Institute for
Interlaboratory Studies

Results of Proficiency Test
Chlorinated Phenols in Textile
December 2023

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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CONTENTS

1 INTRODUCTION

Many countries have adopted environmental standards and requirements restricting the use of harmful chemicals in the production of textiles and clothing. Laws and regulations impose some of these standards and requirements. In addition to mandatory environmental standards and requirements for textiles, some Eco-labelling schemes are imposing environmental requirements for textile products on a voluntary basis, e.g. Milieukeur (Netherlands), Bluesign® (Switzerland) and OEKO-TEX® Standard 100 (Switzerland).

Since 2004 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of Chlorinated Phenols in Textile every year. During the annual proficiency testing program of 2023 it was decided to continue the proficiency test for the determination of Chlorinated Phenols in Textile.

In this interlaboratory study 75 laboratories in 23 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 a laboratory that has performed the tests in accordance with for ISO/IEC17043 relevant requirements of ISO/IEC17025.
It was decided to send one textile sample of 3 grams labelled #23800.
Participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

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

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

A batch of dark blue colored viscose was selected, which was made positive on some Chlorinated Phenols by a third party. The batch was cut into small pieces and after homogenization 105 small plastics bags were filled with approximately 3 grams each and labelled #23800.
The homogeneity of the subsamples was checked by determination of Pentachlorophenol (PCP) using an in house test method on 8 stratified randomly selected subsamples.

	Pentachlorophenol in mg/kg
sample #23800-1	4.3
sample #23800-2	4.5
sample #23800-3	4.7
sample #23800-4	4.9
sample #23800-5	5.0
sample #23800-6	4.6
sample #23800-7	4.9
sample #23800-8	4.8

Table 1: homogeneity test results of subsamples #23800

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility of the reference method in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Pentachlorophenol in mg/kg
r (observed)	0.7
reference method	iis memo 1601
0.3 x R (reference method)	1.1

Table 2: evaluation of the repeatability of subsamples #23800

The calculated repeatability is in agreement with 0.3 times the reproducibility of the reference method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one textile sample labelled #23800 was sent on November 15, 2023.

2.5 ANALYZES

The participants were requested to determine Pentachlorophenol (PCP) and all isomers of Tetra-, Tri-, Di- and Monochlorinated Phenols.
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 and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per determination in appendices 1 and 2 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the result tables in appendices 1 and 2. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5).
For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either ‘unknown’, ‘OK’, ‘suspect’ or ‘not OK’. After removal of outliers, this check was repeated. If a 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:

$z_{(target)}$ = (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. 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. Two 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 73 laboratories reported 120 numerical test results. Observed were 3 outlying test results, which is 2.5%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

The 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 test 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.

In test method DIN50009:21 Appendix B and in test method EN17134-2:23 annex B some precision data is given. Unfortunately, this informative precision data is based on a few components only and the concentration of these components are (far) below the concentration as found in this PT. Therefore, in this PT the test results will not be evaluated against test method DIN50009:21 or EN17134-2 but against the target reproducibility as given in iis memo 1601 (lit.13). In iis memo 1601 an estimated iis target reproducibility based on iis PTs of Pentachlorophenol in Textile from 2004 until 2014 is determined.

Pentachlorophenol (PCP): The group of participants met the target requirements. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the target reproducibility as derived from iis memo 1601.

2,3,4,6-Tetrachlorophenol: The group of participants met the target requirements. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the target reproducibility as derived from iis memo 1601.

Almost all participants agreed on a concentration near or below the limit of detection for all other components 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 derived from reference method are presented in the next table.

Component	unit	n	average	2.8 * sd	R(target)
Pentachlorophenol (PCP)	mg/kg	71	5.82	3.03	4.59
2,3,4,6-Tetrachlorophenol	mg/kg	46	0.097	0.044	0.141

Table 3: reproducibilities of components on sample #23800

Without further statistical calculations it can be concluded that there is a good compliance of the group of participants with the target reproducibilities.

4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2023 WITH PREVIOUS PTS

	December 2023	December 2022	December 2021	December 2020	December 2019
Number of reporting laboratories	73	74	73	69	81
Number of test results	120	145	73	131	120
Number of statistical outliers	3	6	3	1	3
Percentage of statistical outliers	2.5%	4.1%	4.1%	0.8%	2.5%

Table 4: 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.

Component	December 2023	December 2022	December 2021	December 2020	December 2019	2009 - 2018
Pentachlorophenol	19%	18%	21%	16%	25%	15 - 31%
2,3,4,5-Tetrachlorophenol	---	21%	---	16%	---	---
2,3,4,6-Tetrachlorophenol	16%	---	---	---	24%	---

Table 5: development of the uncertainties over the years

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

4.4 EVALUATION OF THE ANALYTICAL DETAILS

Several test methods are reported in this PT: for example test method LFGB B82.02.8 is reported by about 20% of the participants, test method DIN50009 by about 45% of the participants and test method EN17134-2 is used by about 10% of the participants.

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:
- 81% of the participants mentioned that they are ISO/IEC17025 accredited to determine the reported component(s).
- Prior to analysis the samples were further cut by 62% of the participants while 38% used the sample as received.
- The amount of sample intake varied between 0.15 and 3 grams: 35% used a sample intake of 0.5 grams, 50% used 1 gram and 10% used more than 1 grams.
- A large variety of different extraction techniques are used.
- About 95% of the participants used a KOH (mixture) and/or a Hexane (mixture) as extraction solvent.

The calculated reproducibilities are in agreement with the requirements of the target reproducibility, therefore no separate statistical analysis has been performed.

5 DISCUSSION

When the test results of this interlaboratory study were compared to the OEKO-TEX® Standard 100 (see next table) it was noticed that all participants would have rejected the PT sample for all Ecolabel classes for Pentachlorophenol only.
When only 2,3,4,6-Tetrachlorophenol is present all labs would accept this sample for classes 2, 3 and 4. For class 1 different decisions will be made.

Ecolabel	Class 1 Baby clothes in mg/kg	Class 2 Clothes direct skin contact in mg/kg	Class 3 Clothes, no direct contact in mg/kg	Class 4 Decoration material in mg/kg
Pentachlorophenol	0.05	0.5	0.5	0.5
Sum Tetrachlorophenols	0.05	0.5	0.5	0.5

Table 6: OEKO-TEXx® Standard 100

6 CONCLUSION

The majority of the participants has no problem with the determination of Pentachlorophenol or 2,3,4,6-Tetrachlorophenol in Textile in this PT.

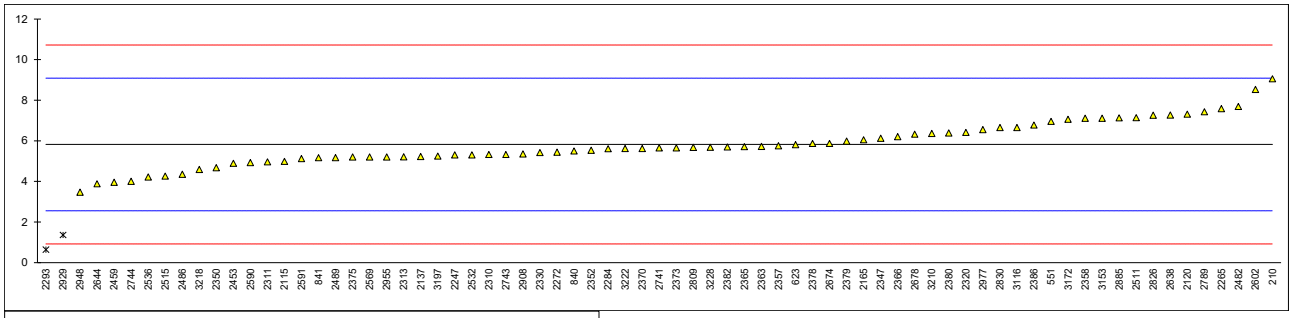
Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

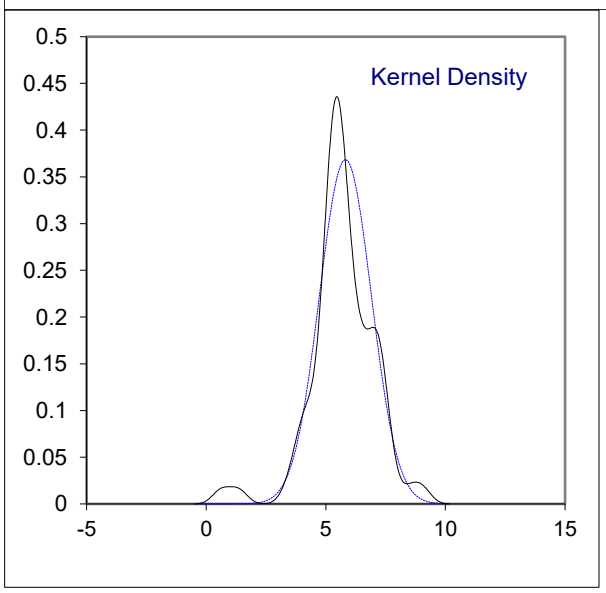
APPENDIX 1

Determination of Pentachlorophenol (PCP) on sample #23800; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	LFGB B82.02.8	9.05		1.98	
339		-----		-----	
551	DIN50009	6.95619		0.69	
623	DIN50009	5.808		-0.01	
840	LFGB B82.02.8	5.5		-0.20	
841	LFGB B82.02.8	5.160		-0.40	
2115	LFGB B82.02.8	4.98		-0.52	
2120	EN17134-2	7.31		0.91	
2137	KS K0733	5.22	C	-0.37	First reported 2.46
2165	DIN50009	6.05		0.14	
2247	DIN50009	5.30		-0.32	
2265	DIN50009	7.58		1.08	
2272	EN17134-2	5.44		-0.23	
2284	LFGB B82.02.8	5.609		-0.13	
2293	DIN50009	0.637	R(0.01)	-3.17	
2310	DIN50009	5.32		-0.31	
2311	DIN50009	4.9614		-0.53	
2313	LFGB B82.02.8	5.21		-0.37	
2320	DIN50009	6.410		0.36	
2330	DIN50009	5.419		-0.25	
2347	ISO17070	6.12		0.18	
2350	DIN50009	4.6701		-0.70	
2352	EN17134-2	5.53		-0.18	
2357	DIN50009	5.751		-0.04	
2358	LFGB B82.02.8	7.11		0.79	
2363	DIN50009	5.72		-0.06	
2365	DIN50009	5.710		-0.07	
2366	DIN50009	6.2		0.23	
2370	DIN50009	5.62		-0.12	
2373	LFGB B82.02.8	5.647		-0.11	
2375	DIN50009	5.2		-0.38	
2378	DIN50009	5.87		0.03	
2379	DIN50009	5.9806		0.10	
2380	DIN50009	6.385		0.35	
2382	EN17134-2	5.69		-0.08	
2386	DIN50009	6.775		0.58	
2453	DIN50009	4.89		-0.57	
2459	LFGB B82.02.8	3.95		-1.15	
2482	EN17134-2	7.691		1.14	
2486	LFGB B82.02.8	4.349		-0.90	
2489	DIN50009	5.17		-0.40	
2511	EN17134-2	7.14		0.81	
2515	DIN50009	4.26		-0.96	
2532	LFGB B82.02.8	5.3		-0.32	
2536	In house	4.214		-0.98	
2561		-----		-----	
2569	LFGB B82.02.8	5.2		-0.38	
2590	DIN50009	4.927		-0.55	
2591	ISO17070	5.117		-0.43	
2602		8.530		1.66	
2638	In house	7.267		0.89	
2644	DIN50009	3.88		-1.19	
2674	In house	5.87		0.03	
2678	§64 LFGB B82.02.8	6.324		0.31	
2741	In house	5.646		-0.11	
2743	DIN50009	5.32		-0.31	
2744	In house	4.00		-1.12	
2789		7.43		0.99	
2809	DIN50009	5.67		-0.09	
2826	DIN50009/EN17134-2	7.262		0.88	
2830	XP G08-015	6.65		0.51	
2885	KS K0733	7.13		0.80	
2908	XP G08-015	5.35	C	-0.29	First reported 2.14
2929	In house	1.3519	R(0.01)	-2.74	
2948	LFGB B82.02.8	3.469		-1.44	
2955	DIN50009	5.2		-0.38	
2977	In house	6.5468		0.44	
3116	DIN50009	6.65		0.51	
3153	LFGB B82.02.8	7.11		0.79	
3172	EN17134-2	7.0573		0.76	
3197	EN17134-2	5.24		-0.36	
3210	In house	6.36		0.33	
3218	DIN50009	4.581		-0.76	
3222	UNI11057	5.617		-0.13	
3228	DIN50009	5.68		-0.09	

normality	OK	
n	71	
outliers	2	
mean (n)	5.8213	
st.dev. (n)	1.08336	RSD = 19%
R(calc.)	3.0334	
st.dev.(iis memo 1601)	1.63314	
R(iis memo 1601)	4.5728	

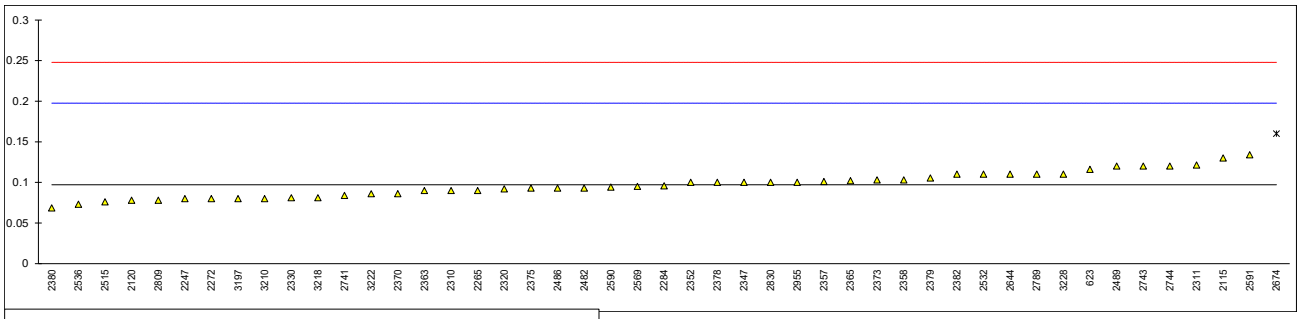


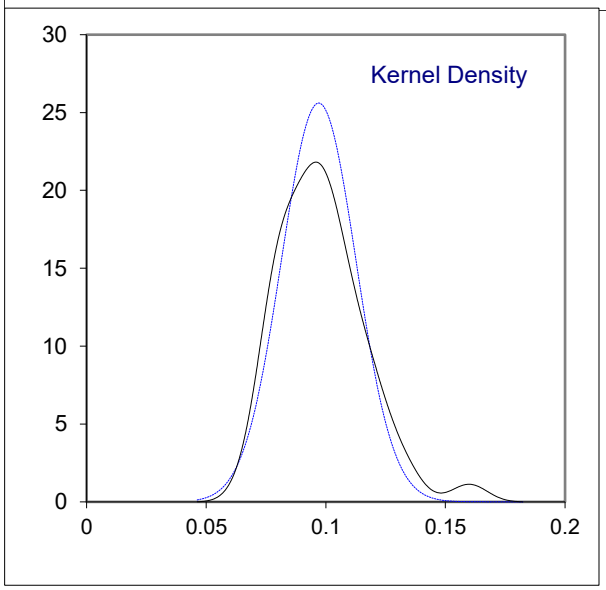


Determination of 2,3,4,6-Tetrachlorophenol on sample #23800; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	LFGB B82.02.8	not detected		-----	
339		-----		-----	
551	DIN50009	not detected		-----	
623	DIN50009	0.116		0.38	
840	LFGB B82.02.8	not detected		-----	
841	LFGB B82.02.8	<0.125		-----	
2115	LFGB B82.02.8	0.13		0.66	
2120	EN17134-2	0.078		-0.38	
2137		-----		-----	
2165	DIN50009	Not Detected		-----	
2247	DIN50009	0.08		-0.34	
2265	DIN50009	0.09		-0.14	
2272	EN17134-2	0.08		-0.34	
2284	LFGB B82.02.8	0.0958		-0.02	
2293		-----		-----	
2310	DIN50009	0.09		-0.14	
2311	DIN50009	0.1212		0.48	
2313	LFGB B82.02.8	Not Detected		-----	
2320	DIN50009	0.092		-0.10	
2330	DIN50009	0.081		-0.32	
2347	ISO17070	0.10		0.06	
2350	DIN50009	<0.1		-----	
2352	EN17134-2	0.10		0.06	
2357	DIN50009	0.101		0.08	
2358	LFGB B82.02.8	0.103		0.12	
2363	DIN50009	0.09		-0.14	
2365	DIN50009	0.102		0.10	
2366		-----		-----	
2370	DIN50009	0.0861		-0.22	
2373	LFGB B82.02.8	0.103		0.12	
2375	DIN50009	0.093		-0.08	
2378	DIN50009	0.1		0.06	
2379	DIN50009	0.1054		0.17	
2380	DIN50009	0.06859		-0.57	
2382	EN17134-2	0.11		0.26	
2386	DIN50009	< 0.100		-----	
2453		-----		-----	
2459	LFGB B82.02.8	ND		-----	
2482	EN17134-2	0.09305		-0.08	
2486	LFGB B82.02.8	0.093		-0.08	
2489	DIN50009	0.12		0.46	
2511		-----		-----	
2515	DIN50009	0.076		-0.42	
2532	LFGB B82.02.8	0.11		0.26	
2536	In house	0.073		-0.48	
2561		-----		-----	
2569	LFGB B82.02.8	0.095		-0.04	
2590	DIN50009	0.094		-0.06	
2591	ISO17070	0.134		0.74	
2602		-----		-----	
2638	In house	not detected		-----	
2644	DIN50009	0.11		0.26	
2674	In house	0.16	R(0.05)	1.25	
2678	§64 LFGB B82.02.8	not detected		-----	
2741	In house	0.084		-0.26	
2743	DIN50009	0.12		0.46	
2744	In house	0.12		0.46	
2789		0.11		0.26	
2809	DIN50009	0.078		-0.38	
2826	DIN50009/EN17134-2	Not detected		-----	
2830	XP G08-015	0.10		0.06	
2885		-----		-----	
2908		-----		-----	
2929	In house	below det. limit		-----	
2948	LFGB B82.02.8	not detected		-----	
2955	DIN50009	0.10		0.06	
2977	In house	not detected		-----	
3116		-----		-----	
3153		-----		-----	
3172	EN17134-2	< 0.01		-----	
3197	EN17134-2	0.08		-0.34	
3210	In house	0.08		-0.34	
3218	DIN50009	0.081		-0.32	
3222	UNI11057	0.086		-0.22	
3228	DIN50009	0.11		0.26	

normality	OK	
n	46	
outliers	1	
mean (n)	0.0970	
st.dev. (n)	0.01558	RSD = 16%
R(calc.)	0.0436	
st.dev.(iis memo 1601)	0.05030	
R(iis memo 1601)	0.1409	





APPENDIX 2 Other reported test results

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

Determination individual and other Chlorinated Phenols on sample #23800; in mg/kg

lab	2345-TeCP	2356-TeCP	234-TCP	235-TCP	236-TCP	245-TCP	246-TCP	345-TCP
210	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
339	-----	-----	-----	-----	-----	-----	-----	-----
551	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
623	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
840	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
841	<0.125	<0.125	<0.125	<0.125	<0.125	<0.125	<0.125	<0.125
2115	-----	-----	-----	-----	-----	-----	-----	-----
2120	< 0,04	< 0,04	< 0,04	< 0,04	< 0,04	< 0,04	< 0,04	< 0,04
2137	-----	-----	-----	-----	-----	-----	-----	-----
2165	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2247	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2265	-----	-----	-----	-----	-----	-----	-----	-----
2272	-----	-----	-----	-----	-----	-----	-----	-----
2284	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
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
2313	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	0.095	Not Detected
2320	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2330	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2347	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2350	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2352	-----	-----	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----	-----	-----
2358	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2363	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2365	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2366	-----	-----	-----	-----	-----	-----	-----	-----
2370	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2373	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05
2375	-----	-----	-----	-----	-----	-----	-----	-----
2378	-----	-----	-----	-----	-----	-----	-----	-----
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2380	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2382	＜0.10	＜0.10	＜0.10	＜0.10	＜0.10	＜0.10	＜0.10	＜0.10
2386	< 0.100	< 0.100	< 0.100	< 0.100	< 0.100	< 0.100	< 0.100	< 0.100
2453	-----	-----	-----	-----	-----	-----	-----	-----
2459	ND	ND	ND	ND	ND	ND	ND	ND
2482	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01
2486	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2489	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2511	-----	-----	-----	-----	-----	-----	-----	-----
2515	-----	-----	-----	-----	-----	-----	-----	-----
2532	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2536	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2561	-----	-----	-----	-----	-----	-----	-----	-----
2569	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2590	-----	-----	-----	-----	-----	-----	-----	-----
2591	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2602	-----	-----	-----	-----	-----	-----	-----	-----
2638	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2644	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2674	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2678	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2741	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2743	-----	not detectable	-----	-----	-----	-----	-----	-----
2744	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2789	-----	-----	-----	-----	-----	-----	-----	-----

lab	2345-TeCP	2356-TeCP	234-TCP	235-TCP	236-TCP	245-TCP	246-TCP	345-TCP
2809	-----	-----	-----	-----	-----	-----	-----	-----
2826	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2830	-----	0.15	-----	-----	-----	-----	-----	-----
2885	-----	Not detected	-----	-----	-----	-----	-----	-----
2908	-----	-----	-----	-----	-----	-----	-----	-----
2929	below detection limit	below detection limit	below detection limit	below detection limit	below detection limit	below detection limit	below detection limit	below detection limit
2948	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2955	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2977	0.1027	not detected	not detected	not detected	not detected	not detected	not detected	not detected
3116	-----	-----	-----	-----	-----	-----	-----	-----
3153	-----	-----	-----	-----	-----	-----	-----	-----
3172	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
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	<0.05
3218	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050
3222	-----	-----	-----	-----	-----	-----	-----	-----
3228	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected

Other reported test results continued

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

Determination individual and other Chlorinated Phenols on sample #23800; in mg/kg

lab	23-DCP	24-DCP	25-DCP	26-DCP	34-DCP	35-DCP	2-CP	3-CP	4-CP
210	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
339	-----	-----	-----	-----	-----	-----	-----	-----	-----
551	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
623	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
840	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
841	<0.125	<0.125	<0.125	<0.125	<0.125	<0.125	<0.125	<0.125	<0.125
2115	-----	-----	-----	-----	-----	-----	-----	-----	-----
2120	< 0,04	< 0,04	< 0,04	< 0,04	< 0,04	< 0,04	< 0,04	< 0,04	< 0,04
2137	-----	-----	-----	-----	-----	-----	-----	-----	-----
2165	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2247	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2265	-----	-----	-----	-----	-----	-----	-----	-----	-----
2272	-----	-----	-----	-----	-----	-----	-----	-----	-----
2284	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2293	-----	-----	-----	-----	-----	-----	-----	-----	-----
2310	not detected	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	Not Detected
2313	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2320	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2330	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2347	Out of cap	Out of cap	Out of cap	Out of cap	Out of cap	Out of cap	Out of cap	Out of cap	Out of cap
2350	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2352	-----	-----	-----	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----	-----	-----	-----
2358	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2363	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2365	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2366	-----	-----	-----	-----	-----	-----	-----	-----	-----
2370	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2373	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05
2375	-----	-----	-----	-----	-----	-----	-----	-----	-----
2378	-----	-----	-----	-----	-----	-----	-----	-----	-----
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2380	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2382	＜0.10	＜0.10	＜0.10	＜0.10	＜0.10	＜0.10	＜0.10	＜0.10	＜0.10
2386	< 0.500	< 0.500	< 0.500	< 0.500	< 0.500	< 0.500	< 0.500	< 0.500	< 0.500
2453	-----	-----	-----	-----	-----	-----	-----	-----	-----
2459	ND	ND	ND	ND	ND	ND	ND	ND	ND
2482	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05
2486	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2489	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2511	-----	-----	-----	-----	-----	-----	-----	-----	-----
2515	-----	-----	-----	-----	-----	-----	-----	-----	-----
2532	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2536	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2561	-----	-----	-----	-----	-----	-----	-----	-----	-----
2569	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2590	-----	-----	-----	-----	-----	-----	-----	-----	-----
2591	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2602	-----	-----	-----	-----	-----	-----	-----	-----	-----
2638	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2644	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2674	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2678	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2741	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2743	-----	-----	-----	-----	-----	-----	-----	-----	-----
2744	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected

lab	23-DCP	24-DCP	25-DCP	26-DCP	34-DCP	35-DCP	2-CP	3-CP	4-CP
2789	-----	-----	-----	-----	-----	-----	-----	-----	-----
2809	-----	-----	-----	-----	-----	-----	-----	-----	-----
2826	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2830	-----	-----	-----	-----	-----	-----	-----	-----	-----
2885	-----	-----	-----	-----	-----	-----	-----	-----	-----
2908	-----	-----	-----	-----	-----	-----	-----	-----	-----
2929	below detection limit	below detection limit	below detection limit	below detection limit	below detection limit	below detection limit	below detection limit	below detection limit	below detection limit
2948	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2955	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2977	-----	-----	-----	-----	-----	-----	-----	-----	-----
3116	-----	-----	-----	-----	-----	-----	-----	-----	-----
3153	-----	-----	-----	-----	-----	-----	-----	-----	-----
3172	< 0.01	< 0.01	-----	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
3197	<0,05	<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	<0.05	<0.05
3218	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050
3222	-----	-----	-----	-----	-----	-----	-----	-----	-----
3228	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected

APPENDIX 3 Analytical details

lab	17025 accr.	Sample preparation	Sample intake (grams)	Extraction technique	Extraction solution
210	Yes	Further cut		---	
339	---	---		---	
551	---	---		---	
623	Yes	Further cut	1	Ultrasonic	hexane
840	Yes	Used as received	1	Ultrasonic	KOH
841	Yes	Used as received	1g	Other	KOH/n-Hexane
2115	Yes	Used as received	0.15 g	Other	Hexane + Triethylamine
2120	No	Used as received	1g	Oven	KOH
2137	Yes	Used as received	1	Ultrasonic	2M KOH
2165	Yes	Further cut	1.0g	Mechanical Shaking	n-hexane
2247	Yes	Further cut	1.5gm	Water bath	n-hexane
2265	Yes	Further cut	0,5	Other	KOH
2272	No	Used as received	1gram	Thermal Desorption	hexane
2284	Yes	Used as received	0.5g	Other	n-hexane
2293	---	---		---	
2310	Yes	Further cut	2	Mechanical Shaking	Hexane
2311	Yes	Further cut	0.5	Thermal Desorption	KOH/ Hexane
2313	Yes	Further cut	1.0 g	Steam distillation	n-Hexane
2320	Yes	Further cut	1g	Mechanical Shaking	Hexane
2330	No	Further cut	1 g	Ultrasonic	Hexane/ KOH
2347	No	Further cut	1.0g	Ultrasonic	
2350	No	Further cut	2.5254 g	Thermal Desorption	KOH (Potassium hydroxide solvent)
2352	Yes	Further cut	1g	Mechanical Shaking	hexane
2357	---	---		---	
2358	Yes	Used as received	1.0	Other	KOH
2363	Yes	Further cut	1g	Oven	n-Hexane
2365	Yes	Further cut	2.5g	Other	1mol/L KOH
2366	No	Further cut	0.5g	Ultrasonic	hexane
2370	Yes	Further cut	1g	Mechanical Shaking	Hexane
2373	Yes	Further cut	0.5g	Ultrasonic	N-Hexane
2375	---	---		---	
2378	Yes	Further cut	0.5g	Mechanical Shaking	hexane
2379	Yes	Further cut	1 g	Other	KOH
2380	Yes	Further cut	1.0 g	Alkaline digestion	KOH
2382	Yes	Further cut	0.5g	Thermal Desorption	1mol/L KOH Solution
2386	Yes	Further cut	0.5 g	Ultrasonic	1 M KOH
2453	Yes	Further cut	+/-0.5g	Thermal Desorption	KOH
2459	Yes	Further cut	1.00 gram	Soxhlet	2M KOH
2482	No	Used as received	0,5	Other	Extraction with 1 M KOH, 90 °C, 16 h
2486	Yes	Used as received	1.0005 gram	Mechanical Shaking	n-Hexane
2489	---	---		---	
2511	Yes	---		Other	According To EN 17134-2:2023
2515	Yes	Used as received	1 gram	Oven	KOH 1M
2532	Yes	Further cut	0.5 grams	Oven	n-Hexane
2536	Yes	Further cut	1.0021	Mechanical Shaking	n-Hexane
2561	---	---		---	
2569	Yes	Further cut	1 gm	Steam distillation	n-Hexane
2590	Yes	Used as received	1g	Mechanical Shaking	hexane
2591	Yes	Further cut	1.0	Basic digestion	
2602	Yes	Used as received	0,5 g	Other	Extraction with KOH, derivatisation with Acetic anhydride, Derivate is extracted with Isooctane
2638	No	Further cut	1 gm	Ultrasonic	Hexane
2644	Yes	Used as received	0.5	Mechanical Shaking	
2674	Yes	Used as received	3.0g	Mechanical Shaking	Hexane
2678	No	Further cut	0.5 grams	Ultrasonic	KOH followed by n-Hexane
2741	Yes	Further cut	0.5 gram	Ultrasonic	Potassium hydroxide
2743	No	Used as received	2	Mechanical Shaking	hexane
2744	Yes	Used as received	1	Mechanical Shaking	Hexane
2789	Yes	Used as received	1	Thermal Desorption	KOH + hexane
2809	Yes	Further cut	0.5	Thermal Desorption	KOH 1M 90oC

lab	17025 accr.	Sample preparation	Sample intake (grams)	Extraction technique	Extraction solution
2826	Yes	Used as received	0.5g	Thermal Desorption	Potassium Hydroxide Solution
2830	Yes	Further cut	1 gramme	Mechanical Shaking	Carbonate de potassium
2885	No	Further cut	0.5 g	Mechanical Shaking	2M KOH
2908	Yes	Used as received	2G	Ultrasonic	hexane
2929	No	Further cut	0,5	Mechanical Shaking	ethyl acetate
2948	Yes	Used as received	1	Mechanical Shaking	KOH followed by N-hexane
2955	Yes	Further cut	1 gm	Mechanical Shaking	Potassium Hydroxide (KOH)
2977	No	Used as received	1g	Thermal Desorption	KOH/hexane
3116	Yes	Used as received	1	Oven	1M KOH
3153	Yes	Further cut	0.5 gram	Steam distillation	N-hexane
3172	Yes	---		---	
3197	Yes	Further cut	0,5 g	Ultrasonic	KOH solution
3210	Yes	Used as received	1g	Ultrasonic	
3218	Yes	Further cut	0.5g	Other	KOH, n-Hexane
3222	Yes	Used as received	1 gram	Ultrasonic	Extraction from the sample with a solution of potassium carbonate, derivatization with acetic anhydride and then extraction with hexane.
3228	Yes	Further cut	0.5 g	Oven	1 mol/L KOH

APPENDIX 4

Number of participants per country

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

APPENDIX 5

Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
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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