

Institute for Interlaboratory Studies

> Results of Proficiency Test Chlorinated Phenols in Leather/Footwear April 2022

Organized by: Institute for Interlaboratory Studies Spijkenisse, the Netherlands

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Report: ii

iis22A06

July 2022

# CONTENTS

1		3
2	SET UP	3
2.1	QUALITY SYSTEM	3
2.2	PROTOCOL	3
2.3	CONFIDENTIALITY STATEMENT	4
2.4	SAMPLES	4
2.5	ANALYZES	5
3	RESULTS	5
3.1	STATISTICS	6
3.2	GRAPHICS	6
3.3	Z-SCORES	7
4	EVALUATION	7
4.1	EVALUATION PER COMPONENT	8
4.2	PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES	8
4.3	COMPARISON OF THE PROFICIENCY TEST OF APRIL 2022 WITH PREVIOUS PTS	9
4.4	EVALUATION OF THE ANALYTICAL DETAILS	9
5	DISCUSSION	10
6	CONCLUSION	10

## Appendices:

1.	Data, statistical and graphic results	11
2.	Other reported test results	15
3.	Analytical details	18
4.	Number of participants per country	19
5.	Abbreviations and literature	20

## 1 INTRODUCTION

Products containing Pentachlorophenol (PCP) may form highly toxic substances when they are incinerated. PCP is also a suspected carcinogen. Since the 1990's many countries have adopted environmental standards and requirements restricting the use of harmful chemicals in the production of textiles and leather consumer products. Laws and regulations impose some of these standards and requirements. In addition to mandatory environmental standards and requirements for leather, there are some Ecolabelling schemes imposing environmental requirements for textile and leather products on a voluntary basis. Well-known Ecolabelling organizations are OEKO-TEX® and Bluesign®.

Since 2016 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of Chlorinated Phenols in Leather/Footwear every year. During the annual proficiency testing program 2021/2022 it was decided to continue the proficiency test for the determination of Chlorinated Phenols in Leather/Footwear.

In this interlaboratory study 70 laboratories in 23 countries registered for participation, see appendix 4 for the number of participants per country. In this report the results of the Chlorinated Phenols in Leather/Footwear 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 leather sample containing some Chlorinated Phenols of 3 grams labelled #22585.

The participants were asked 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 a 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 green leather containing some Chlorinated Phenols was selected. The batch was cut into small pieces and after homogenization 100 small plastics bags were filled with approximately 3 grams each and labelled #22585.

The homogeneity of the subsamples was checked by determination of 2,4,6-Trichlorophenol in accordance with ISO17070 on 8 stratified randomly selected subsamples.

	2,4,6-Trichlorophenol in mg/kg
sample #22585-1	1.327
sample #22585-2	1.325
sample #22585-3	1.256
sample #22585-4	1.191
sample #22585-5	1.257
sample #22585-6	1.323
sample #22585-7	1.241
sample #22585-8	1.470

Table 1: homogeneity test result of subsamples #22585

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

	2,4,6-Trichlorophenol in mg/kg
r (observed)	0.236
reference method	iis memo 1601
0.3 x R (reference method)	0.383

Table 2: evaluation of the repeatability of subsamples #22585

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 of #22585 was sent on March 30, 2022.

## 2.5 ANALYZES

The participants were requested to determine Pentachlorophenol, 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 determination of Chlorinated Phenols in Leather/Footwear and to report some analytical details.

It was explicitly requested to treat the sample as if it was a routine sample, but not to age nor to dry the sample nor to determine volatile matter. The amount of sample was not sufficient to allow aging and/or determine the volatile matter content.

It was also requested to report the test results using the indicated units on the report form and not to round the results, but report as much significant figures as possible and 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) 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:

 $\begin{aligned} |z| &< 1 \quad \text{good} \\ 1 &< |z| &< 2 \quad \text{satisfactory} \\ 2 &< |z| &< 3 \quad \text{questionable} \\ 3 &< |z| \quad & \text{unsatisfactory} \end{aligned}$ 

## 4 EVALUATION

In this proficiency test some problems were encountered with the dispatch of the samples due to COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with another week. Twelve participants reported test results after the extended reporting date and two other participants did not report any test results. Not all participants were able to report all tests requested.

In total 68 participants reported 119 numerical test results. Observed were 5 outlying test results, which is 4.2%. In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

Both 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 methods are also in the tables in together with the original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 5.

Test methods ISO17070:15 and LFGB 82.02-8 mention precision data for Pentachlorophenol. In 2016 iis investigated the reproducibilities of the determination of Pentachlorophenol in textile over 18 determinations in iis PTs conducted from 2004 until 2014. It was observed that the reproducibility as mentioned in ISO17070 is very strict. Therefore, a new target reproducibility on base of the iis PTs was determined and described in iis memo 1601. Although iis memo 1601 is based on iis PTs of Pentachlorophenol in Textile it is decided to use the estimated iis target reproducibility also for the determination of Pentachlorophenol in Leather. Furthermore, it is decided to use the estimated iis target reproducibility for other Chlorinated Phenol components determined in Leather as well.

- <u>Pentachlorophenol</u>: The determination of this component was problematic for a number of laboratories. Five 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.
- <u>2,4,6-Trichlorophenol</u>: The determination of this component was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the target reproducibility derived from the reproducibilities observed in previous as derived from iis memo 1601.

The majority of the participants agreed on a concentration near or below the limit of detection for all other Chlorinated Phenols mentioned in paragraph 2.5. Therefore, no z-scores were calculated for these Chlorinated Phenols. The reported test results of these components 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 from reference methods are presented in the next table.

Component	unit	n	average	2.8 * sd	R(target)
Pentachlorinatedphenol	mg/kg	50	0.18	0.18	0.24
2,4,6-Trichlorophenol	mg/kg	64	0.90	0.75	0.93

 Table 3: reproducibility of tests on sample #22585

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

#### 4.3 COMPARISON OF THE PROFICIENCY TEST OF APRIL 2022 WITH PREVIOUS PTS

	April 2022	May 2021	May 2020	May 2019	April 2018
Number of reporting laboratories	68	70	65	73	72
Number of test results	119	70	125	205	127
Number of statistical outliers	5	1	2	4	4
Percentage of statistical outliers	4.2%	1.4%	1.6%	2.0%	3.1%

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 tests was compared, expressed as relative standard deviation (RSD) of the PTs, see next table.

	April 2022	May 2021	May 2020	2016 – 2019	Target *)
PCP	35%	31%	21%	26 - 41%	35 – 25%
2,3,4,5-TeCP	n.e.	n.e.	18%	n.e.	35 – 25%
2,3,5,6-TeCP	n.e.	n.e.	n.e.	26%	35 – 25%
2,4,6-TCP	30%	n.e.	n.e.	29 - 34%	35 – 25%

Table 5: development of the uncertainties over the years

\* Concentration range 0.5 – 13 mg/kg respectively

The uncertainties observed in this PT are in line with previous PTs.

#### 4.4 EVALUATION OF THE ANALYTICAL DETAILS

Test method ISO17070 is used by about 60% of the reporting participants and test method LFGB B82.02.8 is used by about 5% of the participants. Test methods ISO17070 and LFGB 82.02-8 describe a similar sample pathway to determine PCP. About 20% of the reporting participants mentioned to have used an in-house test method.

For this PT also some analytical details were requested, see appendix 3 for the reported details. Based on the answers given by the participants the following can be summarized:

- About 75% of the participants mentioned that they are accredited for the determination of the reported components.

- About 40% of the participants used the sample as received and about 60% did further cut or grind the sample.

- About 80% of the participants used a sample intake between 0.5 - 1 grams and about 10% used more than 1 grams as sample intake.

- About 60% of the participants used steam distillation as technique to release the Chlorinated Phenols and about 20% used KOH as extraction technique.

- About 20% of the participants used Ultrasonic extraction and about 50% used Mechanical Shaking as technique to extract the Chlorinated Phenols.

The calculated reproducibilities are in agreement with the estimated reproducibilities of the reference method, therefore no separate statistical analysis has been performed.

## 5 DISCUSSION

When the results of this proficiency test were compared to the OEKO-TEX® Leather Standard (see Table 6) it was noticed that not all participants would make identical decisions about the acceptability of the leather for Chlorophenols.

	Baby in mg/kg	Direct skin contact in mg/kg	No direct skin contact in mg/kg	Decoration material in mg/kg
Pentachlorophenol (PCP)	<0.3	<0.5	<0.5	<0.5
Tetrachlorophenols (TeCP), each isomer	<0.5	<0.5	<0.5	<0.5
Trichlorophenols (TrCP), each isomer	<0.5	<1.0	<1.0	<1.0

Table 6: Oeko-Tex® Leather Standard specific limits for Chlorophenols

For Pentachlorophenol almost all participants would have accepted the leather for all classes. However, five participants would have rejected the leather for the class of Baby. For Trichlorophenol seven participants would have accepted the leather for all classes, while twenty-one participants would have rejected the leather for all classes.

#### 6 CONCLUSION

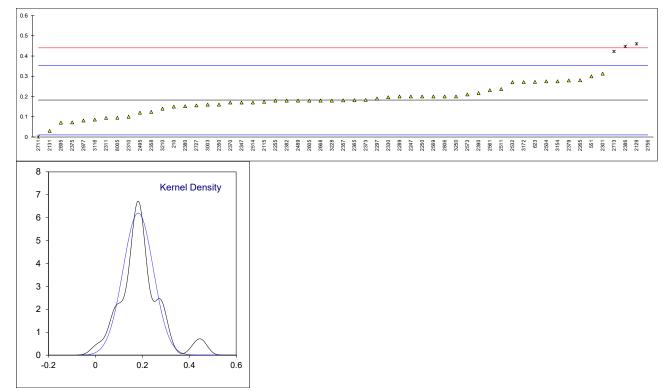
The majority of the participants has no problem with the determination of Pentachlorophenol and 2,4,6-Trichlorophenol in Leather/Footwear.

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.

Determination of Pentachlorophenol (PCP) on sample #22585; results in mg/kg

Ibb         method         value         mark         Z(tory)         remarks           210         ISO17070         0.15         -0.38         -0.38           551         ILFOB B82.02.8         0.2996         -1.38           551         ISO17070         0.460         R(0.05)         -2.3           2131         Inhouse         0.03         -1.77           2145         ISO17070         0.18         -0.03           2247         DIN50090         0.22         0.21           2258         ISO17070         0.19         0.09           2268         ISO17070         0.31281         C         1.52           2261         ISO17070         0.31281         C         1.52           2301         ISO17070         0.31281         C         1.52           2311         ISO17070         0.16         -0.98           2335         ISO17070         0.181         -0.02           2336         ISO17070         0.181         -0.02           2337         ISO17070         0.181         -0.02           2338         ISO17070         0.182         -0.33           2339         ISO17070         0.182				,		2585; results in mg/kg
551       LFGB B22.02.8       0.2996       1.36         281       ISO17070       0.272       1.04         2115       ISO17070       0.174       -0.10         2131       ISO17070       0.272       1.04         2143       ISO17070       0.21       -0.10         2247       DIN5009       0.20       0.21         2255       ISO17070       0.18       -0.03         2261       ISO17070       0.28       1.14         2281       ISO17070       0.28       1.14         2281       ISO17070       0.2       0.21         2283       ISO17070       0.16       -0.03         2301       ISO17070       0.16       -0.14         2331       ISO17070       0.16       -0.21         2347       ISO17070       0.16       -0.23         2347       ISO17070       0.181       -0.02         2358       ISO17070       0.181       -0.02         2358       ISO17070       0.170       -0.14         2351       ISO17070       0.181       -0.03         2364       IN house       0.277       R(0.05)       -0.12         2379				mark		remarks
623       SO17070       0.272       1.04         2115       SO17070       0.460       R(0.05)       3.23         2131       In hause       0.03      77         2145       ISO17070       0.164      77         2145       ISO17070       0.16      03         2250       Inhouse       0.21         2251       ISO17070       0.22       0.21         2252       ISO17070       0.23       0.21         2253       ISO17070       0.24       0.21         2254       ISO17070       0.19       0.09         2251       ISO17070       0.124       -0.04         2253       ISO17070       0.17       -0.14         2311       ISO17070       0.17       -0.14         2331       ISO17070       0.17       -0.14         2341       ISO17070       0.181       -0.02         2355       ISO17070       0.124       -0.68         2354       ISO17070       0.127       -1.13         2355       ISO17070       0.172       -1.28         2354       ISO17070       0.172       -0.40         2355       ISO17070       <						
2115       ISO17070       0.460       R(0.05)       3.23         2131       In house       0.33       -1.77         2135       ISO17070       0.20       0.21         2135       ISO17070       0.18       -0.61         2255       ISO17070       0.18       -0.63         2256       ISO17070       0.28       1.14         2280       ISO17070       0.29       0.21         2281       ISO17070       0.19       C       1.58         2301       ISO17070       0.19       C       1.59         2311       ISO17070       0.19       -       -         2324       ISO17070       0.16       -       0.16         2331       ISO17070       0.16       -       0.26         2333       ISO17070       0.181       -       0.02         2334       ISO17070       0.183       0.01         2335       ISO17070       0.183       0.01         2336       ISO17070       0.183       0.01         2337       ISO17070       0.180       -       0.33         2340       ISO17070       0.181       -       0.35						
213       In Nouse       0.43       -1.77         2145       In Nouse       0.33       -1.77         2145       IN Nouse       0.20       0.21         2251       Di Nouse       0.20       0.21         2252       Di Nouse       0.20       0.21         2255       Di Nouse       0.22       0.21         2265       IN Nouse       0.22       0.21         2267       ISO17070       0.22       0.21         2283       ISO17070       0.19       0.09         2284       ISO17070       0.19       0.09         2311       ISO17070       0.19       0.09         23311       ISO17070       0.196       0.14         2335       ISO17070       0.181       -0.62         2356       ISO17070       0.181       -0.62         2357       ISO17070       0.182       0.00         2366       ISO17070       0.182       -0.35         2370       ISO17070       0.182       -0.35         2386       ISO17070       0.182       -0.35         2387       ISO17070       0.182       -0.35         2380       ISO17070 <t< td=""><td></td><td></td><td></td><td></td><td></td><td></td></t<>						
2131       in house       0.03       -1.77         2246       ISO17070       not detected          2247       ISO1000       0.20       0.21         2255       In house       0.20       0.21         2256       Is house       0.20       0.21         2255       Is house       0.20       0.21         2256       Is house       0.20       0.21         2257       ISO17070       0.18       -0.09         2301       ISO17070       0.31281       C       1.52         2301       ISO17070       0.196       0.16         2301       ISO17070       0.17       -0.14         2301       ISO17070       0.181       -0.06         2311       ISO17070       0.17       -0.14         2345       ISO17070       0.17       -0.14         2347       ISO17070       0.170       -0.14         2348       ISO17070       0.170       -0.14         2347       ISO17070       0.172       -1.28         2348       ISO17070       0.172       -1.31         2349       ISO17070       0.172       -1.32         2348						
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2247     DIN50009     0.20     0.21       2250     In Nouse     0.20     0.21       2251     ISO17070     0.18     -0.03       2283     ISO17070     0.2     0.21       2284     ISO17070     0.19     0.09       2285     DIN50009     0.28     1.14       2280     ISO17070     0.19     0.03       2281     ISO17070     0.191     0.193       2311     ISO17070     0.191     -0.16       23331     ISO17070     0.116     -0.26       2354     ISO17070     0.16     -0.26       2355     ISO17070     0.181     -0.02       2356     ISO17070     0.181     -0.02       2358     ISO17070     0.183     0.01       2351     ISO17070     0.170     -1.43       2351     ISO17070     0.162     -0.33       2351     ISO17070     0.163     -0.01       2351     ISO17070     0.162     -0.33       2361     ISO17070     0.162     -0.33       2371     ISO17070     0.162     -0.33       2380     ISO17070     0.162     -0.33       2380     ISO17070     0.162       2391 <td< td=""><td></td><td></td><td></td><td></td><td></td><td></td></td<>						
2250         IN house         0.20         0.21           2256         ISO17070         0.18         -0.03           2289         ISO17070         0.28         1.14           2237         ISO17070         0.19         0.21           2237         ISO17070         0.19         0.21           2237         ISO17070         0.19         0.122           233         ISO17070         0.194         -1.03           2341         ISO17070         0.196         -0.14           2353         ISO17070         0.181         -0.02           2354         ISO17070         0.181         -0.02           2355         ISO17070         0.182         0.00           2356         ISO17070         0.173         -0.14           2350         ISO17070         0.173         -0.14           2357         ISO17070         0.173         -0.14           2358         ISO17070         0.173         -0.14           2359         ISO17070         0.181         -0.03           2361         In house         0.217         0.40           2451         ISO17070         0.182         -0.35           23						
2255       ISO17070       0.18       -0.03         2286       DISO17070       0.22       0.21         2287       ISO17070       0.19       0.09         2281       ISO17070       0.121       -0.36         2281       ISO17070       0.1221       C         2301       ISO17070       0.0441       -1.03         2311       ISO17070       0.1641       -1.04         2347       ISO17070       0.1641       -0.02         2350       ISO17070       0.181       -0.02         2354       ISO17070       0.182       0.00         2366       ISO17070       0.182       0.00         2366       ISO17070       0.183       0.01         2377       ISO17070       0.183       0.01         2378       ISO17070       0.182       -0.03         2380       ISO17070       0.180       -0.03         2381       ISO17070       0.180       -0.03         2382       ISO17070       0.180       -0.03         2384       ISO17070       0.120       -0.03         2385       ISO17070       0.2366       0.63         2380       ISO17070						
2285       DINS0009       0.28       1.14         2289       ISO17070       0.2       0.21         2287       ISO17070       0.31281       C       1.52         2301       IFG8 B82 02.8       0.1           2311       ISO17070       0.31281       C       1.52         2331       ISO17070       0.941       -1.03         2333       ISO17070       0.16          2334       ISO17070       0.17       -0.14         2335       ISO17070       0.18          2345       ISO17070       0.18          2355       ISO17070       0.17          2356       ISO17070       0.170          2371       ISO17070       0.170          2373       ISO17070       0.172      1.28         2373       ISO17070       0.172      1.28         2379       ISO17070       0.152      0.3         2382       ISO17070       0.168      0.3         2384       ISO17070       0.276       1.08         2371       ISO17070       0.236       0.63 <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>						
2283         ISO17070         0.2         0.21           2297         ISO17070         0.19         0.09           2301         ISO17070         0.31281         C         1.52           2310         ISO17070         0.19         0.09           2331         ISO17070         0.198         0.16           23347         ISO17070         0.16         -0.26           2335         ISO17070         0.16         -0.26           2336         ISO17070         0.181         -0.02           2336         ISO17070         0.181         -0.02           2336         ISO17070         0.17         -1.4           2337         ISO17070         0.170            2337         ISO17070         0.470            2338         ISO17070         0.470            2347         ISO17070         0.470            2348         Inhouse         0.217         -0.3           2359         ISO17070         0.18         -0.03           2349         ISO17070         0.236         0.63           2349         Inhouse         0.17						
2239       ISO17070       0.19       C       1.52         2301       IFGB B82 02.8       0.1       -9.66       -9.66         2311       ISO17070       0.0441       -1.03         2330       ISO17070       0.196       0.16         2331       ISO17070       0.16       -0.26         2336       ISO17070       0.16       -0.26         2337       ISO17070       0.181       -0.02         2338       ISO17070       0.17       -0.14         2349       ISO17070       0.170       -0.14         2351       ISO17070       0.170       -0.14         2353       ISO17070       0.170       -0.14         2354       ISO17070       0.172       -1.28         2379       ISO17070       0.172       -1.3         2380       Inbuse       0.447       R(0.05)       3.08         2396       Inbuse       0.272       -1.3         2390       Inbuse       0.120       -0.72         2445       Inbuse       0.217       0.40         2452       ISO17070       0.180       -0.03         2451       Inbuse       0.275       1.08						
2291     ISO17070     0.19     0.09       2301     ISO17070     0.31281     C     1.52       2310     ISO17070     0.196     0.16       2331     ISO17070     0.196     0.16       2334     ISO17070     0.16     -0.26       2357     ISO17070     0.181     -0.02       2368         2371     ISO17070     0.182     0.00       2385     ISO17070     0.182     0.00       2386     INouse     -0.50        2397     ISO17070     0.170     -0.14       2398     ISO17070     0.182     0.00       2371     ISO17070     0.182     -0.3       2373     ISO17070     0.182     -0.3       2389     ISO17070     0.182     -0.3       2389     INouse     0.247     R(0.05)     3.08       2389     INouse     0.247     R(0.05)     3.08       2390     INouse     0.275     1.08       2491     INOuse     0.17     -0.14       2492     ISO17070     0.2366     0.63       2511     ISO17070     0.27     1.02       2520     ISO17070     0.27     1.02		15017070				
2301       ISO17070       0.31281       C       1.52       First reported 216.91 mg/kg         2311       ISO17070       0.0941       -1.03         2331       ISO17070       0.17       -0.16         2347       ISO17070       0.181       -0.02         2358       ISO17070       0.181       -0.02         2358       ISO17070       0.182       0.00         2366       ISO17070       0.182       0.00         2366       ISO17070       0.183       0.01         2371       ISO17070       0.183       0.01         23737       ISO17070       0.170       -0.14         23737       ISO17070       0.183       0.01         23737       ISO17070       0.184       -0.03         2380       ISO17070       0.184       -0.03         2381       ISO17070       0.184       -0.03         2382       ISO17070       0.18       -0.03         2384       Inhouse       0.27       1.28         2380       ISO17070       0.2366       0.63         2381       IsO17070       0.246          2485       Inhouse       0.17       -0.14 <td></td> <td>10017070</td> <td></td> <td></td> <td></td> <td></td>		10017070				
2310       LFGB B82.02.8       0.1       -0.96         2311       LSO17070       0.196       0.16         2333       LSO17070       0.177       -0.14         2350       LSO17070       0.181       -0.02         2355       LSO17070       0.181       -0.02         2356       LSO17070       0.181       -0.02         2356       LSO17070       0.182       0.00         2357       LSO17070       0.183       0.01         2373       LSO17070       0.170       -0.14         2375       LSO17070       0.172       -1.28         2376       LSO17070       0.152       -0.35         2377       LSO17070       0.152       -0.35         2382       LSO17070       0.152       -0.35         2384       LSO17070       0.180       -0.03         2385       In house       0.275       1.08         2384       LSO17070       0.2366       0.63         2454       In house       0.27       1.02         2551       ISO17070       0.234       0.57         2541       In house       0.27       1.02         2551       ISO170				C		First reported 216 01 mg/kg
2311       ISO17070       0.0941       -1.03         2330       ISO17070       0.17       -0.14         2347       ISO17070       0.181       -0.02         2358       ISO17070       0.181       -0.02         2358       ISO17070       0.182       0.00         2366       ISO17070       0.182       0.00         2366       ISO17070       0.183       0.01         2377       ISO17070       0.183       0.01         2378       ISO17070       0.183       0.01         2379       ISO17070       0.172       -1.28         2380       ISO17070       0.182       -0.03         2380       ISO17070       0.184       -0.03         2380       ISO17070       0.184       -0.03         2381       ISO17070       0.18       -0.03         2384       Inhouse       0.275       1.06         2391       ISO17070       0.2866       0.63         2391       IsO17070       0.2366       0.63         2391       IsO17070       0.27       0.22         2391       ISO17070       0.27       0.22         2391       IsO17070 <td></td> <td></td> <td></td> <td>C</td> <td></td> <td>First reported 210.91 mg/kg</td>				C		First reported 210.91 mg/kg
2330     ISO17070     0.196     0.16       2347     ISO17070     0.16     -0.26       2358     ISO17070     0.181     -0.02       2358     ISO17070     0.181     -0.02       2358     ISO17070     0.182     0.00       2356     IsO17070     0.182     0.00       2371     ISO17070     0.170     -0.14       2373     ISO17070     0.170     -0.14       2374     ISO17070     0.170     -0.14       2375     ISO17070     0.183     0.01       2376     ISO17070     0.183     0.01       2377     ISO17070     0.152     -0.35       2382     ISO17070     0.152     -0.35       2384     ISO17070     0.180     -0.03       2385     In house     0.247     R(0.05)     3.08       2390     In house     0.275     1.08       2541     Iso17070     0.2366     0.63       2541     Iso17070     0.231     0.57       2541     Iso17070     0.2366     0.63       2541     Iso17070     0.277     1.02       2551     ISO17070     0.27     1.02       2551     ISO17070     0.18     -0.03 <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>						
2341     ISO17070     0.17     -0.14       2350     ISO17070     0.181     -0.02       2351     ISO17070     0.182     0.00       2365     ISO17070     0.182     0.00       2366     ISO17070     0.182     0.00       2367     ISO17070     0.170     -0.14       2371     ISO17070     0.172     -1.28       2375     ISO17070     0.152     -0.35       2386     ISO17070     0.152     -0.35       2386     ISO17070     0.160     -0.03       2386     Inhouse     0.217     0.40       2386     Inhouse     0.217     0.40       2386     Inhouse     0.217     0.40       2489     ISO17070     0.180     -0.03       2386     Inhouse     0.217     0.40       2489     ISO17070     0.18     -0.03       2501     ISO17070     0.2366     0.63       2511     ISO17070     0.2366     0.63       2520     ISO17070     0.22     0.21       2531     ISO17070     0.22     0.21       2541     ISO17070     0.22     0.21       2541     ISO17070     0.18     -0.03       254						
2350       ISO17070       0.16       -0.26         2358       ISO17070       0.124       -0.68         2363						
2357     ISO17070     0.181     -0.02       2365     ISO17070     0.182     0.00       2366     ISO17070     0.182     0.00       2367     ISO17070     0.170     -0.14       2371     ISO17070     0.170     -0.14       2375     ISO17070     0.172     -1.28       2376     ISO17070     0.172     -0.35       2380     ISO17070     0.152     -0.35       2380     ISO17070     0.160     -0.03       2386     In house     0.247     R(0.05)       2386     In house     0.217     0.40       2489     ISO17070     0.180     -0.03       2386     In house     0.275     1.08       2489     ISO17070     0.16     -0.72       2504     ISO17070     0.2366     0.63       2511     ISO17070     0.23     0.57       2520     ISO17070     0.22     0.21       2531     ISO17070     0.22     0.21       2541     ISO17070     0.22     0.21       2533     ISO17070     0.22     0.21       2541     ISO17070     0.18     -0.03       2541     ISO17070     0.18       2541 <td< td=""><td></td><td></td><td></td><td></td><td></td><td></td></td<>						
2353						
2363						
2366       In House       <0.50						
2360         In house         <0.50		ISO17070				
2373       ISO17070       0.170       -0.14         2373       ISO17070       0.072       -1.28         2379       ISO17070       0.172       -0.13         2380       ISO17070       0.152       -0.35         2381       ISO17070       0.180       -0.03         2382       ISO17070       0.1447       R(0.05)       3.08         2380       In house       0.217       0.40         2381       IsO17070       0.18       -0.03         2382       ISO17070       0.16       -0.03         2384       In house       0.120       -0.72         2489       ISO17070       0.2366       0.63         2511       ISO17070       0.2366       0.63         2520       ISO17070       0.27       1.02         2531       ISO17070       0.27       0.21         2541       ISO17070       0.21       0.32         25561       ISO17070       0.21       0.32         2569       ISO17070       0.21       0.32         2590           2731       IsO17070       0.18       -0.03         2688       ISO17070						
2373       ISO17070       0.183       0.01         2375       ISO17070       0.2792       1.13         2380       ISO17070       0.152       -0.35         2382       ISO17070       0.180       -0.03         2386       IsO17070       0.180       -0.03         2386       IsO17070       0.180       -0.03         2386       IsO17070       0.180       -0.03         2386       IsO17070       0.180       -0.03         2387       ISO17070       0.180       -0.03         2489       ISO17070       0.18       -0.03         2541       In house       0.275       1.08         2511       ISO17070       0.2366       0.63         2514       In house       0.17       -0.14         2520       ISO17070       0.271       1.02         2561       ISO17070       0.21       0.32         2569       ISO17070       0.21       0.32         2573       ISO17070       0.21       0.32         2585       ISO17070       0.18       -0.03         2686       ISO17070       0.18       -0.03         2711       ISO17070						
2375       ISO17070       0.072       -1.28         2379       ISO17070       0.152       -0.35         2380       ISO17070       0.160       -0.03         2380       In house       0.247       R(0.05)       3.08         2390       In house       0.217       0.40         2421       ISO17070       0.18       -0.03         2435       ISO17070       0.14 detected						
2379     ISO17070     0.2792     1.13       2380     ISO17070     0.152     -0.35       2382     ISO17070     0.180     -0.03       2386     In house     0.247     R(0.05)     3.08       2390     In house     0.217     0.40       2489     ISO17070     not detected			0.072			
2380       ISO17070       0.152       -0.35         2382       ISO17070       0.160       -0.03         2386       In house       0.217       0.40         2381       ISO17070       n0 detected		ISO17070	0.2792			
2386       In house       0.447       R(0.05)       3.08         2390       In house       0.217       0.40         2452       ISO17070       0.18       -0.03         2489       ISO17070       0.18       -0.03         2495       In house       0.120       -0.72         2604       In house       0.275       1.08         2511       ISO17070       Less than 0.1          2520       ISO17070       Less than 0.1          2521       ISO17070       0.21       0.32         2569       ISO17070       0.21       0.32         2569       ISO17070       0.21       0.32         2590           2605       ISO17070       0.18       -0.03         2606       ISO17070       0.18       -0.03         2605       ISO17070       0.18       -0.03         2703       In house       Not detected          2711       ISO17070       0       R(0.05)       2.79         2731       IsO17070       1.56       -0.31          2737       In house       0.2       0.21	2380	ISO17070	0.152		-0.35	
2390     In house     0.217     0.40       2452     ISO17070     not detected	2382	ISO17070	0.180		-0.03	
2452       ISO17070       not detected		In house		R(0.05)	3.08	
2489       ISO17070       0.18       -0.03         2495       In house       0.275       1.08         2514       In house       0.275       1.08         2511       ISO17070       0.2366       0.63         2514       In house       0.17       -0.14         2520       ISO17070       Less than 0.1		In house	0.217		0.40	
2495       In house       0.120       -0.72         2504       In house       0.275       1.08         2511       ISO17070       0.2366       0.63         2514       In house       0.17       -0.14         2520       ISO17070       Less than 0.1						
2504         In house         0.275         1.08           2511         ISO17070         0.2366         0.63           2514         In house         0.17         -0.14           2520         ISO17070         Less than 0.1            2532         ISO17070         0.231         0.57           2561         ISO17070         0.21         0.32           2573         ISO17070         0.21         0.32           2590             2605         ISO17070         0.18         -0.03           2668         ISO17070         0.18         -0.03           2668         ISO17070         0.18         -0.03           2703         In house         Not detected            2711         ISO17070         0         R(0.05)         -2.12           2713         In house         0.422         R(0.05)         -2.12           2721         ISO17070         0.156         -0.31           2737         ISO17070         1.83         C,R(0.01)         19.16           2737         ISO17070         1.83         C,R(0.01)         19.16           2737						
2511       ISO17070       0.2366       0.63         2514       In house       0.17       -0.14         2520       ISO17070       Less than 0.1          2532       ISO17070       0.27       1.02         2561       ISO17070       0.231       0.57         2569       ISO17070       0.21       0.32         2590           2605       ISO17070       0.18       -0.03         2668       ISO17070       0.18       -0.03         2668       ISO17070       0.18       -0.03         2665       ISO17070       0.18       -0.03         2703       In house       Not detected          2711       ISO17070       0       R(0.05)       2.12         2713       In house       not detected          2723       In house       not detected          2734       ISO17070       1.83       C,R(0.01)       19.16         2737       In house       0.2       0.21          2737       In house       0.2       0.21          2736       ISO17070       1.83						
2514       In house       0.17       -0.14         2520       ISO17070       Less than 0.1          2532       ISO17070       0.27       1.02         2561       ISO17070       0.21       0.57         2569       ISO17070       0.21       0.32         2573       ISO17070       0.21       0.32         2580           2605       ISO17070       0.18       -0.03         2668       ISO17070       0.0708       -1.30         2703       In house       Not detected          2711       ISO17070       0       R(0.05)       -2.12         2733       In house       0.422       R(0.05)       2.79         2723       Is house       not detected          2727       ISO17070       0.156       -0.31         2737       In house       0.2       0.21         2737       Is house       not detected          2737       ISO17070       1.83       C.R(0.01)       19.16         2737       Is house       0.2       0.21       -254         2907       0.08105       -1.18						
2520       ISO17070       Less than $0.1$ 2532       ISO17070 $0.27$ $1.02$ 2561       ISO17070 $0.231$ $0.57$ 2569       ISO17070 $0.21$ $0.32$ 2590       SO17070 $0.21$ $0.32$ 2590           2605       ISO17070 $0.18$ $-0.03$ 2686       ISO17070 $0.18$ $-0.03$ 2695       ISO17070 $0.18$ $-0.03$ 2695       ISO17070 $0.0708$ $-1.30$ 2703       In house       Not detected          2711       ISO17070 $0.156$ $-0.31$ 2723       In house       not detected          2734       ISO17070 $1.56$ $-0.31$ 2737            2737       Not detected          2737       0.08105 $-1.18$ 2979       ISO17070       1.83       C,R(0.01)       19.16         2979       ISO17070       0.271       0.281         2979						
2532       ISO17070       0.27       1.02         2561       ISO17070       0.21       0.57         2573       ISO17070       0.21       0.32         2590           2605       ISO17070       0.18       -0.03         2668       ISO17070       0.18       -0.03         2668       ISO17070       0.18       -0.03         2703       In house       Not detected          2711       ISO17070       0       R(0.05)       -2.12         2713       In house       0.422       R(0.05)       -2.72         2723       In house       0.422       R(0.05)       -2.79         2723       Is house       not detected          2734       ISO17070       0.156       -0.31         2737            2736       ISO17070       1.83       C.R(0.01)       19.16         2979       ISO17070       1.83       C.R(0.01)       19.16         2979       ISO17070       Not detected          2980            3016       LFGB B82.02.8						
2561       ISO17070       0.21       0.57         2569       ISO17070       0.2       0.21         2573       ISO17070       0.18       -0.03         2605       ISO17070       0.18       -0.03         2668       ISO17070       0.18       -0.03         2695       ISO17070       0.0708       -1.30         2703       In house       Not detected						
2569       ISO17070       0.2       0.21         2573       ISO17070       0.21       0.32         2590           2605       ISO17070       0.18       -0.03         2686       ISO17070       0.18       -0.03         2695       ISO17070       0.0708       -1.30         2703       In house       Not detected          2711       ISO17070       0       R(0.05)       -2.12         2713       In house       not detected          2723       Is house       not detected          2737       IsO17070       0.156       -0.31         2737       IsO17070       not detected          2737       ISO17070       not detected						
2573       ISO17070       0.21       0.32         2590           2605       ISO17070       0.18       -0.03         2695       ISO17070       0.708       -1.30         2703       In house       Not detected          2711       ISO17070       0       R(0.05)       -2.12         2713       In house       0.422       R(0.05)       2.79         2723       In house       not detected          2734       ISO17070       0.156       -0.31         2734       ISO17070       1.83       C.R(0.01)       19.16         2737            2736       ISO17070       1.83       C.R(0.01)       19.16         2806       In house       0.2       0.21         2977       0.08105       -1.18         2979       ISO17070       Not detected          2980            2980            3003       DIN50009       0.275       1.08         3172       ISO17070       0.271       1.03					0.57	
2590           2605       ISO17070       0.18       -0.03         2668       ISO17070       0.0708       -1.30         2703       In house       Not detected          2711       ISO17070       0       R(0.05)       -2.12         2713       In house       0.422       R(0.05)       2.79         2723       In house       not detected          2727       ISO17070       0.156       -0.31         2734       ISO17070       not detected          2737           2756       ISO17070       1.83       C,R(0.01)       19.16         2979       ISO17070       1.83       C,R(0.01)       19.16         2979       ISO17070       1.83       C,R(0.01)       19.16         2979       ISO17070       1.58       -0.26         3116       LFGB B82.02.8       0.086       -1.12         3154       DIN50009       0.275       1.08         3172       ISO17070       0.2713       1.03         3210       In house       0.18       -0.03         3218       ISO17070       0						
2605       ISO17070       0.18       -0.03         2668       ISO17070       0.18       -0.03         2695       ISO17070       0.0708       -1.30         2703       In house       Not detected          2711       ISO17070       0       R(0.05)       -2.12         2713       In house       0.422       R(0.05)       2.79         2723       In house       not detected          2727       ISO17070       0.156       -0.31         2734       ISO17070       not detected          2737            2756       ISO17070       1.83       C,R(0.01)       19.16       First reported 2.54         2806       In house       0.2       0.21           2977       0.08105       -1.18           2980             2980             2980             3003       DIN50009       0.275       1.08       -112	2573	15017070	0.21		0.32	
2668       ISO17070       0.18       -0.03         2695       ISO17070       0.0708       -1.30         2703       In house       Not detected          2711       ISO17070       0       R(0.05)       2.12         2713       In house       0.422       R(0.05)       2.79         2723       In house       not detected          2727       ISO17070       0.156       -0.31         2734       ISO17070       not detected          2737           2736       ISO17070       1.83       C,R(0.01)       19.16       First reported 2.54         2806       In house       0.2       0.21           2756       ISO17070       1.83       C,R(0.01)       19.16       First reported 2.54         2806       In house       0.2       0.21           2979       ISO17070       Not detected           2980             3003       DIN50009       0.1598       -0.26       -1.12         3154       DIN50009		19017070	0.18		_0.03	
2695       ISO17070       0.0708       -1.30         2703       In house       Not detected          2711       ISO17070       0       R(0.05)       -2.12         2713       In house       0.422       R(0.05)       2.79         2723       In house       not detected          2727       ISO17070       0.156       -0.31         2734       ISO17070       not detected          2737           2756       ISO17070       1.83       C,R(0.01)       19.16       First reported 2.54         2806       In house       0.2       0.21           2977       0.08105       -1.18           2980             3003       DIN50009       0.1598       -0.26       -1.12         3154       DIN50009       0.275       1.08       -1.12         3172       ISO17070       0.2713       1.03          3210       In house       0.14       -0.49          3228       In house       0.18       -0.03 <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>						
2703In houseNot detected2711ISO170700R(0.05)-2.122713In house0.422R(0.05)2.792723In housenot detected2727ISO170700.156-0.312734ISO17070not detected27372736ISO170701.83C,R(0.01)19.16In house0.20.2129770.08105-1.182979ISO17070Not detected29802003DIN500090.15983116LFGB B82.02.80.0863172ISO170700.27133172ISO170700.27133210In house0.143228In house0.183220LFGB B82.02.80.20.21						
$\begin{array}{cccccccccccccccccccccccccccccccccccc$						
2713       In house       0.422       R(0.05)       2.79         2723       In house       not detected          2727       ISO17070       0.156       -0.31         2734       ISO17070       not detected          2737           2756       ISO17070       1.83       C,R(0.01)       19.16         2806       In house       0.2       0.21         2977       0.08105       -1.18         2979       ISO17070       Not detected          2980           3003       DIN50009       0.1598       -0.26         3116       LFGB B82.02.8       0.086       -1.12         3154       DIN50009       0.2713       1.03         3210       In house       0.14       -0.49         3218       ISO17070       <0.050				R(0.05)		
2723In housenot detected2727ISO170700.156-0.312734ISO17070not detected27372756ISO170701.83 $C,R(0.01)$ 19.16First reported 2.542806In house0.20.8105-1.182979ISO17070Not detected29803003DIN500090.15983116LFGB B82.02.80.0863116LFGB B82.02.80.08631172ISO170700.27133210In house0.143218ISO170703228In house0.1832373250LFGB B82.02.80.20.20.21						
2727       ISO17070       0.156       -0.31         2734       ISO17070       not detected          2737           2736       ISO17070       1.83       C,R(0.01)       19.16       First reported 2.54         2806       In house       0.2       0.21          2977       0.08105       -1.18          2980           3003       DIN50009       0.1598       -0.26         3116       LFGB B82.02.8       0.086       -1.12         3154       DIN50009       0.275       1.08         3172       ISO17070       0.2713       1.03         3210       In house       0.14       -0.49         3218       ISO17070       <0.2713				()		
2734       ISO17070       not detected          2737           2756       ISO17070       1.83       C,R(0.01)       19.16       First reported 2.54         2806       In house       0.2       0.21          2977       0.08105       -1.18          2980           3003       DIN50009       0.1598       -0.26         3116       LFGB B82.02.8       0.086       -1.12         3154       DIN50009       0.275       1.08         3172       ISO17070       0.2713       1.03         3218       ISO17070       <0.050						
2737           2756       ISO17070       1.83       C,R(0.01)       19.16       First reported 2.54         2806       In house       0.2       0.21          2977       0.08105       -1.18          2980           3003       DIN50009       0.1598       -0.26         3116       LFGB B82.02.8       0.086       -1.12         3154       DIN5009       0.2713       1.03         3210       In house       0.14       -0.49         3218       ISO17070       <0.050						
2756       ISO17070       1.83       C,R(0.01)       19.16       First reported 2.54         2806       In house       0.2       0.21         2977       0.08105       -1.18         2979       ISO17070       Not detected          2980           3003       DIN50009       0.1598       -0.26         3116       LFGB B82.02.8       0.086       -1.12         3154       DIN50009       0.275       1.08         3172       ISO17070       0.2713       1.03         3210       In house       0.14       -0.49         3218       ISO17070       <0.050						
2806       In house       0.2       0.21         2977       0.08105       -1.18         2979       ISO17070       Not detected          2980           3003       DIN50009       0.1598       -0.26         3116       LFGB B82.02.8       0.086       -1.12         3154       DIN50009       0.275       1.08         3172       ISO17070       0.2713       1.03         3210       In house       0.14       -0.49         3218       ISO17070       <0.050		ISO17070		C,R(0.01)	19.16	First reported 2.54
2977       0.08105       -1.18         2979       ISO17070       Not detected          2980           3003       DIN50009       0.1598       -0.26         3116       LFGB B82.02.8       0.086       -1.12         3154       DIN50009       0.275       1.08         3172       ISO17070       0.2713       1.03         3210       In house       0.14       -0.49         3218       ISO17070       <0.050		In house		. ,		
2980           3003       DIN50009       0.1598       -0.26         3116       LFGB B82.02.8       0.086       -1.12         3154       DIN50009       0.275       1.08         3172       ISO17070       0.2713       1.03         3210       In house       0.14       -0.49         3218       ISO17070       <0.050						
2980           3003       DIN50009       0.1598       -0.26         3116       LFGB B82.02.8       0.086       -1.12         3154       DIN50009       0.275       1.08         3172       ISO17070       0.2713       1.03         3210       In house       0.14       -0.49         3218       ISO17070       <0.050		ISO17070	Not detected			
3116       LFGB B82.02.8       0.086       -1.12         3154       DIN50009       0.275       1.08         3172       ISO17070       0.2713       1.03         3210       In house       0.14       -0.49         3218       ISO17070       <0.050	2980					
3154       DIN50009       0.275       1.08         3172       ISO17070       0.2713       1.03         3210       In house       0.14       -0.49         3218       ISO17070       <0.050	3003					
3172       ISO17070       0.2713       1.03         3210       In house       0.14       -0.49         3218       ISO17070       <0.050		LFGB B82.02.8				
3210     In house     0.14     -0.49       3218     ISO17070     <0.050		DIN50009				
3218       ISO17070       <0.050		ISO17070				
3228     In house     0.18     -0.03       3237         3250     LFGB B82.02.8     0.2     0.21					-0.49	
3237         3250     LFGB B82.02.8     0.2     0.21						
3250 LFGB B82.02.8 0.2 0.21		In house	0.18			
8005 ISO1/070 0.095 -1.02						
	8005	15017070	0.095		-1.02	

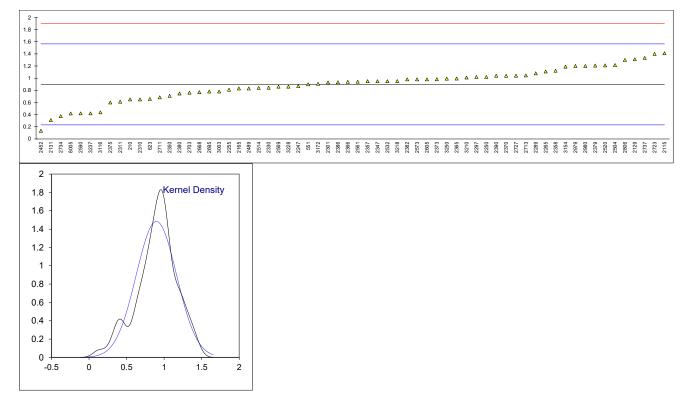
normality n outliers mean (n) st.dev. (n) R(calc.) st.dev.(iis memo 1601) D(iii memo 1601)	OK 50 5 0.1823 0.06433 0.1801 0.08600 0.2408	RSD = 35%
R(iis memo 1601)	0.2408	



# Determination of 2,4,6-Trichlorophenol on sample #22585; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	ISO17070	0.65		-0.74	
551	LFGB B82.02.8	0.8987		0.00	
623 2115	ISO17070 ISO17070	0.657 1.41		-0.72 1.54	
2115	ISO17070	1.310		1.54	
2123	In house	0.31		-1.76	
2165	ISO17070	0.83		-0.20	
2247	DIN50009	0.87		-0.08	
2250	In house	1.02		0.37	
2255	ISO17070	0.81		-0.26	
2265	DIN50009	1.11		0.64	
2289	ISO17070	1.08		0.55	
2293					
2297	ISO17070	1.02		0.37	
2301	ISO17070	0.93		0.10	
2310	LFGB B82.02.8	0.65		-0.74	
2311 2330	ISO17070 ISO17070	0.6126 0.843		-0.85 -0.16	
2330	ISO17070	0.843		0.16	
2350	ISO17070	0.71		-0.56	
2357	ISO17070	0.950		0.16	
2358	ISO17070	1.1235		0.68	
2363					
2365	ISO17070	0.993		0.29	
2366	In house	0.94		0.13	
2370	ISO17070	1.04		0.43	
2373	ISO17070	0.982		0.25	
2375	ISO17070	0.60		-0.89	
2379	ISO17070	1.2076		0.93	
2380	ISO17070	0.747		-0.45	
2382	ISO17070	0.980		0.25	
2386 2390	In house	0.932 1.04		0.10 0.43	
2390	In house ISO17070	0.134		-2.29	
2489	ISO17070	0.83		-0.20	
2495	In house	< 0.05			
2504	In house	1.215	С	0.95	First reported 2.322
2511					
2514	In house	0.84		-0.17	
2520	ISO17070	1.21		0.94	
2532	ISO17070	0.95		0.16	
2561	ISO17070	0.94159		0.13	
2569	ISO17070	0.86		-0.11	
2573	ISO17070	0.98		0.25	
2590	ISO17070	0.420		-1.43	
2605	ISO17070	0.98		0.25	
2668 2695	ISO17070 ISO17070	0.77 0.7795		-0.38 -0.35	
2095	In house	0.7795		-0.35 -0.41	
2703	ISO17070	0.6844		-0.41	
2713	In house	1.044		0.44	
2723	In house	1.4		1.51	
2727	ISO17070	1.041		0.43	
2734	ISO17070	0.375		-1.57	
2737	GB/T22808	1.333		1.31	
2756					
2806	In house	1.3		1.21	
2977	10017070	Not Deter	mined		
2979	ISO17070	1.2	C	0.91	First we wanted 4.0
2980	ISO17070	1.2	С	0.91	First reported 1.8
3003	DIN50009 LFGB B82.02.8	0.78 0.437		-0.35 -1.38	
3116 3154	DIN50009	0.437		-1.38 0.88	
3154 3172	ISO17070	0.9079		0.08	
3210	In house	1.01		0.03	
3210	ISO17070	0.953		0.34	
3228	In house	0.86		-0.11	
3237	ISO17070	0.42		-1.43	
3250	LFGB B82.02.8	0.992		0.28	
8005	ISO17070	0.419		-1.44	

 mean (n) st.dev. (n) R(calc.) st.dev.(iis memo 1601)	64 0 0.8972 0.26898 RSD = 30% 0.7531 0.33322 0.9330
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Summary of other reported Chlorinated Phenols on sample #22585; results in mg/kg

2345TeCP 2346TeCP	= 2,3,4,5-Tetrachlorophenol = 2,3,4,6-Tetrachlorophenol
2356TeCP	= 2,3,5,6-Tetrachlorophenol
234TCP	= 2,3,4-Trichlorophenol
235TCP	= 2,3,5-Trichlorophenol
236TCP	= 2,3,6-Trichlorophenol
245TCP	= 2,4,5-Trichlorophenol
345TCP	= 3,4,5-Trichlorophenol
23DCP	= 2,3-Dichlorophenol
24DCP	= 2,4-Dichlorophenol
25DCP	= 2,5-Dichlorophenol
26DCP	= 2,6-Dichlorophenol
34DCP	= 3,4-Dichlorophenol
35DCP	= 3,5-Dichlorophenol
2CP	= 2-Chlorophenol
3CP	= 3-Chlorophenol
4CP	= 4-Chlorophenol

lab	2345TeCP	2346TeCP	2356TeCP	234TCP	235TCP	236TCP	245TCP	345TCP
210	0.15	nd	nd	nd	nd	nd	nd	nd
551		0.1997					0.1997	
623	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2115		0.094		0.055				
2129	<0,1	0.341	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1
2131	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2165	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2247	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2250		0.10						
2255 2265	not detected < 0.05	not detected < 0.05	not detected < 0.05	not detected < 0.05	not detected < 0.05	not detected < 0.05	not detected < 0.05	not detected < 0.05
2205	< 0,05	< 0,05 0.09	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05
2203								
2297	<0.05	0.07	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2301								
2310	not detected	0.08	not detected					
2311	not detected	0.07038	not detected					
2330	ND	ND	ND	ND	ND	ND	ND	ND
2347	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2350	< 0.125	< 0.125	< 0.125	< 0.125	< 0.125	< 0.125	< 0.125	< 0.125
2357								
2358	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2363								
2365	<0.05	0.071	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2366	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50	<0.50
2370	<0.05	0.0740	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
2373	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2375 2379	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2379	< 0.05	< 0.05	< 0.05	< 0.05	<0.05	<0.05	<0.05	<0.05
2382	<0.00	<0.00	<0.00	<0.00	<0.00	<0.00	<0.00	<0.00
2386	<0.100	<0,20	<0.100	<0.100	<0.100	<0,10	<0,10	<0.100
2390	not detected	0.0985	not detected					
2452	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2489	not detected		not detected					
2495	<0.05	0.320	<0.05	0.951	<0.05	<0.05	<0.05	<0.05
2504				not detected	not detected	not detected	< 0.1	not detected
2511								
2514								
2520	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
2532	not detected		not detected					
2561		0.11498		0.06919				
2569 2573	not detected not detected	 0.08	not detected not detected					
2573		0.08						
2605	<0.1	<0.1	<0.1	 <0.1	<0.1	<0.1	<0.1	<0.1
2668	not detected	0.09	not detected					
2695	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2703	Not detected	0.0417	Not detected					
2711	0	0	0	0	0	0	0	0
2713	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05	<0,05

lab	2345TeCP	2346TeCP	2356TeCP	234TCP	235TCP	236TCP	245TCP	345TCP
2723	not detected	not detected	not detected	not analyzed				
2727	not detected							
2734	not detected							
2737								
2756								
2806	< 0,1	0.1	< 0,1	< 0,1	< 0,1	< 0,1	< 0,1	< 0,1
2977	0.21704	not detected	not detected					
2979	not detected							
2980								
3003		0.1						
3116								
3154	not detected	0.163	not detected	not detected	not detected	not detected	0.11	not detected
3172	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
3210	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
3218	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050
3228	not detected							
3237								
3250								
8005								

#### -- continued --

lab	23DCP	24DCP	25DCP	26DCP	34DCP	35DCP	2CP	3CP	4CP
210	nd	nd	nd	nd	nd	nd	nd	nd	nd
551		0.2996	0.2996						
623	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2115			0.075						0.131
2129	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	<0,1	0.219
2131	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2165	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2247	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2250									0.12
2255	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	0.10
2265	< 0,05	0.29	0.29	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05	< 0,05
2289									
2293									
2297 2301	<0.05 	<0.05	<0.05 	<0.05	<0.05	<0.05 			
2301	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2310	not detected	< 0.01	< 0.01	not detected					
2330	ND	3.668	3.668	ND	ND	ND	ND	ND	0.134
2347									
2350	< 0.125	< 0.125	< 0.125	< 0.125	< 0.125	< 0.125	< 0.125	< 0.125	< 0.125
2357									
2358	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	0.134
2363									
2365	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	0.124
2366									
2370	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	0.126
2373	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2375									
2379	not detected			not detected	0.1129				
2380	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	0.100
2382 2386	<0.100 <0.50	<0.100 0.706	<0.100	<0.100 <0,50	<0.100 <0.50	<0.100 <0,50	<0.100 <0.50	<0.100 <0.50	<0.100 <0,50
2380	not detected	not detected	see comment 0.217	not detected					
2390	not detected		not detected	not detected	not detected	not detected	not detected	not detected	not detected
2489	not detected			not detected					
2495	< 0.05			< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
2504									
2511									
2514									0.10
2520	<0.1	0.59	0.61	16.35	<0.1	<0.1	<0.1	<0.1	<0.1
2532	not detected			not detected					
2561									
2569	not detected			not detected					
2573	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2590									
2605	< 0.1		 					 	
2668	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2695 2703	not detected	not detected	not detected	not detected	not detected	not detected	0.1571	not detected	0.1222
2703	not detected		0.1619 	not detected					
2711									
2723									
2727									
2121									

#### Institute for Interlaboratory Studies

lab	23DCP	24DCP	25DCP	26DCP	34DCP	35DCP	2CP	3CP	4CP
2734	not detected								
2737									
2756									
2806									
2977									
2979	not detected	0.6	0.6	16.5	not detected				
2980		0.6	0.6	16.5					
3003									0.1
3116									
3154	not detected	not detected	0.1	not detected	0.25				
3172	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
3210	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
3218	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050
3228	not detected								
3237									
3250									
8005									

# **APPENDIX 3** Analytical Details

lah	10047005	Comula	Commis		Eutroption to chairma
lab	ISO17025 accredited	Sample preparation	Sample intake (g)	Release technique	Extraction technique
210	Yes	Further cut	intake (g)	Steam distillation	Mechanical Shaking
551	Yes	Further cut	1 g	Steam distillation was skipped	Ultrasonic
623	Yes	Further cut	1 gram	Other	Ultrasonic
2115	Yes	Used as received	1g	Steam distillation	Ultrasonic
2129	Yes	Used as received	0,5g	Other	ASE
2131	Yes	Used as received	1g	Microwave extraction	Other Mashaniaal Shaking
2165 2247	Yes Yes	Further cut Used as received	1.0g	Steam distillation	Mechanical Shaking Other
2250	Yes	Further cut	0.7gm 1	KOH extraction followed by 64 LFGB 82.02.8 Other	Ultrasonic
2255	Yes	Further cut	0.5	Steam distillation	Ultrasonic
2265	No	Used as received	0,5	Other	Ultrasonic
2289	Yes	Further cut	1.0g	Steam distillation	Soxhlet
2293					
2297	Yes	Used as received	1g	Steam distillation	Mechanical Shaking
2301 2310	No Yes	Used as received	1.0030	Steam distillation	Ultrasonic Mechanical Sheking
2310	Yes	Further cut Further cut	2grams 1	Steam distillation Steam distillation	Mechanical Shaking Mechanical Shaking
2330	No	Further cut	1.0000g	Steam distillation	Mechanical Shaking
2347	Yes	Further cut	1.0g		
2350	No	Further cut	2.0083 g	Steam distillation	ASE
2357					
2358	Yes	Used as received	1 g	Steam distillation	
2363					
2365 2366	Yes No	Further cut	1.5g	Steam distillation Other	Mechanical Shaking
2300	Yes	Further cut Further cut	0.5g 1 g	Steam distillation	Ultrasonic Other
2373			2g		
2375	Yes	Further cut	-9 1 gr	Steam distillation	Mechanical Shaking
2379	No	Further cut	1 grams	Steam distillation	Other
2380	Yes	Further cut	1.0 g	Steam distillation	Mechanical Shaking
2382	Yes	Further cut	1.0g	Steam distillation	Steam distillation
2386	Yes	Further cut	0.500	extracted with 1 M KOH	Ultrasonic
2390 2452	Yes No	Further cut Used as received	0.5 gram 1	Alkaline digestion Steam distillation	Ultrasonic Soxhlet
2452	Yes	Further cut	1.0010g	Steam distillation	Mechanical Shaking
2495	Yes	Used as received	0.5	KOH method	Ultrasonic
2504	Yes	Further cut	0.5 grams	KOH extraction	Heating block
2511	Yes	Used as received	0.5 gram		
2514	Yes	Further cut	0.3712 gm	KOH METHOD	Mechanical Shaking
2520	No	Further cut	1gm	Steam distillation	Mechanical Shaking
2532	Yes	Further cut	1 Gram	Steam distillation	Mechanical Shaking
2561 2569	Yes Yes	Used as received Further grinded	1g 1 gm	Steam distillation Steam distillation	Mechanical Shaking Mechanical Shaking
2503	Yes	Used as received	1g	Steam distillation	Mechanical Shaking
2590	Yes	Used as received	1g	Steam distillation	Mechanical Shaking
2605	Yes	Further cut	1.000 g	Steam distillation	
2668	Yes	Further cut	0.5 gms	16 hrs KOH extraction at 90 C for 16 hrs	Mechanical Shaking
2695	Yes	Further cut	1g	Steam distillation	Mechanical Shaking
2703	Yes	Used as received	1g	Steam distillation	Other Machanical Shaking
2711 2713	No Yes	Further cut Further cut	1 0,5 g	Steam distillation Ultrasonic Bath	Mechanical Shaking Mechanical Shaking
2713	Yes	Used as received	0,5 g 2g	Extraction with EtOH/Toluene 34:16 under reflux	Other
2727	Yes	Further cut	29 0.5	Steam distillation	Other
2734	Yes	Used as received	3g	Steam distillation	Mechanical Shaking
2737	Yes	Used as received	1g	KOH extraction in oven 90 degree for 16 hours	
2756	Yes	Used as received			Mechanical Shaking
2806	No	Further cut	2 -	 Dimention with KOU	
2977 2979	No No	Used as received Further cut	3 g 1 g	Digestion with KOH Steam distillation	Thermal Desorption
2979 2980	No	Further cut	1 g 1 gram	Steam distillation	Mechanical Shaking Mechanical Shaking
3003	No	Used as received	2.8 gm	Release Techniques: KOH,	Mechanical Shaking
3116	Yes	Used as received	1 gram	Steam distillation was skipped	Ultrasonic
3154	Yes	Further cut	0,5	KOH extracted in water for 16 h at 90 °C	Other
3172					
3210	Yes	Used as received	1g	Ultrasonic, followed by Liq-Liq extraction.	Mechanical Shaking
3218	Yes	Used as received	1g	Steam distillation	Mechanical Shaking
3228 3237	Yes Yes	Further cut Further cut	1 g 2,5g	 Steam distillation	 Mechanical Shaking
3257	Yes	Used as received	2,5y 1	KOH 16h at 90C (DIN50009	Mechanical Shaking
8005	No	Used as received	' 1 gram	Steam distillation	Mechanical Shaking
			-		U U

#### Number of participants per country

4 labs in BANGLADESH 1 lab in BRAZIL 2 labs in CAMBODIA 3 labs in EGYPT 1 lab in ETHIOPIA 1 lab in FRANCE 5 labs in GERMANY 1 lab in GUATEMALA 3 labs in HONG KONG 7 labs in INDIA 2 labs in INDONESIA 9 labs in ITALY 1 lab in KOREA, Republic of 1 lab in MOROCCO 15 labs in P.R. of CHINA 1 lab in PAKISTAN 1 lab in PORTUGAL 2 labs in SWITZERLAND 1 lab in TAIWAN 2 labs in THAILAND 2 labs in TUNISIA 3 labs in TURKEY 2 labs in UNITED KINGDOM

#### Abbreviations

С	= 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
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
<i>c</i>	

fr. = first reported

## Literature

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