Institute for
Interlaboratory Studies

Results of Proficiency Test
Chlorinated Phenols in Textile
December 2022

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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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 2022/2023 it was decided to continue the proficiency test for the determination of Chlorinated Phenols in Textile.

In this interlaboratory study 76 laboratories in 25 countries registered for participation, see appendix 4 for the number of participants per country. In this report the results of the Chlorinated Phenols in Textile proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.
It was decided to send one textile sample of 3 grams labelled #22805.
The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

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

2.2 PROTOCOL

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

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

A batch of blue/white colored jeans was selected, which was made positive on some Chlorinated Phenols by a third party. The batch was cut into small pieces and after homogenization 100 small plastics bags were filled with approximately 3 grams each and labelled #22805.
The homogeneity of the subsamples was checked by determination of Pentachlorophenol (PCP) and 2,3,4,5-Tetrachlorophenol using an in-house test method on 8 stratified randomly selected subsamples.

	Pentachlorophenol in mg/kg	2,3,4,5-Tetrachlorophenol in mg/kg
sample #22805-1	23.8	14.1
sample #22805-2	24.2	14.6
sample #22805-3	23.1	14.9
sample #22805-4	21.9	13.9
sample #22805-5	23.4	15.2
sample #22805-6	21.6	14.0
sample #22805-7	23.5	15.3
sample #22805-8	24.1	15.7

Table 1: homogeneity test results of subsamples #22805

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

	Pentachlorophenol in mg/kg	2,3,4,5-Tetrachlorophenol in mg/kg
r (observed)	3.3	1.9
reference method	iis memo 1601	iis memo 1601
0.3 x R (reference method)	4.4	3.0

Table 2: evaluation of the repeatabilities of subsamples #22805

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

To each of the participating laboratories one textile sample labelled #22805 was sent on November 9, 2022.

2.5 ANALYZES

The participants were requested to determine the Pentachlorophenol (PCP), 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. Seven 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 74 laboratories reported 145 numerical test results. Observed were 6 outlying test results, which is 4.1%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

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

4.1 EVALUATION PER COMPONENT

In this section the reported test results are discussed per component. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables 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 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 but against the target reproducibility as given in iis memo 1601. 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): This determination was not problematic. Four 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.

2,3,4,5-Tetrachlorophenol: This determination was not problematic. 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.

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 methods are presented in the next table.

Component	unit	n	average	2.8 * sd	R(target)
Pentachlorophenol (PCP)	mg/kg	70	17.75	9.09	11.80
2,3,4,5-Tetrachlorophenol	mg/kg	69	13.01	7.82	9.06

Table 3: reproducibilities of components on sample #22805

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 2022 WITH PREVIOUS PTS

	December 2022	December 2021	December 2020	December 2019	December 2018
Number of reporting laboratories	74	73	69	81	81
Number of test results	145	73	131	120	208
Number of statistical outliers	6	3	1	3	8
Percentage of statistical outliers	4.1%	4.1%	0.8%	2.5%	3.8%

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 2022	December 2021	December 2020	December 2019	2009 - 2018	Target *)
Pentachlorophenol	18%	21%	16%	25%	15 - 31%	29 - 20%
2,3,4,5-Tetrachlorophenol	21%	---	16%	---	---	29 - 20%
2,3,4,6-Tetrachlorophenol	---	---	---	24%	---	29 - 20%

Table 5: development of the uncertainties over the years

*) Concentration range 5 - 50 mg/kg respectively

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

Sample #22805 was used in a previous PT as sample #20750 in iis20A18. The averages found in both PTs for this sample are similar.

		sample #22805			sample #20750		
Component	unit	n	average	R(calc)	n	average	R(calc)
Pentachlorophenol	mg/kg	70	17.75	9.09	67	18.04	8.17
2,3,4,5-Tetrachlorophenol	mg/kg	69	13.01	7.82	63	13.21	6.08

Table 6: comparison of sample #22805 with #20750

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 30% of the participants and test method DIN50009 by about 20% 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:

- About 75% 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 about 70% of the participants while about 30% used the sample as received.
- The amount of sample intake varied between 0.25 and 5 grams: about 35% used a sample intake of 0.5 grams, about 50% used 1 gram and about 15% used more than 1 grams.
- Ultrasonic extraction and mechanical shaking were most often reported as extraction techniques, respectively 40% and 55%.
- About 80% 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.

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 7: Oeko-Tex® Standard 100

6 CONCLUSION

The majority of the participants has no problem with the determination of Pentachlorophenol or 2,3,4,5-Tetrachlorophenol in Textile.

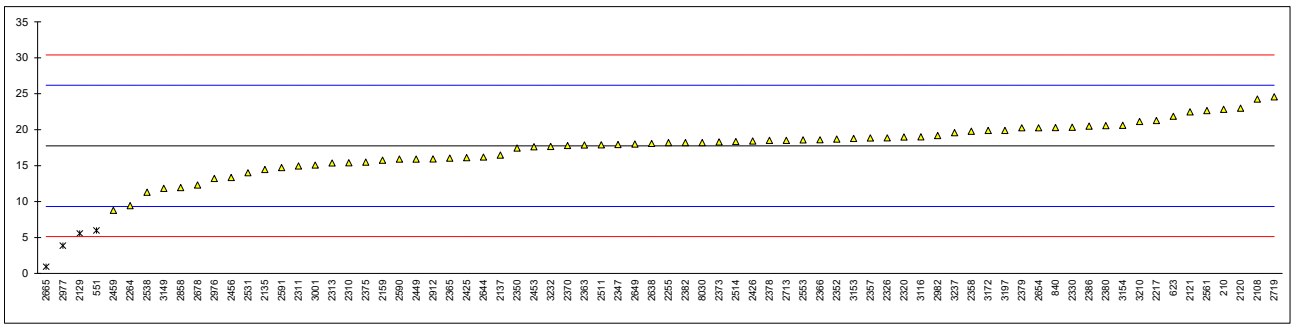
Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

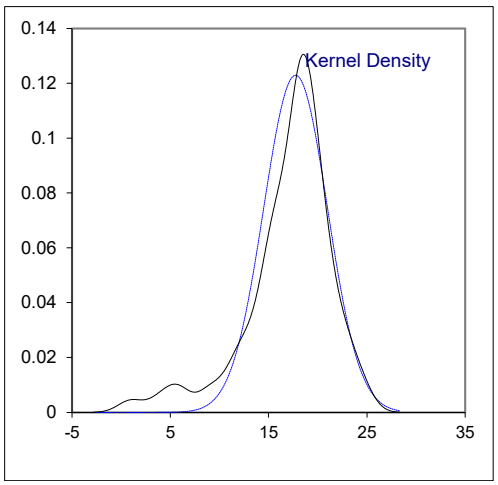
APPENDIX 1

Determination of Pentachlorophenol (PCP) on sample #22805; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	In house	22.84		1.21	
551	§64 LFGB B82.02.8	5.9880	R(0.05)	-2.79	
623	LFGB B82.02.8	21.878		0.98	
840	LFGB B82.02.8	20.31		0.61	
2108	DIN50009	24.26		1.54	
2120	EN17134-2	23		1.25	
2121	In house	22.48		1.12	
2129		5.57	R(0.05)	-2.89	
2135	In house	14.464		-0.78	
2137	KS K0733	16.48		-0.30	
2159	In house	15.75		-0.48	
2217	LFGB B82.02.8	21.27		0.83	
2255	DIN50009	18.20		0.11	
2264	LFGB B82.02.8	9.45		-1.97	
2265		-----		-----	
2310	DIN50009	15.4		-0.56	
2311	DIN50009	14.9557		-0.66	
2313	LFGB B82.02.8	15.36		-0.57	
2320	DIN50009	18.985		0.29	
2326	DIN50009	18.86		0.26	
2330	LFGB B82.02.8	20.355		0.62	
2347	LFGB B82.02.8	17.93		0.04	
2350	In house	17.45		-0.07	
2352	DIN50009	18.70		0.22	
2357	LFGB B82.02.8	18.840		0.26	
2358	LFGB B82.02.8	19.794		0.48	
2363	In house	17.87		0.03	
2365	In house	16.051		-0.40	
2366	LFGB B82.02.8	18.6		0.20	
2370	LFGB B82.02.8	17.8		0.01	
2373	LFGB B82.02.8	18.282		0.13	
2375	In house	15.5		-0.53	
2378	DIN50009	18.51		0.18	
2379	LFGB B82.02.8Mod.	20.2606		0.59	
2380	LFGB B82.02.8Mod.	20.573		0.67	
2382	DIN50009	18.20		0.11	
2386	In house	20.5		0.65	
2425	In house	16.11		-0.39	
2426	ISO17070	18.425		0.16	
2449	LFGB B82.02.8	15.92		-0.44	
2453		17.627		-0.03	
2456	UNI11057	13.35		-1.05	
2459	LFGB B82.02-8	8.79		-2.13	
2511	ISO17070	17.9		0.03	
2514	In house	18.35		0.14	
2531	UNI11057	14		-0.89	
2538	DIN50009	11.3205		-1.53	
2553	CPSD AN 00094	18.57		0.19	
2561		22.646		1.16	
2590	LFGB B82.02.8	15.915		-0.44	
2591	ISO17070Mod.	14.7236		-0.72	
2638	In house	18.08		0.08	
2644	UNI11057	16.20		-0.37	
2649	In house	18		0.06	
2654	XPG08-015	20.27		0.60	
2665	In house	0.935	R(0.05)	-3.99	
2678	§64 LFGB B82.02.8	12.31		-1.29	
2713	DIN50009	18.514		0.18	
2719	In house	24.56		1.62	
2858	In house	11.959		-1.38	
2912	UNI11057	15.941		-0.43	
2976	DIN50009	13.233		-1.07	
2977	In house	3.860	C,R(0.05)	-3.30	first reported 3.3391
2982	LFGB B82.02.8	19.19		0.34	
3001	In house	15.07		-0.64	
3116	DIN50009	19.0		0.30	
3149	In house	11.85		-1.40	
3153	LFGB B82.02.8	18.79		0.25	
3154	DIN50009	20.62		0.68	
3172	KS K0733	19.893		0.51	
3197	LFGB B82.02.8	19.9		0.51	
3210		21.14		0.80	
3230		-----		-----	
3232	ISO17070	17.664		-0.02	

lab	method	value	mark	z(targ)	remarks
3237		19.58		0.43	
8030	DIN50009	18.2014		0.11	
	normality	OK			
	n	70			
	outliers	4			
	mean (n)	17.754			
	st.dev. (n)	3.247	RSD = 18%		
	R(calc.)	9.090			
	st.dev.(iis memo 1601)	4.214			
	R(iis memo 1601)	11.798			

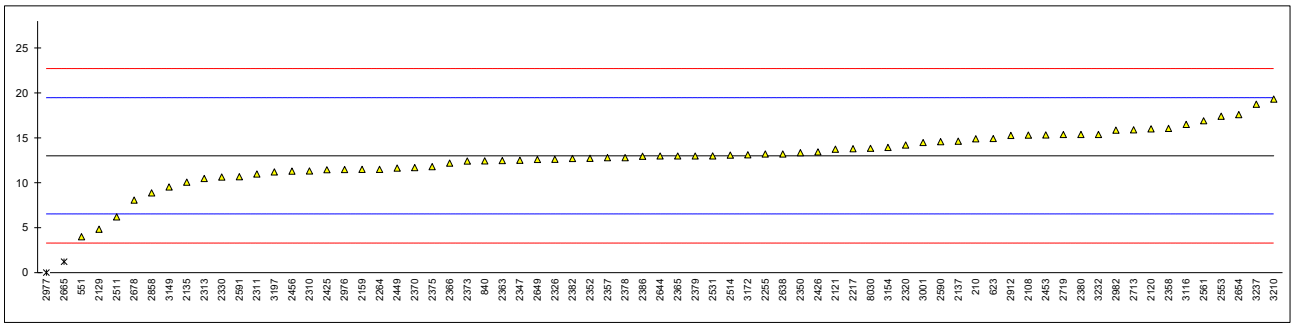


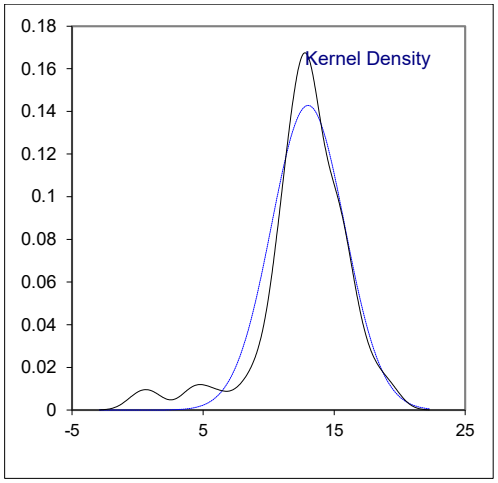


Determination of 2,3,4,5-Tetrachlorophenol on sample #22805; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	In house	14.89		0.58	
551	§64 LFGB B82.02.8	3.9920		-2.79	
623	LFGB B82.02.8	14.931		0.60	
840	LFGB B82.02.8	12.45		-0.17	
2108	DIN50009	15.31		0.71	
2120	EN17134-2	16		0.93	
2121	In house	13.74		0.23	
2129		4.84		-2.52	
2135	In house	10.071		-0.91	
2137	KS K0733	14.63		0.50	
2159	In house	11.51		-0.46	
2217	LFGB B82.02.8	13.8		0.25	
2255	DIN50009	13.21		0.06	
2264	LFGB B82.02.8	11.51		-0.46	
2265		-----		-----	
2310	DIN50009	11.33		-0.52	
2311	DIN50009	10.9774		-0.63	
2313	LFGB B82.02.8	10.48		-0.78	
2320	DIN50009	14.209		0.37	
2326	DIN50009	12.63		-0.12	
2330	LFGB B82.02.8	10.656		-0.73	
2347	LFGB B82.02.8	12.51		-0.15	
2350	In house	13.34		0.10	
2352	DIN50009	12.73		-0.09	
2357	LFGB B82.02.8	12.810		-0.06	
2358	LFGB B82.02.8	16.058		0.94	
2363	In house	12.48		-0.16	
2365	In house	12.983		-0.01	
2366	LFGB B82.02.8	12.2		-0.25	
2370	LFGB B82.02.8	11.7		-0.40	
2373	LFGB B82.02.8	12.426		-0.18	
2375	In house	11.8		-0.37	
2378	DIN50009	12.81		-0.06	
2379	LFGB B82.02.8Mod.	12.9903		0.00	
2380	LFGB B82.02.8Mod.	15.382		0.73	
2382	DIN50009	12.70		-0.09	
2386	In house	12.95		-0.02	
2425	In house	11.45		-0.48	
2426	ISO17070	13.450		0.14	
2449	LFGB B82.02.8	11.63		-0.43	
2453		15.322		0.72	
2456	UNI11057	11.30		-0.53	
2459	LFGB B82.02-8	not detected		-----	possibly a false negative test result?
2511	ISO17070	6.2		-2.10	
2514	In house	13.09		0.03	
2531	UNI11057	13		0.00	
2538		-----		-----	
2553	CPSD AN 00094	17.41		1.36	
2561		16.898		1.20	
2590	LFGB B82.02.8	14.575		0.49	
2591	ISO17070Mod.	10.6809		-0.72	
2638	In house	13.210		0.06	
2644	UNI11057	12.98		-0.01	
2649	In house	12.6		-0.13	
2654	XPG08-015	17.60		1.42	
2665	In house	1.214	R(0.01)	-3.65	
2678	§64 LFGB B82.02.8	8.08		-1.52	
2713	DIN50009	15.899		0.89	
2719	In house	15.38		0.73	
2858	In house	8.890		-1.27	
2912	UNI11057	15.281		0.70	
2976	DIN50009	11.483		-0.47	
2977	In house	0.013	C,R(0.01)	-4.02	first reported not detected
2982	LFGB B82.02.8	15.87		0.89	
3001	In house	14.48		0.46	
3116	DIN50009	16.5		1.08	
3149	In house	9.55		-1.07	
3153		-----		-----	
3154	DIN50009	13.94		0.29	
3172	KS K0733	13.114		0.03	
3197	LFGB B82.02.8	11.2		-0.56	
3210		19.33		1.96	
3230		-----		-----	
3232	ISO17070	15.388		0.74	

lab	method	value	mark	z(targ)	remarks
3237		18.75		1.78	
8030	DIN50009	13.8392		0.26	
	normality	suspect			
	n	69			
	outliers	2			
	mean (n)	13.006			
	st.dev. (n)	2.794	RSD =21%		
	R(calc.)	7.822			
	st.dev.(iis memo 1601)	3.234			
	R(iis memo 1601)	9.056			





APPENDIX 2 Other reported test results

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

Determination individual and other Chlorinated Phenols on sample #22805; in mg/kg

lab	2346-TeCP	2356-TeCP	234-TCP	235-TCP	236-TCP	245-TCP	246-TCP	345-TCP
210	-----	-----	-----	-----	-----	-----	-----	-----
551	0.2994	-----	-----	-----	-----	-----	-----	-----
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
2108	0.03	0.04	-----	-----	-----	-----	-----	-----
2120	< 0,04	0.06	< 0,04	< 0,04	< 0,04	< 0,04	< 0,04	< 0,04
2121	-----	-----	-----	-----	-----	-----	-----	-----
2129	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
2135	0.066	0.067	-----	-----	-----	-----	-----	-----
2137	-----	-----	-----	-----	-----	-----	-----	-----
2159	not determined	not determined	not determined	not determined	not determined	not determined	not determined	not determined
2217	-----	-----	-----	-----	-----	-----	-----	-----
2255	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2264	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2265	-----	-----	-----	-----	-----	-----	-----	-----
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	Not Detected	Not Detected
2320	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2326	ND	ND	ND	ND	ND	ND	ND	ND
2330	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2347	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
2350	<0.125	<0.125	<0.125	<0.125	<0.125	<0.125	<0.125	<0.125
2352	-----	-----	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----	-----	-----
2358	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2363	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2365	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05
2366	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
2370	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2373	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2375	-----	-----	-----	-----	-----	-----	-----	-----
2378	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05
2379	0.0928	0.0773	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	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2386	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2425	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2426	-----	-----	-----	-----	-----	-----	-----	-----
2449	-----	-----	-----	-----	-----	-----	-----	-----
2453	-----	-----	-----	-----	-----	-----	-----	-----
2456	traces	traces	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed
2459	not detected	8.56	not detected	not detected	not detected	not detected	not detected	0.60
2511	-----	-----	-----	-----	-----	-----	-----	-----
2514	-----	-----	-----	-----	-----	-----	-----	-----
2531	-----	-----	-----	-----	-----	-----	-----	-----
2538	-----	-----	-----	-----	-----	-----	-----	-----
2553	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2561	-----	-----	-----	-----	-----	0.07665	-----	-----
2590	0.122	-----	-----	-----	-----	-----	-----	-----
2591	0.1318	0.0925	Not Detected	Not Detected	Not Detected	0.0524	Not Detected	Not Detected
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
2649	-----	-----	-----	-----	-----	-----	-----	-----
2654	-----	-----	-----	-----	-----	-----	-----	-----
2665	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2678	0.112	0.12	not detected	not detected	not detected	not detected	not detected	not detected
2713	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05

lab	2346-TeCP	2356-TeCP	234-TCP	235-TCP	236-TCP	245-TCP	246-TCP	345-TCP
2719	-----	-----	-----	-----	-----	-----	-----	-----
2858	-----	-----	not detected	not detected	not detected	not detected	not detected	not detected
2912	-----	-----	-----	-----	-----	-----	-----	-----
2976	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2977	2.957 C	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2982	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
3001	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
3116	-----	-----	-----	-----	-----	-----	-----	-----
3149	-----	-----	-----	-----	-----	-----	-----	-----
3153	-----	-----	-----	-----	-----	-----	-----	-----
3154	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
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
3230	-----	-----	-----	-----	-----	-----	-----	-----
3232	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
3237	-----	-----	-----	-----	-----	-----	-----	-----
8030	0.1687	0.0915	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected

Lab 2977 first reported 2.6697

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 #22805; in mg/kg

lab	23-DCP	24-DCP	25-DCP	26-DCP	34-DCP	35-DCP	2-CP	3-CP	4-CP
210	-----	-----	-----	-----	-----	-----	-----	-----	-----
551	-----	-----	-----	-----	-----	-----	-----	-----	-----
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
2108	-----	-----	-----	-----	-----	-----	-----	-----	-----
2120	< 0,04	< 0,04	< 0,04	< 0,04	< 0,04	< 0,04	< 0,04	< 0,04	< 0,04
2121	-----	-----	-----	-----	-----	-----	-----	-----	-----
2129	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
2135	-----	-----	-----	-----	-----	-----	-----	-----	-----
2137	-----	-----	-----	-----	-----	-----	-----	-----	-----
2159	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed
2217	-----	-----	-----	-----	-----	-----	-----	-----	-----
2255	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2264	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2265	-----	-----	-----	-----	-----	-----	-----	-----	-----
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	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2326	ND	ND	ND	ND	ND	ND	ND	ND	ND
2330	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2347	-----	-----	-----	-----	-----	-----	-----	-----	-----
2350	<0.125	<0.125	<0.125	<0.125	<0.125	<0.125	<0.125	<0.125	<0.125
2352	-----	-----	-----	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----	-----	-----	-----
2358	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2363	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2365	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05
2366	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5
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	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05	＜0.05
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	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2386	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2425	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2426	-----	-----	-----	-----	-----	-----	-----	-----	-----
2449	-----	-----	-----	-----	-----	-----	-----	-----	-----
2453	-----	-----	-----	-----	-----	-----	-----	-----	-----
2456	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed
2459	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2511	-----	-----	-----	-----	-----	-----	-----	-----	-----
2514	-----	-----	-----	-----	-----	-----	-----	-----	-----
2531	-----	-----	-----	-----	-----	-----	-----	-----	-----
2538	-----	-----	-----	-----	-----	-----	-----	-----	-----
2553	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2561	-----	-----	-----	-----	-----	-----	-----	-----	-----
2590	-----	-----	-----	-----	-----	-----	-----	-----	-----
2591	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
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
2649	-----	-----	-----	-----	-----	-----	-----	-----	-----
2654	-----	-----	-----	-----	-----	-----	-----	-----	-----
2665	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
2678	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2713	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05
2719	-----	-----	-----	-----	-----	-----	-----	-----	-----
2858	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2912	-----	-----	-----	-----	-----	-----	-----	-----	-----
2976	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2977	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed
2982	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
3001	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
3116	-----	-----	-----	-----	-----	-----	-----	-----	-----
3149	-----	-----	-----	-----	-----	-----	-----	-----	-----
3153	-----	-----	-----	-----	-----	-----	-----	-----	-----
3154	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
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
3230	-----	-----	-----	-----	-----	-----	-----	-----	-----
3232	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
3237	-----	-----	-----	-----	-----	-----	-----	-----	-----
8030	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	1g	Ultrasonic	n-Hexane
551	Yes	Further cut	1g	Ultrasonic	KOH Followed by n-hexane
623	Yes	Further cut	1	Ultrasonic	n Hexane
840	Yes	Further cut	1.0	Ultrasonic	KOH
2108	Yes	Used as received	1 g	Ultrasonic	KOH / iso-Octane
2120	No	Other	0,5 g	Oven 90 ºC	KOH
2121	Yes	Used as received	1g	Microwaves extraction	KOH for extraction Iso-Octane to analyze GCMS
2129	Yes	Used as received	0,5 g	ASE	acetone with 0,1% acetic acid
2135	Yes	Used as received	1g	extraction	Basic extraction with Sodiumcarbonate w= 25%
2137	Yes	Used as received	1	Ultrasonic	2M KOH
2159	Yes	Further cut	1 gram	Ultrasonic	KOH - Hexane
2217	Yes	Used as received	0.5022	Mechanical Shaking	hexane
2255	Yes	Further cut	0.5	Ultrasonic	n-Hexane
2264	No	Further cut		Ultrasonic	Potassium carbonate
2265	---	---		---	
2310	Yes	Further cut	2	Mechanical Shaking	Hexane
2311	Yes	Further cut	0.5	Thermal Desorption	KOH/ Hexane
2313	Yes	Further cut	1.0g	Steam distillation	n-Hexane
2320	Yes	Further cut	0.5g	Mechanical Shaking	Hexane
2326	Yes	Further cut	2.0 GM	Ultrasonic	KOH
2330	No	Further cut	2 g	Ultrasonic	KOH and n-Hexane
2347	No	Used as received	1.0g	Mechanical Shaking	
2350	No	Further cut	2.0163 g	Thermal Desorption	KOH
2352	Yes	Further cut	0.9996g	Mechanical Shaking	N-Hexane
2357	---	---		---	
2358	Yes	Used as received	2.5	Other	KOH follow by Hexane
2363	Yes	Further cut	1.9967g	Ultrasonic	H-Hexane
2365	Yes	Further cut	2.0g	Ultrasonic	KOH solution
2366	No	Further cut	1.0g	Mechanical Shaking	KOH, hexane
2370	Yes	Further cut	1 g	Distillation	Water
2373	Yes	Further cut	1g	Ultrasonic	n-Hexane
2375	Yes	Further cut	1 gram	Ultrasonic	KOH Extraction
2378	No	Used as received	0.9g	Mechanical Shaking	N-Hexane
2379	No	Further cut	2 g	Other	KOH
2380	Yes	Further cut	1.0 g	Ultrasonic	KOH Followed by n-hexane
2382	Yes	Further cut	1g	Ultrasonic	Hexane
2386	Yes	Further cut	0.5 g	first Ultrasonic, than Thermal Desorption	
2425	Yes	Further cut	0.5g	Thermal Desorption	1 M KOH solution
2426	Yes	Further cut	0.5 gram	KOH Extraction at 90°C	KOH Extraction. Acetylation / n-Hexane
2449	Yes	Further cut	1 gram	Ultrasonic	n -hexane
2453	No	Further cut	±0.5g	Thermal Desorption	
2456	Yes	Used as received	All quantity dispatched was used.	Ultrasonic	Potassium Carbonate 1.5% w/v
2459	Yes	Further cut	1.00 gram	Ultrasonic	n-Hexane
2511	---	---		---	
2514	Yes	Further cut	0.2530	Mechanical Shaking	n-Hexane
2531	Yes	Used as received	1 g	Ultrasonic	Polycarbonate solution 1.5%
2538	Yes	Further cut	0,5 g	Extraction with KOH, 16 h at 90 °C	Extraction KOH, Derivatisation in Hexane with acetic anhydride
2553	Yes	Further cut	1g	Mechanical Shaking	2M KOH
2561	No	Used as received	1g	Mechanical Shaking	
2590	Yes	Further cut	1g	Mechanical Shaking	hexane
2591	Yes	Further cut	1.0 grams	Other	Technique used to release/extract the Chlorinated Phenols: Basic Digestion
2638	No	Further cut	1 gm	Ultrasonic	Hexane
2644	Yes	Further cut	0.5	Mechanical Shaking	KOH 0.5 M/ hexane
2649	Yes	Further cut	1 g	Ultrasonic	n-Hexane
2654	Yes	Used as received	5 grams	Ultrasonic	K2CO3 1.5%

lab	17025 accr.	Sample preparation	Sample intake (grams)	Extraction technique	Extraction solution
2665	Yes	Further cut	0.5 g	Ultrasonic	Dichloromethane
2678	No	Used as received	2 grams	Ultrasonic	KOH followed by Hexane
2713	No	Further cut	0,5 g	Oven-90 °C - 16h (KOH)	1M KOH-K2CO3/Hexane
2719	Yes	Further cut	0.5g	Ultrasonic	KOH 1M
2858	Yes	Further cut	1.0 gm	Hot chamber/cabinet	KOH + Iso Octane
2912	Yes	Used as received	1g	Ultrasonic	Potassium carbonate solution
2976	Yes	Further cut	0.5029 g	Thermal Desorption	KOH 1M
2977	No	Used as received	0,5 g	Mechanical Shaking	Hexane after derivatization
2982	Yes	Used as received	1.0g	Mechanical Shaking	n-Hexane
3001	No	Used as received	1	Ultrasonic	KOH solution/Hexane
3116	No	Used as received	0.5 gram	Oven	1M KOH
3149	Yes	Used as received	1 g	Soxhlet	Acetone
3153	Yes	Further cut	0.5g	Steam Distillation	n-hexane
3154	Yes	Further cut	0,5 grams	Other	1M KOH, 16 hours at 90 °C
3172	Yes	---		---	
3197	Yes	Further cut	0,5 g	Other	Distilled water/potassium carbonate solution
3210	---	---		---	
3230	---	---		---	
3232	Yes	Further cut	0.5	Mechanical Shaking	K2CO3/n-Hexane
3237	Yes	Further cut	0,5	Mechanical Shaking	Hexane
8030	No	Further cut	1 g	Other	KOH

APPENDIX 4

Number of participants per country

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

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2 ISO5725:86
3 ISO5725 parts 1-6:94
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