Results of Proficiency Test OPP & Chlorinated Phenols in textile December 2018

Organised 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 Ortho-Phenylphenol (OPP), Pentachlorophenol (PCP) and Tetrachlorophenols (TeCPs) in textile every year. During the annual proficiency test program 2018/2019, it was decided to continue this proficiency test and extent the scope with Tri Chlorophenols (TrCPs). Therefore, the proficiency scheme was renamed to OPP and Chlorinated Phenols in textile.
In this interlaboratory study 82 laboratories in 22 different countries registered for participation. See appendix 4 for the number of participants per country. In this report, the results of the 2018 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 organiser of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send two different textile samples, labelled #18650 and #18651 of 3 grams each, which were positive on OPP and/or some Chlorinated Phenols. 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/IEC 17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

The first batch, a light beige cotton fabric positive on OPP obtained from a third party, was cut into pieces. From this batch, after mixing well, 100 subsamples of approximately 3 grams each were packed and labelled #18650. The homogeneity of 8 stratified randomly selected samples was checked by determination of OPP in accordance with an in-house test method. See the following table for the test results.

	OPP in mg/kg
Sample #18650-1	43.0
Sample #18650-2	45.8
Sample #18650-3	42.3
Sample #18650-4	44.0
Sample #18650-5	44.6
Sample #18650-6	44.5
Sample #18650-7	42.5
Sample #18650-8	49.1

Table 1: homogeneity test results of subsamples #18650

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility of the reference method in agreement with the procedure of ISO 13528, Annex B2 in the next table.

	OPP in mg/kg
r (observed)	6.2
reference method	iis memo 1601 (see lit. 18)
0.3 x R (reference method)	7.7

Table 2: evaluation of the repeatability of subsamples #18650

The calculated repeatability of Ortho-Phenylphenol (OPP) was in agreement with 0.3 times the reference method. Therefore, homogeneity of the subsamples was assumed.

The second batch, a black ribbed cotton fabric positive on PCP also obtained from a third party, was cut into pieces. From this batch, after mixing well, 100 subsamples of approximately 3 grams each were packed and labelled #18651. The homogeneity of 8 stratified randomly selected samples was checked by determination of PCP in accordance with an in-house test method for PCP. See the following table for the test results.

	PCP in mg/kg
Sample #18651-1	10.11
Sample #18651-2	10.40
Sample #18651-3	10.70
Sample #18651-4	10.49
Sample #18651-5	10.38
Sample #18651-6	10.76
Sample #18651-7	10.57
Sample #18651-8	10.88

Table 3: homogeneity test results of subsamples #18651

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility of the reference method in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	PCP in mg/kg
r (observed)	0.69
reference method	iis memo 1601 (see lit.18)
0.3 x R (reference method)	2.27

Table 4: evaluation of the repeatability of subsamples #18651

The calculated repeatability of Pentachlorophenol (PCP) was in agreement with 0.3 times the reference method. Therefore, homogeneity of the subsamples was assumed.

To each participating laboratory 1 sample labelled #18650 and 1 sample labelled #18651 were sent on November 14, 2018.

2.5 ANALYSES

The participants were requested to determine on samples #18650 and #18651 the concentrations of Ortho-Phenylphenol (OPP), Pentachlorophenol (PCP), Trichlorophenols, Tetrachlorophenols and other Chlorinated Phenols applying the analysis procedure that is routinely used in the laboratory. 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 samples as if they were routine samples 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 reanalyses). 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 Organization, 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 ISO 5725 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 visualise 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 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 occurred with the dispatch of the samples. However, one participant did not report any test results at all. All other participants reported before the final reporting date. Not all laboratories were able to report all compaonents requested. In total 81 laboratories reported 208 numerical test results. Observed were 8 outlying test results, which is 3.8%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

Not all original 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 SAMPLE AND PER COMPONENT

In this section, the test results are discussed per sample and 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 listed in appendix 5.

Due to the lack of relevant reference test methods and/or precision data for the determination of OPP and 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 OPP/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 to estimate the target reproducibilities for the evaluation of the PT test results by iis from 2015 onwards (see lit.18).

Sample #18650

OPP: This determination may be problematic for a number of laboratories. Five statistical outliers were observed. However, the calculated reproducibility after rejection of the outliers is in agreement with the estimated reproducibility found in previous iis PTs (iis memo 1601).

Other Chlorinated Phenols: The concentrations reported were near or below the detection limit, see appendix 2. Therefore, no z-scores were calculated.

Sample #18651

OPP: This determination was problematic at the low level of 2.1 mg/kg OPP. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the estimated reproducibility found in previous iis PTs (iis memo 1601).

PCP: This determination was not problematic at the level of 9.3 mg/kg PCP. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the estimated reproducibility found in previous iis PTs (iis memo 1601).

Other Chlorinated Phenols: The concentrations reported were near or below the detection limit, see appendix 2. Therefore, no z-scores were calculated.

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.

	unit	n	average	2.8 * sd	R (lit)
OPP	mg/kg	64	20.6	9.1	13.4

Table 5: reproducibility of OPP on sample #18650

	unit	n	average	2.8 * sd	R (lit)
OPP	mg/kg	58	2.13	3.44	1.95
PCP	mg/kg	78	9.28	6.82	6.80

Table 6: reproducibilities of OPP and PCP on sample #18651

Without further statistical calculations, it can be concluded that the total group of participating laboratories have no difficulties with the analysis of OPP at a level of 21 mg/kg and PCP at a level of 9 mg/kg. See also the discussion in paragraphs 4.1 and 5.

4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2018 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.

	December 2018	December 2017	December 2016	November 2015	November 2014	iis memo 1601
OPP	16-58%	39-54%	38%	24%	27%	24%
PCP	26%	28-45%	28%	38%	26%	26%

Table 7: 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 73% of the participating laboratories reported to be accredited for the determination of OPP and/or PCP in textile. The amount of sample intake varied between 0.2 and 3 grams with an average of around 1 gram. Prior to analysis the samples were further cut by 59 participants while 13 participants reported to use the sample as received. Ultrasonic extraction was the most often used technique by 30 participants followed by Steam distillation (18 participants), KOH extraction (14 participants) and Other extraction methods (11 participants).

5 DISCUSSION

The effect of the reported analytical details (see appendix 3) on the determination of OPP in sample #18650 and PCP in sample #18651 were further investigated, see table 8 for OPP and table 9 for PCP.

Analytical Details	unit	n	average	sd
ISO/IEC 17025 accredited	mg/kg	50	20.4	3.51
Not ISO/IEC 17025 accredited	mg/kg	10	21.5	2.49
Ultrasonic extraction	mg/kg	23	21.0	3.89
Steam distillation	mg/kg	14	18.8	2.31
KOH extraction	mg/kg	11	22.5	3.55
<1g sample intake	mg/kg	16	22.0	3.52
1g sample intake	mg/kg	38	20.0	3.27
>1g sample intake	mg/kg	4	20.9	3.38
Further cut (prior to analysis)	mg/kg	50	20.9	2.95
Used as received	mg/kg	8	20.1	4.43

Table 8: effect of analytical details on OPP textile sample #18650

Analytical Details	unit	n	average	sd
ISO/IEC 17025 accredited	mg/kg	60	9.3	2.24
Not ISO/IEC 17025 accredited	mg/kg	13	9.5	3.39
Ultrasonic extraction	mg/kg	30	9.5	2.36
Steam distillation	mg/kg	17	8.3	2.53
KOH extraction	mg/kg	14	10.2	2.84
<1g sample intake	mg/kg	22	9.5	2.59
1g sample intake	mg/kg	42	9.3	2.64
>1g sample intake	mg/kg	6	9.4	1.07
Further cut (prior to analysis)	mg/kg	59	9.4	2.54
Used as received	mg/kg	12	9.3	2.26

Table 9: effect of analytical details on PCP textile sample #18651

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

When the test results of this interlaboratory study were compared to the Ecolabelling Standards and Requirements for Textiles in EU (see table 10) it could be noticed that the majority of the participants was able to detect OPP in both samples #18650 and #18651 and PCP in sample #18651.
All reported test values for OPP were <50.0 mg/kg for both samples #18650 and #18651. Thus, both textile materials would have been accepted based on the OPP analyses for all four classes mentioned in table 10 by all reporting laboratories.

Further it could be noticed that for sample #18650 all reported test values for PCP are below 0.5 mg/kg (except one test result, see appendix 2). Thus, on the basis of PCP level this textile material would have been accepted for Ecolabel classes 2 to 4 at least. For baby clothes the maximum PCP level is 0.05 mg/kg. Based on this PCP level this textile material would have been accepted for Ecolabel class 1 by ten laboratories. For sample #18651 all reported test values for PCP are above 0.5 mg/kg (except one test result, see appendix 1). Thus, this textile material would have been rejected for all classes by all reported laboratories, except one.

Regarding the sum of TeCPs on samples #18650 and #18651 all laboratories would have accepted the samples for Ecolabel Class 2 to 4, based on the sum of TeCPs <0.5 mg/kg. For Ecolabel Class 1 eleven laboratories would accept both samples based on the sum of TeCPs <0.05 mg/kg or a “less than” result.
Regarding the sum of TrCPs on samples #18650 and #18651 all (except for two) 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)
Ortho-Phenylphenol	50.0	100.0	100.0	100.0
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 10: Ecolabelling Standards and Requirements for Textiles in EU

Sample #18650 was used before in Proficiency Test iis16A11 as sample #16645. It is observed that the PT findings of the subsamples of textile containing OPP give a good correlation on the average OPP content, and that the calculated reproducibility in the current PT is much better.

	unit	#18650			#16645		
		n	average	R(calc)	n	average	R(calc)
OPP	mg/kg	64	20.6	9.1	74	18.0	19.3

Table 11: comparison of sample #18650 with #16645

6 CONCLUSION

In this proficiency test, the OPP, PCP, TeCPs and TrCPs content were determined. The variation observed for OPP in sample #18650 in this interlaboratory study is low compared to observations in the previous proficiency tests. However, the variation observed for OPP in sample #18651 is high compared to observations in the previous proficiency tests. This could be due to the low level of OPP in this sample.
The variation observed for PCP in sample #18651 is in line with the observations in the previous proficiency tests.

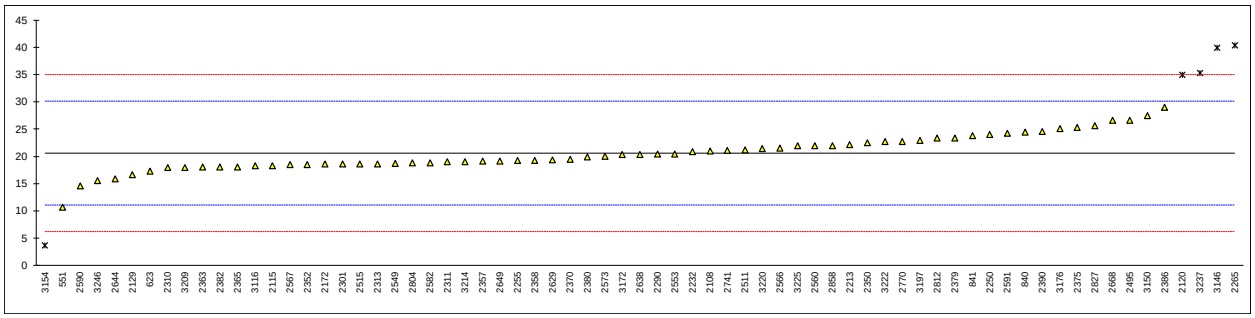
A possible explanation for the variation could be the preparation or the conditioning of the sample and/or by the performance of the analysis by the laboratory. Each laboratory should evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation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

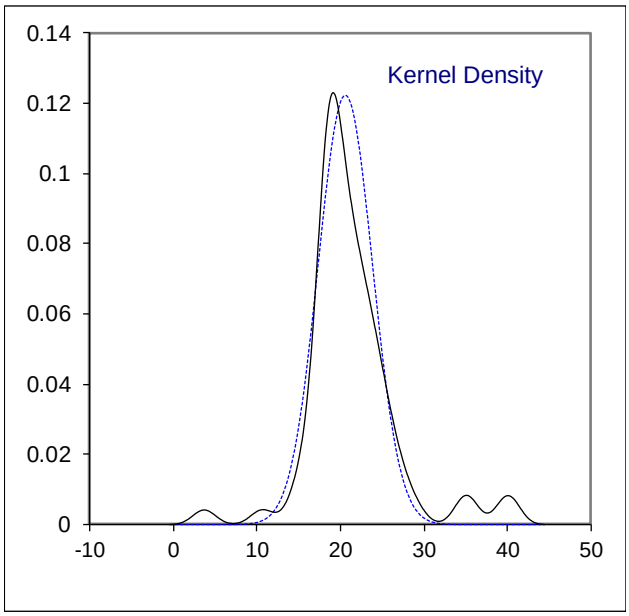
APPENDIX 1

Determination of Ortho-Phenylphenol (OPP) on sample #18650; results in mg/kg

lab	method	value	mark	z(targ)	remarks
213		-----		-----	
551	In house	10.6468		-2.08	
623	LFGB B62.02.8Mod.	17.26		-0.70	
840	LFGB B82.02.8	24.42		0.80	
841	LFGB B82.02.8	23.83		0.67	
2108	In house	20.97		0.07	
2115	LFGB B82.02.8	18.261		-0.49	
2120	LFGB B82.02.8	35.0	R(0.01)	3.01	
2129	EN17070Mod.	16.6		-0.84	
2165		-----		-----	
2172	In house	18.549		-0.43	
2184		-----		-----	
2213	LFGB B82.02.8	22.15		0.32	
2232	LFGB B82.02.8	20.84339		0.05	
2250	In house	24.0		0.71	
2255	In house	19.2		-0.30	
2265		40.383	R(0.01)	4.13	
2290	In house	20.41		-0.04	
2301	LFGB B82.02.8	18.62		-0.42	
2310	LFGB B82.02.8	17.9		-0.57	
2311	LFGB B82.02.8	19.013		-0.33	
2313	LFGB B82.02.8	18.634		-0.41	
2330		-----		-----	
2350	In house	22.5091		0.40	
2352	In house	18.52		-0.44	
2357	In house	19.171		-0.30	
2358	In house	19.3		-0.27	
2363	In house	18.07		-0.53	
2365	In house	18.114		-0.52	
2370	In house	19.5		-0.23	
2375	In house	25.26		0.97	
2379	LFGB B82.02.8	23.406	C	0.58	first reported 1.845
2380	LFGB B82.02.8	19.898		-0.15	
2382	In house	18.11		-0.52	
2386	In house	28.94		1.74	
2390	In house	24.52		0.82	
2453		-----		-----	
2495	In house	26.58		1.25	
2511		21.211		0.12	
2515	LFGB B82.02.8	18.623		-0.42	
2549	In house	18.679		-0.40	
2553	In house	20.42		-0.04	
2560	LFGB B82.02.8	21.94		0.28	
2566	LFGB B82.02.8	21.5		0.19	
2567	In house	18.5		-0.44	
2573	ISO17070	20.03		-0.12	
2582	In house	18.86		-0.37	
2590	ISO17070	14.55		-1.27	
2591	In house	24.264		0.76	
2629	LFGB B82.02.8	19.34	C	-0.27	first reported 5.9
2638	ISO17070	20.3495		-0.06	
2644	UNI11057	15.9		-0.99	
2649	In house	19.19		-0.30	
2654		-----		-----	
2668	ISO17070	26.56		1.24	
2730		-----		-----	
2741	In house	21.06		0.09	
2767		-----		-----	
2770	GB/T20386	22.72		0.44	
2804	In house	18.8		-0.38	
2812	LFGB B82.02.8	23.33		0.57	
2827	LFGB B82.02.8	25.61		1.04	
2830		-----		-----	
2852		-----		-----	
2858	In house	22.0		0.29	
3116	LFGB B82.02.8	18.23		-0.50	
3146	In house	39.90	R(0.01)	4.03	
3150	ISO17070	27.43		1.42	
3153		-----		-----	
3154		3.676	R(0.01)	-3.54	
3172	KS K0733	20.31		-0.06	
3176	LFGB B82.02.8	25.13		0.94	
3179		-----		-----	
3197	LFGB B82.02.8	22.94		0.49	

lab	method	value	mark	z(targ)	remarks
3209	In house	17.92		-0.56	
3210	In house	<40		-----	
3214	ISO17070	19.033		-0.33	
3220	In house	21.425		0.17	
3222	UNI11057Mod.	22.71		0.44	
3225	ISO17070	21.93		0.28	
3237	LFGB B82.02.8	35.29	R(0.01)	3.07	
3246	ISO17070	15.6		-1.05	
	normality	OK			
	n	64			
	outliers	5			
	mean (n)	20.614			
	st.dev. (n)	3.2590	RSD=16%		
	R(calc.)	9.125			
	st.dev.(iis-memo 1601)	4.7841			
	R(iis-memo 1601)	13.395			
Compare					
	R(Horwitz)	5.857			

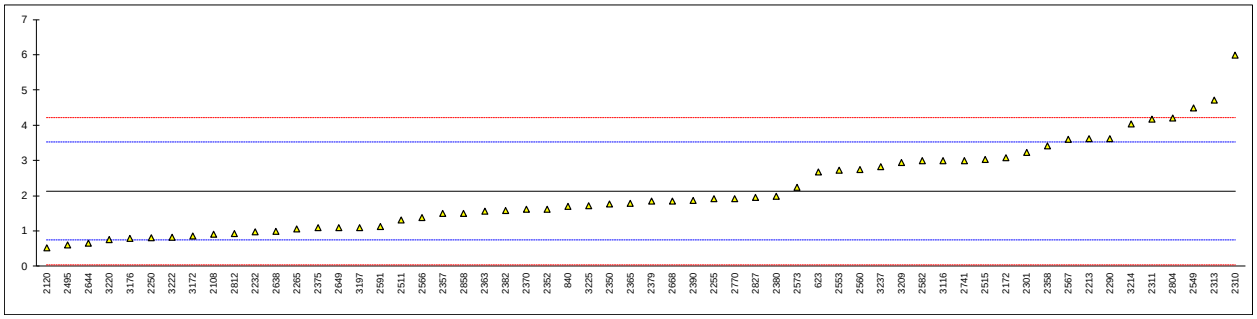


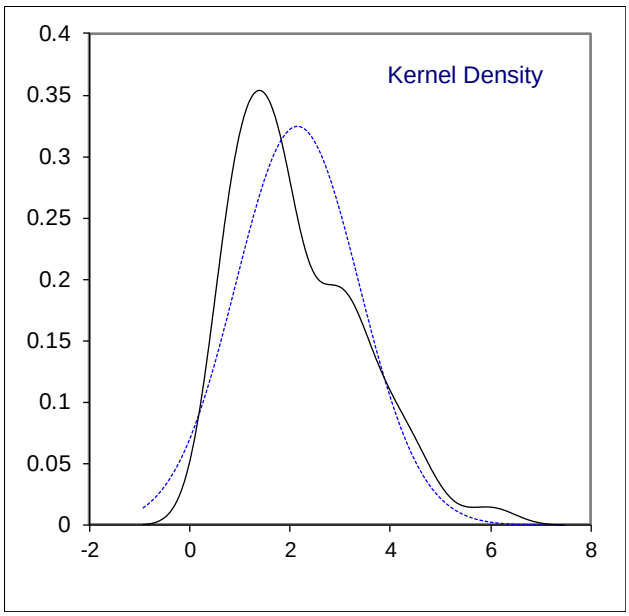


Determination of Ortho-Phenylphenol (OPP) on sample #18651; results in mg/kg

lab	method	value	mark	z(targ)	remarks
213		-----		-----	
551	In house	N.D.		-----	
623		2.68		0.78	
840	LFGB B82.02.8	1.69		-0.64	
841	LFGB B82.02.8	ND		-----	
2108		0.901		-1.77	
2115		-----		-----	
2120	LFGB B82.02.8	0.527		-2.31	
2129	EN17070Mod.	<5		-----	
2165		-----		-----	
2172	In house	3.083		1.36	
2184		-----		-----	
2213	LFGB B82.02.8	3.61		2.12	
2232	LFGB B82.02.8	0.9773249		-1.66	
2250	In house	0.81		-1.90	
2255	In house	1.92		-0.31	
2265		1.054		-1.55	
2290	In house	3.62		2.13	
2301	LFGB B82.02.8	3.23		1.57	
2310	LFGB B82.02.8	5.99		5.54	
2311	LFGB B82.02.8	4.171		2.93	
2313	LFGB B82.02.8	4.712		3.70	
2330		-----		-----	
2350	In house	1.7697		-0.52	
2352	LFGB B82.02.8	1.62		-0.74	
2357	LFGB B82.02.8	1.492		-0.92	
2358	In house	3.42		1.85	
2363	In house	1.57		-0.81	
2365	In house	1.780		-0.51	
2370	In house	1.62		-0.74	
2375	In house	1.09		-1.50	
2379	LFGB B82.02.8	1.845	C	-0.42	first reported 14.808
2380	LFGB B82.02.8	1.985		-0.21	
2382	LFGB B82.02.8	1.58		-0.80	
2386		<0,5		-----	
2390	In house	1.86		-0.39	
2453		-----		-----	
2495	In house	0.60		-2.20	
2511		1.312		-1.18	
2515	LFGB B82.02.8	3.022		1.28	
2549	In house	4.5		3.40	
2553	In house	2.72		0.84	
2560	LFGB B82.02.8	2.74		0.87	
2566	LFGB B82.02.8	1.37		-1.10	
2567	In house	3.6		2.11	
2573	ISO17070	2.24		0.15	
2582	In house	2.993		1.23	
2590		-----		-----	
2591	In house	1.131		-1.44	
2629	LFGB B82.02.8	<0.5	C	-----	first reported 9.8
2638	ISO17070	0.992		-1.64	
2644	UNI11057	0.65		-2.13	
2649	In house	1.10		-1.49	
2654		-----		-----	
2668	ISO17070	1.85		-0.41	
2730		-----		-----	
2741	In house	3.00		1.24	
2767		-----		-----	
2770		1.92		-0.31	
2804	In house	4.20		2.97	
2812		0.92		-1.74	
2827	LFGB B82.02.8	1.95		-0.26	
2830		-----		-----	
2852		-----		-----	
2858	In house	1.50		-0.91	
3116	LFGB B82.02.8	2.997		1.24	
3146		-----		-----	
3150		<1,0		-----	
3153		-----		-----	
3154		-----		-----	
3172		0.861		-1.83	
3176	LFGB B82.02.8	0.789		-1.93	
3179		-----		-----	
3197	LFGB B82.02.8	1.10		-1.49	
3209	In house	2.951		1.17	

lab	method	value	mark	z(targ)	remarks
3210	In house	<40		-----	
3214	ISO17070	4.047		2.75	
3220	In house	0.763		-1.97	
3222	UNI11057Mod.	0.82		-1.89	
3225	ISO17070	1.71		-0.61	
3237	LFGB B82.02.8	2.83		1.00	
3246	ISO17070	Not detected		-----	
	normality	OK			
	n	58			
	outliers	0			
	mean (n)	2.134			
	st.dev. (n)	1.2275	RSD=58%		
	R(calc.)	3.437			
	st.dev.(iis-memo 1601)	0.6960			
	R(iis-memo 1601)	1.949			
Compare					
	R(Horwitz)	0.853			

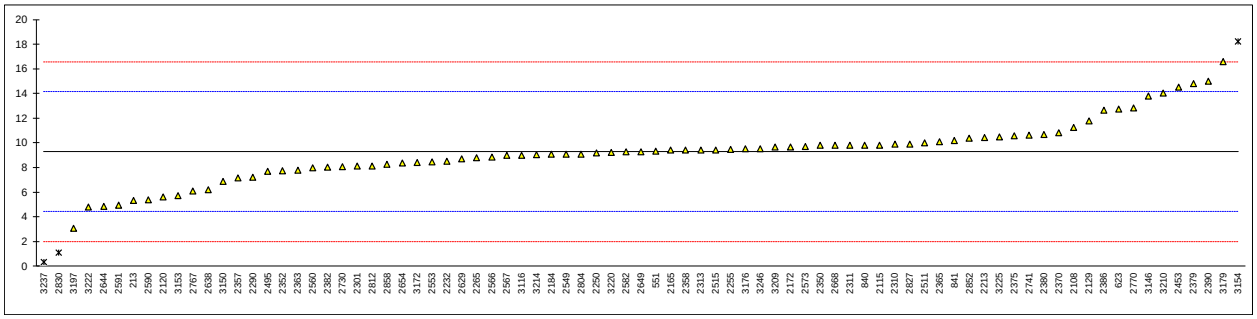


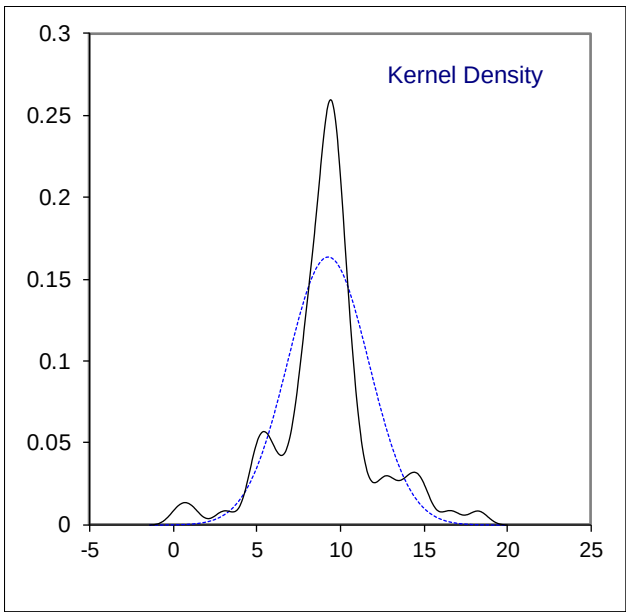


Determination of Pentachlorophenol (PCP) on sample #18651; results in mg/kg

lab	method	value	mark	z(targ)	remarks
213	LFGB B82.02.8	5.35		-1.62	
551	In house	9.3076		0.01	
623		12.76		1.43	
840	LFGB B82.02.8	9.80		0.21	
841	LFGB B82.02.8	10.18		0.37	
2108		11.23		0.80	
2115	LFGB B82.02.8	9.821		0.22	
2120	LFGB B82.02.8	5.60		-1.52	
2129	EN17070Mod.	11.77		1.02	
2165	LFGB B82.02.8	9.4		0.05	
2172	In house	9.661		0.16	
2184	LFGB B82.02.8	9.1		-0.08	
2213	LFGB B82.02.8	10.43		0.47	
2232	LFGB B82.02.8	8.513635		-0.32	
2250	In house	9.2		-0.03	
2255	In house	9.48		0.08	
2265		8.786		-0.20	
2290	In house	7.23		-0.85	
2301	LFGB B82.02.8	8.10		-0.49	
2310	LFGB B82.02.8	9.88		0.25	
2311	LFGB B82.02.8	9.793		0.21	
2313	LFGB B82.02.8	9.412		0.05	
2330		-----		-----	
2350	In house	9.7837		0.21	
2352	LFGB B82.02.8	7.76		-0.63	
2357	LFGB B82.02.8	7.143		-0.88	
2358	In house	9.40		0.05	
2363	LFGB B82.02.8	7.78		-0.62	
2365	LFGB B82.02.8	10.078		0.33	
2370	In house	10.8		0.63	
2375	In house	10.60		0.54	
2379	LFGB B82.02.8	14.808	C	2.28	first reported 23.406
2380	LFGB B82.02.8	10.676		0.57	
2382	LFGB B82.02.8	8.01		-0.52	
2386		12.64		1.38	
2390	In house	14.986		2.35	
2453	LFGB B82.02.8	14.50		2.15	
2495	In house	7.67		-0.66	
2511		10.011		0.30	
2515	LFGB B82.02.8	9.444		0.07	
2549	In house	9.1		-0.08	
2553	In house	8.44		-0.35	
2560	LFGB B82.02.8	7.99		-0.53	
2566	LFGB B82.02.8	8.84		-0.18	
2567	In house	9.0		-0.12	
2573	ISO17070	9.71		0.18	
2582	In house	9.285		0.00	
2590	ISO17070	5.40		-1.60	
2591	In house	4.970		-1.78	
2629	LFGB B82.02.8	8.7		-0.24	
2638	ISO17070	6.215		-1.26	
2644	UNI11057	4.87		-1.82	
2649	In house	9.30		0.01	
2654	XP G 08-015	8.387		-0.37	
2668	ISO17070	9.79		0.21	
2730	XP G 08-015	8.097		-0.49	
2741	In house	10.64		0.56	
2767	LFGB B82.02.8	6.1		-1.31	
2770		12.85		1.47	
2804	In house	9.10		-0.08	
2812		8.11		-0.48	
2827	LFGB B82.02.8	9.91		0.26	
2830	XP G 08-015	1.10	DG(0.05)	-3.37	
2852	XP G 08-015	10.4	C	0.46	first reported 0.52
2858	In house	8.244		-0.43	
3116	LFGB B82.02.8	9.004		-0.11	
3146	In house	13.79		1.86	
3150		6.888		-0.99	
3153	LFGB B82.02.8	5.72		-1.47	
3154	In house	18.25	G(0.05)	3.69	
3172		8.39		-0.37	
3176	LFGB B82.02.8	9.50		0.09	
3179		16.6		3.01	
3197	LFGB B82.02.8	3.08		-2.55	
3209	In house	9.641		0.15	

lab	method	value	mark	z(targ)	remarks
3210	In house	14.02		1.95	
3214	ISO17070	9.058		-0.09	
3220	In house	9.206		-0.03	
3222	UNI11057Mod.	4.81		-1.84	
3225	ISO17070	10.47		0.49	
3237	LFGB B82.02.8	0.349	DG(0.05)	-3.68	
3246	ISO17070	9.50		0.09	
	normality	suspect			
	n	78			
	outliers	3			
	mean (n)	9.282			
	st.dev. (n)	2.4343	RSD=26%		
	R(calc.)	6.816			
	st.dev.(iis-memo 1601)	2.4281			
	R(iis-memo 1601)	6.799			
Compare					
	R(Horwitz)	2.974			





APPENDIX 2: Other reported test results

Determination of Pentachlorophenol, 2,3,4-Trichlorophenol, 2,3,5-Trichlorophenol, 2,3,6-Trichlorophenol, 2,4,5-Trichlorophenol, 2,4,6-Trichlorophenol and 3,4,5-Trichlorophenol on sample #18650; in mg/kg

lab	PCP	234-TCP	235-TCP	236-TCP	245-TCP	246-TCP
213	-----	-----	-----	-----	-----	-----
551	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
623	0.13	n.d.	n.d.	n.d.	n.d.	n.d.
840	not detected	not detected	not detected	not detected	not detected	not detected
841	ND	ND	ND	ND	ND	ND
2108	-----	-----	-----	-----	-----	-----
2115	-----	-----	-----	-----	-----	-----
2120	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05
2129	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
2165	n.d.	-----	-----	-----	-----	-----
2172	-----	-----	-----	-----	-----	-----
2184	Not detected	-----	-----	-----	-----	-----
2213	0.09	<0.05	<0.05	<0.05	<0.05	<0.05
2232	-----	-----	-----	-----	-----	-----
2250	-----	-----	-----	-----	-----	-----
2255	nd	nd	nd	nd	nd	nd
2265	< 0,1	< 0,1	< 0,1	< 0,1	< 0,1	< 0,1
2290	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
2301	ND	ND	ND	ND	ND	ND
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
2313	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2330	-----	-----	-----	-----	-----	-----
2350	<0.125	<0.125	<0.125	<0.125	<0.125	<0.125
2352	-----	-----	-----	-----	-----	-----
2357	ND	ND	ND	ND	ND	ND
2358	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2363	ND	ND	ND	ND	ND	ND
2365	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2370	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2375	-----	-----	-----	-----	-----	-----
2379	14.808	Not detected	Not detected	Not detected	Not detected	Not detected
2380	-----	-----	-----	-----	-----	-----
2382	-----	-----	-----	-----	-----	-----
2386	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
2390	-----	-----	-----	-----	-----	-----
2453	nd [<0.5 mg/kg]	-----	-----	-----	-----	-----
2495	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
2511	-----	-----	-----	-----	-----	-----
2515	-----	-----	-----	-----	-----	-----
2549	ND	ND	ND	ND	ND	ND
2553	ND	ND	ND	ND	ND	ND
2560	0.08	-----	-----	-----	-----	-----
2566	ND	ND	ND	ND	ND	ND
2567	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2573	-----	-----	-----	-----	-----	-----
2582	ND	ND	ND	ND	ND	ND
2590	0.28	-----	-----	-----	-----	-----
2591	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2629	<0.05 mg/kg	<0.05 mg/kg	<0.05 mg/kg	<0.05 mg/kg	<0.05 mg/kg	<0.05 mg/kg
2638	n.d	n.d	n.d	n.d	n.d	n.d
2644	-----	-----	-----	-----	-----	-----
2649	-----	-----	-----	-----	-----	-----
2654	ND	ND	ND	ND	ND	ND
2668	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2730	0.010	-----	-----	-----	-----	-----
2741	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
2767	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2770	ND	ND	ND	ND	ND	ND
2804	-----	-----	-----	-----	-----	-----
2812	-----	-----	-----	-----	-----	-----
2827	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2830	ND	-----	-----	-----	-----	-----
2852	Non détecté	-----	-----	-----	-----	-----
2858	n.d	n.d	n.d	n.d	n.d	n.d
3116	-----	-----	-----	-----	-----	-----
3146	-----	-----	-----	-----	-----	-----
3150	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
3153	<0.5	-----	-----	-----	-----	-----
3154	-----	-----	-----	-----	-----	-----
3172	-----	-----	-----	-----	-----	-----
3176	nd	-----	-----	-----	-----	-----
3179	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1

lab	PCP	234-TCP	235-TCP	236-TCP	245-TCP	246-TCP
3197	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05
3209	-----	-----	-----	-----	-----	-----
3210	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
3214	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
3220	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
3222	-----	-----	-----	-----	-----	-----
3225	ND	ND	ND	ND	ND	ND
3237	0.057	-----	-----	-----	-----	-----
3246	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected

Determination of 3,4,5-Trichlorophenol, 2,3,4,5-Tetrachlorophenol, 2,3,4,6-Tetrachlorophenol, 2,3,5,6-Tetrachlorophenol and Other Chlorophenols on sample #18650; results in mg/kg =continued=

lab	345-TCP	2345-TCP	2346-TCP	2356-TCP	Other
213	-----	-----	-----	-----	-----
551	N.D.	N.D.	N.D.	N.D.	-----
623	n.d.	n.d.	n.d.	n.d.	n.d.
840	not detected	not detected	not detected	not detected	not detected
841	ND	ND	ND	ND	-----
2108	-----	-----	-----	-----	-----
2115	-----	-----	-----	-----	-----
2120	<0,05	<0,05	<0,05	<0,05	<0,05
2129	<0,1	<0,1	<0,1	<0,1	<0,1
2165	-----	-----	-----	-----	-----
2172	-----	-----	-----	-----	-----
2184	-----	-----	-----	-----	-----
2213	<0.05	<0.05	<0.05	<0.05	<0.05
2232	-----	-----	-----	-----	-----
2250	-----	-----	-----	-----	-----
2255	nd	nd	nd	nd	nd
2265	< 0,1	< 0,1	< 0,1	< 0,1	-----
2290	<0.5	<0.5	<0.5	<0.5	<0.5
2301	ND	ND	ND	ND	-----
2310	NOT DETECTED	NOT DETECTED	NOT DETECTED	NOT DETECTED	-----
2311	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2313	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2330	-----	-----	-----	-----	-----
2350	<0.125	<0.125	<0.125	<0.125	<0.125
2352	-----	-----	-----	-----	-----
2357	ND	ND	ND	ND	ND
2358	<0.05	<0.05	<0.05	<0.05	<0.05
2363	ND	ND	ND	ND	ND
2365	<0.05	<0.05	<0.05	<0.05	<0.05
2370	n.d.	n.d.	n.d.	n.d.	n.d.
2375	-----	-----	-----	-----	-----
2379	Not detected	Not detected	Not detected	Not detected	Not test
2380	-----	-----	-----	-----	-----
2382	-----	-----	-----	-----	-----
2386	<0,1	<0,1	<0,1	<0,1	<0,5
2390	-----	-----	-----	-----	-----
2453	-----	-----	-----	-----	-----
2495	<0.5	<0.5	<0.5	<0.5	-----
2511	-----	-----	-----	-----	-----
2515	-----	-----	-----	-----	-----
2549	ND	ND	ND	ND	ND
2553	ND	ND	ND	ND	ND
2560	-----	-----	-----	-----	-----
2566	ND	ND	ND	ND	-----
2567	<0.05	<0.05	<0.05	<0.05	<0.05
2573	-----	-----	-----	-----	-----
2582	ND	ND	ND	ND	ND
2590	-----	-----	-----	-----	-----
2591	<0.05	<0.05	<0.05	<0.05	-----
2629	<0.05 mg/kg	<0.05 mg/kg	<0.05 mg/kg	<0.05 mg/kg	-----
2638	n.d	n.d	n.d	n.d	n.d
2644	-----	-----	-----	-----	-----
2649	-----	-----	-----	-----	-----
2654	ND	ND	ND	ND	ND
2668	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2730	-----	-----	-----	-----	-----
2741	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
2767	<0.1	<0.1	<0.1	<0.1	-----
2770	ND	ND	ND	ND	ND

lab	345-TCP	2345-TCP	2346-TCP	2356-TCP	Other
2804	-----	-----	-----	-----	-----
2812	-----	-----	-----	-----	-----
2827	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2830	-----	ND	ND	ND	-----
2852	-----	Non détecté	Non détecté	Non détecté	-----
2858	n.d	n.d	n.d	n.d	-----
3116	-----	-----	-----	-----	-----
3146	-----	-----	-----	-----	-----
3150	<0,1	<0,1	<0,1	<0,1	<0,1
3153	-----	-----	-----	-----	-----
3154	-----	-----	-----	-----	-----
3172	-----	-----	-----	-----	-----
3176	-----	-----	-----	-----	-----
3179	<0,1	<0,1	<0,1	<0,1	-----
3197	<0,05	<0,05	<0,05	<0,05	<0,05
3209	-----	-----	-----	-----	-----
3210	<0.05	<0.05	<0.05	<0.05	-----
3214	<0.05	<0.05	<0.05	<0.05	<0.05
3220	Not Detected	Not Detected	Not Detected	Not Detected	-----
3222	-----	-----	-----	-----	-----
3225	ND	ND	ND	ND	ND
3237	-----	-----	-----	-----	-----
3246	Not detected	Not detected	Not detected	Not detected	Not detected

Determination of 2,3,4-Trichlorophenol, 2,3,5-Trichlorophenol, 2,3,6-Trichlorophenol, 2,4,5-Trichlorophenol, 2,4,6-Trichlorophenol and 3,4,5-Trichlorophenol on sample #18651; results in mg/kg

lab	234-TCP	235-TCP	236-TCP	245-TCP	246-TCP	345-TCP
213	-----	-----	-----	-----	-----	-----
551	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
623	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
840	not detected	not detected	not detected	not detected	not detected	not detected
841	ND	ND	ND	ND	ND	ND
2108	-----	-----	-----	-----	-----	-----
2115	-----	-----	-----	-----	-----	-----
2120	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05
2129	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
2165	-----	-----	-----	-----	-----	-----
2172	-----	-----	-----	-----	-----	-----
2184	-----	-----	-----	-----	-----	-----
2213	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2232	-----	-----	-----	-----	-----	-----
2250	-----	-----	-----	-----	-----	-----
2255	nd	nd	nd	nd	nd	nd
2265	< 0,1	< 0,1	< 0,1	< 0,1	< 0,1	< 0,1
2290	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
2301	ND	ND	ND	ND	ND	ND
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
2313	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2330	-----	-----	-----	-----	-----	-----
2350	<0.125	<0.125	<0.125	<0.125	<0.125	<0.125
2352	-----	-----	-----	-----	-----	-----
2357	ND	ND	ND	ND	ND	ND
2358	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2363	ND	ND	ND	ND	ND	ND
2365	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2370	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2375	-----	-----	-----	-----	-----	-----
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2380	-----	-----	-----	-----	-----	-----
2382	-----	-----	-----	-----	-----	-----
2386	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
2390	-----	-----	-----	-----	-----	-----
2453	-----	-----	-----	-----	-----	-----
2495	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
2511	-----	-----	-----	-----	-----	-----
2515	-----	-----	-----	-----	-----	-----
2549	ND	ND	ND	ND	ND	ND
2553	ND	ND	ND	ND	ND	ND
2560	-----	-----	-----	-----	-----	-----
2566	ND	ND	ND	ND	ND	ND
2567	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05

lab	234-TCP	235-TCP	236-TCP	245-TCP	246-TCP	345-TCP
2573	-----	-----	-----	-----	-----	-----
2582	ND	ND	ND	ND	ND	ND
2590	-----	-----	-----	-----	-----	-----
2591	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2629	<0.05 mg/kg	<0.05 mg/kg	<0.05 mg/kg	<0.05 mg/kg	<0.05 mg/kg	<0.05 mg/kg
2638	n.d	n.d	n.d	n.d	n.d	n.d
2644	-----	-----	-----	-----	-----	-----
2649	-----	-----	-----	-----	-----	-----
2654	ND	ND	ND	ND	ND	ND
2668	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2730	-----	-----	-----	-----	-----	-----
2741	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
2767	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2770	ND	ND	ND	ND	ND	ND
2804	-----	-----	-----	-----	-----	-----
2812	-----	-----	-----	-----	-----	-----
2827	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2830	-----	-----	-----	-----	-----	-----
2852	-----	-----	-----	-----	-----	-----
2858	n.d	n.d	n.d	n.d	n.d	n.d
3116	-----	-----	-----	-----	-----	-----
3146	-----	-----	-----	-----	-----	-----
3150	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
3153	-----	-----	-----	-----	-----	-----
3154	-----	-----	-----	-----	-----	-----
3172	-----	-----	-----	-----	-----	-----
3176	-----	-----	-----	-----	-----	-----
3179	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
3197	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05
3209	-----	-----	-----	-----	-----	-----
3210	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
3214	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
3220	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
3222	-----	-----	-----	-----	-----	-----
3225	ND	ND	ND	ND	ND	ND
3237	-----	-----	-----	-----	-----	-----
3246	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected

Determination of 2,3,4,5-Tetrachlorophenol, 2,3,4,6-Tetrachlorophenol, 2,3,5,6-Tetrachlorophenol and Other Chlorophenols on sample #18651; results in mg/kg = continued =

lab	2345-TCP	2346-TCP	2356-TCP	Other
213	-----	-----	-----	-----
551	N.D.	N.D.	N.D.	-----
623	n.d.	n.d.	n.d.	n.d.
840	not detected	not detected	not detected	not detected
841	ND	ND	ND	-----
2108	-----	-----	-----	-----
2115	-----	-----	-----	-----
2120	< 0,05	< 0,05	< 0,05	< 0,05
2129	<0,1	<0,1	<0,1	<0,1
2165	-----	-----	-----	-----
2172	-----	-----	-----	-----
2184	-----	-----	-----	-----
2213	<0.05	<0.05	<0.05	<0.05
2232	-----	-----	-----	-----
2250	-----	-----	-----	-----
2255	nd	nd	nd	-----
2265	< 0,1	< 0,1	< 0,1	-----
2290	<0.5	<0.5	<0.5	<0.5
2301	ND	ND	ND	-----
2310	NOT DETECTED	NOT DETECTED	NOT DETECTED	-----
2311	Not Detected	Not Detected	Not Detected	Not Detected
2313	Not Detected	Not Detected	Not Detected	Not Detected
2330	-----	-----	-----	-----
2350	<0.125	<0.125	<0.125	<0.125
2352	-----	-----	-----	-----
2357	ND	ND	ND	ND
2358	<0.05	<0.05	<0.05	<0.05
2363	ND	ND	ND	ND
2365	<0.05	<0.05	<0.05	<0.05
2370	n.d.	n.d.	n.d.	n.d.
2375	-----	-----	-----	-----
2379	Not detected	Not detected	Not detected	Not test
2380	-----	-----	-----	-----

lab	2345-TCP	2346-TCP	2356-TCP	Other
2382	-----	-----	-----	-----
2386	<0,1	<0,1	<0,1	<0,5
2390	-----	-----	-----	-----
2453	-----	-----	-----	-----
2495	<0.5	<0.5	<0.5	-----
2511	-----	-----	-----	-----
2515	-----	-----	-----	-----
2549	ND	ND	ND	ND
2553	ND	ND	ND	ND
2560	-----	-----	-----	-----
2566	ND	ND	ND	-----
2567	<0.05	<0.05	<0.05	<0.05
2573	-----	-----	-----	-----
2582	ND	ND	ND	ND
2590	-----	-----	-----	-----
2591	<0.05	<0.05	<0.05	-----
2629	<0.05 mg/kg	<0.05 mg/kg	<0.05 mg/kg	-----
2638	n.d	n.d	n.d	n.d
2644	-----	-----	-----	-----
2649	-----	-----	-----	-----
2654	ND	ND	ND	ND
2668	Not Detected	Not Detected	Not Detected	Not Detected
2730	-----	-----	-----	-----
2741	< 0.05	< 0.05	< 0.05	< 0.05
2767	<0.1	<0.1	<0.1	-----
2770	ND	ND	ND	ND
2804	-----	-----	-----	-----
2812	-----	-----	-----	-----
2827	Not Detected	Not Detected	Not Detected	Not Detected
2830	ND	ND	ND	-----
2852	Non détecté	Non détecté	Non détecté	-----
2858	n.d	n.d	n.d	-----
3116	-----	-----	-----	-----
3146	-----	-----	-----	-----
3150	<0,1	<0,1	<0,1	<0,1
3153	-----	-----	-----	-----
3154	-----	-----	-----	-----
3172	-----	-----	-----	-----
3176	-----	-----	-----	-----
3179	<0,1	<0,1	<0,1	-----
3197	<0,05	<0,05	<0,05	<0,05
3209	-----	-----	-----	-----
3210	<0.05	<0.05	<0.05	-----
3214	<0.05	<0.05	<0.05	<0.05
3220	Not Detected	Not Detected	Not Detected	-----
3222	-----	-----	-----	-----
3225	ND	ND	ND	ND
3237	-----	-----	-----	-----
3246	Not detected	Not detected	Not detected	Not detected

APPENDIX 3: Analytical details

lab	ISO/IEC17025 accredited	Sample intake (grams)	Extraction technique	Sample preparation
213	---	---	---	---
551	Yes	1.0	Ultrasonic extraction	---
623	Yes	1	Ultrasonic extraction	Further cut
840	Yes	0.5	Ultrasonic extraction	Further cut
841	Yes	about 1	Ultrasonic extraction	Further cut
2108	Yes	1	---	---
2115	Yes	1	Steam distillation	Used as received
2120	No	0,5	Steam distillation	Further cut
2129	Yes	1	Soxhlet / AES extraction	Used as received
2165	Yes	0.5	Ultrasonic extraction	Used as received
2172	Yes	0.205	Other	Further cut
2184	Yes	0.5	Ultrasonic extraction	Used as received
2213	Yes	1	Other	Further cut
2232	Yes	1	KOH extraction	Further cut
2250	Yes	0,5	Ultrasonic extraction	Further cut
2255	Yes	0.5	KOH extraction	Further cut
2265	Yes	0,5	KOH extraction	Further cut
2290	---	---	---	---
2301	Yes	1	Ultrasonic extraction	Further cut
2310	Yes	2	Steam distillation	Further cut
2311	Yes	1	Steam distillation	Further cut
2313	Yes	2.0	Steam distillation	Further cut
2330	---	---	---	---
2350	No	about 0.5 and 2	Ultrasonic extraction	Further cut
2352	Yes	1	Steam distillation	Further cut
2357	Yes	0.5	Steam distillation	Further cut
2358	Yes	1	Ultrasonic extraction	Further cut
2363	Yes	0.5	Ultrasonic extraction	Further cut
2365	Yes	1	Ultrasonic and Steam	Further cut
2370	Yes	1.02	Incubation	Further cut
2375	Yes	2	Ultrasonic extraction	Further cut
2379	No	0.5	KOH extraction	Further cut
2380	Yes	18650=1.0202; 18651=1.0010	Other	Further cut
2382	No	0.5	Steam distillation	Further cut
2386	Yes	0,5	Ultrasonic extraction	Further cut
2390	No	1	Ultrasonic extraction	Further cut
2453	No	1	Steam distillation	Further cut
2495	Yes	1	KOH extraction	Used as received
2511	---	---	---	---
2515	Yes	1.0012 #18650; 1.0015 #18651	KOH extraction	Further cut
2549	Yes	1.01	Steam distillation	Further cut
2553	---	---	---	---
2560	Yes	1.5	Other	Further cut
2566	Yes	1	Other	Further cut
2567	Yes	0.5	KOH extraction	Further cut
2573	Yes	0.5	Steam distillation	Used as received
2582	Yes	Around 1.0	KOH extraction	Further cut
2590	Yes	1	Steam distillation	Further cut
2591	No	1.0	Alkaline Digestion	Further cut
2629	Yes	1.0	Ultrasonic extraction	Further cut
2638	No	1	Ultrasonic extraction	Further cut
2644	Yes	1	Ultrasonic extraction	Used as received
2649	Yes	1	Ultrasonic extraction	Further cut
2654	Yes	2.5	Ultrasonic extraction	Further cut
2668	Yes	0.5	KOH extraction	Further cut
2730	No	2 18650; 1 18561	Ultrasonic extraction	Further cut
2741	No	0.5	Ultrasonic extraction	Further cut
2767	Yes	1	Ultrasonic extraction	Further cut
2770	Yes	1.00	Ultrasonic extraction	Further cut
2804	No	1	Ultrasonic extraction	Further cut

lab	ISO/IEC17025 accredited	Sample intake (grams)	Extraction technique	Sample preparation
2812	No	1	Steam distillation	Further cut
2827	Yes	1	KOH extraction	Further cut
2830	---	---	---	---
2852	Yes	3	Ultrasonic extraction	Used as received
2858	Yes	0.5022	KOH extraction	Further cut
3116	No	1	Incubation	Used as received
3146	Yes	1	KOH extraction	Used as received
3150	Yes	0,5	KOH extraction	Used as received
3153	Yes	0.5	Steam distillation	Further cut
3154	---	---	---	---
3172	---	---	---	---
3176	Yes	1	Ultrasonic extraction	Further cut
3179	Yes	0,6-0,7	KOH extraction	Further cut
3197	Yes	1	Steam distillation	Further cut
3209	Yes	---	Ultrasonic extraction	Used as received
3210	Yes	1	Ultrasonic extraction	Further cut
3214	Yes	1	Steam distillation	Further cut
3220	Yes	1	Ultrasonic extraction	Further cut
3222	Yes	1	Ultrasonic extraction	Further cut
3225	Yes	0.5	Liquid-Liquid extraction	Further cut
3237	No	3	Steam distillation	Used as received
3246	Yes	1.00	Steam distillation	Further cut

APPENDIX 4

Number of participants per country

5 labs in BANGLADESH
1 lab in BRAZIL
2 labs in CAMBODIA
2 labs in FRANCE
9 labs in GERMANY
6 labs in HONG KONG
9 labs in INDIA
2 labs in INDONESIA
6 labs in ITALY
1 lab in KOREA
4 labs in MOROCCO
10 labs in P.R. of CHINA
2 labs in PAKISTAN
2 labs in PORTUGAL
1 lab in SINGAPORE
1 lab in SPAIN
2 labs in SRI LANKA
2 labs in TAIWAN R.O.C.
1 lab in THAILAND
1 lab in TUNISIA
6 labs in TURKEY
7 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:

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