cooking tweezers) that could be used to grab food, e.g. from a pan, barbecue or a pot. The Kernel density plot for this sample also indicated at least two subpopulations, although they were less clearly separated than in case of sample A (see 14.3 Figure 19). The main group of participants (mode with 71% probability) obtained a H_f-value in the range between 12.7 and 13.5 cm.

A smaller subpopulation (mode of 13% probability) reported a H_f-value of 11.3-11.6 cm. The main problem was the definition of the handle. Like for all other samples, the handle was not clearly separated. The latter group probably assumed that the handle of the sample was defined by an indentation in the upper part of the sample whereas the first subgroup probably set the length of the handle by default to 2/3 of the total sample height. Some participants also brought forward the argument that proper handling of the tong was only possible, if it was grasped at about half height of the sample. Otherwise, too much force was required. In conclusion, these laboratories assumed a larger value for the handle and reported even lower H_f-values.

Using the Hampel estimator, a robust mean value of 12.89 cm resulted which was used as assigned value. For the tolerance limits ($|z_U| \leq 2$), values of 12.4 and 13.3 cm were yielded. The lower alarm limit ($z_U \geq -3$) was 12.2 cm. Consequently, all those laboratories, that reported a H_f-value below 12.2 cm, received a z_U-score < -3. For the reasons mentioned above, this does not necessarily mean that the laboratory performance was unacceptable.

The Kernel density plots for the H_f-values of sample B, C and D displayed one main mode with 75-89% probability, indicating homogeneous data populations (see 14.3 Figure 16-Figure 18). For these samples, H_p was much larger than ½ H_n, so that H_f had to be set to $2/3(H_n+H_p)$ and H_f-values below 15 cm resulted. Despite this, a few laboratories reported H_f-values above 19 cm. In contrast to sample A, there was no founded reason why these results were obtained, so they might be regarded as outliers. The same applies for inexplicably low H_f-values.

Conclusions

The majority of laboratories obtained similar results for H_f. The laboratory performance was satisfying. Some difficulties occurred as the instructions gave room for different interpretations (see sample A and E). To avoid this, the instructions should be specified.

9.2.3. Food contact surface area

The results of the statistical evaluation for the surface area measurements are summarized in Table 3-Table 7. The single results reported by the laboratories, the Kernel density plots and the obtained z_U-scores are displayed in the annex (see 14.3.2 Figure 20-Figure 39 and 14.4.2 Table 31-Table 35). All calculations were done with ProLab [12].

To draw the outline of the samples, the laboratories were free to cut the samples into smaller pieces and draw the outline of each of the pieces or to leave the samples uncut. In case of sample A, B and E, 3, 4 and 2 laboratories, respectively, cut the samples prior to drawing. For sample C and D, there were 8 laboratories each that cut the samples before drawing. No significant differences were obtained for the results of cut and uncut samples (Mann-Whitney-Wilcoxon test, $\alpha = 0.05$). Therefore, the data were treated as a single population for statistical evaluation.

NRLs and OCLs were treated as one data population for the evaluation as there was no significant difference in their performance.

Table 3 Summary of the statistical evaluation for the food contact surface area determined for Sample A (all values)

Method DIN 38402 A45	calculation	wrap paper	wrap Al foil	draw shape
Measurand	SA	SA	SA	SA
Robust Mean = Assigned Value [cm²]	130.1	137.4	139.7	134.5
Robust Reproducibility = Target s.d. [cm²]	19.7	22.3	16.7	16.7
Rel. Reproducibility s.d. [%]	15.1	16.2	12.0	12.4
Lower limit of tolerance [cm²] ($z_U \geq -2$)	93.3	96.0	108.0	102.9
Upper limit of tolerance [cm²] ($z_U \leq 2$)	172.9	186.2	175.3	170.2
Lower alarm limit [cm²] ($z_U \geq -3$)	73.5	73.5	91.2	86.1
Upper alarm limit [cm²] ($z_U \leq 3$)	193.0	209.0	192.2	187.1
Number of results	58	53	56	59*
Lab performance				
$\|z_u\|\leq 2$	55 (94.8%)	46 (86.8%)	53 (94.6%)	55 (93.2%)
$2<\|z_u\|\leq 3$	2 (3.4%)	7 (13.2%)	2 (3.6%)	1 (1.7%)
$\|z_u\|>3$	1 (1.7%)	0 (0%)	1 (1.8%)	3 (5.1%)

*3 sample cut; 56 samples uncut

Table 4 Summary of the statistical evaluation for the food contact surface area determined for Sample B

Method DIN 38402 A45	calculation	wrap paper	wrap Al foil	draw shape
Measurand	SA	SA	SA	SA
Robust Mean = Assigned Value [cm²]	89.5	92.6	94.8	86.3
Robust Reproducibility = Target s.d. [cm²]	12.5	16.2	13.6	14.3
Rel. Reproducibility s.d. [%]	14.0	17.5	14.4	16.6
Lower limit of tolerance [cm²] ($z_U \geq -2$)	65.9	62.6	69.2	59.7
Upper limit of tolerance [cm²] ($z_U \leq 2$)	116.6	128.3	124.2	117.7
Lower alarm limit [cm²] ($z_U \geq -3$)	53.3	46.3	55.6	45.3
Upper alarm limit [cm²] ($z_U \leq 3$)	129.3	144.9	138.1	132.4
Number of results	56	53	56	59*
Lab performance				
$\|z_u\|\leq 2$	53 (94.6%)	48 (90.6%)	51 (91.1%)	56 (94.9%)
$2<\|z_u\|\leq 3$	2 (3.6%)	3 (5.7%)	4 (7.1%)	1 (1.7%)
$\|z_u\|>3$	1 (1.8%)	2 (3.8%)	1 (1.8%)	2 (3.4%)

* 4 samples cut; 55 samples uncut

Table 5 Summary of the statistical evaluation for the food contact surface area determined for Sample C

Method DIN 38402 A45	calculation	wrap paper	wrap Al foil	draw shape
Measurand	SA	SA	SA	SA
Robust Mean = Assigned Value [cm²]	143.1	147.9	159.6	128.4
Robust Reproducibility = Target s.d. [cm²]	17.7	12.2	14.6	22.0
Rel. Reproducibility s.d. [%]	12.3	8.3	9.2	17.2
Lower limit of tolerance [cm²] ($z_U \geq -2$)	109.7	124.4	131.6	87.5
Upper limit of tolerance [cm²] ($z_U \leq 2$)	180.9	173.4	190.3	177.0
Lower alarm limit [cm²] ($z_U \geq -3$)	92.0	112.1	116.9	65.3
Upper alarm limit [cm²] ($z_U \leq 3$)	198.8	185.7	204.9	199.7
Number of results	58	54	59	53*
Lab performance				
$\|z_u\|\leq 2$	50 (86.2%)	49 (90.7%)	56 (94.9%)	49 (92.5%)
$2<\|z_u\|\leq 3$	3 (5.2%)	2 (3.7%)	2 (3.4%)	1 (1.9%)
$\|z_u\|>3$	5 (8.6%)	3 (5.6%)	1 (1.7%)	3 (5.7%)

* 8 samples cut; 45 samples uncut

Table 6 Summary of the statistical evaluation for the food contact surface area determined for Sample D

Method DIN 38402 A45	calculation	wrap paper	wrap Al foil	draw shape
Measurand	SA	SA	SA	SA
Robust Mean = Assigned Value [cm²]	137.9	165.3	173.7	149.8
Robust Reproducibility = Target s.d. [cm²]	20.4	11.3	12.5	21.7
Rel. Reproducibility s.d. [%]	14.8	6.8	7.2	14.5
Lower limit of tolerance [cm²] ($z_U \geq -2$)	99.7	143.4	149.4	109.1
Upper limit of tolerance [cm²] ($z_U \leq 2$)	182.1	188.8	199.7	196.9
Lower alarm limit [cm²] ($z_U \geq -3$)	79.2	132.1	136.9	87.2
Upper alarm limit [cm²] ($z_U \leq 3$)	202.9	200.1	212.3	219.0
Number of results	57	54	59	53*
Lab performance				
$\|z_u\|\leq 2$	50 (87.7%)	49 (90.7%)	55 (93.2%)	50 (94.3%)
$2<\|z_u\|\leq 3$	4 (7.0%)	1 (1.9%)	2 (3.4%)	0 (0%)
$\|z_u\|>3$	3 (5.3%)	4 (7.4%)	2 (3.4%)	3 (5.7%)

* 8 samples cut; 45 samples uncut

Table 7 Summary of the statistical evaluation for the food contact surface area determined for Sample E

Method DIN 38402 A45	calculation	wrap paper	wrap Al foil	draw shape
Measurand	SA	SA	SA	SA
Robust Mean = Assigned Value [cm²]	84.4	87.9	90.0	83.8
Robust Reproducibility = Target s.d. [cm²]	11.5	10.5	11.2	14.7
Rel. Reproducibility s.d. [%]	13.6	12.0	12.4	17.5
Lower limit of tolerance [cm²] ($z_U \geq -2$)	62.9	68.0	68.8	56.6
Upper limit of tolerance [cm²] ($z_U \leq 2$)	109.1	110.4	113.9	116.2
Lower alarm limit [cm²] ($z_U \geq -3$)	51.3	57.4	57.6	41.8
Upper alarm limit [cm²] ($z_U \leq 3$)	120.8	121.1	125.2	131.4
Number of results	58	53	58	57*
Lab performance				
$\|z_u\|\leq 2$	49 (84.5%)	39 (73.6%)	48 (82.8%)	48 (84.2%)
$2<\|z_u\|\leq 3$	1 (1.7%)	7 (13.2%)	3 (5.2%)	6 (10.5%)
$\|z_u\|>3$	8 (13.8%)	7 (13.2%)	7 (12.1%)	3 (5.3%)

* 2 samples cut; 55 samples uncut

The calculated z_U-scores indicate a satisfying laboratory performance but it must be noted that the reproducibility standard deviations were rather high and therefore also the tolerance limits were high. Between 73.6% and 94.9% of the laboratories reported satisfactory results ($|z_U|\leq 2$). 0-13.2% of the laboratories obtained questionable results ($2<|z_U|\leq3$) and 0-13.8% reported unsatisfactory results ($|z_U|>3$).

The relative reproducibility standard deviations range from 6.8 to 17.5%. Also the Kernel density plots indicated broad, but homogeneous data distributions for all samples and all measurement methods. To a certain extent, the broad distributions of results are due to different H_f-values that were assumed by the laboratories. It was proven by a rank test that values for the surface area of sample A referring to $H_f \geq 19$ cm differ significantly from those corresponding to $H_f < 16$ cm (Mann-Whitney-Wilcoxon test, $\alpha = 0.05$). In average, surface area values based on $H_f \geq 19$ cm were 19.5% higher than those based on $H_f < 16$ cm.

But also for those laboratories that assumed the same H_f values, the final results for the food contact surface area measurements varied broadly (see Youden plots in 14.3.4). In fact, for sample A-D differences in H_f only had a minor influence on the size of the surface area because the sample part H_p contributes only to a minor extent to the total surface area. For sample E, a major influence of H_f on the value of the surface area is visible. The shape of this sample is similar to a rectangular solid, so there should be a more or less direct proportional correlation between H_f and the surface area. Indeed, the respective Youden plots display a direct correlation (see 14.3.4 Figure 54).

Conclusions

There is a correlation between the food contact surface area and H_f. If higher H_f-values were measured, higher values for the food contact surface area were obtained. This caused a broader distribution of results. The respective reproducibility standard deviations for the determination of the food contact surface area do not only reflect the uncertainty of the surface area measurement but also the bias due to different H_f-values.

Also the obtained z_U-scores do not only reflect the laboratory performance for the surface area measurement but also the bias from the robust mean due to the determination of a different H_f-value. All results would have to be corrected by a bias. This was not possible. The samples were not entirely regular shaped, so the correlation between H_f and the surface area could not be described by a mathematical model. H_f should be marked on all samples before the shipment to the participants for the determination of the reproducibility of the surface area analysis itself.

Although the food contact surface area depends on the value of H_f, in most cases no consequences for the final migration results will arise if laboratories determine different H_f values. If larger H_f values are deterimined, a larger sample part and then also a larger sample surface will be exposed in the migration testing and the absolute migrating amount will be higher. For samples, where the migration is even over the entire surface, both parameters (i.e. surface area and migrated amount) will be higher to the same extent. Hence, the migrated amount in mg/dm² remains the same.

A problem might occur for samples that consist of more than one material, e.g. plastic kitchen spatulas where the lower (functional) part is covered with silicone, baby feeding spoons where parts of the handle are made of different plastics, silicone or thermoplastic elastomers and utensils with a printing on the handle, or materials where the migration ratio depends on the thickness of the material. In these cases, it would be important which value is assumed for H_f and which sample part then would be exposed in a migration testing because the migration will not be even over the surface of the entire sample.

9.2.4. Comparison of the methods for the determination of the food contact surface area

For the comparison of the different methods, only those results were selected where the participants had assumed similar values for H_f. These were between 28 and 40 results for each of the five samples and each of the four methods (see Table 8). For these values, again the robust mean values and robust reproducibility standard deviations were calculated. An overview of the obtained statistical data is given in Table 9 and Table 10.

Table 8 Number of results for the surface area based on the same value of H_f

Sample	selected range of H_f [cm]	Number of results calculation	wrap in paper	wrap in Al foil	draw shape
A	13.6-13.9	32	28	30	32
B	12.2-12.4	34	31	34	36
C	12.5-12.8	39	36	40	36
D	12.7-12.9	35	33	36	32
E	12.8-12.9	38	33	38	36

Table 9 Statistical data for the selected results of the food contact surface area determined by "calculation" and "wrapping in paper" based on the same value of H_f

Sample	selected range of H_f [cm]	calculation			wrap in paper		
		robust mean [cm²]	reprod. s.d. [cm²]	rel. reprod. s.d. [%]	robust mean [cm²]	reprod. s.d. [cm²]	rel. reprod. s.d. [%]
A	13.6-13.9	124.1	14.6	11.7	130.6	10.9	8.3
B	12.2-12.4	89.7	11.0	12.3	92.6	12.2	13.2
C	12.5-12.8	142.5	14.5	10.2	145.0	9.6	6.6
D	12.7-12.9	138.7	15.9	11.4	161.4	8.8	5.4
E	12.8-12.9	89.6	5.8	6.5	91.6	5.2	5.7

Table 10 Statistical data for the selected results of the food contact surface area determined by "wrapping in aluminium foil" and "drawing the shape" based on the same value of H_f

Sample	selected range of H_f [cm]	wrap in Al foil			draw shape		
		robust mean [cm²]	reprod. s.d. [cm²]	rel. reprod. s.d. [%]	robust mean [cm²]	reprod. s.d. [cm²]	rel. reprod. s.d. [%]
A	13.6-13.9	134.5	11.7	8.7	129.5	10.5	8.1
B	12.2-12.4	94.4	9.9	10.5	87.4	12.1	13.9
C	12.5-12.8	155.6	10.6	6.8	127.8	20.6	16.1
D	12.7-12.9	171.4	9.9	5.8	151.0	21.0	13.9
E	12.8-12.9	92.7	6.5	7.0	89.3	9.5	10.6

The relative reproducibility standard deviations for these results ranged between 5.4 and 16.1%. The smallest values were obtained for “wrapping in paper” and “wrapping in aluminium foil”. “Drawing the shape” yielded the best reproducibility for sample A which was a flat spatula. “Calculation” returned the smallest reproducibility standard deviation for sample E which had a rather simple geometric form.

The robust mean values of the food contact surface area of each sample, determined by “calculation”, “wrapping in paper”, “wrapping in aluminium foil” and “drawing the shape”, did not differ significantly (comparison of mean values, $\alpha = 0.05$) but a rank test performed with the single results displayed significant differences (Friedman’s test, $\alpha = 0.05$). “Calculation” and “drawing the shape” tended to return the lowest results whereas “wrapping in aluminium foil” always returned the highest values.

Trueness of results

The true values for the surface areas of the provided samples are unknown. To evaluate the trueness of the performed methods for the determination of the surface area ("calculation", "wrapping in paper", "wrapping in aluminium foil" and "drawing the shape"), the robust mean values were compared to results from a laser scanning. The laser scanning was performed by Creaform Deutschland GmbH on behalf of the German Bundesinstitut für Risikobewertung (BfR). The results are given in Table 11.

Table 11 Results for the food contact surface area measured with a laser scanner (single determination, performed by Creaform Deutschland GmbH)

Sample	laser scanner	
	H_f [cm]	surface area [cm²]
A	13.7	129.18
B	12.3	89.22
C	12.7	130.71
D	12.8	150.87
E	12.9	84.90

A comparison of the data obtained by laser scanner and the other four methods is shown in Figure 3 and Figure 4. For sample A and B all four methods lead to results equal to the result of the laser scanning and all methods show similar relative reproducibility standard deviations (see Figure 3, Figure 4). For the round-shaped samples C and D and also for sample E, the surface area is overestimated when measured by "wrapping in paper" and "wrapping in aluminium foil". With "wrapping in aluminium foil", the overestimation is up to (19.0±8.1)% (see Table 12), probably due to the formation of crinkles. Several laboratories described that aluminium foil heavily crinkled when used for wrapping. If the aluminium foil was too thin, it was impossible to remove the excess aluminium foil of the crinkles with a scalpel or scissors as the foil was easily ruptured. One of the participants recommended the use of thicker aluminium household foil with a surface weight of approx. 0.42 g/dm^2 compared to conventional aluminium household foil with 0.31 g/dm^2. They stated that proper wrapping and cutting is possible with this type of foil. Alternatively, two laboratories suggested redrawing the shape of the crinkled aluminium foil on paper and cutting and weighing the paper afterwards. Thus, it would be possible to disregard the excess foil of the crinkles.

Table 12 Bias of robust mean values for the surface area determined by "calculation", "wrapping in paper", "wrapping in aluminium foil" and "drawing the shape" compared to results obtained by laser scanner

Sample	selected range of H_f [cm]	calculation		wrap in paper		wrap in Al foil		draw shape	
		bias to laser scanner [%]	robust reproducibility s.d. [%]	bias to laser scanner [%]	robust reproducibility s.d. [%]	bias to laser scanner [%]	robust reproducibility s.d. [%]	bias to laser scanner [%]	robust reproducibility s.d. [%]
A	13.6-13.9	-3.9	11.3	1.1	8.4	4.1	9.0	0.3	8.1
B	12.2-12.4	0.5	12.4	3.8	13.7	5.8	11.1	-2.0	13.6
C	12.5-12.8	9.0	11.1	10.9	7.3	19.0	8.1	-2.2	15.7
D	12.7-12.9	-8.1	10.5	7.0	5.8	13.6	6.6	0.1	13.9
E	12.8-12.9	5.5	6.8	7.8	6.1	9.2	7.6	5.1	11.2

For sample C, D and E, "drawing the shape" returned robust mean values closest to the result of the laser scanning but it must be noted that this method showed the highest relative robust reproducibility standard deviations (see Table 10). The robust mean values for sample C, D and E obtained by “calculation” also do not differ significantly from the results obtained with the laser scanner and the respective relative robust reproducibility standard deviations were smaller compared to “drawing the shape”.

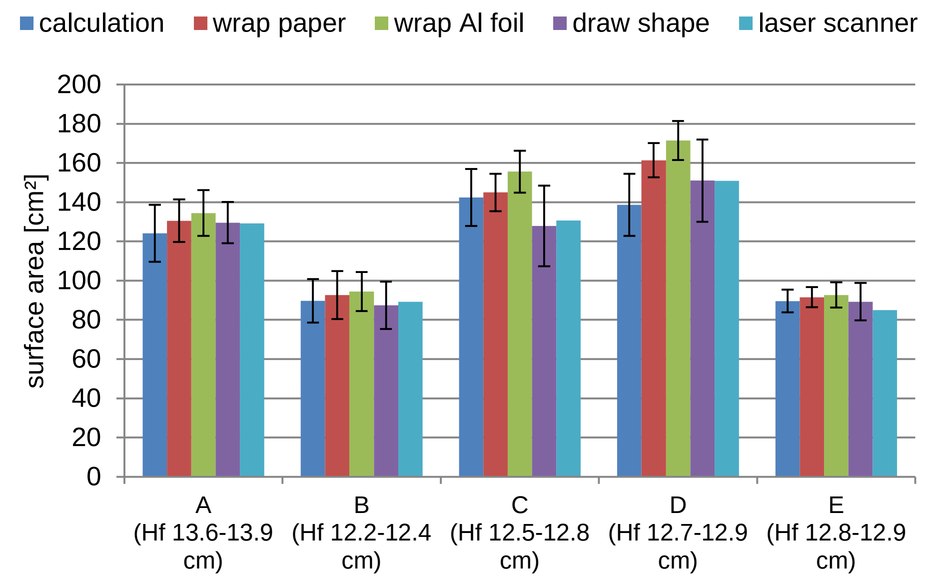


Figure 3 Comparison of results for the surface area determined by "calculation", "wrapping in paper", "wrapping in aluminium foil", "drawing the shape" and by laser scanner. (Data shown: robust mean values ± robust reproducibility s.d., in cm^2)

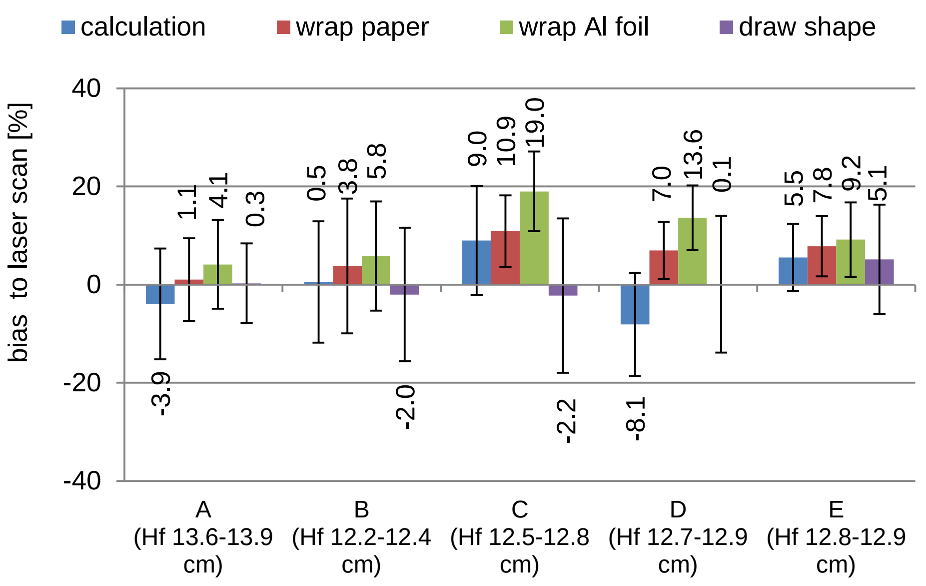


Figure 4 Bias of robust mean values for the surface area determined by "calculation", "wrapping in paper", "wrapping in aluminium foil" and "drawing the shape" compared to results obtained by laser scanner. (Data shown: difference of robust mean values to surface area determined by laser scanner ± robust reproducibility s.d., in %)

Effect on migration results

Overall and specific migration results are calculated as follows.

(1) $$OM\ or\ SM\ \left[\frac{mg}{dm^2}\right] = \frac{x}{S}$$

(2) $$SM\ \left[\frac{mg}{kg}\right] = \frac{x}{S} \cdot \frac{6\ dm^2}{kg}$$

where:

OM: overall migration
SM: specific migration
x: migrated amount [mg]
S: surface area exposed in the migration testing [dm^2] (usually this is the food contact surface area)

In consequence, the trueness of the overall and specific migration results is affected by the systematic bias of the surface area measurement, e.g. the overestimation of the surface area observed for "wrapping in aluminium foil", and the migration measurement. The uncertainty of the overall and specific migration result arises from random effects of the surface area measurement and the determination of the migrated amount as well as “from imperfect correction of the results for systematic effects” (see [6] p. 5).

Trueness of overall and specific migration results

The total systematic bias is calculated as follows ([14] p. 50).

(3) $$\frac{\Delta OM_{sys}}{OM}\ or\ \frac{\Delta SM_{sys}}{SM} = \frac{\Delta x_{sys}}{x} - \frac{\Delta S_{sys}}{S}$$

where:

$\frac{\Delta OM_{sys}}{OM}$: relative systematic bias for the overall migration [%]
$\frac{\Delta SM_{sys}}{SM}$: relative systematic bias for the specific migration [%]
$\frac{\Delta x_{sys}}{x}$: relative systematic bias for the determination of the migrated amount [%]
$\frac{\Delta S_{sys}}{S}$: relative systematic bias for the surface area measurement [%]

Assuming that $\frac{\Delta x_{sys}}{x} = 0$, the following equation results:

(4) $$\frac{\Delta OM_{sys}}{OM}\ or\ \frac{\Delta SM_{sys}}{SM} = -\frac{\Delta S_{sys}}{S}$$

This shows, that if the surface area is systematically overestimated by up to (19.0±8.1)% as observed for "wrapping in aluminium foil" (see Table 12), the overall or specific migration result will be systematically underestimated by the same percentage.

Uncertainty of migration results

Based on the JCGM Guide to the expression of uncertainty in measurement ([6] p. 19) and ISO/TS 21748 ([7] p.16 and p.8), the relative uncertainty of the overall or specific migration result after correction of the systematic bias is calculated as described hereafter.

(5) $$\frac{u_{OM\ or\ SM}}{OM\ or\ SM} = \sqrt{\left(\frac{u(x)}{x}\right)^2 + \left(\frac{u(S)}{S}\right)^2}$$

where:

$u_{OM\ or\ SM}$:	combined uncertainty (standard deviation) of the overall or specific migration result [mg/dm^2 or mg/kg]
OM:	overall migration [mg/dm^2]
SM:	specific migration [mg/kg]
$u_{OM\ or\ SM}$/OM or SM:	combined relative uncertainty (relative standard deviation) of the overall or specific migration result
u(x):	uncertainty associated with x [mg]
u(S):	uncertainty associated with S [dm^2]

For the uncertainties (standard deviations) associated with the measurement of x and S are calculated, also the uncertainty related to the bias (if present and if significant) need to be considered as shown in the following equations.

(6) $$u(x) = \sqrt{u^2(\hat{\delta}_x) + s_R{}^2(x)}$$

(7) $$u(S) = \sqrt{u^2(\hat{\delta}_S) + s_R{}^2(S)}$$

where:

$s_R(x)$:	reproducibility standard deviation for the determination of x [mg]
$s_R(S)$:	reproducibility standard deviation for the determination of S [dm^2]
$u(\hat{\delta}_x)$:	uncertainty (standard deviation) associated with δ_x due to the uncertainty of estimating δ_x by measuring a reference measurement standard or reference material with certified value $\hat{\mu}$ [mg]
δ_x:	bias intrinsic to the measurement method for x [mg]
$u(\hat{\delta}_S)$:	uncertainty (standard deviation) associated with δ_S due to the uncertainty of estimating δ_S by measuring with a reference method [dm^2]
δ_S:	bias intrinsic to the measurement method for S [dm^2]

It should be noted that the measurement uncertainty for the determination of the migrated amount x comprises the entire migration measurement including the migration experiment, treatment of the migration solutions and the quantification of the substances.

The uncertainty associated with the determination of the bias δ_S for the surface area measurement arises from the uncertainty of the laser scanning and the data treatment afterwards. This uncertainty is unknown and cannot be estimated as the laser scanning was done only as a single-fold determination. For the present study, it is assumed that this uncertainty is insignificant compared to $s_R(S)$ and therefore can be disregarded as stated in the JCGM guide ([6] p. 7).

Assuming further that there is no significant bias δ_x for the measurement of x and $u(\hat{\delta}_x) = 0$, equation (5) can be simplified in the following way.

(8) $$\frac{u_{OM\ or\ SM}}{OM\ or\ SM} = \sqrt{\left(\frac{s_R(x)}{x}\right)^2 + \left(\frac{s_R(S)}{S}\right)^2}$$

where:

$s_R(x)/x$: relative reproducibility standard deviation for the determination of x

$s_R(S)/S$: relative reproducibility standard deviation for the determination of S

To illustrate the consequences of systematic and random effects of the surface area measurement for the final overall or specific migration results, examples are given below.

Example 1: Migration of formaldehyde (HCHO) (SML = 15 mg/kg food [1]), surface area determined by "drawing the shape", sample C

In ILC01 2012 on the determination of formaldehyde and melamine in 3% acetic acid migration solutions, a relative reproducibility standard deviation of 12.02% was obtained for the quantification of formaldehyde in a solution that contained about 9.12 mg HCHO/kg [2]. Assuming this as the uncertainty for the migration measurement and considering the highest relative reproducibility standard deviation obtained for the determination of the surface area (i.e. 16.1%, obtained with "drawing the shape" see Table 10), a total uncertainty of 20.1% results for the specific migration. (Note: There is no significant bias for the surface area determined by "drawing the shape". Hence, no bias has to be regarded.)

$$\frac{u_{SM}}{SM} = \sqrt{\left(\frac{s_R(x)}{x}\right)^2 + \left(\frac{s_R(S)}{S}\right)^2} = \sqrt{(12.02\%)^2 + (16.1\%)^2} = 20.1\%$$

$$\underline{\underline{SM = (9.1 \pm 1.8)\frac{mg}{kg}}}$$

Example 2: Migration of formaldehyde (HCHO) (SML = 15 mg/kg food [1]), surface area determined by "wrapping in aluminium foil", sample C

If the surface area in example 1 is determined by "wrapping in aluminium foil" instead, the relative reproducibility standard deviation for the surface area measurement is only 6.8% (see Table 10). This yields a total uncertainty of 13.8% for the result of the specific migration. Finally a specific migration result of (9.1 ± 1.3) mg/kg would be yielded. (Note: The result for the surface area measurement has to be corrected before by the bias, i.e. 19.0% due to the systematic overestimation of the surface area.)

$$\frac{u_{SM}}{SM} = \sqrt{\left(\frac{s_R(x)}{x}\right)^2 + \left(\frac{s_R(S)}{S}\right)^2} = \sqrt{(12.02\%)^2 + (6.8\%)^2} = 13.8\%$$

$$\underline{\underline{SM = (9.1 \pm 1.3)\frac{mg}{kg}}}$$

Example 3: Migration of 2,4-Toluenediamine (2,4-TDA) (SML = 0.01 mg/kg food [1]), surface area determined by "drawing the shape", sample C

The obtained relative reproducibility standard deviations in ILC02 2012 for the determination of four primary aromatic amines in 3% acetic acid migration solutions ranged between 10.39% and 39.90% [13]. The highest value (i.e. 39.90%) was obtained for 2,4-TDA in a migration solution with a concentration level of about 16.69 µg/kg.

Assuming again that the surface area was determined by "drawing the shape" as done in example 1, a total uncertainty of 43.0% results.

$$\frac{u_{SM}}{SM} = \sqrt{\left(\frac{s_R(x)}{x}\right)^2 + \left(\frac{s_R(S)}{S}\right)^2} = \sqrt{(39.90\%)^2 + (16.1\%)^2} = 43.0\%$$

$$\underline{\underline{SM = (16.7 \pm 7.2)\frac{\mu g}{kg}}}$$

Example 1 shows that if the reproducibility of the analysis and of the determination of the surface area are similar that the uncertainty of the result almost doubles. When the reproducibility of the determination of the surface area is smaller than the reproducibility of the analysis then the reproducibility of the determination of the surface area has little effect on the the uncertainty of the result (example 2 and 3). In conclusion, the obtained relative reproducibility standard deviations of 5.4-16.1% for the surface area measurement can be regarded as acceptable.

A problem may be the significant overestimation of the surface area when round-shaped articles are wrapped in aluminium foil. The surface area values determined with the help of this method would need to be corrected by the bias. As the bias is not equal for all sample types, but depends on the sample shape, no general value for the bias can be determined. Therefore, correction of the results will be difficult.

Conclusions

The trueness and precision of the methods depend on the sample shape. For flat samples and simple geometric shapes, all methods return results that do not differ significantly from the laser scanning and show acceptable reproducibility. Hence, all methods are suitable for these types of samples.

For round-shaped samples, the surface area is overestimated when determined by wrapping in aluminium foil and paper. It would be worth to check if the overestimation for wrapping in aluminium foil is less severe when aluminium foil of appropriate thickness is used which is ruptured less easily and therefore easier to cut.

"Drawing the shape" is less suitable for round-shaped samples and for samples with a non-negligible thickness, as the reproducibility standard deviation increases. Considering that the migration measurement itself can be affected by uncertainties of similar levels as those of the determination of the surface area, the larger uncertainties for the surface area measurement are still acceptable.

"Calculation" is suitable for all sample types and shows acceptable reproducibility standard deviations.

9.3. *Evaluation of the questionnaire*

The questionnaire aimed on the evaluation of the performed surface area measurement methods in terms of convenience and feasibility as the methods need to be suitable for routine work. All participants were asked to provide the following information:

- how much time was spent approximately to determine the surface area for each sample and each method
- which of the performed methods was most suitable in their opinion for the particular sample
- which of the performed methods were in use in the laboratory and how often
- their willingness to implement the performed methods in daily laboratory work
- an overall ranking of the methods according to their personal preference

59 laboratories returned the filled questionnaires.

Time spent for the determination

The amount of time spent for the analysis varied broadly. The results reported by the single laboratories ranged between 2 and 210 min (see 14.3.5 Figure 55-Figure 59). To compare the data, robust mean values and robust reproducibility standard deviations were calculated with the help of the ProLab software [12]. The results are shown in Table 13 and Figure 5.

Table 13 Time spent in average for the determination of the surface area

	calculation		wrap in paper		wrap in Al foil		draw shape	
Sample	robust mean [min]	reprod. s.d. [min]	robust mean [min]	reprod. s.d. [min]	robust mean [min]	reprod. s.d. [min]	robust mean [min]	reprod. s.d. [min]
A	32	22	23	15	23	14	19	15
B	36	25	24	13	24	14	19	15
C	26	18	25	17	22	14	21	17
D	35	25	23	16	25	15	23	17
E	15	9	14	8	12	8	12	8

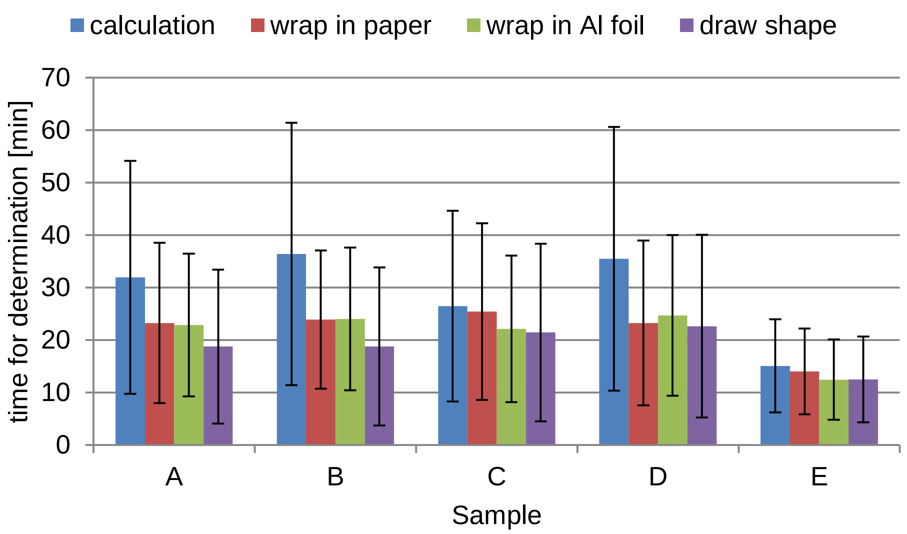


Figure 5 Time spent in average for the determination of the surface area (Data shown: robust mean values ± robust reproducibility s.d.)

The robust mean values indicate that the determinations in average lasted between 12.4 and 36.4 min. Friedman's tests on the single reported values showed significant differences (α = 0.05) between the times needed for the different methods. Most time was needed for "calculation", followed by "wrapping in paper" and "wrapping in aluminium foil". "Drawing the shape" was the fastest approach.

Most suitable method for each sample and overall ranking

To determine the surface area of sample A and B, 40.3% and 33.8% of the laboratories, respectively, stated that "drawing the shape" is the most suitable method, followed by "calculation" and "wrapping in aluminium foil". For sample C and D, "wrapping in aluminium foil" was preferred by 50.8% and 55.7% of the participants, respectively. For sample E, "calculation" was declared as most suitable (see Table 14 and Figure 6). It should be noted, that some laboratories named more than one method as suitable for the respective sample.

Table 14 Most suitable method for the determination of the surface area of the particular sample

	calculation		wrap in paper		wrap in Al foil		draw shape	
sample	number of votes	number of votes [%]	number of votes	number of votes [%]	number of votes	number of votes [%]	number of votes	number of votes [%]
A	20	29.9	5	7.5	15	22.4	27	40.3
B	19	29.2	5	7.7	19	29.2	22	33.8
C	22	36.1	7	11.5	31	50.8	1	1.6
D	14	23.0	8	13.1	34	55.7	5	8.2
E	34	50.0	3	4.4	13	19.1	18	26.5

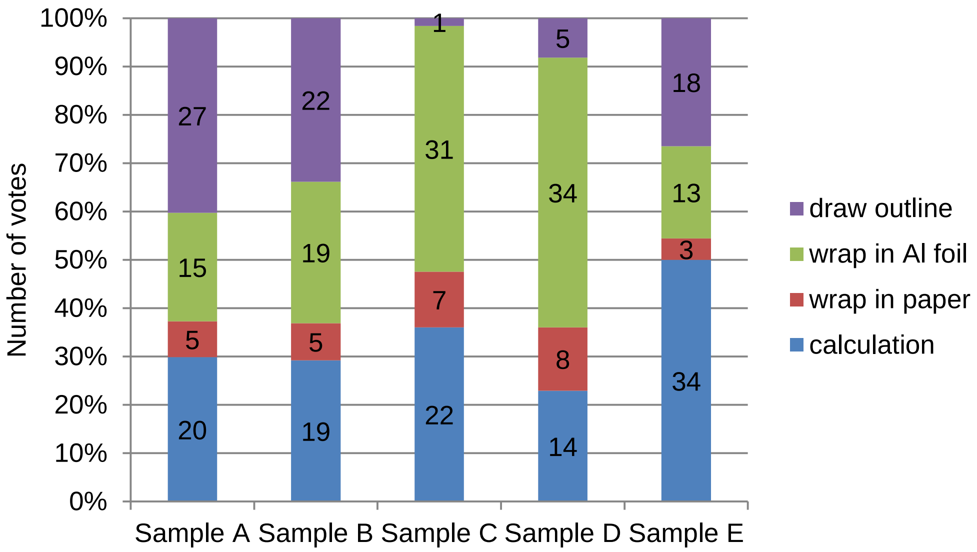


Figure 6 Most suitable method for the determination of the surface area of the particular sample

The results in Figure 6 and Table 14 show that "calculation" and "wrapping in aluminium foil" were regarded as suitable for all types of samples whereas "drawing the shape" was preferred for flat samples and not for the round-shaped samples C and D. It was declared that it was impossible to properly draw the shape of the curved samples. Cutting the items into smaller pieces was claimed as time-consuming and demanding for the equipment and finally did not help much as stated by one laboratory. Apart from this, another laboratory declared that it is difficult to take into account the thickness of the sample when drawing its shape. So this

laboratory normally uses a mixture of “drawing the shape” and “calculation”, using the latter one to calculate the surface area of the sample side parts.
“Wrapping in paper” was generally characterised as inconvenient. Several laboratories declared that paper is less flexible than aluminium foil and therefore wrapping was difficult. In addition, some laboratories reported difficulties to find paper with a uniform surface weight (deviation < 1 %). They finally used paper with a grammage deviation of 1.5-2 %.

These results correspond to the overall ranking of the methods. “Calculation” was the most popular method, followed by “wrapping in aluminium foil” and “drawing the shape”. The worst method according to this subjective ranking was “wrapping in paper”. It must be noted that a large number of participants also declared that “wrapping in aluminium foil” was inconvenient. The main reasons were that especially thin aluminium foil crinkles heavily and is ruptured easily. On the other hand, it was mentioned that aluminium foil is very flexible and easily allowed covering the contour of the samples. Further, one laboratory stated that the surface weight of aluminium foil is very consistent and no problems with humidity would occur in comparison to paper.

Table 15 Results of the overall ranking of the methods for the determination of the surface area on a scale from 1 to 4 where 1 = "the best" and 4 = "the worst"

	calculation		wrap in paper		wrap in Al foil		draw shape	
overall rank	number of votes	number of votes [%]	number of votes	number of votes [%]	number of votes	number of votes [%]	number of votes	number of votes [%]
1	21	35.6	6	10.2	19	32.2	13	22.0
2	15	25.0	12	20.0	15	25.0	18	30.0
3	13	23.6	15	27.3	9	16.4	18	32.7
4	9	16.4	23	41.8	15	27.3	8	14.5
average rank	2.2		3.0		2.3		2.4	

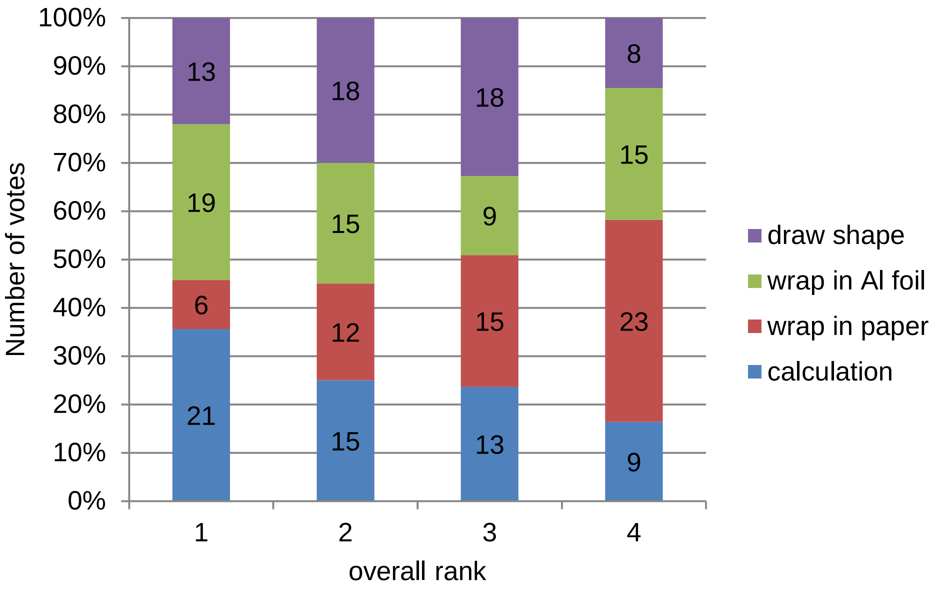


Figure 7 Results of the overall ranking of the methods for the determination of the surface area on a scale from 1 to 4 where 1 = "the best" and 4 = "the worst"

As reasons for their preference and the awarded overall ranks, two participants mentioned the aspect of documentation for quality assurance. They declared to prefer “calculation” and “drawing the shape (on scale paper)” as these methods and the obtained results can be easily documented.

Methods currently in use and willingness to implement the tested methods

“Calculation” is used most often by the laboratories, followed by “drawing the shape” and “wrapping in paper”. 61% of the participants stated to use "calculation" frequently, 20.3% use it at least sometimes. Only three laboratories (5.2 %) declared to frequently use “wrapping in aluminium foil”. Most of the laboratories (75.9 % and 57.1 %, respectively) did not use “wrapping in aluminium foil” or “wrapping in paper” at all but a large percentage would implement “wrapping in aluminium foil”. For “wrapping in paper”, the willingness to implement this method is the lowest (see Table 17). This corresponds to the results discussed before.

Table 16 Current usage of the tested methods for the determination of the surface area

	calculation		wrap in paper		wrap in Al foil		draw shape	
current usage of the method	number of labs	number of labs [%]	number of labs	number of labs [%]	number of labs	number of labs [%]	number of labs	number of labs [%]
frequently	36	61.0	10	17.9	3	5.2	15	25.9
sometimes	12	20.3	5	8.9	5	8.6	23	39.7
hardly	4	6.8	7	12.5	6	10.3	1	1.7
never	7	11.9	32	57.1	44	75.9	18	31.0
not anymore	0	0.0	2	3.6	0	0.0	1	1.7

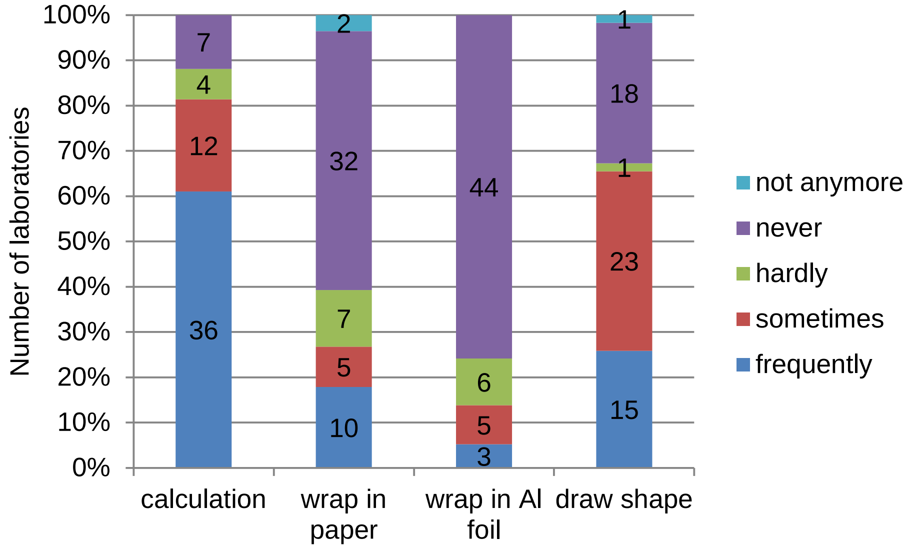


Figure 8 Current usage of the tested methods for the determination of the surface area

Table 17 Willingness of the laboratories to implement the performed methods expressed on a scale from 1 to 6 where 1 = "of course" and 6 = "never"

	calculation		wrap in paper		wrap in Al foil		draw shape	
willingness to implement the methods	number of labs	number of labs [%]	number of labs	number of labs [%]	number of labs	number of labs [%]	number of labs	number of labs [%]
1	26	45.6	8	14.3	18	31.0	17	30.9
2	12	21.1	5	8.9	12	20.7	16	29.1
3	10	17.5	10	17.9	11	19.0	8	14.5
4	3	5.3	9	16.1	3	5.2	5	9.1
5	3	5.3	8	14.3	4	6.9	4	7.3
6	3	5.3	16	28.6	10	17.2	5	9.1
average willingness	2.2		3.9		2.9		2.6	

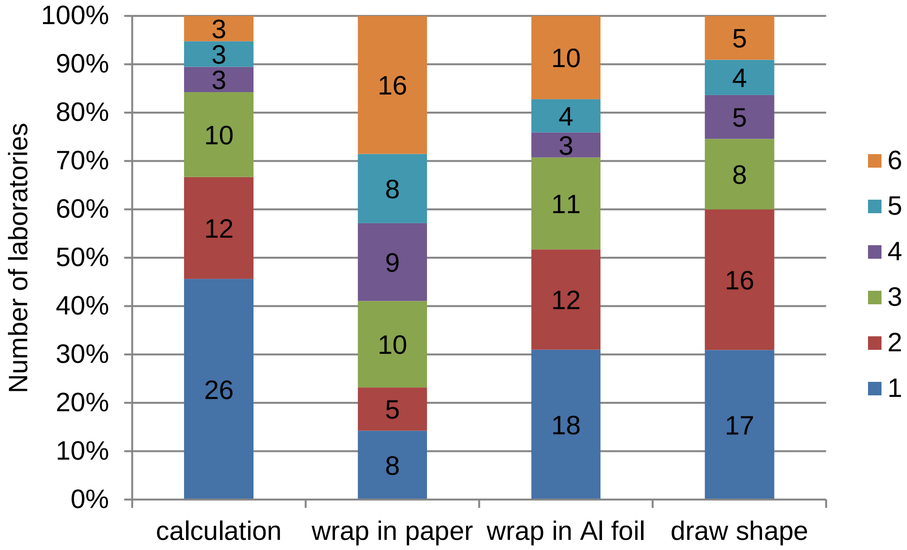


Figure 9 Willingness of the laboratories to implement the performed methods expressed on a scale from 1 to 6 where 1 = "of course" and 6 = "never"

Conclusions

Depending on the sample shape, different methods are preferred by the laboratories.

"Calculation" is generally the most popular method. It is used most often, it is regarded as suitable for all types of samples and it is the method that most laboratories would implement, although it is the most time-consuming one. It is easy to document and therefore also convenient in terms of quality management.

"Drawing the shape" is the fastest method and it is easy to document as well. Whenever possible, i.e. for flat samples and simple geometric shapes, this method or "calculation" are the methods of choice.

For round-shaped samples, "wrapping in aluminium foil" or "calculation" are preferred because aluminium foil is very flexible and easily allows covering the sample contours.

9.4. Voluntary exercise – Envelope Volume

In the voluntary exercise, the participants were asked to determine the envelope volume of the samples on a 2-cm-scale and a 5-cm-scale. The results reported by the laboratories and the respective Kernel density plots are shown in 14.3.3.1-14.3.3.5 Figure 40-Figure 49. In addition, Youden plots are given in 14.3.4 that show the correlation between the envelope volume and the sample height (H_f) with foreseeable food contact.

For the statistical evaluation, robust mean values and the reproducibility standard deviations were calculated as described in section 8.1 and 8.2. They are summarised in Table 18 and Table 19. The calculated z_U-scores for the single laboratories are given in section 14.4.3 Table 36 and Table 37.

Table 18 Summary of the statistical evaluation for the envelope volume determined on a 2-cm-scale

Method DIN 38402 A45	Sample A	Sample B	Sample C	Sample D	Sample E
Measurand	EV 2-cm-scale	EV 2-cm-scale	EV 2-cm-scale	EV 2-cm-scale	EV 2-cm-scale
Assigned Value [cm^3]	168	336	336	336	224
Robust Mean [cm^3]	202.5	237.8	310.1	342.9	131.0
Median [cm^3]	168	168	336	336	120
Robust Reproducibility = Target s.d. [cm^3]	91.6	157.4	141.2	157.6	88.8
Interquartile range [cm^3]	168	168	112	224	72
Rel. Reproducibility s.d. [%]	54.5	46.9	42.0	46.9	39.6
Lower limit of tolerance [cm²] ($z_U \geq -2$)	35	87	102	87	74
Upper limit of tolerance [cm²] ($z_U \leq 2$)	411	747	699	747	450
Lower alarm limit [cm²] ($z_U \geq -3$)	3	9	14	9	12
Upper alarm limit [cm²] ($z_U \leq 3$)	505	915	854	915	549
Number of results	52	52	53	53	53
Lab performance					
$\|z_U\|\leq 2$	42 (80.8%)	48 (92.3%)	48 (90.6%)	49 (92.5%)	38 (71.7%)
$2<\|z_U\|\leq 3$	3 (5.8%)	3 (5.8%)	3 (5.7%)	2 (3.8%)	13 (24.5%)
$\|z_U\|>3$	7 (13.5%)	1 (1.9%)	2 (3.8%)	2 (3.8%)	2 (3.8%)

Table 19 Summary of the statistical evaluation for the envelope volume determined on a 5-cm-scale

Method DIN 38402 A45	Sample A	Sample B	Sample C	Sample D	Sample E
Measurand	EV 5-cm-scale	EV 5-cm-scale	EV 5-cm-scale	EV 5-cm-scale	EV 5-cm-scale
Assigned Value [cm^3]	750	750	750	750	750
Robust Mean [cm^3]	814.9	559.0	751.2	779.1	605.5
Median [cm^3]	750	750	750	750	750
Robust Reproducibility = Target s.d. [cm]	263.7	322.8	345.3	241.8	374.1
Interquartile range [cm^3]	250	375	0	0	375
Rel. Reproducibility s.d. [%]	35.2	43.0	46.0	32.2	49.9
Lower limit of tolerance [cm²] ($z_U \geq -2$)	292	220	199	324	177
Upper limit of tolerance [cm²] ($z_U \leq 2$)	1405	1582	1649	1339	1734
Lower alarm limit [cm²] ($z_U \geq -3$)	67	28	22	98	17
Upper alarm limit [cm²] ($z_U \leq 3$)	1708	1936	2019	1618	2125
Number of results	51	51	52	52	52
Lab performance					
$\|z_U\|\leq 2$	43 (84.3%)	49 (96.1%)	48 (92.3%)	47 (90.4%)	50 (96.2%)
$2<\|z_U\|\leq 3$	2 (3.9%)	1 (2.0%)	1 (1.9%)	4 (7.7%)	2 (3.8%)
$\|z_U\|>3$	6 (11.8%)	1 (2.0%)	3 (5.8%)	1 (1.9%)	0 (0%)

The obtained relative reproducibility standard deviations range from 39.6-54.5% for the envelope volume determined on the 2-cm-scale and from 32.2-49.9% for the 5-cm-scale. They indicate a broad distribution of results. The high values for the interquartile range confirm this observation (see Table 18 and Table 19).

As the reproducibility standard deviations were set as target standard deviations, their high values result in broad ranges of tolerance. Therefore, most of the laboratory results were within the tolerance limits even if the reported values differed by more than a factor of 2 from the assigned value. It should be noted that there were also some samples where almost all laboratories obtained the same value for the envelope volume (see sample C and D, 14.3.3 Figure 44-Figure 47). For these two samples, the interquartile range for the envelope volume determined on the 5-cm-scale was 0 (see Table 19). All in all, the laboratory performance can be regarded as satisfying.

For sample A, the majority of laboratories also obtained the same results for the envelope volume. The Kernel density plots display only one main mode with 81% and 86% probability for the results obtained on the 2-cm-scale and the 5-cm-scale, respectively. The laboratories that reported values outside the calculated tolerance limits were mainly those that had assumed a higher value for H_f (see Youden plots in 14.3.4). For these laboratories, the yielded z_U-scores again misleadingly indicate an unacceptable performance.

For sample B, the reported data display two subpopulations for the results obtained on the 2-cm-scale and for those obtained on the 5-cm-scale. The Youden plots indicate that the differences are not (or not exclusively) correlated to the value of H_f. So the laboratories must have assumed different values for the depth and/or width of the sample. The same applies for sample C and D.

For sample C and D, several subpopulations of data can be distinguished for the results obtained on the 2-cm-scale but almost all laboratories reported the same result for the envelope volume determined on the 5-cm-scale, thus showing that the discrete values of the 5-cm-scale cover deviations of the values measured for depth, width and H_f of the sample.

The results for the envelope volume of sample E which was determined using the 2-cm-scale also show a broad distribution. To a certain extent, this is due to different H_f-values that were assumed by the laboratories (see Youden plots in 14.3.4). But also within laboratories that reported the same value of H_f, there is a high deviation of results. This indicates again, that the laboratories must have assumed different values for the width and the depth of the sample. Especially the low values obtained with the 2-cm-scale as well as the group of results with 375 cm^3 for the 5-cm-scale seem to be inexplicable. It is supposed that they refer to the envelope volume of the compressed tong.

Conclusions

The determination of the envelope volume is convenient because it requires only the determination of H_f and the measurement of the depth and width of the sample.

Despite this, measuring the sample dimensions caused problems. For compressible items like sample E, the instructions should specify if the sample dimensions refer to the compressed or uncompressed article.

The envelope volume is correlated to H_f as well. In consequence, the observed difficulties in the determination of H_f may have severe influence on the envelope volume and on the migration result. For those samples where the sample part defined by H_p contributes only to a minor extent to the food contact surface area (see sample A-D), the envelope volume and therefore the estimated amount of food in contact with the sample will increase more heavily with higher H_f values than the food contact surface area and hence also more heavily than the absolute migration. This will finally lead to smaller specific migration results, expressed in mg/kg. For sample A, the robust mean values of the surface area based on $H_f \geq 19$ cm were in average about 19.5% higher than those based on $H_f < 16$ cm whereas the envelope volume determined on the 2-cm-scale was about 252.8% higher and for the 5-cm-scale the increase was still 78.2%.

As a scale with discrete values for H_f, depth and width is used, some deviations in the measured sample dimensions can be compensated. The effect becomes stronger the rougher the scale is. Therefore, there were less data groups for the results obtained on the 5-cm-scale compared to the 2-cm-scale.

The main problem of using discrete scales is that if the measured values for the sample dimensions differ in a way that different values have to be assigned following the rules laid down in 14.1.7, the resulting envelope volumes can easily differ by a factor of 2. This leads to high measurement uncertainties.

10. Final conclusions

The ILC03 2013 was the first exercise to evaluate the common procedures for the determination of the surface area and also to assess the performance of the participating laboratories.

The participation in the ILC03 2013 (including the voluntary exercise) was satisfactory. The laboratory performance was satisfactory as well.

Difficulties were observed in the determination of H_f. They will not affect migration results, unless the tested articles are multi-material products or have a printing on the handle.

For the determination of the surface area, the trueness and precision of the methods depend on the sample shape. "Calculation" generates accurate results for all sample types. "Drawing the shape" is most convenient and provides accurate results for flat samples that have a negligible thickness. For round-shaped samples, "wrapping in aluminium foil" is most convenient but it overestimates the surface area. The trueness might be improved if a thicker aluminium foil is used. "Wrapping in paper" generates accurate results for flat samples and simple geometric shapes. For round-shaped samples, the surface area is overestimated as well. In general, paper is less convenient for wrapping than aluminium foil as it is less flexible.

With respect to the final migration result, the obtained reproducibility standard deviations for all four approaches to determine the surface area are acceptable because the migration measurement itself can be affected by uncertainties of similar levels as those of the determination of the surface area.

The determination of the envelope volume was convenient because it required only the determination of H_f and the measurement of the depth and width of the sample. Despite this, some difficulties were observed regarding the measurement of the sample dimensions. The determination of the envelope volume is a new approach and most of the laboratories performed this determination for the first time. The laboratory performance is expected to be improved with more training.

To avoid misunderstandings, the instructions for the determination of the surface area and the envelope volume need to be adapted.

11. Future prospects

A follow-up exercise on the determination of the surface area will be launched in 2014. Therefore new samples will be dispatched. This exercise will include the 3D laser scanning performed by the EURL-FCM. To check whether the approach of "wrapping in aluminium foil" can be improved when using a thicker foil, the laboratories will be provided with a special aluminium foil.

In preparation of the follow-up exercise, the reasons why participants obtained unacceptable results need to be figured out and the instructions will be adapted to avoid misunderstandings.

12. Acknowledgements

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

NRLs

Austria	Austrian Agency for Health and Food Safety (AGES) Institut für Lebensmittelsicherheit, Wien
Belgium	Institute of Public Health, ISSP-LP, Bruxelles
Bulgaria	National Centre of Public Health & Analysis , Sofia
Cyprus	State General Laboratory, Nicosia
Czech Republic	National Institute of Public Health, Praha 10
Denmark	National Food Institute, Technical University of Denmark, Department of Food Chemistry, Søborg
Denmark	Danish Veterinary & Food Administration, Laboratory Århus, Lystrup
Estonia	Central Laboratory of Chemistry, Tallinn
Finland	Finnish Customs Laboratory, Espoo
France	Testing Department, Laboratoire National d'Essais, Trappes Cedex
France	SCL Laboratoire de Bordeaux-Pessac, Pessac
Germany	Bundesinstitut für Risikobewertung (BfR) (Federal Institute for Risk Assessment), Berlin
Greece	General Chemical State Laboratory, Laboratory of Articles and Materials in Contact with Foodstuffs, Athens
Hungary	National Food Chain Safety Office, Food and Feed Safety Directorate, Food Toxicological NRL, Budapest
Ireland	Public Analyst's Laboratory, Dublin
Italy	Istituto Superiore di Sanità, Labor Esposizione e rischio da materiali, Roma
Latvia	Institute of Food Safety, Animal Health and Environment (BIOR), Riga
Lithuania	National Public Health Surveillance Laboratory, Laboratory of Chemistry, Vilnius
Luxembourg	Laboratoire National de Santé, Division de Contrôle Alimentaires, Luxembourg (G.D. of Luxembourg)
Malta	Department for Environmental Health, Public Health Laboratory Services, Evans Buildings, Valletta VLT
Poland	National Institute of Public Health - National Institute of Hygiene, Warsaw
Portugal	ESB Escola Superior de Biotecnologia, Universidade Católica Portuguesa, Packaging Department, Porto
Romania	NRL for Food Contact Materials, National Institute of Public Health, Bucharest
Slovakia	Regional Public Health Authority Poprad (RUVZ), Poprad
Slovenia	Institute of Public Health Maribor, Laboratory in Ljubljana, Ljubljana
Spain	Centro Nacional de Alimentación, Agencia Espanola de Seguridad, Alimentaria y Nutrición (AESAN), Majadahonda-Madrid
Sweden	National Food Agency, Uppsala
Switzerland	Official Food Control Authority, Canton of Zurich, Zürich
The Netherlands	Food & Consumer Product Safety Authority (VWA), Ministry of Economic Affairs, Agriculture & Innovation, Groningen
United Kingdom	The Food and Environment Research Agency, York

Guests

Germany	Chemisches und Veterinäruntersuchungsamt Ostwestfalen-Lippe, CVUA-OWL, Detmold
Germany	LANDESUNTERSUCHUNGSANSTALT FÜR DAS GESUNDHEITS- UND VETERINÄRWESEN SACHSEN, FG 2.3 Bedarfsgegenstände und Kosmetische Mittel, Dresden
Germany	Landeshauptstadt Düsseldorf, Der Oberbürgermeister, Amt für Verbraucherschutz, Chemische und Lebensmitteluntersuchung (39/2), Düsseldorf
Germany	Bayerisches Landesamt für Gesundheit und Lebensmittelsicherheit, Erlangen
Germany	Chemisches und Veterinäruntersuchungsamt Stuttgart, Fellbach
Germany	Landesamt für Verbraucherschutz Sachsen-Anhalt, Dezernatsleiter Bedarfsgegenstände, kosmetische Mittel, Rückstände, Kontaminanten, Halle
Germany	Stadt Hamm, Der Oberbürgermeister, Chemisches Untersuchungsamt, Hamm
Germany	Landesuntersuchungsamt Rheinland-Pfalz, Institut für Lebensmittelchemie Koblenz, Koblenz
Germany	Zentrales Institut des Sanitätsdienstes der Bundeswehr KOBLENZ, Laborabteilung III, Lebensmittelchemie und Ökochemie, Koblenz
Germany	Chemisches und Veterinäruntersuchungsamt Rheinland (AöR), Leverkusen
Germany	Niedersächsisches Amt für Verbraucherschutz und Lebensmittelsicherheit (LAVES), Institut für Bedarfsgegenstände, Lüneburg
Germany	Landeslabor Schleswig-Holstein, Neumünster
Germany	Landesamt für Verbraucherschutz (LAV) Abt. B3B, Saarbrücken
Germany	Landesbetrieb Hessisches Landeslabor, Wiesbaden
Italy	IZS Lombardia Emilia Romagna- reparto chimico degli alimenti Bologna, Bologna
Italy	Laboratorio di Sanità Pubblica Area Vasta Toscana Centro - Azienda Sanitaria di Firenze, Firenze
Italy	ARPAL dipartimento di Genova, Genova
Italy	Polo Alimenti - Arpa Piemonte, La Loggia TO
Italy	ASL Milano, Lab. di Prevenzione, Milano
Italy	ASL Provincia di Milano 1, Parabiago MI
Italy	ARPAM Dipartimento Provinciale di Pesaro, Pesaro
Italy	ARPA Lazio di Roma, UO Alimenti, Droghe e Cosmetici, Roma
Italy	ARPA Verona, SL Unita' Operativa Alimenti, Verona
Spain	Laboratorio de Salud Pública de Alicante, Alicante
Spain	Laboratorio Provincial de Salud Pública de Cádiz, Cádiz
Spain	LABORATORIO DE SALUD PÚBLICA, Hospital Monte San Isidro, 1ª planta, León
Spain	Centro Analítico de Inspección y Control de Calidad de Comercio Exterior (SOIVRE), Madrid
Spain	CNTA, San Adrián - Navarra
Spain	Laboratorio de Salud Pública de Valencia, Valencia
United Kingdom	Tayside Scientific Services, Dundee

United Kingdom	Edinburgh Scientific Services, Edinburgh
United Kingdom	Public Analyst - Kent Scientific Services, Kent County Council, Customer and Communities, Regulatory Services Group, Kent
United Kingdom	West Yorkshire Analytical Services, Leeds
United Kingdom	Public Analyst Scientific Services, Norwich
United Kingdom	Quality Manager, Worcestershire Scientific Services, Unit 5 Berkeley Business Park, Worcester

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14. Annexes

14.1. *Invitation letters and documents sent to the participants*

14.1.1. Invitation letter

Ref. Ares(2013)550265 - 05/04/2013

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for Health and Consumer Protection
Chemical Assessment and Testing Unit

EURL
European Union Reference Laboratory for Food Contact Materials

Subject: **Comparative trial ILC 2013 - 03 "Determination of surface contact area" from EURL FOOD CONTACT MATERIALS**

Ispra, 4th of April 2013

Dear Madam, Sir

On behalf of the EURL for food contact materials, I would like to invite you to participate in an inter-laboratory comparison (ILC) exercise for the determination of thesurface contact area of kitchen utensils which is due to start by the beginning of April. In the ILC 2012-01 on "Formaldehyde and melamine in 3% acetic acid migration solution" the laboratories were free to use their own method for the determination of the surface area of a spoon and the results showed substantial variation. In order to reduce that variation of the reported surface area, the NRLs were asked to provide us with their methods. From those methods we extracted four different methods. The rationale you will find in Annex I and the four methods for the determination of the surface area in Annex II. Laboratories are required to perform all four methods in ILC 2013-03 so that we can evaluate the inter-laboratory variation of each method and compare the four methods.

In one of our plenary meetings of our EURL-NRL network in 2012, Els van Hoeck (NRL-BE) presented the Envelope Volume method that is developed by the Council of Europe. This method aims to overcome the complex determination of the surface area of kitchen utensils. Although this Envelope Volume method is not an official EU approach and checking the performance of this method is not part of our work programme 2013, the EURL is of the opinion that with a little more effort and using the supplied kitchen utensils, we can be pro-active in gathering information on the performance of this approach in this ILC 2013-03 (see method description in Annex III).

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. This ILC also does not require particular skills. For these two reasons we assume that all of you 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 12th of April** to: Anja Mieth (anja.mieth@ec.europa.eu).

Direct access EURL: ph: +39.0332.785319; Fax: +39.0332.785707; e-mail: eddo.hoekstra@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 in the week of the 8th of April. 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 **10th of May 2013.**

If you have any question, please contact Eddo Hoekstra (eddo.hoekstra@ec.europa.eu).

Sincerely yours,

Eddo Hoekstra

Dr.ir. Eddo J. Hoekstra
European Union 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)
Via E. Fermi 2749
I-21027 Ispra (VA)
Italy

Annex I Rationale for selection four methods for determination of surface area
Annex II four methods for determination of surface area
Annex III Envelop Volume Method

Cc: MM. M.P. Aguar Fernandez, D. Rembges, C. Simoneau (JRC),
MM. A. Schaefer, B. Schupp, F. Vanhee (SANCO)

14.1.2. Confirmation of participation

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

EURL
European Union Reference Laboratory
for Food Contact Materials

Ispra, 4th of April 2013

Participation to EURL-FCM ILC 2013 - 03
Interlaboratory comparison (ILC) exercise
for the determination of surface contact area
CONFIRMATION OF PARTICIPATION

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

item	YES	NO
I will participate the collaborative trial on the determination of surface contact area and will deliver results on time		
I will participate the collaborative trial on the determination of Envelope Volume method and will deliver results on time		

Kindly send back this proformat to Anja Mieth (anja.mieth@ec.europa.eu) **by the 12th of April.**

Sincerely yours,

Eddo Hoekstra

Direct access EURL: ph: +39.0332.785319; Fax: +39.0332.785707; e-mail: eddo.hoekstra@ec.europa.eu

14.1.3. Shipping kit information

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

Ispra, April 8th, 2013

Shipping kit for interlaboratory comparative testing EURL-FCM ILC03 2013 – Determination of the food contact surface area of kitchen utensils

Shipping kit - samples

- sample A, B, C, D and E – Five different kitchen utensils for the determination of the food contact surface area and the envelope volume

Shipping kit - documentation

- letter of confirmation of receipt ILC 03 2013
- letter with instructions to perform the determination of the food contact surface area (part I) and the envelope volume (part II)
- letter with instructions for the compilation of the results for interlaboratory comparative testing EURL-FCM ILC03 2013
- a print copy of the Excel form "ILC03 2013 test result surface.xls" for information
- electronic Excel file "ILC03 2013 test result surface.xls" will be sent by e-mail
- a print copy of the questionnaire
- electronic Word file "ILC03 2013 questionnaire.doc" will be sent by e-mail

Storage

- the samples A, B, C, D and E should be stored at room temperature

Instructions

Result format: Use the Excel file "ILC03 2013 test result surface.xls" that will be provided by e-mail; Use the printed empty form "ILC03 2013 test result surface.xls" in case of difficulties with the Excel file.

Questionnaire: Use the Word file "ILC03 2013 questionnaire.doc" that will be provided by e-mail; Use the printed empty form "ILC03 2013 questionnaire.doc" in case of difficulties with the Word file.

14.1.4. Confirmation of the sample receipt

EUROPEAN COMMISSION
GENERAL DIRECTORATE JRC
JOINT RESEARCH CENTRE
Institute for Health and Consumer Protection – IHCP
Chemical exposure and testing

EURL
European Union Reference Laboratory
for Food Contact Materials

Ispra, April 8th, 2013

PARTICIPATION TO CRL-FCM ILC03 2013
FOOD CONTACT SURFACE AREA

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:	
E-mail:	
Phone:	

Any remarks ....

Date arrival package ....

Signature ....

Kindly send back this form to: Anja Mieth (anja.mieth@ec.europa.eu)

Sincerely yours,

Eddo Hoekstra

14.1.5. Instructions for compilation of results

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

EURL
European Union Reference Laboratory
for Food Contact Materials

Ispra, April 8th, 2013

Instructions for the compilation of the results for interlaboratory comparative testing EURL-FCM ILC03 2013

DEADLINE: Friday, May 10th 2013

Results requested:

Determination of the Food Contact Surface Area:
Perform one replicate of the determination of the food contact surface area for each sample A, B, C, D and E following the "Instructions for EURL-FCM ILC03 2013 – part I". Report all the data (incl. the height of the sample foreseeably in contact with food) using the unit of measure specified in the file "ILC03 2013 test result surface.xls".

Determination of the Envelope Volume:
Perform one replicate of the determination of the envelope volume for each sample A, B, C, D and E following the "Instructions for EURL-FCM ILC03 2013 – part II". Report all the data using the unit of measure specified in the file "ILC03 2013 test result surface.xls".

Compilation of results
Data generated by the laboratories for the comparative test EURL-FCM ILC03/2013 will be processed by the EURL-FCM using a software package for statistical analyses and professional data handling of interlaboratory tests.

For compilation of results, please fill the Excel file "ILC03 2013 test result surface.xls" and also the Word file "ILC03 2013 questionnaire.doc" and send both files back by e-mail to Anja Mieth (anja.mieth@ec.europa.eu) **by May 10th 2013**.

If you have any question, please contact Eddo Hoekstra (eddo.hoekstra@jrc.ec.europa.eu), phone: +39 0332 785319

Sincerely yours,

Eddo Hoekstra

14.1.6. Instructions for the determination of the food contact surface area

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

Ref. Ares(2013)629423 - 10/04/2013

EURL
European Union Reference Laboratory
for Food Contact Materials

Ispra, April 5th, 2013

Instructions for the EURL-FCM ILC03 2013
– part I. Determination of the Food Contact Surface Area –

I.1 Determination of the sample part that will foreseeably be in contact with food

Scope

This protocol explains how to determine the part of a kitchen utensil that will foreseeably be in contact with food. The description refers to the concept proposed by the Council of Europe.

Reagents and laboratory equipment

- certified ruler (accuracy 0.1 cm)

Procedure

The total length of the sample (incl. the handle) is measured (H_{total}) (for visual explanation see Figure 1). Then the length of the handle is measured (H_{handle}). If the handle is not clearly separated, a default length of 1/3 of H_{total} is assigned. Then, the part reasonably in contact with food (H_r) is determined. It results from: $H_r = H_{total} - H_{handle}$.

Next, the part which is necessarily in contact with food (H_n) is measured and the part that is probably in contact with food (H_p) is calculated. It arises from: $H_p = H_r - H_n$.

If $H_p \leq 0.5\ H_n$, the height of the sample that will foreseeably be in contact with food (H_f) is considered to be equal to H_r. Otherwise, a value of 2/3 of H_r is assigned to H_f.

Number of Replicates and Report

For the ILC, the determination shall be performed a single time. Please report the obtained value for H_f (see Excel file "ILC03 2013 test result surface.xls").

1

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Institute for Health and Consumer Protection – IHCP
Chemical Assessment and testing

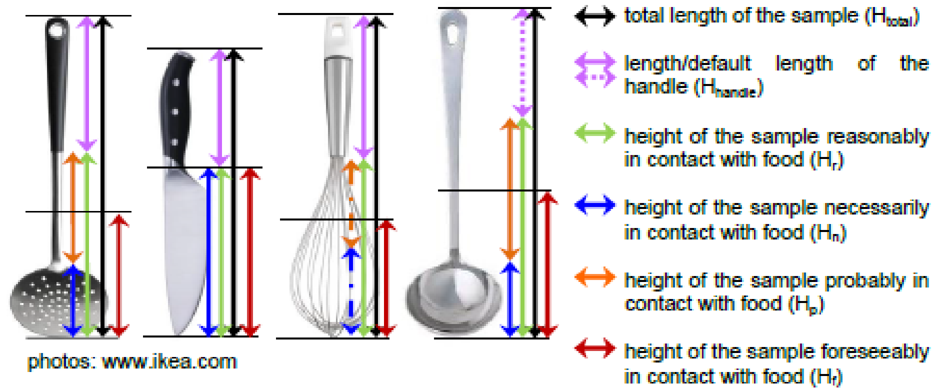


Figure 1 Determination of the sample height foreseeably in contact with food (H_f)

2

I.2 Determination of the food contact surface area by calculation

Scope

This protocol explains how to determine the surface area of a kitchen utensil by calculation after measuring the sample dimensions with a ruler or caliper.

Reagents and laboratory equipment

- certified ruler (accuracy 0.1 cm)
- certified caliper (accuracy 0.01 mm)

Procedure

At first, the part of the sample is determined that will foreseeably be in contact with food. This is done as described in section I.1.

Then the shape of this sample part is broken down to regular geometric shapes (e.g. cylinders, rectangular solids, truncated cones, trapezoids, ellipsoids). If necessary, an irregular shape can be divided into different triangles or trapezoids.

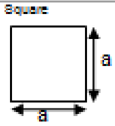
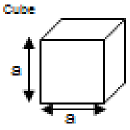
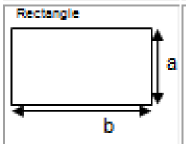
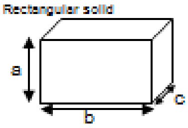
The surface area of each subpart is calculated according to the corresponding mathematical formulas (examples are listed in Table 1). For each subpart, the dimensions needed for calculation are measured with a ruler or caliper.

The total food contact area is the sum of all subareas.

Number of Replicates and Report

For the ILC, the determination shall be performed a single time. Please report the calculated value for the area that will be foreseeably in contact with food (see Excel file "ILC03 2013 test result surface.xls").

Table 1 Surface area formulae for regular geometric shapes

Shape	Surface area formula	Shape	Surface area formula
Square 	Surface = a^2	Cube 	$S = 6a^2$ a: length of the side of each edge of the cube
Rectangle 	$S = ab$	Rectangular solid 	$S = 2ab + 2bc + 2ac = 2(ab + bc + ca)$ a, b, c: lengths of the 3 sides

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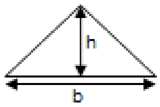
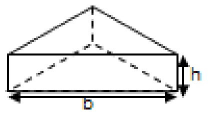
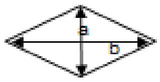
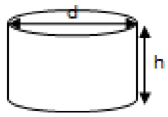
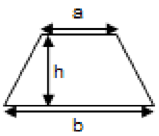
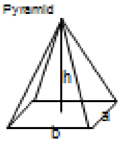
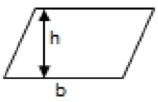
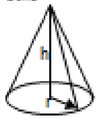
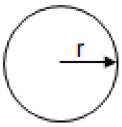
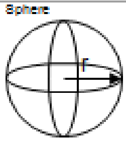
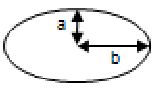
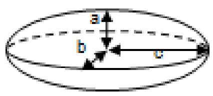
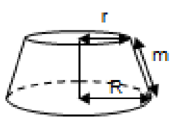
Institute for Health and Consumer Protection – IHCP
Chemical Assessment and testing

Shape	Surface area formula	Shape	Surface area formula
Triangle 	S =bh/2	Prism 	S = 2b + Ph (*b is the area of the base P is the perimeter of the base)*
Diamond 	S = (ab) /2	Cylinder 	Lateral surface area S = πdh S = 2 πrh + 2 πr² S = areas of top and bottom + area of the side S = 2(area of top) + (perimeter of top) · height h: height of the cylinder r : radius of the top
Trapezoid 	S = 1/2 (base 1 + base 2) · height S = h(a+b)/2	Pyramid 	S: Add the Area of the base to the sum of the areas of all of the triangular faces. The areas of the triangular faces will have different formulas for different shaped bases. b is the Area of the base
Parallelogram 	S = base · height S = b h	Cone 	$S = \pi r^2 + \pi rs$ $= \pi r^2 + \pi r\sqrt{r^2 + h^2}$
Circle 	$S = \pi r^2$ r: radius of the circle (distance from the center to any point on the circle)	Sphere 	$S = 4\pi r^2$
Ellipse 	$S = \pi \cdot a \cdot b$ a, b: length of semi-axis	Triaxial Ellipsoid 	$S = 4\pi\left(\frac{(ab)^{1.6} + (ac)^{1.6} + (bc)^{1.6}}{3}\right)^{0.625}$ a, b, c: length of semi-axis in x-, y- and z-direction This equation yields to an approximate value for the surface of a triaxial ellipsoid. The relative error is less than ~1.2 %. (reference: www.numericana.com/answer/ellipsoid.htm#thomsen)
Spherical cap 	$S = \pi \cdot (a^2 + h^2)$ h: height of the spherical cap a: radius of the base of the spherical cap	Truncated cone 	$S_{top} = \pi \cdot r^2$ $S_{base} = \pi \cdot R^2$ $S_{lateral} = \pi \cdot m \cdot (r + R)$ $S_{total} = S_{top} + S_{base} + S_{lateral}$ S_{top}: area of the top (circle) S_{base}: area of the base (circle) $S_{lateral}$: area of the lateral surface

4

I.3 Determination of the food contact surface area by wrapping in paper and direct weighing

Scope

This protocol explains how to determine the surface area of a kitchen utensil by wrapping in paper and weighing the paper.

Reagents and laboratory equipment

- paper with a constant grammage (e.g. 80 g/m^2)
- scissors or scalpel
- balance (accuracy 0.0001 g)

Procedure

At first, the part of the sample is determined that will foreseeably be in contact with food. This is done as described in section I.1.

Before wrapping the sample, the paper must be checked for a sufficiently constant and homogenous grammage. The deviation within each sheet and between different sheets of the same paper batch shall not exceed ±1% of the average grammage.

The surface weight (grammage) of the paper is determined with paper from the same batch. It can be done e.g. by cutting 6 pieces of paper, each with an area of 1 dm². Another approach is to prepare a 5-step calibration (e.g. with pieces of 0.25, 0.5, 1.0, 1.5, 2.0 dm²). The intercept is set to zero. The slope corresponds to the paper grammage.

Then the part of the sample that will foreseeably be in contact with food (see section I.1) is wrapped in paper. Wrapping is done as tight as possible. Excess paper is removed with a scalpel or scissors. Afterwards, the sample is unwrapped and the wrapping material (paper) is weighed. Knowing the paper grammage, the surface area of the sample can be calculated.

NOTE: The term wrapping shall be interpreted in a very broad sense. Important is that the paper that is weighted at the end, represents the foreseeable contact surface area of the article.

Number of Replicates and Report

For the ILC, the determination shall be performed a single time. Please report the obtained value for the area that will be foreseeably in contact with food (see Excel file "ILC03 2013 test result surface.xls").

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I.4 Determination of the food contact surface area by wrapping in aluminium foil and direct weighing

Scope

This protocol explains how to determine the surface area of a kitchen utensil by wrapping in aluminium foil and weighing the aluminium foil.

Reagents and laboratory equipment

- aluminium foil with a constant surface weight (common household aluminium foil is sufficient)
- scissors or scalpel
- balance (accuracy 0.0001 g)

Procedure

At first, the part of the sample is determined that will foreseeably be in contact with food. This is done as described in section I.1.

Before wrapping the sample, the aluminium foil must be checked for a sufficiently constant and homogenous grammage. The deviation within the same batch/roll shall not exceed ±1% of the average grammage.

The surface weight of the aluminium foil is determined with foil of the same batch/roll. It can be done e.g. by cutting 6 pieces of foil, each with an area of 1 dm^2. Another approach is to prepare a 5-step calibration (e.g. with pieces of 0.25, 0.5, 1.0, 1.5, 2.0 dm^2). The intercept is set to zero. The slope corresponds to the surface weight of the aluminium foil.

Then the part of the sample that will foreseeably be in contact with food (see section I.1) is wrapped in aluminium foil. Wrapping is done as tight as possible. Excess aluminium foil is removed with a scalpel or scissors. Afterwards, the sample is unwrapped and the wrapping material (aluminium foil) is weighed. Knowing the surface weight of the aluminium foil, the surface area of the sample can be calculated.

NOTE: The term wrapping shall be interpreted in a very broad sense. Important is that the paper that is weighted at the end, represents the foreseeable contact surface area of the article.

Number of Replicates and Report

For the ILC, the determination shall be performed a single time. Please report the obtained value for the area that will be foreseeably in contact with food (see Excel file "ILC03 2013 test result surface.xls").

I.5 Determination of the food contact surface area by drawing the sample outline on paper

Scope

This protocol explains how to determine the surface area of a kitchen utensil by drawing its outline on paper.

Reagents and laboratory equipment

- paper with a constant grammage (e.g. 80 g/m²)
- pencil
- scissors or scalpel
- balance (accuracy 0.0001 g)

Procedure

At first, the part of the sample is determined that will foreseeably be in contact with food. This is done as described in section I.1.

Before drawing the sample outline on paper, the paper must be checked for a sufficiently constant and homogenous grammage. The deviation within each sheet and between different sheets of the same paper batch shall not exceed ±1% of the average grammage.

The surface weight (grammage) of the paper is determined with paper from the same batch. It can be done e.g. by cutting 6 pieces of paper, each with an area of 1 dm². Another approach is to prepare a 5-step calibration (e.g. with pieces of 0.25, 0.5, 1.0, 1.5, 2.0 dm²). The intercept is set to zero. The slope corresponds to the paper grammage.

Then the sample is placed on a sheet of paper and its outline is drawn on the paper. Only the part of the sample that will foreseeably be in contact with food is outlined (see section I.1). To make sure that the outline is representative for the real surface, the sample can be cut into smaller pieces and the outline of each of the single pieces is drawn on paper. The drawings are cut and weighed. Knowing the paper grammage, the surface area of the sample can be calculated.

NOTE: If the kitchen utensil is cut into smaller pieces, please make sure that the determination procedures I.1-I.4 are performed before with the intact article.

Number of Replicates and Report

For the ILC, the determination shall be performed a single time. Please report the obtained value for the area that will be foreseeably in contact with food (see Excel file "ILC03 2013 test result surface.xls").

14.1.7. Instructions for the determination of the envelope volume ILC03 2013

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Ref. Ares(2013)629423 - 10/04/2013

EURL
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Ispra, April 5th, 2013

Instructions for the EURL-FCM ILC03 2013
– part II. Envelope Volume –

II.1 Calculation of the Envelope Volume (for determination of the specific migration)

The "envelope volume" concept is proposed by the Council of Europe in a draft for a new resolution. It does not represent a way to determine the surface area of a kitchen article but it is useful in assessing the specific migration of samples.

The envelope volume of a kitchen utensil is an estimated value for the amount of food that comes into contact with the article. Limits for the specific migration always refer to mg/kg foodstuff. According to Article 17 of the Regulation 10/2011, "*specific migration values shall be expressed in mg/kg applying the real surface to volume ratio in actual or foreseen use*". This implies that the amount of food is known with which the article will be in contact. If this amount is unknown (e.g. in case of kitchen utensils), usually a value of 6 dm^2 per kg foodstuff is assumed. This is a default value and does not represent real use conditions for all types of samples. The envelope volume offers the possibility to obtain a more reasonable value for the amount of food in contact with the article.

The principal of the determination is as follows. The dimensions (depth x, width y, height z) of the sample part that will be in contact with food are determined on a 5-cm-scale. The envelope volume is the product of $x \cdot y \cdot z$ (in cm^3). Then, the reference weight W_{ref} (kg) results from: envelope volume (cm^3)/1000.
To determine the specific migration SM of a substance from the sample, the migrated mass M of this substance is divided by the reference weight: $SM = M/W_{ref}$

It was suggested to perform also the calculation of the envelope volume on the samples that will be delivered to the NRLs for the Interlaboratory Comparison Exercise. For this exercise, the envelope volume shall be determined using a 2-cm-scale and a 5-cm-scale. A description is given in section II.2.

1

II.2 Operation procedure - Calculation of the envelope volume on a 2-cm-scale and a 5-cm-scale

Scope

This protocol explains how to determine the envelope volume of a kitchen utensil on a 2-cm-scale and a 5-cm-scale.

Reagents and laboratory equipment

- certified ruler (accuracy 0.1 cm)

Procedure

Determination of x-, y- and z-value

The actual depth (x) and width (y) of the sample are measured with a ruler. The z-value, i.e. the height of the sample foreseeably in contact with food (H_f), is determined as described in Instructions, part I, section I.1. The obtained values (0.1 cm accuracy) are transferred to rounded values on a 2-cm-scale and a 5-cm-scale. On the 5-cm-scale, x, y and z can take values of 5, 10, 15, 20, 25 or 30 cm (see Table 1) with 5 cm being the smallest value and 30 cm being the largest value. On the 2-cm-scale, they can take values between 2 cm and 30 cm (see Table 2).

Table 1 Assigned values for x, y and z using a 5-cm-scale

measured values for actual depth (x), width (y) and height (z) [cm]	values to be assigned for x, y or z on the 5-cm-scale
≤ 5 cm	5 cm
5 cm < measured value ≤ 10 cm	10 cm
10 cm < measured value ≤ 15 cm	15 cm
15 cm < measured value ≤ 20 cm	20 cm
20 cm < measured value ≤ 25 cm	25 cm
> 25 cm	30 cm

Table 2 Assigned values for x, y and z using a 2-cm-scale

measured values for actual depth (x), width (y) and height (z) [cm]	values to be assigned for x, y or z on the 2-cm-scale
≤ 2 cm	2 cm
2 cm < measured value ≤ 4 cm	4 cm
4 cm < measured value ≤ 6 cm	6 cm
...	...
24 cm < measured value ≤ 26 cm	26 cm
26 cm < measured value ≤ 28 cm	28 cm
> 28 cm	30 cm

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Calculation of the envelope volume

Having determined the assigned values for x, y and z, the envelope volume is derived from:

$$V_{envelope} = x_{ass} \cdot y_{ass} \cdot z_{ass}$$

$V_{envelope}$: envelope volume [cm^3]
x_{ass}, y_{ass}, z_{ass}: assigned values for x, y and z, respectively [cm]

Number of Replicates and Report

For the ILC, the determination shall be performed a single time. Please report the obtained value for the envelope volume using the 2-cm-scale and the 5-cm-scale (see Excel file "ILC03 2013 test result surface.xls").

3

14.1.8. Excel file for compilation of results

FCM EURL-NRL ILC 2013_003 Food Contact Surface Area

TEST RESULTS

Lab Code - LC00....

I. Determination of the food contact surface area (Please insert your data with 1 decimal place.)

Sample	operation procedure I.1 height foreseeably in food contact (H_f) [cm]	operation procedure I.2 **calculation** Food contact surface area [cm²]	operation procedure I.3 **wrap in paper** Food contact surface area [cm²]	operation procedure I.4 **wrap in Al foil** Food contact surface area [cm²]	operation procedure I.5 **draw shape** Food contact surface area [cm²]	remark*
A						
B						
C						
D						
E						

* Please report whether the article was cut into smaller pieces (p) or left uncut (u) for drawing the shape.

II. Determination of the envelope volume (Please insert your data with 1 decimal place.)

Sample	operation procedure II.2 **2-cm-scale** envelope volume [cm³]	operation procedure II.2 **5-cm-scale** envelope volume [cm³]
A		
B		
C		
D		
E		

....
Place and date

....
Laboratory Manager

....
Signature

14.1.9. Questionnaire

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Questionnaire for the EURL-FCM ILC03 2013

Please evaluate the performed methods for the determination of the surface area by answering the following questions!

1) How much time did you spend approximately to determine the surface area using this method? (hours/min)

	Sample A	Sample B	Sample C	Sample D	Sample E
I.2 calculation					
I.3 wrap in paper					
I.4 wrap in aluminium foil					
I.5 draw shape					

comments: ____________________

2) Please choose the method that is most suitable in your opinion for the particular sample! (Please mark with a cross!)

	Sample A	Sample B	Sample C	Sample D	Sample E
I.2 calculation					
I.3 wrap in paper					
I.4 wrap in aluminium foil					
I.5 draw shape					

comments: ____________________

3) Is this method currently in use in your laboratory? (Please mark with a cross!)

	frequently	sometimes	hardly	not anymore	never
I.2 calculation					
I:3 wrap in paper					
I.4 wrap in aluminium foil					
I.5 draw shape					

comments: ____________________

1

4) Would you implement this method in your daily laboratory work, expressed on a scale from 1 to 6 (1 = of course, 6 = never)?

of course ← 1 — 2 — 3 — 4 — 5 — 6 → never

	implementation
I.2 calculation	
I.3 wrap in paper	
I.4 wrap in aluminium foil	
I.5 draw shape	

comments: ____________________

5) Please rank the methods according to your personal preference (1 = the best, 4 = the worst)!

	rank
I.2 calculation	
I.3 wrap in paper	
I.4 wrap in aluminium foil	
I.5 draw shape	

comments: ____________________

6) **General Comments**

Please feel free to make any additional comment here.

14.2. Results of the homogeneity studies (sample A-E)

Sample A

Figure 10 Dimensions measured for homogeneity testing (Sample A)

Table 20 Measured values – Homogeneity Testing (Sample A)

sample A flat spatula	1	2	3	4	5	6	7	8	9	10
max. width (food contact part) [cm]	5.762	5.708	5.758	5.736	5.750	5.752	5.736	5.724	5.760	5.740
length middle slot [cm]	8.756	8.776	8.790	8.782	8.794	8.784	8.784	8.786	8.804	8.796
max. width handle [cm]	2.716	2.710	2.716	2.726	2.714	2.716	2.730	2.720	2.718	2.740
thickness (handle, top end) [cm]	0.496	0.496	0.510	0.490	0.504	0.508	0.494	0.518	0.492	0.510
thickness (food contact part) [cm]	0.144	0.140	0.142	0.140	0.144	0.144	0.142	0.140	0.142	0.144

Table 21 Statistical evaluation – Homogeneity Testing (Sample A)

sample A flat spatula	mean [cm]	s.d. [cm]	CV [%]
max. width (food contact part) [cm]	5.743	0.017	0.30
length middle slot [cm]	8.785	0.013	0.15
max. width handle [cm]	2.721	0.009	0.33
thickness (handle, top end) [cm]	0.502	0.009	1.88
thickness (food contact part) [cm]	0.142	0.002	1.23

Sample B

Figure 11 Dimensions measured for homogeneity testing (Sample B)

Table 22 Measured values – Homogeneity Testing (Sample B)

sample B fork	1	2	3	4	5	6	7	8	9	10
max. width (food contact part) [cm]	5.112	5.134	5.122	5.134	5.134	5.138	5.142	5.128	5.138	5.138
max. width handle [cm]	2.704	2.716	2.724	2.734	2.734	2.742	2.726	2.732	2.720	2.720
thickness (handle, top end) [cm]	0.510	0.492	0.484	0.492	0.494	0.486	0.492	0.480	0.484	0.508
thickness (prong 3) [cm]	0.266	0.276	0.278	0.266	0.276	0.272	0.286	0.260	0.268	0.262
width prong 3 [cm]	0.772	0.808	0.774	0.786	0.786	0.782	0.782	0.786	0.786	0.780

Table 23 Statistical evaluation – Homogeneity Testing (Sample B)

sample B fork	mean [cm]	s.d. [cm]	CV [%]
max. width (food contact part) [cm]	5.132	0.009	0.18
max. width handle [cm]	2.725	0.011	0.40
thickness (handle, top end) [cm]	0.492	0.010	2.02
thickness (prong 3) [cm]	0.271	0.008	2.98
width prong 3 [cm]	0.784	0.010	1.25

Sample C

Figure 12 Dimensions measured for homogeneity testing (Sample C)

Table 24 Measured values – Homogeneity Testing (Sample C)

sample C oval spoon	1	2	3	4	5	6	7	8	9	10
max. width (food contact part) [cm]	5.998	5.992	6.002	6.022	6.024	5.956	6.016	6.002	6.006	6.040
max. width handle [cm]	2.724	2.720	2.720	2.726	2.726	2.716	2.730	2.720	2.720	2.720
thickness (handle, top end) [cm]	0.488	0.484	0.482	0.484	0.488	0.484	0.494	0.492	0.482	0.482
max. height (food contact part) [cm]	2.198	2.200	2.190	2.196	2.204	2.220	2.186	2.200	2.194	2.198
thickness (food contact part) [cm]	0.130	0.128	0.128	0.118	0.126	0.122	0.116	0.120	0.114	0.122

Table 25 Statistical evaluation – Homogeneity Testing (Sample C)

sample C oval spoon	mean [cm]	s.d. [cm]	CV [%]
max. width (food contact part) [cm]	6.006	0.023	0.38
max. width handle [cm]	2.722	0.004	0.15
thickness (handle, top end) [cm]	0.486	0.004	0.89
max. height (food contact part) [cm]	2.199	0.009	0.42
thickness (food contact part) [cm]	0.122	0.005	4.48

Sample D

Figure 13 Dimensions measured for homogeneity testing (Sample D)

Table 26 Measured values – Homogeneity Testing (Sample D)

sample D rectangular spoon	1	2	3	4	5	6	7	8	9	10
max. width (food contact part) [cm]	6.168	6.154	6.172	6.150	6.178	6.158	6.138	6.146	6.168	6.172
max. width handle [cm]	2.716	2.726	2.740	2.738	2.720	2.720	2.712	2.724	2.726	2.726
thickness (handle, top end) [cm]	0.496	0.506	0.492	0.498	0.498	0.494	0.500	0.508	0.498	0.490
max. height (food contact part) [cm]	2.118	2.120	2.142	2.116	2.118	2.118	2.106	2.114	2.114	2.118
thickness (food contact part) [cm]	0.138	0.134	0.130	0.138	0.140	0.138	0.134	0.128	0.132	0.136

Table 27 Statistical evaluation – Homogeneity Testing (Sample D)

sample D rectangular spoon	mean [cm]	s.d. [cm]	CV [%]
max. width (food contact part) [cm]	6.160	0.013	0.21
max. width handle [cm]	2.725	0.009	0.32
thickness (handle, top end) [cm]	0.498	0.006	1.14
max. height (food contact part) [cm]	2.118	0.009	0.43
thickness (food contact part) [cm]	0.135	0.004	2.90

Sample E

Figure 14 Dimensions measured for homogeneity testing (Sample E)

Table 28 Measured values – Homogeneity Testing (Sample E)

sample E tongs	1	2	3	4	5	6	7	8	9	10
max. width handle [cm]	2.722	2.728	2.720	2.716	2.726	2.710	2.722	2.726	2.726	2.724
min. width (food contact part) left [cm]	1.000	0.992	1.000	1.000	0.998	1.022	1.002	1.010	1.000	1.004
min. width (food contact part) right [cm]	1.000	0.996	1.012	0.996	1.006	1.000	1.016	0.992	1.004	1.004
thickness (food contact part) [cm]	0.426	0.400	0.410	0.424	0.406	0.402	0.400	0.410	0.410	0.408
thickness (food contact part) [cm]	0.410	0.400	0.406	0.424	0.410	0.406	0.402	0.404	0.420	0.412

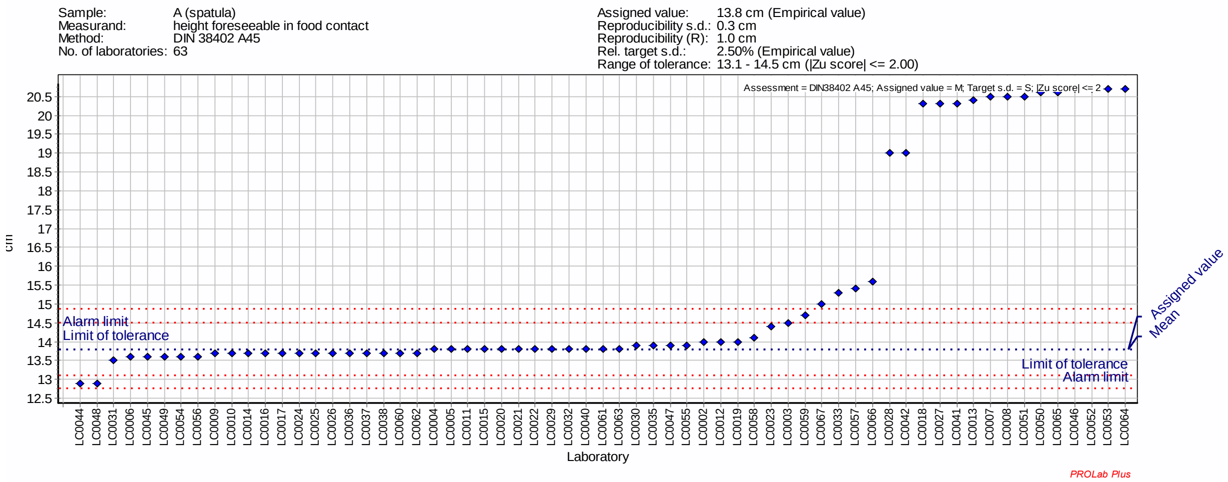
Table 29 Statistical evaluation – Homogeneity Testing (Sample E)

sample E tongs	mean [cm]	s.d. [cm]	CV [%]
max. width handle [cm]	2.722	0.005	0.20
min. width (food contact part) left [cm]	1.003	0.008	0.81
min. width (food contact part) right [cm]	1.003	0.007	0.74
thickness (food contact part) [cm]	0.410	0.009	2.21
thickness (food contact part) [cm]	0.409	0.008	1.87

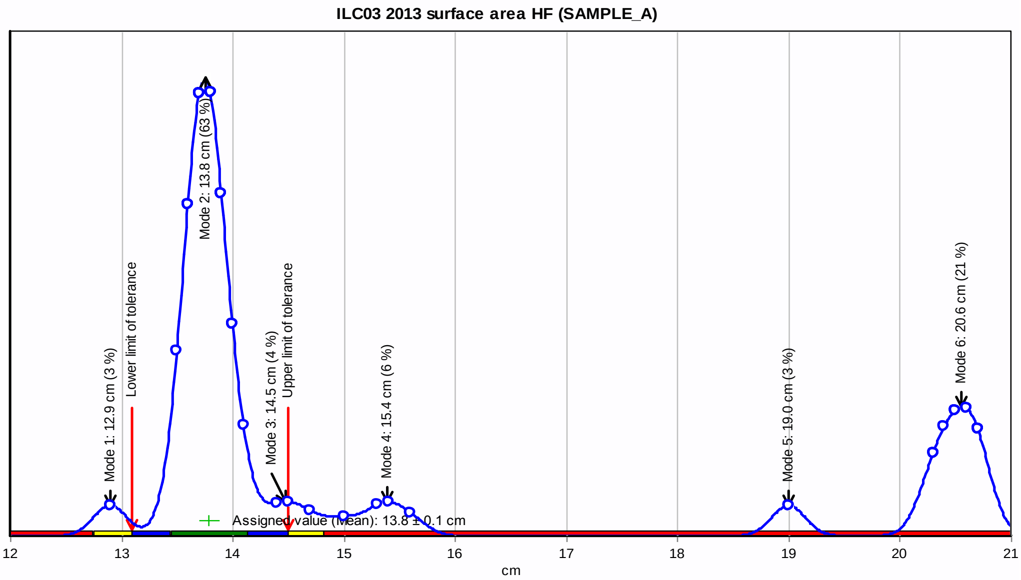
14.3. *Reported results and z_U-scores*

14.3.1. Reported results and z_U-scores for the sample height with foreseeable food contact (H_f)

(a)



(b)



(c)

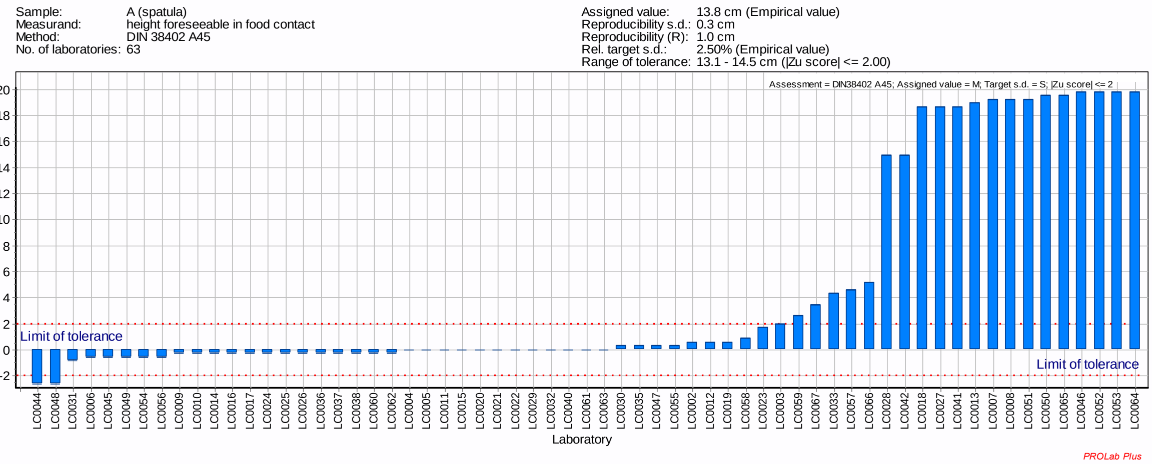


Figure 15 Summary of reported test results for the sample height foreseeably in food contact (H_f) of Sample A (a), Kernel density estimation (b) and z_U-scores (c)

(a)

(b)

(c)

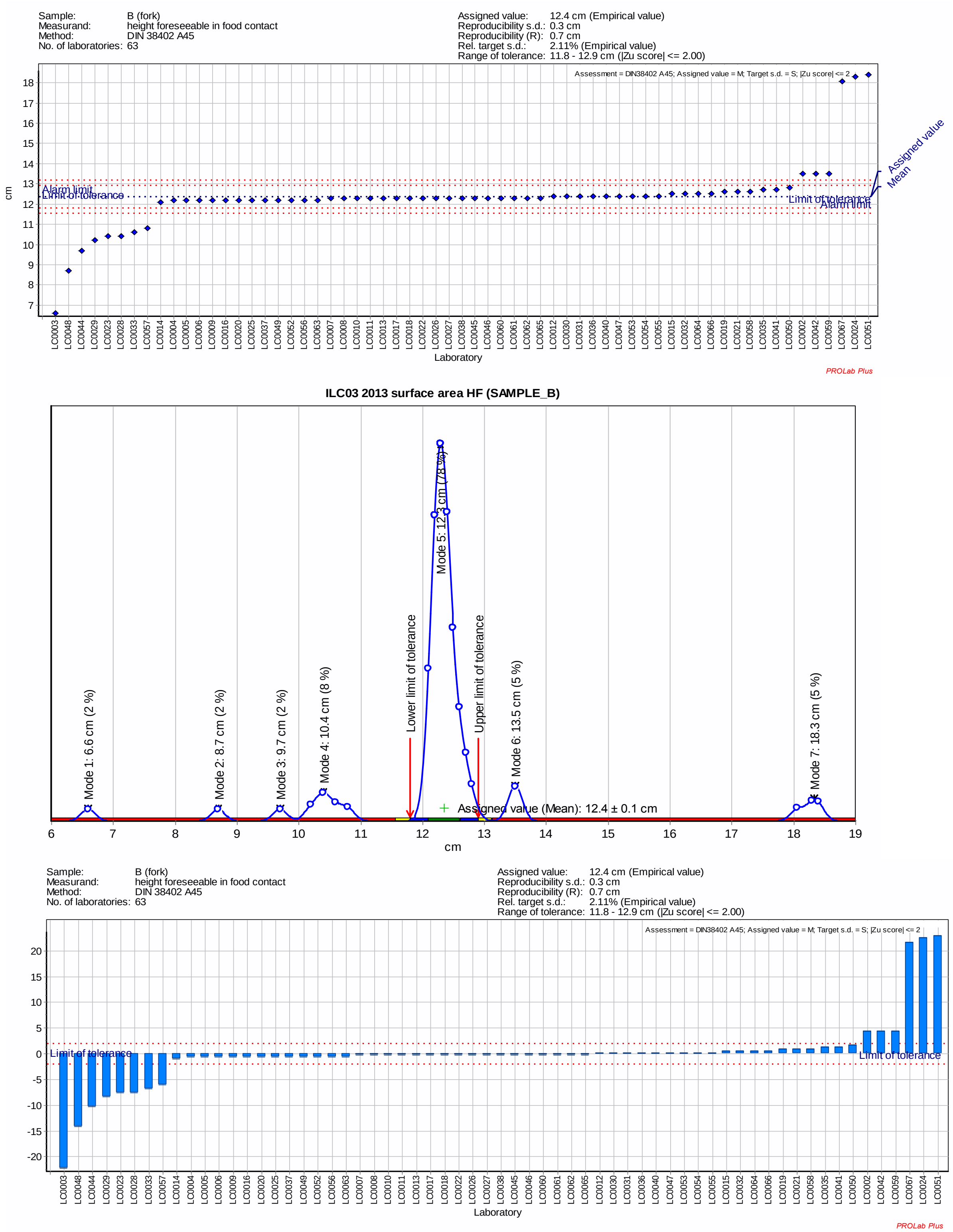


Figure 16 Summary of reported test results for the sample height foreseeably in food contact (H_f) of Sample B (a), Kernel density estimation (b) and z_U-scores (c)

(a)

(b)

(c)

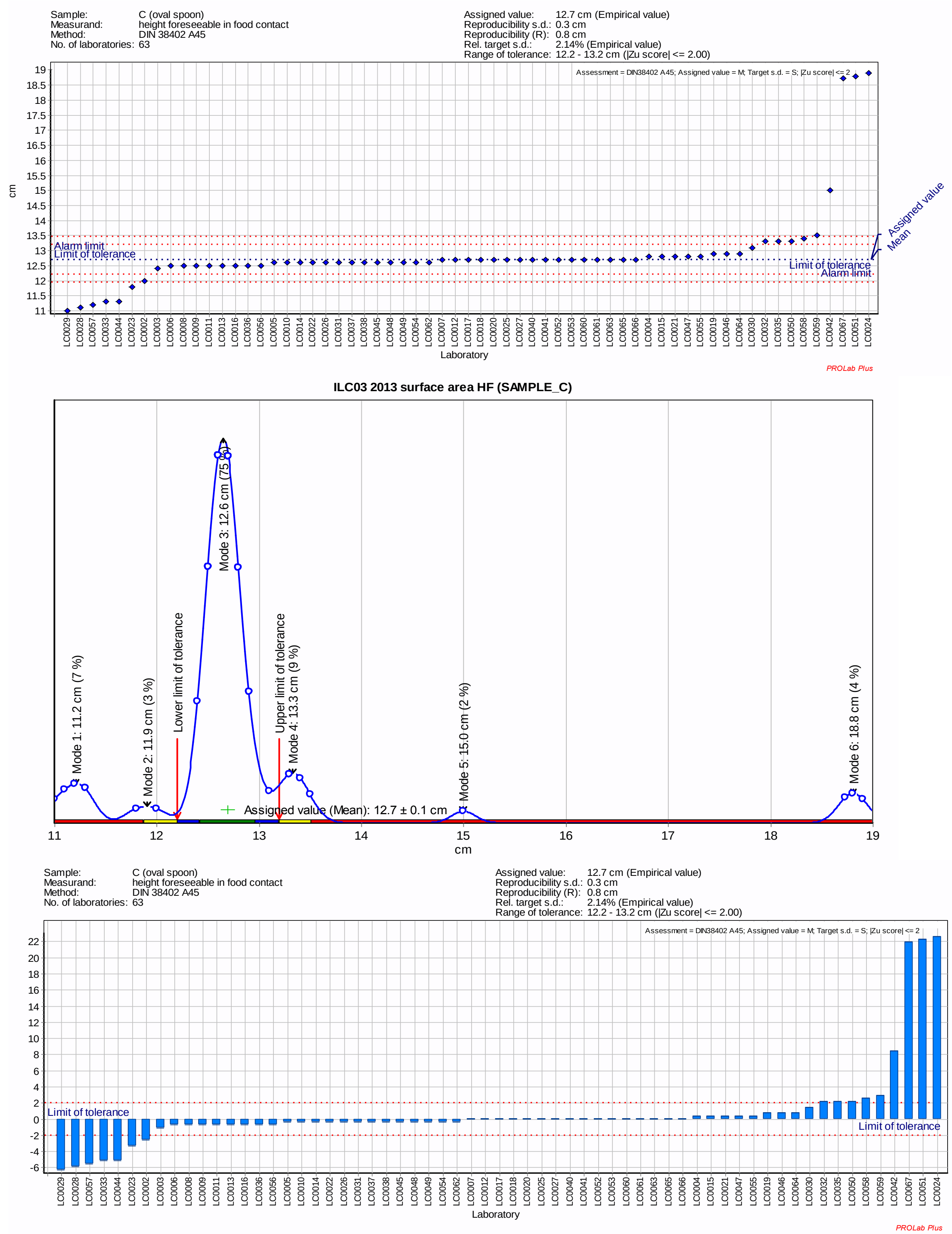


Figure 17 Summary of reported test results for the sample height foreseeably in food contact (H_f) of Sample C (a), Kernel density estimation (b) and z_U-scores (c)

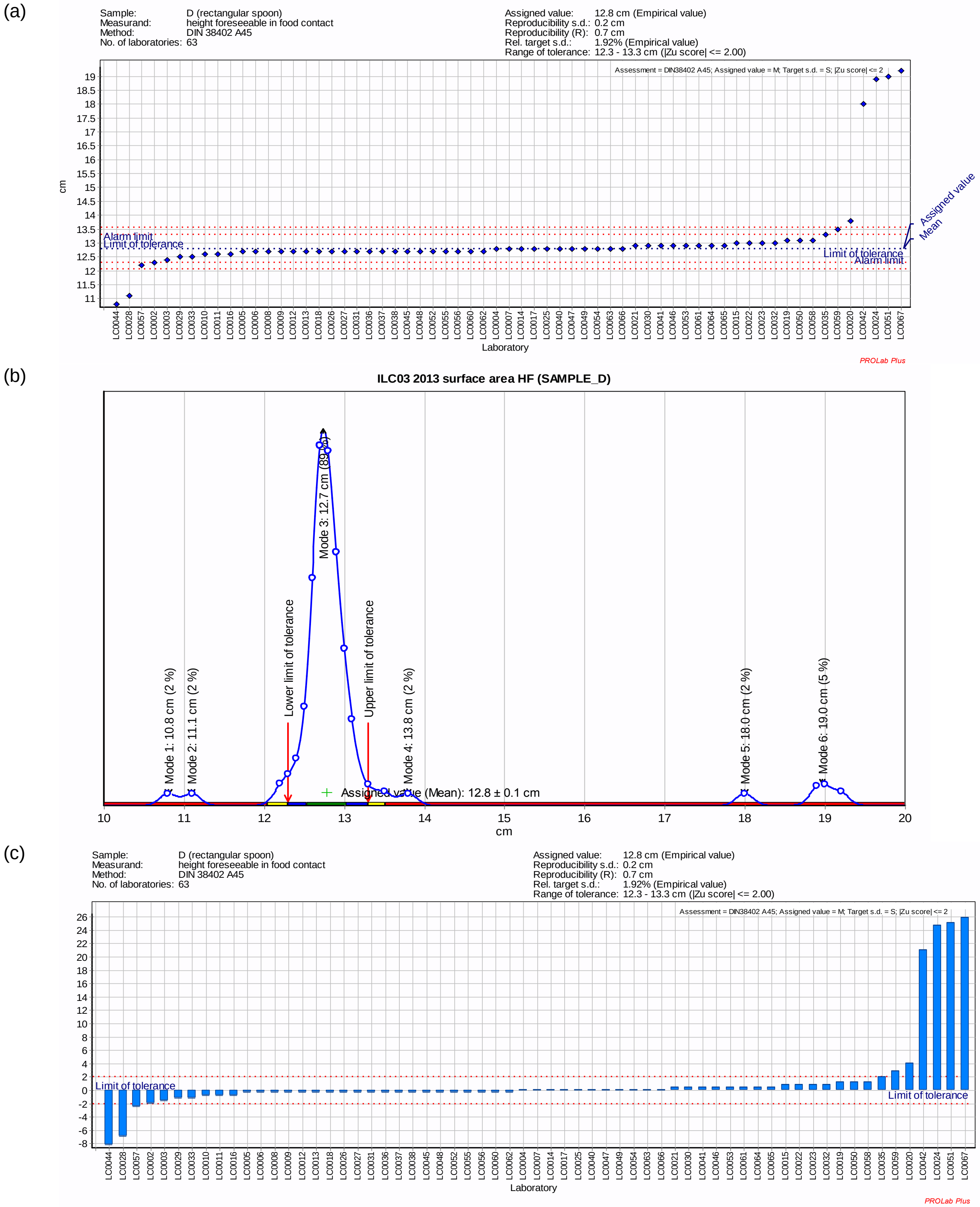


Figure 18 Summary of reported test results for the sample height foreseeably in food contact (H_f) of Sample D (a), Kernel density estimation (b) and z_U-scores (c)

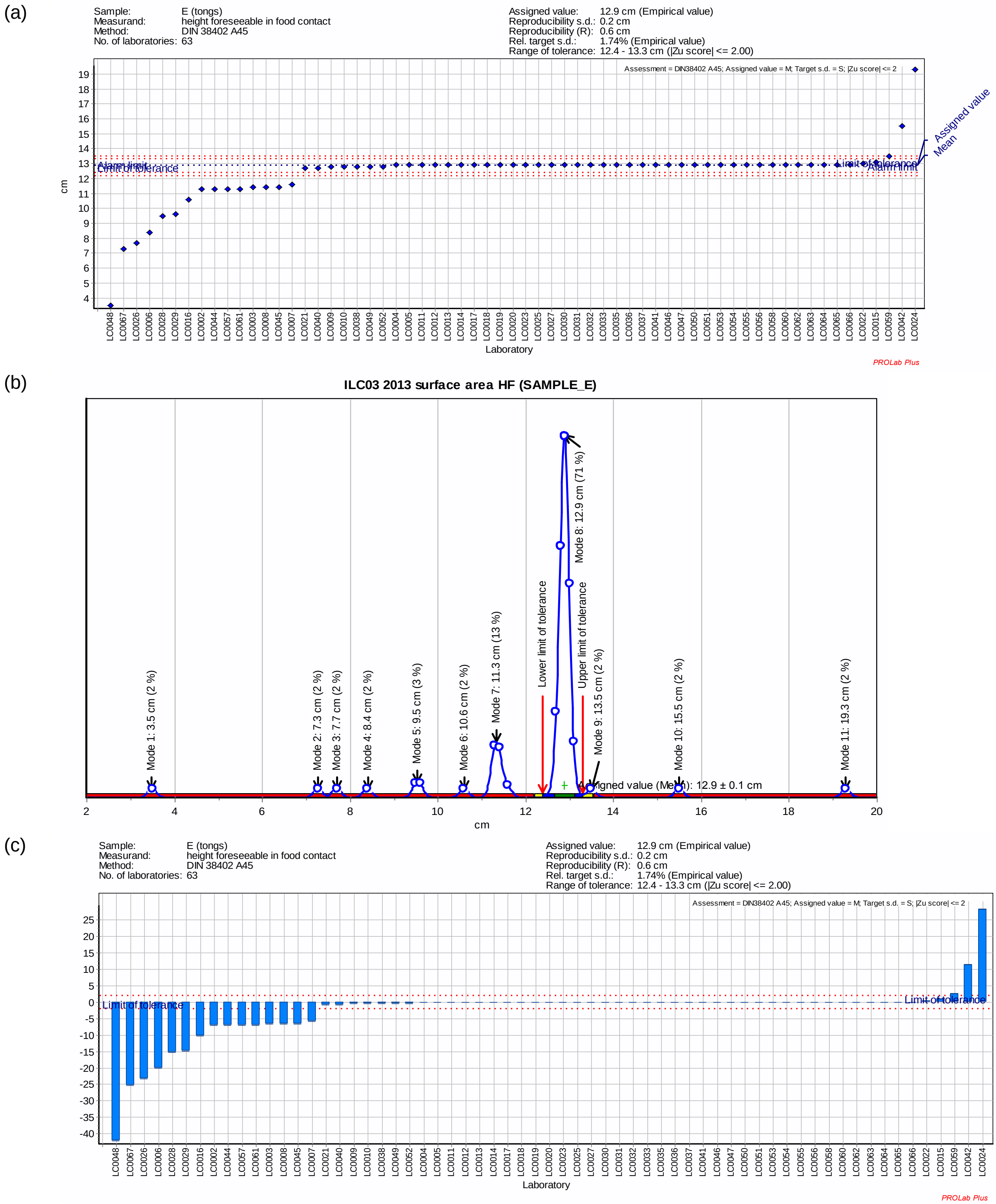


Figure 19 Summary of reported test results for the sample height foreseeably in food contact (H_f) of Sample E (a), Kernel density estimation (b) and z_U-scores (c)

14.3.2. Reported results and z_U-scores for the food contact surface area

14.3.2.1. Reported results and z_U-scores for the food contact surface area of sample A

(a)

(b)

(c)

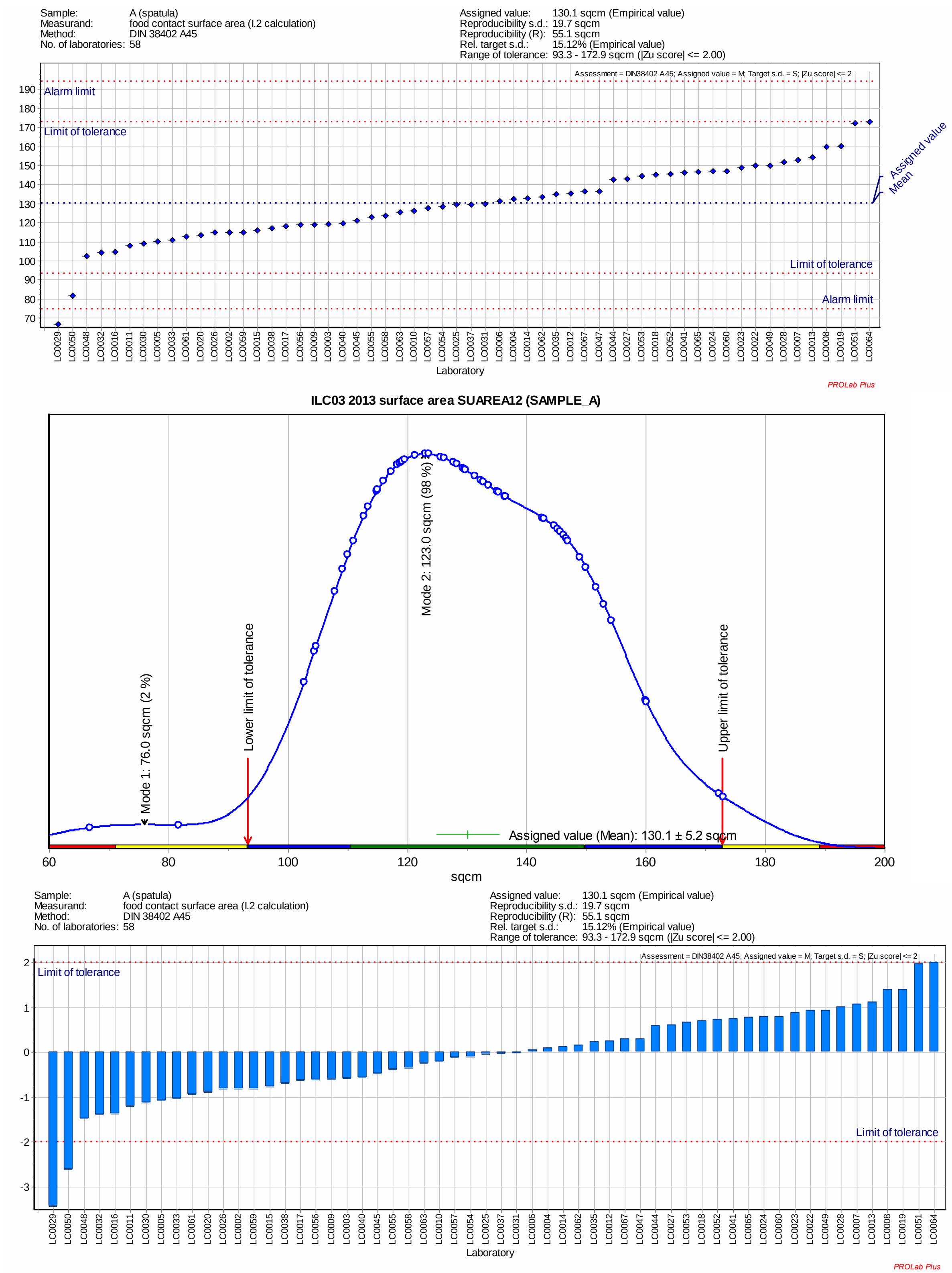


Figure 20 Summary of reported test results for the food contact surface area of Sample A determined by "calculation" (a), Kernel density estimation (b) and z_U-scores (c)

(a) (b) (c)

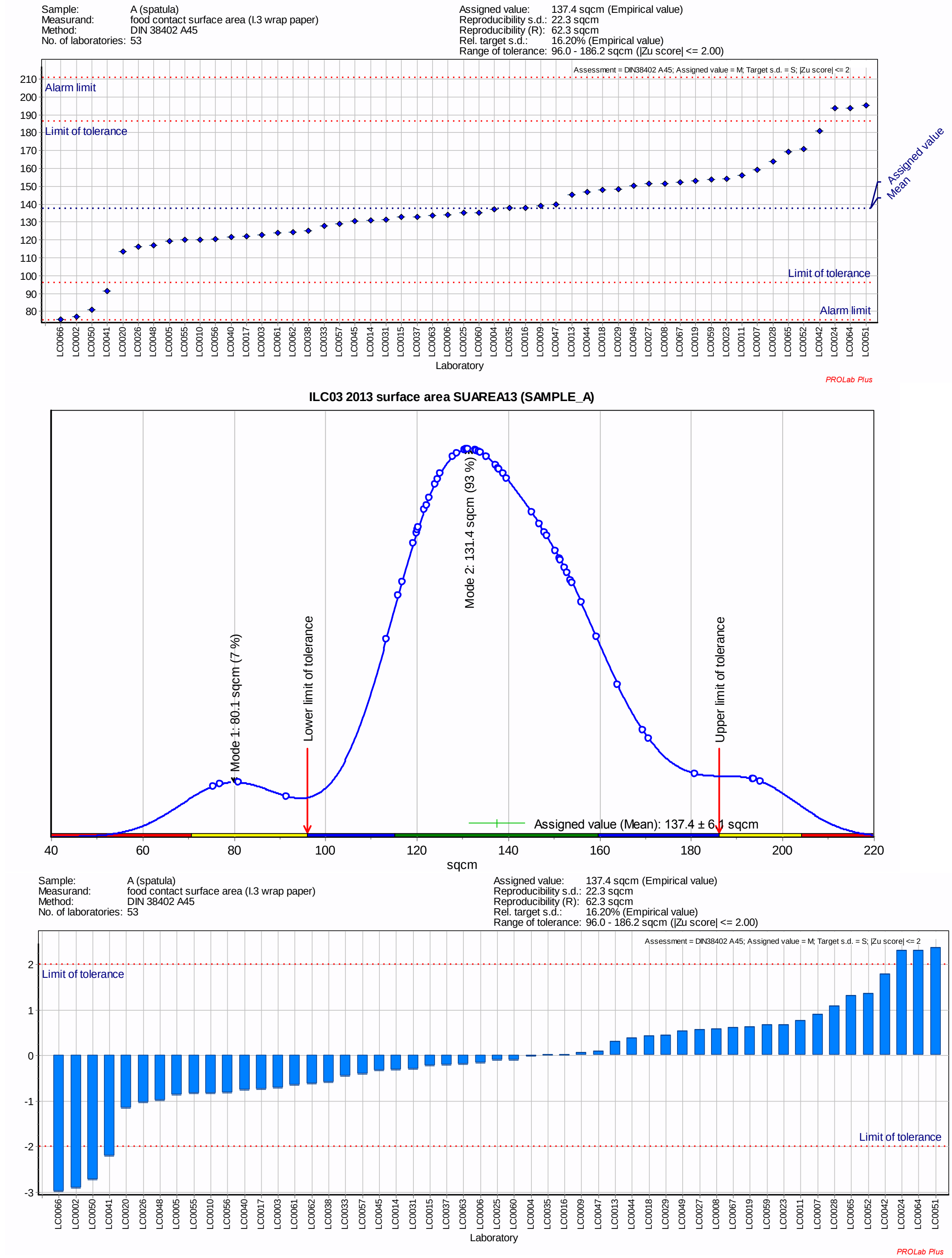


Figure 21 Summary of reported test results for the food contact surface area of Sample A determined by "wrapping in paper" (a), Kernel density estimation (b) and z_U-scores (c)

(a)

(b)

(c)

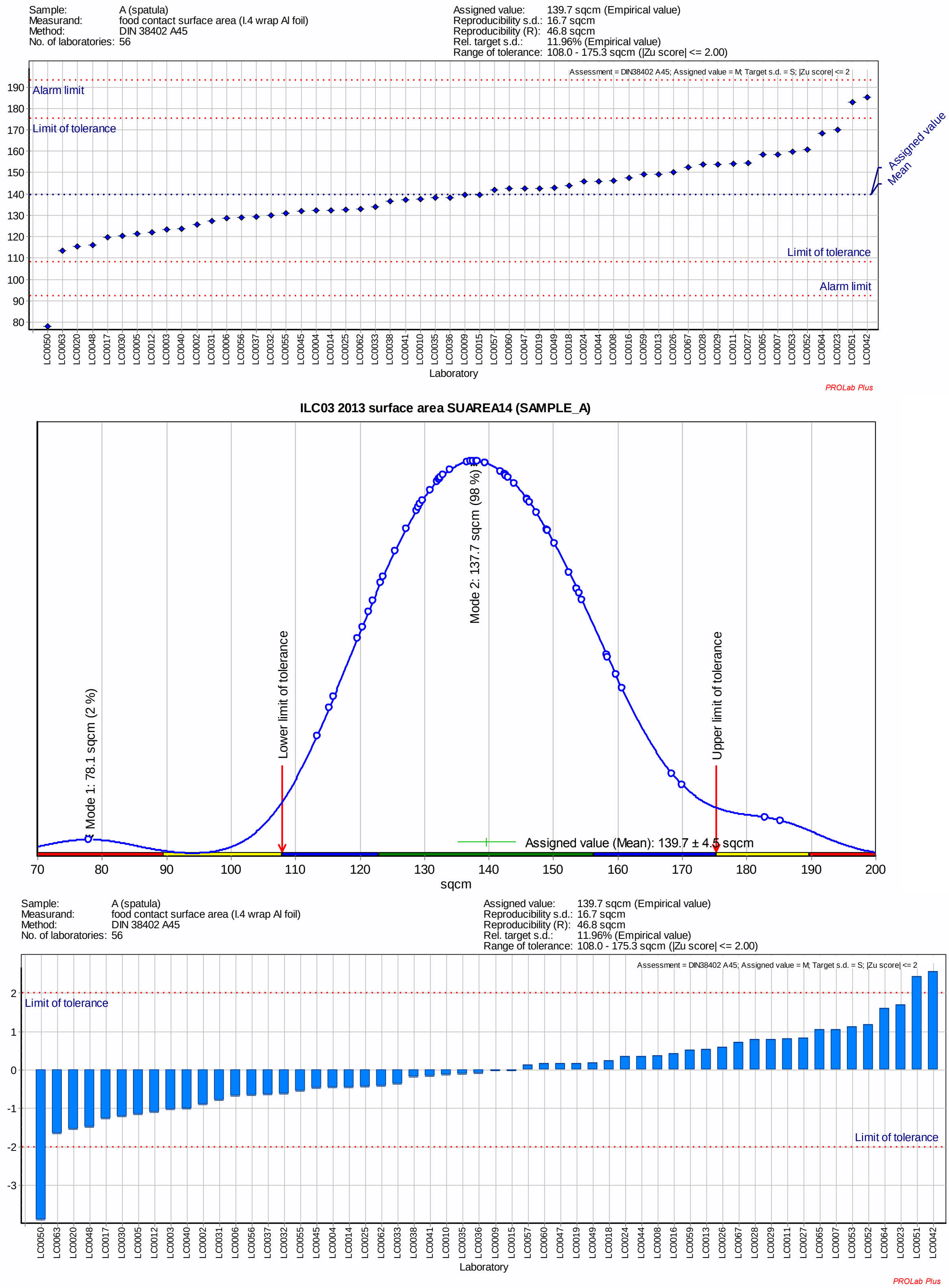


Figure 22 Summary of reported test results for the food contact surface area of Sample A determined by "wrapping in aluminium foil" (a), Kernel density estimation (b) and z_U-scores (c)

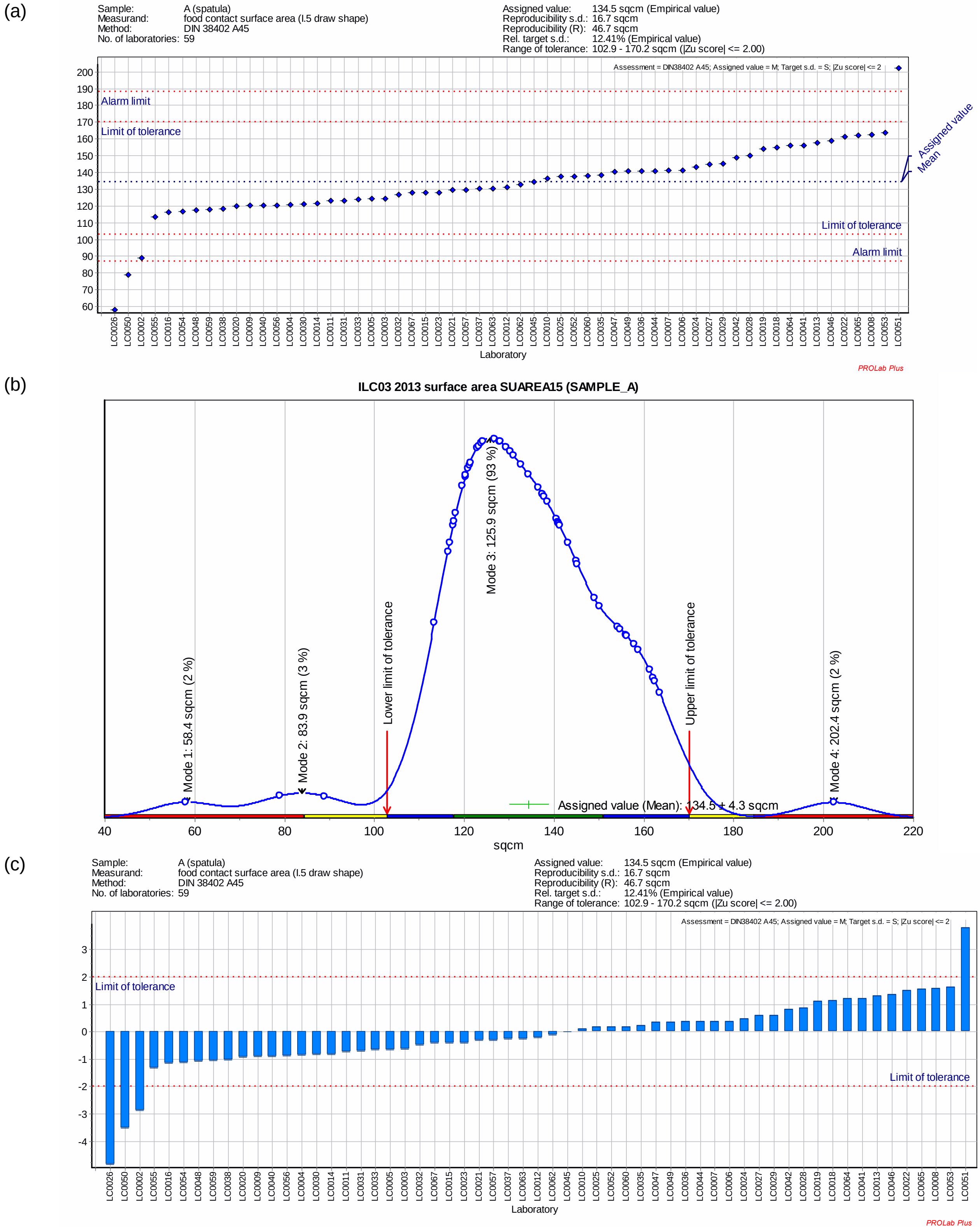
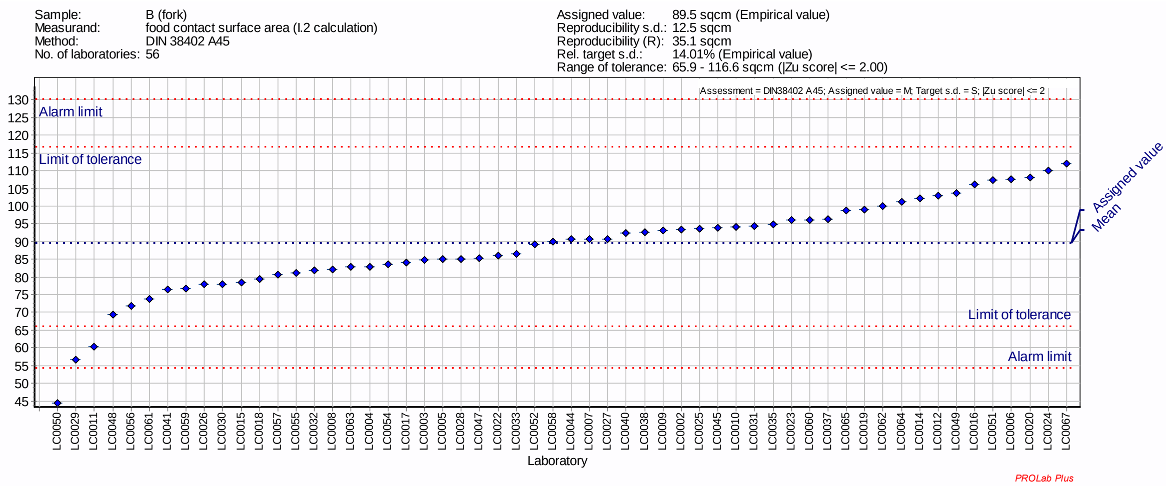


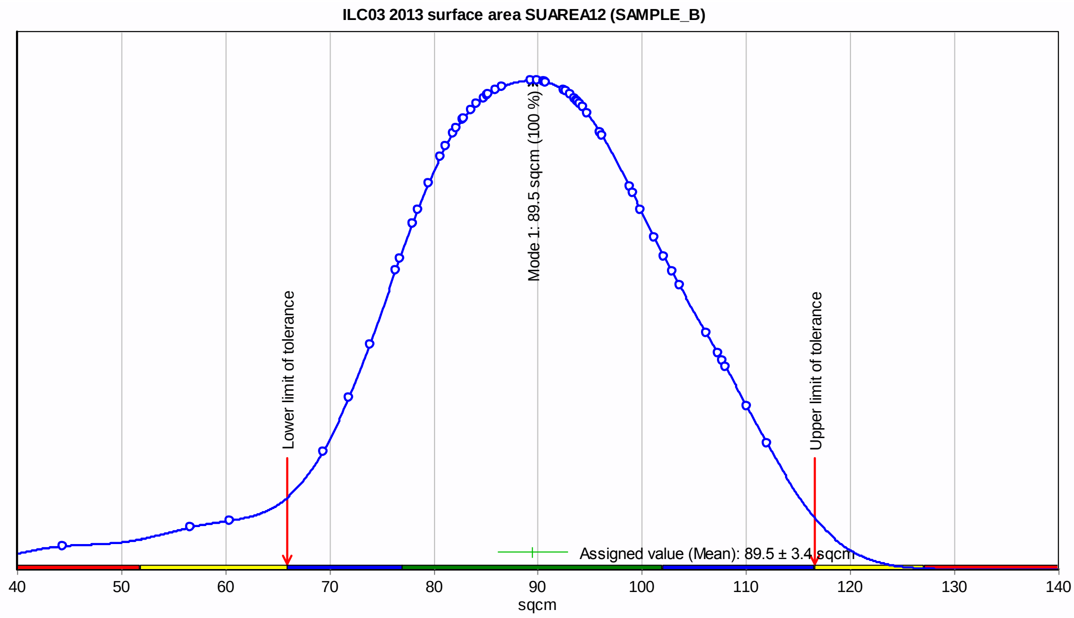
Figure 23 Summary of reported test results for the food contact surface area of Sample A determined by "drawing the shape" (a), Kernel density estimation (b) and z_U-scores (c)

14.3.2.2. Reported results and z_U-scores for the food contact surface area of sample B

(a)



(b)



(c)

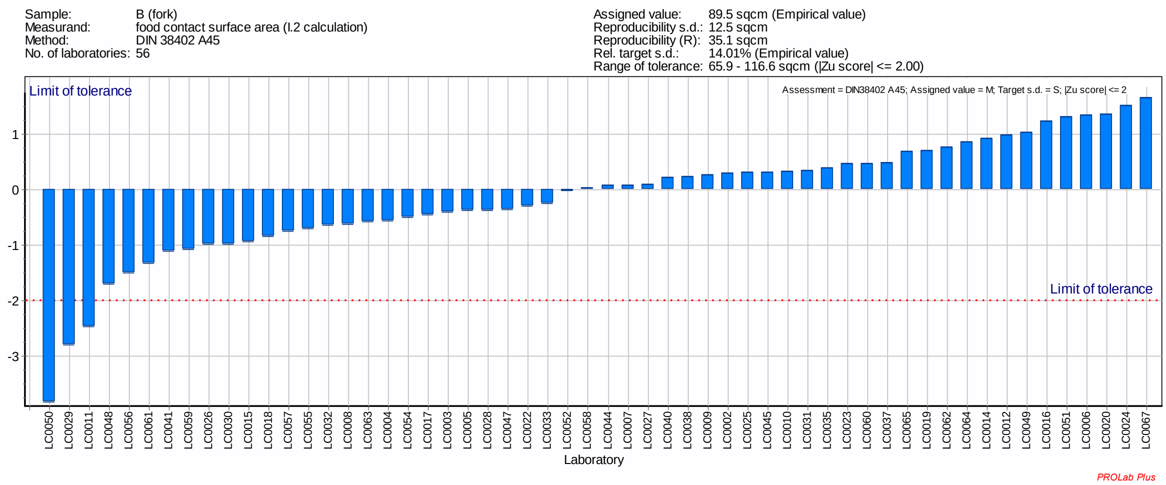


Figure 24 Summary of reported test results for the food contact surface area of Sample B determined by "calculation" (a), Kernel density estimation (b) and z_U-scores (c)

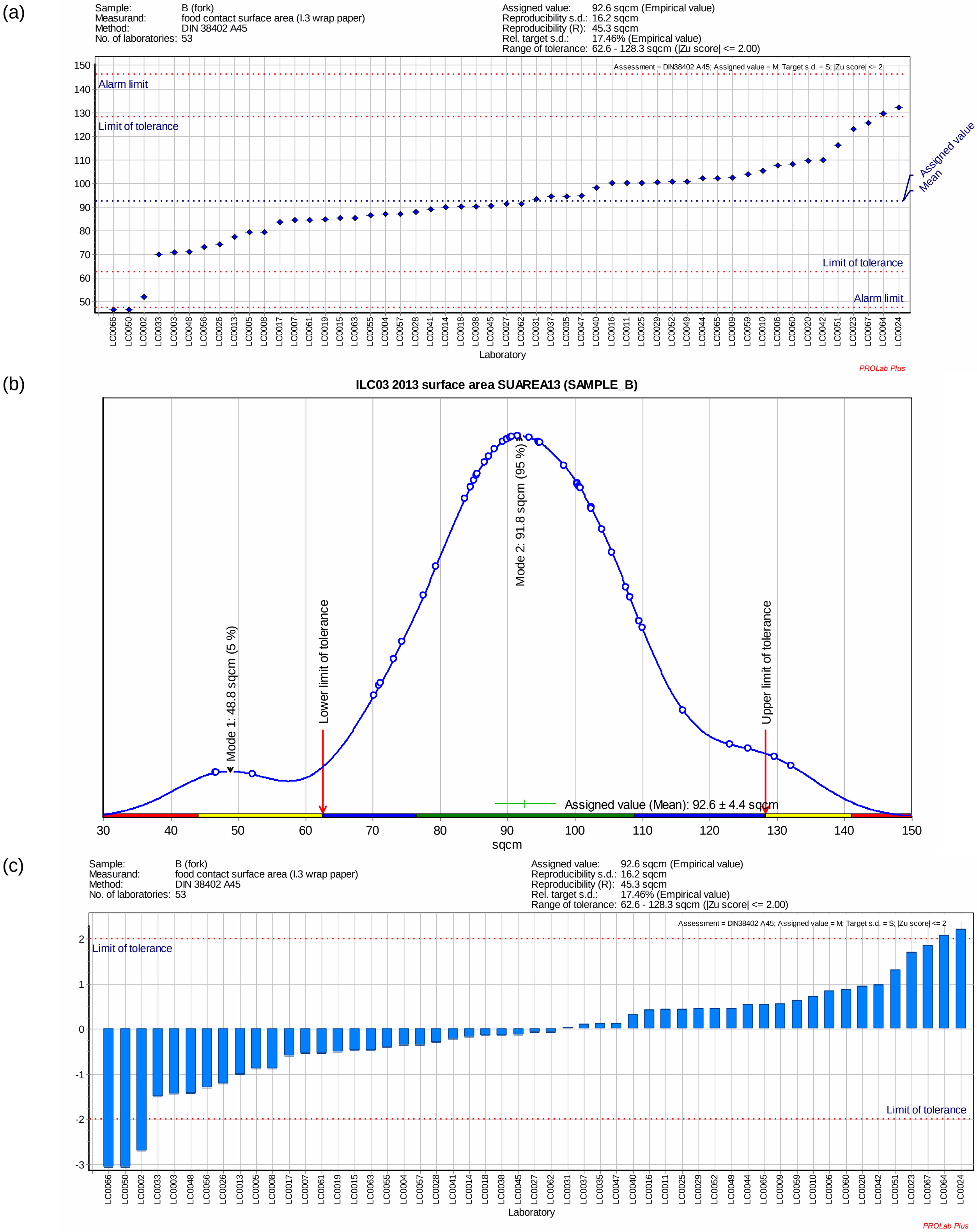


Figure 25 Summary of reported test results for the food contact surface area of Sample B determined by "wrapping in paper" (a), Kernel density estimation (b) and z_U-scores (c)

(a)

(b)

(c)

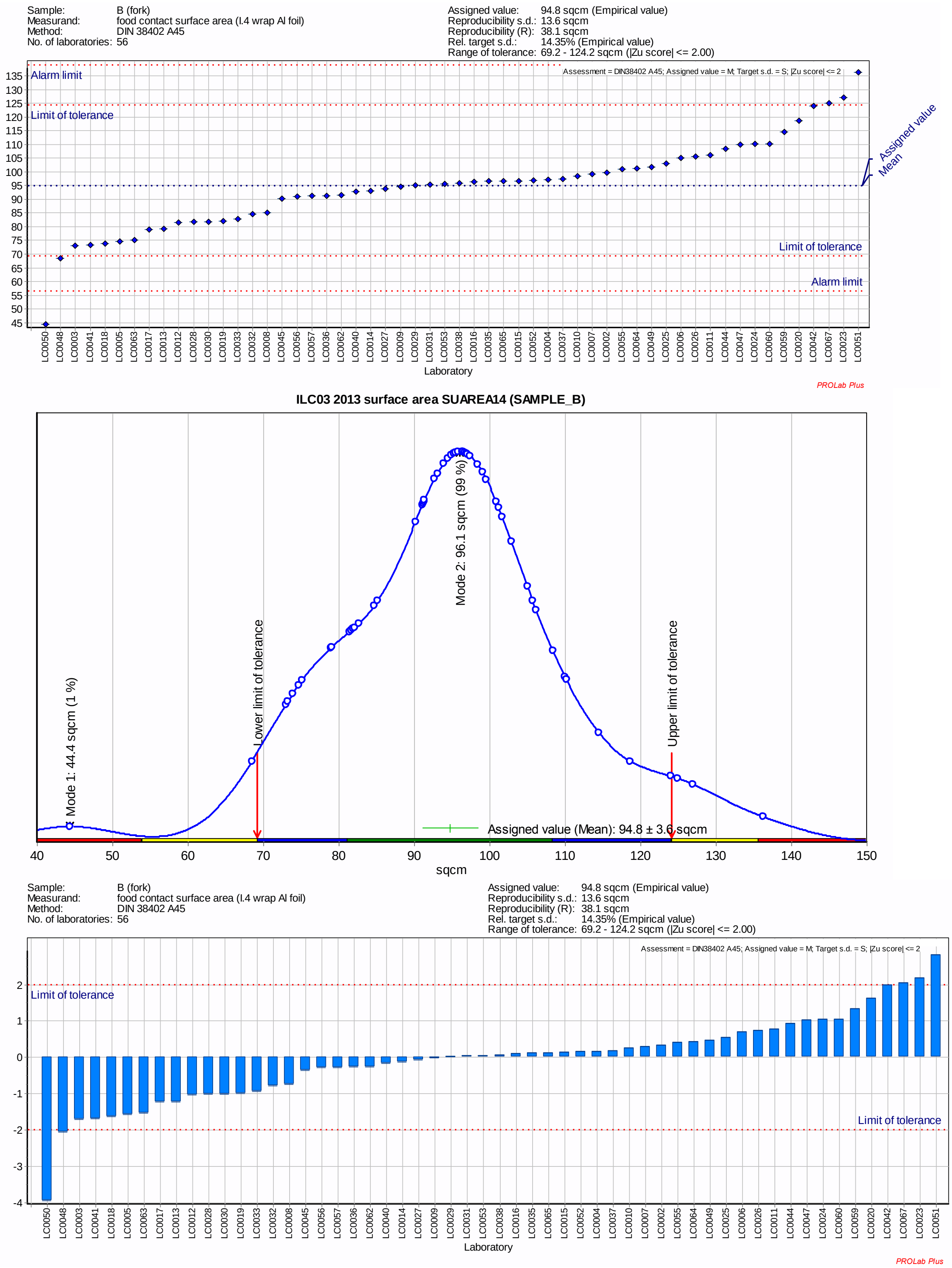


Figure 26 Summary of reported test results for the food contact surface area of Sample B determined by "wrapping in aluminium foil" (a), Kernel density estimation (b) and z_U-scores (c)

(a)

(b)

(c)

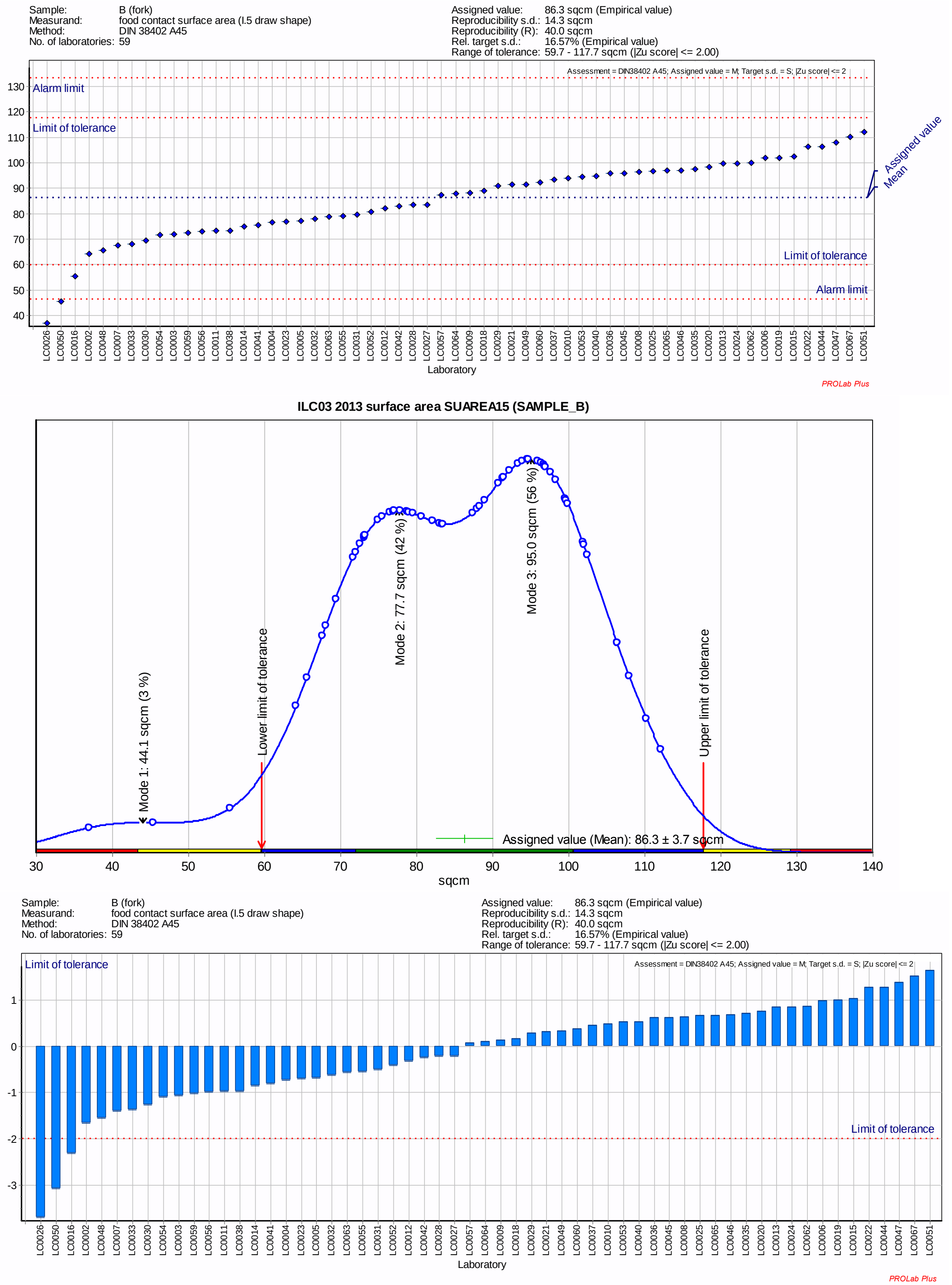
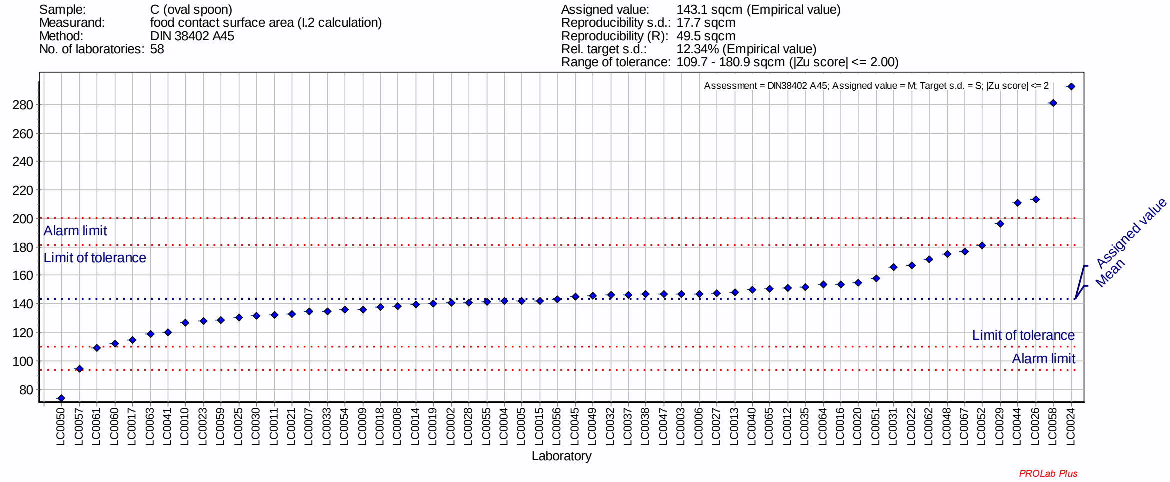


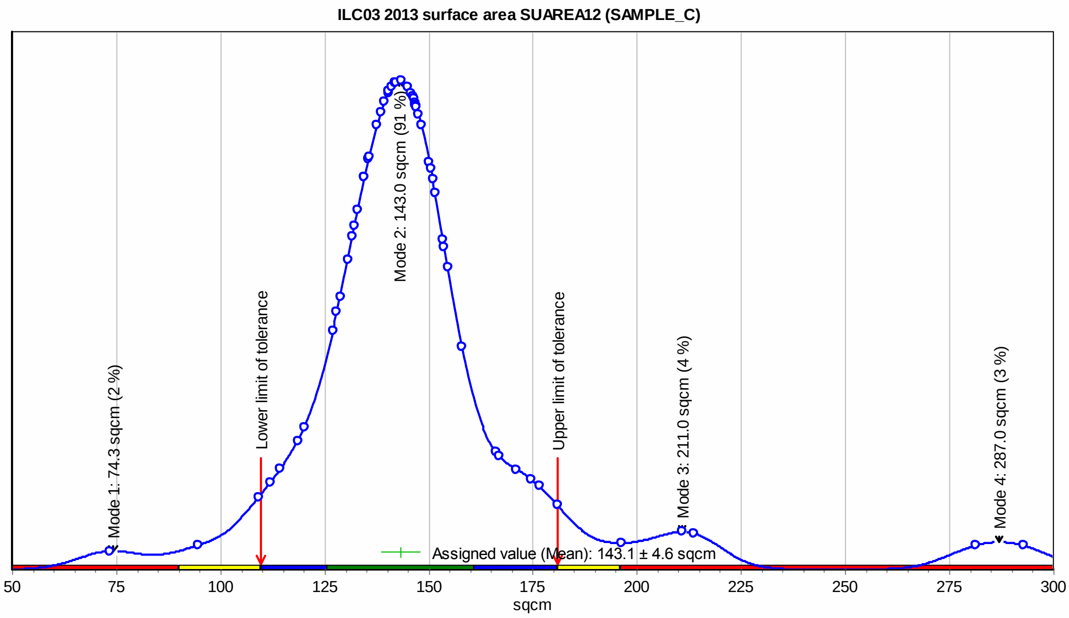
Figure 27 Summary of reported test results for the food contact surface area of Sample B determined by "drawing the shape" (a), Kernel density estimation (b) and z_U-scores (c)

14.3.2.3. Reported results and z_U-scores for the food contact surface area of sample C

(a)



(b)



(c)

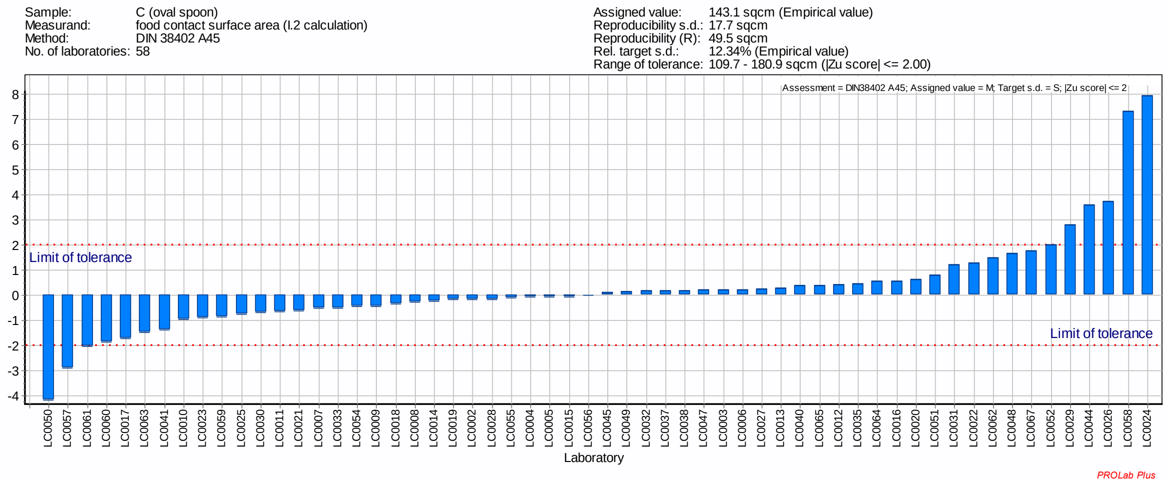


Figure 28 Summary of reported test results for the food contact surface area of Sample C determined by "calculation" (a), Kernel density estimation (b) and z_U-scores (c)

(a)

(b)

(c)

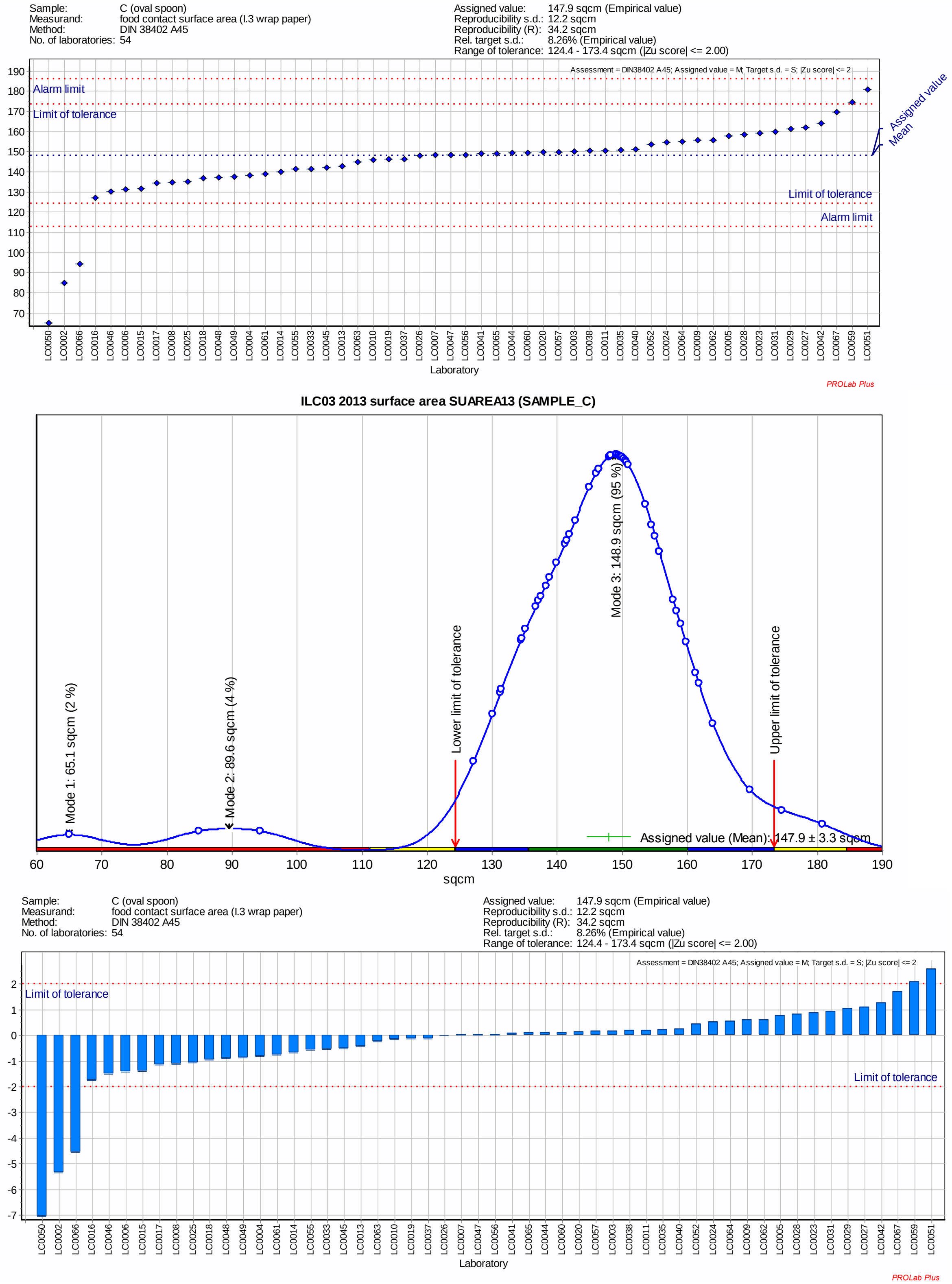


Figure 29 Summary of reported test results for the food contact surface area of Sample C determined by "wrapping in paper" (a), Kernel density estimation (b) and z_U-scores (c)

(a)

(b)

(c)

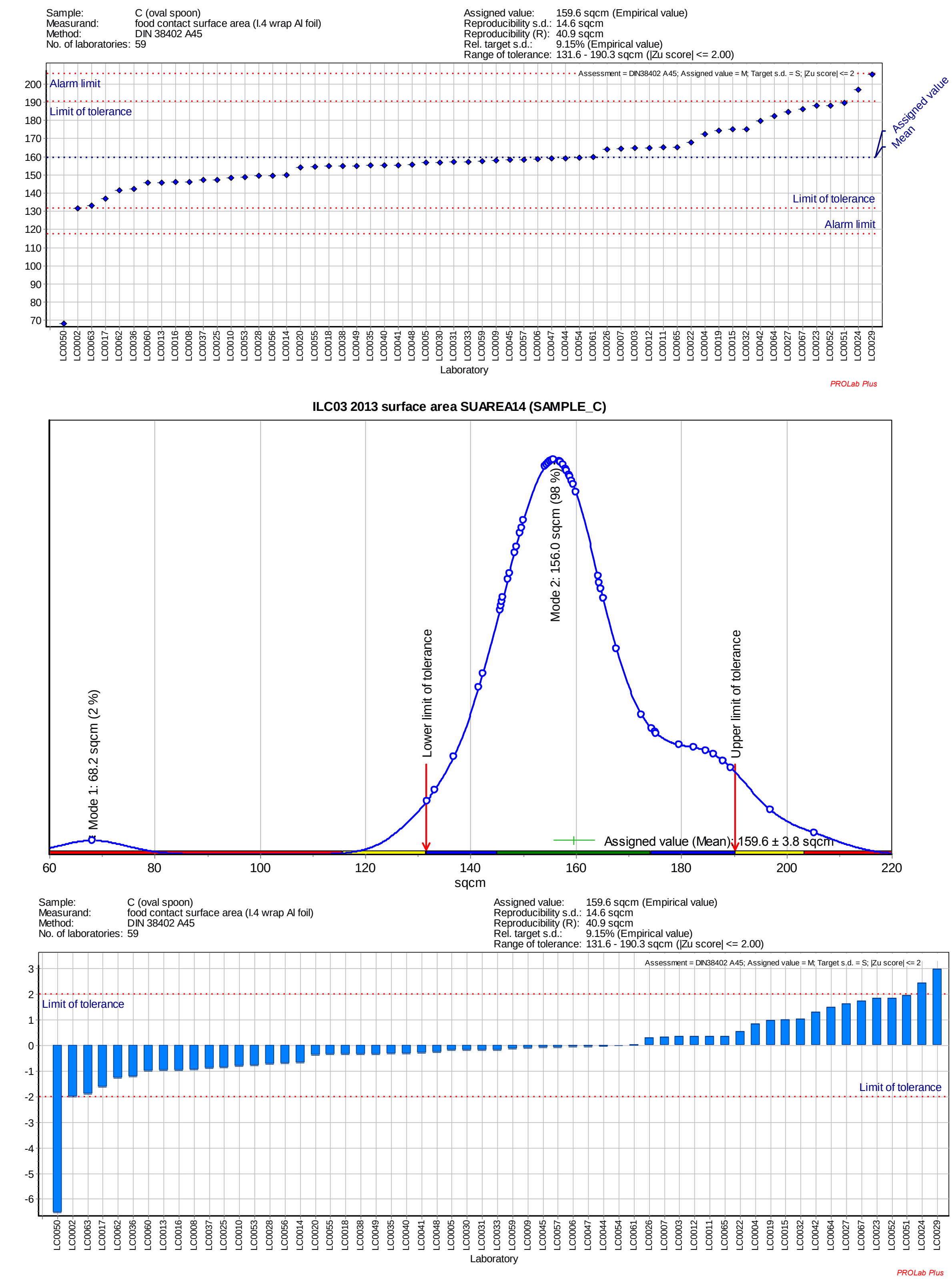


Figure 30 Summary of reported test results for the food contact surface area of Sample C determined by "wrapping in aluminium foil" (a), Kernel density estimation (b) and z_U-scores (c)

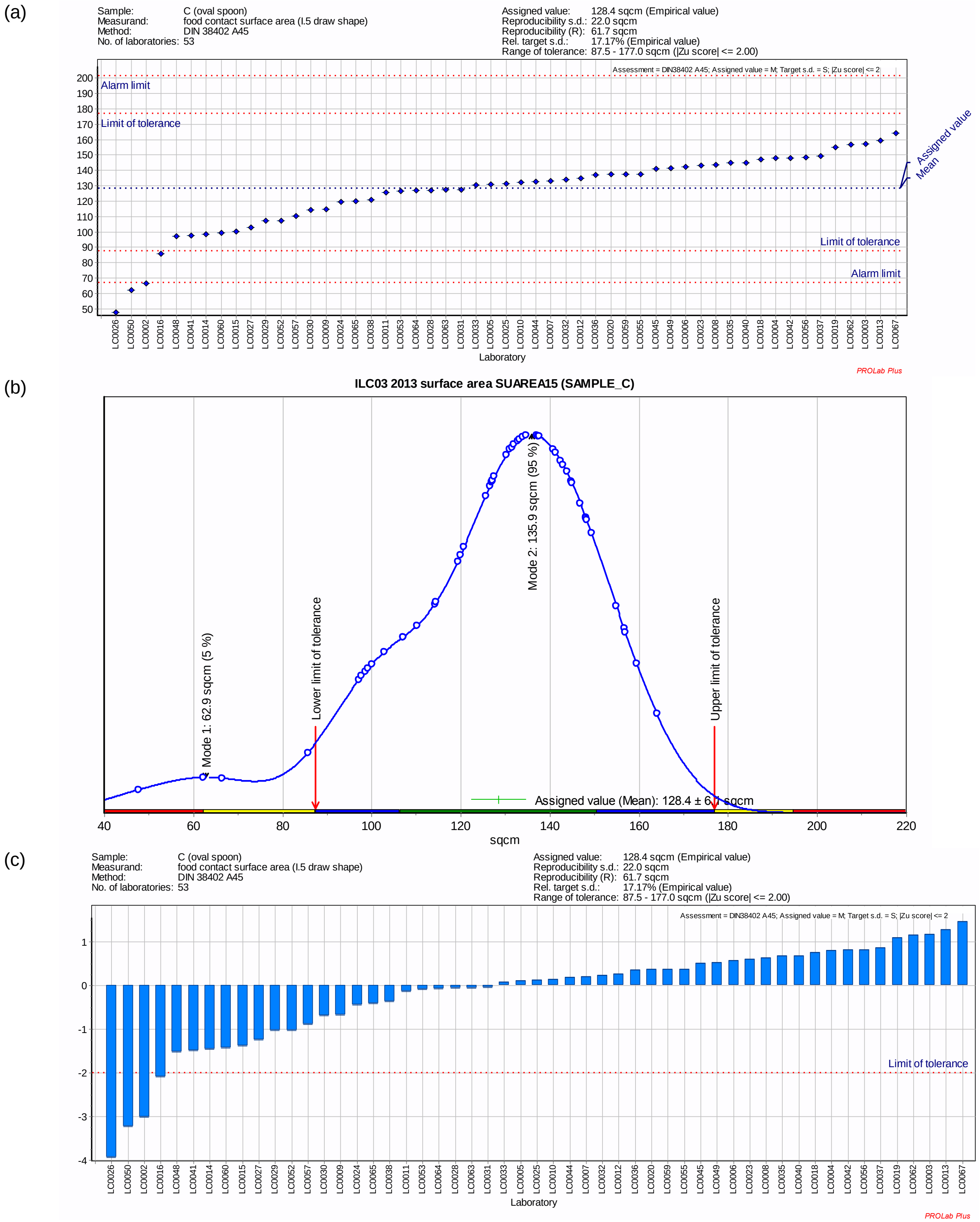


Figure 31 Summary of reported test results for the food contact surface area of Sample C determined by "drawing the shape" (a), Kernel density estimation (b) and z_U-scores (c)

14.3.2.4. Reported results and z_U-scores for the food contact surface area of sample D

(a)

(b)

(c)

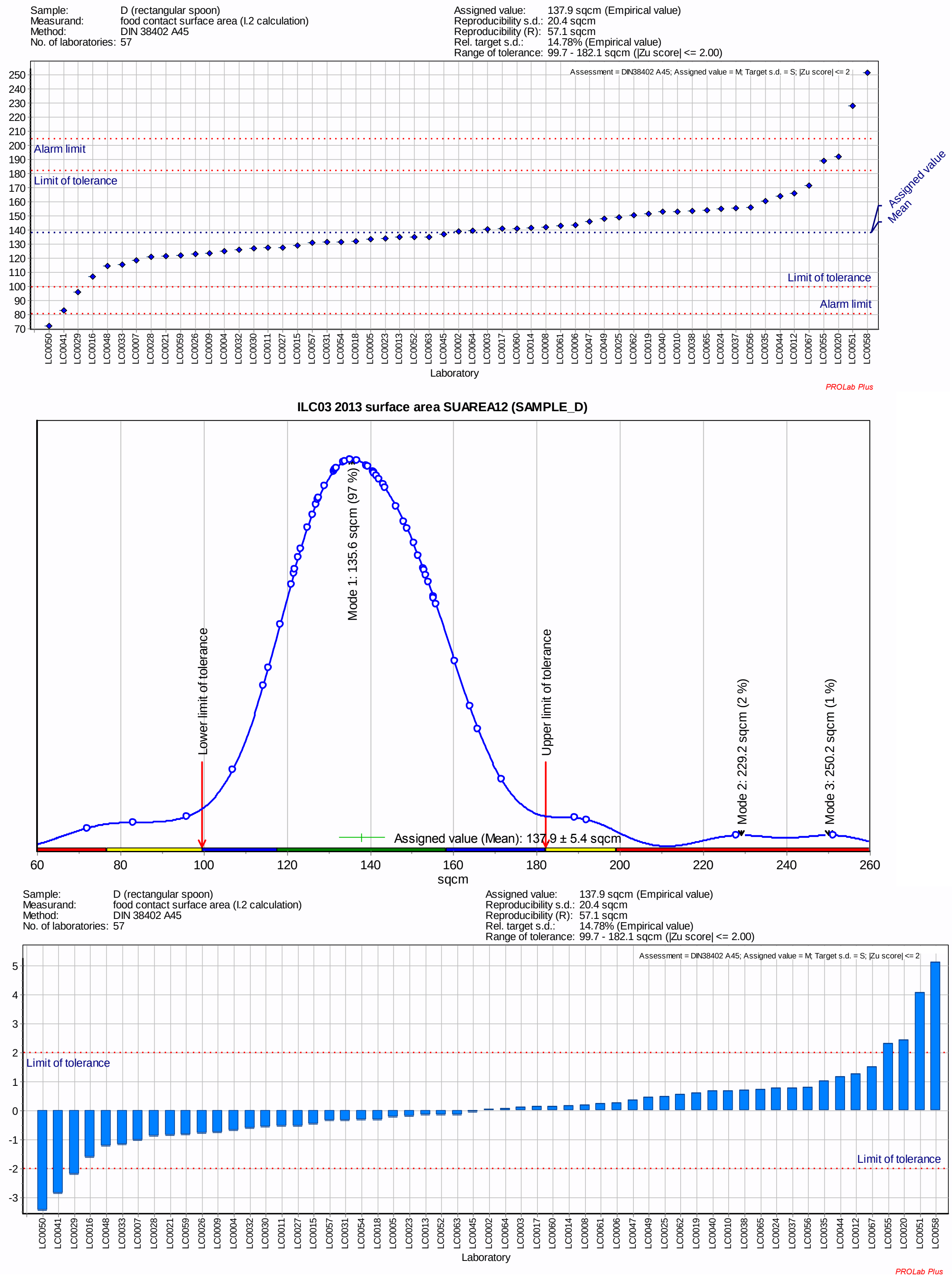


Figure 32 Summary of reported test results for the food contact surface area of Sample D determined by "calculation" (a), Kernel density estimation (b) and z_U-scores (c)

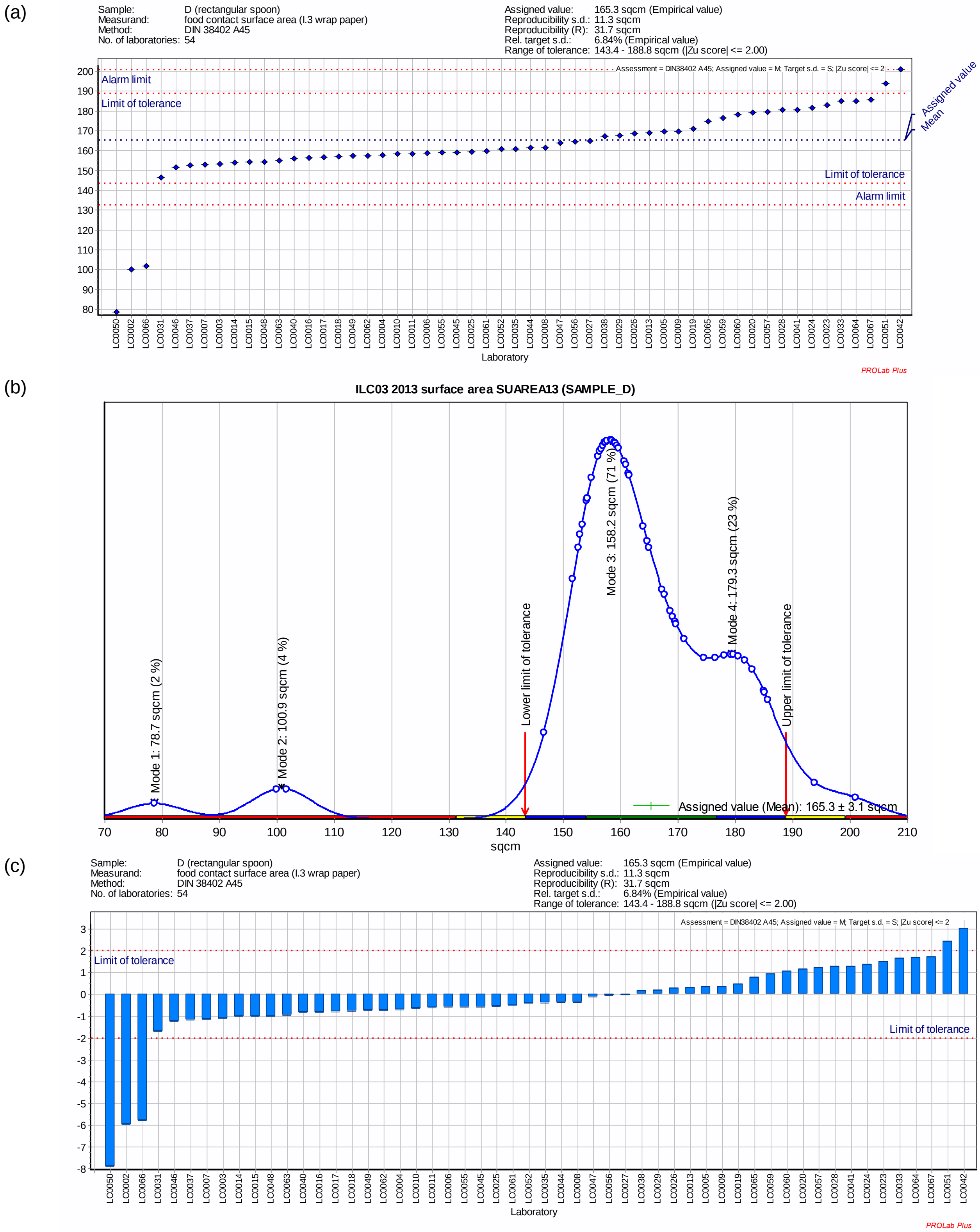


Figure 33 Summary of reported test results for the food contact surface area of Sample D determined by "wrapping in paper" (a), Kernel density estimation (b) and z_U-scores (c)

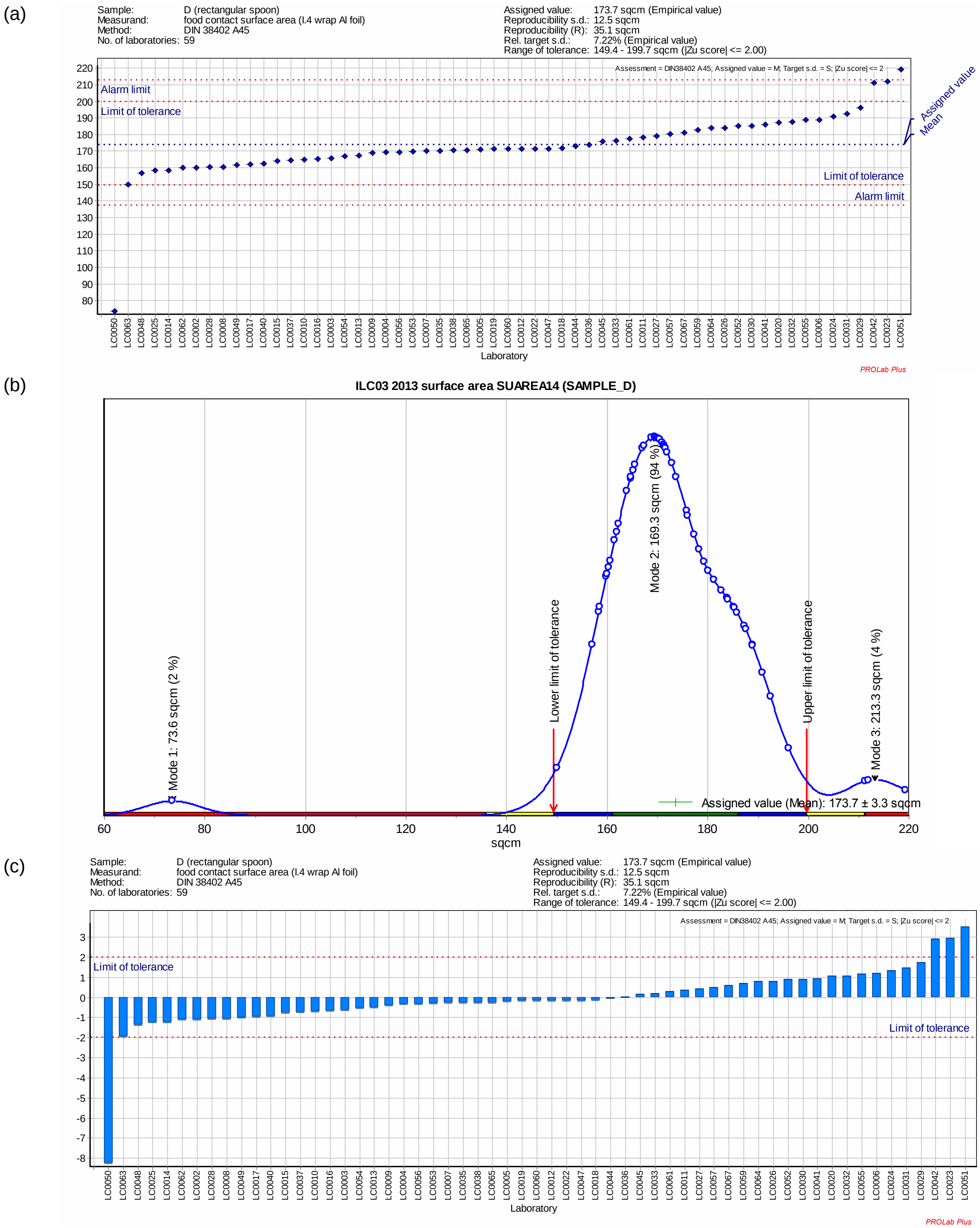


Figure 34 Summary of reported test results for the food contact surface area of Sample D determined by "wrapping in aluminium foil" (a), Kernel density estimation (b) and z_U-scores (c)

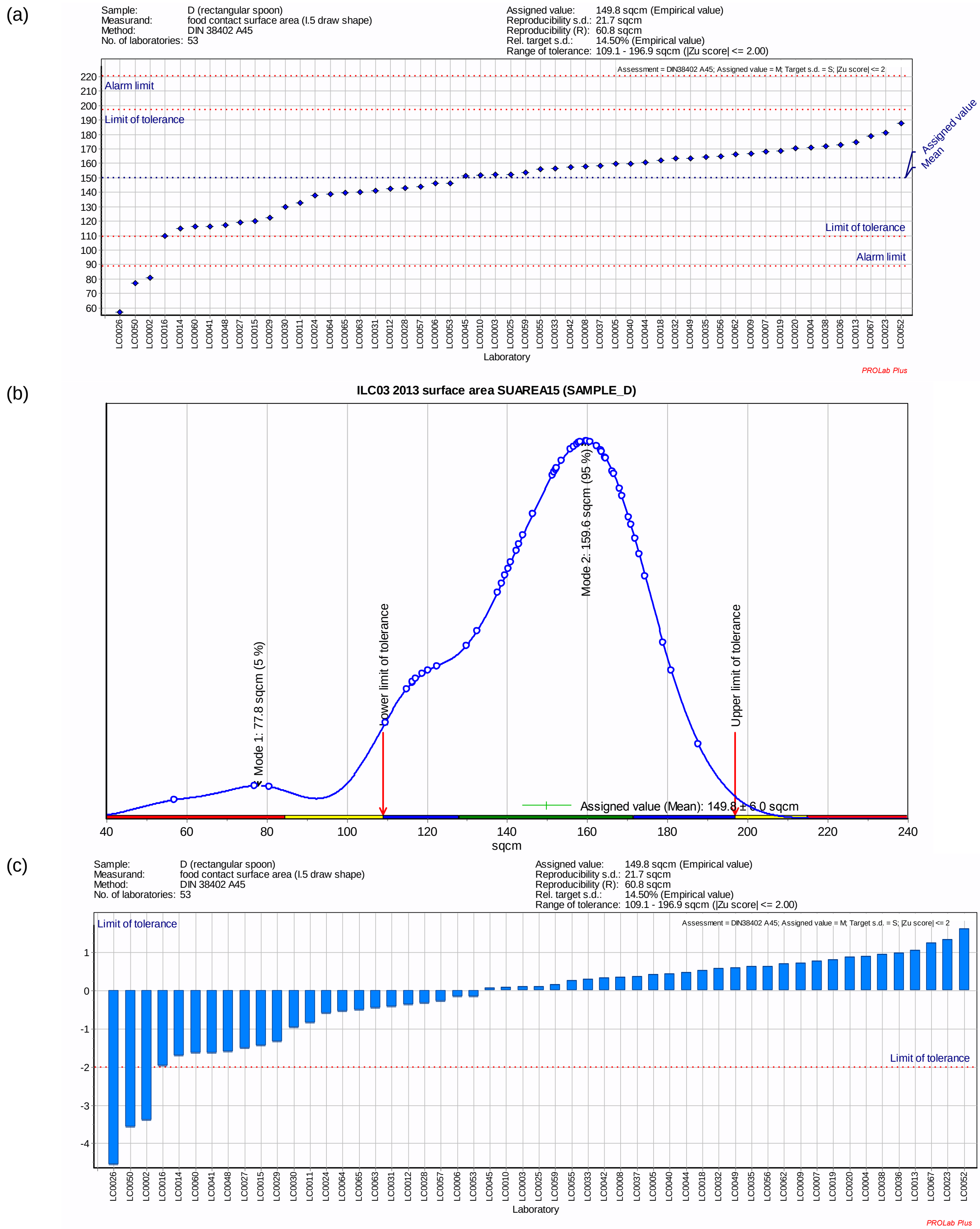


Figure 35 Summary of reported test results for the food contact surface area of Sample D determined by "drawing the shape" (a), Kernel density estimation (b) and z_U-scores (c)

14.3.2.5. Reported results and z_U-scores for the food contact surface area of sample E

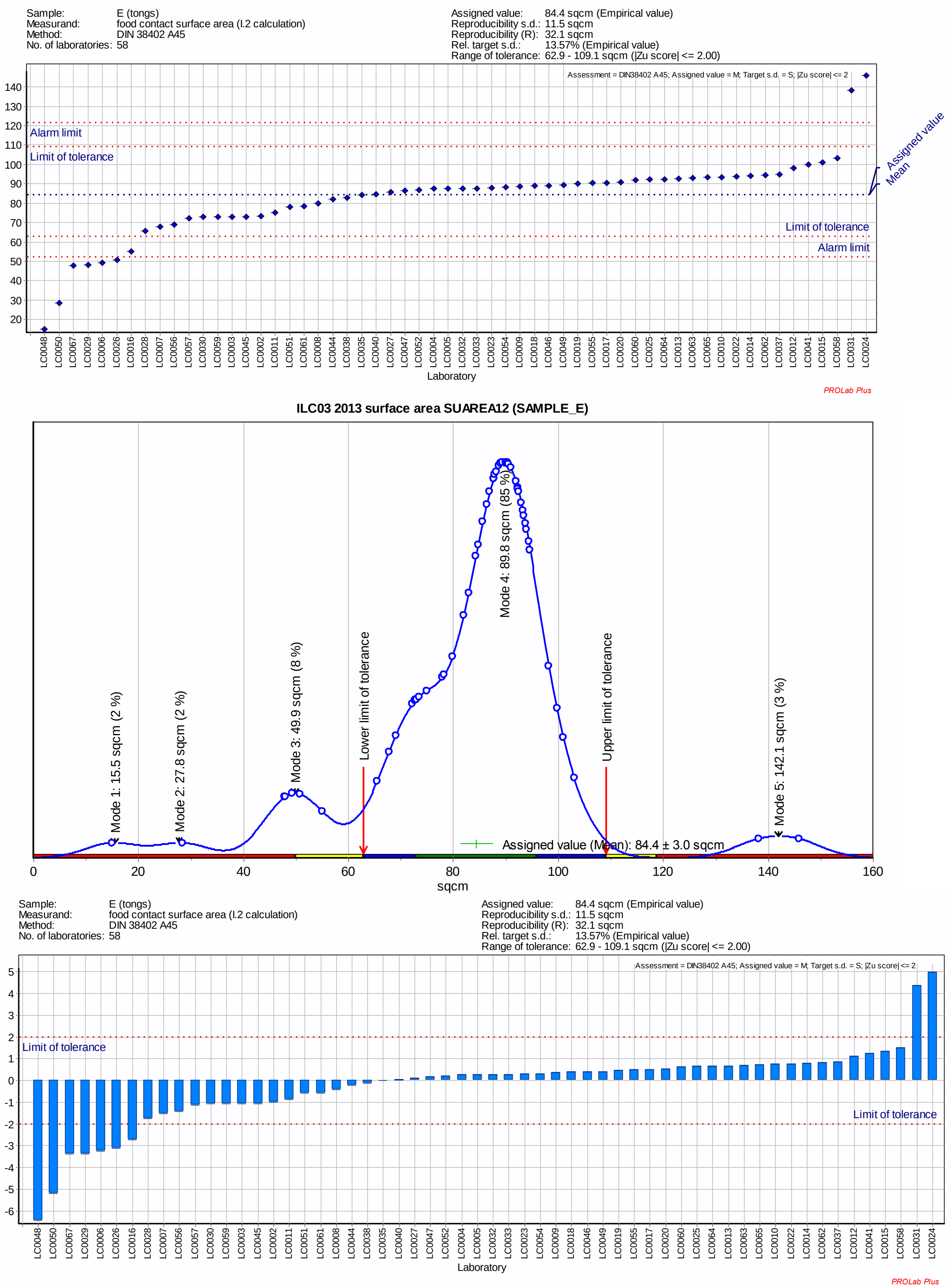


Figure 36 Summary of reported test results for the food contact surface area of Sample E determined by "calculation" (a), Kernel density estimation (b) and z_U-scores (c)

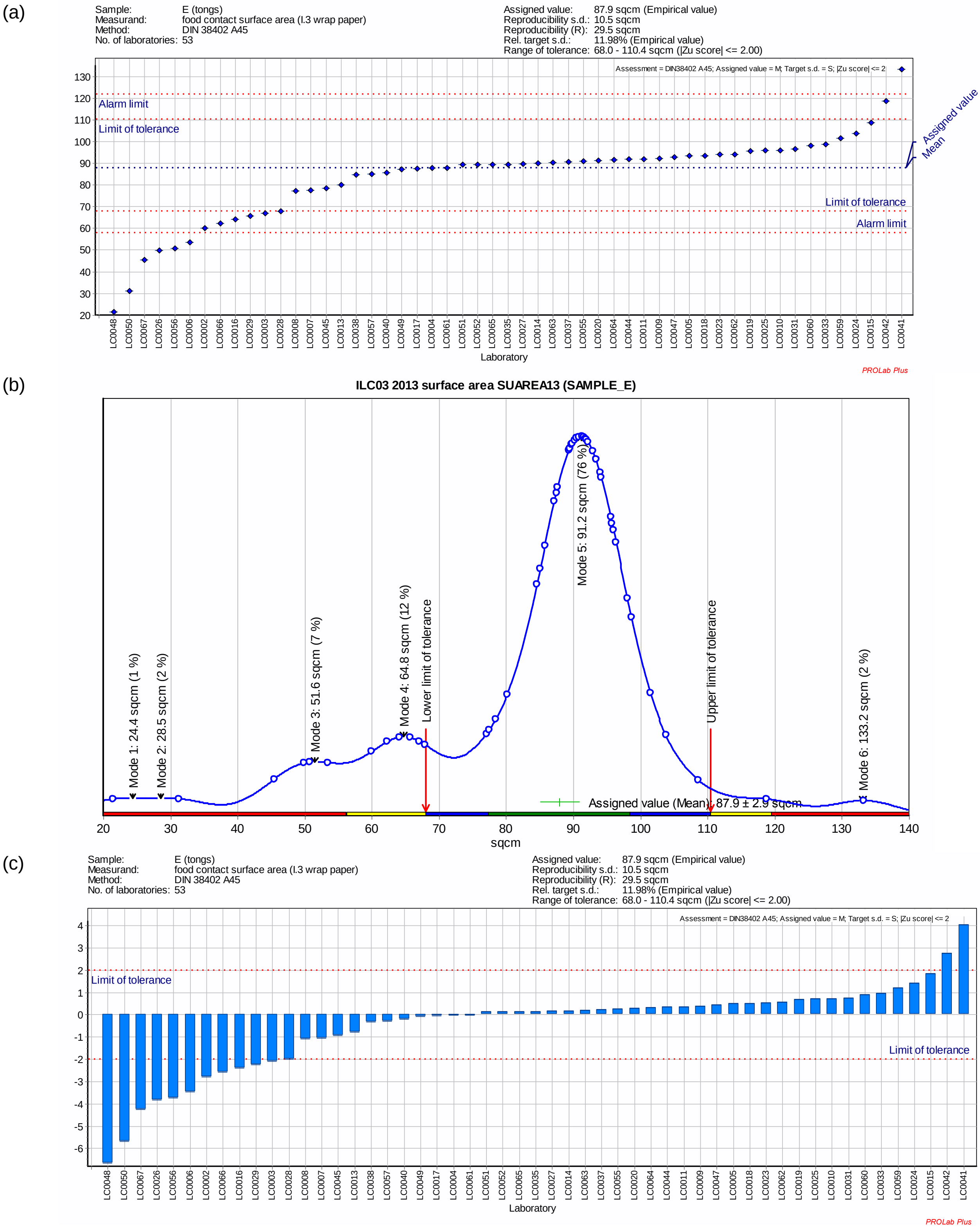


Figure 37 Summary of reported test results for the food contact surface area of Sample E determined by "wrapping in paper" (a), Kernel density estimation (b) and z_U-scores (c)

(a)

(b)

(c)

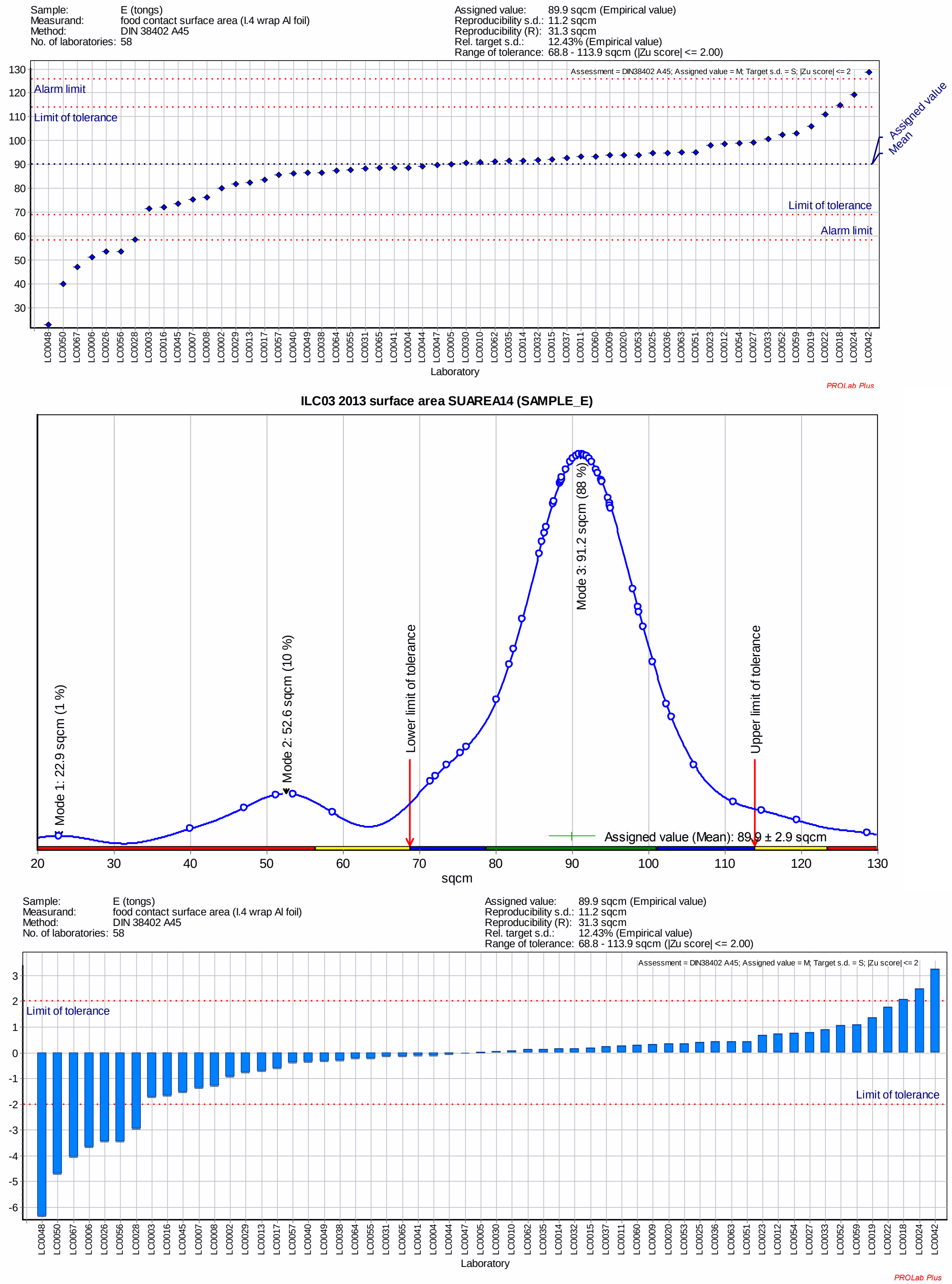


Figure 38 Summary of reported test results for the food contact surface area of Sample E determined by "wrapping in aluminium foil" (a), Kernel density estimation (b) and z_U-scores (c)

(a) (b) (c)

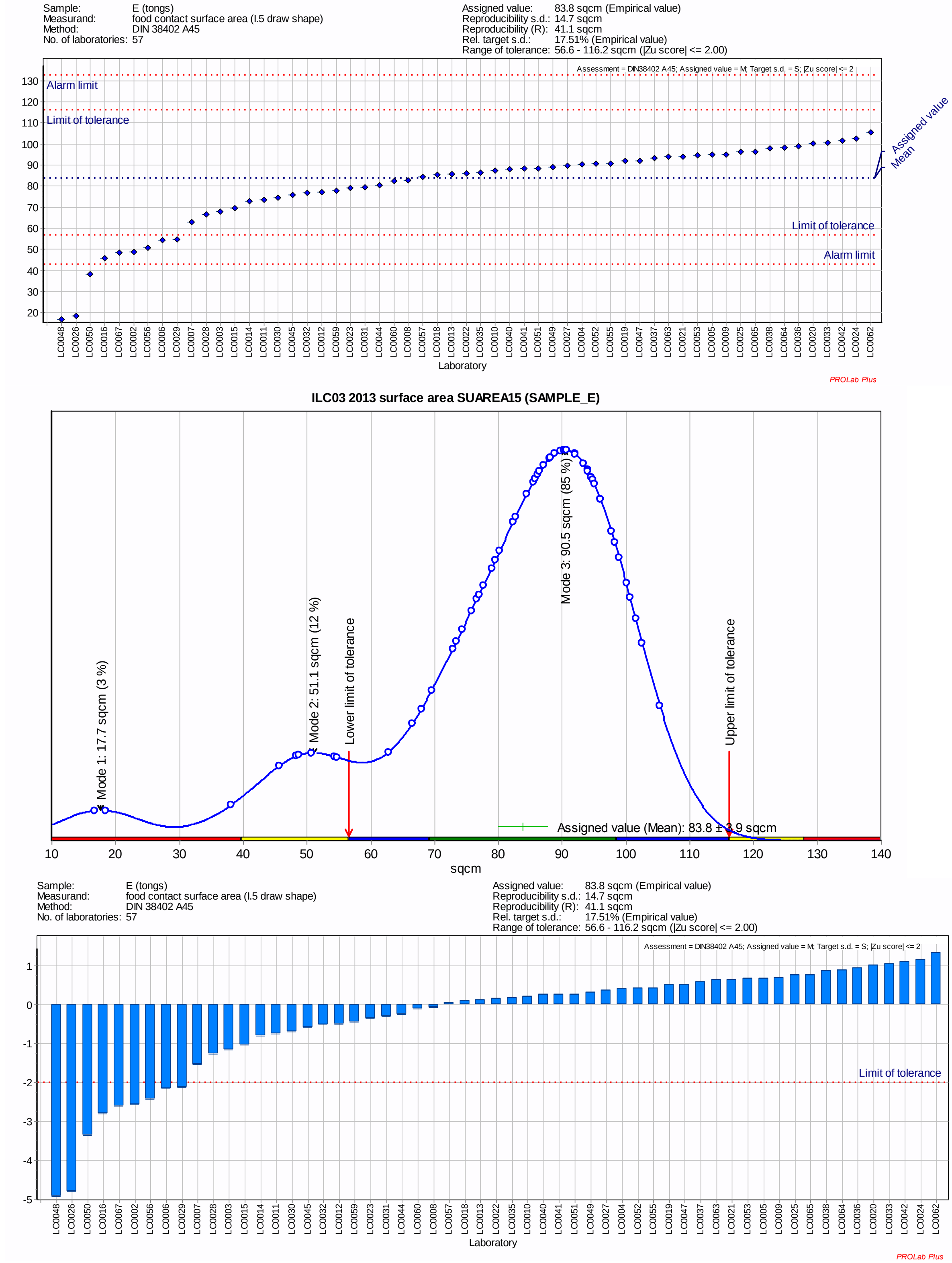


Figure 39 Summary of reported test results for the food contact surface area of Sample E determined by "drawing the shape" (a), Kernel density estimation (b) and z_U-scores (c)

14.3.3. Reported results and z_U-scores for the envelope volume

14.3.3.1. Reported results and z_U-scores for the envelope volume of sample A

(a)
(b)
(c)

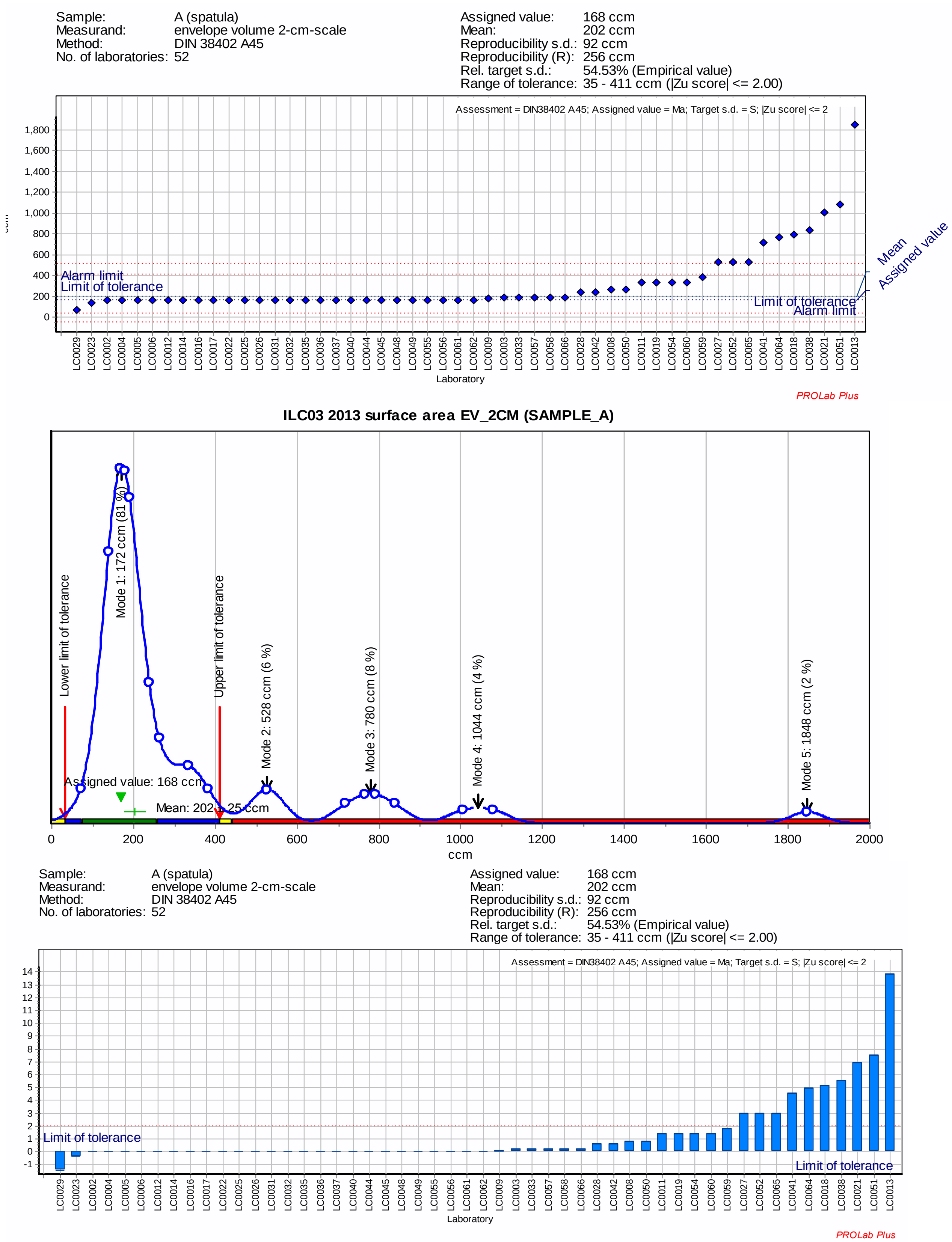
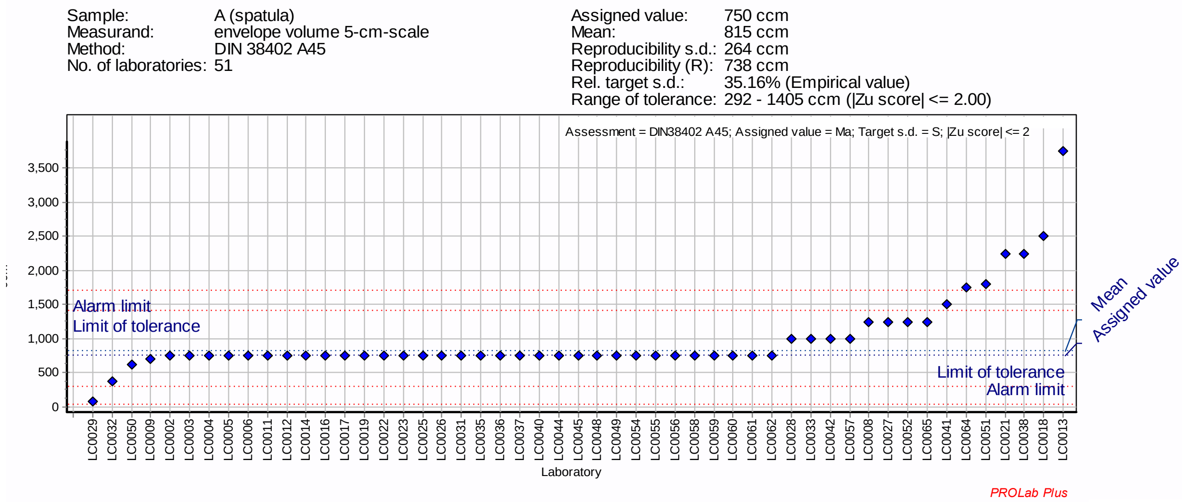
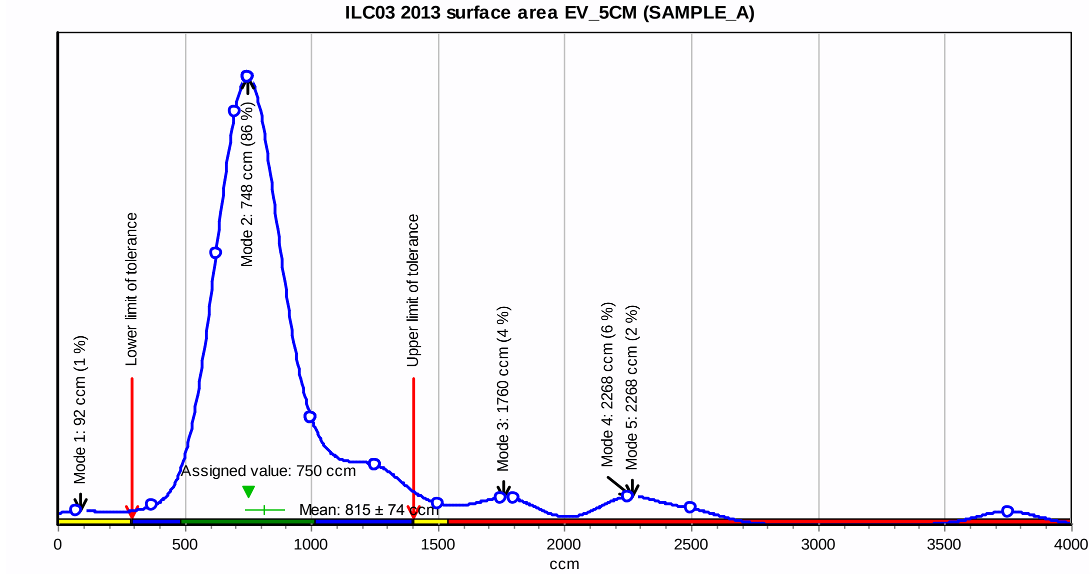


Figure 40 Summary of reported test results for the envelope volume of Sample A determined on a 2-cm-scale (a), Kernel density estimation (b) and z_U-scores (c)

(a)



(b)



(c)

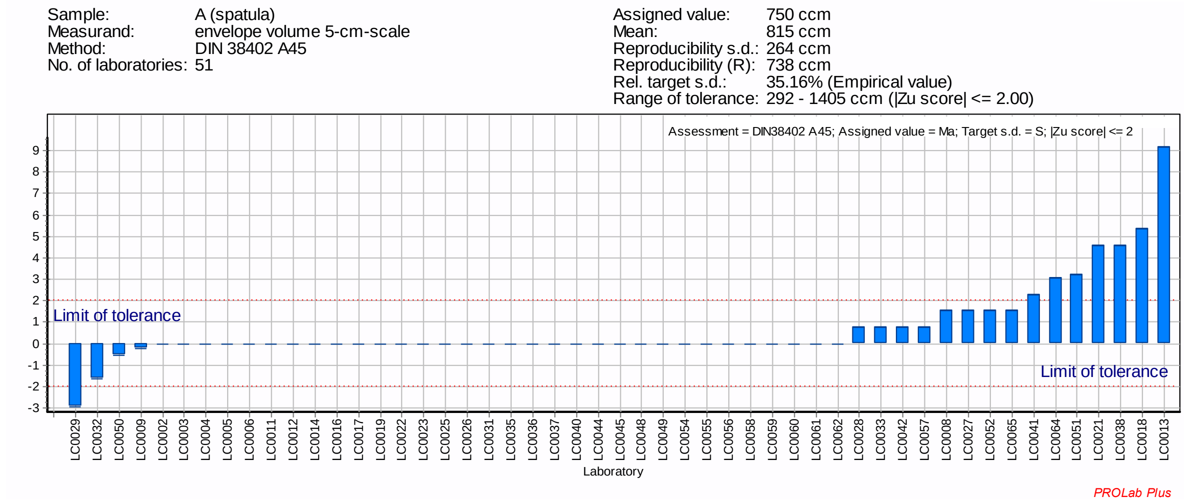


Figure 41 Summary of reported test results for the envelope volume of Sample A determined on a 5-cm-scale (a), Kernel density estimation (b) and z_U-scores (c)

14.3.3.2. Reported results and z_U-scores for the envelope volume of sample B

(a)

(b)

(c)

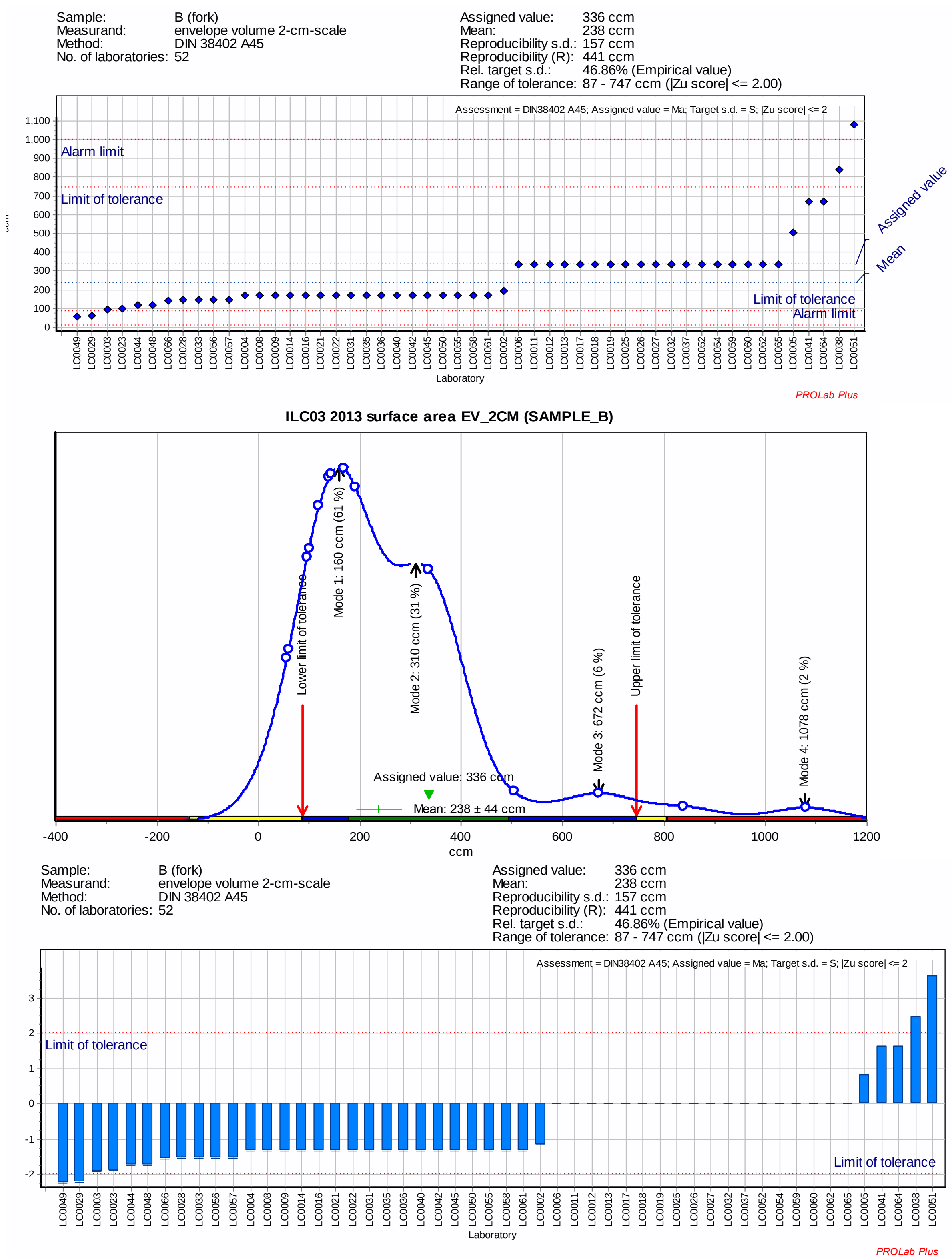


Figure 42 Summary of reported test results for the envelope volume of Sample B determined on a 2-cm-scale (a), Kernel density estimation (b) and z_U-scores (c)

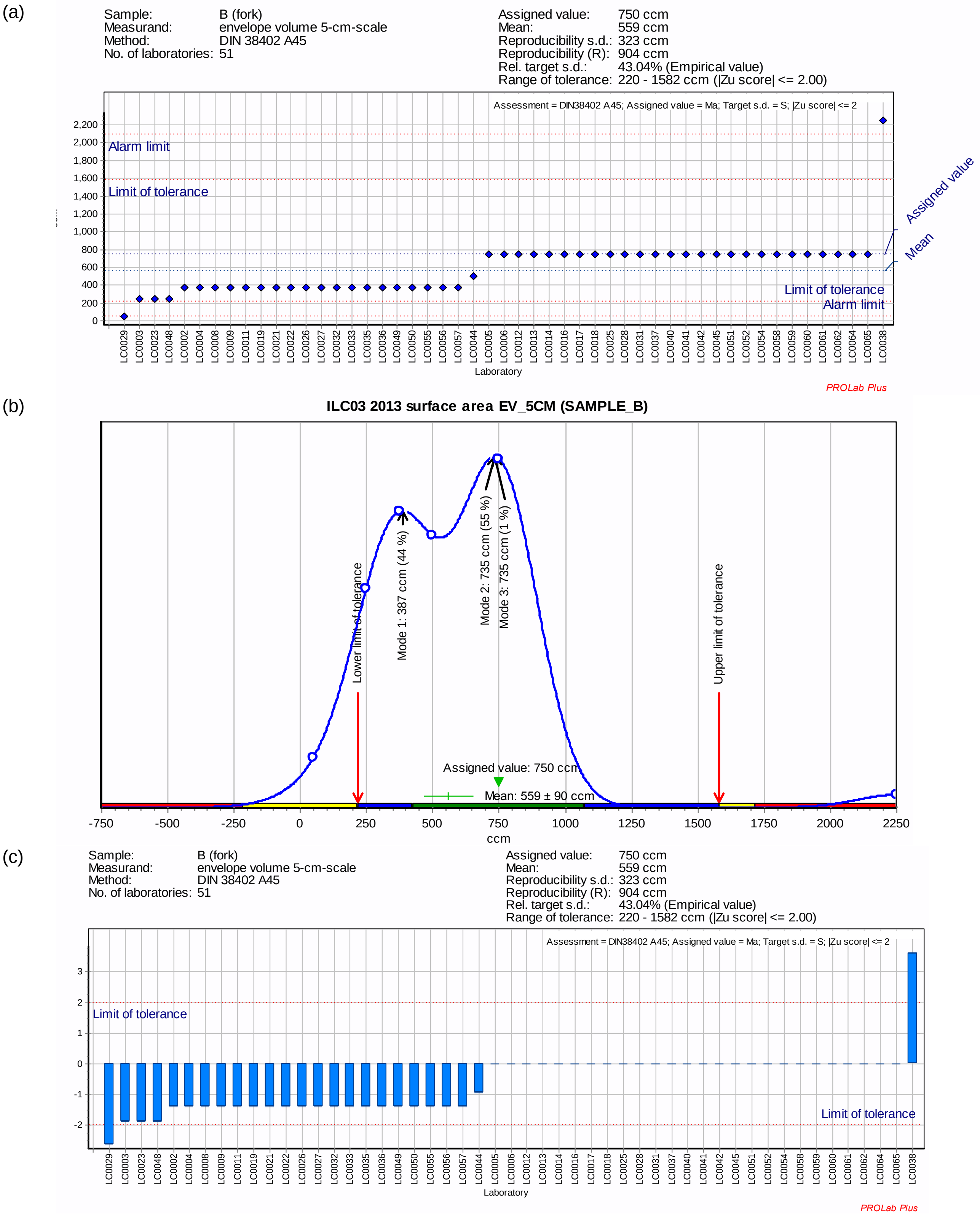


Figure 43 Summary of reported test results for the envelope volume of Sample B determined on a 5-cm-scale (a), Kernel density estimation (b) and z_U-scores (c)

14.3.3.3. Reported results and z_U-scores for the envelope volume of sample C

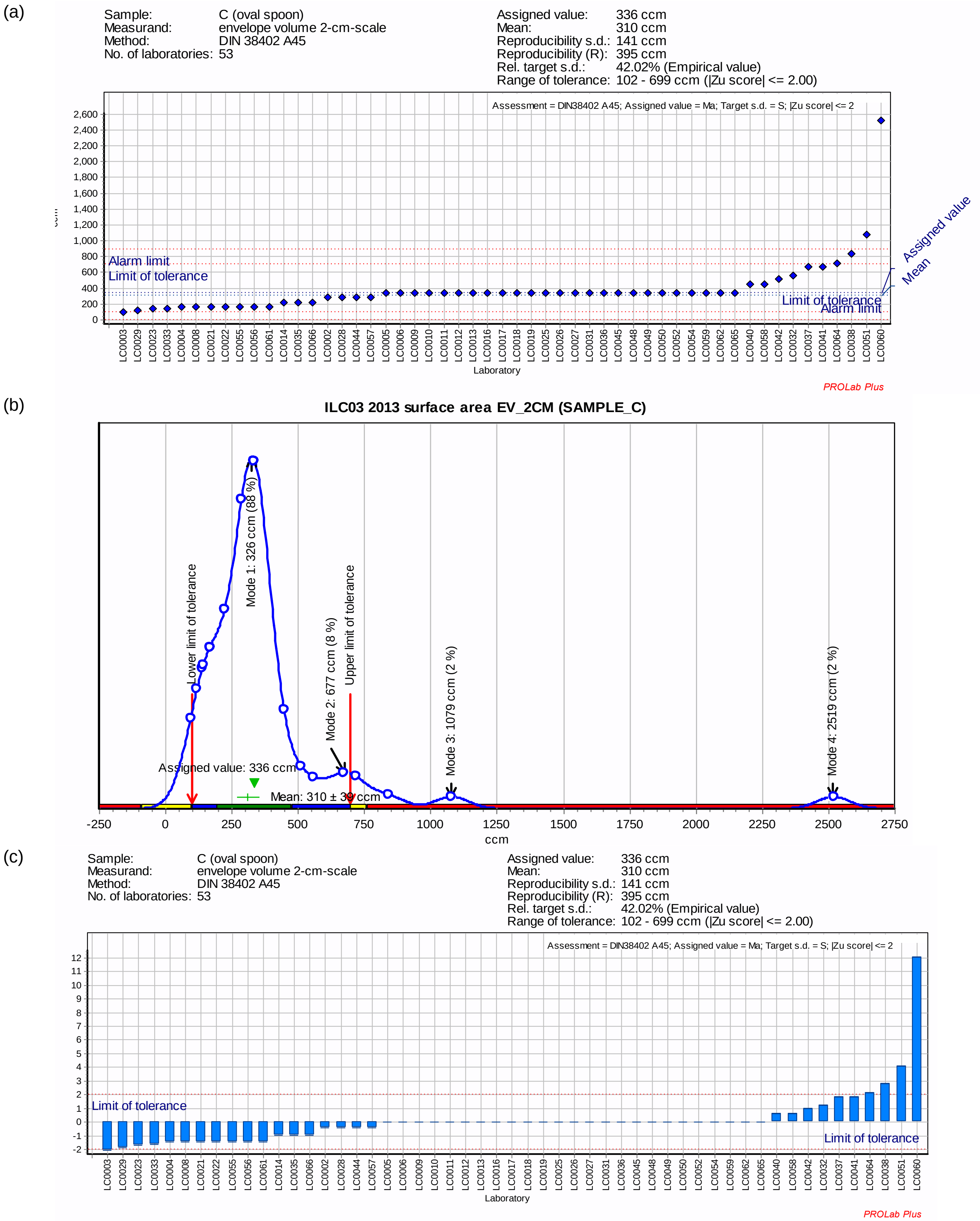


Figure 44 Summary of reported test results for the envelope volume of Sample C determined on a 2-cm-scale (a), Kernel density estimation (b) and z_U-scores (c)

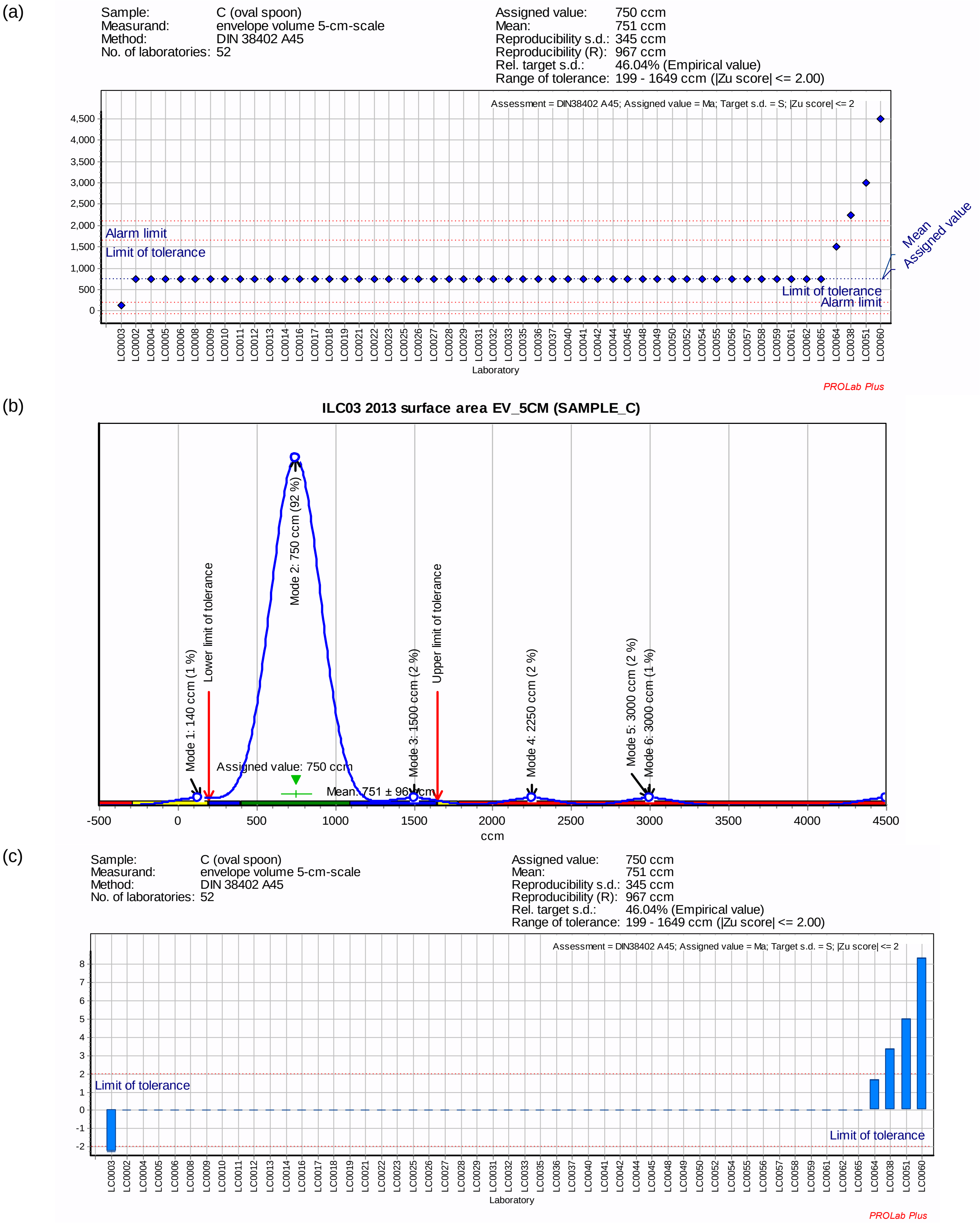


Figure 45 Summary of reported test results for the envelope volume of Sample C determined on a 5-cm-scale (a), Kernel density estimation (b) and z_U-scores (c)

14.3.3.4. Reported results and z_U-scores for the envelope volume of sample D

(a)

(b)

(c)

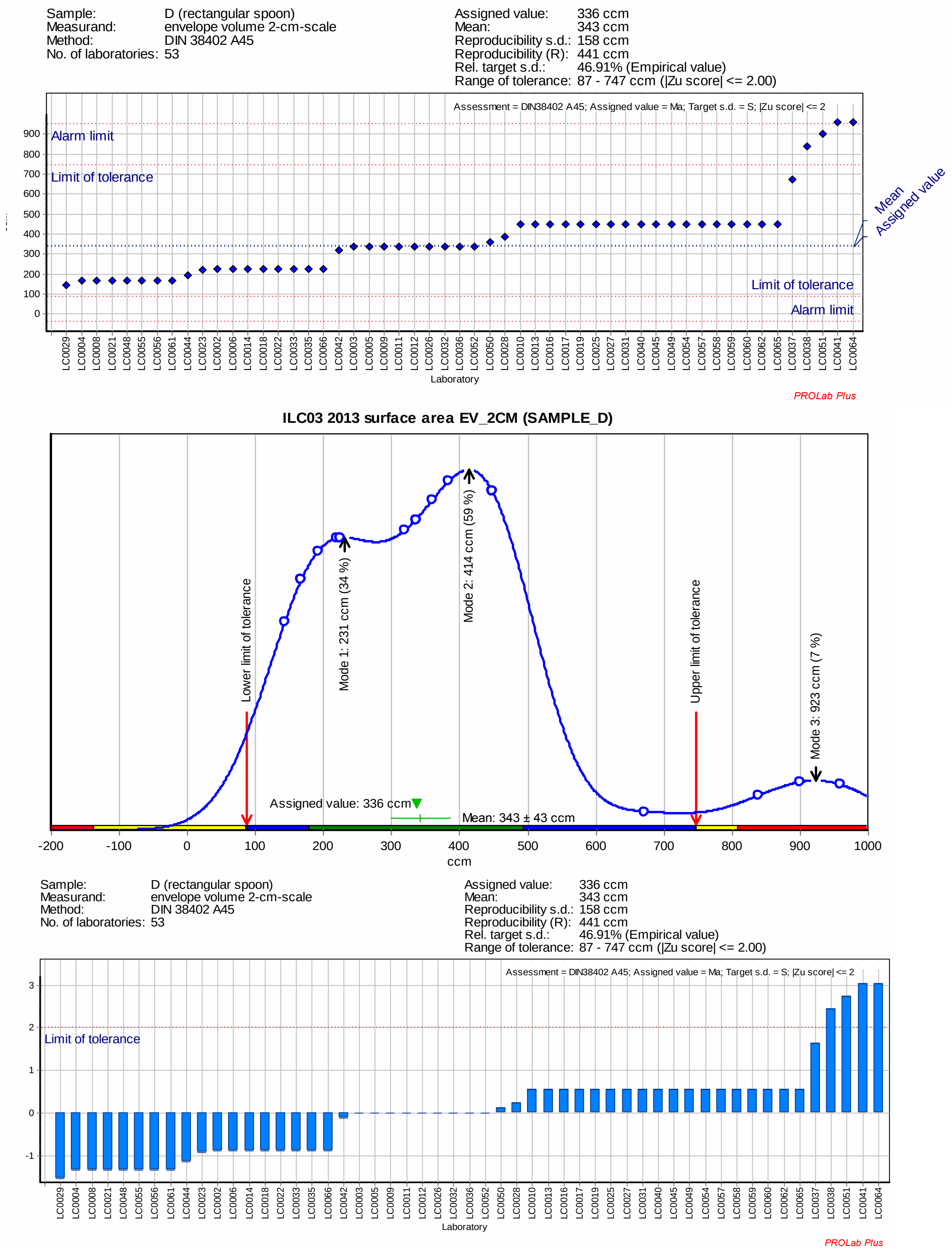
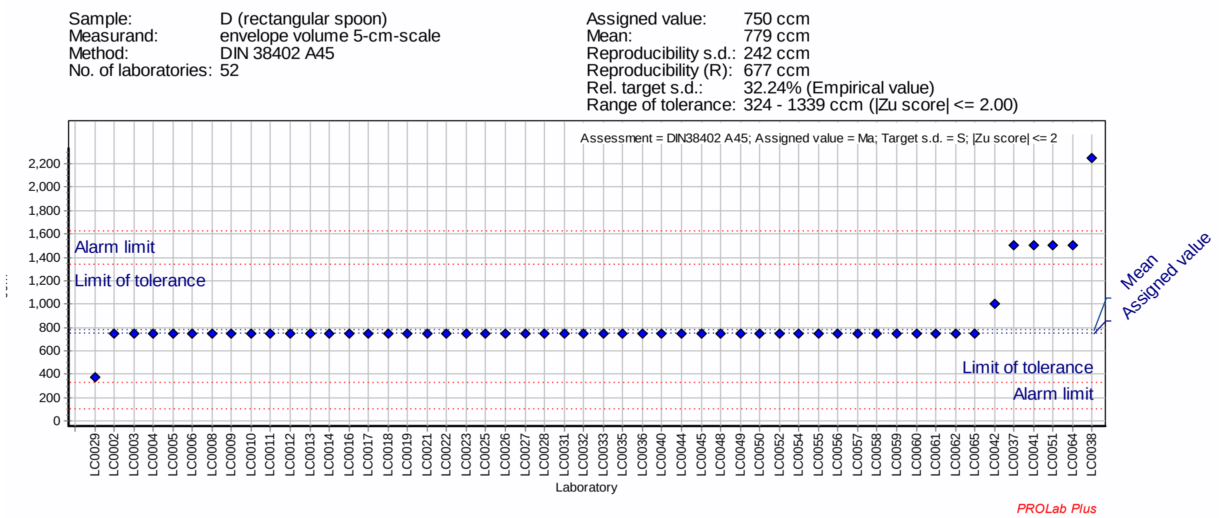
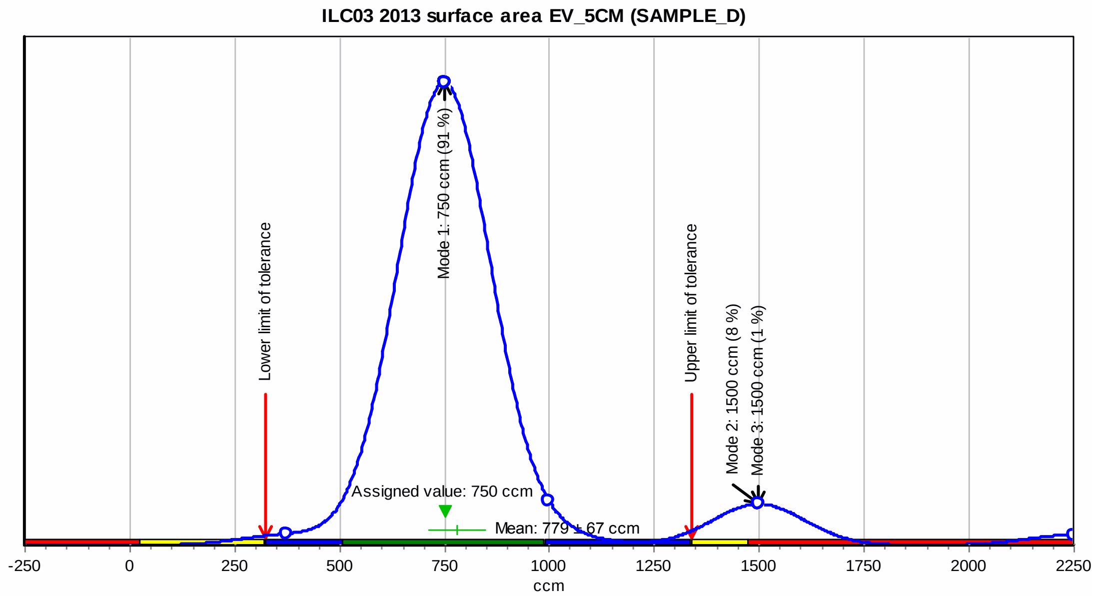


Figure 46 Summary of reported test results for the envelope volume of Sample D determined on a 2-cm-scale (a), Kernel density estimation (b) and z_U-scores (c)

(a)



(b)



(c)

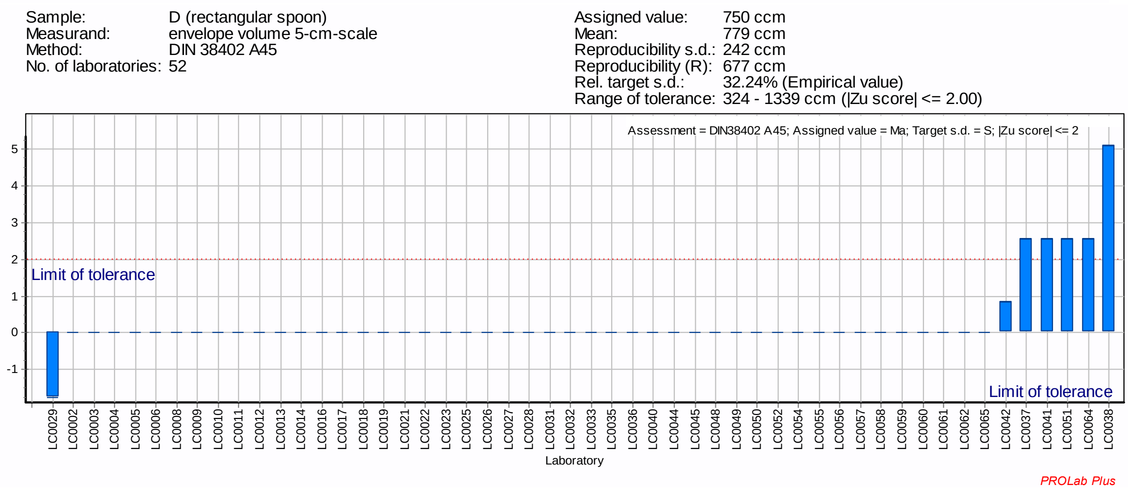
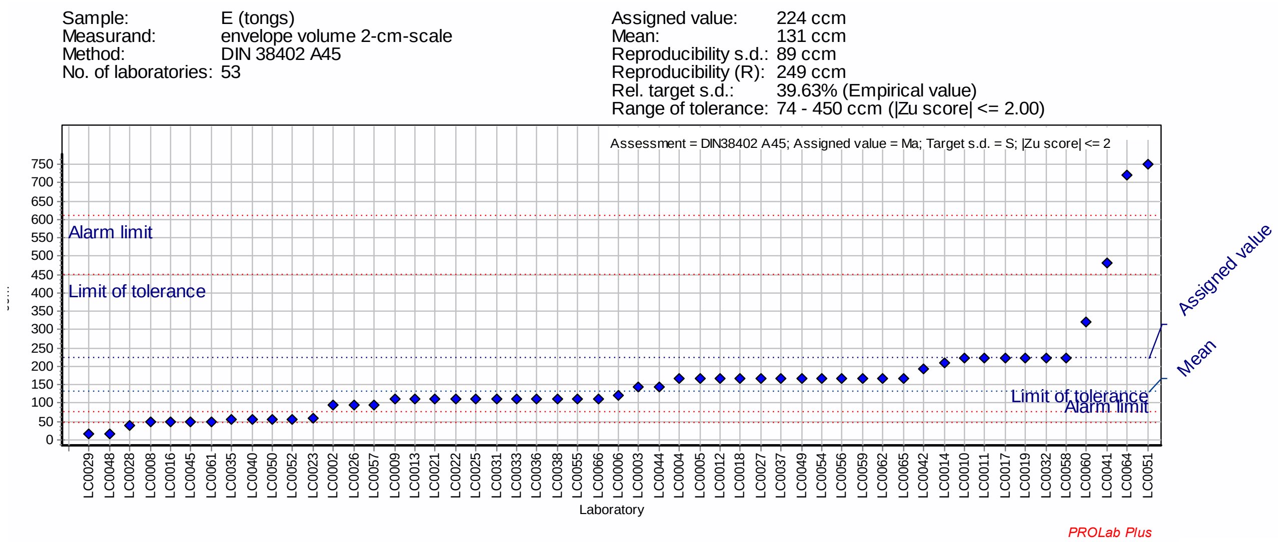


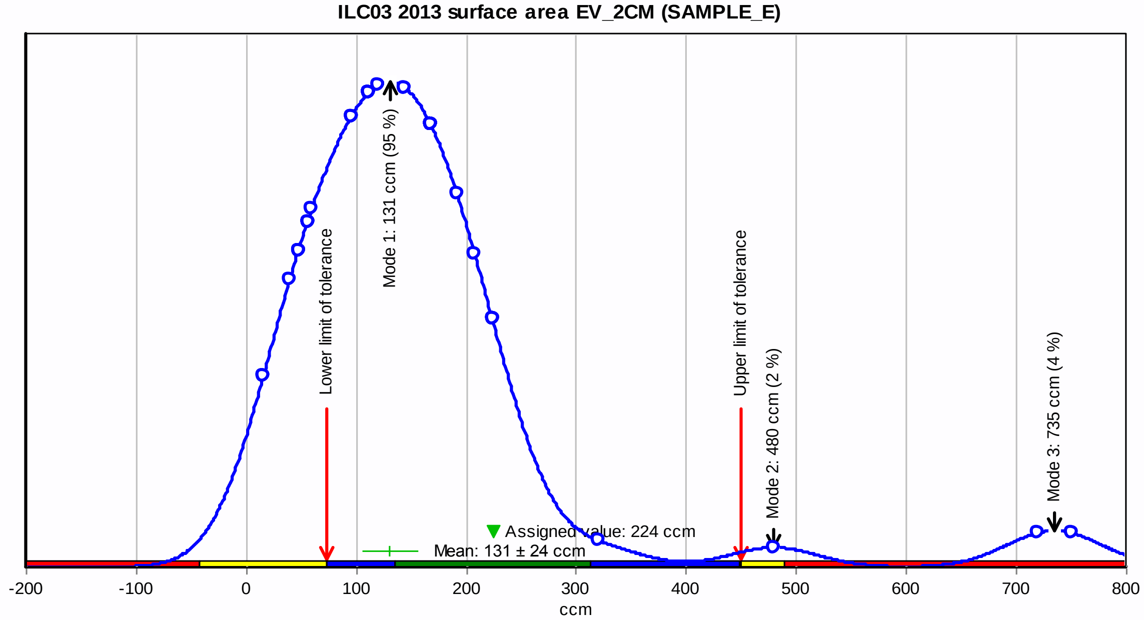
Figure 47 Summary of reported test results for the envelope volume of Sample D determined on a 5-cm-scale (a), Kernel density estimation (b) and z_U-scores (c)

14.3.3.5. Reported results and z_U-scores for the envelope volume of sample E

(a)



(b)



(c)

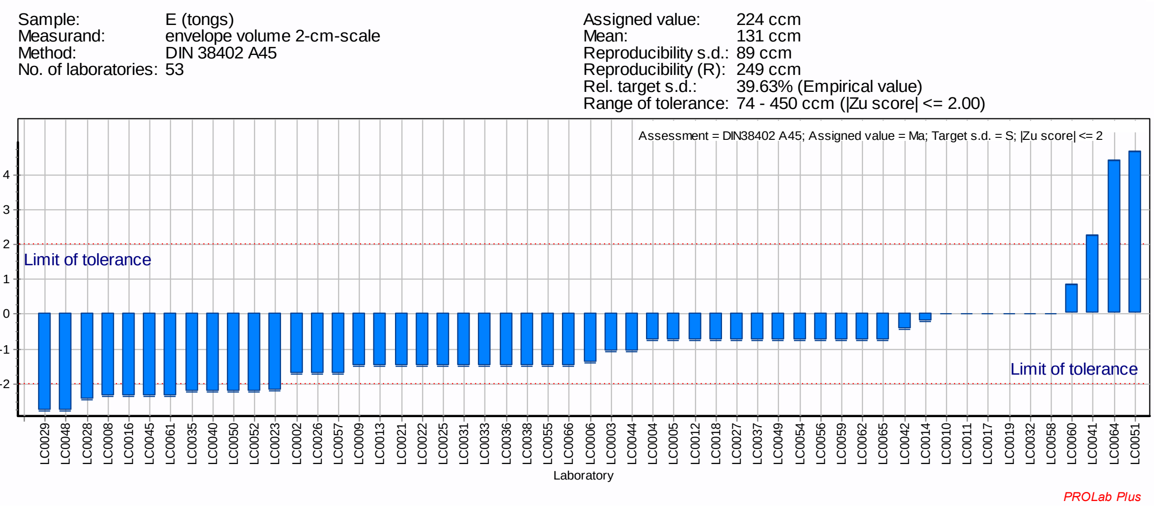
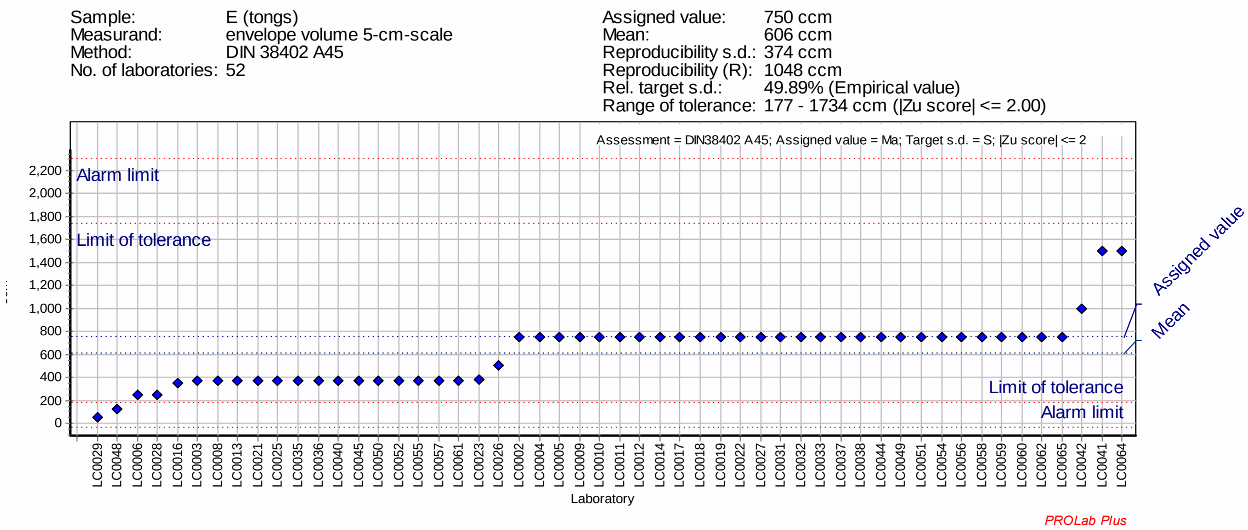
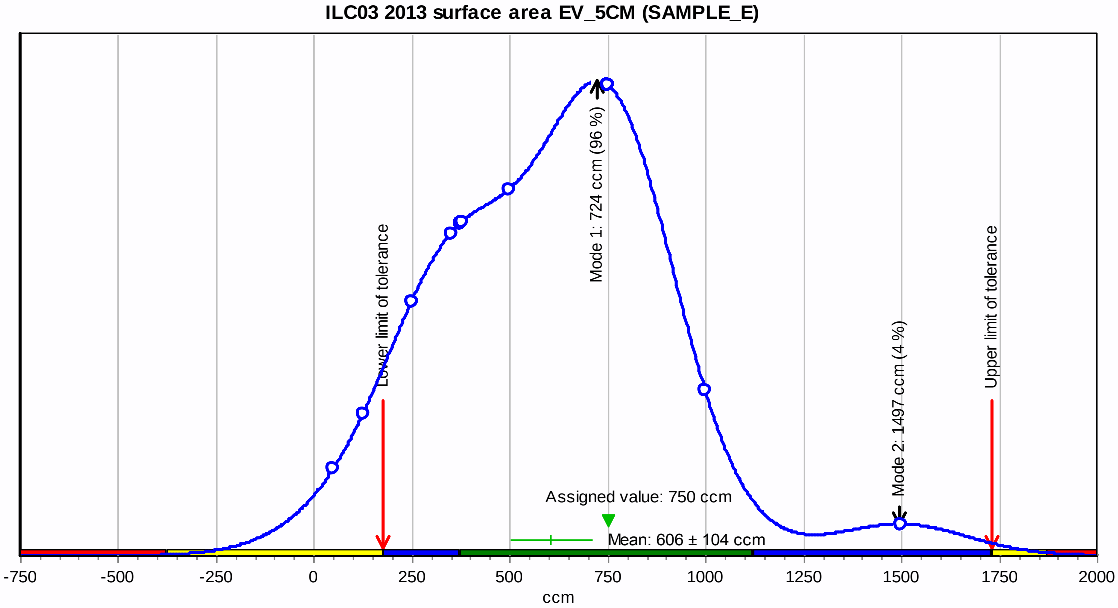


Figure 48 Summary of reported test results for the envelope volume of Sample E determined on a 2-cm-scale (a), Kernel density estimation (b) and z_U-scores (c)

(a)



(b)



(c)

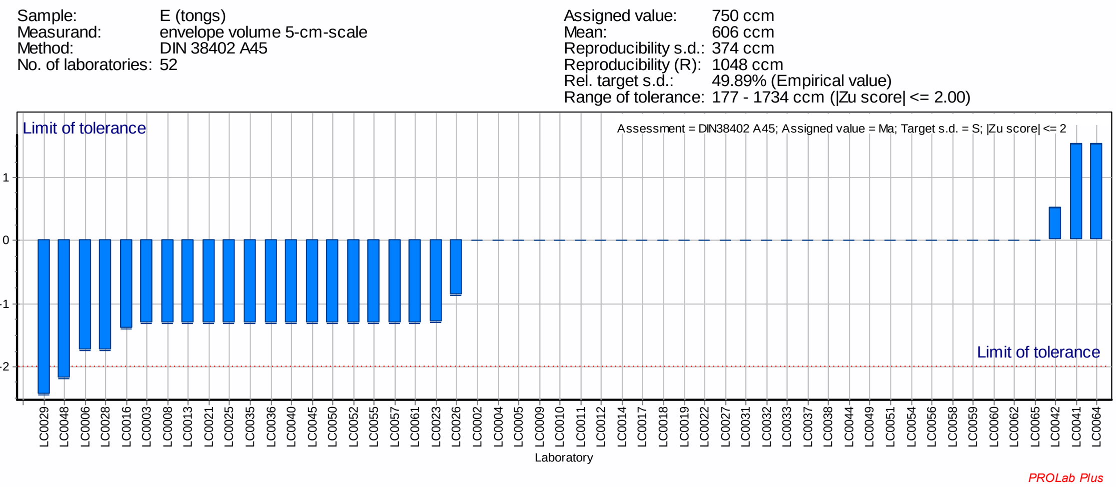
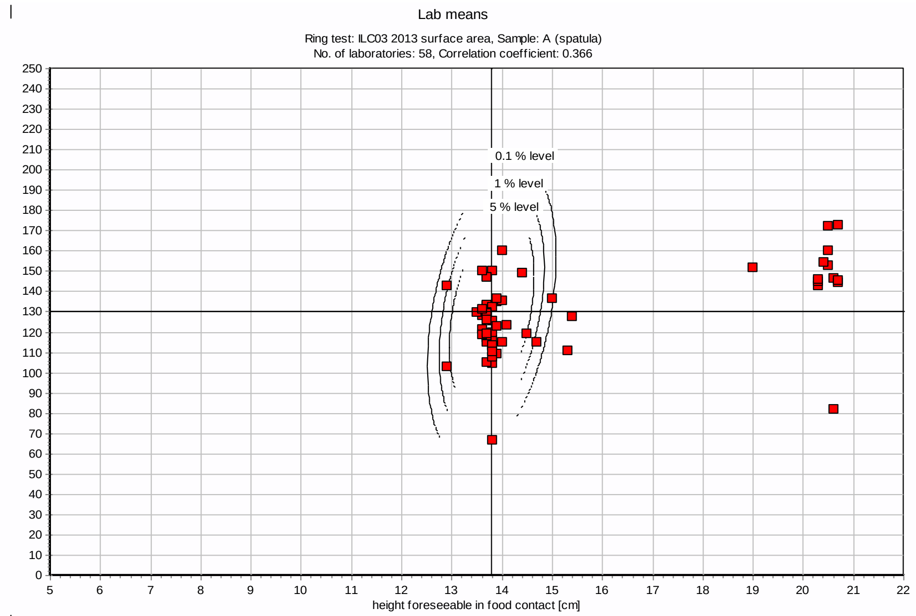


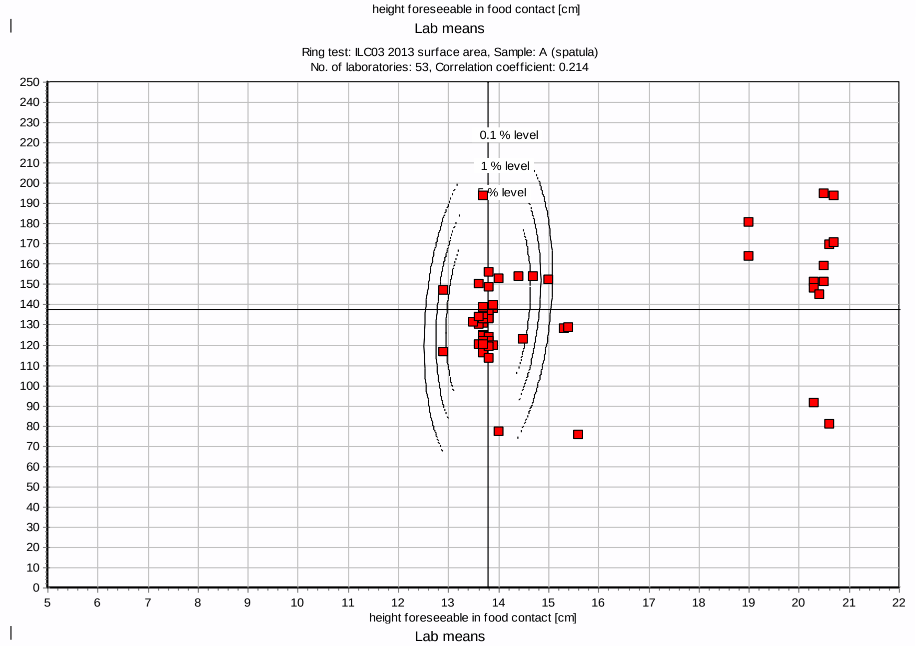
Figure 49 Summary of reported test results for the envelope volume of Sample E determined on a 5-cm-scale (a), Kernel density estimation (b) and z_U-scores (c)

14.3.4. Youden plots for the surface area and the envelope volume of sample A-E

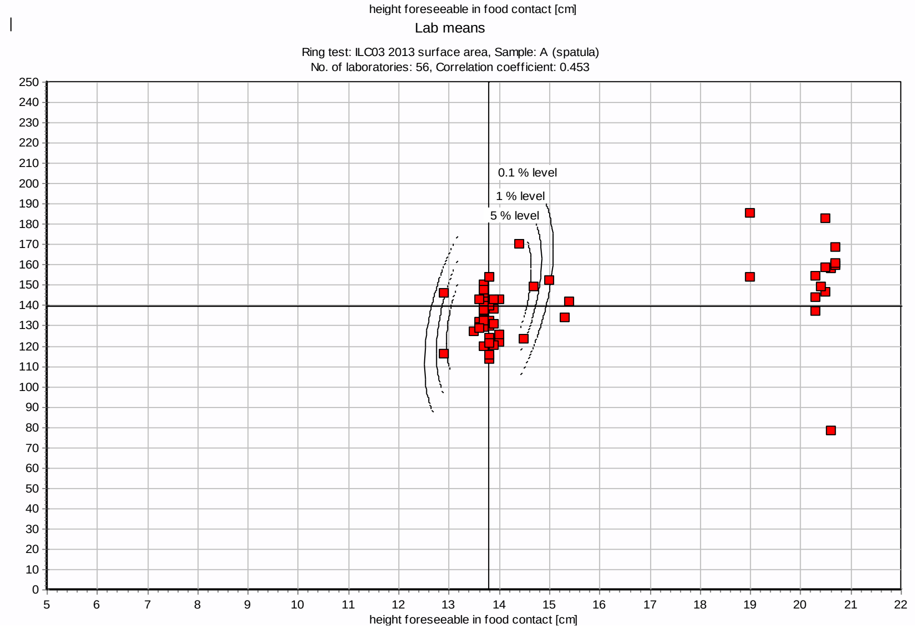
(a)



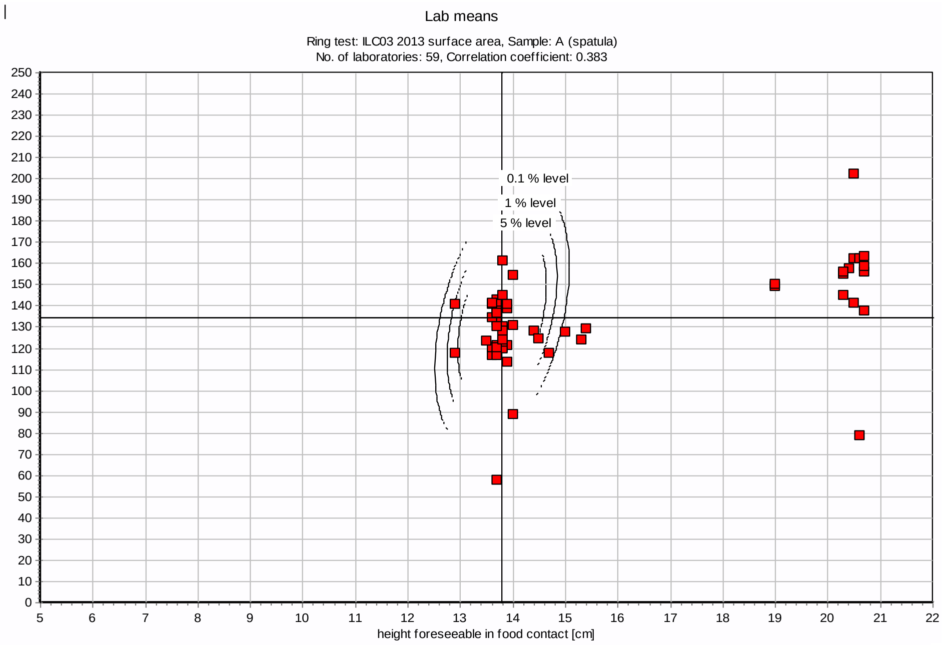
(b)



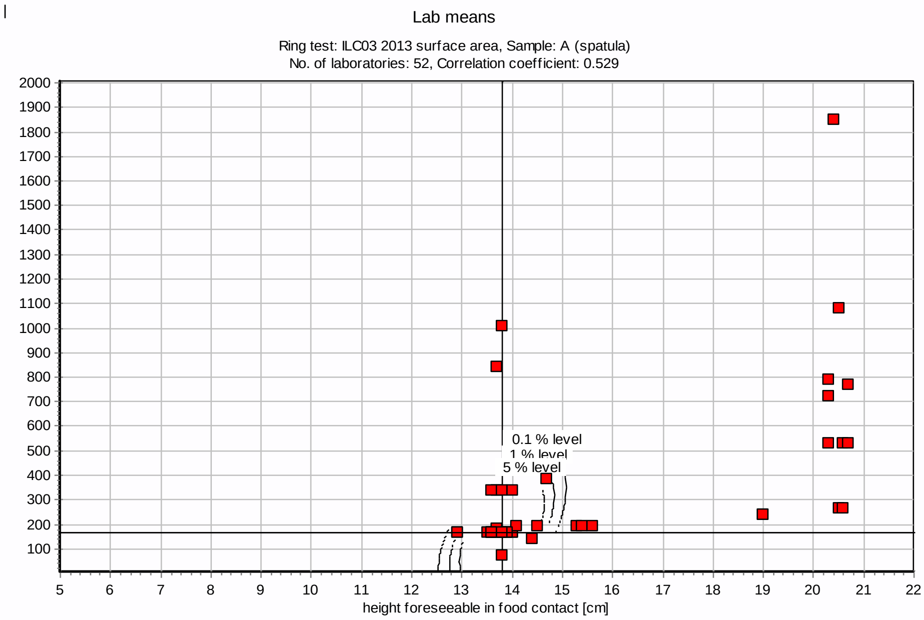
(c)



(d)



(e)



(f)

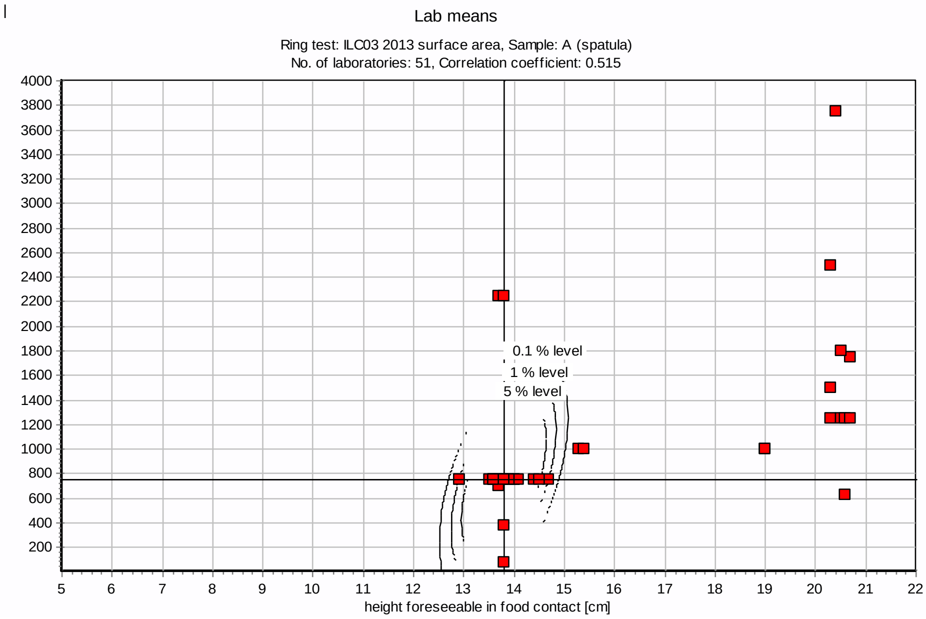
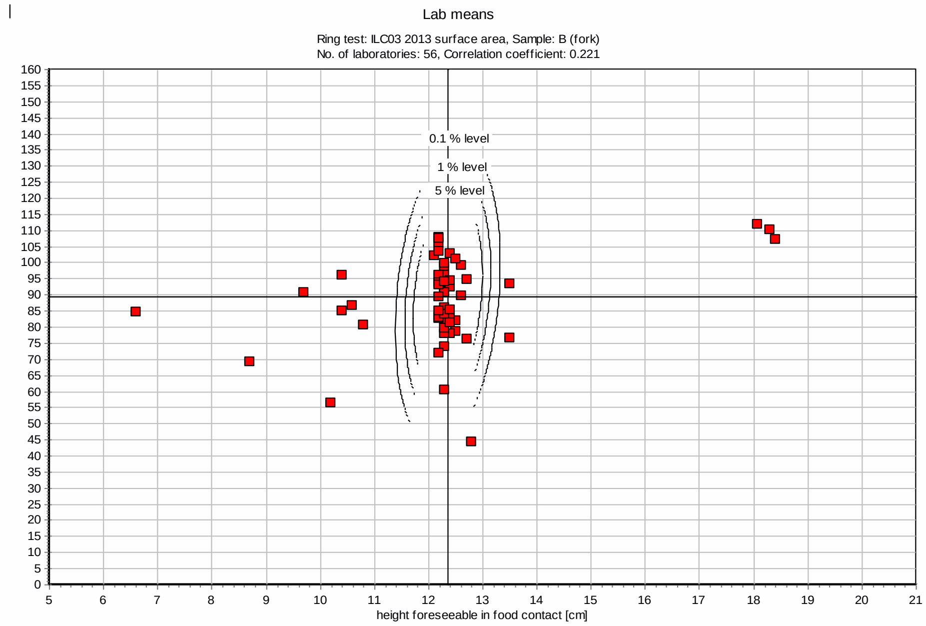
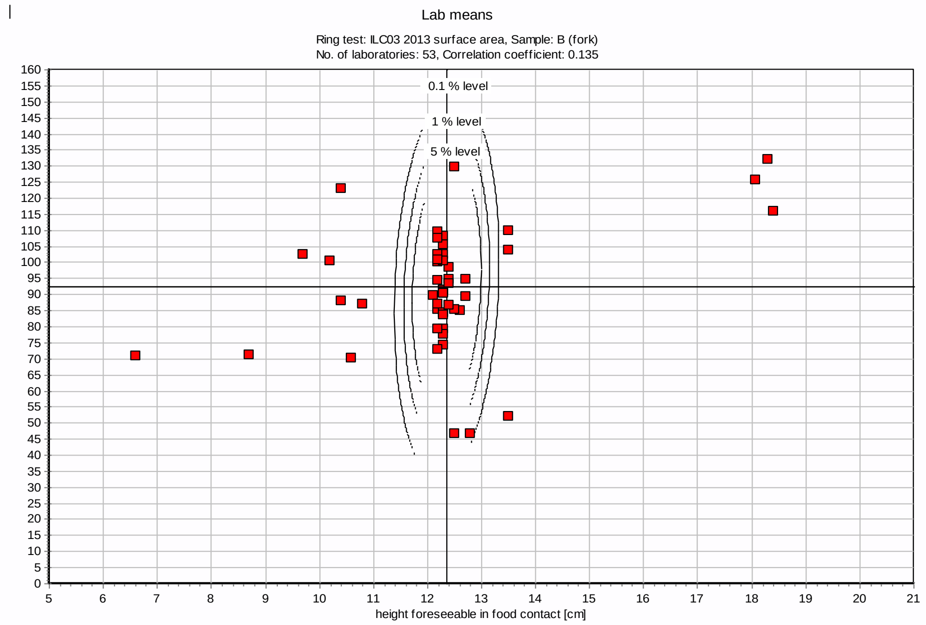


Figure 50 Youden plots for sample A displaying correlation of H_f and the surface area determined by "calculation" (a), "wrapping in paper" (b), "wrapping in aluminium foil" (c) and "drawing the shape" (d) and the envelope volume determined on a 2-cm-scale (e) and on a 5-cm-scale (f). Black solid lines mark the assigned values.

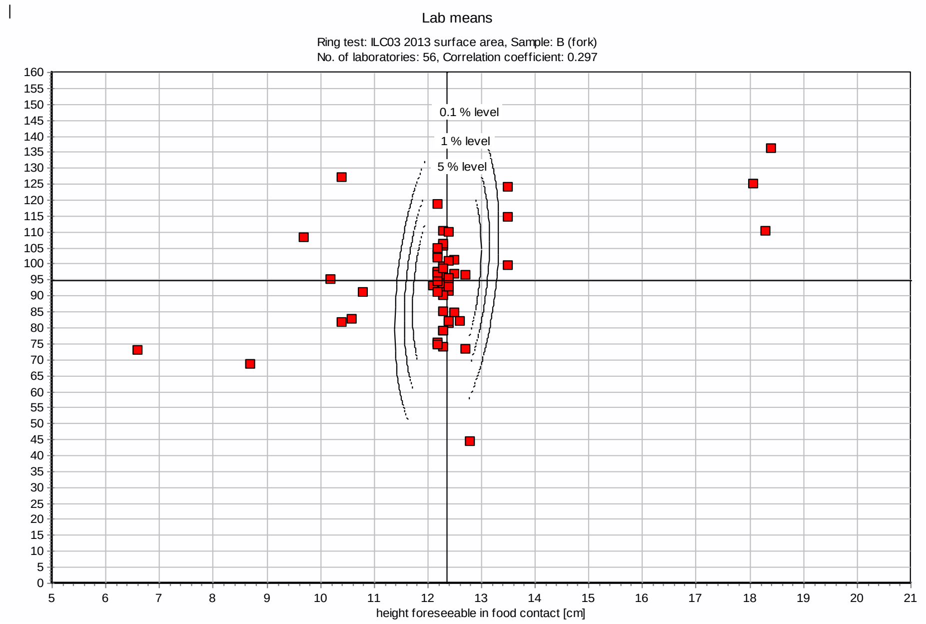
(a)


Lab means
Ring test: ILC03 2013 surface area, Sample: B (fork)
No. of laboratories: 56, Correlation coefficient: 0.221
0.1 % level
1 % level
5 % level
height foreseeable in food contact [cm]

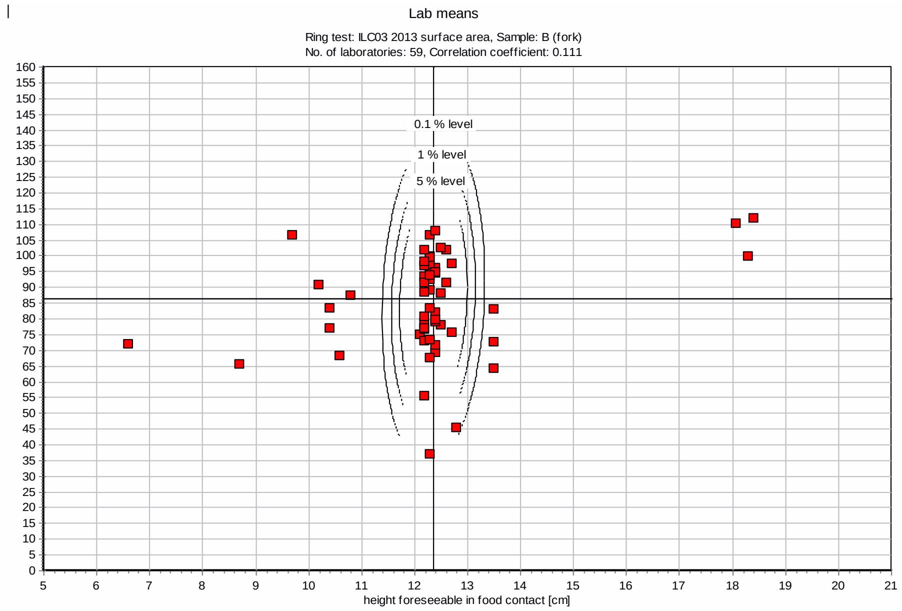
(b)


Lab means
Ring test: ILC03 2013 surface area, Sample: B (fork)
No. of laboratories: 53, Correlation coefficient: 0.135
0.1 % level
1 % level
5 % level
height foreseeable in food contact [cm]

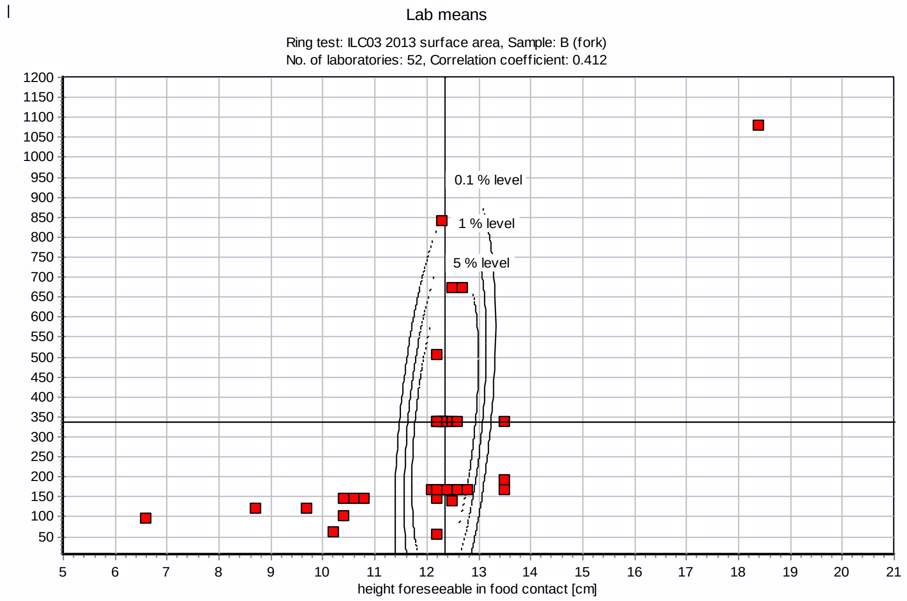
(c)


Lab means
Ring test: ILC03 2013 surface area, Sample: B (fork)
No. of laboratories: 56, Correlation coefficient: 0.297
0.1 % level
1 % level
5 % level
height foreseeable in food contact [cm]

(d)



(e)



(f)

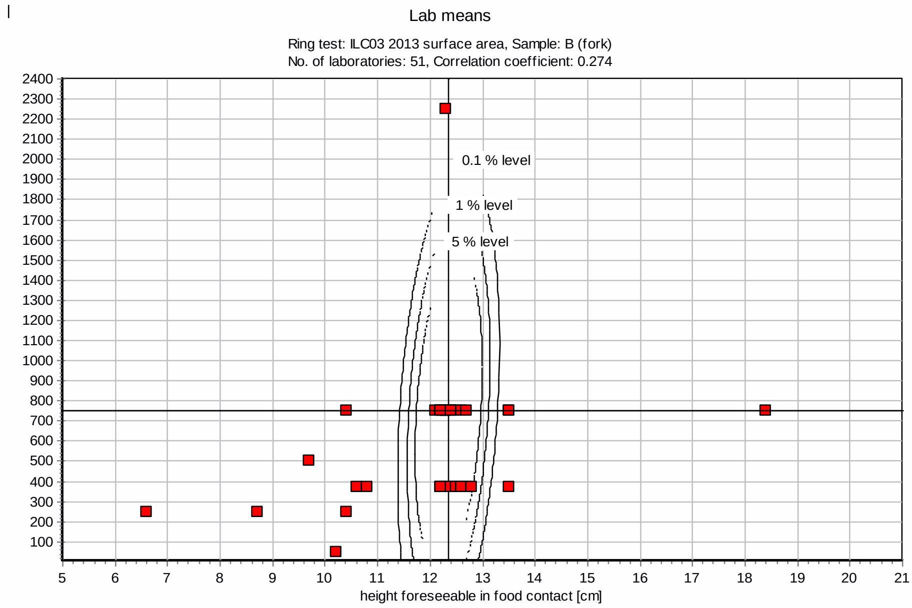
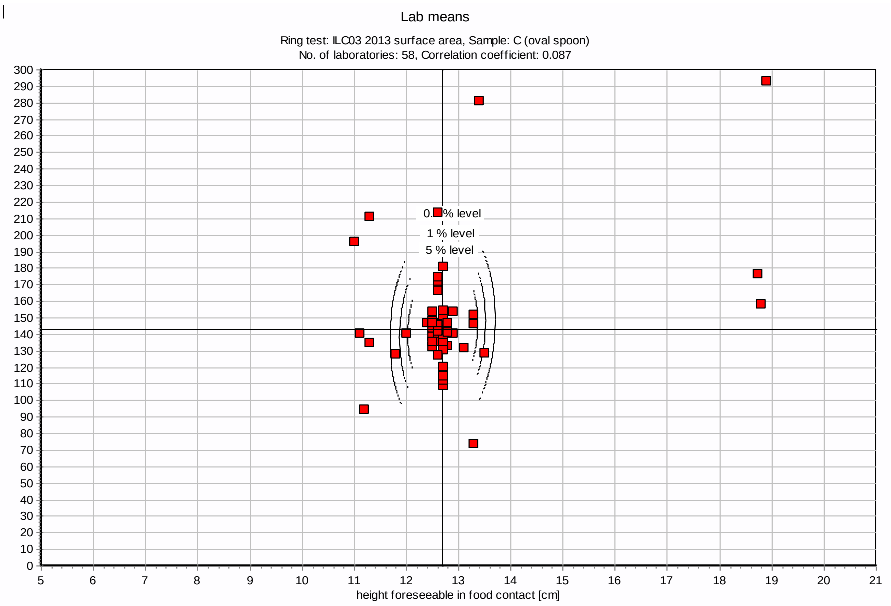
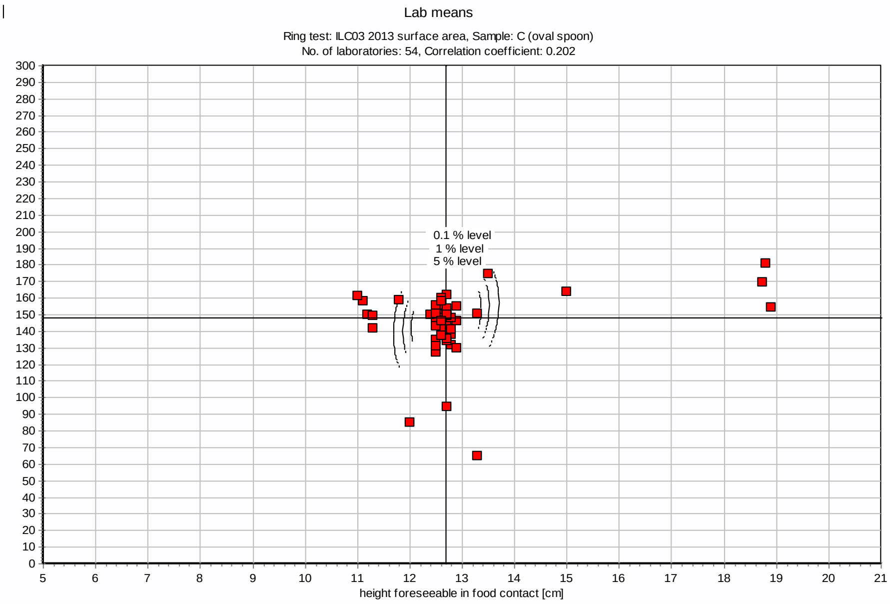


Figure 51 Youden plots for sample B displaying correlation of H_f and the surface area determined by "calculation" (a), "wrapping in paper" (b), "wrapping in aluminium foil" (c) and "drawing the shape" (d) and the envelope volume determined on a 2-cm-scale (e) and on a 5-cm-scale (f). Black solid lines mark the assigned values.

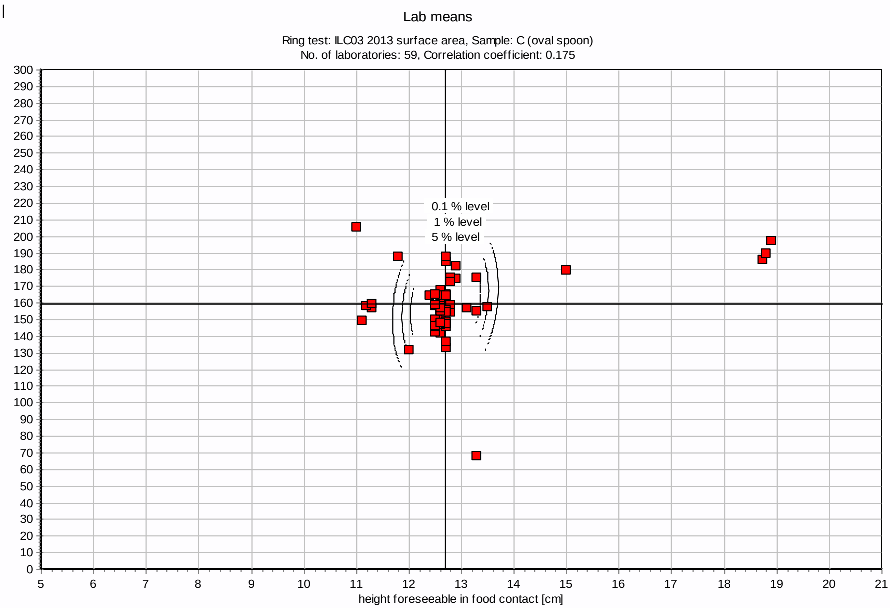
(a)


Lab means
Ring test: ILC03 2013 surface area, Sample: C (oval spoon)
No. of laboratories: 58, Correlation coefficient: 0.087
0.1 % level
1 % level
5 % level
height foreseeable in food contact [cm]

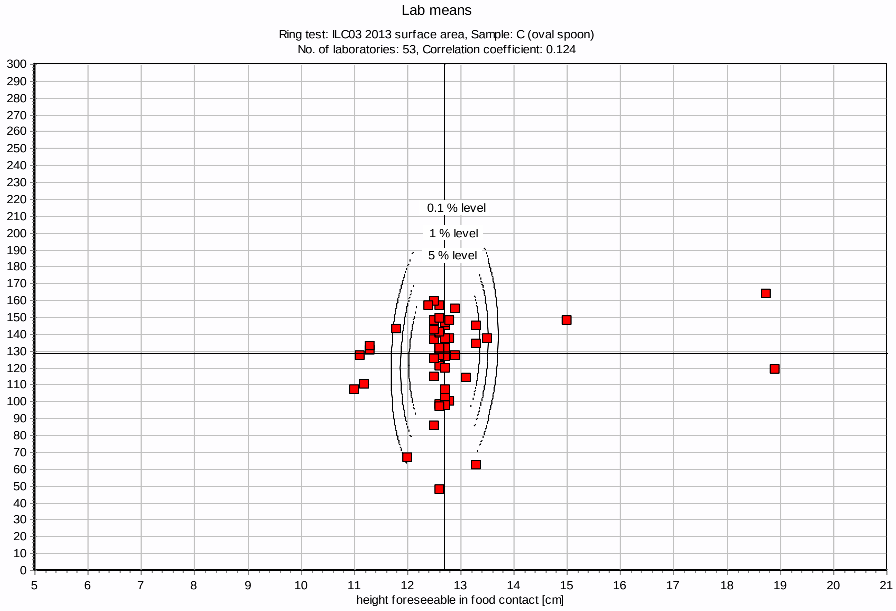
(b)


Lab means
Ring test: ILC03 2013 surface area, Sample: C (oval spoon)
No. of laboratories: 54, Correlation coefficient: 0.202
0.1 % level
1 % level
5 % level
height foreseeable in food contact [cm]

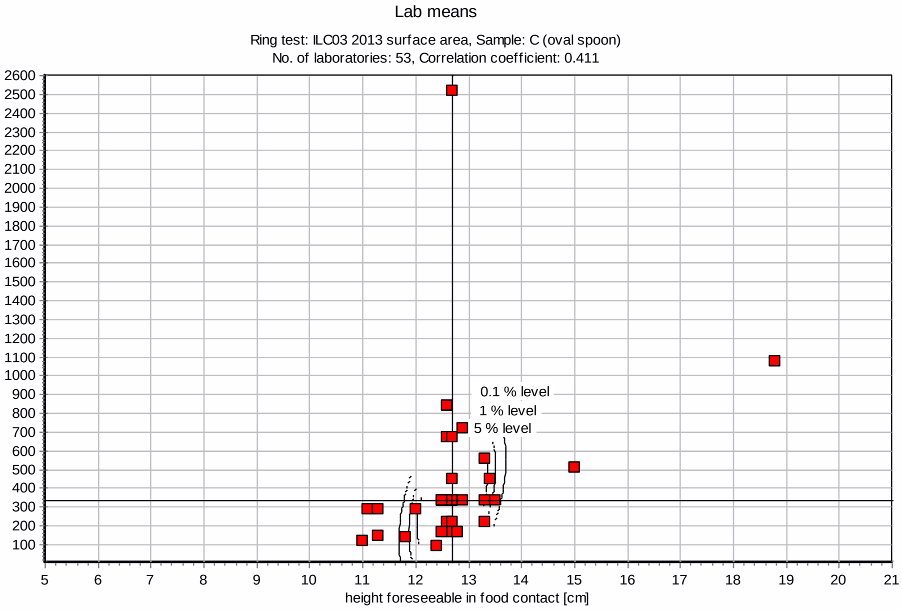
(c)


Lab means
Ring test: ILC03 2013 surface area, Sample: C (oval spoon)
No. of laboratories: 59, Correlation coefficient: 0.175
0.1 % level
1 % level
5 % level
height foreseeable in food contact [cm]

(d)



(e)



(f)

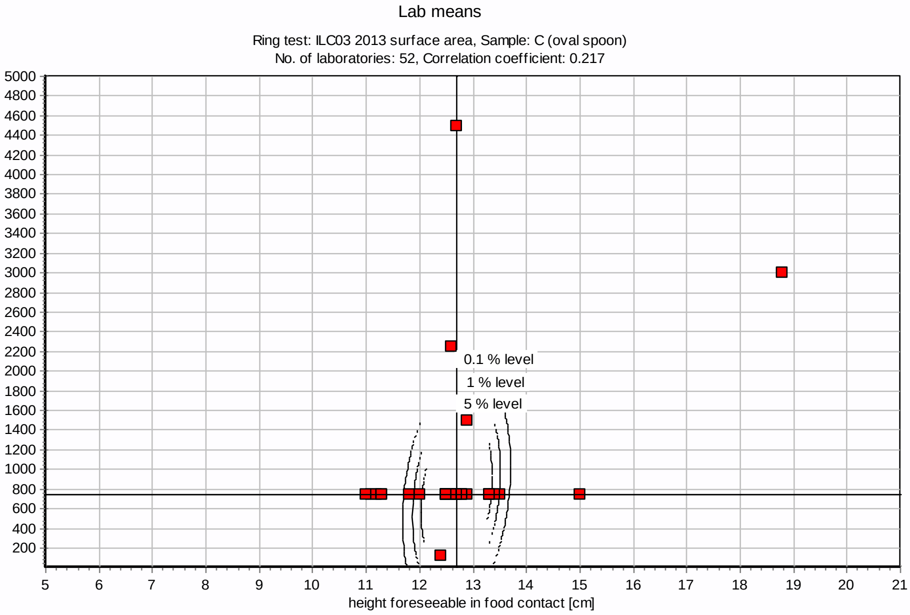
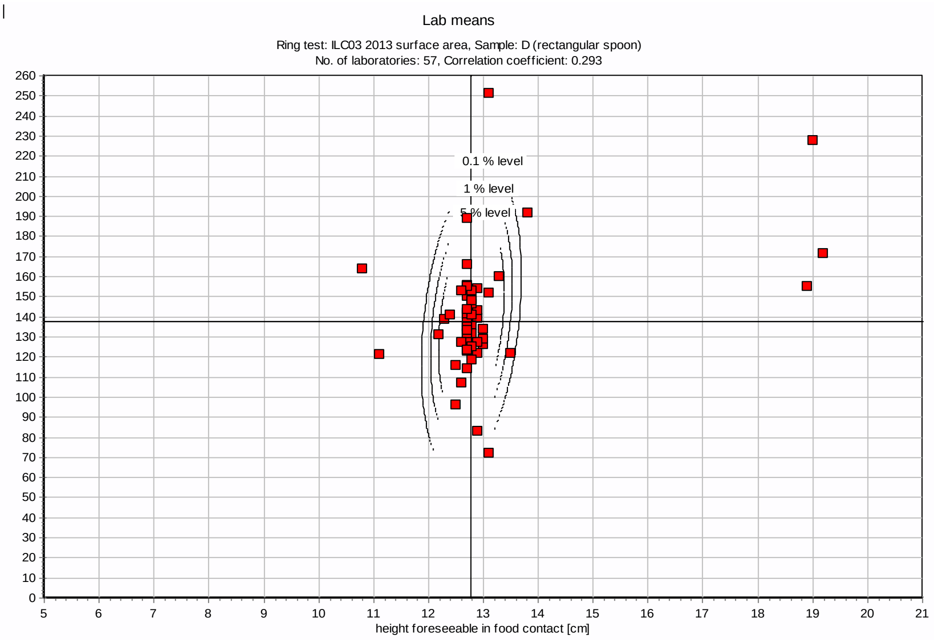
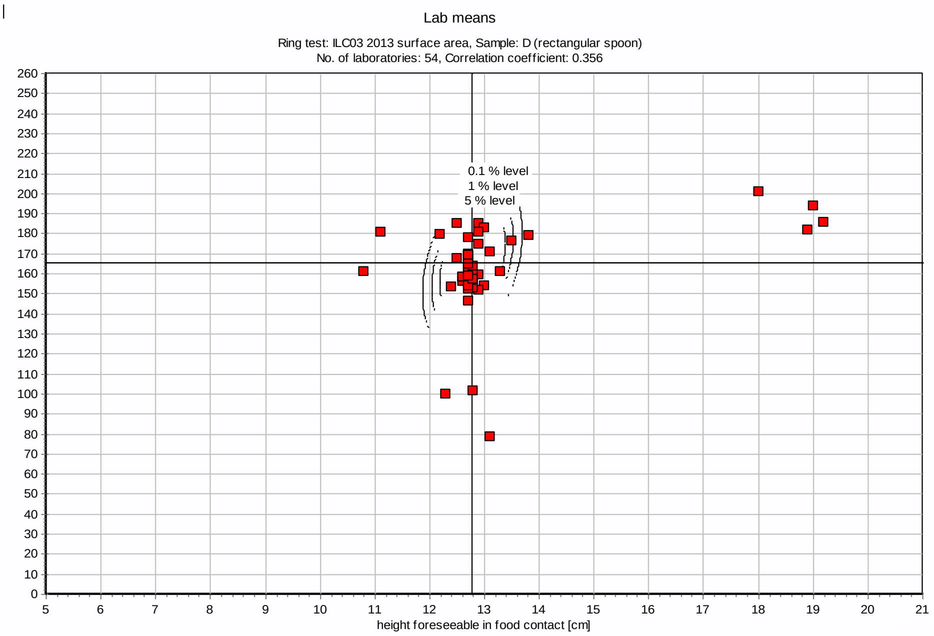


Figure 52 Youden plots for sample C displaying correlation of H_f and the surface area determined by "calculation" (a), "wrapping in paper" (b), "wrapping in aluminium foil" (c) and "drawing the shape" (d) and the envelope volume determined on a 2-cm-scale (e) and on a 5-cm-scale (f). Black solid lines mark the assigned values.

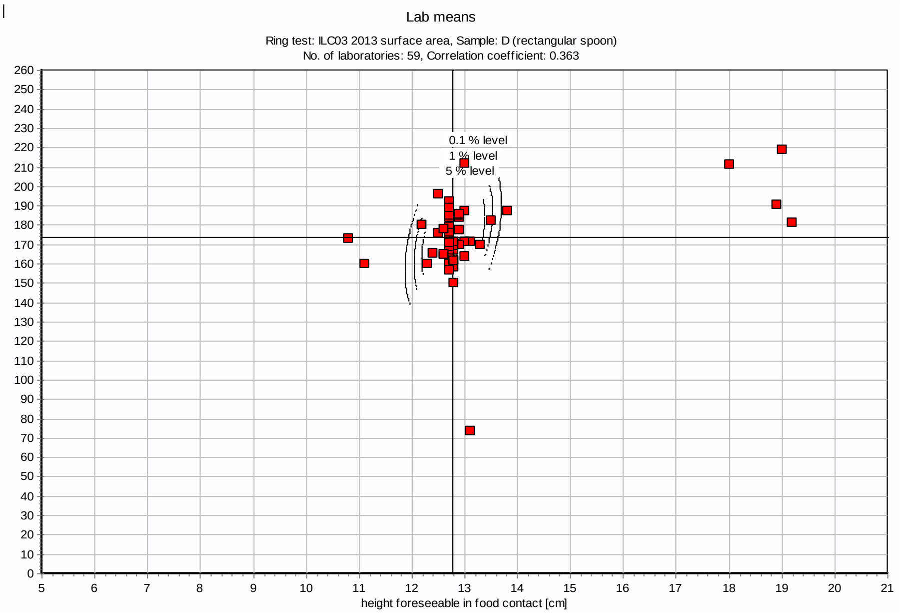
(a)


Lab means
Ring test: ILC03 2013 surface area, Sample: D (rectangular spoon)
No. of laboratories: 57, Correlation coefficient: 0.293
0.1 % level
1 % level
5 % level
height foreseeable in food contact [cm]

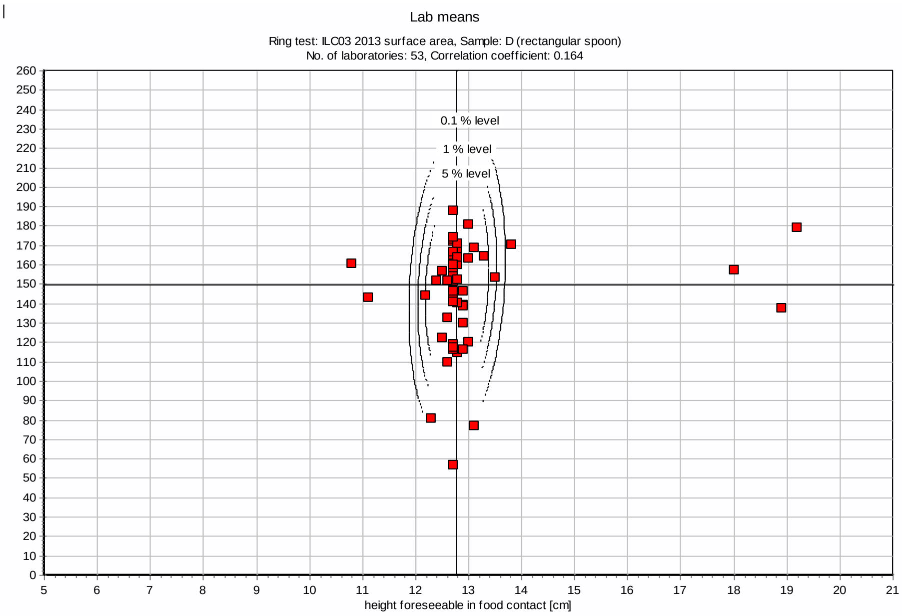
(b)


Lab means
Ring test: ILC03 2013 surface area, Sample: D (rectangular spoon)
No. of laboratories: 54, Correlation coefficient: 0.356
0.1 % level
1 % level
5 % level
height foreseeable in food contact [cm]

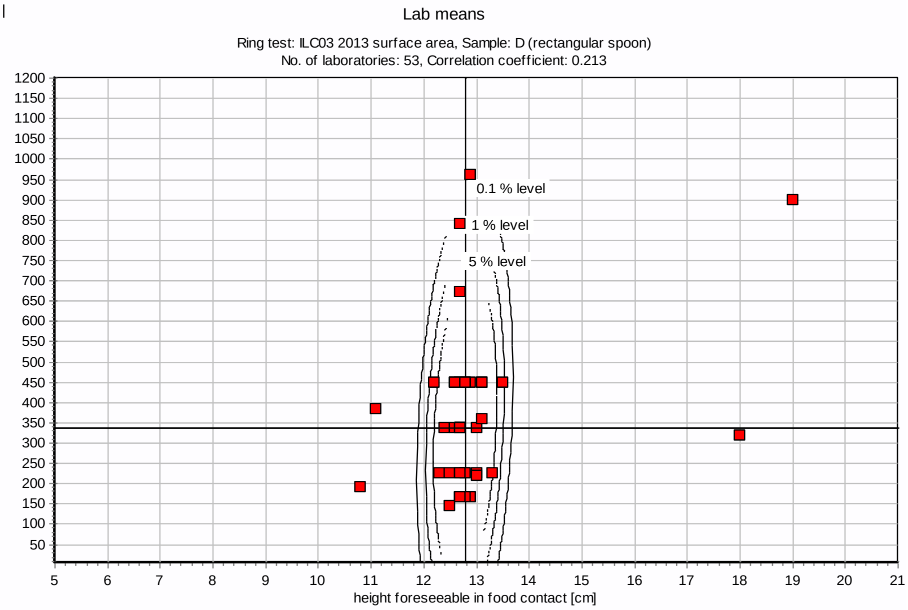
(c)


Lab means
Ring test: ILC03 2013 surface area, Sample: D (rectangular spoon)
No. of laboratories: 59, Correlation coefficient: 0.363
0.1 % level
1 % level
5 % level
height foreseeable in food contact [cm]

(d)



(e)



(f)

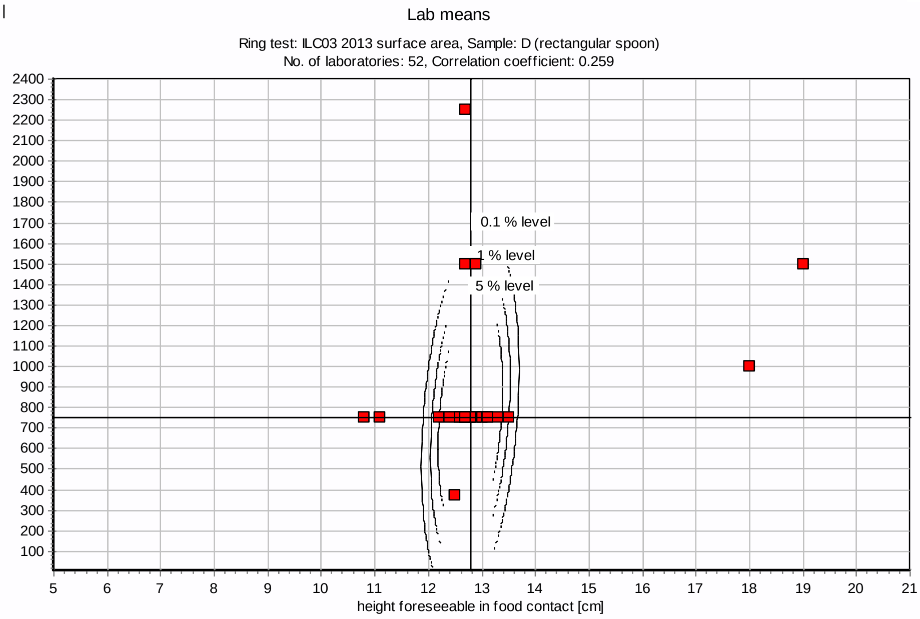
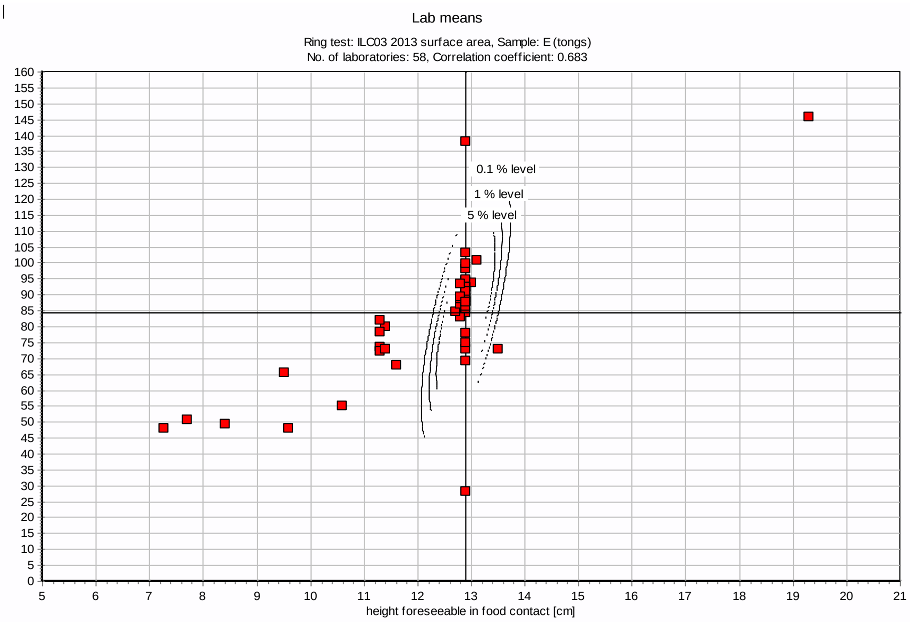
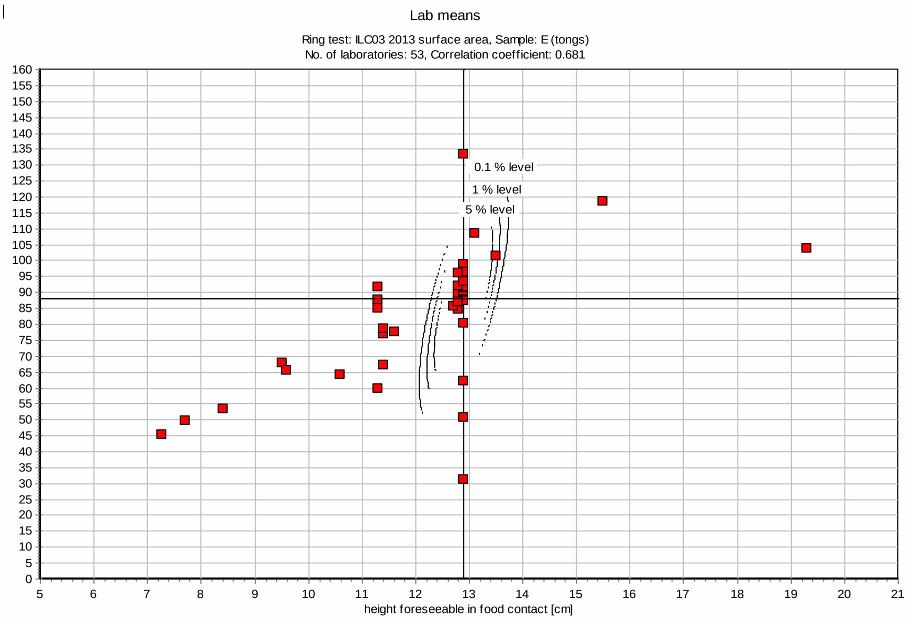


Figure 53 Youden plots for sample D displaying correlation of H_f and the surface area determined by "calculation" (a), "wrapping in paper" (b), "wrapping in aluminium foil" (c) and "drawing the shape" (d) and the envelope volume determined on a 2-cm-scale (e) and on a 5-cm-scale (f). Black solid lines mark the assigned values.

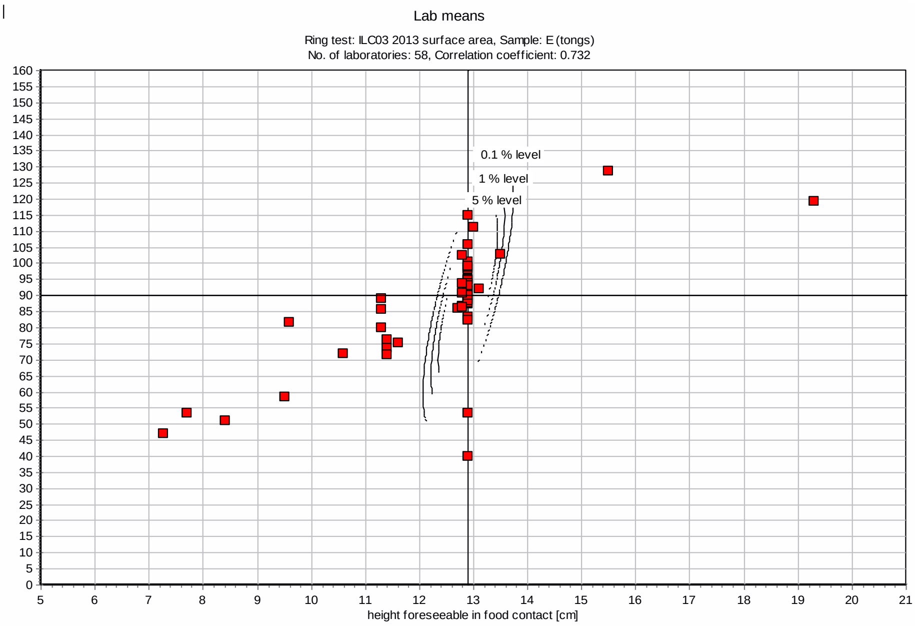
(a)


Lab means
Ring test: ILC03 2013 surface area, Sample: E (tongs)
No. of laboratories: 58, Correlation coefficient: 0.683
0.1 % level
1 % level
5 % level
height foreseeable in food contact [cm]

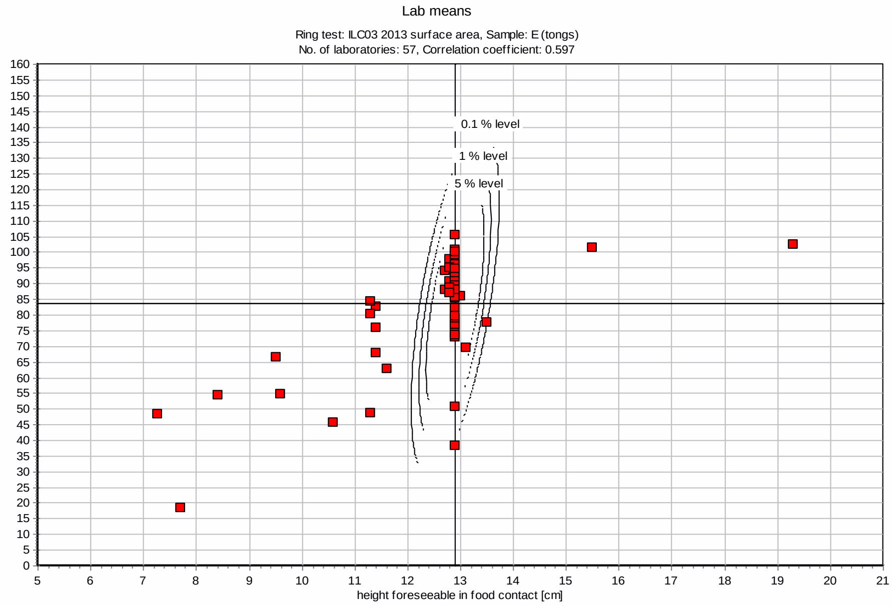
(b)


Lab means
Ring test: ILC03 2013 surface area, Sample: E (tongs)
No. of laboratories: 53, Correlation coefficient: 0.681
0.1 % level
1 % level
5 % level
height foreseeable in food contact [cm]

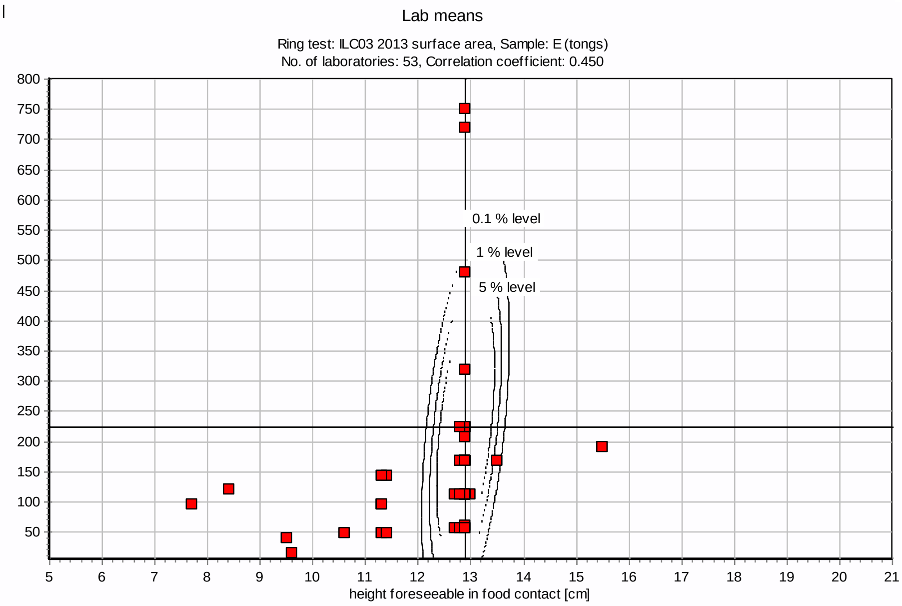
(c)


Lab means
Ring test: ILC03 2013 surface area, Sample: E (tongs)
No. of laboratories: 58, Correlation coefficient: 0.732
0.1 % level
1 % level
5 % level
height foreseeable in food contact [cm]

(d)



(e)



(f)

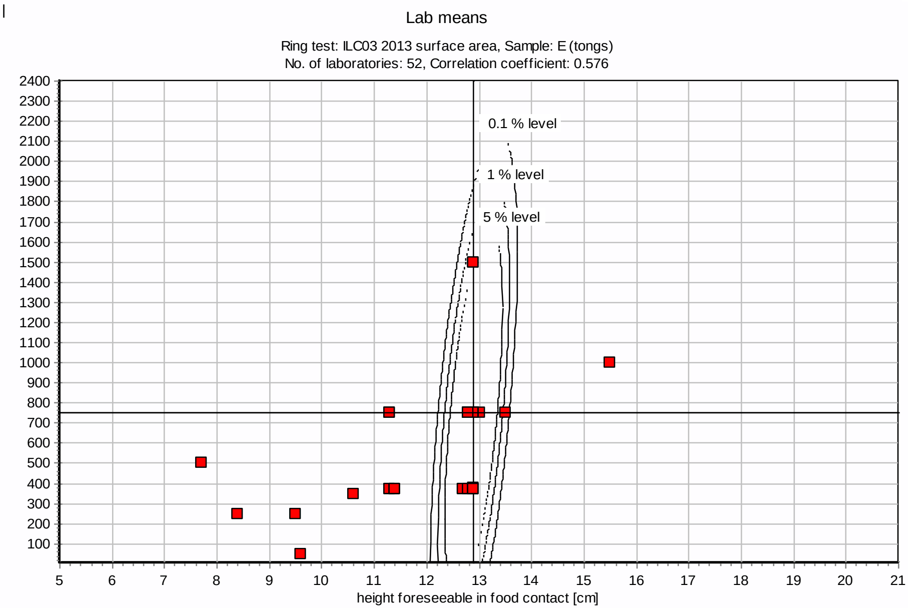
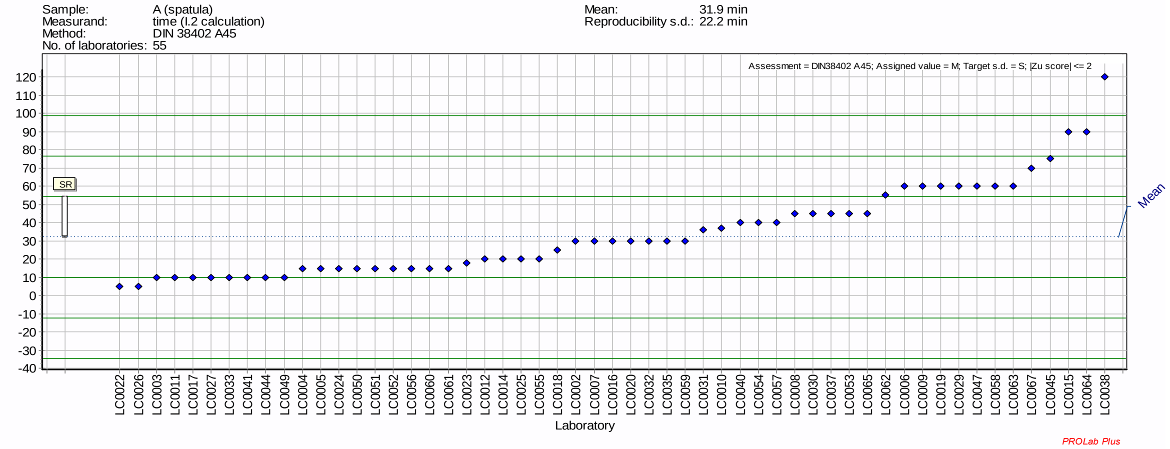


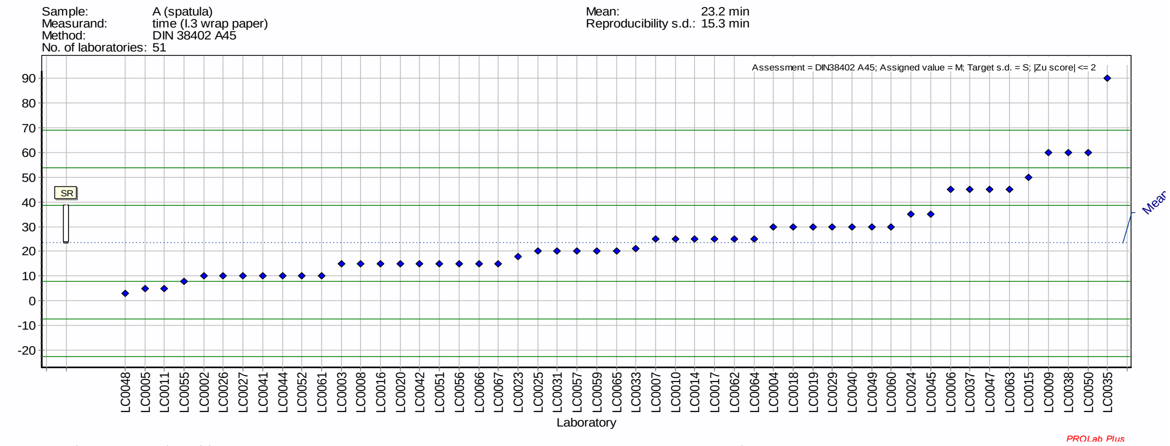
Figure 54 Youden plots for sample E displaying correlation of H_f and the surface area determined by "calculation" (a), "wrapping in paper" (b), "wrapping in aluminium foil" (c) and "drawing the shape" (d) and the envelope volume determined on a 2-cm-scale (e) and on a 5-cm-scale (f). Black solid lines mark the assigned values.

14.3.5. Reported amounts of time spent for the determination of the surface area

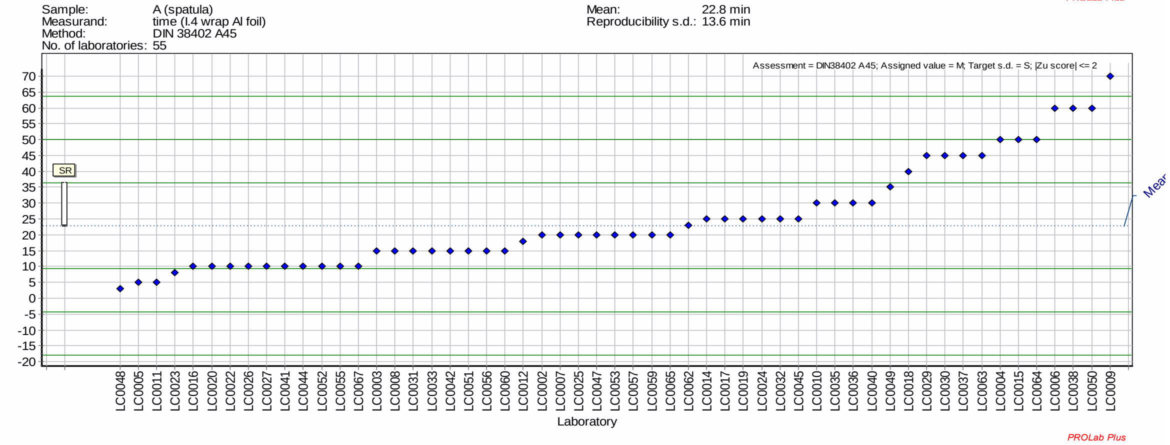
(a)



(b)



(c)



(d)

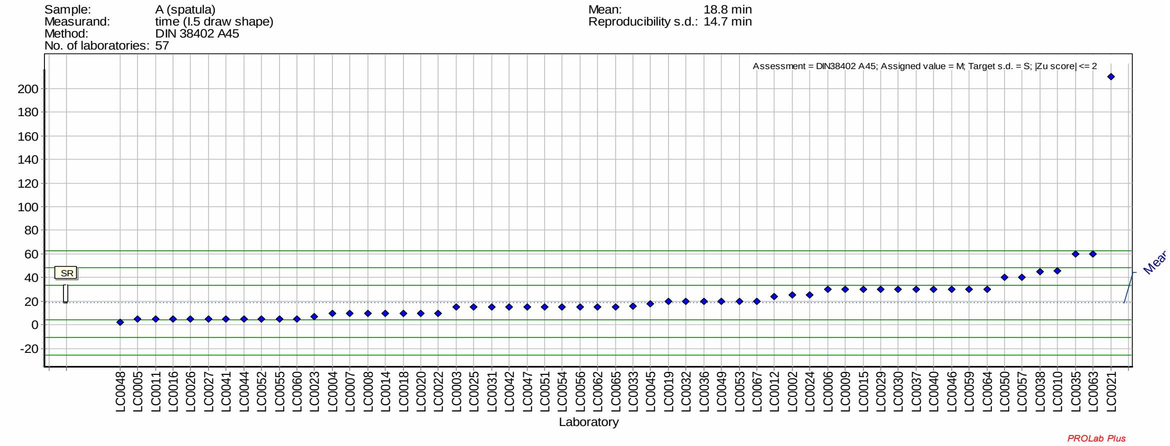
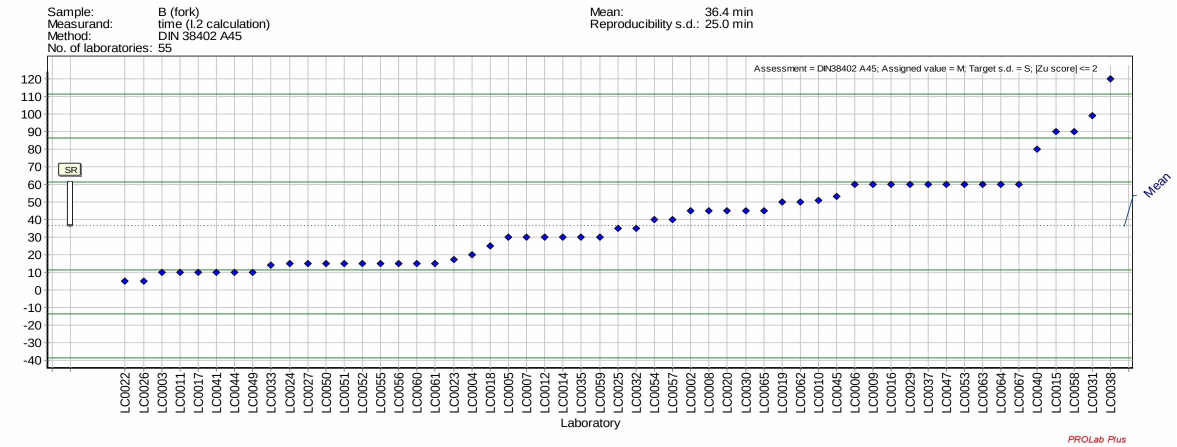
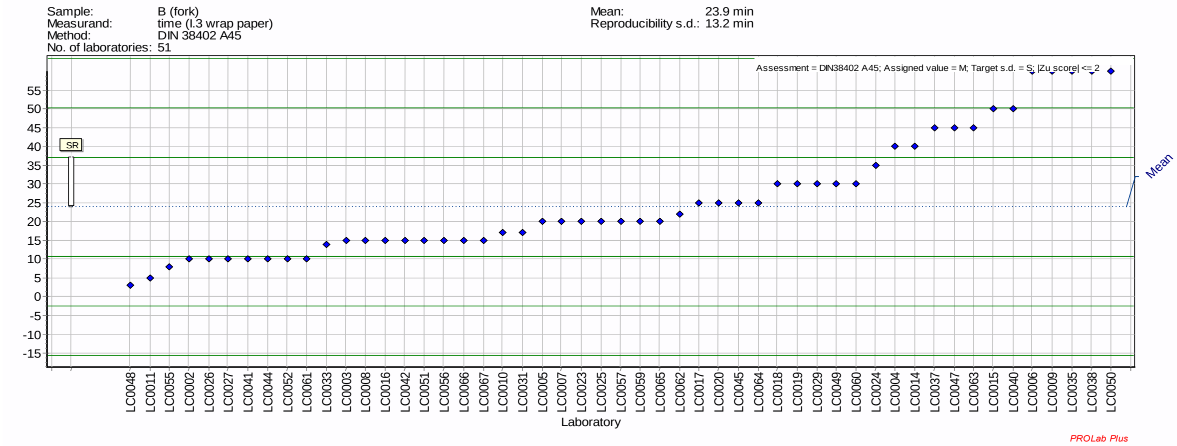


Figure 55 Time spent for the determination of the surface area of sample A by "calculation" (a), "wrapping in paper" (b), "wrapping in aluminium foil" (c) and "drawing the shape" (d)

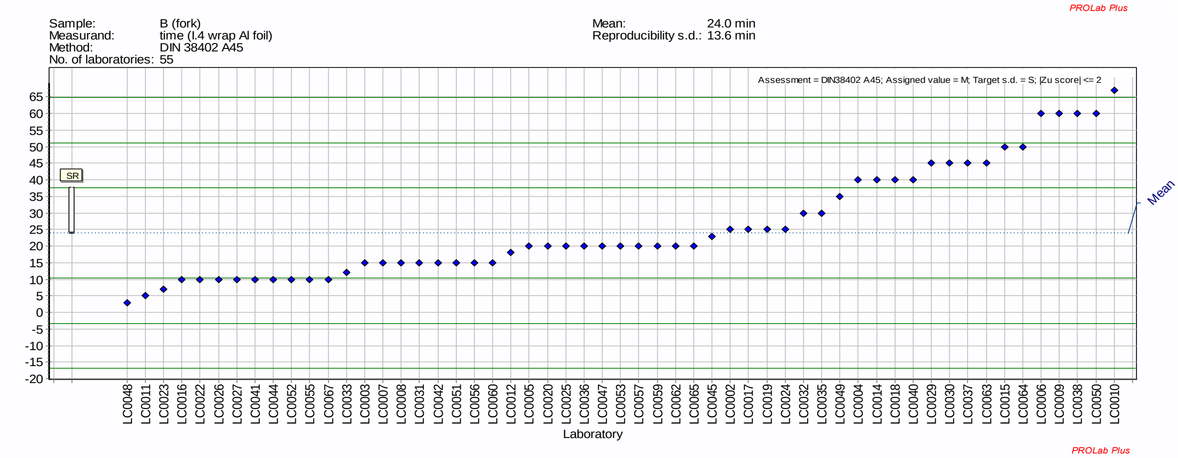
(a)



(b)



(c)



(d)

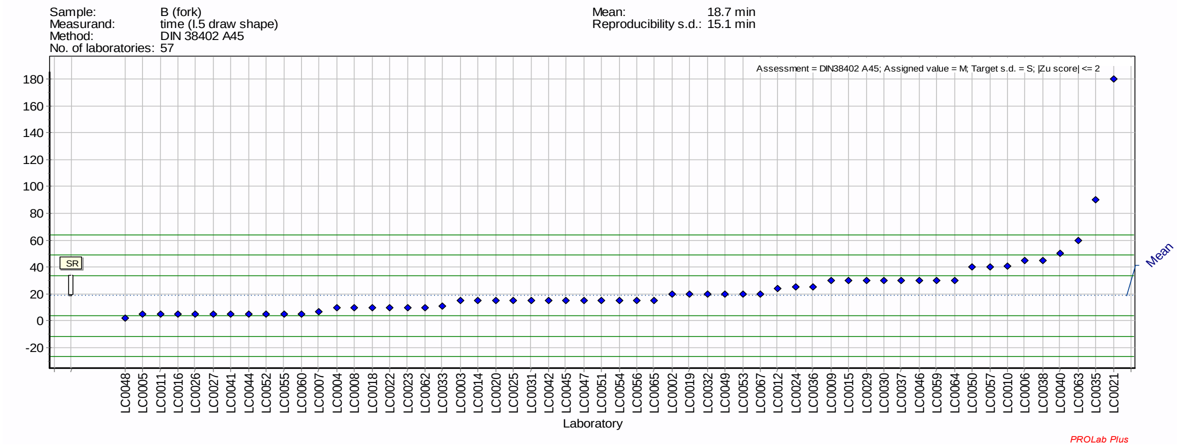


Figure 56 Time spent for the determination of the surface area of sample B by "calculation" (a), "wrapping in paper" (b), "wrapping in aluminium foil" (c) and "drawing the shape" (d)

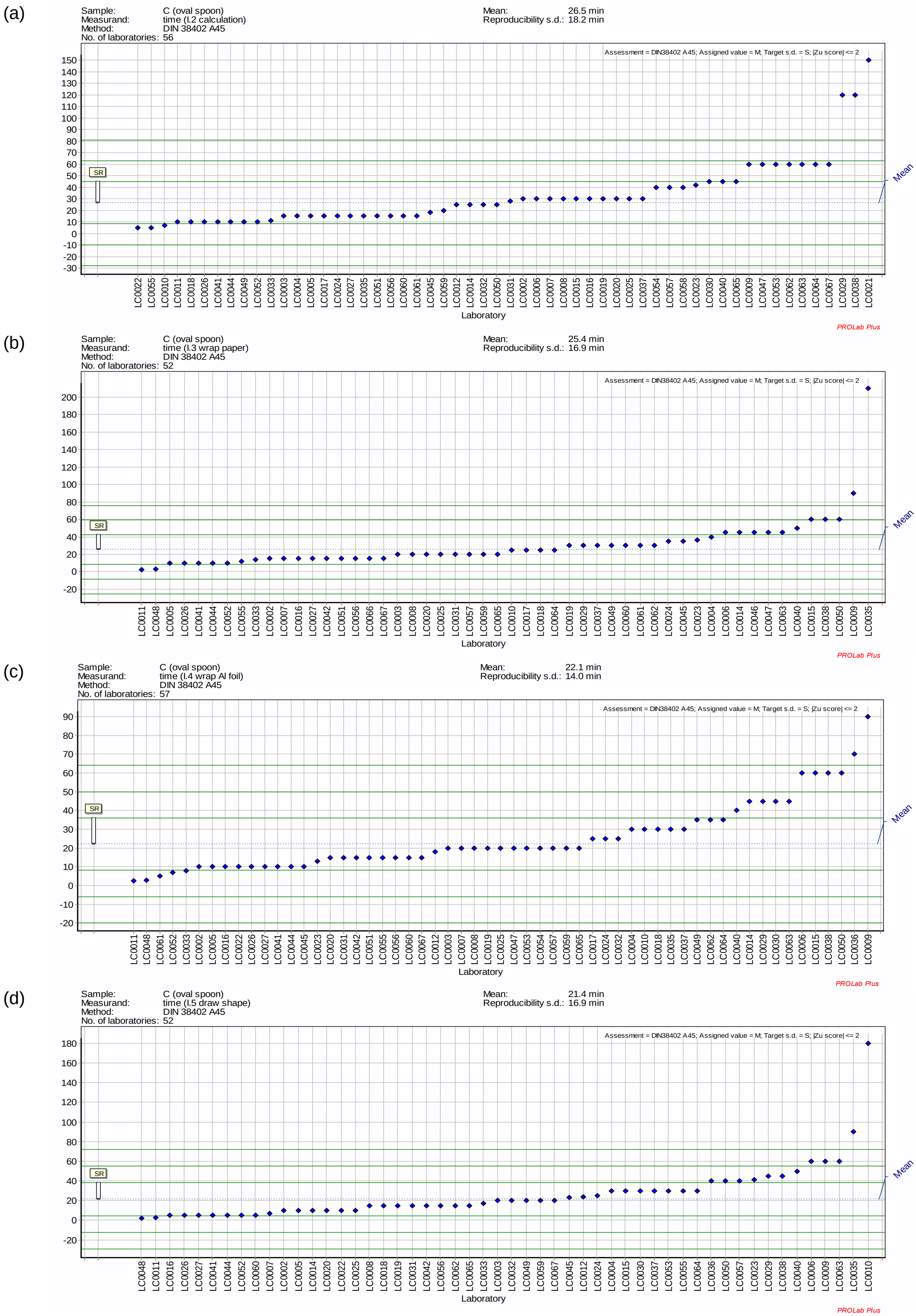


Figure 57 Time spent for the determination of the surface area of sample C by "calculation" (a), "wrapping in paper" (b), "wrapping in aluminium foil" (c) and "drawing the shape" (d)

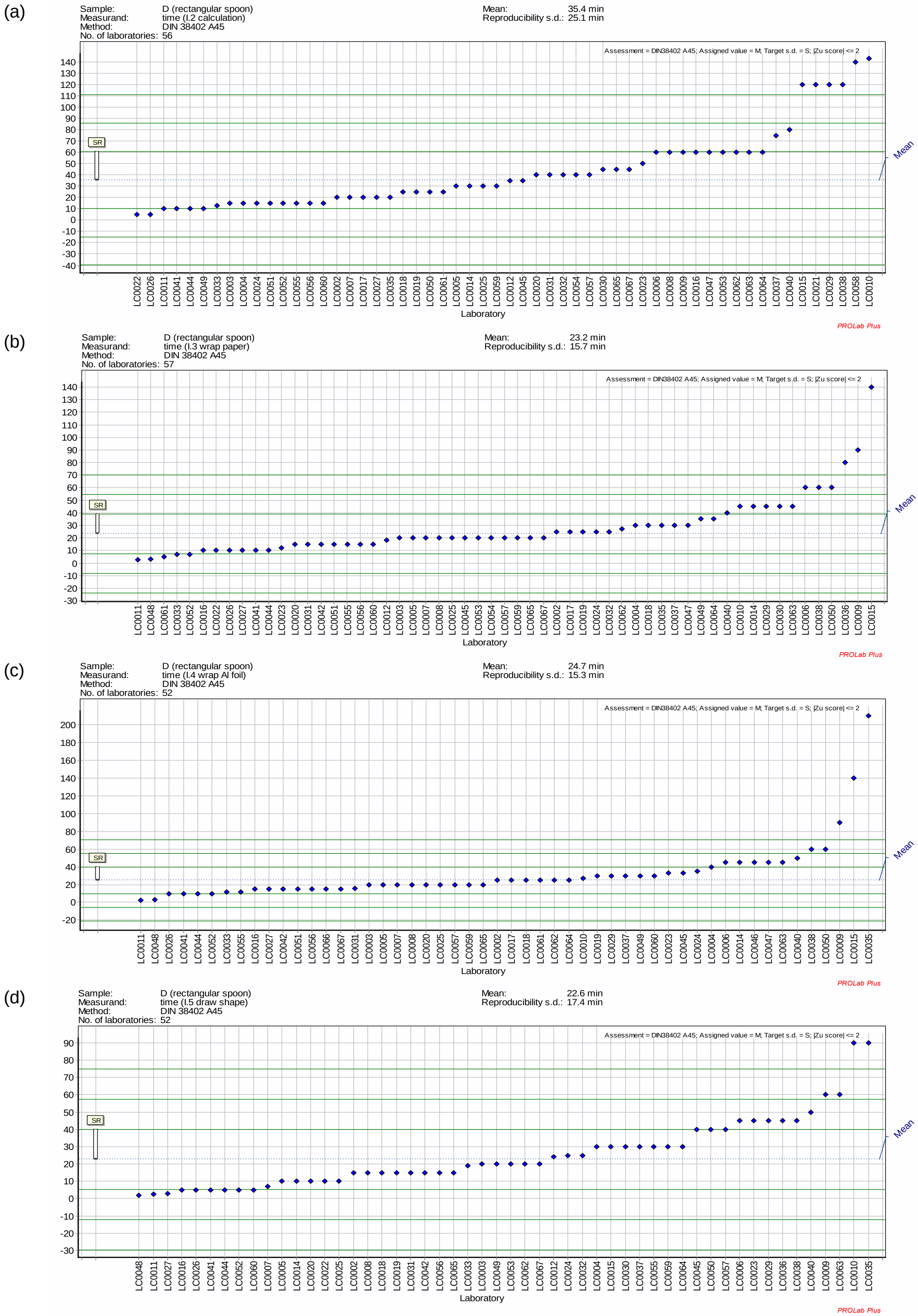


Figure 58 Time spent for the determination of the surface area of sample D by "calculation" (a), "wrapping in paper" (b), "wrapping in aluminium foil" (c) and "drawing the shape" (d)

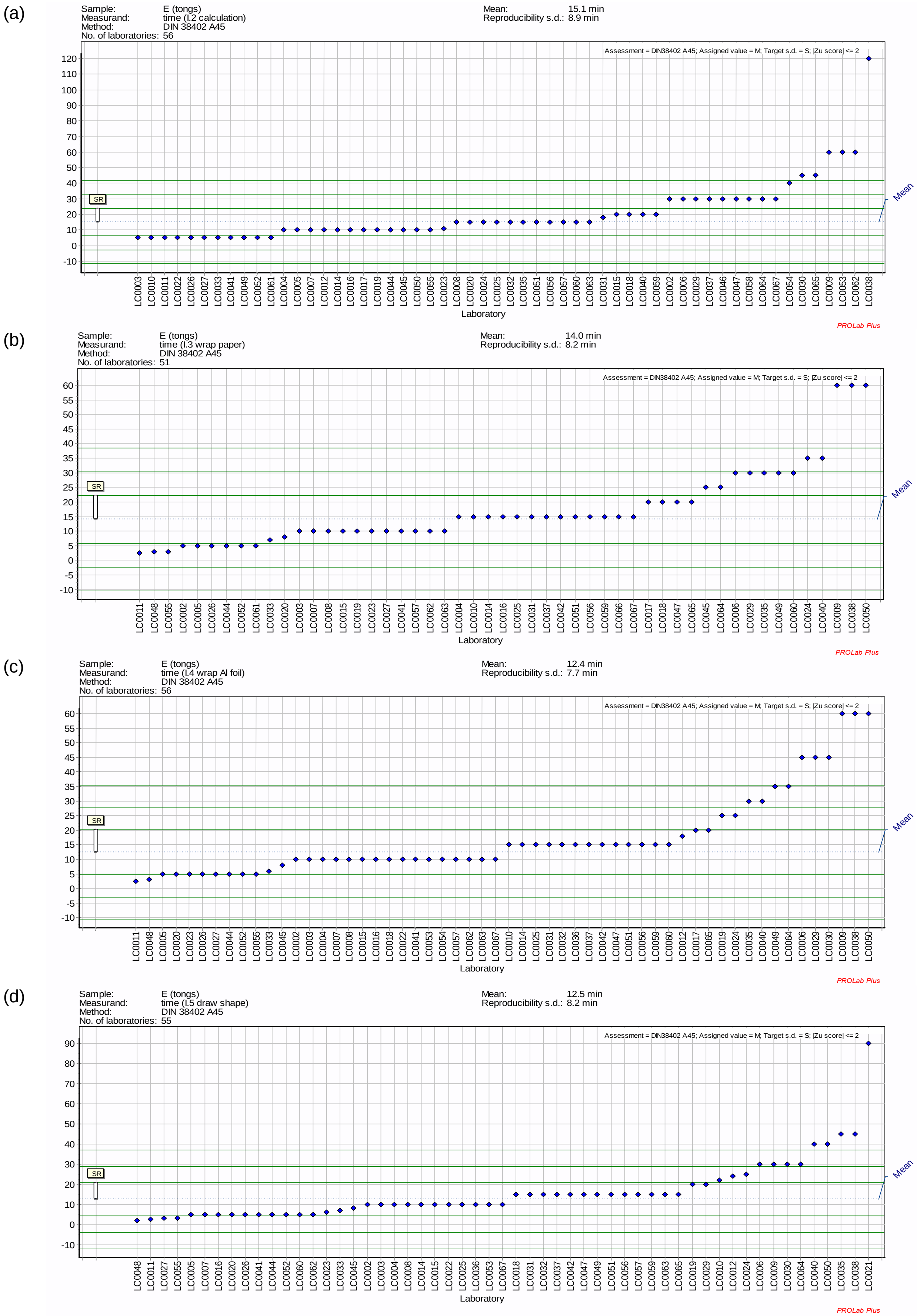


Figure 59 Time spent for the determination of the surface area of sample E by "calculation" (a), "wrapping in paper" (b), "wrapping in aluminium foil" (c) and "drawing the shape" (d)

14.4. Tabulated z_U-scores

14.4.1. Tabulated z_U-scores for the sample height with foreseeable food contact (H_f)

Table 30 z_u scores for the sample height with foreseeable food contact (H_f)

Laboratory	Sample A	Sample B	Sample C	Sample D	Sample E
LC0002	0.602	4.356	-2.572	-1.978	-7.154
LC0003	2	-22.277	-1.084	-1.567	-6.705
LC0004	0.029	-0.586	0.395	0.078	0.026
LC0005	0.029	-0.586	-0.34	-0.332	0.026
LC0006	-0.557	-0.586	-0.712	-0.332	-20.168
LC0007	19.218	-0.199	0.031	0.078	-5.808
LC0008	19.218	-0.199	-0.712	-0.332	-6.705
LC0009	-0.263	-0.586	-0.712	-0.332	-0.423
LC0010	-0.263	-0.199	-0.34	-0.744	-0.423
LC0011	0.029	-0.199	-0.712	-0.744	0.026
LC0012	0.602	0.184	0.031	-0.332	0.026
LC0013	18.932	-0.199	-0.712	-0.332	0.026
LC0014	-0.263	-0.974	-0.34	0.078	0.026
LC0015	0.029	0.564	0.395	0.885	0.908
LC0016	-0.263	-0.586	-0.712	-0.744	-10.295
LC0017	-0.263	-0.199	0.031	0.078	0.026
LC0018	18.646	-0.199	0.031	-0.332	0.026
LC0019	0.602	0.943	0.759	1.289	0.026
LC0020	0.029	-0.586	0.031	4.115	0.026
LC0021	0.029	0.943	0.395	0.482	-0.871
LC0022	0.029	-0.199	-0.34	0.885	0.467
LC0023	1.748	-7.558	-3.316	0.885	0.026
LC0024	-0.263	22.559	22.603	24.704	28.251
LC0025	-0.263	-0.586	0.031	0.078	0.026
LC0026	-0.263	-0.199	-0.34	-0.332	-23.31
LC0027	18.646	-0.199	0.031	-0.332	0.026
LC0028	14.922	-7.558	-5.919	-6.917	-15.232
LC0029	0.029	-8.333	-6.291	-1.155	-14.783
LC0030	0.316	0.184	1.487	0.482	0.026
LC0031	-0.851	0.184	-0.34	-0.332	0.026
LC0032	0.029	0.564	2.216	0.885	0.026
LC0033	4.325	-6.784	-5.175	-1.155	0.026
LC0035	0.316	1.322	2.216	2	0.026
LC0036	-0.263	0.184	-0.712	-0.332	0.026
LC0037	-0.263	-0.586	-0.34	-0.332	0.026
LC0038	-0.263	-0.199	-0.34	-0.332	-0.423
LC0040	0.029	0.184	0.031	0.078	-0.871
LC0041	18.646	1.322	0.031	0.482	0.026
LC0042	14.922	4.356	8.405	21.071	11.492
LC0044	-2.613	-10.27	-5.175	-8.151	-7.154
LC0045	-0.557	-0.199	-0.34	-0.332	-6.705
LC0046	19.791	-0.199	0.759	0.482	0.026
LC0047	0.316	0.184	0.395	0.078	0.026
LC0048	-2.613	-14.143	-0.34	-0.332	-42.158
LC0049	-0.557	-0.586	-0.34	0.078	-0.423
LC0050	19.505	1.701	2.216	1.289	0.026
LC0051	19.218	22.938	22.239	25.108	0.026
LC0052	19.791	-0.586	0.031	-0.332	-0.423
LC0053	19.791	0.184	0.031	0.482	0.026
LC0054	-0.557	0.184	-0.34	0.078	0.026
LC0055	0.316	0.184	0.395	-0.332	0.026
LC0056	-0.557	-0.586	-0.712	-0.332	0.026
LC0057	4.612	-6.009	-5.547	-2.39	-7.154
LC0058	0.889	0.943	2.58	1.289	0.026

Laboratory	Sample A	Sample B	Sample C	Sample D	Sample E
LC0059	2.607	4.356	2.944	2.904	2.672
LC0060	-0.263	-0.199	0.031	-0.332	0.026
LC0061	0.029	-0.199	0.031	0.482	-7.154
LC0062	-0.263	-0.199	-0.34	-0.332	0.026
LC0063	0.029	-0.586	0.031	0.078	0.026
LC0064	19.791	0.564	0.759	0.482	0.026
LC0065	19.505	-0.199	0.031	0.482	0.026
LC0066	5.185	0.564	0.031	0.078	0.026
LC0067	3.466	21.649	21.984	25.916	-25.239

14.4.2. Tabulated z_U-scores for the food contact surface area

Table 31 z_u scores for the food contact surface area of sample A

Lab code	calcu-lation	wrap paper	wrap Al foil	draw shape
LC0002	-0.823	-2.914	-0.896	-2.888
LC0003	-0.595	-0.711	-1.035	-0.644
LC0004	0.101	-0.007	-0.466	-0.86
LC0005	-1.089	-0.875	-1.155	-0.65
LC0006	0.054	-0.171	-0.687	0.383
LC0007	1.068	0.897	1.056	0.372
LC0008	1.395	0.577	0.372	1.571
LC0009	-0.605	0.06	-0.011	-0.898
LC0010	-0.214	-0.831	-0.131	0.114
LC0011	-1.209	0.761	0.804	-0.726
LC0012	0.246		-1.111	-0.219
LC0013	1.124	0.314	0.529	1.307
LC0014	0.124	-0.315	-0.46	-0.828
LC0015	-0.768	-0.224	-0.011	-0.409
LC0016	-1.377	0.023	0.434	-1.145
LC0017	-0.638	-0.74	-1.263	
LC0018	0.704	0.433	0.243	1.139
LC0019	1.4	0.634	0.17	1.106
LC0020	-0.904	-1.159	-1.541	-0.936
LC0021				-0.321
LC0022	0.928			1.504
LC0023	0.881	0.679	1.701	-0.409
LC0024	0.788	2.303	0.349	0.484
LC0025	-0.04	-0.108	-0.447	0.17
LC0026	-0.828	-1.034	0.591	-4.848
LC0027	0.601	0.568	0.832	0.585
LC0028	1.007	1.089	0.787	0.876
LC0029	-3.442	0.45	0.787	0.602
LC0030	-1.138		-1.218	-0.841
LC0031	-0.019	-0.301	-0.788	-0.714
LC0032	-1.393		-0.624	-0.492
LC0033	-1.04	-0.46	-0.359	-0.669
LC0035	0.232	0.019	-0.099	0.226
LC0036			-0.093	0.366
LC0037	-0.024	-0.214	-0.649	-0.27
LC0038	-0.698	-0.59	-0.188	-1.031
LC0040	-0.573	-0.759	-1.01	-0.898
LC0041	0.75	-2.215	-0.156	1.218
LC0042		1.783	2.56	0.815
LC0044	0.587	0.384	0.355	0.366
LC0045	-0.48	-0.339	-0.485	-0.004
LC0046				1.358
LC0047	0.297	0.093	0.164	0.344
LC0048	-1.491	-0.995	-1.496	-1.069
LC0049	0.928	0.532	0.187	0.361
LC0050	-2.627	-2.721	-3.899	-3.516
LC0051	1.965	2.365	2.425	3.8
LC0052	0.727	1.364	1.18	0.17
LC0053	0.675		1.123	1.632
LC0054	-0.1			-1.12
LC0055	-0.383	-0.841	-0.555	-1.335
LC0056	-0.616	-0.822	-0.668	-0.891
LC0057	-0.127	-0.417	0.125	-0.321
LC0058	-0.356			
LC0059	-0.817	0.667	0.523	-1.056
LC0060	0.788	-0.108	0.159	0.187
LC0061	-0.942	-0.648		
LC0062	0.162	-0.624	-0.422	-0.111
LC0063	-0.241	-0.19	-1.661	-0.27
LC0064	2.002	2.307	1.612	1.212
LC0065	0.774	1.311	1.045	1.548
LC0066		-2.987		
LC0067	0.291	0.613	0.721	-0.421

Table 32 z_u scores for the food contact surface area of sample B

Lab code	calcu-lation	wrap paper	wrap Al foil	draw shape
LC0002	0.297	-2.695	0.328	-1.664
LC0003	-0.397	-1.441	-1.706	-1.077
LC0004	-0.559	-0.36	0.158	-0.739
LC0005	-0.372	-0.88	-1.573	-0.694
LC0006	1.345	0.841	0.695	0.992
LC0007	0.09	-0.54	0.294	-1.408
LC0008	-0.618	-0.88	-0.75	0.642
LC0009	0.267	0.555	-0.021	0.125
LC0010	0.334	0.723	0.247	0.482
LC0011	-2.469	0.438	0.776	-0.987
LC0012	0.991		-1.04	-0.318
LC0013		-1	-1.228	0.845
LC0014	0.932	-0.18	-0.131	-0.859
LC0015	-0.932	-0.48	0.131	1.03
LC0016	1.235	0.432	0.111	-2.318
LC0017	-0.457	-0.593	-1.236	
LC0018	-0.847	-0.146	-1.635	0.17
LC0019	0.71	-0.507	-0.993	0.998
LC0020	1.368	0.953	1.625	0.763
LC0021				0.323
LC0022	-0.295			1.279
LC0023	0.482	1.704	2.189	-0.701
LC0024	1.523	2.214	1.048	0.852
LC0025	0.312	0.438	0.552	0.661
LC0026	-0.975	-1.214	0.742	-3.708
LC0027	0.098	-0.073	-0.068	-0.213
LC0028	-0.372	-0.3	-1.024	-0.22
LC0029	-2.792	0.449	0.016	0.285
LC0030	-0.975		-1.008	-1.273
LC0031	0.356	0.04	0.036	-0.506
LC0032	-0.643		-0.789	-0.634
LC0033	-0.244	-1.494	-0.946	-1.37
LC0035	0.393	0.118	0.118	0.718
LC0036			-0.272	0.616
LC0037	0.496	0.113	0.179	0.45
LC0038	0.238	-0.146	0.07	-0.98
LC0040	0.223	0.325	-0.162	0.533
LC0041	-1.111	-0.22	-1.682	-0.814
LC0042		0.976	1.985	-0.25
LC0044	0.083	0.55	0.926	1.279
LC0045	0.319	-0.126	-0.358	0.616
LC0046				0.68
LC0047	-0.355	0.124	1.034	1.381
LC0048	-1.705	-1.427	-2.058	-1.558
LC0049	1.043	0.46	0.471	0.329
LC0050	-3.828	-3.055	-3.947	-3.077
LC0051	1.316	1.317	2.82	1.642
LC0052	-0.015	0.454	0.152	-0.423
LC0053			0.05	0.527
LC0054	-0.499			-1.1
LC0055	-0.703	-0.4	0.417	-0.551
LC0056	-1.493	-1.301	-0.287	-0.995
LC0057	-0.745	-0.36	-0.279	0.068
LC0058	0.031			
LC0059	-1.077	0.639	1.34	-1.032
LC0060	0.482	0.875	1.048	0.38
LC0061	-1.323	-0.54		
LC0062	0.77	-0.073	-0.264	0.865
LC0063	-0.567	-0.473	-1.533	-0.566
LC0064	0.866	2.074	0.437	0.106
LC0065	0.688	0.55	0.118	0.673
LC0066		-3.062		
LC0067	1.663	1.855	2.052	1.523

Table 33 z_u scores for the food contact surface area of sample C

Lab code	calcu-lation	wrap paper	wrap Al foil	draw shape
LC0002	-0.158	-5.351	-1.992	-3.027
LC0003	0.204	0.164	0.34	1.176
LC0004	-0.075	-0.817	0.842	0.81
LC0005	-0.069	0.783	-0.192	0.11
LC0006	0.209	-1.412	-0.063	0.579
LC0007	-0.512	0.023	0.321	0.2
LC0008	-0.278	-1.132	-0.956	0.633
LC0009	-0.446	0.619	-0.106	-0.681
LC0010	-0.967	-0.162	-0.799	0.147
LC0011	-0.655	0.204	0.366	-0.139
LC0012	0.415		0.34	0.258
LC0013	0.273	-0.434	-0.985	1.279
LC0014	-0.224	-0.672	-0.678	-1.458
LC0015	-0.063	-1.395	1.005	-1.385
LC0016	0.564	-1.761	-0.971	-2.089
LC0017	-1.721	-1.14	-1.628	
LC0018	-0.332	-0.944	-0.342	0.76
LC0019	-0.17	-0.128	0.966	1.094
LC0020	0.606	0.141	-0.385	0.369
LC0021	-0.607			
LC0022	1.262		0.529	
LC0023	-0.907	0.869	1.853	0.6
LC0024	7.918	0.525	2.44	-0.442
LC0025	-0.751	-1.081	-0.871	0.126
LC0026	3.733	0.008	0.301	-3.946
LC0027	0.236	1.089	1.638	-1.253
LC0028	-0.158	0.822	-0.728	-0.065
LC0029	2.807	1.05	2.981	-1.038
LC0030	-0.685		-0.192	-0.696
LC0031	1.214	0.932	-0.177	-0.041
LC0032	0.161		1.025	0.229
LC0033	-0.512	-0.545	-0.177	0.077
LC0035	0.447	0.219	-0.32	0.674
LC0036			-1.228	0.353
LC0037	0.172	-0.128	-0.892	0.863
LC0038	0.188	0.188	-0.342	-0.373
LC0040	0.373	0.243	-0.32	0.682
LC0041	-1.38	0.094	-0.299	-1.497
LC0042		1.261	1.312	0.814
LC0044	3.585	0.11	-0.027	0.18
LC0045	0.103	-0.511	-0.092	0.509
LC0046		-1.514		
LC0047	0.199	0.023	-0.049	
LC0048	1.669	-0.91	-0.27	-1.531
LC0049	0.14	-0.885	-0.342	0.53
LC0050	-4.163	-7.053	-6.53	-3.232
LC0051	0.786	2.585	1.951	
LC0052	1.997	0.446	1.853	-1.038
LC0053			-0.778	-0.09
LC0054	-0.452		-0.006	
LC0055	-0.117	-0.562	-0.363	0.377
LC0056	0.013	0.031	-0.706	0.818
LC0057	-2.906	0.157	-0.092	-0.891
LC0058	7.31			
LC0059	-0.859	2.091	-0.142	0.369
LC0060	-1.864	0.125	-0.999	-1.429
LC0061	-2.032	-0.766	0.027	
LC0062	1.474	0.619	-1.285	1.164
LC0063	-1.469	-0.247	-1.885	-0.06
LC0064	0.548	0.564	1.488	-0.07
LC0065	0.389	0.102	0.366	-0.412
LC0066		-4.552		
LC0067	1.769	1.704	1.73	1.467

Table 34 z_u scores for the food contact surface area of sample D

Lab code	calcu-lation	wrap paper	wrap Al foil	draw shape
LC0002	0.05	-5.969	-1.127	-3.397
LC0003	0.126	-1.099	-0.665	0.098
LC0004	-0.676	-0.707	-0.343	0.897
LC0005	-0.231	0.356	-0.227	0.425
LC0006	0.258	-0.588	1.179	-0.166
LC0007	-1.022	-1.127	-0.293	0.774
LC0008	0.194	-0.35	-1.078	0.34
LC0009	-0.76	0.373	-0.401	0.714
LC0010	0.678	-0.633	-0.731	0.085
LC0011	-0.545	-0.624	0.357	-0.849
LC0012	1.266		-0.178	-0.368
LC0013	-0.147	0.321	-0.524	1.05
LC0014	0.163	-1.026	-1.243	-1.713
LC0015	-0.466	-1.017	-0.797	-1.453
LC0016	-1.619	-0.816	-0.698	-1.973
LC0017	0.131	-0.798	-0.97	
LC0018	-0.315	-0.77	-0.137	0.531
LC0019	0.619	0.492	-0.194	0.803
LC0020	2.441	1.183	1.048	0.875
LC0021	-0.849			
LC0022			-0.17	
LC0023	-0.204	1.508	2.945	1.326
LC0024	0.782	1.397	1.324	-0.598
LC0025	0.493	-0.542	-1.259	0.111
LC0026	-0.791	0.287	0.802	-4.555
LC0027	-0.535	-0.03	0.434	-1.516
LC0028	-0.886	1.303	-1.102	-0.333
LC0029	-2.196	0.193	1.731	-1.335
LC0030	-0.571		0.894	-0.976
LC0031	-0.335	-1.712	1.447	-0.431
LC0032	-0.618		1.071	0.574
LC0033	-1.169	1.678	0.188	0.289
LC0035	1.013	-0.405	-0.293	0.625
LC0036			0.011	0.986
LC0037	0.787	-1.163	-0.739	0.362
LC0038	0.701	0.168	-0.269	0.939
LC0040	0.674	-0.844	-0.937	0.429
LC0041	-2.872	1.303	0.94	-1.634
LC0042		3.043	2.891	0.323
LC0044	1.175	-0.359	-0.062	0.463
LC0045	-0.053	-0.569	0.165	0.064
LC0046		-1.246		
LC0047	0.375	-0.122	-0.17	
LC0048	-1.232	-1.017	-1.375	-1.595
LC0049	0.461	-0.734	-1.003	0.587
LC0050	-3.448	-7.915	-8.259	-3.574
LC0051	4.074	2.438	3.505	
LC0052	-0.147	-0.423	0.887	1.614
LC0053			-0.318	-0.166
LC0054	-0.325		-0.549	
LC0055	2.315	-0.579	1.171	0.26
LC0056	0.809	-0.058	-0.343	0.633
LC0057	-0.351	1.226	0.503	-0.284
LC0058	5.127			
LC0059	-0.833	0.953	0.695	0.162
LC0060	0.14	1.089	-0.194	-1.639
LC0061	0.235	-0.515	0.28	
LC0062	0.565	-0.725	-1.135	0.701
LC0063	-0.142	-0.944	-1.952	-0.466
LC0064	0.068	1.687	0.787	-0.549
LC0065	0.728	0.791	-0.269	-0.505
LC0066		-5.805		
LC0067	1.52	1.734	0.58	1.24

Table 35 z_u scores for the food contact surface area of sample E

Lab code	calcu-lation	wrap paper	wrap Al foil	draw shape
LC0002	-1.014	-2.801	-0.932	-2.575
LC0003	-1.06	-2.089	-1.746	-1.164
LC0004	0.265	-0.024	-0.118	0.399
LC0005	0.265	0.486	0.013	0.677
LC0006	-3.249	-3.452	-3.666	-2.164
LC0007	-1.542	-1.046	-1.377	-1.546
LC0008	-0.42	-1.086	-1.301	-0.076
LC0009	0.354	0.379	0.322	0.695
LC0010	0.735	0.717	0.079	0.208
LC0011	-0.875	0.353	0.272	-0.76
LC0012	1.123		0.723	-0.503
LC0013	0.654	-0.776	-0.714	0.121
LC0014	0.775	0.175	0.138	-0.804
LC0015	1.342	1.838	0.188	-1.046
LC0016	-2.72	-2.39	-1.689	-2.803
LC0017	0.492	-0.044	-0.61	
LC0018	0.378	0.486	2.077	0.103
LC0019	0.459	0.682	1.341	0.504
LC0020	0.532	0.299	0.33	1.004
LC0021				0.633
LC0022	0.759		1.767	0.146
LC0023	0.289	0.539	0.673	-0.356
LC0024	4.978	1.411	2.461	1.158
LC0025	0.637	0.699	0.397	0.757
LC0026	-3.119	-3.813	-3.448	-4.802
LC0027	0.103	0.166	0.781	0.368
LC0028	-1.746	-2.009	-2.966	-1.267
LC0029	-3.378	-2.229	-0.771	-2.134
LC0030	-1.079		0.054	-0.694
LC0031	4.354	0.753	-0.147	-0.311
LC0032	0.265		0.163	-0.525
LC0033	0.265	0.957	0.89	1.041
LC0035	-0.003	0.139	0.121	0.164
LC0036			0.414	0.936
LC0037	0.832	0.228	0.221	0.59
LC0038	-0.133	-0.334	-0.317	0.862
LC0040	0.03	-0.214	-0.364	0.263
LC0041	1.245	4.035	-0.128	0.269
LC0042		2.745	3.238	1.096
LC0044	-0.216	0.344	-0.071	-0.26
LC0045	-1.06	-0.946	-1.547	-0.591
LC0046	0.378			
LC0047	0.16	0.441	-0.014	0.51
LC0048	-6.439	-6.66	-6.353	-4.927
LC0049	0.402	-0.074	-0.336	0.312
LC0050	-5.196	-5.678	-4.726	-3.354
LC0051	-0.596	0.13	0.43	0.269
LC0052	0.208	0.13	1.04	0.417
LC0053			0.33	0.664
LC0054	0.305		0.74	
LC0055	0.475	0.255	-0.222	0.424
LC0056	-1.422	-3.723	-3.448	-2.428
LC0057	-1.125	-0.284	-0.402	0.041
LC0058	1.512			
LC0059	-1.069	1.206	1.09	-0.451
LC0060	0.613	0.904	0.288	-0.106
LC0061	-0.568	-0.024		
LC0062	0.807	0.557	0.113	1.331
LC0063	0.694	0.201	0.422	0.627
LC0064	0.645	0.326	-0.232	0.893
LC0065	0.718	0.13	-0.137	0.757
LC0066		-2.57		
LC0067	-3.388	-4.251	-4.055	-2.604

14.4.3. Tabulated z_U-scores for the envelope volume of sample A-E

Table 36 z_u scores for the envelope volume determined on a 2-cm-scale

Laboratory	Sample A	Sample B	Sample C	Sample D	Sample E
LC0002	0	-1.155	-0.41	-0.898	-1.707
LC0003	0.198	-1.925	-2.051	0	-1.067
LC0004	0	-1.348	-1.435	-1.347	-0.747
LC0005	0	0.818	0	0	-0.747
LC0006	0	0	0	-0.898	-1.387
LC0008	0.79	-1.348	-1.435	-1.347	-2.347
LC0009	0.099	-1.348	0	0	-1.493
LC0010			0	0.544	0
LC0011	1.383	0	0	0	0
LC0012	0	0	0	0	-0.747
LC0013	13.832	0	0	0.544	-1.493
LC0014	0	-1.348	-0.957	-0.898	-0.213
LC0016	0	-1.348	0	0.544	-2.347
LC0017	0	0	0	0.544	0
LC0018	5.138	0	0	-0.898	-0.747
LC0019	1.383	0	0	0.544	0
LC0021	6.916	-1.348	-1.435	-1.347	-1.493
LC0022	0	-1.348	-1.435	-0.898	-1.493
LC0023	-0.421	-1.893	-1.675	-0.93	-2.187
LC0025	0	0	0	0.544	-1.493
LC0026	0	0	0	0	-1.707
LC0027	2.964	0	0	0.544	-0.747
LC0028	0.593	-1.54	-0.41	0.233	-2.453
LC0029	-1.444	-2.214	-1.846	-1.539	-2.773
LC0031	0	-1.348	0	0.544	-1.493
LC0032	0	0	1.235	0	0
LC0033	0.198	-1.54	-1.641	-0.898	-1.493
LC0035	0	-1.348	-0.957	-0.898	-2.24
LC0036	0	-1.348	0	0	-1.493
LC0037	0	0	1.853	1.633	-0.747
LC0038	5.533	2.453	2.779	2.45	-1.493
LC0040	0	-1.348	0.618	0.544	-2.24
LC0041	4.545	1.635	1.853	3.033	2.268
LC0042	0.593	-1.348	0.971	-0.128	-0.427
LC0044	0	-1.733	-0.41	-1.155	-1.067
LC0045	0	-1.348	0	0.544	-2.347
LC0048	0	-1.733	0	-1.347	-2.773
LC0049	0	-2.246	0	0.544	-0.747
LC0050	0.79	-1.348	0	0.117	-2.24
LC0051	7.509	3.62	4.103	2.741	4.661
LC0052	2.964	0	0	0	-2.24
LC0054	1.383	0	0	0.544	-0.747
LC0055	0	-1.348	-1.435	-1.347	-1.493
LC0056	0	-1.54	-1.435	-1.347	-0.747
LC0057	0.198	-1.54	-0.41	0.544	-1.707
LC0058	0.198	-1.348	0.618	0.544	0
LC0059	1.778	0	0	0.544	-0.747
LC0060	1.383	0	12.044	0.544	0.851
LC0061	0	-1.348	-1.435	-1.347	-2.347
LC0062	0	0	0	0.544	-0.747
LC0064	4.94	1.635	2.118	3.033	4.395
LC0065	2.964	0	0	0.544	-0.747
LC0066	0.198	-1.572	-0.957	-0.898	-1.493

Table 37 z_u scores for the envelope volume determined on a 5-cm-scale

Laboratory	Sample A	Sample B	Sample C	Sample D	Sample E
LC0002	0	-1.414	0	0	0
LC0003	0	-1.885	-2.267	0	-1.308
LC0004	0	-1.414	0	0	0
LC0005	0	0	0	0	0
LC0006	0	0	0	0	-1.744
LC0008	1.528	-1.414	0	0	-1.308
LC0009	-0.218	-1.414	0	0	0
LC0010			0	0	0
LC0011	0	-1.414	0	0	0
LC0012	0	0	0	0	0
LC0013	9.165	0	0	0	-1.308
LC0014	0	0	0	0	0
LC0016	0	0	0	0	-1.395
LC0017	0	0	0	0	0
LC0018	5.346	0	0	0	0
LC0019	0	-1.414	0	0	0
LC0021	4.583	-1.414	0	0	-1.308
LC0022	0	-1.414	0	0	0
LC0023	0	-1.885	0	0	-1.291
LC0025	0	0	0	0	-1.308
LC0026	0	-1.414	0	0	-0.872
LC0027	1.528	-1.414	0	0	0
LC0028	0.764	0	0	0	-1.744
LC0029	-2.946	-2.639	0	-1.76	-2.442
LC0031	0	0	0	0	0
LC0032	-1.637	-1.414	0	0	0
LC0033	0.764	-1.414	0	0	0
LC0035	0	-1.414	0	0	-1.308
LC0036	0	-1.414	0	0	-1.308
LC0037	0	0	0	2.545	0
LC0038	4.583	3.604	3.336	5.09	0
LC0040	0	0	0	0	-1.308
LC0041	2.291	0	0	2.545	1.524
LC0042	0.764	0	0	0.848	0.508
LC0044	0	-0.943	0	0	0
LC0045	0	0	0	0	-1.308
LC0048	0	-1.885	0	0	-2.18
LC0049	0	-1.414	0	0	0
LC0050	-0.546	-1.414	0	0	-1.308
LC0051	3.208	0	5.003	2.545	0
LC0052	1.528	0	0	0	-1.308
LC0054	0	0	0	0	0
LC0055	0	-1.414	0	0	-1.308
LC0056	0	-1.414	0	0	0
LC0057	0.764	-1.414	0	0	-1.308
LC0058	0	0	0	0	0
LC0059	0	0	0	0	0
LC0060	0	0	8.339	0	0
LC0061	0	0	0	0	-1.308
LC0062	0	0	0	0	0
LC0064	3.055	0	1.668	2.545	1.524
LC0065	1.528	0	0	0	0

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Abstract
This report presents the results of the inter-laboratory comparison on the determination of the food contact surface area of kitchen utensils organised by the EURL-FCM, Ispra (Italy). Four different approaches were tested ("calculation", "wrapping in paper", wrapping in Al foil", "draw the shape"). A voluntary exercise comprised the determination of the envelope volume. The test materials were five different types of plastic kitchen utensils purchased on the Italian market. Results showed a satisfactory laboratory performance. For the determination of the surface area, the trueness and precision of the methods depended on the sample shape. "Calculation" generated accurate results for all sample types. "Drawing the shape" was most convenient and provided accurate results for flat samples that had a negligible thickness. For round-shaped samples, "wrapping in aluminium foil" was most convenient but it overestimated the surface area. "Wrapping in paper" generated accurate results for flat samples and simple geometric shapes. With respect to the effect of the four methods on the uncertainty of the final migration result, the obtained reproducibility standard deviations for all four approaches were acceptable.

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