Results of Proficiency Test
Pesticides in Textile
December 2020

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® system substances list (BSSL) and Oeko-Tex Standard 100 (Switzerland).

Since 2004 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for Pesticides in textile, once every two years. During the annual proficiency testing program of 2020/2021 it was decided to continue the proficiency test for the analysis of Pesticides in textile.
In this interlaboratory study 16 laboratories in 10 different countries registered for participation. See appendix 4 for the number of participants per country. In this report the results of this proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands was the organizer of 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 two different textile samples of approximately 3 grams each both positive on Pesticides, one sample of yellow cotton pieces labelled #20740 and one off white cotton pieces labelled #20741. 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 yellow cotton was selected which was made positive on the herbicide 2,4-Dichlorophenoxy Acetic Acid (2,4-D). This material was cut into small pieces. After homogenization 45 subsamples of approximately 3 grams each were prepared and labelled #20740.
The homogeneity of the subsamples was checked by the determination of 2,4-D using an in-house test method (with Methanol in ultrasonic bath) on 10 stratified randomly selected subsamples.

	2,4-D in mg/kg
sample #20740-1	35.877
sample #20740-2	36.613
sample #20740-3	36.140
sample #20740-4	36.714
sample #20740-5	35.329
sample #20740-6	36.294
sample #20740-7	36.771
sample #20740-8	36.070
sample #20740-9	36.836
sample #20740-10	35.921

Table 1: homogeneity test results of subsamples #20740

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.

	2,4-D in mg/kg
r (observed)	1.354
reference method	Horwitz
0.3 x R (reference method)	2.838

Table 2: evaluation of the repeatability of subsamples #20740

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

Also, a batch of off white cotton, made positive for the insecticide Deltamethrin by a third-party, was selected. This batch was used before in the PT of iis13A05 as sample #13233. The homogeneity was performed and approved in this PT, which was published in January 2014. After homogenization 38 subsamples of approximately 3 grams each were prepared from this batch and labelled #20741.

To each of the participating laboratories 1 sample labelled #20740 and 1 sample labelled #20741 was sent on November 17, 2020.

2.5 ANALYZES

The participants were requested to determine on both samples the concentrations of a limited number of prescribed pesticides (Chlorophenoxy Acids for #20740 and Pyrethroids for #20741), 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 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 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 evaluation.

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 appendix 1 and 2 of this report. The laboratories are represented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that did not report 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 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.

According to ISO5725 the original test results per determination were submitted subsequently to Dixon's, Grubbs' or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of 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 was not negligible and will be used to calculate z'-scores (see also 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 reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. The Kernel Density Graph is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation 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. In some cases, a reproducibility based on former iis proficiency tests could be used.

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.
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. Two participants did not report any test results and not all laboratories were able to report all analyzes requested. In total 14 laboratories reported 25 numerical test results. Observed were 4 outlying results, which is 16% 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 SAMPLE AND PER COMPONENT

In this section the reported test results are discussed per sample. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These methods are also in the table together with the reported test results. The abbreviations used in these tables are explained in appendix 5.

Unfortunately, a suitable reference test method, providing precision data, is not available for all determinations. For the tests, that have no available precision data, the calculated reproducibility was compared against the estimated reproducibility calculated with the Horwitz equation.

Sample #20740

2,4-D: The determination may be problematic at the level of 40 mg/kg. 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 combined with the PT uncertainty (see §3.3).

Sample #20741

Deltamethrin: The determination may be problematic at the level of 6 mg/kg. 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 combined with the PT uncertainty (see §3.3).

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the estimated target reproducibility 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 estimated target reproducibility are compared in the next tables.

Component	unit	n	average	2.8 * sd	R(target)
2,4-D	mg/kg	10	40.1	18.2	12.6

Table 3: reproducibility of pesticides in sample #20740

Component	unit	n	average	2.8 * sd	R(target)
Deltamethrin	mg/kg	11	6.13	5.63	2.98

Table 4: reproducibility of pesticides in sample #20741

Without further statistical calculations, it can be concluded that for the observed pesticides the group of participating laboratories may have difficulties with the analysis.

4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2020 WITH PREVIOUS PTS

	December 2020	December 2018	December 2016	November 2014	November 2013
Number of reporting laboratories	14	14	13	21	22
Number of test results	25	81	109	53	56
Number of statistical outliers	4	15	5	3	6
Percentage of statistical outliers	16%	19%	4.6%	5.7%	10.7%

Table 5: 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 2020	December 2018	December 2016	November 2014	2008 - 2013
Carbaryl	--	--	39%	--	52%
Cyhalothrin-lambda	--	--	--	--	35 - 45%
Cypermethrin (=Σ)	--	--	--	--	15 - 28%
2,4-D	16%	--	--	--	--
4,4'-DDD	--	--	--	29%	38%
Deltamethrin	33%	--	--	--	12 - 31%
Dimethoate	--	--	--	54%	35%
α/β-Endosulfan	--	18 - 34%	27 - 47%	--	15 - 33%
Esfenvalerate	--	--	--	--	22 - 42%
Fenvalerate	--	--	--	--	11 - 37%
Methoxychlor	--	--	--	35%	14 - 28%
Monocrotophos	--	--	--	--	38%
Parathion	--	--	61%	--	73%
Quinalphos	--	35 - 38%	32 - 52%	--	24 - 39%

Table 6: comparison of uncertainties in iis proficiency tests on pesticides in textile

The precision that was found for Deltamethrin was not improved during the present proficiency test. It was the first time that 2,4-D 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:
Nine of the fourteen reporting laboratories mentioned to be accredited for the determination of 2,4-D and eleven of fourteen reporting laboratories mentioned to be accredited for the determination of Deltamethrin according to ISO/IEC17025.

Nine participants used 1 or more grams as sample intake. The other participants used between 0.5 and 1 grams.
For the determination of 2,4-D seven laboratories used Ultrasonic for extraction and for Deltamethrin nine laboratories used Ultrasonic extraction. The other laboratories used a Soxhlet or an accelerated solvent extraction (ASE) or mechanical shaking. The extractions were done at different temperatures and for different lengths of time.
Five participants used Methanol as extraction solvent for the determination of 2,4-D. All other reporting participants used different extraction solvent combinations of Acetone, Acetonitrile and Hexane. For the determination of Deltamethrin, eight reporting participants used Acetone or Acetone combined with Hexane, Acetonitrile or Dichloromethane. The others used Acetonitrile, Methanol or Toluene.
The differences in analytical details did not appear to be of influence on the reported test results, except for the extraction method (see paragraph 5 Discussion).

5 DISCUSSION

Most participants used an in-house method. Therefore, some method details were requested on the report form. Looking at the extraction methods, it appears to be an effect for the determination of Deltamethrin with Ultrasonic extraction. When the laboratories that use Ultrasonic extraction are evaluated separately, the RSD of the group decreases from 33% to 22%. This effect is not seen with the evaluation of the results of the group with Ultrasonic extraction in the determination of 2,4-D. Since these pesticides are very different types of pesticides and structures, the method of extraction may be more or less of influence.

When the results of this interlaboratory study were compared to the standard 100 by OEKO-TEX® (see table 7) and Bluesign® Restricted Substances List (RSL) – Consumer Safety Limits (see table 8), it could be noticed that all laboratories, except two for the determination of 2,4-D, would reject 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 7: OEKO-TEX 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 8: Bluesign®

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

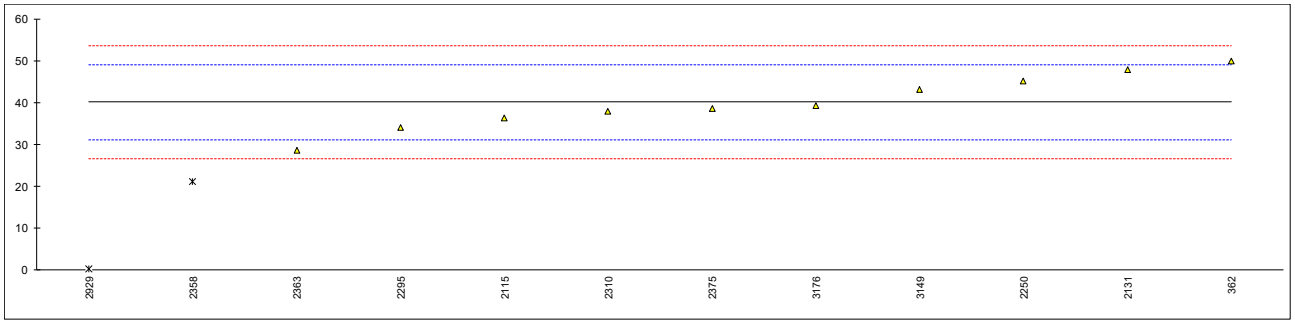
Finally, each laboratory has to evaluate its performance in this study and make decisions about necessary corrective actions. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus improve of the quality of the analytical results.

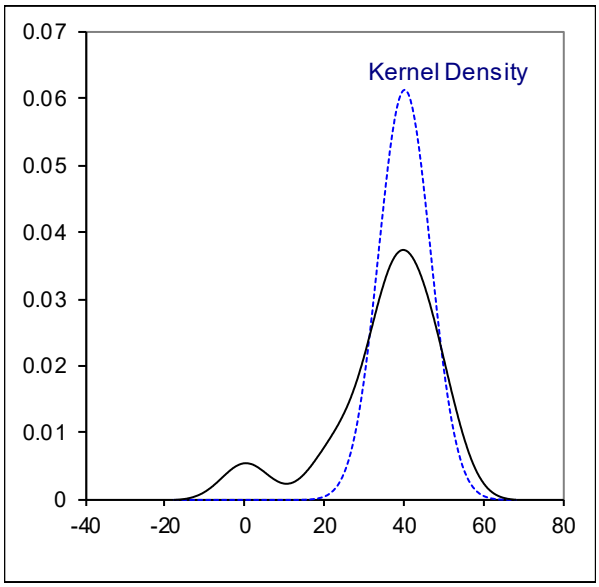
APPENDIX 1

Determination of Cholorophenoxy Acids, 2,4-D (CAS No. 94-75-7) on sample #20740; results in mg/kg

lab	method	value	mark	z'(targ)	remarks
339	EN15662	<0.05		<-8.91	possibly a false negative test result?
362	In house	50.0		2.21	
2115	In house	36.36		-0.83	
2131	In house	47.78		1.71	
2250	In house	45.2	C	1.14	first reported: <0.1
2295	In house	34		-1.35	
2310	In house	37.9		-0.49	
2358	In house	21.1066	G(0.05)	-4.23	
2363	EPA8081B	28.7		-2.53	
2375	In house	38.5		-0.35	
2386		-----		-----	
2390		-----		-----	
2929	In house	0.19	G(0.05)	-8.88	
2939		-----		-----	
3149	In house	43.2		0.69	
3176	In house	39.20		-0.20	

			Ultrasonic extraction only:	
normality	OK		OK	
n	10		6	
outliers	2		1	
mean (n)	40.0840		39.0450	
st.dev. (n)	6.51475	RSD = 16%	7.64166	RSD = 20%
R(calc.)	18.2413		21.3967	
st.dev.(Horwitz')	4.49135		5.30656	
R(Horwitz')	12.5758		14.8584	

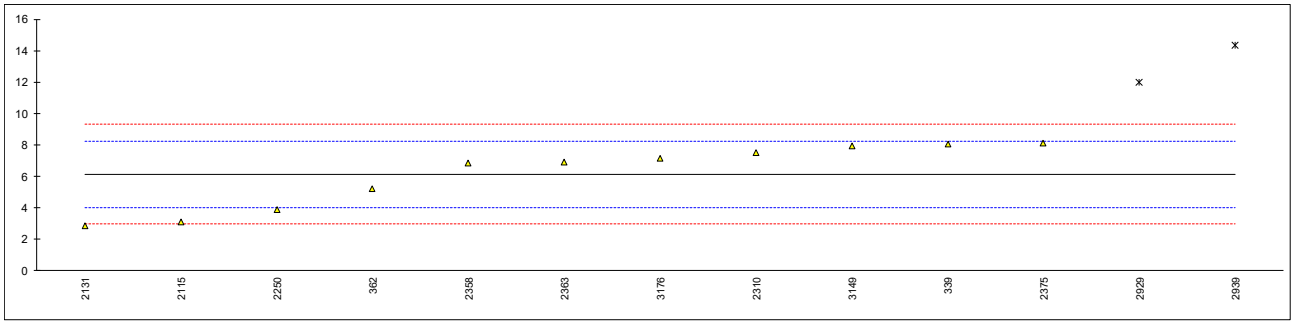


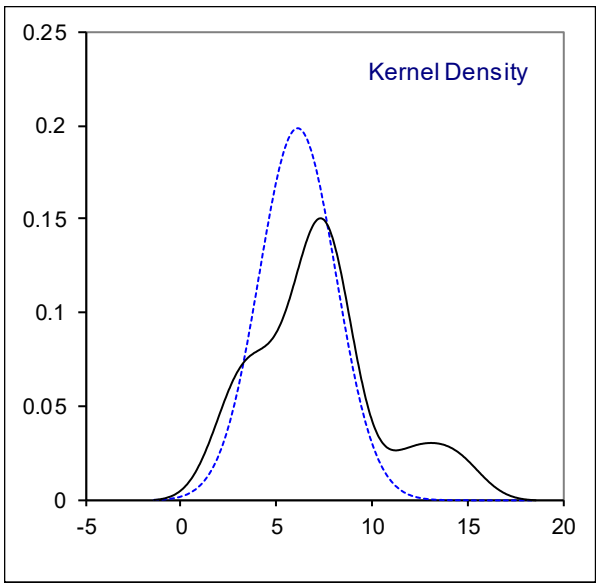


Determination of Pyrethroids, Deltamethrin (CAS No. 52981-63-5) on sample #20741; results in mg/kg

lab	method	value	mark	z'(targ)	remarks
339	EN15662	8.03		1.78	
362	In house	5.2		-0.88	
2115	In house	3.088		-2.86	
2131	In house	2.87		-3.07	
2250	In house	3.9		-2.10	
2295		-----		-----	
2310	In house	7.48		1.26	
2358	In house	6.8351		0.66	
2363	EPA8081B	6.9		0.72	
2375	In house	8.1		1.85	
2386		-----		-----	
2390		-----		-----	
2929	In house	12	G(0.05)	5.51	
2939	In house	14.35	C,G(0.05)	7.72	first reported: 1.435
3149	In house	7.91		1.67	
3176	In house	7.17		0.97	

				Ultrasonic extraction only:	
	normality	OK		OK	
	n	11		7	
	outliers	2		1	
	mean (n)	6.1348		6.5122	
	st.dev. (n)	2.00929	RSD = 33%	1.45434	RSD = 22%
	R(calc.)	5.6260		4.0721	
	st.dev.(Horwitz')	1.06374		1.04391	
	R(Horwitz')	2.9785		2.9230	





APPENDIX 2

Determination of Cholorophenoxy Acids, 2,4,5-T, MCPA, MCPB and Mecoprop on sample #20740; results in mg/kg

lab	2,4,5-T	MCPA	MCPB	Mecoprop	remarks
339	<0.05	<0.05	<0.05	<0.05	
362	-----	-----	-----	-----	
2115	< 0.05	not detected	< 0.05	< 0.05	
2131	not analyzed	not detected	not detected	not detected	
2250	<0,1	<0,1	<0,1	<0,1	
2295	-----	-----	-----	-----	
2310	Not Detected	Not Detected	Not Detected	Not Detected	
2358	n.d.	n.d.	n.d.	n.d.	
2363	<0.5	<0.5	<0.5	<0.5	
2375	-----	-----	-----	-----	
2386	-----	-----	-----	-----	
2390	-----	-----	-----	-----	
2929	0	0	0	0	
2939	-----	-----	-----	-----	
3149	-----	-----	-----	-----	
3176	-----	-----	-----	-----	

Determination of Pyrethroids, Cyfluthrin, Cyhalothrin, Cypermethrin, Esfenvalerate en Fenvalerate on sample #20741; results in mg/kg

lab	Cyfluthrin	Cyhalothrin	Cypermethrin	Esfenvalerate	Fenvalerate	remarks
339	<0.02	<0.02	0.0486	<0.02	<0.02	
362	-----	-----	0.087	-----	-----	
2115	not detected	0.0459	not detected	0.0482	not detected	
2131	not detected	not detected	not detected	not detected	not detected	
2250	-----	-----	-----	-----	-----	
2295	-----	-----	-----	-----	-----	
2310	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	
2358	n.d.	n.d.	n.d.	n.d.	n.d.	
2363	<0.2	<0.2	<0.2	<0.2	<0.2	
2375	-----	-----	-----	-----	-----	
2386	-----	-----	-----	-----	-----	
2390	-----	-----	-----	-----	-----	
2929	0	0.011	0.039	0.012	0.024	
2939	-----	-----	-----	-----	-----	
3149	-----	-----	-----	-----	-----	
3176	-----	-----	-----	-----	-----	

APPENDIX 3 Analytical Details

Cholorophenoxy Acids – sample #20740

Lab	ISO17025 accredited	Intake sample amount	Extraction type	Extraction solvent	Extraction time	Extraction temp.
339	No	Used as received	1	Mechanical Shaking + Centrifugation	H2O/Acetonitrile 50:50	30
362	Yes	Used as received	1g	Ultrasonic	Methanol	60
2115	Yes	Used as received	0.5 g	ASE	Methanol 100% (50 ml)	*
2131	Yes	Used as received	1.5g	Thermal Desorption	Quechers	30 min.
2250	Yes	Used as received	1 and 0,5	Ultrasonic	Dichlormethan: Aceton (1:1)	30 min
2295	Yes	Further cut	1 gram	Ultrasonic	Methanol	1 hr
2310	Yes	Further cut	1 gram	Ultrasonic	Methanol	One hour
2358	Yes	Used as received	1 g	Ultrasonic		1 hour
2363	No	Further cut	1g	Ultrasonic		60min
2375	Yes	Further cut	0,5 grams	Ultrasonic	Methanol	60 mins
2386	---	---		---		
2390	---	---		---		
2929	Yes	Used as received	1	Mechanical Shaking	Aceton/Acetonitrile	60
2939	---	---		---		
3149	No	Used as received	1g	---		
3176	No	Used as received	1	Soxhlet	Hexane/Acetone	360

*) Heat time: 5 minutes; Static Time: 10 minutes

Pyrethroids – sample #20741

Lab	ISO17025 accredited	Intake sample amount	Extraction type	Extraction solvent	Extraction time	Extraction temp.
339	No	Used as received	1	Mechanical shaking + centrifugation	H2O/Acetonitrile 50:50	30
362	Yes	Used as received	1g	Ultrasonic	Methanol	60
2115	Yes	Used as received	0.5 g	ASE	ACETONE 100% (50 ml)	*
2131	No	Used as received	1.5 g	300 rpm shaking for 30 minutes	Quechers	30 Min.
2250	Yes	Used as received	1 gram	Ultrasonic	Acetone/Dichloromethan (1:1)	2 x 30 min
2295	Yes	Further cut	1 gram	Ultrasonic	Methanol	1 hour
2310	Yes	Further cut	2 grams	Ultrasonic	Acetone:Hexane(1:1)	1 hour
2358	Yes	Used as received	1 g	Ultrasonic	/	1 hour
2363	Yes	Further cut	0.5g	Ultrasonic	actone:hexane=1:1	60min
2375	Yes	Further cut	0,5 grams	Ultrasonic	Hexane:Acetone (1:1)	60 mins
2386	---	---		---		
2390	---	---		---		
2929	Yes	Used as received	1	Mechanical Shaking	Aceton/Acetonitrile	60
2939	Yes	Used as received	0.7259	Ultrasonic	Acetone	30
3149	Yes	Further cut	1 g	Soxhlet	Aceton	300
3176	No	Used as received	1	Ultrasonic	Toluen	60

*) Heat time: 5 minutes; Static Time: 10 minutes

APPENDIX 4

Number of participants per country

1 lab in BULGARIA
1 lab in FRANCE
4 labs in GERMANY
1 lab in HONG KONG
1 lab in INDIA
1 lab in ITALY
1 lab in P.R. of CHINA
2 labs 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

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