Results of Proficiency Test
SCCP & MCCP in Polymers
June 2019

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Report: iis19P05

September 2019

CONTENTS

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 2018/2019 it was decided to continue the proficiency test for the analysis of SCCP/MCCP in Polymers.

In this interlaboratory study 50 laboratories from 18 different countries registered for participation. See appendix 3 for the number of participants per country. In this report, the results of the 2019 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. Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send two different plastic samples both positive on SCCP and MCCP. The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation. Participants were also requested to report a number of details of the test method used.

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 organisation 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

Two different PVC batches both artificially fortified with a different level of a commercial mixture of SCCP and MCCP were obtained from a third party. The first PVC batch contained black colored PVC rings/tubes. The second PVC batch contained blue PVC blocks. After homogenisation the batches were separately divided over 100 plastic bags, approximately 3 grams per bag and labelled #19549 and #19550 respectively. The homogeneities of the subsamples #19549 and #19550 were checked by determination of the SCCP content on eight randomly selected subsamples.

	SCCP in mg/kg sample #19549	SCCP in mg/kg sample #19550
Sample 1	950	548
Sample 2	982	537
Sample 3	983	547
Sample 4	953	535
Sample 5	948	529
Sample 6	970	528
Sample 7	938	536
Sample 8	938	540

Table 1: homogeneity test results of the subsamples #19549 and #19550

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

	SCCP in mg/kg sample #19549	SCCP in mg/kg sample #19550
r (observed)	56	21
reference method	Horwitz (n=9)	Horwitz (n=9)
0.3 x R (reference method)	137	84

Table 2: evaluation of repeatabilities of SCCP contents of the subsamples #19549 and #19550

The calculated repeatabilities were in agreement with 0.3 times the target reproducibilities estimated from the Horwitz equation (n=9). Therefore, homogeneity of the subsamples #19549 and #19550 was assumed.

To each of the participating laboratories one sample #19549 and one sample #19550 was sent on May 8, 2019.

2.5 ANALYSES

The participants were requested to determine on both samples the SCCP and MCCP content, applying the analysis procedure that is routinely used in the laboratory. It was also requested to report if the laboratory was accredited to determine the requested components and to report some analytical details of the test method used.

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’ 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 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.kmpd.co.uk/sgs-iis-cts/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by the 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 the data analysis and the original 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 wast 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.

According to ISO5725 the original test results per determination were submitted subsequently to Dixon’s, Grubbs’ and/or Rosner’s outlier tests. Outliers are marked by D(0.01) for Dixon’s test, by G(0.01) or DG(0.01) for Grubbs’s test and by R(0.01) for Rosner’s test. Stragglers are marked by D(0.05) for Dixon’s test, by G(0.05) or DG(0.05) for Grubbs’ test and by R(0.05) for 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 uncertainly 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 target reproducibility by division with 2.8. In case no literature reproducibility was available, other target values are used.
In some cases, a reproducibility based on former iis proficiency tests could be used.

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. 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 no severe problems were encountered with dispatch of the samples. Five participants did not report any test results and three other participants reported test results after the final reporting date. Finally, the 45 reporting laboratories reported 154 numerical results. In the reported test results 9 statistical outliers were observed, which is 5.5%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

All original data sets proved to have a normal Gaussian distribution.

4.1 EVALUATION PER SAMPLE AND PER COMPONENT

For the determination of SCCP/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 plastics. Regretfully, for the determination of SCCP/MCCP content in plastics 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).
Between 56-62% of the participants (dependent on the component) reported to have used ISO18219 as test method and between 27-38% of the participants reported to have used an ‘in house’ test method.

In previous PTs it appeared that the SCCP/MCCP level increased and the variation decreased 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 or THF/ACN as extraction solvent. And the investigated effect of sample pre-treatment showed no obvious improvement in the evaluation, see appendix 1. Therefore, it was decided not to exclude test results for the SCCP/MCCP determination based on these reported analytical details, as is done in previous iis PTs.

Sample #19549

SCCP: This determination may be problematic. One statistical outlier was observed. The observed reproducibility after rejection of the statistical outlier 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. Four statistical outliers were observed. However, the observed reproducibility after rejection of the statistical outliers is in full agreement with the estimated reproducibility calculated using the Horwitz equation (n=9). See §5 for more discussion.

Sample #19550

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

MCCP: This determination may be problematic. Two statistical outliers were observed and one other test result was excluded. 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.

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 test results, the average result, the calculated reproducibility (standard deviation * 2.8) and the estimated target reproducibility are presented in next tables.

Component	unit	n	average	2.8 * sd	R(target)
SCCP	mg/kg	43	1197	886	553
MCCP	mg/kg	31	3268	1187	1299

Table 3: performance overview on samples #19549

Component	unit	n	average	2.8 * sd	R(target)
SCCP	mg/kg	42	488	250	258
MCCP	mg/kg	28	133	124	86

Table 4: performance overview on samples #19550

Without further statistical calculations, it can be concluded that in two cases there is a good compliance of the group of participating laboratories with the target reproducibilities.

4.3 COMPARISON OF THE PROFICIENCY TEST OF JUNE 2019 WITH PREVIOUS PTS

	June 2019	May 2018	May 2017	May 2016	May 2015
Number of reporting laboratories	45	66	55	51	58
Number of test results	154	216	198	184	110
Number of statistical outliers	9	8	10	4	3
Percentage outliers	5.5%	3.6%	4.8%	2.1%	2.7%

Table 5: 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 2019	May 2018	May 2017	April 2016	May 2015	Target
SCCP	18-27%	13-28%	15-23%	23-33%	29%	16-18%
MCCP	13-33%	18%	19-20%	31-39%	19%	14-15%

Table 6: evolution of the observed uncertainties over the years

For the investigated components, the performance 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:

- Thirty-three participants (73%) reported to be ISO/IEC17025 accredited for the determination of total SCCP/MCCP in polymers.
- Twenty-six participants (58%) further cut or further grind the samples prior to analysis. The final estimated sample size reported was most often between 2x3mm.
- Almost all participants used a sample intake of 0.5 grams.
- To release/extract the SCCP/MCCP most participants (64%) reported to have used Toluene as extraction solvent. Six participants used a mixture of Toluene/Hexane, five participants used Hexane and two participants used THF/ACN.
- Almost all participants used an extraction time of 60 minutes and an extraction temperature of 60°C.

The effects of the analytical details is further discussed in §5.

5 DISCUSSION

It was observed that most participants were able to detect SCCP and MCCP in this proficiency test for the determination of total SCCP and MCCP in polymers.
The effect of the reported analytical details (see paragraph 4.4) on SCCP and MCCP were further investigated on those analytical details where it was possible to distinguish two or more meaningful subgroups to compare, see tables 7 and 8 respectively.

sample	analytical details	unit	n	average	RSD (%)
#19549	ISO/IEC17025 accredited	mg/kg	33	1243	23%
#19549	Not ISO/IEC17025 accredited	mg/kg	7	984	44%
#19550	ISO/IEC17025 accredited	mg/kg	33	489	17%
#19550	Not ISO/IEC17025 accredited	mg/kg	6	468	30%
#19549	Further cut or grinded	mg/kg	24	1223	19%
#19549	Used as received	mg/kg	17	1161	36%
#19550	Further cut or grinded	mg/kg	24	500	18%
#19550	Used as received	mg/kg	16	468	19%

Table 7: effect of analytical details on SCCP

sample	analytical details	unit	n	average	RSD (%)
#19549	Further cut or grinded	mg/kg	18	3327	13%
#19549	Used as received	mg/kg	12	3217	13%
#19550	Further cut or grinded	mg/kg	17	132	38%
#19550	Used as received	mg/kg	10	137	24%

Table 8: effect of analytical details on MCCP

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 tend to give higher levels of components, although the effect on the variation is less clear. Please note that the observed effects are not statistically significant.

Sample #19549 was used earlier as sample #17570 in iis17P05 (2017). In PT iis17P05 test values were excluded from the statistical evaluations where participants did not cut or grind the samples or where Hexane was used as extraction solvent. In this 2019 PT, as explained in paragraph 4, test results were not excluded based on the reported analytical details. In table 9 a comparison is given over the two proficiency tests. It is observed that the average levels of SCCP/MCCP are very similar. The observed reproducibility R(calc) for SCCP is in 2019 somewhat worse than in 2017, while the R(calc) for MCCP has improved in 2019.

	Sample #19549				Sample #17570			
	unit	n	average	R(calc)	unit	n	average	R(calc)
SCCP	mg/kg	43	1197	886	mg/kg	42	1291	552
MCCP	mg/kg	31	3268	1187	mg/kg	37	3281	1759

Table 9: comparison of sample #19549 with #17570

6 CONCLUSION

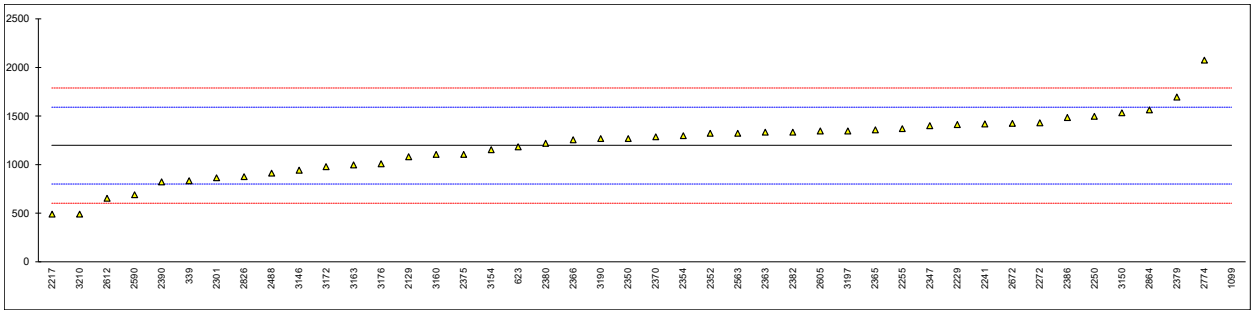
It is clear that the majority of the participants were able to determine total SCCP and total MCCP in the polymer matrix. However, it is noted that there is a large variation in the results dependent on the type of component and sample. Each laboratory has to evaluate its performance in this study and 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 #19549; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339	In house	834		-1.83	
623	ISO18219	1182.20		-0.07	
1099	In house	3921.61	R(0.01)	13.79	
2118		-----		-----	
2129	ISO18219	1079		-0.60	
2217	ISO18219	490.6	C	-3.57	first reported 392.6
2229	In house	1411.5		1.09	
2241	ISO18219	1417.5		1.12	
2250	ISO18219	1494		1.50	
2255	ISO18219	1369.8		0.88	
2267		-----		-----	
2272	ISO18219	1431.14		1.19	
2293	ISO18219	detected		-----	
2301	ISO18219	866.4		-1.67	
2347	In house	1401		1.03	
2350	ISO18219	1268.887		0.37	
2352	In house	1321.2		0.63	
2354	ISO18219	1297.6		0.51	
2357		-----		-----	
2363	ISO18219	1334		0.69	
2365	ISO18219	1357.9		0.82	
2366	ISO18219	1257		0.31	
2369		-----		-----	
2370	ISO18219	1286		0.45	
2375	ISO18219	1107		-0.45	
2379	ISO18219	1694.6		2.52	
2380	ISO18219	1219.148		0.11	
2382	ISO18219	1337.0		0.71	
2386	ISO18219	1484		1.45	
2390	ISO18219	822.9		-1.89	
2488	In house	914.3		-1.43	
2493		-----		-----	
2563	ISO18219	1324.9		0.65	
2590	ISO18219	689.62		-2.57	
2605	ISO18219	1344.53		0.75	
2612	In house	653.7		-2.75	
2672	ISO18219	1426.0		1.16	
2774	In house	2075		4.44	
2826	In house	880	C	-1.60	first reported 1100
2864	In house	1564.52		1.86	
3146	In house	941.1		-1.29	
3150	ISO18219	1531.1		1.69	
3154	ISO18219	1153.07		-0.22	
3160	In house	1105.563		-0.46	
3163	In house	1000		-1.00	
3172	In house	977		-1.11	
3176	ISO18219	1008	C	-0.95	first reported 252
3190	In house	1265.8		0.35	
3197	In house	1345.3		0.75	
3210	In house	494.241		-3.55	

				only toluene(/hexane),THF/ACN		only further cut/grinded	
	normality	OK		OK		OK	
	n	43		32		24	
	outliers	1		3 +9ex		1 +19ex	
	mean (n)	1196.70		1242.94		1222.88	
	st.dev. (n)	316.559	RSD=27%	233.794	RSD=19%	230.657	RSD=19%
	R(calc.)	886.37		654.62		645.84	
	st.dev.(Horwitz n=9)	197.671		204.142		200.974	
	R(Horwitz n=9)	553.48		571.60		563.75	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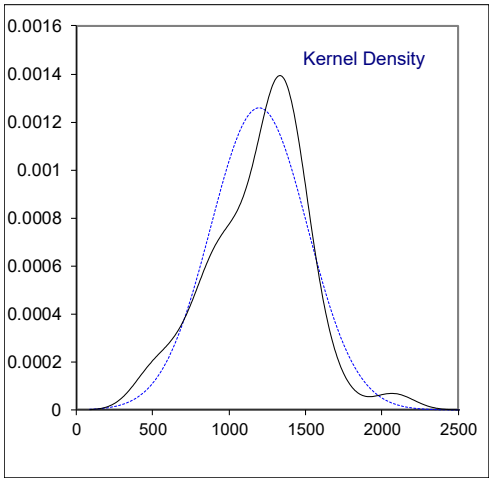
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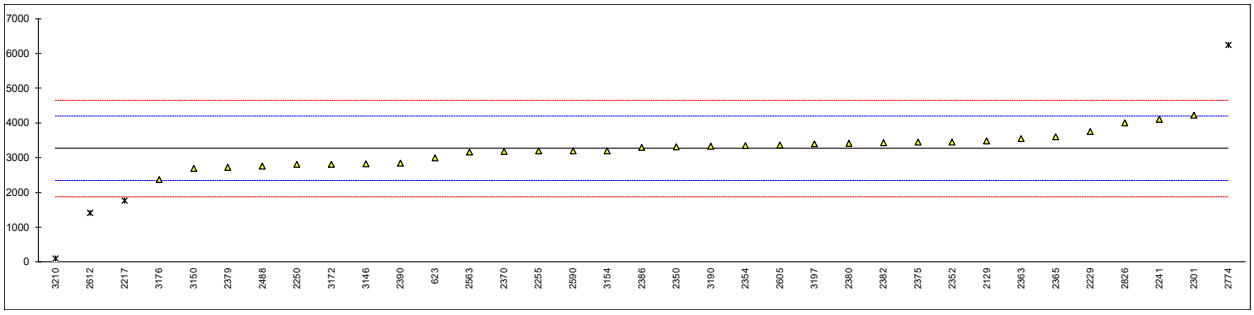
2000
2500

Determination of MCCP on sample #19549; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339		-----		-----	
623	ISO18219	2996.30		-0.58	
1099		-----		-----	
2118		-----		-----	
2129	ISO18219	3489		0.48	
2217	ISO18219	1766.4	C,R(0.05)	-3.24	first reported 1668.6
2229	In house	3748.7		1.04	
2241	ISO18219	4108.7		1.81	
2250	ISO18219	2807		-0.99	
2255	ISO18219	3192		-0.16	
2267		-----		-----	
2272		-----		-----	
2293	ISO18219	detected		-----	
2301	ISO18219	4226.4		2.07	
2347		-----		-----	
2350	ISO18219	3311.133		0.09	
2352	In house	3454.4		0.40	
2354	ISO18219	3349.4		0.18	
2357		-----		-----	
2363	ISO18219	3552		0.61	
2365	ISO18219	3593.1		0.70	
2366		-----		-----	
2369		-----		-----	
2370	ISO18219	3176		-0.20	
2375	ISO18219	3452		0.40	
2379	ISO18219	2717.1	C	-1.19	first reported 5122.7
2380	ISO18219	3411.306		0.31	
2382	ISO18219	3433.0		0.36	
2386	ISO18219	3296		0.06	
2390	ISO18219	2851.0		-0.90	
2488	In house	2760.1		-1.09	
2493		-----		-----	
2563	ISO18219	3160		-0.23	
2590	ISO18219	3194.36		-0.16	
2605	ISO18219	3363.20		0.21	
2612	In house	1406.1	C,R(0.05)	-4.01	first reported 1726.1
2672		-----		-----	reported: no capability for MCCP
2774	In house	6249	R(0.01)	6.43	
2826	In house	4010	C	1.60	first reported 5700
2864		-----		-----	
3146	In house	2827.0		-0.95	
3150	ISO18219	2694.8		-1.23	
3154	ISO18219	3203.74		-0.14	
3160		-----		-----	
3163		-----		-----	
3172	In house	2808		-0.99	
3176	ISO18219	2375		-1.92	
3190	In house	3329.8		0.13	
3197	In house	3405.4		0.30	
3210	In house	95.351	R(0.01)	-6.84	

				only toluene(/hexane),THF/ACN		only further cut/grinded	
	normality	OK		OK		OK	
	n	31		28		18	
	outliers	4		2 +5ex		1 +16ex	
	mean (n)	3267.61		3334.03		3326.64	
	st.dev. (n)	423.822	RSD=13%	384.884	RSD=12%	428.377	RSD=13%
	R(calc.)	1186.70		1077.68		1199.45	
	st.dev.(Horwitz n=9)	464.010		472.010		471.120	
	R(Horwitz n=9)	1299.23		1321.63		1319.14	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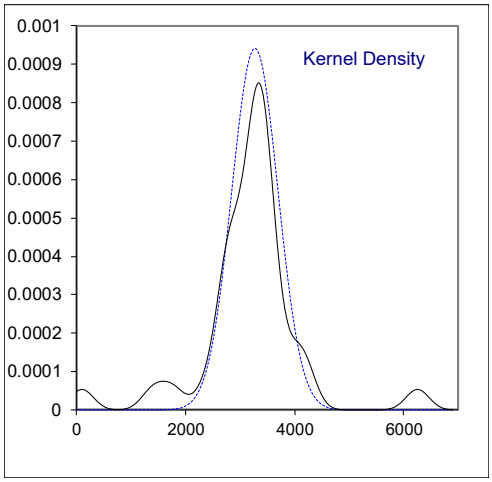
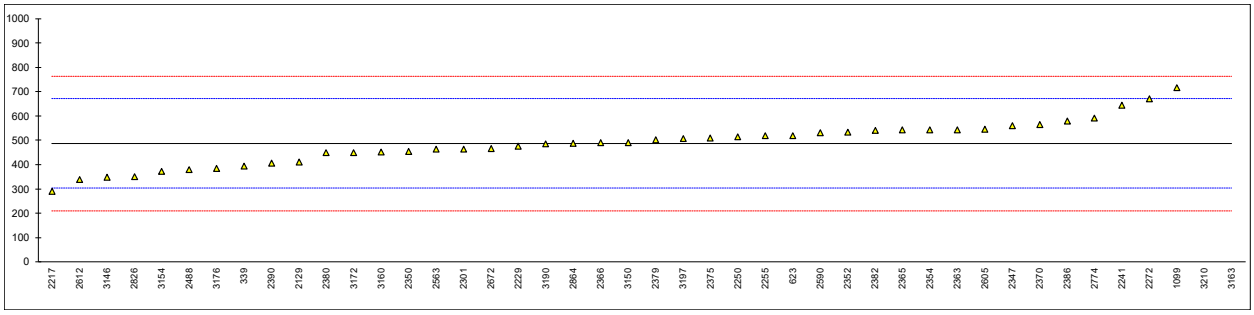
2826
2241
2301
2774

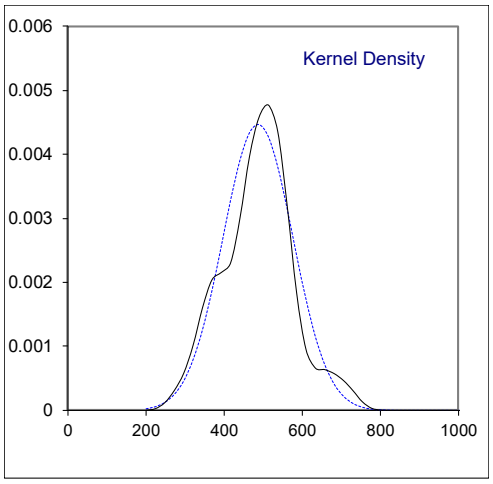

Kernel Density
0.001
0.0009
0.0008
0.0007
0.0006
0.0005
0.0004
0.0003
0.0002
0.0001
0
0
2000
4000
6000

Determination of SCCP on sample #19550; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339	In house	395		-1.00	
623	ISO18219	520.0		0.35	
1099	In house	715.62		2.47	
2118		-----		-----	
2129	ISO18219	410		-0.84	
2217	ISO18219	290.9	C	-2.13	first reported 230.2
2229	In house	475.1		-0.14	
2241	ISO18219	643.4		1.69	
2250	ISO18219	515		0.30	
2255	ISO18219	519.8		0.35	
2267		-----		-----	
2272	ISO18219	669.998		1.98	
2293	ISO18219	detected		-----	
2301	ISO18219	464.0		-0.26	
2347	In house	560		0.78	
2350	ISO18219	453.453		-0.37	
2352	In house	534.8		0.51	
2354	ISO18219	543.6		0.61	
2357		-----		-----	
2363	ISO18219	544		0.61	
2365	ISO18219	542.3		0.59	
2366	ISO18219	491		0.04	
2369		-----		-----	
2370	ISO18219	564		0.83	
2375	ISO18219	510		0.24	
2379	ISO18219	503.1		0.17	
2380	ISO18219	448.566		-0.42	
2382	ISO18219	540.0		0.57	
2386	ISO18219	579		0.99	
2390	ISO18219	406.3		-0.88	
2488	In house	379.82		-1.17	
2493		-----		-----	
2563	ISO18219	463		-0.27	
2590	ISO18219	530.21		0.46	
2605	ISO18219	545.92		0.63	
2612	In house	338.7		-1.62	
2672	ISO18219	465.95		-0.24	
2774	In house	592		1.13	
2826	In house	350		-1.49	
2864	In house	486.88	C	-0.01	first reported 973.76
3146	In house	347.8		-1.52	
3150	ISO18219	491.23		0.04	
3154	ISO18219	371.81		-1.26	
3160	In house	452.142		-0.39	
3163	In house	6250	R(0.01)	62.50	
3172	In house	450		-0.41	
3176	ISO18219	384		-1.12	
3190	In house	486.5		-0.01	
3197	In house	506.6		0.21	
3210	In house	1251.141	R(0.01)	8.28	

				only toluene(/hexane),THF/ACN		only further cut/grinded	
	normality	OK		OK		OK	
	n	42		34		24	
	outliers	2		1 +9ex		1 +19ex	
	mean (n)	487.65		504.09		499.55	
	st.dev. (n)	89.277	RSD=18%	82.831	RSD=16%	92.010	RSD=18%
	R(calc.)	249.98		231.93		257.63	
	st.dev.(Horwitz n=9)	92.205		94.838		94.111	
	R(Horwitz n=9)	258.17		265.55		263.51	

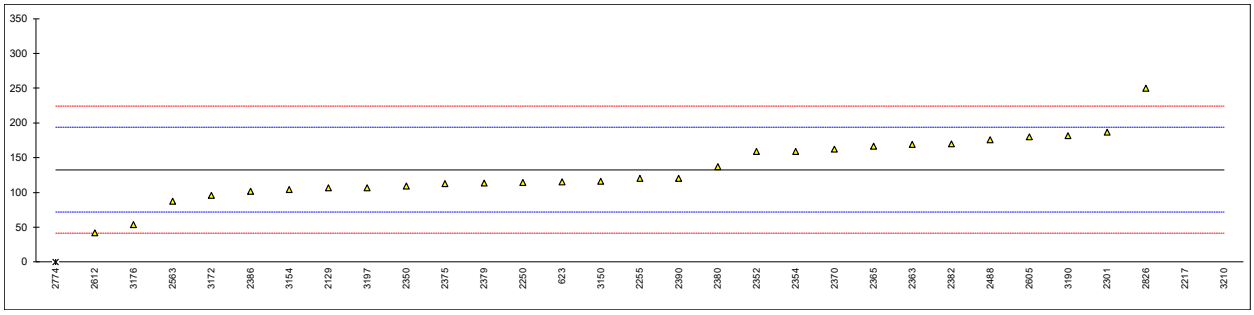

1000
900
800
700
600
500
400
300
200
100
0
2217
2612
3146
2826
3154
2488
3176
339
2390
2129
2380
3172
3160
2350
2563
2301
2672
2229
3190
2864
2366
3150
2379
3197
2375
2250
2255
623
2590
2352
2382
2365
2354
2363
2605
2347
2370
2386
2774
2241
2272
1099
3210
3163

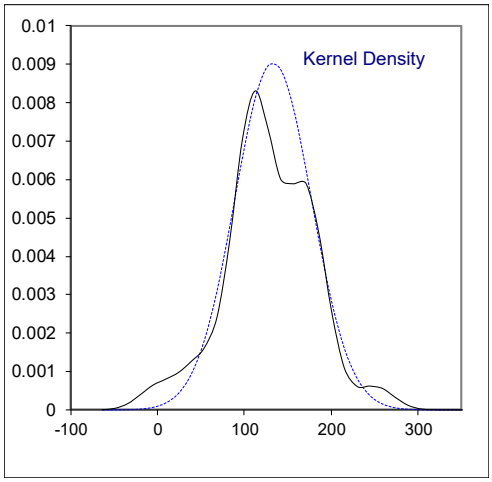

Kernel Density
0.006
0.005
0.004
0.003
0.002
0.001
0
0
200
400
600
800
1000

Determination of MCCP on sample #19550; results in mg/kg

lab	method	value	mark	z(targ)	remarks
339		-----		-----	
623	ISO18219	115.0		-0.58	
1099		-----		-----	
2118		-----		-----	
2129	ISO18219	107		-0.85	
2217	ISO18219	872.6	C,R(0.01)	24.22	first reported 773.04
2229	In house	<200		-----	
2241	ISO18219	<50	C	-----	first reported 335.1
2250	ISO18219	114		-0.62	
2255	ISO18219	120.1		-0.42	
2267		-----		-----	
2272		-----		-----	
2293	ISO18219	detected		-----	
2301	ISO18219	187.0		1.77	
2347		-----		-----	
2350	ISO18219	109.409		-0.77	
2352	In house	159.1		0.86	
2354	ISO18219	159.3		0.87	
2357		-----		-----	
2363	ISO18219	169		1.18	
2365	ISO18219	166.6		1.11	
2366		-----		-----	
2369		-----		-----	
2370	ISO18219	162		0.96	
2375	ISO18219	113		-0.65	
2379	ISO18219	113.6		-0.63	
2380	ISO18219	137.203		0.14	
2382	ISO18219	170.0		1.22	
2386	ISO18219	102		-1.01	
2390	ISO18219	120.2		-0.41	
2488	In house	175.68		1.40	
2493		-----		-----	
2563	ISO18219	87.6		-1.48	
2590		-----		-----	
2605	ISO18219	179.88		1.54	
2612	In house	42.1		-2.97	
2672		-----		-----	reported: no capability for MCCP
2774	In house	0	ex	-4.35	excluded as zero is not a real test value
2826	In house	250		3.84	
2864		-----		-----	
3146		-----		-----	
3150	ISO18219	116.33		-0.54	
3154	ISO18219	104.4		-0.93	
3160		-----		-----	
3163		-----		-----	
3172	In house	96		-1.21	
3176	ISO18219	54		-2.58	
3190	In house	181.5		1.59	
3197	In house	107.2		-0.84	
3210	In house	3613.394	R(0.01)	113.95	

				only toluene(/hexane),THF/ACN		only further cut/grinded	
	normality	OK		not OK		OK	
	n	28		24		17	
	outliers	2 +1ex		1 +6ex		0 +14ex	
	mean (n)	132.83		139.64		132.36	
	st.dev. (n)	44.172	RSD=33%	38.408	RSD=28%	50.872	RSD=38%
	R(calc.)	123.68		107.54		142.44	
	st.dev.(Horwitz n=9)	30.545		31.871		30.453	
	R(Horwitz n=9)	85.53		89.24		85.27	


350
300
250
200
150
100
50
0
2774
2612
3176
2563
3172
2386
3154
2129
3197
2350
2375
2379
2250
623
3150
2255
2390
2380
2352
2354
2370
2365
2363
2382
2488
2605
3190
2301
2826
2217
3210


Kernel Density
0.01
0.009
0.008
0.007
0.006
0.005
0.004
0.003
0.002
0.001
0
-100
0
100
200
300

APPENDIX 2
Analytical details

lab	laboratory ISO/IEC17025 accredited	sample preparation before use	final particle size (mm)	sample intake (g)	extraction solvent	extraction time (min)	extraction temp. (°C)
339	No	Used as received	19549:2x10 19550:4x4	0.5	Toluene	60	60
623	---	Further Cut	2x2	0.5	Toluene	60	60
1099	No	Further Grinded	<1	0.5	Toluene/Hexane	60	60
2118	No	Further Cut	2x3	0.5	Toluene	60	60
2129	Yes	Further Cut	2x3	0.5	Toluene	60	60
2217	No	Used as received	---	0.5	Hexane	60	60
2229	Yes	Further Cut	2x2	0.5	Toluene	60	60
2241	Yes	Further Cut	2x2	0.5	Toluene	60	60
2250	Yes	Used as received	2	0.5	Toluene	60	60
2255	Yes	Further Cut	cut as much as possible	0.5	Toluene	60	60
2267	---	---	---	---	---	---	---
2272	Yes	Further Cut	2x2	0.5	Toluene	60	60
2293	No	Used as received	---	0.5	Toluene/Hexane	60	70
2301	Yes	Used as received	5x5	1	Toluene	60	60
2347	---	---	---	---	---	---	---
2350	No	Further Cut	2x2	0.5	Toluene	60	60
2352	Yes	Further Cut	2x2x2	0.5	Toluene	60	60
2354	Yes	Used as received	5x5	0.5	Toluene	60	60
2357	---	---	---	---	---	---	---
2363	Yes	Further Cut	2x2	0.5	Toluene	60	69
2365	Yes	Further Cut	2x2	0.5	Toluene	60	60
2366	Yes	Further Cut	2x2x2	0.5	Toluene	60	60
2369	---	---	---	---	---	---	---
2370	Yes	Further Cut	3x3	0.5	Toluene	60	60
2375	Yes	Further Cut	2x2	0.5	Toluene	60	60
2379	No	Further Cut	2x2	0.5	Toluene	60	60
2380	Yes	Used as received	2x2	0.5	Toluene	60	60
2382	Yes	Used as received	2x2	0.5	Toluene	60	60
2386	Yes	Used as received	3x3	0.5	Toluene	60	60
2390	Yes	Further Cut	2x2	0.5	Toluene	60	60
2488	Yes	Used as received	---	---	---	---	---
2493	---	---	---	---	---	---	---
2563	Yes	Further Cut	---	0.5	Toluene	60	60
2590	Yes	Used as received	3x3	0.5	Toluene/Hexane	60	60
2605	Yes	Further Cut	2x2	0.5	THF/ACN	60	70
2612	Yes	Further Cut	2	0.5	Hexane	60	60
2672	Yes	Further Cut	<2	0.5	Toluene	60	60
2774	Yes	Used as received	as received	0.3	Toluene	60	60
2826	Yes	Further Cut	---	---	Toluene	60	60
2864	Yes	Used as received	---	0.5	Hexane	120	68
3146	Yes	Used as received	---	0.5	THF/ACN	60	70
3150	Yes	Used as received	---	0.5	Toluene/Hexane	60	60
3154	Yes	Used as received	---	0.5	Toluene	60	60
3160	No	Further Cut	3x3	0.5	Hexane	60	60
3163	No	Further Cut	0.5	0.0005	---	---	---
3172	---	---	---	---	---	---	---
3176	Yes	Further Grinded	5x5	0.5	Hexane	60	60
3190	Yes	Further Cut	2-3	0.5	Toluene/Hexane	75	60
3197	Yes	Used as received	5x5	0.25	Toluene/Hexane	60 and 15	60
3210	No	Used as received	---	0.5	Toluene	60	60

APPENDIX 3

Number of participants per country

2 labs in	BANGLADESH
1 lab in	BELGIUM
2 labs in	FRANCE
10 labs in	GERMANY
1 lab in	GUATEMALA
2 labs in	HONG KONG
2 labs in	HUNGARY
2 labs in	INDONESIA
2 labs in	ITALY
1 lab in	KOREA
13 labs in	P.R. of CHINA
1 lab in	PAKISTAN
1 lab in	POLAND
1 lab in	SPAIN
2 labs in	TAIWAN R.O.C.
1 lab in	THAILAND
2 labs in	THE NETHERLANDS
4 labs in	TURKEY

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

Literature:

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2 ASTM E178:02
3 ASTM E1301:03
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6 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
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8 IP 367:96
9 DIN 38402 T41/42
10 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
11 J.N. Miller, Analyst, 118, 455, (1993)
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13 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry 2002, Analyst 2002, 127, 1359-1364
14 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)
15 ISO/DIS 18219 IUC (2012), Determination of chlorinated hydrocarbons in leather – Chromatographic method for short-chain chlorinated paraffins (SCCP).
16 Mise au point de methodes pour l'analyse de substances critiques issues des rejets industriels et de la fabrication des produits de la filiere cuir, Aurelien Rey, September 26, 2014
17 Dr. R.G. Visser, Addendum to PT report "SCCP&MCCP in polymer April 2016", September 2016