



Institute for  
Interlaboratory Studies

## Results of Proficiency Test PAA from (polyamide) kitchenware October 2023

Organized by: Institute for Interlaboratory Studies  
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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 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 (lit. 13), determining PAA after exposing the kitchenware to acidic test conditions. The limit for PAA is that it should not be present, which means the detection limit applies. In EUR24815 EN2011 it is set as 0.01 mg/kg food or food simulants.

Since 2020 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of PAA from polyamide kitchenware every year. During the annual proficiency testing program 2023 it was decided to continue the proficiency test for the determination of PAA from polyamide kitchenware.

In this interlaboratory study 24 laboratories from 12 countries registered for participation, see appendix 4 for the number of participants per country. In this report the results of the PAA from polyamide kitchenware proficiency test are presented and discussed. This report is also electronically available through the iis website [www.iisnl.com](http://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 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 one sample of kitchenware, a yellow Acrylonitrile Butadiene Styrene (ABS) cup labelled #23725. Although the sample is not made from polyamide, it is positive for PAA and therefore suitable for this proficiency test.

The participants were requested 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](http://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 yellow Acrylonitrile Butadiene Styrene (ABS) cups was selected, which was made positive on PAA by a third party. From the batch 35 plastic bags were filled with one cup and labelled #23725.

The homogeneity of the subsamples was checked by determination of PAA using an in house test method on 8 stratified randomly selected subsamples with the following conditions: article filling, 200 mL of 3% Acetic Acid and 2 hours at 70 °C.

	4,4-Diaminodiphenyl methane µg/L
sample #23725-1	18.4
sample #23725-2	23.7
sample #23725-3	18.2
sample #23725-4	21.2
sample #23725-5	20.5
sample #23725-6	19.2
sample #23725-7	21.1
sample #23725-8	22.2

Table 1: homogeneity test results of subsamples #23725

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

	4,4-Diaminodiphenyl methane µg/L
r (observed)	5.3
reference method	Horwitz
0.3 x R (reference method)	5.0

Table 2: evaluation of the repeatability of subsamples #23725

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.

To each of the participating laboratories one ABS sample labelled #23725 was sent on September 6, 2023.

## 2.5 ANALYZES

The participants were requested to determine 3 different PAA: Aniline (CAS no. 62-53-3), 4,4'-Methylenedianiline (CAS no. 101-77-9) and 2,4-Toluenediamine (CAS no. 95-80-7) using the prescribed test conditions (article filling, single use as migration method and 200 mL of 3% Acetic Acid as simulant for 2 hours at 70 °C). In daily practice, not just one item, but more items for testing would have been sent. However, this sample is positive on PAA. This means that one item of the sample is sufficient for the determination of PAA. It was also requested to report if the laboratory was accredited for the determined components and to report some analytical details.

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 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/](http://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](http://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/](http://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 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.

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 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:

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

The  $Z_{(\text{target})}$  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

## 4 EVALUATION

In this proficiency test no severe problems were encountered with the dispatch of the samples. All participants reported test results before the final reporting date except for one participant who did not report any test results. Not all laboratories were able to report all components requested.

In total 23 participants reported 26 numerical test results. Observed were 2 outlying test results, which is 7.7%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

The data set of 4,4'-Methylenedianiline proved not to have a normal Gaussian distribution. This is referred to as "not OK" or "suspect". The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

### 4.1 EVALUATION PER COMPONENT

In this section the reported 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 together with the original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 5.

The Technical Guidelines of EUR24815 (lit. 13) does not have a clear statement that mentions a repeatability and/or reproducibility at the levels of PAA found in this PT. For these components the calculated reproducibility was compared against the estimated reproducibility calculated with the Horwitz equation.

Aniline: The reporting participants agreed on a value near or below the application range. Therefore, no z-scores are calculated.

4,4'-Methylenedianiline: The group of participants may have had difficulty to meet the target requirements. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated reproducibility calculated with the Horwitz equation.

2,4-Toluediamine: The reporting participants agreed on a value near or below the application range. Therefore, no z-scores are calculated.

### 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the 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 ( $2.8 \times$  standard deviation) and the target reproducibility derived from the reference method are presented in the next table.



Component	unit	n	average	2.8 * sd	R(target)
Aniline	µg/dm <sup>2</sup>	9	<0.5	n.e.	n.e.
4,4'-Methylenedianiline	µg/dm <sup>2</sup>	21	2.9	3.5	3.1
2,4-Toluediamine	µg/dm <sup>2</sup>	6	<0.5	n.e.	n.e.

Table 3: reproducibilities of tests on sample #23725

Without further statistical calculations it can be concluded that for 4,4'-Methylenedianiline there is not a good compliance of the group of participants with the reference method.

#### 4.3 COMPARISON OF THE PROFICIENCY TEST OF OCTOBER 2023 WITH PREVIOUS PTS

	October 2023	October 2022	October 2021	October 2020
Number of reporting laboratories	23	15	20	28
Number of test results	26	20	19	27
Number of statistical outliers	2	1	1	1
Percentage of statistical outliers	7.7%	5.0%	5.3%	3.7%

Table 4: comparison with previous proficiency tests

In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

The performance of the determinations of the proficiency test was compared to uncertainties observed in PTs over the years, expressed as relative standard deviation (RSD) of the PTs, see next table.

Year	Components	Type of migration	Observed RSD%	Target RSD%	Concentration µg/dm <sup>2</sup>
2020	4,4'-Methylenedianiline	immersion	49	22	25
2021	Aniline	immersion	31	29	20
2022	4,4'-Methylenedianiline	article filling	57	38	15
2023	4,4'-Methylenedianiline	article filling	44	39	2.9

Table 5: development of the uncertainties over the years

The uncertainty observed in this PT is much closer to the target uncertainty than in previous PTs.

#### 4.4 EVALUATION OF THE ANALYTICAL DETAILS

The reported details of the determination of PAA per contact surface area of the cup and the reported analytical details that were used by the participants are listed in appendices 2 and 3. Based on the answers given by the participants the following can be summarized:

- Eight participants have used the Technical Guidelines of EUR24815, four participants EN13130-1 and nine other participants an in house test method.
- A majority (14 out of 21) of participants are accredited to determine the components in this PT.

- All participants used 200 mL amount of simulant as was prescribed for this PT.
- The contact surface area varied between 1.2 and 1.8 dm<sup>2</sup>.
- While this sample was not labelled with cleaning before use instructions eight participants did clean the sample prior to the migration step, mostly with (D.I.) water
- Most participants heated the simulant prior to filling the sample with it.
- Almost all participants used an oven for the migration step.
- All participants prevented that the simulant was evaporated during the test by either testing in an airtight container, using an aluminum seal or by covering with glass or plastic wrap.

The influence of these analytical details could not be determined because either the group followed the same analytical procedures or the group of participants is too small for meaningful sub analyzes.

## 5 DISCUSSION

The limit for PAA from 284/2011/EU is stated in mg/kg food. As is mentioned in other Specific Migration methods, such as EN13130-1, the limit expressed in mg/kg can be divided by the conventional conversion factor of 6 in order to express it in mg/dm<sup>2</sup>, see table 6.

Component	Specific Migration Limit in µg/kg	Specific Migration Limit in µg/dm <sup>2</sup>
Total of PAAs	10	1.7

Table 6: Specific Migration maximum limits according to 284/2011/EU

It can be concluded that, based on this limit, all but four reporting participants would reject the sample based on the test results of 4,4'-Methylenedianiline.

## 6 CONCLUSION

Although it can be concluded that most of the participants have no problem with the determination of 4,4'-Methylenedianiline in this PT, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

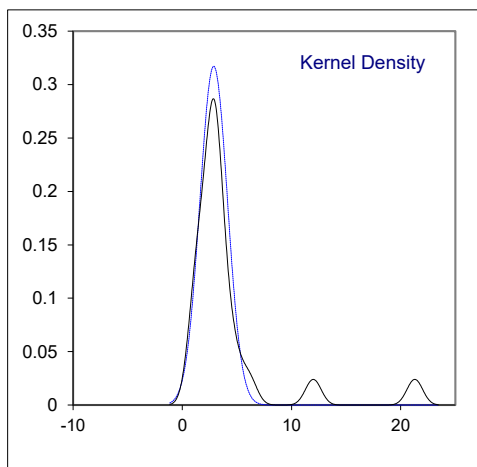
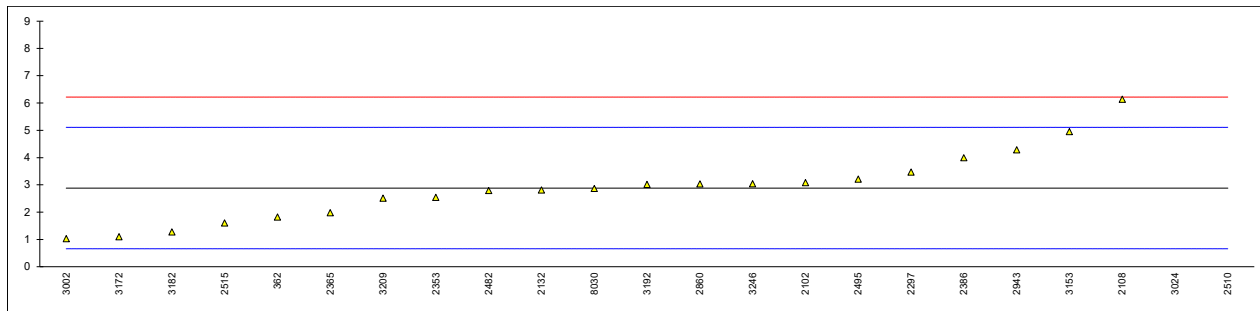
**APPENDIX 1**

Determination of Specific Migration of Aniline (CAS No. 62-53-3) on sample #23725; results in  $\mu\text{g}/\text{dm}^2$  per contact surface

lab	method	value	mark	z(targ)	remarks
362		----		----	
551		----		----	
2102	In house	Not detected		----	
2108	EUR24815 EN2011	not detected		----	
2132	EUR24815 EN2011	<0.002		----	
2297	EUR24815 EN2011	Not detected		----	
2353	EN13130-1	0.319		----	
2365		<2		----	
2386	In house	< 0.25		----	
2482	In house	< 0,27		----	
2495	EUR24815 EN2011	Not Detected		----	
2510	In house	0.2691		----	
2515	EN13130-1	<0.26438		----	
2860	In house	<0.167		----	
2943	In house	Not detected		----	
3002	In house	not detected		----	
3024	EN13130-1	Not Detected		----	
3153		----		----	
3172	EUR24815 EN2011	< 0.28		----	
3182	EUR24815 EN2011	Not analysed		----	
3192	In house	0.015		----	
3209		----		----	
3246		Not detected		----	
8030	EN13130-1	Not detected		----	
	n	9			
	mean (n)	<0.5			

Determination of Specific Migration of 4,4'-Methylenedianiline (CAS No.101-77-9) on sample #23725; results in  $\mu\text{g}/\text{dm}^2$  per contact surface

lab	method	value	mark	z(targ)	remarks
362	In house	1.82		-0.96	
551					
2102	In house	3.080		0.18	
2108	EUR24815 EN2011	6.137		2.93	
2132	EUR24815 EN2011	2.81		-0.07	
2297	EUR24815 EN2011	3.47		0.53	
2353	EN13130-1	2.542		-0.31	
2365		1.98		-0.81	
2386	In house	3.993		1.00	
2482	In house	2.792		-0.08	
2495	EUR24815 EN2011	3.21		0.29	
2510	In house	21.2966	R(0.01)	16.55	
2515	EN13130-1	1.60224		-1.15	
2860	In house	3.035		0.14	
2943	In house	4.2807		1.26	
3002	In house	1.03		-1.67	
3024	EN13130-1	12	C,R(0.01)	8.20	first reported Not Detected
3153	EUR24815 EN2011	4.95	C	1.86	first reported 24.7
3172	EUR24815 EN2011	1.0998		-1.60	
3182	EUR24815 EN2011	1.27		-1.45	
3192	In house	3.016		0.12	
3209	EUR24815 EN2011	2.511		-0.33	
3246		3.0394		0.14	
8030	EN13130-1	2.865		-0.02	
normality		suspect			
n		21			
outliers		2			
mean (n)		2.883			
st.dev. (n)		1.2584	RSD=44%		
R(calc.)		3.524			
st.dev.(Horwitz)		1.1123			
R(Horwitz)		3.115			



Determination of Specific Migration of 2,4-Toluenediamine (CAS No. 95-80-7) on sample #23725;  
results in  $\mu\text{g}/\text{dm}^2$  per contact surface

lab	method	value	mark	z(targ)	remarks
362		----		----	
551		----		----	
2102	In house	Not detected		----	
2108	EUR24815 EN2011	not detected		----	
2132	EUR24815 EN2011	<0.002		----	
2297	EUR24815 EN2011	Not detected		----	
2353	EN13130-1	ND		----	
2365		<2		----	
2386	In house	< 0.25		----	
2482	In house	< 0,27		----	
2495	EUR24815 EN2011	Not Detected		----	
2510		----		----	
2515	EN13130-1	<0.26438		----	
2860	In house	<0.167		----	
2943	In house	Not detected		----	
3002	In house	not detected		----	
3024	EN13130-1	Not Detected		----	
3153		----		----	
3172	EUR24815 EN2011	< 0.28		----	
3182	EUR24815 EN2011	Not detected		----	
3192	In house	not detected		----	
3209		----		----	
3246		Not detected		----	
8030	EN13130-1	Not detected		----	
	n	6			
	mean (n)	<0.5			

**APPENDIX 2**

## Reported intermediate test results on sample #23725

lab	surface area (dm <sup>2</sup> )	volume simulant (mL)	final concentration in simulant (µg/L)			remarks
			Aniline	4,4'-Methylenedianiline	2,4-Toluenediamine	
362	1.52	200	----	13.86	----	
551	-----	-----	----	-----	----	
2102	1.521	200	Not detected	23.42	Not detected	
2108	1.75	200	not detected	53.70	not detected	
2132	1.36	200	<0.002	19.10	<0.002	
2297	1.48	200	Not detected	25.68	Not detected	
2353	1.388	200	2.217	17.645	ND	
2365	1.62	200.0	<2	15.9	<2	
2386	1.6	200	< 2	31.942	< 2	
2482	1.49	200	< 2	20.80	< 2	
2495	1.45	200	Not Detected	23.3	Not Detected	
2510	1.4516	200	0.32557	25.6407	----	
2515	1.513	200	<2	12.121	<2	
2860	1.311	200	<1.000	19.893	<1.000	
2943	1.5	200	Not detected	32.1050	Not detected	
3002	1.2	200.0	not detected	6.16	not detected	
3024	1.44	200.00	Not Detected	Not Detected	Not Detected	
3153	1.3544	200	----	33.5	----	
3172	1.4335	200	< 2	7.8824	< 2	
3182	1.73	200.00	Not analysed	10.94	Not detected	
3192	1.59	200	0.1209	23.9736	not detected	
3209	1.45	200	----	18.21	----	
3246	1.46	200	Not detected	22.19	Not detected	
8030	1.74	200	Not detected	24.925	Not detected	

**APPENDIX 3**

## Summary of reported analytical details

lab	ISO17025 accredited	sample cleaned prior to migration step(s)	simulant preheated	equipment migration	simulant sealed to prevent simulant evaporation
362	---	---	---	---	---
551	---	---	---	---	---
2102	Yes	No	Yes	Oven	Sealed with a watch glass
2108	No	Cleaned with lint-free cloth	Yes	Oven	With plastic wrap
2132	Yes	Used DI water to clean	Yes	Oven	Yes, tested in an airtight container
2297	Yes	No	Yes	Oven	With aluminum seal
2353	No	No	Yes	Oven	Covered by glass
2365	Yes	No	Yes	Oven	Yes, tested in an airtight container
2386	Yes	No	Yes	Incubator	Sealed with a glas plate
2482	No	With a lint-free cloth	Yes	Oven	Covered with a watch glass
2495	Yes	Cleaned with soap	Yes	Oven	Covered with glass laboratory
2510	Yes	No	Yes	Oven	Yes, with aluminum seal
2515	Yes	No	No	Oven	Sealed with PE film
2860	Yes	No	Yes	Oven	Yes, with aluminum seal
2943	Yes	No	Yes	Oven	Yes, tested in an airtight container
3002	Yes	Cleaned with water	Yes	Oven	Yes, tested in an airtight container
3024	Yes	Rinsed with purified water	No	Oven	Sealed with a laboratory Watch Glass
3153	No	No	Yes	Oven	Yes, with aluminum seal
3172	---	No	Yes	Oven	Yes, with aluminum seal
3182	No	With DI water	Yes	Oven	Cover the sample with watch glass
3192	No	No	No	Incubator	Covered with glass
3209	Yes	No	Yes	Oven	Yes, tested in an airtight container
3246	No	Rinse with D.I. water	Yes	Oven	Seal sample with a thick plastic film
8030	Yes	No	Yes	Oven	Yes, tested in an airtight container

## **APPENDIX 4**

### **Number of participants per country**

1 lab in BRAZIL  
1 lab in BULGARIA  
4 labs in GERMANY  
3 labs in HONG KONG  
1 lab in IRELAND  
1 lab in ISRAEL  
4 labs in ITALY  
3 labs in P.R. of CHINA  
1 lab in SERBIA  
2 labs in THAILAND  
1 lab in THE NETHERLANDS  
2 labs in VIETNAM



## 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	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?

### Literature

- 1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
- 2 ISO5725:86
- 3 ISO5725 parts 1-6:94
- 4 ISO13528:05
- 5 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
- 6 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 7 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
- 8 J.N. Miller, Analyst, 118, 455, (1993)
- 9 Analytical Methods Committee, Technical Brief, No 4, January 2001
- 10 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)
- 11 W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
- 12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)
- 13 EUR24815 Technical Guidelines on testing the migration of primary aromatic amines from polyamide kitchenware and of formaldehyde in kitchenware, 1<sup>st</sup> edition 2011 (European Commission Joint Research Center Scientific and Technical Reports)
- 14 Commission regulation (EU) No 287/2011 of 22 March 2011 laying down specific conditions and detailed procedures for the import of polyamide and melamine plastic kitchenware originating in or consigned from the People's Republic of China and Hong Kong Special Administrative Region, China