Institute for
Interlaboratory Studies

Results of Proficiency Test
Pesticides in Textile
December 2023

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: Mrs. A. Ouwerkerk, BSc
Correctors: Mr. R.J. Starink, BSc & Mrs. G.A. Oosterlaken-Buijs, BSc
Approved by: Mr. R.J. Starink, BSc

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CONTENTS

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 2023 it was decided to continue the proficiency test for the determination of Pesticides in Textile.

In this interlaboratory study 11 laboratories in 5 countries registered for participation, see appendix 4 for the number of participants per country. In this report the results of the Pesticides in Textile proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to a laboratory that has performed the tests in accordance with for ISO/IEC17043 relevant requirements of ISO/IEC17025.
It was decided to send one textile sample of approximately 3 grams labelled #23791.
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 blue cotton was selected, which was artificially fortified with some Pesticides. After homogenization 30 small plastic bags were filled with approximately 3 grams each and labelled #23791.
The batch for sample #23791 was used in a previous proficiency test on Pesticides in Textile as sample #12160 in iis12A05. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one textile sample labelled #23791 was sent on November 15, 2023.

2.5 ANALYZES

The participants were requested to determine the concentrations of a limited number of prescribed pesticides.
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 sample as if it was a routine sample and to report the test results using the indicated units on the report form and not to round the results, but report as much significant figures as possible. It was also requested not to report ‘less than’ test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per determination in 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 the criterion of ISO13528, paragraph 9.2.1, was met for all evaluated tests. Therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

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

3.2 GRAPHICS

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

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

3.3 Z-SCORES

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

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

The $z_{(target)}$ scores are listed in the test result tables in appendix 1.

Absolute values for z<2 are very common and absolute values for z>3 are very rare. Therefore, the usual interpretation of z-scores is as follows:

	$\lvert z \rvert$	< 1	good
1 <	$\lvert z \rvert$	< 2	satisfactory
2 <	$\lvert z \rvert$	< 3	questionable
3 <	$\lvert z \rvert$		unsatisfactory

4 EVALUATION

In this proficiency test no problems were encountered with the dispatch of the samples.
Three participants did not report any test results and not all participants were able to report all tests requested.
In total 8 participants reported 30 numerical test results. No outlying test results were observed. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

None of the data sets proved to have a normal Gaussian distribution. These are referred to as “unknown”. The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1 EVALUATION PER COMPONENT

In this section the reported test results are discussed per component. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the table together with the original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 5.

Unfortunately, a suitable reference test method, providing the precision data, is not available for the determinations of Pesticides in Textile.
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). 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.

Cypermethrin: The group of participants met the target requirements. No statistical outliers were observed. The calculated reproducibility is in agreement with the target reproducibility as derived from iis memo 2302.

alpha-Endosulfan I: The group of participants met the target requirements. No statistical outliers were observed. The calculated reproducibility is in agreement with the target reproducibility as derived from iis memo 2302.

beta-Endosulfan II: The group of participants may have had difficulty to meet the target requirements. No statistical outliers were observed. The calculated reproducibility is not in agreement with the target reproducibility as derived from iis memo 2302.

Esfenvalerate: The group of participants met the target requirements. No statistical outliers were observed. The calculated reproducibility is in agreement with the target reproducibility as derived from iis memo 2302.

Fenvalerate: The group of participants met the target requirements. No statistical outliers were observed. The calculated reproducibility is in agreement with the target reproducibility as derived from iis memo 2302.

The participants agreed on a concentration near or below the limit of detection for all other components mentioned in paragraph 2.5. Therefore, no z-scores are calculated for these components. The reported test results are given in appendix 2.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from reference methods are presented in the next table.

Component	unit	n	average	2.8 * sd	R(target)
Cypermetrin	mg/kg	7	284	167	263
alpha-Endosulfan I	mg/kg	5	0.43	0.25	0.40
beta-Endosulfan II	mg/kg	6	0.29	0.39	0.27
Esfenvalerate	mg/kg	6	0.97	0.95	0.89
Fenvalerate	mg/kg	6	1.48	1.20	1.37

Table 1: reproducibilities of tests on sample #23791

Without further statistical calculations it can be concluded that for many 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 2023 WITH PREVIOUS PTS

	December 2023	December 2022	December 2021	December 2020	December 2018
Number of reporting laboratories	8	12	11	14	14
Number of test results	30	45	11	25	81
Number of statistical outliers	0	5	1	4	15
Percentage of statistical outliers	0%	11%	9.1%	16%	19%

Table 2: 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 2023	December 2022	December 2021	December 2020	2007 - 2018
Aldicarb	--	23%	--	--	--
Carbaryl	--	--	--	--	39-52%
Cyhalothrin-lambda	--	--	--	--	35-45%
Cypermethrin	21%	--	--	--	15-28%
2,4-D	--	12%	--	16%	--
4,4'-DDD	--	--	--	--	29-38%
Dichlorprop	--	--	16%	--	--
Deltamethrin	--	--	--	33%	12-31%
Dimethoate	--	--	--	--	35-54%
α/β-Endosulfan	20-49%	14-29%	--	--	15-47%
Esfenvalerate	35%	--	--	--	22-42%
Fenvalerate	29%	--	--	--	11-37%
Methoxychlor	--	--	--	--	14-35%
Monocrotophos	--	--	--	--	38-74%
Parathion	--	--	--	--	61-73%
Quinalphos	--	--	--	--	24-52%

Table 3: development of the uncertainties over the years

The uncertainties observed in this PT are comparable to the uncertainties observed in previous PTs.

Sample #23791 was used in a previous PT as sample #12160 in iis12A05. The averages and calculated reproducibilities for this sample are similar for Esfenvalerate and Fenvalerate in both PTs and is for Cypermethrin in the 2023PT lower compared to the 2012PT. Monocrotophos was detected in the 2012PT but not in the 2023PT. The components alpha-Endosulfan I and,beta-Endosulfan II were only requested in the 2023PT.

		sample #23791			sample #12160		
Component	unit	n	average	R(calc)	n	average	R(calc)
Cypermethrin	mg/kg	7	284	167	14	323	253
Esfenvalerate	mg/kg	6	0.97	0.95	9	0.95	1.10
Fenvalerate	mg/kg	6	1.48	1.20	11	1.63	1.28

Table 4: comparison of sample #23791 with #18645

4.4 EVALUATION OF THE ANALYTICAL DETAILS

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

- Four participants mentioned to be accredited in according with ISO/IEC17025 to determine the reported component(s).
- Prior to analysis the sample was further cut by three participants and five participants used the sample as received.

- Six participants reported to use 0.5 gram as sample intake and two participants to use respectively 1.5 grams and 3 grams.
- Six participants reported to use Ultrasonic for extraction and the other laboratories reported either ASE or Soxhlet technique.
- The participants used an Acetone (mixture) or Methanol as extraction solvent.
- The extraction time varied from 60 minutes to 240 minutes. A majority (5 participants) used an extraction/release time of 60 minutes.
- The extraction temperature varied from 50 to 80 degrees Celsius.

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 5) and Bluesign® Restricted Substances List (RSL) Consumer Safety Limits (see table 6) it could be noticed that all laboratories would have made the same decision and would have rejected the sample.

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

Table 5: 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 6: 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

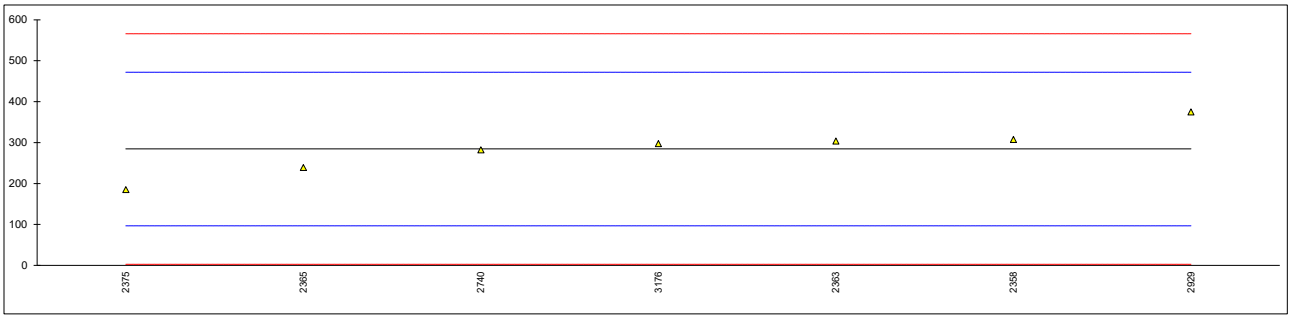
Although it can be concluded that the participants have no problem with the determination on the requested components in this PT, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular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

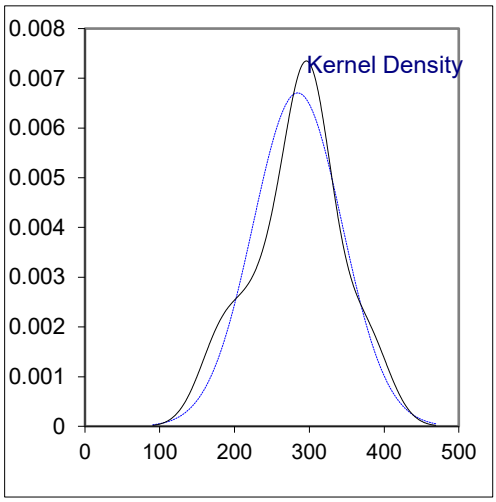
APPENDIX 1

Determination of Cypermetrin (CAS No. 52315-07-8) on sample #23791; results in mg/kg

lab	method	value	mark	z(targ)	remarks
2115		-----		-----	
2265		-----		-----	
2358	In house	308		0.25	
2363	In house	304		0.21	
2365	In house	239.1		-0.48	
2375	In house	185		-1.06	
2386		-----		-----	
2740	In house	282.52		-0.02	
2929	In house	375		0.96	
2977		-----		-----	
3176		297.5	C	0.14	First reported 47.60

normality	unknown
n	7
outliers	0
mean (n)	284.45
st.dev. (n)	59.522 RSD=21%
R(calc.)	166.66
st.dev.(iis memo 2302)	93.867
R(iis memo 2302)	262.83

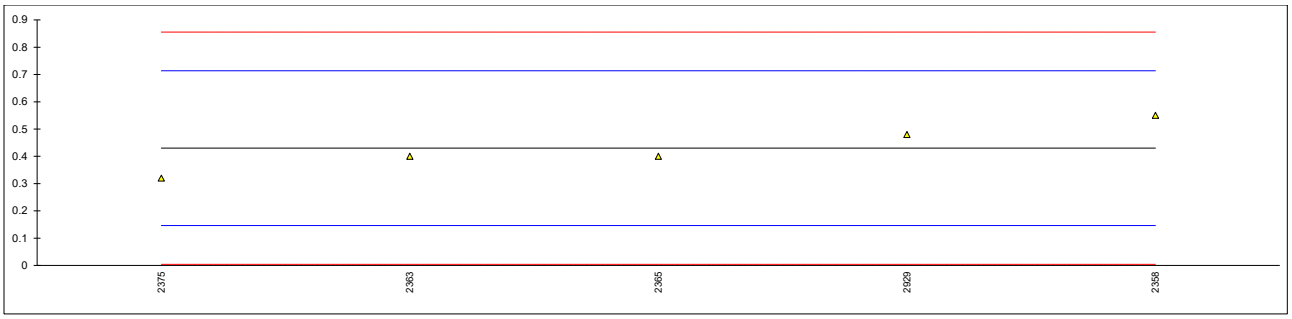




Determination of alpha-Endosulfan I (CAS No. 959-98-8) on sample #23791; results in mg/kg

lab	method	value	mark	z(targ)	remarks
2115		-----		-----	
2265		-----		-----	
2358	In house	0.55		0.85	
2363	In house	0.4		-0.21	
2365	In house	0.4		-0.21	
2375	In house	0.32		-0.78	
2386		-----		-----	
2740		-----		-----	
2929	In house	0.48		0.35	
2977		-----		-----	
3176		-----		-----	

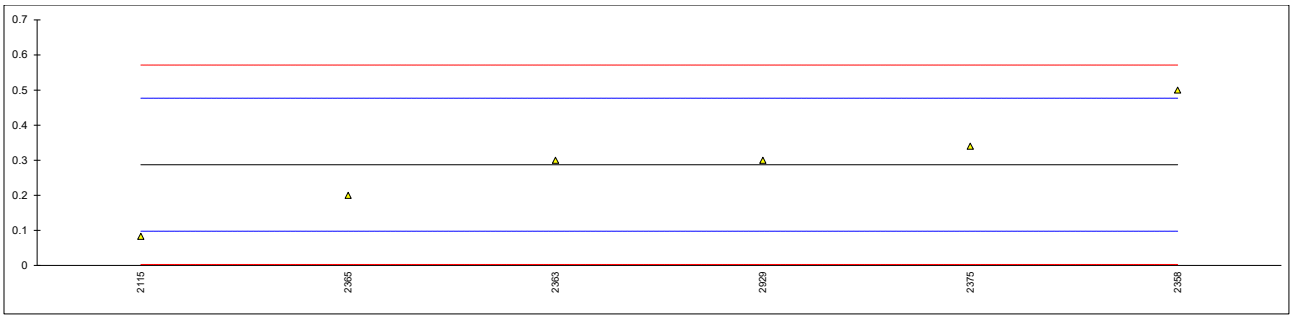
normality	unknown
n	5
outliers	0
mean (n)	0.430
st.dev. (n)	0.0877 RSD=20%
R(calc.)	0.246
st.dev.(iis memo 2302)	0.1419
R(iis memo 2302)	0.397



Determination of beta-Endosulfan II (CAS No. 33213-65-9) on sample #23791; results in mg/kg

lab	method	value	mark	z(targ)	remarks
2115	In house	0.083		-2.15	
2265		-----		-----	
2358	In house	0.50		2.25	
2363	In house	0.3		0.14	
2365	In house	0.2		-0.92	
2375	In house	0.34		0.56	
2386		-----		-----	
2740		-----		-----	
2929	In house	0.30		0.14	
2977		-----		-----	
3176		-----		-----	

normality	unknown	
n	6	
outliers	0	
mean (n)	0.287	
st.dev. (n)	0.1398	RSD=49%
R(calc.)	0.391	
st.dev.(iis memo 2302)	0.0948	
R(iis memo 2302)	0.265	

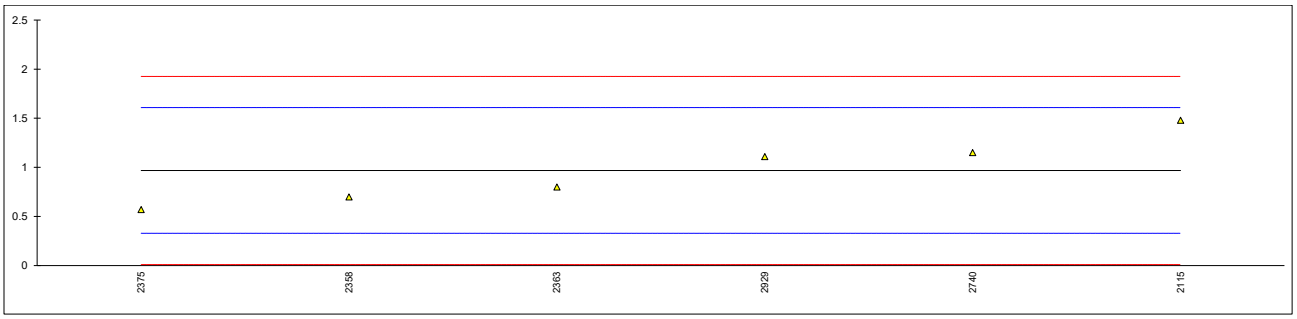


Determination of Esfenvalerate (CAS No. 66230-04-4) on sample #23791; results in mg/kg

lab	method	value	mark	z(targ)	remarks
2115	In house	1.48	C	1.60	First reported 2.297
2265		-----		-----	
2358	In house	0.70		-0.84	
2363	In house	0.8		-0.53	
2365		-----		-----	
2375	In house	0.57		-1.25	
2386		-----		-----	
2740	In house	1.15		0.57	
2929	In house	1.11		0.44	
2977		-----		-----	
3176		-----		-----	

normality	unknown
n	6
outliers	0
mean (n)	0.968
st.dev. (n)	0.3388
R(calc.)	0.949
st.dev.(iis memo 2302)	0.3195
R(iis memo 2302)	0.895

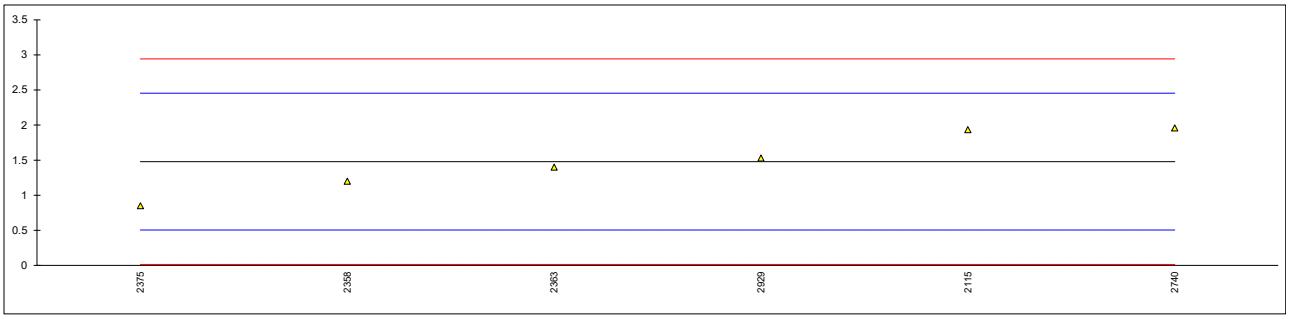
RSD=35%



Determination of Fenvalerate (CAS No. 51630-58-1) on sample #23791; results in mg/kg

lab	method	value	mark	z(targ)	remarks
2115	In house	1.934		0.93	
2265		-----		-----	
2358	In house	1.2		-0.57	
2363	In house	1.4		-0.16	
2365		-----		-----	
2375	In house	0.85		-1.29	
2386		-----		-----	
2740	In house	1.96		0.99	
2929	In house	1.53		0.10	
2977		-----		-----	
3176		-----		-----	

normality	unknown
n	6
outliers	0
mean (n)	1.479
st.dev. (n)	0.4291 RSD=29%
R(calc.)	1.202
st.dev.(iis memo 2302)	0.4881
R(iis memo 2302)	1.367



APPENDIX 2

Determination of other Pesticides on sample #23791; results in mg/kg

lab	Carbaryl	Malathion	Methyl-parathion	Monocrotophos	Parathion	Quinalphos
2115	-----	-----	-----	-----	-----	-----
2265	-----	-----	-----	-----	-----	-----
2358	not detected	not detected	not detected	not detected	not detected	not detected
2363	not detected	not detected	not detected	not detected	not detected	not detected
2365	<0.2	＜0.2	＜0.2	<0.5	＜0.2	＜0.2
2375	-----	-----	-----	-----	-----	-----
2386	-----	-----	-----	-----	-----	-----
2740	-----	-----	-----	-----	-----	-----
2929	not detected	not detected	not detected	not detected	not detected	0.032
2977	-----	-----	-----	-----	-----	-----
3176	-----	-----	-----	-----	-----	-----

APPENDIX 3 Analytical Details

Lab	ISO 17025 accr.	as received or further grinded/cut	Intake sample amount	Extraction type	Extraction solvent	Extraction time	Extraction temp.
2115	Yes	Used as received	1.5 g	ASE	Acetone		
2265	---	---		---			
2358	No	Used as received	0.5	Ultrasonic	Hexane and Acetone (1:1) for GC Methanol for LC	60	50
2363	Yes	Further cut	3g	Ultrasonic	hexane:acetone=1:1	60mins	50℃
2365	Yes	Further cut	0.5g	Ultrasonic	methanol	60min	50℃
2375	Yes	Further cut	0.5 g	Ultrasonic	Hexane/Acetone (1:1)	60 min	50 °C
2386	---	---		---			
2740	No	Used as received	0,5 g	Soxhlet	acetone	240 min	80 °C
2929	No	Used as received	0.5	Ultrasonic	acetone/ ethylacetate	120	60
2977	---	---		---			
3176	No	Used as received	0.5	Ultrasonic	MeOH	60	60

APPENDIX 4

Number of participants per country

4 labs in GERMANY

1 lab in HONG KONG

2 labs in ITALY

2 labs in P.R. of CHINA

2 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

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2 ISO5725:86
3 ISO5725 parts 1-6:94
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Address: Malledijk 18, P.O. Box 200, 3200 AE Spijkenisse, The Netherlands
Telephone number: +31 (0)88 214 45 41
Email address: nl.iis@sgs.com
Website: www.iisnl.com