Results of Proficiency Test
Total Bisphenol A in Polymers
April 2017

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Bisphenol A (BPA) is a chemical that is mainly used in combination with other chemicals to manufacture plastics and resins. For example, BPA is used in polycarbonate, a high performance transparent, rigid plastic. Polycarbonate is used to make food containers, such as returnable beverage bottles, infant feeding (baby) bottles, tableware (plates and mugs) and storage containers. Residues of BPA are also present in epoxy resins used to make protective coatings and linings for food and beverage cans. BPA can migrate in small amounts into food and beverages stored in materials containing the substance.

Bisphenol A is classified in Directive 2009/48/EC under Regulation (EC) No 1272/2008 as toxic. In the absence of any specific requirements, bisphenol A can be contained in toys in concentrations equal to or smaller than the relevant concentration established for the classification of mixtures containing it as CMRs, namely 5 % as from 20 July 2013 and 3 % as from 1 June 2015 respectively. It cannot be excluded that that concentration may lead to increased exposure to bisphenol A, compared to the migration limit of 0.1 mg/l for bisphenol A set by European standards EN 71-9:2005+A1:2007, EN 71-10:2005 and EN 71-11:2005.

Bisphenol A is a chemical that also often can be found in coatings on thermal printing paper. The surface of the paper is coated with a solid-state mixture of a dye and a reactant acid (Bisphenol A). So a large amount of BPA is expected to be found in thermal printing paper. The BPA can transfer readily to the skin in small amounts, especially when the skin is dry and free of grease. On 12 December 2016, the Official Journal of the European Union published Regulation (EU) 2016/2235 to include BPA restriction in Annex XVII to Regulation (EC) No 1907/2006 (REACH Regulation). The new restriction sets forth a threshold limit of 0.02 % (by weight) for Bisphenol A (BPA) present in thermal paper after 2 January 2020.

The determination of Bisphenol A in plastics is known to give problems with the comparability of laboratory results. However, no appropriate Bisphenol A reference materials are yet available. As an alternative, participation in a proficiency test may enable laboratories to check their performance. Therefore, a proficiency test (laboratory-evaluating interlaboratory study) for the determination of Bisphenol A in plastics was organized for the first time by the Institute for Interlaboratory Studies in April 2014.
Since 2014, a proficiency scheme is organized every year by the Institute for Interlaboratory Studies (iis). During the annual proficiency testing program 2016/2017, it was decided to continue the proficiency test for the analysis of Bisphenol A in polymer.
In this interlaboratory study 61 laboratories in 19 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 (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory.
It was decided to send two different samples. The first sample, a Polycarbonate (PC) granulate, was especially prepared by a Chinese factory by addition of Bisphenol A to PC and subsequent homogenization and extrusion. The second sample, a piece of thermal printing paper, positive on BPA was choosen. The participants were requested to report rounded and unrounded test results and also some details of the sample preparation and the test procedure. 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 Organization, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4). This protocol can be downloaded from 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

Two different materials, one batch of Polycarbonate (PC) granulate artificially fortified to be positive on Bisphenol A and one batch of thermal printing paper were selected. Both materials were divided over plastic bags, approx. 3 grams for each sample. The thermal printing paper was wrapped in Aluminium foil to avoid influence of light and heat.
The homogeneity of the subsamples was checked by determination of Bisphenol A (BPA) content on 8 stratified randomly selected subsamples of each batch.

	total BPA in mg/kg		total BPA in mg/kg
Sample #17565-1	2345	Sample #17566-1	15040
Sample #17565-2	2444	Sample #17566-2	15635
Sample #17565-3	2379	Sample #17566-3	15030
Sample #17565-4	2459	Sample #17566-4	15770
Sample #17565-5	2353	Sample #17566-5	16050
Sample #17565-6	2256	Sample #17566-6	15775
Sample #17565-7	2114	Sample #17566-7	15700
Sample #17565-8	2286	Sample #17566-8	15330

Table 1: homogeneity test results of the subsamples #17565 and #17566

From the above test results the repeatabilities were calculated. Comparison of the repeatabilities with 0.3 times the estimated repeatability of EN14372:04 in agreement with the procedure of ISO 13528, Annex B2. Regretfully, EN14372:04 does not mention a reproducibility. Therefore the comparison was made with the repeatability of EN14372:04.

	total BPA in mg/kg	total BPA in mg/kg
r (observed) #17565	312	--
r (observed) #17566	--	1035
reference test method	EN14372:04	EN14372:04
r (ref. test method)	294	1958

Table 2: evaluation of repeatabilities of BPA contents of the subsamples #17565 and #17566

For both samples #17565 and #17566, the observed repeatability of the 8 test results of the homogeneity study is equal or smaller than the repeatability of the reference test method and therefore the homogeneity of subsamples #17565 and #17566 was assumed.

To each of the participating laboratories, one sample of approx. 3 grams PC granulate, labelled #17565 and one sample of approx. 3 grams thermal printing paper (wrapped in aluminium foil) labelled #17566, were sent on April 19, 2017.

2.5 ANALYSES

The participants were requested to determine and report the total Bisphenol A content on both samples #17565 and #17566 applying the analysis procedure that is routinely used in the laboratory. Also some analytical 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 test results, but 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 can not be used for meaningful statistical calculations.

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 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 appendix 1 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 data analysis and the original reported test results 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 March 2017 (iis-protocol, version 3.4).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. Results reported as ‘<…’ or ‘>…” were not used in the statistical evaluation. First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test 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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.

According to ISO 5725 the original test results per determination were submitted subsequently to Dixon’s, Grubbs' and 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 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 reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.
Furthermore, Kernel Density Graphs were made. The Kernel Density Graph is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the target reproducibility (preferably taken from a standardized test method) by division with 2.8. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency tests could be used.

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 results is fit-for-use.

The z-scores were calculated in according to:

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

The $z_{(target)}$ scores are listed in the result tables of 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 interlaboratory study, some problems were encountered. Six participants decided not report any test results and seven other participants reported test results after the final reporting date. Finally, the 55 reporting laboratories reported 108 numerical results. In the reported test results 8 statistical outliers were observed, which is 6.9%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER SAMPLE AND PER COMPONENT

In this section the results are discussed per sample and per component.

Due to the lack of a suitable test method with precision data for the determination of total BPA in polymers, it was decided to use the requirements from the standardised method EN14372:04, “Child use and care articles, Cutlery and feeding utensils, Safety requirements and tests” for evaluation of the results of this interlaboratory study.
Regretfully, only a relative within-laboratory standard deviation RSDr is given in EN14372:04. Multiplication of RSDr by 2.8 gives the repeatability. Multiplication of the repeatability by 3 gives a good estimate of the target reproducibility.

Sample #17565

BPA: The determination of total Bisphenol A in this PC sample was problematic for a number of laboratories at the level of 2124 mg/kg. Four statistical outliers were observed and two other test results were excluded from the statistical evaluation as the test results of these two laboratories on sample #17566 appeared to be statistical outliers and therefore also two test results on sample #1756 were suspect.
However, the calculated reproducibility after rejection of the suspect results is in full agreement with the estimated reproducibility of EN14372:04.

Sample #17566

BPA: The determination of total Bisphenol A in this thermal printing paper sample was not problematic. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the estimated reproducibility of EN14372:04.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibilities as found for the group of participating laboratories and the estimated reproducibilities of EN14372:2004 (R_{target}) in the next tables:

Parameter	Unit	n	Average	2.8 * sd	R (target)
Bisphenol A (total)	mg/kg	48	2124	841	803

Table 3: overview of results for sample #17565

Parameter	Unit	n	Average	2.8 * sd	R (target)
Bisphenol A (total)	mg/kg	50	14575	5090	5509

Table 4: overview of results for sample #17566

4.3 COMPARISON OF THE PROFICIENCY TEST OF MAY 2017 WITH THE PREVIOUS PTS

	May 2017	May 2016	April 2015	April 2014
Number of reporting labs	55	53	53	60
Number of results reported	108	105	104	120
Number of statistical outliers	8	3	6	6
Percentage outliers	6.9%	2.8%	5.5%	4.8%

Table 5: Comparison with previous proficiency tests

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

Parameter	Conc. in mg/kg	May 2017	May 2016	April 2015	April 2014	Est. EN14372
BPA	<1000	n.e.	30%	54%	n.e.	13.5%
BPA	1000 – 2500	14%	24%	23%	34%	13.5%
BPA	>2500	12%	n.e.	n.e.	21%	13.5%

Table 6: Development of relative uncertainties over the years

The uncertainties in the test results of BPA in the 2017 PT iis17P04 have significantly improved in comparison with the previous PTs and are even in line with the uncertainty requirements of the target method (see table 6).

5 DISCUSSION

In this PT also some analytical details were asked (see appendix 2) to use for further statistical analyses. The majority (64%) of the participants is ISO/IEC 17025 accredited for the determination of BPA in polymer, total.

From the reported test methods it appeared that a large majority participants tested the polymers according to an in house test method (47 laboratories = 87%). This led to a variety of reported analytical details, e.g. particle size (cut/grinded or as received), extraction technique and the use of extraction solvent(s).

It is remarkable that for the polycarbonate (PC) and thermal printing paper samples used in this proficiency test, the requested analytical details, mentioned in appendix 2, appeared to have no significant influence on the test result for these samples. Both calculated reproducibilities are in full agreement with the estimated reproducibility mentioned in EN14372:04.

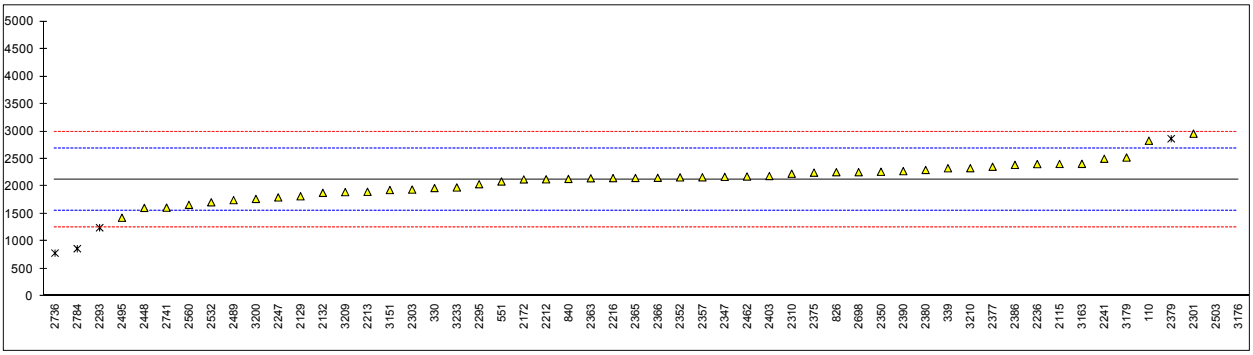
Although, it can be concluded that the group of participants have no problems with the determination of BPA in these samples, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary.
Therefore, participation on a regular basis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

APPENDIX 1

Determination of Total Bisphenol A (BPA) on sample #17565; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	2830.42		2.46	
330	In house	1970		-0.54	
339	In house	2330		0.72	
452		-----		-----	
551	In house	2089.14		-0.12	
623		-----		-----	
826	In house	2257.73		0.47	
840	In house	2136		0.04	
2115	In house	2407.62		0.99	
2129	D7574Mod.	1820		-1.06	
2132	In house	1882.76		-0.84	
2172	In house	2127		0.01	
2212	In house	2128		0.01	
2213	In house	1901		-0.78	
2216	In house	2151		0.09	
2236	In house	2405.89		0.98	
2241	In house	2502.4		1.32	
2247	In house	1799.33		-1.13	
2293	In house	1247.450	C,ex	-3.06	First reported 1095.430, result excluded as #17566 is an outlier
2295	In house	2040		-0.29	
2301	In house	2958.93		2.91	
2303	In house	1941.9		-0.64	
2310	In house	2230		0.37	
2330		-----		-----	
2347	In house	2173		0.17	
2350	In house	2265.9		0.49	
2352	JETRO2009	2164.0		0.14	
2357	JETRO2009	2165.5		0.14	
2363	In house	2147.1		0.08	
2365	EPA3550C	2153.4		0.10	
2366	In house	2156.49		0.11	
2375	In house	2250.0		0.44	
2377	In house	2356.81		0.81	
2379	JETRO2009	2867.33	ex	2.59	Result exluded, as test result on sample #17566 is an outlier
2380	In house	2298.620		0.61	
2386	In house	2391		0.93	
2390	In house	2277.784		0.54	
2403	EPA3550C/EPA8321B	2188.522		0.22	
2448	In house	1609.2774		-1.80	
2462	In house	2180		0.19	
2475		-----		-----	
2489	In house	1750	C	-1.30	First reported 0.175
2492		-----		-----	
2495	In house	1429.57		-2.42	
2503	In house	5085	R(0.01)	10.33	
2532	In house	1711		-1.44	
2560	In house	1663.9250		-1.60	
2698	In house	2258.550		0.47	
2736	In house	786.402	R(0.05)	-4.67	
2741	JETRO2009	1613		-1.78	
2784	In house	866.520	R(0.05)	-4.39	
3146		-----		-----	
3151	In house	1933		-0.67	
3163	In house	2410.169		1.00	
3172		-----		-----	
3176	In house	12640.00	C,R(0.01)	36.67	First reported 12.64
3179	In house	2525		1.40	
3200	In house	1772.2		-1.23	
3209	In house	1895.8		-0.80	
3210	In house	2330.82		0.72	
3233	In house	1980.555		-0.50	

normality	OK
n	48
outliers	4 (+2 excl)
mean (n)	2124.17
st.dev. (n)	300.375
R(calc.)	841.05
R(EN14372:04)	802.94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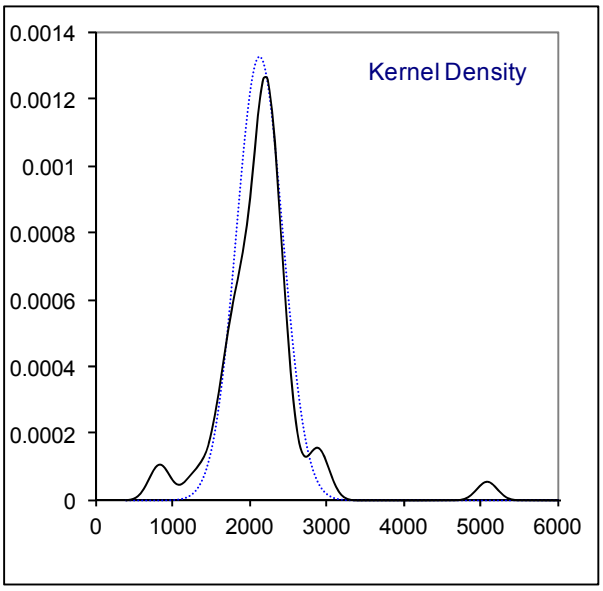
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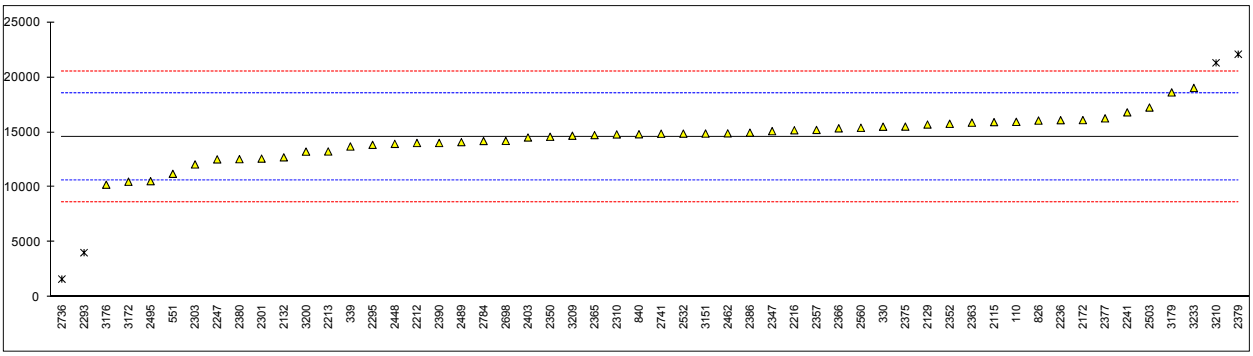
Determination of Total Bisphenol A (BPA) on sample #17566; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	15942.3		0.69	
330	In house	15500		0.47	
339	In house	13700		-0.44	
452		-----		-----	
551	In house	11219.15		-1.71	
623		-----		-----	
826	In house	16058.08		0.75	
840	In house	14815		0.12	
2115	In house	15917.53		0.68	
2129	D7574Mod.	15700		0.57	
2132	In house	12715.78		-0.94	
2172	In house	16098		0.77	
2212	In house	14020		-0.28	
2213	In house	13250		-0.67	
2216	In house	15178		0.31	
2236	In house	16093.36		0.77	
2241	In house	16806.7		1.13	
2247	In house	12529.653		-1.04	
2293	In house	4045.63	C,R(0.01)	-5.35	First reported 2113.46
2295	In house	13853		-0.37	
2301	In house	12601.77	C	-1.00	First reported 21293.53
2303	In house	12070.7		-1.27	
2310	In house	14800		0.11	
2330		-----		-----	
2347	In house	15095		0.26	
2350	In house	14585.0		0.01	
2352	JETRO2009	15766.0		0.61	
2357	JETRO2009	15207.3		0.32	
2363	In house	15869.4		0.66	
2365	EPA3550C	14737.9		0.08	
2366	In house	15354.12		0.40	
2375	In house	15512.2		0.48	
2377	In house	16260		0.86	
2379	JETRO2009	22090.38	R(0.05)	3.82	
2380	In house	12549.880		-1.03	
2386	In house	14960		0.20	
2390	In house	14020.147		-0.28	
2403	JETRO2009	14518.387		-0.03	
2448	In house	13941.589		-0.32	
2462	In house	14900		0.17	
2475		-----		-----	
2489	In house	14100	C	-0.24	First reported 1.41
2492		-----		-----	
2495	In house	10546.94		-2.05	
2503	In house	17245		1.36	
2532	In house	14873		0.15	
2560	In house	15402.8884		0.42	
2698	In house	14212.202		-0.18	
2736	In house	1646.971	R(0.01)	-6.57	
2741	JETRO2009	14863		0.15	
2784	In house	14202.603		-0.19	
3146		-----		-----	
3151	In house	14877		0.15	
3163		-----		-----	
3172	EN14372	10498		-2.07	
3176	In house	10228.000	C	-2.21	First reported 10.228
3179	In house	18620		2.06	
3200	In house	13235.2		-0.68	
3209	In house	14680.2		0.05	
3210	In house	21308.07	R(0.05)	3.42	
3233	In house	19024.445		2.26	

normality	OK
n	50
outliers	4
mean (n)	14575.09
st.dev. (n)	1818.009
R(calc.)	5090.42
R(EN14372:04)	5509.38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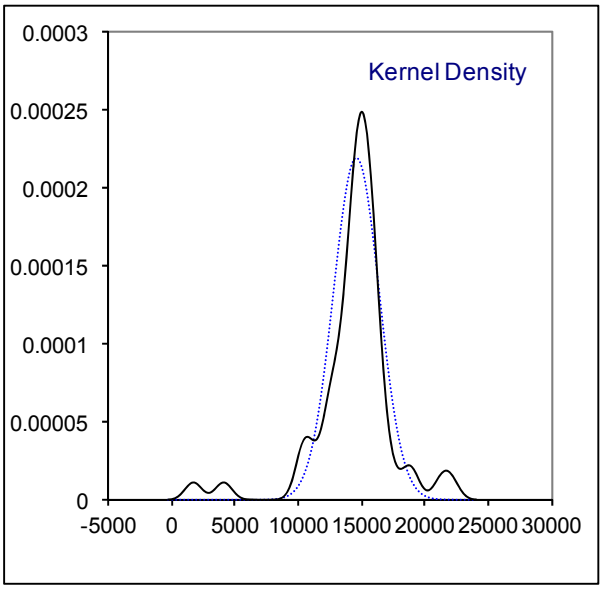
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APPENDIX 2

Method information as reported by the participating laboratories

Lab	Accredited laboratory	sample grinded or cut	final particle size	extraction technique used	extraction solvent used	analysis technique used
110	Yes	Cut	3mm x 3mm	Ultrasonic	Methylene Chloride	30min at 40C
330	Yes	as received		Ultrasonic (plastic). Migration (paper)	#17565 DCM/toluene #17566 ACN	1440 min at 23°C or 30 min at 70°C
339	No	#17565 as received #17566 Cut	#17566 : 2x2 mm	Ultrasonic	Toluene/DCM (50/50)	30 minutes at 70°C
452	---	---		---		
551	No	Cut	3x3 mm	Ultrasonic	Dichlorometane	30 min. / 40ºC
623	---	---		---		
826	Yes	Cut	5mm*5mm	Ultrasonic	DCM/Acetone	30min, 40¡É
840	Yes	Cut	1 mm	Ultrasonic	CH2Cl2	30 min. 40oC
2115	Yes	Used as received		Ultrasonic	Acetonitrile	60°C; 30 min
2129	Yes	Used as received		Ultrasonic	#17565: THF #17566: Methanol	#17565: 30 min room temp #17566: 30 min 60 °C
2132	No	#17565 Grinded #17566 Cut	#17565: <1x 1mm #17566: <3x 3mm	Ultrasonic	#17565: DCM/Acetone; #17566: DCM/CAN	60 mins & 60C
2172	Yes	Cut	5 mm x 5 mm	Ultrasonic	#17565: DCM,Acetone, #17566: THF,ACN	40oC for 60 minutes
2212	Yes	Cut	2mm	Mechanical Shaking	Dichloromethane	30minutes, room temperature
2213	Yes	Cut	5X5 mm	Ultrasonic	Chloroform/Methanol	60mins at 70°C
2216	Yes	Grinded	Ground to powder	Mechanical Shaking	DCM	60 minutes at room temperature
2236	Yes	Cut	2 mm x 2 mm	Ultrasonic	2:1 Chloroform:Methanol	60 minutes at 70 °C
2241	Yes	Cut	2mm*2mm*2mm	Ultrasonic	Dichloromethane	40 degree for 30mins
2247	Yes	#17565 as received #17566 Cut	#17566: 2x2mm	Ultrasonic	chloroform : Methanol 2:1	60 mins at 70 °C
2293	No	Cut	3 x 3 mm	Ultrasonic	Chloroform: methanol 2:1	60 minutes at 70°C
2295	Yes	Cut		Ultrasonic	Acetone, methanol and dicloromethane	1 hour and room temperature
2301	Yes	Cut	5 mm x 5 mm	Ultrasonic	ACN:Water 1:1	60 min (40 C)
2303	No	Cut	2mm	Ultrasonic	Dichloromethane/ethanol	120 minutes / room temperature
2310	No	#17565:as received #17566: Cut	#17566: 5x5 mm	Ultrasonic	Dichloromethane	30 mins & 40°C
2330	---	---		---		
2347	Yes	Cut	2mm*2mm	Ultrasonic	dichloromethane	40°C,30min
2350	No	Cut	2mm*2mm	Ultrasonic	DCM	30 min. / 40 C
2352	Yes	Cut	2mm*2mm	Ultrasonic	Dichloromethane	30min 40ºC
2357	Yes	Cut	#17565 2*2*2mm, #17566 5*5mm	Ultrasonic	dichloromethane	30min,40
2363	No	Cut	2*2mm	Ultrasonic	DCM	30mins40°C
2365	Yes	Cut	2mm*2mm	Ultrasonic	Dichloromethane	40°C - 30 min
2366	Yes	Cut	#17565: 2*2*2mm, #17566: 5*5mm	Ultrasonic	dichloromethane	60°C 30min
2375	Yes	Used as received	2x2 mm	Ultrasonic	Dichloromethane	30 min 40 C
2377	---	---		---		
2379	No	Cut	#17565 2x2 mm. #17566 5x5 mm	Ultrasonic	Dichloromethane	40 °C , 30 minutes
2380	No	Cut	2X2 mm	Ultrasonic	Dichloromethane	40+/-2 °C
2386	Yes	#17565:as received #17566: Cut		Ultrasonic	Dichlormathan	30 minutes 40°C
2390	Yes	#17565:as received #17566: Cut	#17565 = 3.5 mm , #17566 = 5x 5 mm	Ultrasonic	Dichloromethane	40 C for 30 min
2403	Yes	Cut	PC(0.5g) ; Paper(5mm*5mm)	Ultrasonic	Methylene Chloride	60 min, 30 centigrade
2448	No	Cut	-	Stirrer	#17565 DCM/Acetone, #17566: DI-water	the room temperature
2462	Yes	Cut	2mm*2mm	Ultrasonic	DCM	1H 60°C
2475	---	---		---		
2489	No	Cut	2X2 MM	Ultrasonic	Methanol/Chloroform	1 hour, 70 degree
2492	---	---		---		
2495	Yes	Used as received		Ultrasonic	Chloroform/Methanol 2:1	60
2503	Yes	Cut	0.05 grams	Ultrasonic	THF	90 min 70C
2532	Yes	Cut	2mm	Ultrasonic	Chloroform:Methanol (2:1)	1 hour at 70 °C
2560	No	Cut	2-3 mm	Ultrasonic	THF, ACN	60 min and 40 °C
2698	Yes	Cut	1mm	Ultrasonic	Methanol;Methylene chloride	60¡æ 60 minutes
2736	No	Cut		Stirrer	TCE, Methanol	

Lab	Accredited laboratory	sample grinded or cut	final particle size	extraction technique used	extraction solvent used	analysis technique used
2741	Yes	Cut	< 2 x 2 mm	Ultrasonic	Dichloromethane	60 mins, room temperature
2784	No	Cut	0.5x0.5 cm	Ultrasonic	acetonitrile	30 minutes at room temperature
3146	---	---		---		
3151	Yes	Cut		Ultrasonic	Tetrahydrofuran/Acetonitril	30 minutes/40°C
3163	No	Used as received	4 mm	Ultrasonic	Toluene	60 minutes and 60 °C
3172	Yes	Cut		---		
3176	Yes	Cut	about 0,3 cm	Ultrasonic	THF+ACN	30 min,40°C
3179	No	Used as received	2x2mm	Ultrasonic	THF,water,acetonitrile	60min,70°C
3200	---	---		---		
3209	Yes	Cut	2mm X 2mm	Ultrasonic	DCM	60 minutes at room temperature.
3210	No	Cut		Ultrasonic	THF/acetonitrile	60 minutes room temperature
3233	No	Used as received	5 x 5 mm	Ultrasonic	THF then ACN after water bath	40°C - 1H

APPENDIX 3

Number of participating laboratories per country

2 labs in	BANGLADESH
1 lab in	BRAZIL
1 lab in	CAMBODIA, Kingdom of
6 labs in	FRANCE
5 labs in	GERMANY
1 lab in	GUATEMALA
4 labs in	HONG KONG
5 labs in	INDIA
2 labs in	INDONESIA
3 labs in	ITALY
2 labs in	SOUTH KOREA
13 labs in	P.R. of CHINA
1 lab in	PAKISTAN
2 labs in	THAILAND
1 lab in	THE NETHERLANDS
3 labs in	TURKEY
5 labs in	U.S.A.
2 labs in	UNITED KINGDOM
2 labs in	VIETNAM

APPENDIX 4

Abbreviations:

C	= final 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	= probably an error in calculations
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.d.	= not detected

Literature:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, March 2017
2 ASTM E178:02
3 ASTM E1301:03
4 ISO 5725:86
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18 https://chemicalwatch.com/44942/bpa-poised-for-classification-as-category-1-reprotoxin
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