Results of Proficiency Test
Total Bisphenol A in Polymers
June 2020

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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CONTENTS

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 a chemical that also can be found in coatings on thermal printing paper. The Bisphenol A can transfer readily to the skin in small amounts, especially when the skin is dry and free of grease.

Since 2014 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of Total Bisphenol A in polymers every year. During the annual proficiency testing program 2019/2020 it was decided to continue the proficiency test for the analysis of Total Bisphenol A in polymers.
In this interlaboratory study 60 laboratories in 21 different countries registered for participation. See appendix 3 for the number of participants per country. In this report the results of this proficiency test Total Bisphenol A in polymers 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 two different samples, both positive on BPA. The first sample was green Polyproylene (PP) granulates of 3 grams, labelled #20610. The second sample was transparent Polycarbonate (PC) granulates of 3 grams, labelled #20611.
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 a 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

The selected batch for the first sample was green Polypropylene (PP) granulates artificially fortified to be positive on Bisphenol A. From this batch 75 small plastic bags were filled with approximately 3 grams each and labelled #20610. The homogeneity of subsamples was checked by the determination of BPA with an in-house test method on 8 stratified randomly selected subsamples.

	Total BPA in mg/kg
Sample #20610-1	667
Sample #20610-2	665
Sample #20610-3	656
Sample #20610-4	649
Sample #20610-5	636
Sample #20610-6	637
Sample #20610-7	642
Sample #20610-8	658

Table 1: homogeneity test results of subsamples #20610

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

	Total BPA in mg/kg
r (observed)	34
reference test method	EN14372:04
0.3 x R (reference test method)	74

Table 2: evaluation of the repeatability of subsamples #20610

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

The selected batch for the second sample was transparent Polycarbonate (PC) granulates artificially fortified to be positive on Bisphenol A. From this batch 75 small plastic bags were filled with approximately 3 grams each and labelled #20611. The homogeneity of subsamples was checked by the determination of BPA with an in-house test method on 8 stratified randomly selected subsamples.

	Total BPA in mg/kg
Sample #20611-1	2690
Sample #20611-2	2646
Sample #20611-3	2542
Sample #20611-4	2626
Sample #20611-5	2685
Sample #20611-6	2696
Sample #20611-7	2490
Sample #20611-8	2624

Table 3: homogeneity test results of subsamples #20611

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

	Total BPA in mg/kg
r (observed)	207
reference test method	EN14372:04
0.3 x R (reference test method)	298

Table 4: evaluation of the repeatability of subsamples #20611

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 set of samples (1 x #20610 and 1 x #20611) was sent on May 6, 2020.

2.5 ANALYZES

The participants were requested to determine the Total Bisphenol A content on both samples #20610 and #20611. It was also requested to report if the laboratory was accredited for the requested component and to report some analytical details.
It was explicitly requested to treat the samples as if they were routine samples. It was also requested 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 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 appropriate 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 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 reanalyzes). Additional or corrected test results are used for data analysis. The original test 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 organization 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 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 in general 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.

According to ISO5725 the original test results per determination were submitted to Dixon's and/or 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 the averages and the 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. 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 proficienct 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 literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values may be 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 results is fit-for-use.

The z-scores were calculated according to:

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

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

Absolute values for z<2 are very common and absolute values for z>3 are very rare. 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 with the dispatch of the samples due to the COVID-19 pandemic. Four participants did not report any test results. In total 56 participants reported 110 numerical test results. Observed were 4 outlying test results, which is 3.6%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

Both data sets proved to have a normal Gaussian distribution.

4.1 EVALUATION PER SAMPLE

In this section the reported test results are discussed per sample. 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 reported test results. The abbreviations, used in these tables, are explained in appendix 4.

No official test method exists for the determination of the total content of BPA in polymers. It was therefore decided to use the requirements from the test 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, due to the lack of a suitable test method with precision data for the determination of Total BPA in polymers. Regretfully, only a relative within-laboratory standard deviation RSDr is given in EN14372:04. Multiplication of RSDr by 2.8 gives the relative repeatability. Multiplication of the repeatability by 3 gives a good estimate of the relative target reproducibility.

Sample #20610

Total BPA: The determination of Total Bisphenol A in the Polypropylene (PP) sample was very problematic. One statistical outlier was observed. The calculated reproducibility was very large. It was therefore decided to calculate no z-scores. See also the discussion in paragraph 5.

Sample #20611

Total BPA: The determination of Total Bisphenol A in the Polycarbonate (PC) sample was problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated reproducibility of EN14372:04. See also the discussion in paragraph 5.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Component	unit	n	average	2.8 * sd	R(lit)
Total Bisphenol A	mg/kg	54	417	466	(157)

Table 5: overview of results for sample #20610

Component	unit	n	average	2.8 * sd	R(lit)
Total Bisphenol A	mg/kg	52	2666	1229	1008

Table 6: overview of results for sample #20611

Without further statistical calculations, it can be concluded that there is not a good compliance of the group of participating laboratories with the reference test method. See also the discussion in paragraphs 4.1 and 5.

4.3 COMPARISON OF THE PROFICIENCY TEST OF JUNE 2020 WITH PREVIOUS PTS

	June 2020	June 2019	May 2018	May 2017	May 2016
Number of reporting laboratories	56	59	69	55	53
Number of test results	110	117	133	108	105
Number of statistical outliers	4	14	9	8	3
Percentage of statistical outliers	3.6%	10.7%	6.3%	6.9%	2.8%

Table 7: comparison with previous proficiency tests

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

The performance of the determinations of the proficiency test was compared, expressed as relative standard deviation (RDS) of the PTs, see next table.

Matrix	June 2020	June 2019	May 2018	May 2017	May 2016	2015 - 2014	EN14372
Polycarbonate (PC)	16%	18%	21%	14%	n.e.	n.e.	14%
Polyethylene (PE)	n.e.	n.e.	n.e.	n.e.	24%	n.e.	14%
Polypropylene (PP)	40%	n.e.	n.e.	n.e.	30%	34 - 54%	14%
Polyvinylchloride (PVC)	n.e.	18%	n.e.	n.e.	n.e.	21 - 23%	14%
Thermal paper (TP)	n.e.	n.e.	11%	12%	n.e.	n.e.	14%

Table 8: development of uncertainties in BPA in polymers determinations over the years

The uncertainty of BPA in PC did improve compared to previous years (except for PT in 2017). The determination of BPA in PP may be very difficult. The uncertainty of PP is quite large compared to the uncertainty of the PC determination, see also paragraph 6 for the conclusion.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

From the reported test methods, it appeared that almost all participants used for the determination of Total BPA an in-house test method (49 laboratories = 90%).

Also is requested whether a participant is accreditated for the determination of Total BPA and some analytical details. The reported details are given in appendix 2. Based on the answers given by the participants the following can be summarized:

- Thirty-seven participants answered to be ISO/IEC17025 accredited for the determination of Total BPA in polymers (= 67%).
- About 60% of the participants did use the samples #20610 and #20611 as received and about 40% of the participants further cut or further grinded the samples #20610 and #20611 prior to analyses.
- The final estimated particle sizes of samples #20610 and #20611 most often used was smaller than 3x3mm.
- About 85% of the participants used a sample intake between 0.5 and 1.0 grams and about 10% of the reporting participants mentioned to have used <0.5 grams.
- The solvent (mixture) to release the BPA from the samples differs. About 45% of the participants used Dichloromethane and 40% of the participants used Tetrahydrofurane (THF) as solvent. The effect of solvent is further investigated and reported in appendix 1, see also discussion in paragraph 5.
- Almost all participants did use an extraction time between 30 and 60 min.
- About 45% of the participants reported to have used an extraction temperature of 40°C and about 45% mentioned to have used an extraction temperature of 60-70°C.

5 DISCUSSION

In this proficiency test for the determination of Total BPA in polymers two different sample matrices were used: Polypropylene and Polycarbonate granulates.
For both samples #20610 and #20611 not a good compliance of the reproducibility was observed for the group of participating laboratories with the reproducibility as estimated from the reference test method of EN14372:04 (R(lit)). That the observed reproducibilities did not comply to the reference test method EN14372:04 may partly be explained by the matrix of the samples. It occurs that releasing BPA from Polypropylene samples is far more difficult than from Polycarbonate, resulting in a higher relative standard deviation (RSD) of 40% of PP compared to a RSD of 16% of PC.

For sample #20610 the group seems to be divided bimodally (see Kernel plot on page 13). The different solvents used may partly explain the distribution. The group that used DCM as solvent found a lower average than the group that used THF (372 vs 500 mg/kg). The uncertainties of the THF users is smaller (31 vs 39%), but is still large (compared to target of 14% of EN14372:04). Therefore, it was decided not to use one group for assigned values and to calculate no z-scores.

6 CONCLUSION

For the analysis of **Total** BPA from polymers a sound test method which prescribe the analysis of **Total** BPA from different polymers in detail is desirable, especially for other polymers than Polycarbonate. Also, the choice of solvent may play a role in the determination other polymers than in Polycarbonate.

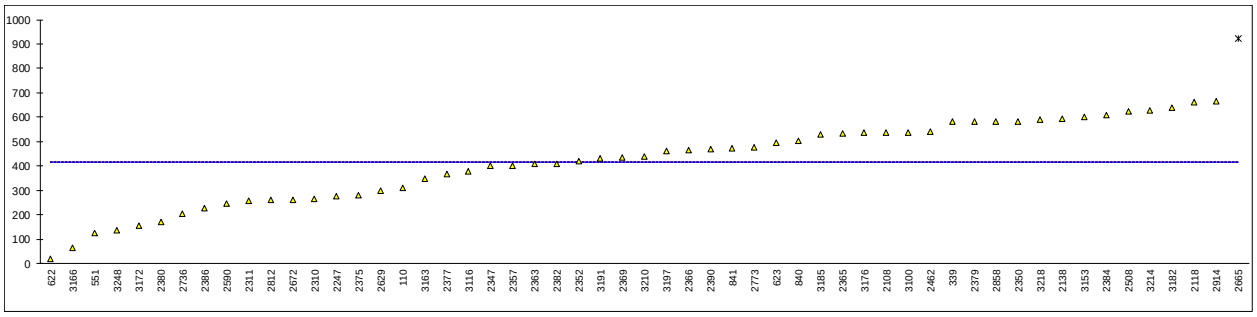
It can be concluded that the group of participants have problems with the determination of Total BPA in the Polypropylene sample in this proficiency test. Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, 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) in Polypropylene sample #20610; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	308.9		-----	
339	In house	580.69		-----	
362		-----		-----	
551	In house	125.0	C	-----	First reported 162.85
622	EN14350-2	20.9128	C	-----	First reported 19.6288
623	In house	495.990		-----	
840	In house	502		-----	
841	In house	473	C	-----	First reported 0.0473 mg/kg
2108	In house	537.77		-----	
2118	In house	660.97		-----	
2131		-----		-----	
2138	In house	592.0		-----	
2247	In house	275.20		-----	
2267		-----		-----	
2310	In house	267		-----	
2311	In house	257		-----	
2347	In house	401.9		-----	
2350	In house	583.14		-----	
2352	JETRO	420		-----	
2357	In house	402		-----	
2363	In house	410		-----	
2365	In house	533.65		-----	
2366	In house	465.1		-----	
2369	In house	436		-----	
2375	In house	279		-----	
2377	In house	367		-----	
2379	JETRO	581.2747		-----	
2380	In house	169.9		-----	
2382	In house	410.2		-----	
2384	In house	610.8264	C	-----	First reported 808.9624
2386	In house	227		-----	
2390		468.1	C	-----	First reported 923.54
2462	EPA3550C/8321B	540		-----	
2475		-----		-----	
2508	In house	622.4		-----	
2590	In house	245.625		-----	
2629	In house	299.15		-----	
2665	In house	923	R(0.05)	-----	
2672	In house	260.8		-----	
2736	In house	203.45		-----	
2773	In house	475.1		-----	
2812	In house	259.6		-----	
2826		-----		-----	
2858	In house	582.80		-----	
2914	In house	664		-----	
3100	In house	538		-----	
3116	In house	379		-----	
3153	In house	600		-----	
3163	In house	350		-----	
3166	In house	63.6		-----	
3172	In house	156.68		-----	
3176	In house	536.80		-----	
3182	In house	639.44		-----	
3185	In house	530		-----	
3191	JETRO	432.1	C	-----	First reported 0.04321 mg/kg
3197	In house	462.2		-----	
3210	In house	438.580		-----	
3214	In house	626.35		-----	
3218	In house	590.79		-----	
3248	In house	135		-----	

				Only DCM		Only THF	
	normality	OK		OK		suspect	
	n	54		23		19	
	outliers	1		1		0	
	mean (n)	416.537		372.450		500.364	
	st.dev. (n)	166.3882	RSD = 40%	144.1405	RSD = 39%	155.2002	RSD = 31%
	R(calc.)	465.887		403.593		434.560	
	st.dev.(EN14372:04)	(56.2325)		(50.2807)		(67.7386)	
	R(EN14372:04)	(157.451)		(140.786)		(189.668)	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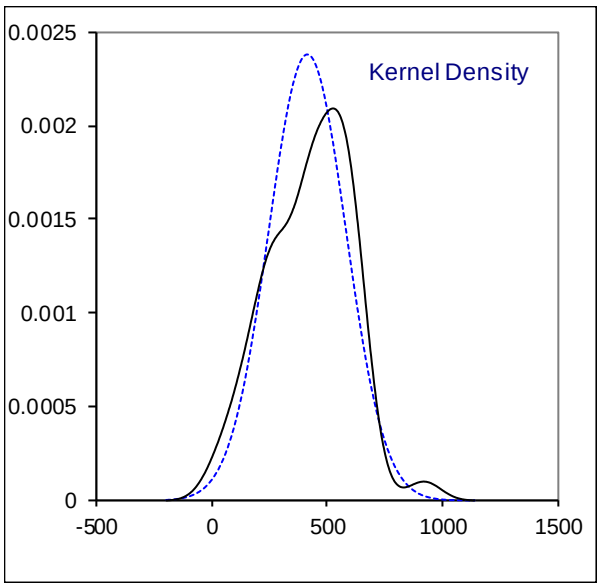
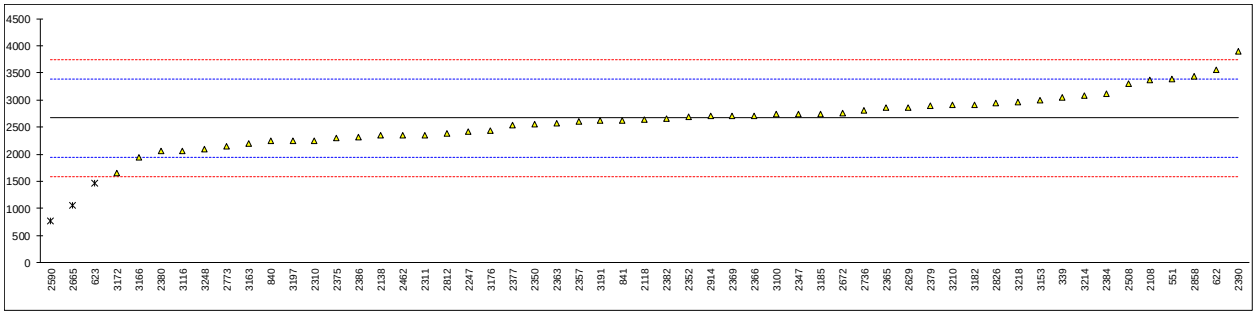

Kernel Density
0.0025
0.002
0.0015
0.001
0.0005
0
-500
0
500
1000
1500

Determination of Total Bisphenol A (BPA) in a Polycarbonate sample #20611; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110		-----		-----	
339	In house	3047.2		1.06	
362		-----		-----	
551	In house	3379.06		1.98	
622	SNI7626-1	3564	C	2.50	First reported 0.3304 mg/kg
623	In house	1466.350	DG(0.05)	-3.33	
840	In house	2243		-1.18	
841	In house	2622	C	-0.12	First reported 0.2622 mg/kg
2108	In house	3362.47		1.94	
2118	In house	2638.46		-0.08	
2131		-----		-----	
2138	In house	2341.7		-0.90	
2247	In house	2409.87		-0.71	
2267		-----		-----	
2310	In house	2250		-1.16	
2311	In house	2355		-0.86	
2347	In house	2738.4		0.20	
2350	In house	2556.71		-0.30	
2352	JETRO	2681		0.04	
2357	In house	2603		-0.17	
2363	In house	2570		-0.27	
2365	In house	2853.19		0.52	
2366	In house	2706.8		0.11	
2369	In house	2703		0.10	
2375	In house	2294		-1.03	
2377	In house	2535		-0.36	
2379	JETRO	2885.6408		0.61	
2380	In house	2059		-1.69	
2382	In house	2650.2		-0.04	
2384	In house	3108.1533		1.23	
2386	In house	2319		-0.96	
2390		3895.54		3.42	
2462	EPA3550C/8321B	2350		-0.88	
2475		-----		-----	
2508	In house	3302.1		1.77	
2590	In house	773.810	G(0.05)	-5.26	
2629	In house	2865.1		0.55	
2665	In house	1058	DG(0.05)	-4.47	
2672	In house	2753		0.24	
2736	In house	2803.83		0.38	
2773	In house	2145.2		-1.45	
2812	In house	2386.3		-0.78	
2826	In house	2943		0.77	
2858	In house	3439.38	C	2.15	First reported 7512.69
2914	In house	2699		0.09	
3100	In house	2733		0.19	
3116	In house	2060		-1.68	
3153	In house	2994		0.91	
3163	In house	2200		-1.29	
3166	In house	1940		-2.02	
3172	In house	1652.5		-2.82	
3176	In house	2432.0		-0.65	
3182	In house	2912.79		0.69	
3185	In house	2747		0.23	
3191	In house	2621.4	C	-0.12	First reported 0.26214 mg/kg
3197	In house	2243.7		-1.17	
3210	In house	2902.070		0.66	
3214	In house	3076.00		1.14	
3218	In house	2967.36		0.84	
3248	In house	2090		-1.60	

				Only DCM		Only THF	
	normality	OK		suspect		OK	
	n	52		28		18	
	outliers	3		1		1	
	mean (n)	2665.964		2636.464		2663.481	
	st.dev. (n)	439.0728	RSD = 16%	417.4771	RSD = 16%	489.6717	RSD = 18%
	R(calc.)	1229.404		1168.936		1371.081	
	st.dev.(EN14372:04)	359.9051		355.9226		359.5700	
	R(EN14372:04)	1007.734		996.583		1006.796	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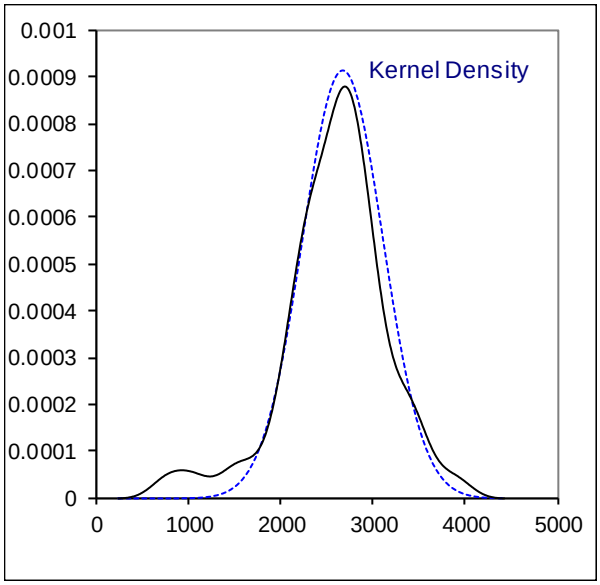
1500
1000
500
0
2590 2665 623 3172 3166 2380 3116 3248 2773 3163 840 3197 2310 2375 2386 2138 2462 2311 2812 2247 3176 2377 2350 2363 2357 3191 841 2118 2382 2352 2914 2369 2366 3100 2347 3185 2672 2736 2365 2629 2379 3210 3182 2826 3218 3153 339 3214 2384 2508 2108 551 2858 622 2390


Kernel Density
0.001
0.0009
0.0008
0.0007
0.0006
0.0005
0.0004
0.0003
0.0002
0.0001
0
0 1000 2000 3000 4000 5000

APPENDIX 2 Analytical details as reported by the participating laboratories

Lab	ISO17025 accr.	sample grinded or cut	final particle size	sample intake (g)	extraction solvent	extraction time (min)	extraction temp (°C)
110	Yes	Used as received	---	1	DCM / Acetone	30	40
339	No	Used as received	0.5 x 0.5 cm	0.5	DCM / Toluene (1:1)	30	70
362	---	---	---	---	---	---	---
551	No	Used as received	---	0.5	---	---	---
622	Yes	Used as received	---	1	HAc ; Ethanol 50%	24 hours	40
623	Yes	Further Cut	2 x 2 mm	1	ACN	60	60
840	Yes	Further Cut	1 x 1 mm	0.5	THF	60	60
841	Yes	Further Cut	2 x 2 mm	0.5	THF	60	60
2108	Yes	Used as received	---	0.5	THF	60	60
2118	No	Used as received	---	0.5	THF/Methanol	60	60
2131	Yes	Used as received	---	0.5	THF/Hexane	60	60
2138	Yes	Used as received	---	0.15 g	THF/Methanol	60	60
2247	Yes	Used as received	<2 mm	0.5	Chloroform/Methanol (2:1)	60	70
2267	---	---	---	---	---	---	---
2310	Yes	Used as received	5 x 5 mm	1	DCM	30	40
2311	Yes	Further Cut	<2 mm	0.5	DCM / Acetone	30	40
2347	No	Further Cut	---	---	---	---	---
2350	No	Further Cut	---	0.5	Methylene chloride	30	40
2352	Yes	Further Cut	2 x 2 mm	1	DCM	30	40
2357	Yes	---	---	---	---	---	---
2363	No	Further Cut	2 x 2 mm	1	DCM	30	40
2365	Yes	Further Cut	2 x 2 mm	0.5	DCM	30	40
2366	No	Further Cut	2 x 2 mm	0.5	DCM	30	40
2369	---	---	---	---	---	---	---
2375	Yes	Further Cut	2 x 2 mm	0.5	DCM	30	40
2377	Yes	Used as received	2 x 2 mm	1	DCM	30	40
2379	Yes	Further Cut	2 x 2 mm	0.5	DCM	30	40
2380	Yes	Used as received	---	0.5	DCM	30	40
2382	Yes	Used as received	2 x 2 mm	1	DCM	30	40
2384	Yes	Further Grinded	<500 um	0.5	DCM	180	40
2386	Yes	Used as received	---	0.2	Methylene Chloride	30	40
2390	Yes	Further Cut	2 x 2 mm	0.5	DCM	30	40
2462	Yes	Further Cut	2 x 2 mm	1	DCM / Methanol (1:1)	60	60
2475	---	---	---	---	---	---	---
2508	Yes	Used as received	---	0.5	THF	60	60
2590	No	Used as received	---	1	THF/ACN	60	60
2629	Yes	Used as received	---	0.5	DCM	60 / 15	40
2665	Yes	Used as received	---	0.5	Xylene; DCM	60	130 / 60
2672	Yes	Used as received	<2 mm	0,1	DCM / Methanol	60	60
2736	No	Further Cut	5 x 5 mm		DCM	60	room temp
2773	No	Further Cut	1 x 1 mm	1	THF/ACN/Water	60	70
2812	No	Used as received	---	1	DCM	30	40
2826	Yes	Further Cut	4 x 4 mm	0.5	DCM / Methanol	30	40
2858	No	Used as received	---	0.5	THF/Hexane	60	60
2914	No	Used as received	---	0.5	Toluene	120	30
3100	Yes	Used as received	---	0.5	THF	30	70
3116	Yes	Used as received	5 x 5 mm	0.5	DCM/Acetone; 1:1 MeOH / water	60	40
3153	No	Used as received	2 x 3 mm	0.2	THF	30	70
3163	No	Further Cut	2 mm	0.2	Toluene	60	60
3166	---	Used as received	---	0.5	DCM	60	ambient
3172	Yes	Used as received	---	1.5	THF/Ethanol	60	25
3176	No	Used as received	---	1	THF/ACN	30	40
3182	No	Used as received	2 x 2 mm	0.5	THF/ACN	30	70
3185	Yes	Further Cut	2 x 2 mm	0.5	THF	30	70
3191	Yes	Used as received	2 x 2 mm	0.1	DCM	60	Normal temp.
3197	Yes	Further Cut	2 x 2 mm	0.5	THF/ACN	30	70
3210	Yes	Used as received	---	0.5	Toluene	60	60
3214	No	Further Cut	< 2 x 2 x 2 mm	0.5	THF/ACN	30	70
3218	Yes	Used as received	0.2 x 0.3 mm	0.5	THF	60	70
3248	Yes	Used as received	---	1	THF	30	40

APPENDIX 3

Number of participants per country

2 labs in BANGLADESH
1 lab in BELGIUM
1 lab in BRAZIL
1 lab in BULGARIA
3 labs in FRANCE
5 labs in GERMANY
5 labs in HONG KONG
4 labs in INDIA
2 labs in INDONESIA
2 labs in ITALY
1 lab in MALAYSIA
13 labs in P.R. of CHINA
1 lab in PAKISTAN
2 labs in SOUTH KOREA
2 labs in SWITZERLAND
1 lab in TAIWAN
2 labs in THAILAND
2 labs in THE NETHERLANDS
4 labs in TURKEY
3 labs in U.S.A.
3 labs 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
E = possibly 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
n.e. = not evaluated
fr. = first reported

Literature

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2 ASTM E178:02
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8 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
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12 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
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19 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)