Results of Proficiency Test SCCP in Polymer June 2020

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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 <u>leather</u> 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.

<u>MCCP:</u> 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

- <u>SCCP:</u> 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.
- <u>MCCP:</u> 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)
МССР	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
МССР	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			
23/5	15018219	390			
23/9	15018219	1211.5789			
∠380 2202	10010210	1120.2			
2302 2386	ISO10219	631 0			
2300	15010219	425.6			
2508	ISO18219	423.0	AV		Test result excluded used a deviating solvent
2563	ISO10219	630 3	ex		Test result excluded, used a deviating solvent
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	mnodoo	565	0A		root rootit oxolidada, dood a domating corrolit
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			
		<b></b>			<u>Only Toluene</u>
	normality	OK			OK
	n	36			29
	outliers	0 (+/excl)			
	mean (n)	//4.423			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) (363.748)
		(302.422)			
<sup>3000</sup> T					0.0012
0500					x x Open
2000					
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1500 +					
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315, 2622	259 316 321 33 33 33 33 33 33 33 53 53 53 53 53 53	323 320 235 235 261 2 261 2 62 5 261 2 261 2 261 2 20 2 20 2 20 2 320	238 238 84( 3197 3197	237 317, 236(	ສູສູສູສູສີສີສີສີສີສີສີສີສີສີສີສີຊີ∓ີສີ -1000 0 1000 2000 3000 400

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

lab	method	value	mark	z(targ)	remarks
110					
339	100 ( 00 ( 0				
623	ISO18219	1484.000			
840	15018219	1338			
041 2118	15016219	2920			
2129	ISO18219	1531			
2247	ISO18219	1531.08			
2250	ISO18219	1128			
2267					
2310	ISO18219	1150			
2311	ISO18219	1227			
2350	ISU18219	1339.216			
2352	ISO18219	2091			
2357	In-house	2677			
2363	ISO18219	2705			
2365	ISO18219	2543.381			
2366					
2370	ISO18219	2250			
2375	ISO18219	1085			First reported 2006 0442
2379	15018219	4330.8973	C,R(0.05)		First reported 3896.8413
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					
2672		690			
2816	In-house	1776.6833			
2826	ISO18219	1292.98			
2835					
2864					
2886	100 100 10				
3154	ISO18219	445.98	R(0.05)		
3103		750 2721			
3197	In-house	1707.0			
3209	In-house	1069.12			
3210	In-house	877.103			
3214					
3228					
3233					Only Talyona
	normality	OK			Orig Toldene
	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)
<sup>5000</sup> ]					0.0007
4500 -					× 0.0006 - Kernel Density
4000					0,0005
3000					



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

110     h-house     202,025     ex     -0.67     Test result excluded, used a deviating solvent       623     ISO18219     242,300     -0.09       441     ISO18219     316     -1.27       1118     ISO18219     316     -0.91       2121     ISO18219     285     0.73       2121     ISO18219     286     0.73       2204     ISO18219     286     0.73       2215     ISO18219     286     0.73       2216     ISO18219     286     0.73       2310     ISO18219     186.8     -0.09       2311     ISO18219     186.8     -0.02       2335     ISO18219     218.725     -0.55       2345     ISO18219     210.7     1.23       2356     ISO18219     20.0     -0.31       2357     In-house     285     0.35       2368     ISO18219     20.0     -0.31       2371     ISO18219     20.0     -0.31       2382     ISO18219     20.0     -0.31       2390     ISO18219     20.0     -0.31       2391     ISO18219     20.0     -0.31       2392     ISO18219     20.0     R(0.01)     7.31       2384 <th>lab</th> <th>method</th> <th>value</th> <th>mark</th> <th>z(targ)</th> <th>remarks</th>	lab	method	value	mark	z(targ)	remarks
339     In-house     141     -2.05       251     ISO18219     306     1.14       441     ISO18219     306     1.14       1218     ISO18219     20.163     -0.91       2118     ISO18219     285.6     0.73       2247     ISO18219     285.6     0.73       2257     ISO18219     285.6     0.73       2257     ISO18219     285.6     0.74       2350     ISO18219     185     -1.20       2351     ISO18219     185     -1.20       2352     In-house     286     0.63       2354     ISO18219     285.7     0.35       2355     ISO18219     290.0     -0.31       2356     ISO18219     230.0     -0.32       2356     ISO18219     230.0     -0.32       2361     ISO18219     23.551     0.33       2370     ISO18219     22.5     0.30       2381     ISO18219     22.5     0.30       2382     ISO18219     22.5     0.30       2383     ISO18219     22.5     0.30       2384     ISO18219     22.5     0.30       2385     ISO18219     22.5     0.30       2386 <t< td=""><td>110</td><td>In-house</td><td>202.0525</td><td>ex</td><td>-0.87</td><td>Test result excluded, used a deviating solvent</td></t<>	110	In-house	202.0525	ex	-0.87	Test result excluded, used a deviating solvent
623     ISO18219     242.300     -0.09       844     ISO18219     313     1.27       118     ISO18219     200.163     -0.91       2129     ISO18219     285.68     0.74       2260     ISO18219     200     -0.91       2271     ISO18219     286.68     0.74       2280     ISO18219     186.8     -0.97       2310     ISO18219     186.8     -0.97       2331     ISO18219     218.725     -0.52       2344     ISO18219     285.68     0.74       2355     ISO18219     296     0.22       2354     ISO18219     298     0.23       2355     ISO18219     298.6     0.46       2356     ISO18219     293.0     -0.03       2376     ISO18219     293.0     -0.03       2380     SO18219     283.5     0.32       2380     ISO18219     283.5     0.32       2380     ISO18219     28.56     w.7.31       2380     ISO18219     28.56     w.7.31       2380     ISO18219     28.56     w.7.31       2381     ISO18219     28.56     w.7.31       2380     ISO18219     28.56     w.7.31 <t< td=""><td>339</td><td>In-house</td><td>141</td><td></td><td>-2.05</td><td></td></t<>	339	In-house	141		-2.05	
840     ISO18219     306     1.14       441     ISO18219     200.163     -0.91       2118     ISO18219     285     0.73       2247     ISO18219     286.68     0.74       2250     ISO18219     286.68     0.74       2267     Torono         2268     ISO18219     186.8     -0.97       2310     ISO18219     186.8     -0.97       2331     ISO18219     187.25     -0.55       2352     In-house     248     0.02       2353     ISO18219     218.725     -0.55       2354     ISO18219     20.0     -0.31       2355     ISO18219     23.00     -0.33       2361     ISO18219     23.00     -0.32       2373     ISO18219     22.07     -0.51       2382     ISO18219     22.07     -0.51       2390     ISO18219     22.07     -0.51       2390     ISO18219     28.05     No.02       2591     ISO18219     28.07     -0.02       2592     ISO18219     24.55     -0.02       2593     ISO18219     24.55     -0.02       2594     ISO18219     24.55     -0.02	623	ISO18219	242.300		-0.09	
B41     ISO18219     313     1.27       118     ISO18219     285.68     0.73       2129     ISO18219     285.68     0.74       2260     ISO18219     285.68     0.74       2270     ISO18219     285.68     0.74       2211     ISO18219     186.8     -0.97       2311     ISO18219     187.25     -0.50       2347     ISO18219     218.725     -0.52       2351     ISO18219     226     0.32       2353     ISO18219     270.986     0.42       2364     ISO18219     270.986     0.42       2375     ISO18219     283.9551     0.33       2380     SO18219     283.9     0.13       2380     SO18219     282.5     0.30       2380     ISO18219     282.5     0.30       2380     ISO18219     282.5     0.32       2380     ISO18219     28.050     R(0.01)       2390     ISO18219     28.050     R(0.01)	840	ISO18219	306		1.14	
2118     ISO18219     200.163     -0.91       2247     ISO18219     285     0.73       2260     ISO18219     200     -0.91       2271     ISO18219     200     -0.91       2281     ISO18219     186.8     -0.97       2310     ISO18219     186.8     -0.97       2331     ISO18219     186.8     -0.97       2332     IsO18219     186.7     -1.20       2335     IsO18219     218.725     -0.55       2336     ISO18219     218.725     -0.30       2337     ISO18219     279.986     0.43       2336     ISO18219     230.0     -0.33       2376     ISO18219     230.0     -0.33       2376     ISO18219     265.5     0.30       2390     ISO18219     265.7     0.30       2390     ISO18219     262.7     -0.51       2390     ISO18219     262.72     0.30       2563     ISO18219     28.725     0.30       2564     In-house     670.722935     R(0.01)     7.03       2571     ISO18219     28.725     N.30     Test result excluded, used a deviating solvent       2612     SO18219     24.55     0.02	841	ISO18219	313		1.27	
2129       ISO18219       285.68       0.73         2250       ISO18219       200       -0.91         2267       ISO18219       200       -0.91         2310       ISO18219       195.68       -0.97         2330       ISO18219       218.7       -0.91         2351       ISO18219       218.7       -0.55         2352       In-house       248       0.02         2354       ISO18219       265       0.35         2365       ISO18219       265       0.32         2376       ISO18219       265       0.33         2385       ISO18219       262.5       0.30         2379       ISO18219       262.5       0.30         2380       ISO18219       262.5       0.30         2380       ISO18219       262.75       exto         2453       ISO18219       280.50       R(0.01)       7.03         2508       ISO18219       280.50       R(0.01)       7.03         2511       n-louse       670.722935       R(0.01)       8.8         2521       ISO18219       245.50       0.30       7         2511       0.13       0.14	2118	ISO18219	200.163		-0.91	
2247 ISO18219 285.68 0.74 2260 ISO18219 200 -0.91 2267 ISO18219 1851.20 2310 ISO18219 1851.20 2352 ISO18219 1851.20 2353 ISO18219 218.725 -0.55 2352 In-house 248 0.02 2354 ISO18219 218.725 -0.55 2355 ISO18219 270.986 0.46 2366 ISO18219 270.986 0.46 2370 ISO18219 270.986 0.46 2370 ISO18219 270.986 0.46 2370 ISO18219 203 -0.65 2370 ISO18219 203 -0.65 2370 ISO18219 26.9.55 0.30 2380 ISO18219 26.5.5 0.30 2422 ISO18219 26.5.5 0.30 2422 ISO18219 26.5.5 0.30 2422 ISO18219 26.5.5 0.30 2422 ISO18219 26.5.5 0.002 250 ISO18219 26.5.7 0.30 2422 ISO18219 26.5.7 0.423 2435 ISO18219 26.5.7 0.423 2435 ISO18219 26.5.7 0.423 2435 ISO18219 28.5.0 0.70 2435 ISO18219 28.5.0 0.70 2432 ISO18219 28.5.0 0.70 2435 ISO18219 28.5.0 0.70 2435 ISO18219 28.5.0 0.70 2435 ISO18219 28.5.0 0.70 2435 ISO18219 28.5.0 0.70 244 ISO18219 28.5.0 0.70 245 ISO18219 24	2129	ISO18219	285		0.73	
2250     ISO18219     200     -0.91       2310     ISO18219     196.8     -0.97       2311     ISO18219     185     -1.20       2350     ISO18219     218.725     -0.55       2351     ISO18219     218.725     -0.52       2351     ISO18219     210.7     1.23       2355     ISO18219     265     0.33       2370     ISO18219     200     -0.33       2371     ISO18219     203     -0.85       23730     ISO18219     203     -0.85       2382     ISO18219     203.     -0.82       2390     ISO18219     202.5     0.30       2390     ISO18219     202.5     0.30       2390     ISO18219     205.5     -0.92       2508     ISO18219     28.050     R(0.05)     -4.23       2505     IsO18219     28.050     R(0.01)     7.03       2521     ISO18219     26.55     ex     -0.3       2521     ISO18219     24.95     -0.02       2531     IsO18219     24.95     -0.02       2535     1079.957     R(0.01)     6.02       2345     In-house     27.51     -0.02       2345     In-house </td <td>2247</td> <td>ISO18219</td> <td>285.68</td> <td></td> <td>0.74</td> <td></td>	2247	ISO18219	285.68		0.74	
2200       ISO18219       196.8       -0.97         2311       ISO18219       185       -1.20         2330       ISO18219       187.25       -0.55         2332       In-house       248       0.02         2331       ISO18219       210.7       1.23         2353       ISO18219       20.00       -0.33         2336       ISO18219       230.0       -0.43         23370       ISO18219       233.0       -0.45         2336       ISO18219       233.0       -0.45         23300       ISO18219       233.0       -0.45         23300       ISO18219       220.7       -0.51         23301       ISO18219       220.7       -0.51         23301       ISO18219       220.5       0.30         25001       ISO18219       28.050       R(0.05)       -4.23         2605       In-house       262.75       ex       0.30         2501       ISO18219       28.050       R(0.05)       -2.88         2501       ISO18219       28.050       R(0.01)       1.6.88         2502       ISO18219       28.55       0.02         2512       ISO18219	2250	ISO18219	200		-0.91	
2310 I SO18219 168 -0.97 2330 ISO18219 185 -1.20 2330 ISO18219 218.725 -0.55 2335 In-house 248 0.02 2334 ISO18219 266 0.35 2335 ISO18219 259 0.23 2336 ISO18219 260 -0.33 2330 ISO18219 230.0 -0.33 2330 ISO18219 262.5 0.30 2336 ISO18219 262.5 0.30 2338 ISO18219 262.5 0.30 2339 ISO18219 262.5 0.30 2360 ISO18219 262.5 0.30 2361 ISO18219 262.5 0.30 2362 ISO18219 262.5 0.30 2362 ISO18219 263.9551 0.32 2503 ISO18219 263.9551 0.32 2505 In-house 262.725 ex 0.30 2622 ISO18219 28.050 R(0.05) 4.23 2635 In-house 262.725 ex 0.30 2642 ISO18219 28.050 R(0.01) 7.03 2642 ISO18219 28.050 -R(0.05) -2.98 161 In-house 670.722935 R(0.01) 8.18 265 ISO18219 3057 R(0.05) -2.98 265 ISO18219 3057 R(0.05) -2.98 266 In-house 27.5 R(0.01) 9.23 3172 ISO18219 162 -1.64 267 ISO18219 30.07 R(0.05) -2.98 175 ISO18219 289.2 ex 0.81 175 Irst result excluded, used a deviating solvent 175 Iso18219 289.2 ex 0.81 176 In-house 27.5 R(0.01) 9.23 3172 ISO18219 289.2 ex 0.81 178 In-house 226.36 -0.21 178 Iso18219 289.2 ex 0.81 178 Iso18219 286 -0.21 178 Iso18219 289.2 ex 0.81 179 Iso18219 289.2 ex 0.81 178 Iso18219 289 Iso18219 289 Iso1820 178 Iso18219 289 Iso1820 198 Iso1820 178 Is	2267	10010010				
2311       ISO18219       1875       -1.20         2332       ISO18219       218.725       -0.55         2334       ISO18219       210.7       1.23         2357       In-house       256       0.35         2361       ISO18219       270.986       0.43         2375       ISO18219       230.0       -0.33         2380       ISO18219       230.0       -0.65         2376       ISO18219       230.0       -0.65         2376       ISO18219       230.0       -0.65         2380       ISO18219       280.7       -0.65         2380       ISO18219       280.7       -0.65         2380       ISO18219       280.7       -0.92         2508       ISO18219       280.5       R(0.05)       -4.23         2505       ISO18219       280.5       ex       7.31         2521       611       R(0.01)       7.03         2521       5018219       245.95       -0.02         2536       1079.597       R(0.01)       8.18         2541       In-house       275       R(0.01)       8.18         2561       10.70.83       -0.32       -0.3	2310	15018219	196.8		-0.97	
2330     IN-Thouse     248     -0.33       2334     IN-Thouse     248     0.02       2335     IN-Thouse     265     0.35       2336     ISO18219     210.7     1.23       2336     ISO18219     230.0     -0.33       2337     IN-Thouse     283.9551     0.33       2336     ISO18219     233     -0.65       2337     ISO18219     233     -0.65       2339     ISO18219     262.5     0.30       2330     ISO18219     262.5     0.30       2330     ISO18219     280.50     R(0.05)     -4.33       2330     ISO18219     280.50     R(0.05)     -4.33       2330     ISO18219     280.50     R(0.01)     7.03       2505     In-house     262.7     0.30     -0.62       2521     SO18219     280.50     R(0.01)     8.18       2826     ISO18219     285.5     ex     7.31       2521     SO18.19     245.95     R0.01     8.18       2826     ISO18219     245.95     R0.01     8.18       2826     ISO18219     29.2     ex     0.41       2836     In-house     27.31     -0.39       23172 <td>2311</td> <td>15018219</td> <td>185</td> <td></td> <td>-1.20</td> <td></td>	2311	15018219	185		-1.20	
2334 ISO18219 245 0 0.23 2335 ISO18219 259 0.23 2336 ISO18219 270.986 0.46 2336 ISO18219 247 0.00 2337 ISO18219 247 0.00 23380 ISO18219 263.9551 0.33 2330	2350	13010219 In house	210.720		-0.55	
2357     In-house     285     0.35       2361     ISO10219     259     0.23       2365     ISO18219     270.986     0.46       2366     ISO18219     230.0     -0.33       2370     ISO18219     203     -0.85       2379     ISO18219     203     -0.85       2386     ISO18219     220.7     -0.51       2390     ISO18219     220.7     -0.51       2390     ISO18219     220.7     -0.51       2508     ISO18219     280.0     R(0.05)     -4.23       2509     ISO18219     280.0     R(0.05)     -4.23       2501     ISO18219     280.5     R(0.07)     -4.23       2612     611     R(0.01)     7.03       2612     611     R(0.01)     7.03       2612     ISO18219     285     -0.02       2816     In-house     670.722935     R(0.01)       2835     1079.597     R(0.01)     8.18       2846     In-house     225.7     8.33       154     ISO18219     28.3     0.70       290     In-house     27.31     -0.38       2172     ISO18219     28.3     0.77       2186     In-house <td>2357</td> <td></td> <td>240</td> <td></td> <td>1.02</td> <td></td>	2357		240		1.02	
2333     15 010219     259     0.23       2336     15 010219     270.986     0.46       2336     15 010219     230.0     -0.33       2337     15 010219     203     -0.85       2338     15 010219     203     -0.85       2339     15 010219     203     -0.85       2330     15 010219     203     -0.85       2330         2330     15 010219     220.7     -0.51       2336     15 018219     220.7     -0.51       2336     15 018219     203.9     1.10       2508     15 018219     303.9     1.10       2509     15 018219     303.9     1.01       2501     15 018219     303.9     1.01       2611     R (0.01)     7.03       2621     15 018219     365     2.28       2636     10 79.597     R (0.01)     16.08       3154     15 018219     283.3     0.70       212     10 rhouse     225.7     -0.02       2335     10 79.597     R (0.01)     9.28       212     10 rhouse     27.51     -0.22       2131     1-house     28.36     -0.21       154     15 018	2357		265		0.35	
2365     ISO 18219     270 986     0.46       2366     ISO 18219     230.0     -0.33       2370     ISO 18219     203     -0.85       2379     ISO 18219     203     -0.85       2380     ISO 18219     203     -0.85       2380     ISO 18219     203     -0.85       2380     ISO 18219     220.7     -0.51       2390     ISO 18219     225.5     0.30       2508     ISO 18219     28.50     R(0.05)     -4.23       2500     ISO 18219     28.050     R(0.05)     -4.23       2605     In-house     262.725     ex     0.30       2612     In-house     260.722935     R(0.01)     7.03       2625     ISO 18219     36.5     .2.28     2.85       2616     In-house     670.722935     R(0.01)     8.16       2835     1079.597     R(0.01)     9.23       3163     In-house     275.73     .0.02       2846     In-house     287.73     .0.38       3172     ISO 18219     28.0     .0.71       3171     In-house     28.3     .0.70       3233     In-house     195.7     .0.21       3233     In-house	2363	ISO18219	203		0.33	
2366     ISO18219     230.0     -0.33       2370     ISO18219     247     0.00       2375     ISO18219     263.9551     0.33       2380	2365	ISO18219	270 986		0.20	
2370 2375       ISO 18219 1SO 18219       247 203       0.00 0.00 0.00 2375         2376       ISO 18219       203       -0.85 0.30 2386         2380       ISO 18219       282.5       0.30 0.05         2380       ISO 18219       282.5       0.30 0.05         2390       ISO 18219       282.5       0.30 0.05         2508       ISO 18219       28.55       ex       7.31 1.00         2509       ISO 18219       28.05       R(0.05)       4.23         2605       In-house       28.2725       ex       0.30 2.28         2612       611       R(0.01)       7.03         2622       ISO 18219       365       2.28         2816       In-house       670 722935       R(0.01)       8.18         2826       ISO 18219       245.95       -0.02         2836       In-house       72.5       R(0.01)       9.23         3154       ISO 18219       93.07       R(0.05)       -2.96         3153       In-house       27.731       -0.33         3172       ISO 18219       28.2       ex       0.81         3230       In-house       27.57.018       -1.74         7       <	2366	ISO18219	230.0		-0.33	
2375       ISO 18219       203       -0.85         2379       ISO 18219       263.9551       0.33         2386       ISO 18219       262.5       0.30         2386       ISO 18219       262.5       0.30         2386       ISO 18219       202.7       -0.51         2390       ISO 18219       203.95       1.00         2508       ISO 18219       280.50       R(0.05)       -4.23         2505       IsO 18219       28.050       R(0.01)       7.03         2621       ISO 18219       26.55       ex       0.30         2621       611       R(0.01)       7.03       Test result excluded, used a deviating solvent         2621       ISO 18219       245.95       -0.02       -0.22         2836       In-house       670.722935       R(0.01)       8.16         2864       In-house       72.5       R(0.01)       9.23         31317       In-house       227.31       -0.38         2101       In-house       227.31       -0.39         2101       In-house       195.7       -0.99         3233       In-house       195.7       -0.21         nortilers       6	2370	ISO18219	247		0.00	
2379     ISO18219     263.9551     0.33       2380         2382     ISO18219     262.5     0.30       2390     ISO18219     220.7     -0.51       2508     ISO18219     255.8     ex     7.31       2509     ISO18219     262.5     0.30       2508     ISO18219     280.50     R(0.05)     -4.23       2605     In-house     262.725     ex     0.30       2612     611     R(0.01)     7.03       2626     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)     8.18       2844     In-house     725     R(0.01)     9.23       3154     ISO18219     93.07     R(0.05)     -2.98       3172     ISO18219     289.2     -0.32       211     In-house     227.31     -0.38       3120     In-house     217.018     -1.74       3231     In-house     236.36     -0.21       3232     In-house     236.36     -0.21       333     outiers     6 (4-4xcl) mean (n)     247.	2375	ISO18219	203		-0.85	
2380	2379	ISO18219	263.9551		0.33	
2382       ISO18219       282.5       0.30         2386       ISO18219       220.7       -0.51         2300       ISO18219       625.58       ex       7.31       Test result excluded, used a deviating solvent         2563       ISO18219       28.050       R(0.05)       -4.23         2605       In-house       28.725       ex       0.30       Test result excluded, used a deviating solvent         2612       611       R(0.01)       7.03       Test result excluded, used a deviating solvent         2612       In-house       670.722935       R(0.01)       8.18         2826       IsO18219       245.95       -0.02         2835       1079.597       R(0.01)       9.18         2836       In-house       424.75          3154       ISO18219       93.07       R(0.05)       -2.98         3154       ISO18219       162       -1.64         3172       ISO18219       289.2       ex       0.81         22810       In-house       157.018       -0.21         22810       In-house       157.018       -0.21         22810       In-house       236.36       -0.21         3233 <t< td=""><td>2380</td><td></td><td></td><td></td><td></td><td></td></t<>	2380					
2386     ISO18219     220.7     -0.51       2390     ISO18219     199.5     -0.92       2508     ISO18219     025.58     ex     7.31       2509     ISO18219     28.050     R(0.05)     -4.23       2605     In-house     282.725     ex     0.30       2612     611     R(0.01)     7.03       2621     ISO18219     365     2.28       2622     ISO18219     365     2.28       2826     ISO18219     245.95     -0.02       2836     In-house     670.722935     R(0.01)     8.18       2826     ISO18219     245.95     -0.02       2835     1079.597     R(0.01)     16.08       2846     In-house     725     R(0.01)     -2.98       2816     ISO18219     93.07     R(0.05)     -2.98       3153     In-house     127.018     -1.64       3197     In-house     127.018     -1.74       3210     In-house     157.018     -1.74       3228     In-house     195.7     -0.99       3233     In-house     236.36     -0.21       normality     suspect     -0.81     -0.21       n     106.339     51.7602	2382	ISO18219	262.5		0.30	
2390     ISO18219     199.5     -0.92       2508     ISO18219     625.58     ex     7.31     Test result excluded, used a deviating solvent       2509     ISO18219     28.050     R(0.05)     -4.23       2605     In-house     262.725     ex     0.30       2612     611     R(0.01)     7.03       2621     SO18219     28.55     rest result excluded, used a deviating solvent       2612     611     R(0.01)     7.03       2621     IsO18219     245.95     -0.02       2835     In-house     670.722935     R(0.01)     16.8       28264     In-house     424.75     3.43       2836     In-house     725     R(0.01)     9.38       2844     In-house     725     R(0.01)     9.33       3172     ISO18219     93.07     R(0.05)     -2.98       3183     IsO18219     28.3     0.70       3200     In-house     227.31     -0.38       3210     In-house     195.7     -0.99       3233     In-house     195.7     -0.99       3234     IsO18219     28.92     ex     0.21       normality     suspect     n     33     0.21       m	2386	ISO18219	220.7		-0.51	
2500     ISO18219     625.58     ex     7.31     Test result excluded, used a deviating solvent       2563     ISO18219     28.050     R(0.05)     -4.23       2605     In-house     262.725     ex     0.30       2612     611     R(0.01)     7.03       2626     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     727     R(0.05)     -2.98       3154     ISO18219     93.07     R(0.05)     -2.98       3153     In-house     725     R(0.01)     9.23       3163     In-house     17.01     -1.64       3210     In-house     157.018     -1.74       3224     ISO18219     289.2     ex     0.81       3214     ISO18219     289.2     ex     0.81       3228     In-house     157.018     -0.21       3233     In-house     236.36     -0.21       3234     In-house     236.36     -0.21       3233     In-house     157.018     -1.74       3233     In-house <td>2390</td> <td>ISO18219</td> <td>199.5</td> <td></td> <td>-0.92</td> <td></td>	2390	ISO18219	199.5		-0.92	
2563 ISO18219 303.9 1.10 2590 ISO18219 28.050 R(0.05) -4.23 2605 In-house 262.725 ex 0.30 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 2846	2508	ISO18219	625.58	ex	7.31	Test result excluded, used a deviating solvent
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	2563	ISO18219	303.9		1.10	
2605 In-house 262.725 ex 0.30 Test result excluded, used a deviating solvent 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 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 157.018 -1.74 3214 ISO18219 289.2 ex 0.81 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. (horwitz n=9) 51.7602 R(Horwitz n=9) 144.929	2590	ISO18219	28.050	R(0.05)	-4.23	
$2612 \\ 2622 \\ SO18219 \\ 365 \\ 2835 \\ 1079.597 \\ R(0.01) \\ 8.18 \\ -0.02 \\ 2835 \\ 1079.597 \\ R(0.01) \\ 16.08 \\ 2864 \\ In-house \\ 424.75 \\ 3.43 \\ 2886 \\ $	2605	In-house	262.725	ex	0.30	Test result excluded, used a deviating solvent
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	2612		611	R(0.01)	7.03	
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	2622	ISO18219	365		2.28	
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	2816	In-house	670.722935	R(0.01)	8.18	
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	2826	15018219	245.95	D(0.04)	-0.02	
$\frac{2806}{3164} = \frac{424, 13}{1000} = \frac{424, 13}{1000} = \frac{1000}{1000} = \frac{10000}{1000} = \frac{1000}{1000} = 100$	2835	In house	1079.597	R(0.01)	16.08	
$\frac{2000}{3154}  SO18219 = 93.07 \\ R(0.05) = 2.98 \\ 3163 \\ In-house = 725 \\ R(0.01) = 9.23 \\ 3172 \\ ISO18219 = 162 \\ 1.64 \\ 3197 \\ In-house = 283.3 \\ 0.70 \\ 3209 \\ In-house = 237.31 \\ 3210 \\ In-house = 157.018 \\ 1.74 \\ 3214 \\ ISO18219 \\ 289.2 \\ ex \\ 0.81 \\ Test result excluded, used a deviating solvent \\ 3228 \\ In-house = 195.7 \\ 0.99 \\ 3233 \\ In-house = 236.36 \\ 0.21 \\ normality \\ n = 33 \\ outliers \\ n = 33 \\ outliers \\ n = 6 \\ (+4excl) \\ mean (n) \\ 247.130 \\ st.dev. (n) \\ 59.4068 \\ R(calc.) \\ 166.339 \\ st.dev. (horwitz n=9) \\ 144.929 \\ R(Horwitz n=9) \\ 144.929 \\ R(Horwitz n=9) \\ R(Horwitz n=9$	2004	III-HOUSE	424.75		3.43	
$\frac{3163}{10} = 10000215 + 10000215 + 10000215 + 10000000000000000000000000000000000$	2000	19018210	03.07	P(0.05)	-2.08	
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	3163		725	R(0.03)	Q 23	
$ \frac{3197}{3209}   n-house \\ 283.3 \\ 3210   n-house \\ 157.018 \\ 1.74 \\ 3214   SO18219 \\ 3228   n-house \\ 195.7 \\ 3233   n-house \\ 236.36 \\ 3233   n-house \\ 236.36 \\ 3233   n-house \\ 236.36 \\ 333 \\ outliers \\ n \\ st.dev. (n) \\ st.dev. (n) \\ st.dev. (n) \\ st.dev. (horwitz n=9) \\ 144.929 \\ 80 \\ 50 \\ 50 \\ 50 \\ 50 \\ 50 \\ 50 \\ 50$	3172	ISO18219	162	1((0.01)	-1 64	
$\frac{3209}{3210} = \frac{11-1000}{11-10000} = \frac{227.31}{157.018} = \frac{-0.38}{-1.74}$ $\frac{3214}{3214} = \frac{15018219}{15018219} = \frac{289.2}{195.7} = \frac{10.08}{-0.999}$ $\frac{3233}{3233} = \frac{11-10000}{11-100000} = \frac{236.36}{-0.21}$ $\frac{11-10000}{10000000000000000000000000000$	3197	In-house	283.3		0.70	
$\frac{3210}{3210}   n-house = \frac{157.018}{157.018} - \frac{1.74}{1.74}$ $\frac{3214}{3214}   SO18219 = 289.2  ex \qquad 0.81  \text{Test result excluded, used a deviating solvent}$ $\frac{3228}{3233}   n-house = \frac{195.7}{2333} - \frac{0.99}{2333} = \frac{10.21}{1000}$ $\frac{1000}{1000} = \frac{1000}{1000} = \frac{1000}{100$	3209	In-house	227.31		-0.38	
3214 ISO 18219 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 $800 = 1000 Kernel Density Kernel Density 0.000 Kernel Density 0.000$	3210	In-house	157.018		-1.74	
$3228  \text{In-house} \qquad 195.7 \qquad -0.99 \\ 3233  \text{In-house} \qquad 236.36 \qquad -0.21 \\ \text{normality} \qquad \text{suspect} \\ n \qquad 33 \\ \text{outliers} \qquad 6  (+4\text{excl}) \\ \text{mean (n)} \qquad 247.130 \\ \text{st.dev. (n)} \qquad 59.4068  \text{RSD} = 24\% \\ \text{R(calc.)} \qquad 166.339 \\ \text{st.dev.(Horwitz n=9)}  51.7602 \\ \text{R(Horwitz n=9)} \qquad 144.929 \\ \text{Kernel Density} \\ \text{sol} \qquad x  x  x  x  x  x  x  x  x  x$	3214	ISO18219	289.2	ex	0.81	Test result excluded, used a deviating solvent
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 R(Horwitz n=9) 144.929	3228	In-house	195.7		-0.99	
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	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 R(Horwitz n=9) 144.929						
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 Kemel Density		normality	suspect			
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 $x \times x \times$		n	33			
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 Remei Density		outliers	6 (+4excl)			
$\begin{array}{c} \text{st.dev. (n)} & 59.4068 \\ \text{R(calc.)} & 166.339 \\ \text{st.dev. (Horwitz n=9)} & 51.7602 \\ \text{R(Horwitz n=9)} & 144.929 \end{array}$		mean (n)	247.130			
R(calc.) = 166.339 st.dev.(Horwitz n=9) 51.7602 R(Horwitz n=9) 144.929		st.dev. (n)	59.4068	RSD = 24%	%	
800 - x x 0.000 - 0.00		R(Calc.)	166.339			
R(HOIWIZ II=9) 144.929		St.dev.(Horwitz n=9)	51.7602			
800 - x x 0.008 - Kernel Density 600 - x x 0.006 - 0.006 - 0.006 - 0.007 - 0.006 - 0.			144.929			
800     x     0.008       700     x     0.007       600     x     x       0.006     -						
700 - x x 0.007 - 0.006 - 0.00	<sup>800</sup>					x Kernel Density
600 - × × 0.006 -	700 -					x 0.007 -
	600 -					× * 0.006 -



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

lab	method	value	mark	z(targ)	remarks
110					
339	10010010				
623	15018219	552.200		-1.02	
840 841	ISO10219	835		-0.02	
2118	ISO18219	591.887		-0.69	
2129	ISO18219	782		0.87	
2247	ISO18219	734.44		0.48	
2250	ISO18219	563		-0.93	
2267	10040040				
2310	ISO18219 ISO18210	701.7		0.21	
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	
2300	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	-0.94	First was acted 4077.0
2390	ISO18219 ISO18210	565.4 1365.61		-0.91	FIRST reported 1077.3
2563	ISO18219	674.6	ex	-0.01	rest result excluded, used a deviating solvent
2590	ISO18219	303.813	R(0.05)	-3.06	
2605					
2612		508		-1.38	
2622	In house	1022 275994		2.96	
2826	ISO18219	808.53		1.09	
2835					
2864					
2886	10010010				
3154	ISO18219	251.85	R(0.05)	-3.49	
3103	IN-NOUSE	750 534		0.61	
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	R5D = 19%	0	
	st dev (Horwitz n=9)	121 6675			
	R(Horwitz n=9)	340.669			
	. ,				
<sup>1600</sup> T					0.0035 1
1400 -					x 0.003 - ∧ Kernel Density



#### APPENDIX 2 Analytical details

Analy	rical actails						
	ISO/IEC17025	sample preparation	final particle	sample		extraction	extraction
lab	accredited	before use	size (mm)	intake (g)	extraction solvent	time (min)	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 2 mm	0.5	Toluene	60	60
2110	No		2 × 5 mm	0.5	Toluono	60	60
2129	Yes	Used as received		0.5		60	60
2247	res		<2 11111	0.3		60	60
2250	Yes	Used as received	2 mm	0.5	Ioluene	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	$0.5 \times 0.5 \text{ cm}$	15	Toluene	60	60
2375	Vec	Further Cut	2 v 2 mm	0.5	Toluene	60	60
2010	No	Further Cut	2 x 2 mm	0.5	Toluono	60	60
23/9	INU		2 X Z IIIIII	0.5	Toluelle	00	00
2300	 \/		 00	 0	 Taluara		
2302	Yes			0.5	Toluene	60	60
2386	Yes	Used as received			Toluene	60	60
2390	Yes	Further Cut	<2 mm	0.5	Ioluene	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	lised as received		0.5	Toluene	60	60
3163	No	Eurther Cut	2 mm	0.0	Toluene	60	60
3172	Vec	Further Cut	2 v 3 mm	2	Hevane	60	60
2107	Vee		5 x 5 mm	0.25		60	60
319/	105 Voo		5 X 5 11111	0.23		60	60
3209	Tes			0.5		00	00
3210	res	Used as received		0.5	TUERA	00	бU 70
3214	Yes	Further Cut	0.5 x 0.5 mm	0.5		60	70
3228	Yes	Used as received	0.5 x 0.5 cm	0.5	I oluene - 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

С	= 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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