

# Results of Proficiency Test Phthalates in Textile April 2022

Organized by: Institute for Interlaboratory Studies

Spijkenisse, The Netherlands

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#### 1 Introduction

Phthalates are commonly used as plasticizers to increase softness of plastic, especially in PVC. In the clothing industry, they can be found in synthetic leather, buttons, coated fabric, plastisol and dye printing.

Since 2019 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of Phthalates in Textile every year. During the annual proficiency testing program 2021/2022 it was decided to continue the proficiency test for the determination of Phthalates in Textile.

In this interlaboratory study 71 laboratories in 27 countries registered for participation, see appendix 4 for the number of participants per country. In this report the results of the proficiency test on Phthalates in Textile are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

## 2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.

It was decided to send two different textile samples positive on Phthalates of 3 grams each, one sample labelled #22565 and the other sample #22566.

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.

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## 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 beige cotton was selected which was made positive on Dibutyl phthalate (DBP) and Di-(2-ethylhexyl) phthalate (DEHP). The batch was cut into small pieces. After homogenization about 90 small plastic bags were filled with approximately 3 grams each and labelled #22565.

The homogeneity of the subsamples was checked by determination of DBP using an inhouse test method on 8 stratified randomly selected subsamples.

	DBP in %M/M
sample #22565-1	0.1289
sample #22565-2	0.1565
sample #22565-3	0.1503
sample #22565-4	0.1509
sample #22565-5	0.1478
sample #22565-6	0.1479
sample #22565-7	0.1484
sample #22565-8	0.1515

Table 1: homogeneity test results of subsamples #22565

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

	DBP in %M/M
r (observed)	0.0227
reference method	iis memo 1701
0.3 x R (reference method)	0.0199

Table 2: evaluation of the repeatability of subsamples #22565

The calculated repeatability is almost in agreement with 0.3 times the corresponding reproducibility of the reference method. The relative standard deviation of the repeatability (RSDr = 5%) is much lower than the relative standard deviation of the reproducibility in iis Phthalates in Textile PTs. The average RSD<sub>R</sub> in iis PTs is about 17%. Therefore, homogeneity of the subsamples was assumed.

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For the second sample a batch of green cotton was selected which was made positive on Di-n-octyl phthalate (DNOP). The batch was cut into small pieces. After homogenization about 100 small plastic bags were filled with approximately 3 grams each and labelled #22566.

The homogeneity of the subsamples was checked by determination of DNOP using an in house test method on 9 stratified randomly selected subsamples.

	DNOP in %M/M
sample #22566-1	0.0999
sample #22566-2	0.0899
sample #22566-3	0.0950
sample #22566-4	0.0901
sample #22566-5	0.0913
sample #22566-6	0.0975
sample #22566-7	0.0893
sample #22566-8	0.0956
sample #22566-9	0.0971

Table 3: homogeneity test results of subsamples #22566

From the above test results the repeatability was calculated and compared with 0.3 times the estimated reproducibility of the reference method in agreement with the procedure of ISO13528. Annex B2 in the next table.

	DNOP in %M/M
r (observed)	0.0110
reference method	iis memo 1701
0.3 x R (reference method)	0.0126

Table 4: evaluation of the repeatability of subsamples #22566

The calculated repeatability is in agreement with 0.3 times the estimated reproducibility of the reference method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one textile sample labelled #22565 and one textile sample labelled #22566 were sent on March 16, 2022.

## 2.5 ANALYZES

The participants were requested to determine on samples #22565 and #22566 sixteen individual Phthalates and the total of other Phthalates:

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

It was requested not to use less than 0.5 gram per determination to ensure the homogeneity. It was also requested to report if the laboratory was accredited for the determined components and to report some analytical details.

It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the test results but report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test 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 method (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

## 3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per determination in appendices 1 and 2 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

## 3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<...' or '>...' were not used in the statistical evaluation.

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

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by F(0.01) for the Rosner's test. Stragglers are marked by F(0.01) 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 of this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, 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 - average of PT)} / \text{target standard deviation}
```

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

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

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

## 4 **EVALUATION**

In this proficiency test some problems were encountered with the dispatch of the samples. Twelve participants reported test results after the final reporting date and two other participants did not report any test results. Not all participants were able to report all components requested.

In total 69 laboratories reported 271 numerical test results. Observed were 7 outlying test results, which is 2.6%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

All data sets proved to have a normal Gaussion distribution.

## 4.1 EVALUATION PER SAMPLE AND PER COMPONENT

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

Two test methods ISO14389 and CPSC-CH-C1001-09.4 are used for determining Phthalates in textile. Regretfully, the CPSC-CH-C1001-09.4 method does not contain any precision statements. The ISO14389:14 method does provide a variety of precision data. There are precision data mentioned for 4 different procedures in ISO14389:14 of which procedure 4 prescribes the extraction with THF followed by precipitation with Acetonitrile. The relative reproducibility for 7 different Phthtalates ranges from 31.5% - 124.9%.

For several years iis organizes PTs on Phthalates in Polymers. In 2017 it was decided to use the iis PT data gathered since 2010 for Phthalates in Polymers to estimate a more realistic target reproducibility, see iis memo 1701 (lit 13). The target reproducibility was estimated as the RSD (relative standard deviation) of 16% of the mean multiplied by 2.8. It was decided to use the estimated iis target reproducibility for Phthalates in Polymers also for the Phthalates in Textile because as explained above test method ISO14389:14 mentioned a variety of precision data.

## sample #22565

<u>DEHP</u>: This determination was not problematic. One statistical outlier was

observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the reproducibility derived from its memo 1701.

<u>DBP</u>: This determination was not problematic. No statistical outliers were

observed. The calculated reproducibility is in good agreement with the

reproducibility derived from iis memo 1701.

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

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## sample #22566

DINP:

This determination was problematic at the low level of 0.028 %M/M. Four 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.

DNOP:

This 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 its memo 1701.

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

## 4.2 Performance evaluation for the group of Laboratories

A comparison has been made between the 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 methods are presented in the next tables.

Component	unit	n	average	2.8 * sd	R(target)
DEHP	%M/M	68	0.117	0.039	0.053
DBP	%M/M	69	0.099	0.047	0.044

Table 5: reproducibilities of components in sample #22565

Component	unit	n	average	2.8 * sd	R(target)
DINP	%M/M	60	0.028	0.016	0.013
DNOP	%M/M	67	0.095	0.037	0.042

Table 6: reproducibilities of components in sample #22566

Without further calculations it can be concluded that for Di-(2-ethylhexyl) phthalate (DEHP), Dibutyl phthalate (DBP) and Di-n-octyl phthalate (DNOP) there is a good compliance of the group of participating laboratories with the target. The problematic component has been discussed in paragraph 4.1.

## 4.3 COMPARISON OF PROFICIENCY TEST OF APRIL 2022 WITH THE PREVIOUS PTS

	April 2022	March 2021	March 2020	March 2019
Number of reporting laboratories	69	68	69	69
Number of test results	271	200	134	188
Number of statistical outliers	7	4	12	8
Percentage of statistical outliers	2.6%	2.0%	9.0%	4.3%

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 tests was compared to the target. The conclusions are given in the following table.

Component	April 2022	March 2021	March 2020	March 2019	Target
BBP	n.e.	14%	n.e.	12%	16%
DEHP	12%	n.e.	n.e.	n.e.	16%
DBP	17%	17%	n.e.	29 - 31%	16%
DINP	21%	n.e.	n.e.	n.e.	16%
DNOP	14%	n.e.	n.e.	n.e.	16%
DCHP	n.e.	16%	n.e.	n.e.	16%
DNHP	n.e.	n.e.	12%	n.e.	16%
DMEP	n.e.	n.e.	17%	n.e.	16%

Table 8: development of uncertainties over the years

The uncertainties (RSD%) of DEHP, DBP and DNOP observed in this PT are in line with previous iis PTs.

## 4.4 EVALUATION OF THE ANALYTICAL DETAILS

The test method ISO14389 is used by about 50% of the reporting participants and the test method CPSC-CH-C1001-09.4 is used by about 25% of the reporting participants. The majority of the other participants used an in house test method.

For this PT also some analytical details were requested. The reported details are given in appendix 3. Based on the answers given by the reporting participants the following can be summarized:

- About 90% of the participants mentioned that they are accredited for the determination of Phthalates in Textile.
- About 45% of the participants used the samples as received and about 50% further cut the samples prior to analysis.
- About 50% of the participants used less than 0.5 grams as sample intake and about 30% used 0.5 grams. It is remarkable that about 50% of the group had used a lower sample intake than it was instructed in the letter of instructions.

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- Almost all of the participants used ultrasonic as technique to release/extract the Phthalates.
- Almost all of the participants used THF or a THF mixture as extraction solvent.
- The extraction time differs from 30 minutes to 2.5 hours. About 80% of the participants used an extraction time of 60 minutes.
- The extraction temperature differs from room temperature to 60°C. About 75% of the participants used an extraction temperature of 60°C.

Although differences are mentioned in the amount of sample intake and the sample preparation a vast majority of the group follow the same analytical procedures. Furthermore, the variation observed in the test results are for three of the four components in line with the target. And the component which was not in line with the target was measured at a low level of Phthalates. Therefore, no separate statistical analysis has been performed.

## 5 DISCUSSION

All reporting participants were able to detect DEHP and DBP in sample #22565 and DINP and DNOP in sample #22566. One lab reported for DINP "not detected" presumable because the test value was below the SML of 0.05%M/M. One must realize that the ecolabelling SML is based on the sum of Phthalates (see Table 9).

When the results of this interlaboratory study were compared to the Ecolabelling Standards and Requirements for Textiles in EU, such as OEKO-TEX® 100 and the similar Bluesign® RSL, it was noticed that all participants would make identical decisions about the acceptability of the textiles for the determined components. All reporting laboratories would have rejected samples #22565 and #22566 for all categories.

Ecolabelling Standard baby in %M/M		in direct skin contact in %M/M	no direct skin contact in %M/M	
Bluesign® RSL	<0.05	<0.05	<0.05	
OEKO-TEX® 100	<0.05	<0.05	<0.05	

Table 9: Maximum allowed level of sum of Phthalates according to Ecolabelling Standards

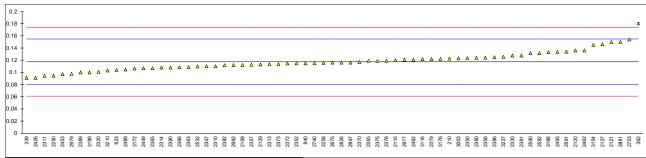
## 6 CONCLUSION

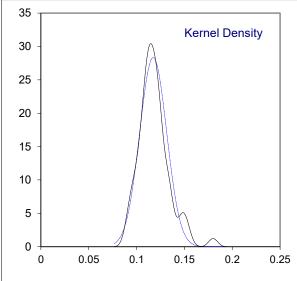
Although, it can be concluded that the majority of the participants has no problem with the determination of the Phthalates in the textile samples of this PT, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore,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

APPENDIX 1
Determination of DEHP - Di-(2-ethylhexyl) phthalate (CAS No. 117-81-7) on sample #22565; results in %M/M

	in %M/M				
lab	method	value	mark	z(targ)	remarks
210	ISO14389	0.12266		0.28	
339	In house	0.091		-1.40	
362	In house	0.18	C,R(0.01)	3.34	First reported 0.22
551					
623	In house	0.104		-0.71	
840	ISO14389	0.115		-0.12	
2115	ISO14389	0.12		0.14	
2120	ISO14389	0.136		1.00	
2121	ISO14389	0.15		1.74	
2129	ISO14389	0.113		-0.23	
2137	IEC62321-8	0.1464		1.55	
2159 2250	ISO14389 ISO14389	0.112 0.0947		-0.28 -1.21	
2258	CPSC-CH-C1001-09.4	0.0947		-0.10	
2265	ISO14389	0.1190		0.09	
2310	CPSC-CH-C1001-09.4	0.1150		-0.39	
2311	ISO14389	0.0943		-1.23	
2313	ISO14389	0.1132		-0.22	
2314	CPSC-CH-C1001-09.4	0.1080		-0.50	
2320	ISO14389	0.100948		-0.87	
2330	CPSC-CH-C1001-09.4	0.1277		0.55	
2347	GB/T20388	0.110		-0.39	
2350	CPSC-CH-C1001-09.4	0.1235		0.33	
2352	ISO14389	0.1150		-0.12	
2357	ISO14389	0.1125		-0.26	
2358	CPSC-CH-C1001-09.4	0.12425		0.37	
2363	CPSC-CH-C1001-09.4	0.1087		-0.46	
2365	ISO14389	0.1069		-0.56	
2366	GB/T20388	0.1084		-0.48	
2370	ISO14389	0.117		-0.02	
2372	CPSC-CH-C1001-09.4	0.114597		-0.15	
2373	CPSC-CH-C1001-09.4	0.1135		-0.20	
2375	CPSC-CH-C1001-09.4	0.119		0.09	
2378	GB/T20388	0.119		0.09	
2379	ISO14389	0.1218		0.24	
2380 2381	CPSC-CH-C1001-09.4	0.12408		0.36 0.55	
2382	CPSC-CH-C1001-09.4 ISO14389	0.1277 0.11174		-0.30	
2386	ISO14389	0.11174		0.40	
2389	CPSC-CH-C1001-09.4	0.1240		-0.92	
2390	CPSC-CH-C1001-09.4	0.108		-0.50	
2426	ISO14389	0.091		-1.40	
2449	CPSC-CH-C1001-09.4	0.1067		-0.57	
2453	ISO14389	0.097		-1.08	
2462	GB/T20388&ISO14389	0.136		1.00	
2489	ISO14389	0.1048		-0.67	
2492	GB/T20388	0.1210		0.20	
2495	ISO14389	0.1336		0.87	
2532	ISO14389	0.1097		-0.41	
2582	ISO14389	0.13143		0.75	
2590	ISO14389	0.1314		0.75	
2591	CPSC-CH-C1001-09.4	0.1341		0.89	
2644	10044200	0.116		0.07	
2675	ISO14389	0.116		-0.07 1.05	
2703 2740	In house	0.154 0.115		1.95	
	ISO14389			-0.12	
2826 2881	ISO14389 In house	0.1160 0.15		-0.07 1.74	
2947	In house	0.13		-0.07	
2947 2977	ISO14389	0.110		0.07	
2979	ISO14389	0.120002		-1.05	
2982	ISO14389	0.11191		-0.29	
3003		0.1230		0.30	
3116	ISO14389	0.1216		0.23	
3154	ISO16181-1	0.145		1.47	
3166	In house	0.1332		0.85	
3172	ISO8124-6	0.10633		-0.59	
3176	ISO14389	0.1221		0.25	
3199	In house	0.100		-0.92	
3210	In house	0.1031		-0.76	
3237	CPSC-CH-C1001-09.4	0.1255		0.44	

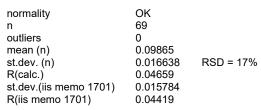
```
normality OK
n 68
outliers 1
mean (n) 0.11732
st.dev. (n) 0.014078
R(calc.) 0.03942
st.dev.(iis memo 1701) 0.018771
R(iis memo 1701) 0.05256
```

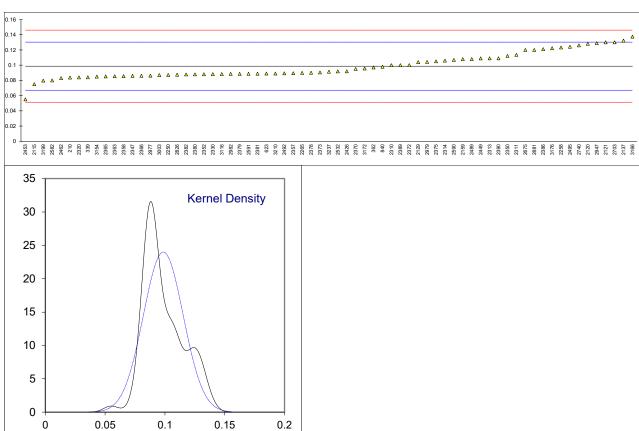




## Determination of DBP - Dibutyl phthalate (CAS No. 84-74-2) on sample #22565; results in %M/M

lab	method	value	mark	z(targ)	remarks
210	ISO14389	0.083751	IIIai N	-0.94	i oniur No
339	In house	0.084		-0.93	
362	In house	0.097		-0.10	
551					
623	In house	0.089	С	-0.61	First reported 0.046
840	ISO14389	0.098		-0.04	
2115	ISO14389	0.075		-1.50	
2120 2121	ISO14389 ISO14389	0.128 0.13		1.86 1.99	
2129	ISO14389	0.13		0.34	
2137	IEC62321-8	0.1322		2.13	
2159	ISO14389	0.108		0.59	
2250	ISO14389	0.0871		-0.73	
2258	CPSC-CH-C1001-09.4	0.1232		1.56	
2265	ISO14389	0.0898		-0.56	
2310	CPSC-CH-C1001-09.4	0.10		0.09	
2311 2313	ISO14389 ISO14389	0.1135 0.1090		0.94 0.66	
2314	CPSC-CH-C1001-09.4	0.1090		0.47	
2320	ISO14389	0.083963	С	-0.93	First reported 0.050554
2330	CPSC-CH-C1001-09.4	0.0883		-0.66	
2347	GB/T20388	0.086		-0.80	
2350	CPSC-CH-C1001-09.4	0.1122		0.86	
2352	ISO14389	0.0880		-0.67	
2357	ISO14389	0.0894		-0.59	
2358 2363	CPSC-CH-C1001-09.4 CPSC-CH-C1001-09.4	0.08558		-0.83 -0.83	
2365	ISO14389	0.0855 0.0852		-0.85	
2366	GB/T20388	0.0861		-0.79	
2370	ISO14389	0.0951		-0.22	
2372	CPSC-CH-C1001-09.4	0.100065		0.09	
2373	CPSC-CH-C1001-09.4	0.0904		-0.52	
2375	CPSC-CH-C1001-09.4	0.105		0.40	
2378	GB/T20388	0.09		-0.55	
2379 2380	ISO14389 CPSC-CH-C1001-09.4	0.0886 0.08792		-0.64 -0.68	
2381	CPSC-CH-C1001-09.4	0.08792		-0.62	
2382	ISO14389	0.08782		-0.69	
2386	ISO14389	0.1211		1.42	
2389	CPSC-CH-C1001-09.4	0.10		0.09	
2390	CPSC-CH-C1001-09.4	0.109		0.66	
2426	ISO14389	0.092		-0.42	
2449	CPSC-CH-C1001-09.4	0.1089		0.65	
2453 2462	ISO14389 GB/T20388&ISO14389	0.055 0.083		-2.77 -0.99	
2489	ISO14389	0.108		0.59	
2492	GB/T20388	0.0893		-0.59	
2495	ISO14389	0.1242		1.62	
2532	ISO14389	0.0919		-0.43	
2582	ISO14389	0.07996		-1.18	
2590	ISO14389	0.1069	С	0.52	First reported 0.1518
2591 2644	CPSC-CH-C1001-09.4	0.0887 		-0.63 	
2644 2675	ISO14389	0.120		1.35	
2703	In house	0.120		1.99	
2740	ISO14389	0.126		1.73	
2826	ISO14389	0.0874		-0.71	
2881	In house	0.12		1.35	
2947	In house	0.129		1.92	
2977	ISO14389	0.086275		-0.78	
2979 2982	ISO14389 ISO14389	0.1042 0.08857		0.35 -0.64	
3003	10014003	0.0870		-0.04 -0.74	
3116	ISO14389	0.0884		-0.65	
3154	ISO16181-1	0.085		-0.86	
3166	In house	0.1374		2.46	
3172	ISO8124-6	0.0956		-0.19	
3176	ISO14389	0.1222		1.49	
3199	In house	0.0798		-1.19 0.61	
3210 3237	In house CPSC-CH-C1001-09.4	0.089 0.0914		-0.61 -0.46	
0201	5. 55 511 51001-05.4	0.0017		-0.40	

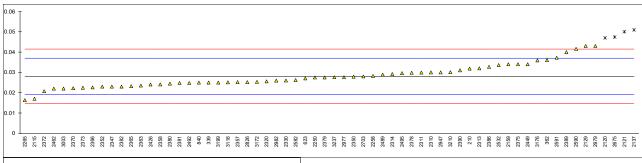


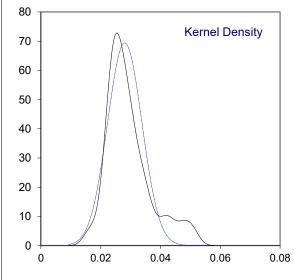


# Determination of DINP - Di-iso-nonyl phthalate (CAS No. 28553-12-0 & 68515-48-0) on sample #22566; results in % M/M

lab	6; results in %M/M	value	mark	z(targ)	remarks
210	ISO14389	0.031866	HIMIT	0.86	i omarko
339	In house	0.025		-0.67	
362	In house	0.036	С	1.78	First reported 0.44
551					•
623	In house	0.027	С	-0.23	First reported 0.104
840	ISO14389	0.025		-0.67	
2115	ISO14389	0.017	D(0.05)	-2.46	
2120	ISO14389	0.047	R(0.05)	4.23	
2121 2129	ISO14389	0.05 0.0430	R(0.05)	4.90 3.34	
2129	IEC62321-8	0.0430	R(0.05)	5.34 5.10	
2159	ISO14389	0.0309	. 1(0.00)	1.33	
2250	ISO14389	0.0275		-0.12	
2258	CPSC-CH-C1001-09.4	0.0282		0.04	
2265	ISO14389	0.0163		-2.61	
2310	CPSC-CH-C1001-09.4	0.03		0.44	
2311	ISO14389	0.0299		0.42	
2313 2314	ISO14389 CPSC-CH-C1001-09.4	0.032 0.0292		0.89 0.26	
2314	ISO14389	0.0292		-0.55	
2330	CPSC-CH-C1001-09.4	0.0260		-0.45	
2347	ISO14389	0.023		-1.12	
2350	CPSC-CH-C1001-09.4	0.0279		-0.03	
2352	ISO14389	0.0230		-1.12	
2357	ISO14389	0.0252		-0.63	
2358	CDSC CH C1001 00 4	0.02408		-0.88 1.01	
2363 2365	CPSC-CH-C1001-09.4 ISO14389	0.0235 0.0232		-1.01 -1.08	
2366	GB/T20388	0.0232		-1.00	
2370	ISO14389	0.0222		-1.30	
2372	CPSC-CH-C1001-09.4	0.020747		-1.62	
2373	CPSC-CH-C1001-09.4	0.0224		-1.25	
2375	CPSC-CH-C1001-09.4	0.034		1.33	
2378	CPSC-CH-C1001-09.4	0.0298		0.40	
2379 2380	EN14372 CPSC-CH-C1001-09.4	0.0275 0.02445		-0.12 -0.80	
2380	CPSC-CH-C1001-09.4 CPSC-CH-C1001-09.4	0.02445		-0.80 -0.72	
2382	ISO14389	0.0248		-0.72 -1.12	
2386	ISO14389	0.0327		1.04	
2389	CPSC-CH-C1001-09.4	0.04		2.67	
2390	CPSC-CH-C1001-09.4	0.031		0.66	
2426	ISO14389	0.024		-0.90	
2449	CPSC-CH-C1001-09.4	0.034		1.33	
2453 2462	GB/T20388&ISO14389	0.022		-1.34	
2489	ISO14389	0.022		0.20	
2492	GB/T20388	0.0248		-0.72	
2495	ISO14389	0.0296		0.35	
2532	ISO14389	0.0336		1.24	
2582	ISO14389	0.02620		-0.41	
2590	ISO14389	0.0416		3.03	
2591 2644	CPSC-CH-C1001-09.4	0.0372		2.05	
2644 2675	ISO14389	0.0474	C,R(0.05)	4.32	First reported 0.123
2703	In house	0.0474	J,11(0.00)	0.00	Thot reported 0.120
2740	ISO14389	<0.03			
2826	ISO14389	0.0252		-0.63	
2881					
2947	In house	0.03		0.44	
2977	ISO14389	0.027603		-0.09	
2979	ISO14389	0.043		3.34	
2982 3003	ISO14389	0.02598 0.0220		-0.46 -1.34	
3116	ISO14389	0.0220		-1.34 -0.65	
3154	.5511000			-0.00	
3166	In house	Not Detected			
3172	ISO8124-6	0.0253		-0.61	
3176	ISO14389	0.0359		1.76	
3199	In house	0.0250		-0.67	
3210	In house	0.0301		0.46	
3237	CPSC-CH-C1001-09.4	0.0276		-0.09	

normality OK n 60 outliers 4 mean (n) 0.02802 st.dev. (n) 0.005752 RSD = 21% R(calc.) 0.01611 st.dev.(iis memo 1701) 0.004483 R(iis memo 1701) 0.01255



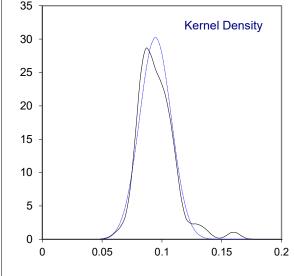


# Determination of DNOP - Di-n-octyl phthalate (CAS No. 117-84-0) on sample #22566; results in %M/M

70 IVI/ IVI	4l d			44	
lab	method	value	mark	z(targ)	remarks
210	ISO14389	0.111429	D(0.04)	1.11	
339	In house	1.11	R(0.01)	67.09	
362	In house	0.086		-0.57	
551	In house	0.006		0.00	
623	In house	0.096		0.09	
840	ISO14389	0.094		-0.04	
2115	ISO14389	0.081		-0.90	
2120	ISO14389	0.124		1.94	
2121 2129	ISO14389	0.11 0.0928		1.02 -0.12	
2129	IEC62321-8	0.0920		1.33	
2159	ISO14389	0.1147		0.56	
2250	ISO14389	0.0891		-0.36	
2258	CPSC-CH-C1001-09.4	0.1061		0.76	
2265	ISO14389	0.1008		0.41	
2310	CPSC-CH-C1001-09.4	0.11		1.02	
2311	ISO14389	0.10699		0.82	
2313	ISO14389	0.1010		0.42	
2314	CPSC-CH-C1001-09.4	0.1021		0.50	
2320	ISO14389	0.077991		-1.10	
2330	CPSC-CH-C1001-09.4	0.0887		-0.39	
2347	ISO14389	0.083		-0.77	
2350	CPSC-CH-C1001-09.4	0.0865		-0.53	
2352	ISO14389	0.0845		-0.67	
2357	ISO14389	0.0850		-0.63	
2358		0.08994		-0.31	
2363	CPSC-CH-C1001-09.4	0.0827		-0.79	
2365	ISO14389	0.0845		-0.67	
2366	GB/T20388	0.0852		-0.62	
2370	ISO14389	0.0902		-0.29	
2372	CPSC-CH-C1001-09.4	0.090609		-0.26	
2373	CPSC-CH-C1001-09.4	0.0821		-0.83	
2375	CPSC-CH-C1001-09.4	0.106		0.75	
2378	CPSC-CH-C1001-09.4	0.0834		-0.74	
2379	EN14372	0.0724		-1.47 0.40	
2380	CPSC-CH-C1001-09.4	0.09312		-0.10	
2381 2382	CPSC-CH-C1001-09.4	0.0931 0.0846		-0.10 -0.66	
2386	ISO14389 ISO14389	0.0040		1.02	
2389	CPSC-CH-C1001-09.4	0.1101		0.36	
2390	CPSC-CH-C1001-09.4	0.078		-1.10	
2426	ISO14389	0.086		-0.57	
2449	CPSC-CH-C1001-09.4	0.0765		-1.20	
2453		0.129		2.27	
2462	GB/T20388&ISO14389	0.082		-0.83	
2489	ISO14389	0.1032		0.57	
2492	GB/T20388	0.0900		-0.30	
2495	ISO14389	0.0827		-0.79	
2532	ISO14389	0.11	С	1.02	First reported 0.138
2582	ISO14389	0.06438		-2.00	
2590	ISO14389	0.0836		-0.73	
2591	CPSC-CH-C1001-09.4	0.1010		0.42	
2644	10011000				
2675	ISO14389	0.0816		-0.86	
2703	In house	0.136		2.74	
2740	ISO14389	0.098		0.23	
2826	ISO14389	0.0907	0.0(0.04)	-0.26	First reported 2.5
2881	In house	0.16	C,R(0.01)	4.32	First reported 2.5
2947	In house	0.097		0.16	
2977 2979	ISO14389 ISO14389	0.097466 0.107	С	0.19 0.82	First reported 0.1404
2979 2982			C	-0.08	First reported 0.1494
3003	ISO14389	0.09334 0.1020		0.49	
3116	ISO14389	0.1020		-0.33	
3154	ISO16181-1	0.0090		0.03	
3166	In house	0.033		1.02	
3172	ISO8124-6	0.0996		0.33	
3176	ISO14389	0.1011		0.43	
3199	In house	0.0903		-0.28	
3210	In house	0.0800		-0.96	
3237	CPSC-CH-C1001-09.4	0.1000		0.36	

normality OK n 67 outliers 2 mean (n) 0.09459 st.dev. (n) 0.013187 RSD = 14% R(calc.) 0.03692 st.dev.(iis memo 1701) 0.015135 R(iis memo 1701) 0.04238





## **APPENDIX 2 Other reported test results**

## Abbreviations of components:

BBP = Benzyl butyl phthalate (CAS No. 85-68-7)

DEHP = Di-(2-ethylhexyl) phthalate (CAS No. 117-81-7)

DBP = Dibutyl phthalate (CAS No. 84-74-2)

DIDP = Di-iso-decyl phthalate (CAS No. 26761-40-0 & 68515-49-1)
DINP = Di-iso-nonyl phthalate (CAS No. 28553-12-0 & 68515-48-0)

DNOP = Di-n-octyl phthalate (CAS No. 117-84-0)

DCHP = Dicyclohexyl phthalate (CAS No. 84-61-7)

DEP = Diethyl phthalate (CAS No. 84-66-2) DMP = Dimethyl phthalate (CAS No. 131-11-3)

DNHP = Di-n-hexyl phthalate (CAS No. 84-75-3)
DIBP = Di-iso-butyl phthalate (CAS No. 84-69-5)

DPHP = Di(2-propylheptyl) phthalate (CAS No. 53306-54-0)

DNPP = Di-n-pentyl phthalate (CAS No. 131-18-0) DUP = Diundecyl phthalate (CAS No. 3648-20-2) DPrP = Di-n-propyl phthalate (CAS No. 131-16-8)

DMEP = Di-(2-methoxyethyl) phthalate (CAS No. 117-82-8)

Other = Total Other Phthalates

## sample #22565; results in %M/M

lab	BBP	DIDP	DINP	DNOP	DCHP	DEP	DMP	DNHP
210								
339	not detected	not detected	not detected	not detected	not detected			not detected
362								
551								
623	0.002	Not detected	0.003	Not detected	Not detected	Not detected	Not detected	Not detected
840	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2115								
2120	< 0.005	< 0.005	< 0.005	< 0.005		< 0.005	< 0.005	< 0.005
2121								
2129								
2137								
2159	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2250								
2258	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2265	< 0.001	< 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	not detected
2311	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2313	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2314								
2320	< 0.003	< 0.005	< 0.005	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003
2330	ND	ND	ND	ND	ND	ND	ND	ND
2347	< 0.003	< 0.005	< 0.005	< 0.003	<0.005			
2350	< 0.015	< 0.015	< 0.015	< 0.015	< 0.015	< 0.015	< 0.015	< 0.015
2352								
2357								
2358	not detected	not analyzed	not detected	not detected	not detected	not analyzed	not analyzed	not detected
2363	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	< 0.005	<0.005
2365	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	< 0.005	<0.005
2366	<0.004	<0.020	<0.015	<0.004	<0.004	<0.004	<0.004	<0.004
2370	<0.00300	<0.00300	<0.00300	<0.00300	<0.00300	<0.00300	<0.00300	<0.00300
2372	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2373	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
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
2380	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2381								
2382	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050
2386	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
2389	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2390	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2426	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2449								
2453								
2462	 Nat data ata d	 Nat data ata d	 Natalataataa	 Nat data ata d	 Natalataataa	 Natalataataa	 Natalataataa	 Natalata atad
2489	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2492	<0.003	<0.01	 <0.01	<0.003	<0.003	<0.003	<0.003	<0.003
2495	<b>~</b> 0.003	<b>~</b> 0.01	<b>\U.U1</b>	<b>~</b> 0.003	<b>~</b> 0.003	<b>~</b> 0.003	<b>~</b> 0.003	<b>~</b> 0.003

lab	BBP	DIDP	DINP	DNOP	DCHP	DEP	DMP	DNHP
2532	Not detected							
2582	Not detected							
2590								
2591	not detected							
2644								
2675								
2703	0.001	Not detected	0.001	Not detected	Not detected	not analyzed	not analyzed	Not detected
2740								
2826	<0.003	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
2881								
2947	not detected							
2977	<0.001	<0.001	<0.001	<0.001	ND	ND	ND	<0.001
2979	0.002	not detected						
2982	not detected							
3003								
3116								
3154								
3166	0.0034							
3172	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005
3176								
3199	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
3210	<0.002	<0.005	<0.005	<0.002	<0.002	<0.002	<0.002	<0.002
3237								

## sample #22565; results in %M/M ---- continued ----

lak	DIDD	DDUD	DNDD	DUD	DD-D	DMED	Othor
lab	DIBP	DPHP	DNPP	DUP	DPrP	DMEP	Other
210							
339	not detected		not detected				
362							
551	 Natalataataa	 N_=4 =  =4==4==1	Not detected	Not detected	 Natalataataat	Not detected	Not doto oto d
623	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
840	not detected	not detected	not detected	not detected	not detected	not detected	
2115							
2120	< 0,005	< 0,005	< 0,005	< 0,005	< 0,005	< 0,005	< 0,005
2121							
2129							
2137	not dotacted	not detected	not detected	not detected	not dotooted	not detected	not detected
2159	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2250 2258	not detected	not detected	not dotacted	not detected	not detected	not dotacted	
2265	< 0.001	< 0.001	not detected < 0.001	< 0.001	< 0.001	not detected < 0,001	not detected
2205	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2310	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2313	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2314							
2320	< 0.003	<0.003	< 0.003	< 0.003	<0.003	< 0.003	< 0.003
2330	ND	ND	ND	ND	ND	ND	ND
2347	< 0.003		< 0.003			< 0.003	
2350	< 0.015	< 0.015	< 0.015	< 0.015	< 0.015	< 0.015	< 0.015
2352							
2357							
2358	not detected	not analyzed	not detected	not analyzed	not analyzed	not analyzed	not analyzed
2363	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2365	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
2366	<0.004	< 0.004	<0.004	<0.004	< 0.004	<0.004	not analysed
2370	< 0.00300	< 0.00300	< 0.00300	< 0.00300	< 0.00300	< 0.00300	< 0.00300
2372	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.214662
2373	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2375							
2378	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	na
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	0.2104
2380	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2381							
2382	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050
2386	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
2389	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2390	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2426	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2449							
2453							
2462		Night details	Night det	Nick detected	Nick detected	 Ni-6 defende	Not detected
2489	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2492							
2495	<0.003	Not detected	< 0.003	Not detected	 Nat data ata d	<0.003	 Niat data ata d
2532	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected

lab	DIBP	DPHP	DNPP	DUP	DPrP	DMEP	Other
2582	Not Detected		Not detected	Not detected	Not detected	Not detected	
2590							
2591	not detected		not detected		not detected	not detected	
2644							
2675							
2703	Not detected	not analyzed	Not detected	not analyzed	not analyzed	Not detected	not analyzed
2740							
2826	<0.003	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
2881							
2947	not detected	not tested	not detected				
2977	<0.001	ND	<0.001	<0.001	ND	<0.001	ND
2979	not detected						
2982	not detected						
3003							
3116							
3154							
3166	Not Detected						
3172	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	
3176							
3199	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
3210	<0.002		<0.002	<0.002	<0.002	<0.002	<0.002
3237							

sample #22566; results in %M/M

lab	BBP	DEHP	DBP	DIDP	DCHP	DEP	DMP	DNHP
210								
339	not detected	not detected	not detected	not detected	not detected			not detected
362								
551								
623	Not detected	Not detected	0.005	Not detected				
840	not detected	not detected	0.008	not detected				
2115								
2120	< 0,005	< 0,005	< 0,005	< 0,005		< 0,005	< 0,005	< 0,005
2121			<0.006					
2129								
2137	not dotootod	not dotootod	0.0126	not dotootod	not dotacted	not dotacted	not dotacted	not detected
2159 2250	not detected	not detected	0.008	not detected				
2258	not detected	not detected	0.0073	not detected				
2265	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
2310	not detected	not detected	0.01	not detected				
2311	Not detected	Not detected	0.0078	Not detected				
2313	Not detected	Not detected	0.0093	Not detected				
2314			0.0091					
2320	<0.003	<0.003	0.005514	<0.005	<0.003	<0.003	<0.003	<0.003
2330	ND	ND	ND	ND	ND	ND	ND	ND
2347	< 0.005	<0.005	< 0.005	< 0.005	<0.005	< 0.005	< 0.005	< 0.005
2350 2352	< 0.015 	< 0.015 	< 0.015 	< 0.015 	< 0.015 	< 0.015 	< 0.015 	< 0.015 
2352								
2357	not detected	not detected	not detected	not analyzed	not detected	not analyzed	not analyzed	not detected
2363	< 0.005	<0.005	< 0.005	<0.005	< 0.005	<0.005	<0.005	< 0.005
2365	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2366	<0.004	<0.004	<0.004	<0.004	<0.004	<0.004	<0.004	<0.004
2370	<0.00300	<0.00300	<0.00300	<0.00300	<0.00300	<0.00300	<0.00300	<0.00300
2372	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2373	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2375			0.009					
2378	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2379 2380	Not detected <0.005	Not detected <0.005	0.0041 <0.005	Not detected <0.005				
2381		~0.003 			<0.003 		~0.003 	
2382	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050
2386	<0.005	<0,005	<0,005	<0.005	<0.005	<0.005	<0.005	<0.005
2389	Not detected	Not detected	0.01	Not detected				
2390	Not detected	Not detected	0.01	Not detected				
2426	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2449			0.009					
2453			0.007					
2462 2489	Not detected	Not detected	0.009	Not detected				
2492	Not detected	Not detected	0.008	Not detected				
2495	<0.003	<0.003	0.003054	<0.003	<0.003	<0.003	<0.003	<0.003
2532	Not detected	Not detected	0.0103	Not detected				
2582	Not detected	Not detected	0.00534	Not detected				
2590			0.00173					
2591	not detected	not detected	0.0055	not detected				
2644								
2675			0.0112					
2703	Not detected	Not detected	0.002	Not detected	Not detected	not analyzed	not analyzed	Not detected
2740 2826		<0.003	 <0.002	 <0.01	 <0.01	<0.01	 <0.01	<0.01
2881	<0.003	<0.003	<0.003	<0.01 	<0.01 	<0.01 	<0.01 	<0.01 
2947	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2977	< 0.001	<0.001	0.010206	< 0.001	ND	ND	ND	< 0.001
2979	0.0013	not detected	0.0013	not detected				
2982	not detected	not detected	0.00713	not detected				
3003								
3116								
3154			0.0048					
3166	Not detected	Not detected	0.0055	Not detected				
3172	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005
3176 3199	 <0.005	 <0.005	0.0058	 <0.005	 <0.005	 <0.005	<0.005	 <0.005
3199	<0.005 <0.002	<0.005 <0.002	0.0139 0.0072	<0.005 <0.005	<0.005 <0.002	<0.005 <0.002	<0.005 <0.002	<0.005 <0.002
3237	<0.002 	<0.002 	0.0072	<0.005 	<0.002 	<0.002 	<0.002 	
3231			3.0001					

sample #22566; results in %M/M ---- continued ----

Sample	#22566; resu	its in %ivi/ivi	continuea				
lab	DIBP	DPHP	DNPP	DUP	DPrP	DMEP	Other
210							
339	not detected		not detected				
362							
551							
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	
2115							
2120	< 0,005	< 0,005	< 0,005	< 0,005	< 0,005	< 0,005	< 0,005
2121							
2129							
2137							
2159	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2250							
2258	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2265	< 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
2313	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2314							
2320	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003	< 0.003
2330	ND	ND	ND	ND	ND	ND	ND
2347	< 0.005	< 0.005	<0.005	<0.005	<0.005	<0.005	
2350	< 0.015	< 0.015	< 0.005	< 0.015	< 0.015	< 0.015	< 0.015
2352							
2357							
2358	not detected	not analyzed	not detected	not analyzed	not analyzed	not analyzed	not analyzed
2363	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2365	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2366	<0.004	<0.004	<0.004	<0.004	<0.004	<0.004	not analysed
2370	<0.00300	<0.00300	<0.00300	<0.00300	<0.00300	<0.00300	<0.00300
2372	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.111356
2373	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2375							
2378	<0.005	< 0.005	< 0.005	<0.005	< 0.005	< 0.005	na
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	0.1041
2380	< 0.005	< 0.005	<0.005	<0.005	<0.005	< 0.005	<0.005
2381							
2382	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050	<0.0050
2386	<0.005	<0.005	<0,005	<0,005	<0,005	<0,005	<0,005
2389						·	·
2390	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2426	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2449							
2453							
2462							
2489	Not Detected	Not Detected	Not Detected	Not Detected	Not detected	Not detected	Not detected
2492							
2495	<0.003		<0.003			<0.003	
2532	Not Detected	Not Detected	Not Detected	Not Detected	Not detected	Not detected	Not detected
2582	Not Detected	Not Analysed	Not detected	Not detected	Not detected	Not detected	
2590							
2591	not detected		not detected		not detected	not detected	
2644							
2675							
2703	Not detected	not analyzed	Not detected	not analyzed	not analyzed	Not detected	not analyzed
2740							
2826	<0.003	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
2881	not detected	not tosted	not detected	not dotacted	not detected	not detected	not detected
2947		not tested	not detected				
2977	< 0.001	ND	<0.001	< 0.001	ND	< 0.001	ND
2979	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2982	not detected	not detected	not detected	not detected	not detected	not detected	not detected
3003							
3116							
3154							
3166	Not Detected						
3172	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	< 0.0005	
3176							
3199	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
3210	<0.002	-0.000	<0.002	<0.002	<0.002	<0.002	<0.002
3237							
3231							

## **APPENDIX 3 Analytical details**

lab	ISO/IEC 17025 accr.	Sample preparation	Sample intake used (grams)	Release/ extraction technique	Extraction solvent	Extraction time (min)	Extraction temperature (°C)
210	No	Further cut					
339	No	Used as received	0.5g	Ultrasonic	thf and hexane	60 min	60°C
362	Yes	Used as received	0.3	Ultrasonic	THF/Hexane	60	60°C
551							
623	Yes	Further cut	0.1 gram	Ultrasonic	THF Hexane	60	60
840	Yes	Further cut	0.5	Ultrasonic	thf-hexan	1hour	60
2115	Yes	Used as received	0.3g	Ultrasonic	THF/Hexane	60 min	60°C
2120	Yes	Used as received	0,3 g	Ultrasonic	THF	60 min	60 °C
2121	Yes	Used as received	Approx. 0,3g	Ultrasonic	THF/Hexane	60 min	60°C
2129	Yes	Further cut	0,5	Ultrasonic	THF	60	60°C
2137	Yes	Used as received	2.5	Ultrasonic	THF/ACN	60	40
2159	Yes	Further cut	0.5	Ultrasonic	THF / ACN (1:2)	60	60
2250	Yes	Used as received	0,3	Ultrasonic	THF/Acetonitril (1:2)	60	60
2258	Yes	Used as received	0.0641 / 0.0609	Ultrasonic	THF/ACN	120 min	40
2265	Yes	Used as received	0,3g	Ultrasonic	THF / Hexan	60	60
2310	Yes	Further cut	0.1 gram	Ultrasonic	THF/Hexane	60	60
2311			0.3	Ultrasonic	THF + Hexane	60	50
2313	Yes	Further cut	0.5g	Ultrasonic	THF / hexane	60 min	60 °C
2314	Yes	Further cut	0.5 gm	Ultrasonic	THF/N-Hexane	60	60
2320	Yes	Further cut	0.5g	Ultrasonic	THF:Hexane	60min	60°C
2330	Yes	Further cut	0.05g	Ultrasonic	THF and Hexane (1:2)	30 min	40°C
2347		Further cut	0.1g	Ultrasonic		60±5min	60±5°C
2350	Yes	Further cut	0.5g	Ultrasonic	THF+ACN	2h 30min	60 °C
2352	Yes	Further cut	0.3g	Ultrasonic	THF+Hexane	60mins	60 °C
2357			-				
2358	Yes	Llood on received	0.05 gram	Mechanical	THF and hexane	2.5 hours	room tomn
2363	Yes	Used as received Further cut	0.05 gram	Shaking Ultrasonic	THE		room temp 60°C
2365	Yes	Used as received	0.3a	Ultrasonic	THF:HEX=1:2	60 min 60min	60°C
2366			J	Ultrasonic	THF and hexane	60	60 C
2370	Yes Yes	Further cut	0.1gram 0.3 g		THE	60 min	60°C
2372	Yes	Further cut Further cut	0.5 g 0.5g	Ultrasonic Ultrasonic	10 mL	60 min	
2372	Yes	Further cut	· ·	Ultrasonic	THF	60MIN	room temp 60±5
2375	Yes	Further cut	0.1g 0.05g	Mechanical Shaking	THF	30 min	room temp
2378	Yes	Used as received	0.3G	Ultrasonic	n-hexan/thf	60	60
2379	Yes	Further cut	0.3 g	Ultrasonic	THF : Hexane	60 min	60 C
2380	Yes	Further cut	0.1 g	Ultrasonic	THF (Tetrahydrofuran)	60 Min	60 °C
2381	Yes	Further cut	.1 gm per trial.	Ultrasonic	THF and hexane	60 minute.	60.
2382	Yes	Further cut	0.1g	Ultrasonic	THF and hexane	60min	60°C
2386	Yes	Further cut	0,5 g	Ultrasonic	THF and hexane	60 min	60 °C
2389	Yes	Further cut	0.5 gram	Ultrasonic	Tetra Hydro Furan	60 min	60C
2390	Yes	Further cut	0.1 g	Ultrasonic	THF	60 minute	60°C
2426	Yes	Further cut	0.25g	Ultrasonic	THF and hexane	30 min	room temp
2449	Yes	Further cut		Ultrasonic	. The direction of the control	60 min	60°C
2449	Yes	Used as received	60-300mg	Ultrasonic	THF/n-hex (1:2)	60	60 60
2462	Yes	Used as received	0.5g	Ultrasonic	Tetrahydrofuran	60±5min	60±5°C
2489	Yes	Further cut	· ·	Ultrasonic	THF/n-Hexane	60 min	
			0.3012g/0.3006g				60 degree
2492	Yes	Used as received	0.6g	Ultrasonic	THF:Hexane (1:2)	60 mins	60°C

lab	ISO/IEC 17025 accr.	Sample preparation	Sample intake used (grams)	Release/ extraction technique	Extraction solvent	Extraction time (min)	Extraction temperature (°C)
2495	Yes	Used as received	0.15	Ultrasonic	THF	60	60
2532	Yes	Further cut	0.3gram	Ultrasonic	THF n-Hexane	60 min	60 °C
2582	Yes	Further cut	0.6003 / 0.6011 g	Ultrasonic	Tetrahydrofuran	60 min	60
2590	Yes	Used as received	0.3	Ultrasonic	thf_hex 1:2	60 min	60°c
2591	Yes	Further cut	0.2 grams	Ultrasonic	THF/n-Hexane		
2644							
2675	No	Further cut	0.5 per extraction	Ultrasonic	THF	60	60
2703	Yes	Further cut	0.5g	Ultrasonic	THF/Hexane	150 min	60°C
2740	Yes	Used as received	approx. 0,5g	Ultrasonic	THF and Hexan	60min	60°C
2826	Yes	Used as received	0.3g	Ultrasonic	Tetrahydrofuran	60mins	60°C
2881	Yes	Used as received	0.5g	Ultrasonic	hexane	1h	room temp
2947	No	Used as received	0.5	Ultrasonic	THF and Hexan	60	60
2977	No	Used as received	3 g	Ultrasonic	THF	60 min	60
2979	No	Further cut	0.3 g	Ultrasonic	THF:Hexane (1:2)	1 hr	60 C
2982	Yes	Used as received	0.5g	Ultrasonic	THF and Hexan	60±5	60±5
3003							
3116	Yes	Used as received	0.6g	Ultrasonic	THF / ACN (1:2)	60 min	60 °C
3154	Yes	Used as received	0,5	Ultrasonic	Toluene	60	60
3166	Yes	Further cut	1.5 grams	extraction	Methylene chloride	60 min	22 C
3172	Yes						
3176	Yes	Used as received	0,3	Ultrasonic	THF / ACN	60	60
3199		Used as received	0.3 grams	Ultrasonic	THF / ACN	120 min	40
3210	Yes	Used as received	1g	Ultrasonic	Toluene	60	60°C
3237	Yes	Used as received	0,25	Ultrasonic	THF/hexan	30	40

## **APPENDIX 4**

## Number of participants per country

- 1 lab in AUSTRIA
- 4 labs in BANGLADESH
- 1 lab in BRAZIL
- 1 lab in BULGARIA
- 1 lab in CAMBODIA
- 1 lab in EGYPT
- 3 labs in FRANCE
- 7 labs in GERMANY
- 1 lab in GUATEMALA
- 4 labs in HONG KONG
- 6 labs in INDIA
- 1 lab in INDONESIA
- 6 labs in ITALY
- 2 labs in KOREA, Republic of
- 1 lab in MOROCCO
- 10 labs in P.R. of CHINA
- 4 labs in PAKISTAN
- 1 lab in POLAND
- 2 labs in PORTUGAL
- 1 lab in SPAIN
- 2 labs in SRI LANKA
- 2 labs in TAIWAN
- 1 lab in THAILAND
- 4 labs in TURKEY
- 2 labs 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

 $\begin{array}{ll} D(0.01) &= \text{outlier in Dixon's outlier test} \\ D(0.05) &= \text{straggler in Dixon's outlier test} \\ G(0.01) &= \text{outlier in Grubbs' outlier test} \\ G(0.05) &= \text{straggler in Grubbs' outlier test} \\ DG(0.01) &= \text{outlier in Double Grubbs' outlier test} \\ DG(0.05) &= \text{straggler in Double Grubbs' outlier test} \\ \end{array}$ 

R(0.01) = outlier in Rosner's outlier test R(0.05) = straggler in Rosner's outlier test

W = test result withdrawn on request of participant ex = test result excluded from statistical evaluation

n.a. = not applicable
n.e. = not evaluated
n.d. = not detected
fr. = first reported

#### Literature

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