# Results of Proficiency Test
# Pesticides in Textile
# December 2021

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AMENDMENT

This amended report replaces the original report iis21T08 of March 2022.

It was discovered that there was an error in the analytical details table in appendix 3. The column headings did not correspond with the details of the column and the column with extraction temperatures was missing. This has been corrected.

The following page in this report has been amended:

- Appendix 1: page 15 (page 14 in the original report)

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# 1 INTRODUCTION

Many countries have adopted environmental standards and requirements restricting the use of harmful chemicals in the production of textiles and clothing. Laws and regulations impose some of these standards and requirements. In addition to mandatory environmental standards and requirements for textile, there are some Ecolabelling schemes imposing environmental requirements for textile products on a voluntary basis. Well known organizations are for instance: Bluesign® (Switzerland), which has created a Bluesign® restricted substances list (RSL) and Oeko-Tex Standard 100 (Switzerland).

Since 2004 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of Pesticides in Textile once every one or two years. During the annual proficiency testing program of 2021/2022 it was decided to continue the proficiency test for the determination of Pesticides in Textile.

In this interlaboratory study 13 laboratories in 9 different countries registered for participation. See appendix 4 for the number of participants per country. In this report the results of the Pesticides 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 the 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 of approximately 3 grams positive on pesticides and labelled #21796.
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 pink cotton which was made positive on Dichlorprop (from the Chlorophenoxy Acid group) was selected. This material was cut into small pieces. After homogenization 50 subsamples of approximately 3 grams each were prepared and labelled #21796.
The homogeneity of the subsamples was checked by the determination of Dichlorprop using an in-house test method (with Methanol in ultrasonic bath) on 8 stratified randomly selected subsamples.

| | Dichlorprop in mg/kg |
|---|---|
| sample #21796-1 | 1.956 |
| sample #21796-2 | 1.936 |
| sample #21796-3 | 1.891 |
| sample #21796-4 | 1.906 |
| sample #21796-5 | 1.883 |
| sample #21796-6 | 1.927 |
| sample #21796-7 | 1.777 |
| sample #21796-8 | 1.693 |

Table 1: homogeneity test results of subsamples #21796

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

| | Dichlorprop in mg/kg |
|---|---|
| r (observed) | 0.252 |
| reference method | Horwitz |
| 0.3 x R (reference method) | 0.229 |

Table 2: evaluation of the repeatability of subsamples #21796

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

To each of the participating laboratories one textile sample labelled #21796 was sent on November 17, 2021.

### 2.5 ANALYZES

The participants were requested to determine the concentrations of a limited number of prescribed pesticides (Chlorophenoxy Acids for #21796), applying the analytical procedure that is routinely used in the laboratory. It was also requested to report if the laboratory was accredited to determine the requested components and to report some analytical details of the test method used.

It was explicitly requested to treat the sample as if it was routine samples and to report the test results using the indicated units on the report form and not to round the results, but to report as much significant figures as possible. It was also requested not to report ‘less than’ results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the 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 the appendices 1 and 2 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 the data analysis and the original results are placed under 'Remarks' in the result tables in appendices 1 and 2. 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, for one or more of the analytes the criterion of ISO13528, paragraph 9.2.1 was not met, therefore, the uncertainty of the assigned value for these analytes is not negligible and will be used to calculate z’-scores (see paragraph 3.3).

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

### 3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

### 3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation of 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.

The standard uncertainly ($u_x$) was calculated from the (target) standard deviation in accordance with ISO13528, paragraph 5.6:

$$u_x = 1.25 * (\text{st.dev } (n)) / \sqrt{n}$$

In ISO13528 is stated that if $u_x \geq 0.3$ * standard deviation for proficiency testing, the uncertainty of the assigned value is not negligible and needs to be included in the interpretation of the results of the proficiency test. Therefore, in this PT report, z'-scores were calculated instead of the usual z-scores. The z'(target)-scores were calculated in accordance with ISO13528 paragraph 9.5:

$$z'(\text{target}) = (\text{test result} - \text{mean of PT}) / \sqrt{((\text{target standard deviation})^2 + (u_x)^2)}$$

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. 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

During the execution of this proficiency test no serious problems occurred with the dispatch of the samples. One participant reported test results after the final reporting date and two participants did not report any test results.
In total 11 laboratories reported 11 numerical test results. Observed was 1 outlying result, which is 9.1% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

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

### 4.1 EVALUATION PER 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 table together with the original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 5.

Unfortunately, a suitable reference test method, providing the precision data, is not available for all determinations. For these tests the calculated reproducibility was compared against the estimated reproducibility calculated with the Horwitz equation.

Dichlorprop: The determination may be problematic at the level of 20 mg/kg. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the estimated reproducibility calculated with the Horwitz equation combined with the uncertainty as explained in paragraph 3.3.

### 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 estimated using the Horwitz equation are presented in the next table.

| Component | unit | n | average | 2.8 * sd | R(target) |
|---|---|---|---|---|---|
| Dichlorprop | mg/kg | 10 | 19.8 | 9.0 | 6.7 |

Table 3: reproducibility of pesticides in sample #21796

Without further statistical calculations it can be concluded that for the observed pesticides the group of participants may have difficulties with the determination.

### 4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2021 WITH PREVIOUS PTS

| | December 2021 | December 2020 | December 2018 | December 2016 | November 2014 |
|---|---|---|---|---|---|
| Number of reporting laboratories | 11 | 14 | 14 | 13 | 21 |
| Number of test results | 11 | 25 | 81 | 109 | 53 |
| Number of statistical outliers | 1 | 4 | 15 | 5 | 3 |
| Percentage of statistical outliers | 9.1% | 16% | 19% | 4.6% | 5.7% |

Table 4: comparison with previous proficiency tests

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

The performance of the determinations of the proficiency test was compared, expressed as relative standard deviation (RSD) or uncertainty of the PTs, see below table.

| | December 2021 | December 2020 | December 2018 | December 2016 | 2008 - 2014 |
|---|---|---|---|---|---|
| Carbaryl | -- | -- | -- | 39% | 52% |
| Cyhalothrin-lambda | -- | -- | -- | -- | 35 - 45% |
| Cypermethrin (=Σ) | -- | -- | -- | -- | 15 - 28% |
| 2,4-D | -- | 16% | -- | -- | -- |
| 4,4'-DDD | -- | -- | -- | -- | 29-38% |
| Dichlorprop | 16% | -- | -- | -- | -- |
| Deltamethrin | -- | 33% | -- | -- | 12 - 31% |
| Dimethoate | -- | -- | -- | -- | 35-54% |
| α/β-Endosulfan | -- | -- | 18 - 34% | 27 - 47% | 15 - 33% |
| Esfenvalerate | -- | -- | -- | -- | 22 - 42% |
| Fenvalerate | -- | -- | -- | -- | 11 - 37% |
| Methoxychlor | -- | -- | -- | -- | 14 - 35% |
| Monocrotophos | -- | -- | -- | -- | 38% |
| Parathion | -- | -- | -- | 61% | 73% |
| Quinalphos | -- | -- | 35 - 38% | 32 - 52% | 24 - 39% |

Table 5: comparison of uncertainties in iis proficiency tests on pesticides in textile

The precision that was found for Dichlorprop is in the range of the precision found for other pesticides over the years. It was the first time that Dichlorprop was present. The relative low number of participating laboratories may (partly) explain for the relatively large variations.

### 4.4 EVALUATION OF ANALYTICAL DETAILS

For this PT some analysis details were requested and listed in appendix 3. From the answers given the following can be summarized:

- Seven of the eleven reporting laboratories mentioned to be accredited for the determination of Dichlorprop according to ISO/IEC17025.
- Seven participants used 1 gram as sample intake. Four other participants used 0.5 gram.
- Seven laboratories used Ultrasonic for extraction and the other laboratories used either a Soxhlet or an accelerated solvent extraction (ASE) or mechanical shaking. The extractions were done at different temperatures and for different lengths of time, although six out of seven laboratories with ultrasonic as extraction technique used 60 minutes at 50°C with Methanol as extraction solvent.

As the majority of the group follow the same analytical procedures no separate statistical analysis has been performed except for the extraction method. Depending on the group of pesticides it may or may not be of influence. For this pesticide the extraction method appears to have an effect, see paragraph 5 Discussion.

## 5 DISCUSSION

Most participants used an in-house method. Therefore, some test method details were requested in this PT. When the laboratories that use Ultrasonic extraction are evaluated separately the RSD of the group decreases from 16% to 11%. It is expected that since there are many different types of pesticides the method of extraction may be more or less of influence depending on the pesticide.

When the results of this interlaboratory study were compared to the standard 100 by OEKO-TEX® (see table 6) and Bluesign® Restricted Substances List (RSL) – Consumer Safety Limits (see table 7) it could be noticed that all laboratories would have rejected the sample.

| Standard 100 by OEKO-TEX® | Baby | Direct skin contact | With no direct skin contact | Decoration material |
|---|---|---|---|---|
| pesticides, total mg/kg | 0.5 | 1.0 | 1.0 | 1.0 |

Table 6: OEKO-TEX® standard 100

| Bluesign® RSL | Baby | Direct skin contact | Occasional skin contact | With no direct skin contact |
|---|---|---|---|---|
| pesticides, total mg/kg | 0.5 | 0.5 | 0.5 | 0.5 |

Table 7: Bluesign® Restricted Substances List (RSL)

Furthermore, the Ecolabelling Standards and Requirements for Textiles in EU only allow 0.5 mg/kg of total pesticides in raw cotton.

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.

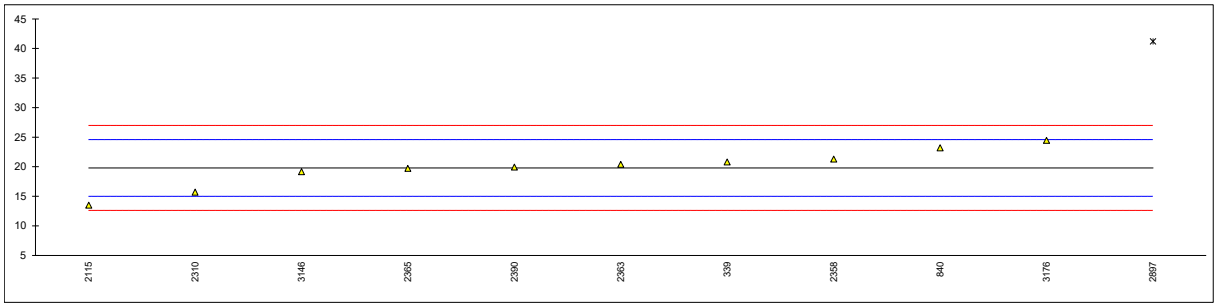
## 6 CONCLUSION

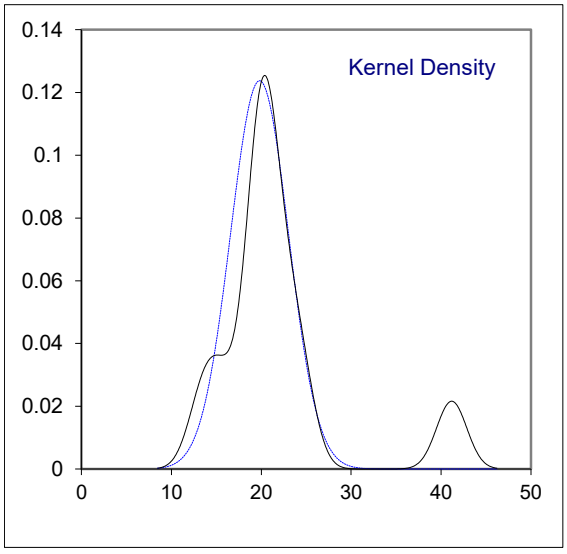
Finally, each laboratory has to evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus improve of the quality of the analytical results.

**APPENDIX 1**

Determination of Dichlorprop (CAS No. 120-36-5), on sample #21796; results in mg/kg

| lab | method | value | mark | z'(targ) | remarks |
|---|---|---|---|---|---|
| 339 | In house | 20.8 | | 0.41 | |
| 840 | In house | 23.2 | | 1.41 | |
| 2115 | In house | 13.51 | | -2.64 | |
| 2310 | In house | 15.7 | C | -1.72 | first reported: 26.9 |
| 2358 | In house | 21.3 | | 0.62 | |
| 2363 | In house | 20.4 | | 0.24 | |
| 2365 | In house | 19.71 | | -0.04 | |
| 2386 | | ----- | | ----- | |
| 2390 | EPA | 19.942 | | 0.05 | |
| 2590 | | ----- | | ----- | |
| 2897 | In house | 41.2 | C,G(0.01) | 8.94 | first reported: 28.42 |
| 3146 | DIN38407F14Mod. | 19.16 | | -0.27 | |
| 3176 | In house | 24.45 | C | 1.94 | first reported: 2.88 |
| | | | | | Ultrasonic extraction only: |
| | normality | OK | | | not OK |
| | n | 10 | | | 7 |
| | outliers | 1 | | | 0 (+4ex) |
| | mean (n) | 19.817 | | | 19.916 |
| | st.dev. (n) | 3.2249 | RSD = 16% | | 2.2849 RSD = 11% |
| | R(calc.) | 9.030 | | | 6.398 |
| | st.dev.(Horwitz ') | 2.3909 | | | 2.3003 |
| | R(Horwitz ') | 6.695 | | | 6.644 |



**APPENDIX 2**

Determination of 2,4,5-T, 2,4-D, MCPA, MCPB and Mecoprop on sample #21796; results in mg/kg

| lab | 2,4,5-T | 2,4-D | MCPA | MCPB | Mecoprop | remarks |
|---|---|---|---|---|---|---|
| 339 | not detected | not detected | not detected | not detected | not detected | |
| 840 | not detected | not detected | not detected | not detected | not detected | |
| 2115 | ----- | ----- | ----- | ----- | ----- | |
| 2310 | Not detected | Not detected | Not detected | Not detected | Not detected | |
| 2358 | not detected | not detected | not detected | not detected | not detected | |
| 2363 | not detected | not detected | not detected | not detected | not detected | |
| 2365 | <0.2 | <0.2 | <0.2 | <0.2 | <0.2 | |
| 2386 | ----- | ----- | ----- | ----- | ----- | |
| 2390 | Not detected | Not detected | Not detected | Not detected | Not detected | |
| 2590 | ----- | ----- | ----- | ----- | ----- | |
| 2897 | not detected | not detected | not detected | not detected | not detected | |
| 3146 | not detected | not detected | not detected | not detected | not detected | |
| 3176 | not detected | ----- | ----- | ----- | ----- | |

**APPENDIX 3 Analytical Details**

Other Chlorophenoxy Acids – sample #21796

| Lab | ISO17025 accredited | Sample preparation | Intake sample | Extraction type | Extraction solvent | Extraction time | Extraction temperature |
|---|---|---|---|---|---|---|---|
| 339 | No | Further cut | 1g | Mechanical Shaking | | | |
| 840 | Yes | Further cut | 0.5g | Ultrasonic | Methanol | 60 minutes | 50°C |
| 2115 | Yes | Used as received | 1 gram | ASE | Methanol | | |
| 2310 | Yes | Further cut | 1 | Ultrasonic | Methanol | 60 minutes | 50 |
| 2358 | Yes | Used as received | 0.5 g | Ultrasonic | Methanol | 60 minutes | 50 degree C |
| 2363 | No | Further cut | about 1g | Ultrasonic | Methanol | 60min | 50℃ |
| 2365 | Yes | Further cut | 1.0g | Ultrasonic | methanol | 60mins | 50℃ |
| 2386 | --- | --- | | --- | | | |
| 2390 | Yes | Further cut | 1 gram | Ultrasonic | Methanol | 60 min | 50 °C? |
| 2590 | --- | --- | | --- | | | |
| 2897 | Yes | Further cut | 0.5g | Mechanical Shaking | Acetonitrile | 5 minutes | Ambient |
| 3146 | No | Used as received | 0.5 | Ultrasonic | Kalium carbonate solution | 30 min | Room temp. |
| 3176 | No | Further cut | 1 | Soxhlet | Acetone | 6 hours | 300 °C |

## APPENDIX 4

### Number of participants per country

1 lab in FRANCE
2 labs in GERMANY
1 lab in HONG KONG
1 lab in INDIA
3 labs in ITALY
2 labs in P.R. of CHINA
1 lab in PAKISTAN
1 lab in TURKEY
1 lab in VIETNAM

**APPENDIX 5**

**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 the statistical evaluation |
| n.a. | = not applicable |
| n.e. | = not evaluated |
| n.d. | = not detected |
| fr. | = first reported |

**Literature**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