Institute for
Interlaboratory Studies

Results of Proficiency Test
SCCP in Leather/Footwear
March 2022

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Report: iis22A04

June 2022

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1 INTRODUCTION

Commercially produced Chlorinated Paraffin's (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 in plasticizers and fire retardants. CPs are classified as persistent and non-biodegradable and they accumulate in the food chain. SCCP was 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 2019 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of SCCP in Leather/Footwear every year. During the annual proficiency testing program 2021/2022 it was decided to continue the proficiency test for the determination of SCCP in Leather/Footwear.

In this interlaboratory study 56 laboratories in 19 countries registered for participation. See appendix 3 for the number of participants per country. In this report the results of the SCCP in Leather/Footwear 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 one leather sample of 3 grams positive on SCCP and labelled #22535.
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

A batch of black colored leather positive on SCCP was selected. The leather was grinded into small pieces and mixed thoroughly. After homogenization 77 plastic bags were filled with approximately 3 grams each and labelled #22535.
The homogeneity of the subsamples was checked by determination of SCCP in accordance with ISO18219 on 10 stratified randomly selected subsamples.

	SCCP in mg/kg
sample #22535-1	131.4
sample #22535-2	125.5
sample #22535-3	133.0
sample #22535-4	132.0
sample #22535-5	116.2
sample #22535-6	123.1
sample #22535-7	129.1
sample #22535-8	116.6
sample #22535-9	125.5
sample #22535-10	131.2

Table 1: homogeneity test results of subsamples #22535

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

	SCCP in mg/kg
r (observed)	17.3
reference method	Horwitz (n=9)
0.3 x R (reference method)	24.6

Table 2: evaluation of the repeatability of subsamples #22535

The calculated repeatability is in agreement with 0.3 times the estimated reproducibility calculated with the Horwitz equation. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample labelled #22535 was sent on February 23, 2022.

2.5 ANALYZES

The participants were requested to determine: SCCP and MCCP. It was noted in the instructions of this PT to not use less than 0.5 grams per determination to ensure the homogeneity. In the instructions was also noted not to dry or age the sample, nor determine volatile matter. 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 sample as if it was a routine sample 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, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

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

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

The z-scores were calculated according to:

$z_{(target)}$ = (test result - average of PT) / target standard deviation

The $z_{(target)}$ scores are listed in the test result tables in appendix 1.

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

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

4 EVALUATION

In this proficiency test some problems were encountered with the dispatch of the samples. Five participants reported test results after the final reporting date and nine other participants were not able to report any test results. Not all participants were able to report all tests requested.
In total 47 participants reported 86 numerical test results. Observed were 3 outlying test results, which is 3.5%. In proficiency studies 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 COMPONENT

In this section the test results are discussed per component. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 4.

For the determination of SCCP and MCCP, ISO18219 is considered to be the official test method. A new version of this method was published in 2021. It was published in two parts. ISO18219-1 describes the determination of SCCP and ISO18219-2 the determination of MCCP. The difference between the two versions is explained in paragraph 5 Discussion. Regretfully, ISO18219 still does not contain any precision data. Therefore, the calculated reproducibility was compared against the estimated reproducibility calculated with the Horwitz equation based on nine components (n=9).

SCCP: This determination may be problematic for a number of laboratories. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated reproducibility calculated with the Horwitz equation (n=9).

MCCP: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility calculated with the Horwitz equation (n=9).

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from reference methods are presented in the next table.

Component	unit	n	average	2.8 * sd	R(target)
SCCP	mg/kg	44	127	90	82
MCCP	mg/kg	39	539	295	281

Table 3: reproducibilities of tests on sample #22535

Without further statistical calculations, it can be concluded that for the SCCP and MCCP determination there is a good compliance of the group of participants with the target reproducibility.

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

	March 2022	February 2021	April 2020	March 2019
Number of reporting laboratories	47	46	53	54
Number of test results	86	82	102	99
Number of statistical outliers	3	6	7	2
Percentage of statistical outliers	3.5%	7.3%	6.9%	2.0%

Table 4: 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	March 2022	February 2021	April 2020	March 2019	Target *)
SCCP	25%	16%	24%	31%	17-24%
MCCP	20%	14%	22%	26%	17-24%

Table 5: development of the uncertainties (RSD) over the years

*) Horwitz based om nine components calculated at respectively 1000 – 100 mg/kg

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

4.4 EVALUATION OF THE ANALYTICAL DETAILS

The reported analytical details from the participants are listed in appendix 2.

- About 83% of the reporting participants mentioned to be accredited for the determination of SCCP and/or MCCP in leather.
- Prior to analysis the samples were further cut or grinded by about 20% of the reporting participants, about 80% used the samples as received.
- The amount of sample intake varied between 0.2 and 1.5 grams, about 80% used 0.5 grams.
- About 85% of the reporting participants used n-Hexane as release solvent. Two laboratories used a combination of Hexane/Dichloromethane, while they reported to have used ISO18219 version 2021. Version 2021 does not describe the use for Hexane/Dichloromethane for clean-up, instead Hexane/Sulfuric Acid is used. Four laboratories reported to have used Toluene, which is not described in the 2015 or 2021 version of method ISO18219. One laboratory using Toluene used an in-house method with Sulfuric Acid clean-up.
- All reporting participants used an extraction time of 60 minutes and an extraction temperature of 60°C.

As the majority of the group follows the same analytical procedures no separate statistical analysis based on these analytical details has been performed.

5 DISCUSSION

In 2021 two new versions were published (ISO18219-1 for SCCP and ISO18219-2 for MCCP) to replace the 2015 version of ISO18219. Different in both procedures is a change in the clean-up step. Instead of using a mixture of n-Hexane/Dichloromethane and solid phase separation (SPE cartridge) as clean-up, the two versions of 2021 use a mixture of n-Hexane/Sulfuric Acid with liquid phase separation as clean-up.

About 70% used ISO18219 version 2021 for determining SCCP/MCCP, about 25% used version 2015 and about 5% used an in house test method.
A separate statistical evaluation was made of the test results of the participants using version 2015 or version 2021 of the method with Hexane only as release solvent. See appendix 1 for the evaluation. Participants that reported to have used an in house method or another solvent than Hexane were not included in this evaluation. The consensus value and variation for both separate evaluations did not differ much. The test results obtained with version 2015 of the method give a slightly higher consensus value. However, this difference in consensus value found for the two method versions does not appear to be significant.

In this proficiency test for the determination of SCCP in leather it was noticed that all reporting participants were able to detect SCCP. The majority of the participants reported also the presence of MCCP.

When the results of this interlaboratory study were compared to the Leather Standard by OEKO-TEX®, it was noticed that all participants, except one, would make an identical decision about the acceptability of the leather for the determined components and would have rejected the sample for all categories.

Ecolabel	baby clothes	in direct skin contact	no direct skin contact
Leather by OEKO-TEX®	<50 mg/kg *)	<50 mg/kg *)	<50 mg/kg *)

Table 6: Leather Standard by OEKO-TEX®

*) This concerns the sum of SCCP and MCCP

6 CONCLUSION

The majority of the participants is able to determine SCCP and MCCP in the leather matrix. The observed reproducibilities in this proficiency test on SCCP in Leather are in line with the reproducibilities of SCCP and MCCP of previous PTs.

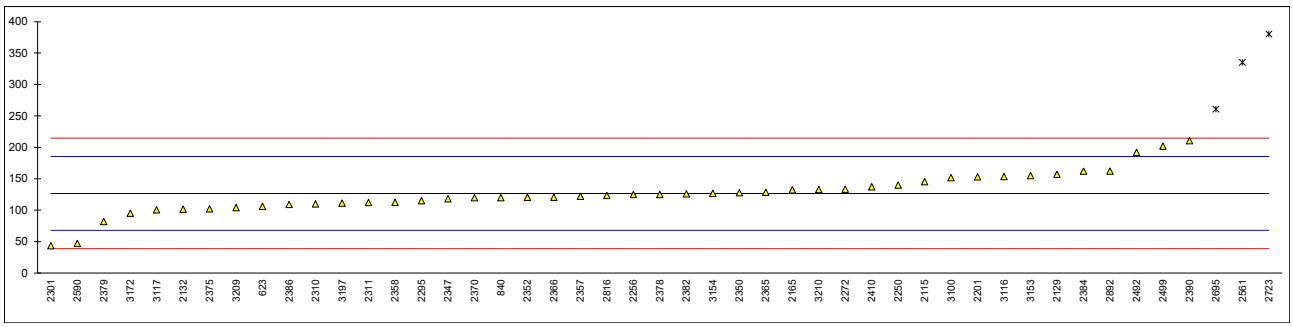
However, each laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

APPENDIX 1

Determination of SCCP on sample #22535; results in mg/kg

lab	method	value	mark	z(targ)	remarks
623	ISO18219-1:2021	106.1		-0.70	
840	ISO18219-1:2021	120		-0.23	
2115	ISO18219-1:2021	145.3		0.63	
2129	ISO18219:2015	157		1.03	
2132	ISO18219-1:2021	101.435		-0.86	
2135		-----		-----	
2165	ISO18219-1:2021	132.2		0.19	
2201	ISO18219:2015	153		0.90	
2241		-----		-----	
2250	ISO18219-1:2021	140		0.45	
2256	ISO18219-1:2021	125		-0.06	
2265		-----		-----	
2272	ISO18219-1:2021	133.5		0.23	
2295	ISO18219-1:2021	115		-0.40	
2301	ISO18219-1:2021	43.57	C	-2.83	first reported: 30.88
2310	ISO18219:2015	110		-0.57	
2311	ISO18219-1:2021	112.1		-0.50	
2330		-----		-----	
2347	ISO18219-1:2021	118		-0.30	
2350	ISO18219:2015	127.7		0.03	
2352	ISO18219-1:2021	120.3		-0.22	
2357	ISO18219-1:2021	122.0		-0.16	
2358	ISO18219:2015	112.47		-0.48	
2363		-----		-----	
2365	ISO18219-1:2021	128.35		0.06	
2366	ISO18219-1:2021	120.7		-0.20	
2370	ISO18219-1:2021	120		-0.23	
2375	ISO18219-1:2021	102		-0.84	
2378	ISO18219-1:2021	125		-0.06	
2379	ISO18219:2015	82.0110		-1.52	
2382	ISO18219-1:2021	126.0		-0.02	
2384	ISO18219-1:2021Mod.	161.86		1.20	
2386	ISO18219:2015	109		-0.60	
2390	ISO18219:2015	210.5		2.86	
2410	ISO18219-1:2021	137		0.35	
2492	ISO18219-1:2021	191.96		2.22	
2499	ISO18219-1:2021	201.91	C	2.56	first reported: 330.18
2561	In house	335.2	C,R(0.01)	7.11	first reported: 412.028
2590	ISO18219-1:2021	47		-2.72	
2695	ISO18219-1:2021	260.58	R(0.01)	4.56	
2723	ISO18219-1:2021	380	R(0.01)	8.63	
2762		-----		-----	
2816	ISO18219:2015	123.309		-0.11	
2892	ISO18219-1:2021	162.315		1.21	
3100	ISO18219-1:2021	151.5		0.85	
3116	ISO18219:2015	153.4		0.91	
3117	ISO18219:2015	100.63		-0.89	
3153	ISO18219-1:2021	155.0		0.97	
3154	ISO18219-1:2021	126.625		0.00	
3172	ISO18219-1:2021	95.137		-1.08	
3185		-----		-----	
3197	ISO18219-1:2021	111.3		-0.52	
3209	In house	104.02		-0.77	
3210	In house	132.69		0.20	
3218		-----		-----	
3228		-----		-----	

			ISO18219-1:2021 Hexane only	ISO18219:2015 Hexane only
normality	suspect		not OK	not OK
n	44		27	10
outliers	3		2	0
mean (n)	126.679		120.816	128.202
st.dev. (n)	32.2465	RSD = 25%	30.6670 RSD = 25%	36.3446 RSD = 28%
R(calc.)	90.290		85.867	101.765
st.dev.(Horwitz n=9)	29.3399		28.1823	29.6392
R(Horwitz n=9)	82.152		78.910	82.990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100
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3197
2311
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2295
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2256
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2365
2165
3210
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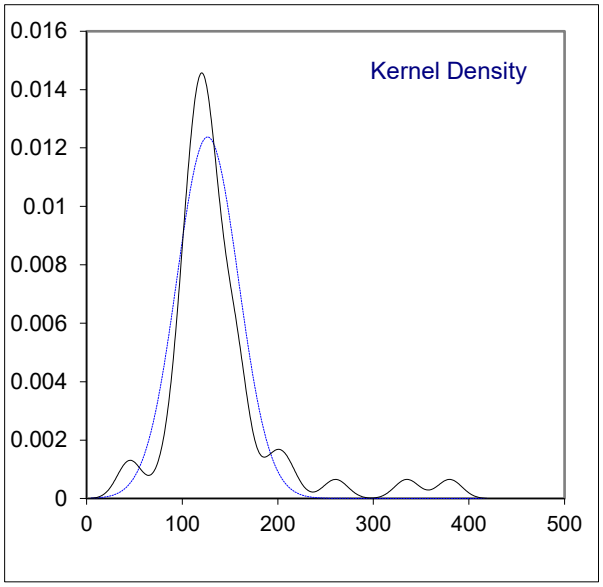
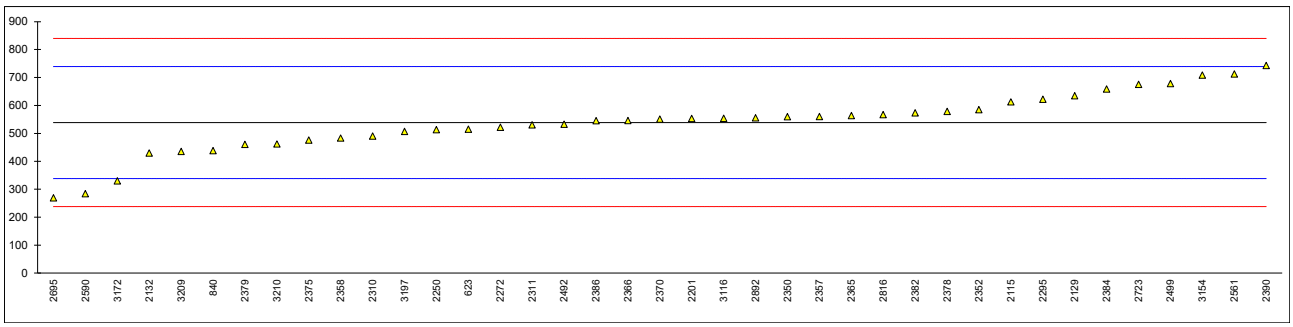
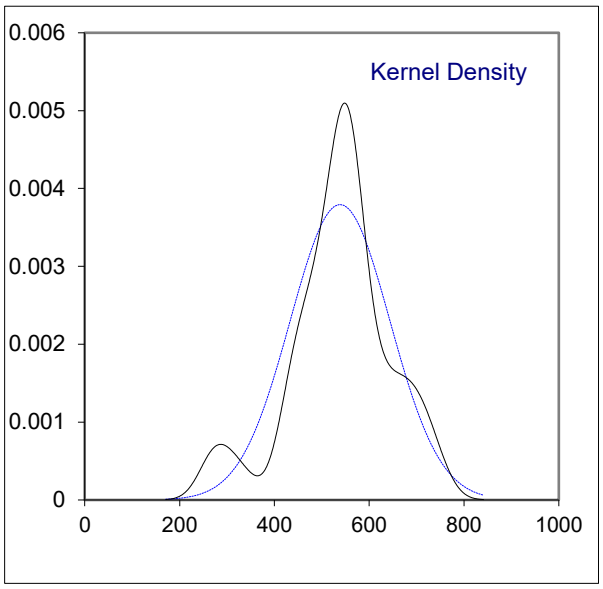

Kernel Density
0.016
0.014
0.012
0.01
0.008
0.006
0.004
0.002
0
0
100
200
300
400
500

Determination of MCCP on sample #22535; results in mg/kg

lab	method	value	mark	z(targ)	remarks
623	ISO18219-2:2021	514.6		-0.24	
840	ISO18219-2:2021	438		-1.00	
2115	ISO18219-2:2021	612.5		0.73	
2129	ISO18219:2015	635		0.96	
2132	ISO18219-2:2021	429.5		-1.09	
2135		-----		-----	
2165		-----		-----	
2201	ISO18219-2:2021	553		0.14	
2241		-----		-----	
2250	ISO18219-2:2021	513		-0.26	
2256		-----		-----	
2265		-----		-----	
2272	ISO18219-2:2021	521.5		-0.17	
2295	ISO18219-2:2021	622	C	0.83	first reported: 290
2301		Not tested		-----	
2310	ISO18219:2015	490		-0.49	
2311	ISO18219-2:2021	530.6		-0.08	
2330		-----		-----	
2347		-----		-----	
2350	ISO18219:2015	559.57		0.21	
2352	ISO18219-2:2021	584.7		0.46	
2357	ISO18219-2:2021	560.0		0.21	
2358	ISO18219:2015	482.81		-0.56	
2363		-----		-----	
2365	ISO18219-2:2021	563.50		0.25	
2366	ISO18219-2:2021	545.8		0.07	
2370	ISO18219-2:2021	551		0.12	
2375	ISO18219-2:2021	476		-0.63	
2378	ISO18219-2:2021	578		0.39	
2379	ISO18219:2015	460.3980		-0.78	
2382	ISO18219-1:2021	573.4		0.35	
2384	ISO18219-2:2021Mod.	658.78		1.20	
2386	ISO18219:2015	545		0.06	
2390	ISO18219:2015	743.0	C	2.04	first reported: 962.3
2410		-----		-----	
2492	ISO18219-2:2021	532.74		-0.06	
2499	ISO18219-2:2021	678.25	C	1.39	first reported: 1123.61
2561	In house	712.576		1.73	
2590	ISO18219-2:2021	284		-2.54	
2695	ISO18219-2:2021	269.39		-2.68	
2723	ISO18219-2:2021	675		1.36	
2762		-----		-----	
2816	ISO18219:2015	566.643		0.28	
2892	ISO18219-2:2021	555.410		0.17	
3100	ISO18219-2:2021	not analyzed		-----	
3116	ISO18219:2015	554.0		0.15	
3117		-----		-----	
3153		-----		-----	
3154	ISO18219-2:2021	708.410		1.69	
3172	ISO18219-2:2021	329.75	C	-2.08	first reported: 249.44
3185		-----		-----	
3197	ISO18219-2:2021	506.9		-0.32	
3209	In house	435.23		-1.03	
3210	In house	461.58		-0.77	
3218		-----		-----	
3228		-----		-----	

			ISO18219-2:2021 Hexane only	ISO18219:2015 Hexane only
normality	OK		OK	not OK
n	39		24	8
outliers	0		0	0
mean (n)	538.757		523.405	550.178
st.dev. (n)	105.25630	RSD = 20%	111.2120 RSD = 21%	87.6693 RSD = 18%
R(calc.)	294.718		311.394	245.474
st.dev.(Horwitz n=9)	100.3503		97.9158	102.1545
R(Horwitz n=9)	280.981		274.164	286.033


900
800
700
600
500
400
300
200
100
0
2695
2590
3172
2132
3209
840
2379
3210
2375
2358
2310
3197
2250
623
2272
2311
2492
2386
2366
2370
2201
3116
2892
2350
2357
2385
2816
2382
2378
2352
2115
2295
2129
2384
2723
2499
3154
2561
2390


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

APPENDIX 2 Analytical details

lab	ISO17025 accredited	sample grinded or cut	intake (g)	release solvent	extraction time (min)	extraction temp (°C)	remarks
623	Yes	Used as received	0.5	hexane	60	60	
840	Yes	Further cut	0.5	HEXANE	60 minutes	60	
2115	No	Used as received	0.5 g	Hexane/Dichloromethane	60 min	60°C	
2129	Yes	Used as received	0.5g	Toluol	60	60	
2132	No	Used as received	1 gram	n-Hexane	60 minutes	60 °C	
2135	---	---					
2165	Yes	Used as received	1.500g	hexane	60 minutes	60°C	
2201	Yes	Used as received	0.5g	Hexane	60min	60 degree	
2241	---	---					
2250	Yes	Used as received	0,5	hexane	60	60	
2256	Yes	Used as received	1.0012 g	n-hexane	60mins	60℃	
2265	---	---					
2272	Yes	Used as received	0.5g	hexane	60min	60°	
2295	Yes	Further cut	0.5 g	nHexane	60 minutes	60 C	
2301	Yes	Used as received	1.0031	hexane	60 min	60c	
2310	Yes	Further cut	0.5	Hexane	60 minutes	60°C	
2311	Yes	Further cut	0.5g	Hexane	60	60	
2330	---	---					
2347	Yes	Used as received	0.5g	Hexane	60min±2min	60℃±2℃	
2350	Yes	Used as received	0.5g	Hexane	60 min	60 ℃	
2352	Yes	Used as received	0.5g	Hexane	60min	60℃	
2357	---	---					
2358	Yes	Used as received	0.5 g	Hexane	60 mins	60 °C	
2363	---	---					
2365	Yes	Used as received	0.5g	n-hexane	60min	60℃	
2366	Yes	Further cut	0.5	n-hexane	60	60	
2370	Yes	Used as received	1 g	Hexane	60 min	60 ℃	
2375	Yes	Used as received	0.5gr	Hexane	60min	60°C	
2378	Yes	Used as received	0.5g	N-hexane	60	60	
2379	No	Further cut	0.5 g	Hexane	60 minutes	60 °C	
2382	Yes	Used as received	0.5g	n-Hexane	60min	60°C	
2384	Yes	Further grinded	0.5g	toluene	60 minutes	60 °C	
2386	Yes	Used as received	0,5 g	n-hexane	60 min	60 °C	
2390	Yes	Used as received	0.5g	n-hexane	60 min	60	
2410	Yes	Used as received	0.5 g	Toluene, Methanol	60 min	(60±2)°C	
2492	Yes	Used as received	0.5g	Hexane/DCM	60 mins	60 °C	
2499	No	Used as received	0.5 g	hexane	60 minutes	60°C	
2561	No	Used as received	1	hexene	60	60	
2590	Yes	Used as received	0.5g	hexane	60min	60°C	
2695	No	Further cut	0.5	Hexane	60	60	
2723	Yes	Used as received	0.5	Hexane	1h	60°C	
2762	---	---					
2816	No	Used as received	1	hexane	60	60	
2892	Yes	Used as received	0.5g	n-hexane	60	60	
3100	Yes	Used as received	0.5g	n-Hexane	(60±2)min	(60±5)℃	
3116	Yes	Used as received	1 gram	n-Hexane	60 mins	60°C	
3117	Yes	Used as received	0.5g	n-Hexane	60 min	60°C	
3153	Yes	Used as received	0.5 gram	N-hexane	60 minutes	60°C	
3154	Yes	Used as received	0,5	n-hexane	60	60	
3172	Yes	---					
3185	---	---					
3197	Yes	Further cut	0,5 g	n-hexane	60 min.	60 C	
3209	Yes	Used as received	0.5	10ml	60min	60℃	
3210	No	Used as received	1 gram	Toluene	60 minutes	60°C	*)
3218	---	---					
3228	Yes	---					

*) Sulfuric acid clean-up made

APPENDIX 3

Number of participants per country

1 lab in CAMBODIA
1 lab in CZECH REPUBLIC
1 lab in DENMARK
1 lab in FRANCE
6 labs in GERMANY
5 labs in HONG KONG
2 labs in INDIA
2 labs in INDONESIA
5 labs in ITALY
2 labs in KOREA, Republic of
1 lab in MALAYSIA
19 labs in P.R. of CHINA
1 lab in PAKISTAN
1 lab in SWITZERLAND
1 lab in TAIWAN
1 lab in THAILAND
3 labs in TURKEY
1 lab in UNITED KINGDOM
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
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?

Literature

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2 ISO5725:86
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11 W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
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