

**Results of Proficiency Test
PCP in leather
April 2016**

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Report: iis16A06

June 2016

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1 INTRODUCTION

Products containing Pentachlorophenol (PCP) may form highly toxic substances when they are incinerated. PCP is also a suspected/probable 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.

Pentachlorophenol is an anti-fungal agent that has been restricted for sale in preparations since 1991, with a maximum amount of 1000 mg/kg. However, in some regions (such as Germany), more stringent limits (a maximum of 5 mg/kg) are placed on its inclusion in finished materials and this lower limit is found in 94/783/EC.

The Institute for Interlaboratory Studies organizes since 2004 a scheme of proficiency test for Orthophenylphenol (OPP), Pentachlorophenol (PCP) and Tetrachlorophenols (TeCP) in textile. In the annual proficiency test program of 2015/2016, it was decided to organise a new proficiency test of Pentachlorophenol (PCP) and Tetrachlorophenols (TeCP) on Leather. In the iis16A06 interlaboratory study 76 laboratories in 19 different countries have registered for participation. See appendix 3 for the number of participants per country. In this report, the results of the 2016 proficiency test are presented and discussed. This report is also available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies in Spijkensisse was the organiser of the proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted to an accredited laboratory. Due to limited availability of samples positive on PCP and/or TeCP on leather it was decided to send one leather sample which was positive on PCP. The participants were requested to report the test results using the indicated units and 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 Spijkensisse, 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 organisation 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 April 2014 (iis-protocol, version 3.3). This protocol is electronically available through the iis website site www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

A batch of dark brown leather positive on PCP was obtained from a third party laboratory. The bulk was cut into pieces. Out of this batch, after mixing well, 85 subsamples of 3 grams each were packed and labelled #16545.

The homogeneity of 8 stratified randomly selected samples was checked by determination of PCP by an accredited third party laboratory. The determination is performed in accordance with an in-house test method for PCP. See the following table for the test results.

	<i>PCP in mg/kg</i>
Sample #16545-1	11.71
Sample #16545-2	13.79
Sample #16545-3	13.37
Sample #16545-4	12.36
Sample #16545-5	12.01
Sample #16545-6	11.18
Sample #16545-7	12.05
Sample #16545-8	11.78

Table 1: homogeneity test results of subsamples #16545

From the above test results of the homogeneity test, the repeatability was calculated and compared with 0.3 times the target reproducibility in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	<i>PCP in mg/kg</i>
r (observed)	2.46
Target	iis-memo (lit.18)
0.3 x R (Target)	2.59

Table 2: evaluation of the repeatability of subsamples #16545.

As target reproducibility the reproducibility of PCP on textile (lit. 18) was taken as it was concluded that the determination of PCP in leather is quite similar to PCP in textile.

The calculated repeatability of Pentachlorophenol (PCP) was in agreement with 0.3 times the target reproducibility. Therefore, homogeneity of the subsamples was assumed.

To each participating laboratory one sample of approx. 3 grams, labelled #16545 was sent on March 23, 2016.

2.5 ANALYSES

The participants were asked to determine the concentration of Pentachlorophenol (PCP), 2,3,4,5-Tetrachlorophenol, 2,3,4,6-Tetrachlorophenol and 2,3,5,6-Tetrachlorophenol on sample #16545 applying the analysis procedure that is routinely used in the laboratory.

To get comparable test results a detailed report form, on which the units were prescribed as well as the reference test methods and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/.

A form to confirm receipt of the samples and a letter of instructions were added to the sample package.

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 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 reanalysis). Additional or corrected test results are used for the data analysis and the original test results are placed under 'Remarks' in the test result tables in appendix 1.

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 April 2014 (iis-protocol, version 3.3).

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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the test results should be used with due care.

According to ISO 5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. 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. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

3.2 GRAPHICS

In order to visualise 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 was projected over the Kernel Density Graph for reference.

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. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation of this interlaboratory study. The target standard deviation was calculated from the literature reproducibility by division with 2.8.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated according to:

$$z_{(\text{target})} = (\text{test result} - \text{average of PT}) / \text{target standard deviation}$$

The $z_{(\text{target})}$ 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.

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 serious problems occurred, although three participants reported the test results after the final reporting date and two participants did not report any test results at all. In total 74 laboratories reported 110 numerical test results. Observed were 3 statistical outlying test results, which is 2.7%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

For Pentachlorophenol (PCP), both existing test methods (LFGB 82.02-8 and ISO17070, the latter test method superseding DIN53313:1996 and DIN14494:2003), mention identical precision data for leather only. These precision data for leather are in full agreement with the Horwitz equation and could be used in the calculation of the z-scores. In the proficiency tests of PCP on textile most participating laboratories reported to use the same two test methods for the determination of PCP. In a recent study (lit. 18), in which reproducibilities of this PCP determination on textile over 18 PTs over 10 years were compared, it was concluded that the published reproducibility of these test methods is much too strict and a more realistic target reproducibility had been determined. This target reproducibility had been used to check the homogeneity of the sub samples of #16545 and to calculate the z-scores.

4.1 EVALUATION PER DETERMINATION

In case the test results of a determination proved to be not to have a normal Gaussian distribution the statistical evaluation of this data set should be used with due care. This data set is referred to as "not OK" or "suspect".

PCP: The determination of this component was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated reproducibility calculated from memo of iis (lit. 18). It is remarkable that two labs reported to shake by hand

while method ISO17070 explicitly mentions to use a mechanical shake and not to try shake by hand as this will produce inconsistent results.

2,3,4,5-TeCP: Sample #16545 did contain very little of this component, which concentration was near or below the detection limit. Therefore no significant conclusions were drawn.

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4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the estimated target reproducibilities (see § 4.1) and the reproducibilities as found for the group of participating laboratories.

The number of test results, the average test results, the calculated reproducibilities (standard deviation*2.8) and the target reproducibilities are compared in the next table:

	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 x sd</i>	<i>R (target)</i>
PCP	mg/kg	72	9.4	10.9	6.9
2,3,4,5-TeCP	mg/kg	52	<1.0	n.a.	n.a.
2,3,4,6-TeCP	mg/kg	53	<1.0	n.a.	n.a.
2,3,5,6-TeCP	mg/kg	51	<1.0	n.a.	n.a.

Table 3: reproducibility of phenols on sample #16545

Without further statistical calculations it can be concluded that for PCP the total group of participating laboratories may have difficulties with the analysis. See also the discussion in paragraphs 4.1 and 6.

5 COMPARISON CURRENT PT WITH PREVIOUS PCP PTS ON TEXTILE

This PCP proficiency test on leather was the first PT organised by the Institute for Interlaboratory Studies on leather and therefore no historical PT data was available. However the uncertainty in the PCP determination in this proficiency test was remarkable comparable to the uncertainties as observed in the previous PCP proficiency tests in textile, see next table;

	<i>Leather</i>	<i>Determination of PCP in Textile</i>						
	<i>April 2016</i>	<i>Nov 2015</i>	<i>Nov 2014</i>	<i>Nov 2013</i>	<i>Nov 2012</i>	<i>Nov 2011</i>	<i>Dec 2010</i>	<i>Period 2009-2004</i>
PCP	41%	38%	26%	20%	16-23%	19-20%	15-24%	19-38%

Table 4: Comparison of uncertainties in iis proficiency tests

6 DISCUSSION

In this proficiency test for the determination of phenols in leather, it was noticed that the participants were able to detect PCP in sample #16545. Regretfully, the observed reproducibility was not in agreement with the target reproducibility. It was observed that the way a sample was extracted affected the level PCP determined and the observed reproducibility. Therefore, a comparison of different sample pathways as reported by the participants (appendix 2) is given in table 5.

<i>PCP Determination pathway</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 x sd</i>	<i>R (target)</i>
Overall	mg/kg	72	9.4	10.9	6.9
Steam Distillation only	mg/kg	26	9.3	8.5	6.8
KOH Extraction only	mg/kg	8	12.0	6.8	8.5
Soxhlet/AES extraction only	mg/kg	9	10.1	3.3	7.3
Ultrasonic extraction only	mg/kg	10	9.6	10.8	7.0

Table 5: Comparison of different sample pathways on the PCP determination

<i>PCP Determination method</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 x sd</i>	<i>R (target)</i>
ISO17070 only	mg/kg	36	9.7	9.1	7.0
LFGB B82.02.8 only	mg/kg	9	9.9	13.0	7.2

Table 6: Comparison of reported test methods on the PCP determination

Test methods ISO17070 and LFGB 82.02-8 describe a similar sample pathway (see table 6); steam distillation to extract the phenols from leather; liquid to liquid extraction to get the phenols in a hydrophobic solvent; acetylation of the phenols to separate the phenols easier by the gas chromatograph.

When the steam distillation is substituted by KOH extraction it could extract also components which interfere with the phenols determination, hence the higher results in the PCP determination and lower reproducibility.

Most labs reported to cut the sample prior the extraction, two labs reported to use the sample as received and one lab reported to grind the sample. The reported PCP level by these three labs does not indicate a remarkable effect on the PCP determination.

It is remarkable that the test results from participants that reported to use a Soxhlet/AES extraction showed the smallest calculated reproducibility that is also in agreement with the requirements of ISO17070:2015. Future proficiency tests should prove whether this is coincidentally or systematically.

APPENDIX 1

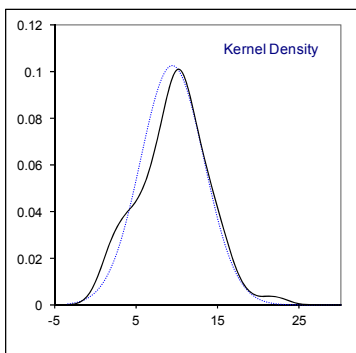
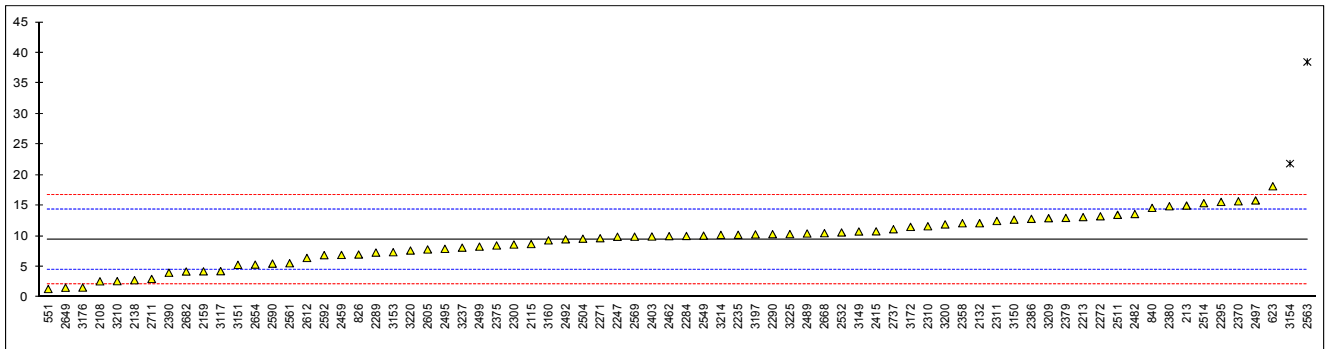
Determination of Pentachlorophenol (PCP) on sample #16545; results in mg/kg

lab	method	value	mark	z(targ)	remarks
213	ISO17070	15		2.28	
551	In house	1.35		-3.28	
623	ISO17070	18.164		3.57	
826	LFGB B82.02.8	6.99		-0.98	
840	ISO17070	14.62		2.13	
2108	ISO17070	2.60		-2.77	
2115	ISO17070	8.708		-0.28	
2132	In house	12.111		1.11	
2138	LFGB B82.02.8	2.7911		-2.69	
2159	LFGB B82.02.8-INH	4.24		-2.10	
2213	ISO17070-INH	13.12		1.52	
2235	ISO17070	10.2171		0.33	
2247	ISO17070	9.9		0.20	
2271	ISO17070	9.66		0.11	
2272	ISO17070	13.2448		1.57	
2284	ISO17070	10.03		0.26	
2289	ISO17070	7.31		-0.85	
2290	ISO17070	10.32		0.38	
2295	In house	15.6		2.53	
2300	ISO17070-INH	8.63	C	-0.31	first reported: 6.18
2310	LFGB B82.02.8	11.62		0.90	
2311	ISO17070	12.48		1.26	
2358	LFGB B82.02.8	12.110		1.10	
2370	LFGB B82.02.8-INH	15.7		2.57	
2375	ISO17070	8.4703		-0.38	
2379	LFGB B82.02.8	12.984		1.46	
2380	LFGB B82.02.8	14.8942		2.24	
2386	In house	12.83		1.40	
2390	In house	4.0152		-2.19	
2403	ISO17070	9.940		0.22	
2415	ISO17070	10.784		0.56	
2459	ISO17070-INH	6.92		-1.01	
2462	ISO17070	10.00		0.24	
2482	In house	13.62		1.72	
2489	ISO17070	10.43		0.42	
2492	In house	9.473		0.03	
2495	ISO17070	7.923		-0.60	
2497	ISO17070	15.84		2.62	
2499	ISO17070	8.281		-0.46	
2504	ISO17070-INH	9.58		0.07	
2511	ISO17070	13.487		1.67	
2514	ISO17070-INH	15.40		2.45	
2532	ISO17070	10.6		0.49	
2549	In house	10.08		0.28	
2561	ISO17070	5.56		-1.56	
2563	ISO17070	38.51	C,R(0.01)	11.86	first reported: 17.555
2569	ISO17070	9.91		0.21	
2590	ISO17070	5.5012		-1.59	
2592	ISO17070	6.89		-1.02	
2605	ISO17070	7.82		-0.64	
2612	ISO17070	6.43		-1.21	
2649	In house	1.56		-3.19	
2654	ISO17070	5.334		-1.66	
2656		----		----	
2668	ISO17070	10.49		0.44	
2682	In house	4.21		-2.11	
2711	CEN/TR 14823	2.99		-2.61	
2737	In house	11.132		0.71	
3117	ISO17070	4.2704		-2.09	
3146		----		----	
3149	In house	10.76		0.55	
3150	ISO17070-INH	12.7		1.35	
3151	In house	5.3000		-1.67	
3153	LFGB B82.02.8	7.37		-0.83	
3154	In house	21.871	R(0.05)	5.08	
3160	ISO17070	9.3020		-0.04	
3172	ISO17070-INH	11.5133		0.86	
3176	ISO17070-INH	1.58	C	-3.19	first reported:0.85
3197	ISO17070	10.28		0.36	
3200	In house	11.92		1.03	
3209	In house	12.933		1.44	
3210	In house	2.632		-2.76	

lab	method	value	mark	z(targ)	remarks
3214	ISO17070	10.20		0.33	
3220	ISO17070-INH	7.64		-0.72	
3225	In house	10.334		0.38	
3237	ISO17070	8.113	C	-0.52	first reported: 1.605

normality OK
 n 72
 outliers 2
 mean (n) 9.40
 st.dev. (n) 3.888
 R(calc.) 10.89
 R(iis memo, lit 18) 6.87

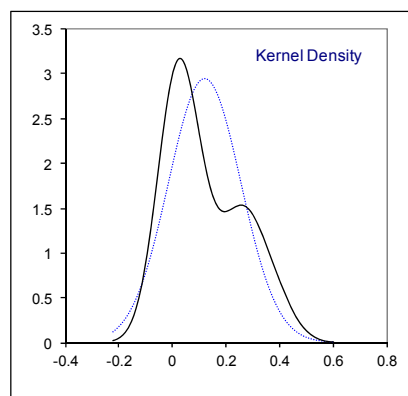
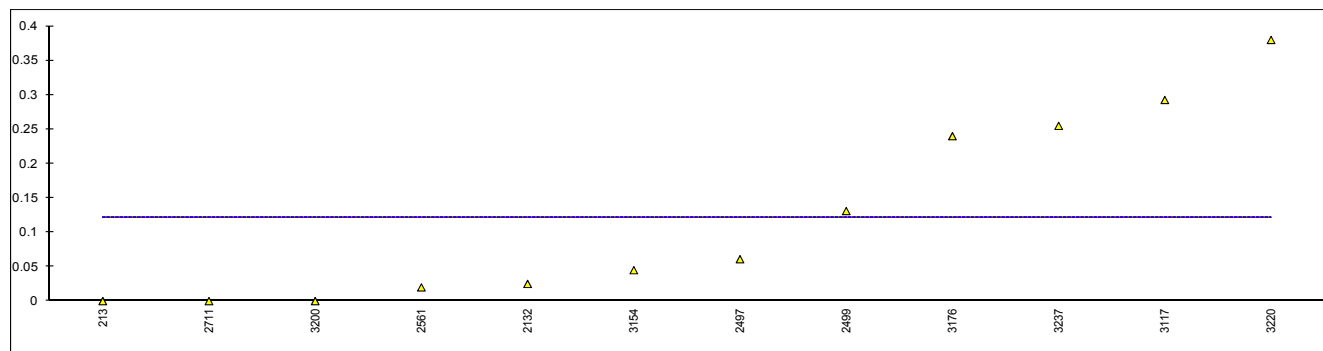
Compare R(ISO17070:2015)=3.16 and R(Horwitz)=3.01



Determination of 2,3,4,5-Tetrachlorophenol on sample #16545; results in mg/kg

lab	method	value	mark	z(targ)	remarks
213	ISO17070	0		----	
551		----		----	
623	ISO17070	n.d.		----	
826	LFGB B82.02.8	n.d.		----	
840	ISO17070	n.d.		----	
2108		----		----	
2115		----		----	
2132	In house	0.025		----	
2138	LFGB B82.02.8	<0.05		----	
2159		----		----	
2213	ISO17070-INH	<0.05		----	
2235		----		----	
2247	ISO17070	n.d.		----	
2271	ISO17070	<0.05		----	
2272		----		----	
2284		----		----	
2289	ISO17070	n.d.		----	
2290	ISO17070	<0.5		----	
2295	In house	n.d.		----	
2300	ISO17070-INH	n.d.	C	----	first reported: 0.41
2310	LFGB B82.02.8	<0.1		----	
2311	ISO17070	n.d.		----	
2358	LFGB B82.02.8	n.d.		----	
2370	LFGB B82.02.8-INH	n.d.		----	
2375		----		----	
2379	LFGB B82.02.8	<0.05		----	
2380	LFGB B82.02.8	n.d.		----	
2386	In house	<0.1		----	
2390	In house	n.d.		----	
2403	ISO17070	n.d.		----	
2415	ISO17070	n.d.		----	
2459	ISO17070-INH	n.d.		----	
2462	ISO17070	n.d.		----	
2482		----		----	
2489	ISO17070	n.d.		----	
2492		----		----	
2495		----		----	
2497	ISO17070	0.061		----	
2499	ISO17070	0.131		----	
2504	ISO17070-INH	<0.1		----	
2511		----		----	
2514		----		----	
2532	ISO17070	n.d.		----	
2549	In house	n.d.		----	
2561	ISO17070	0.02		----	
2563		----		----	
2569	ISO17070	<0.5		----	
2590	ISO17070	<L.O.Q.		----	
2592		----		----	
2605	ISO17070	n.d.		----	
2612	ISO17070	<0.1		----	
2649		----		----	
2654	ISO17070	n.d.		----	
2656		----		----	
2668	ISO17070	n.d.		----	
2682		----		----	
2711	CEN/TR 14823	0		----	
2737		----		----	
3117	ISO17070	0.2926		----	
3146		----		----	
3149		----		----	
3150		----		----	
3151	In house	n.d.		----	
3153		----		----	
3154	In house	0.045		----	
3160		----		----	
3172	ISO17070-INH	<0.05		----	
3176	ISO17070-INH	0.24		----	
3197	ISO17070	n.d.		----	
3200	In house	0		----	
3209	In house	n.d.		----	
3210	In house	<0.500		----	
3214	ISO17070	n.d.		----	
3220	In house	0.38		----	

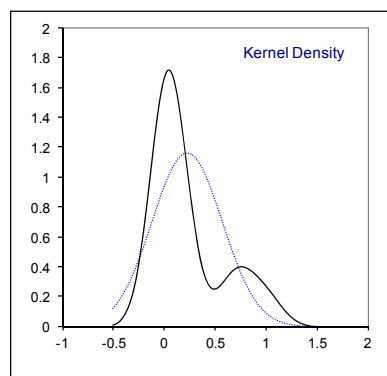
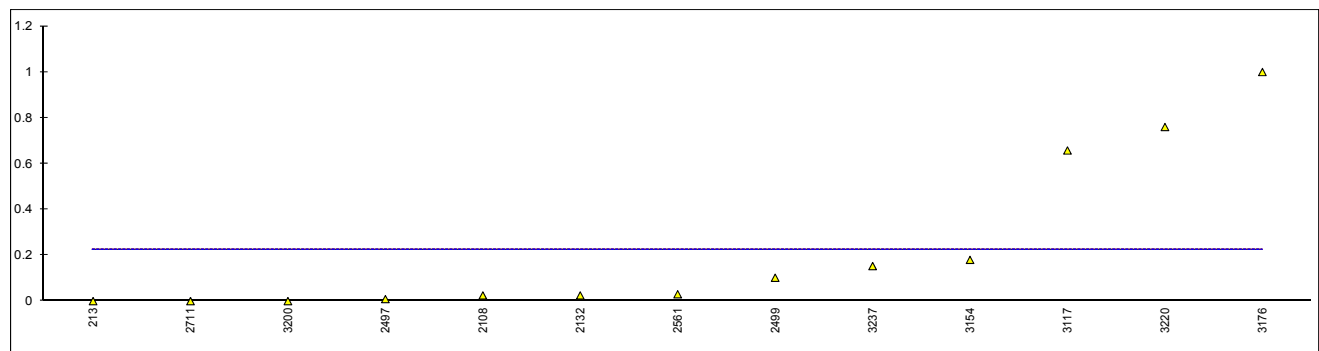
lab	method	value	mark	z(targ)	remarks
3225	In house	n.d.		----	
3237	ISO17070	0.255		----	
	normality	n.a.			
	n	52			
	outliers	n.a.			
	mean (n)	<1.0			12 participants reported a value <0.4
	st.dev. (n)	(0.135)			
	R(calc.)	(0.38)			
	R(Horwitz)	(0.07)			



Determination of 2,3,4,6-Tetrachlorophenol on sample #16545; results in mg/kg

lab	method	value	mark	z(targ)	remarks
213	ISO17070	0		----	
551		----		----	
623	ISO17070	n.d.		----	
826	LFGB B82.02.8	n.d.		----	
840	ISO17070	n.d.		----	
2108	ISO17070	0.025		----	
2115		----		----	
2132	In house	0.025		----	
2138	LFGB B82.02.8	<0.05		----	
2159		----		----	
2213	ISO17070-INH	<0.05		----	
2235		----		----	
2247	ISO17070	n.d.		----	
2271	ISO17070	<0.05		----	
2272		----		----	
2284		----		----	
2289	ISO17070	n.d.		----	
2290	ISO17070	<0.5		----	
2295	In house	n.d.		----	
2300	ISO17070-INH	n.d.	C	----	first reported: 0.6
2310	LFGB B82.02.8	<0.1		----	
2311	ISO17070	n.d.		----	
2358	LFGB B82.02.8	n.d.		----	
2370	LFGB B82.02.8-INH	n.d.		----	
2375		----		----	
2379	LFGB B82.02.8	<0.05		----	
2380	LFGB B82.02.8	n.d.		----	
2386	In house	<0.1		----	
2390	In house	n.d.		----	
2403	ISO17070	n.d.		----	
2415	ISO17070	n.d.		----	
2459	ISO17070-INH	n.d.		----	
2462	ISO17070	n.d.		----	
2482		----		----	
2489	ISO17070	n.d.		----	
2492		----		----	
2495		----		----	
2497	ISO17070	0.009		----	
2499	ISO17070	0.102		----	
2504	ISO17070-INH	<0.1		----	
2511		----		----	
2514		----		----	
2532	ISO17070	n.d.		----	
2549	In house	n.d.		----	
2561	ISO17070	0.03		----	
2563		----		----	
2569	ISO17070	<0.5		----	
2590	ISO17070	<L.O.Q.		----	
2592		----		----	
2605	ISO17070	n.d.		----	
2612	ISO17070	<0.1		----	
2649		----		----	
2654	ISO17070	n.d.		----	
2656		----		----	
2668	ISO17070	n.d.		----	
2682		----		----	
2711	CEN/TR 14823	0		----	
2737		----		----	
3117	ISO17070	0.6574		----	
3146		----		----	
3149		----		----	
3150		----		----	
3151	In house	n.d.		----	
3153		----		----	
3154	In house	0.180		----	
3160		----		----	
3172	ISO17070-INH	<0.05		----	
3176	ISO17070-INH	1.0	C	----	first reported: 0.55
3197	ISO17070	n.d.		----	
3200	In house	0		----	
3209	In house	n.d.		----	
3210	In house	<0.500		----	
3214	ISO17070	n.d.		----	
3220	In house	0.76		----	

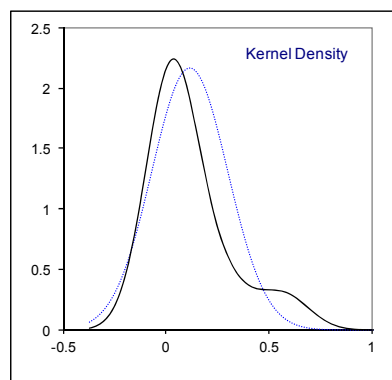
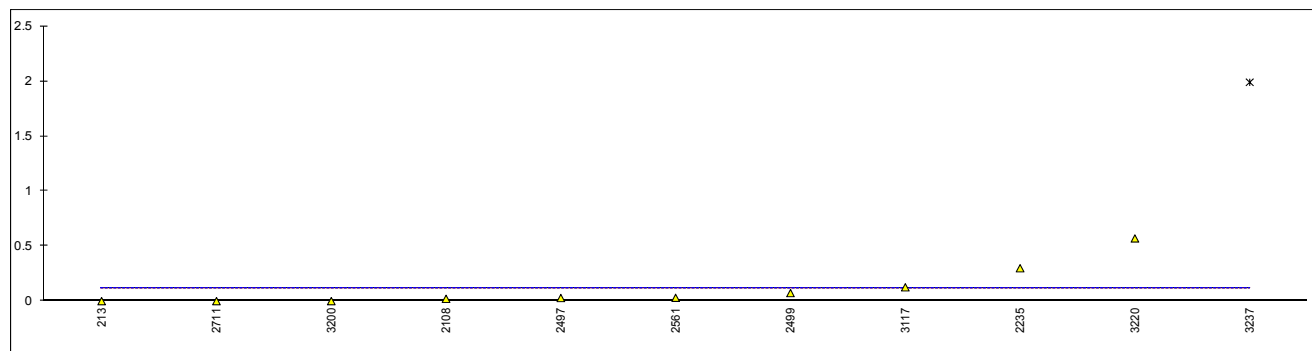
lab	method	value	mark	z(targ)	remarks
3225	In house	n.d.		----	
3237	ISO17070	0.153		----	
	normality	n.a.			
	n	53			
	outliers	n.a.			
	mean (n)	< 1.0			13 participants reported a value ≤ 1.0
	st.dev. (n)	(0.343)			
	R(calc.)	(0.96)			
	R(Horwitz)	(0.10)			



Determination of 2,3,5,6-Tetrachlorophenol on sample #16545; results in mg/kg

lab	method	value	mark	z(targ)	remarks
213	ISO17070	0		----	
551		----		----	
623	ISO17070	n.d.		----	
826	LFGB B82.02.8	n.d.		----	
840	ISO17070	n.d.		----	
2108	ISO17070	0.021		----	
2115		----		----	
2132	In house	<0.01		----	
2138	LFGB B82.02.8	<0.05		----	
2159		----		----	
2213	ISO17070-INH	<0.05		----	
2235	ISO17070	0.2990		----	
2247	ISO17070	n.d.		----	
2271	ISO17070	<0.05		----	
2272		----		----	
2284		----		----	
2289	ISO17070	n.d.		----	
2290	ISO17070	<0.5		----	
2295	In house	n.d.		----	
2300	ISO17070-INH	n.d.		----	
2310	LFGB B82.02.8	<0.1		----	
2311	ISO17070	n.d.		----	
2358	LFGB B82.02.8	n.d.		----	
2370	LFGB B82.02.8-INH	n.d.		----	
2375		----		----	
2379	LFGB B82.02.8	<0.05		----	
2380	LFGB B82.02.8	n.d.		----	
2386	In house	<0.1		----	
2390	In house	n.d.		----	
2403	ISO17070	n.d.		----	
2415	ISO17070	n.d.		----	
2459	ISO17070-INH	n.d.		----	
2462	ISO17070	n.d.		----	
2482		----		----	
2489	ISO17070	n.d.		----	
2492		----		----	
2495		----		----	
2497	ISO17070	0.028		----	
2499	ISO17070	0.073		----	
2504	ISO17070-INH	<0.1		----	
2511		----		----	
2514		----		----	
2532	ISO17070	n.d.		----	
2549	In house	n.d.		----	
2561	ISO17070	0.03		----	
2563		----		----	
2569	ISO17070	<0.5		----	
2590	ISO17070	<L.O.Q.		----	
2592		----		----	
2605	ISO17070	n.d.		----	
2612	ISO17070	<0.1		----	
2649		----		----	
2654	ISO17070	n.d.		----	
2656		----		----	
2668	ISO17070	n.d.		----	
2682		----		----	
2711	CEN/TR 14823	0		----	
2737		----		----	
3117	ISO17070	0.1257		----	
3146		----		----	
3149		----		----	
3150		----		----	
3151	In house	n.d.		----	
3153		----		----	
3154		----		----	
3160		----		----	
3172	ISO17070-INH	<0.05		----	
3176		----		----	
3197	ISO17070	n.d.		----	
3200	In house	0		----	
3209	In house	n.d.		----	
3210	In house	<0.500		----	
3214	ISO17070	n.d.		----	
3220	In house	0.57		----	

lab	method	value	mark	z(targ)	remarks
3225	In house	n.d.		----	
3237	ISO17070	1.99	C, D(0.01)	----	first reported: 0.796, possibly a false positive test result?
	normality	n.a.			
	n	51			
	outliers	(1)			
	mean (n)	< 1.0			10 participants reported a value <0.6
	st.dev. (n)	(0.184)			
	R(calc.)	(0.52)			
	R(Horwitz)	(0.07)			



APPENDIX 2

Details of the test methods used by the participants

lab	Cut/Grinded	Size	How to release the PCP	How to extract the PCP	How to shake the liquid/liquid extraction	Acetylation
213	Cut	3 x 3 mm	Steam distillation	Ultrasonic extraction	Mechanical shaker	Yes
551	Cut	---	Steam distillation	---	Mechanical shaker	Yes
623	Cut	3 x 3 mm	Steam distillation was skipped	Ultrasonic extraction	Mechanical shaker	Yes
826	Cut	3 x 3 mm	Steam distillation	Ultrasonic extraction	Mechanical shaker	Yes
840	Cut	3 x 3 mm	Steam distillation	---	Mechanical shaker	Yes
2108	Cut	---	Steam distillation	---	Mechanical shaker	Yes
2115	Cut	2 mm	Steam distillation	---	Mechanical shaker	Yes
2132	Cut	5 x 5 mm	Ultrasonic extraction	Ultrasonic extraction	Mechanical shaker	Yes
2138	Cut	5 x 5 mm	Steam distillation was skipped	Ultrasonic extraction	Mechanical shaker	Yes
2159	Cut	5 x 5 mm	---	Ultrasonic extraction	Mechanical shaker	Yes
2213	Cut	2-3 mm	KOH extraction	see answer left	Mechanical shaker	Yes
2235	Cut	2-3 mm	Steam distillation	Liquid / Liquid Extraction	Mechanical shaker	Yes
2247	Cut	3-4 mm	Steam distillation	---	Mechanical shaker	Yes
2271	Cut	2 x 2 mm	Steam distillation was skipped	Ultrasonic extraction	Mechanical shaker	Yes
2272	Cut	5 x 5 mm	---	Ultrasonic extraction	Mechanical shaker	Yes
2284	Cut	3 x 3 mm	---	---	Mechanical shaker	Yes
2289	Cut	2 mm	Steam distillation	Soxhlet / AES extraction	Mechanical shaker	Yes
2290	As received	---	---	---	---	No
2295	Cut	---	---	---	Mechanical shaker	No
2300	Cut	5 mm	Soxtherm Extraction	see answer left	Mechanical shaker	Yes
2310	Cut	3 x 3 mm	Steam distillation	Soxhlet / AES extraction	Mechanical shaker	Yes
2311	Cut	3 x 3 mm	Steam distillation	---	Mechanical shaker	Yes
2358	Cut	5 x 5 mm	Steam distillation	Liquid / Liquid Extraction	Mechanical shaker	Yes
2370	Cut	3 x 3 mm	Steam distillation	Steam distillation	Mechanical shaker	No
2375	Cut	3 x 3 mm	Steam distillation	Steam Distillation	Mechanical shaker	Yes
2379	Cut	6 x 6 mm	Steam distillation	Liquid / Liquid Extraction	Mechanical shaker	Yes
2380	Cut	3 x 3 mm	Alkaline digestion was used	see answer left	Mechanical shaker	Yes
2386	Cut	5 x 5 mm	---	Ultrasonic extraction	Mechanical shaker	Yes
2390	Cut	3-4 mm	---	Ultrasonic extraction	No liquid/liquid extraction	No
2403	Cut	2 x 2 mm	Steam distillation	Soxhlet / AES extraction	Mechanical shaker	Yes
2415	Cut	1 mm	Incubating at 90°C for 12 hrs.	Liquid / Liquid Extraction	Mechanical shaker	Yes
2459	Cut	---	Steam distillation	Ultrasonic extraction	Mechanical shaker	Yes
2462	Cut	20	Steam distillation	---	Mechanical shaker	Yes
2482	Cut	---	KOH extraction	see answer left	Mechanical shaker	Yes
2489	Cut	2 x 2 mm	Steam distillation	Soxhlet / AES extraction	Mechanical shaker	Yes
2492	Cut	0.5 mm	---	Soxhlet / AES extraction	Mechanical shaker	Yes
2495	Cut	3 x 3 mm	Steam distillation	---	Mechanical shaker	Yes
2497	Cut	2 mm	Steam distillation	Ultrasonic extraction	Mechanical shaker	Yes
2499	Cut	2 x 2 mm	Steam distillation	---	Mechanical shaker	Yes
2504	Cut	2 x 2 mm	Heating Block	see answer left	Mechanical shaker	Yes
2511	Cut	2-3 mm	---	---	Mechanical shaker	Yes
2514	Cut	2 x 2 mm	KOH extraction	---	Mechanical shaker	Yes
2532	Cut	---	Steam distillation	Soxhlet / AES extraction	Mechanical shaker	Yes
2549	Cut	3 x 3 mm	Incubation	KOH extraction	Mechanical shaker	Yes
2561	Cut	5 x 5 mm	Steam distillation	---	Mechanical shaker	Yes
2563	Cut	3 x 3 mm	---	Soxhlet / AES extraction	Mechanical shaker	Yes
2569	Cut	---	Steam distillation	---	Mechanical shaker	Yes
2590	Cut	2 x 2 mm	Steam distillation was skipped	Ultrasonic extraction	Mechanical shaker	Yes
2592	Cut	< 4 mm	Steam distillation	Ultrasonic extraction	Mechanical shaker	Yes
2605	Cut	5	Steam distillation	---	Mechanical shaker	Yes
2612	Cut	2 x 2 mm	Steam distillation	---	Mechanical shaker	Yes
2649	Cut	5 x 5 mm	---	Ultrasonic extraction	No liquid/liquid extraction	Yes
2654	Grinded	0.8 mm	Steam distillation	Liquid / Liquid Extraction	Mechanical shaker	Yes
2656	---	---	---	---	---	---
2668	Cut	2 x 2 mm	KOH extraction	Ultrasonic extraction	Mechanical shaker	Yes
2682	Cut	2 mm	Steam distillation was skipped	Soxhlet / AES extraction	Shaked by hand	Yes
2711	Cut	---	---	Ultrasonic extraction	Mechanical shaker	Yes
2737	Cut	3 x 3 mm	---	Ultrasonic extraction	Mechanical shaker	Yes
3117	Cut	---	Steam distillation	---	Mechanical shaker	Yes
3146	---	---	---	---	---	---
3149	Cut	3 x 3 mm	---	Soxhlet / AES extraction	Mechanical shaker	Yes
3150	Cut	3 x 3 mm	KOH extraction	see answer left	Mechanical shaker	Yes
3151	Cut	5 x 5 mm	---	Ultrasonic extraction	Mechanical shaker	Yes
3153	As received	2 x 2 mm	Steam distillation	Liquid / Liquid Extraction	Mechanical shaker	Yes
3154	Cut	---	---	Ultrasonic extraction	Mechanical shaker	Yes
3160	Cut	2 x 2 mm	Steam distillation	---	Mechanical shaker	Yes
3172	Cut	5 x 5 mm	---	Ultrasonic extraction	Mechanical shaker	Yes
3176	Cut	5 x 5 mm	---	Ultrasonic extraction	Shaked by hand	Yes
3197	Cut	2-3 mm	Steam distillation	Soxhlet / AES extraction	Mechanical shaker	Yes
3200	Cut	3 x 3 mm	---	Ultrasonic extraction	Mechanical shaker	Yes
3209	Cut	2 x 2 mm	KOH extraction, 12 hrs at 90°C.	see answer left	Mechanical shaker	Yes
3210	Cut	< 25 mm ²	Steam distillation was skipped	Ultrasonic extraction	Mechanical shaker	Yes

lab	Cut/Grinded	Size	How to release the PCP	How to extract the PCP	How to shake the liquid/liquid extraction	Acetylation
3214	Cut	2 x 2 mm	Steam distillation	Soxhlet / AES extraction	Mechanical shaker	Yes
3220	Cut	10 mm ²	KOH extraction	Ultrasonic extraction	Mechanical shaker	Yes
3225	Cut	3 x 3 mm	---	Ultrasonic extraction	Mechanical shaker	Yes
3237	Cut	2 x 2 mm	Steam distillation	---	Mechanical shaker	Yes

APPENDIX 3

Number of participants per country

3 labs in BANGLADESH
1 lab in BRAZIL
2 labs in FRANCE
10 labs in GERMANY
5 labs in HONG KONG
11 labs in INDIA
1 lab in INDONESIA
9 labs in ITALY
2 labs in KOREA
2 labs in MOROCCO
11 labs in P.R. of CHINA
3 labs in PAKISTAN
1 lab in SPAIN
2 labs in TAIWAN R.O.C.
2 labs in THAILAND
1 lab in TUNISIA
6 labs in TURKEY
2 labs in UNITED KINGDOM
3 labs in VIETNAM

APPENDIX 4

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 outlier test
R(0.05)	= straggler in Rosner outlier test
ex	= test result withdrawn on request of participant
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected

Literature:

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- 11 ISO14184-1:1994
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