Results of Proficiency Test
Phthalates in Textile
March 2019

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Phthalates are commonly used as plasticizers to increase softness of plastic, especially in PVC. In the clothing industry, they can be found in synthetic leather, buttons, coated fabric, plastisol and dye printing. However, no appropriate textile reference materials are yet available. As an alternative, participation in a proficiency test may enable laboratories to check and improve their performance. Therefore, on request of some laboratories the Institute for Interlaboratory Studies (iis) decided to set up a proficiency test for the determination of Phthalates in textile during the annual testing program 2018/2019.

In this first interlaboratory study, 71 laboratories in 23 different countries registered for participation. See appendix 4 for the number of participating laboratories per country. In this report, the results of the 2019 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 analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send two different textile samples of 3 grams each, one sample of yellow pieces of textile labelled #19514 and one sample of pink pieces of textile labelled #19515. Both samples #19514 and #19515 were positive on Phthalates. 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/IEC 17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on a regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the 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). 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 first textile batch was a yellow cotton which was enriched with Dibutylphthalate (DBP). The material was cut in small pieces and after homogenization subsamples of 3 grams each were packed in plastic bags and labelled #19514. The homogeneity of the subsamples #19514 was checked by determination of DBP on 10 stratified randomly selected subsamples.

	DBP in %M/M
sample #19514-1	0.0706
sample #19514-2	0.0783
sample #19514-3	0.0784
sample #19514-4	0.0805
sample #19514-5	0.0828
sample #19514-6	0.0810
sample #19514-7	0.0877
sample #19514-8	0.0871
sample #19514-9	0.0860
sample #19514-10	0.0770

Table 1: homogeneity test result of subsamples #19514

From the above test results the relative standard deviation (RSD%) was calculated and compared with 0.3 times the estimated relative standard deviation calculated from the uncertainties (relative in %) of the iis PTs Phthalates in Polymers conducted from 2010-2016 (see iis memo 1701, lit. 14 and paragraph 4.1), and in agreement with the procedure of ISO 13528, Annex B2 in the next table.

	DBP
RSD% (observed)	6
reference method	iis memo 1701
0.3 * RSD% (ref. method)	5

Table 2: evaluation of the repeatability of subsamples #19514

The second textile batch was a pink cotton which was enriched with Benzylbutylphthalate (BBP). The material was cut in small pieces and after homogenization subsamples of 3 grams each were packed in plastic bags and labelled #19515. The homogeneity of the subsamples #19515 was checked by determination of BBP on 10 stratified randomly selected subsamples.

	BBP in %M/M
sample #19515-1	0.1167
sample #19515-2	0.1398
sample #19515-3	0.1100
sample #19515-4	0.1213
sample #19515-5	0.1287
sample #19515-6	0.1332
sample #19515-7	0.1335
sample #19515-8	0.1295
sample #19515-9	0.1352
sample #19515-10	0.1422

Table 3: homogeneity test result of subsamples #19515

From the above test results the relative standard deviation (%RSD) was calculated and compared with 0.3 times the estimated relative standard deviation calculated from the uncertainties (relative in %) of the iis PTs Phthalates in Polymers conducted from 2010-2016 (see iis memo 1701, lit. 14 and paragraph 4.1), and in agreement with the procedure of ISO 13528, Annex B2 in the next table.

	BBP
RSD% (observed)	8
reference method	iis memo 1701
0.3 * RSD% (ref. method)	5

Table 4: evaluation of the repeatability of subsamples #19515

The observed RSD of the homogeneity data was for the samples #19514 and #19515 larger than 0.3 times the estimated relative standard deviation calculated from the uncertainties (relative in %) of the iis PTs conducted from 2010-2016. It should be noted that the PTs mentioned in iis memo 1701 are all PTs of Phthalates in Polymers. Because it is the first time that iis conducted a proficiency test for Phthalates in Textile. Based on the experience of iis an observed RSD of the homogeneity data of ≤8% for PT samples is good enough to continue with this PT of Phthalates in Textile.
To each of the participating laboratories, one sample labelled #19514 and one sample labelled #19515 were sent on February 13, 2019.

2.5 ANALYSES

The participants were requested to determine fourteen individual Phthalates and the total of other Phthalates on samples #19514 and #19515. It was also requested to report if the laboratory was accredited for the determined 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 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 results cannot be used for meaningful statistical evaluations.

To get comparable 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 method (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 and 2 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 original test results are placed under 'Remarks' in the test 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 dataset does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

According to ISO 5725 the original test results per determination were submitted to Dixon’s, Grubbs’ and/or Rosner’s outlier tests. Outliers are marked by D(0.01) for the Dixon’s test, by G(0.01) or DG(0.01) for the Grubbs’ test and by R(0.01) for the Rosner’s test. Stragglers are marked by D(0.05) for the Dixon’s test, by G(0.05) or DG(0.05) for the Grubbs’ test and by R(0.05) for the Rosner’s test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. 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 was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation of 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. In some cases, a reproducibility based on former iis proficiency tests could be used.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test 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.
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 no problems were encountered with the dispatch of the samples. One participant reported after the final reporting date and two participants did not report any test results at all. Finally, 69 laboratories reported 188 numerical results. Observed were 8 statistically outlying test results, which is 4.3% of all results. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

All original data sets given in appendix 1 proved to have a normal Gaussion 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 methods are also in the tables together with the original data. The abbreviations, used in these tables, are listed in appendix 5.

Regretfully, the CPSC method does not contain any precision statements. ISO14389:14 does provide a variety of precision data. There are precision data mentioned for 4 different procedures in ISO14389:14 of which procedure 4, prescribes the extraction with THF followed by precipitation with Acetonitrile. The relative reproducibility for 7 different Phthtalates ranges from 31.5% - 124.9%.
Therefore, it is not surprising that in Annex D of test method ISO14389:14 is mentioned that "*Results indicated that both the four methods for Phthalates and the laboratories' performance have to be drastically improved*".

For several years iis organizes PTs on Phthalates in Polymers. In 2017, it was decided to use the iis PT data gathered since 2010, to estimate a more realistic target reproducibility in polymers (see iis memo 1701, lit 14). The target reproducibility was estimated by the relative standard deviation (16%) of the mean multiplied by 2.8.
It should be noted that the iis-memo 1701 is based on previous iis PTs of Phthalates in Polymers and not based on iis PTs of Phthalates in Textile. Because it is the first time that iis conducted a PT on Phthalates in Textile and test method ISO14389:14 does provide a variety of precision data it was therefore decided to use the estimated iis target reproducibility for the polymer PT also for the textile PT.

Sample #19514

DBP: The determination of DBP may be problematic at a consensus value of 0.04 %M/M. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the target reproducibility derived from the reproducibilities observed in previous iis PTs of Phthalates in Polymers, iis-memo 1701.

The majority of the participants agreed on a concentration near or below the limit of detection for BBP, DEHP, DIDP, DINP, DNOP, DCHP, DEP, DMP, DNHP, DIBP, DPHP, DNPP and DUP. Therefore, no significant conclusions were drawn for these Phthalates. These components are given in appendix 2.

Sample #19515

BBP: The determination of BBP may not be problematic. Six statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the target reproducibility derived from the reproducibilities observed in previous iis PTs of Phthalates in polymers, iis memo 1701.

DBP: The determination of DBP may be problematic at a consensus value of 0.008 %M/M. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the target reproducibility derived from the reproducibilities observed in previous iis PTs of Phthalates in polymers, iis memo 1701.

The majority of the participants agreed on a concentration near or below the limit of detection for DEHP, DIDP, DINP, DNOP, DCHP, DEP, DMP, DNHP, DIBP, DPHP, DNPP and DUP. Therefore, no significant conclusions were drawn for these Phthalates. These components are given in appendix 2.

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 target reproducibility as derived from the reproducibilities observed in previous iis PTs of Phthalates in Polymers, iis-memo 1701. The number of significant test results, the average result, the average result, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility are presented in the next tables.

Component	unit	n	average	2.8 * sd	R (target)
DBP	%M/M	68	0.043	0.038	0.019

Table 5: reproducibilities of tests on sample #19514

Component	unit	n	average	2.8 * sd	R (target)
BBP	%M/M	62	0.094	0.031	0.042
DBP	%M/M	51	0.0084	0.0068	0.0037

Table 6: reproducibilities of tests on sample #19515

Without further calculations, it could be concluded that for Benzylbutylphthalate (BBP) there is a good compliance of the group of participating laboratories with the target. For Dibutylphthalate (DBP) the group of participating laboratories show to have more difficulty in this determination. The problematic tests have been discussed in paragraph 4.1.

4.3 OVERVIEW OF PROFICIENCY TEST OF MARCH 2019

	March 2019
Number of reporting labs	69
Number of test results	188
Number of statistical outliers	8
Percentage outliers	4.3%

Table 7: overview of proficiency test of March 2019

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

The uncertainty of the determinations in this proficiency test, expressed as relative standard deviation (RSD) of the PT iis19A03 are given in below table.

Component	March 2019	Iis Memo 1701
BBP	12%	16%
DBP	29% - 31%	16%

Table 8: overview of relative uncertainties (RSD)

The RSD of Benzylbutylphthalate (BBP) observed in this PT is comparable to the uncertainty as derived from previous iis PTs of Phthalates in Polymers while the RSD of Dibutylphthalate (DBP) observed in this PT is much larger.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

The test method ISO14389 is used by about 60% of the reporting participants, the test method CPSC-CH-C1001-09.3 is used by about 15% of the reporting participants and the test method CPSC-CH-C1001-09.4 is used by about 5% of the reporting participants.
For this PT also some analytical details were requested, see appendix 3. Based on the answers given by the participants the following can be summarized:

- About 80% of the reporting participants mentioned that they are accredited for the determination of Phthalates in Textile.
- About 55% of the reporting participants used less than 0.5 grams as sample intake, about 35% used 0.5 grams and about 5% used more than 0.5 grams as sample intake .
- About 40% of the reporting participants used the samples as received and about 50% further cut the samples prior to analysis.
- Almost all of the reporting participants used ultrasonic as technique to release/extract the Phthalates.
- Almost all of the reporting participants used THF or a THF mixture as extraction solvent.
- The extraction time differs from 30 minutes to 2.5 hours. About 80% of the reporting participants used an extraction time of 60 minutes.

- The extraction temperature differs from room temperature to 70°C. About 70% of the reporting participants used an extraction temperature of 60°C.
- About 5% did not report any analytical details.

When the analytical details where investigated separately, it appeared that the effect on the determination of Phthalates in textile is negligible.

5 DISCUSSION

In this proficiency test for the determination of Phthalates in Textile, it was noticed that the majority of the participants were able to detect the Phthalates present in sample #19514 and #19515. Regretfully, only the observed reproducibility of Benzylbutylphthalate (BBP) in sample #19515 was in agreement with the target reproducibility derived from the reproducibilities observed in previous iis PTs of Phthalates in Polymers (lit. 14, iis-memo 1701).

In this PT, the average of the homogeneity test results are not in line with the average (consensus value) from the PT results. There are several reasons for this, First, the goal of the homogeneity testing is different from the goal of the evaluation of the reported PT results. In order to prove the homogeneity of the PT samples, a test method is selected with a high precision (smallest variation). The accuracy (trueness) of the test method is less relevant. Secondly, the homogeneity testing is done by one laboratory only. The test results of this ISO/IEC 17025 accredited laboratory will have a bias (systematic deviation) depending on the test method used. The desire to detect small variations between the PT samples leads to the use of a sensitive test method with high precision, which may be a test method with significant bias.
Also each test result reported by the laboratories that participate in the PT will have a bias. However, some will have a positive bias and others a negative bias. These different biases compensate each other in the PT average (consensus value). Therefore, the PT consensus value may deviate from the average of the homogeneity test. At the same time the accuracy of the PT consensus value is more reliable than the accuracy of the average of the homogeneity test.

6 CONCLUSION

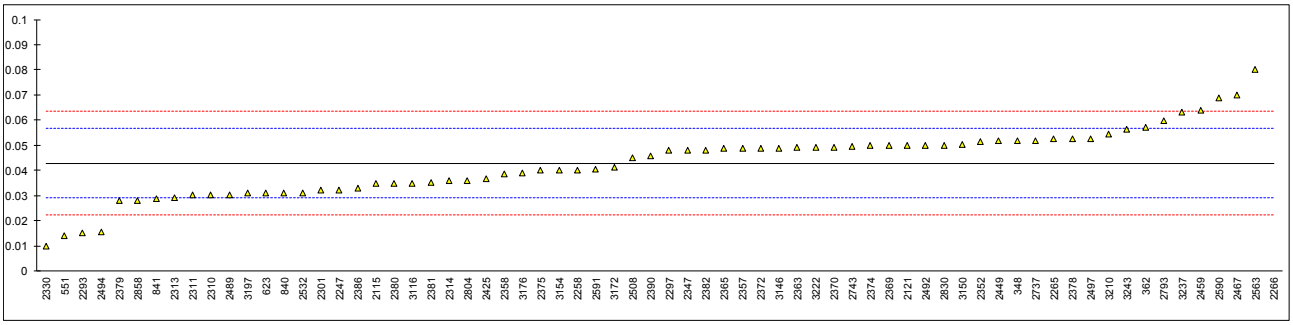
The majority of the participants has no problem with the determination of Benzylbutylphthalate (BBP) in textile. However, the determination of Dibutylphthalate (DBP) was more problematic at the concentration levels in this PT.
Each participating laboratory will have to evaluate its performance in this 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.

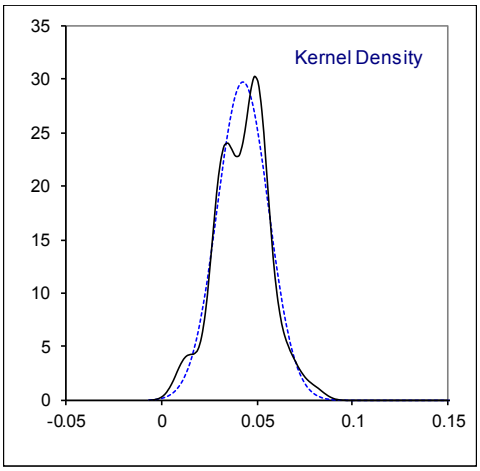
APPENDIX 1

Determination of DBP – Dibutylphthalate on sample #19514; results in %M/M

lab	method	value	mark	z(targ)	remarks
339	In house	<0.001		<-6.10	possibly a false negative test result?
348	CPSC-CH-C1001-09.4	0.0520		1.33	
362	ISO14389	0.057		2.06	
551	In house	0.014099		-4.20	
623	ISO14389	0.031		-1.73	
840	CPSC-CH-C1001-09.3	0.031		-1.73	
841	CPSC-CH-C1001-09.3	0.0287		-2.07	
2115	ISO14389	0.0347		-1.19	
2121	ISO14389	0.05		1.04	
2170		-----		-----	
2247	ISO14389	0.032	C	-1.59	first reported 18.33
2258	CPSC-CH-C1001-09.3	0.040125	C	-0.40	first reported 401.25 %M/M
2265	ISO14389	0.0524		1.39	
2266	ISO14389	0.48	R(0.01)	63.70	
2293	CPSC-CH-C1001-09.3	0.015	C	-4.06	first reported 151.51 %M/M
2297	ISO14389	0.048		0.74	
2301	ISO14389	0.032		-1.59	
2310	ISO14389	0.0302		-1.85	
2311	ISO14389	0.0302		-1.85	
2313	ISO14389	0.0293		-1.98	
2314	ISO14389	0.0358	C	-1.03	first reported 358.22 %M/M
2330	ISO14389	0.01		-4.79	
2347	GB/T20388	0.048		0.74	
2352	GB/T20388	0.0513		1.23	
2357	ISO14389	0.0487		0.85	
2358	ISO14389	0.03867		-0.61	
2363	ISO14389	0.049		0.89	
2365	ISO14389	0.04868		0.84	
2369	ISO14389	0.050		1.04	
2370	ISO14389	0.0491		0.91	
2372	ISO14389	0.0489		0.88	
2374	CPSC-CH-C1001-09.3	0.05		1.04	
2375	ISO14389	0.040		-0.42	
2378	ISO14389	0.0526		1.42	
2379	CPSC-CH-C1001-09.4	0.028		-2.17	
2380	ISO14389	0.0349		-1.16	
2381	CPSC-CH-C1001-09.3	0.035178		-1.12	
2382	ISO14389	0.0481		0.76	
2386	ISO14389	0.033		-1.44	
2390	ISO14389	0.0456		0.40	
2425	In house	0.0369		-0.87	
2449	CPSC-CH-C1001-09.3	0.0517	C	1.28	first reported 0.517
2459	ISO14389	0.0641		3.09	
2467		0.0699		3.94	
2476		-----		-----	
2489	ISO14389	0.0304	C	-1.82	first reported 304 %M/M
2492	In house	0.0500		1.04	
2494	CPSC-CH-C1001-09.3	0.0155		-3.99	
2497	ISO14389	0.0526		1.42	
2508		0.045062		0.32	
2532	ISO14389	0.0312		-1.70	
2563	CPSC-CH-C1001-09.3	0.08		5.41	
2590	ISO14389	0.06899		3.80	
2591	CPSC-CH-C1001-09.3	0.04038		-0.37	
2737	ISO14389	0.052		1.33	
2743	ISO14389	0.049631		0.98	
2793	ISO14389	0.05969		2.45	
2804	In house	0.036		-1.00	
2830	ISO14389	0.050		1.04	
2858		0.028		-2.17	
3116	ISO14389	0.035		-1.15	
3146	In house	0.0489		0.88	
3150	ISO14389	0.0504		1.09	
3154	ISO14389	0.04		-0.42	
3172		0.0413		-0.23	
3176	ISO14389	0.0389		-0.58	
3197	ISO14389	0.0309		-1.75	
3210	ISO/TS 16181	0.0545		1.69	
3222		0.049		0.89	
3237	CPSC-CH-C1001-09.3	0.063		2.93	
3243	In house	0.05634		1.96	

normality	OK	
n	68	
outliers	1	
mean (n)	0.0429	
st.dev. (n)	0.01339	RSD = 31%
R(calc.)	0.0375	
st.dev.(iis-memo 1701)	0.00686	
R(iis-memo 1701)	0.0192	

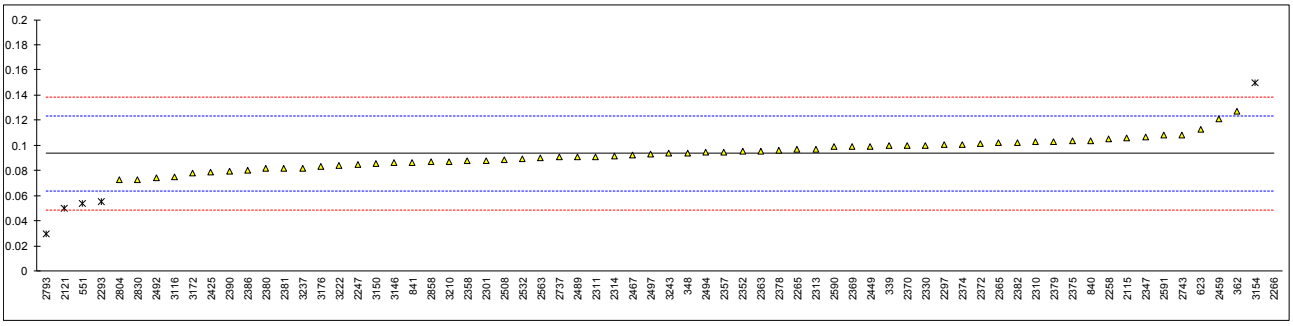


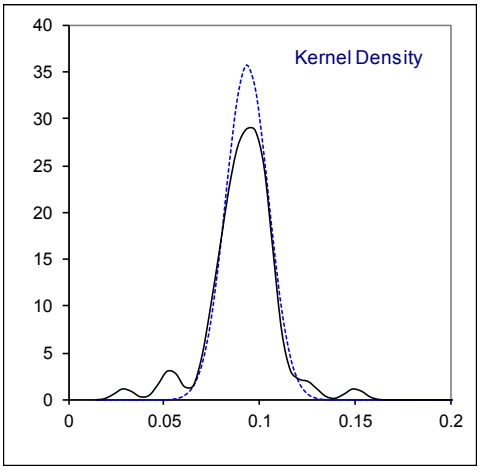


Determination of BBP – Benzylbutylphthalate on sample #19515; results in %M/M

lab	method	value	mark	z(targ)	remarks
339	In house	0.0997		0.41	
348	CPSC-CH-C1001-09.4	0.0937		0.01	
362	ISO14389	0.127		2.23	
551	In house	0.054029	R(0.01)	-2.64	
623	ISO14389	0.113		1.29	
840	CPSC-CH-C1001-09.3	0.104		0.69	
841	CPSC-CH-C1001-09.3	0.0864		-0.48	
2115	ISO14389	0.1059		0.82	
2121	ISO14389	0.05	R(0.01)	-2.91	
2170		-----		-----	
2247	ISO14389	0.085	C	-0.57	first reported 853.22 %M/M
2258	CPSC-CH-C1001-09.3	0.10554	C	0.80	first reported 1055.54 %M/M
2265	ISO14389	0.0965		0.19	
2266	ISO14389	1.18	R(0.01)	72.53	
2293	CPSC-CH-C1001-09.3	0.0556	C,R(0.01)	-2.54	first reported 556.86 %M/M
2297	ISO14389	0.101		0.49	
2301		0.088		-0.37	
2310	ISO14389	0.1028		0.61	
2311	ISO14389	0.091060		-0.17	
2313	ISO14389	0.0970		0.23	
2314	ISO14389	0.0916	C	-0.13	first reported 916.31 %M/M
2330	ISO14389	0.10		0.43	
2347	ISO14389	0.107		0.89	
2352	GB/T20388	0.0950		0.09	
2357	ISO14389	0.0949		0.09	
2358	ISO14389	0.08747		-0.41	
2363	ISO14389	0.095		0.09	
2365	ISO14389	0.10179		0.55	
2369	ISO14389	0.099	C	0.36	first reported as DBP
2370	ISO14389	0.0998		0.41	
2372	ISO14389	0.1014		0.52	
2374	CPSC-CH-C1001-09.3	0.101		0.49	
2375	ISO14389	0.104		0.69	
2378	ISO14389	0.0962		0.17	
2379	CPSC-CH-C1001-09.4	0.103		0.63	
2380	ISO14389	0.0815		-0.81	
2381	CPSC-CH-C1001-09.3	0.081759		-0.79	
2382	ISO14389	0.102		0.56	
2386	ISO14389	0.080		-0.91	
2390	ISO14389	0.0795		-0.94	
2425	In house	0.0785		-1.01	
2449	CPSC-CH-C1001-09.3	0.0995	C	0.39	first reported 0.995
2459	ISO14389	0.1210		1.83	
2467		0.0923		-0.09	
2476		-----		-----	
2489	ISO14389	0.0908	C	-0.19	first reported 908 %M/M
2492	In house	0.0743		-1.29	
2494	CPSC-CH-C1001-09.3	0.0947		0.07	
2497	ISO14389	0.0928		-0.05	
2508		0.0888		-0.32	
2532	ISO14389	0.0893		-0.29	
2563	CPSC-CH-C1001-09.3	0.09		-0.24	
2590	ISO14389	0.09897		0.36	
2591	CPSC-CH-C1001-09.3	0.10782		0.95	
2737	ISO14389	0.0905		-0.21	
2743	ISO14389	0.10854		1.00	
2793	ISO14389	0.0293	R(0.01)	-4.29	
2804		0.073		-1.38	
2830	ISO14389	0.073		-1.38	
2858	In house	0.0871		-0.43	
3116	ISO14389	0.075		-1.24	
3146	In house	0.086		-0.51	
3150	ISO14389	0.0852		-0.56	
3154	ISO14389	0.15	R(0.01)	3.76	
3172		0.07813		-1.03	
3176	ISO14389	0.08325		-0.69	
3197		-----		-----	
3210	ISO/TS 16181	0.0874		-0.41	
3222		0.084		-0.64	
3237	CPSC-CH-C1001-09.3	0.082		-0.78	
3243	In house	0.09347		-0.01	

normality	OK	
n	62	
outliers	6	
mean (n)	0.0936	
st.dev. (n)	0.01117	RSD = 12%
R(calc.)	0.0313	
st.dev.(iis-memo 1701)	0.01498	
R(iis-memo 1701)	0.0419	

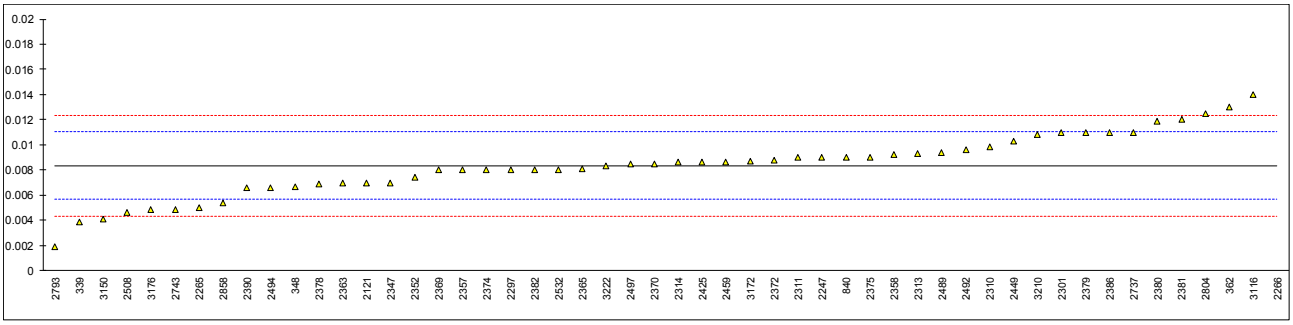


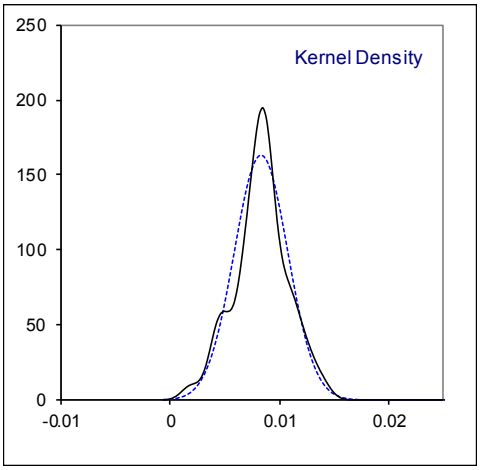


Determination of DBP – Dibutylphthalate on sample #19515; results in %M/M

lab	method	value	mark	z(targ)	remarks
339	In house	0.0039		-3.33	
348	CPSC-CH-C1001-09.4	0.0067		-1.24	
362	ISO14389	0.013		3.47	
551	In house	N.D.		-----	
623	ISO14389	n.d.		-----	
840	CPSC-CH-C1001-09.3	0.009		0.48	
841	CPSC-CH-C1001-09.3	ND		-----	
2115		-----		-----	
2121	ISO14389	0.007		-1.01	
2170		-----		-----	
2247	ISO14389	0.009	C	0.48	first reported 92.48 %M/M
2258	CPSC-CH-C1001-09.3	<0.0050	C	-----	first reported <50 %M/M
2265	ISO14389	0.005		-2.51	
2266	ISO14389	0.08	R(0.01)	53.59	
2293		-----		-----	
2297	ISO14389	0.0080		-0.27	
2301		0.011		1.98	
2310	ISO14389	0.0098		1.08	
2311	ISO14389	0.008973		0.46	
2313	ISO14389	0.0093		0.71	
2314	ISO14389	0.0086	C	0.18	first reported 86.22 %M/M
2330	ISO14389	ND		-----	
2347	ISO14389	0.007		-1.01	
2352	GB/T20388	0.0074		-0.72	
2357	ISO14389	0.008		-0.27	
2358	ISO14389	0.009229		0.65	
2363	ISO14389	0.007		-1.01	
2365	ISO14389	0.00808		-0.21	
2369	ISO14389	0.008	C	-0.27	first reported as BBP
2370	ISO14389	0.00850		0.11	
2372	ISO14389	0.00877		0.31	
2374	CPSC-CH-C1001-09.3	0.008		-0.27	
2375	ISO14389	0.009		0.48	
2378	ISO14389	0.0069		-1.09	
2379	CPSC-CH-C1001-09.4	0.011		1.98	
2380	ISO14389	0.0119		2.65	
2381	CPSC-CH-C1001-09.3	0.012		2.73	
2382	ISO14389	0.0080		-0.27	
2386	ISO14389	0.011		1.98	
2390	ISO14389	0.006578		-1.33	
2425	In house	0.0086		0.18	
2449	CPSC-CH-C1001-09.3	0.0103	C	1.45	first reported 0.153
2459	ISO14389	0.0086		0.18	
2467		< 0.0150		-----	
2476		-----		-----	
2489	ISO14389	0.0094	C	0.78	first reported 93.6 %M/M
2492	In house	0.0096		0.93	
2494	CPSC-CH-C1001-09.3	0.0066		-1.31	
2497	ISO14389	0.00846		0.08	
2508		0.00459		-2.82	
2532	ISO14389	0.008		-0.27	
2563	CPSC-CH-C1001-09.3	n.d		-----	
2590		-----		-----	
2591	CPSC-CH-C1001-09.3	<0.005		-----	
2737	ISO14389	0.011		1.98	
2743	ISO14389	0.00486		-2.62	
2793	ISO14389	0.00192		-4.81	
2804		0.0125		3.10	
2830	ISO14389	ND		-----	
2858	In house	0.0054		-2.21	
3116	ISO14389	0.014		4.22	
3146		-----		-----	
3150	ISO14389	0.0041		-3.18	
3154		-----		-----	
3172		0.00868		0.24	
3176	ISO14389	0.00483		-2.64	
3197		-----		-----	
3210	ISO/TS 16181	0.0108		1.83	
3222		0.0083		-0.04	
3237		-----		-----	
3243	In house	n.d.		-----	

normality	OK	
n	51	
outliers	1	
mean (n)	0.00836	
st.dev. (n)	0.002446	RSD = 29%
R(calc.)	0.00685	
st.dev.(iis-memo 1701)	0.001337	
R(iis-memo 1701)	0.00374	





APPENDIX 2

Abbreviations of components:

BBP = Benzylbutylphthalate
DEHP = Bis-2-ethylhexylphthalate
DBP = Dibutylphthalate
DIDP = Diisodecylphthalate
DINP = Diisononylphthalate
DNOP = Di-n-octylphthalate
DCHP = Dicyclohexylphthalate
DEP = Diethylphthalate
DMP = Dimethylphthalate
DNHP = Di-n-hexylphthalate
DIBP = Diisobutylphthalate
DPHP = Di(2-propylheptyl)phtalate
DNPP = Di-n-pentylphthalate
DUP = Diundecylphthalate

Other reported Phthalates in sample #19514; results in %M/M

Lab	BBP	DEHP	DIDP	DINP	DNOP	DCHP	DEP
339	<0.001	0.0549	<0.005	<0.005	<0.001	<0.001	<0.001
348	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
362	-----	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	-----
551	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
623	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
840	nd	nd	nd	nd	nd	nd	nd
841	ND	ND	ND	ND	ND	ND	ND
2115	-----	-----	-----	-----	-----	-----	-----
2121	-----	-----	-----	-----	-----	-----	-----
2170	-----	-----	-----	-----	-----	-----	-----
2247	ND (< 0.005)	ND (< 0.005)	ND (< 0.005)	ND (< 0.005)	ND (< 0.005)	ND (< 0.005)	ND (< 0.005)
2258	<0.0050 C	<0.0050 C	<0.0050 C	<0.0050 C	<0.0050 C	<0.0050 C	-----
2265	< 0,025	< 0,025	< 0,025	< 0,025	< 0,025	< 0,025	< 0,025
2266	0.02	0	0	0	0	0	-----
2293	-----	-----	-----	-----	-----	-----	-----
2297	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2301	ND	ND	ND	ND	ND	ND	ND
2310	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2311	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2313	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2314	-----	-----	-----	-----	-----	-----	-----
2330	ND	ND	ND	ND	ND	ND	ND
2347	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003
2352	-----	-----	-----	-----	-----	-----	-----
2357	ND	ND	ND	ND	ND	ND	ND
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2363	ND	ND	ND	ND	ND	ND	ND
2365	<0.003	<0.003	<0.005	<0.005	<0.003	<0.003	<0.003
2369	<0.003	<0.003	<0.010	<0.010	<0.003	<0.003	<0.003
2370	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2372	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2374	-----	-----	-----	-----	-----	-----	-----
2375	-----	-----	-----	-----	-----	-----	-----
2378	-----	-----	-----	-----	-----	-----	-----
2379	0.003	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2380	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003
2381	-----	-----	-----	-----	-----	-----	-----
2382	-----	-----	-----	-----	-----	-----	-----
2386	<0,003	<0,003	<0,003	<0,003	<0,003	<0,003	<0,003
2390	-----	-----	-----	-----	-----	-----	-----
2425	ND	ND	ND	ND	ND	ND	ND
2449	-----	-----	-----	-----	-----	-----	-----
2459	-----	-----	-----	-----	-----	-----	-----
2467	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
2476	-----	-----	-----	-----	-----	-----	-----
2489	ND	ND	ND	ND	ND	ND	ND
2492	-----	-----	-----	-----	-----	-----	-----
2494	0.0047	ND	ND	ND	ND	ND	ND
2497	0.00168	-----	-----	-----	-----	-----	-----
2508	-----	-----	-----	-----	-----	-----	-----
2532	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2563	n.d.	n.d	n.d	n.d	n.d	-----	n.d
2590	-----	-----	-----	-----	-----	-----	-----
2591	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2737	ND	ND	ND	ND	ND	ND	ND
2743	-----	-----	-----	-----	-----	-----	-----
2793	-----	-----	-----	-----	-----	-----	-----
2804	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
2830	ND	ND	ND	ND	ND	EI	_
2858	n.d	n.d	n.d	n.d	n.d	n.d	n.d
3116	-----	-----	-----	-----	-----	-----	-----
3146	-----	-----	-----	-----	-----	-----	-----
3150	-----	-----	-----	-----	-----	-----	-----
3154	-----	-----	-----	-----	-----	-----	-----
3172	-----	-----	-----	-----	-----	-----	-----
3176	0.0012	-----	-----	-----	-----	-----	-----
3197	-----	-----	-----	-----	-----	-----	-----
3210	-----	-----	-----	-----	-----	-----	-----
3222	-----	-----	-----	-----	-----	-----	-----
3237	-----	-----	-----	-----	-----	-----	-----
3243	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

Lab 2258: reported <50 %M/M

Other reported Phthalates in sample #19514; results in %M/M ---- continued ----

Lab	DMP	DNHP	DIBP	DPHP	DNPP	DUP	other
339	<0.001	<0.001	<0.001	-----	<0.001	-----	<0.005
348	<0.005	<0.005	<0.005	-----	<0.005	-----	-----
362	< 0.003	-----	< 0.003	-----	-----	-----	-----
551	-----	N.D.	N.D.	N.D.	N.D.	-----	-----
623	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.031
840	nd	nd	nd	nd	nd	nd	-----
841	ND	ND	ND	ND	ND	ND	ND
2115	-----	-----	-----	-----	-----	-----	-----
2121	-----	-----	-----	-----	-----	-----	-----
2170	-----	-----	-----	-----	-----	-----	-----
2247	ND (< 0.005)	ND (< 0.005)	ND (< 0.005)	ND (< 0.005)	ND (< 0.005)	ND (< 0.005)	ND (< 0.005)
2258	-----	<0.0050 C	<0.0050 C	-----	<0.0050 C	-----	-----
2265	< 0,025	< 0,025	< 0,025	< 0,025	< 0,025	< 0,025	-----
2266	-----	0	0	0	0	0	-----
2293	-----	-----	-----	-----	-----	-----	-----
2297	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2301	ND	ND	ND	ND	ND	ND	-----
2310	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	-----
2311	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	-----
2313	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2314	-----	-----	-----	-----	-----	-----	-----
2330	ND	ND	ND	ND	ND	ND	0.01
2347	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003	-----
2352	-----	-----	-----	-----	-----	-----	-----
2357	ND	ND	ND	ND	ND	ND	ND
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2363	ND	ND	ND	ND	ND	ND	ND
2365	<0.003	<0.003	<0.003	<0.005	<0.003	<0.003	-----
2369	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003	-----
2370	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2372	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.0489
2374	-----	-----	-----	-----	-----	-----	-----
2375	-----	-----	-----	-----	-----	-----	-----
2378	-----	-----	-----	-----	-----	-----	-----
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not tested
2380	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003	-----
2381	-----	-----	-----	-----	-----	-----	-----
2382	-----	-----	-----	-----	-----	-----	-----
2386	<0,003	<0,003	<0,003	<0,003	<0,003	<0,003	<0,003
2390	-----	-----	-----	-----	-----	-----	-----
2425	ND	ND	ND	ND	ND	ND	ND
2449	-----	-----	-----	-----	-----	-----	-----
2459	-----	-----	-----	-----	-----	-----	-----
2467	0.0000	0.0000	0.0000	-----	0.0000	0.0000	0.0000
2476	-----	-----	-----	-----	-----	-----	-----
2489	ND	ND	ND	ND	ND	ND	ND
2492	-----	-----	-----	-----	-----	-----	-----
2494	ND	ND	ND	ND	ND	ND	ND
2497	-----	-----	-----	-----	-----	-----	-----
2508	-----	-----	-----	-----	-----	-----	-----
2532	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2563	n.d	n.d	n.d	-----	n.d	-----	-----
2590	-----	-----	-----	-----	-----	-----	-----
2591	<0.005	<0.005	<0.005	-----	<0.005	-----	-----
2737	ND	ND	ND	ND	ND	-----	-----
2743	-----	-----	-----	-----	-----	-----	-----
2793	-----	-----	-----	-----	-----	-----	-----
2804	-----	N.D.	N.D.	N.D.	N.D.	-----	-----
2830	_	_	ND	_	ND	_	_
2858	n.d	n.d	n.d	n.d	n.d	n.d	n.d
3116	-----	-----	-----	-----	-----	-----	-----
3146	-----	-----	-----	-----	-----	-----	-----
3150	-----	-----	-----	-----	-----	-----	-----
3154	-----	-----	-----	-----	-----	-----	-----
3172	-----	-----	-----	-----	-----	-----	-----
3176	-----	-----	-----	-----	-----	-----	-----
3197	-----	-----	-----	-----	-----	-----	-----
3210	-----	-----	-----	-----	-----	-----	-----
3222	-----	-----	-----	-----	-----	-----	-----
3237	-----	-----	-----	-----	-----	-----	-----
3243	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

Lab 2258: reported <50 %M/M

Other reported Phthalates in sample #19515; results in %M/M

Lab	DEHP	DIDP	DINP	DNOP	DCHP	DEP	DMP
339	<0.001	<0.005	<0.005	<0.001	<0.001	<0.001	<0.001
348	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
362	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	-----	< 0.003
551	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	-----
623	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
840	nd	nd	nd	nd	nd	nd	nd
841	ND	ND	ND	ND	ND	ND	ND
2115	-----	-----	-----	-----	-----	-----	-----
2121	-----	-----	-----	-----	-----	-----	-----
2170	-----	-----	-----	-----	-----	-----	-----
2247	ND (< 0.005)	ND (< 0.005)	ND (< 0.005)	ND (< 0.005)	ND (< 0.005)	ND (< 0.005)	ND (< 0.005)
2258	<0.0050 C	<0.0050 C	<0.0050 C	<0.0050 C	<0.0050 C	-----	-----
2265	< 0,025	< 0,025	< 0,025	< 0,025	< 0,025	< 0,025	< 0,025
2266	0	0	0	0	0	-----	-----
2293	-----	-----	-----	-----	-----	-----	-----
2297	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2301	ND	ND	ND	ND	ND	ND	ND
2310	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2311	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2313	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2314	-----	-----	-----	-----	-----	-----	-----
2330	ND	ND	ND	ND	ND	ND	ND
2347	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003
2352	-----	-----	-----	-----	-----	-----	-----
2357	ND	ND	ND	ND	ND	ND	ND
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2363	ND	ND	ND	ND	ND	ND	ND
2365	<0.003	<0.005	<0.005	<0.003	<0.003	<0.003	<0.003
2369	<0.003	<0.010	<0.010	<0.003	<0.003	<0.003	<0.003
2370	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2372	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2374	-----	-----	-----	-----	-----	-----	-----
2375	-----	-----	-----	-----	-----	-----	-----
2378	-----	-----	-----	-----	-----	-----	-----
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2380	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003
2381	-----	-----	-----	-----	-----	-----	-----
2382	-----	-----	-----	-----	-----	-----	-----
2386	<0,003	<0,003	<0,003	<0,003	<0,003	<0,003	<0,003
2390	-----	-----	-----	-----	-----	-----	-----
2425	ND	ND	ND	ND	ND	ND	ND
2449	-----	-----	-----	-----	-----	-----	-----
2459	-----	-----	-----	-----	-----	-----	-----
2467	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
2476	-----	-----	-----	-----	-----	-----	-----
2489	ND	ND	ND	ND	ND	ND	ND
2492	-----	-----	-----	-----	-----	-----	-----
2494	ND	ND	ND	ND	ND	ND	ND
2497	-----	-----	-----	-----	-----	-----	-----
2508	-----	-----	-----	-----	-----	-----	-----
2532	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2563	n.d	n.d	n.d	n.d	-----	n.d	n.d
2590	-----	-----	-----	-----	-----	-----	-----
2591	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2737	ND	ND	ND	ND	ND	ND	ND
2743	-----	-----	-----	-----	-----	-----	-----
2793	-----	-----	-----	-----	-----	-----	-----
2804	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	-----
2830	ND	ND	ND	ND	EI	_	_
2858	n.d	n.d	n.d	n.d	n.d	n.d	n.d
3116	-----	-----	-----	-----	-----	-----	-----
3146	-----	-----	-----	-----	-----	-----	-----
3150	-----	-----	-----	-----	-----	-----	-----
3154	-----	-----	-----	-----	-----	-----	-----
3172	-----	-----	-----	-----	-----	-----	-----
3176	-----	-----	-----	-----	-----	-----	-----
3197	-----	-----	-----	-----	-----	-----	-----
3210	-----	-----	-----	-----	-----	-----	-----
3222	-----	-----	-----	-----	-----	-----	-----
3237	-----	-----	-----	-----	-----	-----	-----
3243	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

Lab 2258: reported <50 %M/M

Other reported Phthalates in sample #19515; results in %M/M ---- continued ----

Lab	DNHP	DIBP	DPHP	DNPP	DUP	other
339	<0.001	<0.001	-----	<0.001	-----	<0.001
348	<0.005	<0.005	-----	<0.005	-----	-----
362	-----	< 0.003	-----	-----	-----	-----
551	N.D.	N.D.	N.D.	N.D.	-----	-----
623	n.d.	n.d.	n.d.	n.d.	n.d.	0.113
840	nd	nd	nd	nd	nd	-----
841	ND	ND	ND	ND	ND	ND
2115	-----	-----	-----	-----	-----	-----
2121	-----	-----	-----	-----	-----	-----
2170	-----	-----	-----	-----	-----	-----
2247	ND (< 0.005)	ND (< 0.005)	ND (< 0.005)	ND (< 0.005)	ND (< 0.005)	ND (< 0.005)
2258	<0.0050 C	<0.0050 C	-----	<0.0050 C	-----	-----
2265	< 0,025	< 0,025	< 0,025	< 0,025	< 0,025	-----
2266	0	0	0	0	0	-----
2293	-----	-----	-----	-----	-----	-----
2297	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2301	ND	ND	ND	ND	ND	-----
2310	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	-----
2311	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	-----
2313	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2314	-----	-----	-----	-----	-----	-----
2330	ND	ND	ND	ND	ND	0.10
2347	<0.003	<0.003	<0.003	<0.003	<0.003	-----
2352	-----	-----	-----	-----	-----	-----
2357	ND	ND	ND	ND	ND	ND
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2363	ND	ND	ND	ND	ND	ND
2365	<0.003	<0.003	<0.005	<0.003	<0.003	-----
2369	<0.003	<0.003	<0.003	<0.003	<0.003	-----
2370	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2372	n.d.	n.d.	n.d.	n.d.	n.d.	0.11017
2374	-----	-----	-----	-----	-----	-----
2375	-----	-----	-----	-----	-----	-----
2378	-----	-----	-----	-----	-----	-----
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not tested
2380	<0.003	<0.003	<0.003	<0.003	<0.003	-----
2381	-----	-----	-----	-----	-----	-----
2382	-----	-----	-----	-----	-----	-----
2386	<0,003	<0,003	<0,003	<0,003	<0,003	<0,003
2390	-----	-----	-----	-----	-----	-----
2425	ND	ND	ND	ND	ND	ND
2449	-----	-----	-----	-----	-----	-----
2459	-----	-----	-----	-----	-----	-----
2467	0.0000	0.0000	-----	0.0000	0.0000	0.0000
2476	-----	-----	-----	-----	-----	-----
2489	ND	ND	ND	ND	ND	ND
2492	-----	-----	-----	-----	-----	-----
2494	ND	ND	ND	ND	ND	ND
2497	-----	-----	-----	-----	-----	-----
2508	-----	-----	-----	-----	-----	-----
2532	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2563	n.d	n.d	-----	n.d	-----	-----
2590	-----	-----	-----	-----	-----	-----
2591	<0.005	<0.005	-----	<0.005	-----	-----
2737	ND	ND	ND	ND	-----	-----
2743	-----	-----	-----	-----	-----	-----
2793	-----	-----	-----	-----	-----	-----
2804	N.D.	N.D.	N.D.	N.D.	-----	-----
2830	_	ND	_	ND	_	_
2858	n.d	n.d	n.d	n.d	n.d	n.d
3116	-----	-----	-----	-----	-----	-----
3146	-----	-----	-----	-----	-----	-----
3150	-----	-----	-----	-----	-----	-----
3154	-----	-----	-----	-----	-----	-----
3172	-----	-----	-----	-----	-----	-----
3176	-----	-----	-----	-----	-----	-----
3197	-----	-----	-----	-----	-----	-----
3210	-----	-----	-----	-----	-----	-----
3222	-----	-----	-----	-----	-----	-----
3237	-----	-----	-----	-----	-----	-----
3243	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

Lab 2258: reported <50 %M/M

APPENDIX 3

Analytical details

Lab	ISO/IEC 17025 accr.	Sample intake used (grams)	Sample preparation	Final estimated particle size	Release/ extraction technique	Extraction solvent	Extraction time (min)	Extrac. temp. (°C)
339	No	0,5	Used as received	5 x 5 mm	Ultrasonic	THF, Methanol (for precipitation)	60	60
348	No	0.5 g	Further Cut	2*2 mm	Ultrasonic	Tetrahydrofuran	180 min	60±5ºC
362	Yes	0.5	Used as received		Ultrasonic	THF	60	70
551	No	0.5g	Used as received		Ultrasonic	Tetrahydrofuran/ Hexane	30	40
623	Yes	0.1	Further Cut	5 mm x 5 mm	Ultrasonic	THF	60	60
840	Yes	0.1 g	---		Mechanical Shaking	THF, n-Hexane	2.5 hours	
841	Yes	about 0.1g	Further Cut	2x2 mm	Ultrasonic	THF, n-Hexane	60	60
2115	---		---		---			
2121	Yes	0.5 g and 5 replicates by sample	Further Cut	ND	Ultrasonic	Hexane/THF	60	60
2170	---		---		---			
2247	Yes	0.5gm	Used as received	<5mm	Ultrasonic	Tetrahydrofuran for extraction n-Hexane for percipitation	30min	25.0
2258	Yes	19514: 0.0640 19515: 0.0623	Used as received	2mm	Ultrasonic	Tetrahydrofurane	60 minutes	40´C
2265	Yes	0,3 g	Further Cut		Ultrasonic	THF first, precipitation with n-hexane	60 min	60 °C
2266	Yes	0.3	Further Cut	0.5 MM	Ultrasonic	THF/Acetone	60 min	60°C
2293	Yes	19514 0.0510 g 19515 0.0501 g	Further Cut	3 x 5 mm	Ultrasonic	Tetrahydrofurane and n-hexane	120 minutes	room temp
2297	Yes	0.5	Used as received		Ultrasonic	THF+HEXANE	60	60
2301	Yes	0.3	Further Cut	2mmx2mm	Ultrasonic	THF	60	60
2310	Yes	0.3g	Further Cut	<2MM	Ultrasonic	THF/Hexane	60	60
2311	Yes	0.3	Further Cut	<2mm	Ultrasonic	THF and n-Hexane	60	60
2313	Yes	0.5 gram	Further Cut	3mm X 3mm	Ultrasonic	THF AND n-HEXANE	1 HOUR	60
2314	Yes	0.3G	Further Cut	2mm X 2mm	Ultrasonic	THF+HEXANE	60	60
2330	No	0.5 grams	Further Cut	5 mm x 5 mm	Ultrasonic	THF and Hexane the ratio 1:2	60 minutes	60 °C
2347	Yes	0.3g	Further Cut	2mm*2mm	Ultrasonic	THF	60min	50
2352	Yes	0.3g	Used as received	£¼5mm¡Á5mm	Ultrasonic	Tetrahydrofuran/ n-Hexane	60minutes	60
2357	Yes	0.3	Further Cut	5mm*5mm*5mm	Ultrasonic	10ml THF and 20ml n-Hexane	60	60
2358	Yes	0.3 grams	Used as received	3mm X 3mm	Ultrasonic	THF, n-hexane	60 minutes	60 °C
2363	Yes	0.3g	Used as received	5mm*5mm	Ultrasonic	THF	60min	60
2365	Yes	0.3g	Further Cut	2mm*2mm*2mm	Ultrasonic	THF/n-Hexane (v/v)=1:2	60min	60
2369	Yes	0.5g	Used as received		Ultrasonic	THF	60min	60
2370	Yes	0.3 g	Used as received	Less than 5 mm	Ultrasonic	THF:Acetonitrile = 10 : 20 mL	60 min	60
2372	No	0.5	Further Cut	2mm	Ultrasonic	THF	60	60
2374	Yes	0.1g	Further Cut	1-2mm	Ultrasonic	THF,Hexane	60min	60
2375	Yes	0.1 g	Further Cut	3mm x 3mm	Ultrasonic	THF / Hexane	60 min	60°C
2378	Yes	0.3g	Used as received	5mm*5mm	Ultrasonic	THF,n-Hexane	60min	60
2379	Yes	0.5 g	Further Cut	5 x 5 mm.	Ultrasonic	Tetrahydrofuran and Hexane	30 min	40
2380	Yes	0.3 gm	Further Cut	5 mm x 5 mm	Ultrasonic	Tetrahydrofuran (THF)	60	60
2381	Yes	.01 gm per trial	Further Cut	`	Ultrasonic	THF & n-Hexan	60	60
2382	Yes	0.5g	Further Cut	2*2mm	Ultrasonic	Tetrahydrofuran	60min	60
2386	Yes	0,5	Used as received	5x5mm	Ultrasonic	THF/n-Hexan	60	60
2390	Yes	0.5 gram	Further Cut	5x5mm	Ultrasonic	THF+n-hexane	60 min	60°C
2425	Yes	0.30 gm	Further Cut	2 mm X 3 mm	Ultrasonic	Tetrahydrofuran +n-hexane	60 ± 5 Minutes	60±5°C
2449	---		---		---			
2459	Yes	0.5	Further Cut	3x3mm	Ultrasonic	THF: n-Hexane	60	60
2467	Yes	0.5	Used as received	as received	Ultrasonic	THF	60	60
2476	---		---		---			

Lab	ISO/IEC 17025 accr.	Sample intake used (grams)	Sample preparation	Final estimated particle size	Release/ extraction technique	Extraction solvent	Extraction time (min)	Extrac. temp. (°C)
2489	Yes	0.5149g/ 0.5379	Further Cut	2x2 mm	Ultrasonic	THF/n-Hexane	60 minutes	60
2492	Yes	0.5 gram for each trial	Used as received	0.5cm	Ultrasonic	THF/n-Hexane	60	60
2494	Yes	0.50 gram	Used as received	5mmx5mm	Ultrasonic	THF:n-hexane	60	60
2497	Yes	2	Used as received		Ultrasonic	THF / Hexane	60	60
2508	Yes	0.4	Used as received		Ultrasonic	THF	60	60
2532	Yes	0.3 grams	Further Cut	< 2 mm	Ultrasonic	THF and n-Hexane	60 Minutes	60 °C
2563	Yes	0,5	Used as received		Ultrasonic	THF	60 Min	RT
2590	Yes	0.3g	Used as received		Ultrasonic	THF/hexane	1 h	60 °C
2591	Yes	0.2 grams	Further Cut		Ultrasonic	Tetrahydrofuran e/n-hexane	2 hours	Room temp
2737	Yes	0.3g	Further Cut	3mm*3mm	Ultrasonic	Tetrahydrofuran and n-hexane	60min	60
2743	Yes	0.5g	Used as received		Ultrasonic	THF. Hexane added after extraction. Then homogeneized.	60 min	60°C
2793	No	0.3	Used as received		Ultrasonic	THF/ ACETONITRILE	60	60
2804	No	0.3	Further Cut	3mm x 3mm	Ultrasonic	THF/ACN	60	60
2830	---		---		---			
2858	Yes	0.3	Used as received	n/a	Ultrasonic	n-hexane and Tetrahydrofuran	60 minutes	60
3116	Yes	0.6 grams	Used as received	5mm x 5mm	Ultrasonic	Tetrahydrofuran, Acetonitrile	60 min	60°C
3146	---	0,5	Used as received	1 * 1 mm	---	THF/Acetonitril	60	70
3150	Yes	0,1 - 0,3	Used as received	5mm * 5mm	Ultrasonic	THF	60	60
3154	---		---		---			
3172	Yes	0.15	Further Cut		Ultrasonic	THF/ACN	60	25
3176	Yes	0,3 grams	Used as received		Ultrasonic	THF/ACN	60 min	60C
3197	Yes	0,3 g	Further Cut	<5 mm	Ultrasonic	THF/n-hexane	60 minutes	60C
3210	Yes	1g	Further Cut	2-3 mm	Ultrasonic	Hexane/Acetone (80/20)	60 min	50°C
3222	No	0.5 g	Used as received		Ultrasonic	THF	60 min	60°C
3237	Yes	0,1 g	Used as received	Used as received	Ultrasonic	ACN,THF	30 min	40
3243	Yes	0.5	Further Cut		Ultrasonic	Dichlormethane	30 min	room temp

APPENDIX 4

Number of participating laboratories per country

4 labs in BANGLADESH
1 lab in BRAZIL
1 lab in BULGARIA
1 lab in CAMBODIA
1 lab in EGYPT
4 labs in FRANCE
8 labs in GERMANY
2 labs in GUATEMALA
4 labs in HONG KONG
7 labs in INDIA
3 labs in INDONESIA
6 labs in ITALY
1 lab in MOROCCO
11 labs in P.R. of CHINA
3 labs in PAKISTAN
1 lab in ROMANIA
1 lab in SINGAPORE
2 labs in SPAIN
2 labs in TAIWAN R.O.C.
1 lab in THAILAND
1 lab in TUNISIA
4 labs in TURKEY
2 labs in VIETNAM

APPENDIX 5

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
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
fr.	= first reported

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