Results of Proficiency Test
OPP and Other Preservatives
in Textile
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Organized by: Institute for Interlaboratory Studies
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CONTENTS

1 INTRODUCTION

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 the determination of Ortho-Phenylphenol (OPP) in Textile every year. During the annual proficiency test program 2021/2022 it was decided to continue the proficiency test of OPP and other Preservatives in Textile.

In this interlaboratory study 27 laboratories in 14 different countries registered for participation. See appendix 4 for the number of participants per country. In this report the results of the OPP and Preservatives in Textile 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 analyzes 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 #21800.
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 blue jeans was selected which was made positive for OPP by a third party. The batch was cut into small pieces. After homogenization 50 small plastics bags were filled with approximately 3 grams each and labelled #21800.
The homogeneity of the subsamples was checked by determination of OPP with an in-house test method based on a KOH extraction on 10 stratified randomly selected subsamples.

	OPP in mg/kg
sample #21800-1	85.5
sample #21800-2	88.9
sample #21800-3	83.2
sample #21800-4	80.9
sample #21800-5	81.2
sample #21800-6	82.4
sample #21800-7	83.5
sample #21800-8	81.2
sample #21800-9	83.7
sample #21800-10	85.2

Table 1: homogeneity test results of subsamples #21800

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

	OPP in mg/kg
r (observed)	6.9
reference method	iis memo 1601
0.3 x R (reference method)	13.2

Table 2: evaluation of the repeatability of subsamples #21800

The calculated repeatability is in agreement with 0.3 times the reproducibility of the reference method. Therefore, homogeneity of the subsamples was assumed.
To each participating laboratory one sample textile labelled #21800 was sent on November 17, 2021.

2.5 ANALYZES

The participants were requested to determine on samples #21800 the concentrations of Ortho-Phenylphenol (OPP), 2-(Thiocyanomethylthio)-Benzothiazole (TCMTB), 4-Chloro-3-Methylphenol (PCMC), 2-Octylisothiazol-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 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/. 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 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. 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:

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

The $z_{(target)}$ scores are listed in the test result tables in appendix 1.

Absolute values for z<2 are very common and absolute values for z>3 are very rare.
The usual interpretation of z-scores is as follows:

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

4 EVALUATION

In this proficiency test no problems occurred with the dispatch of the samples. Two participants were not able to report any test results and all other participants reported before the final reporting date.
In total 25 laboratories reported 24 numerical test results. Two statistical outliers were observed, which is 8.3%. 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.

Unfortunately, a suitable reference test method providing the precision data is not available for the determination of OPP in Textile. Therefore, iis developed a target reproducibility based on iis PT data of OPP proficiency tests from 2004 until 2014. This means that the calculated reproducibility was compared against the estimated reproducibility calculated with a Horwitz-like equation as mentioned in iis memo 1601. This document can be downloaded from de iis website www.iisnl.com (see lit.13).

Ortho-Phenylphenol (OPP): Based on the analytical details two groups of test results could be identified. It appeared that test results obtained from Acrylonitrile and Alkaline extraction differ significantly (see also paragraph 4.2 and 4.4). Therefore, it was decided to evaluate both two groups separately.
For the group using Acrylonitrile extraction this determination may be problematic. One outlier was observed and one other test result was excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the estimated reproducibility calculated from iis memo 1601.
For the group using Alkaline extraction this determination may be problematic. One outlier was observed and two other test results were excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the estimated reproducibility calculated from iis memo 1601.

The concentrations of the other reported preservatives were near or below the detection limit. Therefore, no z-scores were calculated for these components. See appendix 2 for the reported test results.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the estimated target reproducibility 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 are presented in the next table.

Component	unit	n	average	2.8 * sd	R(target)
Ortho-Phenylphenol (OPP) ACN	mg/kg	12	18.7	18.4	12.3
Ortho-Phenylphenol (OPP) KOH	mg/kg	7	57.7	57.7	31.1

Table 3: performance evaluation sample #21800

Without further statistical calculations it can be concluded the participating laboratories have difficulties with the analysis of OPP. See also the discussion in paragraphs 4.1, 4.4 and 5.

4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2021 WITH PREVIOUS PTS

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

Component	December 2021	December 2020	December 2019	December 2018	December 2017	iis memo 1601
Ortho-Phenylphenol (OPP)	35-36%	16-29%	21%	16-58%	39-54%	20-33%

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-four participants reported analytical details. The following can be summarized:

- Nineteen participants were accredited in accordance with ISO/IEC17025 to determine the reported component(s) in textile.
- The samples were further cut prior to analysis by eighteen participants and six participants used the sample as received.
- Eight participants used a sample intake of 0.5-0.75 gram and fourteen participants used about 1 gram. Two participants used 1.5 grams or more.
- Ultrasonic extraction was the most often reported technique to release the components.
- Fourteen participants reported to use Acrylonitrile as extraction solvent and eight participants reported to use KOH for the extraction.

For the second time it is observed that the use of ACN or KOH as extraction solvent has a profound effect on the analysis of OPP content in the sample. In last year PT (iis20A17) this was also observed. And as in last PT, iis decided to evaluate both groups separately.
The other analytical details are not statistically significant given the small subgroups and the variation observed in this proficiency test.

5 DISCUSSION

All participants were able to detect OPP in sample #21800. However, the choice of the extraction solvent was of major influence on the observed OPP amount and thus on the decision to accept or reject the sample when the test results are compared to the Ecolabelling Standards and Requirements for Textiles in EU (see table 5 for the limits).

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

The reported test results for OPP extracted with ACN were much lower than extracted with KOH and in most cases lower than 50.0 mg/kg. Based on this the sample would have been accepted for all four classes mentioned in table 5 by all ACN reporting laboratories except for one laboratory.
The reported test results for OPP extracted with KOH were in most cases above 50.0 mg/kg. Based on this the sample would have been rejected for Class 1 by the KOH reporting laboratories except two laboratories who would have accepted the sample.

6 CONCLUSION

In this proficiency test Ortho-Phenylphenol (OPP) and other Preservatives in Textile were determined. The participating laboratories had some problems in determining OPP although the choice of extraction solvent is of significant influence. It is advised that members of the technical committee take this on-board to discuss and decide the best extraction method for Ortho-Phenylphenol (OPP) determination.

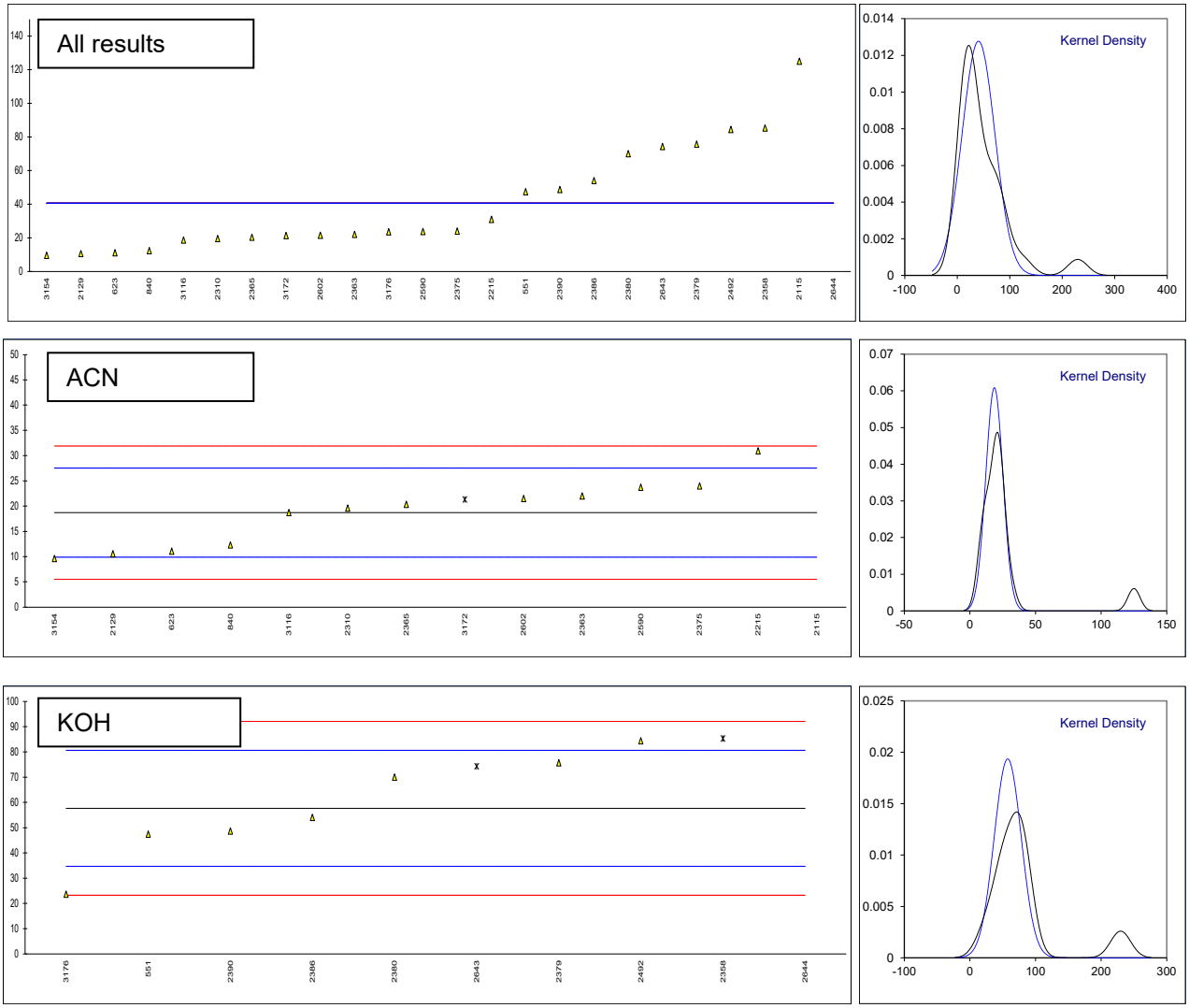
Each laboratory should evaluate its performance in this study and make decisions about possible corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

APPENDIX 1

Determination of Ortho-Phenylphenol (OPP) on sample #21800; results in mg/kg

lab	method	All	mark	z(targ)	ACN	mark	z(targ)	KOH	mark	z(targ)
551	In house	47.398		-----	-----		-----	47.398		-0.90
623	ISO13365	11.065		-----	11.065		-1.73	-----		-----
840	In house	12.3		-----	12.3		-1.45	-----		-----
2115	In house	125	C	-----	125	C,G(0.01)	24.15	-----		-----
2121		-----		-----	-----		-----	-----		-----
2129	ISO13365Mod.	10.564		-----	10.564		-1.85	-----		-----
2215	In house	30.91		-----	30.91		2.78	-----		-----
2265		-----		-----	-----		-----	-----		-----
2310	ISO13365	19.6		-----	19.6		0.21	-----		-----
2358		85.3		-----	-----		-----	85.3	ex	2.41
2363	ISO13365	22		-----	22		0.75	-----		-----
2365	ISO13365	20.34		-----	20.34		0.37	-----		-----
2375	EN17134	24		-----	24		1.21	-----		-----
2379	§64 LFGB B82.02.8	75.6606		-----	-----		-----	75.6606		1.57
2380	In house	70.009		-----	-----		-----	70.009		1.07
2386	In house	54.02		-----	-----		-----	54.02		-0.32
2390	In house	48.68		-----	-----		-----	48.68		-0.78
2492	In house	84.4		-----	-----		-----	84.4		2.33
2590	EN17134	23.713		-----	23.713		1.14	-----		-----
2602	In house/ISO13365-1	21.486		-----	21.486		0.64	-----		-----
2643		74.286		-----	-----		-----	74.286	ex	1.45
2644	In house	229.75	G(0.01)	-----	-----		-----	229.75	G(0.01)	15.00
3116	In house	18.70		-----	18.70		0.00	-----		-----
3154		9.598		-----	9.598		-2.07	-----		-----
3172		21.302		-----	21.302	ex	0.59	-----		-----
3176	In house	23.60		-----	-----		-----	23.60		-2.97
3210	EN13365	<40		-----	<40		-----	-----		-----
	normality	suspect			OK			OK		
	n	23			12			7		
	outliers	1			1 + 1ex			1 + 2ex		
	mean (n)	40.606			18.690			57.681		
	st.dev. (n)	31.2288	RSD = 77%		6.5557	RSD = 35%		20.6189	RSD =36%	
	R(calc.)	87.441			18.356			57.733		
	st.dev.(iis-memo 1601)	(8.5125)			4.4017			11.4719		
	R(iis-memo 1601)	(23.835)			12.325			32.121		

Lab 2115 first reported 106.1
Lab 2358 reported to have used Methanol as extraction solvent, therefore the test result was excluded for statistical evaluation.
Lab 2643 reported to have used Methanol as extraction solvent, therefore the test result was excluded for statistical evaluation
Lab 3172 did not report any analytical details; therefore the test result was excluded for statistical evaluation.


All results
3154 2129 623 840 3116 2310 2365 3172 2602 2363 3176 2590 2375 2215 551 2390 2386 2380 2643 2379 2492 2358 2115 2644
Kernel Density
ACN
3154 2129 623 840 3116 2310 2365 3172 2602 2363 2590 2375 2215 2115
Kernel Density
KOH
3176 551 2390 2386 2380 2643 2379 2492 2358 2644
Kernel Density

APPENDIX 2 Other reported test results

Determination of 2-(Thiocyanomethylthio)-Benzothiazole (TCMTB), 4-Chloro-3-Methylphenol (PCMC), 2-Octylisothiazol-3(2H)-one (OIT), Triclosan (TCS) and eventually other preservatives on sample #21800; in mg/kg

lab	TCMTB	PCMC	OIT	TCS	Other Preservatives
551	-----	-----	-----	-----	-----
623	not detected	not detected	not detected	not detected	not detected
840	not detected	not detected	not detected	not detected	-----
2115	-----	-----	-----	-----	-----
2121	-----	-----	-----	-----	-----
2129	-----	-----	-----	-----	-----
2215	Not detected	Not detected	Not detected	Not detected	Not detected
2265	-----	-----	-----	-----	-----
2310	Not detected	Not detected	Not detected	Not detected	Not detected
2358	not detected	not detected	not detected	not detected	not detected
2363	＜1	＜1	＜1	＜1	＜1
2365	<1.0	<1.0	<1.0	<1.0	-----
2375	-----	-----	-----	-----	-----
2379	Not tested	Not tested	Not tested	Not tested	Not tested
2380	-----	-----	-----	-----	-----
2386	-----	-----	<0,5	< 10	-----
2390	No capability	No capability	No capability	No capability	No capability
2492	-----	-----	-----	-----	-----
2590	-----	-----	-----	-----	-----
2602	-----	-----	-----	-----	-----
2643	-----	-----	-----	-----	-----
2644	-----	-----	-----	-----	-----
3116	not detected (<0.5) C	-----	-----	-----	-----
3154	-----	-----	-----	-----	-----
3172	< 5	< 5	< 5	< 1	-----
3176	-----	-----	-----	-----	-----
3210	<40	<40	<40	not analysed	not analysed

Lab 3116 first reported 22.39

APPENDIX 3 Analytical details

lab	ISO17025 accredited	Sample preparation	Sample intake (grams)	Extraction technique	Extraction solvent
551	Yes	Further cut	1	Alkaline Digestion	KOH followed by n-Hexane
623	Yes	Further cut	1	Ultrasonic	Acetonitrile
840	Yes	Further cut	0.5	Ultrasonic	Acetonitrile
2115	No	Used as received	1g	Ultrasonic	Acetonitrile
2121	---	---		---	---
2129	No	Further cut	0.5 g	Ultrasonic	Acetonitrile
2215	Yes	Further cut	1.0 gram	Ultrasonic	Acetonitrile
2265	---	---		---	---
2310	Yes	Further cut	1	Ultrasonic	Acetonitrile
2358	Yes	Used as received	0.5 grams	Ultrasonic	Methanol
2363	Yes	Further cut	about 1g	Ultrasonic	Acetonitrile
2365	Yes	Further cut	0.5g	Ultrasonic	Acetonitrile
2375	Yes	Further cut	0.5g	Ultrasonic	Acetonitrile
2379	No	Further cut	0.5 gram	Ultrasonic	KOH followed by n-Hexane
2380	Yes	Further cut	1.0 g	Alkaline Digestion	KOH followed by n-Hexane
2386	Yes	Further cut	1g	Ultrasonic	KOH (OPP) Methanol (other substances)
2390	Yes	Further cut	1.0 gram	Ultrasonic	KOH
2492	Yes	Further cut	0.5g	Alkaline Digestion	KOH
2590	No	Used as received	1g	Ultrasonic	Acetonitrile
2602	No	Further cut	0,75 g / 20ml ACN	Ultrasonic	Acetonitrile
2643	Yes	Further cut	2 g	Ultrasonic	Methanol
2644	Yes	Used as received	2 G	Ultrasonic	KOH followed by n-Hexane
3116	No	Used as received	1 gram	Ultrasonic	Acetonitrile
3154	Yes	Used as received	1	Ultrasonic	Acetonitrile
3172	Yes	---		---	---
3176	Yes	Further cut	1	Ultrasonic	KOH followed by n-Hexane
3210	Yes	Further cut	1 g	Ultrasonic	Acetonitrile

APPENDIX 4

Number of participants per country

1 lab in BANGLADESH
1 lab in BRAZIL
2 labs in FRANCE
5 labs in GERMANY
3 labs in HONG KONG
1 lab in INDIA
1 lab in INDONESIA
4 labs in ITALY
1 lab in KOREA, Republic of
3 labs in P.R. of CHINA
1 lab in PAKISTAN
1 lab in THAILAND
2 labs in TURKEY
1 lab 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

Literature

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