Results of Proficiency Test Chlorinated Phenols in Textile December 2019

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2004, the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for Orthophenylphenol (OPP), Pentachlorophenol (PCP) and Tetrachlorophenols (TeCPs) in Textile every year. During the annual proficiency test program 2019/2020 it was decided to separate the proficiency tests on the determination of Orthophenylphenol and Chlorinated Phenols and to continue this proficiency test as Chlorinated Phenols in Textile.

In this interlaboratory study 84 laboratories in 20 different countries registered for participation. See appendix 4 for the number of participants per country.
In this report the results of the 2019 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 3 grams and labelled #19655.
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

The selected batch was an orange cotton hosiery fabric obtained from a third party. The batch was cut into pieces and after mixing well divided over 100 subsamples of approximately 3 grams each and labelled #19655.
The homogeneity of the subsamples was checked by determination of PCP in accordance with an in-house test method on 8 stratified randomly selected subsamples.

	Pentachlorophenol in mg/kg
Sample #19655-1	3.71
Sample #19655-2	3.42
Sample #19655-3	3.38
Sample #19655-4	3.13
Sample #19655-5	2.80
Sample #19655-6	2.92
Sample #19655-7	2.82
Sample #19655-8	3.06

Table 1: homogeneity test results of subsamples #19655

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.

	Pentachlorophenol in mg/kg
r (observed)	0.91
reference method	iis memo 1601 (see lit. 18)
0.3 x R (reference method)	0.82

Table 2: evaluation of the repeatability of subsamples #19655

The calculated repeatability was 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 #19655 was sent on November 13, 2019.

2.5 ANALYZES

The participants were requested to determine on the sample #19655 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 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 evaluation.

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 appropriate 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 reanalyzes). Additional or corrected test results are used for data analysis and 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 dataset does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

According to ISO5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. 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. The Kernel Density Graph 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 was projected over the Kernel Density Graph 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. In some cases, a reproducibility based on former iis proficiency tests could be used.
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 were encountered with the dispatch of the samples. Three participants did not report any test results and two other participants reported the test results after the final reporting date. Finally, 81 laboratories reported 120 numerical test results. Observed were 3 outlying test results, which is 2.5%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

Both original data sets proved to have a normal Gaussian distribution.

4.1 EVALUATION PER COMPONENT

In this section the 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.

Due to the lack of relevant reference test methods and/or precision data for the determination of PCP calculated reproducibilities were compared with reproducibilities estimated from the Horwitz equation until 2015. In 2015, it was decided to estimate a target reproducibility based on iis PT data of PCP proficiency tests from 2004 until 2014. 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 as mentioned in iis memo 1601 to estimate the target reproducibility for the evaluation of the PT test results by iis from 2015 onwards (see lit.18).

For the other components the calculated reproducibility was compared against the reproducibility estimated from the Horwitz equation.

Sample #19655

Pentachlorophenol (PCP): This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated reproducibility derived from iis memo 1601 (see lit. 18).

2,3,4,6-Tetrachlorophenol: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated reproducibility calculated using the Horwitz equation.

Other Chlorinated Phenols: The concentrations reported were near or below the detection limit. Therefore, no z-scores were calculated. 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 reproducibilities and the reproducibilities as found for the group of participating laboratories. The number of significant test results, the average test result, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility are compared in the next table.

Component	unit	n	average	2.8 * sd	R (target)
Pentachlorophenol	mg/kg	80	19.9	14.2	13.0
2,3,4,6-Tetrachlorophenol	mg/kg	37	0.11	0.08	0.07

Table 3: reproducibilities of components on sample #19655

Without further statistical calculations, the group of participating laboratories have no difficulties with the analyzes of PCP and 2,3,4,6-Tetrachlorophenol. See also the discussion in paragraphs 4.1, 4.4 and 5.

4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2019 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 2019	December 2018	December 2017	December 2016	2014-2015	Target
Pentachlorophenol	25%	26%	28-45%	28%	26-38%	26%
2,3,4,6-Tetrachlorophenol	24%	n.e.	n.e.	n.e.	n.e.	22%

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 80% of the reporting laboratories reported to be accredited for the determination of PCP in textile. The amount of sample intake varied between 0.2 and 3 grams with a majority around 1 gram (=55%).
Prior to analysis the samples were further cut by 44 participants while 28 other participants reported to use the sample as received. Ultrasonic extraction and Steam distillation were most often reported techniques for extraction by the participants, respectively 32% and 35%. KOH extraction was reported by 37% of the participants. A number of participants reported also the presence of Orthophenylphenol (OPP).

The effect of the reported analytical details on the determination of PCP in sample #19655 was further investigated, see summary in below table.

Analytical Details	unit	n	average	sd
ISO/IEC17025 accredited	mg/kg	64	20.5	5.0
Not ISO/IEC17025 accredited	mg/kg	9	17.5	3.4
Ultrasonic extraction	mg/kg	24	19.9	6.2
Steam distillation	mg/kg	26	21.3	3.6
<1g sample intake	mg/kg	27	20.2	5.9
1g sample intake	mg/kg	41	20.4	4.1
>1g sample intake	mg/kg	5	17.9	6.0
Further cut (prior to analysis)	mg/kg	44	19.5	4.3
Used as received	mg/kg	28	20.9	5.8

Table 5: effect of analytical details on PCP textile sample #19655

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 6) it could be noticed that almost all participants were able to detect PCP in the sample. The determination of 2,3,4,6-Tetrachlorophenol was much more difficult. Only 39 participants were able to report a numerical test result.

Further it could be noticed that for sample #19655 all reported test values for PCP are above 0.5 mg/kg. Thus, on the basis of PCP level this textile material would have been rejected for all Ecolabel classes.

Regarding the “sum of TeCPs” on samples #19655 all laboratories, except one, would have accepted the samples for Ecolabel Class 2 to 4, based on the sum of TeCPs <0.5 mg/kg. For Ecolabel Class two laboratories would accept the samples based on the sum of TeCPs <0.05 mg/kg or a “less than” test result.

Regarding the “sum of TrCPs” on samples #19655 none of the laboratories reported a positive test result. Thus, all reporting laboratories would have accepted the samples for Ecolabel Class 1 to 4, based on the sum of TrCPs <0.2 mg/kg.

Ecolabel	Class 1 Baby clothes (mg/kg)	Class 2 Clothes direct skin contact (mg/kg)	Class 3 Clothes, no direct contact with skin (mg/kg)	Class 4 Decoration material (mg/kg)
Pentachlorophenol	0.05	0.5	0.5	0.5
Sum of Tetrachlorophenols	0.05	0.5	0.5	0.5
Sum of Trichlorophenols	0.2	2.0	2.0	2.0

Table 6: Ecolabelling Standards and Requirements for Textiles in EU

In this proficiency test, 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 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/IEC17025 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.

6 CONCLUSION

In this proficiency test, the Pentachlorophenol, Tetrachlorophenols and Trichlorophenols content were determined. The variation observed for PCP in sample #19655 is in line with the observations in the previous proficiency tests.

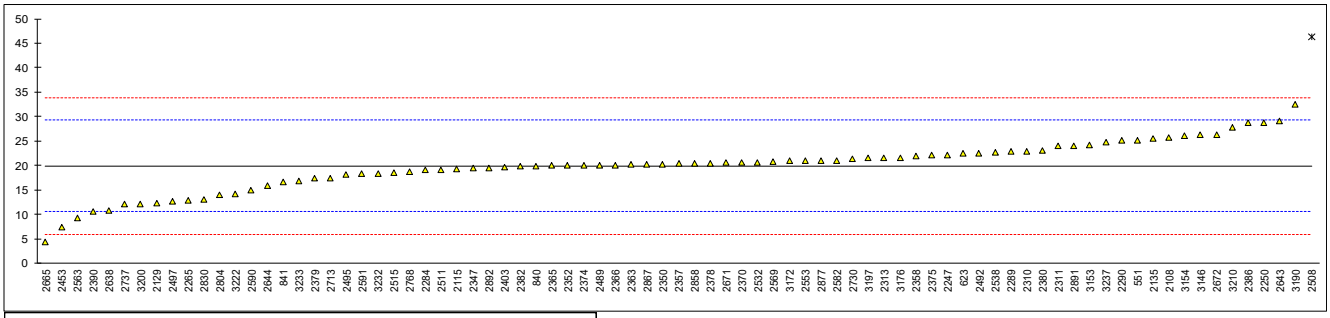
A possible explanation for the variation could be the preparation or the conditioning of the sample and/or by the performance of the analysis by the laboratory. Each laboratory should evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

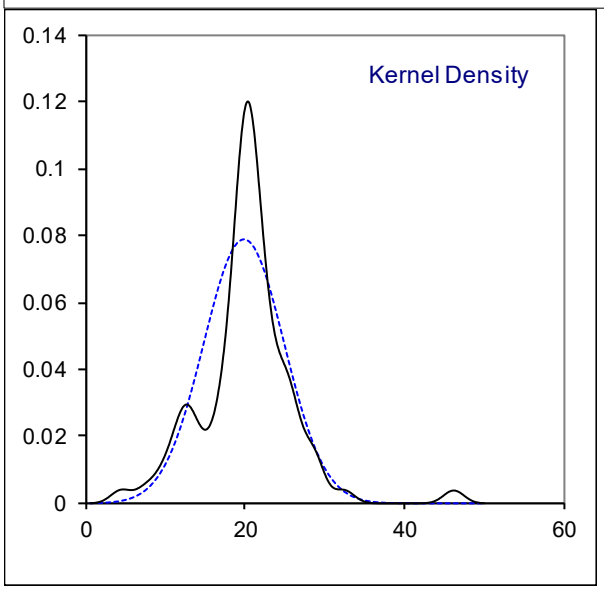
APPENDIX 1

Determination of Pentachlorophenol (PCP) on sample #19655; results in mg/kg

lab	Method	value	mark	z(targ)	remarks
551	In house	25.1950		1.13	
623	LFGB B82.02.8Mod.	22.43		0.54	
840	LFGB B82.02.8	19.9		-0.01	
841	LFGB B82.02.8	16.71		-0.69	
2108	In house	25.726		1.25	
2115	ISO17070	19.3		-0.14	
2129	In house	12.26		-1.65	
2135	In house	25.48	C	1.19	First reported 32.36
2247	In house	22.19		0.48	
2250	In house	28.8		1.91	
2265	In house	12.924		-1.51	
2284	LFGB B82.02.8	19.040		-0.19	
2289	ISO17070	22.85		0.63	
2290	ISO17070	25.12		1.12	
2310	LFGB B82.02.8	22.86		0.63	
2311	LFGB B82.02.8	24.037		0.88	
2313	LFGB B82.02.8	21.56		0.35	
2347	ISO17070	19.5	C	-0.09	First reported <0.5
2350	In house	20.287	C	0.08	First reported 32.6388
2352	LFGB B82.02.8	19.97		0.01	
2357	LFGB B82.02.8	20.40		0.10	
2358	In house	22.034		0.45	
2363	In house	20.24		0.07	
2365	In house	19.97		0.01	
2366	ISO17070	20.14		0.04	
2370	LFGB B82.02.8	20.6		0.14	
2374	In house	20.01		0.02	
2375	LFGB B82.02.8	22.10		0.47	
2378	LFGB B82.02.8	20.44		0.11	
2379	LFGB B82.02.8	17.360		-0.55	
2380	64 LFGB B82.02.8Mod.	23.040		0.67	
2382	LFGB B82.02.8	19.800		-0.03	
2386	ISO17070	28.7		1.89	
2390	ISO17070	10.5790		-2.01	
2403	ISO17070	19.728		-0.04	
2452		-----		-----	
2453	LFGB B82.02.8	7.49	C	-2.68	First reported 5.83
2489	LFGB B82.02.8	20.1		0.04	
2492	In house	22.538		0.56	
2495	In house	18.095		-0.40	
2497	ISO17070	12.687		-1.56	
2508	LFGB B82.02.8	46.31	R(0.01)	5.67	
2511		19.1		-0.18	
2515	In house	18.591		-0.29	
2532	ISO17070	20.7		0.16	
2538	LFGB B82.02.8	22.73		0.60	
2553	In house	20.92		0.21	
2563	ISO17070	9.29		-2.29	
2569	LFGB B82.02.8	20.9		0.21	
2582	In house	21.00	C	0.23	First reported 7.0001
2590	ISO17070	14.964		-1.07	
2591	ISO17070	18.286		-0.35	
2602		-----		-----	
2638	ISO17070	10.718	C	-1.98	First reported 7.38
2643	KS0733	29.08		1.97	
2644	UNI11057	15.96		-0.85	
2665	In house	4.47		-3.33	
2671		20.56		0.13	
2672	In house	26.35		1.38	
2713	In house	17.37		-0.55	
2730	XP G08-15	21.41		0.32	
2737	In house	12.151		-1.67	
2768	LFGB B82.02.8	18.67		-0.27	
2804	In house	14.0		-1.28	
2830	ISO XP G08-15	13.036		-1.48	
2858	In house	20.42		0.10	
2867	In house	20.28		0.07	
2877	ISO17070	20.9263		0.21	
2891	LFGB B82.02.8	24.07	C	0.89	First reported <0.5
2892	LFGB B82.02.8	19.500		-0.09	
3118		-----		-----	
3146	GB/T20386	26.3		1.37	
3153	LFGB B82.02.8	24.25		0.93	
3154	ISO17070	26.07		1.32	

lab	Method	value	mark	z(targ)	Remarks
3172	GB/T20386	20.911		0.21	
3176	LFGB B82.02.8	21.60		0.36	
3190	LFGB B82.02.8	32.520		2.71	
3197	LFGB B82.02.8	21.55		0.35	
3200	LFGB B82.02.8	12.16		-1.67	
3210	In house	27.8		1.69	
3222		14.19		-1.24	
3232	ISO17070	18.29		-0.35	
3233	In house	16.76		-0.68	
3237	ISO17070	24.76		1.04	
	normality	OK			
	n	80			
	outliers	1			
	mean (n)	19.935	RSD = 25%		
	st.dev. (n)	5.0550			
	R(calc.)	14.154			
	st.dev.(iis memo 1601)	4.6498			
	R(iis memo 1601)	13.019			



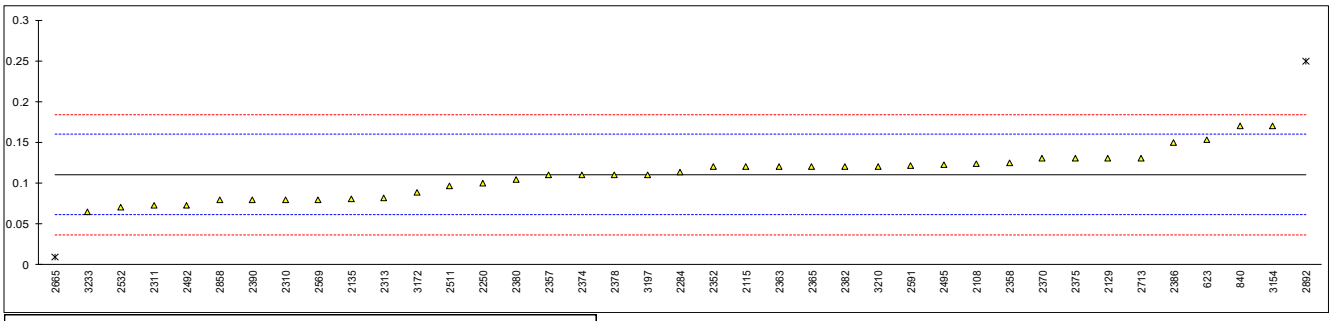


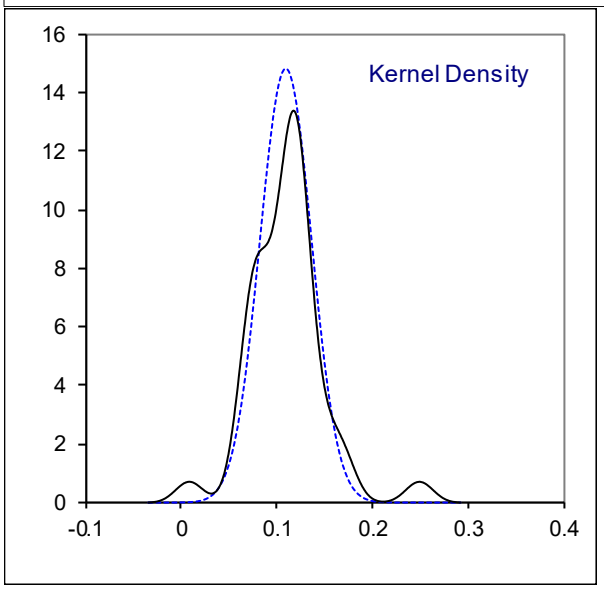
Determination of 2,3,4,6-Tetrachlorophenol on sample #19655; results in mg/kg

lab	method	value	mark	z(targ)	remarks
551	In house	N.D.		-----	
623	LFGB B82.02.8Mod.	0.153		1.73	
840	LFGB B82.02.8	0.17		2.43	
841	LFGB B82.02.8	ND		-----	
2108	In house	0.124		0.56	
2115	ISO17070	0.12		0.39	
2129	In house	0.130		0.80	
2135	In house	0.081		-1.19	
2247	In house	ND		-----	
2250	In house	0.10		-0.42	
2265	In house	< 0,1		-----	
2284	LFGB B82.02.8	0.114		0.15	
2289	ISO17070	<0.05		-----	
2290	ISO17070	<0.5		-----	
2310	LFGB B82.02.8	0.08		-1.23	
2311	LFGB B82.02.8	0.0723		-1.55	
2313	LFGB B82.02.8	0.082		-1.15	
2347	ISO17070	<0.5		-----	
2350	In house	<0.125		-----	
2352	LFGB B82.02.8	0.12		0.39	
2357	LFGB B82.02.8	0.110		-0.01	
2358	In house	0.1252		0.60	
2363	In house	0.12		0.39	
2365	In house	0.12		0.39	
2366	ISO17070	<0.5		-----	
2370	LFGB B82.02.8	0.130		0.80	
2374	In house	0.11		-0.01	
2375	LFGB B82.02.8	0.13		0.80	
2378	LFGB B82.02.8	0.11		-0.01	
2379	LFGB B82.02.8	Not detected		-----	
2380	64 LFGB B82.02.8Mod.	0.105		-0.22	
2382	LFGB B82.02.8	0.120		0.39	
2386	ISO17070	0.15		1.61	
2390	ISO17070	0.07972		-1.24	
2403		-----		-----	
2452		-----		-----	
2453		-----		-----	
2489	LFGB B82.02.8	ND		-----	
2492	In house	0.073		-1.52	
2495	In house	0.123		0.51	
2497		-----		-----	
2508		-----		-----	
2511		0.096		-0.58	
2515		-----		-----	
2532	ISO17070	0.07		-1.64	
2538		-----		-----	
2553	In house	ND		-----	
2563	ISO17070	< 0,1		-----	
2569	LFGB B82.02.8	0.08		-1.23	
2582		-----		-----	
2590		-----		-----	
2591	ISO17070	0.122		0.47	
2602		-----		-----	
2638		-----		-----	
2643		-----		-----	
2644		-----		-----	
2665	In house	0.0095	R(0.05)	-4.10	
2671		-----		-----	
2672	In house	<0.05		-----	
2713	In house	0.13		0.80	
2730		-----		-----	
2737		-----		-----	
2768		-----		-----	
2804		-----		-----	
2830	ISO XP G08-15	ND		-----	
2858	In house	0.079		-1.27	
2867		-----		-----	
2877		-----		-----	
2891	LFGB B82.02.8	< 0,5		-----	
2892	LFGB B82.02.8	0.2500	R(0.01)	5.68	
3118		-----		-----	
3146		-----		-----	
3153		-----		-----	
3154	ISO17070	0.17	C	2.43	First reported 0.01

lab	method	value	mark	z(targ)	remarks
3172	GB/T20386	0.0883		-0.90	
3176		-----		-----	
3190		-----		-----	
3197	LFGB B82.02.8	0.11		-0.01	
3200	LFGB B82.02.8	<0.1		-----	
3210	In house	0.12		0.39	
3222		-----		-----	
3232	ISO17070	<0.05		-----	
3233	In house	0.065		-1.84	
3237		-----		-----	

	normality	OK
	n	37
	outliers	2
	mean (n)	0.1103 RSD = 24%
	st.dev. (n)	0.02693
	R(calc.)	0.0754
	st.dev.(Horwitz)	0.02460
	R(Horwitz)	0.0689





APPENDIX 2

Other reported test results

Determination individual and other Chlorinated Phenols on sample #19655; in mg/kg

lab	2345-TeCP	2356-TeCP	234-TCP	235-TCP	236-TCP	245-TCP	246-TCP	345-TCP	Other
551	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	-----
623	0.154	0.107	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
840	0.10	not det.	not det.	not det.	not det.	not det.	not det.	not det.	-----
841	ND	0.13	ND	ND	ND	ND	ND	ND	-----
2108	-----	-----	-----	-----	-----	-----	-----	-----	23.3 OPP
2115	-----	-----	-----	-----	-----	-----	-----	-----	-----
2129	0.041	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	27.66 OPP
2135	-----	-----	-----	-----	-----	-----	-----	-----	-----
2247	ND	ND	ND	ND	ND	ND	ND	ND	19.7 OPP
2250	-----	-----	-----	-----	-----	-----	-----	-----	28.5 OPP
2265	< 0,1	< 0,1	< 0,1	< 0,1	< 0,1	< 0,1	< 0,1	< 0,1	-----
2284	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2289	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2290	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	-----
2310	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.
2313	-----	-----	-----	-----	-----	-----	-----	-----	-----
2347	<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
2352	-----	-----	-----	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----	-----	-----	-----
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2363	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	NA
2365	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2366	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	out of cap.
2370	0.107	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
2374	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2375	-----	-----	-----	-----	-----	-----	-----	-----	-----
2378	-----	-----	-----	-----	-----	-----	-----	-----	-----
2379	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	-----
2382	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050
2386	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.5
2390	0.01993	-----	-----	-----	-----	-----	-----	-----	-----
2403	-----	-----	-----	-----	-----	-----	-----	-----	-----
2452	-----	-----	-----	-----	-----	-----	-----	-----	-----
2453	-----	-----	-----	-----	-----	-----	-----	-----	-----
2489	ND	ND	ND	ND	ND	ND	ND	ND	17.3 OPP
2492	-----	-----	-----	-----	-----	-----	-----	-----	-----
2495	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2497	-----	-----	-----	-----	-----	-----	-----	-----	9.364 OPP
2508	-----	-----	-----	-----	-----	-----	-----	-----	50.90 OPP
2511	-----	-----	-----	-----	-----	-----	-----	-----	-----
2515	-----	-----	-----	-----	-----	-----	-----	-----	-----
2532	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	17 OPP
2538	-----	-----	-----	-----	-----	-----	-----	-----	-----
2553	ND	ND	ND	ND	ND	ND	ND	ND	ND
2563	-----	-----	-----	-----	-----	-----	-----	-----	-----
2569	ND	ND	ND	ND	ND	ND	ND	ND	ND
2582	-----	-----	-----	-----	-----	-----	-----	-----	7.4998
2590	-----	-----	-----	-----	-----	-----	-----	-----	-----
2591	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	-----
2602	-----	-----	-----	-----	-----	-----	-----	-----	-----
2638	-----	-----	-----	-----	-----	-----	-----	-----	11.492 OPP
2643	-----	-----	-----	-----	-----	-----	-----	-----	-----
2644	-----	-----	-----	-----	-----	-----	-----	-----	19.55 OPP
2665	0.0027	0.0008	0.0019	0	0	0	0	0	0
2671	-----	-----	-----	-----	-----	-----	-----	-----	-----
2672	0.0825	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2713	< 0,05	<0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	-----
2730	-----	-----	-----	-----	-----	-----	-----	-----	-----
2737	-----	-----	-----	-----	-----	-----	-----	-----	-----
2768	-----	-----	-----	-----	-----	-----	-----	-----	-----
2804	-----	-----	-----	-----	-----	-----	-----	-----	-----
2830	ND	ND	ND	ND	ND	ND	ND	ND	ND
2858	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d
2867	-----	-----	-----	-----	-----	-----	-----	-----	-----
2877	-----	-----	-----	-----	-----	-----	-----	-----	-----
2891	<0.5	< 0,5	< 0,5	< 0,5	< 0,5	< 0,5	< 0,5	< 0,5	< 0,5
2892	0.2100	0.1500	-----	-----	-----	-----	-----	-----	-----

lab	2345-TeCP	2356-TeCP	234-TCP	235-TCP	236-TCP	245-TCP	246-TCP	345-TCP	Other
3118	-----	-----	-----	-----	-----	-----	-----	-----	-----
3146	-----	-----	-----	-----	-----	-----	-----	-----	-----
3153	-----	-----	-----	-----	-----	-----	-----	-----	-----
3154	0.005	0.003	-----	-----	-----	-----	0.001	-----	0.012
3172	-----	-----	-----	-----	-----	-----	-----	-----	-----
3176	-----	-----	-----	-----	-----	-----	-----	-----	18.4 OPP
3190	-----	-----	-----	-----	-----	-----	-----	-----	-----
3197	ND	ND	ND	ND	ND	ND	ND	ND	-----
3200	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
3210	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
3222	-----	-----	-----	-----	-----	-----	-----	-----	20.16 OPP
3232	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	-18.71 OPP
3233	-----	-----	-----	-----	-----	-----	-----	-----	20.83 OPP
3237	-----	-----	-----	-----	-----	-----	-----	-----	20.55 OPP

2345-TeCP = 2,3,4,5-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

APPENDIX 3

Analytical details

lab	ISO/IEC17025 accredited	Sample intake (grams)	Extraction technique	Extraction solution	Sample preparation
551	Yes	1.0	Ultrasonic extraction	KOH	Further cut
623	Yes	1	Ultrasonic extraction	KOH	Further cut
840	Yes	1	Ultrasonic extraction	KOH	Further cut
841	Yes	1	Ultrasonic extraction	KOH	Further cut
2108	Yes	0,5	Microwave	KOH	As received
2115	Yes	1	Steam distillation	KOH	Further cut
2129	Yes	0,5	Ultrasonic extraction	KOH	As received
2135	Yes	1	Basic extraction	Sodium Carbonate	As received
2247	Yes	0.5	Other	KOH	Further cut
2250	Yes	0,5	Ultrasonic extraction	KOH	As received
2265	Yes	0,5	Basic extraction (90°)	KOH	Further cut
2284	Yes	1	Other	KOH	As received
2289	Yes	1.0	Steam distillation	Other	Further cut
2290	Yes	---	---	---	---
2310	Yes	2	Steam distillation	Hexane	Further cut
2311	Yes	1	Steam distillation	Hexane	Further cut
2313	Yes	1.0	Steam distillation	Hexane	Further cut
2347	Yes	1.0	Ultrasonic extraction	KOH	As received
2350	No	0.5 & 2	Ultrasonic extraction	KOH	Further cut
2352	Yes	0.5	Steam distillation	Hexane	Further cut
2357	---		---	---	---
2358	Yes	1	Other	KOH	Further cut
2363	Yes	0.5	Ultrasonic extraction	KOH	Further cut
2365	No	0.8023	Ultrasonic extraction	KOH	Further cut
2366	Yes	0.5	Steam distillation	Potassium Carbonate	Further cut
2370	Yes	2	Steam distillation	Sulfuric Acid	Further cut
2374	Yes	1	Ultrasonic extraction	KOH	As received
2375	Yes	1	Ultrasonic extraction	KOH	Further cut
2378	Yes	0.5	Steam distillation	Hexane	Further cut
2379	No	1	Ultrasonic extraction	KOH	Further cut
2380	Yes	1.0	Alkaline digestion	KOH	Further cut
2382	Yes	1.0	Steam distillation	Sulfuric Acid	Further cut
2386	Yes	0.5	Ultrasonic extraction	KOH	Further cut
2390	---		---	---	---
2403	Yes	1	Steam distillation	Other	Further cut
2452	---		---	---	---
2453	---		---	---	---
2489	Yes	0.5810	Steam distillation	Other	Further cut
2492	Yes	0.5	Soxhlet / AES extraction	Other	Further cut
2495	Yes	1.00	KOH extraction	KOH	As received
2497	Yes	0.5	Ultrasonic extraction	MeOH	Further cut
2508	Yes	0.5	Ultrasonic extraction	Other	As received
2511	---		---	---	---
2515	Yes	0.5	KOH extraction (90°C)	KOH	As received
2532	Yes	1	Steam distillation	Sulfuric acid	Further cut
2538	Yes	2	Steam distillation	Other	Further cut
2553	Yes	1	Liquid extraction	KOH	As received
2563	Yes	1,5	Soxhlet / AES extraction	Acetone/HAc	Further cut
2569	Yes	1	Steam distillation	Other	Further cut
2582	Yes	1.0045	Steam distillation	KOH	As received
2590	Yes	1	Steam distillation	K2CO3 Solution	Further cut
2591	Yes	1	Basic Digestion	KOH	Further cut
2602	---		---	---	---
2638	No	1	Ultrasonic extraction	Hexane	Further cut
2643	Yes	0.8	Ultrasonic extraction	KOH	As received
2644	Yes	1	Ultrasonic extraction	KOH	Further cut
2665	Yes	0.5	Ultrasonic extraction	Dichloromethane	As received
2671	Yes	1	Other	KOH	As received

lab	ISO/IEC17025 accredited	Sample intake (grams)	Extraction technique	Extraction solution	Sample preparation
2672	Yes	1	Ultrasonic extraction	Toluene	As received
2713	No	1	KOH extraction	KOH	Further cut
2730	No	0,5	Ultrasonic extraction	---	As received
2737	Yes	1	KOH extraction (90°C	KOH	Further cut
2768	Yes	1	Steam distillation	Sulfuric Acid	As received
2804	No	1	Soxhlet / AES extraction	KOH	As received
2830	Yes	0.5	Steam distillation	K2CO3 Solution	Further cut
2858	Yes	1.0	Other	KOH	As received
2867	Yes	0.5	Basic Extraction	KOH	As received
2877	---		---	---	---
2891	Yes	1,0014	Steam distillation	Sulfuric Acid	Further cut
2892	No	1.0	Other	KOH	As received
3118	---		---	---	---
3146	Yes	0,5	KOH extraction	KOH / Methanol	---
3153	Yes	0.25	Steam distillation	Sulfuric Acid	Further cut
3154	Yes	0,5	Steam distillation	Water	As received
3172	---		---	---	---
3176	Yes	1	Ultrasonic extraction	KOH	As received
3190	Yes	1	Steam distillation	Other	As received
3197	Yes	1	Steam distillation	Other	As received
3200	Yes	0.5	Other	KOH	Further cut
3210	---	1	Ultrasonic extraction	K2CO3 Solution	As received
3222	Yes	2	Ultrasonic extraction	K2CO3 Solution	As received
3232	Yes	0.5	Steam distillation	Other	Further cut
3233	No	1	KOH extraction	KOH	As received
3237	Yes	0,5	Steam distillation	Other	Further cut

APPENDIX 4

Number of participants per country

2 labs in BANGLADESH
2 labs in BRAZIL
3 labs in FRANCE
14 labs in GERMANY
5 labs in HONG KONG
10 labs in INDIA
2 labs in INDONESIA
7 labs in ITALY
1 lab in MOROCCO
15 labs in P.R. of CHINA
2 labs in PAKISTAN
1 lab in PORTUGAL
3 labs in SOUTH KOREA
1 lab in SPAIN
2 labs in SRI LANKA
1 lab in TAIWAN R.O.C.
1 lab in THAILAND
2 labs in TUNISIA
5 labs in TURKEY
5 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
n.a.	= not applicable
n.d.	= not detected
n.e.	= not evaluated
W	= test result withdrawn on request of participant
ex	= test result excluded from the statistical evaluation

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