Institute for
Interlaboratory Studies

Results of Proficiency Test
OPP and other Preservatives
in Leather/Footwear
May 2023

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 1990 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 leather there are some Ecolabelling schemes imposing environmental requirements for textile and leather products on a voluntary basis. Well-known Ecolabelling organizations are OEKO-TEX® and Bluesign®.

Since 2018 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of Ortho-Phenylphenol (OPP) and other Preservatives in Leather/Footwear every year. During the annual proficiency testing program 2022/2023 it was decided to continue the proficiency test for the determination of OPP and other Preservatives in Leather/Footwear.

In this interlaboratory study 27 laboratories in 14 countries registered for participation, see appendix 4 for the number of participants per country. In this report the results of the OPP and other Preservatives in Leather/Footwear 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 the 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 leather sample of 3 grams labelled #23590.
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 organisation 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 white leather containing OPP was obtained from a local supplier. The batch was grinded and after homogenization 60 plastic bags were filled with approximately 3 grams each and labelled #23590.
The homogeneity of the subsamples was checked by determination of OPP according to ISO13665-1 on 8 stratified randomly selected subsamples.

	OPP in mg/kg
sample #23590-1	321.7
sample #23590-2	333.1
sample #23590-3	312.8
sample #23590-4	335.6
sample #23590-5	345.5
sample #23590-6	353.7
sample #23590-7	330.4
sample #23590-8	345.2

Table 1: homogeneity test results of subsamples #23590

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)	37.6
reference method	iis memo 1601
0.3 x R (reference method)	43.0

Table 2: evaluation of the repeatability of subsamples #23590

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 of the participants one sample labelled #23590 was sent on April 19, 2023.

2.5 ANALYZES

The participants were requested to determine the concentrations of Ortho-Phenylphenol (OPP), 2-(Thiocyanomethylthio)-Benzothiazole (TCMTB), 4-Chloro-3-Methylphenol (PCMC), 2-Octylisothiazol-3(2H)-one (OIT), Triclosan (TCS) and Other Preservatives.
To ensure homogeneity it was requested not to use less than 0.5 gram per determination.
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, but not to age nor dry the sample nor to determine volatile matter. The amount of sample was not sufficient to allow aging and/or determine the volatile matter content.
It was requested to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible.
It was also requested not to report ‘less than’ test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kmpd.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 the data analysis and the original 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 wast 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's 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 uncertainly 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 were encountered with the dispatch of the samples. Three participants reported test results after the final reporting date and one other participant did not report any test results. Not all participants were able to report all tests requested. In total 26 participants reported 50 numerical test results. No outlying test results were observed. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

Both data sets proved to have a normal Gaussian distribution.

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 official test method for the determination of total Preservatives content in Leather/Footwear is considered to be test method ISO13365-1. There is also a second method ISO13365-2, which is a test method for perspirated preservatives in Leather/Footwear. Regretfully both test methods do not provide precision data for OPP or any other preservatives. When no test method reproducibility is known the target reproducibility is in general estimated using the Horwitz equation. However, in 2016 iis investigated the reproducibilities of the determination of OPP in textile over 18 determinations in iis PTs conducted from 2004 until 2014. It was observed in these PTs that the estimated reproducibility based on the Horwitz equation was very strict. Therefore, a new target reproducibility on base of the iis PTs was determined and described in iis memo 1601. Although iis memo 1601 is based on iis PTs of OPP in Textile it is decided to use the estimated iis target reproducibility also for the determination of OPP in Leather. Furthermore, it is decided to use the estimated iis target reproducibility for other preservatives determined in Leather as well.

Ortho-Phenylphenol (OPP): This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the target reproducibility as derived from iis memo 1601.

4-Chloro-3-Methylphenol (PCMC): This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the target reproducibility as derived from iis memo 1601.

The majority of the participants agreed on a concentration near or below the limit of detection for all other requested Preservatives mentioned in paragraph 2.5. Therefore, no z-scores are calculated for these components. The reported test results can be found in appendix 2.

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 * standard deviation) and the target reproducibility derived from previous iis PTs are presented in the next table.

Component	unit	n	average	2.8 * sd	R(target)
Ortho-Phenylphenol (OPP)	mg/kg	26	299	116	130
4-Chloro-3-Methylphenol (PCMC)	mg/kg	24	330	85	141

Table 3: reproducibilities of preservatives measured in sample #23590

Without further statistical calculations it can be concluded that there is a good compliance of the group of participants with the target reproducibility of the reference method.

4.3 COMPARISON OF THE PROFICIENCY TEST OF MAY 2023 WITH PREVIOUS PTS

	May 2023	April 2022	May 2021	May 2020	May 2019
Number of reporting laboratories	26	37	34	32	38
Number of test results	50	91	102	59	89
Number of statistical outliers	0	0	5	0	5
Percentage of statistical outliers	0%	0%	4.9%	0%	5.6%

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 tests was compared, expressed as relative standard deviation (RSD) of the PTs, see next table.

Component	May 2023	April 2022	May 2021	May 2020	2019 - 2018	Target *)
OPP	14%	16%	14%	15%	21-23%	14-24%
TCMTB	---	---	30%	---	---	14-24%
PCMC	9%	14%	16%	26%	15-26%	14-24%
OIT	---	28%	25%	---	39%	14-24%

Table 5: development of uncertainties over the years

*) Concentration range 600-15 mg/kg respectively

The uncertainty of OPP observed in this PT is in line with previous iis PTs. The uncertainty of PCMC has been improved.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

Test method ISO13365-1:2020 describes an Acetonitrile solvent extraction for the determination of the solvent extractible content of the preservatives in leather by liquid chromatography. Test method ISO13365-2:2020 describes a test method by artificial perspiration solution aqueous extraction for the determination of the aqueous extractable preservatives in leather by liquid chromatography.
Almost all (88%) of the reporting participants mentioned to use ISO13365-1:2020 and 12% mentioned to use an inhouse method. None of the participants mentioned to use test method ISO13365-2:2020.

Some analytical details were requested, see appendix 3 for the reported answers. Based on the answers given by the participants the following can be summarized:
- 85% of the participants mentioned that they are accredited for the determination of the reported components.
- 72% of the participants used the sample as received.
- Allmost all participants did use a sample intake of 0.5 grams to 1 gram.

As the majority of the group participants follow the same analytical procedures and the performances of the determinations are in line with the target reproducibilities no separate statistical analysis has been performed.

5 DISCUSSION

According to the OEKO-TEX® Ecolabelling Standard and Requirements for leathers in the EU (see Table 6) almost all participants would have rejected the sample for class Baby for OPP. Three participants would have rejected the sample for class Baby only. For the other classes the sample would have been accepted for OPP by all participants.
For PCMC almost all participants would have rejected the sample for all classes. Three participants would have rejected the sample for class Baby only.

Component	Baby in mg/kg	Direct skin contact in mg/kg	No direct skin contact in mg/kg	Decoration material in mg/kg
OPP	<250	<750	<750	<750
TCMTB	<250	<500	<500	<500
PCMC	<150	<300	<300	<300
OIT	<50	<100	<100	<100

Table 6: OEKO-TEX® Ecolabelling Standard and Requirements for leathers in EU

Bluesign meantions two lists regarding chemicals of concern; A Bluesign® Systems Substances List (BSSL) and the Bluesign® Restricted List (RSL). The BSSL contains all chemicals that are restricted or suspected to restricted and are therefore monitored. The RSL is an extract of the BSSL and contains the restricted chemicals with consumer safety limits. The Bluesign RSL mentions only a safety limit for OPP (see Table 7). For OPP all participants would have rejected this sample for all classes.

Component	Class A Next to skin and Baby mg/kg	Class B Occasional skin contact mg/kg	Class C No skin contact mg/kg
OPP	<50	<100	<200

Table 7: Product classes specific limit values, Bluesign® RSL list v13.0

6 CONCLUSION

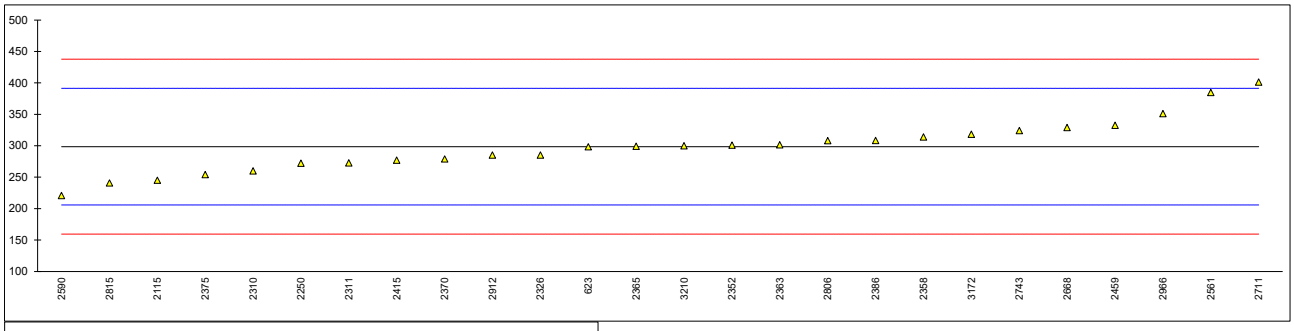
It can be concluded that the majority of the participants have no problems with the determination of OPP and other Preservatives in the sample in this PT.

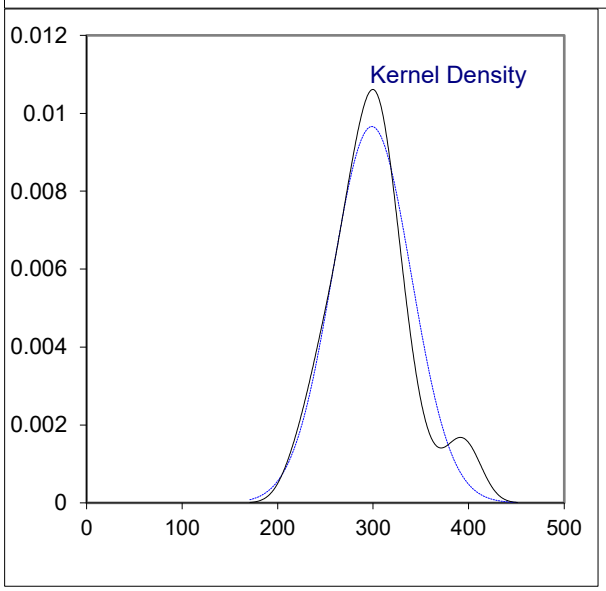
Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

APPENDIX 1

Determination of Ortho-Phenylphenol (OPP) on sample #23590; results in mg/kg

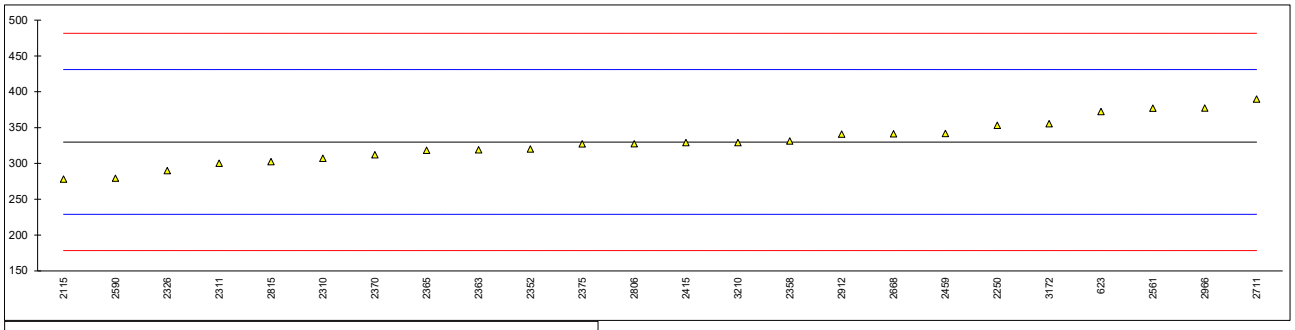
lab	method	value	mark	z(targ)	remarks
551		-----		-----	
623	ISO13365-1:2020	298.33		-0.01	
2115	ISO13365-1:2020	245		-1.15	
2250	ISO13365-1:2020	272		-0.57	
2310	ISO13365-1:2020	260		-0.83	
2311	ISO13365-1:2020	272.57		-0.56	
2326	ISO13365-1:2020	285.26		-0.29	
2352	ISO13365-1:2020	300.8		0.05	
2358	ISO13365-1:2020	314		0.33	
2363	ISO13365-1:2020	301.3		0.06	
2365	ISO13365-1:2020	299.15		0.01	
2370	ISO13365-1:2020	279		-0.42	
2375	ISO13365-1:2020	254		-0.96	
2386	In house	308.310		0.21	
2415	ISO13365-1:2020	277		-0.46	
2459	ISO13365	332.45		0.73	
2561	ISO13365-1:2020	384.80		1.86	
2590	ISO13365-1:2020	220.706		-1.68	
2668	In house	329.03		0.66	
2711	In house	401.3		2.21	
2743	ISO13365-1:2020	324.11		0.55	
2806	ISO13365-1:2020	308.2		0.21	
2815	ISO13365-1:2020	240.7		-1.25	
2912	ISO13365-1:2020	285.102		-0.29	
2966	ISO13365-1:2020	351.18		1.13	
3172	ISO13365-1:2020	318.36		0.43	
3210	ISO13365-1:2020	300		0.03	
	normality	OK			
	n	26			
	outliers	0			
	mean (n)	298.564			
	st.dev. (n)	41.3093	RSD = 14%		
	R(calc.)	115.666			
	st.dev.(iis memo 1601)	46.4023			
	R(iis memo 1601)	129.926			

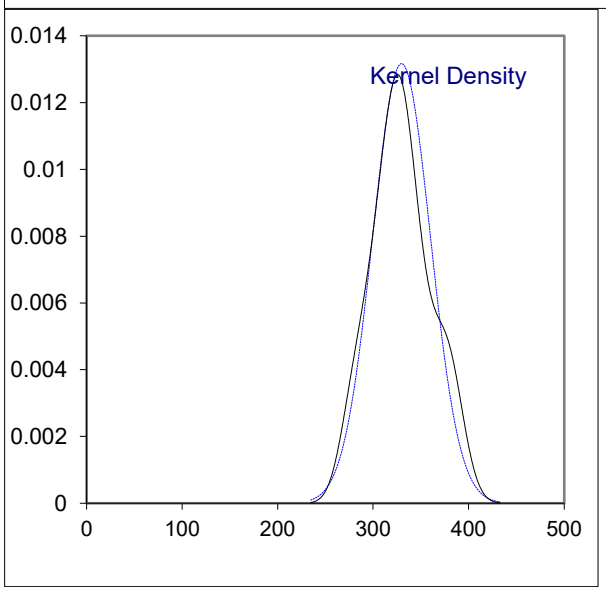




Determination of 4-Chloro-3-Methylphenol (PCMC) on sample #23590; results in mg/kg

lab	method	value	mark	z(targ)	remarks
551		-----		-----	
623	ISO13365-1:2020	372.27		0.84	
2115	ISO13365-1:2020	278		-1.03	
2250	ISO13365-1:2020	353		0.46	
2310	ISO13365-1:2020	307		-0.45	
2311	ISO13365-1:2020	300.22		-0.59	
2326	ISO13365-1:2020	289.75		-0.79	
2352	ISO13365-1:2020	320.1		-0.19	
2358	ISO13365-1:2020	331		0.02	
2363	ISO13365-1:2020	318.8		-0.22	
2365	ISO13365-1:2020	318.15		-0.23	
2370	ISO13365-1:2020	312		-0.35	
2375	ISO13365-1:2020	327		-0.06	
2386	In house	not analyzed		-----	
2415	ISO13365-1:2020	329		-0.02	
2459	ISO13365	341.66		0.23	
2561	ISO13365-1:2020	376.92		0.93	
2590	ISO13365-1:2020	279.149		-1.00	
2668	In house	341.23		0.23	
2711	In house	389.6		1.18	
2743		-----		-----	
2806	ISO13365-1:2020	327.2		-0.05	
2815	ISO13365-1:2020	302.4		-0.54	
2912	ISO13365-1:2020	340.733		0.22	
2966	ISO13365-1:2020	377.28		0.94	
3172	ISO13365-1:2020	355.29		0.50	
3210	ISO13365-1:2020	329		-0.02	
	normality	OK			
	n	24			
	outliers	0			
	mean (n)	329.865			
	st.dev. (n)	30.3003	RSD = 9%		
	R(calc.)	84.841			
	st.dev.(iis memo 1601)	50.5060			
	R(iis memo 1601)	141.417			





APPENDIX 2 Other reported Preservatives

lab	2-(Thiocyanomethylthio)-Benzothiazole (TCMTB)	2-Octylisothiazol-3(2H)-one (OIT)	Triclosan	Other Preservatives
551	-----	-----	-----	-----
623	not detected	not detected	not detected	not detected
2115	1.1	-----	99.9	-----
2250	< 5	< 5	< 5	-----
2310	not detected	not detected	not detected	not detected
2311	Not Detected	Not Detected	Not Detected	Not Detected
2326	1.39	ND	ND	-----
2352	-----	-----	-----	-----
2358	not detected	not detected	not detected	not detected
2363	not detected	not detected	not detected	not analyzed
2365	＜1.0	＜1.0	＜1.0	-----
2370	<2	<2	<2	<2
2375	-----	-----	-----	-----
2386	not analyzed	not analyzed	not analyzed	not analyzed
2415	not detected	not detected	not detected	-----
2459	9.38	Not Detected	Not Performed	Nil
2561	-----	-----	-----	-----
2590	-----	-----	-----	-----
2668	Not detected	Not detected	Not detected	Not detected
2711	Not detected	Not detected	Not analyzed	-----
2743	-----	-----	Not detected	-----
2806	< 10,0	< 10,0	-----	-----
2815	< 20.0	< 30.0	-----	-----
2912	-----	-----	-----	-----
2966	1.68	NOT DETECTED	NOT ANALYZED	NOT ANALYZED
3172	< 5	< 5	-----	-----
3210	<40	<40	-----	-----

APPENDIX 3 Analytical details

lab	ISO 17025 accr.	sample preparation	sample intake (g)
551	-----	-----	-----
623	Yes	Further cut	1
2115	No	Used as received	0.5 g
2250	Yes	Used as received	0.25
2310	Yes	Used as received	1
2311	Yes	Further cut	1
2326	Yes	Used as received	0.5 g
2352	Yes	Used as received	3.05
2358	Yes	Used as received	0.5g
2363	Yes	Further cut	1g
2365	Yes	Used as received	1.0g
2370	Yes	Used as received	1g
2375	Yes	Used as received	0,5 gram
2386	Yes	Further cut	0.5 g
2415	Yes	Used as received	0.5 gram
2459	No	Further cut	1.00 gram
2561	Yes	Used as received	1g
2590	Yes	Used as received	1g
2668	Yes	Used as received	0.5 g
2711	No	Used as received	1,0
2743	Yes	Further cut	1
2806	Yes	Further cut	1,000
2815	Yes	Used as received	1 g
2912	No	Used as received	0.5g
2966	Yes	Used as received	1.010
3172	Yes	-----	
3210	Yes	Used as received	1g

APPENDIX 4

Number of participants per country

1 lab in BRAZIL
1 lab in FRANCE
2 labs in GERMANY
1 lab in HONG KONG
3 labs in INDIA
1 lab in INDONESIA
8 labs in ITALY
3 labs in P.R. of CHINA
2 labs in PAKISTAN
1 lab in SWITZERLAND
1 lab in TAIWAN
1 lab in TURKEY
1 lab in UNITED KINGDOM
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
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
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