

Institute for
Interlaboratory Studies

Results of Proficiency Test Phthalates in Leather/Footwear February 2022

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Phthalates is a restricted substance in a lot of applications. In the EU Phthalates are regulated in polymers by EC 1907/2006 (REACH) with a limit of 0.1%M/M. Furthermore, some Ecolabel organizations have mentioned limits for the use of Phthalates in consumer items like Textile and Leather. Well-known Ecolabelling organizations are OEKO-TEX® and BlueSign®.

Since 2017 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of Phthalates in Leather/Footwear every year. During the annual proficiency testing program 2021/2022 it was decided to continue the proficiency test for the determination of Phthalates in Leather/Footwear.

In this interlaboratory study 49 laboratories in 24 countries registered for participation, see appendix 4 for the number of participants per country. In this report the results of the Phthalates in Leather/Footwear 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 #22505 and #22506. 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

For the first sample a batch of grey leather, positive for Di-iso-butyl phthalate (DIBP) and Di-n-pentyl phthalate (DNPP), was selected. After homogenization 100 small bags were filled with approximately 3 grams each and labelled #22505.

The homogeneity of the subsamples was checked by determination of DIBP and DNPP in accordance with an in-house method on 7 stratified randomly selected subsamples.

	DIBP in %M/M	DNPP in %M/M
sample #22505-1	0.0800	0.0890
sample #22505-2	0.0752	0.0757
sample #22505-3	0.0927	0.0868
sample #22505-4	0.0875	0.0875
sample #22505-5	0.0801	0.0860
sample #22505-6	0.0832	0.0813
sample #22505-7	0.0797	0.0785

Table 1: homogeneity test results of subsamples #22505

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.

	DIBP in %M/M	DNPP in %M/M
r (observed)	0.0163	0.0142
reference method	iis memo 2201	iis memo 2201
0.3 x R (reference method)	0.0146	0.0147

Table 2: evaluation of the repeatabilities of subsamples #22505, see also paragraph 4.1 for more details

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

For the second sample a batch of a brown grinded leather, positive for Benzyl butyl phthalate (BBP) and Di-n-octyl phthalate (DNOP), was selected. After homogenization 80 small bags were filled with approximately 3 grams each and labelled #22506.

The homogeneity of the subsamples was checked by determination of BBP and DNOP in accordance with an in-house method on 10 stratified randomly selected subsamples.

	BBP in %M/M	DNOP in %M/M
sample #22506-1	0.1260	0.1655
sample #22506-2	0.1159	0.1554
sample #22506-3	0.1138	0.1559
sample #22506-4	0.1138	0.1472
sample #22506-5	0.1071	0.1481
sample #22506-6	0.1141	0.1554
sample #22506-7	0.1127	0.1477
sample #22506-8	0.1206	0.1610
sample #22506-9	0.1317	0.1758
sample #22506-10	0.1279	0.1739

Table 3: homogeneity test results of subsamples #22506

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	DNOP in %M/M
r (observed)	0.0220	0.0290
reference method	iis memo 2201	iis memo 2201
0.3 x R (reference method)	0.0209	0.0280

Table 4: evaluation of the repeatabilities of subsamples #22506, see also paragraph 4.1 for more details

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 leather sample labelled #22505 and one leather sample labelled #22506 was sent on February 2, 2022.

2.5 ANALYZES

The participants were requested to determine on samples #22505 and #22506, 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 determined components and some analytical details.

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 evaluations.

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods (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 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, by $G(0.05)$ or $DG(0.05)$ for the Grubbs' test and by $R(0.05)$ for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT, the criterion of ISO13528, paragraph 9.2.1 was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements (derived from e.g. ISO or ASTM test methods), 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 literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, like Horwitz or 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 in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated according to:

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

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

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

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

4 EVALUATION

In this proficiency test no problems were encountered with the dispatch of the samples. Four participants reported test results after the final reporting date and four other participants did not report any test results. Not all laboratories were able to report all components requested. In total 45 laboratories reported 220 numerical test results. Observed were 7 outlying test results, which is 3.2%.

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

Not all 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 original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 5.

Test method ISO16181 is considered to be the official test method for Phthalates in Leather/Footwear. Unfortunately, this test method provides a variety of unclear precision data. Therefore, the target reproducibility used for the evaluation of the test results of Phthalates in Polymers as prescribed in iis memo 1701 was also used for the evaluation of the quality of the test results of Phthalates in Leather/Footwear.

However, with the progress of the iis PTs of Phthalates in Leather/Footwear it was noticed that the relative standard deviations (RSD%) for Phthalates in iis PTs in Polymers is on average somewhat better than in iis PTs in Leather. Therefore, it was decided in 2022 not to use the target reproducibility based on Phthalates in Polymers any longer, but to develop a new target reproducibility based on the iis PTs for Phthalates in PTs in Leather/Footwear with the aim to estimate a more realistic target reproducibility for Phthalates in Leather/Footwear. An iis memo 2201 was made in which the development of the new precision data has been prescribed. The PT data for iis memo 2201 are obtained from 6 iis PTs and 26 different data sets for 12 different Phthalates. It is observed that the relative standard deviation of the subsequent PTs and the individual Phthalates is not dependent on the individual Phthalates and therefore one target reproducibility for all individual Phthalates has been developed. The target reproducibility based on iis memo 2201 was estimated as the relative standard deviation (21%) of the mean multiplied by 2.8. This was used for the evaluation of the quality of the test results in this PT.

sample #22505

DINP: The determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full agreement with the reproducibility derived from iis memo 2201.

DIBP: The determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the reproducibility derived from iis memo 2201.

DNPP: The determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the reproducibility derived from iis memo 2201.

The concentrations of the other reported Phthalates were near or below the detection limit. Therefore, for these components no z-scores were calculated, see appendix 2 for the reported test results.

sample #22506

BBP: The determination was not problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the reproducibility derived from the iis memo 2201.

DNOP: The determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the reproducibility derived from the iis memo 2201.

The concentrations of the other reported Phthalates were near or below the detection limit. Therefore, for these components no z-scores were calculated, see appendix 2 for the reported test results.

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 ($2.8 \times$ standard deviation) and the target reproducibility based on previous proficiency tests are presented in the next tables.

Component	unit	n	average	2.8 * sd	R(target)
DINP	%M/M	41	0.027	0.015	0.016
DIBP	%M/M	45	0.062	0.028	0.037
DNPP	%M/M	40	0.072	0.028	0.042

Table 5: reproducibilities of tests on sample #22505

Component	unit	n	average	2.8 * sd	R(target)
BBP	%M/M	42	0.119	0.046	0.070
DNOP	%M/M	45	0.176	0.111	0.103

Table 6: reproducibilities of tests on sample #22506

Without further statistical calculations, it can be concluded that for all tests there is a good compliance of the group of participants with the reference method.

4.3 COMPARISON OF THE PROFICIENCY TEST OF FEBRUARY 2022 WITH PREVIOUS PTS

	February 2022	March 2021	May 2020	May 2019	April 2018
Number of reporting laboratories	45	46	42	54	66
Number of test results	220	256	180	224	123
Number of statistical outliers	7	12	5	10	2
Percentage of statistical outliers	3.2%	4.7%	2.8%	4.5%	1.6%

Table 7: comparison with previous proficiency tests

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

The performance of the determinations of the proficiency test was compared, expressed as relative standard deviation (RSD) of the PTs, see below table.

Component	February 2022	March 2021	May 2020	May 2019	2017-2018	iis memo 2201
BBP	14%	17%	22%	n.e.	13-16%	21%
DEHP	n.e.	19%	25%	n.e.	n.e.	21%
DBP	n.e.	17-21%	n.e.	n.e.	18%	21%
DIDP	n.e.	38%	n.e.	21%	n.e.	21%
DINP	20%	18%	n.e.	29%	n.e.	21%
DNOP	22%	n.e.	n.e.	n.e.	n.e.	21%
DCHP	n.e.	n.e.	16%	n.e.	21%	21%
DEP	n.e.	n.e.	23-27%	n.e.	n.e.	21%
DMP	n.e.	n.e.	n.e.	33-46%	n.e.	21%
DNHP	n.e.	n.e.	n.e.	14%	n.e.	21%
DNPP	14%	n.e.	n.e.	n.e.	n.e.	21%
DIBP	16%	n.e.	n.e.	n.e.	16%	21%

Table 8: development of uncertainties over the years

The uncertainty (RSD) of the determined Phthalates in this PT is in line with previous PTs.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

The reported analytical details from the participants are listed in appendix 3. Based on the answers given by the participants the following can be summarized:

- About 88% of the reporting participants mentioned that they are accredited for the determination of Phthalates in Leather/Footwear.
- About 70% of the reporting participants used a test portion between 0.5 and 1 grams. About 28% used less sample material and about 2% used a sample intake of 2 – 3 grams.
- A vast majority (about 90%) of the reporting participants used an extraction time of 60 minutes.
- Almost all of the reporting participants used an extraction temperature of 50°C or 60°C.
- About 18% of the reporting laboratories used Hexane/Acetone as solvent mixture to release the Phthalates. Another part of the group used Toluene (35%), THF/Hexane (22%), THF (10%), THF/Acetonitrile (10%) as solvent.

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. In test method ISO16181-1 the sample intake of 1.0 gram is described.

For DINP, DIBP and DNPP found in sample #22505 and for BBP and DNOP found in sample #22506 the calculated reproducibility is in agreement with the requirements target reproducibility, therefore no separate statistical analysis has been performed.

5 DISCUSSION

In this proficiency test for the determination of Phthalates in Leather/Footwear, it was noticed that almost all of the participants were able to detect the Phthalates present in sample #22505 and sample #22506.

When the results of this interlaboratory study were compared to the LEATHER STANDARD by OEKO-TEX® and with the similar BlueSign® systems substances list or BSSL (Table 9), it was noticed that not all participants would make an identical decision about the acceptability of the samples for the determined components.

Six of the reporting laboratories would accept the sample #22505 for all categories while all other laboratories would have rejected the sample #22505 for all categories. Sample #22506 would have been rejected by all the reporting laboratories for all categories.

Ecocert	baby in %M/M	in direct skin contact in %M/M	no direct skin contact in %M/M
BlueSign® BSSL	<0.05	<0.05	<0.05
OEKO-TEX® 100	<0.05	<0.05	<0.05

Table 9: BlueSign® BSSL and LEATHER STANDARD by OEKO-TEX®

6 CONCLUSION

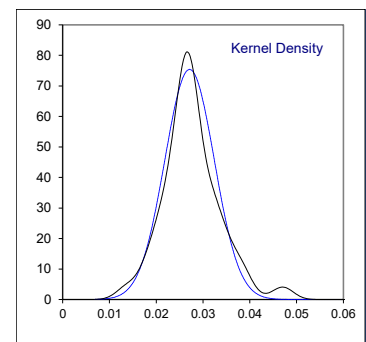
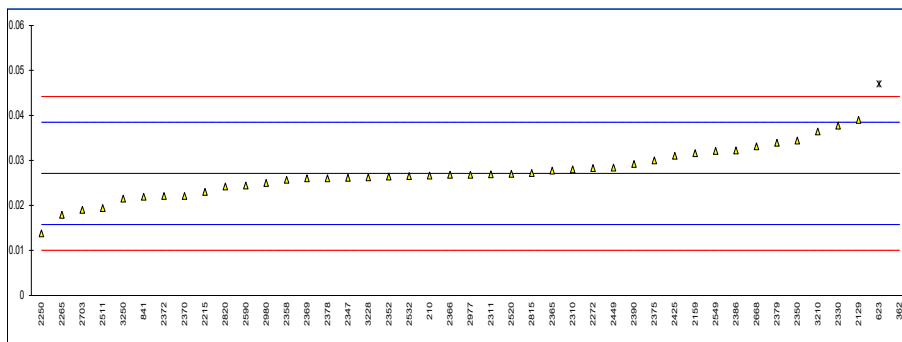
Although it can be concluded that most of the participants have no problem with the determination on Phthalates in Leather/Footwear in this PT, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary.

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 DINP - Di-iso-nonyl phthalate on sample #22505; results in %M/M

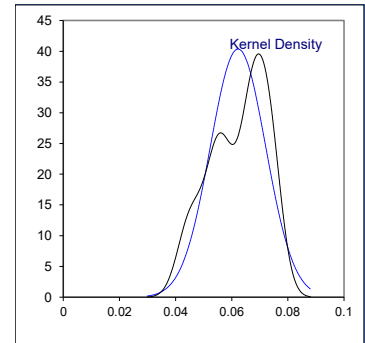
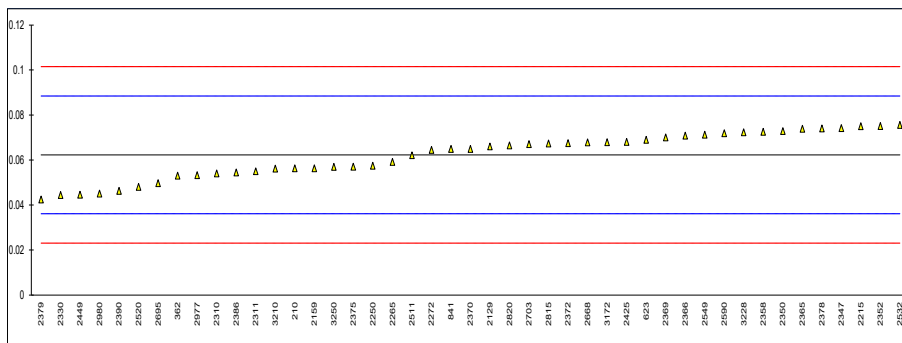
lab	method	value	mark	z(targ)	remarks
210	CPSC-CH-C1001-09.4	0.0266		-0.09	
362	ISO/TS 16181	0.147	R(0.01)	21.06	
551		----		----	
623	In house	0.0470	R(0.05)	3.49	
841	In house	0.0219		-0.91	
2129	ISO14389	0.0390		2.09	
2159	ISO/TS 16181	0.0316		0.79	
2215	ISO/TS 16181	0.023		-0.72	
2250	ISO14389	0.01376		-2.34	
2265	ISO14389	0.0179		-1.62	
2272	ISO/TS 16181	0.0283		0.21	
2310	ISO/TS 16181	0.028		0.16	
2311	ISO/TS 16181	0.0269		-0.04	
2330	ISO14389	0.0377		1.86	
2347	ISO16181	0.0261		-0.18	
2350	CPSC-CH-C1001-09.4	0.0344		1.28	
2352	ISO16181-1	0.0264		-0.12	
2358	ISO/TS 16181	0.02569		-0.25	
2365	ISO/TS 16181	0.0277		0.10	
2366	CPSC 09.4	0.0268		-0.05	
2369	ISO14389	0.026		-0.19	
2370	CNS15138-1	0.0221		-0.88	
2372	ISO14389	0.02209		-0.88	
2375	ISO14389	0.030		0.51	
2378	ISO16181-1	0.026		-0.19	
2379	CPSC-CH-C1001-09.4	0.0339	C	1.19	first reported 339.0154 %M/M
2382		----		----	
2386	ISO/TS 16181	0.0322		0.89	
2390	ISO14389	0.0292		0.37	
2425	In house	0.0310		0.68	
2449	CPS	0.0284		0.23	
2455		----		----	
2511	ISO16181-1	0.0194		-1.35	
2520	ISO/TS 16181	0.027		-0.02	
2532	ISO/TS 16181	0.0265		-0.11	
2549	ISO/TS 16181	0.0321		0.88	
2590	ISO/TS 16181	0.0244		-0.48	
2668	ISO/TS 16181	0.0331		1.05	
2695	ISO/TS 16181	not determined		----	
2703	In house	0.019		-1.42	
2756		----		----	
2815	ISO/TS 16181	0.0272		0.02	
2820	ISO/TS 16181	0.02417	C	-0.52	first reported 0.06959
2977	ISO/TS 16181	0.0268		-0.05	
2980	ISO/TS 16181	0.025	C	-0.37	first reported 0.03
3172	ISO8124-6	< 0.0005		<-4.67	possibly a false negative test result?
3210	In house	0.0364		1.63	
3228	ISO14389	0.0262		-0.16	
3250	ISO14389	0.0215		-0.99	
	normality	OK			
	n	41			
	outliers	2			
	mean (n)	0.02711			
	st.dev. (n)	0.005292	RSD = 20%		
	R(calc.)	0.01482			
	st.dev.(iis memo 2201)	0.005693			
	R(iis memo 2201)	0.01594			



Determination of DIBP - Di-iso-butyl phthalate on sample #22505; results in %M/M

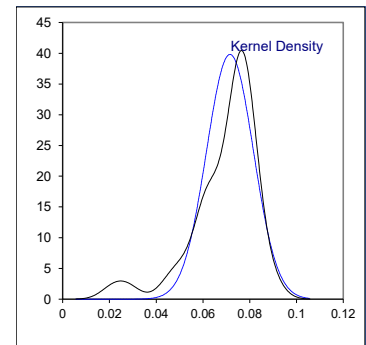
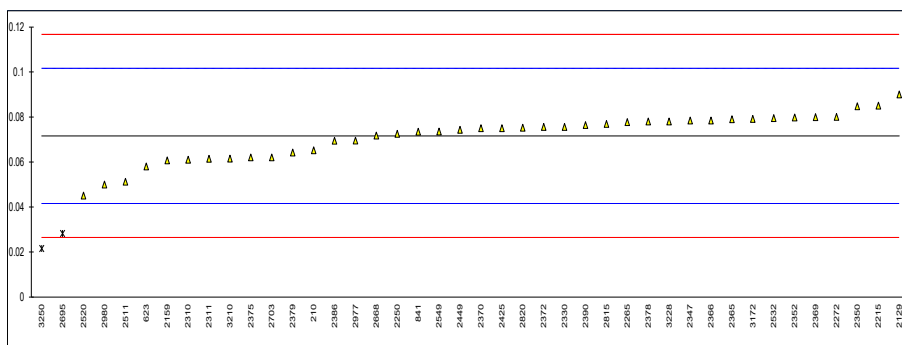
lab	method	value	mark	z(targ)	remarks
210	CPSC-CH-C1001-09.4	0.0563		-0.46	
362	ISO/TS 16181	0.053		-0.71	
551		-----		-----	
623	In house	0.069		0.52	
841	In house	0.0649		0.20	
2129	ISO14389	0.0660		0.29	
2159	ISO/TS 16181	0.0563		-0.46	
2215	ISO/TS 16181	0.075		0.97	
2250	ISO14389	0.05744		-0.37	
2265	ISO14389	0.0591		-0.24	
2272	ISO/TS 16181	0.0645		0.17	
2310	ISO/TS 16181	0.054		-0.63	
2311	ISO/TS 16181	0.0550		-0.55	
2330	ISO14389	0.0444		-1.37	
2347	ISO16181	0.0741		0.91	
2350	CPSC-CH-C1001-09.4	0.0728		0.81	
2352	ISO16181-1	0.0751		0.98	
2358	ISO/TS 16181	0.07255		0.79	
2365	ISO/TS 16181	0.0738		0.88	
2366	CPSC 09.4	0.0708		0.65	
2369	ISO14389	0.07		0.59	
2370	CNS15138-1	0.0649		0.20	
2372	ISO14389	0.06746		0.40	
2375	ISO14389	0.057		-0.40	
2378	ISO16181-1	0.074		0.90	
2379	CPSC-CH-C1001-09.4	0.0424	C	-1.52	first reported 424.3826 %M/M
2382		-----		-----	
2386	ISO/TS 16181	0.0545		-0.59	
2390	ISO14389	0.0463		-1.22	
2425	In house	0.0680		0.44	
2449	CPS	0.0445		-1.36	
2455		-----		-----	
2511	ISO16181-1	0.0621		-0.01	
2520	ISO/TS 16181	0.048	C	-1.09	first reported 0.092
2532	ISO/TS 16181	0.0756		1.02	
2549	ISO/TS 16181	0.0712		0.68	
2590	ISO/TS 16181	0.0719		0.74	
2668	ISO/TS 16181	0.0678		0.42	
2695	ISO/TS 16181	0.04969		-0.96	
2703	In house	0.067		0.36	
2756		-----		-----	
2815	ISO/TS 16181	0.0673		0.39	
2820	ISO/TS 16181	0.06640		0.32	
2977	ISO/TS 16181	0.0532		-0.69	
2980	ISO/TS 16181	0.045	C	-1.32	first reported 0.09
3172	ISO8124-6	0.06788		0.43	
3210	In house	0.0561		-0.47	
3228	ISO14389	0.0723		0.77	
3250	ISO14389	0.0569		-0.41	

normality OK
 n 45
 outliers 0
 mean (n) 0.06226
 st.dev. (n) 0.009881 RSD = 16%
 R(calc.) 0.02767
 st.dev.(iis memo 2201) 0.013074
 R(iis memo 2201) 0.03661



Determination of DNPP - Di-n-pentyl phthalate on sample #22505; results in %M/M

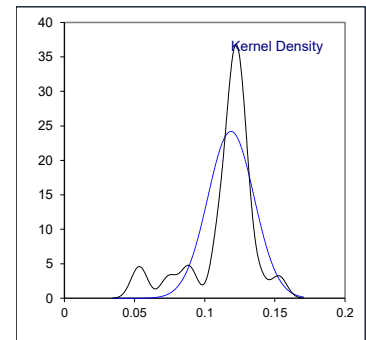
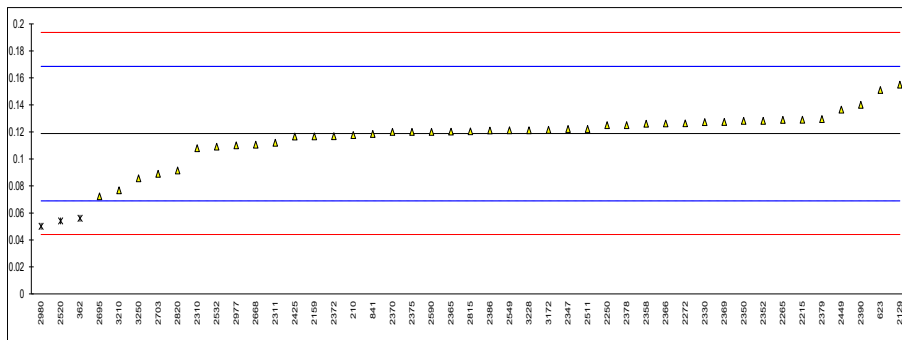
lab	method	value	mark	z(target)	remarks
210	CPSC-CH-C1001-09.4	0.0652		-0.43	
362		----		----	
551		----		----	
623	In house	0.058		-0.90	
841	In house	0.0734		0.12	
2129	ISO14389	0.0900		1.22	
2159	ISO/TS 16181	0.0607		-0.72	
2215	ISO/TS 16181	0.085		0.89	
2250	ISO14389	0.07255		0.06	
2265	ISO14389	0.0777		0.41	
2272	ISO/TS 16181	0.0800		0.56	
2310	ISO/TS 16181	0.061		-0.70	
2311	ISO/TS 16181	0.0614		-0.68	
2330	ISO14389	0.0756		0.27	
2347	ISO16181	0.0784		0.45	
2350	CPSC-CH-C1001-09.4	0.0847		0.87	
2352	ISO16181-1	0.0798		0.55	
2358	ISO/TS 16181	not applicable	C	----	first reported not detected
2365	ISO/TS 16181	0.0790		0.49	
2366	CPSC 09.4	0.0784		0.45	
2369	ISO14389	0.0799		0.55	
2370	CNS15138-1	0.0750		0.23	
2372	ISO14389	0.07554		0.26	
2375	ISO14389	0.062		-0.64	
2378	ISO16181-1	0.078		0.43	
2379	CPSC-CH-C1001-09.4	0.0642	C	-0.49	first reported 642.0898 %M/M
2382		----		----	
2386	ISO/TS 16181	0.0694		-0.15	
2390	ISO14389	0.0764		0.32	
2425	In house	0.0750		0.23	
2449	CPS	0.0743		0.18	
2455		----		----	
2511	ISO16181-1	0.0512		-1.36	
2520	ISO/TS 16181	0.045	C	-1.77	first reported 0.16
2532	ISO/TS 16181	0.0795		0.53	
2549	ISO/TS 16181	0.0735		0.13	
2590		----		----	
2668	ISO/TS 16181	0.0717		0.01	
2695	ISO/TS 16181	0.02821	C,R(0.01)	-2.89	first reported 0.02959
2703	In house	0.062		-0.64	
2756		----		----	
2815	ISO/TS 16181	0.0769		0.35	
2820	ISO/TS 16181	0.07523		0.24	
2977	ISO/TS 16181	0.0695		-0.14	
2980	ISO/TS 16181	0.05	C	-1.44	first reported 0.15
3172	ISO8124-6	0.07914		0.50	
3210	In house	0.0615		-0.67	
3228	ISO14389	0.0780		0.43	
3250	ISO14389	0.0215	R(0.01)	-3.33	
normality		OK			
n		40			
outliers		2			
mean (n)		0.07159			
st.dev. (n)		0.010023	RSD = 14%		
R(calc.)		0.02806			
st.dev.(iis memo 2201)		0.015035			
R(iis memo 2201)		0.04210			



Determination of BBP – Benzyl butyl phthalate on sample #22506; results in %M/M

lab	method	value	mark	z(targ)	remarks
210	CPSC-CH-C1001-09.4	0.1176		-0.05	
362	ISO/TS 16181	0.056	R(0.05)	-2.52	
551		-----		-----	
623	In house	0.1510		1.29	
841	In house	0.1184		-0.02	
2129	ISO14389	0.155		1.45	
2159	ISO/TS 16181	0.1166		-0.09	
2215	ISO/TS 16181	0.129		0.41	
2250	ISO14389	0.1250		0.25	
2265	ISO14389	0.1289		0.41	
2272	ISO/TS 16181	0.1263		0.30	
2310	ISO/TS 16181	0.108		-0.43	
2311	ISO/TS 16181	0.1119		-0.28	
2330	ISO14389	0.1272		0.34	
2347	GB/T32440	0.1221		0.13	
2350	CPSC-CH-C1001-09.4	0.1281		0.37	
2352	ISO16181-1	0.1281		0.37	
2358	ISO/TS 16181	0.12604		0.29	
2365	ISO/TS 16181	0.1202		0.06	
2366	CPSC 09.4	0.1262		0.30	
2369	ISO14389	0.1273		0.34	
2370	CNS15138-1	0.120		0.05	
2372	CNS15138-1	0.1168		-0.08	
2375	ISO14389	0.120		0.05	
2378	ISO16181-1	0.125		0.25	
2379	CPSC-CH-C1001-09.4	0.1294	C	0.43	first reported 1294.2367 %M/M
2382		-----		-----	
2386	ISO/TS 16181	0.1210		0.09	
2390	ISO14389	0.140		0.85	
2425	In house	0.1165		-0.09	
2449	CPS	0.1365		0.71	
2455		-----		-----	
2511	ISO16181-1	0.1221		0.13	
2520	ISO/TS 16181	0.054	C,R(0.05)	-2.60	first reported 0.22
2532	ISO/TS 16181	0.109		-0.39	
2549	ISO/TS 16181	0.1211		0.09	
2590	ISO/TS 16181	0.12	C	0.05	first reported 0.069
2668	ISO/TS 16181	0.1105		-0.33	
2695	ISO/TS 16181	0.072289	C	-1.86	first reported 0.06484
2703	In house	0.089		-1.19	
2756		-----		-----	
2815	ISO/TS 16181	0.1204		0.06	
2820	ISO/TS 16181	0.09140		-1.10	
2977	ISO/TS 16181	0.1100		-0.35	
2980	ISO/TS 16181	0.05	C,R(0.05)	-2.76	first reported 0.21
3172	ISO8124-6	0.12148		0.11	
3210	In house	0.0767		-1.69	
3228	ISO14389	0.1211		0.09	
3250	ISO14389	0.0856		-1.33	

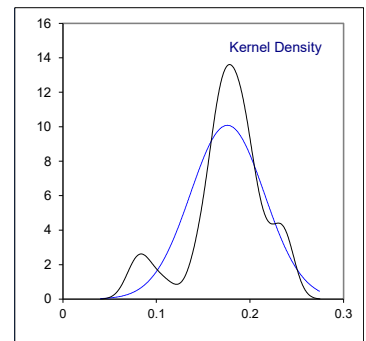
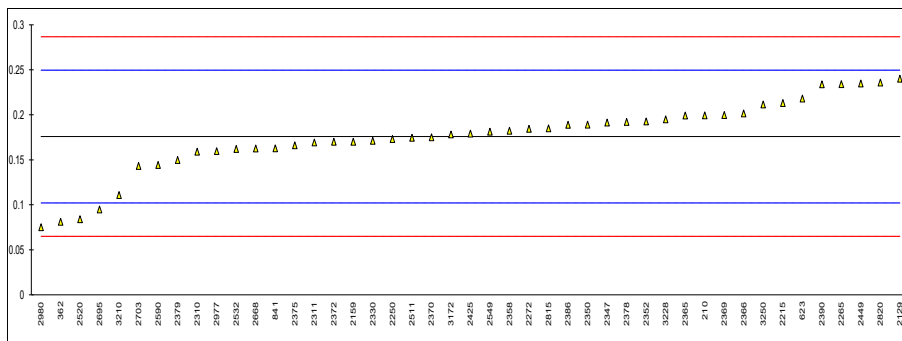
normality suspect
 n 42
 outliers 3
 mean (n) 0.11878
 st.dev. (n) 0.016481 RSD = 14%
 R(calc.) 0.04615
 st.dev.(iis memo 2201) 0.024944
 R(iis memo 2201) 0.06984



Determination of DNOP - Di-n-octyl phthalate on sample #22506; results in %M/M

lab	method	value	mark	z(targ)	remarks
210	CPSC-CH-C1001-09.4	0.1992		0.63	
362	ISO/TS 16181	0.081		-2.57	
551		-----		-----	
623	In house	0.2180		1.14	
841	In house	0.1626		-0.36	
2129	ISO14389	0.240		1.74	
2159	ISO/TS 16181	0.1699		-0.16	
2215	ISO/TS 16181	0.213		1.01	
2250	ISO14389	0.1730		-0.08	
2265	ISO14389	0.2339		1.57	
2272	ISO/TS 16181	0.1844		0.23	
2310	ISO/TS 16181	0.159		-0.46	
2311	ISO/TS 16181	0.1692		-0.18	
2330	ISO14389	0.1709		-0.13	
2347	GB/T32440	0.1912		0.42	
2350	CPSC-CH-C1001-09.4	0.1889		0.35	
2352	ISO16181-1	0.1926		0.45	
2358	ISO/TS 16181	0.1822		0.17	
2365	ISO/TS 16181	0.1990		0.63	
2366	CPSC 09.4	0.2015		0.69	
2369	ISO14389	0.1996		0.64	
2370	CNS15138-1	0.175		-0.02	
2372	CNS15138-1	0.1698		-0.16	
2375	ISO14389	0.166		-0.27	
2378	ISO16181-1	0.192		0.44	
2379	CPSC-CH-C1001-09.4	0.1497	C	-0.71	first reported 1496.9452 %M/M
2382		-----		-----	
2386	ISO/TS 16181	0.1888		0.35	
2390	ISO14389	0.2338		1.57	
2425	In house	0.1790		0.08	
2449	CPS	0.2346		1.59	
2455		-----		-----	
2511	ISO16181-1	0.1745		-0.04	
2520	ISO/TS 16181	0.084	C	-2.49	first reported 0.26
2532	ISO/TS 16181	0.162		-0.38	
2549	ISO/TS 16181	0.1811		0.14	
2590	ISO/TS 16181	0.1442		-0.86	
2668	ISO/TS 16181	0.1625		-0.36	
2695	ISO/TS 16181	0.094837	C	-2.19	first reported 0.08935
2703	In house	0.143		-0.89	
2756		-----		-----	
2815	ISO/TS 16181	0.1849		0.24	
2820	ISO/TS 16181	0.23571		1.62	
2977	ISO/TS 16181	0.1595		-0.44	
2980	ISO/TS 16181	0.075	C	-2.73	first reported 0.26
3172	ISO8124-6	0.17802		0.06	
3210	In house	0.1108		-1.76	
3228	ISO14389	0.195		0.52	
3250	ISO14389	0.2114		0.96	

normality OK
 n 45
 outliers 0
 mean (n) 0.17587
 st.dev. (n) 0.039551 RSD = 22%
 R(calc.) 0.11074
 st.dev.(iis memo 2201) 0.036933
 R(iis memo 2201) 0.10341



APPENDIX 2

Summary of other Phthalates in sample #22505: results in %M/M

BBP = Benzyl butyl phthalate
 DEHP = Di-(2-ethylhexyl) phthalate
 DBP = Dibutyl phthalate
 DIDP = Di-iso-decyl phthalate
 DNOP = Di-n-octyl phthalate
 DCHP = Dicyclohexyl phthalate
 DEP = Diethyl phthalate

Lab	BBP	DEHP	DBP	DIDP	DNOP	DCHP	DEP
210	----	----	----	----	----	----	----
362	----	----	----	----	----	----	----
551	----	----	----	----	----	----	----
623	not detected	not detected	not detected	not detected	not detected	not detected	not detected
841	0.005	0.005	0.005	0.005	0.005	0.005	0.005
2129	----	----	----	----	----	----	----
2159	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2215	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2250	----	----	----	----	----	----	----
2265	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01
2272	----	----	----	----	----	----	----
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	<0.010	<0.003	<0.003	<0.003
2350	< 0.015	< 0.015	< 0.015	< 0.015	< 0.015	< 0.015	< 0.015
2352	----	----	----	----	----	----	----
2358	not detected	not detected	not detected	not detected	not detected	<i>not applicable</i>	<i>not applicable</i>
2365	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050
2366	----	----	----	----	----	----	----
2369	<0.003	<0.003	<0.003	<0.01	<0.003	<0.003	<0.003
2370	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500
2372	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2375	----	----	----	----	----	----	----
2378	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2379	<i><u>0.0037</u></i>	<i><u>0.0022</u></i>	not detected	not detected	not detected	not detected	not detected
2382	----	----	----	----	----	----	----
2386	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
2390	not detected	not detected	0.0214	not detected	not detected	not detected	not detected
2425	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2449	----	----	0.0187	----	----	----	----
2455	----	----	----	----	----	----	----
2511	----	----	----	----	----	----	----
2520	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
2532	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2549	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2590	----	----	0.0651	----	----	----	----
2668	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2695	not determined	not determined	not determined	not determined	not determined	not determined	not determined
2703	----	0.001	----	----	----	----	----
2756	----	----	----	----	----	----	----
2815	not detected	not detected	not detected	not detected	not detected	not detected	----
2820	----	----	----	----	----	----	----
2977	< 0.0010	< 0.0010	< 0.0010	< 0.0010	< 0.0010	not analyzed	not analyzed
2980	< detection limit	< detection limit	< detection limit	< detection limit	< detection limit	< detection limit	< detection limit
3172	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005
3210	<0.002	<0.002	<0.002	<0.005	<0.002	<0.002	<0.002
3228	not detected	not detected	not detected	not detected	not detected	----	----
3250	----	----	----	----	----	----	----

bold, italic and underline results

lab 2358 first reported not detected

lab 2379 first reported 36.9943 %M/M (BBP), 22.4001 %M/M (DEHP)

Summary of other Phthalates in sample #22505: results in %M/M - continued

DMP = Dimethyl phthalate
 DNHP = Di-n hexyl phthalate
 DPHP = Di(2-propylheptyl) phthalate
 DUP = Diundecyl phthalate
 DPrP = Di-n-propyl phthalate
 DMEP = Di-(2-methoxyethyl) phthalate
 Other = Other Phthalates

Lab	DMP	DNHP	DPHP	DUP	DPrP	DMEP	Other
210	----	----	----	----	----	----	----
362	----	----	----	----	----	----	----
551	----	----	----	----	----	----	----
623	not detected	not detected	not detected	not detected	not detected	not detected	not detected
841	0.005	0.005	0.005	0.005	0.005	0.005	0.005
2129	----	----	----	not analyzed	----	----	----
2159	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2215	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2250	----	----	----	----	----	----	----
2265	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01
2272	----	----	----	----	----	----	----
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	----
2350	< 0.015	< 0.015	< 0.015	< 0.015	< 0.015	< 0.015	< 0.015
2352	----	----	----	----	----	----	----
2358	<u>not applicable</u>	<u>not applicable</u>	<u>not applicable</u>	<u>not applicable</u>	<u>not applicable</u>	<u>not applicable</u>	not detected
2365	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050
2366	----	----	----	----	----	----	----
2369	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003
2370	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500
2372	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	0.16509
2375	----	----	----	----	----	----	----
2378	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	----
2379	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2382	----	----	----	----	----	----	----
2386	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	----
2390	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2425	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	0.1740
2449	----	----	----	----	----	----	----
2455	----	----	----	----	----	----	----
2511	----	----	----	----	----	----	----
2520	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
2532	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2549	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2590	----	----	----	----	----	----	----
2668	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2695	not determined	not determined	not determined	not determined	not determined	not determined	not determined
2703	----	----	----	----	----	----	----
2756	----	----	----	----	----	----	----
2815	----	not detected	----	----	----	not detected	----
2820	----	----	----	----	----	----	----
2977	not analyzed	< 0.0010	not analyzed	< 0.0010	not analyzed	< 0.0010	not analyzed
2980	< detection limit	< detection limit	< detection limit	< detection limit	< detection limit	< detection limit	< detection limit
3172	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005
3210	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	----
3228	----	----	----	----	----	not detected	----
3250	----	----	----	----	----	----	----

bold, italic and underline result

lab 2358 first reported not detected

Summary of other Phthalates in sample #22506: results in %M/M

DEHP = Di-(2-ethylhexyl) phthalate
 DBP = Dibutyl phthalate
 DIDP = Di-iso-decyl phthalate
 DINP = Di-iso-nonyl phthalate
 DCHP = Dicyclohexyl phthalate
 DEP = Diethyl phthalate
 DMP = Dimethyl phthalate
 DNHP = Di-n-hexyl phthalate

Lab	DEHP	DBP	DIDP	DINP	DCHP	DEP	DMP	DNHP
210	----	----	----	----	----	----	----	----
362	----	----	----	----	----	----	----	----
551	----	----	----	----	----	----	----	----
623	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
841	0.005	0.005	0.005	0.005	0.005	0.005	0.005	0.005
2129	----	----	----	----	----	----	----	----
2159	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2215	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2250	----	----	----	----	----	----	----	----
2265	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01
2272	----	----	----	----	----	----	----	----
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	<0.003	<0.010	<0.010	<0.003	<0.003	<0.003	<0.003
2350	< 0.015	< 0.015	< 0.015	< 0.015	< 0.015	< 0.015	< 0.015	< 0.015
2352	----	----	----	----	----	----	----	----
2358	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2365	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050
2366	----	----	----	----	----	----	----	----
2369	<0.003	<0.003	<0.01	<0.01	<0.003	<0.003	<0.003	<0.003
2370	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500
2372	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2375	----	----	----	----	----	----	----	----
2378	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2379	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2382	----	----	----	----	----	----	----	----
2386	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
2390	not detected	0.0435	not detected	not detected	not detected	not detected	not detected	not detected
2425	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2449	----	0.0428	----	----	----	----	----	----
2455	----	----	----	----	----	----	----	----
2511	----	----	----	----	----	----	----	----
2520	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
2532	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2549	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2590	----	----	----	----	----	----	----	Not detected
2668	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2695	not determ.	not determ.	not determ.	not determ.	not determ.	not determ.	not determ.	not determ.
2703	----	0.001	----	----	----	----	----	----
2756	----	----	----	----	----	----	----	----
2815	0.0041	not detected	not detected	not detected	not detected	----	----	0.0495
2820	----	0.00126	----	0.00308	----	----	----	----
2977	< 0.0010	< 0.0010	< 0.0010	< 0.0010	not analyzed	not analyzed	not analyzed	< 0.0010
2980	< detect. limit	< detect. limit	< detect. limit	< detect. limit	< detect. limit	< detect. limit	< detect. limit	< detect. limit
3172	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005
3210	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
3228	not detected	not detected	not detected	not detected	----	----	----	----
3250	----	----	----	----	----	----	----	----

Summary of other Phthalates in sample #22506: results in %M/M - continued

DIBP = Di-iso-butyl phthalate
 DPHP = Di(2-propylheptyl) phthalate
 DNPP = Di-n-pentyl phthalate
 DUP = Diundecyl phthalate
 DPrP = Di-n-propyl phthalate
 DMEP = Di-(2-methoxyethyl) phthalate
 Other = Other Phthalates

Lab	DIBP	DPHP	DNPP	DUP	DPrP	DMEP	Other
210	0.0143	----	----	----	----	----	----
362	----	----	----	----	----	----	----
551	----	----	----	----	----	----	----
623	0.0070	not detected	not detected	not detected	not detected	not detected	not detected
841	0.005	0.005	0.005	0.005	0.005	0.005	0.005
2129	----	----	----	not analyzed	----	----	----
2159	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2215	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2250	----	----	----	----	----	----	----
2265	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01	< 0,01
2272	0.0067	----	----	----	----	----	----
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	0.0045	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2347	0.0062	<0.003	<0.003	<0.003	<0.003	<0.003	----
2350	< 0.015	< 0.015	< 0.015	< 0.015	< 0.015	< 0.015	< 0.015
2352	----	----	----	----	----	----	----
2358	0.007086	not detected	not detected	not detected	not detected	not detected	not detected
2365	0.0063	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050
2366	----	----	----	----	----	----	----
2369	0.0057	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003
2370	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500
2372	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	0.2866
2375	----	----	----	----	----	----	----
2378	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	----
2379	<u>0.0074</u>	not detected	<u>0.0042</u>	not detected	not detected	not detected	not detected
2382	----	----	----	----	----	----	----
2386	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	----
2390	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2425	0.0077	Not detected	Not detected	Not detected	Not detected	Not detected	0.3032
2449	----	----	----	----	----	----	----
2455	----	----	----	----	----	----	----
2511	----	----	----	----	----	----	----
2520	0.009	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
2532	0.0107	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2549	0.0078	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2590	0.00340	----	----	----	----	----	----
2668	0.01	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2695	not determ.	not determined	not determined	not determined	not determined	not determined	not determined
2703	0.005	----	0.001	----	----	----	----
2756	----	----	----	----	----	----	----
2815	0.0050	----	not detected	----	----	not detected	----
2820	0.00398	----	0.00153	----	----	----	----
2977	0.0017	not analyzed	< 0.0010	< 0.0010	not analyzed	< 0.0010	not analyzed
2980	0.01	< detection limit	< detection limit	< detection limit	< detection limit	< detection limit	< detection limit
3172	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005
3210	0.0049	<0.002	<0.002	<0.002	<0.002	<0.002	----
3228	not detected	----	not detected	----	----	not detected	----
3250	----	----	----	0.8997	----	----	----

bold, italic and underline result

lab 2379 first reported 73.9154 %M/M (DIBP), 42.0910 %M/M (DNPP)

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				
362	Yes	1g	Toluene	60	(60±5)
551	---				
623	Yes	0.1 gram	THF : Hexane	60	60
841	Yes	0.5 grams	Toluene	60	60
2129	Yes	0,5	THF	60	60
2159	Yes	1	Toluene	60	60
2215	Yes	1g	10ml	60	60
2250	Yes	0,3	THF/ACN (1:2)	60	60
2265	Yes	0,3g	THF/Hexan (1:2)	60	60
2272	Yes	1g	toluene	1hour	60°C
2310	Yes	0.5g	Hexane	60	50
2311	Yes	0.5	Acetone and Hexane	60	50
2330	Yes	0.30 gram	Tetrahydrofuran (THF) : Hexane ratio 1:2	1 hour	60 ± 5
2347	No	0.5g	Toluene	60	60
2350	Yes	0.5 g	THF + ACN	2 h 30 min	60
2352	Yes	0.5g	Toluene	60	60
2358	Yes	1 gram	n-hexane : acetone (80:20)	60	50
2365	Yes	1g	toluene	60	60
2366	Yes				
2369	---				
2370	Yes	0.5 g	THF : Hexane=10 : 20 mL	30	room temperature
2372	Yes	0.3 g	THF	60	60
2375	Yes	0.3g	THF	60	60
2378	Yes	1g	toluene	60	60
2379	Yes	0.1 gram	Tetrahydrofurane / Hexane	60	60
2382	---				
2386	Yes	0,5	n-Hexane/Acetone	60	50
2390	Yes	0.1g	THF/Hexane	60	60
2425	Yes	0.5g	Toluene	60	60
2449	Yes	0.3	THF and ACN	30	60
2455	---				
2511	Yes				
2520	No	0.5 gm	Toulene	60	60
2532	Yes	0.5g	n-Hexane/Acetone 80:20	60	50
2549	Yes	0.5 g	Toluene	60	60
2590	Yes	1g	toluene	60	60
2668	Yes	0.5 gms	THF:Hexane	60	60
2695	No	1	Hexane:Acetone	60	50
2703	Yes	0.5g	THF/Hexane	150	60
2756	---				
2815	Yes	~ 2	80% n-hexane 20% acetone	60	50
2820	Yes	0,5	TOLUENE	60	60
2977	No	1 g	THF:Hexane=1:2	1 hour	60
2980	No	1	hexan/acetone	60	60
3172	---				
3210	Yes	1g	Toluene	60	60
3228	Yes	0.3	THF	60	60
3250	Yes	0.3g	THF/ACN	1 hours	60

APPENDIX 4

Number of participants per country

1 lab in BANGLADESH
1 lab in BRAZIL
1 lab in BULGARIA
2 labs in CAMBODIA
2 labs in EGYPT
1 lab in ETHIOPIA
1 lab in FRANCE
4 labs in GERMANY
1 lab in HONG KONG
5 labs in INDIA
1 lab in INDONESIA
5 labs in ITALY
1 lab in KOREA, Republic of
1 lab in MOROCCO
10 labs in P.R. of CHINA
2 labs in PAKISTAN
1 lab in SWITZERLAND
2 labs in TAIWAN
1 lab in THAILAND
1 lab in TUNISIA
2 labs in TURKEY
1 lab in U.S.A.
1 lab in UNITED KINGDOM
1 lab in VIETNAM

APPENDIX 5

Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?

Literature

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