

Results of Proficiency Test
SCCP in Leather
April 2020

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Commercially produced chlorinated paraffin's (CPs) are classified according to their carbon chain length into Short Chain CPs (SCCP C₁₀-C₁₃), Medium Chain CPs (MCCP C₁₄-C₁₇) and Long Chain CPs (LCCP >C₁₇). 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).

In 2019 on request of several participants the Institute for Interlaboratory Studies (iis) organized a proficiency test for the determination of SCCP content in leather for the first time. During the annual proficiency testing program 2019/2020 it was decided to continue the proficiency test for the analysis of SCCP in leather.

In this interlaboratory study 56 laboratories from 20 different countries registered for participation. See appendix 3 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 artificially fortified with SCCP and MCCP of 3 gram and labelled #20560. 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 some 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 beige colored leather was selected which was made positive on SCCP. The leather was grinded into small pieces and mixed thoroughly. After homogenization the batch was divided over 83 subsamples in small bags of approximately 3 grams each and labelled #20560. The homogeneity was checked by determination of SCCP in accordance with ISO18219 on ten stratified randomly selected subsamples.

	SCCP in mg/kg
sample #20560-1	237.4
sample #20560-2	266.9
sample #20560-3	268.8
sample #20560-4	276.1
sample #20560-5	248.7
sample #20560-6	266.6
sample #20560-7	265.2
sample #20560-8	256.7
sample #20560-9	264.7
sample #20560-10	263.8

Table 1: homogeneity test results of subsamples #20560

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.

	SCCP in mg/kg
r (observed)	31.2
reference method	Horwitz (n=9)
0.3 * R (reference method)	45.6

Table 2: evaluation of the repeatability of subsamples #20560

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

To the participating laboratories one sample labelled #20560 was sent on March 4, 2020.

2.5 ANALYZES

The participants were requested to determine the SCCP and MCCP 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 noted in the instructions of this PT to use no less than 0.5 grams per determination to ensure the homogeneity.

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 were 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 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 reanalyzes). 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 ISO5725 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, 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:

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

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

In this interlaboratory study serious problems were encountered with the dispatch of the samples. A number of participants informed iis that they were not able to report test results due to the measures taken to contain the Covid-19 pandemic in their countries. When the data entry portal was closed twelve participants did not report any test results. For these participants an extra round was prepared on the data entry portal. During the preparation of this PT report it was decided to add these test results in this final report as well.

In total fifty-three participants reported 102 numerical test results. Observed were 7 outlying test results, which is 6.9% of the numerical test results. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

Not all original data sets proved to have a normal Gaussian distribution. These are referred to as “not OK” or “suspect”. The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1 EVALUATION PER COMPONENT

In this section, the test results are discussed per component. The test methods, which were reported by the laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the table together with the original data. The abbreviations, used in these tables, are explained in appendix 4.

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 #20560

SCCP: This determination may be problematic for a number of laboratories. Four statistical outliers were observed. However, the observed reproducibility after rejection of the statistical outliers is in agreement with the estimated reproducibility based on the Horwitz equation (n=9).

MCCP: This determination may be problematic. Three statistical outliers were observed. The observed reproducibility after rejection of the statistical outliers 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 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, calculated using the Horwitz equation are presented in the next table.

component	unit	n	average	2.8 * sd	R(target)
SCCP	mg/kg	49	173	117	107
MCCP	mg/kg	46	710	445	355

Table 3: reproducibilities of tests on samples #20560

Without further statistical calculations, it can be concluded that for sample #20560 there is a compliance of the group of participating laboratories for the SCCP determination with the target reproducibility. Regretfully, this was not the case for the MCCP determination.

4.3 COMPARE OF PROFICIENCY TEST OF APRIL 2020 TO THE PREVIOUS PT

	April 2020	March 2019
Number of reporting laboratories	53	54
Number of test results	102	99
Number of statistical outliers	7	2
Percentage of statistical outliers	6.9%	2.0%

Table 4: comparison with the previous proficiency test

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	April 2020	March 2019	Horwitz (n=9)
SCCP	24%	31%	21-22%
MCCP	22%	26%	17-21%

Table 5: development of uncertainties (RSD) over the years

The group's performance improved compared to last year's performance.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

In this PT, also some analytical details were asked (see appendix 2) to use for further statistical analysis.

Around 75% of the participants mentioned to be accredited for the determination of SCCP in leather.

About 80% of the reporting participants mentioned to use test method ISO18219 for the determination of SCCP. About 15% of the participants reported to have used an in house method.

About 70% of the participants reported to use an intake of 0.5 gram. Remarkably, eleven participants reported to have cut the already grinded sample prior to analysis.

About 70% of the participants used Hexane to release/extract the components. Toluene was used by about 10% of the participants and 8% participant another solvent 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 in leather it was noticed that the use of Hexane only showed no improvement. However, for the determination of MCCP a small improvement is visible (see appendix 1).

5 DISCUSSION

In this proficiency test for the determination of SCCP in leather, it was noticed that all reporting participants were able to detect SCCP. The majority of the participants reported also the presence of MCCP.

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 6: Leather Standard by OEKO-TEX

*) This concerns the sum of SCCP and MCCP

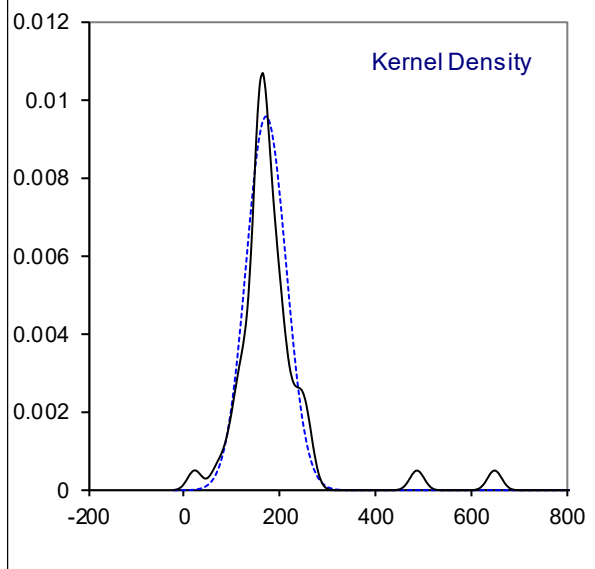
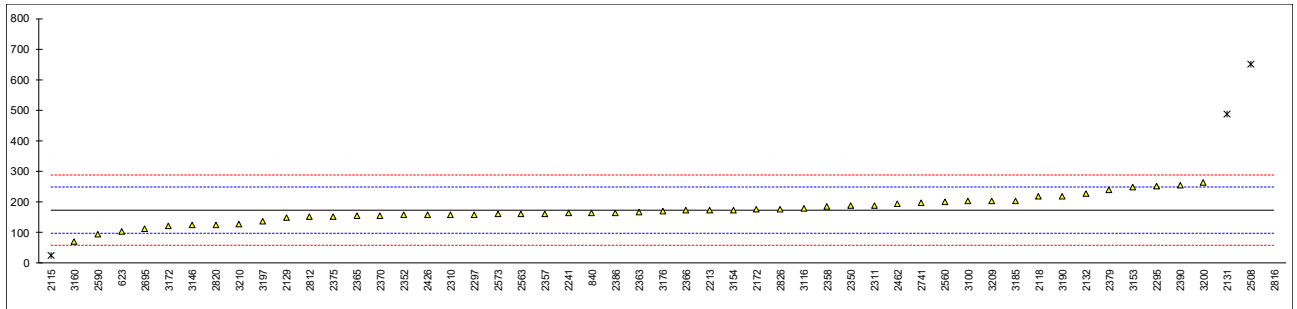
6 CONCLUSION

It is clear that the majority of the participants is able to determine SCCP and MCCP in the leather matrix. The observed reproducibility in this proficiency test on SCCP in Leather improved compared to last year. Each 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

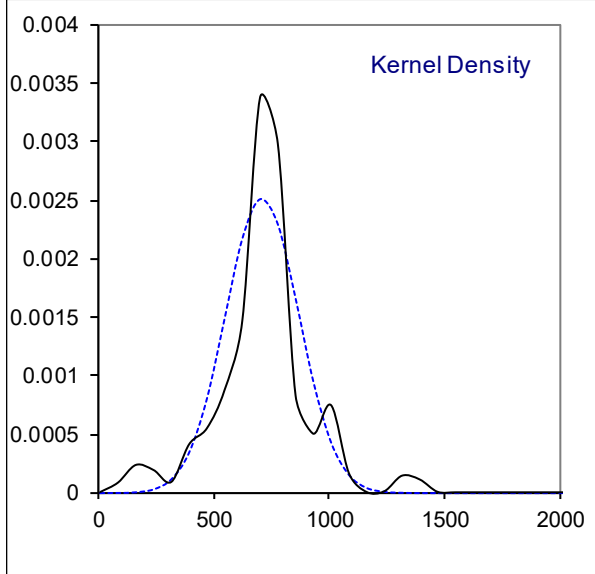
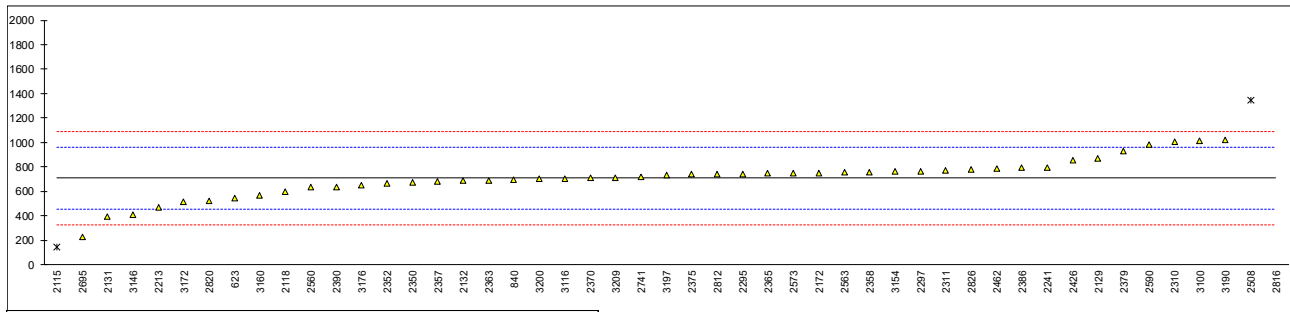
Determination of SCCP on sample #20560; results in mg/kg

lab	method	value	mark	z(targ)	remarks
623	ISO18219	103.43		-1.81	
840	ISO18219	164		-0.22	
2115	ISO18219	23.61	R(0.05)	-3.90	
2118	ISO18219	217.814		1.19	
2129	ISO18219	149		-0.62	
2131	In house	487.5	R(0.01)	8.26	
2132	In house	228		1.45	
2172	ISO18219	176.56		0.11	
2213	ISO18219	172		-0.01	
2241	ISO18219	163.70		-0.23	
2267		----		----	
2295	ISO18219	251.0		2.06	
2297	ISO18219	158.3		-0.37	
2310	ISO18219	158.2		-0.38	
2311	ISO18219	189.1		0.43	
2350	ISO18219	188.511		0.42	
2352	ISO18219	157.4		-0.40	
2357	ISO18219	162.0		-0.28	
2358	ISO18219	186.26		0.36	
2363	ISO18219	167		-0.14	
2365	ISO18219	154.97		-0.46	
2366	ISO18219	171.8		-0.02	
2370	ISO18219	155		-0.46	
2375	ISO18219	151		-0.56	
2379	ISO18219	239.715		1.76	
2386	ISO18219	165		-0.20	
2390	ISO18219	253.0		2.11	
2426	ISO18219	157.65		-0.39	
2462	ISO18219	195		0.59	
2508	ISO18219	649.51	R(0.01)	12.51	
2560	ISO18219	201.10		0.75	
2561		----		----	
2563	ISO18219	161.9		-0.28	
2573	ISO18219	160.3		-0.32	
2590	ISO18219	93.56		-2.07	
2614		----		----	
2695	In house	112.7978269		-1.57	
2741	ISO18219	196.73		0.63	
2812	ISO18219	150.8	C	-0.57	First reported 1004.55
2816	In house	1337	R(0.01)	30.53	
2820	ISO18219	124.8	C	-1.25	First reported 3184.1
2826	ISO18219	177.3781		0.13	
3100	ISO18219	201.6		0.76	
3116	ISO18219	179.4		0.18	
3146	In house	123		-1.30	
3153	ISO18219	247.1		1.96	
3154	ISO18219	172.363		0.00	
3160	In house	70.21		-2.68	
3172	ISO18219	121		-1.35	
3176	ISO18219	169.92		-0.07	
3185	ISO18219	202.94		0.80	
3190	ISO18219	219.71		1.24	
3197	ISO18219	137.2		-0.93	
3200	ISO18219	265.0		2.42	
3209	In house	202.37		0.78	
3210	In house	127.13		-1.19	
					<u>Only Hexane as solvent</u>
	normality	OK			OK
	n	49			37
	outliers	4			0
	mean (n)	172.525			172.651
	st.dev. (n)	41.6611	RSD = 24%		43.3421
	R(calc.)	116.651			121.358
	st.dev.(Horwitz (n=9))	38.1429			38.1666
	R(Horwitz (n=9))	106.800			106.866



Determination of MCCP on sample #20560; results in mg/kg

lab	method	value	mark	z(targ)	remarks
623	ISO18219	542.93		-1.32	
840	ISO18219	694		-0.13	
2115	ISO18219	141.66	R(0.05)	-4.48	
2118	ISO18219	598.311		-0.88	
2129	ISO18219	872		1.27	
2131	In house	392		-2.51	
2132	In house	686		-0.19	
2172	ISO18219	751.66		0.33	
2213	ISO18219	469		-1.90	
2241	ISO18219	795.38		0.67	
2267		----		----	
2295	ISO18219	742.0		0.25	
2297	ISO18219	765.1		0.43	
2310	ISO18219	1009		2.35	
2311	ISO18219	775		0.51	
2350	ISO18219	676.113		-0.27	
2352	In house	669.8		-0.32	
2357	ISO18219	679.0		-0.25	
2358	ISO18219	758.38		0.38	
2363	ISO18219	686		-0.19	
2365	ISO18219	746.16		0.28	
2366	ISO18219	out of capability		----	
2370	ISO18219	710		0.00	
2375	ISO18219	740		0.23	
2379	ISO18219	934.630		1.77	
2386	ISO18219	793		0.65	
2390	ISO18219	635.3		-0.59	
2426	ISO18219	855.17		1.14	
2462	ISO18219	790		0.63	
2508	ISO18219	1348.47	R(0.05)	5.03	
2560	ISO18219	635.20		-0.59	
2561		----		----	
2563	ISO18219	757.4		0.37	
2573	ISO18219	750.1		0.31	
2590	ISO18219	982.82		2.15	
2614		----		----	
2695	In house	224.73	C	-3.83	First reported 22.25844
2741	ISO18219	718.62		0.07	
2812	ISO18219	740.3	C	0.24	First reported 371.26
2816	In house	19495	R(0.01)	148.01	
2820	ISO18219	526.4	C	-1.45	First reported 4887.6
2826	ISO18219	782.8341		0.57	
3100	ISO18219	1010.2		2.36	
3116	ISO18219	707.2		-0.02	
3146	In house	406		-2.40	
3153		----		----	
3154	ISO18219	764.036		0.42	
3160	In house	564.86		-1.15	
3172	ISO18219	516		-1.53	
3176	ISO18219	650.0		-0.48	
3185		----		----	
3190	ISO18219	1023.07		2.46	
3197	ISO18219	731.1		0.16	
3200	ISO18219	704.0		-0.05	
3209	In house	715.20		0.04	
3210		----		----	
					<u>Only Hexane as solvent</u>
	normality	suspect			not OK
	n	46			33
	outliers	3			0
	mean (n)	710.348			735.130
	st.dev. (n)	159.0447	RSD = 22%		156.7877
	R(calc.)	445.325			439.006
	st.dev.(Horwitz (n=9))	126.9180			130.6696
	R(Horwitz (n=9))	355.370			365.875



APPENDIX 2 Analytical details

lab	ISO/IEC17025 accredited	Sample reduced before use	Sample intake (g)	Extraction solvent used	Extraction time and temperature used
623	Yes	Used as received	0.5	n-Hexane	60min - 60°C
840	Yes	Further Cut	0.5	n-Hexane	60min - 60°C
2115	No	Used as received	1	n-Hexane/dichloromethane 1:1	60min - 60°C
2118	No	Used as received	0.8	n-Hexane	60min - 60°C
2129	Yes	Used as received	0.5	Toluene	60min - 60°C
2131	Yes	Used as received	1	n-Hexane/dichloromethane 1:1	
2132	No	Used as received	0.5	n-Hexane	60min - 60°C
2172	Yes	Used as received	0.5	n-Hexane	60min - 60°C
2213	Yes	Further Cut			
2241	Yes	Further Cut	0.3	n-Hexane	60min - 60°C
2267	---	---			
2295	Yes	Further Cut	0.5	n-Hexane	60min - 60°C
2297	Yes	Used as received	0.5	Toluene	60min - 60°C
2310	Yes	Used as received	0.5	n-Hexane	60min - 60°C
2311	Yes	Further Cut	0.5	n-Hexane	60min - 60°C
2350	No	Used as received	0.5	n-Hexane	60min - 60°C
2352	Yes	Used as received	0.5	n-Hexane	60min - 60°C
2357	---	---			
2358	Yes	Used as received	0.5	Toluene	60min - 60°C
2363	Yes	Used as received	0.5	n-Hexane	60min - 60°C
2365	Yes	Used as received	0.5	n-Hexane	60min - 60°C
2366	Yes	Further Cut	0.5	n-Hexane	60min - 60°C
2370	Yes	Used as received	0.5	n-Hexane	60min - 60°C
2375	Yes	Used as received	0.5	n-Hexane	60min - 60°C
2379	No	Used as received	0.5	n-Hexane	60min - 60°C
2386	Yes	Used as received	0.5	n-Hexane	60min - 60°C
2390	Yes	Further Cut	0.5051	n-Hexane	60min - 60°C
2426	Yes	Used as received	0.5033	n-Hexane	60min - 60°C
2462	No	Further Cut	0.5	n-Hexane	60min - 60°C
2508	---	---			
2560	Yes	Used as received	1.0	n-Hexane	60min - 60°C
2561	---	---			
2563	Yes	Further Cut	0.5	n-Hexane	60min - 60°C
2573	Yes	Used as received	0.5	n-Hexane	60min - 60°C
2590	Yes	Other	0.5	n-Hexane	60min - 60°C
2614	---	---			
2695	Yes	Used as received	1	n-Hexane, THF/Methanol mix	60min - 60°C
2741	Yes	Used as received	0.5	n-Hexane	60min - 60°C
2812	No	Used as received	0.5	n-Hexane	60min - 60°C
2816	No	Further Cut	0.5	Pentane/Acetone	225min - 22°C
2820	Yes	Used as received	1	n-Hexane	60min - 60°C
2826	Yes	Further Cut	0.5	Toluene	60min - 60°C
3100	Yes	Used as received	0.5	n-Hexane	60min - 60°C
3116	Yes	Used as received	1	Toluene	60min - 60°C
3146	Yes	Used as received	0.5	n-Hexane + inhouse	30min - 60°C
3153	Yes	Used as received	0.5	n-Hexane	60min - 60°C
3154	Yes	Used as received	1	n-Hexane	60min - 60°C
3160	No	Used as received	0.6	n-Hexane	60min - 60°C
3172	Yes	---			
3176	Yes	Used as received	0.5	n-Hexane/dichloromethane	60min - 60°C
3185	Yes	Used as received	0.5	n-Hexane	60min - 60°C
3190	Yes	Used as received	0.5	n-Hexane	60min - 60°C
3197	Yes	Used as received	0.5	n-Hexane	60min - 60°C
3200	Yes	Used as received	0.5	THF/CAN	
3209	Yes	Used as received	0.5		60min - 60°C
3210	Yes	Used as received	1	n-Hexane	60min - 60°C

APPENDIX 3

Number of participants per country

1 lab in BANGLADESH
1 lab in BELGIUM
1 lab in DENMARK
1 lab in FRANCE
6 labs in GERMANY
5 labs in HONG KONG
4 labs in INDIA
1 lab in INDONESIA
5 labs in ITALY
15 labs in P.R. of CHINA
2 labs in PAKISTAN
1 lab in SOUTH KOREA
1 lab in SPAIN
1 lab in SWITZERLAND
1 lab in TAIWAN R.O.C.
1 lab in THAILAND
1 lab in THE NETHERLANDS
5 labs in TURKEY
1 lab in UNITED KINGDOM
2 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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