

Results of Proficiency Test
Chlorinated Organic Compounds
(COC) in Textile
October 2022

Organized by: Institute for Interlaboratory Studies

Spijkenisse, the Netherlands

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1 Introduction

Since the 1990's many countries have adopted environmental standards and requirements restricting the use of harmful chemicals in the production of textiles and clothing. Laws and regulations impose some of these standards and requirements. In addition to mandatory environmental standards and requirements for textiles, some Eco-labelling schemes are imposing environmental requirements for textile products on a voluntary basis, e.g. Milieukeur (Netherlands), Bluesign® (Switzerland) and Oeko-Tex® Standard 100 (Switzerland).

The determination of Chlorinated Benzenes and Chlorinated Toluenes (Chlorinated Organic Compounds) is known to give problems with the comparability of laboratory results. However, no appropriate reference materials are yet available. As an alternative, participation in a proficiency test may enable laboratories to check their performance. Therefore, a proficiency test (laboratory-evaluating interlaboratory study) for the determination of Chlorinated Organic Compounds in Textile was organized by the Institute for Interlaboratory Studies for the first time in September 2021 on request of many participants. During the annual proficiency testing program 2022/2023 it was decided to continue the proficiency test for the determination of Chlorinated Organic Compounds in Textile.

In this interlaboratory study 75 laboratories in 22 countries registered for participation, see appendix 4 for the number of participants per country. In this report the results of the Chlorinated Organic Compounds 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 gray polyester sample of 5 grams labelled #22730. 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 gray polyester was selected which was made positive on Chlorinated Organic Compounds by a third party. The batch was cut into small pieces and after homogenization 80 small plastic bags were filled with approximately 5 grams each and labelled #22730. The homogeneity of the subsamples was checked by determination of 1,2,4,5-Tetrachlorobenzene in accordance with EN17137 on 6 stratified randomly selected subsamples.

	1,2,4,5-Tetrachlorobenzene in mg/kg
sample #22730-1	184.77
sample #22730-2	185.76
sample #22730-3	187.47
sample #22730-4	187.55
sample #22730-5	189.93
sample #22730-6	186.00

Table 1: homogeneity test results of subsamples #22730

From the above test results the repeatability was 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.

	1,2,4,5-Tetrachlorobenzene in mg/kg
r (observed)	5.1
reference method	Horwitz
0.3 x R (reference method)	11.4

Table 2: evaluation of the repeatability of subsamples #22730

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 sample labelled #22730 was sent on September 21, 2022.

2.5 ANALYZES

The participants were requested to determine on the sample the concentrations of eleven individual Chlorotoluenes and eleven individual Chlorobenzenes.

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 the 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 F(0.01) for the Rosner's test. Stragglers are marked by F(0.01) 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_{\text{(target)}} = \text{(test result - average of PT)} / \text{target standard deviation}
```

The $z_{(target)}$ scores are listed in the test result tables in appendix 1.

Absolute values for z<2 are very common and absolute values for z>3 are very rare. Therefore, the usual interpretation of z-scores is as follows:

```
|z| < 1 good
1 < |z| < 2 satisfactory
2 < |z| < 3 questionable
3 < |z| unsatisfactory
```

4 EVALUATION

In this proficiency test no problems were encountered with the dispatch of the samples. Nine participants reported the 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 components requested.

In total 73 participants reported 325 numerical test results. Observed were 2 outlying test results, which is 0.6%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

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

4.1 EVALUATION PER COMPONENT

In this section the reported test results are discussed per component. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 5.

Test method EN17137 is considered to be the official test method for the determination of Chlorotoluenes and Chlorobenzenes in Textile. The precision data mentioned in EN17137 appendix B.5) is not clear and measured at one concentration level of 0.1 mg/kg. Therefore, it is decided to use the Horwitz equation for estimation of the target reproducibilities and to mention the requirements from EN17137:18 for comparison only.

- 2-Chlorotoluene: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility calculated with the Horwitz equation.
- <u>1,2,4-Trichlorobenzene:</u> This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility calculated with the Horwitz equation
- <u>1,2,3,4-Tetrachlorobenzene:</u> This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated reproducibility calculated with the Horwitz equation.
- <u>1,2,4,5-Tetrachlorobenzene:</u> This determination may be problematic. No statistical outliers were observed. The calculated reproducibility is not at all in agreement with the estimated reproducibility calculated with the Horwitz equation.
- <u>Pentachlorobenzene:</u> This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated reproducibility calculated with the Horwitz equation.
- <u>Hexachlorobenzene:</u> This determination may be problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the estimated reproducibility calculated with the Horwitz equation.

The majority of the participants agreed on a concentration near or below the limit of detection for all other requested components. Therefore, no z-scores are calculated. The 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)
2-Chlorotoluene	mg/kg	66	0.87	0.41	0.40
1,2,4-Trichlorobenzene	mg/kg	45	0.15	0.07	0.09
1,2,3,4-Tetrachlorobenzene	mg/kg	45	0.17	0.09	0.10
1,2,4,5-Tetrachlorobenzene	mg/kg	70	96.7	50.2	21.8
Pentachlorobenzene	mg/kg	26	0.12	0.05	0.07
Hexachlorobenzene	mg/kg	71	28.2	14.9	7.6

Table 3: reproducibilities of components on sample #22730

Without further statistical calculations it can be concluded that for many tests there is a good compliance of the group of participants with the reference method. The problematic tests have been discussed in paragraph 4.1 and 5.

4.3 COMPARISON OF THE PROFICIENCY TEST OF OCTOBER 2022 WITH THE PREVIOUS PT

	October 2022	November 2021
Number of reporting laboratories	73	26
Number of test results	325	93
Number of statistical outliers	2	5
Percentage of statistical outliers	0.6%	5.4%

Table 4: comparison with the previous proficiency test

In proficiency tests, outlier percentages of 3% - 7.5% are guite normal.

The performance of the determinations of the proficiency test was compared, expressed as relative standard deviation (RSD) of the PTS, see next table.

Component	October 2022	November 2021	Target (Horwitz)
2-Chlorotoluene	17%	22%	16%
1,4-Dichlorobenzene		17%	21%
1,2,4-Trichlorobenzene	16%		21%
1,2,3,4-Tetrachlorobenzene	19%		21%
1,2,4,5-Tetrachlorobenzene	19%	19%	8%
Pentachlorobenzene	16%		22%
Hexachlorobenzene	19%	12%	10%

Table 5: development of the uncertainties over the years

4.4 EVALUATION OF ANALYTICAL DETAILS

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:

- A majority (about 90%) of the participants mentioned that they are accredited for the determination of Chlorinated Organic Compounds in Textile.
- About 20% of the participants used the sample as received and about 80% did further cut or further grind the samples prior to analysis.
- About 20% of the participants used 0.5 grams or less of sample intake, about 55% used a sample intake of 1 gram and about 25% used a sample intake of 2 grams or more.
- All participants mentioned to have used Dichloromethane as extraction solvent and almost all participants carried out the extraction at room temperature for 30 minutes.

As the majority of the group follow the same analytical procedures no separate statistical analysis has been performed.

5 DISCUSSION

In this PT the average of the homogeneity test results is not in line with the average (consensus value) from the PT results. There are several reasons for this. First, the goal of the homogeneity testing is different from the goal of the evaluation of the reported PT results. In order to prove the homogeneity of the PT samples, a test method is selected with a high precision (smallest variation). The accuracy (trueness) of the test method is less relevant. Secondly, the homogeneity testing is done by one laboratory only. The test results of this ISO/IEC17025 accredited laboratory will have a bias (systematic deviation) depending on the test method used. The desire to detect small variations between the PT samples leads to the use of a sensitive test method with high precision, which may be a test method with significant bias.

Also, each test result reported by the laboratories that participate in the PT will have a bias. However, some will have a positive bias and others a negative bias. These different biases compensate each other in the PT average (consensus value). Therefore, the PT consensus value may deviate from the average of the homogeneity test. At the same time the accuracy of the PT consensus value is more reliable than the accuracy of the average of the homogeneity test.

For the determination of Chlorinated organic Compounds limits are mentioned in Ecolabelling standards like Oeko-Tex® 100. See table below.

	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)
Sum of Chlorotoluenes and Chlorobenzenes	1.0	1.0	1.0	1.0

Table 6: Oeko-Tex® 100 Ecolabelling Standard and Requirements for Textiles in EU

It appears that all reporting participants would have rejected the sample for all Classes for Chlorotoluenes and Chlorobenzenes.

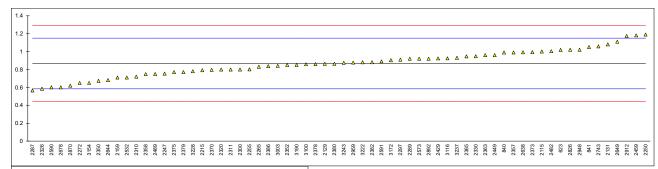
6 CONCLUSION

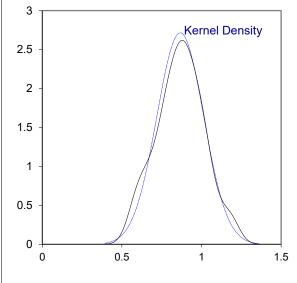
Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

APPENDIX 1
Determination of 2-Chlorotoluene (CAS No. 95-49-8) on sample #22730; results in mg/kg

Deteili	Illitation of	Z-Ciliorotoluene	(CAS NO.	95-49-6)	on sample #22730; results in mg/kg
lab	method	value	mark	z(targ)	remarks
623	EN17137	1.018		1.07	
840	EN17137	0.99		0.87	
841	EN17137	1.05		1.29	
2115	EN17137	1.0		0.94	
2129		0.8628		-0.03	
2131	In house	1.08		1.50	
2159	EN17137	0.71		-1.11	
2215	EN17137	0.791		-0.54	
2232	EN147407				
2247	EN17137	0.754		-0.80	
2250	EN17137	1.190		2.28	
2255	EN17137	0.801		-0.46	
2265	EN17137	0.83		-0.26	
2272	EN17137	0.65		-1.53	
2287		0.5650		-2.13	
2289	EN17137	0.92		0.38	
2291	EN17137	ND			
2293					
2297	EN17137	0.91		0.30	
2300	EN17137	0.8	С	-0.47	first reported: not detected
2301					•
2310	EN17137	0.72		-1.04	
2311	EN17137	0.80		-0.47	
2320	EN17137	0.800		-0.47	
2326	EN17137 EN17137	0.585		-0.47 -1.99	
2330 2350	EN17137 EN17137	0.948 0.673		0.57 -1.37	
2352	EN17137	0.85		-0.12	
2357	EN17137	0.990		0.87	
2358	EN17137	0.7488		-0.83	
2363	EN17137	0.96		0.66	
2365	EN17137	0.946		0.56	
2370	EN17137	0.7957		-0.50	
2373	EN17137	0.9937		0.90	
2375	EN17137	0.77		-0.68	
2378	EN17137	0.86		-0.05	
2379	EN17137	0.7706		-0.68	
2380	EN17137	0.864		-0.02	
2382	EN17137	0.881		0.10	
2386	EN17137	0.838		-0.20	
2429	EN17137	0.923		0.40	
2449	EN17137	0.96		0.66	
2459	EN17137	1.18		2.21	
2482	DIN54232	1.004		0.97	
2489	EN17137	0.75		-0.82	
2532	EN17137	0.71		-1.11	
2573	21111101	0.92		0.38	
2590	EN17137	0.6		-1.88	
2590	EN17137 EN17137	0.89		0.16	
2638	EN17137 EN17137	0.89		0.16	
2643	LIVII 131				
	EN147407	0.60		1.22	
2644	EN17137	0.68		-1.32	
2649	EN17137	1.11		1.72	
2678	EN17137	0.6		-1.88	
2737	EN12=16=				
2743	EN17137	1.05742		1.34	
2768	DIN54232	n.d			
2826	EN17137	1.02		1.08	
2870	EN17137	0.619		-1.75	
2892	EN17137	0.92		0.38	
2912	EN17137	1.174		2.17	
2948		1.02		1.08	
2959	EN17137	0.877		0.07	
3003	DIN54232	0.84		-0.19	
3100	EN17137	0.858		-0.06	
3116		0.924		0.40	
3154	EN17137	0.65	С	-1.53	first reported 0.317
3172	EN17137	0.9026	-	0.25	
3190	EN17137	0.85		-0.12	
3222	EN17137 EN17137	0.88		0.09	
3228	EN17137	0.78		-0.61	
3230	EN147407	0.03		0.45	
3237	EN17137	0.93		0.45	
3243	EN17137	0.874		0.05	

lab	method	value	mark	z(targ)	remarks
3250					
	normality	OK			
	n	66			
	outliers	0			
	mean (n)	0.86683			
	st.dev. (n)	0.146934	RSD = 17%		
	R(calc.)	0.41141			
	st.dev.(Horwitz)	0.141708			
	R(Horwitz)	0.39678			
Compa					
	R(EN17137:18)	0.28470			

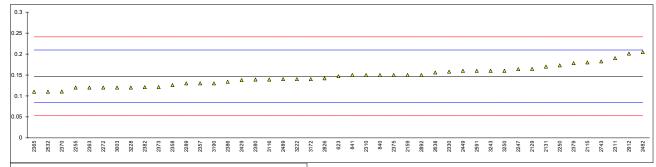


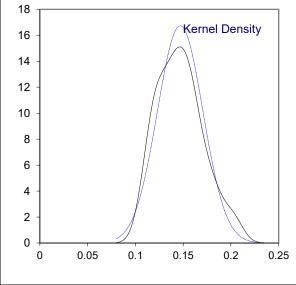


Determination of 1,2,4-Trichlorobenzene (CAS No. 120-82-1) on sample #22730; results in mg/kg

lab	method	value	mark	z(targ)	remarks
623	EN17137	0.147		0.00	
840	EN17137	0.15		0.10	
841	EN17137	0.15		0.10	
2115	EN17137	0.18		1.06	
2129		0.1644		0.56	
2131	In house	0.17		0.74	
2159	EN17137	0.15		0.10	
2215 2232	EN17137	not detected			
2232	EN17137	0.164		0.55	
2250	EN17137	0.173		0.83	
2255	EN17137	0.120		-0.86	
2265	EN17137				
2272	EN17137	0.12		-0.86	
2287		<0.2			
2289	EN17137	0.13		-0.54	
2291	EN17137	ND			
2293					
2297	EN17137	Not detected			
2300	EN17137	Not detected			
2301	EN147407	0.45		0.40	
2310	EN17137	0.15		0.10	
2311 2320	EN17137	0.19		1.38	
2326	EN17137 EN17137	<0.1 ND			
2330	EN17137 EN17137	0.158		0.35	
2350	EN17137	<0.1		0.55	
2352	EN17137				
2357	EN17137	0.130		-0.54	
2358	EN17137	0.1258		-0.67	
2363	EN17137	0.12		-0.86	
2365	EN17137	0.110		-1.18	
2370	EN17137	0.1105		-1.16	
2373	EN17137	0.1212		-0.82	
2375	EN17137	0.15		0.10	
2378	EN17137	<0.2			
2379	EN17137	0.1782		1.00	
2380	EN17137	0.139		-0.25	
2382 2386	EN17137	0.121 0.134		-0.82 -0.41	
2429	EN17137 EN17137	0.134		-0.41	
2449	EN17137 EN17137	0.16		0.42	
2459	EN17137				
2482	DIN54232	0.2045		1.84	
2489	EN17137	0.14		-0.22	
2532	EN17137	0.11		-1.18	
2573		not detected			
2590	EN17137				
2591	EN17137	0.16		0.42	
2638	EN17137	0.156		0.29	
2643					
2644	EN17137	not detected			
2649	EN17137	 Ni-6 defects d			
2678	EN17137	Not detected			
2737 2743	EN17137	0.18242		1.13	
2768	DIN54232	0.16242 n.d		1.13	
2826	EN17137	0.142		-0.16	
2870	EN17137				
2892	EN17137	0.15		0.10	
2912	EN17137	0.201		1.73	
2948					
2959	EN17137				
3003	DIN54232	0.12		-0.86	
3100	EN17137	not detected			
3116		0.139		-0.25	
3154	EN17137	not detected			
3172	EN17137	0.14013		-0.21	
3190	EN17137	0.13		-0.54	
3222	EN17137	0.14		-0.22	
3228 3230	EN17137	0.12		-0.86	
3230 3237	EN17137				
3243	EN17137 EN17137	0.160		0.42	
5275	_1417 107	0.100		0.72	

lab	method	value	mark	z(targ)	remarks
3250		0.16		0.42	
	normality	ОК			
	n	45			
	outliers	0			
	mean (n)	0.14687			
	st.dev. (n)	0.023855	RSD = 16%		
	R(calc.)	0.06679			
	st.dev.(Horwitz)	0.031365			
	R(Horwitz)	0.08782			
Compa					
	R(EN17137:18)	0.04935			

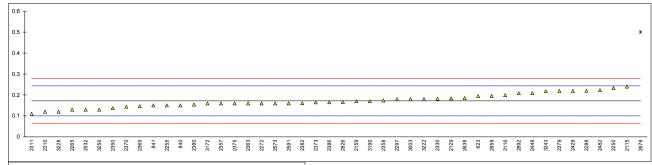


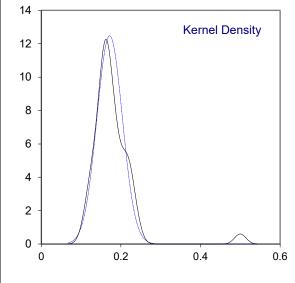


Determination of 1,2,3,4-Tetrachlorobenzene (CAS No. 634–66–2) on sample #22730; results in mg/kg

mg/kg					
lab	method	value	mark	z(targ)	remarks
623	EN17137	0.195		0.65	
840	EN17137	0.15		-0.61	
841	EN17137 EN17137	0.15		-0.61	
2115	EN17137	0.24		1.91	
2129		0.1836		0.33	
2131	In house	not detected			
2159	EN17137	0.17		-0.05	
2215	EN17137	not detected			
2232					
2247	EN17137	<0.1			
2250	EN17137	0.233		1.71	
2255	EN17137	0.15		-0.61	
2265	EN17137	0.13		-1.17	
2272	EN17137	0.16		-0.33	
	EN17137			-0.55	
2287	EN47407	<0.2			
2289	EN17137	0.22		1.35	
2291	EN17137	ND			
2293					
2297	EN17137	0.18		0.23	
2300	EN17137	Not detected			
2301					
2310	EN17137	0.12		-1.44	
2311	EN17137	0.11		-1.72	
2320	EN17137	<0.1			
2326	EN17137	ND			
2330	EN17137	0.182		0.29	
2350	EN17137 EN17137	0.137		-0.97	
	LINIT IST				
2352	EN17127	0.160		0.22	
2357	EN17137	0.160		-0.33	
2358	EN17137	0.1735		0.05	
2363	EN17137	0.16		-0.33	
2365	EN17137	0.146		-0.72	
2370	EN17137	0.1443		-0.77	
2373	EN17137	0.1645		-0.20	
2375	EN17137	0.16		-0.33	
2378	EN17137	<0.2			
2379	EN17137	0.2190		1.32	
2380	EN17137	0.154		-0.50	
2382		0.161		-0.30	
	EN17137				
2386	EN17137	0.165		-0.19	
2429	EN17137	0.219		1.32	
2449	EN17137	0.209		1.04	
2459					
2482	DIN54232	0.2221		1.41	
2489	EN17137	ND			
2532	EN17137	0.13		-1.17	
2573		0.16		-0.33	
2590					
2591	EN17137	0.16		-0.33	
2638	EN17137	0.185		0.37	
2643					
2644	EN17137	not detected			
2644 2649	LIVIT IST				
	EN17107	 0 E	D(0.04)	0.40	
2678	EN17137	0.5	R(0.01)	9.16	
2737					
2743					
2768	DIN54232	n.d			
2826	EN17137	0.166		-0.16	
2870					
2892	EN17137	0.208		1.01	
2912	•				
2948					
2959	EN17137	0.195		0.65	
3003	DIN54232	0.195		0.03	
3100	EN17137	not detected		0.70	
3116	=114=46=	0.199		0.76	
3154	EN17137	not detected			
3172	EN17137	0.15936		-0.35	
3190	EN17137	0.17		-0.05	
3222	EN17137	0.18		0.23	
3228	EN17137	0.12		-1.44	
3230	e :				
3237					
3243	EN17137	0.218	С	1.29	first reported 113.45
0240	LI417 107	0.210	9	1.23	mot reported 1 10.70

lab	method	value	mark	z(targ)	remarks
3250	EN17137	0.13		-1.17	
	normality	ОК			
	n	45			
	outliers	1			
	mean (n)	0.17174			
	st.dev. (n)	0.031988	RSD = 19%		
	R(calc.)	0.08957			
	st.dev.(Horwitz)	0.035823			
	R(Horwitz)	0.10030			
Compa					
	R(EN17137:18)	0.06636			

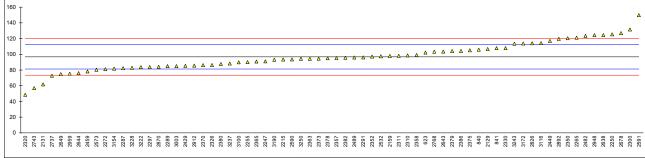


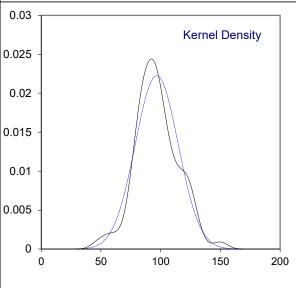


Determination of 1,2,4,5-Tetrachlorobenzene (CAS No. 95-94-3) on sample #22730; results in mg/kg

mg/kg					
lab	method	value	mark	z(targ)	remarks
			man		Tomano
623	EN17137	102.182		0.70	
840	EN17137	105.9		1.18	
841	EN17137	107.85		1.43	
2115					
2129		106.67		1.28	
	la barra				
2131	In house	61.73		-4.50	
2159	EN17137	97.84		0.14	
2215	EN17137	93.27		-0.45	
2232					
	EN147407				
2247	EN17137	90.982		-0.74	
2250	EN17137	125.338		3.68	
2255	EN17137	90.0		-0.87	
2265	EN17137	121.20		3.14	
2272	EN17137	81.02		-2.02	
2287		82.20		-1.87	
2289	EN17137	84.93		-1.52	
2291	EN17137	96.05		-0.09	
	LIVIT 101				
2293					
2297	EN17137	83.75		-1.67	
2300	EN17137	131.49		4.47	
2301					
2310	EN17137	98.5		0.23	
2311	EN17137	98.06		0.17	
2320	EN17137	48.436		-6.21	
2326	EN17137	86.28		-1.35	
2330	EN17137	107.858		1.43	
2350	EN17137	120.42		3.04	
2352	EN17137	97.06		0.04	
2357	EN17137	95.210		-0.20	
2358	EN17137	99.0514		0.30	
2363	EN17137	94.31		-0.31	
2365	EN17137	90.780		-0.77	
2370	EN17137	86.0856		-1.37	
2373	EN17137	94.4736		-0.29	
2375	EN17137	105		1.06	
2378	EN17137	95		-0.22	
2379	EN17137	104.1418		0.95	
2380	EN17137	87.657		-1.17	
2382	EN17137	95.571		-0.15	
2386	EN17137	104.2	С	0.96	first reported 44.15
2429	EN17137	85.276		-1.47	·
2449	EN17137	117		2.60	
2459	EN17137	78.17		-2.39	
2482	DIN54232	123.4		3.43	
2489	EN17137	95.9		-0.11	
2532	EN17137	97.37		0.08	
2573		80.27		-2.12	
	EN147407		•		C 1 11010
2590	EN17137	93.4	С	-0.43	first reported 4.912
2591	EN17137	149.75		6.81	
2638	EN17137	124.5		3.57	
2643	EN17137	103.37		0.85	
2644	EN17137	75.98		-2.67	
2649	EN17137	74.88		-2.81	
2678	EN17137	127		3.89	
2737	EN17137	72.6675		-3.10	
2743	EN17137	56.93474		-5.12	
2768	DIN54232	103.2763		0.84	
2826	EN17137	114	_	2.22	
2870	EN17137	84	С	-1.64	first reported 18.343
2892	EN17137	119.60		2.94	
2912	EN17137	85.583		-1.43	
2948		124.23		3.53	
	EN17107				
2959	EN17137	75.321		-2.75	
3003	DIN54232	85		-1.51	
3100	EN17137	89.745		-0.90	
3116		114.3		2.26	
3154	EN17137	81.666		-1.94	
3172	EN17137	113.73		2.18	
3190	EN17137	93.02		-0.48	
3222	EN17137	83.63		-1.69	
3228	EN17137	82.81		-1.79	
3230	EN147407			4.40	
3237	EN17137	88.16	_	-1.10	
3243	EN17137	113.45	С	2.15	first reported 0.218

lab	method	value	mark	z(targ)	remarks
3250	EN17137	94.24		-0.32	
	normality	OK			
	n	70			
	outliers	0			
	mean (n)	96.7447			
	st.dev. (n)	17.94425	RSD = 19%		
	R(calc.)	50.2439			
	st.dev.(Horwitz)	7.77822			
	R(Horwitz)	21.7790			
Compa					
	R(EN17137:18)	36.5695			

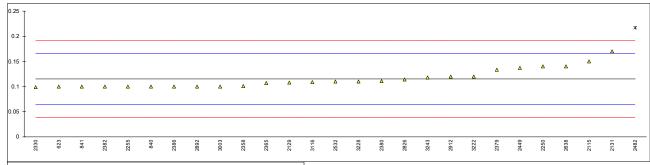


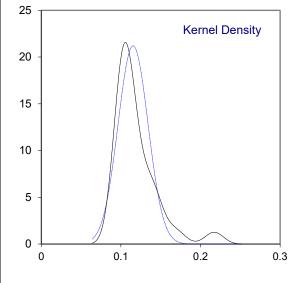


Determination of Pentachlorobenzene (CAS No. 608-93-5) on sample #22730; results in mg/kg

1-1-				-(4)	and the second s
lab	method	value	mark	z(targ)	remarks
623	EN17137	0.1		-0.60	
840	EN17137	0.10		-0.60	
841	EN17137	0.10		-0.60	
2115	EN17137	0.15		1.36	
2129		0.108		-0.29	
2131	In house	0.17	С	2.14	first reported 0.19
2159	EN17137	not determined			
2215	EN17137	not detected			
2232	LIVI7 107				
2247	EN17137	<0.1			
2250	EN17137 EN17137	0.14		0.97	
2255	EN17137	0.10		-0.60	
2265					
2272					
2287		<0.2			
2289					
2291	EN17137	ND			
2293					
2297	EN17137	Not detected			
2300	EN17137	Not detected			
2301					
2310	EN17137	not detected			
2311	EN17137	<0.1			
2320	EN17137	<0.1			
2326	EN17137 EN17137	ND			
2330	EN17137 EN17137	0.099		-0.64	
2350	EN17137 EN17137	<0.1		-0.04	
	LINIT 131				
2352					
2357	EN147407				
2358	EN17137	0.1009		-0.56	
2363	EN17137	not detected			
2365	EN17137	0.107		-0.32	
2370	EN17137	<0.1			
2373	EN17137	not detected			
2375					
2378	EN17137	<0.2			
2379	EN17137	0.1334		0.71	
2380	EN17137	0.111		-0.17	
2382	EN17137	0.100		-0.60	
2386	EN17137	0.100		-0.60	
2429	211111101				
2449	EN17137	0.137		0.85	
2459	EN17137	ND			
2482	DIN54232	0.2172	R(0.01)	3.99	
2489	EN17137	ND	13(0.01)	3.99	
		0.11			
2532	EN17137			-0.21	
2573		not detected			
2590	EN147407				
2591	EN17137	not detected			
2638	EN17137	0.14		0.97	
2643					
2644	EN17137	not detected			
2649					
2678	EN17137	Not detected			
2737					
2743					
2768	DIN54232	n.d			
2826	EN17137	0.114		-0.05	
2870					
2892	EN17137	0.10		-0.60	
2912	EN17137	0.120	С	0.18	first reported 0.166
2948			-		
2959					
3003	DIN54232	0.1		-0.60	
3100	EN17137	not detected		-0.00	
3116	LINII IJI	0.109		-0.25	
	EN17197			-0.25	
3154	EN17137	not detected			
3172	EN147407				
3190	EN17137	<0.1			
3222	EN17137	0.12		0.18	
3228	EN17137	0.11		-0.21	
3230					
3237					
3243	EN17137	0.118		0.11	

lab	method	value	mark	z(targ)	remarks
3250					
	normality	not OK			
	n	26			
	outliers	1			
	mean (n)	0.11528			
	st.dev. (n)	0.018815	RSD = 16%		
	R(calc.)	0.05268			
	st.dev.(Horwitz)	0.025533			
	R(Horwitz)	0.07149			
Compa					
	R(EN17137:18)	0.74079			

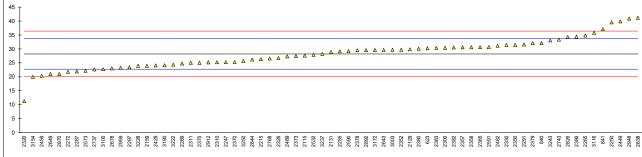


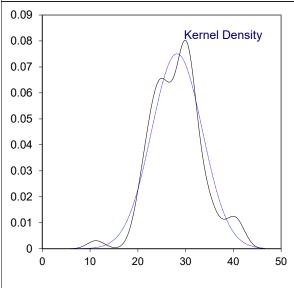


Determination of Hexachlorobenzene (CAS No. 118-74-1) on sample #22730; results in mg/kg

lab	method	value	mark	z(targ)	remarks
623	EN17137	30.229	mant	0.74	Tomana
840	EN17137	32.08		1.42	
841	EN17137	37.06		3.24	
2115	EN17137	27.6		-0.22	
2129	In the case of	29.833		0.59	
2131 2159	In house EN17137	28.88 23.93		0.25 -1.57	
2215	EN17137 EN17137	26.32		-0.69	
2232	LIVIT 101			-0.00	
2247	EN17137	25.231		-1.09	
2250	EN17137	39.508		4.14	
2255	EN17137	29.12		0.33	
2265	EN17137	34.80		2.41	
2272 2287	EN17137	21.79 21.92		-2.35 -2.30	
2289	EN17137	24.75		-1.27	
2291	EN17137	31.59		1.24	
2293					
2297	EN17137	23.42	_	-1.75	
2300	EN17137	30.38	С	0.79	first reported 46.75
2301 2310	EN17137	 25.2		 -1.10	
2311	EN17137 EN17137	24.99		-1.18	
2320	EN17137	11.225		-6.22	
2326	EN17137	26.73		-0.54	
2330	EN17137	31.418		1.17	
2350	EN17137	31.42		1.18	
2352	EN17137	29.69		0.54	
2357 2358	EN17137 EN17137	30.560 30.5841		0.86 0.87	
2363	EN17137	30.37		0.79	
2365	EN17137	30.655		0.90	
2370	EN17137	25.3026		-1.06	
2373	EN17137	27.4445		-0.28	
2375	EN17137	25 20 F		-1.18	
2378 2379	EN17137 EN17137	29.5 32.0589		0.47 1.41	
2380	EN17137 EN17137	30.099		0.69	
2382	EN17137	30.551		0.86	
2386	EN17137	34.38	С	2.26	first reported 17.53
2429	EN17137	24.065		-1.52	
2449	EN17137	39.87		4.27	
2459 2482	EN17137 DIN54232	20.35 31.14		-2.88 1.07	
2489	EN17137	27.3		-0.33	
2532	EN17137	27.86		-0.13	
2573		22.14		-2.22	
2590	EN17137	29.2	С	0.36	first reported 0.971
2591	EN17137	30.68		0.90	
2638 2643	EN17137 EN17137	41.173 29.62		4.75 0.52	
2644	EN17137 EN17137	26.2		-0.74	
2649	EN17137	21		-2.64	
2678	EN17137	23		-1.91	
2737	EN17137	22.6353		-2.04	
2743 2768	EN17137 DIN54232	33.33943 26.5692		1.88 -0.60	
2826	EN17137	34.3		2.23	
2870	EN17137	21	С	-2.64	first reported 76.522
2892	EN17137	29.56		0.49	·
2912	EN17137	25.153		-1.12	
2948	EN147407	40.89		4.64	
2959 3003	EN17137 DIN54232	23.168 29.67		-1.85 0.53	
3100	EN17137	29.67 22.710		-2.01	
3116	=	35.78		2.77	
3154	EN17137	19.966		-3.02	
3172	EN17137	29.608		0.51	
3190	EN17137	24.12		-1.50 1.40	
3222 3228	EN17137 EN17137	24.38 23.92		-1.40 -1.57	
3230	21417 107	23.92		-1.57	
3237	EN17137	28.2		0.00	
3243	EN17137	33.04		1.77	

lab	method	value	mark	z(targ)	remarks
3250	EN17137	25.71		-0.92	
	normality	suspect			
	n	71			
	outliers	0			
	mean (n)	28.2104			
	st.dev. (n)	5.31849	RSD = 19%		
	R(calc.)	14.8918			
	st.dev.(Horwitz)	2.73036			
	R(Horwitz)	7.6450			
Compa					
	R(EN17137:18)	14.4550			





APPENDIX 2 Other reported components

Abbreviations of components

3-CT = 3-Chlorotoluene CAS No. 108-41-8 4-CT = 4-Chlorotoluene CAS No. 106-43-4 23-DCT = 2,3-Dichlorotoluene CAS No. 32768-54-0 24-DCT = 2,4-Dichlorotoluene CAS No. 95-73-8 25-DCT = 2,5-Dichlorotoluene CAS No. 19398-61-9 26-DCT = 2,6-Dichlorotoluene CAS No. 118-69-4 34-DCT = 3,4-Dichlorotoluene CAS No. 95-75-0 236-TCT = 2,3,6-Trichlorotoluene CAS No. 2077-46-5 245-TCT = 2,4,5-Trichlorotoluene CAS No. 6639-30-1 **PentaCT** = Pentachlorotoluene CAS No. 877-11-2 12-DCB = 1,2-Dichlorobenzene CAS No. 95-50-1 13-DCB = 1,3-Dichlorobenzene CAS No. 541-73-1 = 1,4-Dichlorobenzene CAS No. 106-46-7 14-DCB 123-TCB = 1,2,3-Trichlorobenzene CAS No. 87-61-6 135-TCB = 1,3,5-Trichlorobenzene CAS No. 108-70-3 1235-TeCB = 1,2,3,5-Tetrachlorobenzene CAS No. 634-90-2

Determination of other Chlorinated Organic Compounds (COC) on sample #22730; in mg/kg

lab	3-CT	4-CT	23-DCT	24-DCT	25-DCT	26-DCT	34-DCT	236-TCT
623	Not Detected							
840	not detected							
841	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04
2115								
2129	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
2131	not detected							
0450	not							
2159	determined							
2215	not detected							
2232			< 0.1	< 0.1	< 0.1		< 0.1	
2247			***	***	***	< 0.1	***	< 0.1
2250 2255	not detected Not Detected							
2265	Not Detected							
2272								
2287	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
2289								
2291	ND							
2293								
2297	Not detected							
2300	Not detected							
2301								
2310	not detected							
2311	Not Detected							
2320	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2326	ND							
2330	Not Detected							
2350	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2352								
2357								
2358	not detected							
2363	not detected							
2365	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2370	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2373	not detected							
2375								
2378	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
2379	Not detected							
2380	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1 <0.100	<0.1
2382 2386	<0.100 < 0.1							
2386 2429	< 0.1	< 0.1	< 0.1					
2429								

lab	3-CT	4-CT	23-DCT	24-DCT	25-DCT	26-DCT	34-DCT	236-TCT
2449								
2459	ND							
2482	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2489	ND							
2532	Not Detected							
2573	not detected							
2590								
2591	not detected							
2638	not detected							
2643								
2644	not detected							
2649								
2678	Not detected							
2737								
2743		0.13292		0.17895				
2768	n.d							
2826	Not detected							
2870								
	not							
2892	determined							
2912								
2948								
2959								
3003								
3100	not detected							
3116 3154	not detected	not detected	not detected	not dotacted	not detected	not dotacted	not detected	not dotacted
3172	not detected < 0.025							
3172	< 0.025 < 0.1	< 0.025	< 0.025 < 0.1	< 0.025 < 0.1	< 0.025 <0.1	< 0.025 < 0.1	< 0.025	< 0.025 < 0.1
3222	<0.1 							
3222	not detected							
3220	not detected							
3237	0.93							
3231	not							
3243	determined							
3250								
5250								

Determination of other Chlorinated Organic Compounds (COC) on sample #22730; in mg/kg

lah	245 TOT	DontoCT	42 DCB	42 DCB	44 DCB	422 TCB	425 TOD	4225 ToCB
623	245-TCT Not Detected	PentaCT Not Detected	Not Detected	Not Detected	14-DCB 0.311	Not Detected	Not Detected	1235-TeCB Not Detected
840	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
841	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04	< 0.04
2115								85.34
2129	<0,1	<0,1	<0,1	<0,1	0.0938	<0,1	<0,1	106.67
2131	not detected	not detected	not detected	not detected	0.08	not detected	0.05	not detected
0.4=0	not	not	not	not	not	not	not	not
2159	determined	determined	determined	determined	determined	determined	determined	determined
2215 2232	not detected	not detected	not detected	not detected	not detected 0.198	not detected	not detected	not detected
2232	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
2250	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2255	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2265								
2272								
2287	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
2289 2291	ND	ND	ND	ND	ND	ND	ND	ND
2293								
2297	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2300	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2301					0.211			
2310	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2311 2320	Not Detected <0.1	Not Detected <0.1	Not Detected <0.1	Not Detected <0.1	<0.1 <0.1	Not Detected <0.1	Not Detected <0.1	Not Detected <0.1
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
2350	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2352								
2357								
2358 2363	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2365 2365	not detected <0.1	not detected <0.1	not detected <0.1	not detected <0.1	not detected <0.1	not detected <0.1	not detected <0.1	not detected <0.1
2370	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2373	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2375								
2378	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2
2379	Not detected	Not detected	Not detected	Not detected	0.0986	Not detected	0.0876	Not detected
2380 2382	<0.1 <0.100	<0.1 <0.100	<0.1 <0.100	<0.1 <0.100	<0.1 <0.100	<0.1 <0.100	<0.1 <0.100	<0.1 <0.100
2386	< 0.100	< 0.100	< 0.100	< 0.100	< 0.100	< 0.1	< 0.1	< 0.100
2429								
2449								
2459	ND	ND	ND					
2482	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2489 2532	ND Not Detected	ND Not Detected	ND Not Detected	ND Not Detected	ND Not Detected	ND Not Detected	ND Not Detected	Not Detected
2573	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
2638	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2643	not detected	not dotacted	not dotacted	not detected	not detected	not dotacted	not dotacted	not detected
2644 2649	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
2737								
2743		0.02570					0.04596	
2768	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d
2826 2870	Not detected	Not detected	Not detected	Not detected	Not detected 0.65 C fr 1.034	Not detected	Not detected	Not detected
2070	not		not	not	not	not	not	not
2892	determined	0.10	determined	determined	determined	determined	determined	determined
2912								
2948								
2959								
3003 3100	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
3116						not detected		
3154	not detected	not detected	not detected	not detected	not detected	not detected	not detected	
3172	< 0.025	< 0.025	< 0.025	< 0.025	< 0.025	< 0.025	< 0.025	< 0.025
3190	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
3222 3228	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
3228 3230	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
0200								

lab	245-TCT	PentaCT	12-DCB	13-DCB	14-DCB	123-TCB	135-TCB	1235-TeCB
3237								88.16
	not							
3243	determined							
3250								

APPENDIX 3 Analytical details

lab	ISO/IEC17025 accredited	Sample	Sample intake	Release/extract solvent	Extraction Time	Extraction temperature
623	Yes	Further cut	1	DCM suprasolve	30	33
840	Yes	Further cut	0.5	dicloromethane	30	room temperature
841	Yes	Further cut	0.5 grams	solvent DCM	30 minutes	room temperature
2115	No	Further cut	0.5 g	DCM	30 min	25 °C
2129	Yes	Used as received	0,5g	DCM	30min	Room temperature
2131	Yes	Used as received	1g	Dichloromethane	30 Minutes	Ambient
2159	Yes	Further cut	1 g	DCM	30 minutes	25 °C
2215	Yes	Further cut	2g	dichloromethane	30min	
2232	Yes	Further cut	1g	Dichloromethane	30	Room Temperature
2247	Yes	Further cut	0.5gm	DCM	30±2min	25±2
2250	Yes	Further cut	0,5; 0,1	DCM	30	roomtemperature Roomtemperature
2255	Yes	Used as received	1.0	DCM	30	(25-27)
2265	Yes	Further cut	1g	Dichloromethane	30 min	25°C
2272	Yes	Used as received	2g	Dichloromethane	30min	room temperature laboratory ambient
2287	No	Used as received	0.5g	dichloromethane	30 min	temperature
2289	Yes	Further cut	2.0g	Dichloromethanol	30mins	25 _i æ
2291	Yes	Further cut	0.2g	Dichloromethane	30min	25 temperature
2293						
2297	Yes	Further cut	1gram	Dichloromethane	30	room temperature
2300	Yes	Further cut	2 grams.	Dichloromethane.	30 minutes.	35-40°C
2301	No	Further cut	1 gram	Dichloromethane	30 min	room temperature Room temperature
2310	Yes	Further cut	1	DCM	30	at 22°C
2311	Yes	Further cut	1	Dichloromethane	30	Room temp.
2320	Yes	Further cut	1.0g 2.0 GM	Dichloromethane	30min	Room Temperature ROOM
2326	Yes	Further cut		DICHLOROMETHANE	30 MIN	TEMPERATURE
2330	Yes	Further cut	1	Dichloromethane	30 minutes	Room Temperature
2350	Yes	Further cut	2 g	Methylene chloride	30 min	room temperature
2352 2357	Yes 	Further cut	1g	Dichloromethane	30min	Room temperature
2358	Yes	Further cut	2.0	DCM	30	ambient
2363	Yes	Further cut	3g	DCM	30mins	room temperature
2365	Yes	Further cut	1.0g	Dichloromethane	30min	30°C Room temp (about
2370	Yes	Further cut	1.0058 grams	Dichloromethane	30 minutes	32°C)
2373	Yes	Further cut	2g	dichloromethane	30 minutes	room temperature
2375	Yes	Further cut	2 g	Dichloromethane dichloromethane	30 min 30	20-25 C laboratory ambient
2378	No	Used as received Further cut	2g			temperature
2379	Yes	Further cut	1 g	dichloromethane	30 minutes 30+/-1	25 C Laboratory temperature (22+/-2)
2380	Yes	Further cut	1.0 g	Dichloromethane (DCM)	minute	°C
2382	Yes	Further cut	1.0g	Dichloromethane	30	Room temperature
2386	Yes	Further cut	0.5 g - 2.0 g	Dichlormethane	30 min	25
2429	Yes	Further cut	0.5g	Dichloromethane	30min	25°C
2449	No	Further cut	1.0 gram	DCM	30 min	Room temperature
2459	Yes	Further cut	1.00 gm	Dichloromethane	60 min	60 °C
2482	Yes	Used as received	1.0	Dichloromethane	30	Room temperature
2489	Yes	Further cut	2.0070g	Dichloromethane	30 minutes	room temperature
2532	Yes	Further cut	1 gram	Dichloromethane (10 ml)	30 minutes	Room Temp
2573	Yes	Further cut	1g	dichloromethane	30minutes	room temperature Ambient
2590	Yes	Used as received	1g	DCM	30 min	temperature Ultrasonic bath,
2591	Yes	Further cut	2.0 grams	20 mL	30 min	room temperature
2638	No	Further cut	1 gm	Dichloro methane	30 min	room temperature
2643	Yes	Further cut	0.5 g	Dichloromethane	30	room temperature
2644	Yes	Further cut	1 g	dichloromethane (10 ml)	30 min	TA
2649						

	ISO/IEC17025		Sample		Extraction	Extraction
lab	accredited	Sample	intake	Release/extract solvent	Time	temperature
2678	No	Used as received	1 gram	Dichloromethane	30 min	room temperature
2737	Yes	Further cut	1g	Dichloromethane	30min	30°C
2743	Yes	Further cut	2 grams	Dichloromethane	30 min	25 C
2768	Yes	Used as received	0.5 gram	Dichloromethane	1 hour	25°C +_ 2
2826	Yes	Further cut	0.5 gram	Dichloromethane	30	Room temperature Laboratory ambient
2870	Yes	Further cut	2 gm	Dichloromethane	30 minutes	temperature
2892	Yes	Further cut	0.5092 gram	Dichloromethane (DCM)	30 minutes	Room temperature
2912	Yes	Used as received	1	Dichloromethane	30	Room Temperature
2948			1grams	Dichloromethane	30	Room temperature
2959						
3003	Yes	Used as received	5 grm	Dichloromethane	30 min	40
3100	Yes	Further cut	0.5g	Dichloromethane	30min	36°C±2°C
3116	Yes	Used as received	1 gram	Dichloromethane	30 minutes	Room temperature uncontrolled (RT -
3154	Yes	Further cut	1 g	Dichloromethane	30 min	40 °C)
3172						
3190	Yes	Further cut	1g	Dichloromethane	30	room temperature
3222	Yes	Further cut	1 g	Dichloromethane	60	ambient laboratory ambient
3228	Yes	Further cut	1	dichloromethane	30	temperature(22.2°C)
3230						
3237	Yes	Further cut	0,5	diklorometan	30	21
3243	Yes	Further cut	1 g	Dichlormethane	0,5 h	20 °C
3250	Yes	Used as received	1g	Dichloromethane	30min	room temperature

APPENDIX 4

Number of participants per country

- 4 labs in BANGLADESH
- 2 labs in CAMBODIA
- 7 labs in GERMANY
- 1 lab in GUATEMALA
- 3 labs in HONG KONG
- 7 labs in INDIA
- 2 labs in INDONESIA
- 7 labs in ITALY
- 1 lab in JAPAN
- 3 labs in KOREA, Republic of
- 1 lab in MAURITIUS
- 19 labs in P.R. of CHINA
- 5 labs in PAKISTAN
- 1 lab in SINGAPORE
- 1 lab in SPAIN
- 1 lab in SRI LANKA
- 1 lab in SWITZERLAND
- 1 lab inTAIWAN
- 1 lab in THAILAND
- 1 lab inTUNISIA
- 3 labs in TURKEY
- 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 D(0.01) = outlier in Grubbs' outlier test D(0.05) = straggler in Grubbs' outlier test D(0.05) = outlier in Double Grubbs' outlier test D(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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