Results of Proficiency Test
Phthalates in Textile
March 2021

Organized 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.

Since 2019 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Phthalates in Textile. During the annual proficiency testing program 2020/2021 it was decided to continue the proficiency test for the analysis of Phthalates in Textile.

In this interlaboratory study 69 laboratories in 23 different countries registered for participation. See appendix 4 for the number of participants per country. In this report the results of the proficiency test on Phthalates in Textile 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 textile samples both positive on Phthalates of approximately 3 grams each, one sample of yellow cotton pieces labelled #21535 and one sample of pink cotton pieces labelled #21536. 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

A batch of yellow cotton was selected which was made positive on Dibutyl phthalate (DBP). The batch was cut into small pieces. After homogenization 85 subsamples of approximately 3 grams each were prepared and labelled #21535.
This batch has been used before in the PT on Phthalates in Textile iis19A03. The batch was tested and found to be homogeneous in this PT.

A batch of pink cotton was selected which was made positive on Benzyl butyl phthalate (BBP) and Dicyclohexyl phthalate (DCHP). The batch was cut into small pieces. After homogenization 90 subsamples of approximately 3 grams each were prepared and labelled #21536.
The homogeneity of the subsamples was checked by determination of BBP and DCHP using an in-house test method on 8 stratified randomly selected subsamples.

	BBP in %M/M	DCHP in %M/M
sample #21536-1	0.141938	0.081103
sample #21536-2	0.144117	0.082788
sample #21536-3	0.124857	0.071861
sample #21536-4	0.130011	0.075943
sample #21536-5	0.137008	0.080602
sample #21536-6	0.141815	0.083005
sample #21536-7	0.132425	0.076195
sample #21536-8	0.127159	0.073655

Table 1: homogeneity test results of subsamples #21536

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), in agreement with the procedure of ISO13528, Annex B2 in the next table.

	BBP	DCHP
RSD% (observed)	5%	5%
reference method	iis memo 1701	iis memo 1701
0.3 x RSD% (reference method)	5%	5%

Table 2: evaluation of the relative standard deviation (RSD) of subsamples #21536

The observed RSD% is in agreement with 0.3 times the estimated RSD% of the reference method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample labelled #21535 and one sample labelled #21536 were sent on February 10, 2021.

2.5 ANALYZES

The participants were requested to determine on samples #21535 and #21536 sixteen individual Phthalates and the total of other Phthalates:

BBP - Benzyl butyl phthalate CAS No. 85-68-7
DEHP - Di-(2-ethylhexyl) phthalate CAS No. 117-81-7
DBP - Dibutyl phthalate CAS No. 84-74-2
DIDP - Di-iso-decyl phthalate CAS No. 26761-40-0 & 68515-49-1
DINP - Di-iso-nonyl phthalate CAS No. 28553-12-0 & 68515-48-0
DNOP - Di-n-octyl phthalate CAS No. 117-84-0
DCHP - Dicyclohexyl phthalate CAS No. 84-61-7
DEP - Diethyl phthalate CAS No. 84-66-2
DMP - Dimethyl phthalate CAS No. 131-11-3
DNHP - Di-n-hexyl phthalate CAS No. 84-75-3
DIBP - Di-iso-butyl phthalate CAS No. 84-69-5
DPHP - Di(2-propylheptyl) phthalate CAS No. 53306-54-0
DNPP - Di-n-pentyl phthalate CAS No. 131-18-0
DUP - Diundecyl phthalate CAS No. 3648-20-2
DPrP - Di-n-propyl phthalate CAS No. 131-16-8
DMEP - Di-(2-methoxyethyl) phthalate CAS No. 117-82-8

It was also requested not to use less than 0.5 gram per determination to ensure the homogeneity.

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 test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the appropriate reference test 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 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.

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, 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:

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

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 interlaboratory study some problems were encountered with the dispatch of the samples. Seven participants reported after the final reporting date and one other participant did not report any test results. In total 68 laboratories reported 200 numerical test results. Observed were 4 outlying test results, which is 2.0%. 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. The 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 5.

Two test methods ISO14389 and CPSC-CH-C1001-09.4 are used for determining Phthalates in Textile. Regretfully, CPSC-CH-C1001-09.4 method does not contain any precision statements and 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 for polymers to estimate a more realistic target reproducibility in polymers (see iis memo 1701, lit 14). The target reproducibility was estimated as the RSD (relative standard deviation) of 16% of the mean multiplied by 2.8. Since test method ISO14389:14 cannot be used because of the 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 #21535

DBP: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full agreement with the estimated reproducibility based on iis memo 1701.

The majority of the participants agreed on a concentration near or below the limit of detection for all other Phthalates mentioned in paragraph 2.5. Therefore, no z-scores were calculated for these Phthalates. The test results of these components are given in appendix 2.

Sample #21536

BBP: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated reproducibility based on iis memo 1701.

DCHP: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in full agreement with the estimated reproducibility based on iis memo 1701.

The majority of the participants agreed on a concentration near or below the limit of detection for all other Phthalates mentioned in paragraph 2.5. Therefore, no z-scores were calculated for these Phthalates. The test results of 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, 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	66	0.029	0.014	0.013

Table 3: reproducibility on sample #21535

Component	unit	n	average	2.8 * sd	R(target)
BBP	%M/M	66	0.110	0.042	0.049
DCHP	%M/M	64	0.067	0.030	0.030

Table 4: reproducibility on sample #21536

Without further calculations, it could be concluded that for Dibutyl phthalate (DBP), Benzyl butyl phthalate (BBP) and Dicyclohexyl phthalate (DCHP) there is a good compliance of the group of participating laboratories with the target.

4.3 COMPARISON OF PROFICIENCY TEST OF MARCH 2021 WITH THE PREVIOUS PTS

	March 2021	March 2020	March 2019
Number of reporting laboratories	68	69	69
Number of test results	200	134	188
Number of statistical outliers	4	12	8
Percentage of statistical outliers	2.0%	9.0%	4.3%

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 tests was compared, expressed as relative standard deviation (RSD) of the PTs, see next table.

Component	March 2021	March 2020	March 2019	iis memo 1701
BBP	14%	n.e.	12%	16%
DBP	17%	n.e.	29% - 31%	16%
DCHP	16%	n.e.	n.e.	16%
DNHP	n.e.	12%	n.e.	16%
DMEP	n.e.	17%	n.e.	16%

Table 6: development of uncertainties over the years

The uncertainties of BBP, DBP and DCHP observed in this PT are in line with the requirements mentioned in the target. The observed incertainty of DBP have been strongly improved in this PT.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

The test method ISO14389 is used by about 60% of the reporting participants and the test method CPSC-CH-C1001-09.4 is used by about 30% of the reporting participants. The majority of the other participants used an in-house test method.

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 97% of the participants mentioned that they are accredited for the determination of Phthalates in Textile.
- About 45% of the participants used the samples as received and about 55% further cut the samples prior to analysis.
- About 63% of the participants used less than 0.5 grams as sample intake, about 32% used 0.5 grams and about 5% used more than 0.5 grams as sample intake. It is remarkable that the majority had used a lower sample intake as it was instructed in the letter of instructions.
- Almost all of the participants used ultrasonic as technique to release/extract the Phthalates.
- Almost all of the participants used THF or a THF mixture as extraction solvent.

- The extraction time differs from 30 minutes to 3 hours. About 80% of the reporting participants used an extraction time of 60 minutes.
- The extraction temperature differs from room temperature to 60°C. About 80% of the reporting participants used an extraction temperature of 60°C.

To extract the requested components mentioned in paragraph 2.5 from a textile, the extraction solvent, the extraction conditions and the contact surface area could be important variables. The influence of different test conditions can not be determined in this PT, because the majority of the participants tested the sample in the same conditions, namely on a sample of 0.5 grams or less with a THF or THF mixture extraction by ultrasonic at 60° for 60 minutes.

5 DISCUSSION

All reporting participants were able to detect DBP in sample #21535 and BBP and DCHP in sample #21536.
When the results of this interlaboratory study were compared to the Ecolabelling Standards and Requirements for Textiles in EU, such as OEKO-TEX® 100 and the similar Bluesign® BSSL, it was noticed that not all participants would make identical decisions about the acceptability of the textiles for the determined components. All reporting laboratories would have approved sample #21535 for all categories, except one laboratory. All reporting laboratories would have rejected sample #21536 for all categories.

Ecolabel	baby clothes	in direct skin contact	no direct skin contact
Bluesign® BSSL	<0.05 mg/kg	<0.05 mg/kg	<0.05 mg/kg
Oeko-Tex 100	<0.05 mg/kg	<0.05 mg/kg	<0.05 mg/kg

Table 7: Bluesign® BSSL and Ecolabelling Standards and Requirements for Textiles in EU

Sample #21535 was used earlier as sample #19514 in iis19A03 (2019). This was the first PT iis organized for Phthalates in Textile. In table 8 a comparison is given over the two proficiency tests.

	Sample #21535				Sample #19514			
	unit	n	average	R(calc)	unit	n	average	R(calc)
DBP	%M/M	66	0.029	0.014	%M/M	68	0.043	0.038

Table 8: comparison of sample #21535 with #19514

It is observed that the average level of DBP in the 2021 PT is lower and the observed reproducibility R(calc) for DBP has improved significantly in 2021. The variation in the test results of the participants in 2021 is much smaller than in 2019. PTs that are organized the first time often show a larger variation than PTs that have been held multiple times. 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. The average and smaller variation of the 2021 PT are within the average and variation of the 2019 PT and therefore comparable.

6 CONCLUSION

Although, it can be concluded that the majority of the participants has no problem with the determination of the Phthalates in the textile samples of this PT, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary.

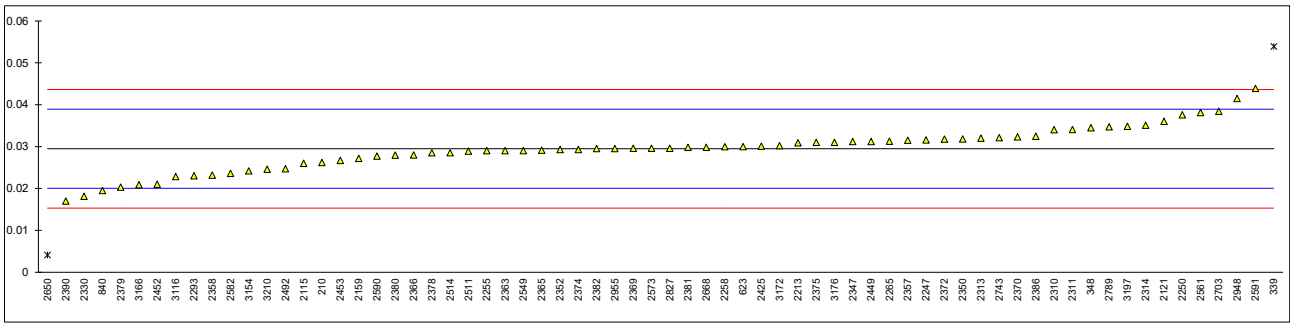
Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

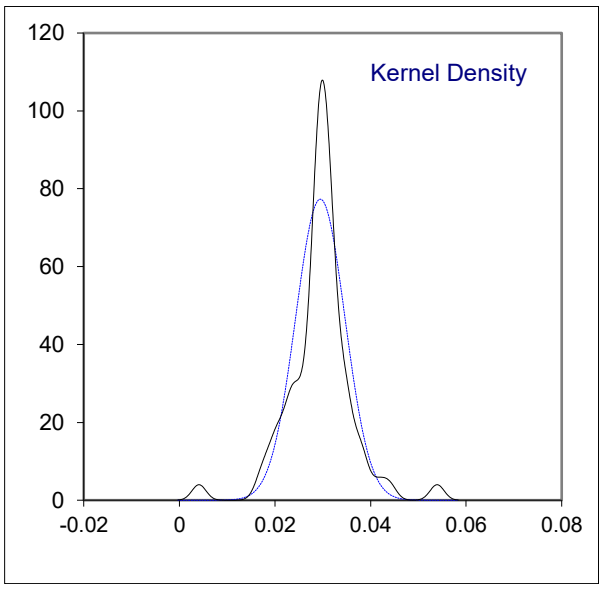
APPENDIX 1

Determination of DBP - Dibutyl phthalate on sample #21535; results in %M/M

lab	method	value	mark	z(targ)	remarks
210	CPSC-CH-C1001-09.4	0.026176		-0.70	
339	In house	0.0539	R(0.01)	5.18	
348	CPSC-CH-C1001-09.4	0.0345		1.06	
623	ISO14389	0.0300		0.11	
840	ISO14389	0.0195		-2.12	
2115	ISO14389	0.026		-0.74	
2121	ISO14389	0.036		1.38	
2159	ISO14389	0.0272		-0.48	
2213	CPSC-CH-C1001-09.4	0.0309	C	0.30	first reported: 309.95 %M/M
2247	ISO14389	0.0316		0.45	
2250	ISO14389	0.03757		1.72	
2255	CPSC-CH-C1001-09.4	0.029		-0.10	
2258	CPSC-CH-C1001-09.4	0.029951		0.10	
2265	ISO14389	0.03125		0.38	
2293	ISO14389	0.02303		-1.37	
2310	CPSC-CH-C1001-09.4	0.034		0.96	
2311	ISO14389	0.03406		0.97	
2313	ISO14389	0.0320		0.53	
2314	ISO14389	0.0351		1.19	
2330	ISO14389	0.01812	C	-2.41	first reported: 0.01573
2347	ISO14389	0.0312		0.36	
2350	CPSC-CH-C1001-09.4	0.0318		0.49	
2352	ISO14389	0.0293		-0.04	
2357	ISO14389	0.0315		0.43	
2358	ISO14389	0.023162		-1.34	
2363	ISO14389	0.029		-0.10	
2365	CPSC-CH-C1001-09.4	0.02913		-0.07	
2366	ISO14389	0.028		-0.31	
2369	CPSC-CH-C1001-09.4	0.0296		0.03	
2370	ISO14389	0.0323		0.60	
2372	CPSC-CH-C1001-09.4	0.03176		0.48	
2374	ISO14389	0.0293		-0.04	
2375	CPSC-CH-C1001-09.4	0.031		0.32	
2378	ISO14389	0.0285		-0.21	
2379	ISO14389	0.0203		-1.95	
2380	CPSC-CH-C1001-09.4	0.02797		-0.32	
2381	CPSC-CH-C1001-09.4	0.0298		0.07	
2382	CPSC-CH-C1001-09.4	0.02950		0.00	
2386	ISO14389	0.03244		0.63	
2390	CPSC-CH-C1001-09.4	0.017	C	-2.65	first reported: 0.0124
2425	In house	0.0301		0.13	
2449		0.0312		0.36	
2452	ISO14389	0.021		-1.80	
2453	CPSC-CH-C1001-09.4	0.0267		-0.59	
2492	In house	0.0247		-1.01	
2511	ISO14389	0.0289		-0.12	
2514	ISO14389	0.0285	C	-0.21	first reported: 284.95 %M/M
2549	ISO14389	0.029		-0.10	
2561	CPSC-CH-C1001-09.4	0.0381		1.83	
2573	ISO14389	0.0296		0.03	
2582	CPSC-CH-C1001-09.4	0.02360		-1.25	
2590	ISO14389	0.02770		-0.38	
2591	CPSC-CH-C1001-09.4	0.04388		3.05	
2650	ISO14389	0.004122	C,R(0.01)	-5.38	first reported: 0.00399
2668	ISO14389	0.0298		0.07	
2671		-----		-----	
2703	In house	0.03841		1.89	
2743	ISO14389	0.032112		0.56	
2789	ISO14389	0.0347		1.11	
2827	ISO14389	0.0296		0.03	
2948	ISO14389	0.0415		2.55	
2955	CPSC-CH-C1001-09.4	0.0295		0.00	
3116	ISO14389	0.02283		-1.41	
3154	ISO16181-1(draft)	0.0242		-1.12	
3166	In house	0.0209		-1.82	
3172	ISO8124-6	0.0302		0.15	
3176	ISO14389	0.031		0.32	
3197	ISO14389	0.0348		1.13	
3210	In house	0.0246		-1.03	

normality	OK	
n	66	
outliers	2	
mean (n)	0.0295	
st.dev. (n)	0.00516	RSD = 17%
R(calc.)	0.0144	
st.dev.(iis memo 1701)	0.00472	
R(iis memo 1701)	0.0132	

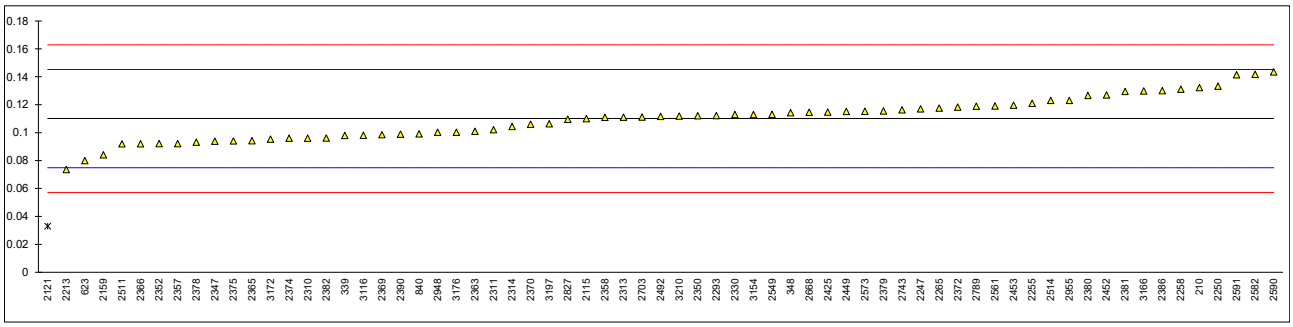


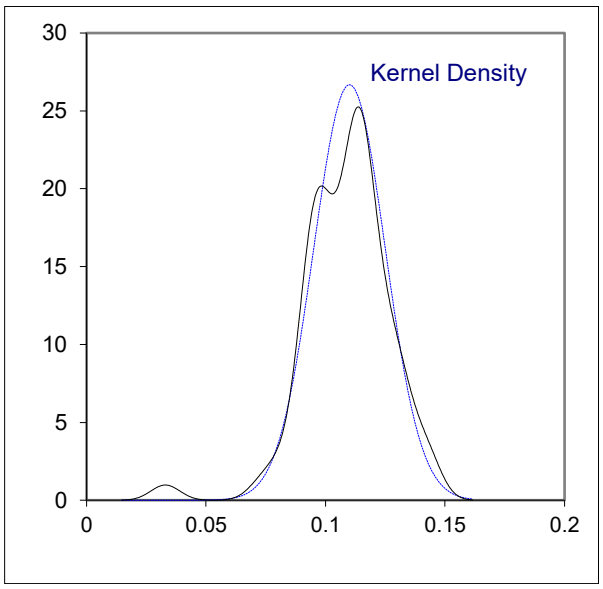


Determination of BBP - Benzyl butyl phthalate on sample #21536; results in %M/M

lab	method	value	mark	z(targ)	remarks
210	CPSC-CH-C1001-09.4	0.132194		1.26	
339	In house	0.0979		-0.69	
348	CPSC-CH-C1001-09.4	0.1142		0.23	
623	ISO14389	0.080	C	-1.71	first reported: 0.2170
840	ISO14389	0.0991		-0.62	
2115	ISO14389	0.11		0.00	
2121	ISO14389	0.033	R(0.01)	-4.38	
2159	ISO14389	0.0841		-1.47	
2213	CPSC-CH-C1001-09.4	0.0735	C	-2.08	first reported: 735 %M/M
2247	ISO14389	0.1169		0.39	
2250	ISO14389	0.1333		1.32	
2255	CPSC-CH-C1001-09.4	0.121		0.62	
2258	CPSC-CH-C1001-09.4	0.131096		1.19	
2265	ISO14389	0.11748		0.42	
2293	ISO14389	0.1122		0.12	
2310	CPSC-CH-C1001-09.4	0.096		-0.80	
2311	ISO14389	0.1020		-0.46	
2313	ISO14389	0.1110		0.05	
2314	ISO14389	0.1043		-0.33	
2330	ISO14389	0.11287		0.16	
2347	ISO14389	0.0938		-0.92	
2350	CPSC-CH-C1001-09.4	0.1120		0.11	
2352	ISO14389	0.0921		-1.02	
2357	ISO14389	0.0921		-1.02	
2358	ISO14389	0.1108645		0.05	
2363	ISO14389	0.101		-0.51	
2365	CPSC-CH-C1001-09.4	0.09416		-0.90	
2366	CPSC-CH-C1001-09.4	0.092		-1.03	
2369	CPSC-CH-C1001-09.4	0.0985		-0.66	
2370	ISO14389	0.106		-0.23	
2372	CPSC-CH-C1001-09.4	0.1182		0.46	
2374	ISO14389	0.0959		-0.80	
2375	CPSC-CH-C1001-09.4	0.094		-0.91	
2378	ISO14389	0.0931		-0.96	
2379	ISO14389	0.1154		0.30	
2380	CPSC-CH-C1001-09.4	0.12665		0.94	
2381	CPSC-CH-C1001-09.4	0.1295		1.10	
2382	CPSC-CH-C1001-09.4	0.0961		-0.79	
2386	ISO14389	0.13001		1.13	
2390	CPSC-CH-C1001-09.4	0.0989		-0.63	
2425	In house	0.1147		0.26	
2449		0.1151	C	0.29	first reported as DBP
2452		0.127		0.96	
2453	CPSC-CH-C1001-09.4	0.1196		0.54	
2492	In house	0.1116		0.09	
2511	ISO14389	0.0919		-1.03	
2514	ISO14389	0.1230	C	0.73	first reported: 1230.25 %M/M
2549	ISO14389	0.1130		0.17	
2561	CPSC-CH-C1001-09.4	0.119		0.51	
2573	ISO14389	0.1152		0.29	
2582	CPSC-CH-C1001-09.4	0.14175		1.80	
2590	ISO14389	0.14339		1.89	
2591	CPSC-CH-C1001-09.4	0.14135		1.78	
2650		-----		-----	
2668	ISO14389	0.1146		0.26	
2671		-----		-----	
2703	In house	0.11109		0.06	
2743	ISO14389	0.116317		0.36	
2789	ISO14389	0.1189		0.50	
2827	ISO14389	0.1096		-0.03	
2948	ISO14389	0.1002		-0.56	
2955	CPSC-CH-C1001-09.4	0.1230		0.73	
3116	ISO14389	0.09804		-0.68	
3154	ISO16181-1(draft)	0.11295		0.16	
3166	In house	0.1298		1.12	
3172	ISO8124-6	0.0953		-0.84	
3176	ISO14389	0.1002		-0.56	
3197	ISO14389	0.1063		-0.21	
3210	In house	0.1118		0.10	

normality	OK	
n	66	
outliers	1	
mean (n)	0.1101	
st.dev. (n)	0.01496	RSD = 14%
R(calc.)	0.0419	
st.dev.(iis memo 1701)	0.01761	
R(iis memo 1701)	0.0493	

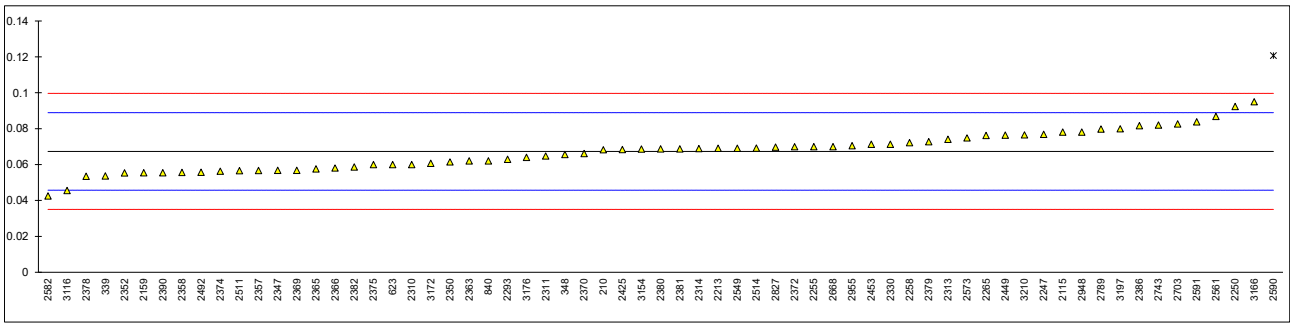


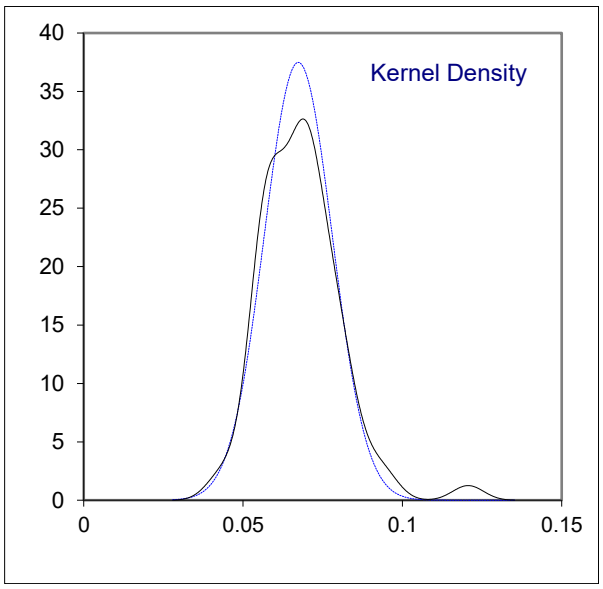


Determination of DCHP - Dicyclohexyl phthalate on sample #21536; results in %M/M

lab	method	value	mark	z(targ)	remarks
210	CPSC-CH-C1001-09.4	0.068186		0.08	
339	In house	0.0536		-1.27	
348	CPSC-CH-C1001-09.4	0.0656		-0.16	
623	ISO14389	0.060	C	-0.68	first reported: 0.1290
840	ISO14389	0.062		-0.49	
2115	ISO14389	0.078		0.99	
2121		-----		-----	
2159	ISO14389	0.0554		-1.11	
2213	CPSC-CH-C1001-09.4	0.069	C	0.15	first reported: 690 %M/M
2247	ISO14389	0.0768		0.88	
2250	ISO14389	0.0923		2.32	
2255	CPSC-CH-C1001-09.4	0.07		0.25	
2258	CPSC-CH-C1001-09.4	0.072176		0.45	
2265	ISO14389	0.07614		0.82	
2293	ISO14389	0.0629		-0.41	
2310	CPSC-CH-C1001-09.4	0.06		-0.68	
2311	ISO14389	0.0647		-0.24	
2313	ISO14389	0.0740		0.62	
2314	ISO14389	0.0689		0.15	
2330	ISO14389	0.07123		0.36	
2347	ISO14389	0.0568		-0.98	
2350	CPSC-CH-C1001-09.4	0.0614		-0.55	
2352	ISO14389	0.0553		-1.12	
2357	ISO14389	0.0567		-0.99	
2358	ISO14389	0.0556012		-1.09	
2363	ISO14389	0.062		-0.49	
2365	CPSC-CH-C1001-09.4	0.05753		-0.91	
2366	CPSC-CH-C1001-09.4	0.058		-0.87	
2369	CPSC-CH-C1001-09.4	0.0568		-0.98	
2370	ISO14389	0.0661		-0.11	
2372	CPSC-CH-C1001-09.4	0.06988		0.24	
2374	ISO14389	0.0563		-1.02	
2375	CPSC-CH-C1001-09.4	0.060		-0.68	
2378	ISO14389	0.0535		-1.28	
2379	ISO14389	0.0727		0.50	
2380	CPSC-CH-C1001-09.4	0.06867		0.12	
2381	CPSC-CH-C1001-09.4	0.0687		0.13	
2382	CPSC-CH-C1001-09.4	0.0586		-0.81	
2386	ISO14389	0.08164		1.33	
2390	CPSC-CH-C1001-09.4	0.0554		-1.11	
2425	In house	0.0684		0.10	
2449		0.0763		0.83	
2452		-----		-----	
2453	CPSC-CH-C1001-09.4	0.0712		0.36	
2492	In house	0.0557		-1.08	
2511	ISO14389	0.0566		-1.00	
2514	ISO14389	0.0691	C	0.16	first reported: 690.76 %M/M
2549	ISO14389	0.0690		0.15	
2561	CPSC-CH-C1001-09.4	0.0868		1.81	
2573	ISO14389	0.0748		0.69	
2582	CPSC-CH-C1001-09.4	0.04250		-2.30	
2590	ISO14389	0.12064	R(0.01)	4.95	
2591	CPSC-CH-C1001-09.4	0.08374		1.52	
2650		-----		-----	
2668	ISO14389	0.070		0.25	
2671		-----		-----	
2703	In house	0.08255		1.41	
2743	ISO14389	0.082012		1.36	
2789	ISO14389	0.0797		1.15	
2827	ISO14389	0.0696		0.21	
2948	ISO14389	0.0780		0.99	
2955	CPSC-CH-C1001-09.4	0.0705		0.29	
3116	ISO14389	0.04552		-2.02	
3154	ISO16181-1(draft)	0.0686		0.12	
3166	In house	0.0949		2.56	
3172	ISO8124-6	0.0607		-0.62	
3176	ISO14389	0.064		-0.31	
3197	ISO14389	0.0799		1.17	
3210	In house	0.0765		0.85	

normality	OK	
n	64	
outliers	1	
mean (n)	0.0673	
st.dev. (n)	0.01064	RSD = 16%
R(calc.)	0.0298	
st.dev.(iis memo 1701)	0.01077	
R(iis memo 1701)	0.0302	





APPENDIX 2

Abbreviations of components:

BBP = Benzyl butyl phthalate
DEHP = Di-(2-ethylhexyl) phthalate
DBP = Dibutyl phthalate
DIDP = Di-iso-decyl phthalate
DINP = Di-iso-nonyl phthalate
DNOP = Di-n-octyl phthalate
DCHP = Dicyclohexyl phthalate
DEP = Diethyl phthalate
DMP = Dimethyl phthalate
DNHP = Di-n-hexyl phthalate
DIBP = Di-iso-butyl phthalate
DPHP = Di-(2-propylheptyl) phtalate
DNPP = Di-n-pentyl phthalate
DUP = Diundecyl phthalate
DPrP = Di-n-propyl phthalate
DMEP = Di-(2-methoxyethyl) phthalate
Other = Total Other Phthalates

Other reported Phthalates in sample #21535; results in %M/M

lab	BBP	DEHP	DIDP	DINP	DNOP	DCHP	DEP	DMP
210	-----	0.004215	-----	-----	-----	-----	-----	-----
339	<0.001	<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	<0.005
623	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
840	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2115	-----	-----	-----	-----	-----	-----	-----	-----
2121	-----	-----	-----	-----	-----	-----	-----	-----
2159	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
2213	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2247	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2250	-----	-----	-----	-----	-----	-----	-----	-----
2255	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2258	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2265	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01
2293	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2310	not detected	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	not detected
2313	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2314	-----	-----	-----	-----	-----	-----	-----	-----
2330	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2347	<0.003	<0.003	<0.005	<0.005	<0.003	<0.003	<0.003	<0.003
2350	-----	-----	-----	-----	-----	-----	-----	-----
2352	-----	-----	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----	-----	-----
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2363	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2365	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2366	<0.004	<0.004	<0.02	<0.02	<0.004	<0.004	<0.004	<0.004
2369	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2370	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500
2372	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2374	-----	-----	-----	-----	-----	-----	-----	-----
2375	-----	-----	-----	-----	-----	-----	-----	-----
2378	-----	-----	-----	-----	-----	-----	-----	-----
2379	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2380	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2381	-----	-----	-----	-----	-----	-----	-----	-----
2382	<0.0030	<0.0030	<0.0030	<0.0030	<0.0030	<0.0030	<0.0030	<0.0030
2386	<0,003	<0,003	<0,003	<0,003	<0,003	<0,003	<0,003	<0,003
2390	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2425	ND	ND	ND	ND	ND	ND	ND	ND
2449	-----	-----	-----	-----	-----	-----	-----	-----
2452	< det. limit	< det. limit	< det. limit	< det. limit	< det. limit	-----	-----	-----
2453	0.0016	-----	-----	-----	-----	-----	-----	-----
2492	not detected	not detected	not detected	not detected	not detected	not detected	-----	-----
2511	-----	-----	-----	-----	-----	-----	-----	-----
2514	-----	-----	-----	-----	-----	-----	-----	-----
2549	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2561	-----	-----	-----	-----	-----	-----	-----	-----
2573	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2582	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not reported
2590	-----	-----	-----	-----	-----	-----	-----	-----
2591	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2650	0.043082 C	-----	-----	-----	-----	-----	-----	-----
2668	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2671	-----	-----	-----	-----	-----	-----	-----	-----
2703	-----	-----	-----	-----	-----	-----	-----	-----
2743	0.025223	0.000184	-----	-----	-----	-----	-----	-----
2789	-----	-----	-----	-----	-----	-----	-----	-----
2827	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2948	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2955	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
3116	-----	-----	-----	-----	-----	-----	-----	-----
3154	-----	-----	-----	-----	-----	-----	-----	-----
3166	<0.002	<0.002	<0.05	<0.05	<0.002	-----	<0.002	<0.002
3172	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
3176	-----	-----	-----	-----	-----	-----	-----	-----
3197	<0,0050	<0,0050	<0,0050	<0,0050	<0,0050	<0,0050	<0,0050	<0,0050
3210	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020

Lab 2213 first reported for all analytes in this table: <50%M/M
Lab 2650 first reported for BBP: 0.11437

Other reported Phthalates in sample #21535; results in %M/M ---- continued ----

lab	DNHP	DIBP	DPHP	DNPP	DUP	DPrP	DMEP	Other
210	-----	-----	-----	-----	-----	-----	-----	-----
339	<0.001	<0.001	-----	<0.001	-----	<0.001	<0.001	<0.005
348	<0.005	<0.005	-----	<0.005	-----	-----	<0.005	-----
623	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
840	not detected	not detected	not detected	not detected	not detected	not detected	not detected	-----
2115	-----	-----	-----	-----	-----	-----	-----	-----
2121	-----	-----	-----	-----	-----	-----	-----	-----
2159	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005	not applicable
2213	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2247	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2250	-----	-----	-----	-----	-----	-----	-----	-----
2255	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2258	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2265	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01
2293	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2310	not detected	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	not detected
2313	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2314	-----	-----	-----	-----	-----	-----	-----	-----
2330	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2347	<0.003	<0.003	<0.005	<0.003	<0.003	<0.003	<0.003	-----
2350	-----	-----	-----	-----	-----	-----	-----	-----
2352	-----	-----	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----	-----	-----
2358	n.d.	n.d.	N/A	n.d.	n.d.	n.d.	n.d.	0.023162
2363	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2365	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2366	<0.004	<0.004	<0.004	<0.004	<0.004	<0.004	<0.004	out of cap.
2369	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2370	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500
2372	not detected	not detected	not detected	not detected	not detected	not detected	not detected	0.03176
2374	-----	-----	-----	-----	-----	-----	-----	-----
2375	-----	-----	-----	-----	-----	-----	-----	-----
2378	-----	-----	-----	-----	-----	-----	-----	-----
2379	not detected	not detected	not detected	not detected	not detected	not detected	not detected	-----
2380	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	-----
2381	-----	-----	-----	-----	-----	-----	-----	-----
2382	<0.0030	<0.0030	<0.0030	<0.0030	<0.0030	<0.0030	<0.0030	<0.0030
2386	<0,003	<0,003	<0,003	<0,003	<0,003	<0,003	<0,003	<0,003
2390	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2425	ND	ND	ND	ND	ND	ND	ND	ND
2449	-----	-----	-----	-----	-----	-----	-----	0.0312
2452	-----	< det. limit	-----	< det. limit	-----	-----	< det. limit	-----
2453	-----	-----	-----	-----	-----	-----	-----	-----
2492	-----	not detected	-----	-----	-----	-----	-----	-----
2511	-----	-----	-----	-----	-----	-----	-----	-----
2514	-----	-----	-----	-----	-----	-----	-----	-----
2549	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2561	-----	-----	-----	-----	-----	-----	-----	-----
2573	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2582	Not detected	Not detected	Not reported	Not detected	Not detected	Not detected	Not reported	-----
2590	-----	-----	-----	-----	-----	-----	-----	-----
2591	not detected	not detected	-----	not detected	-----	-----	not detected	-----
2650	-----	-----	-----	-----	-----	-----	-----	-----
2668	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2671	-----	-----	-----	-----	-----	-----	-----	-----
2703	-----	-----	-----	-----	-----	-----	-----	-----
2743	-----	-----	-----	-----	-----	-----	-----	-----
2789	-----	-----	-----	-----	-----	-----	-----	-----
2827	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2948	not detected	not detected	not detected	not detected	not detected	not detected	not detected	0.0415
2955	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
3116	-----	-----	-----	-----	-----	-----	-----	-----
3154	-----	-----	-----	-----	-----	-----	-----	-----
3166	<0.002	<0.002	-----	<0.002	-----	-----	-----	-----
3172	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	-----
3176	-----	-----	-----	-----	-----	-----	-----	-----
3197	<0,0050	<0,0050	<0,0050	<0,0050	<0,0050	<0,0050	<0,0050	-----
3210	<0.0020	<0.0020	-----	<0.0020	<0.0020	-----	<0.0020	-----

Lab 2213 first reported for all analytes in this table: <50%M/M

Other reported Phthalates in sample #21536; results in %M/M

lab	DEHP	DBP	DIDP	DINP	DNOP	DEP	DMP	DNHP
210	-----	0.003989	-----	-----	-----	-----	-----	-----
339	<0.001	0.00239	<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	<0.005
623	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
840	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2115	-----	0.003	-----	-----	-----	-----	-----	-----
2121	-----	-----	-----	-----	-----	-----	-----	-----
2159	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
2213	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2247	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2250	-----	-----	-----	-----	-----	-----	-----	-----
2255	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2258	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2265	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01
2293	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2310	not detected	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	not detected
2313	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2314	-----	-----	-----	-----	-----	-----	-----	-----
2330	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2347	<0.003	<0.003	<0.005	<0.005	<0.003	<0.003	<0.003	<0.003
2350	-----	-----	-----	-----	-----	-----	-----	-----
2352	-----	-----	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----	-----	-----
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2363	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2365	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2366	<0.015	<0.015	<0.015	<0.015	<0.015	<0.015	<0.015	<0.015
2369	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2370	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500
2372	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2374	-----	-----	-----	-----	-----	-----	-----	-----
2375	-----	-----	-----	-----	-----	-----	-----	-----
2378	-----	-----	-----	-----	-----	-----	-----	-----
2379	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2380	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2381	-----	-----	-----	-----	-----	-----	-----	-----
2382	<0.0030	<0.0030	<0.0030	<0.0030	<0.0030	<0.0030	<0.0030	<0.0030
2386	<0,003	0.00321	<0,003	<0,003	<0,003	<0,003	<0,003	<0,003
2390	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2425	ND	ND	ND	ND	ND	ND	ND	ND
2449	-----	-----	-----	-----	-----	-----	-----	-----
2452	< det. limit	< det. limit	< det. limit	< det. limit	< det. limit	-----	-----	-----
2453	-----	0.0042	-----	-----	-----	-----	-----	-----
2492	not detected	not detected	not detected	not detected	not detected	-----	-----	-----
2511	-----	-----	-----	-----	-----	-----	-----	-----
2514	-----	-----	-----	-----	-----	-----	-----	-----
2549	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2561	-----	-----	-----	-----	-----	-----	-----	-----
2573	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2582	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not reported	Not detected
2590	-----	0.00305	-----	-----	-----	-----	-----	-----
2591	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2650	-----	0.128528	-----	-----	-----	-----	-----	-----
2668	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2671	-----	-----	-----	-----	-----	-----	-----	-----
2703	-----	-----	-----	-----	-----	-----	-----	-----
2743	-----	-----	-----	-----	-----	-----	-----	-----
2789	-----	-----	-----	-----	-----	-----	-----	-----
2827	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2948	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2955	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
3116	-----	-----	-----	-----	-----	-----	-----	-----
3154	-----	0.00553	-----	-----	-----	-----	-----	-----
3166	<0.002	0.0031	<0.05	<0.05	<0.002	<0.002	<0.002	<0.002
3172	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
3176	-----	0.0019	-----	-----	-----	-----	-----	-----
3197	<0,0050	<0,0050	<0,0050	<0,0050	<0,0050	<0,0050	<0,0050	<0,0050
3210	<0.0020	0.0043	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020	<0.0020

Lab 2115 first reported for DBP: 0.0069
Lab 2213 first reported for all analytes in this table: <50%M/M
Lab 2650 first reported for DBP: 0.03254

Other reported Phthalates in sample #21536; results in %M/M ---- continued ----

lab	DIBP	DPHP	DNPP	DUP	DPrP	DMEP	Other
210	-----	-----	-----	-----	-----	-----	-----
339	<0.001	-----	<0.001	-----	<0.001	<0.001	<0.005
348	<0.005	-----	<0.005	-----	-----	<0.005	-----
623	not detected	not detected	not detected	not detected	not detected	not detected	not detected
840	not detected	not detected	not detected	not detected	not detected	not detected	-----
2115	-----	-----	-----	-----	-----	-----	-----
2121	-----	-----	-----	-----	-----	-----	-----
2159	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005	not applicable
2213	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2247	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2250	-----	-----	-----	-----	-----	-----	-----
2255	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2258	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2265	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01
2293	not detected	not detected	not detected	not detected	not detected	not detected	not detected
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	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2347	<0.003	<0.005	<0.003	<0.003	<0.003	<0.003	-----
2350	-----	-----	-----	-----	-----	-----	-----
2352	-----	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----	-----
2358	n.d.	N/A	n.d.	n.d.	n.d.	n.d.	0.1664657
2363	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2365	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2366	<0.015	<0.015	<0.015	<0.015	<0.015	<0.015	out capability
2369	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2370	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500
2372	not detected	not detected	not detected	not detected	not detected	not detected	0.18808
2374	-----	-----	-----	-----	-----	-----	-----
2375	-----	-----	-----	-----	-----	-----	-----
2378	-----	-----	-----	-----	-----	-----	-----
2379	not detected	not detected	not detected	not detected	not detected	not detected	-----
2380	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	-----
2381	-----	-----	-----	-----	-----	-----	-----
2382	< 0.0030	< 0.0030	< 0.0030	< 0.0030	< 0.0030	< 0.0030	< 0.0030
2386	<0,003	<0,003	<0,003	<0,003	<0,003	<0,003	<0,003
2390	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2425	ND	ND	ND	ND	ND	ND	ND
2449	-----	-----	-----	-----	-----	-----	0.1914
2452	< det. limit	-----	< det. limit	-----	-----	< det. limit	-----
2453	-----	-----	-----	-----	-----	-----	-----
2492	not detected	-----	-----	-----	-----	-----	-----
2511	-----	-----	-----	-----	-----	-----	-----
2514	-----	-----	-----	-----	-----	-----	-----
2549	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2561	-----	-----	-----	-----	-----	-----	-----
2573	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2582	not detected	not detected	not detected	not detected	not detected	not detected	-----
2590	-----	-----	-----	-----	-----	-----	-----
2591	not detected	-----	not detected	-----	-----	not detected	-----
2650	-----	-----	-----	-----	-----	-----	-----
2668	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2671	-----	-----	-----	-----	-----	-----	-----
2703	-----	-----	-----	-----	-----	-----	-----
2743	-----	-----	-----	-----	-----	-----	-----
2789	-----	-----	-----	-----	-----	-----	-----
2827	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2948	not detected	not detected	not detected	not detected	not detected	not detected	0.1782
2955	not detected	not detected	not detected	not detected	not detected	not detected	not detected
3116	-----	-----	-----	-----	-----	-----	-----
3154	-----	-----	-----	-----	-----	-----	-----
3166	<0.002	-----	<0.002	-----	-----	-----	-----
3172	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
3176	-----	-----	-----	-----	-----	-----	-----
3197	<0,0050	<0,0050	<0,0050	<0,0050	<0,0050	<0,0050	-----
3210	<0.0020	-----	<0.0020	<0.0020	-----	<0.0020	-----

Lab 2213 first reported for all analytes in this table: <50%M/M

APPENDIX 3

Analytical details

lab	ISO/IEC 17025 accr.	Sample preparation	Sample intake used (grams)	Release/ extraction technique	Extraction solvent	Extraction time (min)	Extraction temperature (°C)
210	Yes	---	---	---	---	---	---
339	No	Used as received	0.5	Ultrasonic	THF	60	60
348	Yes	Further cut	0.5	Thermal Desorption	THF	180	60±5
623	Yes	Further cut	0.1	Ultrasonic	THF Hexane	60	60
840	Yes	Further cut	0.5	Ultrasonic	THF-Hexane	60	60
2115	Yes	Used as received	0.3	Ultrasonic	THF	60	60
2121	Yes	Further cut	0,5 *)	Ultrasonic	Hexane/THF	60	60
2159	Yes	Used as received	0,5	Ultrasonic	THF/ACN	30	40
2213	Yes	Used as received	0.3	Ultrasonic	THF+Hexane	30	Room Temp
2247	Yes	Used as received	1-2	Ultrasonic	THF & n-Hexane	30.0	Room temp
2250	Yes	Used as received	0,3	Ultrasonic	THF/Acetonitril (1:2)	60	60
2255	Yes	Further cut	0.5	Ultrasonic	THF/ACN	120	60
2258	Yes	Used as received	0.06	Thermal Desorption	THF/ACN	120	40
2265	Yes	Further cut	0,3	Ultrasonic	10mL THF 20mL n-Hexan for precip.	60	60
2293	Yes	Used as received	0.3001	Ultrasonic	THF and n-Hexane	60	60
2310	Yes	Further cut	0.1	Ultrasonic	THF/HEXANE	60	60
2311	Yes	Further cut	0.3	Ultrasonic	THF + Hexane	60	50
2313	Yes	Further cut	0.5	Ultrasonic	THF and Hexane	60	60
2314	Yes	Further cut	0.5	Ultrasonic	THF/N-HEXANE	60	60
2330	Yes	Further cut	0.30	Ultrasonic	THF : n-Hexane (1:2)	60	60 ± 5
2347	Yes	Further cut	0.3	---	30mL	60	60
2350	Yes	Used as received	0.5	Ultrasonic	THF + ACN	2 h	60
2352	Yes	Further cut	0.3	Ultrasonic	THF, Hexane	60	60
2357	---	---	---	---	---	---	---
2358	Yes	Used as received	0.3	Ultrasonic	THF, n-hexane	60	60
2363	Yes	Further cut	0.3	Ultrasonic	30ml	60	60
2365	Yes	Further cut	0.1	Ultrasonic	THF: n-hexane =1:1	60	60
2366	Yes	Used as received	ISO14389:0.3g CPSC..-09.4: 0.05	Soxhlet	THF and hexane	60	60
2369	---	---	---	---	---	---	---
2370	Yes	Further cut	0.5	Ultrasonic	THF：ACN=10：20mL	60	60
2372	Yes	Used as received	0.5	Ultrasonic	THF	60	RT
2374	Yes	Further cut	0.5	Ultrasonic	THF, Hexane	60	60
2375	Yes	Further cut	0.05	Mechanical Shaking	THF	30	Room temp
2378	Yes	Further cut	0.3	---	THF	60	60
2379	Yes	Further cut	0.3	Ultrasonic	THF : Hexane	60	60
2380	Yes	Used as received	0.5	Ultrasonic	THF	60	60
2381	Yes	Further cut	0.1	Ultrasonic	THF, n-Hexane.	60	60
2382	Yes	Used as received	0.1	Ultrasonic	THF n-Hexane	60	60
2386	Yes	Used as received	0,5	Ultrasonic	THF	60	60
2390	Yes	Further cut	0.1	Ultrasonic	THF : n-Hexane	60	60
2425	Yes	Further cut	0.3	Ultrasonic	THF: n-hexane	60	60
2449	Yes	---		---			
2452	Yes	Used as received	0.3	Ultrasonic	THF+HEXANE	60	60

lab	ISO/IEC 17025 accr.	Sample preparation	Sample intake used (grams)	Release/ extraction technique	Extraction solvent	Extraction time (min)	Extraction temperature (°C)
2453	Yes	Further cut	100mg-300mg	Ultrasonic			
2492	No	Used as received	0.3	Ultrasonic	THF:n-Hex (1:2)	60	60
2511	---	---		---			
2514	Yes	Further cut	0.2632/ 0.2791	Ultrasonic	THF:N-HEXANE=1:2	60	60
2549	Yes	Used as received	0.5	Ultrasonic	THF	60	60
2561	Yes	Used as received	0.2	Ultrasonic	10ml THF, 20ml Hexane with ISTD	60 **)	60
2573	Yes	Used as received	0.25g	Ultrasonic	THF and n-Hexane	60	60
2582	Yes	Further cut	#21535 -0.5365 g #21536 - 0.5181 g	Ultrasonic	THF	30	25
2590	Yes	Further cut	0.3g	Ultrasonic	THF_Xeh	60	60
2591	Yes	Further cut	0.2 grams	Ultrasonic	THF/n-hexane	120	Room temp
2650	Yes	Used as received	3	Ultrasonic	THF	60	60
2668	Yes	Further cut	0.3 gms	Ultrasonic	THF:Hexane	60	60
2671	---	---		---			
2703	Yes	Further cut	#21535 0.1048g, #21536 0.1026g	Ultrasonic	THF/Hexane	150	60
2743	Yes	Used as received	0,3	Ultrasonic	THF/Hexane	60	60
2789	Yes	Used as received	0.3	Ultrasonic	ACN / THF	60	60
2827	Yes	Further cut	0.5g	Ultrasonic	THF/HEXANE	60 MINS	60
2948	---	---	0.30	---	THF/ACN	60	60
2955	Yes	Further cut	0.5	Ultrasonic	THF/ACN	120	60
3116	Yes	Used as received	0.6 grams	Ultrasonic	2:1 ACN / THF	60	60°C
3154	Yes	---		---			
3166	Yes	Used as received	0.5 g	---	methylene chloride	1 h	Ambient
3172	Yes	Further cut	0.15	Ultrasonic	THF to extract, ACN to precipitate	60	25
3176	Yes	Used as received	0,3	Ultrasonic	THF	60	60
3197	Yes	Further cut	0,3 g	Ultrasonic	THF/n-hexane	60	60
3210	Yes	Used as received	1g	Ultrasonic	80% Hexane 20% Acetone	60	50

*) lab 2121 reported: 5 replicates per sample
**) lab 2561 reported: ultrasonic, shake 30 secs, settle 30 mins

APPENDIX 4

Number of participants per country

6 labs in BANGLADESH
1 lab in CAMBODIA
3 labs in FRANCE
4 labs in GERMANY
2 labs in GUATEMALA
3 labs in HONG KONG
10 labs in INDIA
1 lab in INDONESIA
4 labs in ITALY
1 lab in MOROCCO
11 labs in P.R. of CHINA
3 labs in PAKISTAN
1 lab in PORTUGAL
1 lab in SOUTH KOREA
4 labs in SPAIN
1 lab in SRI LANKA
2 labs in TAIWAN
1 lab in THAILAND
2 labs in TUNISIA
4 labs in TURKEY
1 lab in U.S.A.
2 labs in UNITED KINGDOM
1 lab 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

Literature

1 iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation, June 2018
2 ISO5725:86
3 ISO5725 parts 1-6:94
4 ISO13528:05
5 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
6 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
7 P.L. Davies, Fr Z. Anal. Chem, 351, 513, (1988)
8 J.N. Miller, Analyst, 118, 455, (1993)
9 Analytical Methods Committee, Technical brief, No 4, January 2001
10 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)
11 Horwitz, R. Albert, J. AOAC Int, 79, 3, 589, (1996)
12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)
13 iis memo 1701 Precision data of Phthalates in Polymers