Institute for
Interlaboratory Studies

# Results of Proficiency Test
# SCCP/MCCP in Leather/Footwear
# April 2023


**Organized by:** Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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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 and MCCP in Leather/Footwear every year. During the annual proficiency testing program 2022/2023 it was decided to continue the proficiency test for the determination of SCCP/MCCP in Leather/Footwear.

In this interlaboratory study 46 laboratories in 16 countries registered for participation, see appendix 3 for the number of participants per country. In this report the results of the SCCP/MCCP 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 labelled #23540.
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 brown colored leather positive on SCCP and MCCP was selected. The leather was grinded and mixed thoroughly. After homogenization 65 small plastic bags were filled with approximately 3 grams each and labelled #23540.
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 #23540-1 | 242.4 |
| sample #23540-2 | 237.3 |
| sample #23540-3 | 232.7 |
| sample #23540-4 | 246.2 |
| sample #23540-5 | 237.7 |
| sample #23540-6 | 240.2 |
| sample #23540-7 | 230.6 |
| sample #23540-8 | 241.6 |
| sample #23540-9 | 238.9 |
| sample #23540-10 | 239.0 |

Table 1: homogeneity test results of subsamples #23540

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) | 12.7 |
| reference method | Horwitz (n=9) |
| 0.3 x R (reference method) | 42.2 |

Table 2: evaluation of the repeatability of subsamples #23540

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 leather sample labelled #23540 was sent on March 8, 2023.

### 2.5 ANALYZES

The participants were requested to determine SCCP and MCCP. To ensure homogeneity it was requested not to use less than 0.5 grams of the sample per determination. It was also requested to report if the laboratory was accredited to determine the reported component and to report some analytical details.

It was explicitly requested to treat the sample as if it was a routine sample but not to age nor to dry the sample nor to determine volatile matter. The amount of sample was not sufficient to allow aging and/or determine the volatile matter content.

Furthermore, it was also requested 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 (derived from e.g. ISO or ASTM test methods), 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:

| | | | |
|---|---|---|---|
| | $\lvert z \rvert$ | < 1 | good |
| 1 < | $\lvert z \rvert$ | < 2 | satisfactory |
| 2 < | $\lvert z \rvert$ | < 3 | questionable |
| 3 < | $\lvert z \rvert$ | | unsatisfactory |

## 4 EVALUATION

In this proficiency test no problems were encountered with the dispatch of the samples. All participants were able to report test results. Five of the participants reported test results after the final reporting date. Not all participants were able to report all tests requested.
In total 46 participants reported 90 numerical test results. Observed were 3 outlying test results, which is 3.3%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.
All data sets proved to have a normal Gaussian distribution.

### 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 test method ISO18219 is considered to be the official test method. The latest version of this method was published in 2021, however the determination of SCCP and MCCP has been published in two separate parts. Part 1 describes the determination of SCCP and part 2 the determination of MCCP, more detail is given in paragraph 5.
Regretfully, also the 2021 versions of test method ISO18219 do not describe 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 was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated reproducibility calculated with the Horwitz equation based on nine components.

MCCP: This determination may be problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated reproducibility calculated with the Horwitz equation based on nine components.

### 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 | 45 | 254.8 | 142.8 | 148.8 |
| MCCP | mg/kg | 42 | 1095 | 609 | 513 |

Table 3: reproducibilities of tests on sample #23540

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

### 4.3 COMPARISON OF THE PROFICIENCY TEST OF APRIL 2023 WITH PREVIOUS PTS

| | April 2023 | March 2022 | February 2021 | April 2020 | March 2019 |
|---|---|---|---|---|---|
| Number of reporting laboratories | 46 | 47 | 46 | 53 | 54 |
| Number of test results | 90 | 86 | 82 | 102 | 99 |
| Number of statistical outliers | 3 | 3 | 6 | 7 | 2 |
| Percentage of statistical outliers | 3.3% | 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 to uncertainties observed in PTs over the years, expressed as relative standard deviation (RSD) of the PTs, see next table.

| Component | April 2023 | March 2022 | February 2021 | April 2020 | March 2019 | Target *) |
|---|---|---|---|---|---|---|
| SCCP | 20% | 25% | 16% | 24% | 31% | 17-24% |
| MCCP | 20% | 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

About 90% of the participants reported to use test method ISO18219 version 2021 for determining SCCP/MCCP. One participant reported to use ISO18219 version 2015.
For this PT some analytical details were requested which are listed in appendix 2. Based on the answers given by the participants the following can be summarized:

- About 85% of the 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 15% of the reporting participants and about 85% used the samples as received.
- The amount of sample intake varied between 0.2 and 1 grams. About 85% of the participants reported to use 0.5 grams.
- About 85% of the reporting participants used n-Hexane as release solvent.
- 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 versions of test method ISO18219 was published. ISO18219 part 1 describes the determination of SCCP and ISO18219 part 2 the determination of MCCP. Compared to the 2015 version a change is made in the clean-up step. Both parts of the 2021 versions of test method ISO18219 describe the use of a mixture of n-Hexane and Sulfuric Acid with liquid phase separation while the 2015 version of SO18219 describes the use of a mixture of n-Hexane and Dichloromethane and solid phase separation (SPE cartridge).

When the results of this interlaboratory study were compared to the Leather Standard by OEKO-TEX® (see table 6) it was noticed that all participants would have made identical decisions about the acceptability of the leather for the determined components and would have rejected the sample for all categories.

| Components | baby clothes in mg/kg | in direct skin contact in kg/mg | no direct skin contact in mg/kg |
|---|---|---|---|
| Sum SCCP and MCCP | <50 | <50 | <50 |

Table 6: Leather Standard by OEKO-TEX®

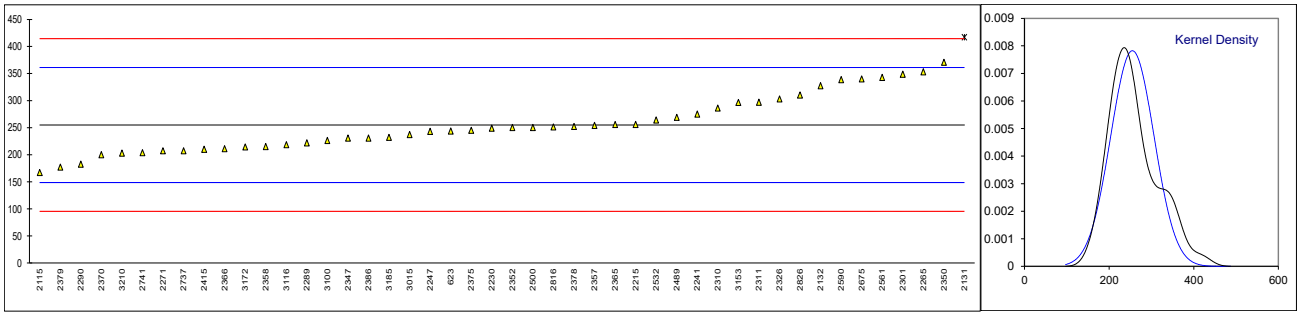
## 6 CONCLUSION

Allmost all of the participants are able to determine SCCP and MCCP in the leather matrix. The observed reproducibilities in this proficiency test on SCCP and MCCP 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

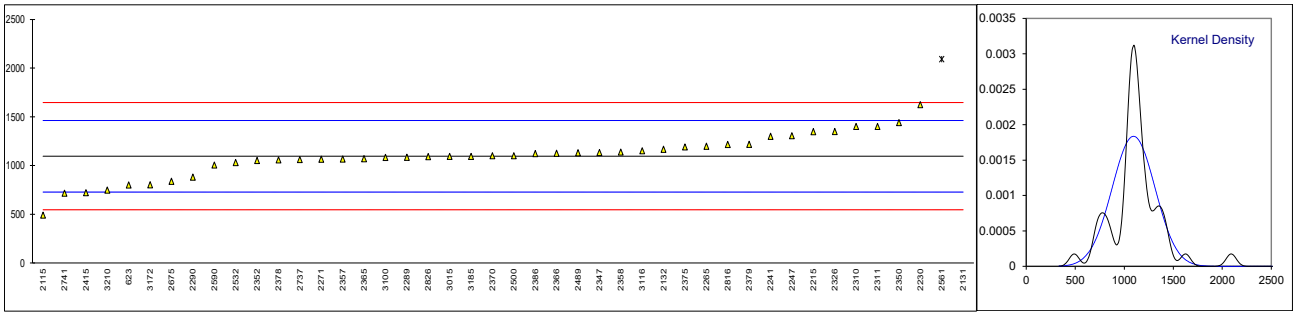
Determination of SCCP on sample #23540; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 623 | ISO18219-1:2021 | 243.64 | | -0.21 | |
| 2115 | ISO18219-1:2021 | 167.1 | | -1.65 | |
| 2131 | In house | 417 | R(0.05) | 3.05 | |
| 2132 | ISO18219-1:2021 | 327.44 | | 1.37 | |
| 2215 | ISO18219-1:2021 | 255.98 | | 0.02 | |
| 2230 | ISO18219-1:2021 | 249 | | -0.11 | |
| 2241 | ISO18219-1:2021 | 275.08 | | 0.38 | |
| 2247 | ISO18219-1:2021 | 243.03 | | -0.22 | |
| 2265 | ISO18219-1:2021 | 353.0 | | 1.85 | |
| 2271 | ISO18219-1:2021 | 207.1 | | -0.90 | |
| 2289 | ISO18219-1:2021 | 222 | | -0.62 | |
| 2290 | ISO18219-1:2021 | 182.5 | | -1.36 | |
| 2301 | ISO18219-1:2021 | 348.55 | | 1.76 | |
| 2310 | ISO18219:2015 | 286 | | 0.59 | |
| 2311 | ISO18219-1:2021 | 297.1 | | 0.80 | |
| 2326 | ISO22818:2021 | 303 | C | 0.91 | First reported 342.4 |
| 2347 | ISO18219-1:2021 | 230.5 | | -0.46 | |
| 2350 | ISO18219-1:2021 | 370.8 | | 2.18 | |
| 2352 | GB/T38405:2019 | 250.2 | | -0.09 | |
| 2357 | ISO18219-1:2021 | 254.0 | | -0.02 | |
| 2358 | ISO18219-1:2021 | 215.25 | | -0.75 | |
| 2365 | ISO18219-1:2021 | 255.6 | | 0.01 | |
| 2366 | ISO18219-1:2021 | 211 | | -0.83 | |
| 2370 | ISO18219-1:2021 | 200 | | -1.03 | |
| 2375 | ISO18219-1:2021 | 245 | | -0.19 | |
| 2378 | ISO18219-1:2021 | 252 | | -0.05 | |
| 2379 | ISO18219-1:2021 | 177.01 | | -1.46 | |
| 2386 | ISO18219-1:2021 | 230.6 | | -0.46 | |
| 2415 | ISO18219-1:2021 | 209.84 | | -0.85 | |
| 2489 | ISO18219-1:2021 | 269 | | 0.27 | |
| 2500 | ISO18219-1:2021 | 250.23 | | -0.09 | |
| 2532 | ISO18219-1:2021 | 264 | | 0.17 | |
| 2561 | ISO18219-1:2021 | 342.8033 | | 1.66 | |
| 2590 | ISO18219-1:2021 | 338.55 | | 1.58 | |
| 2675 | ISO18219-1:2021 | 340.064 | | 1.60 | |
| 2737 | ISO18219-1:2021 | 207.23 | | -0.90 | |
| 2741 | ISO18219-1:2021 | 203.81 | | -0.96 | |
| 2816 | ISO18219-1:2021 | 251.4 | C | -0.06 | First reported 405.758 |
| 2826 | ISO18219-1:2021 | 310.3454 | | 1.04 | |
| 3015 | ISO18219-1:2021 | 237.27 | | -0.33 | |
| 3100 | ISO18219-1:2021 | 226.36 | | -0.54 | |
| 3116 | ISO18219-1:2021 | 218.47 | | -0.68 | |
| 3153 | ISO18219-1:2021 | 296.4 | | 0.78 | |
| 3172 | ISO18219-1:2021 | 214.2 | | -0.76 | |
| 3185 | ISO18219-1:2021 | 232.11 | | -0.43 | |
| 3210 | ISO22818 | 202.87 | | -0.98 | |
| | | | | | |
| | normality | OK | | | |
| | n | 45 | | | |
| | outliers | 1 | | | |
| | mean (n) | 254.832 | | | |
| | st.dev. (n) | 50.9930 | RSD = 20% | | |
| | R(calc.) | 142.780 | | | |
| | st.dev.(Horwitz n=9) | 53.1273 | | | |
| | R(Horwitz n=9) | 148.756 | | | |



## Determination of MCCP on sample #23540; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 623 | ISO18219-2:2021 | 800.36 | | -1.61 | |
| 2115 | ISO18219-2:2021 | 490.5 | | -3.30 | |
| 2131 | In house | 5190 | R(0.01) | 22.33 | |
| 2132 | ISO18219-2:2021 | 1166.75 | | 0.39 | |
| 2215 | ISO18219-2:2021 | 1347.58 | | 1.38 | |
| 2230 | ISO18219-2:2021 | 1625 | | 2.89 | |
| 2241 | ISO18219-2:2021 | 1298.67 | | 1.11 | |
| 2247 | ISO18219-2:2021 | 1305.40 | | 1.15 | |
| 2265 | ISO18219-2:2021 | 1197.71 | | 0.56 | |
| 2271 | ISO18219-2:2021 | 1065.2 | | -0.16 | |
| 2289 | ISO18219-2:2021 | 1085 | | -0.06 | |
| 2290 | ISO18219-2:2021 | 880.3 | | -1.17 | |
| 2301 | | ----- | | ----- | |
| 2310 | ISO18219:2015 | 1400 | | 1.66 | |
| 2311 | ISO18219-2:2021 | 1400.2 | | 1.66 | |
| 2326 | ISO22818:2021 | 1348.7 | C | 1.38 | First reported 1713.0 |
| 2347 | ISO18219-2:2021 | 1132.9 | | 0.21 | |
| 2350 | ISO18219-2:2021 | 1440.3 | | 1.88 | |
| 2352 | ISO18219-2:2021 | 1051.0 | | -0.24 | |
| 2357 | ISO18219-2:2021 | 1066.0 | | -0.16 | |
| 2358 | ISO18219-2:2021 | 1137.75 | | 0.23 | |
| 2365 | ISO18219-2:2021 | 1069.1 | | -0.14 | |
| 2366 | ISO18219-2:2021 | 1126 | | 0.17 | |
| 2370 | ISO18219-2:2021 | 1100 | | 0.03 | |
| 2375 | ISO18219-2:2021 | 1190 | | 0.52 | |
| 2378 | ISO18219-2:2021 | 1058 | | -0.20 | |
| 2379 | ISO18219-2:2021 | 1217.21 | | 0.67 | |
| 2386 | ISO18219-2:2021 | 1122.9 | | 0.15 | |
| 2415 | ISO18219-2:2021 | 720.400 | | -2.04 | |
| 2489 | ISO18219-2:2021 | 1130 | | 0.19 | |
| 2500 | ISO18219-2:2021 | 1100.31 | | 0.03 | |
| 2532 | ISO18219-2:2021 | 1030 | | -0.36 | |
| 2561 | ISO18219-2:2021 | 2091.113 | R(0.01) | 5.43 | |
| 2590 | ISO18219-2:2021 | 1004.33 | | -0.50 | |
| 2675 | ISO18219-2:2021 | 839.085 | | -1.40 | |
| 2737 | ISO18219-2:2021 | 1061.34 | | -0.19 | |
| 2741 | ISO18219-2:2021 | 715.46 | | -2.07 | |
| 2816 | ISO18219-2:2021 | 1216.0 | C | 0.66 | First reported 1719.53 |
| 2826 | ISO18219-2:2021 | 1090.2523 | | -0.03 | |
| 3015 | ISO18219-2:2021 | 1093.16 | | -0.01 | |
| 3100 | ISO18219-2:2021 | 1083.24 | | -0.07 | |
| 3116 | ISO18219-2:2021 | 1151.18 | | 0.30 | |
| 3153 | | ----- | | ----- | |
| 3172 | ISO18219-2:2021 | 801.85 | | -1.60 | |
| 3185 | ISO18219-2:2021 | 1094.56 | | 0.00 | |
| 3210 | ISO22818 | 747.56 | | -1.90 | |
| | | | | | |
| | normality | OK | | | |
| | n | 42 | | | |
| | outliers | 2 | | | |
| | mean (n) | 1095.268 | | | |
| | st.dev. (n) | 217.4321 | RSD = 20% | | |
| | R(calc.) | 608.810 | | | |
| | st.dev.(Horwitz n=9) | 183.3447 | | | |
| | R(Horwitz n=9) | 513.365 | | | |



## **APPENDIX 2** Analytical details

| lab | ISO17025 accredited | sample grinded or cut | intake (g) | release solvent | extraction time (min) | extraction temp (°C) |
|---|---|---|---|---|---|---|
| 623 | Yes | Further cut | 0.5 | n-Hexane | 60 | 60 |
| 2115 | No | Used as received | 0.5 | n-Hexane | 60 | 60 |
| 2131 | --- | Used as received | 1 | n-Hexane | 60 | 60 |
| 2132 | Yes | Used as received | 0.5 | n-Hexane | 60 | 60 |
| 2215 | Yes | Used as received | 0.5 | n-Hexane | 60 | 60 |
| 2230 | Yes | Used as received | 0.4925 | -- | 60 | 60 |
| 2241 | Yes | Used as received | 0.5 | n-Hexane | 60 | 60 |
| 2247 | Yes | Used as received | 0.5 | n-Hexane | 60 | 60 |
| 2265 | No | Used as received | 0,5 | n-Hexane | 60 | 60 |
| 2271 | Yes | Used as received | 0.5 | n-Hexane | 60 | 60 |
| 2289 | Yes | Further cut | 0.5 | n-Hexane | 60 | 60 |
| 2290 | Yes | --- | | | | |
| 2301 | --- | --- | | | | |
| 2310 | Yes | Further cut | 0.5 | n-Hexane | 60 | 60 |
| 2311 | Yes | Further cut | 0.5 | n-Hexane | 60 | 60 |
| 2326 | Yes | Used as received | 0.5 | n-Hexane | 60 | 60 |
| 2347 | Yes | Used as received | 0.25 | n-Hexane | 60 | 60 |
| 2350 | Yes | Used as received | 0.5 | n-Hexane | 60 | 60 |
| 2352 | Yes | Used as received | 0.5 | n-Hexane | 60 | 60 |
| 2357 | --- | --- | | | | |
| 2358 | No | Used as received | 0.5 | n-Hexane | 60 | 60 |
| 2365 | Yes | Further cut | 0.5 | n-Hexane | 60 | 60 |
| 2366 | Yes | Further cut | 0.5 | Toluene | | |
| 2370 | Yes | Used as received | 1 | n-Hexane | 60 | 60 |
| 2375 | Yes | Used as received | 0.5 | n-Hexane | 60 | 60 |
| 2378 | Yes | Used as received | 0.5 | n-Hexane | 60 | 60 |
| 2379 | No | Further grinded | 0.5 | n-Hexane | 60 | 60 |
| 2386 | Yes | Used as received | 0.5 | Toluene | 60 | 60 |
| 2415 | Yes | Used as received | 0.5 | n-Hexane | 60 | 60 |
| 2489 | Yes | Used as received | 0.5026 | n-Hexane | 60 | 60 |
| 2500 | Yes | Used as received | 0.5 | n-Hexane | 60 | 60 |
| 2532 | Yes | Used as received | 0.5 | n-Hexane | 60 | 60 |
| 2561 | No | Used as received | 1 | n-Hexane | 60 | 60 |
| 2590 | --- | --- | | | | |
| 2675 | No | Used as received | 0.5 | n-Hexane | 60 | 60 |
| 2737 | Yes | Used as received | 0.5 | n-Hexane | 60 | 60 |
| 2741 | Yes | Used as received | 0.5 | n-Hexane | 60 | 60 |
| 2816 | Yes | Used as received | 0.5 | n-Hexane | 60 | 60 |
| 2826 | Yes | Used as received | 0.5002 | Toluene | 60 | 60 |
| 3015 | Yes | Used as received | 0.5 | n-Hexane | 60 | 60 |
| 3100 | Yes | Used as received | >0.5 | n-Hexane | 60 | 60 |
| 3116 | Yes | Used as received | 1 | n-Hexane | 60 | 60 |
| 3153 | Yes | Used as received | 0.5 | n-Hexane | 60 | 60 |
| 3172 | Yes | --- | | | | |
| 3185 | Yes | Used as received | 0.5 | n-Hexane | 60 | 60 |
| 3210 | Yes | Used as received | 1 | Toluene *) | 60 | 60 |

*) Sulfuric acid clean-up made

## APPENDIX 3

### Number of participants per country

| | |
|---|---|
| 1 lab | in DENMARK |
| 1 lab | in FRANCE |
| 3 labs | in GERMANY |
| 5 labs | in HONG KONG |
| 5 labs | in INDIA |
| 2 labs | in INDONESIA |
| 3 labs | in ITALY |
| 1 lab | in KOREA, Republic of |
| 15 labs | in P.R. of CHINA |
| 1 lab | in PAKISTAN |
| 1 lab | in SWITZERLAND |
| 2 labs | in TAIWAN |
| 1 lab | in THAILAND |
| 1 lab | in TURKEY |
| 1 lab | in UNITED KINGDOM |
| 3 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 |

### Literature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