Results of Proficiency Test
SCCP in Polymer
June 2020

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). Since 2017 SCCP is banned under the Stockholm Convention on Persistent Organic Pollutants (annex A).

Since 2015 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of SCCP/MCCP content in Polymers. During the annual proficiency testing program 2019/2020 it was decided to continue the proficiency test for the analysis of SCCP/MCCP in Polymers.
In this interlaboratory study 46 laboratories in 19 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 samples both positive on SCCP and MCCP. The first sample was a Thermo Plastic Elastomer (TPU) red granulate sample of 3 grams, labelled #20615. The second sample was a Polyvinylchloride (PVC) green rings sample of 3 grams, labelled #20616.
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 colored Thermo Plastic Elastomer (TPU) was selected which was made positive on SCCP and MCCP by a third-party laboratory. After homogenization 75 small plastic bags were filled with approximately 3 grams each and labelled #20615. 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 #20615-1	1688
Sample #20615-2	1633
Sample #20615-3	1618
Sample #20615-4	1626
Sample #20615-5	1664
Sample #20615-6	1641
Sample #20615-7	1646
Sample #20615-8	1710

Table 1: homogeneity test results of the subsamples #20615

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility estimated from the Horwitz equation (n=9) in agreement with the procedure of ISO13528, Annex B2.

	total SCCP in mg/kg
r (observed)	89
reference method	Horwitz (n=9)
0.3 x R (reference method)	218

Table 2: evaluation of the repeatability of the subsamples #20615

The calculated repeatability was in agreement with 0.3 times the reproducibility estimated from the Horwitz equation (n=9). 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 75 small plastic bags were filled with approximately 3 grams each and labelled #20616. The homogeneity of the subsamples was checked by determination of the total SCCP content using an in-house test method on 10 stratified randomly selected subsamples.

	total SCCP in mg/kg
Sample #20616-1	312
Sample #20616-2	317
Sample #20616-3	308
Sample #20616-4	324
Sample #20616-5	310
Sample #20616-6	321
Sample #20616-7	305
Sample #20616-8	317
Sample #20616-9	312
Sample #20616-10	305

Table 3: homogeneity test results of the subsamples #20616

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility estimated from the Horwitz equation (n=9) in agreement with the procedure of ISO13528, Annex B2.

	total SCCP in mg/kg
r (observed)	18
reference method	Horwitz (n=9)
0.3 x R (reference method)	53

Table 4: evaluation of the repeatability of the subsamples #20616

The calculated repeatability was in agreement with 0.3 times the reproducibility estimated from the Horwitz equation (n=9). Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one set of samples (1 x #20615 and 1 x #20616) was sent on May 6, 2020.

2.5 ANALYSES

The participants were requested to determine on both samples the total SCCP and total MCCP content on both samples. It was also requested to report if the laboratory was accredited for the requested determined components and to report some analytical details.

It was explicitly requested to treat the samples as if they were routine samples. It was also requested to report the test results using the indicated units on the report form and not to round the results, but report as much significant figures as possible and 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 appropriate reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The participating laboratories 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 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 reanalysis). 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 Organization, 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 in general 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.

According to ISO5725 the original test results per determination were submitted to Dixon's and/or Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of the averages and the standard deviations.

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

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

3.2 GRAPHICS

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

Furthermore, Kernel Density Graphs were made. The Kernel Density Graph is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

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

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

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test results 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 result tables in appendix 1.

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

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

4 EVALUATION

In this interlaboratory study some problems were encountered with the dispatch of the samples due to the COVID-19 pandemic. Three participants did not report any test results. In total 43 participants reported 152 numerical test results. Observed were 10 outlying test results, which is 6.6%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

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

4.1 EVALUATION PER 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 ISO18219 is considered to be the official test method. However, this method is developed for the determination of SCCP/MCCP in leather and therefore it is unknown if it is applicable for other matrices like polymers. Regretfully, for the determination of total SCCP/MCCP content in polymers no official test method is available. Therefore, the target requirements in this study were estimated using the Horwitz equation based on nine components (n=9).

Sample #20615

SCCP: This determination was very problematic. No statistical outliers were observed, but seven test results were excluded. The group seems divided bimodally, therefore no z-scores were calculated. See §5 for more discussion.

MCCP: This determination was very problematic. Two statistical outliers were observed and two other test results were excluded. The group seems divided bimodally, therefore no z-scores were calculated. See §5 for more discussion.

Sample #20616

SCCP: This determination may be problematic. Six statistical outliers were observed and four other test results were excluded. However, the observed reproducibility after rejection of the suspect data is not in agreement with the estimated reproducibility calculated using the Horwitz equation (n=9). See §5 for more discussion.

MCCP: This determination was not problematic. Two statistical outliers were observed and one other test result was excluded. However, the observed reproducibility after rejection of the suspect data is in full agreement with the estimated reproducibility calculated using the Horwitz equation (n=9). See §5 for more discussion.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the estimated target reproducibility using the Horwitz equation (n=9) 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 are presented in next tables.

Component	unit	n	average	2.8 * sd	R(lit)
SCCP	mg/kg	36	774	1129	(382)
MCCP	mg/kg	29	1664	1915	(732)

Table 5: performance overview on sample #20615

Component	unit	n	average	2.8 * sd	R(lit)
SCCP	mg/kg	33	247	166	145
MCCP	mg/kg	30	676	360	341

Table 6: performance overview on sample #20616

Without further statistical calculations, it can be concluded that there is not a good compliance of the group of participating laboratories with the reference test method. See also the discussion in paragraphs 4.1 and 5.

4.3 COMPARISON OF THE PROFICIENCY TEST OF JUNE 2020 WITH PREVIOUS PTS

	June 2020	June 2019	May 2018	May 2017	May 2016
Number of reporting laboratories	43	45	66	55	51
Number of test results	152	154	216	198	184
Number of statistical outliers	10	9	8	10	4
Percentage of statistical outliers	6.6%	5.5%	3.6%	4.8%	2.1%

Table 7: comparison with previous proficiency tests

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

The uncertainties determined in this PT are compared with the relative standard deviations as found in previous years and with the target requirements based on the Horwitz equation in the next table.

Component	June 2020	June 2019	May 2018	May 2017	2015 - 2016	Target
SCCP	24-52%	18-27%	13-28%	15-23%	23-33%	16-18%
MCCP	19-41%	13-33%	18%	19-20%	19-39%	14-15%

Table 8: the observed uncertainties over the years

Only for the PVC sample (#20616) the reproducibility of the group is similar in comparison with previous years.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

For this proficiency test some analytical details were requested, see appendix 2 for the reported answers. Based on the answers the following can be summarized: Two participants of the forty-three did not report any analytical details.
Around 60% of the participants (dependent on the component) reported to have used ISO18219 as test method and between 30-40% of the participants reported to have used an ‘in-house’ test method.
- About 80% of the participants reported to be ISO/IEC17025 accredited for the determination of total SCCP/MCCP in polymers.
- About 60% of the participants reported to have further cut or further grind the samples prior to analysis. The final estimated sample size reported was most often below 2x3mm.
- About 75% of the participants used a sample intake of 0.5 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.

It was observed that most participants were able to detect SCCP and MCCP in this proficiency test for the determination of total SCCP and total MCCP in polymers.
The effect of some of the reported analytical details (see paragraph 4.4) on SCCP were further investigated on those analytical details where it was possible to distinguish two or more meaningful subgroups to compare, see table 9.

sample	analytical details	unit	n	average	RSD (%)
#20615	ISO/IEC17025 accredited	mg/kg	27	811	46
#20615	Not ISO/IEC17025 accredited	mg/kg	7	614	87
#20616	ISO/IEC17025 accredited	mg/kg	27	248	23
#20616	Not ISO/IEC17025 accredited	mg/kg	5	241	34
#20615	Further cut or grinded	mg/kg	21	849	49
#20615	Used as received	mg/kg	7	570	82
#20616	Further cut or grinded	mg/kg	20	251	24
#20616	Used as received	mg/kg	4	252	37

Table 9: effect of analytical details on SCCP

It is observed that accredited laboratories yield higher levels of SCCP with less variation between the laboratories. Further cutting or further grinding the samples before use tends to give higher levels of components and a smaller reproducibility in sample #20615 (TPU polymer). The reproducibility in sample 20616 (PVC) was already lower and the effect of further cutting or grinding is less profound. Please note that the observed effects are not statistically significant.

5 DISCUSSION

In previous PTs it appeared that the SCCP and MCCP levels did increase and the variations did decrease when the samples were cut or grinded before use or when Toluene or THF/ACN was used as extraction solvent. However, in this PT almost all participants have used Toluene as extraction solvent. And the investigated effect of sample treatment showed no obvious improvement in the evaluation, see sample #20616 in appendix 1. Therefore, it was decided not to exclude test results for the SCCP/MCCP determination based on these reported analytical details like it was done in previous iis PTs.

In this proficiency test for the determination of Total SCCP and Total MCCP in polymers two different polymers were used. Sample #20615 is made of Thermo Plastic Elastomer (TPU) and sample #20616 of Polyvinylchloride (PVC). The observed reproducibility of sample #20615 was much higher than the reproducibility of sample #20616. The difference may be explained by the difference in matrices of the samples. It occurs that releasing SCCP and MCCP from TPU samples is far more difficult than from PVC.

Furthermore, the test results reported for sample #20615 seem to give a bimodal distribution (see Kernel plot on page 13). A clear cause cannot be identified (e.g. effect of sample treatment). Therefore, it was decided not to use a specific group for assigned values and to calculate no z-scores.

6 CONCLUSION

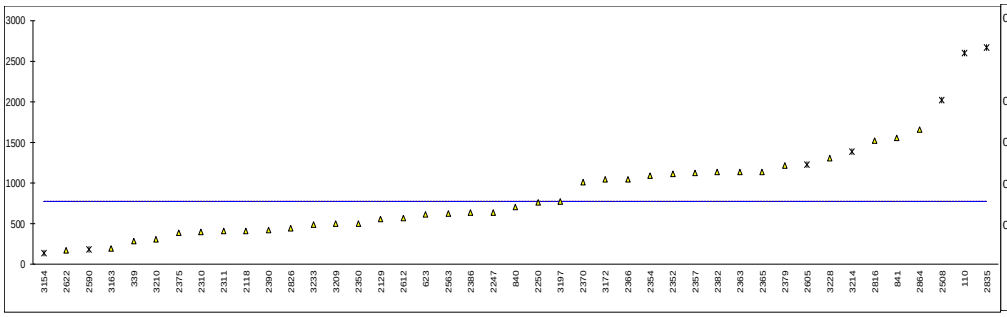
It is clear that the majority of the participants were able to determine total SCCP and total MCCP in the polymer matrix. However, it is noted that there is a large variation in the results dependent on the type of component and matrix of polymer. For the analysis of Total SCCP from polymers a sound test method which prescribe the analysis of Total SCCP from different polymers in detail is desirable, especially for other polymers than PVC. Also, the choice of solvent may play a role in the determination other polymers than in PVC.

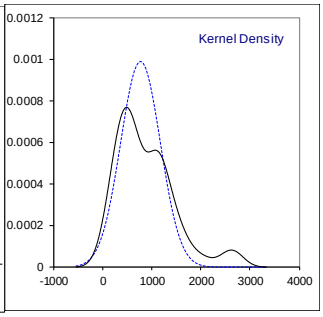
Each laboratory has to evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and the quality of the analytical results.

APPENDIX 1

Determination of SCCP on sample #20615; results in mg/kg

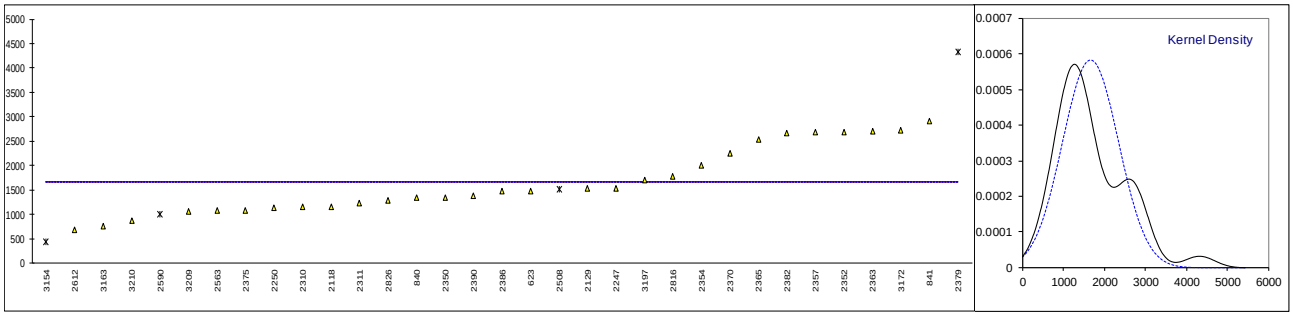
lab	method	value	mark	z(targ)	remarks
110	In-house	2597.7495	ex	-----	Test result excluded, used a deviating solvent
339	In-house	289		-----	
623	ISO18219	611.200		-----	
840	ISO18219	700		-----	
841	ISO18219	1551		-----	
2118	ISO18219	412.884		-----	
2129	ISO18219	559		-----	
2247	ISO18219	638.66		-----	
2250	ISO18219	760		-----	
2267		-----		-----	
2310	ISO18219	394.6		-----	
2311	ISO18219	408		-----	
2350	ISO18219	505.294		-----	
2352	In-house	1115		-----	
2354	ISO18219	1085.1		-----	
2357	In-house	1127		-----	
2363	ISO18219	1135		-----	
2365	ISO18219	1139.582		-----	
2366	ISO18219	1050.0		-----	
2370	ISO18219	1010		-----	
2375	ISO18219	390		-----	
2379	ISO18219	1211.5789		-----	
2380		-----		-----	
2382	ISO18219	1130.3		-----	
2386	ISO18219	631.9		-----	
2390	ISO18219	425.6		-----	
2508	ISO18219	2017.67	ex	-----	Test result excluded, used a deviating solvent
2563	ISO18219	630.3		-----	
2590	ISO18219	178.900	ex	-----	Test result excluded as more test results were statistical outliers
2605	In-house	1221.949	ex	-----	Test result excluded, used a deviating solvent
2612		565		-----	
2622	ISO18219	174		-----	
2816	In-house	1520.50008		-----	
2826	ISO18219	442.3		-----	
2835		2662.338	ex	-----	Test result excluded, used a deviating solvent
2864	In-house	1660.58		-----	
2886		-----		-----	
3154	ISO18219	141.74	ex	-----	Test result excluded as more test results were statistical outliers
3163	In-house	200		-----	
3172	ISO18219	1048		-----	
3197	In-house	768.0		-----	
3209	In-house	496.21		-----	
3210	In-house	306.943		-----	
3214	ISO18219	1390.5	ex	-----	Test result excluded, used a deviating solvent
3228	In-house	1300.9		-----	
3233	In-house	485.78		-----	
					Only Toluene
	normality	OK			OK
	n	36			29
	outliers	0 (+7excl)			0 (+2excl)
	mean (n)	774.423			730.101
	st.dev. (n)	403.3920	RSD = 52%		361.8575 RSD = 50%
	R(calc.)	1129.498			1013.201
	st.dev.(Horwitz n=9)	(136.5792)			(129.9099)
	R(Horwitz n=9)	(382.422)			(363.748)





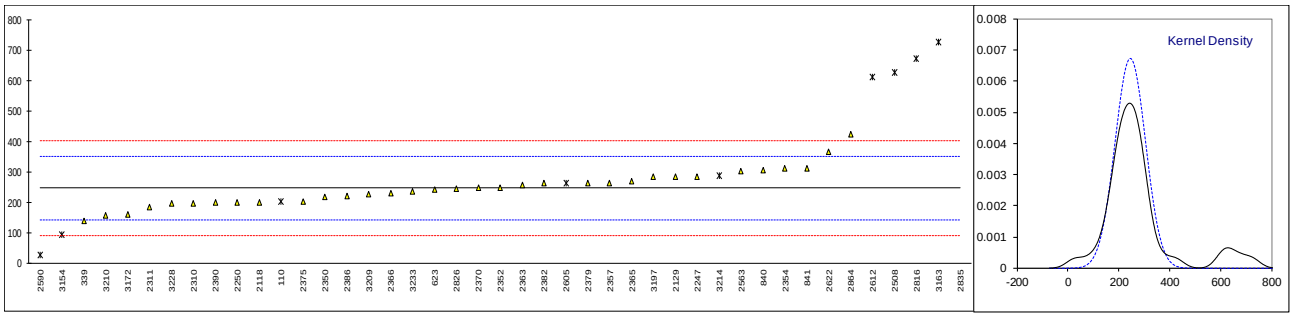
Determination of MCCP on sample #20615; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
339		-----		-----	
623	ISO18219	1484.000		-----	
840	ISO18219	1338		-----	
841	ISO18219	2920		-----	
2118	ISO18219	1150.521		-----	
2129	ISO18219	1531		-----	
2247	ISO18219	1531.08		-----	
2250	ISO18219	1128		-----	
2267		-----		-----	
2310	ISO18219	1150		-----	
2311	ISO18219	1227		-----	
2350	ISO18219	1339.216		-----	
2352	In-house	2691		-----	
2354	ISO18219	2014.2		-----	
2357	In-house	2677		-----	
2363	ISO18219	2705		-----	
2365	ISO18219	2543.381		-----	
2366		-----		-----	
2370	ISO18219	2250		-----	
2375	ISO18219	1085		-----	
2379	ISO18219	4330.8973	C,R(0.05)	-----	First reported 3896.8413
2380		-----		-----	
2382	ISO18219	2670.4		-----	
2386	ISO18219	1476		-----	
2390	ISO18219	1386.5		-----	
2508	ISO18219	1510.21	ex	-----	Test result excluded, used a deviating solvent
2563	ISO18219	1081.2		-----	
2590	ISO18219	995.862	ex	-----	Test result excluded as more test results were statistical outliers
2605		-----		-----	
2612		690		-----	
2622		-----		-----	
2816	In-house	1776.6833		-----	
2826	ISO18219	1292.98		-----	
2835		-----		-----	
2864		-----		-----	
2886		-----		-----	
3154	ISO18219	445.98	R(0.05)	-----	
3163	In-house	750		-----	
3172	ISO18219	2721		-----	
3197	In-house	1707.0		-----	
3209	In-house	1069.12		-----	
3210	In-house	877.103		-----	
3214		-----		-----	
3228		-----		-----	
3233		-----		-----	
					Only Toluene
	normality	OK			OK
	n	29			24
	outliers	2 (+2excl)			2 (+1 excl)
	mean (n)	1664.220			1621.404
	st.dev. (n)	683.8390	RSD = 41%		656.5080 RSD = 40%
	R(calc.)	1914.749			1838.222
	st.dev.(Horwitz n=9)	(261.5842)			(255.8561)
	R(Horwitz n=9)	(732.436)			(716.397)



Determination of SCCP on sample #20616; results in mg/kg

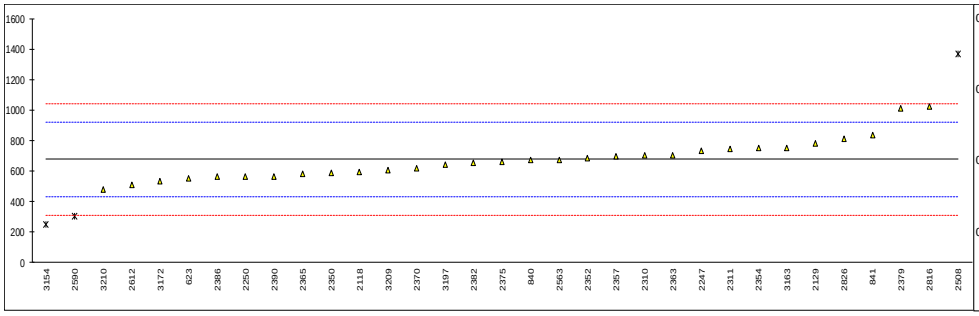
lab	method	value	mark	z(targ)	remarks
110	In-house	202.0525	ex	-0.87	Test result excluded, used a deviating solvent
339	In-house	141		-2.05	
623	ISO18219	242.300		-0.09	
840	ISO18219	306		1.14	
841	ISO18219	313		1.27	
2118	ISO18219	200.163		-0.91	
2129	ISO18219	285		0.73	
2247	ISO18219	285.68		0.74	
2250	ISO18219	200		-0.91	
2267		-----		-----	
2310	ISO18219	196.8		-0.97	
2311	ISO18219	185		-1.20	
2350	ISO18219	218.725		-0.55	
2352	In-house	248		0.02	
2354	ISO18219	310.7		1.23	
2357	In-house	265		0.35	
2363	ISO18219	259		0.23	
2365	ISO18219	270.986		0.46	
2366	ISO18219	230.0		-0.33	
2370	ISO18219	247		0.00	
2375	ISO18219	203		-0.85	
2379	ISO18219	263.9551		0.33	
2380		-----		-----	
2382	ISO18219	262.5		0.30	
2386	ISO18219	220.7		-0.51	
2390	ISO18219	199.5		-0.92	
2508	ISO18219	625.58	ex	7.31	Test result excluded, used a deviating solvent
2563	ISO18219	303.9		1.10	
2590	ISO18219	28.050	R(0.05)	-4.23	
2605	In-house	262.725	ex	0.30	Test result excluded, used a deviating solvent
2612		611	R(0.01)	7.03	
2622	ISO18219	365		2.28	
2816	In-house	670.722935	R(0.01)	8.18	
2826	ISO18219	245.95		-0.02	
2835		1079.597	R(0.01)	16.08	
2864	In-house	424.75		3.43	
2886		-----		-----	
3154	ISO18219	93.07	R(0.05)	-2.98	
3163	In-house	725	R(0.01)	9.23	
3172	ISO18219	162		-1.64	
3197	In-house	283.3		0.70	
3209	In-house	227.31		-0.38	
3210	In-house	157.018		-1.74	
3214	ISO18219	289.2	ex	0.81	Test result excluded, used a deviating solvent
3228	In-house	195.7		-0.99	
3233	In-house	236.36		-0.21	
	normality	suspect			
	n	33			
	outliers	6 (+4excl)			
	mean (n)	247.130			
	st.dev. (n)	59.4068	RSD = 24%		
	R(calc.)	166.339			
	st.dev.(Horwitz n=9)	51.7602			
	R(Horwitz n=9)	144.929			

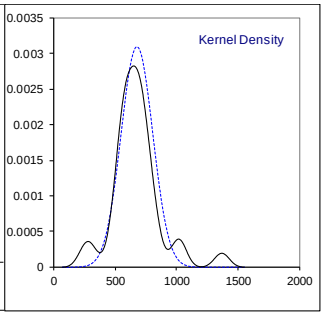


Determination of MCCP on sample #20616; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
339		-----		-----	
623	ISO18219	552.200		-1.02	
840	ISO18219	674		-0.02	
841	ISO18219	835		1.31	
2118	ISO18219	591.887		-0.69	
2129	ISO18219	782		0.87	
2247	ISO18219	734.44		0.48	
2250	ISO18219	563		-0.93	
2267		-----		-----	
2310	ISO18219	701.7		0.21	
2311	ISO18219	745		0.57	
2350	ISO18219	589.020		-0.71	
2352	In-house	683		0.06	
2354	ISO18219	748.5		0.60	
2357	In-house	695		0.16	
2363	ISO18219	702		0.21	
2365	ISO18219	580.831		-0.78	
2366		-----		-----	
2370	ISO18219	620		-0.46	
2375	ISO18219	659		-0.14	
2379	ISO18219	1008.5975		2.73	
2380		-----		-----	
2382	ISO18219	655.2		-0.17	
2386	ISO18219	561.6		-0.94	
2390	ISO18219	565.4	C	-0.91	First reported 1077.3
2508	ISO18219	1365.61	ex	5.67	Test result excluded, used a deviating solvent
2563	ISO18219	674.6		-0.01	
2590	ISO18219	303.813	R(0.05)	-3.06	
2605		-----		-----	
2612		508		-1.38	
2622		-----		-----	
2816	In-house	1023.275884		2.86	
2826	ISO18219	808.53		1.09	
2835		-----		-----	
2864		-----		-----	
2886		-----		-----	
3154	ISO18219	251.85	R(0.05)	-3.49	
3163	In-house	750		0.61	
3172	ISO18219	534		-1.17	
3197	In-house	644.5		-0.26	
3209	In-house	608.15		-0.56	
3210	In-house	478.060		-1.63	
3214		-----		-----	
3228		-----		-----	
3233		-----		-----	

normality	not OK
n	30
outliers	2 (+1 excl)
mean (n)	675.883
st.dev. (n)	128.6666 RSD = 19%
R(calc.)	360.267
st.dev.(Horwitz n=9)	121.6675
R(Horwitz n=9)	340.669





APPENDIX 2
Analytical details

lab	ISO/IEC17025 accredited	sample preparation before use	final particle size (mm)	sample intake (g)	extraction solvent	extraction time (min)	extraction temp. (°C)
110	Yes	Further Cut	2 x 2 mm	1	Dichloromethane - Hexane	60	50
339	No	Used as received	---	0.5	Toluene	60	60
623	Yes	Further Cut	2 x 2 mm	0.5	Hexane	60	60
840	Yes	Further Cut	2 x 2 mm	0.5	Toluene	60	60
841	Yes	Further Cut	2 x 2 mm	0.5	Toluene - Hexane	60	60
2118	No	Further Cut	2 x 3 mm	0.5	Toluene	60	60
2129	Yes	Used as received	---	0.5	Toluene	60	60
2247	Yes	Further Cut	<2 mm	0.3	Toluene - Hexane	60	60
2250	Yes	Used as received	2 mm	0.5	Toluene	60	60
2267	---	---	---	---	---	---	---
2310	Yes	Further Cut	2 x 2 mm	0.5	Toluene	60	60
2311	Yes	Further Cut	<3 mm	0.5	Toluene	60	60
2350	Yes	Further Cut	2 x 2 mm	0.5	Toluene	60	60
2352	Yes	Further Cut	<2 x 2 x 2mm	0.5	Toluene	60	60
2354	Yes	Further Cut	5 x 5mm	0.5	Toluene	60	60
2357	---	---	---	---	---	---	---
2363	Yes	Further Cut	2mm	0.5	Toluene	60	60
2365	Yes	Further Cut	1~2mm	0.5	Toluene	60	60
2366	Yes	Further Cut	2 x 2 x 2mm	0.5	Toluene	60	60
2370	Yes	Further Cut	0.5 x 0.5 cm	1.5	Toluene	60	60
2375	Yes	Further Cut	2 x 2 mm	0.5	Toluene	60	60
2379	No	Further Cut	2 x 2 mm	0.5	Toluene	60	60
2380	---	---	---	---	---	---	---
2382	Yes	Further Cut	2 x 2 mm	0.5	Toluene	60	60
2386	Yes	Used as received	---	---	Toluene	60	60
2390	Yes	Further Cut	<2 mm	0.5	Toluene	60	60
2508	Yes	Used as received	---	0.5	Dichloromethane - Hexane	60	60
2563	Yes	Used as received	---	0.5	Toluene	60	60
2590	Yes	Further Cut	0.2 x 0.2 mm	0.5	Toluene - Hexane	60	60
2605	Yes	Further Cut	2 x 2 mm	0.5	THF/ACN	60	70
2612	---	---	---	---	---	---	---
2622	No	Used as received	---	0.8	Hexane	60	50-60
2816	No	Used as received	2 mm	0.5	Pentane - Acetone	240	Room
2826	Yes	Used as received	4 x 4 mm	0.5	Toluene	60	60
2835	Yes	Further cut	1 mm	0.1	Dichloromethane - Hexane	15	100
2864	Yes	Further Grinded	<1 mm	0.05	Acetone – Hexane	360	150
2886	---	---	---	---	---	---	---
3154	Yes	Used as received	---	0.5	Toluene	60	60
3163	No	Further Cut	2 mm	0.2	Toluene	60	60
3172	Yes	Further Cut	3 x 3 mm	2	Hexane	60	60
3197	Yes	Used as received	5 x 5 mm	0.25	Toluene	60	60
3209	Yes	Used as received	---	0.5	Toluene	60	60
3210	Yes	Used as received	---	0.5	Toluene	60	60
3214	Yes	Further Cut	0.5 x 0.5 mm	0.5	THF/ACN	60	70
3228	Yes	Used as received	0.5 x 0.5 cm	0.5	Toluene - Hexane	60	60
3233	No	Used as received	---	0.5	Toluene	60	60

APPENDIX 3

Number of participants per country

1 lab in BANGLADESH
1 lab in BELGIUM
2 labs in DENMARK
3 labs in FRANCE
7 labs in GERMANY
2 labs in HONG KONG
3 labs in INDIA
1 lab in INDONESIA
3 labs in ITALY
9 labs in P.R. of CHINA
1 lab in PAKISTAN
1 lab in SINGAPORE
1 lab in SOUTH KOREA
3 labs in TAIWAN
1 lab in THAILAND
2 labs in THE NETHERLANDS
2 labs in TURKEY
1 lab in U.S.A.
2 labs in VIETNAM

APPENDIX 4

Abbreviations

C = final test result after checking of first reported suspect test result
D(0.01) = outlier in Dixon's outlier test
D(0.05) = straggler in Dixon's outlier test
G(0.01) = outlier in Grubbs' outlier test
G(0.05) = straggler in Grubbs' outlier test
DG(0.01) = outlier in Double Grubbs' outlier test
DG(0.05) = straggler in Double Grubbs' outlier test
R(0.01) = outlier in Rosner's outlier test
R(0.05) = straggler in Rosner's outlier test
n.a. = not applicable
n.e. = not evaluated
n.d. = not detected
W = test result withdrawn on request of participant
ex = test result excluded from statistical evaluation

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