Institute for
Interlaboratory Studies

Results of Proficiency Test
Specific Migration on
Food Contact Materials
October 2024

Preview

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

During the contact of materials with food, molecules can migrate from the food contact 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. 13 and lit. 14) 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. The EU10/2011 regulation was amended regarding Metals in September 2020 with EU 2020/1245 (lit. 15) and was amended regarding Phthalates in July 2023 with EU 2023/1442 (lit 16).

Since 2012 the Institute of Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of Specific Migration on Food Contact Materials every year. During the annual proficiency testing program of 2024 it was decided to continue the proficiency test for the determination of Specific Migration on Food Contact Materials.

In this interlaboratory study 35 laboratories in 19 countries registered for participation, see appendix 5 for the number of participants per country. In this report the results of the Specific Migration proficiency test are presented and discussed.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to a laboratory that has performed the tests in accordance with for ISO/IEC17043 relevant requirements of ISO/IEC17025.
It was decided to send two different samples. The first sample was a green melamine bowl labelled #24720 for the determination of migratable Formaldehyde. The second sample was a beige polypropylene cup labelled #24721 for the determination of migratable metals.
The participants were requested 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 under 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 Organization, Statistics and Evaluation' of October 2024 (iis-protocol, version 4.0). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

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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

For the first sample a batch of green Melamine containing a detectable level of Formaldehyde was obtained from the local market. The subsamples were labelled #24720. The homogeneity of the subsamples was checked by determination of the Specific Migration of Formaldehyde using an in-house test method on 8 stratified randomly selected subsamples. Migration conditions: article filling, 3% M/V Acetic Acid, 120 minutes at 70 °C.

	Formaldehyde in mg/dm^2
sample #24720-1	0.517
sample #24720-2	0.495
sample #24720-3	0.495
sample #24720-4	0.522
sample #24720-5	0.509
sample #24720-6	0.390 (D0.01)
sample #24720-7	0.476
sample #24720-8	0.478

Table 1: homogeneity test results of subsamples #24720

Sample 6 was marked as a Dixon statistical outlier.

From the above test results the repeatability was calculated and compared with 0.3 times the estimated reproducibility calculated with the Horwitz equation in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Formaldehyde in mg/dm^2
r (observed)	0.051
reference method	Horwitz
0.3 x R (reference method)	0.074

Table 2: evaluation of the repeatability of subsamples #24720

The calculated repeatability is in agreement with 0.3 times the estimated reproducibility calculated with the Horwitz equation. Therefore, homogeneity of the subsamples was assumed.

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For the second sample a batch of beige polypropylene cups containing a detectable level of some metals was prepared by a third party. The subsamples were labelled #24721.
The homogeneity of the subsamples was checked by determination of the Specific Migration of Cobalt and Lithium using an in house test method on 8 stratified randomly selected subsamples. Migration conditions: article filling, 200 mL, 3% M/V Acetic Acid, 1 hour at 100 °C.

	Cobalt in mg/dm^2	Lithium in mg/dm^2
sample #24721-1	0.111	0.179
sample #24721-2	0.117	0.191
sample #24721-3	0.114	0.185
sample #24721-4	0.117	0.180
sample #24721-5	0.110	0.189
sample #24721-6	0.108	0.180
sample #24721-7	0.119	0.187
sample #24721-8	0.107	0.181

Table 3: homogeneity test results of subsamples #24721

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

	Cobalt in mg/dm^2	Lithium in mg/dm^2
r (observed)	0.013	0.013
reference method	Horwitz	Horwitz
0.3 x R (reference method)	0.021	0.032

Table 4: evaluation of the repeatabilities of subsamples #24721

The calculated repeatabilities are in agreement with 0.3 times the corresponding estimated reproducibility calculated with the Horwitz equation. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one bowl labelled #24720 and one cup labelled #24721 were sent on September 4, 2024.

2.5 ANALYZES

The participants were requested to determine on sample #24720: Formaldehyde using the prescribed test conditions (article filling, repeated use, 2 hours at 70 °C and 3% M/V Acetic Acid as simulant).
For sample #24721 it was requested to determine: 9 different Metals (Aluminum as Al, Barium as Ba, Cobalt as Co, Copper as Cu, Iron as Fe, Lithium as Li, Manganese as Mn, Nickel as Ni and Zinc as Zn) using the prescribed conditions (article filling, single use, 2 hours at 100 °C and 3% M/V Acetic Acid as simulant).

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It was also requested to report for both samples if the laboratory was accredited for the determined component and to report some analytical details.

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 test results but report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot 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 reference test methods (when applicable) 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 are 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 appendices 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 the original test results are placed under 'Remarks' in the result tables in appendices 1 and 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 Organization, Statistics and Evaluation' of October 2024 (iis-protocol, version 4.0).
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,

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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.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. 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. This 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 (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

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 (derived from e.g. ISO or ASTM test methods), the

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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, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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. Therefore, the usual interpretation of z-scores is as follows:

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

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