Results of Proficiency Test
Phosphorus Flame retardants
and ADCA in Polymers
February 2017

Organised by: **Institute for Interlaboratory Studies (iis)**
                              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. Well known organophosphate esters are: Tris(2-chloroethyl)phosphate (TCEP), Tris(1,3-dichloroisopropyl)phosphate (TDCPP) and Tris(chloropropyl)phosphate (TCPP).
However, production and use has been in decline since the 1980s, when TCEP has been progressively replaced by other flame retardants. TCEP was comprehensively evaluated under the EU existing substances regulation (EEC) 793/93 in 2009. TCEP is classified under Regulation (EC) No 1272/2008 as a carcinogenic, mutagenic and toxic substance. In March 2012, the European Union decided to lower the limit of TCEP in toys (5 mg/kg) .

Regretfully, no certified reference materials (CRMs) for TCEP, TDCPP and TCPP are available to optimise the determination of Phosphorus flame retardants. As an alternative, participation in a proficiency test may enable the laboratories to check their performance and thus to increase this comparability. Therefore, a proficiency testing scheme (laboratory-evaluating interlaboratory study) for the determination of Phosphorus flame retardants was started by the Institute for Interlaboratory Studies in 2014. During this proficiency test in 2014 only TCEP was requested to be analyzed. This proficiency test was continued in the 2015 and 2016 PT program (in 2016 the scope was extended with TDCPP and TCPP). During the annual proficiency testing program 2016/2017, it was decided to continue the PT for the analysis of Phosphorus Flame retardants and to extend the scope with V6 (2,2'-bis(chloromethyl)tri-methylene bis(bis(2-chloroethyl)phosphate) and only in this PT with ADCA (Diazene-1,2-dicarboxamide). In the interlaboratory study of February 2017, 42 laboratories from 15 different countries registered for participation. See appendix 3 for the number of participants per country. In this report the results of the 2017 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 in Spijkenisse was the organizer of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO17025 accredited laboratory. It was decided to send two different polymer samples, both positive on Phosphorus Flame retardants, of approx. 3 grams each and resp. labelled #17500 and #17501. 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/IEC 17043:2010. 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 a 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 April 2014 (iis-protocol, version 3.3). 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

For the first sample a batch of small plastic (red) pieces, artificially fortified to be positive on TCEP, TDCPP and TCPP was selected. Samples of approx. 3 gram each were prepared and labelled #17500. Six stratified randomly selected samples were tested using an in house test method to check the homogeneity of the batch.

	TCEP in mg/kg	TDCPP in mg/kg
Sample #17500-1	199	125
Sample #17500-2	192	124
Sample #17500-3	190	123
Sample #17500-4	187	126
Sample #17500-5	195	121
Sample #17500-6	195	120

Table 1: homogeneity test results of subsamples #17500

From the test results of table 1, the repeatability was calculated and compared with 0.3 times the corresponding estimated target reproducibility in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	TCEP in mg/kg	TDCPP in mg/kg
r (observed)	12	6
reference test method	EN71-11:2005	EN71-11:2005
0.3 x R (reference test method)	13	8

Table 2: repeatability of subsamples #17500

The calculated repeatability of the test results was in agreement with 0.3 times the estimated reproducibility mentioned in the reference method EN71-11.
Therefore, homogeneity of the subsamples was assumed.

For the second sample a batch of small polyester (black) pieces, obtained from a third party laboratory, positive on TCEP, TCPP and ADCA (Diazene-1,2-dicarboxamide) was selected. Samples of approx. 3 gram each were prepared and labelled #17501. Seven stratified randomly selected samples were tested using an in house test method to check the homogeneity of the batch.

	TCEP in mg/kg	ADCA in mg/kg
Sample #17501-1	8756	7436
Sample #17501-2	8610	7251
Sample #17501-3	8739	7571
Sample #17501-4	8764	7415
Sample #17501-5	8675	7742
Sample #17501-6	8683	7280

Table 3: homogeneity test results of subsamples #17501

From the test results of table 1, the repeatability was calculated and compared with 0.3 times the corresponding estimated target reproducibility in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	TCEP in mg/kg	ADCA in mg/kg
r (observed)	166	516
reference test method	EN71-11:2005	EN71-11:2005
0.3 x R (reference test method)	570	488

Table 4: repeatability of subsamples #17501

The calculated repeatability of the test results was in agreement with 0.3 times the estimated reproducibility mentioned in the reference method EN71-11.
Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories a set of samples (1 sample labelled #17500, containing approx. 3 grams of polymer and 1 sample labelled #17501 containing approx. 3 grams of polyester) was sent on January 18, 2017. A letter of instructions was added to the sample package.

2.5   ANALYSES

The participants were requested to determine one to all of the following components: Tris(2-chloroethyl)phosphate (TCEP) (CAS No. 115-96-8), Tris(1,3-dichloro-2-propyl)- phosphate (TDCPP) (CAS No. 13674-87-8), Tris(1-chloro-2-propyl)phosphate (TCPP) (CAS No. 13674-84-5), 2,2'-bis(chloromethyl)tri-methylene bis(bis(2-chloroethyl)phosphate (V6) (CAS No. 38051-10-4) and Diazene-1,2-dicarboxamide (ADCA) (CAS No. 123-77-3) applying the analysis procedure that is routinely used in the laboratory. Also some method details were requested to be reported.
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 results cannot be used for meaningful statistical evaluation.

To get comparable results a detailed report form and a letter of instructions are prepared. 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 were 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.iisn.com.

3   RESULTS

During five weeks after sample dispatch, the 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 sample and per component in the appendix 1 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 reanalyses). 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 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 April 2014 (iis-protocol, version 3.3).

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.

In accordance to ISO 5725 the original test results per determination were submitted subsequently to Dixon's, Grubbs' and 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. 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 significant 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.

3.2 GRAPHICS

In order to visualise 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 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 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. 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, e.g. ISO reproducibilities, 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. In some cases, a reproducibility based on former iis proficiency tests could be used.

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 should be done in order to evaluate whether the reported test results are fit-for-purpose.

The z-scores were calculated in accordance with:

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

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:

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

4   EVALUATION

During the execution of this proficiency test no problems occurred. Forty participants reported test results of which one participant after the final reporting deadline. Two other participants did not report any test results at all. Finally, the 40 participants did report 239 numerical test results. Observed were 18 outlying test results, which is 7.5% of the numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

One participant mentioned that during the processing of sample #17501 a different coloration of the extract was present, depending on the temperature and the amount of extract.

For the determination of TCEP, TDCPP, TCPP, V6 and ADCA no standard test method is available. Most participating laboratories therefore had to perform an in house method. This will consist of a preparation/extraction step and an analytical step. Method EN71-11 describes the analytical determination of TCEP after migration/extraction and has a precision statement for TCEP. That is the reason that in this report EN71-11 is used as reference test method (for the analytical determination). It is also possible to use the more strict reproducibility calculated with the Horwitz equation. However, it was decided to use the precision statement for TCEP in EN71-11 also as reference for TDCPP, TCPP and ADCA.

Regretfully in EN71-11:2005, no reproducibility requirements for TCEP are mentioned, but only the standard deviation for the repeatability. The target reproducibility is estimated as follows: the standard deviation was multiplied with 2.8 to get the target repeatability. This

was multiplied with 3 to get an estimate of the target reproducibility.

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

4.1   EVALUATION ANALYTICAL DETAILS

For this PT some analytical details were requested (see appendix 2). Questions like: Is your laboratory accredited in accordance with ISO/IEC17025 and some specific questions with regards to the analytical details of the test method used.
Based on the answers given by the participants the following can be summarized:
Twenty-four of the forty reporting participants mentioned that they are accredited for determination of P-flame retardants in polymer.
Thirty-three participants mentioned that they have cut/grinded the sample before use, the other seven participants used the sample as received.
All, except two, participants reported to have used ultrasonic as technique to release/extract the analytes. Two participants used Thermal Desorption as technique.
Thirteen participants used Toluene as extraction solvent, seventeen used THF and/or ACN as extraction solvent. Seven participants used another solvent mixture, for instant hexane, methanol, ethylacetate and/or acetone.
When evaluating the above differences in the execution of the test, no clear correlation was found between these test conditions and the reported test results.

4.2   EVALUATION PER SAMPLE AND PER COMPONENT

In this section, the results are discussed per sample and per component. All statistical results reported on the sample #17500 and #17501 are summarised in appendix 1 and analytical details provided by the participants are summarised in appendix 2. The Abbreviations, used in these tables, are listed in Appendix 3.

Sample #17500

TCEP:　　　　The determination of this component was problematic at the measured level of 187 mg/kg. Five statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers was not in agreement with the estimated target reproducibility of EN71-11:2005.

TDCPP:　　　 The determination of this component was problematic at the measured level of 124 mg/kg. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers was not in agreement with the estimated target reproducibility of EN71-11:2005.

TCPP:　　　　The determination of this component was problematic at the measured level of 158 mg/kg. Only one statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier was not in

agreement with the estimated target reproducibility of EN71-11:2005.

V6 / ADCA: The concentrations of these two components were near or below the detection limit. Therefore no significant conclusions were drawn.

Sample #17501

TCEP: The determination of this component was problematic at the measured level of 8090 mg/kg. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers was not in agreement with the estimated target reproducibility of EN71-11:2005.

TDCPP: The determination of this component was problematic at the measured level of 46 mg/kg. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers was not in agreement with the estimated target reproducibility of EN71-11:2005.

TCPP: The determination of this component was problematic at the measured level of 7250 mg/kg. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers was not in agreement with the estimated target reproducibility of EN71-11:2005.

V6:   The concentration of this component was near or below the detection limit. Therefore no significant conclusions were drawn.

ADCA: Six participants reported a positive concentration of this component between 2737 – 5840.24 mg/kg. Four other participants reported a false negative test result. No reliable consensus value could be determined, therefore no z-scores were calculated.

4.3 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the calculated reproducibilities estimated from EN71-11:05 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 (EN71-11), are compared in the next tables.

Parameter	unit	n	average	2.8 * sd	R (target)
TCEP	mg/kg	35	187	69	41
TDCPP	mg/kg	36	124	51	27
TCPP	mg/kg	37	158	68	35

Table 5: observed reproducibilities of samples #17500

Parameter	unit	n	average	2.8 * sd	R (target)
TCEP	mg/kg	37	8086	2912	1766
TDCPP	mg/kg	35	45.7	17.5	10.0
TCPP	mg/kg	35	7250	2576	1584
ADCA	mg/kg	6	(4508)	(3106)	(985)

Table 6: observed reproducibilities of samples #17501

(result between brackets are for comparison only)

Without further statistical calculations, it can be concluded that the group of participating laboratories have some problems with the analysis of TCEP, TDCPP, TCPP and ADCA in polymer at these concentration levels. See also the discussion in paragraphs 4.2 and 5.

4.4   COMPARISON OF THE PROFICIENCY TEST OF FEBRUARY 2017 WITH THE PREVIOUS PT

	February 2017	February 2016	February 2015	February 2014
Number of reporting labs	40	31	33	23
Number of results reported	239	61	32	23
Number of statistical outliers	18	9	2	1
Percentage outliers	7.5%	14.8%	6.3%	4.3%

Table 7: Comparison with previous proficiency test

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

The uncertainty in the test results of TCEP in the iis17P01 PT did not improve compared to the previous PTs. However, the uncertainty of the test results of TDCPP in iis17P01 PT did improve slightly. TCPP in the iis17P01 was determined for the first time. It is noticeable that the uncertainty was similar to the uncertainty of TCEP and/or TDCPP.

Parameter	February 2017	February 2016	February 2015	February 2014	Est. EN71-11
TCEP	13%	9%	12%	23%	7.8%
TDCPP	13-14%	15%	n.e.	n.e.	7.8%
TCPP	13-15%	n.e.	n.e.	n.e.	7.8%

Table 8: Development of relative uncertainties over the years

5   DISCUSSION

The materials used in this PT were a PVC granulate and polyester pieces. In order to extract the requested components (see chapter 2.6) from a solid like a polymer, the extraction solvent, the extraction conditions and the contact surface area will be important variables.
In previous proficiency tests on Phosphorus Flame retardants it appeared that the choice of the extraction solvent (see PT report iis14P01) and the grain size of the granulate (see PT report iis15P01) were the most important variables. This was mainly caused by the matrix of

the samples used in the proficiency tests: in PT iis14P01 a foam block was used as sample and in PT iis15P01 a high density plastic was used as sample.

In the PT of 2016, a PVC sample positive on TCEP and TDCPP was used. It appeared that none of the requested analytical details was dominant as the calculated reproducibility for the TCEP determination, using all reported test results and thus including all different test details, was almost in agreement with the estimated reproducibility limits of EN71-11.

In the PT of 2017 two different samples were used, a PVC sample (#17500) positive on TCEP, TDCPP and TCPP and a polyester sample (#17501) positive on TCEP, TDCPP, TCPP and ADCA was used.
The observed large variation could not be explained from the reported analytical details.
It is noticeable that the uncertainties of the TCEP, TDCPP and TCPP were similar for both samples.

Due to the lack of a suitable test method, with precision data for the determination of TDCPP, TCPP and ADCA, it was decided to compare the group performance for the TDCPP and TCPP determination with the precision statement for TCEP of EN71-11. In this PT for the first time the component TCPP was requested to be determined. Also for this new component the precision from EN71-11 was used as target.

6   CONCLUSION

In this proficiency test the TCEP, TDCPP, TCPP and ADCA in polymers were determined. The variations observed in this interlaboratory study can be caused by the preparation or the conditioning of the sample and/or by the performance of the analysis. Consequently, the reproducibility cannot be improved by only one change in the analysis. 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 increase of the quality of the analytical results.

APPENDIX 1

Determination of Tris(2-chloro-ethyl)phosphate (TCEP) CAS no.115-96-8 in sample #17500;
results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	36.4	R(0.01)	-10.32	
339	In house	288	R(0.05)	6.93	
840	In house	177.27		-0.66	
2108	EN71-11	154.81		-2.20	
2129	prEN ISO17881-2Mod.	245.04		3.98	
2132	In house	182		-0.34	
2165	In house	193		0.42	
2172	In house	202.071		1.04	
2184	In house	195		0.55	
2213	In house	159.8		-1.86	
2215	In house	189		0.14	
2241	In house	180.0		-0.48	
2247	In house	212.847		1.78	
2295	In house	193		0.42	
2297	EN71-11	187.4		0.03	
2303	In house	101.5	R(0.05)	-5.86	
2352	In house	185.0		-0.13	
2358	In house	206.105		1.31	
2363	In house	184		-0.20	
2365	In house	180.1		-0.47	
2366	In house	204.93		1.23	
2375	In house	172.7		-0.98	
2380	In house	216.34		2.02	
2384	In house	147.09		-2.73	
2386	In house	207.6		1.42	
2389	In house	195.87		0.61	
2462	In house	260	R(0.05)	5.01	
2492	In house	120.1		-4.58	
2602	In house	155.3		-2.17	
2749		-----		-----	
2770	In house	288.40	R(0.05)	6.96	
3100	In house	172.68		-0.98	
3146		-----		-----	
3153	In house	225		2.61	
3163	In house	184		-0.20	
3172	GB/T24279	183.88		-0.21	
3179	In house	161.49		-1.75	
3185	In house	171.06	C	-1.09	First reported 289.07
3200	In house	175.6		-0.78	
3209	In house	197.10		0.70	
3210	In house	228.90		2.88	
3228	In house	197		0.69	

			Only Toluene	Only ACN/THF	Other solvents
normality		suspect	OK	OK	not OK
n		35	9	16	7
outliers		5	4	1	0
mean (n)		186.945	190.597	182.872	195.325
st.dev. (n)		24.4935	28.0905	27.9997	11.4556
R(calc.)		68.582	78.653	78.399	32.076
R(EN71-11:05)		40.829	41.626	39.939	42.659
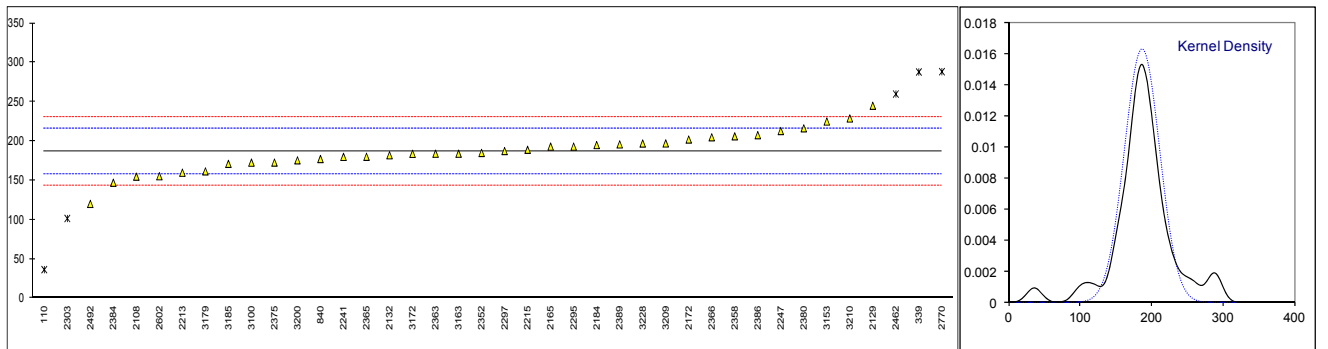
Determination of Tris(1,3-dichloro-2-propyl)phosphate (TDCPP) CAS no.13674-87-8 in sample #17500; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	88.8		-3.66	
339	In house	144		2.04	
840	In house	113.25		-1.13	
2108	EN71-11	69.20	R(0.05)	-5.68	
2129	prEN ISO17881-2Mod.	138.26		1.45	
2132	In house	124		-0.02	
2165	In house	127		0.29	
2172	In house	129.32		0.53	
2184	In house	136		1.22	
2213	In house	115.1		-0.94	
2215	In house	118		-0.64	
2241	In house	117.0		-0.75	
2247	In house	113.58		-1.10	
2295	In house	145		2.14	
2297	EN71-11	151.9		2.86	
2303	In house	43.9	R(0.05)	-8.29	
2352	In house	116.4		-0.81	
2358	In house	127.195		0.31	
2363	In house	113		-1.16	
2365	In house	105.6		-1.92	
2366	In house	114.29		-1.02	
2375	In house	92.3		-3.29	
2380	In house	109.16		-1.55	
2384	In house	80.50		-4.51	
2386	In house	126.7		0.26	
2389	In house	110.65		-1.40	
2462	In house	140		1.63	
2492		-----		-----	
2602	In house	144.2		2.06	
2749		-----		-----	
2770	In house	160.65	C	3.76	
3100	In house	117.24		-0.72	
3146		-----		-----	
3153	In house	113		-1.16	
3163	In house	34	R(0.05)	-9.31	
3172	GB/T24279	137.20		1.34	
3179	In house	121.14		-0.32	
3185	In house	156.90		3.37	
3200	In house	125.4		0.12	
3209	In house	124.62		0.04	
3210	In house	144.58		2.10	
3228	In house	130		0.60	

				Only Toluene	Only ACN/THF	Other solvents
	normality	OK		OK	OK	suspect
	n	36		13	14	7
	outliers	3		0	2	0
	mean (n)	124.220		119.178	130.974	121.406
	st.dev. (n)	18.0911		22.3251	15.3393	14.9584
	R(calc.)	50.655		62.510	42.950	41.884
	R(EN71-11:05)	27.130		26.028	28.605	26.515

Determination of Tris(1-chloro-2-propyl)phosphate (TCPP) CAS no.13674-84-5 in sample #17500; results in mg/kg

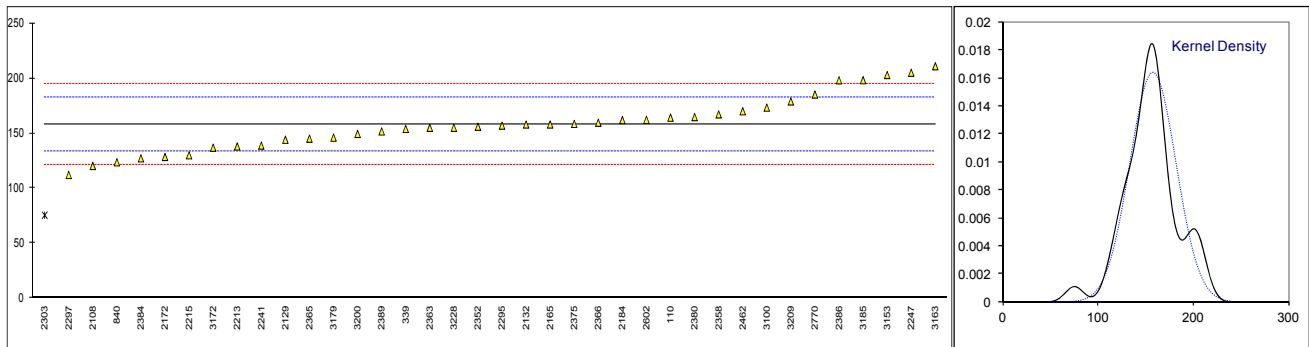| lab	method	value	mark	z(targ)	remarks
110	In house	164.2		0.50	
339	In house	154		-0.33	
840	In house	123.61		-2.79	
2108	EN71-11	120.31		-3.06	
2129	prEN ISO17881-2Mod.	144.12		-1.13	
2132	In house	158		0.00	
2165	In house	158		0.00	
2172	In house	128.472		-2.40	
2184	In house	162		0.32	
2213	In house	138		-1.62	
2215	In house	130		-2.27	
2241	In house	138.8		-1.56	
2247	In house	205.00	C	3.81	First reported 210.92
2295	In house	157		-0.08	
2297	EN71-11	112.1		-3.72	
2303	In house	75.5	R(0.05)	-6.69	
2352	In house	156.0		-0.16	
2358	In house	167.199		0.75	
2363	In house	155		-0.24	
2365	In house	145.1		-1.05	
2366	In house	159.61		0.13	
2375	In house	158.5		0.04	
2380	In house	164.74		0.55	
2384	In house	127.21		-2.50	
2386	In house	198.2		3.26	
2389	In house	151.71		-0.51	
2462	In house	170		0.97	
2492		-----		-----	
2602	In house	162.2		0.34	
2749		-----		-----	
2770	In house	185.25	C	2.21	
3100	In house	173.34		1.24	
3146		-----		-----	
3153	In house	203		3.65	
3163	In house	211		4.30	
3172	GB/T24279	136.90		-1.71	
3179	In house	146.04		-0.97	
3185	In house	198.28		3.27	
3200	In house	149.4		-0.70	
3209	In house	178.97		1.70	
3210		-----		-----	
3228	In house	155		-0.24	

|			Only Toluene	Only ACN/THF	Other solvents
normality	OK		OK	OK	not OK
n	37		13	14	7
outliers	1		0	1	0
mean (n)	158.007		153.711	157.884	164.373
st.dev. (n)	24.2845		16.6498	30.5879	15.7120
R(calc.)	67.997		46.619	85.646	43.994
R(EN71-11:05)	34.509		33.570	34.482	35.899

Determination of 2,2'-bis(chloromethyl)tri-methylene bis(bis(2-chloroethyl)phosphate) (**V6**) CAS no. 38051-10-4 and Diazene-1,2-dicarboxamide (**ADCA**) CAS no. 123-77-3 in sample #17500; results in mg/kg

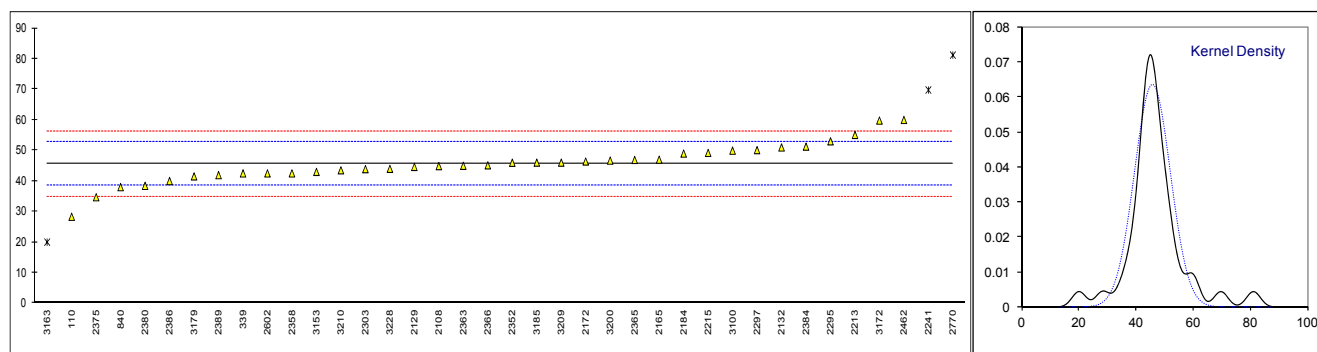| lab	method	V6	mark	z(targ)	method	ADCA	mark	z(targ)
110		-----		-----		-----		-----
339		-----		-----	IEC62321-6Mod.	< 250		-----
840		ND		-----		-----		-----
2108		-----		-----		-----		-----
2129		-----		-----		-----		-----
2132		-----		-----		-----		-----
2165		-----		-----		-----		-----
2172		ND		-----		-----		-----
2184		-----		-----		-----		-----
2213		<5		-----		<5		-----
2215		<5		-----		-----		-----
2241		<5		-----		<5		-----
2247		ND		-----		-----		-----
2295		-----		-----		-----		-----
2297		<50		-----		<50		-----
2303		-----		-----		-----		-----
2352		-----		-----		-----		-----
2358		-----		-----		-----		-----
2363	In house	N.D.		-----		-----		-----
2365	In house	ND		-----	In house	<500		-----
2366		Out Cap		-----		Out Cap		-----
2375		-----		-----		-----		-----
2380		-----		-----		-----		-----
2384		-----		-----		-----		-----
2386		< 10		-----		-----		-----
2389		-----		-----		-----		-----
2462		-----		-----		-----		-----
2492		-----		-----		-----		-----
2602		-----		-----		-----		-----
2749		-----		-----		-----		-----
2770		-----		-----	In house	N.D.		-----
3100		<5		-----		<5		-----
3146		-----		-----		-----		-----
3153		-----		-----		-----		-----
3163		-----		-----		-----		-----
3172	GB/T24279	Nd		-----	GB/T24279	nd		-----
3179	In house	<5		-----		-----		-----
3185	In house	ND		-----	In house	ND		-----
3200		ND		-----		-----		-----
3209		Not detected		-----		-----		-----
3210		-----		-----		-----		-----
3228		-----		-----		-----		-----
	normality	unknown			normality	unknown		
	n	7			n	6		
	outliers	n.a.			outliers	n.a.		
	mean (n)	<50			mean (n)	<500		
	st.dev. (n)	n.a.			st.dev. (n)	n.a.		
	R(calc.)	n.a.			R(calc.)	n.a.		
	R(lit)	n.a.			R(lit)	n.a.		

Determination of Tris(2-chloro-ethyl)phosphate (TCEP) CAS no.115-96-8 in sample #17501; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	6227.4		-2.95	
339	In house	11600	R(0.05)	5.57	
840	In house	7510.75		-0.91	
2108	EN71-11	9234.74		1.82	
2129	prEN ISO17881-2Mod.	10244.53		3.42	
2132	In house	7544		-0.86	
2165	In house	8710		0.99	
2172	In house	8052.32		-0.05	
2184	In house	8850		1.21	
2213	In house	7672		-0.66	
2215	In house	7910		-0.28	
2241	In house	7705.5		-0.60	
2247	In house	9076.82		1.57	
2295	In house	6960		-1.79	
2297	EN71-11	8013.1		-0.12	
2303	In house	6476.55		-2.55	
2352	In house	7610.0		-0.75	
2358	In house	8562.93		0.76	
2363	In house	7510		-0.91	
2365	In house	6953.7		-1.80	
2366	In house	7556.43		-0.84	
2375	In house	8575		0.78	
2380	In house	7313.48		-1.22	
2384	In house	5955.71		-3.38	
2386	In house	8141		0.09	
2389	In house	7168.5		-1.45	
2462	In house	9500		2.24	
2492	In house	8724.4		1.01	
2602	In house	10189		3.33	
2749		-----		-----	
2770	In house	12016.0	R(0.05)	6.23	
3100	In house	6884.95		-1.90	
3146		-----		-----	
3153	EN71-11	7577		-0.81	
3163	In house	3608	R(0.05)	-7.10	
3172	GB/T24279	8595.8		0.81	
3179	In house	9872		2.83	
3185	In house	8591.70		0.80	
3200	In house	8063.3		-0.04	
3209	In house	7754.3		-0.53	
3210	In house	9337.46		1.98	
3228	In house	8560		0.75	

			Only Toluene	Only ACN/THF	Other solvents
normality	OK		OK	OK	not OK
n	37		11	17	7
outliers	3		2	0	0
mean (n)	8086.06		7676.85	8140.57	8366.78
st.dev. (n)	1039.895		1259.318	982.626	571.156
R(calc.)	2911.71		3526.09	2751.35	1599.24
R(EN71-11:05)	1766.00		1676.62	1777.90	1827.30
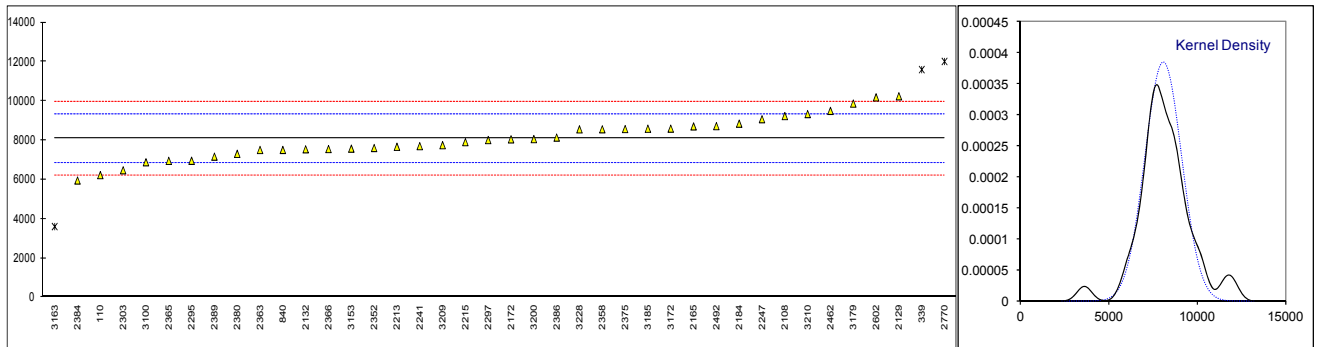
Determination of Tris(1,3-dichloro-2-propyl)phosphate (TDCPP) CAS no.13674-87-8 in sample #17501; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	28.31		-4.87	
339	In house	42.5		-0.89	
840	In house	38.00		-2.15	
2108	EN71-11	44.90		-0.21	
2129	prEN ISO17881-2Mod.	44.62		-0.29	
2132	In house	51		1.50	
2165	In house	47		0.38	
2172	In house	46.40		0.21	
2184	In house	49		0.94	
2213	In house	55.1		2.65	
2215	In house	49.2		0.99	
2241	In house	69.8	R(0.05)	6.78	
2247	In house	nd		-----	
2295	In house	53		2.06	
2297	EN71-11	50.12	C	1.25	First reported 6557.6
2303	In house	43.9		-0.49	
2352	In house	46.0		0.10	
2358	In house	42.517		-0.88	
2363	In house	45		-0.18	
2365	In house	46.9		0.35	
2366	In house	45.14		-0.15	
2375	In house	34.7		-3.08	
2380	In house	38.43		-2.03	
2384	In house	51.27	C	1.58	First reported 25.87
2386	In house	40.0		-1.59	
2389	In house	41.93		-1.05	
2462	In house	60		4.03	
2492		-----		-----	
2602	In house	42.5		-0.89	
2749		-----		-----	
2770	In house	81.23	C,R(0.01)	9.99	First reported 7936.1
3100	In house	49.95		1.21	
3146		-----		-----	
3153	In house	43		-0.75	
3163	In house	20	R(0.05)	-7.20	
3172	GB/T24279	59.80		3.97	
3179	In house	41.53		-1.16	
3185	In house	46.03		0.10	
3200	In house	46.7		0.29	
3209	In house	46.03		0.10	
3210	In house	43.53		-0.60	
3228	In house	44		-0.47	

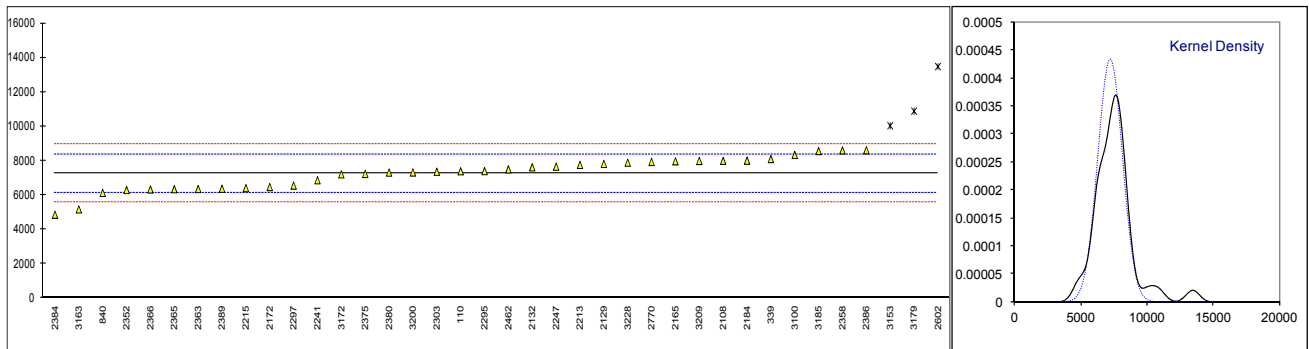|			Only Toluene	Only ACN/THF	Other solvents
normality		suspect	not OK	OK	OK
n		35	13	14	7
outliers		3	1	1	0
mean (n)		45.657	44.375	48.233	42.735
st.dev. (n)		6.2324	7.3307	5.1378	4.6865
R(calc.)		17.451	20.526	14.386	13.122
R(EN71-11:05)		9.972	9.692	10.534	9.333

Determination of Tris(1-chloro-2-propyl)phosphate (TCPP) CAS no.13674-84-5 in sample #17501; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	7387.9		0.24	
339	In house	8100		1.50	
840	In house	6131.29		-1.98	
2108	EN71-11	7997.67		1.32	
2129	prEN ISO17881-2Mod.	7818.02		1.00	
2132	In house	7623		0.66	
2165	In house	7970		1.27	
2172	In house	6472.40		-1.38	
2184	In house	8010		1.34	
2213	In house	7761		0.90	
2215	In house	6410		-1.49	
2241	In house	6866.8		-0.68	
2247	In house	7657.7	C	0.72	First reported 11278.32
2295	In house	7400		0.26	
2297	EN71-11	6557.6	C	-1.23	First reported 50.1
2303	In house	7361.55		0.20	
2352	In house	6300.0		-1.68	
2358	In house	8602.58		2.39	
2363	In house	6356		-1.58	
2365	In house	6345.0		-1.60	
2366	In house	6329.74		-1.63	
2375	In house	7236		-0.03	
2380	In house	7313.48		0.11	
2384	In house	4860.98		-4.23	
2386	In house	8612		2.41	
2389	In house	6373.8		-1.55	
2462	In house	7500		0.44	
2492		-----		-----	
2602	In house	13479	R(0.01)	11.01	
2749		-----		-----	
2770	In house	7936.1	C	1.21	First reported 81.23
3100	In house	8338.57		1.92	
3146		-----		-----	
3153	In house	10035	R(0.05)	4.92	
3163	In house	5171		-3.68	
3172	GB/T24279	7210.67	C	-0.07	First reported 2710.67
3179	In house	10886	R(0.05)	6.43	
3185	In house	8568.60		2.33	
3200	In house	7320.4		0.12	
3209	In house	7991.5		1.31	
3210		-----		-----	
3228	In house	7890		1.13	

|			Only Toluene	Only ACN/THF	Other solvents
normality	OK		OK	OK	OK
n	35		13	13	7
outliers	3		0	2	0
mean (n)	7250.90		6899.92	7523.62	7813.48
st.dev. (n)	920.122		928.246	638.561	789.034
R(calc.)	2576.34		2599.09	1787.97	2209.30
R(EN71-11:05)	1583.60		1506.94	1643.16	1706.46

Determination of 2,2'-bis(chloromethyl)tri-methylene bis(bis(2-chloroethyl)phosphate) (**V6**) CAS no. 38051-10-4 in sample #17501; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
339		-----		-----	
840		ND		-----	
2108		-----		-----	
2129		-----		-----	
2132		-----		-----	
2165		-----		-----	
2172		ND		-----	
2184		-----		-----	
2213		<5		-----	
2215		<5		-----	
2241		<5		-----	
2247		ND		-----	
2295		-----		-----	
2297		<50		-----	
2303		-----		-----	
2352		-----		-----	
2358		-----		-----	
2363	In house	N.D.		-----	
2365	In house	ND		-----	
2366		Out Cap		-----	
2375		-----		-----	
2380		-----		-----	
2384		-----		-----	
2386		< 10		-----	
2389		-----		-----	
2462		-----		-----	
2492		-----		-----	
2602		-----		-----	
2749		-----		-----	
2770		-----		-----	
3100		<5		-----	
3146		-----		-----	
3153		-----		-----	
3163		-----		-----	
3172	GB/T24279	nd		-----	
3179	In house	<5		-----	
3185	In house	ND		-----	
3200		ND		-----	
3209		Not detected		-----	
3210		-----		-----	
3228		-----		-----	

normality	unknown
n	7
outliers	n.a.
mean (n)	<50
st.dev. (n)	n.a.
R(calc.)	n.a.
R(lit)	n.a.

Determination of Diazene-1,2-dicarboxamide (**ADCA**) CAS no. 123-77-3 in sample #17501; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
339	In house	2737		-----	
840		-----		-----	
2108		-----		-----	
2129		-----		-----	
2132		-----		-----	
2165		-----		-----	
2172		-----		-----	
2184		-----		-----	
2213		<5		-----	False negative test result?
2215		-----		-----	
2241		<5		-----	False negative test result?
2247		-----		-----	
2295		-----		-----	
2297		<50		-----	False negative test result?
2303		-----		-----	
2352		-----		-----	
2358		-----		-----	
2363		-----		-----	
2365	In house	4196.8		-----	
2366		Out Cap		-----	
2375	In house	5554		-----	
2380		-----		-----	
2384		-----		-----	
2386		-----		-----	
2389		-----		-----	
2462		-----		-----	
2492		-----		-----	
2602		-----		-----	
2749		-----		-----	
2770	In house	4299.175		-----	
3100		4423.40		-----	
3146		-----		-----	
3153		-----		-----	
3163		-----		-----	
3172	GB/T24279	nd		-----	False negative test result?
3179		-----		-----	
3185	In house	5840.24		-----	
3200		-----		-----	
3209		-----		-----	
3210		-----		-----	
3228		-----		-----	

normality	unknown
n	6
outliers	0
mean (n)	(4508.44)
st.dev. (n)	(1109.327)
R(calc.)	(3106.12)
R(EN71-11:05)	(984.64)
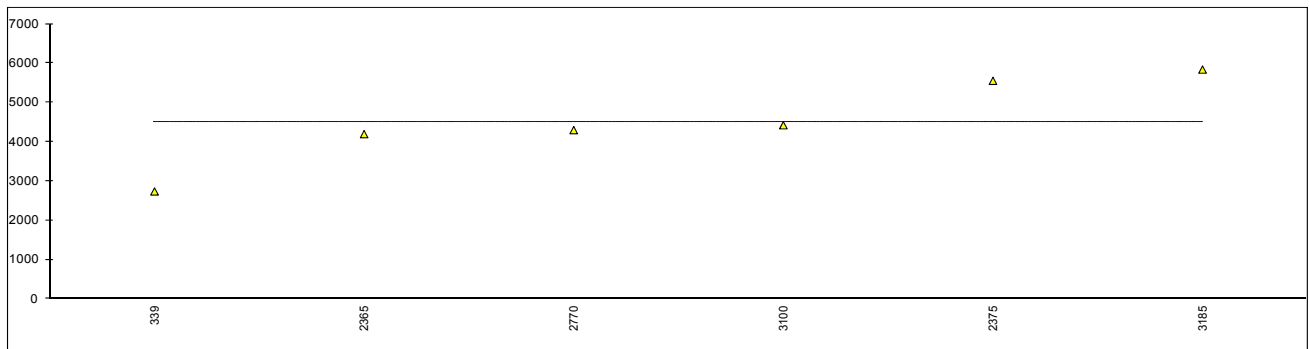
APPENDIX 2
Analytical details

lab	1. Laboratory ISO/IEC17025 accredited?	2. Was the sample grinded prior to analysis?	3. What was the final estimated particle size before analysis?	4. Which technique was used to extract the analyte(s)?	5. What solvent (mixture) was used to release the analyte(s)?	6. What was the extraction time (minutes) and temperature (°C)?	7. Which calibrant(s) was/were used?	Remarks on Additional Questions
110	Yes	Cut	3mm x 3mm	Ultrasonic	Toluene	60 min; 60C	Accustandard (custome certified reference standards)	
339	No	Used as received	Not measured	Ultrasonic	Toluene	60 minutes at 60°C	Calibrants from Sigma Aldrich, purity from 85 to 99%	Answers given for flame retardants testing in GC-MS, not for ADCA
840	Yes	Cut	2mm x 2mm	Ultrasonic	toluene	60min and 60°C	Chemservice	
2108	Yes	Cut	3 x 3 mm	Ultrasonic	ACN	1h; 40°C	Campro / Dr. Ehrenstorfer	
2129	No	Used as received		Ultrasonic	Toluene	60 minutes, 60 °C	TDCCP(Ehrenstorfer; 95,6%), TCPP(Ehrenstorfer; 99,5%), TCEP(LGC; 99,0%)	dissolve in methanol (1/10), filtrate
2132	No	Cut	less than 4mm x 4mm	Ultrasonic	THF	30 min, 40C	TCEP: Fluka, 98.4%; TDCPP: Chiron, >96%; TCPP: Dr. E, 99.5%	
2165	No	Cut	3mm*3mm	Ultrasonic	Hexane:Acetone :MTBE (1:1:1)	180minutes, 60°C	Dr.Ehrenstorfer, >98.5%	
2172	Yes	Cut	2mm*2mm	Ultrasonic	ACN and THF	40 oC. 30min		
2184	Yes	Used as received	3mm X 3mm	Ultrasonic	Hexane : Acetone : MTBE (1 : 1 : 1)	3 hrs, 60 deg C	Dr. Ehrenstorfer> 99%	
2213	Yes	Cut	2mm to 3 mm	Ultrasonic	Acetonitrile	1 hr and 40°C	DR EHRENSTORFER	
2215	No	Cut	2mm*2mm	Ultrasonic				
2241	Yes	Cut	1mm*1mm	Ultrasonic	acetonitrile	60min at 40	Dr. EHRENSTORFER	
2247	No	Cut	approx 2 mm	Ultrasonic	THF and Acetonitrile 1:2	1 hr (30 mins + 30 mins)	Chem services, Dr. Erhenstoper and Toronto chemical purity 95 to 99%	
2295	No	Cut	<4 mm	Ultrasonic	Tetrahidrofuran (THF)	60 min and 40C	Dr. Ehrenstorfer, Purity =>95%	
2297	Yes	Cut	less than 3mm	Ultrasonic	acetonitrile	60 min at 40 °C.		
2303	No	Cut	4mm	Ultrasonic	THF	30 minute @ 40C	Dr Ehrenstorfer	
2352	Yes	Cut	2mm*2mm*2mm	Ultrasonic	Toluene	60 minutes, 60 degee centigrade		
2358	Yes	Cut	5mm X 5mm	Ultrasonic	ethyl acetate and n-hexane (1:1-V:V)	60 minutes. 50 degree C		
2363	No	Cut	1mm*1mm*1mm	Ultrasonic	Toluene	60min 60°C	TCEP,TDCPP(TCI 97%);TCPP(Dr/Ehrenstorfer 99.5%)	
2365	Yes	Cut	1mm*1mm	Ultrasonic	Toluene	60min,60°C	DR,99%	
2366	Yes	Cut	2mm*2mm	Ultrasonic	Toluene	60min,50°C	None	None
2375	No	Cut	2mmX2mm	Ultrasonic	Ethylacetate : Hexane (1:1)	60 min 50 C	-	-
2380	No	Cut	2x2 mm	Ultrasonic	Toluene	60 minutes & 60 °C	TCEP: Chem Service 100%, TCPP: Dr. Ehrenstorfer, 99.5%, TDCPP: Chiron, 96.0%	
2384	Yes	Cut	3mm x 3mm x 3mm	Ultrasonic	toluene	60min, 60°C	-	

lab	1. Laboratory ISO/IEC17025 accredited?	2. Was the sample grinded prior to analysis?	3. What was the final estimated particle size before analysis?	4. Which technique was used to extract the analyte(s)?	5. What solvent (mixture) was used to release the analyte(s)?	6. What was the extraction time (minutes) and temperature (°C)?	7. Which calibrant(s) was/were used?	Remarks on Additional Questions
2386	Yes	Other (mention below)	17500: grinded. 17501: cut	Ultrasonic	n-Hexane/ethyl acetate 1:1 (v/v)	50 °C / 1h		
2389	No	Used as received	3 X 3 mm	Ultrasonic	n-Hexane , Ethyl Acetate	1 hour , 50 C	TCEP (Aldrich , 97%), TDCPP (Chemservice, 96%) , TCPP (Fluka 97.5 %)	
2462	Yes	Cut	2mm*2mm	Ultrasonic	Toluene	1h 60°C		
2492	Yes	Cut	0.5cm	Ultrasonic	THF	60min at 60°C	Campro Scientific	
2602	Yes	Cut	3 mm x 3 mm	Ultrasonic	Acetonitril	60 min and 40°C	TCEP: Campro 99,0% / TDCPP: Campro 95,6% / TCPP: Sigma-Aldrich 97,5%	temperature ultrasonic: started with 40°C but at the end about 60°C
2749	---	---		---				
2770	No	Cut	3mm*3mm	Ultrasonic	Toluene	70°C - 60min	TCEP:0.25g,99.0%,Dr.E; TCPP:0.25g,95.5%,Dr.E;TDCPP:0.25g,95.6%,Dr.E;ADCA:100ug/mL,1mL,MANHAGE	None
3100	Yes	Cut	2mm*2mm	Ultrasonic	TFH&ACN	70°C - 60min	Brand :Dr.Ehrenstorfer	
3146	---	---		---				
3153	Yes	Cut	2mm x 2mm	Ultrasonic	EN71-11: Acetonitrile; In house: Tetrahydrofuran	EN71-11: 60 min at 40oC; In house: 30 min at 70oC	TCEP: AccuStandard 100%; TDCPP: Fluka 95.6%; TCPP: Fluka 97.5%	
3163	No	Cut	1mm	Thermal Desorption	none	None	custom mix	
3172	Yes	Grinded		Ultrasonic	THF - Mixture(Hexane: Acetone 7:3)	1h - 30°C		
3179	Yes	Used as received	not applicable	Thermal Desorption				
3185	Yes	Cut	2mm*2mm	Ultrasonic	Tetrahydrofuran and Acetonitrile	1hour,70°C	TCEP:Accustandard 100%;TDCPP:Dr. Ehrenstorfer 96%£»TCPP:Dr. Ehrenstorfer 99.5%	
3200	Yes	Cut	5mm	Ultrasonic	toluene	2h	DR.Ehrenstorfer Gmbrh£¬99%	
3209	No	Cut	3mm X 3mm	Ultrasonic	THF+Acetonitrile	30 minutes at 40°C	Dr.E 96%/95.5%	
3210	No	Used as received		Ultrasonic	THF/Acétonitirle	60 min at 60°C	TCEP : Sigma aldrich purity : 98 ; TDCPP : Sigma aldrich purity 97%	THF for dissolution ; acetonitrile for precipitation
3228	Yes	Used as received	3mm*3mm	Ultrasonic	Hexane:Acetone:Methanol(1:1:1)	3Hrs, 60ºC	Dr. Ehrenstorfer >99%	

APPENDIX 3

Number of participants per country

    1 lab in BANGLADESH

    2 labs in FRANCE

    6 labs in GERMANY

    5 labs in HONG KONG

    2 labs in INDIA

    1 lab in ITALY

    1 lab in MALAYSIA

    16 labs in P.R. of CHINA

    1 lab in PAKISTAN

    1 lab in SWITZERLAND

    1 lab in THE NETHERLANDS

    2 labs in TURKEY

    1 lab in U.S.A.

    1 lab in UNITED KINGDOM

    1 lab in VIETNAM

APPENDIX 4

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
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
ex	= test result excluded from statistical evaluation

Literature:

1   iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, April 2014

2   K. Bester and K. Vorkamp, Anal Bioanal Chem 405, 6519-6527 (2013)

3   ISO 5725:86

4   ISO 5725, parts 1-6:94

5   M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)

6   W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)

7   IP 367:96

8   DIN 38402 T41/42

9   P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)

10   J.N. Miller, Analyst, 118, 455, (1993)

11   Analytical Methods Committee Technical Brief, No 4 January 2001

12   P.J. Lowthian and M. Thompson, The Royal Society of Chemistry 2002, Analyst 2002, 127, 1359-1364.

13   R.G. Visser, Reliability of proficiency test results for metals and phthalates in plastics, Accred Qual Assur, 14, 29-34 (2009)

14   F. Cordeiro, I. Verbist, P. Robouch, T. Linsinger, M.B. de la Calle, IMEP-26: Determination of brominated flame retardants in plastic, EUR 24874 EN2011

15   Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), 165-172, (1983)