# Results of Proficiency Test
# Specific Migration (fcm)
# September 2019

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. R.J. Starink
Correctors: ing. A.S. Noordman-de Neef & ing. C.M. Nijssen-Wester
Report: iis19P10SM

November 2019

**CONTENTS**

# 1 INTRODUCTION

During the contact of food with materials like kitchenware, molecules can migrate from the material to the food. Because of this, in many countries regulations are made to ensure food safety. The framework Regulation (EU) No. 10/2011 (lit. 4) applies to all food contact materials and describes a large number of requirements, e.g. limits for Overall Migration and Specific Migration limits for certain constituents. Article 11 (and Annex II) of this regulation describes the Specific Migration limit, expressed in mg/kg food or food simulant. For DEHP the Specific Migration limit is 1.5 mg/kg food and for Diallyl Phthalate is ”Not Detected”.

Since 2012, the Institute of Interlaboratory Studies (iis) organizes a proficiency test scheme for food contact materials every year. During the annual proficiency testing program 2019/2020, it was decided to continue the proficiency test for the determination of Specific Migration on food contact materials.
In the interlaboratory study of September 2019, 20 laboratories from 9 different countries participated (see appendix 4). In this report, the results of the 2019 proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

# 2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send one sample, a cup, labelled #19621, artificially fortified with Bis-2-Ethylhexyl Phthalate (DEHP) and Diallyl Phthalate (DAP) and to prescribe a number of test conditions (migration method, type of simulant, exposure time and temperature). Participants were also requested to report some intermediate test results and to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

## 2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls in the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant’s data. Feedback from the participants on the reported data is encouraged and customer’s satisfaction is measured on regular basis by sending out questionnaires.

## 2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report ‘iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation’ of June 2018 (iis protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

## 2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

## 2.4 SAMPLES

A batch of 50 pink colored polypropylene (food) cups containing a relevant concentration of DEHP and DAP was prepared by a third party. The subsamples were labelled #19621. The homogeneity was checked by determination of the Specific Migration of DEHP and DAP by an in-house test method on 8 stratified randomly selected cups. Migration conditions: 230 ml, 50% Ethanol, 60 min at 70°C.

| | DAP in mg/dm$^2$ | DEHP in mg/dm$^2$ |
|---|---|---|
| Sample #19621-1 | 0.2127 | 0.2077 |
| Sample #19621-2 | 0.2171 | 0.1931 |
| Sample #19621-3 | 0.2338 | 0.2120 |
| Sample #19621-4 | 0.2403 | 0.2026 |
| Sample #19621-5 | 0.2244 | 0.1989 |
| Sample #19621-6 | 0.2360 | 0.2156 |
| Sample #19621-7 | 0.2374 | 0.2171 |
| Sample #19621-8 | 0.2193 | 0.2055 |

Table 1: homogeneity test results on the subsamples #19621

From the above test results, the repeatabilities were calculated and compared to 0.3 times the corresponding reproducibility of the reference method in agreement with the procedure of ISO13528, Annex B2 in the next table.

| | DAP in mg/dm$^2$ | DEHP in mg/dm$^2$ |
|---|---|---|
| r(observed) | 0.030 | 0.023 |
| reference method | Horwitz | Horwitz |
| 0.3 x R (reference method) | 0.038 | 0.035 |

Table 2: evaluation of the repeatabilities of subsamples #19621

The calculated repeatabilities were in good agreement with 0.3 times the corresponding reproducibility of the reference method, estimated from the Horwitz equation. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample #19621 was sent on August 28, 2019.

### 2.5 ANALYSES

The participants were requested to determine 16 different Phthalates on sample #19621 using the prescribed test conditions (article filling, single use, 60 minutes at 70°C and 50% Ethanol as simulant). It was also requested to report if the laboratory was accredited for the requested components that were determined. Also, some analytical details were requested.

It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the results, but report as much significant figures as possible. It was also requested not to report ‘less than’ results, which are above the detection limit, because such test results can’t be used for meaningful statistical evaluations.

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the appropriate reference test methods that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The participating laboratories were also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

## 3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per determination in appendix 1 and 2 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1 or 2. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

### 3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report ‘iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation’ of June 2018 (iis-protocol, version 3.5).
For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as ‘<…’ or ‘>…’ were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either ‘unknown’, ‘OK’, ‘suspect’ or ‘not OK’. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

According to ISO5725 the original test results per determination were submitted to Dixon’s, Grubbs’ and/or Rosner’s outlier tests. Outliers are marked by D(0.01) for the Dixon’s test, by G(0.01) or DG(0.01) for the Grubbs’ test and by R(0.01) for the Rosner’s test. Stragglers are marked by D(0.05) for the Dixon’s test, by G(0.05) or DG(0.05) for the Grubbs’ test and by R(0.05) for the Rosner’s test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.
For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT the criterion of ISO13528, paragraph 9.2.1 was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

### 3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis.

The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. The Kernel Density Graph is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve was projected over the Kernel Density Graph for reference.

### 3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency tests could be used.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated according to:

$z_{(target)}$ = (test result - average of PT) / target standard deviation

The $z_{(target)}$ scores are listed in the test result tables in appendix 1.
Absolute values for z<2 are very common and absolute values for z>3 are very rare.

The usual interpretation of z-scores is as follows:

| | |
|---|---|
| \| z \| < 1 | good |
| 1 < \| z \| < 2 | satisfactory |
| 2 < \| z \| < 3 | questionable |
| 3 < \| z \| | unsatisfactory |

## 4 EVALUATION

In this interlaboratory study, no problems were encountered with the dispatch of the samples. One participant reported test results after the final reporting date and two other participants did not report any test result at all. Not all laboratories were able to report all components requested.
Finally, in total over 54 (intermediate) test results were reported of which 26 test results in mg/dm$^2$. Observed were 3 outlying test results, which is 11.5% of the statistically evaluated numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

The two data sets proved to have a normal Gaussian distribution.

### 4.1 EVALUATION PER COMPONENT

The determination of Specific Migration requires additional analytical testing following the migration step, while the determination of the Overall (also called global, or total) Migration requires weighing as only quantitative analytical technique. This makes the Specific Migration of Phthalates from food contact materials more difficult than determination of the overall migration.

In the past, iis has found that the Overall and Specific Migration methods, limits and calculations are mixed up and used inappropriately by participants. So iis issued a White Paper on this subject in February 2018 (White Paper on the determination of Overall and Specific Migration on food contact materials, lit. 19) to help participants understand the differences between the two methods, the units used for reporting and the regulated limits. The test results of the Specific Migration reported in mg/dm$^2$ were used for the statistical evaluation.

For the determination of Specific Migration, several standardized test methods exist. The most relevant literature is test method EN13130 part 1. Method EN13130-1 describes how the Specific Migration test should be performed. Regretfully no reference test method is available with precision requirements for the migration of Phthalates from food contact materials. Therefore, it was decided to estimate the target reproducibilities from the Horwitz equation.

DEHP: This determination may be problematic. Two statistical outliers were observed and three other test results were excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the estimated reproducibility using the Horwitz equation.

DAP: This determination may be problematic. No statistical outliers were observed, but one test results was excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the estimated reproducibility using the Horwitz equation.

Other Phthalates: The majority of participants agreed on a concentration near or below the limit of detection for the other requested Phthalates, see appendix 2.

### 4.2 PERFORMANCE EVALUATION OF THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility of the relevant reference method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average test result, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from the estimated target reproducibility are presented in the next tables.

| Component | unit | n | average | 2.8 * sd | R (target) |
|---|---|---|---|---|---|
| DEHP | mg/dm$^2$ | 12 | 0.337 | 0.320 | 0.178 |
| DAP | mg/dm$^2$ | 8 | 0.200 | 0.133 | 0.114 |

Table 3: Reproducibilities of components on sample #19621

Without further statistical calculations, it can be concluded that for Phthalates present in the sample there is not a good compliance of the group of laboratories with the relevant target reproducibility (see for discussion paragraph 4.1 and 5).

### 4.3 COMPARISON OF PROFICIENCY TEST OF SEPTEMBER 2019 TO PREVIOUS PROFICIENCY TESTS

The evolution of the uncertainty for Specific Migration in mg/dm$^2$ as observed in this proficiency scheme and the comparison with the findings in previous rounds is listed in table 4.

| Year | Components | Type of migration | Observed RSD% | Target RSD% | Concentration range mg/dm$^2$ |
|---|---|---|---|---|---|
| 2012 | Formaldehyde | article filling | 41 – 47 | 14 – 20 | 0.2 – 3 |
| 2013 | Formaldehyde | article filling | 41 – 61 | 14 – 20 | 0.2 – 3 |
| 2014 | Bisphenol-A | total immersion | 44 – 52 | 14 – 20 | 0.2 – 3 |
| 2015 | DEHP | total immersion | 34 – 40 | 14 – 20 | 0.2 – 3 |
| 2016 | Metals | total immersion | 29 – 30 | 14 – 20 | 0.2 – 3 |
| 2017 | Bisphenol-A | article filling | 33 – 50 | 20 – 33 | 0.009 – 0.2 |
| 2018 | Metals | article filling | 21 – 35 | 17 – 38 | 0.003 – 0.6 |
| 2019 | DEHP/DAP | article filling | 24 – 34 | 19 – 20 | 0.20 – 0.34 |

Table 4: comparison of the uncertainties in % for Specific Migration in the present and previous PTs

From the above table, it is clear that the performance of this PT is in line with the previous PTs. It also shows that the strict requirements, estimated from the Horwitz equation will not be met with higher concentrations.

### 4.4 EVALUATION OF THE ANALYTICAL DETAILS

The reported analytical details that were used by the participants are listed in appendix 3. About 75% of the reporting laboratories are accredited for the determination of the Specific Migration of Phthalates.

About 25% of the reporting participants mentioned to have used test method EN13130-1 for the Specific Migration and 30% of the reporting participants mentioned to have used an ‘in house’ method. One participant reported to have used EN1186 (which is an Overall migration method), one reported to have used CPSC-CH-C1001-09.4 (which is a migration for Child safety) and one other used ISO18856 (which is for Phthalates in water) and five other participants did not mention a test method.

From the intermediate results: concentration in mg/L, surface and used volume of simulant, the test results in mg/dm$^2$ were calculated for both added Phthalates. It appeared that for three laboratories the reported test values were different than the iis calculated results (two for DEHP and one for DAP) see appendices 1 and 3.

One laboratory only reported results in mg/L with the comment “for food contact plastic articles when they are fillable the Specific Migration test result shall be expressed as mg/kg (mg/L) rather than mg/dm$^2$”. This is remarkable as test method EN13130-1, which is considered to be the official test method, calculates the Specific Migration in mg/dm$^2$. Also, when using article filling (variable surface to volume ratio), the results in mg/L simulant is not the same as the results in mg/kg food (see White Paper on the determination of Overall and Specific Migration on food contact materials, lit. 19).

Ten participants (66%) reported not to clean the sample before the determination of the Specific Migration and five participants (33%) reported to clean the cup. One of these participants reported to clean the cup with lint-free cloth. The four other participants reported to clean the cup with water, which is not in line with test method EN13130-1 paragraph 15.5.
All participants preheated the simulant solution to 70°C.

Thirteen participants (81%) used an oven for the test. Two participants (13%) reported to have used an incubator and one used a water bath.
About eight participants (50%) used Aluminum seal to close the cup. Six participants (38%) used a watch glass (or petri dish). One participant (7%) used a plastic film.

## 5 DISCUSSION

The limits for specific migration for DEHP and DAP are mentioned in mg/kg food. As it is mentioned in EN13130-1, the limits expressed in mg/kg shall be divided by the conventional conversion factor of 6 in order to express them in mg/dm$^2$, see table 5.

| Component | Specific Migration Limit in mg/kg | Specific Migration Limit in mg/dm$^2$ |
|---|---|---|
| DEHP | 1.5 | 0.25 |
| DAP | Not Detected | Not Detected |

Table 5: Specific Migration maximum limits according to 10/2011/EU

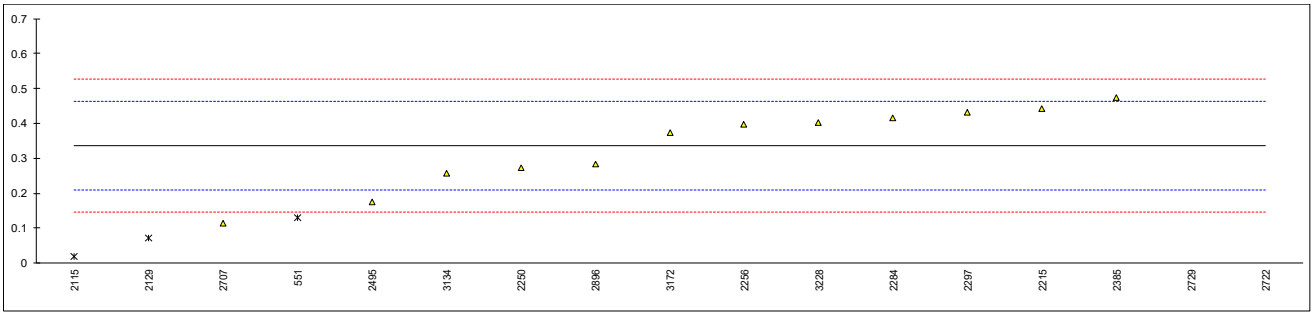
Twelve laboratories would reject the sample for DEHP, while five would accept the sample. As the limit for DAP is specified as Not Detected, it is difficult to mention the number of laboratories that would reject the sample. Nine participants provided a numerical test result and based on this detected test result the sample would have been rejected.

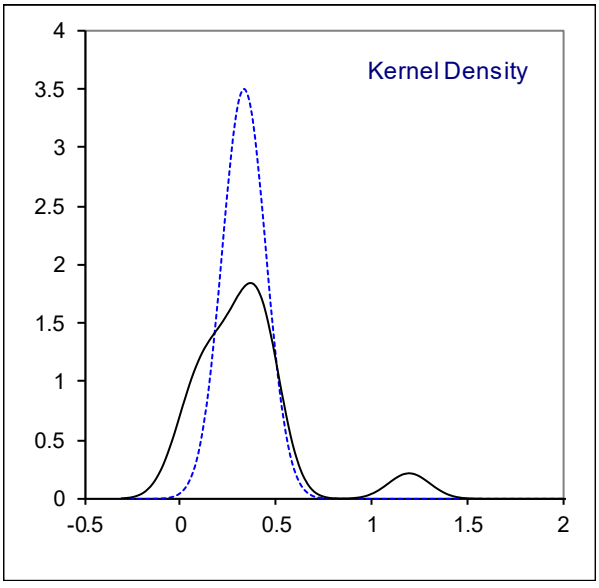
Each laboratory should evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and the quality of the analytical results.

**APPENDIX 1**

Determination of Specific Migration of DEHP - Bis-2-ethylhexyl Phthalate (CAS No.117-81-7) on sample #19621; results in mg/dm$^2$ per contact surface

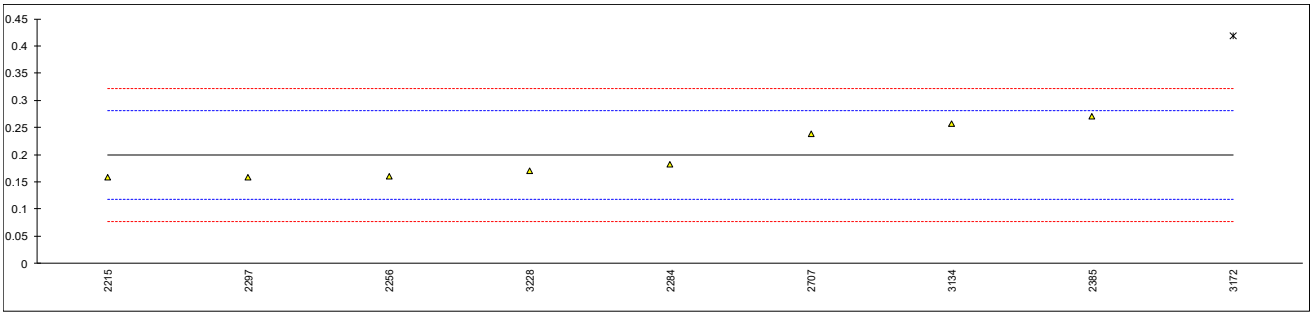
| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 362 | | ----- | | ----- | |
| 551 | In-House | 0.1311 | ex,E | -3.24 | Test result excluded, iis calculated a different mg/dm$^2$ content: 0.1387 |
| 2115 | EN13130-1/EN1186-9 | 0.02 | G(0.01) | -4.99 | |
| 2129 | EN13130-1 | 0.0725 | ex | -4.16 | Test result excluded, deviating surface/volume ratio. |
| 2215 | | 0.443 | | 1.67 | |
| 2250 | DIN EN 1186 | 0.273 | | -1.00 | |
| 2256 | EN13130-1 | 0.397 | | 0.95 | |
| 2284 | | 0.415 | | 1.23 | |
| 2297 | | 0.431 | | 1.49 | |
| 2385 | In-House | 0.475 | | 2.18 | |
| 2495 | CPSC-CH-C1001-09.4 | 0.1751 | | -2.55 | |
| 2573 | | ----- | | ----- | |
| 2707 | In-House | 0.115 | | -3.49 | |
| 2722 | EN13130-1 | 5.22 | ex,E | 76.94 | Test result excluded, iis calculated a different mg/dm$^2$ content: 5.7470 |
| 2729 | In-House | 1.2 | G(0.01) | 13.60 | |
| 2896 | In-House | 0.2843 | | -0.83 | |
| 3134 | | 0.256 | | -1.27 | |
| 3163 | | ----- | | ----- | |
| 3172 | | 0.3745 | | 0.59 | |
| 3228 | ISO18856 | 0.402 | | 1.03 | |
| | | | | | |
| | normality | OK | | | |
| | n | 12 | | | |
| | outliers | 2 (+3ex) | | | |
| | mean (n) | 0.3367 | RSD = | 34% | |
| | st.dev. (n) | 0.11416 | | | |
| | R(calc.) | 0.3197 | | | |
| | st.dev.(Horwitz) | 0.06347 | | | |
| | R(Horwitz) | 0.1777 | | | |

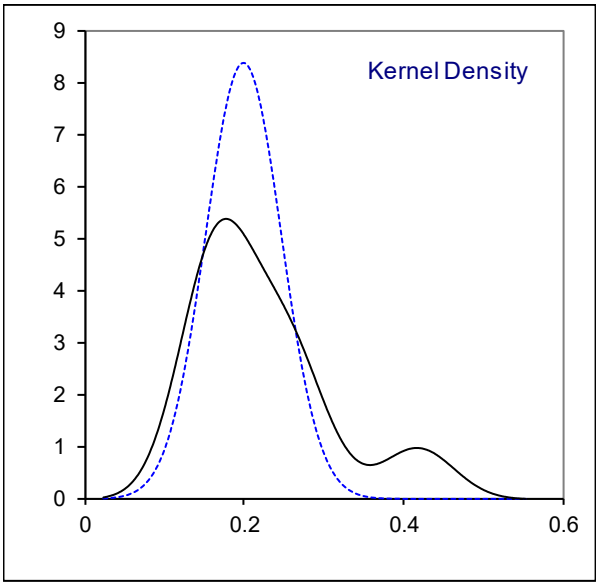


Determination of Specific Migration of DAP – Diallyl Phthalate (CAS No.131-17-9) on sample #19621; results in mg/dm$^2$ per contact surface

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 362 | | ----- | | ----- | |
| 551 | | ----- | | ----- | |
| 2115 | | ----- | | ----- | |
| 2129 | | ----- | | ----- | |
| 2215 | | 0.159 | | -1.00 | |
| 2250 | | ----- | | ----- | |
| 2256 | EN13130-1 | 0.16 | | -0.97 | |
| 2284 | | 0.183 | | -0.41 | |
| 2297 | | 0.159 | | -1.00 | |
| 2385 | In-House | 0.271 | | 1.75 | |
| 2495 | | ----- | | ----- | |
| 2573 | | ----- | | ----- | |
| 2707 | In-House | 0.238 | | 0.94 | |
| 2722 | | ----- | | ----- | |
| 2729 | | ----- | | ----- | |
| 2896 | | ----- | | ----- | |
| 3134 | | 0.257 | | 1.41 | |
| 3163 | | ----- | | ----- | |
| 3172 | | 0.419 | C,ex,E | 5.39 | First reported 0.465, test result excluded, different mg/dm$^2$: 0.4650 |
| 3228 | ISO18856 | 0.170 | | -0.73 | |
| | | | | | |
| | normality | OK | | | |
| | n | 8 | | | |
| | outliers | 0 (+1ex) | | | |
| | mean (n) | 0.1996 | RSD = | 24% | |
| | st.dev. (n) | 0.04764 | | | |
| | R(calc.) | 0.1334 | | | |
| | st.dev.(Horwitz) | 0.04071 | | | |
| | R(Horwitz) | 0.1140 | | | |



**APPENDIX 2**

Determination of Specific Migration of Other Phthalates on sample #19621; results in mg/dm$^2$ per contact surface

**Abbreviations of components:**

BBP = Benzylbutylphthalate
DBP = Dibutylphthalate
DIDP = Diisodecylphthalate
DINP = Diisononylphthalate
DNOP = Di-n-octylphthalate
DCHP = Dicyclohexylphthalate
DEP = Diethylphthalate
DMP = Dimethylphthalate
DNHP = Di-n-hexylphthalate
DIBP = Diisobutylphthalate
DPHP = Di(2-propylheptyl) phtalate
DNPP = Di-n- pentylphthalate
DUP = Diundecylphthalate
DPRP = Diproylphthalate

| Lab | BBP | DBP | DIDP | DINP | DNOP | DCHP | DEP |
|---|---|---|---|---|---|---|---|
| 362 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 551 | N.D. | N.D. | N.D. | ----- | ----- | ----- | ----- |
| 2115 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2129 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2215 | ND | ND | ND | ND | ND | ND | ND |
| 2250 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2256 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2284 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2297 | nd | nd | nd | nd | nd | nd | nd |
| 2385 | <0,001 | <0,001 | <0,001 | <0,001 | <0,001 | <0,001 | <0,001 |
| 2495 | <0.02 | <0.02 | <0.02 | <0.02 | <0.02 | <0.02 | <0.02 |
| 2573 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2707 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2722 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2729 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2896 | 0 | 0 | ----- | ----- | 0 | 0 | 0 |
| 3134 | N.D. | N.D. | N.D. | N.D. | N.D. | N.A. | N.D. |
| 3163 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3172 | < 0.01 | < 0.01 | < 0.01 | < 0.01 | < 0.01 | < 0.01 | < 0.01 |
| 3228 | n.d. | n.d. | n.d. | n.d. | n.a. | n.a. | n.a. |

| Lab | DMP | DNHP | DIBP | DPHP | DNPP | DUP | DPRP |
|---|---|---|---|---|---|---|---|
| 362 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 551 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2115 | ----- | ----- | 0.004 | ----- | ----- | ----- | ----- |
| 2129 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2215 | ND | ND | ND | ND | ND | ND | ND |
| 2250 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2256 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2284 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2297 | nd | nd | nd | nd | nd | nd | nd |
| 2385 | <0,001 | <0,001 | <0,001 | <0,001 | <0,001 | <0,001 | <0,001 |
| 2495 | <0.02 | <0.02 | <0.02 | ----- | <0.02 | ----- | <0.02 |
| 2573 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2707 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2722 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2729 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2896 | 0 | 0 | 0 | ----- | ----- | ----- | ----- |
| 3134 | N.D. | N.A. | N.D. | N.A. | N.A. | N.A. | N.A. |
| 3163 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3172 | < 0.01 | < 0.01 | < 0.01 | < 0.01 | < 0.01 | < 0.01 | < 0.01 |
| 3228 | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. | n.a. |

## APPENDIX 3 ANALYTICAL DETAILS

### Details on final concentration, surface area and volume of simulant reported on DEHP

| lab | surface area in $dm^2$ | volume simulant in ml | Surface to volume ration in $dm^2/100ml$ | final conc. in simulant in mg/l | reported Specific Migration in $mg/dm^2$ | iis calculated Specific Migration in $mg/dm^2$ | Difference absolute |
|---|---|---|---|---|---|---|---|
| 362 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 551 | 1.56 | 240 | 0.65 | 0.90125 | 0.1311 | 0.1387 | 0.0076 |
| 2115 | 1.58 | 200 | 0.79 | 0.15 | 0.02 | 0.0190 | -0.0010 |
| 2129 | 2.04 | 200 | 1.02 | 0.74 | 0.0725 | 0.0726 | 0.0001 |
| 2215 | 1.5 | 200 | 0.75 | 3.315 | 0.443 | 0.4420 | -0.0010 |
| 2250 | 1.6748 | 220 | 0.76127 | 2.08 | 0.273 | 0.2732 | 0.0002 |
| 2256 | 1.49 | 200 | 0.745 | 2.96 | 0.397 | 0.3973 | 0.0003 |
| 2284 | 1.67 | 250 | 0.668 | 2.775 | 0.415 | 0.4154 | 0.0004 |
| 2297 | 1.846 | 250 | 0.7384 | 3.18 | 0.431 | 0.4307 | -0.0003 |
| 2385 | 1.75 | 240 | 0.72917 | 3.47 | 0.475 | 0.4759 | 0.0009 |
| 2495 | 1.371 | 200 | 0.6855 | 1.1993 | 0.1751 | 0.1750 | -0.0002 |
| 2573 | 1.67 | 240 | 0.69583 | 3.214 | ----- | 0.4619 | ----- |
| 2707 | 1.41 | 200 | 0.705 | 0.8108 | 0.115 | 0.1150 | 0.0000 |
| 2722 | 1.63 | 220 | 0.74091 | 42.58 | 5.22 | 5.7470 | 0.5270 |
| 2729 | 1.25 | 250 | 0.5 | 5.9671 | 1.2 | 1.1934 | -0.0066 |
| 2896 | 1.86 | 250 | 0.744 | 2.115 | 0.2843 | 0.2843 | 0.0000 |
| 3134 | 1.541 | 220 | 0.70045 | 1.793 | 0.256 | 0.2560 | 0.0000 |
| 3163 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3172 | 1.6975 | 250 | 0.679 | 2.5428 | 0.3745 | 0.3745 | 0.0000 |
| 3228 | 1.71 | 250 | 0.684 | 2.75 | 0.402 | 0.4021 | 0.0001 |

### Details on final concentration, surface area and volume of simulant reported on DAP

| lab | surface area in $dm^2$ | volume simulant in ml | Surface to volume ration in $dm^2/100ml$ | final conc. in simulant in mg/l | reported Specific Migration in $mg/dm^2$ | iis calculated Specific Migration in $mg/dm^2$ | Difference absolute |
|---|---|---|---|---|---|---|---|
| 362 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 551 | 1.56 | 240 | 0.65 | ----- | ----- | ----- | ----- |
| 2115 | 1.58 | 200 | 0.79 | ----- | ----- | ----- | ----- |
| 2129 | 2.04 | 200 | 1.02 | ----- | ----- | ----- | ----- |
| 2215 | 1.5 | 200 | 0.75 | 1.188 | 0.159 | 0.1584 | -0.0006 |
| 2250 | 1.6748 | 220 | 0.76127 | ----- | ----- | ----- | ----- |
| 2256 | 1.49 | 200 | 0.745 | 1.19 | 0.16 | 0.1597 | -0.0003 |
| 2284 | 1.67 | 250 | 0.668 | 1.224 | 0.183 | 0.1832 | 0.0002 |
| 2297 | 1.846 | 250 | 0.7384 | 1.12 | 0.159 | 0.1517 | -0.0073 |
| 2385 | 1.75 | 240 | 0.72917 | 1.98 | 0.271 | 0.2715 | 0.0005 |
| 2495 | 1.371 | 200 | 0.6855 | ----- | ----- | ----- | ----- |
| 2573 | 1.67 | 240 | 0.69583 | 1.336 | ----- | 0.1920 | ----- |
| 2707 | 1.41 | 200 | 0.705 | 1.6758 | 0.238 | 0.2377 | -0.0003 |
| 2722 | 1.63 | 220 | 0.74091 | ----- | ----- | ----- | ----- |
| 2729 | 1.25 | 250 | 0.5 | ----- | ----- | ----- | ----- |
| 2896 | 1.86 | 250 | 0.744 | ----- | ----- | ----- | ----- |
| 3134 | 1.541 | 220 | 0.70045 | 1.802 | 0.257 | 0.2573 | 0.0003 |
| 3163 | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3172 | 1.6975 | 250 | 0.679 | 3.1574 | 0.419 | 0.4650 | 0.0460 |
| 3228 | 1.71 | 250 | 0.684 | 1.16 | 0.170 | 0.1696 | -0.0004 |

## ANALYTICAL DETAILS - continued -

| lab | accredited acc. ISO/IEC17025 | sample cleaned prior to the migration step | simulant preheated to 70°C | Equipment used | Sample sealed during test |
|---|---|---|---|---|---|
| 362 | --- | --- | --- | --- | --- |
| 551 | Yes | No | Yes | Oven | Yes, with Aluminum Seal |
| 2115 | --- | --- | --- | --- | --- |
| 2129 | Yes | No | Yes | Oven | Yes, with Aluminum Seal |
| 2215 | No | No | Yes | Water bath | Yes, with Aluminum Seal |
| 2250 | Yes | No | Yes | Oven | Yes, with plastic film |
| 2256 | Yes | No | Yes | Oven | Yes, with Aluminum Seal |
| 2284 | Yes | No | Yes | Oven | Yes, with Aluminum Seal |
| 2297 | No | Yes, with water | Yes | Incubator | Yes, with Aluminum Seal |
| 2385 | Yes | Yes, with water | Yes | Oven | Yes, with watch glass |
| 2495 | Yes | --- | --- | Oven | Yes, with watch glass |
| 2573 | Yes | No | Yes | Oven | Yes, with glass |
| 2707 | Yes | Yes, with (not specified) | Yes | Oven | Yes, with Aluminum Seal |
| 2722 | No | No | Yes | Incubator | Yes, with watch glass |
| 2729 | --- | --- | --- | --- | --- |
| 2896 | Yes | Yes, with water | Yes | Oven | Yes, with glass petri dish |
| 3134 | No | No | Yes | Oven | Yes, with glass |
| 3163 | --- | --- | --- | --- | --- |
| 3172 | Yes | No | Yes | Oven | No |
| 3228 | Yes | Yes, with lint-free cloth | Yes | Oven | Yes, with Aluminum Seal |

## APPENDIX 4

### Number of participating laboratories per country

1 lab in BRAZIL

1 lab in BULGARIA

3 labs in GERMANY

1 lab in GREECE

1 lab in HONG KONG

4 labs in ITALY

7 labs in P.R. of CHINA

1 lab in SOUTH KOREA

1 lab in THE NETHERLANDS

**APPENDIX 5**

**Abbreviations**:

C = final test result after checking of first reported suspect test result
D(0.01) = outlier in Dixon's outlier test
D(0.05) = straggler in Dixon's outlier test
G(0.01) = outlier in Grubbs' outlier test
G(0.05) = straggler in Grubbs' outlier test
DG(0.01) = outlier in Double Grubbs' outlier test
DG(0.05) = straggler in Double Grubbs' outlier test
R(0.01) = outlier in Rosner's outlier test
R(0.05) = straggler in Rosner's outlier test
E = possibly an error in calculation
W = test result withdrawn on request of the participant
ex = test result excluded from statistical evaluation
n.a. = not applicable
n.e. = not evaluated
n.d. = not detected
fr. = first reported

**Literature**:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2 EN13130-1 Materials and articles in contact with foodstuffs – Plastics substances subject to limitation
3 Commission regulation (EU) No 1935/2004 of 27 October 2004, on materials and articles intended to come into contact with food
4 Commission regulation (EU) No 10/2011 of 14 January 2011 on plastic materials and articles intended to come into contact with food
5 ASTM E178:02
6 ASTM E1301:03
7 ISO 5725:86
8 ISO 5725, parts 1-6, 1994
9 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
10 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
11 IP 367/96
12 DIN 38402 T41/42
13 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
14 J.N. Miller, Analyst, 118, 455, (1993)
15 Analytical Methods Committee Technical Brief, No 4, January 2001
16 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364 (2002)
17 R.G. Visser, Accred Qual Assur, 14:29-34 (2009)
18 Bernard Rosner, Technometrics, 25(2), pp. 165-172, (1983)
19 White Paper on the determinations of Overall and Specific Migration on food contact materials, February 2018, www.iisnl.com/pdf/WhitePaper.pdf (or www.iisnl.com on News and Report page in News)