Institute for
Interlaboratory Studies

Results of Proficiency Test
Chlorinated Phenols
in Leather/Footwear
May 2023

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. G.A. Oosterlaken-Buijs
Correctors: ing. M. Meijer & ing. R.J. Starink
Approved by: ing. A.S. Noordman-de Neef

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CONTENTS

1 INTRODUCTION

Products containing Pentachlorophenol (PCP) may form highly toxic substances when they are incinerated. PCP is also a suspected carcinogen. Since the 1990's many countries have adopted environmental standards and requirements restricting the use of harmful chemicals in the production of textiles and leather consumer products. 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 2016 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of Chlorinated Phenols 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 Chlorinated Phenols in Leather/Footwear.

In this interlaboratory study 56 laboratories in 21 countries registered for participation, see appendix 4 for the number of participants per country. In this report the results of the Chlorinated Phenols 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 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 leather sample of 3 grams labelled #23585.
The participants were asked 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 yellow leather containing some Chlorinated Phenols was obtained from a third party. The batch was cut into small pieces and after homogenization 80 small plastics bags were filled with approximately 3 grams each and labelled #23585.
The batch for sample #23585 was used in a previous proficiency test on Chlorinated Phenols in Leather/Footwear as sample #17545 in iis17A07. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample of #23585 was sent on April 19, 2023.

2.5 ANALYZES

The participants were requested to determine Pentachlorophenol (PCP) and all isomers of Tetra-, Tri-, Di- and Monochlorophenols.
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 to 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 also requested to report the test results using the indicated units on the report form and not to round the results but report as much significant figures as possible and 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 data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner’s outlier test can be used. Outliers are marked by D(0.01) for the Dixon’s test, by G(0.01) or DG(0.01) for the Grubbs’ test and by R(0.01) for the Rosner’s test. Stragglers are marked by D(0.05) for the Dixon’s test, by G(0.05) or DG(0.05) for the Grubbs’ test and by R(0.05) for the Rosner’s test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT the criterion of ISO13528, paragraph 9.2.1, was met for all evaluated tests. Therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements (derived from e.g. ISO or ASTM test methods), the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated according to:

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

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. Therefore, 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. Five participants reported test results after the final reporting date and two other participants did not report any test results. Not all participants were able to report all tests requested. In total 54 participants reported 99 numerical test results. No outlying test results were observed. In proficiency tests, 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 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.

Test methods ISO17070:15 and LFGB 82.02-8 mention precision data for Pentachlorophenol. In 2016 iis investigated the reproducibilities of the determination of Pentachlorophenol in textile over 18 determinations in iis PTs conducted from 2004 until 2014. It was observed that the reproducibility as mentioned in ISO17070 is 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 Pentachlorophenol in Textile it is decided to use the estimated iis target reproducibility also for the determination of Pentachlorophenol in Leather/Footwear. Furthermore, it is decided to use the estimated iis target reproducibility for other Chlorinated Phenol components determined in Leather/Footwear as well.

Pentachlorophenol: The determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the target reproducibility as derived from iis memo 1601.

2,4,6-Trichlorophenol: The 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 Chlorinated Phenols mentioned in paragraph 2.5. Therefore, no z-scores are calculated for these components. The reported test results are given in appendix 2.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the 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 from the reference method are presented in the next table.

Component	unit	n	average	2.8 * sd	R(target)
Pentachlorophenol (PCP)	mg/kg	53	10.1	10.6	7.3
2,4,6-Trichlorophenol	mg/kg	46	1.00	0.84	1.03

Table 1: reproducibilities of components on sample #23585

Without further statistical calculations it can be concluded that for 2,4,6-Trichlorophenol there is a good compliance of the group of participating laboratories with the target reproducibility, but not for Pentachlorophenol. The problematic tests have been discussed in paragraph 4.1.

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	54	68	70	65	73
Number of test results	99	119	70	125	205
Number of statistical outliers	0	5	1	2	4
Percentage of statistical outliers	0.0%	4.2%	1.4%	1.6%	2.0%

Table 2: 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 test was compared to uncertainties observed in PTs over the years, expressed as relative standard deviation (RSD) of the PTs, see next table.

	May 2023	April 2022	May 2021	2016 – 2020	Target *)
PCP	37%	35%	31%	21 - 41%	41 – 25%
2,3,4,5-TeCP	n.e.	n.e.	n.e.	n.e.	41 – 25%
2,3,5,6-TeCP	n.e.	n.e.	n.e.	26%	41 – 25%
2,4,6-TCP	30%	30%	n.e.	29 - 34%	41 – 25%

Table 3: development of the uncertainties over the years

*) Concentration range 0.5 – 13 mg/kg respectively

The uncertainties observed in this PT are in line with previous iis PTs.

Sample #23585 was used in a previous PT as sample #17545 in iis17A07. The average and the calculated reproducibility for Pentachlorophenol (PCP) in the 2023 PT are in line with the 2017 PT.

Component	unit	sample #23585			sample #17545		
		n	average	R(calc)	n	average	R(calc)
Pentachlorophenol	mg/kg	53	10.1	10.6	70	9.8	9.9

Table 4: comparison of sample #23585 with #17545

4.4 EVALUATION OF THE ANALYTICAL DETAILS

Test method ISO17070 is used by about 70% of the reporting participants and test method LFGB B82.02.8 is used by about 2% of the participants. Test methods ISO17070 and LFGB 82.02-8 describe a similar sample pathway to determine PCP. About 13% of the reporting participants mentioned to have used an in-house test method. And about 13% of the reporting participants used DIN50009 which is a method for textile.

For this PT some analytical details were requested which are listed in appendix 3. Based on the answers given by the participants the following can be summarized:
- 87% of the participants mentioned that they are accredited for the determination of the reported component(s).
- 27% of the participants used the sample as received and 73% did further cut or grind the sample.
- The sample intake varied from 0.2 grams to 2 grams. 39% of the participants used a sample intake of 0.5 grams and 53% used 1 gram.
- 65% of the participants used steam distillation and 13% used KOH as technique to release the Chlorinated Phenols.
- 19% of the participants used Ultrasonic and 57% used Mechanical Shaking as technique to extract the Chlorinated Phenols.

When the analytical details were investigated separately, it appeared that the effect on the determination of Chlorinated Phenols in Leather/Footwear is negligible.

5 DISCUSSION

When the results of this proficiency test were compared to the OEKO-TEX® Leather Standard (see table 5) it was noticed that not all participants would make identical decisions about the acceptability of the sample for Chlorinated Phenols.
For Pentachlorophenol all participants would have rejected the sample for all classes.
For 2,4,6-Trichlorophenol almost all participants (except one) would have rejected the sample for class 1. However, 23 participants would have accepted the sample for the other classes.

	Class 1 Baby in mg/kg	Class 2 Direct skin contact in mg/kg	Class 3 No direct skin contact in mg/kg	Class 4 Decoration material in mg/kg
Pentachlorophenol (PCP)	<0.3	<0.5	<0.5	<0.5
Tetrachlorophenols (TeCP), each isomer	<0.5	<0.5	<0.5	<0.5
Trichlorophenols (TrCP), each isomer	<0.5	<1.0	<1.0	<1.0
Dichlorophenols (DCP), each isomer	<1.0	<1.0	<1.0	<1.0
Monochlorophenols (MCP), each isomer	<2.0	<2.0	<2.0	<2.0

Table 5: OEKO-TEX® Leather Standard specific limits for Chlorophenols

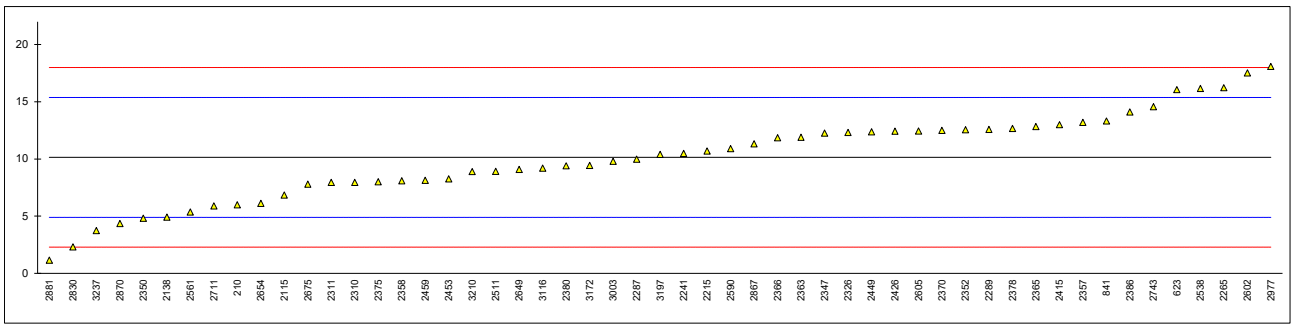
6 CONCLUSION

Although it can be concluded that most of the participants have no problem with the determination on Pentachlorophenol and 2,4,6-Trichlorophenol 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 Pentachlorophenol (PCP) on sample #23585; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	ISO17070	6		-1.58	
551		-----		-----	
623	ISO17070	16.065		2.27	
841	ISO17070	13.321		1.22	
2115	ISO17070	6.85		-1.26	
2138	KS K0733	4.907		-2.00	
2215	ISO17070	10.69		0.21	
2241	ISO17070	10.487		0.13	
2265	DIN50009	16.225		2.33	
2287	DIN50009	9.989		-0.06	
2289	ISO17070	12.58		0.93	
2293		-----		-----	
2310	ISO17070	7.95		-0.84	
2311	ISO17070	7.950		-0.84	
2326	DIN50009	12.32	C	0.83	first reported 14.248
2347	ISO17070	12.26		0.81	
2350	ISO17070	4.8167		-2.03	
2352	ISO17070	12.56		0.93	
2357	ISO17070	13.201		1.17	
2358	ISO17070	8.1		-0.78	
2363	In house	11.902		0.67	
2365	ISO17070	12.840		1.03	
2366	In house	11.86		0.66	
2370	ISO17070	12.5		0.90	
2375	ISO17070	8.0		-0.82	
2378	ISO17070	12.67		0.97	
2380	ISO17070	9.403		-0.28	
2386	DIN50009	14.110		1.52	
2415	ISO17070	13.00		1.09	
2426	ISO17070	12.422		0.87	
2449	ISO17070	12.38		0.86	
2453	DIN50009	8.269		-0.71	
2459	ISO17070	8.13		-0.77	
2511	ISO17070	8.92		-0.47	
2538	DIN50009	16.16		2.30	
2561	ISO17070	5.3661665		-1.82	
2590	ISO17070	10.899		0.29	
2592		-----		-----	
2602	In house	17.525		2.82	
2605	ISO17070	12.44		0.88	
2649	In house	9.09		-0.40	
2654	ISO17070	6.12		-1.54	
2675	ISO17070	7.797		-0.89	
2711	ISO17070	5.90		-1.62	
2743	ISO17070	14.57	C	1.69	first reported 1.254
2830	ISO17070	2.316		-2.99	
2867	ISO17070	11.332		0.46	
2870	ISO17070	4.36		-2.21	
2881	In house	1.15		-3.43	
2977	In house	18.090		3.04	
3003	DIN50009	9.8		-0.13	
3116	LFGB B82.02.8	9.20		-0.36	
3172	ISO17070	9.4396		-0.27	
3197	ISO17070	10.4		0.10	
3210	In house	8.89		-0.48	
3237	ISO17070	3.73		-2.45	
	normality	OK			
	n	53			
	outliers	0			
	mean (n)	10.1368			
	st.dev. (n)	3.79338	RSD = 37%		
	R(calc.)	10.6215			
	st.dev.(iis memo 1601)	2.61683			
	R(iis memo 1601)	7.3271			
compare					
	R(ISO17070:15)	3.4281			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2453
3210
2511
2649
3116
2380
3172
3003
2287
3197
2241
2215
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2363
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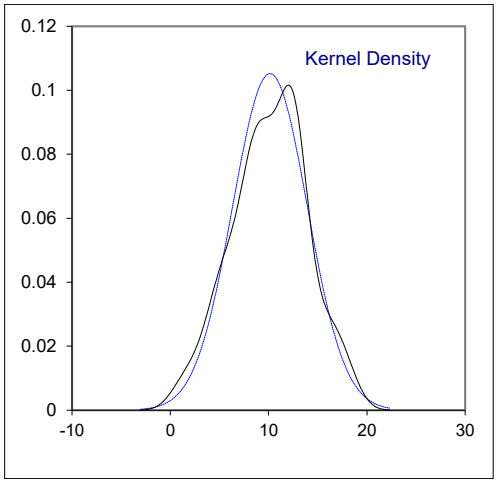
Kernel Density
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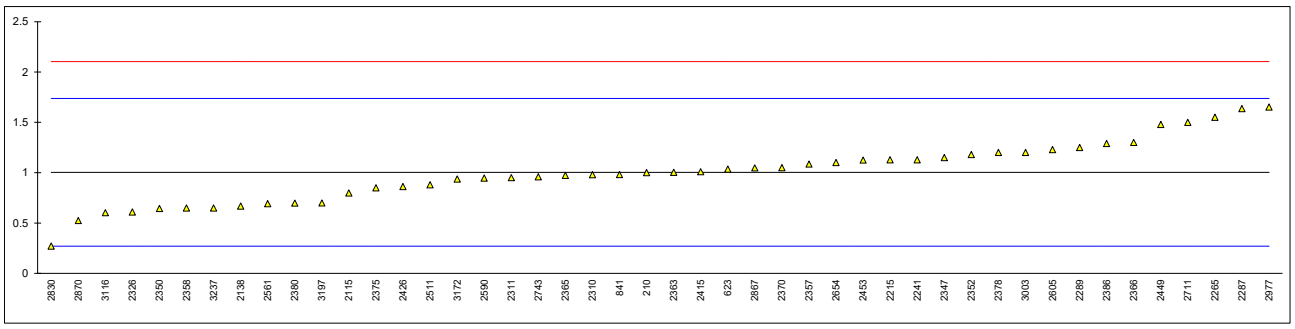
0.1
0.08
0.06
0.04
0.02
0
-10
0
10
20
30

Determination of 2,4,6-Trichlorophenol on sample #23585; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	ISO17070	1		-0.01	
551		-----		-----	
623	ISO17070	1.036		0.09	
841	ISO17070	0.982		-0.06	
2115	ISO17070	0.8		-0.56	
2138	KS K0733	0.668		-0.92	
2215	ISO17070	1.128		0.34	
2241	ISO17070	1.128		0.34	
2265	DIN50009	1.550		1.49	
2287	DIN50009	1.637		1.73	
2289	ISO17070	1.25		0.67	
2293		-----		-----	
2310	ISO17070	0.98		-0.06	
2311	ISO17070	0.951		-0.14	
2326	DIN50009	0.61	C	-1.07	first reported 2.131
2347	ISO17070	1.15		0.40	
2350	ISO17070	0.6457		-0.98	
2352	ISO17070	1.18		0.48	
2357	ISO17070	1.086		0.22	
2358	ISO17070	0.65		-0.97	
2363	In house	1.004		0.00	
2365	ISO17070	0.974		-0.08	
2366	In house	1.30		0.81	
2370	ISO17070	1.05		0.13	
2375	ISO17070	0.85		-0.42	
2378	ISO17070	1.2		0.54	
2380	ISO17070	0.6982		-0.83	
2386	DIN50009	1.290		0.78	
2415	ISO17070	1.01		0.02	
2426	ISO17070	0.863		-0.38	
2449	ISO17070	1.48		1.30	
2453	DIN50009	1.126		0.33	
2459	ISO17070	Not Detected		-----	
2511	ISO17070	0.88		-0.34	
2538		-----		-----	
2561	ISO17070	0.6927075		-0.85	
2590	ISO17070	0.947		-0.15	
2592		-----		-----	
2602		-----		-----	
2605	ISO17070	1.23		0.62	
2649		-----		-----	
2654	ISO17070	1.10		0.26	
2675	ISO17070	not detected		-----	
2711	ISO17070	1.50		1.35	
2743	ISO17070	0.96	C	-0.12	first reported 0.191
2830	ISO17070	0.27		-2.00	
2867	ISO17070	1.048		0.12	
2870	ISO17070	0.524		-1.31	
2881		-----		-----	
2977	In house	1.653		1.77	
3003	DIN50009	1.2		0.54	
3116	LFGB B82.02.8	0.603		-1.09	
3172	ISO17070	0.9374		-0.18	
3197	ISO17070	0.7		-0.83	
3210	In house	<0.05		-----	
3237	ISO17070	0.65		-0.97	
	normality	OK			
	n	46			
	outliers	0			
	mean (n)	1.0037	RSD = 30%		
	st.dev. (n)	0.29979			
	R(calc.)	0.8394			
	st.dev.(iis memo 1601)	0.36655			
	R(iis memo 1601)	1.0263			
compare					
	R(ISO17070:15)	0.1566			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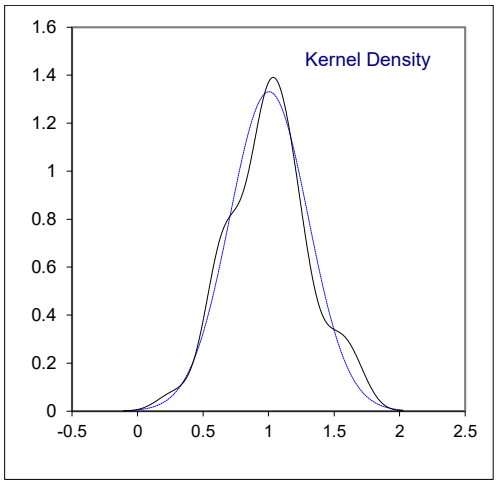
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1.2
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0.6
0.4
0.2
0
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APPENDIX 2 Other reported test results

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

23DCP = 2,3-Dichlorophenol
24DCP = 2,4-Dichlorophenol
25DCP = 2,5-Dichlorophenol
26DCP = 2,6-Dichlorophenol
34DCP = 3,4-Dichlorophenol
35DCP = 3,5-Dichlorophenol
2CP = 2-Chlorophenol
3CP = 3-Chlorophenol
4CP = 4-Chlorophenol

Other reported Chlorinated Phenols on sample #23585; results in mg/kg

lab	2345TeCP	2346TeCP	2356TeCP	234TCP	235TCP	236TCP	245TCP	345TCP
210	-----	-----	-----	-----	-----	-----	-----	-----
551	-----	-----	-----	-----	-----	-----	-----	-----
623	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
841	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2115	-----	0.036	-----	-----	-----	-----	-----	-----
2138	-----	-----	-----	-----	-----	-----	-----	-----
2215	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2241	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2265	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05
2287	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
2289	-----	-----	-----	-----	-----	-----	-----	-----
2293	-----	-----	-----	-----	-----	-----	-----	-----
2310	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2311	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2326	ND	ND	ND	ND	ND	ND	ND	ND
2347	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2350	< 0.125	< 0.125	< 0.125	< 0.125	< 0.125	< 0.125	< 0.125	< 0.125
2352	-----	-----	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----	-----	-----
2358	-----	-----	-----	-----	-----	-----	-----	-----
2363	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2365	＜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
2370	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2375	-----	-----	-----	-----	-----	-----	-----	-----
2378	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05
2380	<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
2415	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2426	ND	ND	ND	ND	ND	ND	ND	ND
2449	-----	-----	-----	-----	-----	-----	-----	-----
2453	-----	-----	-----	-----	-----	-----	-----	-----
2459	not detected	not detected	not detected	0.92	not detected	not detected	not detected	not detected
2511	-----	-----	-----	-----	-----	-----	-----	-----
2538	-----	-----	-----	-----	-----	-----	-----	-----
2561	-----	-----	-----	-----	-----	-----	-----	-----
2590	-----	-----	-----	-----	-----	-----	-----	-----
2592	-----	-----	-----	-----	-----	0.5050	-----	-----
2602	-----	-----	-----	-----	-----	-----	-----	-----
2605	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10
2649	-----	-----	-----	-----	-----	-----	-----	-----
2654	-----	-----	-----	-----	-----	-----	-----	-----
2675	not detected	not detected	not detected	1.440	not detected	not detected	not detected	not detected
2711	<0.5	<0.5	<0.5	<0.5	not analyzed	<0.5	<0.5	<0.5
2743	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2830	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2867	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2870	-----	-----	-----	-----	-----	-----	-----	-----
2881	-----	-----	-----	-----	-----	-----	-----	-----
2977	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
3003	-----	-----	-----	-----	-----	-----	-----	-----
3116	-----	-----	-----	-----	-----	-----	-----	-----
3172	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
3197	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05
3210	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	1.25
3237	nd	nd	nd	nd	nd	nd	nd	nd

Other reported Chlorinated Phenols on sample #23585; results in mg/kg -- continued --

lab	23DCP	24DCP	25DCP	26DCP	34DCP	35DCP
210	-----	-----	-----	-----	-----	-----
551	-----	-----	-----	-----	-----	-----
623	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
841	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2115	-----	-----	-----	-----	-----	-----
2138	-----	-----	-----	-----	-----	-----
2215	not detected	not detected	not detected	not detected	not detected	not detected
2241	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2265	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05
2287	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
2289	-----	-----	-----	-----	-----	-----
2293	-----	-----	-----	-----	-----	-----
2310	not detected	not detected	not detected	not detected	not detected	not detected
2311	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2326	ND	ND	ND	ND	ND	ND
2347	out of capability	out of capability	out of capability	out of capability	out of capability	out of capability
2350	< 0.125	< 0.125	< 0.125	< 0.125	< 0.125	< 0.125
2352	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----
2358	-----	-----	-----	-----	-----	-----
2363	not detected	not detected	not detected	not detected	not detected	not detected
2365	＜0.1	＜0.1	＜0.1	＜0.1	＜0.1	＜0.1
2366	out capability	out capability	out capability	out capability	out capability	out capability
2370	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2375	-----	-----	-----	-----	-----	-----
2378	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05
2380	<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
2415	not detected	not detected	not detected	not detected	not detected	not detected
2426	ND	ND	ND	ND	ND	ND
2449	-----	-----	-----	-----	-----	-----
2453	-----	-----	-----	-----	-----	-----
2459	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2511	-----	-----	-----	-----	-----	-----
2538	-----	-----	-----	-----	-----	-----
2561	-----	-----	-----	-----	-----	-----
2590	-----	-----	-----	-----	-----	-----
2592	-----	-----	-----	-----	-----	-----
2602	-----	-----	-----	-----	-----	-----
2605	<0.10	<0.10	<0.10	<0.10	<0.10	<0.10
2649	-----	-----	-----	-----	-----	-----
2654	-----	-----	-----	-----	-----	-----
2675	not detected	not detected	not detected	not detected	not detected	not detected
2711	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed
2743	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2830	not detected	not detected	not detected	not detected	not detected	not detected
2867	not detected	not detected	not detected	not detected	not detected	not detected
2870	-----	-----	-----	-----	-----	-----
2881	-----	-----	-----	-----	-----	-----
2977	not determined	not determined	not determined	not determined	not determined	not determined
3003	-----	-----	-----	-----	-----	-----
3116	-----	-----	-----	-----	-----	-----
3172	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
3197	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05
3210	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
3237	nd	nd	nd	nd	nd	nd

Other reported Chlorinated Phenols on sample #23585; results in mg/kg -- continued --

lab	2CP	3CP	4CP
210	-----	-----	-----
551	-----	-----	-----
623	Not detected	Not detected	Not detected
841	<0.05	<0.05	<0.05
2115	-----	-----	0.052
2138	-----	-----	-----
2215	not detected	not detected	not detected
2241	<0.1	<0.1	<0.1
2265	< 0,05	< 0,05	< 0,05
2287	<0.5	<0.5	＜0.5
2289	-----	-----	-----
2293	-----	-----	-----
2310	not detected	not detected	not detected
2311	Not Detected	Not Detected	Not Detected
2326	ND	ND	ND
2347	out of capability	out of capability	out of capability
2350	< 0.125	< 0.125	< 0.125
2352	-----	-----	-----
2357	-----	-----	-----
2358	-----	-----	-----
2363	not detected	not detected	not detected
2365	＜0.1	＜0.1	＜0.1
2366	out capability	out capability	out capability
2370	<0.05	<0.05	0.0690
2375	-----	-----	-----
2378	＜0.05	＜0.05	＜0.05
2380	<0.05	<0.05	<0.05
2386	< 0.1	< 0.1	< 0.1
2415	not detected	not detected	not detected
2426	ND	ND	ND
2449	-----	-----	0.06
2453	-----	-----	-----
2459	Not Detected	Not Detected	Not Detected
2511	-----	-----	-----
2538	-----	-----	-----
2561	-----	-----	-----
2590	-----	-----	-----
2592	-----	-----	-----
2602	-----	-----	-----
2605	<0.10	<0.10	<0.10
2649	-----	-----	-----
2654	-----	-----	-----
2675	not detected	not detected	not detected
2711	not analyzed	not analyzed	not analyzed
2743	Not detected	Not detected	Not detected
2830	not detected	not detected	not detected
2867	not detected	not detected	not detected
2870	-----	-----	-----
2881	-----	-----	-----
2977	not determined	not determined	not determined
3003	-----	-----	0.064
3116	-----	-----	-----
3172	< 0.05	< 0.05	< 0.05
3197	<0,05	<0,05	<0,05
3210	<0.05	<0.05	<0.05
3237	nd	nd	nd

APPENDIX 3 Analytical Details

lab	ISO17025 accredited	Sample preparation	Sample intake (g)	Release technique	Extraction technique
210	Yes	Further cut		Steam distillation	---
551	---	---		---	---
623	Yes	Further cut	1	Steam distillation	Soxhlet
841	Yes	Used as received	1g	Steam distillation	---
2115	Yes	Used as received	0.5 g	---	---
2138	Yes	Used as received	about 0.5g	Steam distillation was skipped	Ultrasonic
2215	Yes	Further cut	1g	Steam distillation	Mechanical Shaking
2241	Yes	Further cut	0.5g.	Steam distillation	Mechanical Shaking
2265	No	Further cut	0,5	extraction with Potassium hydroxide 90°C for 16 hours in drying oven	Mechanical Shaking
2287	No	Further cut	0.5g	Steam distillation was skipped	Mechanical Shaking
2289	Yes	Further cut	1.0g	Steam distillation	Mechanical Shaking
2293	---	---		---	---
2310	Yes	Further cut	2	Steam distillation	Mechanical Shaking
2311	Yes	Further cut	1	Steam distillation	Mechanical Shaking
2326	No	Further cut	0.5 G	KOH extraction	Ultrasonic
2347	Yes	Further cut	0.5g	Steam distillation was skipped	Mechanical Shaking
2350	Yes	Further cut	0.5038 g (as test) / 1.9962 g Total 2.5 g	Steam distillation	Mechanical Shaking
2352	Yes	Further cut	0.5g	Steam distillation	Mechanical Shaking
2357	---	---		---	---
2358	Yes	Used as received	1.0g	Steam distillation	Steam distillation
2363	Yes	Further cut	1g	0.1M,KOH,extract 1h, 90℃ 16h	0.1M,KOH,extract 1h, 90℃ 16h
2365	Yes	Further cut	0.5g	Steam distillation	Mechanical Shaking
2366	No	Further cut	0.5g	KOH method	Ultrasonic
2370	Yes	Further cut	1g	Steam distillation	Distillation
2375	Yes	Further cut	1 gram	Steam distillation	Mechanical Shaking
2378	Yes	Further cut	0.5g	Steam distillation	Mechanical Shaking
2380	Yes	Further cut	1.0 g	Steam distillation	Mechanical Shaking
2386	Yes	Further cut	0.5 g	KOH extraction 60min ultrasonic + 16h oven	Ultrasonic
2415	Yes	Further cut	0.5	Other	Thermal Desorption
2426	Yes	Used as received	0.5 gram	Steam distillation	Mechanical Shaking
2449	Yes	Further cut	0.5 gram	Steam distillation was skipped	Ultrasonic
2453	No	Used as received	±1g	Other	Thermal Desorption
2459	Yes	Further cut	1.0 gram	Steam distillation	Stirrer
2511	Yes	Used as received		---	---
2538	Yes	Further cut	0,2 - 0,5 g	Other	Other
2561	Yes	Used as received	1g	Steam distillation	Vapodest
2590	Yes	Used as received	1g	Steam distillation was skipped	Ultrasonic
2592	Yes	Used as received	1.0160	Steam distillation	Mechanical Shaking
2602	Yes	Further cut	0,5	Extraktion with KOH, derivatisation with Acetic anhydride, Derivate is extracted with Isooctane	Mechanical Shaking
2605	Yes	---	2.0000	Steam distillation	Mechanical Shaking
2649	Yes	Further cut	1 gram	---	Ultrasonic
2654	Yes	Further cut	1 gram	Steam distillation	Liquid/liquid extraction
2675	Yes	Further cut	1g	Steam distillation	Mechanical Shaking
2711	No	Further cut	1.0 g	Steam distillation	Mechanical Shaking
2743	Yes	Further cut	1g	Steam distillation	Mechanical Shaking
2830	Yes	Further cut		Steam distillation	---
2867	Yes	Further cut	0.2g	Steam distillation	Mechanical Shaking
2870	Yes	Used as received	1gm	Steam distillation	Mechanical Shaking
2881	Yes	Used as received	1g	Steam distillation	Mechanical Shaking
2977	No	Used as received	1g	Steam distillation was skipped	Mechanical Shaking
3003	Yes	Further grinded	1 gram	Steam distillation was skipped	Thermal Desorption
3116	Yes	Used as received	1	---	Ultrasonic
3172	Yes	---		---	---
3197	Yes	Further cut	0,5 g	Steam distillation	Mechanical Shaking
3210	Yes	Further cut	1g	Steam distillation was skipped	Ultrasonic
3237	Yes	Further cut	0,5	Steam distillation	Mechanical Shaking

APPENDIX 4

Number of participants per country

3 labs in	BANGLADESH
1 lab in	BRAZIL
1 lab in	FRANCE
5 labs in	GERMANY
1 lab in	GUATEMALA
2 labs in	HONG KONG
3 labs in	INDIA
1 lab in	INDONESIA
7 labs in	ITALY
1 lab in	JAPAN
2 labs in	KOREA, Republic of
3 labs in	MOROCCO
12 labs in	P.R. of CHINA
4 labs in	PAKISTAN
1 lab in	POLAND
1 lab in	PORTUGAL
1 lab in	TAIWAN
1 lab in	TUNISIA
3 labs in	TURKEY
1 lab in	UNITED KINGDOM
2 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
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?

Literature

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