Results of Proficiency Test
PAA from polyamide kitchenware
June 2020

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Some Primary Aromatic Amines (PAA) are considered to be carcinogenic or suspected to be carcinogenic. PAA can be released from food contact materials, like kitchenware such as spoons, due to impurities or breakdown products present in the polyamide. These PAA together with other precursors present in food can form N-Nitrosamines upon ingestion (through metabolic activation), which are potent carcinogens for animals (and most likely also for humans). In the market some batches of polyamide kitchenware were found to release high levels of PAA in the food. In 2011 the European Commission issued regulation 284/2011 to lay down specific conditions and detailed procedures for the import of polyamide and melamine kitchenware. In support of this, to enhance harmonization of sampling and its testing, EUR24815: Technical Guidelines on testing the migration of primary aromatic amines from polyamide kitchenware was made public (see lit. 2), determining PAA after exposing the kitchenware to acidic test conditions. The limits for PAA is that it should not be present, which means the detection limit applies. In EU284/2011 it is set as 0.01 mg/kg food.

In 2020 the Institute of Interlaboratory Studies (iis) decided to organize a proficiency test scheme for PAA in Kitchenware during the annual testing program of 2019/2020.
In the interlaboratory study 31 laboratories from 14 different countries participated. See appendix 4 for the number of participants per country. In this report the results of this 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 of kitchenware, the nylon part of a soup spoon, labelled #20635, positive for 4,4'-Methylenedianiline 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 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. 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 37 black nylon soup spoons containing a relevant concentration of 4,4'-Methylenedianiline was obtained from a third party. The subsamples were labelled #20635. The homogeneity of the subsamples was checked by determination of the Specific Migration of PAA by an in-house test method on 4 stratified randomly selected spoons. Migration conditions were: 3% Acetic Acid and 120 minutes at 100°C.

	4,4'-Methylenedianiline in µg/L
Sample #20635-1	100.74
Sample #20635-2	91.18
Sample #20635-3	115.54
Sample #20635-4	126.96

Table 1: homogeneity test results on the subsamples #20635

From the above test results the relative standard deviation was calculated and compared to 0.3 times the relative standard deviation of the reference method in agreement with the procedure of ISO13528, Annex B2 in the next table.

	4,4'-Methylenedianiline in µg/L
RSD_r (observed)	14.6%
reference method	EUR24815, Annex 1.4*)
RSD_R (reference method)	12-68%
0.3 x RSD_R (reference method)	4-20%

Table 2: evaluation of the relative standard deviation of subsamples #20635

*) Annex 1.4 mentions an S_R of 3-17% at a level of 25 µg/L, S_R was extrapolated to 100 µg/L.

The calculated relative standard deviation of the repeatability was in agreement with 0.3 times the relative standard deviation of the reproducibility of the reference method, estimated from EUR28415 Annex 1.4. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample #20635 was sent on May 20, 2020.

2.5 ANALYZES

The participants were requested to determine 3 different PAA: Aniline (CAS no. 63-53-3), 4,4'-Methylenediamine (CAS no. 101-77-9) and 2,4-Toluenediamine (CAS no. 95-80-7) on sample #20635 using the prescribed test conditions (immersion, 120 minutes at 100°C and 3% Acetic Acid 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 sample as if it was a routine sample 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:

$\lvert z \rvert < 1$	good
$1 < \lvert z \rvert < 2$	satisfactory
$2 < \lvert z \rvert < 3$	questionable
$3 < \lvert z \rvert$	unsatisfactory

4 EVALUATION

In this proficiency test no severe problems were encountered with the dispatch of the samples. Three participants did not report at all and none of the participants reported the test results after the final reporting date. Not all laboratories were able to report all components requested.
Finally, in total over 54 (intermediate) test results were reported of which 27 test results in mg/dm^2. Observed was one outlying test result, which is 3.7% of the statistically evaluated numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

The data set proved to have a normal Gaussian distribution.

4.1 EVALUATION PER COMPONENT

In this section the test results are discussed per component. The test methods, which were used by the various laboratories, were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables in appendix 1 together with the reported test results. The abbreviations used in these tables are explained in appendix 5.

The Technical Guidelines of EUR28415 (lit. 2) does not have a clear statement that mentions a repeatability and/or reproducibility at the levels of PAA found in this PT. Therefore, it was decided to use a target reproducibility derived from the Horwitz equation. This target is dependent on the measured concentration of PAA in the simulant, the average surface area and the average volume that was used for migration by the participants.

4,4'-Methylenediamine: This determination may be very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the target reproducibility estimated from the Horwitz equation.

Other PAA: The majority of participants agreed on a concentration near or below the limit of detection for the other requested PAA. The test results are given in appendix 2.

4.2 PERFORMANCE EVALUATION OF THE GROUP OF LABORATORIES

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

Component	unit	n	average	2.8 * sd	R(target)
4,4'-Methylenediamine	µg/dm^2	26	24.57	33.55	15.25

Table 3: Overview of results for sample #20635

Without further statistical calculations, it can be concluded that for the PAA 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 OVERVIEW OF PROFICIENCY TEST OF JUNE 2020

The evolution of the uncertainty for Specific Migration in mg/dm^2 as observed in this proficiency scheme is listed in table 4.

Year	Components	Type of migration	Observed RSD%	Target RSD%	Concentration mg/dm^2
2020	4,4'-Methylenediamine	Immersion	49	22	25

Table 4: uncertainty in % for Specific Migration of PAA

4.4 EVALUATION OF THE ANALYTICAL DETAILS

The reported analytical details that were used by the participants are listed in appendix 3. About 71% of the reporting laboratories are accredited for the determination of the PAA in Kitchenware.

Sixteen participants mentioned to have used the Technical Guidelines of EUR24815. The following methods were only used once or twice by the participants: EN13130-1, EN1186, EN28410 or in house. Six participants did not report a test method.

All reporting participants reported the surface area and volume of simulant used. Remarkably, one laboratory (lab 551) reported to have used a surface area of 6 dm^2, two used a surface area of 4 dm^2 and one only 0.6 dm^2, when in fact iis calculated a surface area

of the spoon and handle of around 3 dm^2. Without using the results above 3 dm^2 and below 1 dm2, the average used surface area was 2.48 dm^2.
Three laboratories (labs 2250, 3233 and 3237) used volume of simulant between 1660 and 2000 mL. This is high in comparison to the average of the volume used by the other 25 laboratories, which was 600 mL.

Remarkably, seven laboratories reported to have used water for cleaning or a rigorous cleaning protocol involving detergent, although the instruction for the PT did not include additional cleaning. EUR28415 states the following: “Dust may be removed by wiping the sample with a lint-free cloth of brushing with a soft brush. If articles are labelled with an instruction that they should be cleaned before use then this instruction should be followed before testing”. The use of different methods and cleaning procedures to release the PAA do not have a significant effect on the reproducibility of the group.

From the intermediate results reported for concentration in mg/L simulant, surface and used volume of simulant, the test results in mg/dm^2 were calculated for the PAA present. It appeared that for four laboratories (2247, 2375, 3218 and 3237) the reported test values were different than the iis calculated results, see appendices 1 and 3. All reporting laboratories reported the test results of the first migration step.

5 DISCUSSION

The limits for PAA from 284/2011/EU are stated in mg/kg food. As is mentioned in other Specific Migration methods, such as EN13130-1, the limits expressed in mg/kg can 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 µg/dm^2
Total of PAAs	0.01	1.7

Table 5: Specific Migration maximum limits according to 287/2011/EU

All but one participant would reject the sample for PAA based on this limit.

6 CONCLUSION

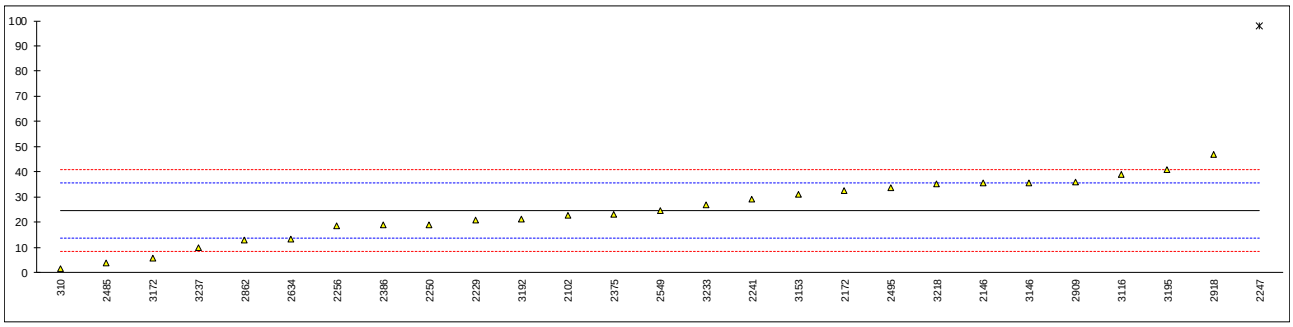
Each laboratory should evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and the quality of the analytical results.

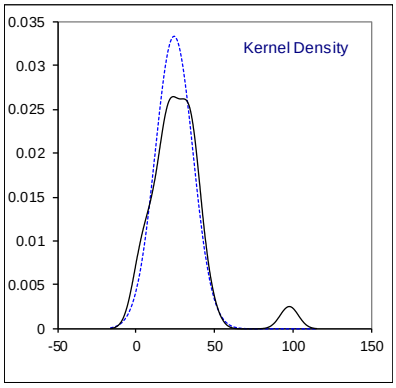
APPENDIX 1

Determination of Specific Migration of 4,4'-Methylenedianiline (CAS No.101-77-9) on sample #20635; results in µg/dm^2 per contact surface

lab	method	value	mark	z(targ)	remarks
310	EUR24815 EN2011	1.57	C	-4.22	first reported: 0.79
551		-----		-----	
2102	EUR24815 EN2011	22.76		-0.33	
2146		35.41		1.99	
2172		32.73		1.50	
2229	EUR24815 EN2011	20.76		-0.70	
2241	EUR24815 EN2011	29.25		0.86	
2247	EN1186	97.88	R(0.01), E	13.46	possible calculation error, iis calculated: 16.31
2250	EUR24815 EN2011	19.107		-1.00	
2256	EUR24815 EN2011	18.7		-1.08	
2366		-----		-----	
2375	EUR24815 EN2011	23.11	E	-0.27	possible calculation error, iis calculated: 49.33
2386		19		-1.02	
2475		-----		-----	
2482		-----		-----	
2485	In house	3.77		-3.82	
2495	EUR24815 EN2011	33.77		1.69	
2549	In house	24.57		0.00	
2634	EN24810	13.46		-2.04	
2862	EUR24815 EN2011	12.8	C	-2.16	first reported: 6.5
2909		36		2.10	
2918		46.9		4.10	
3116	EUR24815 EN2011	39.09		2.67	
3146	EN13030-1	35.5		2.01	
3153	EUR24815 EN2011	30.93		1.17	
3172	EUR24815 EN2011	5.73		-3.46	
3192	EUR24815 EN2011	21.4031		-0.58	
3195		40.7		2.96	
3218	EUR24815 EN2011	35.19	E	1.95	possible calculation error, iis calculated: 62.89
3233	EUR24815 EN2011	26.72	C	0.39	first reported: 0.027
3237	EUR24815 EN2011	9.85	E	-2.70	possible calculation error, iis calculated: 37.78

normality	OK	
n	26	
outliers	1	
mean (n)	24.568	
st.dev. (n)	11.9812	RSD = 49%
R(calc.)	33.547	
st.dev.(Horwitz)	5.4479	
R(Horwitz)	15.254	





APPENDIX 2

Determination of Final concentration of 4,4'-Methylenedianiline (CAS No.101-77-9), Aniline (CAS No. 62-53-3) and 2,4-Toluenediamine (CAS No. 95-80-7) on sample #20635; results in µg/L simulant

Lab	Aniline	mark	4,4'-Methylenedianiline	mark	2,4-Toluenediamine	mark	remarks
310	2.74		7.59		nd		
551	28.01	C	-----		-----		first reported: 22.8129
2102	0.64		83.41		0		
2146	1.89		132.77		-----		
2172	-----		196.389		-----		
2229	ND		210.20		ND		
2241	<10		148.00		<10		
2247	ND		103.60		ND		
2250	< 1		43.5354		< 1		
2256	-----		112.6		-----		
2366	-----		-----		-----		
2375	nd		138.63		nd		
2386	< 2		114		< 2		
2475	-----		-----		-----		
2482	-----		-----		-----		
2485	< LOQ		19.5		not detectable		
2495	<2		230.76		<2		
2549	0		90.1		0		
2634	less than LOQ		34.53		less than LOQ		
2862	-----		38.9		-----		
2909	1.64		95.26		0		
2918	<LOD		216.5		<LOD		
3116	-----		244.70		-----		
3146	1.3		207		n.d.		
3153	1.30		133.0		0		
3172	< 0.05		17.2		< 0.05		
3192	0.6572		64.3055		<LOD		
3195	1.58		169.4		<2		
3218	-----		211.15		-----		
3233	<0.01		48.6	C	<0.01		first reported: 0.049
3237	-----		60.26		-----		

Determination of Specific Migration of Aniline (CAS No. 62-53-3) and 2,4-Toluenediamine (CAS No. 95-80-7) on sample #20635; results in µg/dm^2 per contact surface

Lab	Aniline	mark	2,4-Toluenediamine	mark	remarks
310	0.57	C	nd		first reported: 0.28
551	4.20	C, possible false positive?	-----		first reported: 3.8021
2102	0.17		0		
2146	0.503		-----		
2172	-----		-----		
2229	ND		ND		
2241	<2		<2		
2247	ND		ND		
2250	< 0,439		< 0,439		
2256	-----		-----		
2366	-----		-----		
2375	nd		nd		
2386	< 0,5		< 0,5		
2475	-----		-----		
2482	-----		-----		
2485	< LOQ		not detectable		
2495	<0.28		<0.28		
2549	ND		ND		
2634	less than LOQ		less than LOQ		
2862	-----		-----		
2909	0.82		<1		
2918	<LOD		<LOD		
3116	-----		-----		
3146	0.16		n.d.		
3153	0.30		0		
3172	< 0.05		< 0.05		
3192	0.2188		<LOD		
3195	0.38		<0.5		
3218	-----		-----		
3233	<0.01		<0.01		
3237	-----		nd		

APPENDIX 3 ANALYTICAL DETAILS

Details on final concentration, surface area and volume of simulant reported on 4,4'-Methylene-dianiline

lab	surface area in dm^2	volume simulant in mL	surface to volume ration in dm^2/100mL	final conc. in simulant in µg/L	reported Spec. Migration in µg/dm^2	iis calculated Spec. Migration in µg/dm^2	difference absolute
310	2.90	600	206.9	7.59	1.57	1.57	0.00
551	6	900	-----	-----	-----	-----	-----
2102	2.43	663	272.8	83.41	22.76	22.76	0.00
2146	2.25	600.0	266.7	132.77	35.41	35.41	0.00
2172	2.25	375	166.7	196.389	32.73	32.73	0.00
2229	1.59	157	98.7	210.20	20.76	20.76	0.00
2241	2.53	500.00	197.6	148.00	29.25	29.25	0.00
2247	2.54	400.0	157.5	103.60	97.88	16.31	81.57
2250	4.0101	1760	438.9	43.5354	19.107	19.11	0.00
2256	0.6009	100	166.4	112.6	18.7	18.74	-0.04
2366	n	-----	-----	-----	-----	-----	-----
2375	2.81	1000	355.9	138.63	23.11	49.33	-26.22
2386	4	670	167.5	114	19	19.10	-0.10
2475	n	-----	-----	-----	-----	-----	-----
2482	n	-----	-----	-----	-----	-----	-----
2485	2.65	514	194.0	19.5	3.77	3.78	-0.01
2495	2.05	300.0	146.3	230.76	33.77	33.77	0.00
2549	2.22	600	270.3	90.1	24.57	24.35	0.22
2634	2.6	1000	384.6	34.53	13.46	13.28	0.18
2862	2.7	885	327.8	38.9	12.8	12.75	0.05
2909	2	750	375.0	95.26	36	35.72	0.28
2918	2.31	500	216.5	216.5	46.9	46.86	0.04
3116	2.01	335	166.7	244.70	39.09	40.78	-1.69
3146	2.64	440	166.7	207	35.5	34.50	1.00
3153	2.58	600	232.6	133.0	30.93	30.93	0.00
3172	2.55	850	333.3	17.2	5.73	5.73	0.00
3192	2.4036	800	332.8	64.3055	21.4031	21.40	0.00
3195	2.54	610	240.2	169.4	40.7	40.68	0.02
3218	2.854	850	297.8	211.15	35.19	62.89	-27.70
3233	2.91	1660	570.4	48.6	26.72	27.72	-1.00
3237	3.19	2000	627.0	60.26	9.85	37.78	-27.93

ANALYTICAL DETAILS - continued -

Details on the test procedure

lab	Accred. for ISO17025	sample cleaned prior to the migration step	part exposed to the simulant	step used for reporting results	remarks
310	No	No	only spoon part	First migration step	
551	No	No	the one intended to come in contact with food.	First migration step	Reference method: Regulamento EU Nº 10/2011 GMC nº 39/2019 German BFR XXI
2102	No	No	The part intended to come into contact with food and approx.. 2 cm of the part just above	First migration step	
2146	Yes	Yes, brushed clean from dust	Food contact area and 1 cm of the handle.	First migration step	
2172	Yes	No	soup spoon body exclude spoon handle	First migration step	
2229	Yes	No	the bottom of spoon	First migration step	
2241	Yes	No	spoon	First migration step	
2247	Yes	Yes, cleaned by Lint free tissue paper	Main part of spoon excluding handle	First migration step	ND: Not Detected <10µg/L
2250	Yes	Yes,	according instructions	First migration step	
2256	Yes	No	spoon	First migration step	
2366	---	---		---	
2375	Yes	No	Spoon and Handle	First migration step	
2386	Yes	Yes, not specified	Functional part hot water	First migration step	
2475	---	---		---	
2482	---	---		---	
2485	Yes	Yes, cleaned with water	Spoon without the shaft	First migration step	
2495	Yes	Yes, gentle cleaned with cold water and soap	Ladle without handle.	First migration step	
2549	Yes	No	Down side part of the spoon	First migration step	
2634	Yes	Yes, Cleaned with distilled water	Only the area that comes with contact with the food.	First migration step	
2862	Yes	No	The nylon part of the spoon.	First migration step	
2909	Yes	No	round part supposed to come into contact with food + 2 cm handle	First migration step	
2918	Yes	Yes, not specified	soup ladle (complete) + 2 cm of the handle	First migration step	
3116	No	Yes, not specified	The nylon part.	First migration step	
3146	No	Yes, short "household cleaning" with warm water and sih liquid	Nylon part as requested	First migration step	
3153	No	Yes, dust removed by wiping with a lint-free cloth	The round part of the spoon	First migration step	
3172	Yes	Yes, cleaned with distilled water only	Functional part + 2 cm of handle	First migration step	
3192	Yes	No	Spoon + 5,71 cm from the Handle	First migration step	
3195	Yes	No	part necessary in contact with food + handle up to 2cm height.	First migration step	
3218	Yes	Yes, with a dilute and warm solution of a commercial detergent and then rinse immediately with plenty of running water and with distilled water	the head and tail of the soup spoon	First migration step	
3233	No	Yes, cleaned with water	we immersed the sample with the simulant, except the piece of the handle	First migration step	
3237	No	No	Whole part	First migration step	

APPENDIX 4

Number of participating laboratories per country

1 lab in BRAZIL
1 lab in FINLAND
2 labs in FRANCE
7 labs in GERMANY
2 labs in HONG KONG
2 labs in INDIA
1 lab in ISRAEL
2 labs in ITALY
1 lab in LUXEMBOURG
6 labs in P.R. of CHINA
1 lab in SLOVENIA
1 lab in SWITZERLAND
2 labs in THE NETHERLANDS
2 labs in TURKEY

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

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