



Institute for
Interlaboratory Studies

Results of Proficiency Test Phosphorus Flame Retardants in Textile March 2023

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Organophosphate esters (OPs) are widely used as flame retardants in various consumer and industrial products such as plastics, electronic equipment, furniture, textiles and building materials. However, production and use has been in decline since the 1980s, when Tris(2-chloro-ethyl) phosphate (TCEP) has been progressively replaced by other flame retardants. There is evidence that TCEP is a carcinogenic, mutagenic and toxic substance for children and therefore banned in toys.

On request of a number of participants the Institute for Interlaboratory Studies (iis) decided to organize a proficiency scheme for the determination of Phosphorus Flame Retardants in Textile. In this first interlaboratory study 37 laboratories in 14 countries registered for participation, see appendix 4 for the number of participants per country. In this report the results of the Phosphorus Flame Retardants 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 an ISO/IEC17025 accredited laboratory. It was decided to send one textile sample of 3 grams labelled #23525. 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 beige Polyester pieces was selected which was made positive on Phosphorus Flame Retardants by a third-party laboratory. After homogenization 55 plastic bags were filled with approximately 3 grams each and labelled #23525.

The homogeneity of the subsamples was checked by determination of TCEP according to ISO17881-1 and ISO17881-2 on 8 stratified randomly selected subsamples.

	TCEP in mg/kg
sample #23525-1	10.0
sample #23525-2	9.7
sample #23525-3	9.9
sample #23525-4	9.6
sample #23525-5	10.0
sample #23525-6	9.8
sample #23525-7	9.7
sample #23525-8	10.0

Table 1: homogeneity test results of subsamples #23525

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

	TCEP in mg/kg
r (observed)	0.4
reference test method	ISO17881-2:16
0.3 x R (reference test method)	0.8

Table 2: evaluation of the repeatability of subsamples #23525

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

To each of the participating laboratories one textile sample labelled #23525 was sent on February 8, 2023.

2.5 ANALYZES

The participants were requested to determine:

TBEP – Tris(2-butoxyethyl) Phosphate, CAS No. 78-51-3

TBP – Tributyl Phosphate, CAS No. 126-73-8

TiBP – Triisobutyl Phosphate, CAS No. 126-71-6

TCP – Tricresyl Phosphate, CAS No. 1330-78-5

TCEP – Tris(2-chloroethyl) Phosphate, CAS No. 115-96-8

TCPP – Tris(1-chloro-2-propyl) Phosphate, CAS No. 13674-84-5

TDCPP – Tris(1,3-dichloro-2-propyl) Phosphate, CAS No. 13674-87-8

TPP – Triphenyl Phosphate, CAS No. 115-86-6

IPTPP – Isopropylated triphenyl Phosphate, CAS No. 68937-41-7

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 test 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 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 reanalyzes). Additional or corrected test results are used for data analysis and the original test 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 in 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. Three participants did not report any test results. Not all participants were able to report all components requested.

In total 34 participants reported 34 numerical test results. Observed was 1 outlying test result, which is 2.9%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

The data set proved to have a normal Gaussian distribution.

4.1 EVALUATION PER COMPONENT

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

Test method ISO17881-2 is considered to be the official test method for the determination of Phosphorus Flame Retardants in Textile. In method ISO17881-2 precision data for TCEP is described in Annex B Table B.1. The target RSD of 10.2% was calculated from the nine CV_R for TCEP as mentioned in test method ISO17881-2. The target reproducibility was calculated as follows: $10.2\% \times PT \text{ mean} \times 2.8$.

sample #23525

TCEP: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ISO17881-2:16.

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 ($2.8 \times$ standard deviation) and the target reproducibility as derived from the reference method is presented in the next table.

Component	unit	n	average	2.8 * sd	R(lit)
TCEP	mg/kg	33	9.2	4.2	2.6

Table 3: reproducibility of the component on sample #23525

Without further statistical calculations it can be concluded that for TECP there is not a good compliance of the group of participating laboratories with the reference test method.

4.3 OVERVIEW OF THE PROFICIENCY TEST OF MARCH 2023

	March 2023
Number of reporting laboratories	34
Number of test results	34
Number of statistical outliers	1
Percentage of statistical outliers	2.9%

Table 4: overview of this Proficiency Test

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

The uncertainty of the determination in this proficiency test was expressed as relative standard deviation (RSD) of the PT, see next table.

Component	March 2023	Target
TCEP	16%	10.2%

Table 5: uncertainty of this Proficiency Test

4.4 EVALUATION OF THE ANALYTICAL DETAILS

Test method ISO17881-2 is used by more than 50% of the reporting participants. About 40% used an in house method and a few used EN71-11.

The reported analytical details from the participants are listed in appendix 3. Based on the answers given by the participants the following can be summarized:

- Seventy-six percent of the participants mentioned to be accredited for the determination of Phosphorus Flame Retardants in Textile.
- Prior to analysis the samples were further cut or grinded by sixty-two percent of the reporting participants, thirty-eight percent used the samples as received.
- The amount of sample intake varied between 0.2 and 2 grams, fifteen percent used 0.2-0.3 grams, forty-five percent used 0.5 grams, thirty-six percent used 1 gram and one laboratory used 2 grams.
- All reporting laboratories reported to have used ultrasonic as technique to release/extract the analytes.
- About fifty percent used Acetone or Acetone followed by Acrylonitrile as an extraction solvent, about twenty percent used a combination of Hexane with Ethyl Acetate, about ten percent used Toluene and about fifteen percent used THF or a THF mixture with Acrylonitrile.
- A vast majority (about eighty percent) of the reporting laboratories used an extraction time of 60 minutes. The extraction temperature differs between room temperature and 70 °C. Sixty-four percent an extraction temperature between 40 and 50 °C, thirty-three percent used an extraction temperature between 60 and 70 °C.

The majority of the group follow the same analytical procedures except for the choice of extraction solvent. Therefore, no separate statistical analysis has been performed. The participants chose more than seven different solvents or solvent mixtures. When analyzed separately over the use of solvents the subgroups will become too small for meaning full conclusions.

5 DISCUSSION

It is remarkably that the analytical conditions as reported by the participants with regards to sample intake and extraction solvent had varied so much in this proficiency test, see the analytical details mentioned in appendix 3. Also, considering the fact that eighteen participants reported to have performed ISO17881-2. Furthermore, sixteen participants are accredited for the determination of Phosphorus Flame Retardants in Textile.

TCEP was comprehensively evaluated under the EU regulation REACH 1907/2006. TCEP is classified under Regulation EC 1272/2008 of substances and mixtures as a carcinogenic, mutagenic and toxic substance. Furthermore, the limits for TCEP, TCPP and TDCP have been set under Regulation 2014/79/EU amending Appendix C of Annex II to Directive 2009/48/EC, see table 6.

Furthermore, the EU released version 3 of a Screening report for TCEP, TCPP and TDCP in April 2018 and a Regulatory strategy for flame retardants from ECHA in March 2023. There is evidence that TCEP is hazardous for children and data is now gathered for adults. A general overview of TCEP, TCPP and TDCP requirements on articles in the EU and the USA is given in the table below.

Region	Scope	Reference	Limit
EU	All articles	REACH candidate list	TCEP: 0.1% by weight
	Toys intended for children under 36 months and in toys intended to be put in the mouth	Toy Directive 2009/48/EC	TCEP, TCPP and TDCP: 5 mg/kg (each)
USA	Children's product and residential upholstered furniture	Various law states in US	TCEP, TCPP and TDCP: 0.1% by weight (each)

Table 6: Limits for Phosphorus Flame Retardants

All participants would have rejected the sample based on the limit of 5 mg/kg for toys intended for children or intended to be put in the mouth but would have accepted it for all other applications.

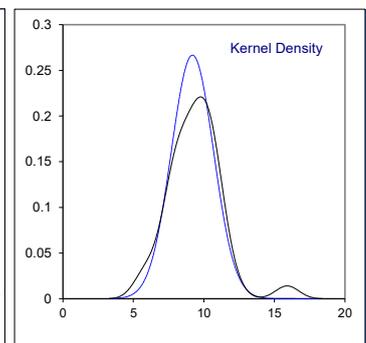
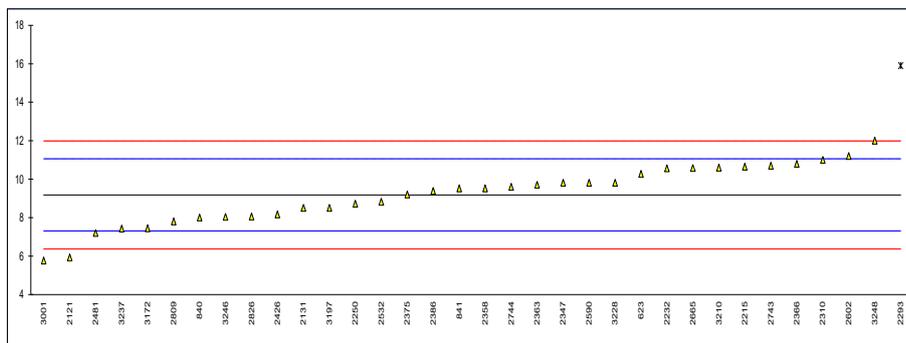
6 CONCLUSION

In the first PT on the determination of Phosphorus Flame Retardants in Textile all reporting participants have identified TCEP correctly. However, each 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 Tris(2-chloroethyl) Phosphate (TCEP) CAS no.115-96-8 in sample #23525; results in mg/kg

lab	method	value	mark	z(targ)	remarks
623	In house	10.27		1.16	
840	In house	8.0		-1.26	
841	In house	9.517		0.36	
2115		-----		-----	
2121	ISO17881-2	5.927		-3.47	
2131	In house	8.50		-0.73	
2215	In house	10.64		1.56	
2232	ISO17881-2	10.56		1.47	
2250	In house	8.72		-0.49	
2265		-----		-----	
2293	ISO17881-2	15.9	R(0.01)	7.17	
2310	ISO17881-2	11		1.94	
2347	In house	9.8		0.66	
2358	ISO17881-2	9.52		0.36	
2363	In house	9.7		0.55	
2366	In house	10.79		1.72	
2375	ISO17881-2	9.2		0.02	
2386	In house	9.38		0.21	
2426	ISO17881-2	8.16		-1.09	
2481	In house	7.2		-2.12	
2532	ISO17881-2	8.82		-0.39	
2561		-----		-----	
2590	ISO17881-2	9.8	C	0.66	first reported: 3.321
2602	In house	11.2		2.16	
2665	EN71-11	10.57		1.48	
2743	ISO17881-2	10.69	C	1.61	first reported: 19.12
2744	ISO17881-2	9.6		0.45	
2809	ISO17881-2	7.8		-1.47	
2826	ISO17881-2	8.0483		-1.21	
3001	ISO17881-2	5.77		-3.64	
3172	ISO17881-2	7.4395		-1.86	
3197	ISO17881-2	8.5		-0.73	
3210	In house	10.59		1.50	
3228	EN71-11	9.80		0.66	
3237	ISO17881-2	7.43	C	-1.87	first reported: 2.24
3246		8.037		-1.22	
3248	ISO17881-2	12		3.01	
					<u>Only ISO17881-2</u>
	normality	OK		OK	
	n	33		17	
	outliers	1		1	
	mean (n)	9.181		8.839	
	st.dev. (n)	1.4963	RSD = 16%	1.7188	RSD = 19%
	R(calc.)	4.190		4.813	
	st.dev.(ISO17881-2:16)	0.9365		0.9016	
	R(ISO17881-2:16)	2.622		2.524	



APPENDIX 2 Other reported Phosphorus Flame Retardants; results in mg/kg

TBEP	= Tris(2-butoxyethyl) Phosphate, CAS No. 78-51-3
TBP	= Tributyl Phosphate, CAS No. 126-73-8
TiBP	= Triisobutyl Phosphate, CAS No. 126-71-6
TCP	= Tricresyl Phosphate, CAS No. 1330-78-5
TCPP	= Tris(1-chloro-2-propyl) Phosphate, CAS No. 13674-84-5
TDCPP	= Tris(1,3-dichloro-2-propyl) Phosphate, CAS No. 13674-87-8
TPP	= Triphenyl Phosphate, CAS No. 115-86-6
IPTPP	= Isopropylated triphenyl Phosphate, CAS No. 68937-41-7

lab	TBEP	TBP	TiBP	TCP	TCPP	TDCPP	TPP	IPTPP
623	Not Detected							
840	not detected	----						
841	not detected	not detected	not detected	----	not detected	not detected	not detected	----
2115	----	----	----	----	----	----	----	----
2121	----	----	----	----	----	----	----	----
2131	not analysed	not analysed	not analysed	not analysed	not detected	not detected	not analysed	not analysed
2215	Not analyzed	Not analyzed	Not analyzed	Not detected				
2232	----	----	----	----	----	----	----	9.42
2250	< 5	< 5	< 5	< 5	< 5	< 5	< 5	----
2265	----	----	----	----	----	----	----	----
2293	----	----	----	----	0	0	0	----
2310	not detected							
2347	out of cap.	out of cap.	out of cap.	out of cap.	<5	<5	out of cap.	out of cap.
2358	not detected							
2363	<5	<5	<5	<5	<5	<5	<5	<5
2366	----	----	----	----	----	----	----	----
2375	----	----	----	----	----	----	----	----
2386	< 5	< 5	< 5	< 5	< 5	< 5	< 5	not analyzed
2426	Not Detected							
2481	----	----	----	----	n.d. <LD=2	n.d. <LD=2	----	----
2532	----	----	----	Not Detected				
2561	----	----	----	----	----	----	----	----
2590	----	----	----	----	----	----	----	----
2602	----	not detected	----	----	not detected	not detected	not detected	----
2665	not detected	not detected	not detected	not detected	0.0265	not detected	not detected	not analyzed
2743	----	----	----	----	not detected	not detected	----	----
2744	not analyzed							
2809	----	----	----	----	----	----	----	----
2826	----	----	----	----	----	----	----	----
3001	not detected	not detected	not detected	not detected	not applic.	not detected	not detected	not applic.
3172	< 1	< 1	----	----	< 1	< 1	< 1	< 1
3197	----	<1	----	<1	<1	<1	<1	<1
3210	----	----	----	----	----	----	----	----
3228	not detected	not detected	not applic.	not detected				
3237	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
3246	not analyzed	not analyzed	not analyzed	not detected				
3248	----	not detected						

APPENDIX 3 Analytical details

lab	ISO17025 accredited	sample preparation	intake (g)	release technique	release/extract solvent	extraction time (min)	extraction temp (°C)
623	Yes	Further cut	1	Ultrasonic	Ethyl acetate : Hexane (1:1)	60	50
840	Yes	Further cut	0.5	Ultrasonic	ethylacetate-n-hexane (1:1)	60	50
841	Yes	Further cut	0.5 g	Ultrasonic	ethylacetate/n-hexane 1:1	60	50
2115	---	---	---	---	---	---	---
2121	No	Used as received	1 g	Ultrasonic	Acetone	40 + 20 min	40°C
2131	No	---	1g	Ultrasonic	Toluene/Methanol	60	60
2215	No	Used as received	0.5g	Ultrasonic	toluene	120 minutes	70°C
2232	Yes	Further cut	1g	Ultrasonic	acetone	40minutes	40°C
2250	Yes	Further cut	0,5 g	Ultrasonic	Acetonitril bzw. Aceton	30 min	40°C
2265	---	---	---	---	---	---	---
2293	Yes	Further cut	0.2 g	Ultrasonic	5 mL THF with 10 mL ACN	30 minutes	70°C
2310	Yes	Further cut	0.5	Ultrasonic	Toluene	60	60
2347	No	Further cut	0.3g	Ultrasonic	---	60min	50°C
2358	Yes	Used as received	1g	Ultrasonic	Ethyl acetate/Hexane (1:1)	60	50
2363	Yes	Further grinded	2	Ultrasonic	Methylbenzene	60	60
2366	Yes	Further cut	0.5g	Ultrasonic	Ethyl acetate:n-hexane=1:1	60min	50°C
2375	Yes	Further cut	0,5 gram	Ultrasonic	Toluene	60	60
2386	Yes	Used as received	ca 1.0 g	Ultrasonic	Ethylacetate/n-Hexane	60 min	50 °C
2426	Yes	Further cut	0.2 gms	Ultrasonic	THF	60 Min	70 °C
2481	No	Further cut	0.5g	Ultrasonic	Toluene	60 minutes	60°C
2532	Yes	Further cut	0.2 grams	Ultrasonic	THF: ACN:water [1:2:3]	90 minutes	70 °C
2561	---	---	---	---	---	+	---
2590	Yes	Used as received	1g	Ultrasonic	acetone - acetonitrile	40 + 20 min	40°C
2602	No	Used as received	0,5	Ultrasonic	acetonitrile, diluted 1:1 with toluene after extraction	60	40
2665	Yes	Further cut	0,5	Ultrasonic	Acetonitrile	60	40
2743	Yes	Used as received	1g	Ultrasonic	Acetone	40+20 min	40°C
2744	Yes	Used as received	0,5	Ultrasonic	Acetonitrile/Acetone	1 hour	40°C
2809	Yes	Further cut	0.5	Ultrasonic	MeOH	30	Room Temp
2826	Yes	Used as received	1g	Ultrasonic	Acetone	60 mins	40°C
3001	No	Further cut	1 gr	Ultrasonic	acetone	60	40
3172	Yes	---	---	---	---	---	---
3197	Yes	Further cut	0,2 g	Ultrasonic	THF/ACN	30+30 min.	70
3210	No	Further cut	1 gram	Ultrasonic	Acetonitrile	60 minutes	60 °C
3228	Yes	Further cut	0.5	Ultrasonic	Acetonitrile	60	40
3237	Yes	Used as received	0,5	Ultrasonic	Acetone	60	40
3246	Yes	Used as received	1.00g	Ultrasonic	Acetone	60 min	40°C
3248	Yes	Used as received	0.5	Ultrasonic	acetone followed by acetonitrile	60	40

APPENDIX 4

Number of participants per country

3 labs in FRANCE

5 labs in GERMANY

1 lab in GUATEMALA

3 labs in HONG KONG

2 labs in INDIA

1 lab in INDONESIA

4 labs in ITALY

5 labs in P.R. of CHINA

1 lab in PAKISTAN

1 lab in SINGAPORE

1 lab in SWITZERLAND

5 labs in TURKEY

1 lab in UNITED KINGDOM

4 labs in VIETNAM

APPENDIX 5

Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported

Literature

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