Results of Proficiency Test
Phthalates in Textile
March 2020

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. G.A. Oosterlaken-Buijs
Correctors: ing. A.S. Noordman-de Neef & ing R.J. Starink

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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) organized an interlaboratory study for the determination of Phthalates in Textile in 2019. During the annual proficiency testing program 2019/2020 it was decided to continue the proficiency test for the analysis of Phthalates in Textile.

In this interlaboratory study 74 laboratories in 28 different countries registered for participation. See appendix 4 for the number of participating laboratories per country. In this report the results of this 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/IEC17025 accredited laboratory. It was decided to send two different textile samples both positive on Phthalates of approximately 3 grams each, one sample of brown cotton pieces labelled #20540 and one sample of green/blue cotton pieces labelled #20541. 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 brown cotton was selected which was made positive on Di-n-hexyl phthalate (DNHP) by iis. The material was cut into small pieces. After homogenization 100 subsamples of approximately 3 grams each were prepared and labelled #20540. The homogeneity of subsamples #20540 was checked by determination of DNHP using an in-house test method on 10 stratified randomly selected subsamples.

	DNHP in %M/M
sample #20540-1	0.14616
sample #20540-2	0.14901
sample #20540-3	0.14962
sample #20540-4	0.15369
sample #20540-5	0.15121
sample #20540-6	0.15740
sample #20540-7	0.14866
sample #20540-8	0.15725
sample #20540-9	0.14927
sample #20540-10	0.14796

Table 1: homogeneity test results of subsamples #20540

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.

	DNHP
RSD% (observed)	3%
reference method	iis memo 1701
0.3 * RSD% (ref. method)	5%

Table 2: evaluation of the repeatability of subsamples #20540

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

A batch green/blue cotton was selected which was made positive on Di-(2-methoxyethyl) phthalate (DMEP). The batch was cut into small pieces. After homogenization 150 subsamples of approximately 3 grams each were prepared and labelled #20541. The homogeneity of subsamples #20541 was checked by determination of DMEP using an in-house test method on 9 stratified randomly selected subsamples.

	DMEP in %M/M
sample #20541-1	0.15019
sample #20541-2	0.14133
sample #20541-3	0.16039
sample #20541-4	0.15063
sample #20541-5	0.14655
sample #20541-6	0.15831
sample #20541-7	0.16639
sample #20541-8	0.15908
sample #20541-9	0.14410

Table 3: homogeneity test results of subsamples #20541

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.

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

Table 4: evaluation of the repeatability of subsamples #20541

The observed RSD% is larger than 0.3 times estimated RSD% of the reference method. It should be noted that the PTs mentioned in iis memo 1701 are all PTs of Phthalates in Polymers. Based on the experience of iis an observed RSD of the homogeneity data of ≤6% for PT samples is sufficient to continue with these subsamples.

To each of the participating laboratories one sample labelled #20540 and one sample labelled #20541 were sent on February 12, 2020.

2.5 ANALYZES

The participants were requested to determine on samples #20540 and #20541 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 requested, to ensure homogeneity, to not use less than 0.5 grams per determination. It was also requested to report if the laboratory was accredited to determine the reported components 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 ISO5725 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:

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

4 EVALUATION

In this interlaboratory study some problems were encountered with the dispatch of the samples due to the COVID-19 pandemic. Seven participants reported after the final reporting date and five participants did not report any test results at all. In total 69 laboratories reported 134 numerical test results. Observed were 12 outlying test results, which is 9.0% of the numerical test 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 in appendix 1. The abbreviations, used in these tables, are explained in appendix 5.

Regretfully, the CPSC-CH-C1001-09.3 or 4 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 as the RSD (relative standard deviation) of 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 second time that iis conducted a PT on Phthalates in Textile and test method ISO14389:14 does provide a variety of precision data only it was therefore again decided to use the estimated iis target reproducibility for the polymer PT also for the textile PT.

Sample #20540

DNHP: This determination may be problematic for a number of laboratories. Six statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in 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 #20541

DMEP: This determination may be problematic for a number of laboratories. Six statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in 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)
DNHP	%M/M	60	0.096	0.032	0.043

Table 5: reproducibility on sample #20540

Component	unit	n	average	2.8 * sd	R(target)
DMEP	%M/M	62	0.101	0.049	0.045

Table 6: reproducibility on sample #20541

Without further calculations, it could be concluded that for Di-n-hexyl phthalate (DNHP) and Di-(2-methoxyethyl) phthalate (DMEP) there is a good compliance of the group of participating laboratories with the target.

4.3 COMPARISON OF THE PROFICIENCY TEST OF MARCH 2020 WITH THE PREVIOUS PT

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

Table 7: comparison with previous proficiency test

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

Table 8: development of uncertainties over the years

Both uncertainties of DNHP and DMEP observed in this PT are close to or in line with the requirements mentioned in the target.

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 90% of the participants mentioned that they are accredited for the determination of Phthalates in Textile.
- About 55% of the 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 45% of the participants used the samples as received and about 55% further cut the samples prior to analysis.
- 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 70°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 effect of further cutting/further grinding on the determination of DNHP in sample #20540 and DMEP in sample #20541 was further investigated, see tables 9 and 10 respectively. It appeared that the effect of further reducing of the sample size on the determination of DNHP and DMEP in this PT is small and not statistically significant.

DNHP	unit	n	average	2.8 * sd
Overall test results	%M/M	60	0.096	0.032
Further cut/grinded	%M/M	28	0.096	0.030
Used as received	%M/M	26	0.095	0.033

Table 9: effect of analytical details on DNHP in sample #20540

DMEP	unit	n	average	2.8 * sd
Overall test results	%M/M	62	0.101	0.049
Further cut/grinded	%M/M	32	0.102	0.058
Used as received	%M/M	24	0.099	0.038

Table 10: effect of analytical details on DMEP in sample #20541

5 DISCUSSION

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/IEC17025 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

Although, it can be concluded that the majority of the participants has no problem with the determination of Di-n-hexyl phthalate (DNHP) and Di-(2-methoxyethyl) phthalate (DMEP) in the 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.

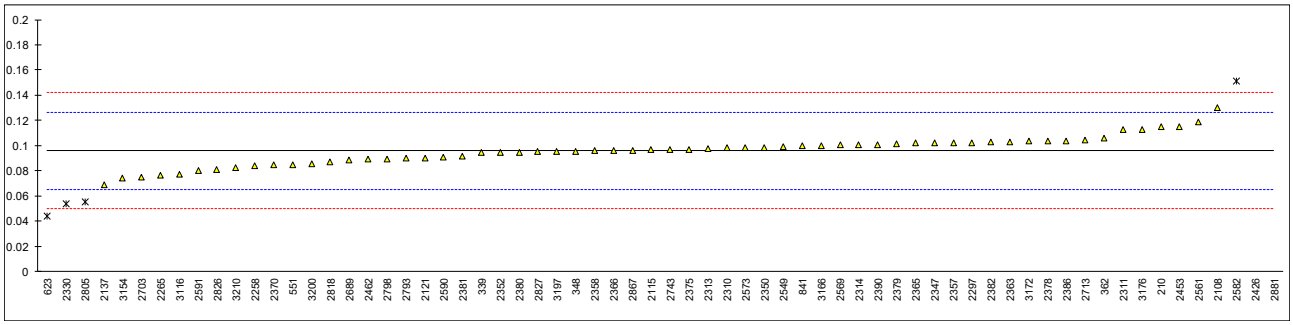
APPENDIX 1

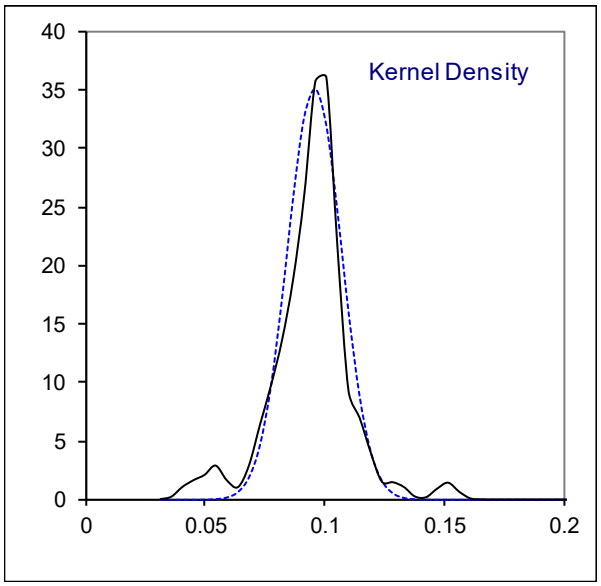
Determination of DNHP – Di-n-hexyl phthalate on sample #20540; results in %M/M

lab	method	value	mark	z(targ)	remarks
210	ISO14389	0.114855		1.23	
339	In house	0.0945		-0.09	
348	CPSC-CH-C1001-09.4	0.0951		-0.05	
362	In house	0.106		0.66	
551	In house	0.0849		-0.72	
623	ISO14389	0.0439	R(0.05)	-3.39	
841	ISO14389	0.0999		0.26	
2108	ISO14389	0.130		2.22	
2115	ISO14389	0.0965		0.04	
2121	ISO14389	0.09		-0.38	
2137	IEC62321-8	0.0691		-1.75	
2258	CPSC-CH-C1001-09.3	0.08414		-0.77	
2265	ISO14389	0.0762		-1.28	
2267		-----		-----	
2297	ISO14389	0.1023		0.42	
2310	CPSC-CH-C1001-09.3	0.098		0.14	
2311	CPSC-CH-C1001-09.3	0.1130		1.11	
2313	ISO14389	0.0975		0.10	
2314	ISO14389	0.1010		0.33	
2330	ISO14389	0.0536	C,R(0.05)	-2.76	first reported 0.0477
2347	ISO14389	0.102		0.40	
2350	CPSC-CH-C1001-09.4	0.0987		0.18	
2352	ISO14389	0.0946		-0.09	
2357	ISO14389	0.1020		0.40	
2358	ISO14389	0.09593		0.00	
2363	ISO14389	0.103		0.46	
2365	ISO14389	0.1019		0.39	
2366	ISO14389	0.096		0.01	
2370	ISO14389	0.0847		-0.73	
2375	ISO14389	0.097		0.07	
2378	ISO14389	0.1040		0.53	
2379	ISO14389	0.1016		0.37	
2380	ISO14389	0.09468		-0.08	
2381	CPSC-CH-C1001-09.3	0.0915		-0.29	
2382	ISO14389	0.1028		0.45	
2386	ISO14389	0.104		0.53	
2390	CPSC-CH-C1001-09.3	0.101		0.33	
2426	CPSC-CH-C1001-09.4	0.223695	R(0.01)	8.33	
2449		-----		-----	
2452		-----		-----	
2453	CPSC-CH-C1001-09.3	0.115		1.24	
2462	GB/T20388-ISO14389	0.089		-0.45	
2476		-----		-----	
2549	ISO14389	0.099		0.20	
2561	ISO14389	0.1187		1.49	
2569	ISO14389	0.1006		0.31	
2573	CPSC-CH-C1001-09.4	0.0983		0.16	
2582	ISO14389	0.151184	R(0.05)	3.60	
2590	ISO14389	0.0910		-0.32	
2591	CPSC-CH-C1001-09.3	0.07999		-1.04	
2689	CPSC-CH-C1001-09.3	0.0884		-0.49	
2703	In house	0.07484		-1.37	
2713	ISO14389	0.1043	C	0.55	first reported 1043 %M/M
2743	ISO14389	0.096621		0.05	
2789		-----		-----	
2793	ISO14389	0.0899		-0.39	
2798	ISO14389	0.0891		-0.44	
2804		-----		-----	
2805		0.0554	R(0.05)	-2.64	
2812		-----		-----	
2818	ISO14389	0.087		-0.58	
2826	ISO14389	0.0812		-0.96	
2827	ISO14389	0.095		-0.06	
2867	ISO14389	0.096		0.01	
2881	In house	1.2	C,R(0.01)	71.95	first reported 0.24
2912		-----		-----	
3116	ISO14389	0.07738		-1.21	
3154	In house	0.0742		-1.41	
3166	In house	0.1002		0.28	
3172	ISO8124-6	0.1038		0.51	
3176	CPSC-CH-C1001-09.4	0.113		1.11	
3197	ISO14389	0.0950		-0.06	
3200	CPSC-CH-C1001-09.3	0.0858		-0.66	
3210	In house	0.0827		-0.86	

normality	OK
n	60
outliers	6
mean (n)	0.0959
st.dev. (n)	0.01137
R(calc.)	0.0318
st.dev.(iis memo 1701)	0.01535
R(iis memo 1701)	0.0430

RSD = 12%



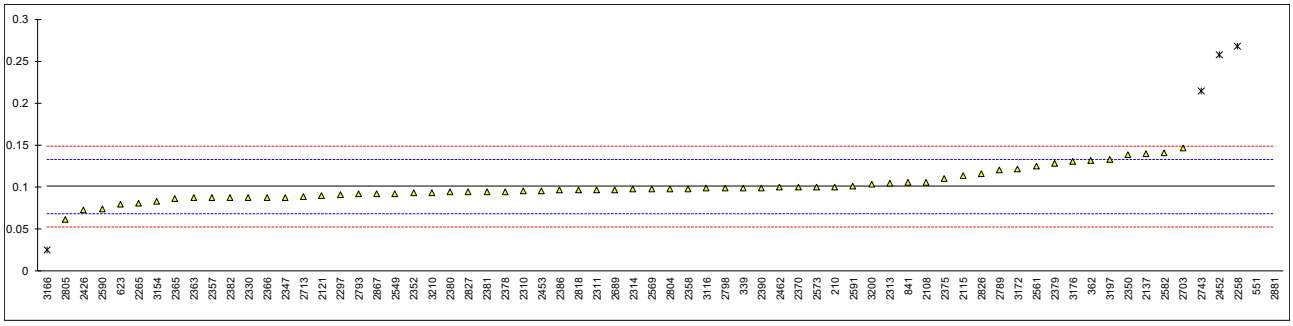


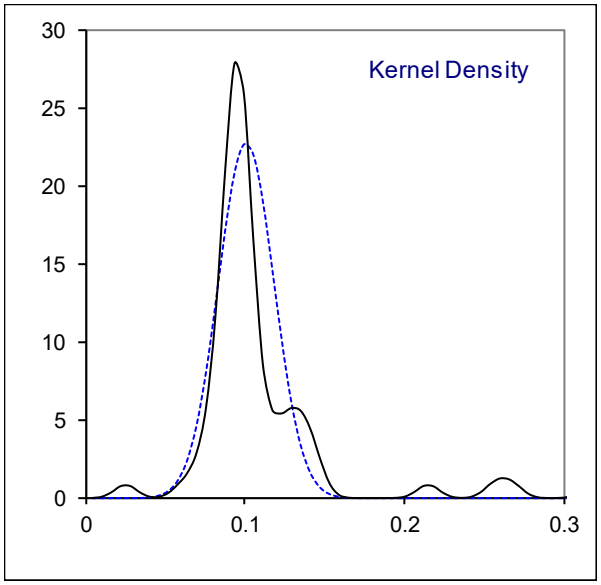
Determination of DMEP – Di-(2-methoxyethyl) phthalate on sample #20541; results in %M/M

lab	method	value	mark	z(targ)	remarks
210	ISO14389	0.100394		-0.02	
339	In house	0.0989		-0.12	
348		-----		-----	,
362	In house	0.132		1.94	
551	In house	0.3179	C,R(0.01)	13.46	first reported 0.1673
623	ISO14389	0.0791		-1.35	
841	ISO14389	0.1051		0.27	
2108	ISO14389	0.106		0.32	
2115		0.113		0.76	
2121	ISO14389	0.09		-0.67	
2137	ISEC62321-8	0.1399		2.43	
2258		0.267326	C,R(0.01)	10.33	first reported 0.37376
2265	ISO14389	0.0804		-1.26	
2267		-----		-----	
2297	ISO14389	0.091		-0.61	
2310	CPSC-CH-C1001-09.3	0.095		-0.36	
2311	CPSC-CH-C1001-09.3	0.0967		-0.25	
2313	ISO14389	0.104		0.20	
2314	ISO14389	0.0980		-0.17	
2330	ISO14389	0.0877		-0.81	
2347	GB/T20388	0.088		-0.79	
2350	CPSC-CH-C1001-09.4	0.1382		2.32	
2352	ISO14389	0.0931		-0.48	
2357	ISO14389	0.0870		-0.86	
2358	ISO14389	0.09811		-0.17	
2363	ISO14389	0.087		-0.86	
2365	ISO14389	0.0865		-0.89	
2366	ISO14389	0.088		-0.79	
2370	ISO14389	0.0998		-0.06	
2375	ISO14389	0.110		0.57	
2378	ISO14389	0.0948		-0.37	
2379	ISO14389	0.1280		1.69	
2380		0.09385		-0.43	
2381	CPSC-CH-C1001-09.3	0.0945		-0.39	
2382	ISO14389	0.0873		-0.84	
2386	ISO14389	0.096		-0.30	
2390		0.099		-0.11	
2426	CPSC-CH-C1001-09.4	0.073058		-1.72	
2449		-----		-----	
2452	ISO14389	0.257	R(0.01)	9.69	
2453	ISO14389	0.095		-0.36	
2462	GB/T20388-ISO14389	0.0995		-0.08	
2476		-----		-----	
2549	ISO14389	0.0923		-0.53	
2561	ISO14389	0.1252		1.51	
2569	ISO14389	0.098		-0.17	
2573	CPSC-CH-C1001-09.4	0.1001		-0.04	
2582	ISO14389	0.141049		2.50	
2590	ISO14389	0.0740		-1.66	
2591	CPSC-CH-C1001-09.3	0.10111		0.02	
2689	CPSC-CH-C1001-09.3	0.0971		-0.23	
2703	In house	0.14589		2.80	
2713	ISO14389	0.0890	C	-0.73	first reported 890 %M/M
2743	ISO14389	0.214938	C,R(0.01)	7.08	first reported 0.188749
2789		0.12		1.19	
2793	ISO14389	0.0915		-0.58	
2798	ISO14389	0.0986		-0.14	
2804	ISO14389	0.098		-0.17	
2805		0.0616		-2.43	
2812		-----		-----	
2818	ISO14389	0.096		-0.30	
2826	ISO14389	0.116		0.94	
2827	ISO14389	0.0939		-0.43	
2867	ISO14389	0.092		-0.55	
2881	In house	1.5	C,R(0.01)	86.76	first reported 0.56
2912		-----		-----	
3116	ISO14389	0.09849		-0.14	
3154	In house	0.0827		-1.12	
3166	In house	0.0251	R(0.01)	-4.69	semi-quantitative estimate
3172	ISO8124-6	0.1214		1.28	
3176	CPSC-CH-C1001-09.4	0.131		1.87	
3197	ISO14389	0.1329		1.99	
3200	CPSC-CH-C1001-09.3	0.1038		0.19	
3210	In house	0.0935		-0.45	

normality	OK
n	62
outliers	6
mean (n)	0.1008
st.dev. (n)	0.01752
R(calc.)	0.0491
st.dev.(iis memo 1701)	0.01613
R(iis memo 1701)	0.0452

RSD = 17%





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 #20540; results in %M/M

Lab	BBP	DEHP	DBP	DIDP	DINP	DNOP	DCHP	DEP
210	-----	-----	-----	-----	-----	-----	-----	-----
339	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	-----
348	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
362	<0.003	<0.003	<0.003	<0.010	<0.010	<0.003	<0.003	-----
551	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
623	ND	ND	ND	ND	ND	ND	ND	ND
841	ND	ND	ND	ND	ND	ND	ND	ND
2108	-----	-----	-----	-----	-----	-----	-----	-----
2115	-----	-----	-----	-----	-----	-----	-----	-----
2121	-----	-----	-----	-----	-----	-----	-----	-----
2137	-----	-----	-----	-----	-----	-----	-----	-----
2258	ND	ND	ND	ND	ND	ND	ND	ND
2265	< 0,025	< 0,025	< 0,025	< 0,025	< 0,025	< 0,025	< 0,025	< 0,025
2267	-----	-----	-----	-----	-----	-----	-----	-----
2297	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
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	-----	-----	-----	-----	-----	-----	-----	-----
2314	-----	-----	-----	-----	-----	-----	-----	-----
2330	ND	ND	ND	ND	ND	ND	ND	ND
2347	<0.003	<0.003	<0.003	<0.005	<0.005	<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	<0.003	<0.003	<0.003	<0.005	<0.005	<0.003	<0.003	<0.003
2366	<0.004	<0.004	<0.004	<0.004	<0.004	<0.004	<0.004	<0.004
2370	< 0.0030	< 0.0030	< 0.0030	< 0.0030	< 0.0030	< 0.0030	< 0.0030	< 0.0030
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	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050
2386	<0,03	<0,03	<0,03	<0,03	<0,03	<0,03	<0,03	<0,03
2390	-----	-----	-----	-----	-----	-----	-----	-----
2426	ND	ND	ND	ND	ND	ND	ND	ND
2449	-----	-----	-----	-----	-----	-----	-----	-----
2452	-----	-----	-----	-----	-----	-----	-----	-----
2453	-----	-----	-----	-----	-----	-----	-----	-----
2462	-----	-----	-----	-----	-----	-----	-----	-----
2476	-----	-----	-----	-----	-----	-----	-----	-----
2549	ND	ND	ND	ND	ND	ND	ND	ND
2561	<100	<100	<100	<100	<100	<100	<100	<100
2569	ND	ND	ND	ND	ND	ND	ND	ND
2573	ND	ND	ND	ND	ND	ND	ND	ND
2582	ND	ND	ND	ND	ND	ND	ND	ND
2590	-----	-----	-----	-----	-----	-----	-----	-----
2591	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2689	ND	ND	ND	ND	ND	ND	ND	ND
2703	-----	-----	-----	-----	-----	-----	-----	-----
2713	<0.005 C	<0.005 C	<0.005 C	<0.005 C	<0.005 C	<0.005 C	-----	-----
2743	-----	-----	-----	-----	-----	-----	-----	-----
2789	-----	-----	-----	-----	-----	-----	-----	-----
2793	-----	-----	-----	-----	-----	-----	-----	-----
2798	-----	-----	-----	-----	-----	-----	-----	-----
2804	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	-----	-----
2805	Non Detected	Non Detected	Non Detected	Non Detected	Non Detected	Non Detected	Non Detected	Non Detected
2812	Not Dedected	Not Dedected	Not Dedected	Not Dedected	Not Dedected	Not Dedected	-----	-----
2818	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2826	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	-----
2827	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2867	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d
2881	0.0	0.0058	0.000032	0.0	0.0	0.0	0.0	0.0
2912	-----	-----	-----	-----	-----	-----	-----	-----
3116	-----	-----	-----	-----	-----	-----	-----	-----
3154	-----	-----	-----	-----	-----	-----	-----	-----
3166	<0.002	<0.002	<0.002	<0.05	<0.05	<0.002	-----	<0.002
3172	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
3176	-----	-----	-----	-----	-----	-----	-----	-----
3197	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D
3200	<0.0100	<0.0100	<0.0100	<0.0100	<0.0100	<0.0100	<0.0100	<0.0100
3210	<0.002	<0.002	<0.002	<0.005	<0.005	<0.002	<0.002	<0.002

Lab 2713: first reported <50 %M/M

Other reported Phthalates in sample #20540; results in %M/M ---- continued ----

Lab	DMP	DIBP	DPHP	DNPP	DUP	DPrP	DMEP	Other
210	-----	-----	-----	-----	-----	-----	0.006213	-----
339	-----	<0.01	-----	<0.01	-----	-----	0.0011	-----
348	<0.005	<0.005	-----	<0.005	-----	-----	-----	-----
362	<0.003	<0.003	-----	-----	-----	-----	0.0038	-----
551	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	-----
623	ND	ND	ND	ND	ND	ND	0.0150	ND
841	ND	ND	ND	ND	ND	ND	ND	ND
2108	-----	-----	-----	-----	-----	-----	-----	-----
2115	-----	-----	-----	-----	-----	-----	0.0040	-----
2121	-----	-----	-----	-----	-----	-----	-----	-----
2137	-----	-----	-----	-----	-----	-----	-----	-----
2258	ND	ND	ND	ND	ND	ND	0.02510	ND
2265	-----	< 0,025	-----	< 0,025	< 0,025	< 0,025	< 0,025	-----
2267	-----	-----	-----	-----	-----	-----	-----	-----
2297	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.1023
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	<0.005	Not Detected
2313	-----	-----	-----	-----	-----	-----	-----	-----
2314	-----	-----	-----	-----	-----	-----	-----	-----
2330	ND	ND	ND	ND	ND	ND	ND	0.0477
2347	<0.003	<0.003	<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.	0.09593
2363	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2365	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003
2366	<0.004	<0.004	<0.004	<0.004	<0.004	<0.004	<0.004	-----
2370	< 0.0030	< 0.0030	< 0.0030	< 0.0030	< 0.0030	< 0.0030	< 0.0030	< 0.0030
2375	-----	-----	-----	-----	-----	-----	-----	-----
2378	-----	-----	-----	-----	-----	-----	-----	-----
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	0.0130	Not tested
2380	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	-----
2381	-----	-----	-----	-----	-----	-----	-----	-----
2382	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050
2386	<0,03	<0,03	<0,03	<0,03	<0,03	<0,03	<0,03	<0,03
2390	-----	-----	-----	-----	-----	-----	-----	-----
2426	ND	ND	ND	ND	ND	ND	ND	0.223695
2449	-----	-----	-----	-----	-----	-----	-----	-----
2452	-----	-----	-----	-----	-----	-----	-----	-----
2453	-----	-----	-----	-----	-----	-----	-----	-----
2462	-----	-----	-----	-----	-----	-----	-----	-----
2476	-----	-----	-----	-----	-----	-----	-----	-----
2549	ND	ND	ND	ND	ND	ND	ND	ND
2561	<100	<100	-----	-----	-----	-----	<100	-----
2569	ND	ND	ND	ND	ND	ND	ND	0.1006
2573	ND	ND	ND	ND	ND	ND	ND	ND
2582	ND	ND	ND	ND	ND	ND	ND	ND
2590	-----	-----	-----	-----	-----	-----	-----	-----
2591	<0.005	<0.005	-----	<0.005	-----	-----	<0.005	-----
2689	ND	ND	ND	ND	ND	ND	ND	-----
2703	-----	-----	-----	-----	-----	-----	0.00138	-----
2713	-----	<0.005 C	-----	<0.005 C	-----	-----	<0.005 C	-----
2743	-----	-----	-----	-----	-----	-----	-----	-----
2789	-----	-----	-----	-----	-----	-----	0.0022	-----
2793	-----	-----	-----	-----	-----	-----	0.0014	-----
2798	-----	-----	-----	-----	-----	-----	-----	-----
2804	-----	<0.005	-----	<0.005	-----	-----	<0.005	<0.005
2805	Non Detected	Non Detected	-----	Non Detected	-----	-----	Non Detected	-----
2812	-----	Not Dedected	-----	-----	-----	-----	-----	-----
2818	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2826	-----	<0.005	<0.005	-----	<0.005	-----	0.005	-----
2827	Not Detected	Not Detected	Not Detected	Not Detected	-----	Not Detected	Not Detected	-----
2867	n.d	n.d	n.d	n.d	n.d	n.d	n.d	-----
2881	0.0002	0.0098	0.0	0.0	0.0	0.0	0.003	-----
2912	-----	-----	-----	-----	-----	-----	-----	-----
3116	-----	-----	-----	-----	-----	-----	-----	-----
3154	-----	-----	-----	-----	-----	-----	-----	-----
3166	<0.002	<0.002	-----	<0.002	-----	-----	-----	-----
3172	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	0.1038
3176	-----	-----	-----	-----	-----	-----	-----	-----
3197	N.D	N.D	N.D	N.D	N.D	N.D	0.0026	-----
3200	<0.0100	<0.0100	<0.0100	<0.0100	<0.0100	<0.0100	<0.0100	<0.0100
3210	<0.002	<0.002	-----	<0.002	<0.002	-----	<0.002	-----

Lab 2713 first reported <50 %M/M

Other reported Phthalates in sample #20541; results in %M/M

Lab	BBP	DEHP	DBP	DIDP	DINP	DNOP	DCHP	DEP
210	-----	-----	-----	-----	-----	-----	-----	-----
339	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	-----
348	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
362	<0.003	<0.003	<0.003	<0.010	<0.010	<0.003	<0.003	-----
551	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
623	ND	ND	ND	ND	ND	ND	ND	ND
841	ND	ND	ND	ND	ND	ND	ND	ND
2108	-----	-----	-----	-----	-----	-----	-----	-----
2115	-----	-----	-----	-----	-----	-----	-----	-----
2121	-----	-----	-----	-----	-----	-----	-----	-----
2137	-----	-----	-----	-----	-----	-----	-----	-----
2258	ND	ND	ND	ND	ND	ND	ND	ND
2265	< 0,025	< 0,025	< 0,025	< 0,025	< 0,025	< 0,025	< 0,025	< 0,025
2267	-----	-----	-----	-----	-----	-----	-----	-----
2297	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
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	-----	-----	-----	-----	-----	-----	-----	-----
2314	-----	-----	-----	-----	-----	-----	-----	-----
2330	ND	ND	ND	ND	ND	ND	ND	ND
2347	<0.003	<0.003	<0.003	<0.005	<0.005	<0.003	-----	-----
2350	-----	-----	-----	-----	-----	-----	-----	-----
2352	-----	-----	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----	-----	-----
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2363	£¼0.005	£¼0.005	<0.005	£¼0.005	£¼0.005	£¼0.005	£¼0.005	£¼0.005
2365	<0.003	<0.003	<0.003	<0.005	<0.005	<0.003	<0.003	<0.003
2366	<0.004	<0.004	<0.004	<0.004	<0.004	<0.004	<0.004	<0.004
2370	< 0.0030	< 0.0030	< 0.0030	< 0.0030	< 0.0030	< 0.0030	< 0.0030	< 0.0030
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	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050
2386	<0,03	<0,03	<0,03	<0,03	<0,03	<0,03	<0,03	<0,03
2390	-----	-----	0.0047	-----	-----	-----	-----	-----
2426	ND	ND	ND	ND	ND	ND	ND	ND
2449	-----	-----	-----	-----	-----	-----	-----	-----
2452	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2453	-----	-----	-----	-----	-----	-----	-----	-----
2462	-----	-----	-----	-----	-----	-----	-----	-----
2476	-----	-----	-----	-----	-----	-----	-----	-----
2549	ND	ND	ND	ND	ND	ND	ND	ND
2561	<100	<100	<100	<100	<100	<100	<100	<100
2569	ND	ND	ND	ND	ND	ND	ND	ND
2573	ND	ND	ND	ND	ND	ND	ND	ND
2582	ND	ND	ND	ND	ND	ND	ND	ND
2590	-----	-----	-----	-----	-----	-----	-----	-----
2591	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2689	ND	ND	ND	ND	ND	ND	ND	ND
2703	-----	-----	-----	-----	-----	-----	-----	-----
2713	<0.005 C	<0.005 C	<0.005 C	<0.005 C	<0.005 C	<0.005 C	-----	-----
2743	-----	-----	-----	-----	-----	-----	-----	-----
2789	-----	-----	-----	-----	-----	-----	-----	-----
2793	-----	-----	-----	-----	-----	-----	-----	-----
2798	-----	-----	-----	-----	-----	-----	-----	-----
2804	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	-----	-----
2805	Non Detected	Non Detected	Non Detected	Non Detected	Non Detected	Non Detected	Non Detected	Non Detected
2812	Not Dedected	Not Dedected	Not Dedected	Not Dedected	Not Dedected	Not Dedected	-----	-----
2818	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2826	<0.005	<0.005	0.006	<0.005	<0.005	<0.005	<0.005	-----
2827	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2867	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d
2881	0.0	0.02	0.0002	0.0	0.0	0.0	0.0	0.0
2912	-----	-----	-----	-----	-----	-----	-----	-----
3116	-----	-----	-----	-----	-----	-----	-----	-----
3154	-----	-----	-----	-----	-----	-----	-----	-----
3166	<0.002	<0.002	<0.002	<0.05	<0.05	<0.002	-----	<0.002
3172	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
3176	-----	-----	-----	-----	-----	-----	-----	-----
3197	N.D	N.D	N.D	N.D	N.D	N.D	N.D	N.D
3200	<0.0100	<0.0100	<0.0100	<0.0100	<0.0100	<0.0100	<0.0100	<0.0100
3210	<0.002	<0.002	<0.002	<0.005	<0.005	<0.002	<0.002	<0.002

Lab 2713 first reported <50 %M/M

Other reported Phthalates in sample #20541; results in %M/M ---- continued ----

Lab	DMP	DNHP	DIBP	DPHP	DNPP	DUP	DPrP	other
210	-----	-----	-----	-----	-----	-----	-----	-----
339	-----	<0.01	<0.01	-----	<0.01	-----	-----	-----
348	<0.005	<0.005	<0.005	-----	<0.005	-----	-----	-----
362	<0.003	<0.003	<0.003	-----	-----	-----	-----	-----
551	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	-----
623	ND	ND	ND	ND	ND	ND	ND	ND
841	ND	ND	ND	ND	ND	ND	ND	ND
2108	-----	-----	-----	-----	-----	-----	-----	-----
2115	-----	-----	-----	-----	-----	-----	-----	-----
2121	-----	-----	-----	-----	-----	-----	-----	-----
2137	-----	-----	-----	-----	-----	-----	-----	-----
2258	ND	0.007524	ND	ND	ND	ND	ND	ND
2265	-----	< 0,025	< 0,025	-----	< 0,025	< 0,025	< 0,025	-----
2267	-----	-----	-----	-----	-----	-----	-----	-----
2297	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.091
2310	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2311	Not Detected	<0.005	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2313	-----	-----	-----	-----	-----	-----	-----	-----
2314	-----	-----	-----	-----	-----	-----	-----	-----
2330	ND	ND	ND	ND	ND	ND	ND	0.0877
2347	-----	-----	<0.003	-----	<0.003	-----	-----	-----
2350	-----	-----	-----	-----	-----	-----	-----	-----
2352	-----	-----	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----	-----	-----
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.09811
2363	£¼0.005	£¼0.005	£¼0.005	£¼0.005	£¼0.005	£¼0.005	£¼0.005	£¼0.005
2365	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003
2366	<0.004	<0.004	<0.004	<0.004	<0.004	<0.004	<0.004	-----
2370	< 0.0030	< 0.0030	< 0.0030	< 0.0030	< 0.0030	< 0.0030	< 0.0030	< 0.0030
2375	-----	-----	-----	-----	-----	-----	-----	-----
2378	-----	-----	-----	-----	-----	-----	-----	-----
2379	Not detected	0.0044	Not detected	Not detected	Not detected	Not detected	Not detected	Not tested
2380	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	-----
2381	-----	-----	-----	-----	-----	-----	-----	-----
2382	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050
2386	<0,03	<0,03	<0,03	<0,03	<0,03	<0,03	<0,03	<0,03
2390	-----	-----	-----	-----	-----	-----	-----	-----
2426	ND	ND	ND	ND	ND	ND	ND	0.073058
2449	-----	-----	-----	-----	-----	-----	-----	-----
2452	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2453	-----	0.005	-----	-----	-----	-----	-----	-----
2462	-----	-----	-----	-----	-----	-----	-----	-----
2476	-----	-----	-----	-----	-----	-----	-----	-----
2549	ND	ND	ND	ND	ND	ND	ND	ND
2561	<100	<100	<100	-----	-----	-----	-----	-----
2569	ND	ND	ND	ND	ND	ND	ND	0.098
2573	ND	ND	ND	ND	ND	ND	ND	ND
2582	ND	ND	ND	ND	ND	ND	ND	ND
2590	-----	-----	-----	-----	-----	-----	-----	-----
2591	<0.005	<0.005	<0.005	-----	<0.005	-----	-----	-----
2689	ND	ND	ND	ND	ND	ND	ND	-----
2703	-----	-----	-----	-----	-----	-----	-----	-----
2713	-----	<0.005 C	<0.005 C	-----	<0.005 C	-----	-----	-----
2743	-----	0.003271	-----	-----	-----	-----	-----	-----
2789	-----	-----	-----	-----	-----	-----	-----	-----
2793	-----	0.0014	-----	-----	-----	-----	-----	-----
2798	-----	-----	-----	-----	-----	-----	-----	-----
2804	-----	-----	<0.005	-----	<0.005	-----	-----	<0.005
2805	Non Detected	Non Detected	Non Detected	-----	Non Detected	-----	-----	-----
2812	-----	-----	Not Dedected	-----	-----	-----	-----	-----
2818	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2826	-----	<0.005	<0.005	<0.005	-----	<0.005	-----	-----
2827	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	-----	Not Detected	-----
2867	n.d	n.d	n.d	n.d	n.d	n.d	n.d	-----
2881	0.001	0.01	0.0061	0.0	0.0	0.0	0.0	-----
2912	-----	-----	-----	-----	-----	-----	-----	-----
3116	-----	-----	-----	-----	-----	-----	-----	-----
3154	-----	-----	-----	-----	-----	-----	-----	-----
3166	<0.002	<0.002	<0.002	-----	-----	-----	-----	-----
3172	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	0.1214
3176	-----	-----	-----	-----	-----	-----	-----	-----
3197	N.D	N.D	N.D	N.D	N.D	N.D	N.D	-----
3200	<0.0100	<0.0100	<0.0100	<0.0100	<0.0100	<0.0100	<0.0100	<0.0100
3210	<0.002	<0.002	<0.002	-----	<0.002	<0.002	-----	-----

Lab 2713 first 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	Extrac. time (min)	Extrac. temp. (°C)
210	Yes	3	Further Cut		Ultrasonic	THF/HEXANE		
339	No		---		---			
348	Yes	0.5	Further Cut	2*2 mm	Ultrasonic	THF	180	60±5
362	No	0.10	Used as received	5mm	Ultrasonic	THF-Hexane	60	60
551	No	0.5	Used as received		Ultrasonic	THF/Hexane (1:1)	60	60
623	Yes	0.1	Further Cut	5 mm x 5 mm	Ultrasonic	THF	60	60
841	Yes	0.3	Used as received	5X5mm	Ultrasonic	THF	60	60
2108	Yes	0,5	Used as received		Ultrasonic	THF	60	60
2115	Yes	0.3	Used as received	0.5cmx1cm	Ultrasonic	THF/Hexan	60	60
2121	Yes	0.5	Used as received	0,5 to 1 cm²	Ultrasonic	THF / Hexane	60	60
2137	Yes	0.5	Further Cut		Ultrasonic	THF	60	40
2258	Yes	0.2988	Used as received	5mm*5mm	Ultrasonic	Tetrahydrofuran	120	40
2265	Yes	0.3	Further Cut	2-4mm	Ultrasonic	10ml THF for extraction, then precipitation with 20ml n-hexane	60	60
2267	---		---		---			
2297	Yes	0.5	Used as received	0.2	Ultrasonic	THF+Hexane	60	40
2310	Yes	0.1	Further Cut	<3mm	Ultrasonic	THF/HEXANE	60	60
2311	Yes	0.1	Further Cut	<2mm	Ultrasonic	THF & n-Hexane	60	60
2313	Yes	0.3	Further Cut	3mm	Ultrasonic	THF and n-hexane	60	60
2314	Yes	0.5	Further Cut	2mm*2mm	Ultrasonic	THF/N-Hexane	60	60
2330	Yes	0.50	Further Cut	less than 2 mm x 2 mm	Ultrasonic	THF : n-Hexane ratio 1:2	60	60
2347	Yes	0.3	Further Cut	2mm*2mm*2 mm	Mechanical Shaking	THF		
2350	Yes	0.5	Further Grinded	2mm X 2mm X 2 mm	Ultrasonic	THF+ACN	2 h	60
2352	Yes	0.3	Further Cut	<5 x <5mm	Ultrasonic	Tetrahydrofuran and Hexane	60	60
2357	---		---		---			
2358	Yes	0.3	Used as received	3mm x 3mm	Ultrasonic	THF	60	60
2363	Yes	about 1.2	Further Grinded	0.5-1.5mm	Ultrasonic	THF	60±5	60±2
2365	Yes	0.5	Used as received	5mm*5mm	Ultrasonic	Tetrahydrofuran:n-hexane(1:1)	60	60
2366	No	0.3	Used as received	as received	Ultrasonic	V(THF:n-hexane)=1:2	60	60±5
2370	Yes	0.3	Used as received	less than 5 mm	Ultrasonic	THF : Acetonitrile = 10 : 20 mL	60	60
2375	Yes	0,5	Further Cut	3 mm * 3 mm	Ultrasonic	THF	60	60
2378	Yes	0.3	Used as received	5mm*5mm	Ultrasonic	THF:Hexane=1:2	60	60
2379	Yes	0.5	Further Cut	5*5 mm.	Ultrasonic	THF : Hexane (1 : 2)	60	60
2380	Yes	0.5	Used as received	(3-5)mm x (3-5)mm	Ultrasonic	Tetrahydrofuran (THF)	60	60
2381	Yes	0.5	Further Cut		Ultrasonic	THF&n-hexane.	60	60
2382	Yes	0.1	Further Cut	5mm*5mm	Ultrasonic	Tetrahydrofuram 5ml + Hexane 5ml	60	60
2386	Yes	0.3	Further Cut	3x3mm	Ultrasonic	Tetrahydrofuran	60	60
2390	Yes	0.1	Further Cut		Ultrasonic	THF, n-Hexane	60	60
2426	Yes	#20540=0.0513 #20541=0.0525	Further Cut	3x3 mm	Ultrasonic	THF than n-Hexane for precipitation	30	25

Lab	ISO/IEC 17025 accr.	Sample intake used (grams)	Sample preparation	Final estimated particle size	Release/ extraction technique	Extraction solvent	Extrac. time (min)	Extrac. temp. (°C)
2449	---		---		---			
2452	---		---		---			
2453	---		---		---			
2462	---		---		---			
2476	---		---		---			
2549	Yes	0.5	Used as received	Used as received	Ultrasonic	THF:n Hexane (1:2)	60	60
2561	---		---		---			
2569	Yes	0.3	Used as received		Ultrasonic	THF +ACN	60	60
2573	Yes	0.5	Used as received	Used as received	Ultrasonic	THF/Hexane	30	40
2582	Yes	#20540=0.6080 #20541=0.6083	Further Cut	Less than 5 mm in the greatest dimension	Ultrasonic	THF	1h±5min	60 ± 5
2590	Yes	0.3	Used as received		Ultrasonic	THF	60	60
2591	Yes	0.2	Further Cut		Ultrasonic	THF/n-Hexane		
2689	No	0.3	Further Cut	3mm*3mm*3 mm	Ultrasonic	THF:ACN = 1:2	60	40
2703	Yes	#20540=0.5007 #20541=0.5067	Further Cut	0.5cm2	Ultrasonic	THF/Hexane	150	63
2713	No	0.3	Further Cut	2mm x 2 mm	Ultrasonic	Tetrahydrofurane / Acetonitrile (1/2)	60	60
2743	Yes	0.5	Further Cut	less than 5 mm	Ultrasonic	THF (tetrahydrofurane) labelled with the internal standard	60	60
2789	Yes	0.3	Used as received		Ultrasonic	Tetrahydrofurane	60	60
2793	---		---		---			
2798	Yes	0.3	Used as received	5mm*5mm	Ultrasonic	Hexane:THF=2:1	60	60
2804	No	0.3	Further Cut	5mm x 5mm	Ultrasonic	THF/ACN	60	60
2805	Yes	0.5	Further Cut	(2x2)mm	Ultrasonic	THF : Hexan (1:1)	60	70
2812	Yes	0.3	Used as received	5x5	Ultrasonic	THF/Hexane	60	60
2818	Yes	0.2	Used as received	<5*5mm	Ultrasonic	Tetrahydrofuran	60	60
2826	No	0.05	Further Cut	2mm x 2mm	Ultrasonic	THF	180	Room temperature (25°C)
2827	Yes	0.3	Further Cut	5mm X 5mm	Ultrasonic	THF : n-HEXANE (1:2)	60	60
2867	Yes	0.3g	Further Cut	5mmx5mm	Ultrasonic	Tetrahydrofuran	60	60
2881	Yes	Brown=0.6-0.8g Green/blue= 0.5-0.7g	Further Cut	0,5 mm	Ultrasonic	acetonitrile	60	60
2912	---		---		---			
3116	Yes	0.6	Used as received	5mm x 5mm	Ultrasonic	Tetrahydrofuran and Acetonitrile	60	60
3154	Yes		Used as received		Ultrasonic	Toluene	60	60
3166	Yes	0.5	Used as received		Mechanical Shaking	DCM		Ambient
3172	Yes	1	Further Cut	2x2mm	Ultrasonic	THF-ACN 1:2	60	25
3176	Yes	0.5	Used as received		Ultrasonic	THF / ACN	30	room temperature
3197	Yes	0.3	Further Cut	<5 mm	Ultrasonic	THF/N-HEXANE	60	60
3200	Yes	0.05	Used as received		Ultrasonic	5ml THF + 5ml N-hexane	1h	Normal atmospheric temperature
3210	Yes	1	Used as received	< 4mm	Ultrasonic	Hexane/Acetone	60	50

APPENDIX 4

Number of participating laboratories per country

2 labs in BANGLADESH
1 lab in BRAZIL
1 lab in BULGARIA
1 lab in CAMBODIA
1 lab in EGYPT
3 labs in FRANCE
4 labs in GERMANY
1 lab in GUATEMALA
4 labs in HONG KONG
7 labs in INDIA
1 lab in INDONESIA
5 labs in ITALY
1 lab in MOROCCO
16 labs in P.R. of CHINA
3 labs in PAKISTAN
1 lab in POLAND
1 lab in PORTUGAL
2 labs in SOUTH KOREA
3 labs in SPAIN
1 lab in SRI LANKA
1 lab in TAIWAN R.O.C.
1 lab in THAILAND
1 lab in THE NETHERLANDS
2 labs in TUNISIA
5 labs in TURKEY
1 lab in U.S.A.
2 labs in UNITED KINGDOM
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

Literature

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