Results of Proficiency Test
Chlorinated Phenols
in Leather/Footwear
May 2021

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. C.M. Nijssen-Wester
Correctors: ing. R.J. Starink & ing. G.A. Oosterlaken-Buijs
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CONTENTS

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 OekoTex® and Bluesign®.

Since 2016 the Institute for Interlaboratory Studies (iis) organizes a proficiency test for the determination of Pentachlorophenol (PCP) and Tetrachlorophenols (TeCPs) in Leather/Footwear every year. In 2018 the scope of the scheme was extended with Trichlorophenols (TrCPs). During the annual proficiency testing program 2020/2021 it was decided to continue the proficiency test for the analysis of Chlorinated Phenols in Leather/Footwear.

In this interlaboratory study 72 laboratories in 21 different countries registered for participation. See appendix 4 for the number of participants per country. In this report the results of this 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. 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 of 3 grams labelled #21585.
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 dark brown leather positive on Pentachlorophenol (PCP) was selected. After homogenization 100 small bags were filled with approximately 3 grams each and labelled #21585.
The batch for sample #21585 was used in a previous proficiency test on Chlorinated Phenols in Leather/Footwear (as sample #16545 in iis16A06). Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample labelled #21585 was sent on April 14, 2021.

2.5 ANALYZES

The participants were requested to determine: Pentachlorophenol (PCP), 2,3,4,5-Tetrachlorophenol, 2,3,4,6-Tetrachlorophenol, 2,3,5,6-Tetrachlorophenol, 2,3,4-Trichlorophenol, 2,3,5-Trichlorophenol, 2,3,6-Trichlorophenol, 2,4,5-Trichlorophenol, 2,4,6-Trichlorophenol, 3,4,5-Trichlorophenol and other Chlorinated 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 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 in appendix 1 and 2 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no re-analyzes). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the 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 Organization, Statistics and Evaluation’ of June 2018 (iis-protocol, version 3.5).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as ‘<…’ or ‘>…” were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either ‘unknown’, ‘OK’, ‘suspect’ or ‘not OK’. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner’s outlier test can be used. Outliers are marked by D(0.01) for the Dixon’s test, by G(0.01) or DG(0.01) for the Grubbs’ test and by R(0.01) for the Rosner’s test. Stragglers are marked by D(0.05) for the Dixon’s test, by G(0.05) or DG(0.05) for the Grubbs’ test and by R(0.05) for the Rosner’s test. Both outliers and stragglers were not included in the calculations of the averages and the 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. The Kernel Density Graph 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 proficienct test (PT) against the literature requirements, 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 may be 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 results 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 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

In this interlaboratory study no problems were encountered with the dispatch of the samples. Ten participants reported test results after the final reporting date and two participants did not report any test results at all. Not all participants were able to report all components requested.
In total 70 participants reported 70 numerical results. Observed was 1 outlying test result, which is 1.4% of the numerical results. In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

The data set 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. The abbreviations, used in these tables, are listed in appendix 5.

The test methods LFGB 82.02-8 and ISO17070:15 mention identical precision data for Pentachlorophenol (PCP). Most participating laboratories in this PT reported to have used one of these two test methods. In iis memo 1601, in which the reproducibilities of the PCP determination on textile over 18 PTs from 2004 until 2014 were compared, it was concluded that the published reproducibility of these test methods is in practice too strict and a more realistic target reproducibility was determined. As it was assumed that the variation in the PT test results will be dependent on the concentration, this resulted in a Horwitz-like equation to estimate the target reproducibilities for the evaluation of the PT test results by iis from 2015 onwards (iis memo 1601, see lit.16). Although iis memo 1601 is based on previous iis PTs of PCP in Textile and not based on iis PTs of PCP in Leather/Footwear, it was decided to use the estimated iis target reproducibility of PCP both in textile PTs and leather PTs. It was also decided to use the estimated iis target reproducibility of PCP also for other Chlorinated Phenols components.

PCP: The determination of this component was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the target reproducibility derived from the reproducibilities observed in previous iis PTs, iis memo 1601.

The majority of the participants agreed on a concentration near or below the limit of detection for all other TeCPs and TCPs mentioned in paragraph 2.5. Therefore, no z-scores were calculated for these Chlorinated Phenols.
Seven participants reported also the presence of other Chlorinated Phenols at different concentration levels (see appendix 2).

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the estimated target reproducibility based on former iis proficiency tests 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 based on previous proficiency tests are presented in the next table.

Component	unit	n	average	2.8 * sd	R(target)
PCP	mg/kg	69	7.94	6.99	5.96

Table 1: reproducibility of tests on sample #21585

Without further statistical calculations, it could be concluded that for PCP there is almost a good compliance of the group of participating laboratories with the target.

4.3 COMPARISON OF THE PROFICIENCY TEST OF MAY 2021 WITH PREVIOUS PTS

	May 2021	May 2020	May 2019	April 2018	April 2017
Number of reporting laboratories	70	65	73	72	72
Number of test results	70	125	205	127	107
Number of statistical outliers	1	2	4	4	2
Percentage of statistical outliers	1.4%	1.6%	2.0%	3.1%	1.9%

Table 2: 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.

	May 2021	May 2020	May 2019	2016 – 2018	Target 1.3 – 13 mg/kg
PCP	31%	21%	26%	26 - 41%	35 - 25%
2,3,4,5-TeCP	n.e.	18%	n.e.	n.e.	35 – 25%
2,3,5,6-TeCP	n.e.	n.e.	26%	n.e.	35 - 25%
2,4,6-TCP	n.e.	n.e.	34%	29%	35 - 25%

Table 3: development of the uncertainties over the years

The uncertainty for PCP observed in this PT has not improved when compared with the previous PTs.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

The test method ISO17070 is used by about 70% of the reporting participants and test method LFGB B82.02.8 is used by than 10% of the reporting participants. Test methods ISO17070 and LFGB 82.02-8 describe a similar sample pathway to determine PCP: steam distillation to extract the phenols from leather, liquid to liquid extraction to get the phenols in a hydrophobic solvent and acetylation of the phenols (with a mechanical shaker) to separate the phenols easier by the gas chromatograph.

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

- About 90% of the reporting participants mentioned that they are accredited for the determination of the reported components.
- About 25% of the reporting participants used the sample as received and about 75% of the reporting participants did further cut or grind the sample.
- About 85% of the reporting participants used a sample intake between 0.5 - 1 grams and about 15% used more than 1 grams as sample intake.
- About 65% of the reporting participants used Steam distillation as technique to release the Chlorinated Phenols and about 10% reported to have skipped the Steam distillation.
- About 25% of the reporting participants used Ultrasonic extraction and about 50% used Mechanical Shaking as technique to extract the Chlorinated Phenols.

When the analytical details were investigated separately, it appeared that the effect on the determination of Chlorinated Phenols in Leather/Footwear is negligible.

5 DISCUSSION

In table 6 the limits mentioned in Oeko-Tex® Leather Standard are mentioned. It was noticed that all participants would make identical decisions about the acceptability of the leather for Chlorophenols.

Chlorinated Phenols in mg/kg	Class I Baby	Class II Direct skin contact	Class III No direct skin contact	Class IV Decoration material
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 4: Product classes specific limit values, Oeko-Tex® Leather Standard

Chlorinated Phenols in mg/kg	Class A Next to skin and Baby	Class B Occasional skin contact	Class C No skin contact
Pentachlorophenol (PCP), sum isomers	<0.5	<0.5	<0.5
Tetrachlorophenols (TeCP), sum isomers	<0.5	<0.5	<0.5
Trichlorophenols (TrCP), sum isomers	<0.5	<0.5	<0.5

Table 5: Product classes specific limit values, Bluesign® RSL list

For the determination of PCP, all participants would reject the sample for all classes.

Sample #21585 was used earlier as sample #16545 in the PT iis16A06 (2016). In table 6 a comparison is given over the two proficiency tests.

	Sample #21585				Sample #16545			
	unit	n	average	R(calc)	unit	n	average	R(calc)
PCP	mg/kg	69	7.94	6.99	mg/kg	72	9.40	10.89

Table 6: comparison of sample #21585 with #16545

It is observed that the average level of PCP in the 2021 PT is lower and the observed reproducibility R(calc) for PCP has improved significantly in 2021 compared to the 2016 PT. The variation in the test results of the participants in 2021 is smaller than in 2016. This is not uncommon. Each time that a laboratory participates in a PT it has the opportunity to learn from the evaluation of the results and improve the analysis. The average and smaller variation of the 2021 PT are within the average and variation of the 2016 PT and therefore comparable.

6 CONCLUSION

The majority of the participants has no problem with the determination of Pentachlorophenol in Leather/Footwear. Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary.

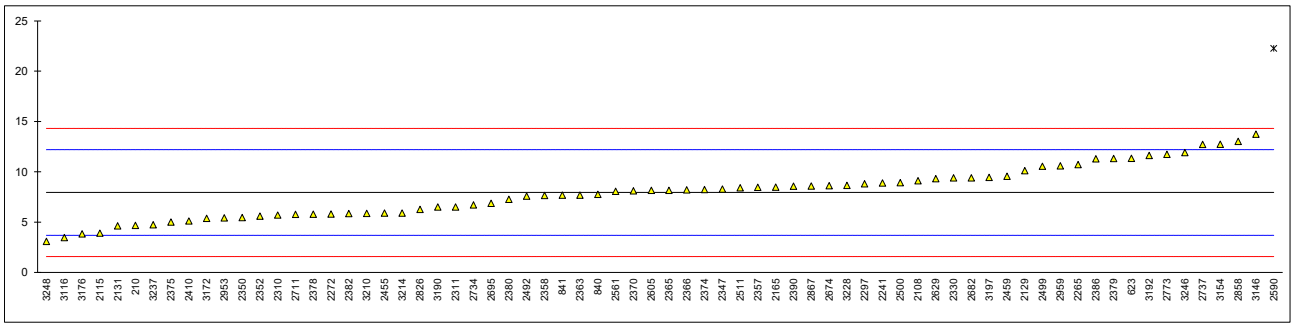
Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

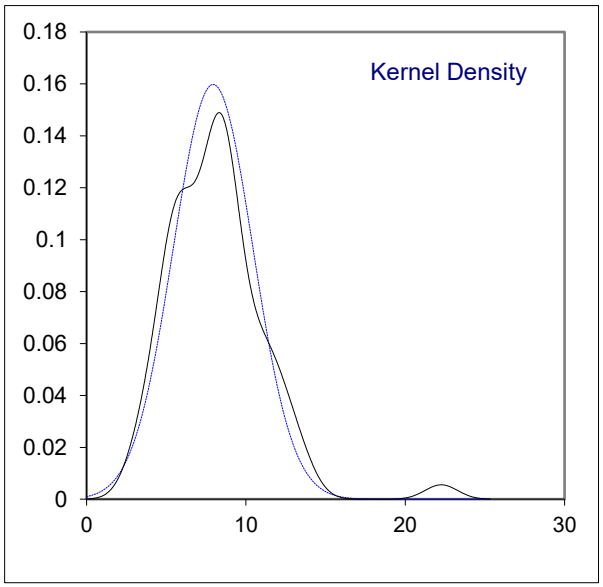
APPENDIX 1

Determination of Pentachlorophenol (PCP) on sample #21585; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	ISO17070	4.67		-1.54	
623	LFGB B82.02.8	11.334	C	1.59	first reported: 15.131
840	ISO17070	7.75		-0.09	
841	ISO17070	7.679		-0.12	
2108	In house	9.1033		0.54	
2115	ISO17070	3.90		-1.90	
2129	ISO17070	10.10		1.01	
2131	OEKO-TEX M-7	4.62		-1.56	
2165	ISO17070	8.47		0.25	
2241	ISO17070	8.88		0.44	
2265	ISO17070	10.712		1.30	
2272	ISO17070	5.80		-1.01	
2297	ISO17070	8.81		0.41	
2310	ISO17070	5.69		-1.06	
2311	ISO17070	6.5002		-0.68	
2330	ISO17070	9.399		0.68	
2347	ISO17070	8.28		0.16	
2350	LFGB B82.02.8	5.4566		-1.17	
2352	LFGB B82.02.8	5.585		-1.11	
2357	ISO17070	8.460		0.24	
2358	ISO17070	7.6401		-0.14	
2363	ISO17070	7.68		-0.12	
2365	ISO17070	8.1612		0.10	
2366	ISO17070	8.20		0.12	
2370	ISO17070	8.10		0.07	
2374	ISO17070	8.231		0.13	
2375	ISO17070	5.0		-1.38	
2378	GB/T22808	5.79		-1.01	
2379	ISO17070	11.3175		1.59	
2380	ISO17070	7.269		-0.32	
2382	LFGB B82.02.8	5.85		-0.98	
2386	In house	11.28		1.57	
2390	ISO17070	8.57		0.29	
2410	ISO17070	5.1		-1.34	
2455	ISO17070	5.895		-0.96	
2459	ISO17070	9.5430		0.75	
2492	In house	7.577		-0.17	
2499	ISO17070	10.5441		1.22	
2500	ISO17070	8.912		0.45	
2511	ISO17070	8.4		0.21	
2561	ISO17070	8.057		0.05	
2582		-----		-----	
2590		22.263	C,R(0.01)	6.73	first reported: 28.587
2605	ISO17070	8.151		0.10	
2629	ISO17070	9.31		0.64	
2674	ISO17070	8.61		0.31	
2682		9.40		0.68	
2695	ISO17070	6.86		-0.51	
2711	ISO17070	5.77		-1.02	
2734	ISO17070	6.7		-0.59	
2737	ISO17070	12.72		2.24	
2773	ISO17070	11.72		1.77	
2806		-----		-----	
2826	ISO17070	6.2537		-0.79	
2858	In house	13.03		2.39	
2867	ISO17070	8.58		0.30	
2953	ISO17070	5.42		-1.19	
2959	In house	10.584		1.24	
3116	LFGB B82.02.8	3.462		-2.11	
3146	DIN50009	13.739		2.72	
3154	In house	12.73	C	2.25	first reported: 0.3
3172	ISO17070	5.3586		-1.22	
3176	In house	3.815		-1.94	
3190	ISO17070	6.50		-0.68	
3192	In house	11.62		1.73	
3197	ISO17070	9.44		0.70	
3210	In house	5.86		-0.98	
3214	LFGB B82.02.8	5.898		-0.96	
3228	ISO17070	8.64		0.33	
3237	ISO17070	4.73		-1.51	
3246		11.90		1.86	
3248	In house	3.08		-2.29	

normality	OK	
n	69	
outliers	1	
mean (n)	7.9449	
st.dev. (n)	2.49668	RSD = 31%
R(calc.)	6.9907	
st.dev.(iis memo 1601)	2.12732	
R(iis memo 1601)	5.9565	





APPENDIX 2

Summary of all other reported Chlorinated Phenols on sample #21585; results in mg/kg

lab	Other reported Chlorinated Phenols
2129	4-Chloro-3-methylphenol = 21.14
2265	Other: 0.132
2455	3,4,5-Trichlorophenol = 0.536
2826	2,3,4,5-Tetrachlorophenol = 0.1595, Other: 0.7340
2953	4-Chloro-3-methylphenol = 137.1
3146	Other: 7.340
3248	3,4,5-Trichlorophenol = 0.34

APPENDIX 3 Analytical Details

lab	ISO17025 accredited	Sample preparation	Sample intake (g)	Release technique	Extraction technique
210	Yes	---		---	---
623	Yes	Further cut	1	---	Ultrasonic
840	Yes	Further cut	1	Steam distillation	Mechanical Shaking
841	Yes	Further cut	1	Steam distillation	Mechanical Shaking
2108	Yes	Used as received	1	Steam distillation	---
2115	Yes	Used as received	1	---	---
2129	Yes	Used as received	0,5	KOH-method	Ultrasonic
2131	Yes	Used as received	1	Microwave extr. with KOH	---
2165	Yes	Further cut	2	Steam distillation	---
2241	Yes	Further cut	0.3	Steam distillation	Mechanical Shaking
2265	No	Further cut	0,5	90°C, 16h	---
2272	Yes	Further cut	1	Steam distillation was skipped	Ultrasonic
2297	Yes	Used as received	1	Steam distillation	Mechanical Shaking
2310	Yes	Further cut	3mm*3mm	Steam distillation	Mechanical Shaking
2311	Yes	Further cut	1	Steam distillation	Mechanical Shaking
2330	No	Further cut	0.5	Steam distillation	Mechanical Shaking
2347	Yes	Further cut	1	---	Mechanical Shaking
2350	No	Further cut	2.0083	Steam distillation	ASE
2352	Yes	Further cut	0.5	Steam distillation	Mechanical Shaking
2357	---	---		---	---
2358	Yes	Used as received	1	Steam distillation	Mechanical Shaking
2363	Yes	Further cut	1	Steam distillation	---
2365	Yes	Further cut	2.0	Steam distillation	Thermal Desorption
2366	Yes	Further cut	0.5	Steam distillation	Steam distillation
2370	Yes	Further cut	2	Steam distillation	Distillation
2374	Yes	Further cut	1	Steam distillation	Mechanical Shaking
2375	Yes	---		---	---
2378	Yes	Further cut	1	Steam distillation	Mechanical Shaking
2379	No	Further cut	1	Steam distillation	Liquid liquid extraction
2380	Yes	Further cut	1.0	Steam distillation	Steam distillation
2382	Yes	Further cut	0.5	Steam distillation	Ultrasonic
2386	Yes	Further cut	0,5	Steam distillation was skipped	Ultrasonic
2390	Yes	Used as received	1.0094	Alkaline digestion	Ultrasonic
2410	Yes	Used as received	1.0	Steam distillation was skipped	Soxhlet
2455	Yes	Further cut	1.0	Steam distillation	Mechanical Shaking
2459	Yes	Further cut	1	Ultrasonic	Ultrasonic
2492	Yes	Used as received	0.5	Steam distillation	Mechanical Shaking
2499	Yes	Further cut	0.7	Steam distillation	Mechanical Shaking
2500	Yes	Used as received	2	Steam distillation was skipped	Ultrasonic
2511	---	---		---	---
2561	Yes	Used as received	1	Steam distillation	---
2582	---	---		---	---
2590	Yes	Further cut	1	Steam distillation	Mechanical Shaking
2605	Yes	Further cut	2	Steam distillation	Distillation
2629	Yes	Further grinded	0.5	Steam distillation	---
2674	Yes	Further cut	2.0	Steam distillation was skipped	Mechanical Shaking
2682	---	---		---	---
2695	Yes	Further cut	1,0286	Steam distillation	Mechanical Shaking
2711	No	Further cut	0.828	Steam distillation	Mechanical Shaking
2734	Yes	Further cut	2.5	Steam distillation	Mechanical Shaking
2737	Yes	Used as received	1	KOH extraction in oven 90°/16h	---
2773	Yes	Further cut	2.0000	KOH extr. 16 hrs/90℃ then extr. with n-hexane	---
2806	---	---		---	---
2826	Yes	Further cut	1	Steam distillation	Mechanical Shaking
2858	Yes	Used as received	1.0441	Dry cabinet chamber.	Thermal Desorption
2867	Yes	Further cut	0.5	With 1 mol/L KOH in oven at 90°/16h	Ultrasonic
2953	No	Further cut	1	Steam distillation	---
2959	No	Further cut	0.5	KOH Extraction, 90℃, 16h	---
3116	Yes	Used as received	1	---	Ultrasonic
3146	Yes	Further cut	0.5	Digestion with KOH	Mechanical Shaking
3154	Yes	Further cut	0,5	---	---
3172	---	---		---	---
3176	Yes	Further cut	1	Steam distillation was skipped	Ultrasonic
3190	Yes	Further cut	1	Steam distillation	Mechanical Shaking
3192	Yes	Further cut	0,5	1 M KOH for 16h in oven at 90°C	---
3197	Yes	Used as received	1	Steam distillation	Soxhlet
3210	Yes	Further cut	1	Ultrasonic 2h then extr. with hexane	Ultrasonic
3214	Yes	Further cut	1	Steam distillation	Thermal Desorption
3228	Yes	Further cut	2.5	Steam distillation	Mechanical Shaking
3237	Yes	Used as received	0,5	Steam distillation	Mechanical Shaking
3246	Yes	Used as received	0.5	KOH 1M in 16h at 90°C then extr. with n-hexane	Mechanical Shaking
3248	Yes	Further cut	1	Steam distillation was skipped	Mechanical Shaking

APPENDIX 4

Number of participants per country

2 labs in BANGLADESH
1 lab in CAMBODIA
1 lab in FRANCE
7 labs in GERMANY
5 labs in HONG KONG
3 labs in INDIA
1 lab in INDONESIA
9 labs in ITALY
1 lab in MOROCCO
21 labs in P.R. of CHINA
3 labs in PAKISTAN
2 labs in SOUTH KOREA
1 lab in SRI LANKA
1 lab in SWITZERLAND
2 labs in TAIWAN
1 lab in THAILAND
1 lab in TUNISIA
4 labs in TURKEY
1 lab in U.S.A.
1 lab in UNITED KINGDOM
4 labs in VIETNAM

APPENDIX 5

Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported

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