Institute for
Interlaboratory Studies

Results of Proficiency Test
SCCP/MCCP in Textile
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Organized by: Institute for Interlaboratory Studies
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Author: ing. A. Ouwerkerk
Correctors: ing. G.A. Oosterlaken-Buijs & ing. R.J. Starink
Approved by: ing. R.J. Starink

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1 INTRODUCTION

Commercially produced Chlorinated Paraffins (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 as plasticizers or 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). SCCP (chlorine content >48%) are listed by the Stockholm Convention on Persistent Organic Pollutants. In Europe SCCP as constituents of articles are prohibited according to regulation 2019/1021 of the European Parliament and of the Council of 20 June 2019 on persistent organic pollutants. Articles containing SCCP in concentrations lower than 0.15% by weight are allowed. Furthermore, it became industrial practice to restrict MCCP as well.

Since 2021 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of SCCP and MCCP in Textile every year. During the annual proficiency testing program of 2023 it was decided to continue the proficiency test for the determination of SCCP/MCCP in Textile.

In this interlaboratory study 31 laboratories in 12 countries registered for participation, see appendix 3 for the number of participants per country. In this report the results of the SCCP/MCCP in Textile proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to a laboratory that has performed the tests in accordance with for ISO/IEC17043 relevant requirements of ISO/IEC17025.
It was decided to send one textile sample containing SCCP and MCCP of approximately 3 grams labelled #23735.
The participants were requested 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 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 brown colored cotton textile was selected which was artificially fortified with SCCP and MCCP. After homogenization 45 small plastic bags were filled with approximately 3 grams each and labelled #23735.
The homogeneity of the subsamples was checked by the determination of SCCP content in accordance with ISO18219 on 8 stratified randomly selected subsamples.

	SCCP in mg/kg
sample #23735-1	83.8
sample #23735-2	85.3
sample #23735-3	86.2
sample #23735-4	87.3
sample #23735-5	84.2
sample #23735-6	83.3
sample #23735-7	83.9
sample #23735-8	84.1

Table 1: homogeneity test results of subsamples #23735

From the above test results the repeatability was calculated and compared with 0.3 times the estimated reproducibility calculated with the Horwitz equation (based on 9 components) in agreement with the procedure of ISO13528, Annex B2 in the next table.

	SCCP in mg/kg
r (observed)	3.8
reference method	Horwitz (n=9)
0.3 x R (reference method)	17.5

Table 2: evaluation of the repeatability of subsamples #23735

The calculated repeatability is in agreement with 0.3 times the estimated reproducibility calculated with the Horwitz equation (based on 9 components). Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one textile sample labelled #23735 was sent on September 13, 2023.

2.5 ANALYZES

The participants were requested to determine SCCP, CAS No. 85535-84-8 and MCCP, CAS No. 85535-85-9.
It was requested not to use less than 0.5 gram per determination to ensure homogeneity.
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 and to report the test results using the indicated units on the report form and not to round the test results, but 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 reporting units are given as well as the reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per determination in 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 reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the result tables in 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.

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 averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

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

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements (derived from e.g. ISO or ASTM test methods), the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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

The z-scores were calculated according to:

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

The $z_{(target)}$ scores are listed in the test result tables in appendix 1.

Absolute values for z<2 are very common and absolute values for z>3 are very rare. Therefore, the usual interpretation of z-scores is as follows:

	|z|	< 1	good
1 <	|z|	< 2	satisfactory
2 <	|z|	< 3	questionable
3 <	|z|		unsatisfactory

4 EVALUATION

In this proficiency test no problems were encountered with the dispatch of the samples. One participant reported test results after the final reporting date and one other participant did not report any test results.
In total 30 participants reported 44 numerical test results. Observed were 3 outlying test results, which is 6.8%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

Both data sets proved to have a normal Gaussian distribution.

4.1 EVALUATION PER COMPONENT

In this section the reported test results are discussed per component. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 4.

Since 2021 test method ISO22818 is available for the determination of SCCP and MCCP in textile products out of different matrices, especially in polymer of the coated fabrics, prints made of polymer and buttons made of polymer (e.g. PVC).
The precision data mentioned in test method ISO22818:21 is for two specific types of coated fabrics which is not the same as the material of the PT sample. Therefore it was decided to follow the procedure as used in previous PTs and to use the Horwitz equation (based on nine components) for estimation of the target reproducibilities and to mention the requirements from ISO22818:21, sample A for comparison only.

SCCP: The group of participants met the target requirements. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility calculated with the Horwitz equation (based on 9 components), but not with the requirements of ISO22818:21, sample A. About half of the reporting participants reported ‘less than’, ‘not detected’ or ‘below detection limit’ test results. If these test results are taken into account, the average will probably somewhat lower.

MCCP: The group of participants met the target requirements. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated reproducibility calculated with the Horwitz equation (based on 9 components) and with the requirements of ISO22818:21, sample A.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from reference methods are presented in the next table.

Component	Unit	n	average	2.8 * sd	R(target)
SCCP	mg/kg	14	59.2	45.0	43.1
MCCP	mg/kg	27	248	97	145

Table 3: reproducibilities of components on sample #23735

Without further statistical calculations it can be concluded that for both components there is a good compliance of the group of participating laboratories with the reference test method.

4.3 COMPARISON OF THE PROFICIENCY TEST OF OCTOBER 2023 WITH PREVIOUS PTS

	October 2023	October 2022	November 2021
Number of reporting laboratories	30	29	11
Number of test results	44	58	21
Number of statistical outliers	3	3	0
Percentage of statistical outliers	6.8%	5.2%	0%

Table 4: comparison with previous proficiency tests

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

The performance of the determinations of the proficiency test was compared to uncertainties observed in PTs over the years, expressed as relative standard deviation (RSD) of the PTs, see next table.

Component	October 2023	October 2022	November 2021
SCCP	27%	14%	23%
MCCP	14%	16%	39%

Table 5: development of the uncertainties over the years

The uncertainties observed in this PT are comparable to the uncertainties observed in previous PTs.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

Test method ISO22818 is used by the majority of the reporting participants. Some participants used test method ISO18219-1 which is a method for the determination of SCCP in leather. Test method ISO18219-1 does not contain precision data.

For this PT some analytical details were requested which are listed in appendix 2. Based on the answers given by the participants the following can be summarized:
- 77% of the participants are accredited to determine the reported components.
- 60% used the sample as received and 40% further cut the sample prior to analysis.
- 76% used 0.5 grams of sample intake and 24% used between 1 and 2 grams.
- 74% used Toluene as extraction solvent, 19% used Hexane or a Hexane mixture.
- almost all participants used an extraction time of 60 minutes and an extraction temperature of 60°C.

For SCCP and MCCP the calculated reproducibility is in agreement with the requirements of the target reproducibility, therefore no separate statistical analysis has been performed.

5 DISCUSSION

In Regulation (EU) 2019/1021 of the European Parliament and of the Council of 20 June 2019 on persistent organic pollutant it is mentioned that articles containing SCCP in concentrations lower than 0.15% by weight are allowed. In this PT about half of the reporting participants reported a numerical value for SCCP and the other half reported ‘less than’, ‘not detected’ or ‘below detection limit’ test results. When the results of this interlaboratory study were compared to the regulation, it was noticed that all reporting participants would have accepted sample #23735, assuming that ‘below detection limit’ and ‘not detected’ are test results below the calculated average.

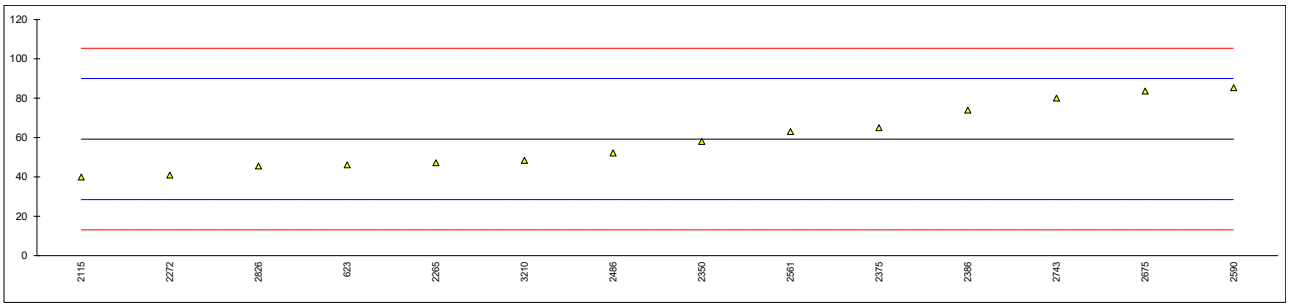
6 CONCLUSION

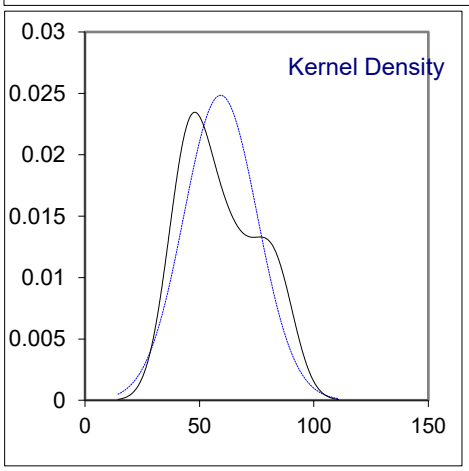
Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

APPENDIX 1

Determination of SCCP, CAS No. 85535-84-8 on sample #23735; results in mg/kg

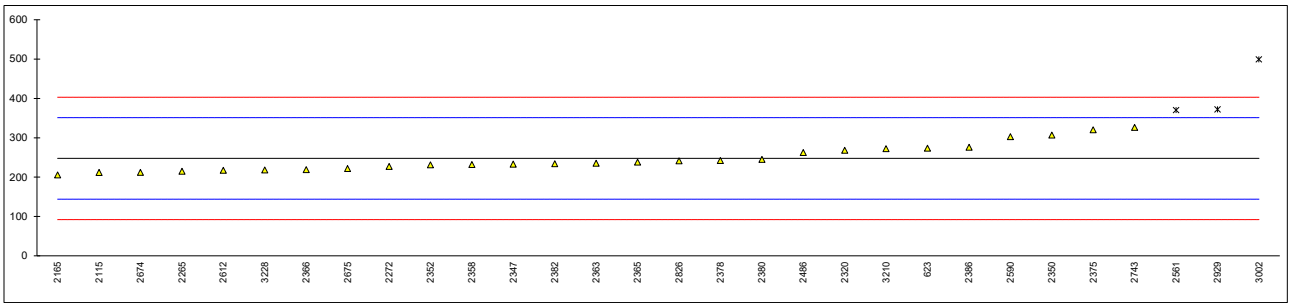
Lab	method	value	mark	z(targ)	remarks
623	ISO22818	46.14		-0.85	
2115	ISO18219-1:2021	39.9		-1.26	
2165	ISO22818	Not Detected		-----	
2265	ISO22818	47.18		-0.78	
2272	ISO22818	40.9		-1.19	
2320	ISO22818	<50		-----	
2347	ISO22818	<50		-----	
2350	ISO22818	58.0		-0.08	
2352		-----		-----	
2358	ISO22818	Not detected		-----	
2363	ISO22818	<50		-----	
2365	ISO22818	＜50		-----	
2366	ISO22818	<50		-----	
2375	ISO22818	65		0.38	
2378	ISO22818	<50		-----	
2380	ISO18219-1:2021	<50		-----	
2382	ISO22818	<50		-----	
2386	ISO18219-1:2021	73.9		0.95	
2486	ISO22818	52.14		-0.46	
2561	In house	63.1		0.25	
2590	ISO18219-1:2021	85.3		1.70	
2612	In house	< Limit of quantitation		-----	
2674	ISO18219-1:2021	not detected		-----	
2675	ISO22818	83.551		1.58	
2743	ISO22818	80.084		1.36	
2826	ISO22818	45.61		-0.89	
2886		-----		-----	
2929	In house	< detection limit		-----	
3002	ISO18219-1:2021	not detected		-----	
3210	ISO22818	48.369		-0.71	
3228	ISO22818	not detected		-----	
	normality	OK			
	n	14			
	outliers	0			
	mean (n)	59.227			
	st.dev. (n)	16.0551	RSD=27%		
	R(calc.)	44.954			
	st.dev.(Horwitz n=9)	15.3804			
	R(Horwitz n=9)	43.065			
compare					
	R(ISO22818:21)	31.011	ISO22818:21 sample A, Table C.1		

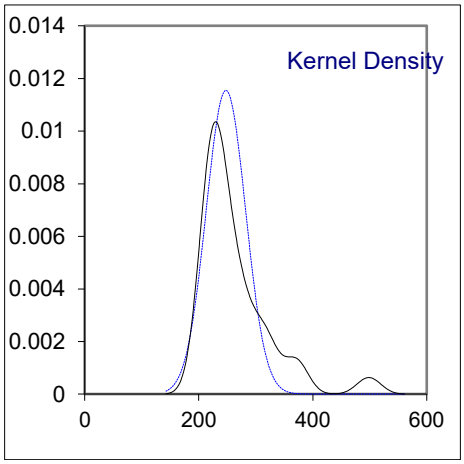




Determination of MCCP, CAS No. 85535-85-9 on sample #23735; results in mg/kg

lab	method	value	mark	z(targ)	remarks
623	ISO22818	273.40		0.50	
2115	ISO18219-2:2021	211.7		-0.69	
2165	ISO22818	205.3		-0.82	
2265	ISO22818	214.71		-0.63	
2272	ISO22818	227.0		-0.40	
2320	ISO22818	268.2		0.40	
2347	ISO22818	233		-0.28	
2350	ISO22818	306.70		1.14	
2352	ISO22818	231		-0.32	
2358	ISO22818	231.94		-0.30	
2363	ISO22818	235		-0.24	
2365	ISO22818	238.0		-0.19	
2366	ISO22818	219		-0.55	
2375	ISO22818	320		1.40	
2378	ISO22818	242		-0.11	
2380	ISO18219-2:2021	244.902		-0.05	
2382	ISO22818	234.0		-0.26	
2386	ISO18219-2:2021	276		0.55	
2486	ISO22818	262.23		0.28	
2561	In house	370	DG(0.05)	2.36	
2590	ISO18219-2:2021	302.9		1.07	
2612	In house	217.21		-0.59	
2674	ISO18219-2:2021	212.3		-0.68	
2675	ISO22818	221.726		-0.50	
2743	ISO22818	326.203		1.52	
2826	ISO22818	240.98		-0.13	
2886		-----		-----	
2929	In house	372.11	DG(0.05)	2.40	
3002	ISO18219-2:2021	498.7	G(0.01)	4.84	
3210	ISO22818	272.104		0.47	
3228	ISO22818	218.2		-0.57	
	normality	OK			
	n	27			
	outliers	3			
	mean (n)	247.619			
	st.dev. (n)	34.5521	RSD=14%		
	R(calc.)	96.746			
	st.dev.(Horwitz n=9)	51.8471			
	R(Horwitz n=9)	145.172			
Compare					
	R(ISO22818:21)	111.627	ISO22818:21 sample A, Table C.1		





APPENDIX 2 Analytical details

lab	ISO/IEC 17025 accr.	sample preparation before use	sample intake (g)	extraction solvent	extraction time (min)	extraction temp. (°C)
623	Yes	Further cut	0.5	toluene	60	60
2115	Yes	Used as received	0.5 g	Hexane	60 min	60°C
2165	Yes	Used as received	1g	Toluene	60min±2	60°C±2
2265	No	Used as received	0,5	toluene	60	60
2272	Yes	Used as received	0.5g	5mL	1hour	60℃
2320	Yes	Further cut	0.5g	Hexane	60 min	60°C
2347	Yes	Used as received	0.5g	methylbenzene	/	/
2350	No	Further cut	0.501 g	Hexane	60 mins	60 ℃
2352	Yes	Used as received	0.5g	toluene	60min	60℃
2358	Yes	Used as received	0.5	Toluene	60	60
2363	Yes	Used as received	0.5g	Toluene	1h	60℃
2365	Yes	Used as received	0.5g	Toluene	60min	60℃
2366	No	Further cut				
2375	No	Further cut	0,5 gram	Toluene	60 min	60 °C
2378	Yes	Used as received	0.5g	toluene	60min	60 °C
2380	Yes	Further cut	0.5 g	Toluene	60 Minutes	60 °C
2382	Yes	Further cut	0.5g	Toluene	60 minutes	60°C
2386	Yes	Used as received	1 g	Toluene	60 min	60°C
2486	Yes	Used as received	1.0065g	Toluene	60 minutes	60°C
2561	No	Used as received	1g	hexane	60	60
2590	Yes	Further cut	1g	Toluene	60 min	60 °C
2612	Yes	Used as received	0,5176g and 0,5213 g Double determination, the average value was formed for the result	Toluene	60 minutes	60°C
2674	Yes	Used as received	2.0	Toluene	60	60
2675	No	Used as received	0.5 g			
2743	No	Used as received	0.5	Toluene	60	60
2826	Yes	Used as received	0.5g	Toluene	60 minutes	60°C
2886	---	---				
2929	Yes	Further cut	0.5	Ethylacetate/Acetone 1:1	60	60
3002	Yes	Further cut	0.495 g	30 mL:10 mL THF+20 mL hexane	30 min vortex	Room temp
3210	Yes	Further cut	1 gram	Toluene	60 min	60°C
3228	Yes	Further cut	0.5	toluene	60	60

APPENDIX 3

Number of participants per country

2 labs in	BANGLADESH
1 lab in	DENMARK
1 lab in	FRANCE
5 labs in	GERMANY
2 labs in	HONG KONG
1 lab in	INDONESIA
4 labs in	ITALY
1 lab in	KOREA, Republic of
11 labs in	P.R. of CHINA
1 lab in	SRI LANKA
1 lab in	TURKEY
1 lab in	UNITED KINGDOM

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
E	= calculation difference between reported test result and result calculated by iis
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
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?

Literature

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