Results of Proficiency Test
Phthalates in Leather
May 2020

CONTENTS

Appendices:

1   INTRODUCTION

The determination of Phthalates on leather is known to give problems with the comparability of laboratory results. However, no appropriate leather reference materials are yet available. As an alternative, participation in a proficiency test may enable laboratories to check and improve their performance. Therefore, on request of several participants, the Institute for Interlaboratory Studies organize an interlaboratory study for the determination of Phthalates in leather since 2017 every year. During the annual testing program of 2019/2020 it was decided to continue the proficiency test for the analysis of Phthalates in Leather.

In this interlaboratory study 44 laboratories in 22 countries registered. See appendix 4 for the number of participating laboratories per country. In this report the results of this proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2   SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send to each participant two different leather samples of 3 grams each labelled #20600 and #20601. 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 can be downloaded via the FAQ page of the iis website www.iisnl.com.

2.3   CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4   SAMPLES

A batch of black leather was made positive on Di-(2-ethylhexyl) phthalate (DEHP) and Diethyl phthalate (DEP). After grinding and homogenization 75 small bags were filled with approximately 3 grams each and labelled #20600.The homogeneity of the subsamples was checked by the determination of DEHP and DEP in accordance with test method ISO/TS16181 on eight stratified randomly selected subsamples.

	DEHP in %M/M	DEP in %M/M
Sample #20600-1	0.1326	0.0696
Sample #20600-2	0.1350	0.0716
Sample #20600-3	0.1267	0.0670
Sample #20600-4	0.1288	0.0673
Sample #20600-5	0.1259	0.0699
Sample #20600-6	0.1276	0.0677
Sample #20600-7	0.1436	0.0744
Sample #20600-8	0.1310	0.0679

Table 1: homogeneity test results of the subsamples #20600

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

	DEHP in %M/M	DEP in %M/M
r (observed)	0.0163	0.0071
reference method	iis memo 1701*)	iis memo 1701*)
0.3 * R (reference method)	0.0177	0.0093

Table 2: evaluation of repeatabilities of the subsamples #20600

*) see literature 15

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

A second batch of black leather was made positive on Benzyl butyl phthalate (BBP) and Dicyclohexyl phthalate (DCHP). After grinding and homogenization 80 small bags were filled with approximately 3 grams each and labelled #20601.The homogeneity of the subsamples was checked by the determination of BBP and DCHP in accordance with test method ISO/TS16181 on eight stratified randomly selected subsamples.

	BBP in %M/M	DCHP in %M/M
Sample #20601-1	0.1554	0.1049
Sample #20601-2	0.1510	0.1057
Sample #20601-3	0.1610	0.1106
Sample #20601-4	0.1638	0.1104
Sample #20601-5	0.1692	0.1155
Sample #20601-6	0.1566	0.1087
Sample #20601-7	0.1621	0.1088
Sample #20601-8	0.1632	0.1110

Table 3: homogeneity test results of the subsamples #20601

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	DCHP in %M/M
r (observed)	0.0160	0.0093
reference method	iis memo 1701*)	iis memo 1701*)
0.3*R (reference method)	0.0215	0.0147

Table 4: evaluation of repeatabilities of the subsamples #20601

*) see literature 15

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

To each of the participating laboratories one sample of approximately 3 grams, labelled #20600 and one sample of approximately 3 grams, labelled #20601 was sent on April 16, 2020.

2.5 ANALYZES

The participants were requested to determine on both samples #20600 and #20601, fourteen individual Phthalates (see appendices 1 and 2) and eventually other Phthalates when identified. It was also requested to report if the laboratory was accredited for the requested components and some method details were asked.

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 can not be used for meaningful statistical evaluation. Futhermore, to ensure the homogeneity it was requested to not use less than 0.5 gram per determination.

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

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

Directly after the deadline a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or corrected test results are used for the data analysis and the original results are placed under 'Remarks' in the result tables in appendices 1 or 2. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organisation of this proficiency test wast 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.

According to ISO5725 the original test results per determination were submitted subsequently to Dixon's, Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

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

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are on the X-axis.

The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. The Kernel Density Graph is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the target reproducibility by division with 2.8. In case no literature reproducibility was available, other target values are used.

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

The z-scores were calculated according to:

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

The $z_{(target)}$ scores are listed in the test result tables of 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 severe problems were encountered with the dispatch of the samples. Four participants reported the test results after the final reporting date and two other participants did not report at all.
Finally, 42 laboratories reported 180 numerical test results. Observed were 5 outlying test results, which is 2.8%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

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

4.1   EVALUATION PER SAMPLE AND PER COMPONENT

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

For many years iis organizes PTs on Phthalates in Polymers. In 2017 it was decided to use the iis PT data gathered since 2010 to estimate a more realistic target reproducibility in polymers (see iis memo 1701, lit. 15). The target reproducibility was estimated by the relative standard deviation (16%) of the mean multiplied by 2.8.
It should be noted that the iis-memo 1701 is based on previous iis PTs of Phthalates in Polymers and not based on iis PTs of Phthalates in Leather. Because only data of three PTs on Phthalates in Leather are available and test method ISO/TS16181:11 provides a variety of precision data it was therefore decided to use the estimated iis target reproducibility for the polymer PT also for the Leather PT.
NB. The target reproducibility from iis memo 1701 has also been used for the textile PT on Phthalates.

Sample #20600

DEHP: The determination was problematic at the level of 0.12 %M/M. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the target reproducibility derived from the iis-memo 1701. See for more discussion paragraph 5.

DEP: The determination was problematic at the level of 0.04 %M/M. No statistical outliers were observed. The calculated reproducibility is not in agreement with the target reproducibility derived from the iis-memo 1701. See for more discussion paragraph 5.

For all other Phthalates the group of participants agreed on a concentration below 0.01 %M/M. Therefore, no significant conclusions were drawn for these Phthalates, see appendix 2 for the reported test results.

Sample #20601

BBP: The determination was problematic at the level of 0.14 %M/M. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the target reproducibility derived from the iis-memo 1701. See for more discussion paragraph 5.

DCHP: The determination was not problematic at the level of 0.10 %M/M. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in full agreement with the target reproducibility derived from the iis-memo 1701. See for more discussion paragraph 5.

DEP: The majority of the group was able to detect DEP. However, the determination was problematic at the level of 0.008 %M/M. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the target reproducibility derived from the iis-memo 1701. See for more discussion paragraph 5.

For all other Phthalates the group of participants agreed on a concentration below 0.01 %M/M. Therefore, no significant conclusions were drawn for these Phthalates, 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 averages, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from reference test method are presented in the next table.

Component	unit	n	average	2.8 * sd	R(target)
DEHP	%M/M	41	0.123	0.085	0.055
DEP	%M/M	35	0.044	0.033	0.020

Table 5: reproducibilities of tests for sample #20600

Component	unit	n	average	2.8 * sd	R(target)
BBP	%M/M	40	0.135	0.082	0.061
DCHP	%M/M	35	0.096	0.042	0.043
DEP	%M/M	24	0.008	0.005	0.004

Table 6: reproducibilities of tests for sample #20601

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

4.3   COMPARISON OF THE PROFICIENCY TEST OF MAY 2020 WITH PREVIOUS PTS

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

Table 7: comparison with previous proficiency tests

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

Component	May 2020	May 2019	April 2018	April 2017	iis memo 1701	ISO/TS 16181
BBP	22%	n.e.	16%	13%	16%	10%
DEHP	25%	n.e.	n.e.	n.e.	16%	10%
DBP	n.e.	n.e.	n.e.	18%	16%	10%
DIDP	n.e.	21%	n.e.	n.e.	16%	10%
DINP	n.e.	29%	n.e.	n.e.	16%	10%
DCHP	16%	n.e.	21%	n.e.	16%	10%
DEP	23-27%	n.e.	n.e.	n.e.	16%	10%
DMP	n.e.	33-46%	n.e.	n.e.	16%	10%
DNHP	n.e.	14%	n.e.	n.e.	16%	10%
DIBP	n.e.	n.e.	n.e.	16%	16%	10%

Table 8: development of uncertainties over the years

The uncertainty (RSD) of the PT is in line with previous PTS and in general higher than the target method.

4.4   EVALUATION OF THE ANALYTICAL DETAILS

For this PT some analytical details were requested. The answers are given in appendix 3.
Based on the answers given by the participants the following can be summarized:
- About 83% of the reporting participants mentioned that they are accredited for the
  determination of Phthalates in Leather.
- About 45% of the reporting participants used a test portion between 0.5 and 1 grams. About
  26% used less sample material and about 14% used a sample intake of 2 – 3 grams.
- About 76% of the reporting participants used an extraction time of 60 minutes.
- About 73% of the reporting participants used an extraction temperature of 50°C or 60°C.
- About 29% of the reporting laboratories used Hexane/Acetone as solvent mixture to release
  the Phthalates. Another part of the group (45%) used THF or mixtures with THF 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. Method ISO/TS16181:11 describes a sample intake of 2
grams. Test method CPSC-CH-C1001-09.3 mentions an intake of only 0.05 gram. However,
this test method describes also that 10 mL of THF can be added for every 0.1 gram of extra
sample intake.

When the test results of participants with THF (or THF mixtures) and Hexane/Acetone are
evaluated separately (see appendix 1 for the results), the calculated reproducibilities are
slightly smaller or the same compared to the whole group. The use of different solvent
mixtures to release the Phthalates do not have a significant effect on the reproducibility nor
on the released Phthalates.

Sample #20601 was also used in a previous iis PT iis18A09 as sample #18555. The results
found in both PTs are in line. In the current PT of 2020 a part of the group detected also
DEP. This component was not observed in the 2018 PT.

Component	Sample #20601				Sample #18555			
	unit	n	average	R(calc)	unit	n	average	R(calc)
BBP	%M/M	40	0.135	0.082	%M/M	63	0.150	0.066
DCHP	%M/M	35	0.095	0.042	%M/M	58	0.100	0.057
DEP	%M/M	24	0.008	0.005	%M/M	33	<0.005	n.e.

Table 9: comparison of sample #20601 with #18555

5   DISCUSSION

In this proficiency test for the determination of Phthalates in Leather, it was noticed that the majority of the participants were able to detect the Phthalates present in sample #20600 and sample #20601. Regretfully, four of the five observed reproducibilities were not in agreement with the target reproducibility mentioned in iis memo 1701.

In this PT, the average of the homogeneity test results is not in line with the average (consensus value) from the PT results. There are several reasons for this. First, the goal of the homogeneity testing is different from the goal of the evaluation of the reported PT results. In order to prove the homogeneity of the PT samples, a test method is selected with a high precision (smallest variation). The accuracy (trueness) of the test method is less relevant. Secondly, the homogeneity testing is done by one laboratory only. The test results of this ISO/IEC17025 accredited laboratory will have a bias (systematic deviation) depending on the test method used. The desire to detect small variations between the PT samples leads to the use of a sensitive test method with high precision, which may be a test method with significant bias.
Also, each test result reported by the laboratories that participate in the PT will have a bias. However, some will have a positive bias and others a negative bias. These different biases compensate each other in the PT average (consensus value). Therefore, the PT consensus value may deviate from the average of the homogeneity test. At the same time the accuracy of the PT consensus value is more reliable than the accuracy of the average of the homogeneity test.

6   CONCLUSION

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

APPENDIX 1

Determination of DEHP - Di-(2-ethylhexyl) phthalate on sample #20600; results in %M/M

lab	method	value	mark	z(targ)	remarks
210	ISO14389	0.1122		-0.56	
348	CPSC-CH-C1001-09.4	0.1185		-0.24	
362	ISO14389	0.0014	R(0.05)	-6.18	
551	In house	0.09684	C	-1.34	First reported 0.8986
622	ISO/TS 16181	0.0328	C	-4.59	First reported 0.0442
623	ISO14389	0.1603		1.88	
840	ISO14389	0.1434		1.02	
2115	ISO14389	0.173		2.52	
2129	In house	0.132		0.44	
2137	KS M1991	0.11		-0.67	
2250	ISO14389	0.1153		-0.40	
2255	ISO/TS 16181	0.1235		0.01	
2267		-----		-----	
2347	ISO/TS 16181	0.1165		-0.34	
2352	ISO/TS 16181	0.1184		-0.25	
2358	ISO/TS 16181	0.1384		0.77	
2363	ISO14389	0.116		-0.37	
2366	ISO14389	0.117		-0.32	
2370	CPSC-CH-C1001-09.4	0.118		-0.27	
2375	ISO/TS 16181	0.152		1.46	
2378	ISO/TS 16181	0.1174		-0.30	
2379	ISO/TS 16181	0.1153		-0.40	
2386	ISO/TS 16181	0.11515		-0.41	
2390	ISO14389	0.10557		-0.90	
2449	CPSC-CH-C1001-09.4	0.058633	C	-3.28	First reported 586.33 %M/M
2455		-----		-----	
2489	ISO14389	0.1874		3.25	
2499	ISO/TS 16181	0.0888		-1.75	
2511	ISO14389	0.141		0.90	
2514	ISO/TS 16181	0.123		-0.01	
2532	ISO/TS 16181	0.1435		1.03	
2563	ISO14389	0.11		-0.67	
2590	ISO/TS 16181	0.0700		-2.70	
2695	ISO/TS 16181	0.12142		-0.09	
2736	In house	0.127		0.19	
2773	ISO/TS 16181	0.1450		1.10	
2789	ISO/TS 16181	0.0861		-1.88	
2881	In house	0.153		1.51	
3154	In house	0.141		0.90	
3172	ISO/TS 16181	0.1455		1.13	
3176	ISO14389	0.1862		3.19	
3197	ISO/TS 16181	0.14857	C	1.28	First reported 1485.7 %M/M
3210	In house	0.1176		-0.29	
3243	In house	0.11155		-0.59	

					Only THF used:	Only Hexane/Acetone used:
	normality	suspect			OK	not OK
	n	41			18	15
	outliers	1			1	0
	mean (n)	0.12324			0.13160	0.11642466
	st.dev. (n)	0.030358	RSD=25%		0.031789	0.030027
	R(calc.)	0.08500			0.08901	0.08408
	st.dev.(iis memo 1701)	0.019718			0.021056	0.018628
	R(iis memo 1701)	0.05521			0.05896	0.05216
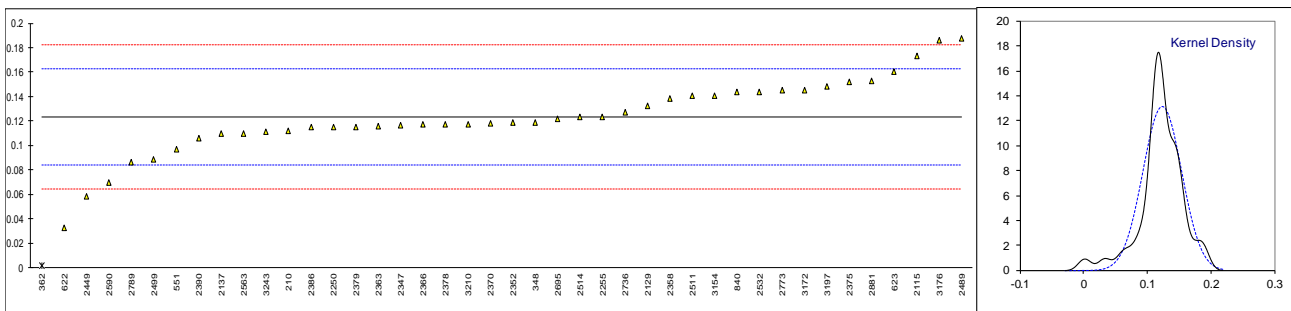
Determination of DEP – Diethyl phthalate on sample #20600; results in %M/M

lab	method	value	mark	z(targ)	remarks
210	ISO14389	0.049		0.72	
348	CPSC-CH-C1001-09.4	0.0526		1.24	
362		-----		-----	
551	In house	0.03233	C	-1.65	First reported 0.8139
622		-----		-----	
623	ISO14389	0.0230		-2.98	
840	ISO14389	0.036		-1.13	
2115	ISO14389	0.049		0.72	
2129	In house	0.0565		1.79	
2137	KS M1991	0.04		-0.56	
2250	ISO14389	0.0421		-0.26	
2255	ISO/TS 16181	0.0475		0.51	
2267		-----		-----	
2347	ISO/TS 16181	0.0372		-0.96	
2352	ISO/TS 16181	0.0435		-0.06	
2358	ISO/TS 16181	N/A		-----	
2363	ISO14389	0.041		-0.41	
2366	ISO14389	0.0437		-0.03	
2370	CPSC-CH-C1001-09.4	0.0360		-1.13	
2375	ISO/TS 16181	0.046		0.30	
2378	ISO/TS 16181	0.0430		-0.13	
2379	ISO/TS 16181	0.0309		-1.85	
2386	ISO/TS 16181	0.05958		2.23	
2390	ISO14389	0.0296		-2.04	
2449		-----		-----	
2455		-----		-----	
2489	ISO14389	0.0455		0.23	
2499		-----		-----	
2511		-----		-----	
2514	ISO/TS 16181	0.047		0.44	
2532	ISO/TS 16181	0.047		0.44	
2563	ISO14389	0.024		-2.83	
2590	ISO/TS 16181	0.0520		1.15	
2695	ISO/TS 16181	0.04676		0.41	
2736	In house	0.0294		-2.07	
2773	ISO/TS 16181	0.0480		0.58	
2789		-----		-----	
2881	In house	0.046		0.30	
3154	In house	0.0652		3.03	
3172	ISO/TS 16181	0.0473		0.48	
3176	ISO14389	0.0586		2.09	
3197	ISO/TS 16181	0.04862	C	0.67	First reported 486.2 %M/M
3210	In house	0.0739		4.27	
3243	In house	0.01917		-3.52	

					Only THF used:	Only Hexane/Acetone used:
	normality	OK			OK	not OK
	n	35			17	11
	outliers	0			0	0
	mean (n)	0.04391			0.04131	0.04829
	st.dev. (n)	0.011669	RSD=27%		0.010586	0.010740
	R(calc.)	0.03267			0.02964	0.03007
	st.dev.(iis memo 1701)	0.007026			0.006610	0.007726
	R(iis memo 1701)	0.01967			0.01851	0.02163
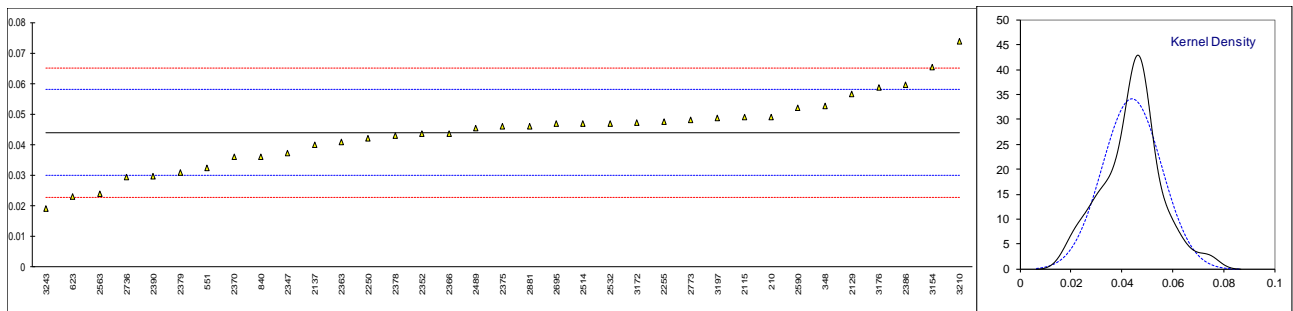
Determination of BBP – Benzyl butyl phthalate on sample #20601; results in %M/M

lab	method	value	mark	z(targ)	remarks
210	ISO14389	0.1486		0.61	
348	CPSC-CH-C1001-09.4	0.1387		0.15	
362	ISO14389	0.0011	R(0.05)	-6.20	
551	In house	0.07082	C	-2.98	First reported 0.8342
622	ISO/TS 16181	0.0248	C,R(0.05)	-5.11	First reported 0.0376
623	ISO14389	0.1681		1.51	
840		0.1768		1.91	
2115	ISO14389	0.165		1.37	
2129	In house	0.143		0.35	
2137	KS M1991	0.11		-1.17	
2250	ISO14389	0.1518		0.76	
2255	ISO/TS 16181	0.152		0.77	
2267		-----		-----	
2347	CPSC-CH-C1001-09.4	0.1393		0.18	
2352	ISO/TS 16181	0.1388		0.16	
2358	ISO/TS 16181	0.1630		1.27	
2363	ISO14389	0.140		0.21	
2366	ISO14389	0.145		0.44	
2370	CPSC-CH-C1001-09.4	0.154		0.86	
2375	ISO/TS 16181	0.158		1.04	
2378	ISO/TS 16181	0.1343		-0.05	
2379	ISO/TS 16181	0.1305		-0.23	
2386	ISO/TS 16181	0.12086		-0.67	
2390	ISO14389	0.1178		-0.81	
2449	CPSC-CH-C1001-09.4	0.073057	C	-2.88	First reported 730.57 %M/M
2455		-----		-----	
2489	ISO14389	0.1544		0.88	
2499	ISO/TS 16181	0.0881		-2.18	
2511	ISO14389	0.132		-0.16	
2514	ISO/TS 16181	0.152		0.77	
2532	ISO/TS 16181	0.1659		1.41	
2563	ISO14389	0.1		-1.63	
2590	ISO/TS 16181	0.088	C	-2.19	First reported 0.062
2695		0.14019		0.22	
2736	In house	0.134		-0.07	
2773	ISO/TS 16181	0.1270		-0.39	
2789	ISO/TS 16181	0.0631		-3.34	
2881	In house	0.160	C	1.13	First reported 0.25
3154	In house	0.1948		2.74	
3172	ISO/TS 16181	0.1350		-0.02	
3176	ISO14389	0.1270		-0.39	
3197	ISO/TS 16181	0.16669	C	1.44	First reported 1666.9 %M/M
3210		0.1271		-0.38	
3243	In house	0.12193		-0.62	

					Only THF used:	Only Hexane/Acetone used:
	normality	OK			suspect	suspect
	n	40			18	14
	outliers	2			1	1
	mean (n)	0.13542			0.13670	0.13527
	st.dev. (n)	0.029320	RSD=22%		0.025466	0.029304
	R(calc.)	0.08210			0.07130	0.08205
	st.dev.(iis memo 1701)	0.021667			0.021873	0.021644
	R(iis memo 1701)	0.06067			0.06124	0.06060

Determination of DCHP – Dicyclohexyl phthalate on sample #20601; results in %M/M

lab	method	value	mark	z(targ)	remarks
210	ISO14389	0.065		-2.00	
348	CPSC-CH-C1001-09.4	0.0833		-0.80	
362		-----		-----	
551	In house	0.07869	C	-1.10	First reported 0.7494
622		-----		-----	
623	ISO14389	0.0983		0.18	
840		0.1220		1.73	
2115	ISO14389	0.123		1.80	
2129	In house	0.103		0.49	
2137		-----		-----	
2250	ISO14389	0.1006		0.33	
2255	ISO/TS 16181	0.0998		0.28	
2267		-----		-----	
2347	CPSC-CH-C1001-09.4	0.0856		-0.65	
2352	ISO/TS 16181	0.0973		0.12	
2358	ISO/TS 16181	N/A		-----	
2363	ISO14389	0.091		-0.29	
2366	ISO14389	0.092		-0.23	
2370	CPSC-CH-C1001-09.4	0.0971		0.10	
2375	ISO/TS 16181	0.109		0.88	
2378	ISO/TS 16181	0.0962		0.05	
2379	ISO/TS 16181	0.0927		-0.18	
2386	ISO/TS 16181	0.09273		-0.18	
2390	ISO14389	0.0789		-1.09	
2449	CPSC-CH-C1001-09.4	0.065735	C	-1.95	First reported 657.35 %M/M
2455		-----		-----	
2489	ISO14389	0.0986		0.20	
2499		-----		-----	
2511	ISO14389	0.092		-0.23	
2514	ISO/TS 16181	0.105		0.62	
2532	ISO/TS 16181	0.1056		0.66	
2563	ISO14389	0.064		-2.06	
2590	ISO/TS 16181	0.0470	R(0.05)	-3.17	
2695		0.10029		0.31	
2736	In house	0.093		-0.16	
2773	ISO/TS 16181	0.0880		-0.49	
2789		-----		-----	
2881	In house	0.11	C	0.95	First reported 0.24
3154	In house	0.1139		1.20	
3172	ISO/TS 16181	0.0908		-0.31	
3176	ISO14389	0.1280		2.13	
3197	ISO/TS 16181	0.10851	C	0.85	First reported 1085.1 %M/M
3210		0.0901		-0.35	
3243	In house	0.083		-0.82	

				Only THF used:	Only Hexane/Acetone used:
normality	OK			OK	OK
n	35			17	11
outliers	1			0	0
mean (n)	0.09551			0.09514	0.09988
st.dev. (n)	0.015047	RSD=16%		0.017739	0.006806
R(calc.)	0.04213			0.04967	0.01906
st.dev.(iis memo 1701)	0.015281			0.015222	0.015981
R(iis memo 1701)	0.04279			0.04262	0.04475
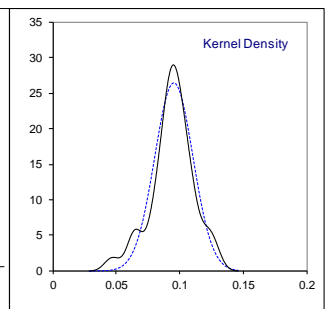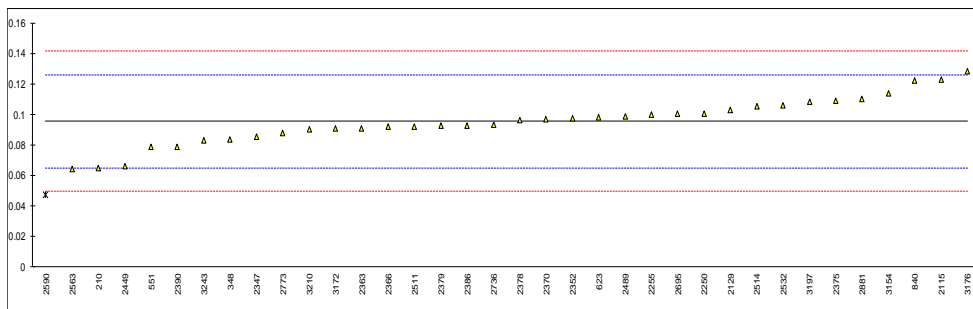
Determination of DEP – Diethyl phthalate on sample #20601; results in %M/M

lab	method	value	mark	z(targ)	remarks
210	ISO14389	0.00908	C	0.90	First reported 0.0124
348	CPSC-CH-C1001-09.4	0.0076		-0.27	
362		-----		-----	
551	In house	0.00846	C	0.41	First reported 0.1282
622		-----		-----	
623	ISO14389	0.0062		-1.37	
840		0.0090		0.84	
2115	ISO14389	0.026	C,R(0.01)	14.22	First reported 0.0167
2129	In house	0.00440		-2.79	
2137		-----		-----	
2250	ISO14389	0.0054		-2.00	
2255	ISO/TS 16181	0.01		1.62	
2267		-----		-----	
2347	CPSC-CH-C1001-09.4	0.0087		0.60	
2352	ISO/TS 16181	0.0094		1.15	
2358	ISO/TS 16181	N/A		-----	
2363	ISO14389	0.008		0.05	
2366	ISO14389	<0.01		-----	
2370	CPSC-CH-C1001-09.4	<0.00500		-----	
2375	ISO/TS 16181	0.009		0.84	
2378	ISO/TS 16181	0.0091		0.91	
2379	ISO/TS 16181	0.0097		1.39	
2386	ISO/TS 16181	0.00457		-2.65	
2390		-----		-----	
2449		-----		-----	
2455		-----		-----	
2489	ISO14389	0.0097		1.39	
2499		-----		-----	
2511		-----		-----	
2514	ISO/TS 16181	0.011		2.41	
2532	ISO/TS 16181	0.0088		0.68	
2563	ISO14389	< 0,005		-----	
2590	ISO/TS 16181	0.00570		-1.76	
2695		0.00543		-1.97	
2736	In house	<0.01		-----	
2773	ISO/TS 16181	0.0090		0.84	
2789		-----		-----	
2881	In house	<0.013		-----	
3154	In house	0.00765		-0.23	
3172	ISO/TS 16181	< 0.005		-----	
3176	ISO14389	0.0061		-1.45	
3197	ISO/TS 16181	0.00853	C	0.47	First reported 85.3 %M/M
3210		<0.001		<-5.46	Possibly a false negative test result?
3243	In house	n.d.		-----	

				Only THF used:	Only Hexane/Acetone used:
	normality	OK		OK	OK
	n	24		9	9
	outliers	1		1	0
	mean (n)	0.00794		0.00727	0.008556
	st.dev. (n)	0.001843	RSD=23%	0.001830	0.0021299
	R(calc.)	0.00516		0.00512	0.005964
	st.dev.(iis memo 1701)	0.001270		0.001163	0.0013689
	R(iis memo 1701)	0.00356		0.00326	0.003833
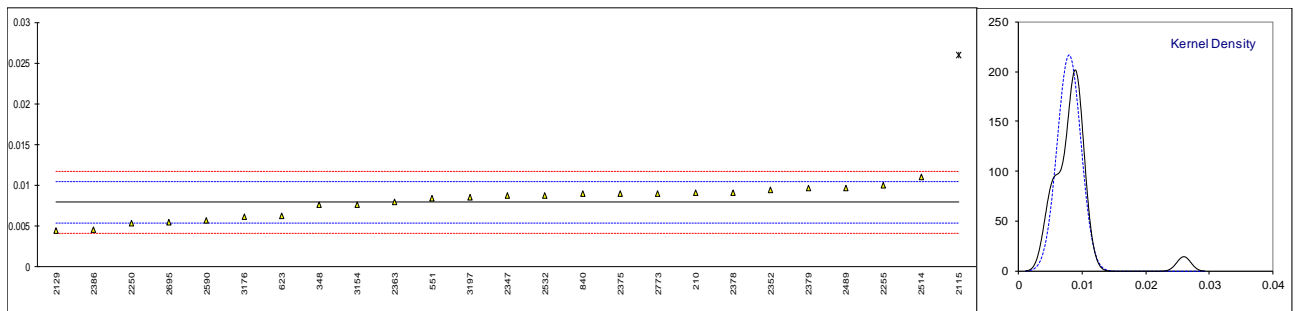
APPENDIX 2

Summary of other Phthalates in sample #20600: results in %M/M

BBP	= Benzylbutylphthalate	**DNOP**	= Di-n-Octylphthalate
DBP	= Dibutylphthalate	**DCHP**	= Dicyclohexylphthalate
DIDP	= Diisodecylphthalate	**DMP**	= Dimethylphthalate
DINP	= Diisononylphthalate	**DNHP**	= Di-n Hexylphthalate

Lab	BBP	DBP	DIDP	DINP	DNOP	DCHP	DMP	DNHP
210	-----	-----	-----	-----	-----	-----	-----	-----
348	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
362	-----	-----	-----	-----	-----	-----	-----	-----
551	-----	-----	-----	-----	-----	-----	-----	-----
622	0	0	0	0	0	-----	-----	0
623	ND	ND	ND	0.0093	ND	ND	ND	ND
840	not detected	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2115	-----	-----	-----	-----	-----	-----	-----	-----
2129	<0,002	<0,002	<0,002	0.0104	<0,002	<0,002	<0,002	<0,002
2137	-----	-----	-----	-----	-----	-----	-----	-----
2250	< 0,005	< 0,005	< 0,005	< 0,005	< 0,005	< 0,005	< 0,005	< 0,005
2255	n.d	n.d	n.d	0.011	n.d	n.d	n.d	n.d
2267	-----	-----	-----	-----	-----	-----	-----	-----
2347	<0.003	<0.003	<0.010	<0.010	<0.003	<0.003	<0.003	<0.003
2352	-----	-----	-----	0.0079	-----	-----	-----	-----
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2363	<0.005	<0.005	<0.005	0.007	<0.005	<0.005	<0.005	<0.005
2366	<0.01	<0.01	<0.02	<0.02	<0.01	<0.01	<0.01	<0.01
2370	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500
2375	-----	-----	-----	0.009	-----	-----	-----	-----
2378	-----	-----	-----	0.0081	-----	-----	-----	-----
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2386	<0,003	<0,003	<0,003	0.00733	<0,003	<0,003	<0,003	<0,003
2390	-----	0.10455	-----	-----	-----	-----	-----	-----
2449	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	-----	n.d.
2455	-----	-----	-----	-----	-----	-----	-----	-----
2489	ND	ND	ND	0.0071	ND	ND	ND	ND
2499	n.d	n.d.	n.d.	n.d.	n.d.	-----	-----	n.d.
2511	-----	-----	-----	-----	-----	-----	-----	-----
2514	-----	-----	-----	0.011	-----	-----	-----	-----
2532	<0.005	<0.005	<0.005	0.01	<0.005	<0.005	<0.005	<0.005
2563	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2590	-----	-----	-----	-----	-----	-----	-----	-----
2695	-----	-----	-----	0.01146	-----	-----	-----	-----
2736	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	-----	-----
2773	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2789	-----	-----	-----	0.0109	-----	-----	-----	-----
2881	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
3154	-----	-----	-----	-----	-----	-----	-----	-----
3172	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
3176	-----	-----	-----	-----	-----	-----	-----	-----
3197	ND	ND	ND	0.01051　C	ND	ND	ND	ND
3210	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
3243	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

Lab 3197: first reported 105.1%

Summary of other Phthalates in sample #20600: results in %M/M - continued

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

Lab	DIBP	DPHP	DNPP	DUP	DPrP	DMEP	Other
210	-----	-----	-----	-----	-----	-----	-----
348	<0,005	-----	<0,005	-----	-----	-----	-----
362	-----	-----	-----	-----	-----	-----	-----
551	-----	-----	-----	-----	-----	-----	-----
622	0	-----	-----	-----	-----	-----	-----
623	ND	ND	ND	ND	ND	ND	ND
840	not detected	not detected	not detected	not detected	not detected	not detected	-----
2115	-----	-----	-----	-----	-----	-----	-----
2129	<0,002	<0,002	<0,002	-----	<0,002	<0,002	<0,002
2137	-----	-----	-----	-----	-----	-----	-----
2250	< 0,005	< 0,005	< 0,005	< 0,005	< 0,005	< 0,005	< 0,005
2255	n.d	n.d	n.d	n.d	n.d	n.d	n.d
2267	-----	-----	-----	-----	-----	-----	-----
2347	<0.003	<0.010	<0.003	<0.003	<0.003	<0.003	-----
2352	-----	-----	-----	-----	-----	-----	-----
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	N/A
2363	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	0.164
2366	<0.01	<0.02	<0.01	<0.01	<0.01	<0.01	<0.01
2370	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500
2375	-----	-----	-----	-----	-----	-----	-----
2378	-----	-----	-----	-----	-----	-----	-----
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not tested
2386	<0,003	<0,003	<0,003	<0,003	<0,003	<0,003	<0,003
2390	-----	-----	-----	-----	-----	-----	-----
2449	n.d.	-----	n.d.	-----	-----	-----	-----
2455	-----	-----	-----	-----	-----	-----	-----
2489	ND	ND	ND	ND	ND	ND	ND
2499	n.d.	-----	n.d	n.d.	-----	-----	-----
2511	-----	-----	-----	-----	-----	-----	-----
2514	-----	-----	-----	-----	-----	-----	-----
2532	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2563	n.d.	-----	n.d.	-----	n.d.	n.d.	-----
2590	-----	-----	-----	-----	-----	-----	-----
2695	-----	-----	-----	-----	-----	-----	-----
2736	<0.01	-----	-----	-----	-----	-----	-----
2773	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	0.1930
2789	-----	-----	-----	-----	-----	-----	-----
2881	0.00	0.00	0.00	0.00	0.00	0.00	0.00
3154	-----	-----	-----	-----	-----	-----	-----
3172	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
3176	-----	-----	-----	-----	-----	-----	-----
3197	ND	ND	ND	ND	ND	ND	ND
3210	<0.001	-----	<0.001	<0.001	-----	<0.001	-----
3243	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

Summary of other Phthalates in sample #20601: results in %M/M

DEHP = Bis-2-ethylhexylphthalate   **DNOP** = Di-n-Octylphthalate
DBP = Dibutylphthalate   **DMP** = Dimethylphthalate
DIDP = Diisodecylphthalate   **DNHP** = Di-n-Hexylphthalate
DINP = Diisononylphthalate

Lab	DEHP	DBP	DIDP	DINP	DNOP	DMP	DNHP
210	-----	-----	-----	-----	-----	-----	-----
348	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
362	-----	-----	-----	-----	-----	-----	-----
551	-----	-----	-----	-----	-----	-----	-----
622	0	0	0	0	0	-----	-----
623	ND	ND	ND	0.0109	ND	ND	ND
840	not detected	not detected	not detected	not detected	not detected	not detected	not detected
2115	-----	-----	-----	-----	-----	-----	-----
2129	<0,002	<0,002	<0,002	0.0102	<0,002	<0,002	<0,002
2137	0.09	-----	-----	-----	-----	-----	-----
2250	< 0,005	< 0,005	< 0,005	< 0,005	< 0,005	< 0,005	< 0,005
2255	n.d	n.d	n.d	0.011	n.d	n.d	n.d
2267	-----	-----	-----	-----	-----	-----	-----
2347	<0.005	<0.005	<0.005	<0.010	<0.005	<0.005	<0.005
2352	-----	-----	-----	0.0078	-----	-----	-----
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2363	<0.005	<0.005	<0.005	0.008	<0.005	<0.005	<0.005
2366	<0.01	<0.01	<0.02	<0.02	<0.01	<0.01	<0.01
2370	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500
2375	-----	-----	-----	0.009	-----	-----	-----
2378	-----	-----	-----	0.0084	-----	-----	-----
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2386	<0,003	<0,003	<0,003	0.00590	<0,003	<0,003	<0,003
2390	-----	0.0901	-----	-----	-----	-----	-----
2449	0.007324	n.d.	n.d.	<0.0050	n.d.	-----	n.d.
2455	-----	-----	-----	-----	-----	-----	-----
2489	ND	ND	ND	0.009	ND	ND	ND
2499	n.d.	n.d.	n.d.	n.d	n.d.	-----	n.d.
2511	-----	-----	-----	-----	-----	-----	-----
2514	-----	-----	-----	0.011	-----	-----	-----
2532	<0.005	<0.005	<0.005	0.01	<0.005	<0.005	<0.005
2563	n.d.	< 0,005	n.d.	n.d.	n.d.	n.d.	n.d.
2590	-----	-----	-----	-----	-----	-----	-----
2695	-----	-----	-----	0.01148	-----	-----	-----
2736	<0.01	<0.01	<0.01	<0.01	<0.01	-----	-----
2773	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2789	-----	-----	-----	-----	-----	-----	-----
2881	0.00	0.00	0.00	0.00	0.00	0.00	0.00
3154	-----	-----	-----	-----	-----	-----	-----
3172	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
3176	-----	-----	-----	-----	-----	-----	-----
3197	ND	ND	ND	0.01081 C	ND	ND	ND
3210	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
3243	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

Lab 3197: First reported 108.1%

Summary of other Phthalates in sample #20601: results in %M/M - continued

DIBP = Diisobutylphthalate
DPHP = Di(2-propylheptyl)phthalate
DNPP = Di-n-Pentylphthalate
DUP = Diundecylphthalate

DPrP = Di-n-Propylphthalate
DMEP = Di(2-methoxyethyl)phthalate
Other = other Phthalates

Lab	DIBP	DPHP	DNPP	DUP	DPrP	DMEP	Other
210	-----	-----	-----	-----	-----	-----	-----
348	<0,005	-----	<0,005	-----	-----	-----	-----
362	-----	-----	-----	-----	-----	-----	-----
551	-----	-----	-----	-----	-----	-----	-----
622	0	-----	-----	-----	-----	-----	-----
623	ND	ND	ND	ND	ND	ND	ND
840	not detected	not detected	not detected	not detected	not detected	not detected	-----
2115	-----	-----	-----	-----	-----	-----	-----
2129	<0,002	<0,002	<0,002	-----	<0,002	<0,002	<0,002
2137	-----	-----	-----	-----	-----	-----	-----
2250	< 0,005	< 0,005	< 0,005	< 0,005	< 0,005	< 0,005	< 0,005
2255	n.d	n.d	n.d	n.d	n.d	n.d	n.d
2267	-----	-----	-----	-----	-----	-----	-----
2347	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	-----
2352	-----	-----	-----	-----	-----	-----	-----
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	N/A
2363	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	0.247
2366	<0.01	<0.02	<0.01	<0.01	<0.01	<0.01	<0.01
2370	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500	<0.00500
2375	-----	-----	-----	-----	-----	-----	-----
2378	-----	-----	-----	-----	-----	-----	-----
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not tested
2386	<0,003	<0,003	<0,003	<0,003	<0,003	<0,003	<0,003
2390	-----	-----	-----	-----	-----	-----	-----
2449	n.d.	-----	n.d.	-----	-----	-----	-----
2455	-----	-----	-----	-----	-----	-----	-----
2489	ND	ND	ND	ND	ND	ND	ND
2499	n.d	-----	n.d	n.d	-----	-----	-----
2511	-----	-----	-----	-----	-----	-----	-----
2514	-----	-----	-----	-----	-----	-----	-----
2532	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2563	< 0,005	-----	n.d.	-----	n.d.	n.d.	-----
2590	-----	-----	-----	-----	-----	-----	-----
2695	-----	-----	-----	-----	-----	-----	-----
2736	<0.01	-----	-----	-----	-----	-----	-----
2773	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	0.2240
2789	-----	-----	-----	-----	-----	-----	-----
2881	0.00	0.00	0.00	0.00	0.00	0.00	0.00
3154	-----	-----	-----	-----	-----	-----	-----
3172	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
3176	-----	-----	-----	-----	-----	-----	-----
3197	ND	ND	ND	ND	ND	ND	ND
3210	<0.001	-----	<0.001	<0.001	-----	<0.001	-----
3243	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

APPENDIX 3 Analytical details

lab	ISO/IEC17025 accredited for Phthalates	Sample intake (in grams)	Solvent (mixture) used to release the analytes	Extraction time (in minutes)	Extraction temperature (in °C)
210	No	--	--	--	--
348	Yes	0.5	THF	180	60±5
362	No	1	THF / Hexane	60	60
551	Yes	0.5	--	--	--
622	Yes	1	Hexane / Acetone	60	50
623	Yes	0.1	THF / Hexane	60	60
840	Yes	0.5	THF / Hexane	60	60
2115	Yes	0.3	THF / Hexane	60	60
2129	Yes	0.5	THF	60	60
2137	Yes	0.5	THF	60	40
2250	Yes	0.3	THF / ACN	60	60
2255	Yes	0.5	Hexane / Acetone	60	50
2267	---	--	--	--	--
2347	Yes	0.3	--	--	--
2352	Yes	0.5	Hexane / Acetone	60	50
2358	Yes	1	Hexane / Acetone	60	50
2363	Yes	2	TFT	60	60
2366	No	0.3	THF / Hexane	60	60
2370	Yes	0.1	THF / Hexane	30	room
2375	Yes	0.1	Hexane / Acetone	60	50
2378	Yes	2	Hexane	60	50
2379	No	0.5	Hexane / Acetone	60	50
2386	Yes	0.5	Hexane / Acetone	60	50
2390	Yes	0.1	THF	60	60
2449	---	0.3	THF / ACN	120	40
2455	---	--	--	--	--
2489	Yes	0.1 / 0.3	THF / Hexane	60	60
2499	Yes	3	Hexane / Acetone	60	50
2511	---	--	--	--	--
2514	Yes	0.3	Hexane / Acetone	60	50
2532	Yes	1	Hexane / Acetone	60	50
2563	Yes	0.3	THF	60	60
2590	---	--	--	--	--
2695	Yes	2	Hexane / Acetone	60	50
2736	Yes	0.5	THF / ACN	60	room
2773	Yes	0.3	THF / Hexane	30	60
2789	Yes	2	Hexane	60	60
2881	Yes	0.5	Hexane	60	60
3154	Yes	0.5	Toluene	60	50
3172	Yes	1	THF / ACN	60	25
3176	Yes	0.3	THF / ACN	60	60
3197	Yes	2	ACN / Hexane	60	50
3210	Yes	1	Hexane / Acetone	60	50
3243	Yes	1	Dichlormethane	30	room

APPENDIX 4

Number of participating laboratories per country

2 labs in BANGLADESH
1 lab in BRAZIL
1 lab in BULGARIA
1 lab in FRANCE
6 labs in GERMANY
1 lab in HONG KONG
3 labs in INDIA
2 labs in INDONESIA
5 labs in ITALY
1 lab in MOROCCO
5 labs in P.R. of CHINA
2 labs in PAKISTAN
1 lab in POLAND
1 lab in SOUTH KOREA
2 labs in SPAIN
1 lab in TAIWAN
1 lab in THAILAND
1 lab in THE NETHERLANDS
1 lab in TUNISIA
3 labs in TURKEY
2 labs in U.S.A.
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

Literature

1   iis, Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation, June 2018
2   ISO/TS16181:11
3   P.L. Davies, Fr Z. Anal. Chem, 351, 513, (1988)
4   W.J. Conover, Practical; Nonparametric Statistics, J. Wiley and Sons, NY, 302, (1971)
5   ISO5725:86
6   ISO5725:94, parts 1-6
7   ISO13528:05
8   M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
9   W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
10   G. Rohm, J. Bohnen and H. Kruessmann, GIT Labor-Fachzeitschrift, 1080, 11, (1997)
11   Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)
12   Analytical Methods Committee, Technical letter, No 4, January 2001
13   P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)
14   Horwitz, W and Albert, R, J. AOAC Int, 79, 3, 589, (1996)
15   iis memo 1701: Precision data of Phthalates in plastic, www.iisnl.com, News and Report page (July 2017)