Results of Proficiency Test
SCCP in Leather
March 2019

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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CONTENTS

1 INTRODUCTION

Commercially produced chlorinated paraffin's (CPs) are classified according to their carbon chain length into Short Chain CPs (SCCP C_{10}-C_{13}), Medium Chain CPs (MCCP C_{14}-C_{17}) and Long Chain CPs (LCCP >C_{17}). The Chlorine content of these mixtures can vary from 30-70% depending on the application. Technical CPs are used in plasticizers and fire retardants. CPs are classified as persistent and non-biodegradable and they accumulate in the food chain. SCCPs were categorized in group 2B as possibly carcinogenic to humans from the International Agency for Research on Cancer (IARC). Since 2017, SCCP is banned under the Stockholm Convention on Persistent Organic Pollutants (annex A).

On request of a number of participants, the Institute for Interlaboratory Studies (iis) decided to organize for the first time a proficiency scheme for the determination of SCCP content in leather during the annual proficiency testing program 2018/2019. In this interlaboratory study 54 laboratories from 17 different countries registered for participation (see appendix 3).
In this report, the results of the 2019 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 gram, artificially fortified with SCCP and MCCP, labelled #19525. The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation. Participants were also requested to report a number of analytical details of the test method used.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

A batch of black colored leather was made positive on SCCP. The leather material was grinded into small pieces and mixed thoroughly. In total 76 bags were filled with 3 grams leather and labelled #19525. The homogeneity of the subsamples was checked by determination of SCCP in accordance with ISO18219 on ten stratified randomly selected samples. See the following table for the test results.

	SCCP in mg/kg
sample #19525-1	268.7
sample #19525-2	265.9
sample #19525-3	252.4
sample #19525-4	248.5
sample #19525-5	266.8
sample #19525-6	263.8
sample #19525-7	270.0
sample #19525-8	266.1
sample #19525-9	260.0
sample #19525-10	270.8

Table 1: homogeneity test results of subsamples #19525

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

	SCCP in mg/kg
r (observed)	21.0
reference method	Horwitz (9 components)
0.3 * R (reference method)	45.9

Table 2: evaluation of the repeatability of subsamples #19525

The calculated repeatability was in agreement with 0.3 times the corresponding reproducibility of the reference method. Therefore, homogeneity of the subsamples #19525 was assumed.

To each of the participating laboratories one sample labelled #19525 containing approximately 3 grams of leather was sent on March 6, 2019.

2.5 ANALYSES

The participants were requested to determine the MCCP and SCCP content, applying the analysis procedure that is routinely used in the laboratory. It was also requested to report if the laboratory was accredited for the determined components 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 or to dry the sample. It was also requested to report the test results using the indicated units on the report form and not to round the test results, but to report as much significant figures as possible. It was also requested not to report ‘less than’ test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reported units are given as well as the appropriate 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 sample and per component in the appendix 1 of this report. The laboratories are represented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that did not report 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 reanalyzis). 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 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.

In accordance 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. 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 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. ISO reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.
The target standard deviation was calculated from the literature reproducibility by division with 2.8. In general, when no literature reproducibility is available, another target 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 should be done in order to evaluate whether the reported test results are fit-for-purpose.

The z-scores were calculated in accordance with:

$z_{(target)}$ = (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.
The usual interpretation of z-scores is as follows:

	$\lvert z \rvert < 1$	good
$1 <$	$\lvert z \rvert < 2$	satisfactory
$2 <$	$\lvert z \rvert < 3$	questionable
$3 <$	$\lvert z \rvert$	unsatisfactory

4 EVALUATION

In this interlaboratory study, no problems were encountered with the dispatch of the samples. All registered participants reported test results. Three participants reported after the final reporting date. Finally, 54 laboratories reported 99 numerical results. In the reported test results 2 statistical outliers were observed, which is 2.0%.
In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

Both original data sets given in appendix 1 proved to have a normal Gaussian distribution.

4.1 EVALUATION PER COMPONENT

In this section, the test results are discussed per component.

For the determination of SCCP (and MCCP), ISO18219 is considered to be the official test method. Regretfully, ISO18219 does not contain any precision data. Therefore, the target requirements in this study were estimated using the Horwitz equation based on nine components (n=9).

Sample #19525

SCCP: This determination may be problematic. One statistical outlier was observed. The observed reproducibility after rejection of the statistical outlier is not in agreement with the estimated reproducibility based on the Horwitz equation (n=9).

MCCP: This determination may be problematic. One statistical outlier was observed. The observed reproducibility after rejection of the statistical outlier is not in agreement with the estimated reproducibility based on the Horwitz equation (n=9).

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the estimated target reproducibility using the Horwitz equation (n=9) and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average result, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility, calculated using the Horwitz equation are presented in the next table.

component	unit	n	average	2.8 * sd	R(target)
SCCP	mg/kg	52	264	229	153
MCCP	mg/kg	45	1010	741	479

Table 3: reproducibilities of tests on samples #19525

Without further statistical calculations, it can be concluded that there is for sample #19525 no compliance of the group of participating laboratories for the SCCP and MCCP determination with the target reproducibility.

4.3 OVERVIEW OF PROFICIENCY TEST OF MARCH 2019

	March 2019
Number of reporting labs	54
Number of test results	99
Number of statistical outliers	2
Percentage outliers	2.0%

Table 4: overview of proficiency test of March 2019

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

The uncertainties observed in this PT are compared with the relative standard deviation and with the target requirements based on the Horwitz equation in the next table.

Component	March 2019	Horwitz (n=9)
SCCP	31%	21%
MCCP	26%	17%

Table 5: overview of relative uncertainties (RSD)

No precision data is mentioned in ISO18219 and therefore it was decided to use the precision requirements based on Horwitz (n=9) as also been used in proficiency test SCCP in Polymer (iis18P05). Unfortunately, the group was not able to meet these requirements.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

The majority of the participants (85%) reported to have used ISO18219 as test method and 14% reported to have used an ‘in house’ test method. The details of the methods that were reported by the participants are listed in appendix 2.

Based on the answers given by the participants the following can be summarized:

- Forty-five of the participants reported to have an ISO/IEC17025 accreditation for the determination of total SCCP in polymers (= 83%).
- Remarkably, nine participants reported to have cut the already grinded sample prior to analysis.
- Almost all participants used a sample intake between 0.5 and 1.0 grams of the sample.
- Forty-five of the participants used Hexane to release/extract the components. Toluene was used by 5 other participants and one participant used THF/ACN to release/extract the SCCP/MCCP components.
- Almost all participants used an extraction time of 60 minutes and an extraction temperature of 60°C.

When the effect of the extraction solvent was investigated on the determination of SCCP and MCCP in leather it was noticed that the use of Hexane showed some improvement for both components on the reproducibility (see table 6 and appendix 1).

		All reported data			Only Hexane used as solvent		
component	unit	n	average	2.8 * sd	n	average	2.8 * sd
SCCP	mg/kg	52	264	229	43	277	203
MCCP	mg/kg	45	1010	741	37	1009	649

Table 6: performance overview on samples #19525

5 DISCUSSION

In this proficiency test for the determination of SCCP in leather, it was noticed that all participants, except one, were able to detect SCCP. Many participants reported also the presence of MCCP. Remarkably, one laboratory reported only MCCP and no SCCP. This is remarkable as both are forbidden components.

When the results of this interlaboratory study were compared to the Leather Standard by Oeko-Tex, it was noticed that all participants would make an identical decision about the acceptability of the leather for the determined components. All reporting laboratories would have rejected the sample for all categories.

Ecolabel	baby clothes	in direct skin contact	no direct skin contact
Leather by Oeko-Tex	<50 mg/kg *)	<50 mg/kg *)	<50 mg/kg *)

Table 7: Leather Standard by OEKO-TEX

*) This concerns the sum of SCCP and MCCP

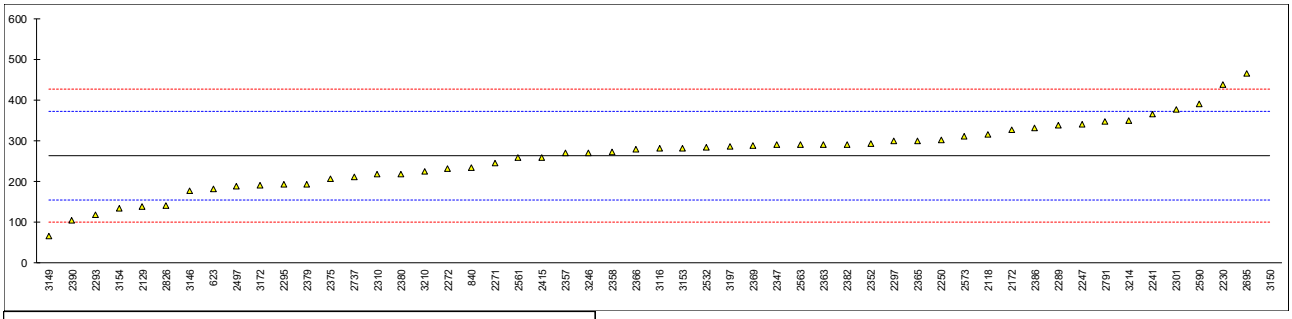
6 CONCLUSION

It is clear is that the majority of the participants is able to determine SCCP and MCCP in the leather matrix. The observed reproducibility in the first proficiency test on SCCP in Leather is large. Eeach laboratory has to evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and the quality of the analytical results.

APPENDIX 1

Determination of SCCP on sample #19525; results in mg/kg

lab	method	value	mark	z(targ)	remarks
623	ISO18219	180.67		-1.52	
840	ISO18219	233		-0.56	
2118	ISO18219	314.68		0.94	
2129	ISO18219Mod.	139		-2.28	
2172	In house	327.51		1.17	
2230	ISO18219	437		3.17	
2241	ISO18219	366		1.87	
2247	ISO18219	341.5		1.43	
2250	ISO18219	301		0.69	
2271	ISO18219	246.3		-0.31	
2272	ISO18219	232.721		-0.56	
2289	ISO18219	338		1.36	
2293	ISO18219	117.668		-2.67	
2295	ISO18219	193		-1.29	
2297	ISO18219	298.9		0.65	
2301	ISO18219	377.5		2.09	
2310	ISO18219	218.2		-0.83	
2347	ISO18219	290		0.48	
2352	ISO18219	293.6		0.55	
2357	ISO18219	270.4		0.13	
2358	ISO18219	273.55		0.18	
2363	ISO18219	291		0.50	
2365	ISO18219	298.9		0.65	
2366	ISO18219	278.2		0.27	
2369	ISO18219	289		0.47	
2375	ISO18219	207		-1.03	
2379	ISO18219	193.810		-1.28	
2380	ISO18219	218.9		-0.82	
2382	ISO18219	291		0.50	
2386	ISO18219	331		1.23	
2390	ISO18219	105.4		-2.89	
2415	ISO18219	259.9		-0.07	
2497	ISO18219	189.118		-1.36	
2532	ISO18219	283		0.36	
2561	In house	259.7	C	-0.07	First reported <100
2563	ISO18219	290.8		0.50	
2573	ISO18219	310.2		0.85	
2590	ISO18219	390.25		2.32	
2695	ISO18219	464	C	3.67	First reported 2321.0
2737	ISO18219	210.3		-0.97	
2791	In house	346.33		1.52	
2826	In house	142		-2.22	
3116	ISO18219	281.5		0.33	
3146	In house	176.8		-1.59	
3149	In house	66		-3.61	
3150	In house	2165.3	R(0.01)	34.79	
3153	ISO18219	282.3		0.34	
3154	ISO18219	133.416		-2.38	
3160		-----		-----	
3172	ISO18219	191		-1.33	
3197	ISO18219	285.3		0.40	
3210	ISO18219	225.54		-0.69	
3214	ISO18219	350		1.58	
3246	ISO18219	271		0.14	
					Only Hexane as solvent
	normality	OK			OK
	n	52			43
	outliers	1			1
	mean (n)	263.52	RSD = 31%		276.78 RSD = 26%
	st.dev. (n)	81.750			72.629
	R(calc.)	228.90			203.36
	st.dev.(Horwitz n=9)	54.661			56.990
	R(Horwitz n=9)	153.05			159.57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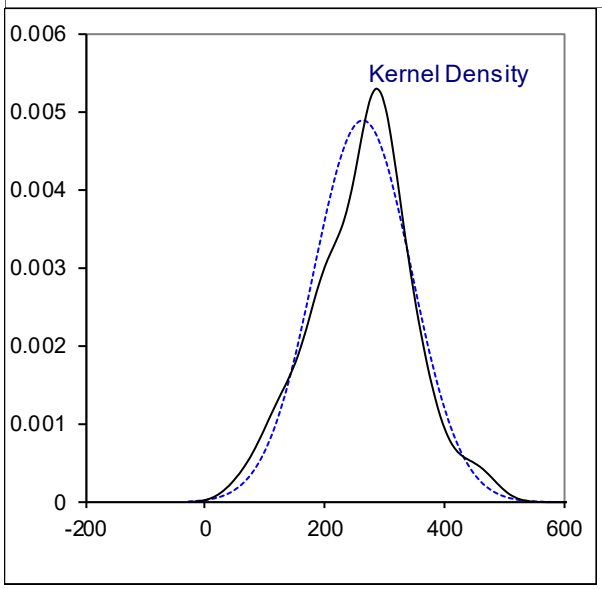
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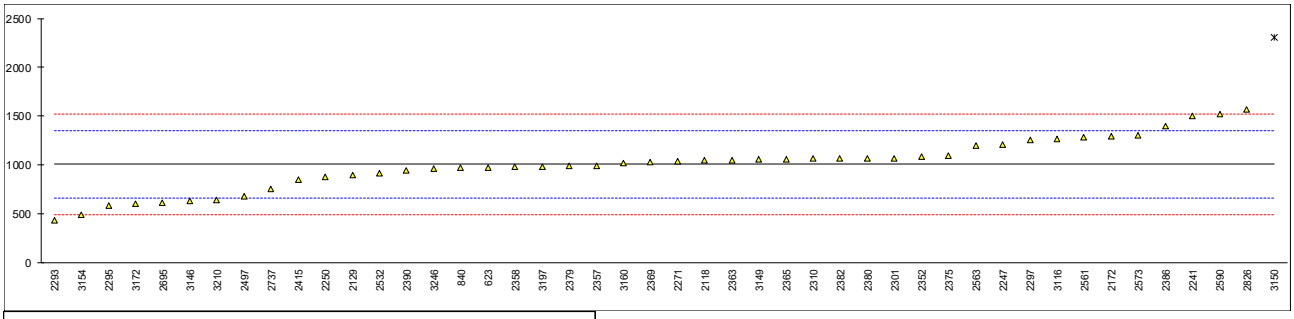
200
400
600
Kernel Density

Determination of MCCP on sample #19525; results in mg/kg

lab	method	value	mark	z(targ)	remarks
623	ISO18219	972.72		-0.22	
840	ISO18219	971		-0.23	
2118	ISO18219	1052.23		0.25	
2129	ISO18219Mod.	898		-0.65	
2172	In house	1299.33		1.69	
2230		-----		-----	
2241	ISO18219	1502		2.88	
2247	ISO18219	1214.1		1.20	
2250	ISO18219	882		-0.75	
2271	ISO18219	1036.2		0.16	
2272		-----		-----	
2289		-----		-----	
2293	ISO18219	440.300		-3.33	
2295	ISO18219	583		-2.49	
2297	ISO18219	1256.6		1.44	
2301	ISO18219	1070.2		0.35	
2310	ISO18219	1068		0.34	
2347		-----		-----	
2352	In house	1088.6		0.46	
2357	ISO18219	994.0		-0.09	
2358	ISO18219	983.15		-0.15	
2363	ISO18219	1053		0.25	
2365	ISO18219	1060.7		0.30	
2366		-----		-----	Out of capacity
2369	ISO18219	1036		0.15	
2375	ISO18219	1094		0.49	
2379	ISO18219	990.389		-0.11	
2380	ISO18219	1068.9		0.35	
2382	ISO18219	1068		0.34	
2386	ISO18219	1404		2.31	
2390	ISO18219	951.0		-0.34	
2415	ISO18219	850.2		-0.93	
2497	ISO18219	678.707		-1.93	
2532	ISO18219	920		-0.52	
2561	In house	1284.54	C	1.61	First reported 1486.8
2563	ISO18219	1204.8		1.14	
2573	ISO18219	1303.5		1.72	
2590	ISO18219	1525.07		3.01	
2695	ISO18219	612	C	-2.32	First reported 3058.5
2737	ISO18219	755.7		-1.48	
2791		-----		-----	
2826	In house	1570		3.28	
3116	ISO18219	1270		1.52	
3146	In house	637.5		-2.17	
3149	In house	1060		0.29	
3150	In house	2309.3	R(0.01)	7.60	
3153		-----		-----	
3154	ISO18219	496.079		-3.00	
3160	In house	1024.83		0.09	
3172	ISO18219	603		-2.38	
3197	ISO18219	985.2		-0.14	
3210	ISO22818draft	648.47		-2.11	
3214		-----		-----	
3246	ISO18219	964		-0.27	
					Only Hexane as solvent
	normality	OK			OK
	n	45			37
	outliers	1			1
	mean (n)	1009.58	RSD = 26%		1008.98 RSD = 23%
	st.dev. (n)	264.522			231.815
	R(calc.)	740.66			649.08
	st.dev.(Horwitz n= 9)	171.085			170.999
	R(Horwitz n=9)	479.04			478.80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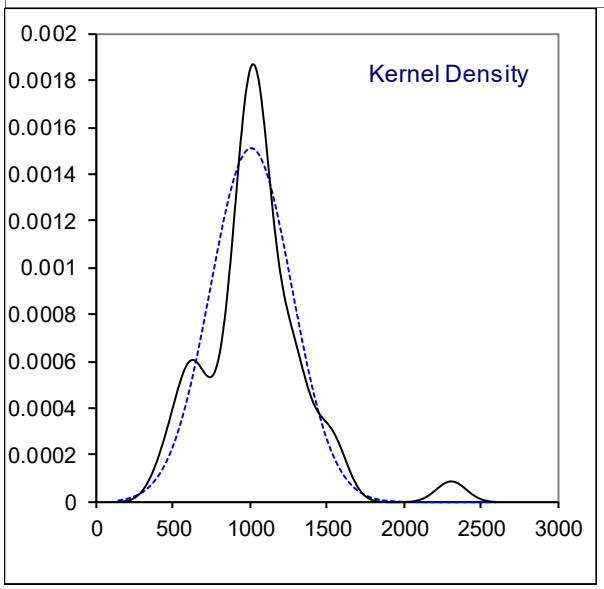
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0.0002
0
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APPENDIX 2
Analytical details

lab	ISO/IEC17025 accredited	grain size reduced before use	reduced to particle size (mm)	Sample intake (g)	Extraction solvent used	Extraction time and temperature used
623	Yes	Further Cut	3 mm x 3 mm	0.5	Hexane	60min - 60°C
840	Yes	Used as received		0.5	Hexane	60min - 60°C
2118	No	Used as received		0.5 g	hexane	60min - 60°C
2129	Yes	Used as received		0,5 g	Toluene	60min - 60°C
2172	Yes	Further Cut	5mm*5mm	0.5g	Hexane	60min - 60°C
2230	Yes	Used as received		1g	Hexane	60min - 60°C
2241	Yes	Used as received		0.5g	Toluene	60min - 60°C
2247	---	Used as received		0.5g	Hexane	60min - 60°C
2250	Yes	Used as received		0.5 g	Hexane	60min - 60°C
2271	Yes	Used as received	3mmx3mm	0.5g	Hexane	60min - 60°C
2272	Yes	Used as received	< 2mm-3mm	0.5gram	Hexane	60min - 60°C
2289	Yes	Further Cut	2mm*2mm	0.5g	Hexane	60min - 60°C
2293	No	Used as received	3 X 5 mm	0.5002 g	10 mL n-hexane	60min - 60°C
2295	Yes	Further Cut		0.5 gram	Hexane	60min - 60°C
2297	Yes	Used as received		1g	Toluene	60min - 60°C
2301	Yes	Used as received	2mm x 2mm	1	Hexane	60min - 60°C
2310	Yes	Used as received		0.5 gram	Hexane	60min - 60°C
2347	Yes	Used as received	2x22mm	0.5g		60min - 60°C
2352	Yes	Used as received	<3x3mm	0.5g	Hexane	60min - 60°C
2357	Yes	Used as received		0.5	Hexane	60min - 60°C
2358	Yes	Used as received	<5mmX5mm	0.58	Hexane	60min - 60°C
2363	Yes	Used as received		0.5g	Hexane	60min - 60°C
2365	Yes	Used as received		0.5g	Hexane	60min - 60°C
2366	Yes	Further Cut	3mm*3mm	0.5 gram	Hexane	60min - 60°C
2369	Yes	Used as received		0.5g	Hexane	60min - 60°C
2375	Yes	Used as received	Small pieces	0,5 g	Hexane	60min - 60°C
2379	No	Used as received	-	0.5 g	Hexane	60min - 60°C
2380	Yes	Used as received	Smashed.	0.5	Hexane	60min - 60°C
2382	Yes	Further Grinded	2mm*2mm	0.5g	Hexane	60min - 60°C
2386	Yes	Used as received	2mm	0,5	Hexane	60min - 60°C
2390	Yes	Used as received	< 2mm	0.5054 gram	Hexane	60min - 60°C
2415	Yes	Used as received	3x3mm	0.5g	Hexane	60min - 60°C
2497	Yes	Used as received		2	Toluene	60min - 60°C
2532	No	Used as received	< 2mm	0.5 gram	Hexane	60min - 60°C
2561	Yes	Used as received		1 g	Hexane	60min - 60°C
2563	Yes	Used as received		0,5 g	Hexane	60min - 60°C
2573	Yes	Used as received		0.5g	Hexane	60min - 60°C
2590	Yes	Used as received	<2 x 3 mm.	1.5 g	Hexane	60min - 60°C
2695	Yes	Used as received		0.500g	Hexane	60min - 60°C
2737	Yes	Further Cut		0.5grams	Hexane	60min - 60°C
2791	No	Used as received		0.5	1:1-Acetone:Hexane	30min - 50°C
2826	Yes	Used as received		0.5	Toluene	60min - 60°C
3116	Yes	Used as received		1.0 g	Hexane	60min - 60°C
3146	Yes	Used as received		0,5	THF/Acetonitrile	60min - 70°C
3149	---	---				
3150	Yes	Used as received	5x5mm	0,5	Hexane	60min - 60°C
3153	Yes	Used as received	2mm x 2mm	0.5g	Hexane	60min - 60°C
3154	Yes	Used as received				
3160	No	Used as received		0,5 gr	Hexane	60min - 60°C
3172	Yes	Further Cut	3x3mm	0.5	Hexane	60min - 60°C
3197	Yes	Used as received		0,25 g	Hexane	60min - 60°C
3210	No	Used as received		0.5g	Hexane	60min - 60°C
3214	Yes	Further Cut	< 5mm x 5mm	0.5	Hexane	60min - 60°C
3246	Yes	Used as received	0.5x0.5mm	0.50g	Hexane	60min - 60°C

APPENDIX 3

Number of participants per country

1 lab in	BANGLADESH
1 lab in	BELGIUM
1 lab in	FRANCE
8 labs in	GERMANY
1 lab in	GUATEMALA
4 labs in	HONG KONG
4 labs in	INDIA
2 labs in	INDONESIA
4 labs in	ITALY
16 labs in	P.R. of CHINA
1 lab in	PAKISTAN
1 lab in	SPAIN
2 labs in	TAIWAN R.O.C.
1 lab in	THAILAND
3 labs in	TURKEY
1 lab 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's outlier test
R(0.05)	= straggler in Rosner's outlier test
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation

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