Results of Proficiency Test Phthalates in Leather March 2021

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### **1** INTRODUCTION

Phthalates is a restricted substance in a lot of applications. In the EU Phthalates are restricted in polymers by Regulation EC 1907/2006 Annex XVII with a limit of 0.1% M/M. Furthermore, some Ecolabel organizations have restrictions for the use of Phthalates in consumer items like Textile and Leather. The Oekotex® for Leather label has a limit for Phthalates of 0.05% M/M and the Bluesign® has a limit of 50 mg/kg.

Since 2017 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of Phthalates in Leather every year. During the annual proficiency testing program of 2020/2021 it was decided to continue the proficiency test for the analysis of Phthalates in Leather.

In this interlaboratory study 50 laboratories in 23 countries registered for participation. See appendix 4 for the number of participants 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 analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send two different leather samples of 3 grams each labelled #21515 and #21516. The samples were positive on some Phthalates. The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

### 2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/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 black grinded leather was selected which was made positive for Di-(2-ethylhexyl) phthalate (DEHP) and Di-iso-nonyl phthalate (DINP) by iis. After homogenization the batch was divided over 80 subsamples in small bags of approximately 3 grams each and labelled #21515.

The homogeneity of the subsamples was checked by determination of DEHP and DINP in accordance with test method ISO/TS16181 on 7 stratified randomly selected subsamples.

	DEHP in %M/M	DINP in %M/M
Sample #21515-1	0.0963	0.0833
Sample #21515-2	0.1041	0.0931
Sample #21515-3	0.1021	0.0925
Sample #21515-4	0.0911	0.0835
Sample #21515-5	0.0945	0.0916
Sample #21515-6	0.0989	0.0889
Sample #21515-7	0.0970	0.0855

Table 1: homogeneity test results of the subsamples #21515

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

	DEHP in %M/M	DINP in %M/M
r (observed)	0.0124	0.0119
reference method	iis memo 1701*)	iis memo 1701*)
0.3 x R (reference method)	0.0131	0.0119

Table 2: evaluation of repeatabilities of the subsamples #21515

\*) see literature 15

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibility of the reference method. Therefore, homogeneity of the subsamples was assumed.

A second batch of a red grinded leather was selected which was made positive for Benzyl butyl phthalate (BBP) and Dibutyl phthalate (DBP) by iis. After homogenization the batch was divided over 80 subsamples in small bags of approximately 3 grams each and labelled #21516.

The homogeneity of the subsamples was checked by determination of BBP and DBP in accordance with test method ISO/TS16181 on 8 stratified randomly selected subsamples.

	BBP in %M/M	DBP in %M/M
Sample #21516-1	0.1553	0.1751
Sample #21516-2	0.1542	0.1765
Sample #21516-3	0.1530	0.1765
Sample #21516-4	0.1660	0.1818
Sample #21516-5	0.1506	0.1562
Sample #21516-6	0.1480	0.1629
Sample #21516-7	0.1558	0.1753
Sample #21516-8	0.1498	0.1649

Table 3: homogeneity test results of the subsamples #21516

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

	BBP in %M/M	DBP in %M/M		
r (observed)	0.0155	0.0245		
reference method	iis memo 1701*)	iis memo 1701*)		
0.3 x R (reference method)	0.0207	0.0230		

Table 4: evaluation of repeatabilities of the subsamples #21516

\*) see literature 15

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibility of the reference method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample labelled #21515 and one sample labelled #21516 was sent on January 27, 2021.

#### 2.5 ANALYZES

The participants were requested to determine on both samples #21515 and #21516, sixteen individual Phthalates (see appendices 1 and 2) and eventually other Phthalates when identified. It was also requested to report if the laboratory was accredited for the requested components and some method details were asked.

Furthermore, to ensure the homogeneity it was requested to not use less than 0.5 gram per determination. And not to dry or age the sample, nor determine volatile matter.

It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the test results but report as much significant figures as possible. It was also requested not to report 'less than' results which are above the detection limit, because such results cannot be used for meaningful statistical evaluation.

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods 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 results of the participants were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per sample and determination in appendices 1 and 2 of this report. The laboratories are presented by the 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 the data analysis and the original results are placed under 'Remarks' in the result tables in appendices 1 or 2. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

### 3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<...' or '>...' were not used in the statistical evaluation.

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

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test 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 analysis 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 standard. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. The Kernel Density Graph 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 in this interlaboratory study. The target standard deviation was calculated from the target reproducibility by division with 2.8. In case no literature reproducibility was available, other target values are used, like an estimated reproducibility based on former iis proficiency tests.

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

The z-scores were calculated according to:

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

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

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

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

### 4 EVALUATION

During the execution of this proficiency test some problems occurred with the dispatch of the samples due to the COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with another week. Four participants did not report any test results and three other participants reported the test results after the extended final reporting date. Not all laboratories were able to report all components requested.

Finally, 46 laboratories reported 256 numerical test results. Observed were 12 outlying test results, which is 4.7%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

Not all original data sets proved to have a normal Gaussian distribution. These are referred to as "not OK" or "suspect". The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

### 4.1 EVALUATION PER SAMPLE AND PER COMPONENT

In this section the reported test results are discussed per sample and per component. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the reported test results in appendix 1. The abbreviations, used in these tables, are explained in appendix 5.

For many years iis organizes PTs on Phthalates in Polymers. In 2017 it was decided for the Phthalates in polymers PT to use the iis PT data to estimate a more realistic target

reproducibility (see iis memo 1701, lit. 15). The target reproducibility was estimated as the relative standard deviation (16%) of the mean multiplied by 2.8.

Test method ISO/TS16181 provides a variety of precision data and therefore it was decided to continue to use the estimated iis target reproducibility from the polymers PT also for the Leather PT. It is observed that the previous iis Leather PT data is in line with the estimated target reproducibility from PTs on Phthalates in Polymers, see table 8 for the observed relative standard deviations over the years.

Please note that the target reproducibility from iis memo 1701 has also been used for the textile PT on Phthalates.

### Sample #21515

- <u>DEHP:</u> The determination may be problematic at the level of 0.08 %M/M. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the reproducibility derived from iis memo 1701.
- <u>DBP:</u> The determination may be problematic at the level of 0.01 %M/M. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the reproducibility derived from iis memo 1701.
- <u>DIDP:</u> The determination may be problematic at the level of 0.02 %M/M. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the reproducibility derived from iis memo 1701.
- <u>DINP:</u> The determination may be problematic at the level of 0.08 %M/M. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the reproducibility derived from iis memo 1701.

For all other Phthalates the group of participants agreed on a concentration close to or below than 0.005 %M/M. Therefore, these Phthalates were not further evaluated. The reported test results are given in appendix 2.

### Sample #21516

- <u>BBP:</u> The determination was not problematic at the level of 0.16 %M/M. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the reproducibility derived from the iis memo 1701.
- <u>DBP:</u> The determination was not problematic at the level of 0.12 %M/M. One statistical outlier was observed and one other test result was excluded. The calculated reproducibility after rejection of the suspect data is in agreement with the reproducibility derived from the iis memo 1701.

For all other Phthalates the group of participants agreed on a concentration close to or below than 0.005 %M/M. Therefore, these Phthalates were not further evaluated. The reported test results are given in appendix 2.

### 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Component	unit	n	average	2.8 * sd	R(target)
DEHP	%M/M	44	0.082	0.043	0.037
DBP	%M/M	38	0.010	0.006	0.004
DIDP	%M/M	32	0.019	0.020	0.008
DINP	%M/M	41	0.084	0.043	0.038

 Table 5: reproducibilities of tests for sample #21515

Component	unit	n	average	2.8 * sd	R(target)
BBP	%M/M	44	0.157	0.076	0.070
DBP	%M/M	44	0.116	0.054	0.052

Table 6: reproducibilities of tests for sample #21516

Without further statistical calculations, it can be concluded that the total group of participating laboratories may have difficulties with the analysis of Phthalates in leather, see also the discussion in paragraphs 4.1 and 5.

### 4.3 COMPARISON OF THE PROFICIENCY TEST OF MARCH 2021 WITH PREVIOUS PTs

	March 2021	May 2020	May 2019	April 2018	April 2017
Number of reporting laboratories	46	42	54	66	41
Number of test results	256	180	224	123	127
Number of statistical outliers	12	5	10	2	9
Percentage of statistical outliers	4.7%	2.8%	4.5%	1.6%	7.1%

Table 7: comparison with previous proficiency tests

The performance of the determinations of the proficiency test was compared, expressed as relative standard deviation (RSD) of the PTs over the years and to the target method, see below table.

Component	March 2021	May 2020	May 2019	April 2018	April 2017	iis memo 1701
BBP	17%	22%	n.e.	16%	13%	16%
DEHP	19%	25%	n.e.	n.e.	n.e.	16%
DBP	17-21%	n.e.	n.e.	n.e.	18%	16%
DIDP	38%	n.e.	21%	n.e.	n.e.	16%
DINP	18%	n.e.	29%	n.e.	n.e.	16%
DCHP	n.e.	16%	n.e.	21%	n.e.	16%
DEP	n.e.	23-27%	n.e.	n.e.	n.e.	16%
DMP	n.e.	n.e.	33-46%	n.e.	n.e.	16%
DNHP	n.e.	n.e.	14%	n.e.	n.e.	16%
DIBP	n.e.	n.e.	n.e.	n.e.	16%	16%

 Table 8: development of uncertainties over the years

The uncertainty (RSD) of the PT is in line with previous PTS and except for DIDP in line with the target method, see also the discussion in paragraph 5.

#### 4.4 EVALUATION OF THE ANALYTICAL DETAILS

For this PT some analytical details were requested. The answers are given in appendix 3. Based on the answers given by the participants the following can be summarized: - About 90% of the reporting participants mentioned that they are accredited for the determination of Phthalates in Leather.

- About 58% of the reporting participants used a test portion between 0.5 and 1 grams. About 30% used less sample material and about 12% used a sample intake of 2 - 3 grams.

- About 88% of the reporting participants used an extraction time of 60 minutes.

- About 90% of the reporting participants used an extraction temperature of 50°C or 60°C.

- About 38% of the reporting laboratories used Hexane/Acetone as solvent mixture to release the Phthalates. Another part of the group used THF/Hexane (38%), THF (12%) or other mixtures such as THF/Acetonitrile or Toluene as solvent (8%).

Looking at the analytical details, it may be remarkable that several participants used a sample intake of less than 0.5 grams. This deviates with the instruction "Please note, to ensure the homogeneity, do not use less than 0.5 gram per determination" in the accompanied letter of instructions. Method ISO/TS16181:11 describes a sample intake of 2 grams. Test method CPSC-CH-C1001-09.3 mentions an intake of only 0.05 gram. However, this test method describes also that 10 mL of THF can be added for every 0.1 gram of extra sample intake.

Looking at the statistical evaluation of the components of both samples, the group is almost within the target reproducibility for these components. Therefore, the use of different solvent mixtures to release the Phthalates does not have a significant effect on the reproducibility nor on the released Phthalates.

### 5 DISCUSSION

During the PT an email was received about the quantification of DIDP in relation to the quantification of DINP. DINP can be quantified by two calibration standards, both CAS numbers are mentioned on the report form of the PT. The standard containing DINP of CAS number 28553-12-0 contains also DIDP. The confusion caused by this standard may have contributed to the larger observed reproducibility in this PT for DIDP. Therefore, iis advise to discuss this subject with the analytical working group on the analysis of DINP and DIDP.

In this proficiency test for the determination of Phthalates in Leather, it was noticed that the majority of the participants were able to detect the Phthalates present in sample #21515 and sample #21516.

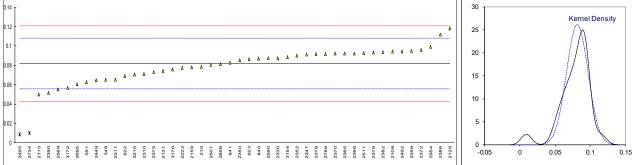
### 6 CONCLUSION

Although it can be concluded that most of the participants have no problem with the determination on Phthalates in Leather in this PT, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

#### **APPENDIX 1**

Determination of DEHP - Di-(2-ethylhexyl) phthalate on sample #21515; results in %M/M

		Di-(2-ethyll		late on	sample #21515; results in %M/M
lab	method	value	mark	z(targ)	remarks
210	CPSC-CH-C1001-09.4	0.078281		-0.26	
348	CPSC-CH-C1001-09.4	0.06492		-1.28	
551	In house	0.06237		-1.47	
622	ISO/TS 16181	0.0689		-0.97	
623 840	ISO14389	0.0865 0.0869		0.37 0.40	
	In house	0.08248		0.40	
841 2108	In house	0.094		0.07	
2108	ISO14389 ISO14389	0.05		-2.42	
2129	ISO14389	0.118		2.78	
2131	In house	0.074		-0.58	
2159	ISO/TS 16181	0.0778		-0.29	
2223	In house	0.07743		-0.32	
2310	ISO/TS 16181	0.071		-0.81	
2311	ISO14389	0.0652		-1.26	
2330	ISO/TS 16181	0.0875		0.45	
2347	ISO/TS 16181	0.0910		0.72	
2350	CPSC-CH-C1001-09.4	0.0853		0.28	
2352	ISO/TS 16181	0.0901		0.65	
2358	ISO/TS 16181	0.09487		1.01	
2365	ISO/TS 16181	0.0922		0.81	
2366	ISO14389	0.0923		0.82	
2370	CNS15138-1	0.0920	0	0.79	
2372	ISO14389	0.09597	С	1.10	first reported: 959.7 %M/M
2375	ISO/TS 16181	0.073		-0.66	
2378	ISO/TS 16181	0.0915		0.76	
2379	ISO/TS 16181	0.0930		0.87	
2382 2386	ISO14389 ISO/TS 16181	0.0936 0.1119		0.92 2.32	
2380	ISO14389	0.0513		-2.32	
2390	ISO/TS 16181	0.0648		-2.32	
2455				-1.25	
2492	In house	0.0943		0.97	
2495	ISO14389	0.0085	R(0.01)	-5.60	
2501	ISO/TS 16181	0.0806		-0.08	
2511	ISO/TS 16181	0.0927		0.85	
2569	ISO/TS 16181	0.055		-2.04	
2582					
2590	ISO/TS 16181	0.0874		0.44	
2695	ISO/TS 16181	0.06026		-1.64	
2734	ISO/TS 16181	0.010	R(0.01)	-5.48	
2789	CPSC-CH-C1001-09.4	0.0915		0.76	
2806	10.04 4000				
2858	ISO14389	0.081		-0.05	
2945	In house			 1 22	
2954	In house	0.099		1.33	
3154 3172	ISO16181-1 Draft ISO8124-6	0.0884 0.05665	С	0.52 -1.91	first reported: <0.005
3172	ISO/TS 16181	0.05665	C	-0.43	first reported: 762.66 %M/M
3210	In house	0.0707	0	-0.43	
0210		0.07.07		5.04	
	normality	OK			
	n	44			
	outliers	2			
	mean (n)	0.08163			
	st.dev. (n)	0.015261	RSD = 19%		
	R(calc.)	0.04273			
	st.dev.(iis memo 1701)	0.013060			
	R(iis memo 1701)	0.03657			
<sup>0.14</sup> T					30
					Kernel Density



# Determination of DBP - Dibutyl phthalate on sample #21515; results in %M/M

lab	method	value	mark	z(targ)	remarks
210	CPSC-CH-C1001-09.4	0.010823		0.82	
348	CPSC-CH-C1001-09.4	0.00804		-1.00	
551	In house	0.00877		-0.52	
622	ISO/TS 16181	0.0105		0.61	
623 840	ISO14389	0.0150		3.55	
840 841	In house In house	0.0097 0.0142		0.09 3.03	
2108	ISO14389	0.0142		0.28	
2100	ISO14389	0.006		-2.33	
2129					
2131	In house	not detected			
2159	ISO/TS 16181	0.0084		-0.76	
2223	In house	0.00555		-2.62	
2310	ISO/TS 16181	0.011		0.94	
2311 2330	ISO14389 ISO/TS 16181	0.0113 0.0041	R(0.01)	1.13 -3.57	
2347	ISO/TS 16181	0.0095	1((0.01)	-0.04	
2350					
2352	ISO/TS 16181	0.0098		0.15	
2358	ISO/TS 16181	0.009857		0.19	
2365	ISO/TS 16181	0.0096		0.02	
2366	ISO14389	0.0096		0.02	
2370 2372	CNS15138-1 ISO14389	0.00900 0.009223	С	-0.37 -0.23	first reported: 92.23 %M/M
2372	ISO/TS 16181	0.009223	C	-0.23 0.28	list reported. 92.25 %M/M
2378	ISO/TS 16181	0.0097		0.09	
2379	ISO/TS 16181	0.0111		1.00	
2382	ISO14389	0.0096		0.02	
2386	ISO/TS 16181	0.0091		-0.31	
2390					
2449	ISO/TS 16181	0.0136		2.63	
2455 2492	In house	 0.0092		-0.24	
2492 2495	ISO14389	0.0924	R(0.01)	-0.24 54.11	
2501	ISO/TS 16181	0.0100	1((0.01)	0.28	
2511	ISO/TS 16181	0.0083		-0.83	
2569	ISO/TS 16181	0.012		1.59	
2582					
2590	ISO/TS 16181	0.00681		-1.80	
2695	ISO/TS 16181	0.00753		-1.33	
2734	ISO/TS 16181	0.017	R(0.01)	4.85	
2789 2806	CPSC-CH-C1001-09.4	0.0096		0.02	
2858	ISO14389	0.010		0.28	
2945					
2954	In house	not detected			
3154	ISO16181-1 Draft	0.00809		-0.97	
3172	ISO8124-6	0.0063		-2.13	
3176	ISO/TS 16181	0.007	С	-1.68	first reported: 67.2 %M/M
3210	In house	0.0098		0.15	
	normality	suspect			
	n	38			
	outliers	3			
	mean (n)	0.00957			
	st.dev. (n)	0.002019	RSD = 21%		
	R(calc.)	0.00565			
	st.dev.(iis memo 1701)	0.001531			
	R(iis memo 1701)	0.00429			
0.02					300 Kernel Density
0.018 -					x 250
0.016 -					A       /\
0.014					200 -
0.01 -				·	
0.008 -					
0.006 -	<u> </u>				
0.004 - X					
0.002 -					50 -
0 0 0	ы N О Б Б В 4 - Л - О ~		0 & N 0 * * *	) <del>~</del> ∞ ~	
2330	2115 2172 2590 2595 348 2695 2695 348 2511 2511 2511 2513 2513 2513 2513 2513	2492 2372 2347 2365 2365 2365 2365 2382 2382 2382 2382 2382	840 2378 2352 3210 2358 2358 2358 2108 2108	2501 2858 622 622	

# Determination of DIDP - Di-iso-decyl phthalate on sample #21515; results in %M/M

lab	method	value	mark	z(targ)	remarks
210					
348 551					
622	ISO/TS 16181	0.0153		-1.16	
623	ISO14389	0.0180		-0.26	
840	In house	0.0198		0.34	
841 2108	In house ISO14389	0.01553 0.02		-1.08 0.40	
2100	ISO14389	0.011		-2.59	
2129					
2131	In house	not detected			
2159 2223	ISO/TS 16181 In house	0.0076 0.0145	С	-3.72 -1.43	first reported: <0.01
2310	ISO/TS 16181	0.0143	C	-2.92	list reported. <0.01
2311	ISO14389	0.0108		-2.66	
2330	ISO/TS 16181	0.0323		4.49	
2347 2350	ISO/TS 16181	0.0260		2.40	
2350	ISO/TS 16181	0.0260		2.40	
2358	ISO/TS 16181	n.d.			
2365	ISO/TS 16181	0.0244		1.87	
2366	ISO14389	0.0259		2.37	
2370 2372	CNS15138-1 ISO14389	0.0202 0.02033	С	0.47 0.51	first reported: 203.3 %M/M
2375	ISO/TS 16181	0.014	0	-1.59	
2378	ISO/TS 16181	0.0260		2.40	
2379	ISO/TS 16181	0.0367		5.96	
2382 2386	ISO14389 ISO/TS 16181	0.0257		2.30 2.13	
2300	ISO14389	0.0252 0.0133		-1.83	
2449					
2455					
2492 2495	In house ISO14389	not detected <0.001		 <-5.92	possibly a false negative test result?
2501					
2511 2569	ISO/TS 16181 ISO/TS 16181	0.0197 0.0104		0.30 -2.79	
2582					
2590	ISO/TS 16181	0.0244		1.87	
2695	ISO/TS 16181	0.01370		-1.69	
2734 2789	ISO/TS 16181 CPSC-CH-C1001-09.4	0.141 0.0162	R(0.01)	40.65 -0.86	
2806	01 00 011 01001 00.4				
2858 2945	ISO14389	0.022		1.07	
2954	In house	not applicable			
3154 3172	ISO8124-6	 0.0119		-2.29	
3176	ISO/TS 16181	0.010	С	-2.92	first reported: 99.6 %M/M
3210	In house	0.0144		-1.46	
	normality	OK			
	n outliers	32 1			
	mean (n)	0.01879			
	st.dev. (n)	0.007068	RSD = 38	3%	
	R(calc.)	0.01979			
	st.dev.(iis memo 1701) R(iis memo 1701)	0.003006 0.00842			
		5.000TE			
0.05 T					60
0.045 +					50 - Kernel Density
0.04 +					▲   \ \
0.035 - 0.03 -					۸ 40 -
0.025				۵ ۸	30 -
0.02 -		. 4	<u>م م م</u>	۸.	
0.015 +	<u>۸ ۸ ۸ ۸</u>	۵ ۵ ۵ <sup>۵</sup>			20 -
0.01	<u>A</u> <u>A</u>				
0	76 69 90 95 75 75 75 75	2223 622 789 623 623	72 70 88	2858 2365 2365 2365 2365	
2159 2310	3176 2569 2311 2115 2390 2390 2330 2337 2337 2337 2337 2337 2337 233	2223 622 841 2788 623 2511 2511	840 2108 2370 2372	2858 2365 2590 2386 2386 2386	

# Determination of DINP - Di-iso-nonyl phthalate on sample #21515; results in %M/M

lab	method	value	mark	z(targ)	remarks	
210	CPSC-CH-C1001-09.4	0.080528		-0.25		
348 551	CPSC-CH-C1001-09.4 In house	0.06788 0.05748		-1.20 -1.97		
622	ISO/TS 16181	0.0476		-2.71		
623	ISO14389	0.0970		0.97		
840	In house	0.0892		0.39		
841 2108	In house ISO14389	0.0884 0.075		0.33 -0.67		
2115	ISO14389	0.067		-1.26		
2129	ISO14389	0.143	R(0.05)	4.40		
2131 2159	In house ISO/TS 16181	not detected 0.0819		-0.15		
2223	In house	0.07834		-0.13		
2310	ISO/TS 16181	0.075		-0.67		
2311	ISO14389	0.0752		-0.65		
2330 2347	ISO/TS 16181 ISO/TS 16181	0.0829 0.0863		-0.08 0.18		
2350	CPSC-CH-C1001-09.4	0.0827		-0.09		
2352	ISO/TS 16181	0.0872		0.24		
2358 2365	ISO/TS 16181	0.08858		0.34 0.21		
2365	ISO/TS 16181 ISO14389	0.0868 0.0867		0.21		
2370	CNS15138-1	0.0817		-0.17		
2372	ISO14389	0.07953	С	-0.33	first reported: 795.3	%M/M
2375 2378	ISO/TS 16181 ISO/TS 16181	0.082 0.0873		-0.14 0.25		
2378	ISO/TS 16181	0.1248		3.04		
2382	ISO14389	0.0867		0.20		
2386	ISO/TS 16181	0.1140		2.24		
2390 2449	ISO14389 ISO/TS 16181	0.0429 0.0562	R(0.05)	-3.06 -2.07		
2455	130/13 10101			-2.07		
2492	In house	0.0898		0.44		
2495	ISO14389	0.0782		-0.43		
2501 2511	ISO/TS 16181 ISO/TS 16181	0.1080 0.1025		1.79 1.38		
2569	ISO/TS 16181	0.074		-0.74		
2582						
2590 2695	ISO/TS 16181 ISO/TS 16181	0.0917 0.06349		0.58 -1.52		
2095	ISO/TS 16181	0.493	R(0.01)	30.45		
2789	CPSC-CH-C1001-09.4	0.0918		0.58		
2806	10.01.1000					
2858 2945	ISO14389	0.101		1.27 		
2954	In house	not applicable				
3154 3172	ISO16181-1 Draft ISO8124-6	0.1016 0.1024		1.31 1.37		
3172	ISO/TS 16181	0.07	С	-1.04	first reported: 696 %	M/M
3210	In house	0.0734	-	-0.79		
	normality	OK				
	normality n	OK 41				
	outliers	3				
	mean (n)	0.08395				
	st.dev. (n) R(calc.)	0.015323 0.04290	RSD = 18	3%		
	st.dev.(iis memo 1701)	0.04290				
	R(iis memo 1701)	0.03761				
<sup>0.16</sup> T						35
0.14 -					×	30 - Kernel Density
0.12 -					۵۵	25 -
0.1					<u>۸</u> ۸ ۸ ۸ ۸	20 -
0.08				<u></u>		20 -
0.06						15 -
0.04 <b>x</b>						10 -
0.02 -						5-
0						
2390 622 2449	2695 2695 2115 2115 348 348 3210 3210 2310 2108 2310 2310 2310 2311 2310 2311 2310	2375 210 2159 2159 2159 2375 2375 2375 2330 23360 23360 23360 23360 23360 23360 23360 23360 23360 23360 23360 23370 23370 23370 23370 237700 2377000 2377000 23770000000000	2382 2365 2352 2378 2378 841	2356 840 2492 2590 2590 2789	623 2858 3154 3172 2511 2511 2501 2501 2379 2379 2379 2379 2379	0 0.05 0.1 0.15 0.2

# Determination of BBP – Benzyl butyl phthalate on sample #21516; results in %M/M

lab	method	value	mark	z(targ)	remarks
210	CPSC-CH-C1001-09.4	0.212562		2.21	
348	CPSC-CH-C1001-09.4	0.13121		-1.03	
551 622	In house ISO/TS 16181	0.11296 0.1626		-1.75 0.22	
622	ISO14389	0.1626	С	0.22	first reported: 0.2570
840	In house	0.200	C	-0.01	liist reported. 0.2370
841	In house	0.1557		-0.05	
2108	ISO14389	0.144		-0.52	
2115	ISO14389	0.11		-1.87	
2129	ISO14389	0.170		0.52	
2131		0.137		-0.80	
2159 2223	ISO/TS 16181	0.1116 0.1790		-1.81 0.87	
2223	In house ISO/TS 16181	0.1790		-0.65	
2311	ISO14389	0.1400		-1.57	
2330	ISO/TS 16181	0.1751		0.72	
2347	GB/T32440	0.1750		0.71	
2350	CPSC-CH-C1001-09.4	0.1603		0.13	
2352	ISO/TS 16181	0.1733		0.65	
2358 2365	ISO/TS 16181	0.1906 0.1794		1.34 0.89	
2365	ISO/TS 16181 ISO14389	0.1794 0.1783		0.89	
2370	CNS15138-1	0.1703		0.00	
2372	ISO14389	0.15379	С	-0.13	first reported: 1537.9 %M/M
2375	ISO/TS 16181	0.123		-1.35	
2378	ISO/TS 16181	0.1755		0.73	
2379	ISO/TS 16181	0.1447		-0.49	
2382	ISO14389	0.1760		0.75	
2386 2390	ISO/TS 16181 ISO14389	0.1955 0.165	С	1.53 0.32	first reported: 0.0779
2390	ISO/TS 16181	0.105	C	-1.61	lifst reported. 0.0779
2455					
2492	In house	0.2003		1.72	
2495	ISO14389	0.1440		-0.52	
2501	ISO/TS 16181	0.1681		0.44	
2511	ISO/TS 16181	0.179		0.87	
2569 2582		0.1165 		-1.61	
2592	ISO/TS 16181	0.1510		-0.24	
2695	ISO/TS 16181	0.04547	R(0.01)	-4.44	
2734	ISO/TS 16181	0.037	R(0.01)	-4.78	
2789	CPSC-CH-C1001-09.4	0.1237	· · · ·	-1.33	
2806					
2858	ISO14389	0.135		-0.88	
2945 2954	In house	 0.198		 1.63	
2954 3154	In house ISO16181-1 Draft	0.198	C	0.36	first reported as DEHP
3172	ISO8124-6	0.1638	0	0.27	
3176	ISO14389	0.134	С	-0.92	first reported: 1335.18 %M/M
3210	In house	0.1492		-0.31	·
		<b></b>			
	normality	OK			
	n outliers	44 2			
	mean (n)	∠ 0.15704			
	st.dev. (n)	0.027024	RSD = 17%	'n	
	R(calc.)	0.07567			
	st.dev.(iis memo 1701)	0.025126			
	R(iis memo 1701)	0.07035			
<sup>0.25</sup> T					16
					A 14 - Kernel Density
0.2					
				<u>م م م</u>	
0.15 -			a a •		
					8 -
0.1					6 -
					4 -
0.05 × ×					
734 0	1159 551 551 311 375 375 375 375 348 348 858 858 858 858 310	210 210 210 372 372 372	840 350 622 172 390	154 501 129 352	
N X N	N - N N N N N N N N N N N N N N N N N N	• • • * * * * * *	ิง ผี > ผี	m a à ă	N N N N N N N N N N N N N N N N N N N
•					

# Determination of DBP - Dibutyl phthalate on sample #21516; results in %M/M

lab	method	value	mark z	z(targ)	remarks
210 348	CPSC-CH-C1001-09.4 CPSC-CH-C1001-09.4	0.150206 0.10486		1.81 -0.62	
540 551	In house	0.10488		-0.02 -1.16	
622	ISO/TS 16181	0.0750		-2.23	
623	ISO14389	0.162	С	2.44	first reported: 0.1865
840	In house	0.0995		-0.91	
841	In house	0.11488		-0.09	
2108	ISO14389	0.112		-0.24	
2115 2129	ISO14389 ISO14389	0.098 0.104		-0.99 -0.67	
2123	10014009	0.093		-1.26	
2159	ISO/TS 16181	0.1011		-0.82	
2223	In house	0.1324		0.85	
2310	ISO/TS 16181	0.125		0.46	
2311	ISO14389	0.1046		-0.64	
2330	ISO/TS 16181	0.1102		-0.34	
2347 2350	GB/T32440 CPSC-CH-C1001-09.4	0.1352 0.1364		1.00 1.07	
2352	ISO/TS 16181	0.1304		1.32	
2358	ISO/TS 16181	0.1269		0.56	
2365	ISO/TS 16181	0.1381		1.16	
2366	ISO14389	0.1368		1.09	
2370	CNS15138-1	0.1100		-0.35	
2372	ISO14389	0.10093	С	-0.83	first reported: 1009.3 %M/M
2375 2378	ISO/TS 16181 ISO/TS 16181	0.117 0.1395		0.03 1.24	
2378	ISO/TS 16181	0.0909		-1.37	
2382	ISO14389	0.1396		1.24	
2386	ISO/TS 16181	0.1598		2.33	
2390	ISO14389	0.092	С	-1.31	first reported: 0.0605
2449	ISO/TS 16181	0.1004		-0.86	
2455	In house				
2492 2495	In house ISO14389	0.1226 0.1126		0.33 -0.21	
2501	ISO/TS 16181	0.1366		1.08	
2511	ISO/TS 16181	0.1088		-0.41	
2569		0.1032		-0.71	
2582					
2590	ISO/TS 16181	0.0997		-0.90	
2695	ISO/TS 16181	0.04695	R(0.05)	-3.73	test result evaluated five out of eiv test results were sufficient
2734 2789	ISO/TS 16181 CPSC-CH-C1001-09.4	0.060 0.1069	ex	-3.03 -0.51	test result excluded, five out of six test results were outliers
2806	01 00-011-01001-09.4			-0.51	
2858	ISO14389	0.110		-0.35	
2945					
2954	In house	0.112		-0.24	
3154	ISO16181-1 Draft	0.1068		-0.52	
3172 3176	ISO8124-6 ISO14389	0.1327 0.109	С	0.87 -0.40	first reported: 1094 69 % M/M
3210	In house	0.109	C	-0.40	first reported: 1084.68 %M/M
0210	Innouse	0.1117		0.07	
	normality	OK			
	n	44			
	outliers	1 (+1ex)			
	mean (n)	0.11647	DCD - 170/		
	st.dev. (n) R(calc.)	0.019446 0.05445	RSD = 17%		
	st.dev.(iis memo 1701)	0.03443			
	R(iis memo 1701)	0.05218			
	· · · /				
<sup>0.2</sup> T					25
0.18 -					Kernel Density
0.16 -					
0.14 -					
0.12			<u> </u>	<u>م م م</u>	<u> </u>
0.1 -					
0.08					
0.06					
0.04 +					5
0.02					
2695 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0	2379 2131 551 2115 2115 840 2449 2449 2159 2158 2158 2158	3154 3154 2789 2789 2778 3176 3176	2858 2330 2108 2954 2954 2495 2495	2375 3210 2492 2310	0         0         0.05         0.1         0.15         0.2         0.25
(N (N			ar ar ar ar ar A		

### **APPENDIX 2**

Summary of other Phthalates in sample #21515: results in %M/M

BBP	= Benzylbutylphthalate
	- Din Octubrith alata

DNOP	= Di-n-Octylphthalate
DCHP	= Dicyclohexylphthalate
DEP	= Diethylphthalate

- DEP = Dietnyiphthalate DMP = Dimethylphthalate DNHP = Di-n Hexylphthalate DIBP = Diisobutylphthalate

Lab	BBP	DNOP	DCHP	DEP	DMP	DNHP	DIBP
210							
348	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
551							
622	0.0009	0.0000	0.0000	0.0000	0.0000	0.0000	0.0006
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	not detected
841	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003
2108							
2100							0.0011
2113							
2123	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2159	< 0.0050	<0,0050	<0.0050	<0,0050	<0,0050	<0,0050	<rl< td=""></rl<>
2223	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
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
2330	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2347	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003
2350							
2352							
2358	n.d.	n.d.	N/A	N/A	N/A	N/A	n.d.
2365	< 0.003	<0.003	< 0.003	< 0.003	< 0.003	< 0.003	<0.003
2366	< 0.004	< 0.004	not determ.	< 0.004	< 0.004	< 0.004	< 0.004
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	not detected
2375							
2378							
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2382	< 0.0030	< 0.0030	< 0.0030	< 0.0030	< 0.0030	< 0.0030	< 0.0030
2386	<0.005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
2390							
2449							
2455							
2492	not detected	not detected					not detected
2495	< 0.001	< 0.001	<0.001	<0.001	<0.001	<0.001	< 0.001
2501							
2511							
2569	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2582							
2590							
2695	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2734	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2789							
2806							
2858	n.d	n.d	n.d	n.d	n.d	n.d	n.d
2945							
2954	not detected	not detected	not applicable	not detected	not detected	not applicable	not applicable
3154							
3172	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
3176							
3210	not detected		not detected	not detected	not detected	not detected	not detected

### Summary of other Phthalates in sample #21515: results in %M/M - continued

- DPHP = Di(2-propylheptyl)phthalate
- DNPP = Di-n-Pentylphthalate
- DUP = Diundecylphthalate
- DPrP = Di-n-Propylphthalate
- DMEP = Di(methoxyethyl)phthalate Other = Other Phthalates

Lab	DPHP	DNPP	DUP	DPrP	DMEP	Other
210						
348		<0.005			<0.005	
551						
622		0.0000			0.0000	
623	not detected	not detected	not detected	not detected	not detected	not detected
840	not detected	not detected	not detected	not detected	not detected	
841	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	
2108						
2115						
2129						
2131	not analysed	not detected	not detected	not detected	not detected	not detected
2159	<0.0050	<rl< td=""><td><rl< td=""><td><rl< td=""><td><rl< td=""><td>Not applicable</td></rl<></td></rl<></td></rl<></td></rl<>	<rl< td=""><td><rl< td=""><td><rl< td=""><td>Not applicable</td></rl<></td></rl<></td></rl<>	<rl< td=""><td><rl< td=""><td>Not applicable</td></rl<></td></rl<>	<rl< td=""><td>Not applicable</td></rl<>	Not applicable
2223	< 0.001	< 0.001		< 0.001	< 0.001	
2310	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2311	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2330	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2347	<0.010	< 0.003	< 0.003	<0.003	<0.003	
2350						
2352						
2358	N/A	N/A	N/A	N/A	N/A	N/A
2365	< 0.003	< 0.003	< 0.003	< 0.003	<0.003	< 0.003
2366	<0.004	< 0.004	<0.004	< 0.004	<0.004	not determined
2370	<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
2375						
2378						
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not tested
2382	<0.0030	< 0.0030	<0.0030	<0.0030	<0.0030	<0.0030
2386	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
2390						
2449						0.1346
2455						
2492						
2495		<0.001		<0.001	<0.001	
2501						
2511						
2569	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	0.1514
2582						
2590						
2695	not detected	not detected	not analyzed	not detected	not detected	not detected
2734	not detected	not detected	not detected	not detected	not detected	not detected
2789						
2806 2858						
	n.d 	n.d	n.d	n.d	n.d	n.d
2945 2954						
2954 3154	not applicable	not applicable	not applicable	not applicable	not applicable	not applicable
3154	< 0.005	< 0.005	< 0.005	< 0.005	 < 0.005	
3172	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	
3210		not detected	not detected	not detected	not detected	
3210		not detected	not delected	not delected	not detected	

#### Summary of other Phthalates in sample #21516: results in %M/M

- DEHP = Bis-2-ethylhexylphthalate
- DIDP = Diisodecylphthalate
- DINP = Diisononylphthalate
- DNOP = Di-n-Octylphthalate
- DCHP = Dicyclohexylphthalate
- DEP = Diethylphthalate

DMP = Dimethylphthalate

DNHP = Di-n-Hexylphthalate

Lab	DEHP	DIDP	DINP	DNOP	DCHP	DEP	DMP	DNHP
210								
348	<0.005	<0.005	<0.005	<0.005	< 0.005	<0.005	< 0.005	<0.005
551								
622	0.0004	0.0000	0.0025	0.0000	0.0000	0.000	0.0000	0.0000
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
841	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	<0.003
2108								
2115								
2129								
2131	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2159	<rl< td=""><td><rl< td=""><td><rl< td=""><td><rl< td=""><td><rl< td=""><td><rl< td=""><td><rl< td=""><td><rl< td=""></rl<></td></rl<></td></rl<></td></rl<></td></rl<></td></rl<></td></rl<></td></rl<>	<rl< td=""><td><rl< td=""><td><rl< td=""><td><rl< td=""><td><rl< td=""><td><rl< td=""><td><rl< td=""></rl<></td></rl<></td></rl<></td></rl<></td></rl<></td></rl<></td></rl<>	<rl< td=""><td><rl< td=""><td><rl< td=""><td><rl< td=""><td><rl< td=""><td><rl< td=""></rl<></td></rl<></td></rl<></td></rl<></td></rl<></td></rl<>	<rl< td=""><td><rl< td=""><td><rl< td=""><td><rl< td=""><td><rl< td=""></rl<></td></rl<></td></rl<></td></rl<></td></rl<>	<rl< td=""><td><rl< td=""><td><rl< td=""><td><rl< td=""></rl<></td></rl<></td></rl<></td></rl<>	<rl< td=""><td><rl< td=""><td><rl< td=""></rl<></td></rl<></td></rl<>	<rl< td=""><td><rl< td=""></rl<></td></rl<>	<rl< td=""></rl<>
2223	<0.001	<0.01	<0.01	<0.001	<0.001	<0.001	<0.001	<0.001
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
2330	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2347	<0.003	<0.010	<0.010	<0.003	<0.003	<0.003	<0.003	<0.003
2350								
2352								
2358	n.d.	n.d.	n.d.	n.d.	N/A	N/A	N/A	N/A
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	not determ.	< 0.004	< 0.004	< 0.004
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
2375 2378								
2378	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2382	< 0.0030	< 0.0030	< 0.0030	< 0.0030	< 0.0030	< 0.0030	< 0.0030	< 0.0030
2386	<0.005	<0,005	<0,005	<0.005	<0.005	<0,005	<0.005	<0.005
2390								
2449								
2455								
2492	not detected	not detected	not detected	not detected				
2495	< 0.001	< 0.001	< 0.001	< 0.001	<0.001	<0.001	<0.001	<0.001
2501								
2511								
2569	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2582								
2590								
2695	not detected	not detected	not detected	not detected	not detected	not detected	not detected	0.02100
2734	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2789								
2806								
2858	n.d	n.d	n.d	n.d	n.d	n.d	n.d	n.d
2945								
2954	not detected	not applicable	not applicable	not detected	not applicable	not detected	not detected	not applicable
3154								
3172	<0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
3176								
3210	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected

#### Summary of other Phthalates in sample #21516: results in %M/M - continued

- DIBP = Diisobutylphthalate
- DPHP = Di(2-propylheptyl)phthalate DNPP = Di-n-Pentylphthalate
- DUP = Diundecylphthalate
- DPrP = Di-n-Propylphthalate
- DMEP = Di(2-methoxyethyl)phthalate
- Other = Other Phthalates

210 348 551	DIBP  <0.005	DPHP	DNPP	DUP	DPrP	DMEP	Other
348 551							
551	<0.005		<0.005			<0.005	
022	0.0001		0.0000			0.0000	
623	not detected	not detected	not detected		not dotostad	not detected	0.0045
				not detected	not detected		
	not detected	not detected	not detected	not detected	not detected	not detected	
841	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003	
2108							
2115							
2129							
2131	not detected	not analysed	not detected	not detected	not detected	not detected	not detected
2159	<rl< td=""><td><rl< td=""><td><rl< td=""><td><rl< td=""><td><rl< td=""><td><rl< td=""><td>Not applicable</td></rl<></td></rl<></td></rl<></td></rl<></td></rl<></td></rl<>	<rl< td=""><td><rl< td=""><td><rl< td=""><td><rl< td=""><td><rl< td=""><td>Not applicable</td></rl<></td></rl<></td></rl<></td></rl<></td></rl<>	<rl< td=""><td><rl< td=""><td><rl< td=""><td><rl< td=""><td>Not applicable</td></rl<></td></rl<></td></rl<></td></rl<>	<rl< td=""><td><rl< td=""><td><rl< td=""><td>Not applicable</td></rl<></td></rl<></td></rl<>	<rl< td=""><td><rl< td=""><td>Not applicable</td></rl<></td></rl<>	<rl< td=""><td>Not applicable</td></rl<>	Not applicable
2223	<0.001	<0.001	<0.001		<0.001	<0.001	
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
2330	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2347	< 0.003	<0.010	< 0.003	< 0.003	< 0.003	< 0.003	
2350							
2352							
	n.d.	N/A	 N/A	 N/A	 N/A	N/A	 N/A
	<0.003	-	-	-	-		
2365		< 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	not determined
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	not detected
2375							
2378							
	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not tested
2382	<0.0030	<0.0030	<0.0030	<0.0030	<0.0030	<0.0030	<0.0030
2386	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
2390							
2449							
2455							
2492	not detected						
2495	< 0.001		<0.001		<0.001	<0.001	
2501							
2511							
2569	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	0.2197
2582							
2590							
2695	not detected	not detected	not detected	not analyzed	not detected	not detected	not detected
2734	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2789							
2806							
2858	n.d	n.d	n.d	n.d	n.d	n.d	n.d
2945							
2954	not applicable	not applicable	not applicable	not applicable	not applicable	not applicable	not applicable
3154							
3172	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	
3176							
3210	not detected		not detected	not detected	not detected	not detected	

# **APPENDIX 3 Analytical details**

lab	ISO/IEC17025 accredited	Sample intake (in grams)	Solvent (mixture) used to release the analytes	Extraction time (in minutes)	Extraction temp. (in °C)
210	Yes				
348	Yes	0.5	THF	180	60±5
551					
622	Yes	1	n-hexane:acetone (80:20)	60	50
623	Yes	0.1	THF Hexane	60	60
840	Yes	0.5	THF-HEXANE	60	60
841	Yes	0.5	Tetrahydrofuran/ n-hexane	60	60
2108	Yes	0,5	THF/Hexane	1h	60
2100	Yes	0.3	THF	60	60
2129	Yes	0,5	THF	1h	60
2120	Yes	0.5	THF:Hexane 1:2	1h	60
2151	Yes	1,0	Hexane/Acetone	1 hour	50
2133	Yes	0.3	first THF, later addition of i-octane	1 hour	60
2223	Yes	0.5	Acetone and Hexane	60	50
2310	Yes	0.3	THE and Hexane	60	60
2311		0.50	n-Hexane:Acetane 80:20	60	50
2330	No				
		1 0.5	25mL THF + ACN	60 2 h	50
2350	Yes				60
2352	Yes	1.0	25mL	60	50
2358	Yes	2	n-hexane / acetone	60	30
2365	Yes	1.0	Acetone: N-hexane=1:1	60	50
2366	Yes	0.3	THF:n-hexane(1:2)	60	60
2370	Yes	0.5	THF : Hexane = 10 : 20 mL	30	room temperature
2372	No	0.5	THE	60	60
2375	Yes	0.1	Hexane : Acetone (4:1)	60	50
2378	Yes	2	40 ml of n-hexane/acetone	60	50
2379	No	0.5	Hexane : Acetone 80 : 20	60	50
2382	No	0.1	Tetrahydrofuran and Hexane	60	60
2386	Yes	0,5	n-Hexane/Acetone (80:20)	60	50
2390	Yes	0.1	Tetra hydro furan (THF) and n-hexane	60	60
2449					
2455					
2492	Yes	0.3	THF:n-Hex (1:2)	60	60
2495	Yes	0.15	THF/Hexane	60	60
2501	Yes	2	hexane acetone	60	50
2511					
2569	Yes	1	Hexane : Acetone	1 hr	50
2582					
2590	Yes	1.25	thf:hex	60	60
2695	Yes	3	HEXANE:ACETONE (80:20)	60	50
2734	Yes	3	n-hexane 80% / acetone 20%	60	50
2789	Yes	0.05	Acetonitrile / Tetrahidrofuran	30	Ambient
2806					
2858	Yes	0.5	THF+n-Hexane	60	60
2945					
2954	Yes	0,1	THF/hexane	120	40
3154	Yes				
3172					
3176	Yes	0,5	N-Hexane / Acetone	60	50
3210	Yes	1	Toluene	60	60

#### **APPENDIX 4**

#### Number of participants per country

1 lab in BANGLADESH 1 lab in BRAZIL 1 lab in CAMBODIA 1 lab in FRANCE 4 labs in GERMANY 2 labs in HONG KONG 3 labs in INDIA 2 labs in INDONESIA 7 labs in ITALY 1 lab in MOROCCO 7 labs in P.R. of CHINA 2 labs in PAKISTAN 1 lab in SERBIA 1 lab in SOUTH KOREA 2 labs in SPAIN 1 lab in SRI LANKA 2 labs in SWITZERLAND 2 labs in TAIWAN 1 lab in THAILAND 1 lab in TUNISIA 3 labs in TURKEY 1 lab in U.S.A. 3 labs in VIETNAM

### **APPENDIX 5**

#### Abbreviations

С	= 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

### Literature

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