Results of Proficiency Test
Chlorinated Phenols in Textile
December 2021

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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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 Chlorinated Phenols in Textile every year. During the annual proficiency test program 2021/2022 it was decided to continue the proficiency test of Chlorinated Phenols in Textile.

In this interlaboratory study 75 laboratories in 25 different countries registered for participation. See appendix 4 for the number of participants per country. In this report the results of the Chlorinated Phenols 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 positive on Chlorinated Phenols labelled #21805.
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 a black knitted cotton with a rib pattern was obtained from a third party. The batch was cut into small pieces and after homogenization 96 small plastics bags were filles with approximately 3 grams each and labelled #21805.
The homogeneity of the subsamples was checked by determination of Pentachlorophenol (PCP) in accordance with an in-house test method on 8 stratified randomly selected subsamples.

	Pentachlorophenol in mg/kg
sample #21805-1	5.71
sample #21805-2	5.96
sample #21805-3	6.48
sample #21805-4	6.09
sample #21805-5	6.31
sample #21805-6	5.86
sample #21805-7	5.76
sample #21805-8	5.80

Table 1: homogeneity test result of subsamples #21805

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.

	Pentachlorophenol in mg/kg
r (observed)	0.78
reference method	iis memo 1601 (see lit. 13)
0.3 x R (reference method)	1.41

Table 2: evaluation of the repeatability of subsamples #21805

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 participating laboratories one sample of #21805 was sent on November 17, 2021.

2.5 ANALYZES

The participants were requested to determine the concentrations of Pentachlorophenol (PCP), Tetrachlorophenols, Trichlorophenols and other Chlorinated Phenols.
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-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 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 dataset 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) test 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 test can be used. For larger data sets (above 20test 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 were encountered with the dispatch of the samples. Two participants did not report any test results and one other participant reported the test results after the final reporting date.

In total 73 laboratories reported 73 numerical test results. Observed were 3 outlying test results, which is 4.1%. In proficiency tests 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 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 in appendix 1 together with the original data. The abbreviations, used in these tables, are explained in appendix 5.

Unfortunately, a suitable reference test method providing the precision data is not available for the determination of Chlorinated Phenols in Textile. Therefore, iis developed a target reproducibility based on iis PT data of PCP 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).

Pentachlorophenol (PCP): This determination was not problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated reproducibility derived from iis memo 1601 (see lit. 13).

The concentrations of the other reported chlorinated phenols 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)
Pentachlorophenol	mg/kg	70	5.06	2.97	4.06

Table 3: reproducibility of component on sample #21805

Without further statistical calculations, it can be concluded that the group of participating laboratories have no difficulties with the analyzes of PCP in textile. See also the discussion in paragraphs 4.1 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 similar in comparison with the uncertainties observed in previous PTs, see the table below.

Component	December 2021	December 2020	December 2019	December 2018	2014 - 2016	Target
Pentachlorophenol	21%	16%	25%	26%	26-45%	29%
2,3,4,5-Tetrachlorophenol	n.e.	16%	n.e.	n.e.	n.e.	29%
2,3,4,6-Tetrachlorophenol	n.e.	n.e.	24%	n.e.	n.e.	29%

Table 4: comparison of uncertainties in iis proficiency tests

4.4 EVALUATION OF ANALYTICAL DETAILS

The reported analytical details from the participants are listed in appendix 3. About 84% of the reporting laboratories reported to be accredited for the determination of Chlorinated Phenols in textile.
The amount of sample intake varied between 0.5 and 3 grams, about 85% of the reporting laboratories used between 0.5 and 1 gram.
Prior to analysis the samples were further cut by about 76% of the participants while 22% of the other participants reported to use the sample as received.
Ultrasonic extraction and mechanical shaking were most often reported techniques for extraction by the participants, respectively 39% and 23%.

It appeared that the effect of the analytical details on the determination of PCP is small and not statistically significant.

5 DISCUSSION

When the test results of this interlaboratory study were compared to the Ecolabelling Standards and Requirements for Textiles in EU (see table 5) it could be noted that all participants were able to detect PCP in the sample and would have rejected the PT sample for all Ecolabel classes.

Ecolabel	Class 1 Baby clothes	Class 2 Clothes direct skin contact	Class 3 Clothes, no direct contact with skin	Class 4 Decoration material
Pentachlorophenol (mg/kg)	0.05	0.5	0.5	0.5

Table 5: Ecolabelling Standards and Requirements for Textiles in EU

6 CONCLUSION

In this proficiency test the Pentachlorophenol content was determined. The variation observed for PCP in sample #21805 is in line with previous iis proficiency tests.

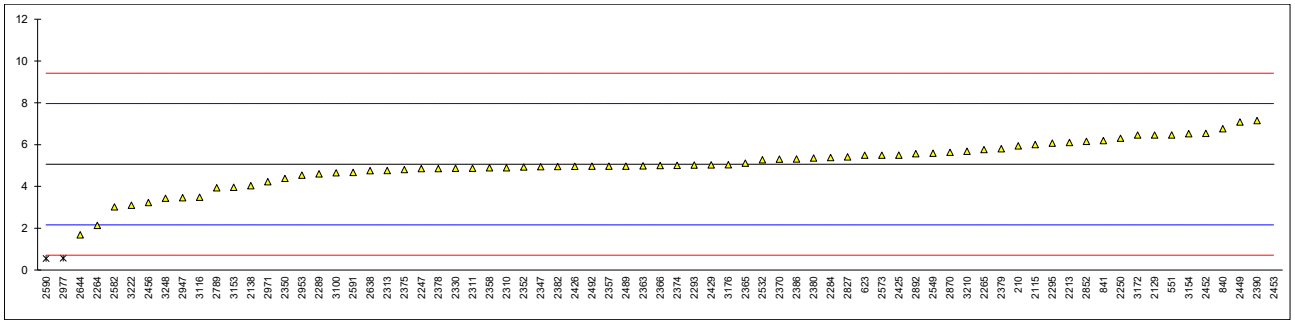
Each laboratory should evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

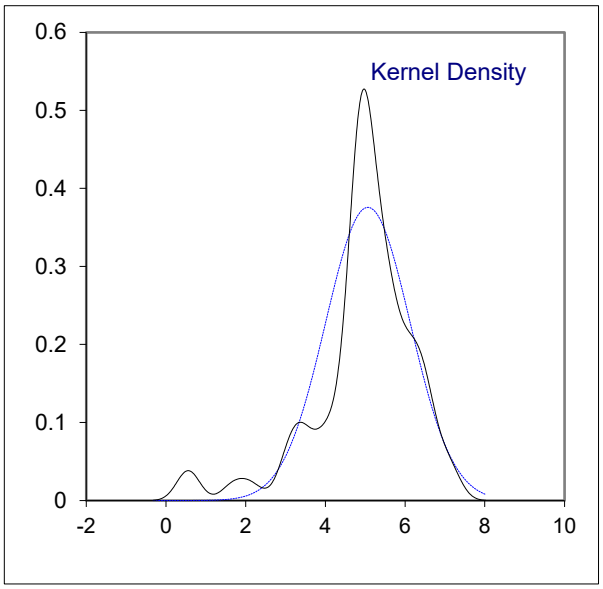
APPENDIX 1

Determination of Pentachlorophenol (PCP) on sample #21805; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	LFGB B82.02.8	5.94		0.60	
551	LFGB B82.02.8	6.465		0.96	
623	LFGB B82.02.8	5.489		0.29	
840	LFGB B82.02.8	6.76		1.17	
841	LFGB B82.02.8	6.191		0.78	
2115	LFGB B82.02.8	6.0	C	0.64	first reported 8.44
2129		6.458		0.96	
2138	KS K0733	4.031	C	-0.71	first reported 1.968
2213	In house	6.1		0.71	
2247	LFGB B82.02.8	4.85		-0.15	
2250	In house	6.3		0.85	
2264	LFGB B82.02.8	2.13		-2.02	
2265		5.758		0.48	
2284	LFGB B82.02.8	5.381		0.22	
2289	LFGB B82.02.8	4.60		-0.32	
2293	LFGB B82.02.8	5.0154		-0.03	
2295	In house	6.07		0.69	
2310	LFGB B82.02.8	4.89		-0.12	
2311	LFGB B82.02.8	4.866		-0.14	
2313	LFGB B82.02.8	4.76		-0.21	
2330	LFGB B82.02.8	4.864		-0.14	
2347	In house	4.94		-0.09	
2350	In house	4.379		-0.47	
2352	In house	4.925		-0.10	
2357	LFGB B82.02.8	4.970		-0.07	
2358	LFGB B82.02.8	4.889		-0.12	
2363	In house	4.981		-0.06	
2365	In house	5.1141		0.03	
2366	ISO17070	5.0		-0.04	
2370	In house	5.30		0.16	
2374	LFGB B82.02.8	5.002		-0.04	
2375	In house	4.8		-0.18	
2378	GB/T18414-1	4.85		-0.15	
2379	LFGB B82.02.8	5.7988		0.51	
2380	LFGB B82.02.8	5.358		0.20	
2382	LFGB B82.02.8	4.951		-0.08	
2386	In house	5.31		0.17	
2390	In house	7.15		1.44	
2425	LFGB B82.02.8	5.498		0.30	
2426	In house	4.958		-0.07	
2429		5.027		-0.03	
2449	LFGB B82.02.8	7.08		1.39	
2452	XPG08-015	6.532		1.01	
2453	LFGB B82.02.8	1698.1	C,R(0.01)	1166.85	first reported 1.79
2456	UNI11057	3.225		-1.27	
2489	LFGB B82.02.8	4.97		-0.07	
2492	In house	4.967		-0.07	
2532	LFGB B82.02.8	5.27		0.14	
2549	LFGB B82.02.8	5.583		0.36	
2561		-----		-----	
2573	LFGB B82.02.8	5.49		0.29	
2582	In house	3.019		-1.41	
2590	LFGB B82.02.8	0.547	R(0.01)	-3.11	
2591	ISO17070	4.670		-0.27	
2638	In house	4.75		-0.22	
2644	In house	1.68		-2.33	
2678		-----		-----	
2789	In house	3.928		-0.78	
2827	In house	5.411		0.24	
2852	In house	6.15		0.75	
2870	In house	5.63		0.39	
2892	LFGB B82.02.8	5.562		0.34	
2947	In house	3.46	C	-1.11	first reported 1.299
2953	In house	4.54	C	-0.36	first reported 11.11
2971	DIN50009	4.22		-0.58	
2977	ISO17070/UNI11057	0.5667	C,R(0.01)	-3.10	first reported 2.1911
3100	LFGB B82.02.8	4.650		-0.29	
3116	LFGB B82.02.8	3.483		-1.09	

lab	method	value	mark	z(targ)	remarks
3153	LFGB B82.02.8	3.95		-0.77	
3154	DIN50009	6.52		1.00	
3172	UNI11057	6.4525		0.96	
3176	In house	5.03		-0.02	
3210	In house	5.68		0.42	
3222	UNI11057	3.097		-1.36	
3248	LFGB B82.02.8	3.43		-1.13	
	normality	suspect			
	n	70			
	outliers	3			
	mean (n)	5.065			
	st.dev. (n)	1.0618	RSD = 21%		
	R(calc.)	2.973			
	st.dev.(iis memo 1601)	1.4509			
	R(iis memo 1601)	4.063			





APPENDIX 2 Other reported test results

2345-TeCP = 2,3,4,5-Tetrachlorophenol
2346-TeCP = 2,3,4,6-Tetrachlorophenol
2356-TeCP = 2,3,5,6-Tetrachlorophenol
234-TCP = 2,3,4-Trichlorophenol
235-TCP = 2,3,5-Trichlorophenol
236-TCP = 2,3,6-Trichlorophenol
245-TCP = 2,4,5-Trichlorophenol
246-TCP = 2,4,6-Trichlorophenol
345-TCP = 3,4,5-Trichlorophenol
Other = Other Chlorinated Phenols

Determination individual and other Chlorinated Phenols on sample #21805; in mg/kg

lab	2345-TeCP	2346-TeCP	2356-TeCP	234-TCP	235-TCP	236-TCP	245-TCP	246-TCP	345-TCP	Other
210	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
551	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
623	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.
840	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	-----
841	<0.125	<0.125	<0.125	<0.125	<0.125	<0.125	<0.125	<0.125	<0.125	<0.125
2115	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2129	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2138	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
2213	0	0	0	0	0	0	0	0	0	0
2247	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.
2250	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2264	0.24	0.24	0.24	< 0.24	<0.24	<0.24	<0.24	<0.24	<0.24	<0.24
2265	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05
2284	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2289	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2293	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.
2295	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2310	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.
2311	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.
2313	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.
2330	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.
2347	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	-----
2350	< 0.125	< 0.125	< 0.125	< 0.125	< 0.125	< 0.125	< 0.125	< 0.125	< 0.125	< 0.125
2352	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2358	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.
2363	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.
2365	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2366	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	out cap
2370	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2374	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.
2375	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2378	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2379	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	-----
2380	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2382	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05
2386	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
2390	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.
2425	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2426	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.
2429	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.
2449	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2452	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	-----
2453	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2456	Not Det.	Not Det.	Not Det.	-----	-----	-----	-----	-----	-----	-----
2489	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
2492	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2532	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.
2549	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.
2561	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2573	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.
2582	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	-----
2590	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----

lab	2345-TeCP	2346-TeCP	2356-TeCP	234-TCP	235-TCP	236-TCP	245-TCP	246-TCP	345-TCP	Other
2591	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	-----
2638	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.
2644	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.
2678	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2789	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2827	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.
2852	<LQ=0.05	<LQ=0.05	<LQ=0.05	<LQ=0.05	<LQ=0.05	<LQ=0.05	<LQ=0.05	<LQ=0.05	<LQ=0.05	<LQ=0.05
2870	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2892	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2947	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	<0.05	<0.05	<0.05	< 0.05
2953	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
2971	0.007	-----	0.011	-----	-----	-----	-----	-----	-----	-----
2977	< LOQ	Not Det.	< LOQ	-----	-----	-----	-----	-----	-----	-----
3100	＜0.5	＜0.5	＜0.5	＜0.5	＜0.5	＜0.5	＜0.5	＜0.5	＜0.5	-----
3116	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3153	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3154	0.28	0	0	0	0	0	0	0	0	0
3172	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	-----
3176	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3210	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
3222	-----	-----	-----	-----	-----	-----	-----	-----	-----	-----
3248	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.	Not Det.

APPENDIX 3 Analytical details

lab	ISO/IEC17025 accredited	Sample	Sample intake (grams)	Extraction technique	Extraction solution
210	Yes	---		---	
551	Yes	Further cut	1	Ultrasonic	KOH
623	Yes	Further cut	1 gram	Ultrasonic	n-Hexane
840	Yes	Further cut	0.5	Ultrasonic	KOH
841	Yes	Further cut	2 grams	Ultrasonic	Potassium hydroxide 1M (KOH 1M)
2115	Yes	Used as received	1 g	Other	Oven
2129	Yes	Used as received	~0,5 g	ASE	acetic acid in acetone
2138	Yes	Further cut	about 0.5 g	Ultrasonic	potassium hydroxide(KOH)
2213	Yes	Further cut	2	Mechanical Shaking	n-Hexane
2247	Yes	Further cut	0.5gm	Mechanical Shaking	n-hexane
2250	Yes	Used as received	0,5	Ultrasonic	n-hexane
2264	No	Further cut	0.5	---	
2265	Yes	Further cut	0,27g	Mechanical Shaking	KOH K2CO3 n-Hexane acetic anhydride
2284	Yes	Further cut	1g	Mechanical Shaking	N-hexane
2289	Yes	Further cut	1.0g	Other	water steam distillation with sulphuric acid
2293	Yes	Further cut	1 gram	owen during 16 hours	KOH 1 Molar
2295	Yes	Further cut	1 gram	Mechanical Shaking	KOH, n-hexane
2310	Yes	Further cut	2	Steam Distillation	Hexane
2311	Yes	Further cut	1	Ultrasonic	Alkaline digestion (1M KOH)
2313	Yes	Further cut	1.0g	Steam Distillation	n-Hexane
2330	No	Further cut	2 grams	Ultrasonic	0.1 M KOH
2347	---	Further cut	1.0g	Ultrasonic	Hexane
2350	No	Further cut	2.0142 g	Ultrasonic	KOH
2352	Yes	Further cut	0.5g	Mechanical Shaking	Hexane
2357	---	---		---	
2358	Yes	Used as received	1 gram	Incubation in oven and then methanol shaking	potassium hydroxide solution, liquid-liquid extraction with hexane
2363	Yes	Further cut	about 1g	Ultrasonic, Oven	KOH/n-hexane
2365	Yes	Further cut	1.0g	Other	1mol/L KOH
2366	No	Further cut	0.5g	Mechanical Shaking	1mol/L H2S04
2370	Yes	Further cut	1 g	Mechanical Shaking	Hexane
2374	Yes	Further cut	1g sample	Ultrasonic	Hexane
2375	Yes	Further cut	1g	Ultrasonic	KOH Solution
2378	Yes	Further cut	0.5g	Ultrasonic	n-hexan
2379	No	Further cut	0.5 gram	Modified KOH Method	KOH
2380	Yes	Further cut	1.0 g	Alkaline digestion	n-Hexane
2382	Yes	Further cut	0.5g	steam distillation	n-hexane
2386	Yes	Further cut	0,5g	Ultrasonic	KOH
2390	Yes	Further cut	1.0 gram	---	KOH
2425	Yes	Further cut	0.5 g	Ultrasonic	KOH AND n-Hexane
2426	Yes	Further cut	1.0g	KOH Ectraction (90°C)	KOH Solution
2429	Yes	Further cut	0.5g	Mechanical Shaking	hexane
2449	No	Further cut	1 gram	Mechanical Shaking	n hexane
2452	No	Used as received	1	Ultrasonic	K2CO3 (15g/L)
2453	---	---		---	
2456	Yes	Used as received	All sample provided	Ultrasonic	Potassium carbonate 1.5%
2489	Yes	Further cut	1.0010g	Disdillation	1m H2SO4/n-Hexane
2492	Yes	Further cut	0.5g	Mechanical Shaking/microwave extraction	KOH(1M)
2532	---	---		---	
2549	Yes	Further cut	1.0	Mechanical Shaking	n-Hexane
2561	---	---		---	

lab	ISO/IEC17025 accredited	Sample	Sample intake (grams)	Extraction technique	Extraction solution
2573	Yes	Used as received	1g	ASE	acetone
2582	Yes	Further cut	1.0072g	Mechanical Shaking/KOH extraction	n-hexane
2590	Yes	Used as received	0.5g	Mechanical Shaking	
2591	Yes	Further cut	1.0 gram	Other	Technique used to release/extract the Chlorinated Phenols: Basic Digestion
2638	No	Further cut	1 gm	Ultrasonic	Hexane
2644	Yes	Used as received	2 g	Ultrasonic	KOH
2678	---	---		---	
2789	Yes	Used as received	1.03	Oven	dissolution KOH
2827	Yes	Further cut	0.5g	Mechanical Shaking	Hexane
2852	Yes	Used as received	0.5 g	Ultrasonic	K2CO3
2870	Yes	Further grinded	1 gm	Microwave extraction	Isooctane
2892	Yes	Further cut	1.0	16h / 90 °C	KOH/N-Hexane
2947	No	Used as received	1	Microwave	KOH 2M
2953	Yes	Further cut	1	Ultrasonic	ethanol/methanol
2971	Yes	Further cut	0.5082	At 90°C oven stay for 16h	KOH, n-Hexane
2977	No	Used as received	All	Thermal Desorption	Sulfuric acid, Potassium Carbonate
3100	Yes	Further cut	0.6315g	Other	water
3116	No	Used as received	1 gram	Oven	2M potassium hydroxide
3153	Yes	Further cut	0.5g	Steam distillation	N-hexane
3154	Yes	Further cut	1,8 g	16 h - 90 °C oven	1 mol/l KOH
3172	Yes	---		---	
3176	Yes	Further cut	1	Ultrasonic	Hexane / KOH
3210	Yes	Further cut	1 g	Ultrasonic	Hexane
3222	Yes	Further cut	1 g	Ultrasonic	Potassium Carbonate
3248	Yes	Used as received	1.0g	Ultrasonic	Potassium Hydroxide

APPENDIX 4

Number of participants per country

1 lab in AUSTRIA
2 labs in BANGLADESH
1 lab in BRAZIL
1 lab in CAMBODIA
1 lab in FRANCE
5 labs in GERMANY
1 lab in GUATEMALA
5 labs in HONG KONG
10 labs in INDIA
1 lab in INDONESIA
8 labs in ITALY
2 labs in KOREA, Republic of
2 labs in MOROCCO
14 labs in P.R. of CHINA
4 labs in PAKISTAN
1 lab in PERU
1 lab in PORTUGAL
2 labs in SPAIN
1 lab in SRI LANKA
1 lab in TAIWAN
1 lab in THAILAND
2 labs in TUNISIA
3 labs in TURKEY
1 lab in UNITED KINGDOM
4 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

Literature

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