# Results of Proficiency Test Chlorinated Organic Compounds (COC) in Textile November 2021

**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 Toluenes 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 in November 2021 on request of many participants.

In this new interlaboratory study 26 laboratories in 13 different 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 textile sample of 5 grams labelled #21900.
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 pink polyester fabric positive on Chlorinated Organic Compounds obtained from a third party was selected. The batch was cut into small pieces and after homogenization 40 small plastic bags were filled with approximately 5 grams each labelled #21900.
The homogeneity of the subsamples was checked by determination of 2-Chlorotoluene in accordance with EN17137 on 8 stratified randomly selected subsamples.

| | 2-Chlorotoluene in mg/kg |
|---|---|
| sample #21900-1 | 1.71 |
| sample #21900-2 | 1.67 |
| sample #21900-3 | 1.74 |
| sample #21900-4 | 1.71 |
| sample #21900-5 | 1.64 |
| sample #21900-6 | 1.72 |
| sample #21900-7 | 1.87 |
| sample #21900-8 | 1.60 |

Table 1: homogeneity test results of subsamples #21900

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

| | 2-Chlorotoluene in mg/kg |
|---|---|
| r (observed) | 0.22 |
| reference method | Horwitz |
| 0.3 x R (reference method) | 0.21 |

Table 2: evaluation of the repeatability of subsamples #21900

The calculated repeatability is in agreement with 0.3 times the estimated reproducibility calculated with the Horwitz equation. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one textile sample labelled #21900 was sent on October 6, 2021.

### 2.5 ANALYZES

The participants were requested to determine on the sample the concentrations of eleven individual Chlorotoluenes and eleven individual Chlorobenzenes.
It was also requested to report if the laboratory was accredited to determine the requested 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 to report as much significant figures as possible. It was also requested not to report “less than” test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluation.

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

## 3 RESULTS

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

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

During the execution of this proficiency test no problems were encountered with the dispatch of the samples. Three participants reported the test results after the final reporting date and all other participants reported in time. Not all participants were able to report all components requested.
In total 26 participants reported 93 numerical test results. Observed were 5 outlying test results, which is 5.4%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

All data sets proved to have a normal Gaussian distribution.

### 4.1 EVALUATION PER COMPONENT

In this section the reported test results are discussed per component. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables in appendix 1 together with the original data. 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 was 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 may be problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the estimated reproducibility calculated with the Horwitz equation or with the requirements of EN17137:18.

1,4-Dichlorobenzene: 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, but not with the requirements of EN17137:18.

1,2,4,5-Tetrachlorobenzene: This determination may be problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the estimated reproducibility calculated with the Horwitz equation or with the requirements of EN17137:18.

Hexachlorobenzene: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers in agreement with the estimated reproducibility calculated with the Horwitz equation and with the requirements of EN17137:18.

The concentrations of the other Chlorotoluenes and Chlorobenzenes were near or below the detection limits. Therefore, no z-scores are calculated. See appendix 2 for the reported test results.

### 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the estimated target reproducibilities and the reproducibilities as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility estimated with the Horwitz equation are compared in the next table.

| Component | unit | n | average | 2.8 * sd | R(target) |
|---|---|---|---|---|---|
| 2-Chlorotoluene | mg/kg | 23 | 5.93 | 3.66 | 2.03 |
| 1,4-Dichlorobenzene | mg/kg | 22 | 0.173 | 0.082 | 0.101 |
| 1,2,4,5-Tetrachlorobenzene | mg/kg | 23 | 57.6 | 30.3 | 14.0 |
| Hexachlorobenzene | mg/kg | 20 | 0.410 | 0.137 | 0.210 |

Table 3: reproducibilities of components on sample #21900

Without further statistical calculations, the group of participating laboratories may have difficulties with the analyzes of 2-Chlorotoluene and 1,2,4,5-Tetrachlorobenzene. See also the discussion in paragraphs 4.1 and 5.

### 4.3 OVERVIEW OF THE PROFICIENCY TEST OF NOVEMBER 2021

| | November 2021 |
|---|---|
| Number of reporting laboratories | 26 |
| Number of test results | 93 |
| Number of statistical outliers | 5 |
| Percentage of statistical outliers | 5.4% |

Table 4: overview of the proficiency test

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

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

| Component | November 2021 |
|---|---|
| 2-Chlorotoluene | 22% |
| 1,4-Dichlorobenzene | 17% |
| 1,2,4,5-Tetrachlorobenzene | 19% |
| Hexachlorobenzene | 12% |

Table 5: Observed uncertainties in PT of 2021

### 4.4 EVALUATION OF ANALYTICAL DETAILS

The reported analytical details from the participants are listed in appendix 3. The following can be summarized:

- About 80% of the reporting participants reported to be accredited for the determination of Chlorinated Organic Compounds in Textile.
- The sample intake was mostly about 1 or 2 grams, 46% and 33% of the reporting participants respectively.
- Prior to analysis the sample was further cut by about 50% of the participants and the other participants reported to use the sample as received.
- Almost all reporting participants mentioned to have used Dichloromethane as extraction solvent and carried out the extraction at room temperature for 30 minutes.

It appeared that the effect of the reported analytical details on the determination of Chlorotoluenes and Chlorobenzenes is small and not statistically significant.

## 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.

All reporting participants would have rejected the sample for all Ecolabel Classes based on the sum of Chlorotoluenes and Chlorobenzenes is more than 1.0 mg/kg (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: Ecolabelling Standards and Requirements for Textiles in EU

## 6 CONCLUSION

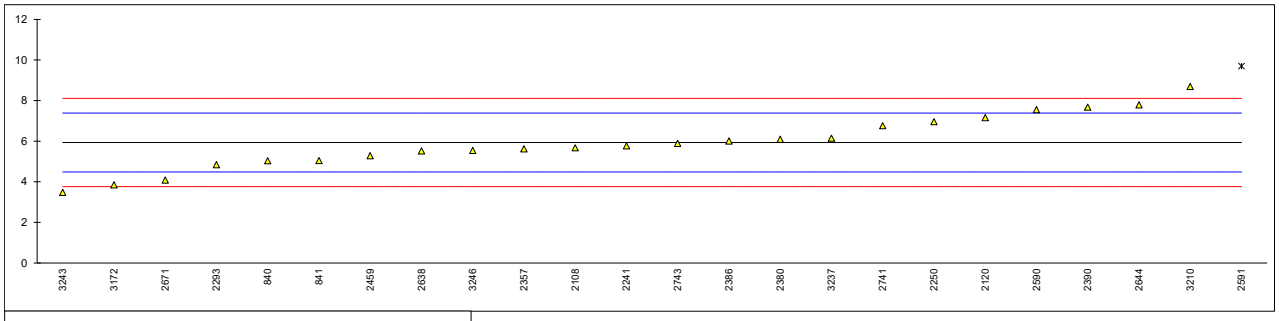
Almost all participants were able to detect 2-Chlorotoluene, 1,4-Dichlorobenzene, 1,2,4,5-Tetrachlorobenzene and Hexachlorobenzene correctly in the PT sample.
Each laboratory should evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

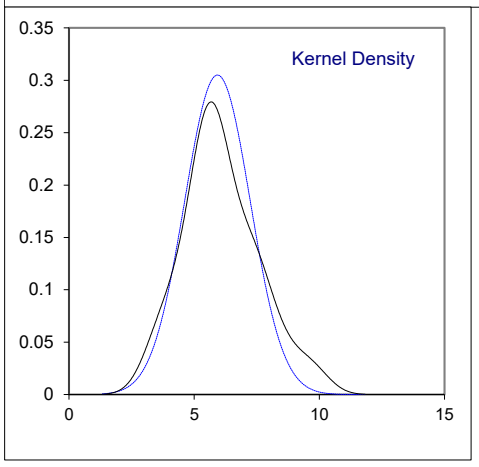
**APPENDIX 1**

Determination of 2-Chlorotoluene (CAS No. 95-49-8) on sample #21900; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 840 | EN17137 | 5.03 | | -1.24 | |
| 841 | EN17137 | 5.04 | | -1.23 | |
| 2108 | EN17137 | 5.67 | | -0.36 | |
| 2120 | EN17137 | 7.16 | | 1.69 | |
| 2241 | EN17137 | 5.763 | | -0.23 | |
| 2250 | EN17137 | 6.957 | | 1.41 | |
| 2293 | EN17137 | 4.8359 | | -1.51 | |
| 2357 | EN17137 | 5.62 | | -0.43 | |
| 2380 | EN17137 | 6.093 | | 0.22 | |
| 2386 | EN17137 | 6.005 | | 0.10 | |
| 2390 | EN17137 | 7.672 | | 2.40 | |
| 2459 | EN17137 | 5.28 | | -0.90 | |
| 2590 | EN17137 | 7.552 | | 2.23 | |
| 2591 | EN17137 | 9.7 | R(0.05) | 5.19 | |
| 2638 | | 5.52 | | -0.57 | |
| 2644 | EN17137 | 7.79 | | 2.56 | |
| 2671 | EN17137 | 4.08 | C | -2.55 | First reported ND |
| 2741 | EN17137 | 6.759 | | 1.14 | |
| 2743 | EN17137 | 5.885699 | C | -0.06 | First reported 13.350709 |
| 3116 | | ----- | | ----- | |
| 3172 | EN17137 | 3.8444 | | -2.87 | |
| 3192 | | ----- | | ----- | |
| 3210 | EN17137 | 8.6901 | | 3.80 | |
| 3237 | EN17137 | 6.14 | | 0.29 | |
| 3243 | EN17137 | 3.47 | | -3.39 | |
| 3246 | | 5.54 | | -0.54 | |

| | | | |
|---|---|---|---|
| | normality | OK | |
| | n | 23 | |
| | outliers | 1 | |
| | mean (n) | 5.9303 | |
| | st.dev. (n) | 1.30791 | RSD = 22% |
| | R(calc.) | 3.6621 | |
| | st.dev.(Horwitz) | 0.72583 | |
| | R(Horwitz) | 2.0323 | |
| Compare | | | |
| | R(EN17137:18) | 1.9478 | |

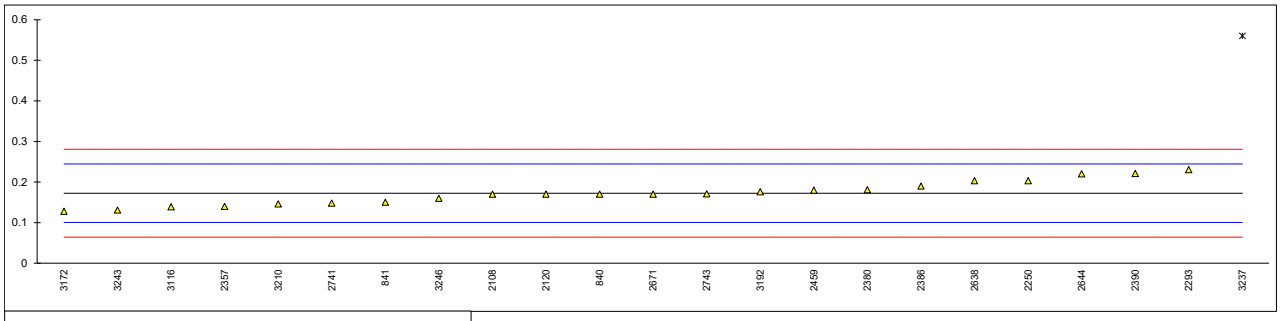


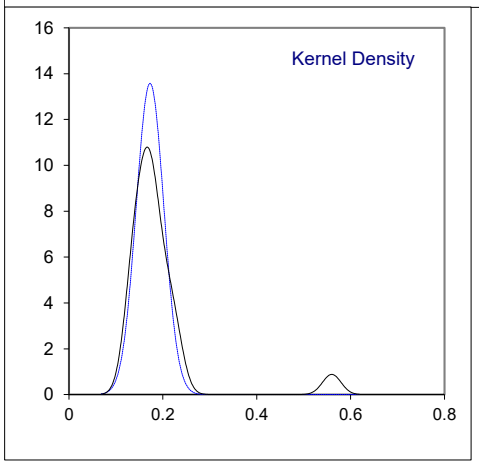



Determination of 1,4-Dichlorobenzene (CAS No. 106-46-7) on sample #21900; results in mg/kg

| lab | method | value | mark | z(targ) | Remarks |
|---|---|---|---|---|---|
| 840 | EN17137 | 0.17 | | -0.07 | |
| 841 | EN17137 | 0.15 | | -0.63 | |
| 2108 | EN17137 | 0.17 | | -0.07 | |
| 2120 | EN17137 | 0.17 | | -0.07 | |
| 2241 | EN17137 | <0.5 | | ----- | |
| 2250 | EN17137 | 0.203 | | 0.85 | |
| 2293 | EN17137 | 0.2304 | | 1.61 | |
| 2357 | EN17137 | 0.14 | | -0.91 | |
| 2380 | EN17137 | 0.181 | | 0.23 | |
| 2386 | EN17137 | 0.190 | | 0.48 | |
| 2390 | EN17137 | 0.221 | | 1.35 | |
| 2459 | EN17137 | 0.18 | | 0.21 | |
| 2590 | | ----- | | ----- | |
| 2591 | EN17137 | n.d. | | ----- | |
| 2638 | | 0.203 | C | 0.85 | First reported Not detected |
| 2644 | EN17137 | 0.22 | | 1.32 | |
| 2671 | EN17137 | 0.17 | | -0.07 | |
| 2741 | EN17137 | 0.148 | | -0.68 | |
| 2743 | EN17137 | 0.170619 | | -0.06 | |
| 3116 | DIN54232 | 0.1390 | | -0.93 | |
| 3172 | EN17137 | 0.12807 | | -1.24 | |
| 3192 | EN17137 | 0.176 | | 0.09 | |
| 3210 | EN17137 | 0.1461 | | -0.74 | |
| 3237 | EN17137 | 0.56 | R(0.01) | 10.77 | |
| 3243 | EN17137 | 0.131 | | -1.16 | |
| 3246 | | 0.16 | | -0.35 | |

| | | | |
|---|---|---|---|
| | normality | OK | |
| | n | 22 | |
| | outliers | 1 | |
| | mean (n) | 0.17260 | |
| | st.dev. (n) | 0.029384 | RSD = 17% |
| | R(calc.) | 0.08227 | |
| | st.dev.(Horwitz) | 0.035975 | |
| | R(Horwitz) | 0.10073 | |
| Compare | | | |
| | R(EN17137:18) | 0.04697 | |

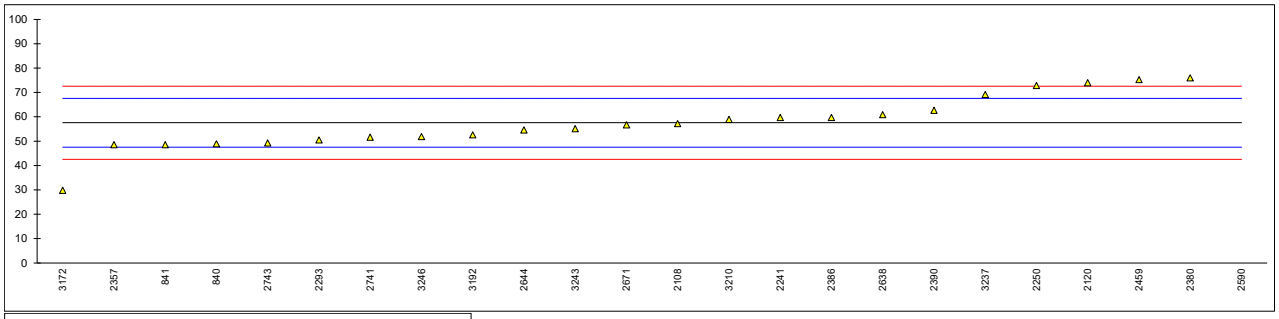


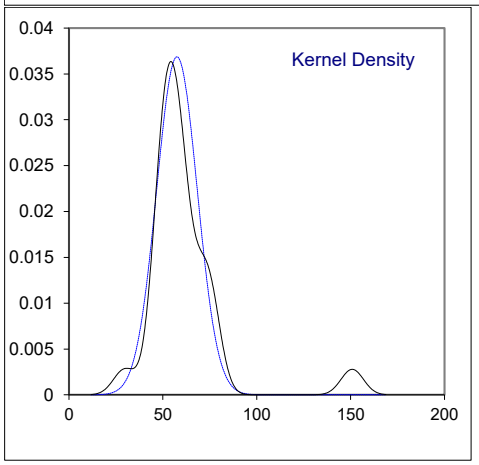



Determination of 1,2,4,5-Tetrachlorobenzene (CAS No. 95-94-3) on sample #21900; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 840 | EN17137 | 48.90 | | -1.73 | |
| 841 | EN17137 | 48.53 | | -1.81 | |
| 2108 | EN17137 | 57.19 | | -0.08 | |
| 2120 | EN17137 | 74 | | 3.28 | |
| 2241 | EN17137 | 59.699 | | 0.43 | |
| 2250 | EN17137 | 72.8 | C | 3.04 | First reported 80.7 |
| 2293 | EN17137 | 50.440 | | -1.42 | |
| 2357 | EN17137 | 48.52 | | -1.81 | |
| 2380 | EN17137 | 75.968 | | 3.68 | |
| 2386 | EN17137 | 59.728 | | 0.43 | |
| 2390 | EN17137 | 62.700 | | 1.03 | |
| 2459 | EN17137 | 75.22 | | 3.53 | |
| 2590 | EN17137 | 150.897 | C,R(0.01) | 18.65 | First reported 86.873 |
| 2591 | EN17137 | n.d. | | ----- | Possibly a false negative test result? |
| 2638 | | 60.86 | C | 0.66 | First reported 58.1 |
| 2644 | EN17137 | 54.58 | C | -0.60 | First reported 27.29 |
| 2671 | EN17137 | 56.7 | C | -0.17 | First reported ND |
| 2741 | EN17137 | 51.640 | | -1.18 | |
| 2743 | EN17137 | 49.254945 | C | -1.66 | First reported Not detected |
| 3116 | | ----- | | ----- | |
| 3172 | EN17137 | 29.777 | | -5.55 | |
| 3192 | EN17137 | 52.589 | | -1.00 | |
| 3210 | EN17137 | 58.9041 | | 0.27 | |
| 3237 | EN17137 | 69.16 | | 2.32 | |
| 3243 | EN17137 | 55.1 | | -0.49 | |
| 3246 | | 51.85 | | -1.14 | |

| | | | |
|---|---|---|---|
| | normality | OK | |
| | n | 23 | |
| | outliers | 1 | |
| | mean (n) | 57.5700 | |
| | st.dev. (n) | 10.81708 | RSD = 19% |
| | R(calc.) | 30.2878 | |
| | st.dev.(Horwitz) | 5.00473 | |
| | R(Horwitz) | 14.0132 | |
| Compare | | | |
| | R(EN17137:18) | 21.7615 | |

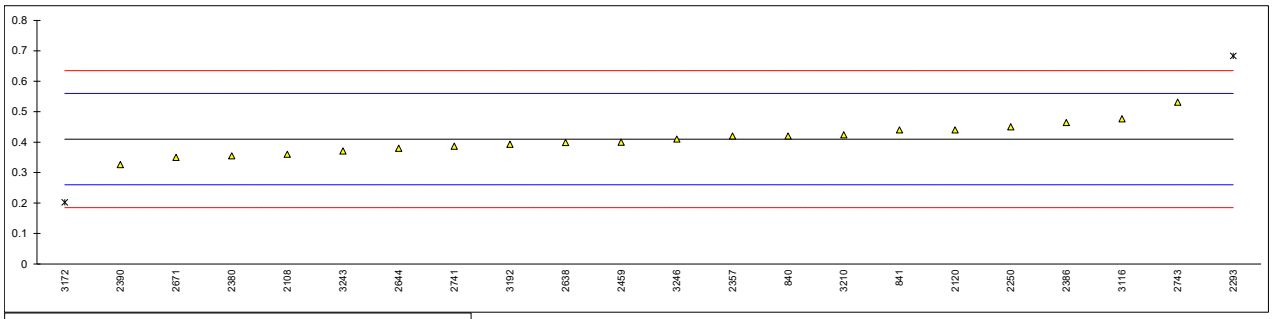


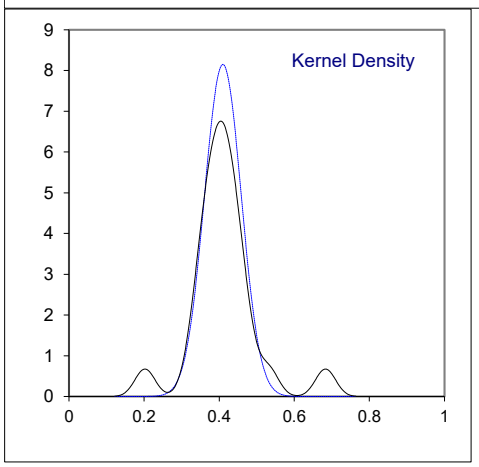



Determination of Hexachlorobenzene (CAS No. 118-74-1) on sample #21900; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 840 | EN17137 | 0.42 | | 0.14 | |
| 841 | EN17137 | 0.44 | | 0.40 | |
| 2108 | EN17137 | 0.36 | | -0.66 | |
| 2120 | EN17137 | 0.44 | | 0.40 | |
| 2241 | EN17137 | <0.5 | | ----- | |
| 2250 | EN17137 | 0.450 | | 0.54 | |
| 2293 | EN17137 | 0.6834 | R(0.01) | 3.65 | |
| 2357 | EN17137 | 0.42 | | 0.14 | |
| 2380 | EN17137 | 0.355 | | -0.73 | |
| 2386 | EN17137 | 0.464 | | 0.72 | |
| 2390 | EN17137 | 0.3261 | | -1.12 | |
| 2459 | EN17137 | 0.40 | | -0.13 | |
| 2590 | | ----- | | ----- | |
| 2591 | EN17137 | n.d. | | ----- | Possibly a false negative test result? |
| 2638 | | 0.399 | C | -0.14 | First reported Not detected |
| 2644 | EN17137 | 0.38 | | -0.40 | |
| 2671 | EN17137 | 0.35 | | -0.80 | |
| 2741 | EN17137 | 0.387 | | -0.30 | |
| 2743 | EN17137 | 0.531588 | | 1.62 | |
| 3116 | DIN54232 | 0.4766 | | 0.89 | |
| 3172 | EN17137 | 0.2022 | R(0.05) | -2.77 | |
| 3192 | EN17137 | 0.393 | | -0.22 | |
| 3210 | EN17137 | 0.4241 | | 0.19 | |
| 3237 | | ----- | | ----- | |
| 3243 | EN17137 | 0.371 | | -0.52 | |
| 3246 | | 0.41 | | 0.00 | |

| | | | |
|---|---|---|---|
| | normality | OK | |
| | n | 20 | |
| | outliers | 2 | |
| | mean (n) | 0.40987 | |
| | st.dev. (n) | 0.048967 | RSD = 12% |
| | R(calc.) | 0.13711 | |
| | st.dev.(Horwitz) | 0.075001 | |
| | R(Horwitz) | 0.21000 | |
| Compare | | | |
| | R(EN17137:18) | 0.21002 | |



## 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
123-TCB = 1,2,3-Trichlorobenzene CAS No. 87–61–6
124-TCB = 1,2,4-Trichlorobenzene CAS No. 120–82–1
135-TCB = 1,3,5-Trichlorobenzene CAS No. 108–70–3
1234-TeCB = 1,2,3,4-Tetrachlorobenzene CAS No. 634–66–2
1235-TeCB = 1,2,3,5-Tetrachlorobenzene CAS No. 634–90–2
PentaCB = Pentachlorobenzene CAS No. 608–93–5

Determination of other Chlorinated Organic Compounds (COC) on sample #21900; in mg/kg

| lab | 3-CT | 4-CT | 23-DCT | 24-DCT | 25-DCT | 26-DCT | 34-DCT | 236-TCT | 245-TCT |
|---|---|---|---|---|---|---|---|---|---|
| 840 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 |
| 841 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 |
| 2108 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2120 | <0,10 | <0,10 | <0,10 | 0.16 | <0,10 | <0,10 | <0,10 | <0,10 | <0,10 |
| 2241 | <0.5 | <0.5 | <0.5 | <0.5 | <0.5 | <0.5 | <0.5 | <0.5 | <0.5 |
| 2250 | ----- | ----- | ----- | 0.110 | ----- | ----- | ----- | ----- | ----- |
| 2293 | not detected | not detected | not detected | not detected | not detected | not detected | not detected | not detected | not detected |
| 2357 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2380 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2386 | <0,05 | <0,05 | <0,05 | 0.081 | <0,05 | <0,05 | <0,05 | <0,05 | <0,05 |
| 2390 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2459 | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| 2590 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2591 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2638 | Not detected | Not detected | Not detected | Not detected | Not detected | Not detected | Not detected | Not detected | Not detected |
| 2644 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2671 | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| 2741 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 |
| 2743 | Not detected | 0.545371 | Not detected | 0.143545 | Not detected | Not detected | Not detected | Not detected | Not detected |
| 3116 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3172 | < 0.05 | < 0.05 | < 0.05 | < 0.05 | < 0.05 | < 0.05 | < 0.05 | < 0.05 | < 0.05 |
| 3192 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3210 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 |
| 3237 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3243 | ----- | ----- | ----- | 0.063 | ----- | ----- | ----- | ----- | ----- |
| 3246 | not detected | not detected | not detected | not detected | not detected | not detected | not detected | not detected | not detected |

Determination of other Chlorinated Organic Compounds (COC) on sample #21900; in mg/kg

| lab | PentaCT | 12-DCB | 13-DCB | 123-TCB | 124-TCB | 135-TCB | 1234-TeCB | 1235-TeCB | PentaCB |
|---|---|---|---|---|---|---|---|---|---|
| 840 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 |
| 841 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 |
| 2108 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2120 | <0,10 | <0,10 | <0,10 | <0,10 | 0.13 | <0,10 | <0,10 | <0,10 | <0,10 |
| 2241 | <0.5 | <0.5 | <0.5 | <0.5 | <0.5 | <0.5 | <0.5 | <0.5 | <0.5 |
| 2250 | ----- | ----- | ----- | ----- | 0.127 | 0.105 | ----- | ----- | ----- |
| 2293 | not detected | not detected | not detected | not detected | not detected | not detected | not detected | not detected | not detected |
| 2357 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2380 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2386 | <0,05 | <0,05 | <0,05 | 0.050 | 0.107 | <0,05 | 0.077 | <0,05 | 0.048 |
| 2390 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2459 | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| 2590 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2591 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. | 71.0 | n.d. |
| 2638 | Not detected | Not detected | Not detected | Not detected | Not detected | Not detected | Not detected | Not detected | Not detected |
| 2644 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 2671 | ND | ND | ND | ND | ND | ND | ND | ND | ND |
| 2741 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 |
| 2743 | Not detected | Not detected | Not detected | Not detected | 0.174097 | 0.115960 | Not detected | *Not detectable* | Not detected |
| 3116 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 3172 | < 0.05 | < 0.05 | < 0.05 | < 0.05 | 0.0581 | 0.0567 | < 0.05 | < 0.05 | < 0.05 |
| 3192 | ----- | < 0,05 | < 0,05 | 0.050 | 0.101 | 0.071 | 0.064 | < 0,05 | 0.052 |
| 3210 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 | <0.1 |
| 3237 | ----- | ----- | ----- | ----- | 0.49 | 0.45 | ----- | ----- | ----- |
| 3243 | ----- | ----- | ----- | ----- | 0.085 | 0.079 | 0.065 | ----- | ----- |
| 3246 | not detected | not detected | not detected | not detected | not detected | not detected | not detected | not detected | not detected |

## APPENDIX 3 Analytical details

| lab | ISO/IEC17025 accredited | Sample | Sample intake | Release/extract solvent | Extraction Time | Extraction temperature |
|---|---|---|---|---|---|---|
| 840 | Yes | Further cut | 0.5075 | DCM | 30 | Room temperature |
| 841 | Yes | Used as received | 1 grams | Dichloromethane | 30 minutes | room temperature |
| 2108 | Yes | Used as received | 2 g | Dichloromethane | 30 min | room temperature |
| 2120 | No | Used as received | 1 g | Dichloromethane | 30 min | Ambient |
| 2241 | Yes | Used as received | 1 grams | dichloromethane | 30 minutes | room temperature |
| 2250 | Yes | Used as received | 1 | Dichloromethane | 30 | 20 |
| 2293 | Yes | Used as received | 2 grams | Dichloromethane | 30 minutes | Room temperature |
| 2357 | --- | --- | | | | |
| 2380 | Yes | Further cut | 1.0 g | Dichloromethane | 30 minutes | Room temperature |
| 2386 | Yes | Further cut | 2,0 | Dichloromethane | 30 | 20 |
| 2390 | Yes | Further cut | 2.0 gram | Dichloromethane | 30 minutes | Room temperature |
| 2459 | No | Further cut | 2.0 grams | Dichloromethane | 30 minutes | Room temperature |
| 2590 | Yes | Used as received | 1g | DCM | 30 min | room temperature |
| 2591 | --- | --- | 2.0 | Dichloromethane | 30 min | |
| 2638 | No | Further cut | 1 gm | Dichloromethane | 30 minutes | Room temperature |
| 2644 | Yes | Used as received | 1 g | dichloromethane | 30 minutes | 25°C |
| 2671 | Yes | Used as received | 1 gram | DCM | 30 mins | Room Temperature |
| 2741 | Yes | Further cut | 0.5 | Dichloromethane | 30 minutes | Room temperature |
| 2743 | Yes | Used as received | 2g | DCM | 30min | Room temp. |
| 3116 | Yes | Used as received | 1 gram | Dichloromethane | 30 minutes | room temperature |
| 3172 | Yes | --- | | | | |
| 3192 | Yes | Further cut | 0,5 g | Dichloromethane | 30 minutes | room temperature |
| 3210 | No | Further cut | 2grammes | Dichloromethane | 30 minutes | 40°C |
| 3237 | Yes | Used as received | 0,5 gr | DCM | 20 | Room temperature |
| 3243 | Yes | Further cut | 1.5 | Dichloromethane | 30 min | 20 °C |
| 3246 | Yes | Further cut | 1.00 | Dichloromethane | 30min | room temperature |

## APPENDIX 4

### Number of participants per country

1 lab in BANGLADESH
1 lab in FRANCE
5 labs in GERMANY
1 lab in GUATEMALA
1 lab in HONG KONG
1 lab in INDIA
4 labs in ITALY
2 labs in P.R. of CHINA
3 labs in PAKISTAN
1 lab in PORTUGAL
1 lab in SPAIN
1 lab in TURKEY
4 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 |

### Literature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