Institute for
Interlaboratory Studies

Results of Proficiency Test
SCCP in Polymers
June 2022

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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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. 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 in Polymers. During the annual proficiency testing program 2021/2022 it was decided to continue the proficiency test for the determination of SCCP/MCCP in Polymers.

In this interlaboratory study 42 laboratories in 19 countries registered for participation, see appendix 3 for the number of participants per country. In this report the results of the SCCP 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 different polymer samples of approximately 3 grams each, both positive on SCCP and MCCP, labelled #22630 and #22631.
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 yellow Acrylonitrile Butadiene Styrene (ABS) granulates was selected which was made positive on SCCP and MCCP by a third-party laboratory. After homogenization 60 small plastic bags were filled with approximately 3 grams each and labelled #22630.
The homogeneity of the subsamples was checked by determination of the SCCP content using an in-house test method on 8 stratified randomly selected subsamples.

	SCCP in mg/kg
sample #22630-1	1049
sample #22630-2	1020
sample #22630-3	1028
sample #22630-4	1007
sample #22630-5	1031
sample #22630-6	1044
sample #22630-7	1031
sample #22630-8	985

Table 1: homogeneity test results of subsamples #22630

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

	SCCP in mg/kg
r (observed)	58
reference test method	ISO22818:21
0.3 x R (reference test method)	161

Table 2: evaluation of the repeatability of subsamples #22630

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

For the second sample a batch of green colored PVC rings was selected which was made positive on SCCP and MCCP by a third-party laboratory. After homogenization 60 small plastic bags were filled with approximately 3 grams each and labelled #22631.
The homogeneity of the subsamples was checked by determination of the SCCP content using an in-house test method on 10 stratified randomly selected subsamples.

	SCCP in mg/kg
sample #22631-1	2336
sample #22631-2	2384
sample #22631-3	2239
sample #22631-4	2382
sample #22631-5	2244
sample #22631-6	2325
sample #22631-7	2376
sample #22631-8	2320
sample #22631-9	2357
sample #22631-10	2382

Table 3: homogeneity test results of subsamples #22631

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

	SCCP in mg/kg
r (observed)	153
reference test method	ISO22818:21
0.3 x R (reference test method)	367

Table 4: evaluation of the repeatability of subsamples #22631

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

To each of the participating laboratories one sample labelled #22630 and one sample labelled #22631 was sent on May 11, 2022.

2.5 ANALYZES

The participants were requested to determine: SCCP and MCCP. 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 ‘<…’ 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.
Five participants reported test results after the final reporting date and five other participants were not able to report any test results. Not all participants were able to report all tests requested.
In total 37 participants reported 134 numerical test results. Observed were 5 outlying test results, which is 3.7%. 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 this test method was used for the evaluation.

sample #22630

SCCP: This determination was very problematic. No statistical outliers were observed. The calculated reproducibility is not at all in agreement with the requirements of ISO22818:21. It was decided not to calculate z-scores due to the large variation in the reported test results, see paragraph 4.4 for more discussion.

MCCP: This determination was very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of ISO22818:21. It was decided not to calculate z-scores due to the large variation in the reported test results, see paragraph 4.4 for more discussion.

sample #22631

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

MCCP: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not 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 reference methods are presented in next tables.

Component	unit	n	average	2.8 * sd	R(lit)
SCCP	mg/kg	36	637	1014	(333)
MCCP	mg/kg	29	1622	2431	(731)

Table 5: performance overview on sample #22630

For results between brackets no z-scores are calculated.

Component	unit	n	average	2.8 * sd	R(lit)
SCCP	mg/kg	35	2262	1438	1184
MCCP	mg/kg	29	6161	3398	2777

Table 6: performance overview on sample #22631

Without further statistical calculations it can be concluded that for SCCP and MCCP there is not a good compliance of the group of participants with the reference test method. The problematic tests have been discussed in paragraphs 4.1 and 4.4.

4.3 COMPARISON OF THE PROFICIENCY TEST OF JUNE 2022 WITH PREVIOUS PTS

	June 2022	May 2021	June 2020	June 2019	May 2018
Number of reporting laboratories	37	57	43	45	66
Number of test results	134	204	152	154	216
Number of statistical outliers	5	8	10	9	8
Percentage of statistical outliers	3.7%	3.9%	6.6%	5.5%	3.6%

Table 7: 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	June 2022	May 2021	June 2020	June 2019	2015 - 2018	target
SCCP	23-57%	15-20%	24-52%	18-27%	13-33%	19%
MCCP	20-54%	20%	19-41%	13-33%	18-39%	16%

Table 8: development of the uncertainties over the years

Only for the PVC sample #22631 the uncertainties observed in this PT are comparable to the uncertainties observed in previous PTs.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

About 39% of the participants reported to have used ISO22818 as test method and 53% 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:

- About 72% of the reporting participants mentioned that they are accredited to determine the reported component(s).
- About 47% of the reporting participants used the sample as received and about 53% did further cut or further grind the samples prior to analysis.
- Almost all the reporting participants used a sample intake between 0.5 - 1 grams.
- About 85% 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.

The test results of the determination of SCCP/MCCP in ABS sample #22630 shows a large variation. The effect of some of the reported analytical details on the SCCP determination was further investigated to possibly explain the high variation in the test results.

subgroup	n	SCCP in mg/kg	RSD (%)
ISO22818	14	565	56
ISO18219	19	671	61
Used as received	16	737	62
Further cut/grinded	17	588	44
Toluene/Toluene & Hexane	30	642	55

Table 9: effect of analytical details on the determination of SCCP in ABS granulates sample #22630

Remarkably, further cutting or grinding the ABS granulates sample before use tends to give a lower level of SCCP and a smaller reproducibility. Please note that the observed effect is not statistically significant. The large variation in the test results of the determination of SCCP in sample #22630 is not caused by just one critical point in the analysis. A clear cause cannot be identified and therefore it was decided not to use a specific group of test results to calculate the assigned values to calculate the z-scores.

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 make identical decisions about the acceptability of the samples for SCCP.
For the PVC sample #22630 all reporting laboratories would have accepted this sample for SCCP.
For the ABS sample #22631 almost all reporting laboratories would have rejected this sample, however two laboratories would have accepted this sample for SCCP.

In this proficiency test two different polymers were used. Sample #22630 is made of Acrylonitrile Butadiene Styrene (ABS) and sample #22631 of Polyvinylchloride (PVC). The observed reproducibility of the ABS sample #22630 was much higher than the reproducibility of the PVC sample #22631. It occurs that releasing SCCP and MCCP from an ABS matrix is more difficult than from PVC.

6 CONCLUSION

It can be concluded that most of the participants were able to determine SCCP and/or MCCP in this PT. However, it is noted that there is a large difference in the evaluations depending on the type of the polymer. For the analysis of SCCP from polymers a sound test method which prescribe the analysis of SCCP from different polymers in detail is desirable, especially for other polymers than PVC.

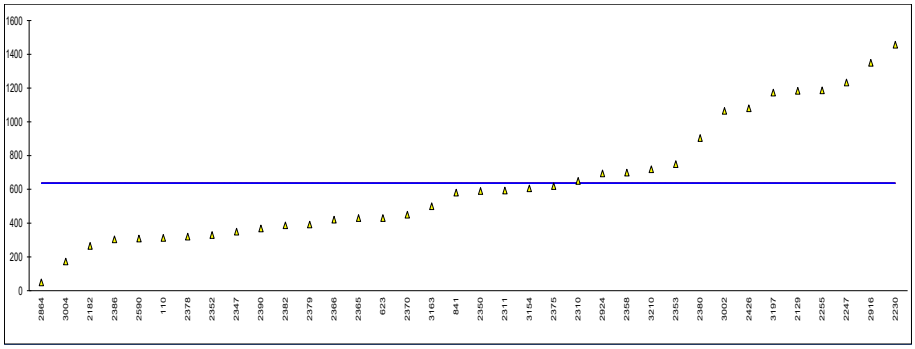
Each laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

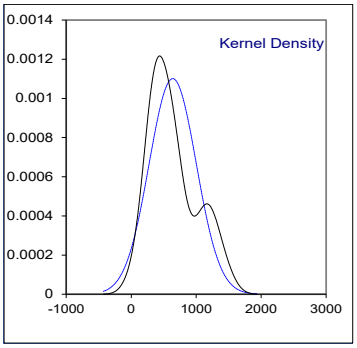
APPENDIX 1

Determination of SCCP on sample #22630; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO18219-1:2021	312.3		-----	
623	ISO22818	430.7		-----	
841	ISO22818	581		-----	
2129	ISO18219-1:2021	1184		-----	
2182	ISO18219:2015	265.2		-----	
2230	ISO18219-1:2021	1457		-----	
2247	ISO22818	1233.02		-----	
2255	ISO18219-1:2021	1187.0		-----	
2265		-----		-----	
2267		-----		-----	
2310	ISO18219-1:2021	650		-----	
2311	ISO18219-1:2021	593.3		-----	
2330		-----		-----	
2347	ISO18219-1:2021	349.7		-----	
2350	ISO18219-1:2021	589.8		-----	
2352	ISO22818	329.1		-----	
2353	ISO22818	750		-----	
2358	ISO18219-1:2021	700		-----	
2363		-----		-----	
2365	ISO18219-1:2021	430.49		-----	
2366	ISO18219-1:2021	421		-----	
2370	ISO22818	450		-----	
2375	ISO22818	619		-----	
2378	ISO18219-1:2021	320		-----	
2379	ISO22818	391.9994		-----	
2380	ISO18219:2015	904.04		-----	
2382	ISO22818	387.0		-----	
2386	ISO18219-1:2021	303.9		-----	
2390	ISO22818	368.1		-----	
2426	ISO18219	1080.73		-----	
2531		-----		-----	
2590	ISO22818	308.46		-----	
2864	ISO18219-1:2021	48.75		-----	
2886		-----		-----	
2916	ISO18219-1:2021	1350		-----	
2924	IEC62321-14	694.5		-----	
3002	In house	1065		-----	
3004	ISO22818	173.04		-----	
3154	ISO18219-1:2021	606.00		-----	
3163	In house	500		-----	
3197	ISO22818	1173.4		-----	
3210	ISO22818	719.44		-----	

	normality	OK	
	n	36	
	outliers	0	
	mean (n)	636.860	
	st.dev. (n)	362.3149	RSD=57%
	R(calc.)	1014.482	
	st.dev.(ISO22818:21)	(119.0929)	
	R(ISO22818:21)	(333.460)	

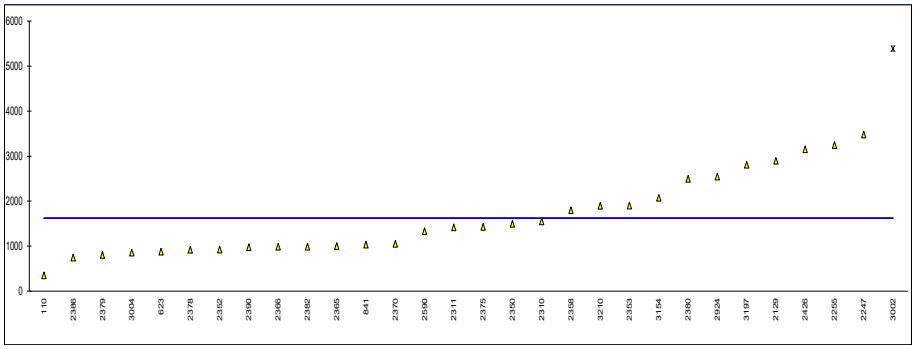


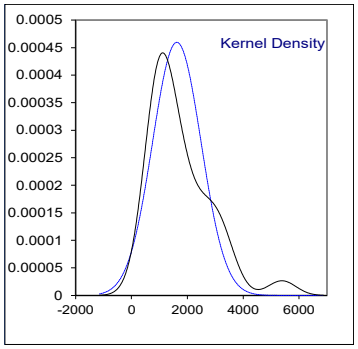


Determination of MCCP on sample #22630; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO18219-2:2021	350.0		-----	
623	ISO22818	878.9		-----	
841	ISO22818	1034		-----	
2129	ISO18219-2:2021	2895		-----	
2182		-----		-----	
2230		-----		-----	
2247	ISO22818	3480.85		-----	
2255	ISO18219-2:2021	3247.0		-----	
2265		-----		-----	
2267		-----		-----	
2310	ISO18219-2:2021	1550		-----	
2311	ISO18219-2:2021	1418		-----	
2330		-----		-----	
2347		-----		-----	
2350	ISO18219-2:2021	1497.38		-----	
2352	ISO22818	924.3		-----	
2353	ISO22818	1900		-----	
2358	ISO18219-2:2021	1800		-----	
2363		-----		-----	
2365	ISO18219-2:2021	1001.88		-----	
2366	ISO18219-2:2021	987		-----	
2370	ISO22818	1050		-----	
2375	ISO22818	1430		-----	
2378	ISO18219-2:2021	920		-----	
2379	ISO22818	803.7854		-----	
2380	ISO18219:2015	2494.80		-----	
2382	ISO22818	989.0		-----	
2386	ISO18219-2:2021	746.6		-----	
2390	ISO22818	978.3		-----	
2426	ISO18219	3154.06		-----	
2531		-----		-----	
2590	ISO22818	1334.60		-----	
2864		-----		-----	
2886		-----		-----	
2916		-----		-----	
2924	IEC62321-14	2546.3		-----	
3002	In house	5389	R(0.01)	-----	
3004	ISO22818	856.32		-----	
3154	ISO18219-2:2021	2073.02		-----	
3163		-----		-----	
3197	ISO22818	2809.2		-----	
3210	ISO22818	1897.30		-----	

	normality	OK
	n	29
	outliers	1
	mean (n)	1622.331
	st.dev. (n)	868.2245 RSD=54%
	R(calc.)	2431.029
	st.dev.(ISO22818:21)	(261.1953)
	R(ISO22818:21)	(731.347)



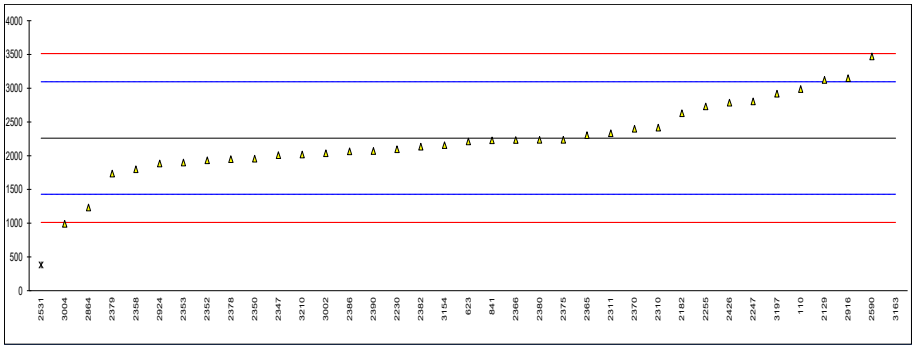


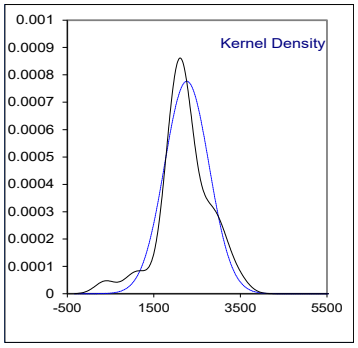
Determination of SCCP on sample #22631; results in mg/kg

ab	method	value	mark	z(targ)	remarks
110	ISO18219-1:2021	2987.4		1.71	
623	ISO22818	2212.4		-0.12	
841	ISO22818	2227		-0.08	
2129	ISO18219-1:2021	3122		2.03	
2182	ISO18219:2015	2630.6	C	0.87	first reported 4998.2
2230	ISO18219-1:2021	2096		-0.39	
2247	ISO22818	2805.39		1.28	
2255	ISO18219-1:2021	2730.0		1.11	
2265		-----		-----	
2267		-----		-----	
2310	ISO18219-1:2021	2417		0.37	
2311	ISO18219-1:2021	2333.6		0.17	
2330		-----		-----	
2347	ISO18219-1:2021	2008.3		-0.60	
2350	ISO18219-1:2021	1957.2		-0.72	
2352	ISO22818	1933.2		-0.78	
2353	ISO22818	1900		-0.86	
2358	ISO18219-1:2021	1800		-1.09	
2363		-----		-----	
2365	ISO18219-1:2021	2305.06		0.10	
2366	ISO18219-1:2021	2232		-0.07	
2370	ISO22818	2400		0.33	
2375	ISO22818	2238		-0.06	
2378	ISO18219-1:2021	1950		-0.74	
2379	ISO22818	1736.8446		-1.24	
2380	ISO18219:2015	2235.53		-0.06	
2382	ISO22818	2134.0		-0.30	
2386	ISO18219-1:2021	2065.3		-0.47	
2390	ISO22818	2069.8		-0.45	
2426	ISO18219	2788.64		1.24	
2531	In house	381.02	C,R(0.05)	-4.45	first reported 652
2590	ISO22818	3472.49	C	2.86	first reported 5387.15
2864	ISO18219-1:2021	1234.82	C	-2.43	first reported 767.17
2886		-----		-----	
2916	ISO18219-1:2021	3148		2.09	
2924	IEC62321-14	1884.7		-0.89	
3002	In house	2036		-0.53	
3004	ISO22818	990.51		-3.01	
3154	ISO18219-1:2021	2157.54		-0.25	
3163	In house	6600	R(0.01)	10.25	
3197	ISO22818	2919.7		1.55	
3210	ISO22818	2018.64		-0.58	

	normality	OK
	n	35
	outliers	2
	mean (n)	2262.219
	st.dev. (n)	513.6375
	R(calc.)	1438.185
	st.dev.(ISO22818:21)	423.0350
	R(ISO22818:21)	1184.498

RSD=23%

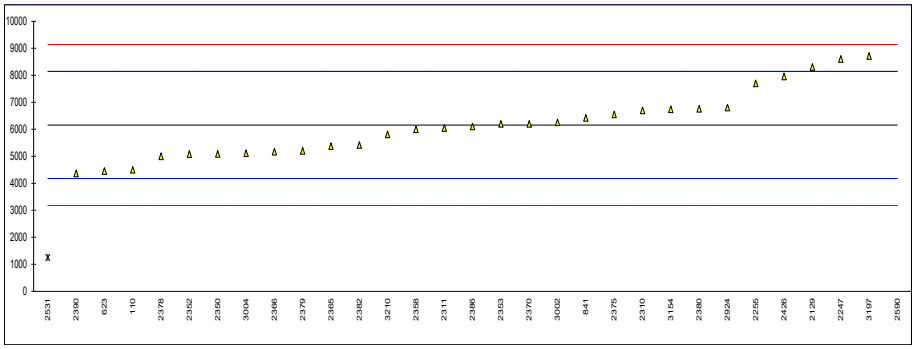


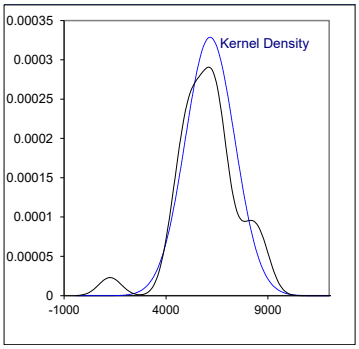


Determination of MCCP on sample #22631; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO18219-2:2021	4499.1		-1.68	
623	ISO22818	4454.7		-1.72	
841	ISO22818	6421		0.26	
2129	ISO18219-2:2021	8303		2.16	
2182		-----		-----	
2230		-----		-----	
2247	ISO22818	8607.35		2.47	
2255	ISO18219-2:2021	7700.0		1.55	
2265		-----		-----	
2267		-----		-----	
2310	ISO18219-2:2021	6698		0.54	
2311	ISO18219-2:2021	6045.8		-0.12	
2330		-----		-----	
2347		-----		-----	
2350	ISO18219-2:2021	5089.94		-1.08	
2352	ISO22818	5080.0		-1.09	
2353	ISO22818	6200		0.04	
2358	ISO18219-2:2021	6000		-0.16	
2363		-----		-----	
2365	ISO18219-2:2021	5377.40		-0.79	
2366	ISO18219-2:2021	5168		-1.00	
2370	ISO22818	6200		0.04	
2375	ISO22818	6553		0.40	
2378	ISO18219-2:2021	5000		-1.17	
2379	ISO22818	5199.8750		-0.97	
2380	ISO18219:2015	6760.59		0.60	
2382	ISO22818	5419.0		-0.75	
2386	ISO18219-2:2021	6103.3		-0.06	
2390	ISO22818	4366.8		-1.81	
2426	ISO18219	7960.72		1.81	
2531	In house	1254.5	R(0.05)	-4.95	
2590	ISO22818	22619.94	C,R(0.01)	16.59	first reported 37483.57
2864		-----		-----	
2886		-----		-----	
2916		-----		-----	
2924	IEC62321-14	6805.0		0.65	
3002	In house	6260		0.10	
3004	ISO22818	5114.31		-1.05	
3154	ISO18219-2:2021	6741.30		0.59	
3163		-----		-----	
3197	ISO22818	8716.0		2.58	
3210	ISO22818	5813.12		-0.35	

normality	OK	
n	29	
outliers	2	
mean (n)	6160.596	
st.dev. (n)	1213.4878	RSD=20%
R(calc.)	3397.766	
st.dev.(ISO22818:21)	991.8559	
R(ISO22818:21)	2777.197	





APPENDIX 2 Analytical details

lab	ISO/IEC17025 accredited	sample preparation before use	sample intake (g)	extraction solvent	extraction time (minutes)	extraction temp. (°C)
110	Yes	Used as received	0.5 g	toluene/hexane	60 min	60°C
623	Yes	Further cut	0.5	toluene	60	60
841	Yes	Further cut	0.5 g	Toluene	60 min	60°C
2129	Yes	Used as received	0.4g	Toluene	60min	60°C
2182	---	---				
2230	Yes	Used as received	1.005	toluene	60min	60°C
2247	Yes	Used as received	0.5g	Toluene & n-Hexane	60.0	60.0
2255	Yes	Further cut	0.5	n-Hexane	60	60
2265	---	---				
2267	---	---				
2310	Yes	Further cut	0.5	Toluene	60	60
2311	No	Further cut	0.5	Toluene	60	60
2330	---	---				
2347	Yes	Further cut	0.5g			
2350	Yes	Further cut	0.5g	Hexane	60min	60°C
2352	Yes	Further cut	0.5g	Toluene	60min	60℃
2353	No	Further cut	0.5	Toluene	60	60
2358	Yes	Further cut	0.5	Toluene	60	60
2363	---	---				
2365	Yes	Further cut	0.5g	Toluene	60min	60℃
2366	Yes	Further cut	0.5	toluene	60	60
2370	Yes	Used as received	1 g	Toluene	60 min	60℃
2375	No	Further cut	0.5g	Toluene	60min	60°C
2378	No	Used as received				
2379	No	Further cut	0.5 g	Toluene	60 min	60 °C
2380	Yes	Used as received	0.5 g	Toluene	60 Min	60 °C
2382	Yes	Used as received	0.5g	Toluene	60min	60°C
2386	Yes	Used as received	0,5 g	Toluol	60 min	60 °C
2390	Yes	Further cut/Used as received	0.5g	Toluene / n-Hexane	60min	60°C
2426	Yes	Further cut	0.5g	Toluene / n-Hexane	60min	60°C
2531	No	Further cut	0.5	Toluene	30, 3 times repeated	60
2590	Yes	Used as received	1g	toluene	60 min	60°C
2864	Yes	Used as received	0.5	Hexane	60	60
2886	---	---				
2916	No	Used as received	0.25	Toluene	60	60
2924	Yes	Used as received	0.1 g	Toluene	60 min	60
3002	Yes	Used as received	0.3 g	THF + Hexane	30 min	RT
3004	No	Further grinded	0.5g	toluene	60min	60℃
3154	Yes	Used as received	0.5 g	Toluene	60	60
3163	No	---	0.02g	ethanol	2h	100C
3197	Yes	Used as received	0,5 g	Toluene/n-hexane	60 + 15 min	60 C
3210	No	Further cut	0.5 g	Toluene	60 min	60°C

APPENDIX 3

Number of participants per country

2 labs in BANGLADESH
1 lab in CAMBODIA
1 lab in DENMARK
1 lab in FRANCE
5 labs in GERMANY
3 labs in HONG KONG
3 labs in INDIA
1 lab in INDONESIA
3 labs in ITALY
1 lab in JAPAN
2 labs in KOREA, Republic of
7 labs in P.R. of CHINA
2 labs in PAKISTAN
3 labs in TAIWAN
1 lab in THAILAND
2 labs in THE NETHERLANDS
2 labs in TURKEY
1 lab in U.S.A.
1 lab 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
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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