Institute for
Interlaboratory Studies

# Results of Proficiency Test
# SCCP/MCCP in Textile
# October 2022


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

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# 1 INTRODUCTION

Commercially produced Chlorinated Paraffins (CPs) are classified according to their carbon chain length into Short Chain CPs (SCCP $C_{10}$-$C_{13}$), Medium Chain CPs (MCCP $C_{14}$-$C_{17}$) and Long Chain CPs (LCCP >$C_{17}$). The Chlorine content of these mixtures can vary from 30-70% depending on the application. Technical CPs are used as plasticizers or fire retardants. CPs are classified as persistent and non-biodegradable and they accumulate in the food chain. SCCPs were categorized in group 2B as possibly carcinogenic to humans from the International Agency for Research on Cancer (IARC). SCCP (chlorine content >48%) are listed by the Stockholm Convention on Persistent Organic Pollutants. In Europe SCCP as constituents of articles are prohibited according to regulation 2019/1021 of the European Parliament and of the Council of 20 June 2019 on persistent organic pollutants. Articles containing SCCP in concentrations lower than 0.15% by weight are allowed. Furthermore, it became industrial practice to restrict MCCP as well.

In 2021 the Institute for Interlaboratory Studies (iis) organized a proficiency test for the determination of SCCP and MCCP in Textile for the first time. During the annual proficiency testing program 2022/2023 it was decided to continue the proficiency test for the determination of SCCP/MCCP in Textile.

In this interlaboratory study 33 laboratories in 15 countries registered for participation, see appendix 3 for the number of participants per country. In this report the results of the SCCP/MCCP in Textile 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 textile sample containing SCCP and MCCP of approximately 3 grams labelled #22735.
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 green colored cotton textile was selected which was artificially fortified with SCCP and MCCP. After homogenization 50 small plastic bags were filled with approximately 3 grams each and labelled #22735.
The homogeneity of the subsamples was checked by the determination of SCCP content in accordance with ISO18219 on 8 stratified randomly selected subsamples.

| | SCCP in mg/kg |
|---|---|
| sample #22735-1 | 101.6 |
| sample #22735-2 | 108.4 |
| sample #22735-3 | 108.6 |
| sample #22735-4 | 108.6 |
| sample #22735-5 | 114.5 |
| sample #22735-6 | 109.3 |
| sample #22735-7 | 103.1 |
| sample #22735-8 | 120.5 |

Table 1: homogeneity test results of subsamples #22735

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

| | SCCP in mg/kg |
|---|---|
| r (observed) | 16.8 |
| reference method | Horwitz (n=9) |
| 0.3 x R (reference method) | 21.7 |

Table 2: evaluation of the repeatability of subsamples #22735

The calculated repeatability is in agreement with 0.3 times the estimated reproducibility calculated with the Horwitz equation (based on 9 components). Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one textile sample labelled #22735 was sent on September 21, 2022.

### 2.5 ANALYZES

The participants were requested to determine SCCP, CAS No. 85535-84-8 and MCCP, CAS No. 85535-85-9.
It was requested not to use less than 0.5 gram per determination to ensure homogeneity.
It was also requested to report if the laboratory was accredited for the determined 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 (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:

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

## 4 EVALUATION

In this proficiency test no problems were encountered with the dispatch of the samples. One participant reported test results after the final reporting date and four participants did not report any test results.
In total 29 participants reported 58 numerical test results. Observed were 3 outlying test results, which is 5.2%. 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 reported 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.

Since 2021 test method ISO22818 is available for the determination of SCCP and MCCP in textile products out of different matrices, especially in polymer of the coated fabrics, prints made of polymer and buttons made of polymer (e.g. PVC).
The precision data mentioned in test method ISO22818:21 is for two specific types of coated fabrics which is not the same as the material of the PT sample. Because this is the second PT on SCCP/MCCP in Textile, it is decided to use the Horwitz equation (based on nine components) for estimation of the target reproducibilities and to mention the requirements from ISO22818:21 for comparison only. However, the test results of the group participants followed very well the described precision statements of ISO22818:21.

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 9 components) and with the requirements of ISO22818:21.

MCCP: This determination was not problematic. Two 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 (based on 9 components) and with the requirements of ISO22818:21.

### 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 | 28 | 68.2 | 27.3 | 48.6 |
| MCCP | mg/kg | 27 | 365 | 159 | 202 |

Table 3: reproducibilities of components on sample #22735

Without further statistical calculations it can be concluded that for both components there is a good compliance of the group of participating laboratories with the reference test method.

### 4.3 COMPARISON OF THE PROFICIENCY TEST OF OCTOBER 2022 WITH THE PREVIOUS PT

| | October 2022 | November 2021 |
|---|---|---|
| Number of reporting laboratories | 29 | 11 |
| Number of test results | 58 | 21 |
| Number of statistical outliers | 3 | 0 |
| Percentage of statistical outliers | 5.2% | 0% |

Table 4: comparison with the previous proficiency test

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 | October 2022 | November 2021 | Target (Horwitz) | ISO22818 |
|---|---|---|---|---|
| SCCP | 14% | 23% | 25% | 18.7% |
| MCCP | 16% | 39% | 20% | 16.1% |

Table 5: development of the uncertainties over the years

The uncertainties observed in this PT are lower than the uncertainties observed in the first PT.

### 4.4 EVALUATION OF THE ANALYTICAL DETAILS

The majority of the reporting participants mentioned to have used test method ISO22818. Some participants used the test method ISO18219-1 which is a method for the determination of SCCP in leather. Test method ISO18219-1 does not contain precision data.

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 65% of the participants are accredited to determine the reported components.
- About 40% used the sample as received, about 55% further cut the sample and about 5% further grinded the sample prior to analysis.
- About 85% used 0.5 grams of sample intake, about 15% used 1 gram.
- About 80% used Toluene as extraction solvent, about 20% used Hexane or a Hexane mixture.
- Almost all participants used an extraction time of 60 minutes and an extraction temperature of 60°C.

For SSCP and MCCP the calculated reproducibility is in agreement with the requirements of the target reproducibility, therefore no separate statistical analysis has been performed.

## 5 DISCUSSION

All reporting participants were able to detect SCCP and MCCP in the sample.

In Regulation (EU) 2019/1021 of the European Parliament and of the Council of 20 June 2019 on persistent organic pollutant it is mentioned that articles containing SCCP in concentrations lower than 0.15% by weight are allowed. When the results of this interlaboratory study were compared to this regulation, it was noticed that all reporting participants would have accepted sample #22735 based on the test results of SCCP.

In this PT, the average of the homogeneity test results are not in line with the average (consensus value) from the PT results. There are several reasons for this. First, the goal of the homogeneity testing is very different from the goal of the evaluation of the reported PT results. In order to prove the homogeneity of the PT samples, a test method is selected with a high precision (smallest variation). The accuracy (trueness) of the test method is less relevant.
Secondly, the homogeneity testing is done by one laboratory only. The test results of this (ISO/IEC 17025 accredited) laboratory will have a bias (systematic deviation) depending on the test method used. The desire to detect small variations between the PT samples leads to the use of a sensitive test method with high precision, which may be a test method with significant bias.
Also each test result reported by the laboratories that participate in the PT will have a bias. However, some will have a positive bias and others a negative bias. These different biases compensate each other in the PT average (consensus value). Therefore, the PT consensus value may deviate from the average of the homogeneity test. At the same time the accuracy of the PT consensus value is more reliable than the accuracy of the average of the results of the homogeneity test.

## 6 CONCLUSION

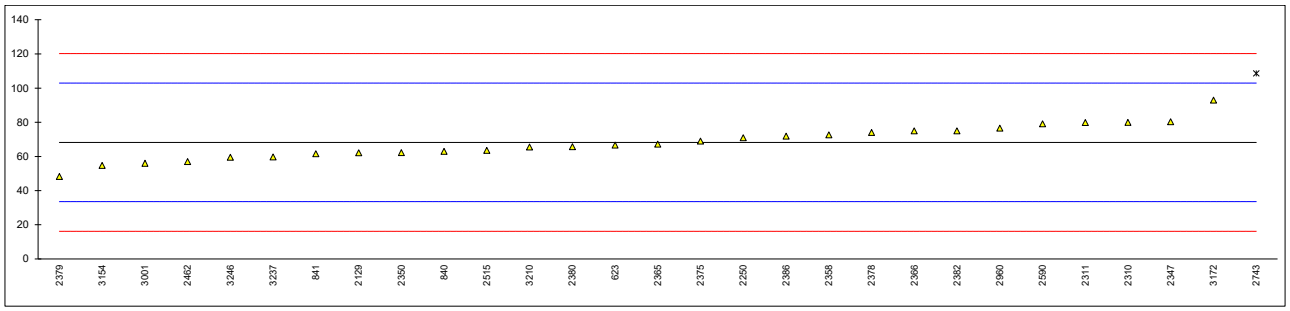
Although it can be concluded that most of the participants have no problem with the determination of SCCP and/or MCCP in this PT, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

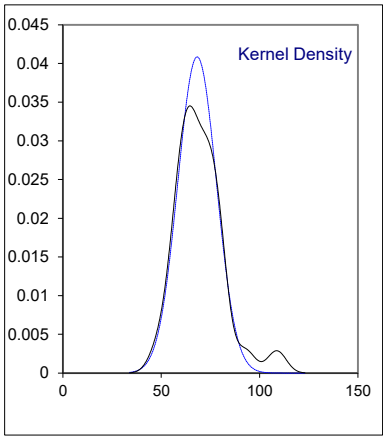
## APPENDIX 1

Determination of SCCP, CAS No. 85535-84-8 on sample #22735; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 623 | ISO22818 | 66.7 | | -0.09 | |
| 840 | ISO22818 | 63 | | -0.30 | |
| 841 | ISO18219-1:2021 | 61.57 | | -0.38 | |
| 2129 | ISO18219-1:2021 | 62.10 | | -0.35 | |
| 2250 | ISO18219-1:2021 | 71 | | 0.16 | |
| 2265 | | ----- | | ----- | |
| 2310 | ISO18219-1:2021 | 80 | | 0.68 | |
| 2311 | ISO22818 | 79.87 | | 0.67 | |
| 2326 | | ----- | | ----- | |
| 2330 | | ----- | | ----- | |
| 2347 | ISO22818 | 80.26 | | 0.69 | |
| 2350 | ISO22818 | 62.2 | | -0.35 | |
| 2358 | ISO18219-1:2021 | 72.62 | | 0.25 | |
| 2365 | ISO22818 | 67.2 | | -0.06 | |
| 2366 | ISO22818 | 75 | | 0.39 | |
| 2375 | ISO22818 | 69 | | 0.05 | |
| 2378 | ISO22818 | 74 | | 0.33 | |
| 2379 | ISO18219-1:2021 | 48.2369 | | -1.15 | |
| 2380 | ISO18219:2015 | 65.704 | | -0.14 | |
| 2382 | ISO22818 | 75.0 | | 0.39 | |
| 2386 | ISO18219-1:2021 | 71.9 | | 0.21 | |
| 2462 | ISO18219-1:2021 | 57 | C | -0.65 | first reported not detected |
| 2515 | ISO22818 | 63.56 | | -0.27 | |
| 2561 | | ----- | | ----- | |
| 2590 | ISO22818 | 79.108 | | 0.63 | |
| 2743 | ISO22818 | 108.66043 | C,R(0.05) | 2.33 | first reported 21.73209 |
| 2960 | ISO18219-1:2021 | 76.6 | | 0.48 | |
| 3001 | ISO18219-1:2021 | 56 | | -0.70 | |
| 3154 | ISO22818 | 54.767 | | -0.78 | |
| 3172 | ISO18219-1:2021 | 92.923 | | 1.42 | |
| 3210 | ISO22818 | 65.509 | | -0.16 | |
| 3237 | ISO18219-1:2021 | 59.7 | | -0.49 | |
| 3246 | ISO22818 | 59.47 | | -0.50 | |

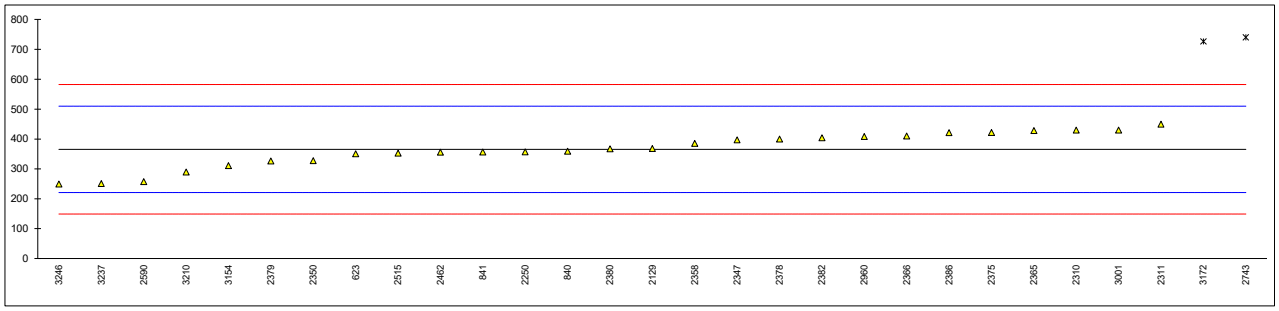
| | | | |
|---|---|---|---|
| | normality | OK | |
| | n | 28 | |
| | outliers | 1 | |
| | mean (n) | 68.214 | |
| | st.dev. (n) | 9.7632 | RSD=14% |
| | R(calc.) | 27.337 | |
| | st.dev.(Horwitz n=9) | 17.3417 | |
| | R(Horwitz n=9) | 48.557 | |
| Compare | | | |
| | R(ISO22818:21) | 35.717 | ISO22818:21 sample A, Table C.1 |

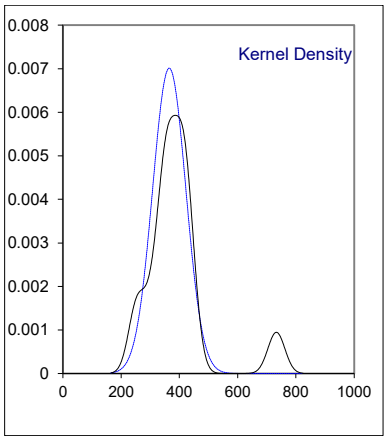


## Determination of MCCP, CAS No. 85535-85-9 on sample #22735; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 623 | ISO22818 | 350.7 | | -0.20 | |
| 840 | ISO22818 | 359 | | -0.09 | |
| 841 | ISO18219-2:2021 | 356.27 | | -0.13 | |
| 2129 | ISO18219-2:2021 | 368.41 | | 0.04 | |
| 2250 | ISO18219-2:2021 | 357 | | -0.12 | |
| 2265 | | ----- | | ----- | |
| 2310 | ISO18219-2:2021 | 430 | | 0.90 | |
| 2311 | ISO22818 | 450.1 | | 1.17 | |
| 2326 | | ----- | | ----- | |
| 2330 | | ----- | | ----- | |
| 2347 | ISO22818 | 396.88 | | 0.44 | |
| 2350 | ISO22818 | 327.47 | | -0.53 | |
| 2358 | ISO18219-2:2021 | 385.34 | | 0.28 | |
| 2365 | ISO22818 | 428.0 | | 0.87 | |
| 2366 | ISO22818 | 410 | | 0.62 | |
| 2375 | ISO22818 | 422 | | 0.78 | |
| 2378 | ISO22818 | 400 | | 0.48 | |
| 2379 | ISO18219-2:2021 | 326.6272 | | -0.54 | |
| 2380 | ISO18219:2015 | 367.322 | | 0.03 | |
| 2382 | ISO22818 | 404.0 | | 0.54 | |
| 2386 | ISO18219-2:2021 | 421 | | 0.77 | |
| 2462 | ISO18219-2:2021 | 356 | | -0.13 | |
| 2515 | ISO22818 | 352.6 | | -0.18 | |
| 2561 | | ----- | | ----- | |
| 2590 | ISO22818 | 257.506 | | -1.50 | |
| 2743 | ISO22818 | 740.34437 | C,R(0.01) | 5.20 | first reported 148.06887 |
| 2960 | ISO18219-2:2021 | 408.5 | | 0.60 | |
| 3001 | ISO18219-2:2021 | 430 | | 0.90 | |
| 3154 | ISO22818 | 310.898 | | -0.76 | |
| 3172 | ISO18219-2:2021 | 726.62 | C,R(0.01) | 5.01 | first reported 568.51 |
| 3210 | ISO22818 | 289.442 | | -1.05 | |
| 3237 | ISO18219-2:2021 | 251.0 | | -1.59 | |
| 3246 | ISO22818 | 249.49 | | -1.61 | |
| | | | | | |
| | normality | OK | | | |
| | n | 27 | | | |
| | outliers | 2 | | | |
| | mean (n) | 365.391 | | | |
| | st.dev. (n) | 56.9001 | RSD=16% | | |
| | R(calc.) | 159.320 | | | |
| | st.dev.(Horwitz n=9) | 72.1548 | | | |
| | R(Horwitz n=9) | 202.034 | | | |
| Compare | | | | | |
| | R(ISO22818:21) | 164.718 | ISO22818:21 sample A, Table C.1 | | |



## APPENDIX 2 Analytical details

| lab | ISO/IEC17025 accredited | sample preparation before use | sample intake (g) | extraction solvent | extraction time (minutes) | extraction temp. (°C) |
|---|---|---|---|---|---|---|
| 623 | Yes | Further cut | 0.5 gr | Toluene | 1 Hour | 60°C |
| 840 | Yes | Further cut | 0.5 | toluene | 60 | 60 |
| 841 | Yes | Further cut | 0.5 grams | toluene solvent | 60 minutes | 60°C |
| 2129 | Yes | Used as received | 1g | Toluene | 60min | 60°C |
| 2250 | Yes | Further cut | 0,5 | Hexane | 60 | 60 |
| 2265 | --- | --- | | | | |
| 2310 | Yes | Further cut | 0.5 | Hexane | 60 | 60 |
| 2311 | No | Further cut | 0.5 | Toluene | 60 | 60 |
| 2326 | --- | --- | | | | |
| 2330 | --- | --- | | | | |
| 2347 | No | Used as received | 0.5g | | 60min | 60℃ |
| 2350 | Yes | Further cut | 0.5g | Hexane | 60 min | 60℃ |
| 2358 | Yes | Used as received | 0.5 | Toluene | 60 | 60 |
| 2365 | Yes | Further cut | 0.5g | Toluene | 60min | 60℃ |
| 2366 | No | Further cut | 0.5g | toluene | 60min | 60℃ |
| 2375 | No | Further cut | 0.5 g | Toluene | 60 min | 60°C |
| 2378 | No | Used as received | 0.5g | toluene | 60 | 60 |
| 2379 | No | Further cut | 0.5 g | Toluene | 60 minutes | 60°C |
| 2380 | Yes | Further cut | 0.5 g | Toluene | 60 Minute | 60°C |
| 2382 | Yes | Further cut | 0.5g | Toluene | 60 | 60 |
| 2386 | Yes | Used as received | 1 | Toluene | 60 | 60 |
| 2462 | Yes | Further cut | 0.5g | Hexane | 60min | 60°C |
| 2515 | Yes | Used as received | 1gram | Toluene | 60 min ± 2min | 60°C ± 2°C |
| 2561 | --- | --- | | | | |
| 2590 | No | Further grinded | 0.5g | toluene | 60 | 60°C |
| 2743 | No | Used as received | 0.5 grams | Toluene and then hexane | 60 min | 60°C |
| 2960 | Yes | Used as received | 0.5g | Toluene | 1h | 60 |
| 3001 | No | Used as received | 0,5 gr | Toluene | 60 | 60 |
| 3154 | Yes | Used as received | 0,5 | Toluene | 60 | 60 |
| 3172 | --- | --- | | | | |
| 3210 | No | Further cut | 1 gram | Toluene | 60 minutes | 60°C |
| 3237 | Yes | Further grinded | 0,5 | Hexane:Acetone (1:1) | 30 | 21 |
| 3246 | Yes | Used as received | 0.5g | Toluene | 1h | 60°C |

## APPENDIX 3

## Number of participants per country

1 lab in BANGLADESH
1 lab in CAMBODIA
1 lab in FRANCE
5 labs in GERMANY
1 lab in HONG KONG
2 labs in INDIA
1 lab in INDONESIA
3 labs in ITALY
1 lab in KOREA, Republic of
7 labs in P.R. of CHINA
1 lab in PAKISTAN
1 lab in THAILAND
3 labs in TURKEY
1 lab in UNITED KINGDOM
4 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 |
| E | = calculation difference between reported test result and result calculated by iis |
| 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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