



Institute for  
Interlaboratory Studies

## Results of Proficiency Test Pesticides in Textile December 2022

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

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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. Since 2020 this scheme is organized every year. During the annual proficiency testing program of 2022/2023 it was decided to continue the proficiency test for the determination of Pesticides in Textile.

In this interlaboratory study 14 laboratories in 8 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](http://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 textile samples of approximately 3 grams each labelled #22795 and #22796 respectively.

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](http://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 PT sample a batch of green cotton was selected, which was made positive on Aldicarb (from the Carbamates group) and 2,4-D (from the Chorophenoxy Acids group). This material was cut into small pieces. After homogenization 40 small plastic bags were filled with approximately 3 grams each and labelled #22795.

The homogeneity of the subsamples was checked by the determination of Aldicarb using an in-house test method on 8 stratified randomly selected subsamples.

	Aldicarb in mg/kg
sample #22795-1	0.98
sample #22795-2	1.03
sample #22795-3	1.07
sample #22795-4	1.11
sample #22795-5	1.10
sample #22795-6	1.11
sample #22795-7	1.13
sample #22795-8	1.10

Table 1: homogeneity test results of subsamples #22795

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

	Aldicarb in mg/kg
r (observed)	0.14
reference method	iis memo 2302 *)
0.3 x R (reference method)	0.30

Table 2: evaluation of the repeatability of subsamples #22795

\*) see chapter 4.1 for more information

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

For the second PT sample a batch of off-white cotton was selected, which was made positive on alpha- and beta-Endosulfan (from the Organochlorines group). This material was cut into small pieces. After homogenization 40 small plastic bags were filled with approximately 3 grams each and labelled #22796.

The homogeneity of the subsamples was checked by the determination of beta-Endosulfan II using an in-house test method on 8 stratified randomly selected subsamples.

	beta-Endosulfan II in mg/kg
sample #22796-1	0.66
sample #22796-2	0.79
sample #22796-3	0.68
sample #22796-4	0.72
sample #22796-5	0.78
sample #22796-6	0.66
sample #22796-7	0.76
sample #22796-8	0.68

Table 3: homogeneity test results of subsamples #22796

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

	beta-Endosulfan II in mg/kg
r (observed)	0.15
reference method	iis memo 2302 *)
0.3 x R (reference method)	0.20

Table 4: evaluation of the repeatability of subsamples #22796

\*) see chapter 4.1 for more information

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

To each of the participating laboratories two textile samples labelled #22795 and #22796 respectively were sent on November 9, 2022.

## 2.5 ANALYZES

The participants were requested to determine the concentrations of a limited number of prescribed pesticides (Carbamates and Chlorophenoxy Acids for #22795 and Organochlorines for #22796).

To ensure homogeneity it was requested not to use less than 0.5 gram per determination. 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 samples as if they were routine samples and 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. 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/](http://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](http://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/](http://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. However, the z-scores in this PT are calculated with a target reproducibility based on the reproducibility of the group (including the uncertainty) over the past years, therefore the uncertainty is already present in the target reproducibility.

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 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.

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:

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

The  $Z_{(\text{target})}$  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. Two participants did not report any test results and not all participants were able to report all tests requested.

In total 12 participants reported 45 numerical test results. Observed were 5 outlying test results, which is 11%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

Not all data sets proved to have a normal Gaussian distribution. These are referred to as “not OK” or “suspect”. The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.



#### 4.1 EVALUATION PER 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 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 the determinations of Pesticides in Textile. Up until 2021 the performance was calculated by using the (strict) estimated reproducibility calculated with the Horwitz equation. The uncertainty of the group was not negligible and this uncertainty was added to the target reproducibility by calculating z'-scores.

In 2023 iis decided to use the iis PT data gathered since 2007 to estimate a more realistic target reproducibility (see iis memo 2302, lit. 13). This target reproducibility is based on the variation of the group over the years and as such this group uncertainty is already accounted for and does not have to be compensated by calculating z'-scores. Furthermore, the variation appears not to be dependent on component or groups of components. Therefore, it was decided to calculate the average reproducibility over all components and to use one target reproducibility for all pesticides measured in the PT samples.

This estimated target reproducibility was calculated from the relative standard deviation of 33% of the mean multiplied by 2.8.

##### **sample #22795**

Aldicarb: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the target reproducibility as derived from iis memo 2302.

2.4-D: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the target reproducibility as derived from iis memo 2302.

##### **sample #22796**

alpha-Endosulfan I: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the target reproducibility as derived from iis memo 2302.

beta-Endosulfan II: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the target reproducibility as derived from iis memo 2302.

## 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 ( $2.8 \times$  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)
Aldicarb	mg/kg	12	1.43	0.93	1.33
2,4-D	mg/kg	11	37.3	12.1	34.5

Table 5: reproducibilities of tests on sample #22795

Component	unit	n	average	2.8 * sd	R(target)
alpha-Endosulfan I	mg/kg	8	0.50	0.20	0.46
beta-Endosulfan II	mg/kg	9	1.39	1.14	1.28

Table 6: reproducibilities of tests on sample #22796

Without further statistical calculations it can be concluded that for all tests there is a good compliance of the group of participants with the reference test method.

## 4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2022 WITH PREVIOUS PTS

	December 2022	December 2021	December 2020	December 2018	December 2016
Number of reporting laboratories	12	11	14	14	13
Number of test results	45	11	25	81	109
Number of statistical outliers	5	1	4	15	5
Percentage of statistical outliers	11%	9.1%	16%	19%	4.6%

Table 7: 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.

	December 2022	December 2021	December 2020	December 2018	2007 - 2016	iis memo 2302
Aldicarb	23%	--	--	--	--	33%
Carbaryl	--	--	--	--	39-52%	33%
Cyhalothrin-lambda	--	--	--	--	35-45%	33%
Cypermethrin (=Σ)	--	--	--	--	15-28%	33%
2,4-D	12%	--	16%	--	--	33%
4,4'-DDD	--	--	--	--	29-38%	33%

	December 2022	December 2021	December 2020	December 2018	2007 - 2016	iis memo 2302
Dichlorprop	--	16%	--	--	--	33%
Deltamethrin	--	--	33%	--	12-31%	33%
Dimethoate	--	--	--	--	35-54%	33%
$\alpha/\beta$ -Endosulfan	14-29%	--	--	18-34%	15-47%	33%
Esfenvalerate	--	--	--	--	22-42%	33%
Fenvalerate	--	--	--	--	11-37%	33%
Methoxychlor	--	--	--	--	14-35%	33%
Monocrotophos	--	--	--	--	38-74%	33%
Parathion	--	--	--	--	61-73%	33%
Quinalphos	--	--	--	35-38%	24-52%	33%

Table 8: development of the uncertainties over the years

The precision that was found for Aldicarb is in the range of the precision found for other pesticides over the years. It was the first time that Aldicarb was present. The uncertainties observed in this PT are comparable to the uncertainties observed in previous PTs.

#### 4.4 EVALUATION OF THE ANALYTICAL DETAILS

For this PT some analytical details were requested which are given in appendix 3. Based on the answers given by the participants the following can be summarized:

- Eight participants mentioned to be accredited for the determination of Carbamates and Chlorophenoxy Acids according to ISO/IEC17025 and nine participants mentioned to be accredited for the determination of Organochlorines according to ISO/IEC17025.
- Seven participants reported to use 1 gram as sample intake and six participants to use 0.5 gram. One participant reported to use 3 grams for the determination of Organochlorines.
- Seven participants reported to use Ultrasonic for extraction and the other laboratories reported either a Soxhlet, mechanical shaking or a stirrer. The extractions were done at different temperatures and for different lengths of time.

The influence of these analytical details could not be determined because the group of participants is too small for further sub analyzes.

## 5 DISCUSSION

When the results of this interlaboratory study were compared to the standard 100 by OEKO-TEX® (see table 9) and Bluesign® Restricted Substances List (RSL) – Consumer Safety Limits (see table 10) it could be noticed that all laboratories would have made the same decision and would have rejected both samples.

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 9: 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 10: 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.

## 6 CONCLUSION

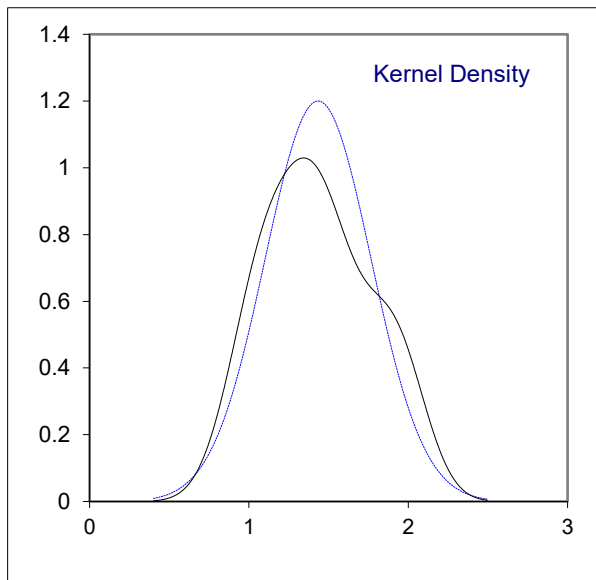
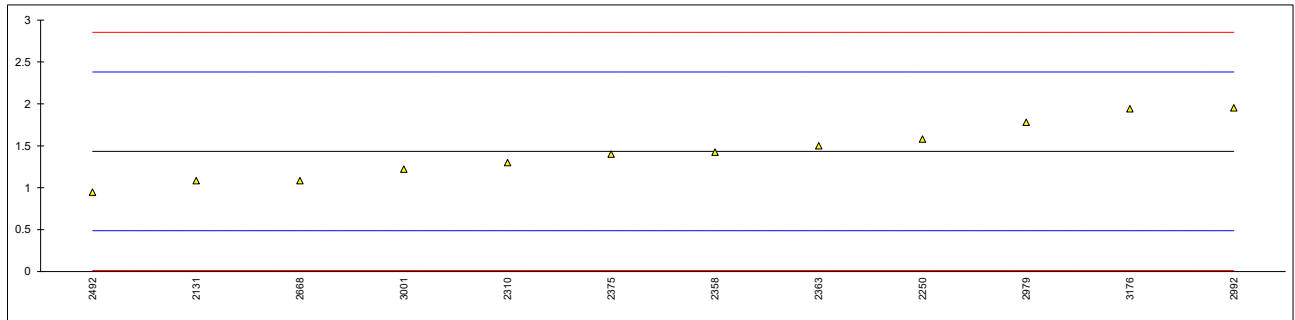
Although it can be concluded that the participants have no problem with the determination on the requested components in this PT, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular 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 Aldicarb (CAS No. 116-06-3-5) on sample #22795; results in mg/kg**

lab	method	value	mark	z(targ)	remarks
2131	In house	1.085		-0.74	
2250		1.58		0.31	
2310	In house	1.3		-0.28	
2358	In house	1.4235		-0.02	
2363	In house	1.5	C	0.14	first reported: 2.32
2375	In house	1.4		-0.07	
2386		----		----	
2459		----		----	
2492	In house	0.947		-1.03	
2668	In house	1.085		-0.74	
2979	In house	1.78		0.73	
2992	In house	1.9529		1.10	
3001	In house	1.22		-0.45	
3176	In house	1.94	C	1.07	first reported: 2.46

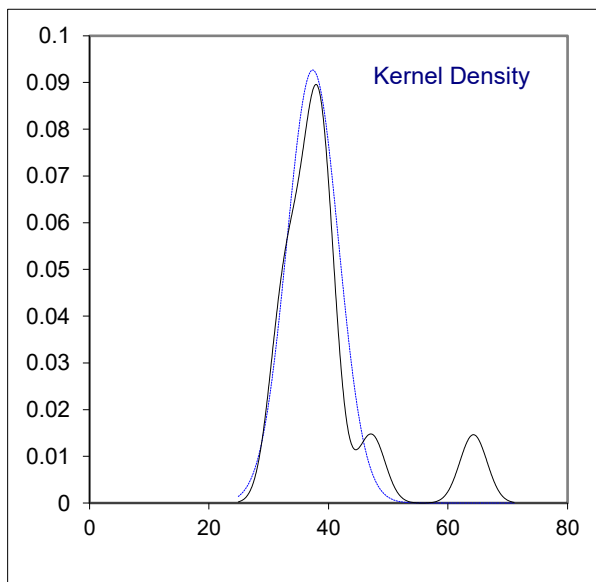
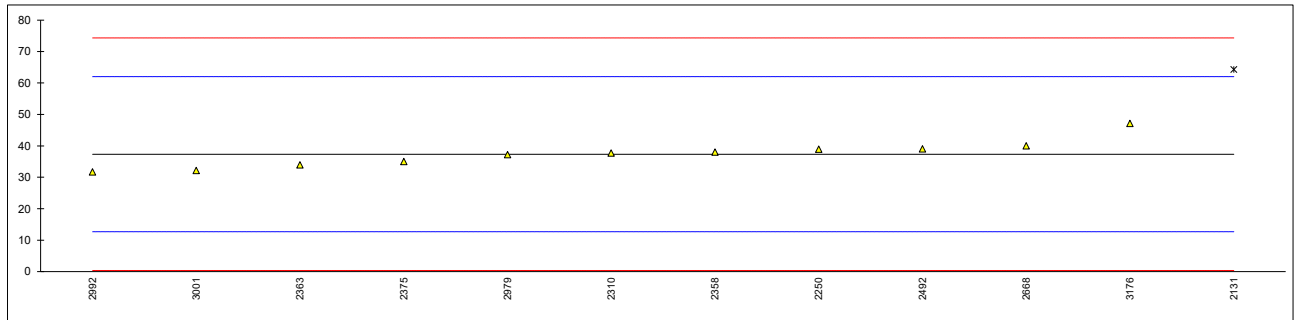
normality OK  
 n 12  
 outliers 0  
 mean (n) 1.435  
 st.dev. (n) 0.3325 RSD = 23%  
 R(calc.) 0.931  
 st.dev.(iis memo 2302) 0.4734  
 R(iis memo 2302) 1.325



Determination of 2,4-D (CAS No. 94-75-7) on sample #22795; results in mg/kg

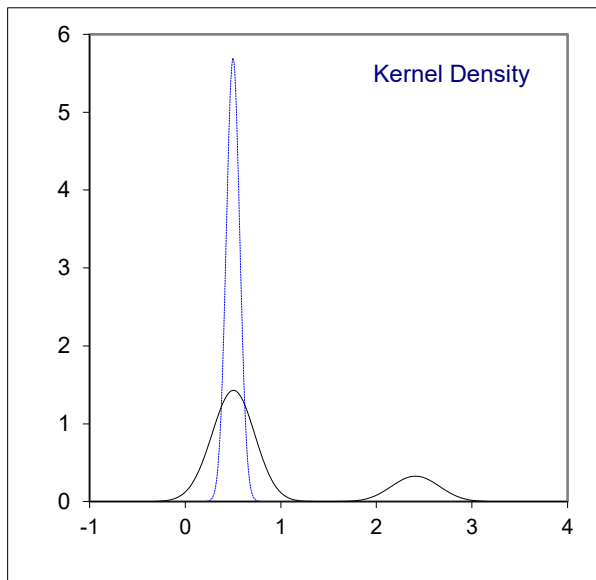
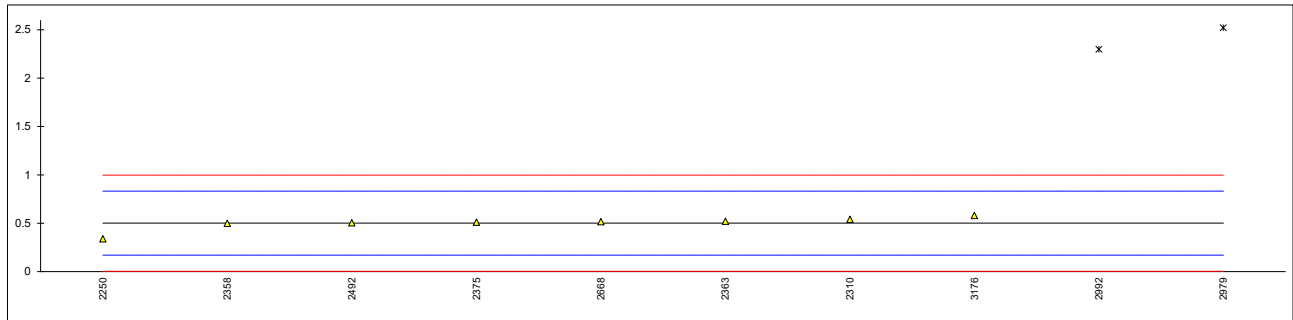
lab	method	value	mark	z(targ)	remarks
2131		64.25	D(0.05)	2.18	
2250		38.9		0.13	
2310	In house	37.7		0.03	
2358	In house	38		0.05	
2363	In house	33.93		-0.28	
2375	In house	35		-0.19	
2386		----		----	
2459		----		----	
2492	In house	39.020		0.14	
2668	In house	40	C	0.22	first reported: Not Detected
2979	In house	37.2		-0.01	
2992	In house	31.7019		-0.46	
3001	In house	32.14		-0.42	
3176	In house	47.16		0.80	

normality suspect  
 n 11  
 outliers 1  
 mean (n) 37.341  
 st.dev. (n) 4.3044 RSD = 12%  
 R(calc.) 12.052  
 st.dev.(iis memo 2302) 12.3226  
 R(iis memo 2302) 34.503



Determination of alpha-Endosulfan I (CAS No. 959-98-8) on sample #22796; results in mg/kg

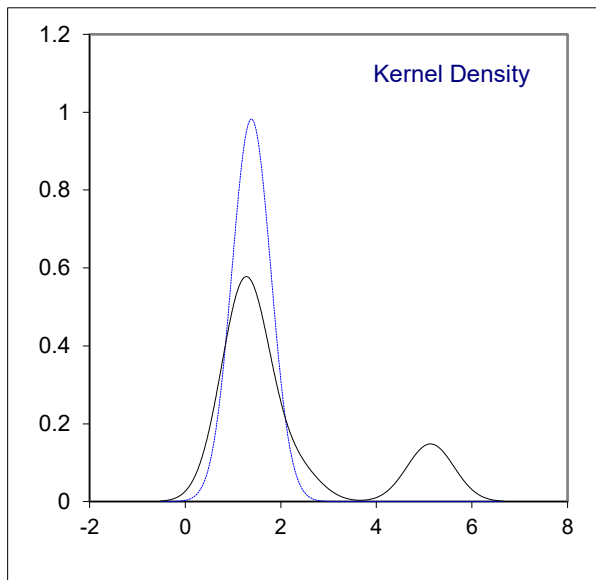
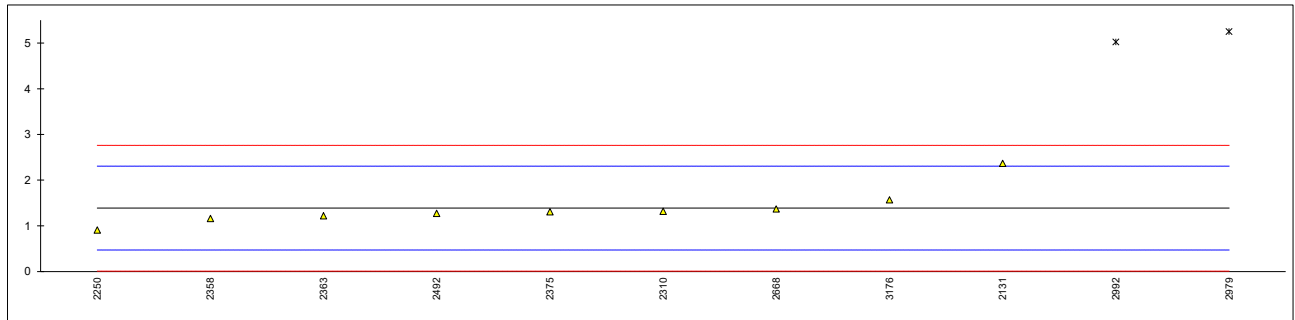
lab	method	value	mark	z(targ)	remarks
2131	In house	-----		-----	
2250	In house	0.34		-0.97	
2310	In house	0.54		0.23	
2358	In house	0.50		-0.01	
2363	In house	0.52		0.11	
2375	In house	0.51		0.05	
2386		-----		-----	
2459		-----		-----	
2492	In house	0.505		0.02	
2668	In house	0.515		0.08	
2979	In house	2.52	C.DG(0.01)	12.20	first reported:1.692
2992	In house	2.298	DG(0.01)	10.86	
3001	In house	not applicable		-----	
3176	In house	0.58		0.48	
normality		not OK			
n		8			
outliers		2			
mean (n)		0.501			
st.dev. (n)		0.0700	RSD = 14%		
R(calc.)		0.196			
st.dev.(iis memo 2302)		0.1654			
R(iis memo 2302)		0.463			



Determination of beta-Endosulfan II (CAS No. 33213-65-9) on sample #22796; results in mg/kg

lab	method	value	mark	z(targ)	remarks
2131	In house	2.365		2.13	
2250	In house	0.91		-1.04	
2310	In house	1.32		-0.15	
2358	In house	1.16		-0.50	
2363	In house	1.22		-0.37	
2375	In house	1.31		-0.17	
2386		----		----	
2459		----		----	
2492	In house	1.273		-0.25	
2668	In house	1.37		-0.04	
2979	In house	5.25	C,DG(0.01)	8.43	first reported: 4.334
2992	In house	5.021	DG(0.01)	7.93	
3001	In house	not applicable		----	
3176	In house	1.57		0.40	

normality not OK  
 n 9  
 outliers 2  
 mean (n) 1.389  
 st.dev. (n) 0.4063 RSD = 29%  
 R(calc.) 1.138  
 st.dev.(iis memo 2302) 0.4583  
 R(iis memo 2302) 1.283





**APPENDIX 2**

Determination of Carbofuran, Carbaryl, Ethienocarb, Fenocarb, Methomyl and Oxamyl on sample #22795; results in mg/kg

lab	Carbofuran	Carbaryl	Ethienocarb	Fenocarb	Methomyl	Oxamyl
2131	----	----	----	----	----	----
2250	----	not detected	----	----	----	----
2310	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed	not analyzed
2358	not detected	not detected	not detected	not detected	not detected	not detected
2363	not analyzed	<1.0	not analyzed	not analyzed	not analyzed	not analyzed
2375	----	----	----	----	----	----
2386	----	----	----	----	----	----
2459	----	----	----	----	----	----
2492	----	----	----	----	----	----
2668	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2979	not detected	not detected	not detected	not detected	not detected	not detected
2992	not detected	not detected	----	not detected	not detected	not detected
3001	not detected	not detected	not applicable	not detected	not detected	not detected
3176	----	----	----	----	----	----

Determination of 2,4,5-T, Dichlorprop, MCPA, MCPB and Mecoprop on sample #22795; results in mg/kg

lab	2,4,5-T	Dichlorprop	MCPA	MCPB	Mecoprop
2131	----	----	----	----	----
2250	not detected	not detected	not detected	not detected	not detected
2310	not detected	not detected	not detected	not detected	not detected
2358	not detected	not detected	not detected	not detected	not detected
2363	<1.0	<1.0	<1.0	<1.0	<1.0
2375	----	----	----	----	----
2386	----	----	----	----	----
2459	----	----	----	----	----
2492	----	----	----	----	----
2668	Not detected	Not detected	Not detected	Not detected	Not detected
2979	not detected	not detected	not detected	not detected	not detected
2992	not detected	not detected	not detected	not detected	not detected
3001	not detected	not detected	not detected	not detected	not detected
3176	----	----	----	----	----

Determination of Aldrin, Chlordane, Heptachlor and Hexachlorocyclopentadiene on sample #22796; results in mg/kg

lab	Aldrin	Chlordane	Heptachlor	Hexachlorocyclopentadiene
2131	----	----	----	is not analysed
2250	not detected	not detected	not detected	----
2310	not detected	not detected	not detected	not analyzed
2358	not detected	not detected	not detected	not detected
2363	not detected	not detected	not detected	not applicable
2375	----	----	----	----
2386	----	----	----	----
2459	----	----	----	----
2492	----	----	----	----
2668	Not detected	Not detected	Not detected	Not detected
2979	not detected	not detected	not detected	not detected
2992	not detected	not detected	not detected	----
3001	not detected	not applicable	not detected	not applicable
3176	----	----	----	----

**APPENDIX 3 Analytical Details**

## Carbamates and Chlorophenoxy Acids – sample #22795

Lab	ISO 17025 accredited	as received or further grinded/cut	Intake sample amount	Extraction type	Extraction solvent	Extraction time	Extraction temp.
2131	Yes	As received	1g	Mechanical Shaking	20 ml Water 4.5 ml Acetonitrile	30 + 15 Minutes (45 Min)	Room Temperature, about 20 - 22 °C
2250	Yes	As received	0,5g	Ultrasonic	Methanol	120 minutes	60°C
2310	Yes	Further cut	1	---	Methanol	60	50
2358	No	As received	1.0	Ultrasonic	Methanol	60	50
2363	Yes	Further cut	1.0g	Ultrasonic	MeOH	60mins	50°C
2375	Yes	Further cut	0.5 gram	Ultrasonic	Water/Acetonitrile (2:1)	20 min	35 C
2386	---	---	---	---	---	---	---
2459	---	---	---	---	---	---	---
2492	Yes	As received	1g	Soxhlet	Methanol	180 mins	150 °C
2668	Yes	Further cut	0.5 gms	Ultrasonic	Methanol	60 min	50C
2979	No	As received	0.5 g	Ultrasonic	Methanol	1 hour	50 C
2992	Yes	Further cut	0.5 and 1.0 g	Mechanical Shaking	10 mL Acetonitrile + 10 mL Water	1470 minutes (24,5 h)	room temperature
3001	No	As received	1	Stirrer	Water/acetonitrile	1	room temperature
3176	No	As received	0,5	Ultrasonic	MeOH	50	60

## Organochlorines – sample #22796

Lab	ISO 17025 accredited	as received or further grinded/cut	Intake sample amount	Extraction type	Extraction solvent	Extraction time	Extraction temp.
2131	Yes	As received	1g	Mechanical Shaking	Water & Acetonitrile Acetone/Dichloromethane	45 Min	Room Temperature 20-22 °C
2250	Yes	Further cut	1.0	Ultrasonic Mechanical	(1:1)	2 x 30	room temperature
2310	Yes	Further cut	1	Shaking	Hexane:Acetone	60 minutes	50
2358	No	As received	1.0	Ultrasonic	Methanol	60	60
2363	Yes	Further cut	3g	Ultrasonic	Acetone: n-hexan=1 : 1	60mins	50°C
2375	Yes	Further cut	0.5 gram	Ultrasonic	Hexane/Acetone (1:1)	60 min	50 C
2386	---	---	---	---	---	---	---
2459	---	---	---	---	---	---	---
2492	Yes	As received	1g	Soxhlet	acetone Ethyl acetate:n-Hexane	180 mins	150 °C
2668	Yes	Further cut	0.5 gms	Ultrasonic	(1:1)	30 min	30C
2979	Yes	As received	0.5 g	Ultrasonic	Methanol	1 hour	50 C
2992	Yes	Further cut	0.5 and 1.0 g	Mechanical Shaking	10 mL Acetonitrile + 10 mL Water	1470 minutes (24.5 h)	room temperature
3001	No	As received	1	Stirrer	water/acetonitrile	1	room temperature
3176	No	As received	0,5	Ultrasonic	MeOH	50	60

## **APPENDIX 4**

### **Number of participants per country**

1 lab in EGYPT

3 labs in GERMANY

2 labs in HONG KONG

2 labs in INDIA

1 lab in P.R. of CHINA

1 lab in PAKISTAN

1 lab in SWITZERLAND

3 labs in TURKEY

## 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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