Results of Proficiency Test OPP & Other Preservatives in Textile December 2020

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since the 1990's many countries have adopted environmental standards and requirements restricting the use of harmful chemicals in the production of textiles and clothing. Laws and regulations impose some of these standards and requirements. In addition to mandatory environmental standards and requirements for textiles, some Eco-labelling schemes are imposing environmental requirements for textile products on a voluntary basis, e.g. Milieukeur (Netherlands), Bluesign© (Switzerland) and Oeko-Tex© Standard 100 (Switzerland).
Since 2004 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for Ortho-Phenylphenol (OPP) in Textile every year. In 2019 it was decided to separate the proficiency tests on the determination of Ortho-Phenylphenol and Chlorinated Phenols in Textile. During the annual proficiency test program 2020/2021 it was decided to continue the proficiency test of OPP & other Preservatives in Textile.

In this interlaboratory study 28 laboratories in 13 different countries registered for participation. 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 analysis for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send one textile sample of approximately 3 grams labelled #20745.
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, 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 light beige cotton was selected which was made positive for OPP by a third party. After cutting the cotton into small pieces and homogenization the batch was divided over 50 subsamples in small bags of approximately 3 grams each and labelled #20745.
The homogeneity of the subsamples was checked by determination of OPP with an in-house test method on 8 stratified randomly selected subsamples.

	OPP in mg/kg
Sample #20745-1	43.0
Sample #20745-2	45.8
Sample #20745-3	42.3
Sample #20745-4	44.0
Sample #20745-5	44.6
Sample #20745-6	44.5
Sample #20745-7	42.5
Sample #20745-8	49.1

Table 1: homogeneity test results of subsamples #20745

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

	OPP in mg/kg
r (observed)	6.2
reference method	iis memo 1601 (see lit. 16)
0.3 x R (reference method)	7.7

Table 2: evaluation of the repeatability of subsamples #20745

The calculated repeatability of Ortho-Phenylphenol (OPP) was in agreement with 0.3 times the estimated reproducibility of the reference method. Therefore, homogeneity of the subsamples was assumed.

To each participating laboratory 1 sample labelled #20745 was sent on November 18, 2020.

2.5 ANALYZES

The participants were requested to determine on samples #20745 the concentrations of Ortho-Phenylphenol (OPP), 2-(Thio Cyano Methyl Thio)-Benzothiazole (TCMTB), 4-Chloro-3-Methyl Phenol (PCMC), 2-Octyl Iso Thiazol-3(2H)-one (OIT), Triclosan (TCS) and eventually other Preservatives detected. It was also requested to report if the laboratory was accredited to determine the requested components and to report some analytical details of the test method used.

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 to 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 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 reanalysis). Additional or corrected test results are used for data analysis and the original test results are placed under ‘Remarks’ in the test result tables in appendix 1. 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.

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 of 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.
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

During the execution of this proficiency test no problems occurred with the dispatch of the samples. Two participants reported test results after the final reporting date and one other participant was not able to report any test results at all. Not all laboratories were able to report all components requested.
In total 27 laboratories reported 25 numerical test results. Two statistical outliers were observed, which is 8.0%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

Not all data sets proved to have a normal Gaussian distribution. These are 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 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 original data. The abbreviations, used in these tables, are explained in appendix 5.

Participants use different methods to determine OPP. Since 2019 the test method EN17134 is available for OPP and TCS in Textile. This test method describes an extraction with Acetonitrile using Ultrasonic. Unfortunately, no precision data is mentioned in this method. Other methods that are used by the participants are ISO13365 (Determination of the Preservative (TCMTB, PCMC, OPP, OIT) content in Leather) and in-house test methods.

Due to the lack of relevant reference test methods and/or precision data for the determination of OPP in Textile, the calculated reproducibility was compared with an estimated target reproducibility based on iis PT data of OPP/PCP proficiency tests from 2004 until 2014, iis memo 1601 (see lit.16). As it was assumed that the variation in the PT test results will be dependent on the concentration, this resulted in a Horwitz-like equation to estimate the target reproducibilities for the evaluation of the PT test results by iis from 2015 onwards.

Sample #20745

OPP: Taking into account the reported analytical details, two groups of results were identified. Test results obtained with Acrylonitrile extraction and test results obtained with Alkaline extraction differed significantly (see also paragraph 4.2 and 4.4). Therefore, these groups were evaluated separately.
For the group using Acrylonitrile extraction this determination may not be problematic. One outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated reproducibility calculated from iis memo 1601.
For the group using Alkaline extraction this determination may not be problematic. One outlier was observed and three other test results were excluded for not reporting analytical details or reporting to have used Hexane as Extraction solvent. The calculated reproducibility after rejection of the suspect data is in agreement with the estimated reproducibility calculated from iis memo 1601.

Other Preservatives: The reported concentrations of all other components were near or below the detection limit. Therefore, no z-scores were calculated. The reported test values are given in appendix 2.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the estimated target reproducibility calculated with the equation mentioned in iis memo 1601 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 estimated reproducibility are presented in next table.

Component	unit	n	average	2.8 * sd	R(target)
Ortho-Phenylphenol (OPP) ACN	mg/kg	15	6.2	5.0	4.8
Ortho-Phenylphenol (OPP) KOH	mg/kg	5	24.4	10.8	15.5

Table 3: performance evaluation sample #20745

Without further statistical calculations it can be concluded that based on the extraction method used the participating laboratories have no difficulties with the analysis of OPP. See also the discussion in paragraphs 4.1, 4.2, 4.4 and 5.

4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2020 WITH PREVIOUS PTS

In this PT, the observed variation expressed as the relative standard deviation RSD of the test results is similar in comparison with the uncertainties observed in previous PTs, see the table below.

Component	December 2020	December 2019	December 2018	December 2017	December 2016	iis memo 1601
Ortho-Phenylphenol (OPP)	16-29%	21%	16-58%	39-54%	38%	24%

Table 4: comparison of uncertainties in iis proficiency tests over the years

4.4 EVALUATION OF ANALYTICAL DETAILS

The reported analytical details from the participants are listed in appendix 3. In total twenty-six participants reported analytical details:

- to determine the reported component(s) in textile sixteen participants (62%) were accredited in accordance with ISO/IEC17025 and ten participants were not accredited (38%)
- prior to analysis the samples were further cut by sixteen participants (61%), one participant did further grind (4%) while nine participants (35%) used the sample as received
- five participants (19%) used a sample intake of 0.5-0.75 gram and seventeen participants (66%) used around 1 gram. Four participants (15%) used around 1.5 gram or more.
- to extract the components the most often used technique was Ultrasonic extraction by twenty-three participants (88%). Other techniques like Mechanical Shaking and Alkaline Digestion was used by three participants (12%)
- eighteen participants (69%) used Acrylonitrile as extraction solvent, six participants (23%) used KOH and two participants (8%) used Hexane.

It is observed that the use of ACN extraction solvent or KOH extraction solvent gives a significantly different average of the test results of the group. Therefore, iis decided to evaluate both groups separately. Please note that the observed effects for the other analytical details are not statistically significant given the variation found in these small groups.

5 DISCUSSION

In this PT, the average of the homogeneity test results is not in line with the average (consensus value) from the PT results. There are several reasons for this. First, the goal of the homogeneity testing is very different from the goal of the evaluation of the reported PT results. In order to prove the homogeneity of the PT samples, a test method is selected with a high precision (smallest variation). The accuracy (trueness) of the test method is less relevant.
Secondly, the homogeneity testing is done by one laboratory only. The test results of this (ISO/IEC 17025 accredited) laboratory will have a bias (systematic deviation) depending on the test method used. The desire to detect small variations between the PT samples leads to the use of a sensitive test method with high precision, which may be a test method with significant bias.
Also each test result reported by the laboratories that participate in the PT will have a bias. However, some will have a positive bias and others a negative bias. These different biases compensate each other in the PT average (consensus value). Therefore, the PT consensus value may deviate from the average of the homogeneity test. At the same time the accuracy of the PT consensus value is more reliable than the accuracy of the average of the results of the homogeneity test.

Sample #20745 was used before in Proficiency Test iis16A11 as sample #16645 and in iis18A14 as sample #18650. In these PTs the extraction solvent was not requested, only the extraction method. Since the publication of EN17134 in 2019 more laboratories may have switched from Alkaline extraction to Acetonitrile extraction. This can be of influence on the test result. Therefore, the test results of this PT cannot be compared with the previous PTs.

When the test results of this interlaboratory study were compared to the Ecolabelling Standards and Requirements for Textiles in EU (see table 7) it could be noticed that all of the participants were able to detect OPP in sample #20745.

Ecolabel	Class 1 Baby clothes (mg/kg)	Class 2 Clothes direct skin contact (mg/kg)	Class 3 Clothes, no direct skin contact (mg/kg)	Class 4 Decoration material (mg/kg)
Ortho-Phenylphenol (OPP)	50.0	100.0	100.0	100.0

Table 5: Ecolabelling Standards and Requirements for Textiles in EU

All reported test results for OPP, except one, were <50.0 mg/kg for sample #20745. Based on this the textile material would have been accepted for all four classes mentioned in table 5 by all reporting laboratories, except one.

6 CONCLUSION

In this proficiency test OPP and other Preservatives in Textile were determined. The participating laboratories had no problems in determining the Ortho-Phenylphenol (OPP), although the choice of extraction solvent can be of significant influence. It is advised that members of the technical committee to take this on-board and to discuss and decide the best extraction method for Ortho-Phenylphenol (OPP) determination.
Other Preservatives in the sample were not detected or were close or underneath the detection limit.

Each laboratory should evaluate its performance in this study and make decisions about possible corrective actions. 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.

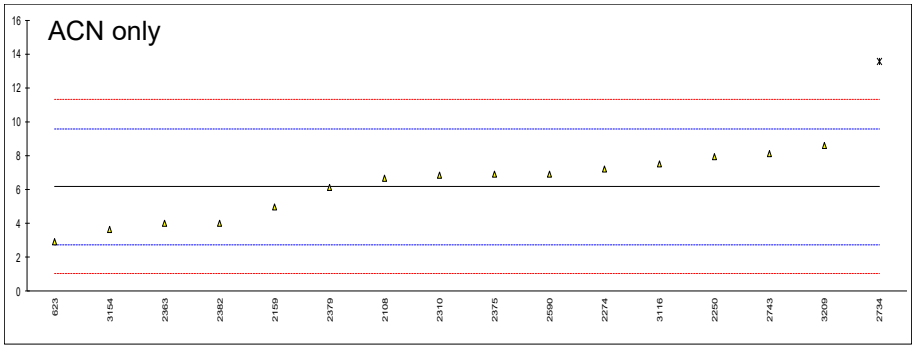
APPENDIX 1

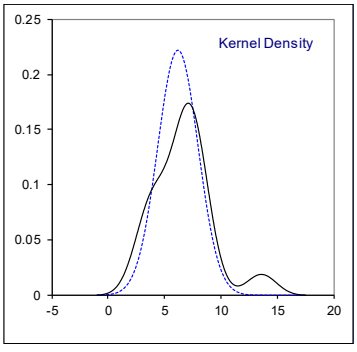
Determination of Ortho-Phenylphenol (OPP) on sample #20745; results in mg/kg

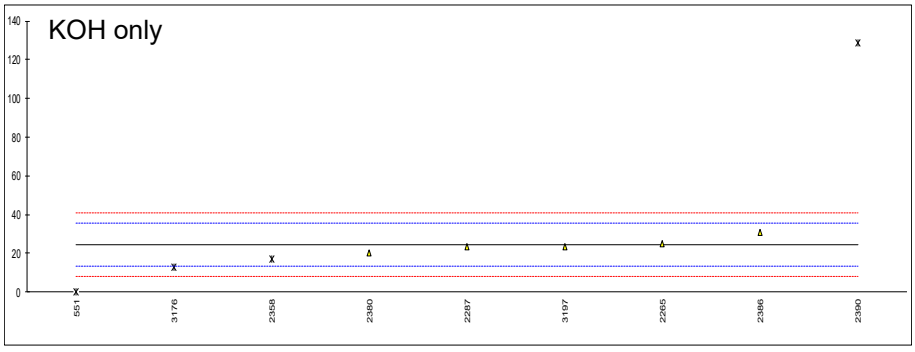
lab	method	ACN only	mark	z(targ)	KOH only	mark	z(targ)	remarks
551	ISO17070	-----		-----	0.26	G(0.05)	-4.37	
623	ISO13365	2.910		-1.89	-----		-----	
2108	In house	6.64		0.28	-----		-----	
2159	In house	4.955		-0.70	-----		-----	
2250	EN17134	7.903		1.02	-----		-----	
2265	In house	-----		-----	24.939		0.09	
2274	EN17134	7.179		0.60	-----		-----	
2287		-----		-----	23.25		-0.22	
2310	ISO13365	6.83		0.39	-----		-----	
2358	In house	-----		-----	17.1637	ex	-1.32	*)
2363	ISO13365	4.01		-1.25	-----		-----	
2375	EN17134	6.92		0.45	-----		-----	
2379	In house	6.132		-0.01	-----		-----	
2380	64LFGB B82.02.8Mod.	-----		-----	20.138		-0.78	
2382	ISO13365	4.03		-1.24	-----		-----	
2386	In house	-----		-----	30.61	C	1.11	fr. 26.51
2390	In house	-----		-----	128.38	ex,C	18.79	fr. 45.7813, **)
2590	EN17134	6.930		0.45	-----		-----	
2644		-----		-----	-----		-----	
2734	EN17134	13.57	G(0.05)	4.33	-----		-----	
2743	EN17134	8.1310		1.15	-----		-----	
3116	In house	7.533		0.81	-----		-----	
3154		3.64		-1.47	-----		-----	
3172	In house	<5		-----	-----		-----	
3176	In house	-----		-----	13.04	ex,C	-2.06	fr. 33.24, **)
3197	In house	-----		-----	23.30		-0.21	
3209	In house	8.57		1.41	-----		-----	
3210	EN13365	<40		-----	-----		-----	
	normality	OK			not OK			
	n	15			5			
	outliers	1			1 (+3ex)			
	mean (n)	6.154			24.447			
	st.dev. (n)	1.7934	RSD = 29%		3.8581	RSD = 16%		
	R(calc.)	5.022			10.803			
	st.dev.(iis-memo 1601)	1.7122			5.5304			
	R(iis-memo 1601)	4.794			15.485			

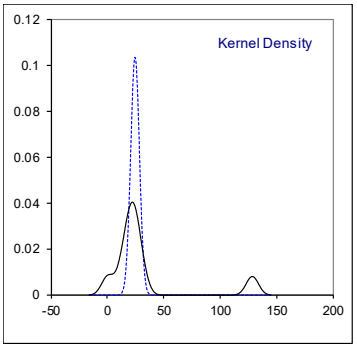
*) Lab 2358 did not report any analytical details, therefore the test result was excluded. Based on the result it is expected that a KOH extraction has been used.

**) Lab 2390 reported n-Hexane as extraction solvent, therefore the test result was excluded.
Lab 3176 reported Hexane as extraction solvent, therefore the test result was excluded.









APPENDIX 2 Other reported test results

Determination of 2-(Thio Cyano Methyl Thio)-Benzothiazole (TCMTB), 4-Chloro-3-Methyl Phenol (PCMC), 2-Octyl Iso Thiazol-3(2H)-one (OIT), Triclosan (TCS) and Other Preservatives on sample #20745; in mg/kg

lab	TCMTB	PCMC	OIT	TCS	Other Preservatives
551	-----	-----	-----	-----	-----
623	not detected	not detected	not detected	not detected	not detected
2108	-----	-----	-----	-----	-----
2159	<0,5	<0,5	<0,5	<0,5	-----
2250	-----	-----	-----	-----	-----
2265	-----	-----	-----	-----	-----
2274	not determined	not determined	not determined	not determined	not determined
2287	-----	-----	-----	-----	-----
2310	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2358	n.d.	n.d.	n.d.	n.d.	n.d.
2363	<1	<1	<1	<1	<1
2375	-----	-----	-----	-----	-----
2379	Not detected	Not detected	Not detected	Not detected	Not tested
2380	-----	-----	-----	-----	-----
2382	-----	-----	-----	-----	-----
2386	not detected	not detected	not detected	not detected	not detected
2390	-----	-----	-----	-----	-----
2590	-----	-----	-----	-----	-----
2644	-----	-----	-----	-----	-----
2734	n.d	n.d.	n.d.	n.d.	n.d.
2743	-----	-----	-----	-----	-----
3116	-----	-----	-----	-----	-----
3154	-----	-----	-----	-----	-----
3172	< 5	< 5	< 5	< 5	< 5
3176	-----	-----	-----	-----	-----
3197	-----	-----	-----	-----	-----
3209	-----	-----	-----	-----	-----
3210	<40	<40	<40	-----	-----

APPENDIX 3 Analytical details

lab	ISO17025 accredited	Sample preparation	Sample intake (grams)	Extraction technique	Extraction solvent
551	No	Further cut	1 g	Ultrasonic	KOH 1M
623	Yes	Further cut	1 gram	Ultrasonic	Acetonitrile
2108	Yes	Further cut	1 g	Ultrasonic	Acetonitrile
2159	Yes	Further cut	1 Gram	Ultrasonic	ACN
2250	Yes	Further cut	1	Ultrasonic	Acetonitril
2265	No	Used as received	0,5g	Mechanical Shaking	KOH extracted with n-Hexane
2274	No	Used as received	1.0g	Ultrasonic	Acetonitrile
2287	No	Further cut	1g	Alkaline digestion	1 mol/L-potassium hydroxide solution
2310	No	Further cut	One gram	Ultrasonic	Acetonitrile
2358	---	---		---	
2363	No	Further cut	1g	Ultrasonic	ACN
2375	No	Further cut	0,5 grams	Ultrasonic	Acetonitrile
2379	No	Further cut	0.5 gram	Ultrasonic	Acetonitrile
2380	Yes	Further cut	1.0 g	Alkaline digestion	KOH / n-hexane
2382	Yes	Further cut	2.0g	Ultrasonic	Acetonitrile
2386	Yes	Further cut	0.5 g	Ultrasonic	KOH 1M (OPP) Methanol (Triclosan)
2390	Yes	Further grinded	1.0gm	Ultrasonic	n-hexane
2590	Yes	Further cut	1 g	Ultrasonic	ACN
2644	---	---		---	
2734	Yes	Used as received	2,5 g	Ultrasonic	Acetonitrile
2743	No	Used as received	0.75 g	Ultrasonic	Acetonitrile
3116	No	Used as received	1 grams	Ultrasonic	Acetonitrile
3154	Yes	Used as received	1 g	Ultrasonic	Acetonitrile
3172	Yes	Further cut	2 grams	ultrasonic	Acetonitrile
3176	Yes	Further cut	1	Ultrasonic	Hexane
3197	Yes	Used as received	1	Ultrasonic	1 M KOH
3209	Yes	Used as received	1.4230g	Ultrasonic	Acetonitrile
3210	Yes	Used as received	1g	Ultrasonic	Acetonitrile

APPENDIX 4

Number of participants per country

1 lab in BANGLADESH
1 lab in BRAZIL
1 lab in FRANCE
5 labs in GERMANY
2 labs in HONG KONG
1 lab in INDIA
1 lab in INDONESIA
5 labs in ITALY
1 lab in JAPAN
4 labs in P.R. of CHINA
1 lab in PAKISTAN
1 lab in THAILAND
4 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
n.a.	= not applicable
n.d.	= not detected
n.e.	= not evaluated
f.r.	= first reported
W	= test result withdrawn on request of participant
ex	= test result excluded from the statistical evaluation

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