Results of Proficiency Test Pesticides in Textile December 2012

Organised by: Institute for Interlaboratory Studies Spijkenisse, the Netherlands

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1 INTRODUCTION

Since the 1990's, 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 textiles, there are some Ecolabelling schemes imposing environmental requirements for textile products on a voluntary basis. Well-known programs are Milieukeur (the Netherlands) and Öko-Tex Standard 100 (Germany).

The Institute for Interlaboratory Studies organizes since 2004 a scheme of proficiency test for Pesticides in textile. As part of the annual proficiency test program 2012/2013, the institute decided to continue this proficiency test on Pesticides in Textile.

In this 2012 interlaboratory study 22 laboratories in 10 different countries participated. See appendix 4 for the number of participants per country.

In this report, the results of this proficiency test are presented and discussed.

2 SET UP

The Institute for Interlaboratory Studies in Spijkenisse was the organiser of this proficiency test. Sample preparation and analyses were subcontracted to an accredited laboratory. It was decided to use two different textile samples in this PT, both positive on a number of pesticides. The participants were requested to report rounded and unrounded results. The unrounded 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 guide 43, ILAC-G13:2007 and ISO/IEC 17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially 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 organisation 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 January 2010 (iis-protocol, version 3.2).

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

Two different textile samples, both positive on a number of pesticides, were prepared by a third party. Sample #12160 was a cotton fabric fortified with Cypermethrin, Monocrotophos and Fenvalerate. Sample #12161 was a cotton fabric fortified with Fenvalerate and Cyhalothrin-lambda. The two samples were each cut into pieces, well mixed and divided over 35 subsamples of 5 grams each. The samples were tested for homogeneity by determination of a pesticide in accordance with an in house test method on 4 stratified randomly selected samples. See the following tables for the test results:

	Cypermethrin in mg/kg
Sample #12160-1	188.6
Sample #12160-2	193.8
Sample #12160-3	184.5
Sample #12160-4	190.2

Table 1: homogeneity test results of sub samples #12160

	Fenvalerate in mg/kg
Sample #12161-1	2.34
Sample #12161-2	2.32
Sample #12161-3	2.42
Sample #12161-4	2.29

Table 2: homogeneity test results of sub samples #12161

From the above results of the homogeneity tests, the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibility of the reference method in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Cypermethrin in mg/kg	Fenvalerate in mg/kg
r (observed)	10.8	0.16
reference method	Horwitz	Horwitz
0.3 x R (reference method)	11.6	0.28

Table 3: repeatabilities of the sub samples #12160 and #12161

The calculated repeatabilities were less than 0.3 times the estimated reproducibility calculated using the Horwitz equation. Therefore, homogeneity of subsamples #12160 and #12161 was assumed.

In total approx. 5 grams of each of the samples #12160 and #12161 were sent to the participating laboratories on November 7, 2012.

2.5 ANALYSES

The participants were asked to determine the concentrations of a limited number of prescribed pesticides, applying the analytical procedure that is routinely used in the laboratory.

To get comparable results a detailed report form, was sent together with the set of samples. On the report forms the requested pesticides, including the units and questions about the analytical details, were pre-printed. In addition, a letter of instructions was sent along.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered. The original data are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder fax was sent to the laboratories that had not reported results at that moment. Shortly after the deadline, the available results were screened for suspect data. A 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 results. Additional or corrected results are used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. 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. After removal of outliers, this check was repeated. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.

In accordance to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs 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. When the uncertainty passed the evaluation no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

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

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of January 2010 (iisprotocol, version 3.2).

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 analysis results are plotted. The corresponding laboratory numbers are under 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 standard. 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 (see appendix 5, nos.15-16).

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 spread of this interlaboratory study. The target standard deviation was calculated from the target reproducibility (preferable from a standard method) by division with 2.8.

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

$$u_x = 1.23 * (st.dev (n)) / \sqrt{n}$$

In ISO13528 is stated that if $u_x \ge 0.3^*$ standard deviation for proficiency testing, the uncertainly of the assigned value is not negligible and need to be included in the interpretation of the results of the proficiency test. In the cases that the uncertainty is not negligible (see appendix 1) the z'-scores were calculated in stead of the usual z-scores.

The $z_{(target)}$ -scores were calculated in accordance with:

 $z_{(target)}$ = (result - average of PT) / target standard deviation

The z'_(target) were calculated in accordance with ISO13528 paragraph 7.6:

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

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 the fit-for-useness of the reported test result.

Absolute values for z<2 are very common and absolute values for z>3 are very rare. The evaluation of $z'_{(target)}$ is not different as for common z-scores and both are evaluated as follows:

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

4 EVALUATION

During the execution of this proficiency test no serious problems occurred. Four participants did not report any test results. Three other participants reported the test results after the final reporting date. The 18 participants reported 106 numerical test results. Observed were 16 statistical outlying results, which is 15.1% of the numerical results. In proficiency studies, outlier percentages of 3 % - 7.5 % are quite normal.

All data sets proved to have a normal distribution.

Due to the lack of relevant standard test methods for the determination of pesticides with precision data, the calculated reproducibilities were compared with the reproducibilities calculated using Horwitz, see also paragraph 5.

4.1 EVALUATION PER SAMPLE AND PESTICIDE

All statistical results reported on the textile samples are summarised in appendix 1 and relevant method information is summarized in appendix 3 and all other positively reported pesticide test results are listed in appendix 2.

Textile #12160:

<u>Cypermethrin</u>: The determination of this pesticide was very problematic at the level of 323 mg/kg. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the estimated target reproducibility (Horwitz').

- Esfenvalarate: The determination of this pesticide may be problematic at the low level of 0.95 mg/kg. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated target reproducibility (Horwitz').
- <u>Fenvalerate</u>: The determination of this pesticide may be very problematic at the low level of 1.63 mg/kg. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the estimated target reproducibility (Horwitz').
- <u>Monocrotophos</u>: The determination of this pesticide may be very problematic at the level of 5.76 mg/kg. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the estimated target reproducibility (Horwitz').

Textile #12161:

- Esfenvalarate: The determination of this pesticide may be problematic at the low level of 0.65 mg/kg. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated target reproducibility (Horwitz).
- <u>Fenvalerate</u>: The determination of this pesticide may be problematic for a number of laboratories at the low level of 1.2 mg/kg. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the estimated target reproducibility (Horwitz).
- Lambda-cyhalothrin: The determination of this pesticide may be very problematic at the level of 9.17 mg/kg. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the estimated target reproducibility (Horwitz').

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the very strict reproducibilities as estimated by the Horwitz equation and the reproducibilities as found for the group of participating laboratories.

The number of significant results, the average results, the calculated reproducibilities (standard deviation*2.8) and the target reproducibilities (estimated via the Horwitz equation), are compared in the next 2 tables.

Parameter	Unit	n	Average	2.8 * sd	R or R' (target)
Cypermethrin	mg/kg	14	323	253	103
Esfenvalerate	mg/kg	9	0.95	1.10	0.62
Fenvalerate	mg/kg	11	1.63	1.28	0.83
Monocrotophos	mg/kg	14	5.76	6.18	2.84

 Table 4: reproducibilities of pesticides in sample #12160

Parameter	Unit	n	Average	2.8 * sd	R or R' (target)
Esfenvalerate	mg/kg	9	0.65	0.40	0.31
Fenvalerate	mg/kg	10	1.18	0.44	0.52
Lambda-cyhalothrin	mg/kg	15	9.17	11.43	4.67

Table 5: reproducibilities of pesticides in sample #12161

Without further statistical calculations it can be concluded that for all determined pesticides the group of participating laboratories has difficulties with the analysis. See also the discussion in paragraphs 4.1 and 5.

5 DISCUSSION

When the results of this interlaboratory study were compared to the Ecolabelling Standards and Requirements for Textiles in EU (see table 6), it could be noticed that all of the reporting laboratories would make the same decision about the acceptability of the textiles for the determined parameters. All participants would reject the textiles.

Ecolabel	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: Ecolabelling Standards and Requirements for Textiles in EU

General

In this proficiency test for the determination of pesticides in textile, all the participants identified all spiked pesticides correctly. The spreads of the group regretfully could not be compared with the precision of a Standard Test Method because of the lack of a suitable test method with precision data.

The majority of the participants used in house methods and different standards to quantify the requested components (see appendix 3).

An explanation for the relative large spreads found may be the fact that the purity of available calibration standards may vary strongly. And it is to be expected that the test results of laboratories, that used identical calibrants, will be closer than the test results of laboratories that used a different calibrants. Significant different results for most components were found by two of the three participants that used a calibration standard from Accustandard.

During this proficiency test Fenvalerate was one of the positive components. One must keep in mind that Fenvalerate is a mixture of 4 isomers (R-R, S-S, S-R and R-S). One of the isomers is also known as Esfenvalerate (S-S isomer). So the concentration found for Esfenvalerate cannot be larger than the concentration found for Fenvalerate. Three laboratories reported a higher value for Esfenvalerate than for Fenvalerate which is in principle not possible.

The spreads that were found for the pesticides Monocrotophos, Esfenvalerate and Fenvalerate during the present proficiency test did improve, while the spreads of other pesticides did not improve in comparison with the spreads as observed in the previous rounds. The relative low number of participating laboratories may (partly) explain for the relatively large spreads.

	November	November	November	February	February	February	February
	2012	2011	2010	2010	2009	2008	2007
Carbaryl			146				
Cyhalothrin-	125		114			99	
lambda							
Cypermetrin (=Σ)	78			41			77
4,4-DDD			106-327				
Deltamethrin		33				104	
Dimethoate					98		
α/β-Endosulfan		75-93		41-55	58		59
Fenvalerate	37-78		32		66-103	90	
Esfenvalerate	62-115		116				
Malathion							
Methoxychlor		61	78			40	
Methylparathion							
Monocrotophos	107						207
Parathion			204				
Quinalfos		67-110		66			79-125

table 7: Comparison of relative standard deviations (RSDs in %) in iis proficiency tests

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 Cypermethrin on sample #12160; results in mg/kg

lab	method	value	mark	z'(targ)	remarks
2115	mounou		man	_ (tu: g/	Tomano
2129		350 218		0 75	
2139	in house	789.4	DG(0.05)	12.69	
2295	in house	14	DG(0.05)	-8.39	
2310	in house	251		-1.95	
2358	in house	271.4		-1.40	
2359	EPA8081B	255		-1.84	
2365	EPA8081B	272.8		-1.36	
2370	in house	250		-1.98	
2375	INH-210	269.36		-1.45	
2379					
2428	GB/T18412.1&3	417.50		2.58	
2492	OekoTex201	167.87		-4.21	
2493	DIN 100 407 0				
2508	DIN38407-2	396.94		2.02	
3117		324.0		0.03	
3140		400		4.41	
3205	in house	355	DG(0.05)	0.88	
3205	III IIOuse			0.00	
3220	in house	4.6	DG(0.05)	-8 65	
3242	in house	452 30	20(0.00)	3 52	
				0.0-	
	normality	OK			
	n	14			
	outliers	4			
	mean (n)	322.74			
	st.dev. (n)	90.463			
	R(calc.)	253.30			
	R(Horwitz)	60.61			
	R(Horwitz')	102.99			
	U(mean)	29.74			





Determination of Esfenvalerate on sample #12160; results in mg/kg

lab	method	value	mark	z'(targ)	remarks
2115					
2129		1.798		3.82	
2139	in house	3.4	DG(0.01)	11.05	
2295	in house	1.2		1.13	
2310	in house	0.82		-0.58	
2358	in house	0.7		-1.13	
2359	EPA8081B	0.85		-0.45	
2365	EPA8081B	1.09		0.63	
2370	in house	0.398		-2.49	
2375	INH-210	0.91		-0.18	
2379	00/740440400				
2428	GB/118412.1&3	n.d.			
2492	OekoTex201	<1.0			
2493	DIN 20407 0	4 1 2		14.24	
2000	DIN30407-2	4.13	DG(0.01)	14.54	
2146					
3140					
3205	in house	<01			
3218	III IIOUSE	<0.1			
3220	in house	n d			
3242	in house	0.78		-0.76	
0212	in nouce	0.10		0.10	
	normality	ОК			
	n	9			
	outliers	2			
	mean (n)	0.950			
	st.dev. (n)	0.3914			
	R(calc.)	1.096			
	R(Horwitz)	0.429			
	R(Horwitz')	0.621			
	U(mean)	0.160			
4.5 T					
4					x
2.5					
0.0					×





Determination of Fenvalerate on sample #12160; results in mg/kg

lab	method	value	mark	z'(targ)	remarks
2115					
2129		2.620	C,G(0.05)	3.34	First reported 3.391
2139	in house	2.4		2.59	
2295	in house	n.d.			
2310	in house	1.60		-0.11	
2358	in house	1.5		-0.45	
2359	EPA8081B	1.42		-0.72	
2365	EPA8081B	1.71		0.26	
2370	in house	1.21		-1.43	
2375	INH-210	0.99		-2.17	
2379					
2428	GB/118412.1&3	n.a.			
2492	Oekorexzur	<1.0			
2493	DIN20407 2	2.0		1.24	
2000	DIN30407-2	2.0		1.24	
3146		23	C	2 26	Reported as sum esfenvalerate + fenvalerate
3172		3 420	C G(0.05)	6.04	Reported as sum esfenvalerate + fenvalerate
3205	in house	17	C, C(0.00)	0.23	First reported 3.4
3218	in nouce		U U		
3220	in house	n.d.			
3242	in house	1.125		-1.71	
	normality	OK			
	n	11			
	outliers	2			
	mean (n)	1.632			
	st.dev. (n)	0.4575			
	R(calc.)	1.281			
	R(Horwitz)	0.679			
	R(Horwitz')	0.829			
	U(mean)	0.170			



Determination of Monocrotophos on sample #12160; results in mg/kg

lah	mathad	value	mark	='/tora)	romarka
0115	metriod	value	IIIdIK	z (lary)	Tellidiks
2110		7.24		1 46	
2129	in house	7.24		3 20	
2109	in house	9.0 14.5	C(0.05)	9.63	
2295	in house	5 50	G(0.05)	-0.25	
2358	in house	5.50		-0.25	
2350	EPA8081B	5 33		-0.00	
2365	EP48081B	5.66		-0.42	
2370	in house	7 51		1 73	
2375	INH-210	2.06		-3.65	
2379	111210				
2428	GB/T18412 1&3	9 14		3 34	
2492	OekoTex201	4.06		-1.68	
2493					
2508	DIN38407-2	6.20		0.44	
3117					
3146	INH-06	5.8		0.04	
3172	EPA8081A/GBT18412.1	0	ex	-5.68	Result excluded, zero is not a real result
3205	in house	6.0	С	0.24	First reported 14.6
3218					•
3220	in house	n.d.			
3242	in house	1.405		-4.30	
	normality	OK			
	n	14			
	outliers	2			
	mean (n)	5.758			
	st.dev. (n)	2.2056			
	R(calc.)	6.176			
	R(Horwitz)	1.982			
	R(Horwitz')	2.837			
	U(mean)	0.700			



Determination of Esfenvalerate on sample #12161; results in mg/kg

lab	method	value	mark	z(targ)	remarks
2115					
2129		0.728		0.69	
2139	in house	2.9	DG(0.01)	20.22	
2295	in house	0.62	. ,	-0.28	
2310	in house	0.54		-1.00	
2358	in house	0.6		-0.46	
2359	EPA8081B	0.80		1.34	
2365	EPA8081B	0.88		2.05	
2370	in house	0.401		-2.25	
2375	INH-210	0.59		-0.55	
2379					
2428	GB/T18412.1&3	n.d.			
2492	OekoTex201	<1.0			
2493					
2508	DIN38407-2	2.78	DG(0.01)	19.14	
3117			()		
3146					
3172					
3205	in house	<0.1		<-4.97	False negative?
3218					-
3220	in house	n.d.			
3242	in house	0.705		0.48	
	normality	OK			
	n	9			
	outliers	2			
	mean (n)	0.652			
	st.dev. (n)	0.1439			
	R(calc.)	0.403			
	R(Horwitz)	0.311			



Determination of Fenvalerate on sample #12161; results in mg/kg

lab	method	value	mark	z(targ)	remarks
2115					
2129		2.344	C,DG(0.01)	6.33	First reported 2.163
2139	in house	2.6	DG(0.01)	7.72	
2295	in house	n.d.			
2310	in house	1.10		-0.43	
2358	in house	1.2		0.11	
2359	EPA8081B	1.10		-0.43	
2365	EPA8081B	1.26		0.44	
2370	in house	1.24		0.33	
2375	INH-210	1.06		-0.65	
2379					
2428	GB/T18412.1&3	n.d.			
2492	OekoTex201	<1.0			
2493					
2508	DIN38407-2	1.32		0.76	
3117			0.000.00		
3146		2	C,G(0.01)	4.46	Reported as sum estenvalerate + tenvalerate
3172		0.85	C	-1.79	Reported as sum estenvalerate + tenvalerate
3205	in house	1.4	C	1.20	First reported 3.6
3218	in house				
3220	in house	n.a. 1 265		0.46	
3242	in nouse	1.205		0.46	
	normality	ОК			
	n	10			
	outliers	3			
	mean (n)	1.179			
	st.dev. (n)	0.1570			
	R(calc.)	0.439			
	R(Horwitz)	0.515			





Determination of Lambda-cyhalothrin on sample #12161; results in mg/kg

lah	method	valuo	mark	z'(targ)	remarks
2115	method	value	IIIdi K	2 (tary)	Telliarks
2129		11 32		1 29	
2139	in house	35.1	G(0.01)	15.54	
2295	in house	0.35	0(0.01)	-5.28	
2310	in house	8.52		-0.39	
2358	in house	8.1		-0.64	
2359	EPA8081B	7.63		-0.92	
2365	EPA8081B	7.47		-1.02	
2370	in house	9.77		0.36	
2375	INH-210	6.61		-1.53	
2379					
2428	GB/T18412.1&3	15.66		3.89	
2492	OekoTex201	6.55		-1.57	
2493					
2508	DIN38407-2	10.58		0.85	
3117	10111.000	17.6		5.06	
3140		11.2		1.22	
2205	EFA0001A/GB110412.1	24.220	G(0.05)	9.02	Eirst reported 22.6
3200	ITTIOUSE	0.0	C	-1.90	First reported 23.0
3220	in house	n d			
3242	in house	10.12		0.57	
0212	in nouce	10.12		0.07	
	normality	OK			
	n	15			
	outliers	2			
	mean (n)	9.165			
	st.dev. (n)	4.0819			
	R(calc.)	11.429			
	R(Horwitz)	2.942			
	R(Horwitz')	4.672			
	U(mean)	1.296			



APPENDIX 2

Summary of all other reported pesticides; results in mg/kg

	#12160						
lab	Deltametrin	Cyhalotrin	λ-cyhalotrin	Parathion	Quinalphos		
2115							
2129	<0.1		<0.1	<0.1	0.427		
2139					0.2		
2295					0.17		
2310							
2358							
2359							
2365	<0.05		<0.2	<0.2	<0.2		
2370							
2375							
2379							
2428							
2492	<1.0		<1.0	<1.0	<1.0		
2493							
2508	0.0	0.0	0.0	0.0	0.0		
3117							
3146	<0.5	<0.5	<0.5	<0.5	<0.5		
3172		0.130					
3205	<0.1	<0.1	<0.1	<0.05	0.21		
3218							
3220							
3242					0.22		

	#12161							
lab	Cypermetrin	Deltametrin	Cyhalotrin	Monocroptos	Parathion	Quinalphos		
2115								
2129	<0.1	<0.1		<0.1	<0.1	<0.1		
2139						0.1		
2295								
2310								
2358								
2359								
2365	<0.05	<0.05		<0.2	<0.2	<0.2		
2370								
2375								
2379								
2428								
2492	<1.0	<1.0		<1.0	<1.0	<1.0		
2493								
2508	0.0	0.0	0.0	0.0	0.0	0.0		
3117								
3146	<0.5	<0.5	1.0	<0.5	<0.5	<0.5		
3172			0.857					
3205	<0.1	<0.1	<0.1	<0.1	<0.05	0.05		
3218								
3220								
3242								

APPENDIX 3 Details of the methods used by the participants:

212b Fervalerate Dr. Ehrensdorfer single 00306 Esterwalerate Dr. Ehrensdorfer single 80429 Lambda-cyhalothrin Fluka single 10912 Cypermethrin Inst.for org. Ind. Chem miture No 1Fr10 2139 Fervalerate Accustd Single Lot 21201114 Esfemvalerate Accustd Single Lot 21201135 Lambda-cyhalothrin Accustd Single Lot 21201135 Cypermethrin Accustd Single Lot 21201028-01 23205 Cypermethrin Accustd Single Lot 212071028-01 23301 Fervalerate Accustd Single Lot 212071028-01 23305 Fervalerate Chem service PS1032 Lambda-cyhalothrin Accustd Single PS1032 Lambda-cyhalothrin Accustd Single PS1032 PS1032 Lambda-cyhalothrin Chem service PS2018 Monocrotophos Chem service Single 446-15A Lambda-cyhalothrin<	<u>Lab</u> 2115	<u>component</u>	Brand name	composition	Batch
Efenvalerate Dr. Enrensdorfer single 80429 Lambda-cyhalothrin Filka single STE8038X Cypermethrin Inst.of org. Ind. Chem mikure No. 1Fr/10 2138 Fenvalerate Accustd Single Lot 21211148 Ederwalerate Accustd Single Lot 21201215 Lambda-cyhalothrin Accustd Single Lot 21201205-01 Monocotophos Accustd Single Lot 21201205-01 Z00 Cypermethrin Accustd Single Lot 21201205-01 2301 Forvalerate Accustd Single Lot 21201205-01 2310 Forvalerate Accustd Single Lot 21201205-01 2311 Forvalerate Accustd Single Particity 2104104-05 2323 Forvalerate Accustd Single Particity 2104104-05 2324 Lambda-cyhalothrin Chem service Mikure 446-15A 2325 Fervalerate Chem service Mikure 446-15A 2325	2129	Fenvalerate	Dr. Ehrensdorfer	single	00308
Lambda-cyhalothrin Fuka single STE8038X Monocrotophos Dr. Ehrensofrer single 10912 Cypermethrin Inst.of org. Ind. Chem mikture No117/10 2139 Ferwalerate Accustd Single Lot 21211143 Lambda-cyhalothrin Accustd Single Lot 212001520-01 Monocrotophos Accustd Single Lot 212001520-01 2200 Cypermethrin Accustd Single Lot 212001520-01 2301 Ferwalerate Accustd Single Lot 21200120-01 2303 Ferwalerate Accustd Single Proceeding 2304 Lambda-cyhalothrin Accustd Single Proceeding 2305 Ferwalerate Chem service PS1032 Proceeding 2305 Ferwalerate Chem service Monocrotophos Chem service PS108 2304 Cypermethrin Chem service single 440-5A 2305 Ferwalerate Chem service single 50014A <td></td> <td>Esfenvalerate</td> <td>Dr. Ehrensdorfer</td> <td>single</td> <td>80429</td>		Esfenvalerate	Dr. Ehrensdorfer	single	80429
Monocrotophos Dr. Ehrensdorfer single 10912 Cypermethnin Inst.of org. Ind. Chem mixture No/ 1F/10 2139 Fervalerate Accustd Single Lot 212011143 Esfenvalerate Accustd Single Lot 21201215 Lambda-cyhalothrin Accustd Single Lot 21041054-05 Cypermethnin Accustd Single Lot 212071028-01 2205 A A A 2310 Fervalerate Accustd Single Lot 212071028-01 2326 Amocrotophos Accustd Single A Cypermethnin Accustd Single P A Cypermethnin Accustd Single P PS032 Lambda-cyhalothrin Chem service Monocrotophos Chem service PS008 Cypermethnin Chem service Single 446-15A Lambda-cyhalothrin Chem service Single 446-15A Lambda-cyhalothrin Chem service Single 90917 <		Lambda-cyhalothrin	Fluka	single	STE8038X
CypermethrinInst.of org. Ind. ChemmixtureNo/ 1Fr/102139FervalerateAccustdSingleLot 21201215EstervalerateAccustdSingleLot 212061215MonocrotophosAccustdSingleLot 212061350-01MonocrotophosAccustdSingleLot 212001250-01CypermethrinAccustdSingleLot 212071028-012280ErravalerateAccustdSingleLot 212071028-012310FenvalerateAccustdSingleTerravelMonocrotophosAccustdSingleTerravelCypermethrinAccustdSinglePS10322381FenvalerateChem servicePS1032MonocrotophosChem servicePS10323393FenvalerateChem serviceMixtureMonocrotophosChem serviceSingle446-14A3304Eambda-cyhaldthrinChem serviceSingle446-145Chem serviceSingle446-14A440-045AChem serviceSingle446-14A440-045AChem serviceSingle99172305FervalerateDr. EhrensdorferSingle909172306Chem serviceSingle90362307Chem serviceSingle90362308FervalerateDr. EhrensdorferSingle90362309Dr. EhrensdorferSingle90362309Dr. EhrensdorferSingle90362309Dr. EhrensdorferSingle <td></td> <td>Monocrotophos</td> <td>Dr. Ehrensdorfer</td> <td>single</td> <td>10912</td>		Monocrotophos	Dr. Ehrensdorfer	single	10912
2139 Fervalerate Accustd Single Lot 212011148 Esfervalerate Accustd Single Lot 2120611350-01 Lambda-cyhalothrin Accustd Single Lot 212061350-01 Qypermethrin Accustd Single Lot 212071028-01 2205		Cypermethrin	Inst.of org. Ind. Chem	mixture	No/ 1F/10
Image: start startAccustSingleLot 212081215Lambda-cyhalothrinAccustdSingleLot 21041054-05CypermethrinAccustdSingleLot 211041054-052296AccustdSingleLot 212071028-012310FervalerateAccustdSingleImage: start st	2139	Fenvalerate	Accustd	Single	Lot 212111148
Lambda-cyhalothrin Accustd Single Lot 212061350-01 Moncorotopnos Accustd Single Lot 212071028-01 2295 Cypermethrin Accustd Single Lot 212071028-01 2295 Accustd Single Lambda-cyhalothrin Accustd Single Image: Accustd Im		Esfenvalerate	Accustd	Single	Lot 212081215
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Cypermethrin Accustd Single Lot 212071028-01 2295 Image: Construction of the service Single Image: Construction of the service 2310 Fervalerate Accustd Single Image: Construction of the service 2368 Fervalerate Chem service PS1032 2378 Fervalerate Chem service PS2018 Monocrotophos Chem service PS2018 Monocrotophos Chem service PS1032 Cypermethrin Chem service PS1068 Cypermethrin Chem service Mixture 446-15A Lambda-cyhalothrin Chem service single 445-149A Monocrotophos Chem service Mixture 440-85A 2355 Esfenvalerate Dr. Ehrensdorfer single 80917 Fervalerate Dr. Ehrensdorfer single 90506 Monocrotophos Dr. Ehrensdorfer single 10912 Cypermethrin Dr. Ehrensdorfer single 420-85A 2370 Esferovalerate <td< td=""><td></td><td>Monocrotophos</td><td>Accustd</td><td>Single</td><td>Lot 211041054-05</td></td<>		Monocrotophos	Accustd	Single	Lot 211041054-05
2285		Cypermethrin	Accustd	Single	Lot 212071028-01
2310 Ferwalerate Accustd Single Lambda-cyhalothrin Accustd Single Cypermethrin Accustd Single 2358 Ferwalerate Chem service PS1032 Lambda-cyhalothrin Chem service PS2018 Monocrotophos Chem service PS509 Cypermethrin Chem service PS1088 2359 Ferwalerate Chem service PS1088 2369 Ferwalerate Chem service Single 446-54A Monocrotophos Chem service single 446-54A Monocrotophos Chem service single 2365 Esferwalerate Dr. Ehrensdorfer single 6moveratephos Dr. Ehrensdorfer single 90506 Monocrotophos Dr. Ehrensdorfer single 10912 Cypermethrin Dr. Ehrensdorfer single 10912 Copermethrin Dr. Ehrensdorfer single 10912 Copermethrin Chem service Mixture S288234VV Lambda-cyhalothrin Dr. Ehrensdorfer single 10912 Copermethrin Dr. Ehrensdorfer Single 3288234VV Lambda-cyhalothrin Fluka Mixture <t< td=""><td>2295</td><td></td><td></td><td></td><td></td></t<>	2295				
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Lambda-cyhalothrinDr. Ehrensdorfersingle90506MonocrotophosDr. Ehrensdorfersingle10912CypermethrinChem serviceMixture440-85A2370EsfenvalerateFlukaMixtureSZB8234XVFenvalerateFlukaMixtureSZB8308XVLambda-cyhalothrinFlukamixtureSZB8200X2375CypermethrinDr. Ehrensdorfer70417EsfenvalerateDr. EhrensdorferSingle804292379Dr. EhrensdorferMix of 1220326CYOrganophosphorusDr. EhrensdorferMix of 3010118TO2492Dr. EhrensdorferMix of 3010118TO2493Dr. EhrensdorferMix of 3010118TO2493Dr. Ehrensdorfermixture10118TO2493Dr. Ehrensdorfermixture10118TO2493Dr. Ehrensdorfermixture10118TO2508Pest mix 15 + 34Neochemamixture31170Dr. EhrensdorfermixtureAll in one, selfmade3172CypermethrinAccustdSingleLot 212021183-01Lambda-cyhalothrinAccustdSingle00914IOMix 192Dr. Ehrensdorfersingle00914IOMix 195Dr. EhrensdorferMixure010111O3218Dr. EhrensdorferMixure01011IO3242Dr. EhrensdorferMixure01011IO		Fenvalerate	Dr. Ehrensdorfer	Mixture	00308
MonocrotophosDr. Ehrensdorfersingle10912CypermethrinChem serviceMixture440-85A2370EsfenvalerateFlukaMixtureSZB8234XVFenvalerateFlukaMixtureSZB8308XVLambda-cyhalothrinFlukamixtureSZB8200X2375CypermethrinDr. Ehrensdorfer70417EsfenvalerateDr. EhrensdorferSingle8042923792428PytrethroidDr. EhrensdorferMix of 1220326CYOrganophosphorusDr. EhrensdorferMix of 3010118TO2492 </td <td></td> <td>Lambda-cyhalothrin</td> <td>Dr. Ehrensdorfer</td> <td>single</td> <td>90506</td>		Lambda-cyhalothrin	Dr. Ehrensdorfer	single	90506
CypermethrinChem serviceMixture440-85A2370EsfenvalerateFlukaMixtureSZB8234XVFenvalerateFlukaMixtureSZB8200XLambda-cyhalothrinFlukamixtureSZB8200X2375CypermethrinDr. Ehrensdorfer70417EsfenvalerateDr. EhrensdorferSingle8042923792428PytrethroidDr. EhrensdorferMix of 1220326CYOrganophosphorusDr. EhrensdorferMix of 3010118TO2492 </td <td></td> <td>Monocrotophos</td> <td>Dr. Ehrensdorfer</td> <td>single</td> <td>10912</td>		Monocrotophos	Dr. Ehrensdorfer	single	10912
2370EsfenvalerateFlukaMixtureSZB8234XVFenvalerateFlukaMixtureSZB8308XVLambda-cyhalothrinFlukamixtureSZBA200X2375CypermethrinDr. EhrensdorfermixtureSZBA200X2379Dr. EhrensdorferSingle804292379Dr. EhrensdorferMix of 1220326CYOrganophosphorusDr. EhrensdorferMix of 3010118TO2492Dr. EhrensdorferMix of 3010118TO2493Neochemamixture10118TO2508Pest mix 15 + 34Neochemamixture3117Dr. EhrensdorfermixtureAll in one, selfmade3117Dr. EhrensdorferSingleLot 212021183-013146Dr. EhrensdorferSingleLot 212021183-013172CypermethrinAccustdSingleLot 212041156-01FenvalerateDr. EhrensdorferMixure104201OMix 192Dr. EhrensdorferMixure104201OMix 195Dr. EhrensdorferMixure010111O3218Dr. EhrensdorferMixure010111O3220Dr. EhrensdorferMixure010111O3242Dr. EhrensdorferMixure010111O		Cypermethrin	Chem service	Mixture	440-85A
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3218 Dr. Ehrensdorfer Mixture Of Office 3220 Dr. Ehrensdorfer		Mix 192		Mixture	0101110
3220 Dr. Ehrensdorfer 3242	3218				
3242	3220		Dr. Ehrensdorfer		
	3242				

APPENDIX 4

Number of participants per country

4 labs in GERMANY 2 labs in HONG KONG

- 1 lab in HUNGARY
- 3 labs in INDIA
- 2 labs in ITALY
- 1 lab in KOREA
- 5 labs in P.R. of CHINA
- 1 lab in TAIWAN R.O.C.
- 1 lab in THAILAND
- 2 labs in TURKEY

APPENDIX 5

Abbreviations:

- C = final result after checking of first reported suspect 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
- n.a. = not applicable
- n.d. = not detected
- W = withdrawn on request of the participant
- fr. = first reported

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