

Institute for Interlaboratory Studies

Results of Proficiency Test SCCP/MCCP in Polymers June 2023



CONTENTS

1		3
2	SET UP	3
2.1	QUALITY SYSTEM	3
2.2	PROTOCOL	4
2.3	CONFIDENTIALITY STATEMENT	4
2.4	SAMPLES	4
2.5	ANALYZES	4
3	RESULTS	5
3.1	STATISTICS	5
3.2	GRAPHICS	6
3.3	Z-SCORES	6
4	EVALUATION	7
4.1	EVALUATION PER SAMPLE AND PER COMPONENT	7
4.2	PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES	8
4.3	COMPARISON OF THE PROFICIENCY TEST OF JUNE 2023 WITH PREVIOUS PTS	9
4.4	EVALUATION OF THE ANALYTICAL RESULTS	10
5	DISCUSSION	10
6	CONCLUSION	10

Appendices:

1.	Data, statistical and graphic results	11
2.	Analytical details	15
3.	Number of participants per country	16
4.	Abbreviations and literature	17

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. SCCP 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 2015 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of SCCP/MCCP in Polymers. During the annual proficiency testing program 2022/2023 it was decided to continue the proficiency test for the determination of SCCP/MCCP in Polymers.

In this interlaboratory study 46 laboratories in 18 countries registered for participation, see appendix 3 for the number of participants per country. In this report the results of the SCCP/MCCP in Polymers 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 an ISO/IEC17025 accredited laboratory. It was decided to send two PVC polymer samples containing SCCP and MCCP labelled #23620 and #23621 respectively.

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

For the first sample a batch of red PVC blocks containing SCCP and MCCP was obtained from a third-party laboratory. After homogenization 60 small plastic bags were filled with approximately 3 grams each and labelled #23620.

The batch for sample #23620 was used in a previous proficiency test on SCCP/MCCP in Polymers as sample #17571 in iis17P05. Therefore, homogeneity of the subsamples was assumed.

For the second sample a batch of black PVC blocks containing SCCP and MCCP was obtained from a third-party laboratory. After homogenization 60 small plastic bags were filled with approximately 3 grams each and labelled #23621.

The batch for sample #23621 was used in a previous proficiency test on SCCP/MCCP in Polymers as sample #19549 in iis19P05. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories two PVC samples labelled #23620 and #23621 respectively were sent on May 24, 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 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 samples as if they were routine samples 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 'c - ' as 's - ' were not used in the statistical

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, 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:

 $\begin{aligned} |z| &< 1 \quad \text{good} \\ 1 &< |z| &< 2 \quad \text{satisfactory} \\ 2 &< |z| &< 3 \quad \text{questionable} \\ 3 &< |z| \quad & \text{unsatisfactory} \end{aligned}$

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 two other participants did not report any test results. Not all participants were able to report all tests requested. In total 44 participants reported 168 numerical test results. Observed were 9 outlying test results, which is 5.4%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

All data sets proved to have a normal Gaussian distribution.

4.1 EVALUATION PER SAMPLE AND PER COMPONENT

In this section the reported test results are discussed per sample and 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.

For the determination of SCCP and MCCP in leather test method ISO18219 is considered to be the official test method. It is unknown if it is applicable for other matrices like polymers. Since 2021 test method ISO22818 became available for the determination of SCCP and MCCP in textile products made of different matrices, especially mentioned is polymer of the coated fabrics, prints made of polymer and buttons made of polymer (e.g. PVC). For the evaluation of the test results in this PT the relative standard deviation (RSD) of SCCP and the RSD of MCCP in polyester textile coated with PVC mentioned in test method ISO22818 was used for the evaluation.

sample #23620	
<u>SCCP</u> :	This determination was not problematic. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO22818:21.
MCCP:	This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ISO22818:21.
sample #23621	
<u>SCCP</u> :	This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO22818:21.
MCCP:	This determination was not problematic. Two statistical outliers were

<u>MCCP</u>: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO22818:21.

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 the reference method are presented in next tables.

Component	unit	n	average	2.8 * sd	R(lit)
SCCP	mg/kg	38	741	325	388
МССР	mg/kg	40	2045	923	922

 Table 1: reproducibilities of components on sample #23620

Component	unit	n	average	2.8 * sd	R(lit)
SCCP	mg/kg	41	1177	626	616
MCCP	mg/kg	40	3109	1078	1402

Table 2: reproducibilities of components on sample #23621

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

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

	June 2023	June 2022	May 2021	June 2020	June 2019
Number of reporting laboratories	44	37	57	43	45
Number of test results	168	134	204	152	154
Number of statistical outliers	9	5	8	10	9
Percentage of statistical outliers	5.4%	3.7%	3.9%	6.6%	5.5%

Table 3: 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 with uncertainties observed in PTs over the years, expressed as relative standard deviation (RSD) of the PTs, see next table.

Component	June 2023	June 2022	May 2021	June 2020	2015 - 2019	target
SCCP	16-19%	23-57%	15-20%	24-52%	13-33%	19%
MCCP	12-16%	20-54%	20%	19-41%	13-39%	16%

Table 4: development of the uncertainties over the years

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

Sample #23620 was used in a previous PT as sample #17571 in iis17P05. The averages found in both PTs for this sample are similar. The calculated reproducibility for the components in this sample improved in the 2023 PT compared to the 2017 PT.

		sample #23620			Sa	ample #175	71
Component	unit	n	average	R(calc)	n	average	R(calc)
SCCP	mg/kg	38	741	325	43	844	552
MCCP	mg/kg	40	2045	923	38	2184	1198

Table 5: comparison of sample #23620 with #17571

Sample #23621 was used in a previous PT as sample #19549 in iis19P05. The averages found in both PTs for this sample are similar. The calculated reproducibility for the components in this sample improved in the 2023 PT compared to the 2019 PT.

		sample #23621			Sa	ample #1954	49
Component	unit	n	average	R(calc)	n	average	R(calc)
SCCP	mg/kg	41	1177	626	43	1197	886
MCCP	mg/kg	40	3109	1078	31	3268	1187

 Table 6: comparison of sample #23621 with #19549

4.4 EVALUATION OF THE ANALYTICAL DETAILS

About 66% of the participants reported to have used ISO22818 as test method and 18% of the participants reported to have used ISO18219. For this PT some analytical details were requested, the reported details are given in appendix 2. Based on the answers given by the participants the following can be summarized:

- 77% of the participants mentioned that they are accredited to determine the reported component(s).
- 38% of the participants used the sample as received and 62% did further cut or further grind the samples prior to analysis.
- 81% of the participants used a sample intake between 0.5 1 grams.
- 90% of the participants reported to have used Toluene or Toluene/Hexane as extraction solvent.
- Almost all participants used an extraction time of 60 minutes and an extraction temperature of 60 °C.

For SCPP and MCCP the calculated reproducibility is in agreement with the requirements of the target reproducibility, therefore no further 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. When the results of this interlaboratory study were compared to this regulation, it was noticed that not all participants would have made identical decisions about the acceptability of the samples for SCCP. For sample #23620 almost all reporting laboratories, except three, would have accepted this sample for SCCP.

For sample #23621 almost all reporting laboratories, except five, would have accepted this sample for SCCP.

6 CONCLUSION

It can be concluded that most of the participants have no problem with the determination of SCCP and/or MCCP in this PT. However, each 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 #23620; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339	In house	463 1	С	-2 01	First reported 664
623	ISO22818	1259 68	C R(0.01)	3 74	First reported 1077 13
840	15022818	780	0,11(0.01)	0.74	
Q/1	19022010	735		0.20	
1000	13022010	2050 74		-0.03	
1099		2000.74	R(0.01)	9.50	
2117	15018219-1:2021	932		1.37	
2232					
2265	10000010				
2297	ISO22818	820		0.57	
2310	ISO18219-1:2021	741		0.00	
2311	ISO22818	613.6		-0.92	
2326	ISO22818	1916.7	C,R(0.01)	8.48	First reported 19217.4
2347	ISO22818	797.13		0.40	
2350	ISO22818	998		1.85	
2352	ISO22818	700		-0.30	
2353	ISO22818	806.14		0.47	
2355	ISO22818	845.5800		0.75	
2357	ISO22818	725.1		-0.12	
2358	ISO22818	806.14		0.47	
2363	ISO22818	756.372		0.11	
2365	ISO22818	704.1		-0.27	
2366	ISO22818	703		-0.28	
2370	ISO22818	765		0.17	
2375	ISO22818	618		-0.89	
2378	ISO22818	754		0.09	
2380	ISO18219-1-2015	652.86		-0.64	
2384	In house	758 56	C	0.12	First reported 1158 93
2386	ISO18219-1·2021	704	0	_0.12	
2488	ISO18219-1.2021	997 63		1.85	
2531	100 102 13	007.00	\٨/	1.00	Test result withdrawn, reported 1408
2501	15022818	500 20	vv	1.00	rest result withdrawn, reported 1430
2090	13022010	390.29		-1.09	
2049	16000010	702 1427		0.27	
2131	15022010	792.1427		0.37	
2009	15022010	740		-0.01	
2820	15018219-1:2021	537	0	-1.47	First new outs of 444
2929	In nouse	790	C	0.35	First reported 414
2959	ISO22818	697		-0.32	
2960	18022818	828.1		0.63	
3003	In house	5/3		-1.21	
3004	ISO18219-1:2021	578.50		-1.18	
3018	ISO22818	905.33		1.18	
3163	In house	610000	R(0.01)	4394.39	
3179	ISO18219-1:2021	646		-0.69	
3210	ISO22818	792.41		0.37	
3214	ISO22818	728.2		-0.10	
3218	ISO22818	799.50		0.42	
	normality	OK			
	n	38			
	outliers	4			
	mean (n)	741.415			
	st.dev. (n)	116.0461	RSD = 16%		
	R(calc.)	324.929			
	st.dev.(ISO22818:21)	138.6447			
	R(ISO22818:21)	388.205			



Determination of MCCP, CAS No 85535-85-9 on sample #23620; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339	In house	2472.8	С	1.30	First reported 4083
623	ISO22818	2245.63	-	0.61	· ······
840	ISO22818	1994		-0.15	
841	ISO22818	1883		-0.49	
1099					
2117	ISO18219-2-2021	2038		-0.02	
2232					
2265					
2297	ISO22818	2188		0.43	
2310	ISO18219-2:2021	1700		-1.05	
2311	ISO22818	1436.4		-1.85	
2326	ISO22818	2822.1	С	2.36	First reported 5684.0
2347	ISO22818	2128.09		0.25	
2350	ISO22818	2484		1.33	
2352	ISO22818	1853		-0.58	
2353	ISO22818	2171.43		0.38	
2355	ISO22818	2049.9700		0.02	
2357	ISO22818	1812.1		-0.71	
2358	ISO22818	2171.43		0.38	
2363	ISO22818	1901.544		-0.44	
2365	ISO22818	1768.9		-0.84	
2366	ISO22818	1778		-0.81	
2370	ISO22818	1910		-0.41	
2375	ISO22818	1814		-0.70	
2378	ISO22818	1828		-0.66	
2380	ISO18219-1:2015	2088.55		0.13	
2384	In house	2198.40	С	0.47	First reported 2271.35
2386	ISO18219-2:2021	2464		1.27	
2488	ISO18219	1291.59		-2.29	
2531	In house	2259		0.65	
2590	ISO22818	2737.51	С	2.10	First reported 3013.73
2649					•
2737	ISO22818	2067.4555		0.07	
2809	ISO22818	2034		-0.03	
2826	ISO18219-2:2021	1720		-0.99	
2929	In house	670	C,R(0.01)	-4.18	First reported 3975
2959	ISO22818	2066		0.06	
2960	ISO22818	2122.2		0.23	
3003	In house	2400		1.08	
3004	ISO22818	2266.55		0.67	
3018	ISO22818	2408.0		1.10	
3163					
3179	ISO18219-2:2021	1340		-2.14	
3210	ISO22818	1835.24		-0.64	
3214	ISO22818	1981.6		-0.19	
3218	ISO22818	2065.04		0.06	
	normality.	OK			
	normality				
	outliere	40 1			
	outilers	1			
	nicali (II) st dov. (n)	2044.000	DSD - 160/		
	R(calc)	022 614	10%		
	et dev (ISO22212-21)	322.014			
	R(ISO22010.21)	929.2270			
	110022010.21)	521.000			



Determination of SCCP, CAS No 85535-84-8 on sample #23621; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339	In house	694.4	С	-2.19	First reported 722
623	ISO22818	1619.66		2.01	
840	ISO22818	1309		0.60	
841	ISO22818	1116		-0.28	
1099	In house	3346.98	R(0.01)	9.86	
2117	ISO18219-1:2021	1496		1.45	
2232	ISO18219-1:2021	908		-1.22	
2265	10000040				
2297	15022818	1297		0.54	
2310	ISO 102 19-1.202 1	1100		-0.06	
2326	ISO22010 ISO22818	1656 5	C	-0.20	First reported 20187 9
2347	ISO22818	1282 32	0	0.48	
2350	ISO22818	1473		1.34	
2352	ISO22818	1071		-0.48	
2353	ISO22818	1031.22		-0.66	
2355	ISO22818	1245.5000		0.31	
2357	ISO22818	1222.1		0.20	
2358	ISO22818	1301.22		0.56	
2363	ISO22818	1230.754		0.24	
2365	ISO22818	1101.2		-0.35	
2366	ISO22818	1125		-0.24	
2370	ISO22818	1280		0.47	
2375	15022818	1025		-0.69	
23/8	15022818	1082		-0.43	
2300 2384	15016219-1.2015	1020.13	C	-0.00	First reported 1582 73
2386	ISO18219-1-2021	1086	0	-0.41	This reported 1002.75
2488	ISO18219	1388 58		0.96	
2531	10010210		W		Test result withdrawn reported 2259
2590	ISO22818	1009.14		-0.76	
2649					
2737	ISO22818	1330.9821		0.70	
2809		1140		-0.17	
2826	ISO18219-1:2021	925		-1.15	
2929	In house	664		-2.33	
2959	ISO22818	1056		-0.55	
2960	15022818	1310.1		0.60	
3003		890 1099 55		-1.30	
3004	15022010	1614 7		-0.40	
3163	In house	684000	R(0.01)	3101.90	
3179	ISO18219-1:2021	951		-1.03	
3210	ISO22818	1076.83		-0.46	
3214	ISO22818	1115.3		-0.28	
3218	ISO22818	1299.76		0.56	
	normality	OK			
	n "	41			
	outliers	2			
	mean (n)	11/1.1/0	PSD - 10%		
	SLUEV. (II) R(calc.)	223.3002	KOD - 19%		
	st dev (ISO22818·21)	220.041			
	R(ISO22818:21)	616.366			
2000 т					0.0025



Determination of MCCP, CAS No 85535-85-9 on sample #23621; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339	In house	3892.4	С	1.56	First reported 5908
623	ISO22818	3308.53		0.40	·
840	ISO22818	3202		0.18	
841	ISO22818	2931		-0.36	
1099					
2117	ISO18219-2:2021	3179		0.14	
2232	ISO18219-2:2021	2970		-0.28	
2265					
2297	ISO22818	3110		0.00	
2310	ISO18219-2:2021	2988		-0.24	
2311	ISO22818	2293.9		-1.63	
2326	ISO22818	3351.2	С	0.48	First reported 10957.7
2347	ISO22818	3321.94		0.42	
2350	ISO22818	3337		0.45	
2352	ISO22818	2750		-0.72	
2353	ISO22818	3379.13		0.54	
2355	ISO22818	3350.3400		0.48	
2357	ISO22818	2971.1		-0.28	
2358	ISO22818	3379.13		0.54	
2363	ISO22818	3020.256		-0.18	
2365	ISO22818	2830.6		-0.56	
2366	ISO22818	2763		-0.69	
2370	ISO22818	3150		0.08	
2375	ISO22818	2960		-0.30	
2378	ISO22818	2782		-0.65	
2380	ISO18219-1:2015	3359.75		0.50	
2384	In house	2923.72	С	-0.37	First reported 3627.05
2386	ISO18219-2:2021	3158		0.10	
2488	ISO18219	2183.83		-1.85	
2531	In house	3012		-0.19	
2590	ISO22818	3796.70		1.37	
2649					
2737	ISO22818	2954.5274		-0.31	
2809		2950		-0.32	
2826	ISO18219-2:2021	2688		-0.84	
2929	In house	823	C,R(0.01)	-4.57	First reported 5872
2959	ISO22818	2501		-1.22	
2960	ISO22818	3430.2		0.64	
3003	In house	3500		0.78	
3004	ISO22818	3865.41	0	1.51	F : 1 1 1015 0
3018	15022818	3857	C	1.49	First reported 4345.0
3163	10010010 0:0001	4740		0.70	
31/9	15018219-2:2021	1743	K(U.U5)	-2.13	
3210	15022818	2826.24		-0.57	
3214	15022818	2945.0		-0.33	
3218	15022818	3206.08		0.19	
	pormality	OK			
	normanty	40			
	outliers	2			
	mean (n)	3109 465			
	st dev (n)	384 0343	RSD = 12%		
	R(calc.)	1077 816	100 - 12/0		
	st dev (ISO22818-21)	500 6239			
	R(ISO22818·21)	1401 747			



APPENDIX 2 Analytical details

lab	ISO/IEC17025 accredited	sample preparation before use	sample intake (g)	extraction solvent	extraction time (minutes)	extraction temp. (°C)
339	Yes	Further cut	0.5	Toluène	60	60
623	Yes		0.5	toluene	60	60
840	Yes	Further cut	0.5	TOLUENE	60	60
841	Yes	Further cut	0.5g	Toluene/ n-Hexane	60 min	60°C
1099	No	Used as received	2 g	toluene into hexane	60 +/- 2	60 +/- 2
2117	Yes	Used as received	~0,5	Toluene	60	60
2232	Yes	Used as received	1g	Toluene	60min	60C
2297	Yes	Used as received	0.5	toluene	60	60
2310	Yes	Further cut	0.5	Toluene	60	60
2311	Yes	Further cut	0.5	Toluene	60	60
2326	Yes	Further cut	0.5 gm	Toulene / n-Hexane	60	60
2347	Yes	Further cut	0.50g	/	60mins	60°C
2350	No	Further cut	0.5 g	Toluene	60 min	60 °C
2352	Yes	Further cut	0.5g	toluene	60min	60°C
2353	Yes	Used as received	0.5	Toluene	60	60
2355	Yes	Further cut	0.5g	Toluene	60min	60°C
2358	Yes	Used as received	0.5	toluene	60	60
2363	Yes	Further cut	0.5g	toluene	60mins	60°C
2365	Yes	Further cut	0.5g	Toluene	60min	60°C
2366	No	Further cut	0.5	Toluene	60	60
2370	Yes	Further cut	1.5g	Toluene	60 min	60°C
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	Used as received	0.5 g	Toluene	60 Minute	60 °C
2384	Yes	Further grinded	0.3g	toluene	60 min	60 °C
2386	Yes	Used as received	1 g	Toluole	60 min	60°C
2488	Yes	Used as received	0.25 g	n-Hexane	60 min	60 °C
2590	No	Used as received	1g	toluene	60min	60°C
2737	Yes	Further cut	0.5	Toluene	60min	60°C
2809	Yes	Further cut	0.5	Toluene	60	60
2826	Yes	Used as received	0.2523 & 0.2547g	Toluene	60 minutes	60°C
2929	Yes	Further cut	0,2	Ethylacetate / Acetone 1:1	60	60
2959	Yes	Further cut	1	Toluene	60	60
2960	Yes	Used as received	0.5	Toluene+Hexane	60min	60
3003	No	Further cut	0.5 gm	n hexane	1 hr sonication	60 °C
3004	No	Further grinded	0.5g	toluene	60min	60°C
3179	Yes	Used as received	~ 0.5 g	toluene	60 min	60 °C
3210	Yes	Further cut	0.5 grams	Toluene	60 minutes	60°C
3214	Yes	Further cut	0.5 gram	toluene	60 min	60 degree
3218	Yes	Used as received	0.5g	Toluene	60min.	60°C

APPENDIX 3

Number of participants per country

3 labs in BANGLADESH

- 2 labs in FRANCE
- 6 labs in GERMANY
- 3 labs in HONG KONG
- 2 labs in INDIA
- 1 lab in INDONESIA
- 2 labs in ITALY
- 1 lab in JAPAN
- 1 lab in KOREA, Republic of
- 1 lab in MALAYSIA
- 13 labs in P.R. of CHINA
 - 1 lab in PAKISTAN
 - 1 lab in POLAND
 - 1 lab in SINGAPORE
- 2 labs in TAIWAN
- 1 lab in THE NETHERLANDS
- 2 labs in TURKEY
- 3 labs in VIETNAM

APPENDIX 4

Abbreviations

С	= 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

- 1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
- 2 ISO5725:86
- 3 ISO5725 parts 1-6:94
- 4 ISO13528:05
- 5 M. Thompson and R. Wood, J. AOAC Int, <u>76</u>, 926, (1993)
- 6 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 7 P.L. Davies, Fr. Z. Anal. Chem, <u>331</u>, 513, (1988)
- 8 J.N. Miller, Analyst, <u>118</u>, 455, (1993)
- 9 Analytical Methods Committee, Technical Brief, No 4, January 2001
- 10 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, <u>127</u>, 1359-1364, (2002)
- 11 W. Horwitz and R. Albert, J. AOAC Int, <u>79, 3</u>, 589-621, (1996)
- 12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, <u>25(2)</u>, 165-172, (1983)