Institute for
Interlaboratory Studies

Results of Proficiency Test
Total Bisphenol A in Polymers
June 2022

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. R.J. Starink
Correctors: ing. M. Meijer & Mrs. E.R. Montenij-Bos
Approved by: ing. A.S. Noordman-de Neef

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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. 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 2021/2022 it was decided to continue the proficiency test for the determination of Total Bisphenol A in Polymers.

In this interlaboratory study 64 laboratories in 23 countries registered for participation, see appendix 3 for the number of participants per country. In this report the results of the Total Bisphenol A in Polymers proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.
It was decided to send two different polymer samples of 3 grams each labelled #22625 and #22626 respectively.
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

For the first sample a batch of transparent Polycarbonate (PC) granulates was selected, which was made positive on Bisphenol A by a third-party laboratory. After homogenization 80 small plastic bags were filled with approximately 3 grams each and labelled #22625.
The batch for sample #22625 was used in a previous proficiency test on Total Bisphenol A in Polymers (as sample #18565 in iis18P04). Therefore, homogeneity of the subsamples was assumed.

For the second sample a batch of yellow Polyethylene (PE) granulates was selected, which was made positive on Bisphenol A by a third-party laboratory. After homogenization 80 small plastic bags were filled with approximately 3 grams each and labelled #22626.
The homogeneity of the subsamples was checked by the determination of Total BPA content using an in-house test method on 8 stratified randomly selected subsamples.

	Total BPA in mg/kg
sample #22626-1	2121
sample #22626-2	2065
sample #22626-3	2184
sample #22626-4	2073
sample #22626-5	2135
sample #22626-6	2149
sample #22626-7	2087
sample #22626-8	2250

Table 1: homogeneity test results of subsamples #22626

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

	Total BPA in mg/kg
r (observed)	174
reference test method	EN14372:04
0.3 x R (reference test method)	242

Table 2: evaluation of the repeatability of subsamples #22626

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 sample PC labelled #22625 and one sample PE labelled #22626 was sent on May 11, 2022.

2.5 ANALYZES

The participants were requested to determine the Total Bisphenol A content on both samples #22625 and #22626. 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 and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report ‘less than’ test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

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

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per determination in 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 and 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 Organisation, Statistics and Evaluation’ of June 2018 (iis-protocol, version 3.5).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

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

3.2 GRAPHICS

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

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve (dotted line) was projected over the Kernel Density

Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

3.3 Z-SCORES

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

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated according to:

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

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

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

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

4 EVALUATION

In this proficiency test some problems were encountered with the dispatch of the samples. Eight participants reported test results after the final reporting date and seven other participants were not able to report any test results.
In total 57 participants reported 112 numerical test results. Observed were 10 outlying test results, which is 8.9%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

Both data sets proved to have a normal Gaussian distribution.

4.1 EVALUATION PER SAMPLE AND PER COMPONENT

In this section the reported test results are discussed per sample and per component. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 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 #22625

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

sample #22626

Total BPA: The determination of Total Bisphenol A in the Polyethylene (PE) 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.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Component	unit	n	average	2.8 * sd	R(lit)
Total Bisphenol A	mg/kg	49	819	343	310

Table 3: reproducibilities of BPA determination on sample #22625

Component	unit	n	average	2.8 * sd	R(lit)
Total Bisphenol A	mg/kg	53	1656	934	626

Table 4: reproducibilities of BPA determination on sample #22626

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF JUNE 2022 WITH PREVIOUS PTS

	June 2022	May 2021	June 2020	June 2019	May 2018
Number of reporting laboratories	57	53	56	59	69
Number of test results	112	106	110	117	133
Number of statistical outliers	10	5	4	14	9
Percentage of statistical outliers	8.9%	4.7%	3.6%	10.7%	6.3%

Table 5: comparison with previous proficiency tests

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

The performance of the determinations of the proficiency test was compared to uncertainties observed in PTs over the years, expressed as relative standard deviation (RSD) of the PTs, per matrix see next table.

Matrix	June 2022	May 2021	June 2020	June 2019	2014 - 2018	EN14372
Polycarbonate (PC)	15%	13%	16%	18%	14 - 21%	14%
Polyethylene (PE)	20%	n.e.	n.e.	n.e.	24%	14%
Polypropylene (PP)	n.e.	33%	40%	n.e.	34 - 54%	14%
Polyvinylchloride (PVC)	n.e.	n.e.	n.e.	18%	21 - 23%	14%
Thermal paper (TP)	n.e.	n.e.	n.e.	n.e.	11 - 12%	14%

Table 6: development of uncertainties in BPA in polymers determinations over the years

The uncertainty of BPA in PC was in line with previous years. The determination of BPA in PE improved but is still high for the group of participants compared to the reference test method EN14372.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

It appeared that about 80% of the participants used an in-house test method for the determination of Total BPA. For this PT some analytical details were requested, the reported details are given in appendix 2. Based on the answers given by the participants the following can be summarized:

- About 55% of the participants answered to be ISO/IEC17025 accredited for the determination of Total BPA in polymers.
- About 50% of the participants did use the samples as received and about 45% of the participants further cut or further grinded the samples prior to analyses.
- About 70% of the participants used a sample intake between 0.5 and 1.0 grams and about 25% of the reporting participants mentioned to have used <0.5 grams.

- The solvent (mixture) to release the BPA from the samples differs. About 35% of the participants used Dichloromethane and 55% of the participants used Tetrahydrofurane (THF) as solvent.
- Almost all participants did use an extraction time between 30 and 60 min.
- About 40% of the participants reported to have used an extraction temperature of 40°C and about 55% mentioned to have used an extraction temperature of 60-70°C

No effect was observed on the averages or variation between reported test results. Therefore, no further investigations were done.

5 DISCUSSION

In this proficiency test the determination of Total BPA in Polymers two different sample matrices were used: Polycarbonate (#22625) and Polyethylene (#22626). For the PC sample the observed reproducibility was somewhat larger compared to the reproducibility as estimated from the reference test method of EN14372:04, but in line with previous findings for PC. For the PE sample the observed reproducibility was somewhat larger compared to the reproducibility as estimated from the reference test method of EN14372:04, but in line with the previous findings for PE.
It occurs that releasing BPA from Polyethylene is more difficult than from Polycarbonate resulting in a higher relative standard deviation (RSD) of 20% compared to an RSD of 15% from the PC matrix for the same group of participants.

Sample #22625 was used earlier as sample #18565 in the PT iis18P04.
In table 7 a comparison is given over the two proficiency tests.

	Sample #22625				Sample #18565			
	unit	n	average	R(calc)	unit	n	average	R(calc)
Total BPA	mg/kg	49	819	343	mg/kg	62	929	534

Table 7: comparison of sample #22625 with #18565

It is observed that the average level of Total BPA in the 2022 PT is somewhat lower but in line with the 2018 PT and the observed reproducibility R(calc) for BPA has improved in 2022. This is not uncommon. Each time that a laboratory participates in a PT it has the opportunity to learn from the evaluation of the results and improve the analysis.

6 CONCLUSION

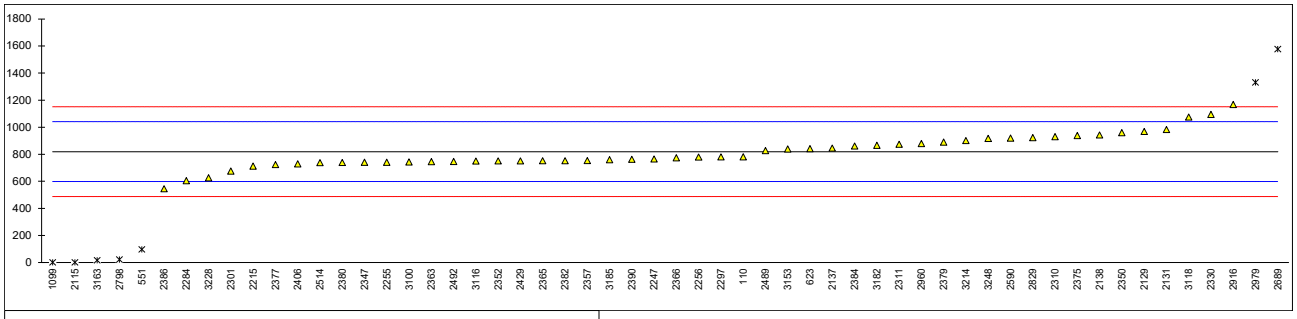
For the analysis of Total BPA from polymers a sound test method which prescribe the analysis of Total BPA from different polymers in detail is desirable, especially for other polymers than Polycarbonate.

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 a PC sample #22625; results in mg/kg

Lab	method	value	mark	z(targ)	remarks
110	In house	780.62		-0.35	
339		-----		-----	
551	JETRO2009	96.76	R(0.01)	-6.53	
623	In house	840.38		0.19	
1099	In house	0.536	R(0.01)	-7.40	
2115	In house	0.7	R(0.01)	-7.40	
2129	D7574Mod	968		1.34	
2131	In house	982		1.47	
2137	KS M1991	845.40		0.24	
2138	IEC62321-13	942.2		1.11	
2215	In house	713		-0.96	
2247	In house	764.89		-0.49	
2255	EN14372	741.0		-0.71	
2256	In house	778.5		-0.37	
2265		-----		-----	
2267		-----		-----	
2284	In house	605		-1.94	
2293		-----		-----	
2297	In house	780		-0.36	
2301	In house	676		-1.30	
2310	In house	930		1.00	
2311	In house	873.79		0.49	
2330	JETRO2009	1094.87		2.49	
2347	EPA3550C	740.3		-0.71	
2350	In house	959.25		1.26	
2352	JETRO2009	750.10		-0.63	
2357	In house	753.1		-0.60	
2363	In house	745		-0.67	
2365	In house	751.247		-0.62	
2366	In house	773.31		-0.42	
2375	In house	938		1.07	
2377	In house	723.38		-0.87	
2379	JETRO2009	890.07		0.64	
2380	In house	738.3		-0.73	
2382	In house	752.2		-0.61	
2384	In house	860.1331		0.37	
2386	In house	545.1		-2.48	
2390	In house	762.17		-0.52	
2406	In house	729		-0.82	
2429	In house	750.6		-0.62	
2475		-----		-----	
2489	In house	827		0.07	
2492	In house	746.09		-0.66	
2514	EN14372	737.78		-0.74	
2572		-----		-----	
2590	In house	919.118		0.90	
2689	In house	1576.3	R(0.01)	6.84	
2798	In house	21.71	R(0.01)	-7.21	
2829	In house	922.2		0.93	
2916	In house	1167		3.14	
2960		879.2		0.54	
2977	In house	<LOQ		-----	
2979	In house	1330	C,R(0.01)	4.62	First reported 2825
3100	In house	743.5		-0.69	
3116	In house	749.97		-0.63	
3118	In house	1074.7905		2.31	
3153	In house	838.8		0.18	
3163	EN14372	16	R(0.01)	-7.26	
3182	In house	865.46		0.42	
3185	In house	758.98		-0.55	
3201		-----		-----	
3214	In house	900.52		0.73	
3228	In house	625		-1.76	
3248	In house	917	C	0.88	First reported 0.092 mg/kg
	normality	OK			
	n	49			
	outliers	7			
	mean (n)	819.374			
	st.dev. (n)	122.4847	RSD = 15%		
	R(calc.)	342.957			
	st.dev.(EN14372:04)	110.6155			
	R(EN14372:04)	309.723			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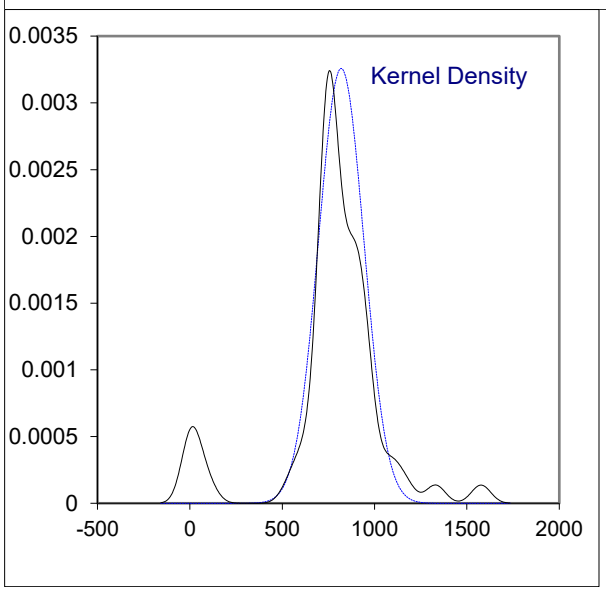
Kernel Density
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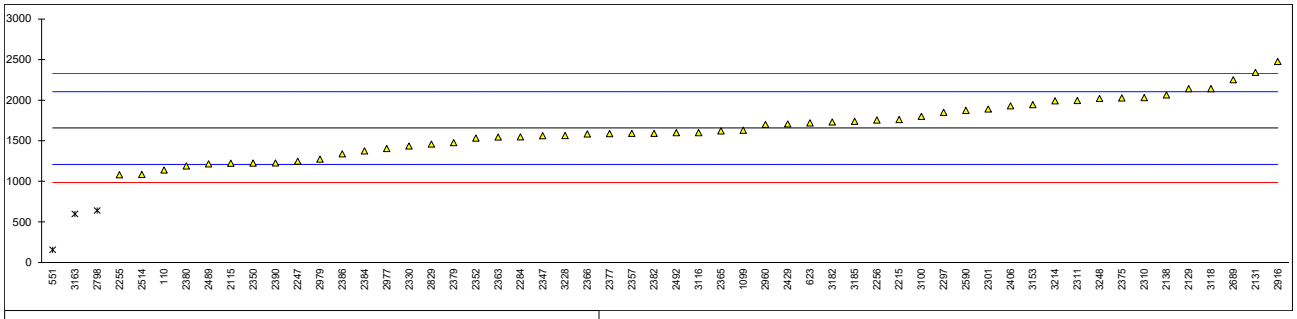
0.003
0.0025
0.002
0.0015
0.001
0.0005
0
-500
0
500
1000
1500
2000

Determination of Total Bisphenol A (BPA) in a PE sample #22626; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	In house	1138.10		-2.32	
339		-----		-----	
551	JETRO2009	155.82	R(0.05)	-6.71	
623	In house	1718.76		0.28	
1099	In house	1627.9	C	-0.13	First reported 16278.7
2115	In house	1222		-1.94	
2129	D7574Mod	2141		2.17	
2131	In house	2340		3.06	
2137		-----		-----	
2138	IEC62321-13	2064.1		1.82	
2215	In house	1760		0.46	
2247	In house	1246.96		-1.83	
2255	EN14372	1081.0		-2.57	
2256	In house	1756		0.44	
2265		-----		-----	
2267		-----		-----	
2284	In house	1546		-0.49	
2293		-----		-----	
2297	In house	1850		0.87	
2301	In house	1890		1.04	
2310	In house	2031		1.67	
2311	In house	1994.66		1.51	
2330	JETRO2009	1433.67		-1.00	
2347	EPA3550C	1561.5		-0.42	
2350	In house	1224.42		-1.93	
2352	JETRO2009	1531.41		-0.56	
2357	In house	1589.3		-0.30	
2363	In house	1544		-0.50	
2365	In house	1620.99		-0.16	
2366	In house	1582.28		-0.33	
2375	In house	2028		1.66	
2377	In house	1586.65		-0.31	
2379	JETRO2009	1475.10		-0.81	
2380	In house	1190.2		-2.09	
2382	In house	1590.1		-0.30	
2384	In house	1374.1696		-1.26	
2386	In house	1337.4		-1.43	
2390	In house	1224.90		-1.93	
2406	In house	1930		1.22	
2429	In house	1706.3		0.22	
2475		-----		-----	
2489	In house	1215		-1.97	
2492	In house	1598.75		-0.26	
2514	EN14372	1084.36		-2.56	
2572		-----		-----	
2590	In house	1873.522		0.97	
2689	In house	2250.3		2.66	
2798	In house	640.02	R(0.05)	-4.55	
2829	In house	1457.0		-0.89	
2916	In house	2475		3.66	
2960		1700.1		0.20	
2977	In house	1403.7948		-1.13	
2979	In house	1272		-1.72	
3100	In house	1798.0		0.63	
3116	In house	1599.80		-0.25	
3118	In house	2141.8795		2.17	
3153	In house	1944.0		1.29	
3163	EN14372	599	R(0.05)	-4.73	
3182	In house	1729.71		0.33	
3185	In house	1739.04		0.37	
3201		-----		-----	
3214	In house	1991.72		1.50	
3228	In house	1562		-0.42	
3248	In house	2020	C	1.63	First reported 0.202 mg/kg
	normality	OK			
	n	53			
	outliers	3			
	mean (n)	1656.488			
	st.dev. (n)	333.7439	RSD = 20%		
	R(calc.)	934.483			
	st.dev.(EN14372:04)	223.6259			
	R(EN14372:04)	626.152			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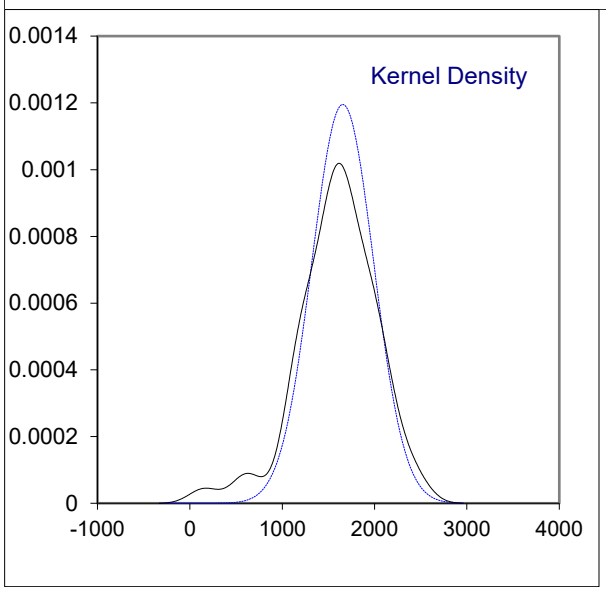
500
0
551 3163 2798 2255 2514 110 2380 2489 2115 2350 2390 2247 2979 2386 2384 2977 2330 2829 2379 2352 2363 2284 2347 3228 2366 2377 2357 2382 2492 3116 2365 1099 2960 2429 623 3182 3185 2256 2215 3100 2297 2590 2301 2406 3153 3214 2311 3248 2375 2310 2138 2129 3118 2689 2131 2916


Kernel Density
0.0014
0.0012
0.001
0.0008
0.0006
0.0004
0.0002
0
-1000
0
1000
2000
3000
4000

APPENDIX 2
Analytical details as reported by the participating laboratories

lab	ISO17025 accr.	sample used as received or grinded/cut	sample intake (g)	extraction solvent	extraction time (min)	extraction temp (°C)
						40°C DCM
110	Yes	Used as received	0.5 g	DCM and Acetone	30 min	25°C Acetone
339	---	---				
551	No	Used as received	1g	Dichloromethane	30	40
623	Yes	Further cut	1	THF	60	60
1099	No	Used as received	0,5	MeOH/H2O	60	40
2115	No	Used as received	1 gram	MeOH-H2O	60 min	60°C
2129	Yes	Other	0,4g	THF	30 min	Room temp.
2131	Yes	Used as received	0.5g	THF	60 Min	60 C
2137	Yes	Used as received	1	DCM/MeOH	5	
2138	No	Used as received	0.15 ~ 0.40	THF / MeOH:Water(1:1)	60	40
				Dichloromethane for 22625		40°C #22625
2215	No	Further cut	1g	Tetrahydrofuran for 22626	60 minutes.	60°C #22626
2247	Yes	Further cut	1.5 g	THF:ACN:Water (1:2:3)	60.0	70.0
2255	Yes	Further cut	0.5	THF+MeOH+ACN	60	70
2256	Yes	Further cut	1.0012 g/1.0031 g	DCM/THF	60mins	40℃
2265	---	---				
2267	---	---				
2284	No	Further cut	0.5	THF	60 min	60°c
2293	---	---				
2297	Yes	Used as received	0.5	DCM	30	40
2301	No	Used as received	1 gram	THF:Methanol	60 min	60c
2310	Yes	Used as received	1	THF	60	60
2311	Yes	Further cut	0.5	THF/Methanol	60	60
2330	Yes	Further cut	0.50 g	Dichloromethan	30 min	40 °C
2347	No	Further cut	0.5g	ACN:water 1:1	30min	40℃
2350	Yes	Further cut	0.5g	DCM	30 min	40
2352	Yes	Further cut	1g	Dichloromethane	30min	40℃
2357	---	---				
2363	Yes	Further cut	1g	DCM	30mins	40°C
2365	Yes	Further cut	1.0g	dichloromethane	30min	40℃
2366	No	Further cut	0.5	DCM	30min	40
2375	Yes	Further cut	0.5gr	THF	60min	60°C
2377	Yes	Used as received	1.0g	DCM	30	40
2379	Yes	Used as received	0 2 grams	DCM	30 minutes	40 C
2380	Yes	Used as received	0.5 g	Dichloromethane	30 Minute	40 °C
2382	Yes	Further cut	1.000g	DCM	30min	40 °C
2384	Yes	Further grinded	0.5g	Dichloromethane	180	40
2386	Yes	Used as received	0,5 g	Dichloromethane	30 min	40 °C
2390	Yes	Further cut	1g	Tetrahydrofuran	60	60
			#22625: 0.5g	#22625: Dichloromethane	60 min #22625	#22625: 40°C
2406	No	Further cut	#22626: 0.4g	#22626: Tetrahydrofuran	30 min #22626	#22626: 60°C
2429	Yes	Used as received	0.5001g	THF	30 minutes	70°C
2475	---	---				
2489	Yes	Further cut	0.5005g/0.5009g	THF/ACN/WATER	30 MINUTES	70 DEGREE
2492	Yes	Used as received	0.3 g	THF	60 mins	60 degree
2514	Yes	Further cut	0.5 g	THF:ACN:Water = 1:2:3	1.0 hr	70°C
2572	---	---				
						40°C DCM
2590	No	Used as received	0.5g	DCM #22625, THF #22626	60 min	60°C THF
2689	Yes	Further cut	0.1g	THF	60mins	60°C
2798	No	Further cut	0.25	Methanol	60min	70°C
2829	No	Further cut	0.200 g	THF/Hexane	60 min	60°C
2916	No	Used as received	0.1	Hexane / Acetone 80/20	60	60
2960	Yes	Used as received	0.05g	Tetrahydrofuran/Methanol	30min	60 °C
2977	No	Used as received	All	Methanol 100%	60 min	60 °C
2979	No	Used as received	0.5	DCM	30	40
3100	Yes	Used as received	0.5g	THF/Tetrahydrofuran	30mins	70℃
				THF / Methanol, 1:1 #22625		40°C #22625
3116	No	Used as received	1g	DCM / Acetone, 1:1 #22626	60 minutes	60°C #22626
3118	Yes	Further cut	0.5 gram	THF:ACN:Water 1:2:3	30 minutes	70 degree
3153	No	Used as received	0.2 gram	Tetrahydrofuran (THF)	30 minutes	70°C
3163	No	Other	0.02g	ethanol	2h	100C
3182	No	Used as received	0.1 gram	THF : ACN : water (1:2:3)	30 minutes	70 degree Celsius
3185	Yes	Used as received	0.5g	Tetrahydrofuran	30min	70°C
3201	---	---				
3214	Yes	Further cut	0.5	THF:ACN:H2O=1:2:3	30	70
3228	Yes	Used as received	0.25	THF	60	60
3248	Yes	Used as received	1g	THF	1 hour	60°C

APPENDIX 3

Number of participants per country

3 labs in	BANGLADESH
1 lab in	BRAZIL
1 lab in	CAMBODIA
1 lab in	EGYPT
2 labs in	FRANCE
5 labs in	GERMANY
1 lab in	GUATEMALA
6 labs in	HONG KONG
4 labs in	INDIA
3 labs in	INDONESIA
4 labs in	ITALY
3 labs in	KOREA, Republic of
1 lab in	MALAYSIA
18 labs in	P.R. of CHINA
1 lab in	PAKISTAN
1 lab in	POLAND
1 lab in	SWITZERLAND
1 lab in	TAIWAN
2 labs in	THAILAND
2 labs in	THE NETHERLANDS
1 lab in	TURKEY
1 lab in	U.S.A.
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
E	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
SDS	= Safety Data Sheet

Literature

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