Results of Proficiency Test
Chlorinated Phenols in Textile
December 2020

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since the 1990's 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 analysis of Chlorinated Phenols in Textile every year. In 2019 it was decided to separate the proficiency tests on the determination of Ortho-Phenylphenol and Chlorinated Phenols in Textile. During the annual proficiency test program 2020/2021 it was decided to continue the proficiency test of Chlorinated Phenols in Textile.

In this interlaboratory study 76 laboratories in 26 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 3 grams and labelled #20750.
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 a blue jeans hosiery fabric obtained from a third party. The batch was cut into small pieces and after homogenization divided over 100 subsamples of approximately 3 grams each and labelled #20750.
The homogeneity of the subsamples was checked by determination of Pentachlorophenol (PCP) and 2,3,4,5-Tetrachlorophenol (TeCP) in accordance with an in-house test method on 10 stratified randomly selected subsamples.

	Pentachlorophenol in mg/kg	2,3,4,5-Tetrachlorophenol in mg/kg
Sample #20750-1	23.8	14.1
Sample #20750-2	24.2	14.6
Sample #20750-3	23.1	14.9
Sample #20750-4	21.9	13.9
Sample #20750-5	23.4	15.2
Sample #20750-6	21.6	14.0
Sample #20750-7	23.5	15.3
Sample #20750-8	24.1	15.7
Sample #20750-9	20.9	14.0
Sample #20750-10	21.8	13.8

Table 1: homogeneity test results of subsamples #20750

From the above test results the repeatabilities were 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	2,3,4,5-Tetrachlorophenol in mg/kg
r (observed)	3.3	1.9
reference method	iis memo 1601 (see lit. 18)	iis memo 1601 (see lit. 18)
0.3 x R (reference method)	4.4	3.0

Table 2: evaluation of the repeatability of subsamples #20750

The calculated repeatabilities were in agreement with 0.3 times the estimated reproducibility of the reference method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample of #20750 was sent on November 18, 2020.

2.5 ANALYZES

The participants were requested to determine on the sample #20750 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 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 the 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.

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. 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 (dotted line) was projected over the Kernel Density Graph (smooth line) 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. Seven participants did not report any test results and five other participants reported the test results after the final reporting date. Finally, 69 laboratories reported 131 numerical test results. Observed was 1 outlying test results, which is 0.8%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

Not all data sets proved to have a normal Gaussian distribution. These are referred to as “not OK” or “suspect”. The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1 EVALUATION PER COMPONENT

In this section the 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.

Due to the lack of relevant reference test methods and/or precision data for the determination of PCP, calculated reproducibilities were compared with an estimated target reproducibility based on iis PT data of OPP/PCP proficiency tests from 2004 until 2014, iis memo 1601 (see lit.18). 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 to estimate the target reproducibility for the evaluation of the PT test results by iis from 2015 onwards This iis memo was also used for the evaluation of 2,3,4,5-TeCP.

Sample #20750

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

2,3,4,5-Tetrachlorophenol: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility derived from iis memo 1601 (see lit. 18).

Other Chlorinated Phenols: The concentrations of the other chlorinated phenols 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, 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	67	18.0	8.2	12.0
2,3,4,5-Tetrachlorophenol	mg/kg	63	13.2	6.1	9.2

Table 3: reproducibilities of components on sample #20750

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2020 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 2020	December 2019	December 2018	December 2017	2014 - 2016	Target
Pentachlorophenol	16%	25%	26%	28-45%	26-38%	26%
2,3,4,5-Tetrachlorophenol	16%	n.e.	n.e.	n.e.	n.e.	26%
2,3,4,6-Tetrachlorophenol	n.e.	24%	n.e.	n.e.	n.e.	26%

Table 4: comparison of uncertainties in iis proficiency tests

The uncertainty in this proficiency test is smaller than observed in previous Chlorinated Phenols in textile proficiency tests.

4.4 EVALUATION OF ANALYTICAL DETAILS

The reported analytical details from the participants are listed in appendix 3. About 75% 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 75% of the reporting laboratories used between 0.5 and 1 gram.
Prior to analysis the samples were further cut by about 60% of the participants while 30% of the other participants reported to use the sample as received.
Ultrasonic extraction, Steam distillation and alkaline digestion were most often reported techniques for extraction by the participants, respectively 40%, 20% and 15%.

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 noticed that almost all participants were able to detect PCP and 2,3,4,5-Tetrachlorophenol in the sample.

Further it could be noticed that for sample #20750 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 #20750 all laboratories would have rejected the sample for all Ecolabel Classes, based on the sum of TeCPs <0.5 mg/kg.
Regarding the “sum of TrCPs” on samples #20750 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 5: Ecolabelling Standards and Requirements for Textiles in EU

6 CONCLUSION

In this proficiency test, the Pentachlorophenol, Tetrachlorophenols and Trichlorophenols content were determined. The variation observed for PCP in sample #20750 is better in comparison with the observations in the previous proficiency tests.

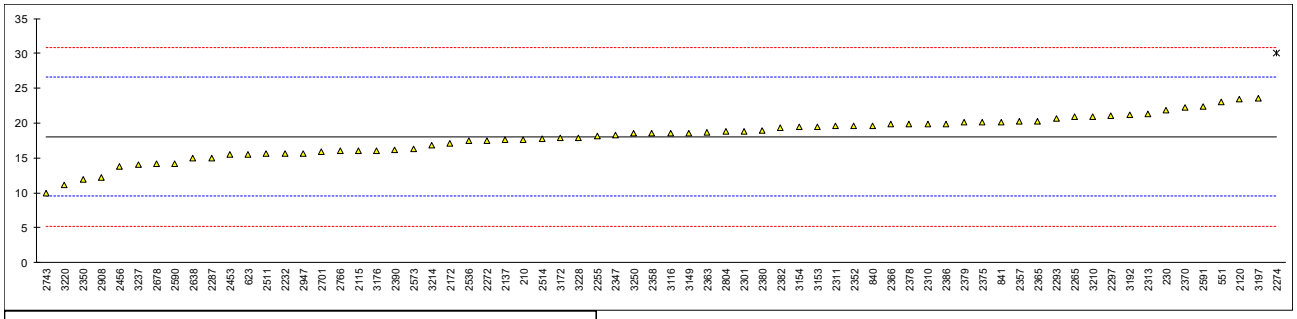
Each laboratory should evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

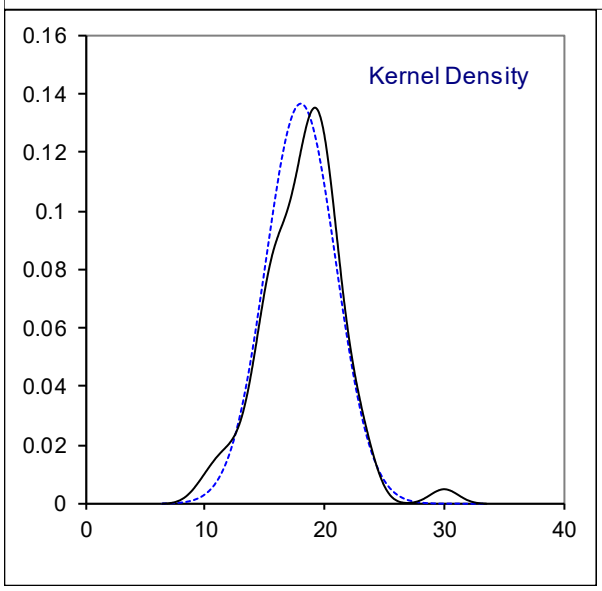
APPENDIX 1

Determination of Pentachlorophenol (PCP) on sample #20750; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	In house	17.58		-0.11	
230	LFGB B82.02.8	21.88044		0.90	
551		23.0		1.16	
623	LFGB B82.02.8	15.470		-0.60	
840	LFGB B82.02.8	19.66		0.38	
841	LFGB B82.02.8	20.121		0.49	
2115	LFGB B82.02.8	16.05		-0.47	
2120	LFGB B82.02.8	23.4		1.25	
2129		-----		-----	
2137	KS K0733	17.560		-0.11	
2172	In house	17.045		-0.23	
2232	In house	15.65447		-0.56	
2255	LFGB B82.02.8	18.20		0.04	
2265	In house	20.892		0.67	
2272	XP G08-015	17.53		-0.12	
2274		30.022	R(0.01)	2.80	
2287	LFGB B82.02.8	15.00		-0.71	
2293	In house	20.667		0.61	
2297	LFGB B82.02.8	21.01		0.69	
2301	LFGB B82.02.8	18.82		0.18	
2310	LFGB B82.02.8	19.9		0.43	
2311	LFGB B82.02.8	19.5513		0.35	
2313	LFGB B82.02.8	21.33		0.77	
2347	LFGB B82.02.8	18.3		0.06	
2350	In house	11.913		-1.44	
2352	In house	19.57		0.36	
2357	In house	20.20		0.50	
2358	In house	18.5537		0.12	
2363	In house	18.7		0.15	
2365	In house	20.21		0.51	
2366	LFGB B82.02.8	19.8		0.41	
2370	LFGB B82.02.8	22.2		0.97	
2375	In house	20.1		0.48	
2378	LFGB B82.02.8	19.87		0.43	
2379	LFGB B82.02.8	20.0919		0.48	
2380	LFGB B82.02.8Mod.	18.943		0.21	
2382	LFGB B82.02.8	19.37		0.31	
2386	In house	19.92		0.44	
2390	LFGB B82.02.8	16.19833		-0.43	
2449		-----		-----	
2452		-----		-----	
2453	LFGB B82.02.8	15.44		-0.61	
2456	UNI11057	13.72		-1.01	
2459		-----		-----	
2511		15.578		-0.58	
2514	In house	17.799		-0.06	
2536	In house	17.481		-0.13	
2561		-----		-----	
2573	LFGB B82.02.8	16.33		-0.40	
2590	LFGB B82.02.8	14.227		-0.89	
2591		22.364		1.01	
2614		-----		-----	
2638	In house	14.943		-0.73	
2644		-----		-----	
2678	UNI11057	14.16		-0.91	
2701		15.93		-0.49	
2743	ISO17070	10.0004		-1.88	
2766	LFGB B82.02.8	16.03		-0.47	
2804	DIN50009	18.8		0.18	
2908		12.22		-1.36	
2947	In house	15.68		-0.55	
3116	LFGB B82.02.8	18.600		0.13	
3118		-----		-----	
3149	In house	18.6		0.13	
3153	LFGB B82.02.8	19.45		0.33	
3154	In house	19.41	C	0.32	First reported 1.91
3172	In house	17.9		-0.03	
3176	In house	16.09		-0.46	
3192	DIN50009	21.15		0.73	
3197	LFGB B82.02.8	23.50		1.28	
3210	In house	20.95		0.68	
3214	LFGB B82.02.8	16.79		-0.29	
3220	In house	11.164		-1.61	
3228	In house	17.9		-0.03	

lab	method	value	mark	z(targ)	remarks
3237	LMBG B82.02-8	14		-0.95	
3250	LFGB B82.02.8	18.53		0.11	
	normality	OK			
	n	67			
	outliers	1			
	mean (n)	18.045			
	st.dev. (n)	2.9194	RSD = 16%		
	R(calc.)	8.174			
	st.dev.(iis memo 1601)	4.2722			
	R(iis memo 1601)	11.962			

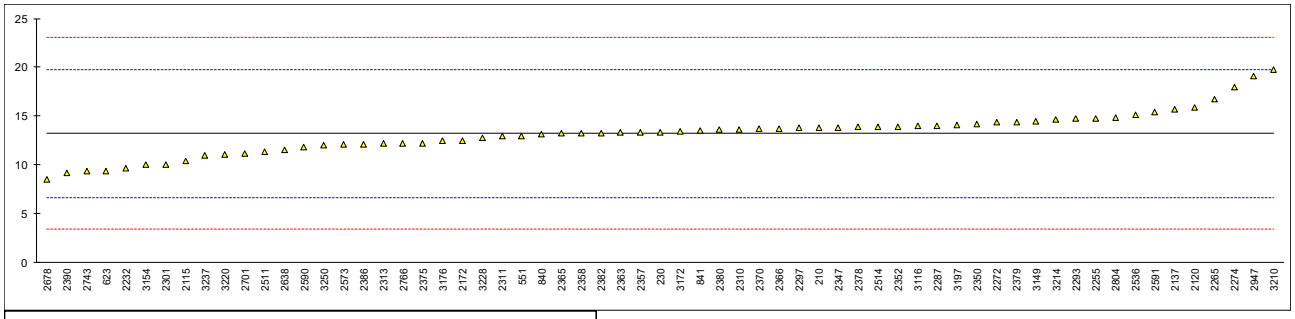


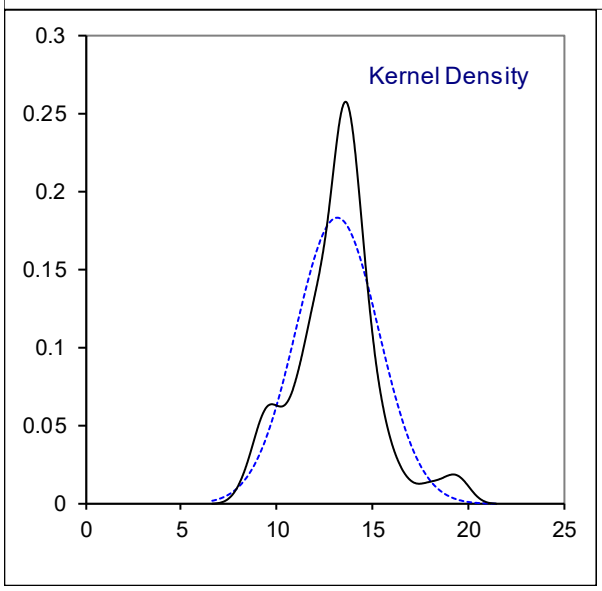


Determination of 2,3,4,5-Tetrachlorophenol on sample #20750; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	In house	13.8		0.18	
230	LFGB B82.02.8	13.37849		0.05	
551		0.13		-0.07	
623	LFGB B82.02.8	9.366		-1.17	
840	LFGB B82.02.8	13.11		-0.03	
841	LFGB B82.02.8	13.544		0.10	
2115	LFGB B82.02.8	10.45		-0.84	
2120	LFGB B82.02.8	15.9	C	0.82	First reported <0.03
2129		-----		-----	
2137	KS K0733	15.691		0.76	
2172	In house	12.508		-0.22	
2232	In house	9.65789		-1.08	
2255	LFGB B82.02.8	14.75		0.47	
2265	In house	16.693		1.06	
2272	XP G08-015	14.39		0.36	
2274		17.974		1.45	
2287	LFGB B82.02.8	14.00		0.24	
2293	In house	14.721		0.46	
2297	LFGB B82.02.8	13.78		0.17	
2301	LFGB B82.02.8	10.01		-0.98	
2310	LFGB B82.02.8	13.6		0.12	
2311	LFGB B82.02.8	12.9998		-0.07	
2313	LFGB B82.02.8	12.17		-0.32	
2347	LFGB B82.02.8	13.8		0.18	
2350	In house	14.204		0.30	
2352	In house	13.94		0.22	
2357	In house	13.31		0.03	
2358	In house	13.2264		0.00	
2363	In house	13.3		0.03	
2365	In house	13.21		0.00	
2366	LFGB B82.02.8	13.7		0.15	
2370	LFGB B82.02.8	13.7		0.15	
2375	In house	12.2		-0.31	
2378	LFGB B82.02.8	13.87		0.20	
2379	LFGB B82.02.8	14.4067		0.36	
2380	LFGB B82.02.8Mod.	13.578		0.11	
2382	LFGB B82.02.8	13.28		0.02	
2386	In house	12.14		-0.33	
2390	LFGB B82.02.8	9.19863		-1.22	
2449		-----		-----	
2452		-----		-----	
2453		-----		-----	
2456		-----		-----	
2459		-----		-----	
2511		11.387		-0.56	
2514	In house	13.874		0.20	
2536	In house	15.173		0.60	
2561		-----		-----	
2573	LFGB B82.02.8	12.13		-0.33	
2590	LFGB B82.02.8	11.787		-0.44	
2591		15.381		0.66	
2614		-----		-----	
2638	In house	11.536		-0.51	
2644		-----		-----	
2678	UNI11057	8.50		-1.44	
2701		11.12		-0.64	
2743	ISO17070	9.3632		-1.17	
2766	LFGB B82.02.8	12.19		-0.31	
2804	DIN50009	14.8		0.48	
2908		-----		-----	
2947	In house	19.06		1.78	
3116	LFGB B82.02.8	13.972		0.23	
3118		-----		-----	
3149	In house	14.5		0.39	
3153		-----		-----	
3154	In house	9.99	C	-0.98	First reported 1.31
3172	In house	13.4		0.06	
3176	In house	12.44		-0.24	
3192		-----		-----	
3197	LFGB B82.02.8	14.1	C	0.27	First reported 19.1
3210	In house	19.72		1.98	
3214	LFGB B82.02.8	14.66		0.44	
3220	In house	11.038		-0.66	
3228	In house	12.8		-0.13	

lab	method	value	mark	z(targ)	remarks
3237	LMBG B82.02-8	11		-0.68	
3250	LFGB B82.02.8	12.01		-0.37	
	normality	suspect			
	n	63			
	outliers	0			
	mean (n)	13.214			
	st.dev. (n)	2.1724	RSD = 16%		
	R(calc.)	6.083			
	st.dev.(iis memo 1601)	3.2782			
	R(iis memo 1601)	9.179			





APPENDIX 2 Other reported test results

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 #20750; in mg/kg

lab	2346-TeCP		2356-TeCP	234-TCP	235-TCP	236-TCP	245-TCP	246-TCP	345-TCP	Other
210	-----		-----	-----	-----	-----	-----	-----	-----	-----
230	not det.		not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.
551	-----		-----	-----	-----	-----	-----	-----	-----	-----
623	0.147		0.050	not det.	not det.	not det.	0.105	not det.	not det.	not det.
840	0.27		0.16	not det.	not det.	not det.	not det.	not det.	not det.	not det.
841	0.261		0.157	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2115	< 0.02	C	0.02	not det.	not det.	not det.	not det.	not det.	not det.	-----
2120	<0.03		< 0,03	< 0,10	< 0,10	< 0,10	< 0,10	< 0,10	< 0,10	< 0,20
2129	-----		-----	-----	-----	-----	-----	-----	-----	-----
2137	-----		-----	-----	-----	-----	-----	-----	-----	-----
2172	-----		-----	-----	-----	-----	-----	-----	-----	-----
2232	-----		-----	-----	-----	-----	-----	-----	-----	0.14289
2255	<0.05		<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	0.16
2265	< 0,1		< 0,1	< 0,1	< 0,1	< 0,1	< 0,1	< 0,1	< 0,1	< 0,1
2272	-----		-----	-----	-----	-----	-----	-----	-----	-----
2274	ND		ND	ND	ND	ND	ND	ND	ND	ND
2287	-----		-----	-----	-----	-----	-----	-----	-----	-----
2293	not det.		not det.	not det.	not det.	not det.	not det.	not det.	not det.	0.315
2297	<0.5		<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	-----
2301	not det.		not det.	not det.	not det.	not det.	not det.	not det.	not det.	0.12
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	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	-----
2350	0.187		-----	-----	-----	-----	-----	-----	-----	-----
2352	-----		-----	-----	-----	-----	-----	-----	-----	-----
2357	-----		-----	-----	-----	-----	-----	-----	-----	-----
2358	n.d.		n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2363	<0.05		<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2365	<0.05		<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2366	<0.5		<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	ND
2370	<0.05		<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2375	-----		-----	-----	-----	-----	-----	-----	-----	-----
2378	-----		-----	-----	-----	-----	-----	-----	-----	-----
2379	0.0545		0.0686	not det.	not det.	not det.	not det.	not det.	not det.	ND
2380	<0.05		<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	-----
2382	<0.010		<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010	<0.010
2386	<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.
2449	-----		-----	-----	-----	-----	-----	-----	-----	-----
2452	-----		-----	-----	-----	-----	-----	-----	-----	-----
2453	-----		-----	-----	-----	-----	-----	-----	-----	-----
2456	-----		13.88	-----	-----	-----	-----	-----	-----	-----
2459	1.9375		-----	-----	-----	-----	-----	-----	-----	-----
2511	-----		-----	-----	-----	-----	-----	-----	-----	-----
2514	-----		-----	-----	-----	-----	-----	-----	-----	0.183
2536	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.
2590	-----		-----	-----	-----	-----	-----	-----	-----	-----
2591	not det.		not det.	-----	-----	-----	-----	-----	-----	-----
2614	-----		-----	-----	-----	-----	-----	-----	-----	-----
2638	not det.		not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.
2644	-----		-----	-----	-----	-----	-----	-----	-----	-----
2678	not det.		not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.
2701	-----		-----	-----	-----	-----	-----	-----	-----	-----

lab	2346-TeCP	2356-TeCP	234-TCP	235-TCP	236-TCP	245-TCP	246-TCP	345-TCP	Other
2743	-----	0.0691	-----	-----	-----	-----	0.0720	-----	0.0704
2766	ND	ND	ND	ND	ND	ND	ND	ND	ND
2804	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	-----
2908	-----	9.24	-----	-----	-----	-----	-----	-----	-----
2947	below 0.05	below 0.05	below 0.05	below 0.05	below 0.05	below 0.05	below 0.05	below 0.05	below 0.05
3116	0.1050	0.0860	-----	-----	-----	-----	-----	-----	-----
3118	-----	-----	-----	-----	-----	-----	-----	-----	-----
3149	-----	-----	-----	-----	-----	-----	-----	-----	-----
3153	-----	-----	-----	-----	-----	-----	-----	-----	-----
3154	-----	-----	-----	-----	-----	-----	-----	-----	-----
3172	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
3176	-----	-----	-----	-----	-----	-----	-----	-----	-----
3192	-----	-----	-----	-----	-----	-----	-----	-----	-----
3197	ND	ND	ND	ND	ND	ND	ND	ND	-----
3210	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100	<0.100
3214	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	-----
3220	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.
3228	not det.	not det.	not det.	not det.	not det.	not det.	not det.	not det.	-----
3237	-----	-----	-----	-----	-----	-----	-----	-----	-----
3250	-----	-----	-----	-----	-----	-----	-----	-----	-----

Lab 2115: first reported 15.9

APPENDIX 3 Analytical details

lab	ISO/IEC17025 accredited	Sample	Sample intake (grams)	Extraction technique	Extraction solution
210	No	---		---	
230	Yes	Further cut	1.0000	Ultrasonic	Hexane
551	---	---		---	
623	Yes	Further cut	1 gram	Ultrasonic	n-Hexane
840	Yes	Further cut	1	Ultrasonic	KOH
841	Yes	Further cut	1.0054 grams	Ultrasonic	n-hexane
2115	Yes	As received	1 g	ASE	
2120	No	Further cut	1 gram	Steam distillation	
2129	---	---		---	
2137	Yes	As received	3	Ultrasonic	HEXANE
2172	Yes	Further cut	0.5g	Oven extract	KOH
2232	Yes	Further cut	1	Alkaline incubation	KOH hexane
2255	Yes	Further cut	0.5	Mechanical Shaking	DCM/Hexane
2265	Yes	As received	0,1 - 0,5 g	Mechanical Shaking	KOH / n-Hexane
2272	Yes	As received	1 gram	Ultrasonic	K2CO3 solution
2274	No	As received	0.5 g.	Thermostatic batch	0.5 % NH3 solution.
2287	No	Further cut	1.0 g	Alkaline digestion	1 mol/L-KOH solution
2293	Yes	As received	1.0 gram	Mechanical Shaking	n-Hexane
2297	Yes	As received	1g	---	
2301	No	Further cut	1 gram	Mechanical Shaking	KOH & Hexane
2310	Yes	Further cut	2 grams	Steam distillation	Hexane
2311	Yes	Further cut	1	KOH extraction	Hexane
2313	Yes	Further cut	0.5grams	Steam distillation	n-Hexane
2347	Yes	Further cut	0.5g	---	
2350	No	As received	2 g	Thermal Desorption	KOH
2352	Yes	Further cut	0.5g	Mechanical Shaking	Hexane
2357	---	---		---	
2358	---	---		---	
2363	Yes	Further cut	0.5g	Ultrasonic	KOH
2365	Yes	Further cut	1.0g	Oven extract	1mol/L KOH
2366	Yes	Further cut	0.5g	Ultrasonic	KOH
2370	Yes	Further cut	1.5 g	Steam distillation	H2O
2375	Yes	Further cut	0,5 grams	Ultrasonic	KOH Solution
2378	Yes	As received	2.5g	Mechanical Shaking	N-hexane
2379	No	Further cut	0.5 g	Alkaline digestion	KOH
2380	Yes	Further cut	1.0 g	Alkaline digestion + derivatization	n-hexane
2382	Yes	Further cut	2.5g	Steam distillation	Hexane
2386	Yes	Further cut	0.5 g	Ultrasonic	KOH (1M)
2390	Yes	Further cut	1.0gm	Ultrasonic	n-hexane
2449	---	---		---	
2452	---	---		---	
2453	---	---		---	
2456	Yes	Further cut	5g	Ultrasonic	K2CO3 1.5%
2459	Yes	Further cut	1.0 g	Ultrasonic	N-Hexane
2511	---	---		---	
2514	---	---		---	
2536	Yes	Further cut	1.0017	Thermal Desorption	KOH solution
2561	---	---		---	
2573	Yes	As received	0.5g	ASE	acetone
2590	---	---		---	
2591	Yes	As received	1.0	---	
2614	---	---		---	
2638	No	Further cut	Approx 1 gm	Ultrasonic	Hexane
2644	---	---		---	
2678	No	As received	1g	Thermal Desorption, Ultrasonic	Hexane
2701	Yes	As received	0.5021 g	Incubation	KOH
2743	Yes	As received	0.6	Steam distillation	Hexane
2766	Yes	Further cut	2.0 gms	Alkaline digestion	KOH
2804	Yes	As received	1g	Oven heating	KOH

lab	ISO/IEC17025 accredited	Sample	Sample intake (grams)	Extraction technique	Extraction solution
2908	Yes	Further cut	0.5554 grams	Ultrasonic	n-Hexane
2947	No	As received	1g	Microwave	KOH
3116	Yes	As received	1 grams	Oven incubation	KOH
3118	---	---		---	
3149	Yes	Further cut	1g	Soxhlet	Acetone
3153	Yes	Further cut	0.5g	Steam distillation	n-hexane
3154	Yes	---		---	
3172	Yes	Further cut	0.5	Ultrasonic	KOH
3176	Yes	Further cut	1	Ultrasonic	Hexane
3192	Yes	Further cut	0,5 g	Oven extract	KOH
3197	Yes	As received	1 g	Steam distillation	Water/ K2CO3
3210	Yes	As received	1 gram	Ultrasonic	K2CO3
3214	Yes	Further cut	0.5 g	Thermal Desorption	H2SO4
3220	Yes	Further cut	1 GRAMS	Mechanical Shaking	HEXANE
3228	Yes	Further cut	0.5	Oven extraction	KOH
3237	Yes	Further cut	0,5	Steam distillation	Hexane
3250	Yes	Further cut	1g	Mechanical Shaking	n-hexane

APPENDIX 4

Number of participants per country

1 lab in AUSTRIA
4 labs in BANGLADESH
1 lab in BRAZIL
1 lab in CAMBODIA
1 lab in FRANCE
6 labs in GERMANY
1 lab in GUATEMALA
4 labs in HONG KONG
6 labs in INDIA
3 labs in INDONESIA
6 labs in ITALY
1 lab in JAPAN
1 lab in MAURITIUS
2 labs in MOROCCO
14 labs in P.R. of CHINA
4 labs in PAKISTAN
2 labs in PORTUGAL
1 lab in SINGAPORE
3 labs in SOUTH KOREA
1 lab in SPAIN
2 labs in TAIWAN
1 lab in THAILAND
3 labs in TUNISIA
4 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
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

Literature

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2 Oeko-Tex© Standard 100; January 2020
3 Bluesign© label, BSSL, version 6.0, July 2016
4 Impacts of Environmental Standards and requirements in EU Countries, August 1999
5 Horwitz, Journal of AOAC International, 79.3, (1996)
6 P.L. Davies, Fr Z. Anal. Chem, 351, 513, (1988)
7 W.J. Conover, Practical Nonparametric Statistics, J. Wiley&Sons, NY., 302, (1971)
8 ISO5725:86
9 ISO5725. parts 1-6:94
10 ISO13528:05
11 M. Thompson and R. Wood, J. AOAC Int., 76, 926, (1993)
12 Analytical Methods Committee, Technical Brief, No 4, January 2001
13 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst 2002, 127, 1359-1364 (2002)
14 Official Journal of the European Communities L133/29, May 2002
15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)
16 iis memo 1601: Precision data of OPP/PCP in textile, February 18, 2016