Results of Proficiency Test
Bisphenol A in Plastic
May 2014

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. R.J. Starink
Corrector: dr. R.G. Visser & ing. N. Boelhouwer
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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 and vats. 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.

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 by the Institute for Interlaboratory Studies in April 2014.

In this new proficiency test iis14P04, 66 laboratories in 19 different countries did participate. See appendix 3 for the number of participating laboratories per country.
In this report the results of the 2014 proficiency test are presented and discussed.

2 SET UP

The Institute for Interlaboratory Studies in Spijkenisse was the organiser of this proficiency test. It was decided to send two different plastic samples. The first sample, a polypropylene (PP) granulate, was especially prepared by a Chinese factory by addition of Bisphenol A to PP. The second sample, a PVC granulate, was especially prepared by a Chinese factory by addition of Bisphenol A to PVC and subsequent homogenization. Analyses for fit-for-use and homogeneity were subcontracted. The participants were asked to report the analytical results with one extra figure using the indicated units on the report form. These results with an extra figure are 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. Also customer's satisfaction is measured on a regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol can be downloaded via the FAQ page of the iis website http://www.iisnl.com.

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 samples, one polypropylene and one PVC batch both artificially fortified to be positive on Bisphenol A (with respective approx. 0.21 %M/M and 0.39%M/M), were selected. Both materials were divided over plastic bags, approx. 3 grams for each sample.
The homogeneity of the subsamples was checked by determination of Bisphenol A (BPA) content on 7 stratified randomly selected subsamples.

	BPA in %M/M		BPA in %M/M
Sample #14067-1	0.2121	Sample #14068-1	0.4007
Sample #14067-2	0.2065	Sample #14068-2	0.3872
Sample #14067-3	0.2184	Sample #14068-3	0.3827
Sample #14067-4	0.2073	Sample #14068-4	0.3832
Sample #14067-5	0.2135	Sample #14068-5	0.3986
Sample #14067-6	0.2149	Sample #14068-6	0.3880
Sample #14067-7	0.2087	Sample #14068-7	0.3990

Table 1: homogeneity test results of the subsamples #14067 and #14068

From the above test results the repeatabilities were calculated and compared with 0.3 times the estimated reproducibility of EN14372:04 in agreement with the procedure of ISO 13528, Annex B2 in the next table;

	BPA in %M/M	BPA in %M/M
r (observed) #14067	0.0122	--
r (observed) #14068	--	0.0219
reference method	EN14372:04	EN14372:04
0.3 x R (ref. method)	0.0240	0.0443

Table 2: evaluation of repeatabilities of BPA contents of the subsamples #14067 and #14068

As the observed repeatabilities of the results of the homogeneity tests were all in agreement with the target precision data, the homogeneity of subsamples #14067 and #14068 was assumed.

To each of the participating laboratories, one sample of approx. 3 grams granulate, labelled #14067 and one sample of approx. 3 grams granulate (labelled #14068) were sent on April 30, 2014.

2.5 ANALYSIS

The participants were requested to determine and report the total Bisphenol A content on both samples #14067 and #14068.
The participants were explicitly asked to treat the samples as if they were routine samples and to report the analytical results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible.
The participants were also asked 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 results a detailed report form, on which the units were prescribed and a report form on which some analytical details were requested, was sent together with each set of samples. Also a letter of instructions was added to the package.
The laboratories were asked to complete the report form with the requested details of the methods used.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered. The original data are tabulated in the appendices of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder fax was sent to those laboratories that had not yet reported. Shortly after the deadline, the available results were screened for suspect data. A result was called suspect in case the Huber Elimination Rule (a robust outlier test, see lit.5) found it to be an outlier. The laboratories that produced these suspect data were asked to check the results. Additional or corrected data are placed under 'Remarks' in the result tables in appendix 1. A list of abbreviations used in the tables can be found in appendix 3.

3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3) For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded 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 (1986 and 1994, lit.8 and 9) the original results per determination were submitted subsequently to Dixon's, Grubbs' and Rosner 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 General ESD test (ref. 15). 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 General ESD test (ref. 15). 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 under the X-axis.

The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3, nos.13-14). Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. These results in an evaluation independent of the spread of this interlaboratory study. The target standard deviation was calculated from the literature reproducibility by division with 2.8.

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.
In case no literature reproducibility was available, other target values were used. In some cases literature repeatability is available; in other cases a reproducibility of a former iis proficiency test could be used and also the Horwitz equation can be used to estimate target reproducibility.

The z-scores were calculated according to:

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

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:

	$\lvert z\rvert < 1$	good
$1 <$	$\lvert z\rvert < 2$	satisfactory
$2 <$	$\lvert z\rvert < 3$	questionable
$3 <$	$\lvert z\rvert$	unsatisfactory

4 EVALUATION

In this interlaboratory study no problems were encountered during the execution.

Five participants did not report any test results due to several unknown reasons. Finally, 60 laboratories reported 120 numerical results. Observed were 6 statistically outlying test results, which is 4.8% of all results. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER SAMPLE

In this section the results are discussed per sample.
Almost all participants reported to have used an in house test method. However, when evaluating the test method details provided by the participants, differences are not large. The majority of the participants reported to have used an ultrasonic bad as technique for release of the BPA.
Due to the lack of a suitable test method with precision data, 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. For comparison the estimated reproducibility calculated using the Horwitz equation is also given.

Sample #14067

Bisphenol A: The determination of BPA was very problematic at the level of 0.145 %M/M. Three statistical outliers were detected. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the estimated reproducibility of EN14372:04. See also discussion (chapter 5)

Sample #14068

Bisphenol A: The determination of BPA was problematic at the level of 0.305 %M/M. Three statistical outliers were detected. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated reproducibility of EN14372:04. See also discussion (chapter 5)

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	%M/M	57	0.145	0.138	0.055

Table 4: overview of results for sample #14067

Parameter	Unit	n	Average	2.8 * sd	R (target)
Bisphenol A	%M/M	57	0.305	0.175	0.115

Table 5: overview of results for sample #14068

5 DISCUSSION

A number of different test methods were reported to have been used. Most often “in house” (49 laboratories) was mentioned as test method used, followed by JETRO2009 (5 laboratories) and EPA3550C (3 laboratories).
From the analytical details in appendix 2, it can be noticed that several different extraction techniques and solvents were used. It is remarkable to see that when only the reported results were evaluated of the laboratories that used Ultrasonic as release technique the calculated reproducibility was smaller (and the consensus value was higher) for sample #14067, while this had no consequences for the spread and consensus value of sample #14068.
When for both samples the data set of the group, that used Ultrasonic as release technique, was evaluated separately for the type of solvent used. It is surprisingly to see that only the calculated reproducibility of sample #14068 for the group that used THF as solvent meets the estimated reproducibility of EN14372:04. The three other data sets did not improve or even got worse.
Not surprisingly, the choice of solvent used is of utmost importance.

Parameter	Unit	n	Average	2.8 * sd	R (target)
Bisphenol A (only ultrasonic)	%M/M	49	0.151	0.116	0.057
Bisphenol A (DCM-ultrasonic)	%M/M	31	0.151	0.119	0.057
Bisphenol A (THF-ultrasonic)	%M/M	18	0.149	0.109	0.056

Table 6: overview of separate evaluation for sample #14067

Parameter	Unit	n	Average	2.8 * sd	R (target)
Bisphenol A (only ultrasonic)	%M/M	51	0.305	0.172	0.115
Bisphenol A (DCM-ultrasonic)	%M/M	34	0.295	0.191	0.112
Bisphenol A (THF-ultrasonic)	%M/M	18	0.326	0.107	0.123

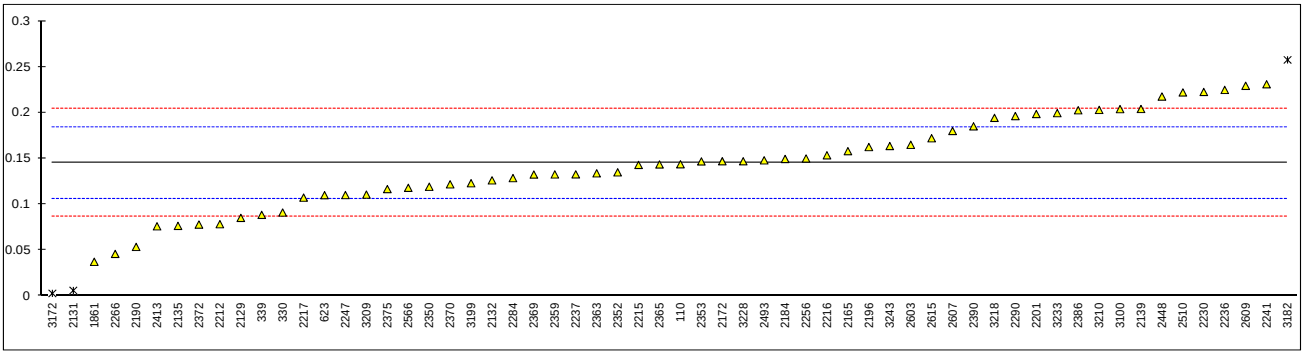
Table 7: overview of separate evaluation for sample #14068

It can be concluded that the observed spread in this interlaboratory study may not be caused by just one critical point in the analysis. Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary.

APPENDIX 1

Determination of Total Bisphenol A (BPA) on sample #14067; results in %M/M

lab	method	value	mark	z(targ)	remarks
110	INH-3352	0.143674	C	-0.08	Reported 1436.74 (unit error?)
330	in house	0.091		-2.76	
339	in house	0.08847		-2.89	
623	in house	0.11		-1.80	
1861	in house	0.03729		-5.50	
2129	in house	0.0852		-3.06	
2131	in house	0.0060	C,R(0.05)	-7.10	First reported 1195
2132	in house	0.1261105		-0.97	
2135	in house	0.07649	C	-3.50	Reported 764.9 (unit error?)
2139	INH-1997	0.204		3.00	
2165	in house	0.158		0.65	
2172	in house	0.147		0.09	
2184	in house	0.1495		0.22	
2190	in house	0.05352		-4.68	
2196	GB/T29609	0.1625		0.88	
2201		0.1985		2.72	
2212	in house	0.07834		-3.41	
2215	in house	0.143		-0.11	
2216	in house	0.1535		0.42	
2217	in house	0.1073		-1.93	
2230	EN14372	0.22261		3.95	
2236	in house	0.2248		4.06	
2237	in house	0.1327		-0.64	
2241	in house	0.231		4.38	
2247	in house	0.1101		-1.79	
2256	in house	0.15		0.25	
2266	in house	0.045867		-5.07	
2284	in house	0.1286	C	-0.85	First reported 0.0513
2290	in house	0.1963		2.61	
2295		-----		-----	
2350	JETRO 2009	0.1192		-1.33	
2352	JETRO 2009	0.1349		-0.52	
2353	in house	0.1468		0.08	
2359	JETRO 2009	0.1326		-0.64	
2363	JETRO 2009	0.1338		-0.58	
2365	EPA3550C	0.14352		-0.09	
2369	in house	0.1325		-0.65	
2370	in house	0.12181		-1.19	
2372	in house	0.0779		-3.43	
2375	JETRO 2009	0.1166		-1.46	
2386	in house	0.2026		2.93	
2390	INH-229	0.1851		2.04	
2413	in house	0.076	C	-3.53	First reported 0.319
2448	in house	0.2176		3.69	
2469		-----		-----	
2493	in house	0.148		0.14	
2510	in house	0.2221		3.92	
2566	in house	0.1180		-1.39	
2603	in house	0.1649482		1.01	
2607	in house	0.18		1.78	
2609	in house	0.2293		4.29	
2615	EPA3550C	0.1721		1.37	
2616		-----		-----	
3100	EPA3550C	0.2039		3.00	
3163		-----	W	-----	Results withdrawn, reported first 4300 mg/kg
3172	in house	0.0028	R(0.05)	-7.26	
3182	in house	0.2576	R(0.05)	5.74	
3199	CPSD-AN-00169	0.123006		-1.13	
3209	in house	0.11058	C	-1.77	Reported 1105.8 (unit error?)
3210	in house	0.203		2.95	
3218	in house	0.1943		2.51	
3220		-----		-----	
3228	in house	0.147		0.09	
3233	in house	0.1995		2.77	
3243	in house	0.1636	C	0.94	First reported 0.196
	normality	OK			
	n	57			
	outliers	3			
	mean (n)	0.14519			
	st.dev. (n)	0.049106			
	R(calc.)	0.13750			
	R(EN14372:04)	0.05488			Compare R(Horwitz) = 0.02174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0.25
0.2
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0
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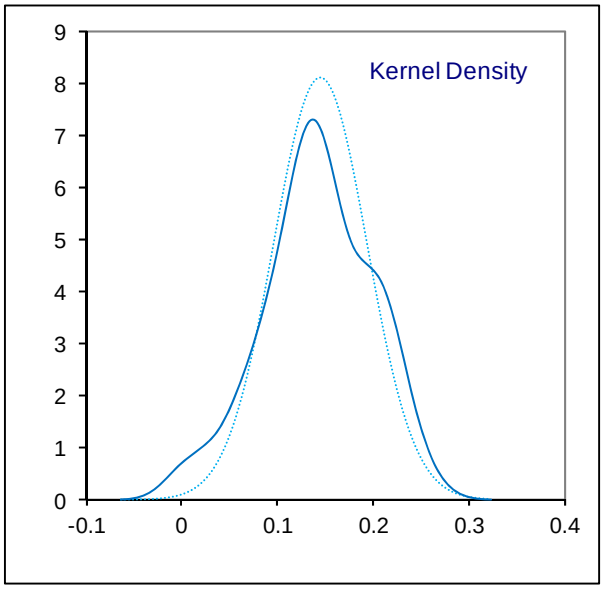
Kernel Density
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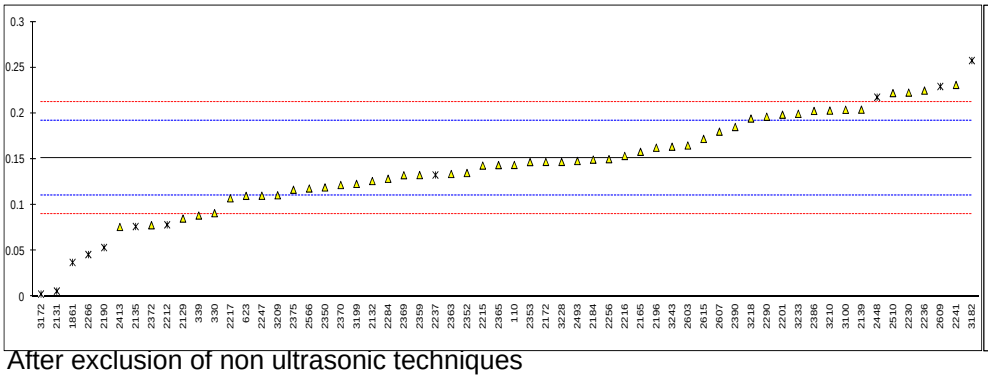
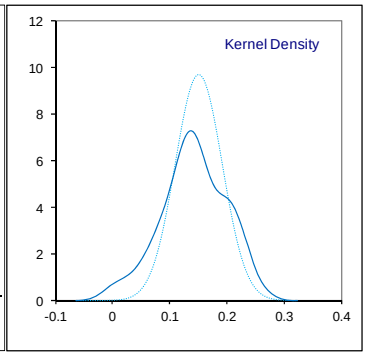
8
7
6
5
4
3
2
1
0
-0.1
0
0.1
0.2
0.3
0.4

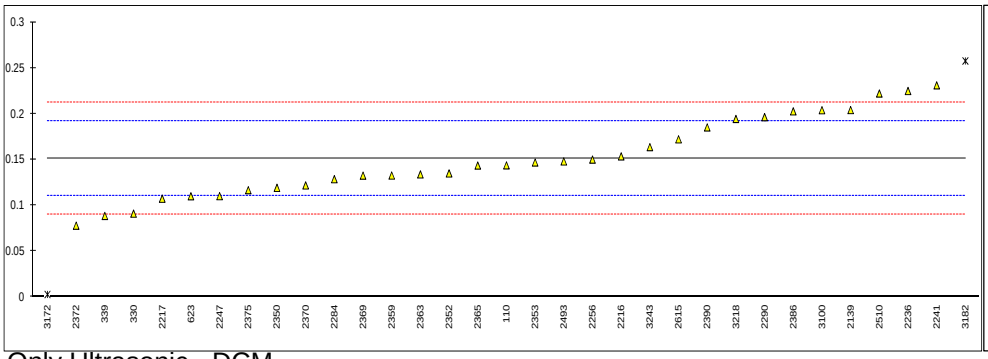
Determination of Total Bisphenol A (BPA) on sample #14067; results in %M/M (evaluated per technique and solvent)

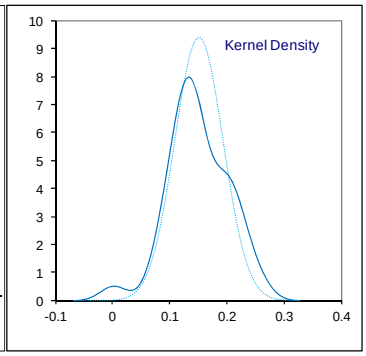




After exclusion of non ultrasonic techniques

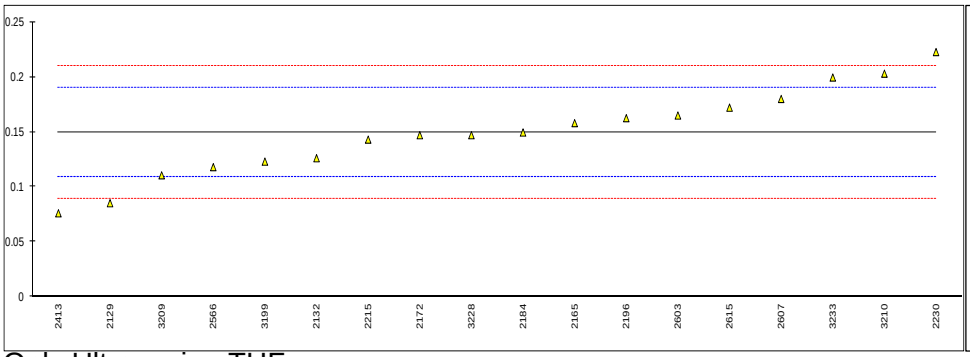
normality	OK	
n	49	
outliers	2	+ 9 excl.
mean (n)	0.1511	
st.dev. (n)	0.04127	
R(calc.)	0.1156	
R(EN14372:04)	0.0571	Compare R(Horwitz) = 0.0225

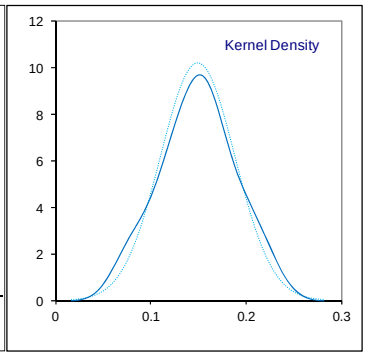




Only Ultrasonic - DCM

Normality	OK	
N	31	
Outliers	2	
mean (n)	0.1513	
st.dev. (n)	0.04239	
R(calc.)	0.1187	
R(EN14372:04)	0.0572	Compare R(Horwitz) = 0.0225



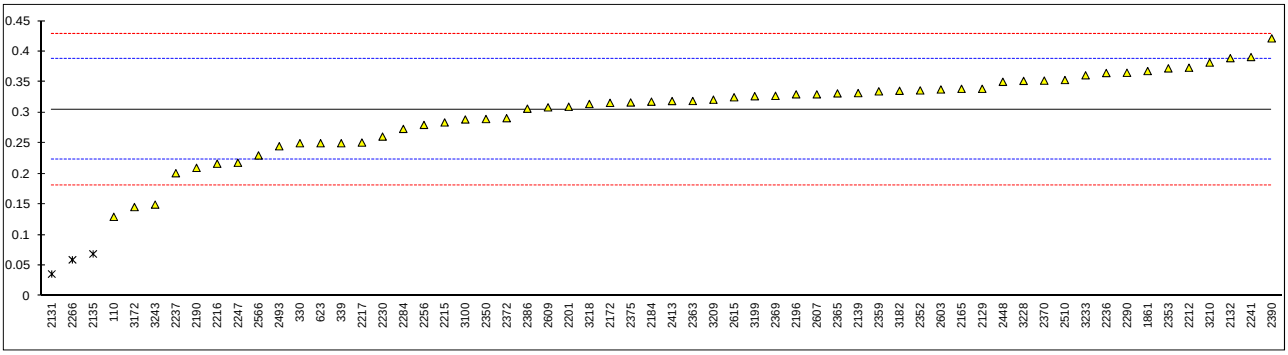


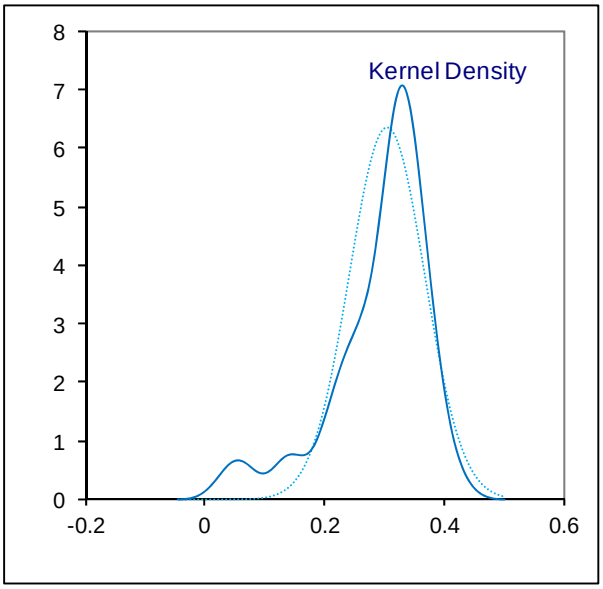
Only Ultrasonic - THF

normality	OK	
n	18	
outliers	0	
mean (n)	0.1493	
st.dev. (n)	0.03909	
R(calc.)	0.1094	
R(EN14372:04)	0.0564	Compare R(Horwitz) = 0.0223

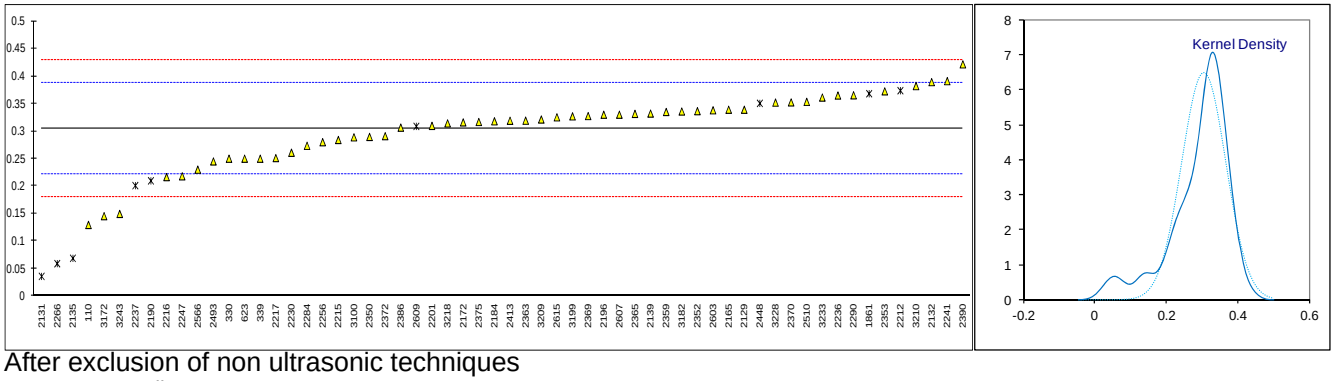
Determination of Total Bisphenol A (BPA) on sample #14068; results in %M/M

lab	method	value	mark	z(targ)	remarks
110	INH-3352	0.129535	C	-4.26	Reported 1295.35 (unit error?)
330	in house	0.25		-1.34	
339	in house	0.25		-1.34	
623	in house	0.25		-1.34	
1861	in house	0.3681		1.53	
2129	in house	0.339		0.83	
2131	in house	0.0359	C,R(0.05)	-6.54	First reported 7145
2132	in house	0.3892075		2.04	
2135	in house	0.06877	C,R(0.05)	-5.74	Reported 687.7 (unit error?)
2139	INH-1997	0.332		0.66	
2165	in house	0.339		0.83	
2172	in house	0.316		0.27	
2184	in house	0.3180		0.32	
2190	in house	0.20964		-2.32	
2196	GB/T29609	0.33		0.61	
2201		0.3097		0.11	
2212	in house	0.3736		1.67	
2215	in house	0.284		-0.51	
2216	in house	0.2165		-2.15	
2217	in house	0.2511		-1.31	
2230	EN14372	0.26080		-1.07	
2236	in house	0.3649		1.45	
2237	in house	0.2009		-2.53	
2241	in house	0.391		2.09	
2247	in house	0.2180		-2.11	
2256	in house	0.28		-0.61	
2266	in house	0.059114	R(0.05)	-5.97	
2284	in house	0.2734	C	-0.77	First reported 0.2055
2290	in house	0.3653		1.46	
2295		-----		-----	
2350	JETRO 2009	0.2896		-0.37	
2352	JETRO 2009	0.3364		0.76	
2353	in house	0.3725		1.64	
2359	JETRO 2009	0.3349		0.73	
2363	JETRO 2009	0.3191		0.34	
2365	EPA3550C	0.33154		0.64	
2369	in house	0.3275		0.55	
2370	in house	0.3524275		1.15	
2372	in house	0.291		-0.34	
2375	JETRO 2009	0.3167		0.28	
2386	in house	0.3065		0.04	
2390	INH-229	0.4216		2.83	
2413	in house	0.319	C	0.34	First reported 0.076
2448	in house	0.3505		1.10	
2469		-----		-----	
2493	in house	0.245		-1.46	
2510	in house	0.3534		1.17	
2566	in house	0.2300		-1.82	
2603	in house	0.3379483909		0.80	
2607	in house	0.33		0.61	
2609	in house	0.3087		0.09	
2615	EPA3550C	0.3252		0.49	
2616		-----		-----	
3100	EPA3550C	0.2889		-0.39	
3163		-----	W	-----	Results withdrawn, first reported 49700 mg/kg
3172	in house	0.14551		-3.87	
3182	in house	0.3358		0.75	
3199	CPSD-AN-00169	0.326929		0.53	
3209	in house	0.32122	C	0.39	Reported 3212.2 (unit error?)
3210	in house	0.382		1.87	
3218	in house	0.3142		0.22	
3220		-----		-----	
3228	in house	0.352		1.14	
3233	in house	0.3612		1.36	
3243	in house	0.1495		-3.78	
	normality	OK			
	n	57			
	outliers	3			
	mean (n)	0.30503			
	st.dev. (n)	0.062632			
	R(calc.)	0.17537			
	R(EN14372:04)	0.11530			Compare R(Horwitz) = 0.04085


0.45
0.4
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
2131 2266 2135 110 3172 3243 2237 2190 2216 2247 2566 2493 330 623 339 2217 2230 2284 2256 2215 3100 2350 2372 2386 2609 2201 3218 2172 2375 2184 2413 2363 3209 2615 3199 2369 2196 2607 2365 2139 2359 3182 2352 2603 2165 2129 2448 3228 2370 2510 3233 2236 2290 1861 2353 2212 3210 2132 2241 2390

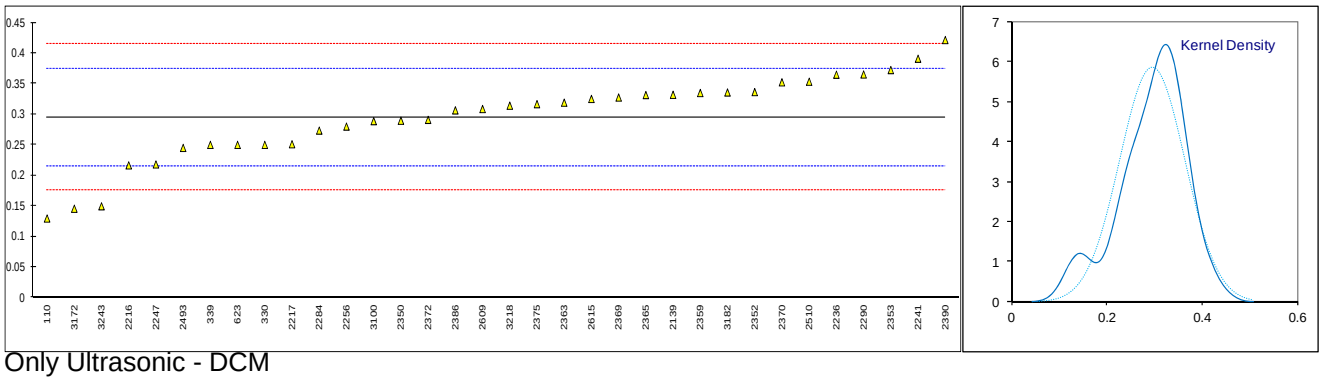

Kernel Density
8
7
6
5
4
3
2
1
0
-0.2
0
0.2
0.4
0.6

Determination of Total Bisphenol A (BPA) on sample #14068; results in %M/M (evaluated per technique and solvent)



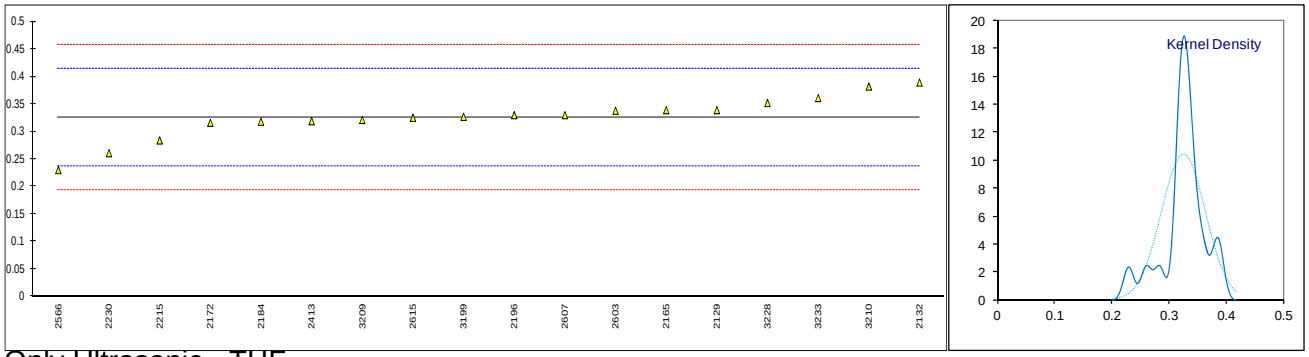
After exclusion of non ultrasonic techniques

normality	suspect	
n	51	
outliers	2	+ 7 excl.
mean (n)	0.3054	
st.dev. (n)	0.06148	
R(calc.)	0.1721	
R(EN14372:04)	0.1154	Compare R(Horwitz) = 0.0409



Only Ultrasonic - DCM

normality	OK	
n	34	
outliers	0	
mean (n)	0.2952	
st.dev. (n)	0.06819	
R(calc.)	0.1909	
R(EN14372:04)	0.1116	Compare R(Horwitz) = 0.0397



Only Ultrasonic - THF

normality	suspect	
n	18	
outliers	0	
mean (n)	0.3256	
st.dev. (n)	0.03837	
R(calc.)	0.1074	
R(EN14372:04)	0.1231	Compare R(Horwitz) = 0.0432

APPENDIX 2
Method information

Lab	maximum particle size	technique to release BPA	extraction solvent used	quantification technique	recovery checked
110	3x3 mm	Ultrasonic	Dichloromethane	LC-MS	Yes, 106%
330	As received	Ultrasonic	Dichloromethane	HPLC/MS/MS	Yes
339	As received	Ultrasonic	Methanol/Dichloromethane	HPLC/MS/MS	No
623	5x5 mm	Ultrasonic	Dichloromethane	LC-MS	Yes, 112
1861	<200 ul	Ultrasonic	Acetonitrile	--	No
2129	3 mm	Ultrasonic	THF/methanol	LC/MS/MS	No
2131	--	Soxhlet	Dichlormethane	LC/MS/MS	No
2132	As received	Ultrasonic	THF	LC/MS/MS	No
2135	As received	Ultrasonic	Methanol	GC/MS	No
2139	90 µm	Ultrasonic	Dichloromethane	LC-FLD	No
2165	3x3x3 mm	Ultrasonic	THF	LC/MS/MS	No
2172	<2 mm	Ultrasonic	THF	LC-MS	Yes, 103
2184	3x3x3 mm	Ultrasonic	THF	LC/MS/MS	No
2190	As received	ASE	Acetone/Hexane	GC/MS (Sim)	No
2196	1 mm	Ultrasonic	THF/methanol	HPLC	--
2201	--	--	--	--	--
2212	2 mm	Shaking	Dichloromethane	HPLC-MS	Yes, 100.6%
2215	3x3 mm	Ultrasonic	THF	HPLC-MS	No
2216	ground	--	Dichloromethane	HPLC-MS	Yes, 110%
2217	--	Ultrasonic	Dichloromethane	HPLC-DAD	Yes, 107%
2230	--	Ultrasonic	THF	--	Yes, 103%
2236	As received	Ultrasonic	Chloroform/methanol	HPLC-FLD	Yes, 101.6%
2237	>1mm	Ultrasonic	Toluene	GC/MSD	Yes, 94%
2241	2x2x2 mm	Ultrasonic	Dichloromethane	LC	No
2247	As received	Ultrasonic	Chloroform/methanol	LC/MS/MS	No
2256	5x5 mm	Ultrasonic	Dichloromethane	HPLC-MS	Yes, 85%
2266	As received	Ultrasonic	Methanol	GC-MS	No
2284	5x5 mm	Ultrasonic	Methanol/Dichloromethane	HPLC-FLD	No
2290	3x3 mm	Ultrasonic	Chloroform/methanol	LC/MS/MS	Yes, 95%
2295	--	--	--	--	--
2350	5x5 mm	Ultrasonic	Dichloromethane	LC/DAD/MS	Yes, 111%
2352	2x2 mm	Ultrasonic	Dichloromethane/acetone	--	Yes, 98.9%
2353	5x5 mm	Ultrasonic	Dichloromethane	LC/MS/MS	No
2359	2x2x2 mm	Ultrasonic	Dichloromethane	HPLC/DAD/MS	Yes, 91.3%
2363	<2x2 mm	Ultrasonic	Dichloromethane	HPLC-FLD	--
2365	2x2 mm	Ultrasonic	Dichloromethane	HPLC/MS/FLD	Yes, 100.6%
2369	2x2x2 mm	Ultrasonic	Dichloromethane	HPLC-FLD/LC/MS/MS	Yes, 98.2 %
2370	5x5 mm	Ultrasonic	Dichloromethane	LC-MS	Yes, 97.4 %
2372	3 mm	Ultrasonic	Dichloromethane	LC/MS/MS	Yes, 108%
2375	2x2 mm	Ultrasonic	Dichloromethane	HPLC/DAD/MS	Yes, 95%
2386	As received	Ultrasonic	Dichloromethane	LC/MS/MS	No
2390	As received	Ultrasonic	Dichloromethane	HPLC-DAD/MS	Yes, 133%
2413	As received	Ultrasonic	THF	GC-MS	No
2448	As received	Reflux	Cyclohexane	HPLC-DAD	Yes, 119%
2469	--	--	--	--	--
2493	<0.1 mm	Ultrasonic	Dichloromethane	GC-MS	No
2510	1x1x1 mm	Ultrasonic	Dichloromethane	HPLC-FLD	No
2566	5x5 mm	Ultrasonic	THF	LC-MS	Yes, 95%
2603	--	Ultrasonic	THF/methanol	--	No
2607	As received	Ultrasonic	THF	HPLC-FLD	Yes, 90%
2609	As received (#14067)	Dissolving	Toluene	HPLC-FLD	No
	1 mm (#14068)	Ultrasonic	Dichloromethane	HPLC-FLD	No
2615	0.1x0.1 mm	Ultrasonic	THF	HPLC-FLD	No
2616	--	--	--	--	--
3100	3x3 mm	Ultrasonic	Chloroform/methanol	HPLC-FLD/MS/MS	Yes, 105%
3163	As received	Thermaldesorption	--	GC/MS	--
3172	--	Ultrasonic	Dichloromethane	LC-FLD/MS	No
3182	No	Ultrasonic	Chloroform/methanol	HPLC	No
3199	As received	Ultrasonic	THF	HPLC/MS	Yes, 94.5%
3209	5x5 mm	Ultrasonic	THF	HPLC/MS/MS	Yes, 95%
3210	As received	Ultrasonic	THF	HPLC-Fluorescence	No
3218	As received	Ultrasonic	Chloroform/methanol	HPLC-FLD	Yes, 98%
3220	--	--	--	--	--
3228	3x3x3 mm	Ultrasonic	THF	LC/MS/MS	No
3233	As received	Ultrasonic	THF	LC/MS	Yes, 95.6%
3243	<2 mm	Ultrasonic	Dichloromethane	GC-MS	No

APPENDIX 3

Number of participating laboratories per country

6 labs in	FRANCE
5 labs in	GERMANY
5 labs in	HONG KONG
2 labs in	HUNGARY
3 labs in	INDIA
1 lab in	INDONESIA
1 lab in	IRELAND
3 labs in	ITALY
3 labs in	KOREA
20 labs in	P.R. of CHINA
1 lab in	PAKISTAN
1 lab in	SAUDI ARABIA
1 lab in	SWITZERLAND
3 labs in	TAIWAN R.O.C.
2 labs in	THAILAND
1 lab in	THE NETHERLANDS
2 labs in	TURKEY
5 labs in	U.S.A.
1 lab in	VIETNAM

APPENDIX 4

Abbreviations:

C = final result after checking of first reported suspect 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' outlier test
R(0.05) = straggler in Rosner' outlier test
n.a. = not applicable
n.d. = not detected
fr = first reported result

Literature:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, January 2010
2 ASTM E178-02
3 ASTM E1301-03
4 ISO 5725: 1986
5 ISO 5725, parts 1-6, 1994
6 2001/804/EC, Official Journal of the European Communities, L304/26, (2001)
7 98/485/EC, Official Journal of the European Comminities, L217/35, (1998)
8 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
9 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
10 IP 367/84
11 DIN 38402 T41/42
12 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
13 J.N. Miller, Analyst, 118, 455, (1993)
14 ASTM F963:"standard consumer safety specification on toy safety"
15 Analytical Methods Committee Technical brief, No4 January 2001.
16 The Royal Society of Chemistry 2002, Analyst 2002, 127 pages 1359-1364, P.J. Lowthian and M. Thompson
17 ISO/FDIS 13528, 2005e, Statistical methods for use in proficiency testing by interlaboratory comparisons
18 R.G. Visser, Reliability of proficiency test results for metals and phthalates in plastics, Accred Qual Assur, 14:29-34 (2009)
19 Annex XVII to REACH Regulation 1907/2006
20 Guideline on the interpretation of the concept "which can be placed in the mouth" as laid down in the entry 52 of Annex XVII to REACH Regulation 1907/2006, published July 18, 2012 on http://echa.europe.eu
21 16 C.F.R. Part 1501 and 16 C.F.R. 1500.50-53
22 16 C.F.R. 1199, Federal Register /Vol. 78, No. 31 /Thursday, February 14, 2013
23 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, T*echnometrics*, 25(2), pp. 165-172, (1983)