Results of Proficiency Test
Total SCCP in Polymers
May 2021

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. SCCPs were categorized in group 2B as possibly carcinogenic to humans from the International Agency for Research on Cancer (IARC). SCCPs (chlorine content >48%) are listed by the Stockholm Convention on Persistent Organic Pollutants. In Europe SCCPs 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 SCCPs in concentrations lower than 0.15% by weight are allowed. Furthermore, it became industrial practice to restrict MCCPs as well.

Since 2015 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of total SCCP in Polymers. During the annual proficiency testing program 2020/2021 it was decided to continue the proficiency test for the analysis of SCCP/MCCP in Polymers.

In this interlaboratory study 58 laboratories in 18 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 (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, positive on SCCP and MCCP, labelled #21605 and #21606.
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 white colored PVC rings was selected which was made positive on SCCP and MCCP by a third-party laboratory. After homogenization 80 small plastic bags were filled with approximately 3 grams each and labelled #21605.
The homogeneity of the subsamples was checked by determination of the total SCCP content using an in-house test method on 8 stratified randomly selected subsamples.

	total SCCP in mg/kg
Sample #21605-1	751
Sample #21605-2	797
Sample #21605-3	806
Sample #21605-4	792
Sample #21605-5	774
Sample #21605-6	781
Sample #21605-7	788
Sample #21605-8	761

Table 1: homogeneity test results of the subsamples #21605

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.

	total SCCP in mg/kg
r (observed)	19
reference test method	ISO22818:21
0.3 x R (reference test method)	123

Table 2: evaluation of the repeatability of the subsamples #21605

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 blocks was selected which was made positive on SCCP and MCCP by a third-party laboratory. After homogenization 80 small plastic bags were filled with approximately 3 grams each and labelled #21606.
The homogeneity of the subsamples was checked by determination of the total SCCP content using an in-house test method on 8 stratified randomly selected subsamples.

	total SCCP in mg/kg
Sample #21606-1	2359
Sample #21606-2	2435
Sample #21606-3	2389
Sample #21606-4	2386
Sample #21606-5	2355
Sample #21606-6	2416
Sample #21606-7	2360
Sample #21606-8	2391

Table 3: homogeneity test results of the subsamples #21606

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.

	total SCCP in mg/kg
r (observed)	80
reference test method	ISO22818:21
0.3 x R (reference test method)	375

Table 4: evaluation of the repeatability of the subsamples #21606

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 #21605 and one sample labelled #21606 was sent on May 5, 2021.

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 requested 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, e.g. ISO reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In 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 interlaboratory study some problems were encountered with the dispatch of the samples. Eleven participants reported test results after the final reporting date and one other participant did not report any test results. Not all participants were able to report all tests requested.
In total 57 participants reported 204 numerical test results. Observed were 8 outlying test results, which is 3.9%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

Not all 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 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. The abbreviations, used in these tables, are explained in appendix 4.

For the determination of total SCCP and total MCCP in leather ISO18219 is considered to be the official test method, it is unknown if it is applicable for other matrices like polymers.
Since begin 2021 the test method ISO22818 is available for the determination of total SCCP and total 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).

For the evaluation of the test results in this PT the relative standard deviation (RSD) of total SCCP and the RSD of total MCCP in polyester textile coated with PVC mentioned in this test method was used for the evaluation.

Sample #21605

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

MCCP: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ISO22818:21.

Sample #21606

SCCP: This determination was not problematic. Three 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 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 literature reference test methods (in casu ISO test methods) are presented in next tables.

Component	unit	n	average	2.8 * sd	R(lit)
SCCP	mg/kg	55	639	353	334
MCCP	mg/kg	45	1073	591	484

Table 5: performance overview on sample #21605

Component	unit	n	average	2.8 * sd	R(lit)
SCCP	mg/kg	53	2067	842	1082
MCCP	mg/kg	43	7445	4145	3356

Table 6: performance overview on sample #21606

Without further statistical calculations, it can be concluded that for SCCP there is a good compliance of the group of participating laboratories with the reference test method. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF MAY 2021 WITH PREVIOUS PTS

	May 2021	June 2020	June 2019	May 2018	May 2017
Number of reporting laboratories	57	43	45	66	55
Number of test results	204	152	154	216	198
Number of statistical outliers	8	10	9	8	10
Percentage of statistical outliers	3.9%	6.6%	5.5%	3.6%	4.8%

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, expressed as relative standard deviation (RSD) of the PTs, see next table.

Component	May 2021	June 2020	June 2019	May 2018	2015 - 2017	target
SCCP	15-20%	24-52%	18-27%	13-28%	15-33%	19%
MCCP	20%	19-41%	13-33%	18%	19-39%	16%

Table 8: development of the uncertainties over the years

The uncertainties observed for SCCP in this PT is comparable with the target uncertainty. The uncertainty observed for MCCP in this PT is slightly higher than the target uncertainty.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

The test method ISO18219 is used by the majority of the reporting participants.
For this PT some analytical details were requested which are given in appendix 2. Based on the answers given by the participants the following can be summarized:

- About 85% of the reporting participants mentioned that they are accredited to determine the reported component(s).
- About 25% of the reporting participants used the sample as received and about 70% did further cut or further grind the samples prior to analysis.
- Almost all of the reporting participants used a sample intake between 0.5 - 1 grams.
- About 75% of the participants reported to have used Toluene as extraction solvent.
- Almost all participants used an extraction time of 60 minutes and an extraction temperature of 60°C.

Since the majority of the laboratories used the same method (ISO18219) to extract and determine SCCP and/or MCCP, no major differences are found in the measuring procedures. Therefore, the differences cannot be used to prove an effect on the determination on total SCCP and/or total MCCP in Polymers and therefore are negligible.

5 DISCUSSION

All reporting participants were able to detect SCCP and/or MCCP in sample #21605 and sample #21606.

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 SCCPs 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 sample #21605 all reporting laboratories (except one) would have accepted this sample for SCCP.
For sample #21606 almost all reporting laboratories would have rejected this sample, three other reporting laboratories would have accepted this sample for SCCP.

6 CONCLUSION

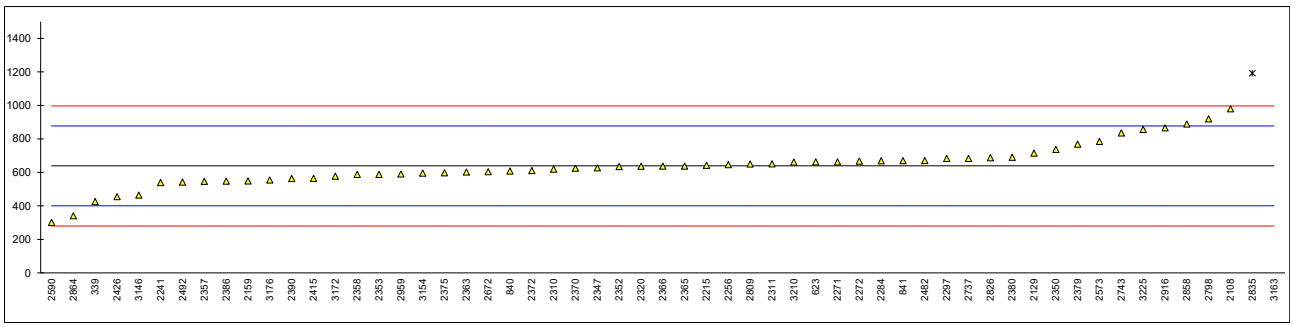
Although it can be concluded that most of the participants have no problem with the determination of total SCCP and/or total MCCP in this PT, each laboratory will have to evaluate its performance in this study and make decisions about necessary corrective actions.
Therefore, participation on a regular 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 #21605; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339	In house	425.7		-1.78	
623	ISO18219	662.90		0.20	
840	ISO18219	608	C	-0.26	first reported 964
841	ISO18219	669.86		0.26	
2108	In house	979.68		2.85	
2129	ISO18219	715		0.64	
2159	ISO18219	549.01		-0.75	
2215	In house	642.39		0.03	
2241	ISO18219	539.89		-0.83	
2256	ISO18219	645.93		0.06	
2271	ISO18219	663.1		0.20	
2272		667.5		0.24	
2284	In house	669		0.25	
2297	in house	683		0.37	
2310	ISO18219	620		-0.16	
2311	ISO18219	651.7		0.11	
2320	ISO18219	636.42		-0.02	
2347	ISO18219	627		-0.10	
2350	ISO18219	737.5		0.83	
2352	In house	634.8		-0.03	
2353	ISO18219	588		-0.43	
2357	ISO18219	547.0		-0.77	
2358	ISO18219	588		-0.43	
2363	In house	602		-0.31	
2365	ISO18219	637.57		-0.01	
2366	ISO18219	637.1		-0.01	
2370	ISO18219	625		-0.12	
2372	ISO18219	611.4		-0.23	
2375	ISO18219	598.0		-0.34	
2379	ISO18219	768.65	C	1.09	first reported 1092.8464
2380	ISO18219	689.98		0.43	
2386	ISO18219	548		-0.76	
2390	ISO18219	563.8		-0.63	
2415	In house	564.5		-0.62	
2426	ISO18219	455.09		-1.54	
2482	ISO18219Mod.	671		0.27	
2492	In house	542.0		-0.81	
2573	ISO18219	785.1		1.23	
2590	ISO18219	301.832	C	-2.82	first reported 284.171
2672	ISO18219	604.0		-0.29	
2737	ISO22818	684.470		0.38	
2743	ISO18219	835.404703		1.65	
2798		919.51		2.35	
2809	ISO18219	651.0		0.10	
2816		-----		-----	
2826	ISO18219-1	687.9257		0.41	
2835	EPA8270E	1191.965	R(0.01)	4.63	
2858	In house	888.50	C	2.09	first reported 1344.9
2864	In house	341.09		-2.49	
2916	In house	866		1.90	
2959		591		-0.40	
3146	In house	464.55		-1.46	
3154	ISO18219	595.999		-0.36	
3163	In house	35000	R(0.01)	287.66	
3172	ISO18219	577.12		-0.52	
3176	ISO18219	554.40		-0.71	
3210	ISO22818	662.36		0.20	
3225	ISO18219	856.6		1.82	

	normality	suspect	
	n	55	
	outliers	2	
	mean (n)	638.770	
	st.dev. (n)	126.0045	RSD = 20%
	R(calc.)	352.813	
	st.dev.(ISO22818:21)	119.4499	
	R(ISO22818:21)	334.460	
	compare		
	R(Horwitz n=9)	324.711	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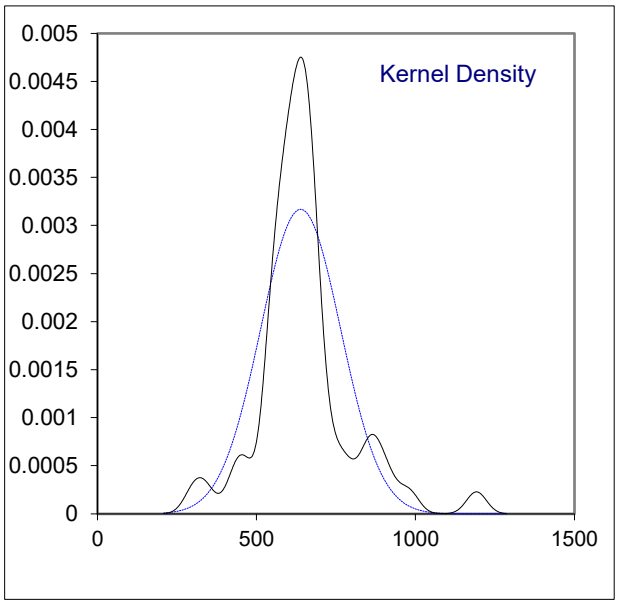
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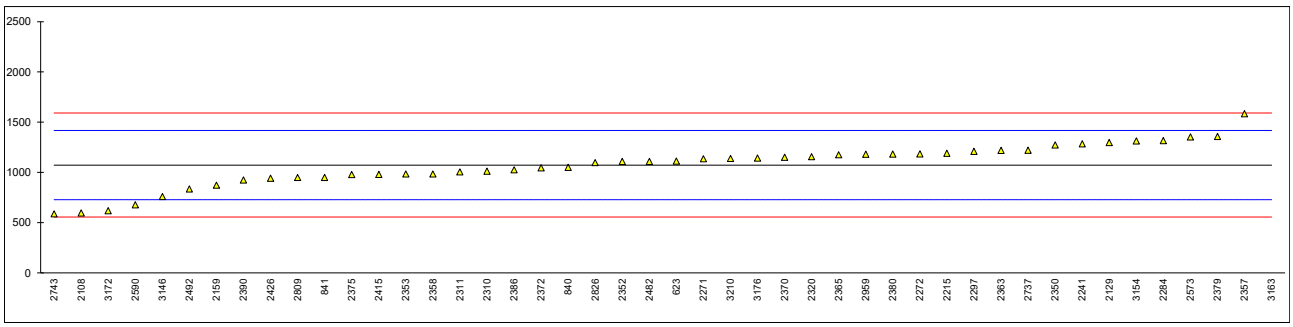
0.0005
0
0
500
1000
1500

Determination of MCCP on sample #21605; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339		-----		-----	
623	ISO18219	1112.0		0.23	
840	ISO18219	1051	C	-0.13	first reported 1960
841	ISO18219	950.56		-0.71	
2108	In house	596.9		-2.76	
2129	ISO18219	1298		1.30	
2159	ISO18219	873.52		-1.15	
2215	In house	1190.22		0.68	
2241	ISO18219	1284.62	C	1.23	first reported 1691.77
2256		-----		-----	
2271	ISO18219	1135.3		0.36	
2272		1184.2		0.64	
2284	In house	1316		1.41	
2297	In house	1210		0.79	
2310	ISO18219	1012		-0.35	
2311	ISO18219	1005.3		-0.39	
2320	ISO18219	1156.24		0.48	
2347		-----		-----	
2350	ISO18219	1273.32		1.16	
2352	In house	1109.6		0.21	
2353	ISO18219	984		-0.51	
2357	ISO18219	1583.9		2.96	
2358	ISO18219	984		-0.51	
2363	In house	1219		0.85	
2365	ISO18219	1176.08		0.60	
2366	ISO18219	not analyzed		-----	
2370	ISO18219	1150		0.45	
2372	ISO18219	1046		-0.16	
2375	ISO18219	978.0		-0.55	
2379	ISO18219	1358.1955		1.65	
2380	ISO18219	1182.43		0.63	
2386	ISO18219	1025		-0.28	
2390	ISO18219	924.8		-0.86	
2415	In house	979.5		-0.54	
2426	ISO18219	942.03		-0.76	
2482	ISO19219Mod.	1110		0.21	
2492	In house	835.6		-1.37	
2573	ISO18219	1353.8		1.63	
2590		678.038		-2.29	
2672		-----		-----	
2737	ISO22818	1221.545		0.86	
2743	ISO18219	587.216055		-2.81	
2798		-----		-----	
2809	ISO18219	948.8		-0.72	
2816		-----		-----	
2826	ISO18219-1	1097.113		0.14	
2835		Not Determine		-----	
2858		-----		-----	
2864		-----		-----	
2916		-----		-----	
2959		1181		0.63	
3146	In house	759.23		-1.82	
3154	ISO18219	1313.607		1.39	
3163		53000	R(0.01)	300.62	
3172	ISO18219	618.83		-2.63	
3176	ISO18219	1143.20		0.41	
3210	ISO22818	1139.34		0.38	
3225		-----		-----	

	normality	OK
	n	45
	outliers	1
	mean (n)	1072.867
	st.dev. (n)	211.1424 RSD = 20%
	R(calc.)	591.199
	st.dev.(ISO22818:21)	172.7317
	R(ISO22818:21)	483.649
	compare	
	R(Horwitz n=9)	504.432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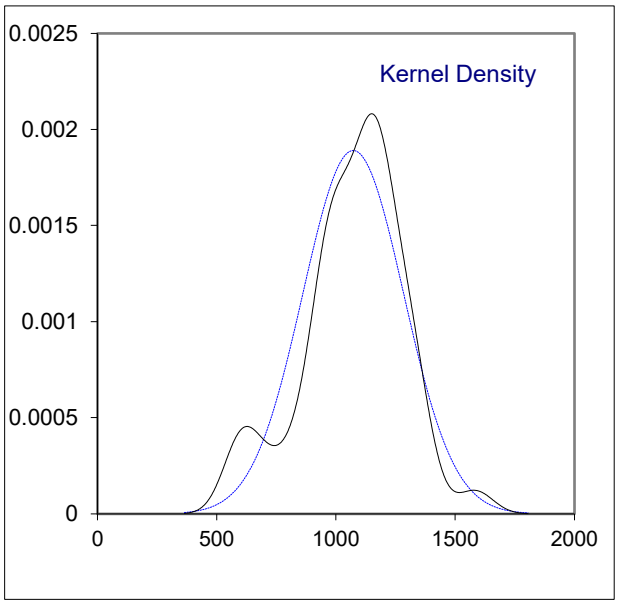
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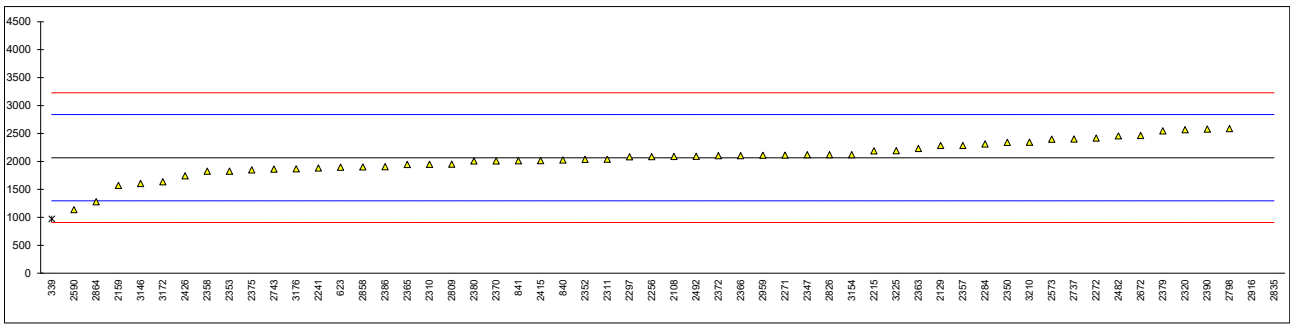
1000
1500
2000

Determination of SCCP on sample #21606; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339	In house	972	R(0.05)	-2.83	
623	ISO18219	1897.60		-0.44	
840	ISO18219	2026	C	-0.11	first reported 3420
841	ISO18219	2013.70		-0.14	
2108	In house	2089.9		0.06	
2129	ISO18219	2284		0.56	
2159	ISO18219	1570.98		-1.28	
2215	In house	2190.43		0.32	
2241	ISO18219	1882.89		-0.48	
2256	ISO18219	2085.68		0.05	
2271	ISO18219	2113.0		0.12	
2272		2416.4		0.91	
2284	In house	2312		0.64	
2297	In house	2085		0.05	
2310	ISO18219	1949		-0.30	
2311	ISO18219	2038.2		-0.07	
2320	ISO18219	2568.21		1.30	
2347	ISO18219	2120		0.14	
2350	ISO18219	2340.9		0.71	
2352	In house	2037.2		-0.08	
2353	ISO18219	1824		-0.63	
2357	ISO18219	2284.9		0.56	
2358	ISO18219	1824		-0.63	
2363	In house	2232		0.43	
2365	ISO18219	1947.49		-0.31	
2366	ISO18219	2102.3		0.09	
2370	ISO18219	2010		-0.15	
2372	ISO18219	2102		0.09	
2375	ISO18219	1850.0		-0.56	
2379	ISO18219	2546.68	C	1.24	first reported 3526.2756
2380	ISO18219	2007.10		-0.15	
2386	ISO18219	1907		-0.41	
2390	ISO18219	2577.9		1.32	
2415	In house	2016.5		-0.13	
2426	ISO18219	1744.29		-0.83	
2482	ISO18219Mod.	2458		1.01	
2492	In house	2092.4		0.07	
2573	ISO18219	2399.3		0.86	
2590	ISO18219	1137.072	C	-2.41	first reported 1037.774
2672	ISO18219	2464		1.03	
2737	ISO22818	2401.945		0.87	
2743	ISO18219	1861.036513		-0.53	
2798		2587.74		1.35	
2809	ISO18219	1952.0		-0.30	
2816		-----		-----	
2826	ISO18219-1	2120.029		0.14	
2835	EPA8270E	6365.03	R(0.01)	11.12	
2858	In house	1901.10		-0.43	
2864	In house	1281.14		-2.03	
2916	In house	5166	R(0.01)	8.02	
2959		2112		0.12	
3146	In house	1604.24		-1.20	
3154	ISO18219	2120.439		0.14	
3163		-----		-----	
3172	ISO18219	1636.63		-1.11	
3176	ISO18219	1867.90		-0.51	
3210	ISO22818	2342.44		0.71	
3225	ISO18219	2193	C	0.33	first reported 3479.1
	normality	suspect			
	n	53			
	outliers	3			
	mean (n)	2066.597			
	st.dev. (n)	300.6827	RSD = 15%		
	R(calc.)	841.912			
	st.dev.(ISO22818:21)	386.4537			
	R(ISO22818:21)	1082.070			
	compare				
	R(Horwitz n=9)	880.358			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3500
3000
2500
2000
1500
1000
500
0
339
2590
2864
2159
3146
3172
2426
2358
2353
2375
2743
3176
2241
623
2858
2386
2365
2310
2809
2390
2370
841
2415
840
2352
2311
2297
2256
2108
2492
2372
2366
2959
2271
2347
2826
3154
2215
3225
2363
2129
2357
2284
2350
3210
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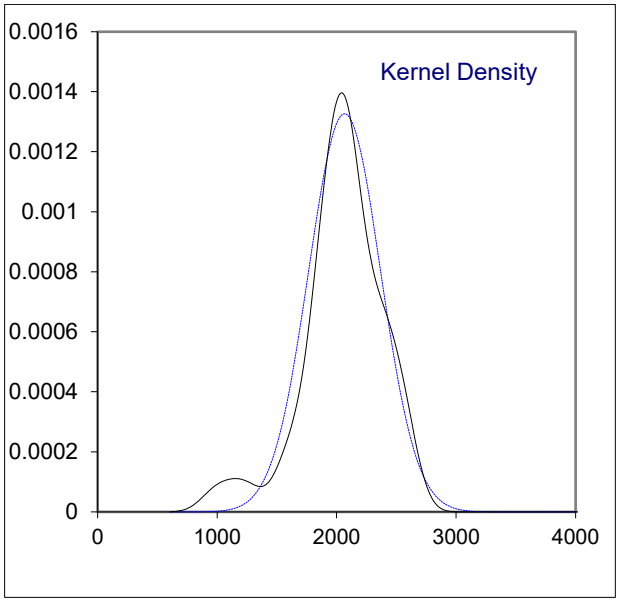
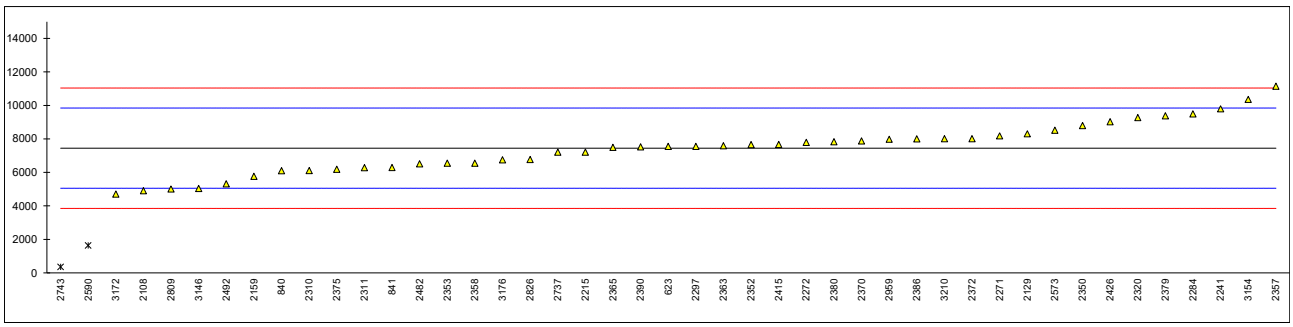
2272
2482
2672
2379
2320
2390
2798
2916
2835


Kernel Density
0.0016
0.0014
0.0012
0.001
0.0008
0.0006
0.0004
0.0002
0
0
1000
2000
3000
4000

Determination of MCCP on sample #21606; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339		-----		-----	
623	ISO18219	7551.60		0.09	
840	ISO18219	6103	C	-1.12	first reported 12200
841	ISO18219	6290.64		-0.96	
2108	In house	4907.92		-2.12	
2129	ISO18219	8303		0.72	
2159	ISO18219	5766.75		-1.40	
2215	In house	7210.49		-0.20	
2241	ISO18219	9794.00		1.96	
2256		-----		-----	
2271	ISO18219	8180.9		0.61	
2272		7792.7		0.29	
2284	In house	9493		1.71	
2297	In house	7555		0.09	
2310	ISO18219	6111		-1.11	
2311	ISO18219	6289.0		-0.96	
2320	ISO18219	9279.34		1.53	
2347		-----		-----	
2350	ISO18219	8798.34		1.13	
2352	In house	7659.2		0.18	
2353	ISO18219	6550		-0.75	
2357	ISO18219	11149.0		3.09	
2358	ISO18219	6550		-0.75	
2363	In house	7591		0.12	
2365	ISO18219	7494.50		0.04	
2366	ISO18219	not analyzed		-----	
2370	ISO18219	7880		0.36	
2372	ISO18219	8018		0.48	
2375	ISO18219	6190.0		-1.05	
2379	ISO18219	9382.4290		1.62	
2380	ISO18219	7831.43		0.32	
2386	ISO18219	8002		0.46	
2390	ISO18219	7530.2		0.07	
2415	In house	7666.4		0.18	
2426	ISO18219	9029.57		1.32	
2482	ISO18219Mod.	6517		-0.77	
2492	In house	5317.4		-1.78	
2573	ISO18219	8520.9		0.90	
2590	ISO18219	1639.603	C,R(0.05)	-4.84	first reported 1528.732
2672		-----		-----	
2737	ISO22818	7210.155		-0.20	
2743	ISO18219	359.742333	C,R(0.05)	-5.91	first reported 1550.850258
2798		-----		-----	
2809	ISO18219	5007.2		-2.03	
2816		-----		-----	
2826	ISO18219-1	6768.293		-0.56	
2835		-----		-----	
2858		-----		-----	
2864		-----		-----	
2916		-----		-----	
2959		7981		0.45	
3146	In house	5042.92		-2.00	
3154	ISO18219	10363.942		2.43	
3163		-----		-----	
3172	ISO18219	4707	C	-2.28	first reported 3540.35
3176	ISO18219	6752.40		-0.58	
3210	ISO22818	8017.25		0.48	
3225		-----		-----	

	normality	OK
	n	43
	outliers	2
	mean (n)	7445.486
	st.dev. (n)	1480.3082 RSD = 20%
	R(calc.)	4144.863
	st.dev.(ISO22818:21)	1198.7233
	R(ISO22818:21)	3356.425
	compare	
	R(Horwitz n=9)	2615.257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10000
8000
6000
4000
2000
0
2743
2590
3172
2108
2809
3146
2492
2159
840
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2375
2311
841
2482
2353
2358
3176
2826
2737
2215
2365
2390
623
2297
2363
2352
2415
2272
2380
2370
2959
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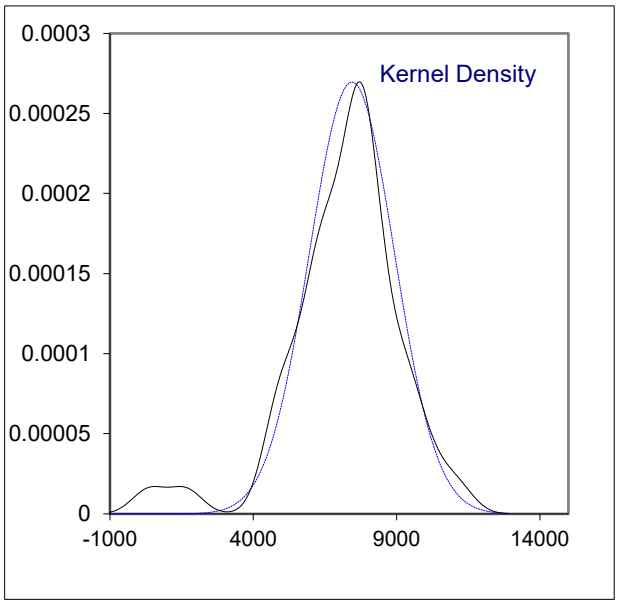
2320
2379
2284
2241
3154
2357


Kernel Density
0.0003
0.00025
0.0002
0.00015
0.0001
0.00005
0
-1000
4000
9000
14000

APPENDIX 2 Analytical details

lab	ISO/IEC17025 accredited	sample preparation before use	sample intake (g)	extraction solvent	extraction time (minutes)	extraction temp. (°C)
339	No	Used as received	0.5	Toluene	60	60
623	Yes	Further cut	0.5	toluene	60	60
840	Yes	Further cut	0.5	toluene	60	60
841	Yes	Further cut	0.5	Toluene	60	60
2108	Yes	Used as received	1	Dichloromethane/Hexane (1:1;V.v)	60	60
2129	Yes	Used as received	1	Toluol	60	60
2159	Yes	Further cut	1	Toluene	60	60
2215	Yes	Further cut	1	toluene	60	60
2241	Yes	Further cut	0.3	Toluene.	60	60
2256	No	Further cut	1.0	Toluene	60	60
2271	Yes	Further cut	0.5	Toluene	60	60
2272	No	Further cut	0.5	toluene	60	60
2284	Yes	Further cut	0.5	Toluene	60	60
2297	Yes	Used as received	1	toluene	1hr	60
2310	Yes	Further cut	0.5	Toluene	60	60
2311	Yes	Further cut	0.5	Toluene	60	60
2320	No	Further cut	0.5	Toluene	60	60
2347	Yes	Further cut	0.1	Toluene	60	60±2
2350	Yes	Further cut	0.5	Hexane	60	60
2352	Yes	Further cut	0.5	Toluene	60	60
2353	Yes	Further cut	0.5	Toluene	60	60
2357	---	---				
2358	---	---				
2363	Yes	Further cut	0.5	toluene	60	60
2365	Yes	Further cut	0.5	Toluene	60	60
2366	Yes	Further cut	0.5	toluene	60	60
2370	Yes	Further cut	1	Toluene	60	60
2372	Yes	Used as received	0.5	Toluene	60	60
2375	Yes	Further cut	0.5	Toluene	60	60
2379	No	Further cut	0.5	Toluene	60	60
2380	Yes	Used as received	0.5	Toluene	60	60
2386	Yes	Used as received	0,5	toluene	60	60
2390	Yes	Further cut	0.5	Toluene	60	60
2415	Yes	Further cut	0.25	Toluene	60	60
2426	Yes	Further cut	#21605 = 0.5013g #21606 = 0.2550g	Toluene + n-Hexane	60min Toluene & 15min after addition of n-Hexane	60
2482	Yes	Used as received	0.5	Toluene	60	60
2492	Yes	Used as received	0.5	DCM:Hexane (1:1)	60	60
2573	Yes	Used as received	0.5	Toluene	60	60
2590	Yes	Further cut	0.5	hexane	60	60
2672	Yes	Further cut	0,5	toluene	60	60
2737	Yes	Used as received	#21605 0.5gram #21606 1.0 gram	Toluene	60	60
2743	Yes	Used as received	0,5	Hexane	60	60
2798	Yes	Used as received	0.5072	Toluene	60	60
2809	No	Further cut	0.5	n-hexane	60	60
2816	---	---				
2826	Yes	Used as received	0.5	Toluene	60	60
2835	Yes	Further cut	0.5	Hexane	60	60
2858	Yes	Used as received	1.0	n-Hexane+Dichloromethane (1:1 mix)	60	60
2864	Yes	Further cut	0.1	hexean	2hr	69
2916	No	Further grinded	0,5	n-Hexane	360	69
2959	No	Further cut	#21605: 0.5030g #21606: 0.5028g	Toluene	60	60
3146	Yes	Used as received	0,5	THF	30	70
3154	Yes	Used as received	0,5	Toluene	60	60
3163	No	Further cut	0.0005	-	-	-
3172	Yes	---				
3176	Yes	Further cut	0,5	Hexane	60	60
3210	Yes	Further cut	1	Toluène	60	60
3225	Yes	Further cut	0.5	Toluene	60	60

APPENDIX 3

Number of participants per country

2 labs in	BANGLADESH
1 lab in	DENMARK
2 labs in	FRANCE
8 labs in	GERMANY
5 labs in	HONG KONG
2 labs in	INDIA
1 lab in	INDONESIA
3 labs in	ITALY
17 labs in	P.R. of CHINA
2 labs in	PAKISTAN
1 lab in	SINGAPORE
1 lab in	SOUTH KOREA
1 lab in	SRI LANKA
3 labs in	TAIWAN
1 lab in	THAILAND
1 lab in	THE NETHERLANDS
3 labs in	TURKEY
4 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
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

Literature

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7 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
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12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)