Results of Proficiency Test
Phthalates in Leather
May 2019

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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CONTENTS

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 organise an interlaboratory study for the determination of Phthalates in leather since 2017 every year. During the annual testing program of 2018/2019, it was decided to continue the proficiency test for the analysis of Phthalates in Leather.
In this interlaboratory study, 56 laboratories in 26 different countries registered. See appendix 4 for the number of participating laboratories per country. In this report, the results of the 2019 proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands was the organizer of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send to each participant two different leather samples of 3 grams each, respectively labelled #19543 and #19544. 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/IEC 17043:2010. 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 organisation 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

Two different batches of leather both made to be positive with a number of Phthalates were prepared.
The first batch a beige colored leather was grinded and subsequently homogenized. Out of the batch approximately 100 subsamples of 3 grams were packed in plastic bags and labelled #19543.
The homogeneity of the subsamples #19543 was checked by the determination of Diisodecylphthalate (DIDP) and Di-n-hexylphthalate (DNHP) on ten stratified randomly selected samples. The determinations were performed in accordance with ISO/TS16181. See the following table for the test results.

	DIDP in %M/M	DNHP in %M/M
Sample #19543-1	0.2290	0.1779
Sample #19543-2	0.2291	0.1792
Sample #19543-3	0.2101	0.1598
Sample #19543-4	0.2305	0.1713
Sample #19543-5	0.2192	0.1631
Sample #19543-6	0.2258	0.1737
Sample #19543-7	0.2252	0.1693
Sample #19543-8	0.2280	0.1659
Sample #19543-9	0.2119	0.1564
Sample #19543-10	0.2061	0.1749

Table 1: homogeneity test results of the subsamples #19543

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.

	DIDP in %M/M	DNHP in %M/M
r (observed)	0.0253	0.0216
reference method	iis memo 1701*)	iis memo 1701*)
0.3*R (reference method)	0.0298	0.0227

Table 2: evaluation of repeatabilities of the subsamples #19543
*) see literature 15

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibility of the reference method. Therefore, the homogeneity of subsamples #19543 was assumed.

The second batch a black colored leather was grinded and subsequently homogenized. Out of the batch approximately 100 subsamples of 3 grams were packed in plastic bags and labelled #19544.

The homogeneity of the subsamples #19544 was checked by the determination of Diisononylphthalate (DINP) and Dimethylphthalate (DMP) on four stratified randomly selected samples. The determinations were performed in accordance with ISO/TS16181. See the following table for the test results.

	DINP in %M/M	DMP in %M/M
Sample #19544-1	0.0967	0.1425
Sample #19544-2	0.0982	0.1425
Sample #19544-3	0.1005	0.1406
Sample #19544-4	0.1058	0.1434

Table 3: homogeneity test results of the subsamples #19544

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.

	DINP in %M/M	DMP in %M/M
r (observed)	0.0112	0.0033
reference method	iis memo 1701*)	iis memo 1701*)
0.3*R (reference method)	0.0135	0.0191

Table 4: evaluation of repeatabilities of the subsamples #19544

*) see literature 15

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibility of the reference method. Therefore, the homogeneity of subsamples #19544 was assumed.

To each of the participating laboratories one sample of approximately 3 grams, labelled #19543 and one sample of approximately 3 grams, labelled #19544 was sent on April 17, 2019.

2.5 ANALYSES

The participants were requested to determine on both samples #19543 and #19544, fourteen individual Phthalates (see appendices 1 and 2) and when identified other Phthalates. It was also requested to report if the laboratory was accredited for the requested components and some method details were the requested to be reported.

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.

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 individual laboratories 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 reanalyses). 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 ISO 5725 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 visualise 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:

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

The $z_{(target)}$ scores are listed in the 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 problems were encountered with sample transport. Four participants reported the test results after the final reporting date and two other participants did not report at all. Finally, 54 laboratories reported 224 numerical results. Observed were 10 statistically outlying test results, which is 4.5% of all results. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

All original data sets proved to have a normal Gaussian distribution.

4.1 EVALUATION PER SAMPLE AND PER COMPONENT

In this section, the reported test results are discussed per sample and per component. The test methods, which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the original data. The abbreviations, used in these tables, are listed 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 two 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 #19543

DIDP: The determination of DIDP was problematic at the level of 0.17 %M/M. Three 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 §5).

DMP: The majority of the group was able to detect DMP at the level of 0.014 %M/M. Regretfully, the calculated reproducibility is very large at this low level. Therefore, no z-scores were calculated.

DNHP: The determination of DNHP was not problematic at the level of 0.12 %M/M. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the target reproducibility derived from the iis-memo 1701(see for more discussion §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 results.

Sample #19544

DINP: The determination of DINP was problematic at the level of 0.11 %M/M. One statistical outlier was observed and one other test result was excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the target reproducibility derived from the iis-memo 1701(see for more discussion §5).

DMP: The determination of DMP was very problematic at the level of 0.10 %M/M. One statistical outlier was observed and one other test result was excluded. The calculated reproducibility after rejection of the suspect data is not at all in agreement with the target reproducibility derived from the iis-memo 1701(see for more discussion §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 results.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Component	unit	n	average	2.8 * sd	R (target)
DIDP	%M/M	45	0.171	0.102	0.077
DMP	%M/M	34	0.014	0.017	(0.006)
DNHP	%M/M	44	0.125	0.049	0.056

Table 5: reproducibilities of tests for sample #19543

Component	unit	n	average	2.8 * sd	R (target)
DINP	%M/M	47	0.106	0.087	0.048
DMP	%M/M	44	0.095	0.087	0.043

Table 6: reproducibilities of tests for sample #19544

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 2019 WITH PREVIOUS PTS

	May 2019	April 2018	April 2017
Number of reporting labs	54	66	41
Number of results reported	224	123	127
Number of statistical outliers	10	2	9
Percentage outliers	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 2019	April 2018	April 2017	iis memo 1701	ISO/TS 16181
BBP	n.e.	16%	13%	16%	10%
DBP	n.e.	n.e.	18%	16%	10%
DIDP	21%	n.e.	n.e.	16%	10%
DINP	29%	n.e.	n.e.	16%	10%
DCHP	n.e.	21%	n.e.	16%	10%
DMP	33-46%	n.e.	n.e.	16%	10%
DNHP	14%	n.e.	n.e.	16%	10%
DIBP	n.e.	n.e.	16%	16%	10%

Table 8: development of uncertainties over the years

4.4 EVALUATION OF THE ANALYTICAL DETAILS

For this PT some analytical details were requested, see appendix 3. Based on the answers given by the participants the following can be summarized:

- About 63% of the reporting participants mentioned that they are accredited for the determination of Phthalates in Leather.
- About 57% of the reporting participants used a test portion between 0.5 and 1 grams. About 27% used less sample material.
- About 75% of the reporting participants used an extraction time of 60 minutes
- About 80% of the reporting participants used an extraction temperature of 50°C or 60°C.
- About 57% of the reporting laboratories used Hexane/Acetone as solvent mixture to release the Phthalates. The other part (43%) of the reporting laboratories used THF as solvent.

Looking at the analytical details, it may be remarkable that a number of 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 but describes also that for samples larger

than 0.05 grams, which is the case here, 10 ml of THF can be added for every 0.1 gram of extra sample intake. When the test results of participants with a reported sample intake of 0.5 gram and higher and reported to have used ISO/TS:16181 as test method are evaluated separately, the calculated reproducibilities are all smaller, but are still not in agreement with the reproducibility limits of iis memo 1701, except for DNHP #19543 which is still in agreement (see in appendix 1). The sample intake of <0.5 grams appears to have a negative influence on the variation in the test results.
The use of different solvent mixtures to release the Phthalates do not have a significant effect on the varaibility.

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 #19543 and sample #19544. Regretfully, three of the four 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/IEC 17025 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

Again it is concluded that the sample intake has a significant effect on the variation, see also PT:iis18A09 of 2018. Therefore, participants are strongly advised to read the PT instructions carefully and to use a sample size that is at least 0.5 grams.

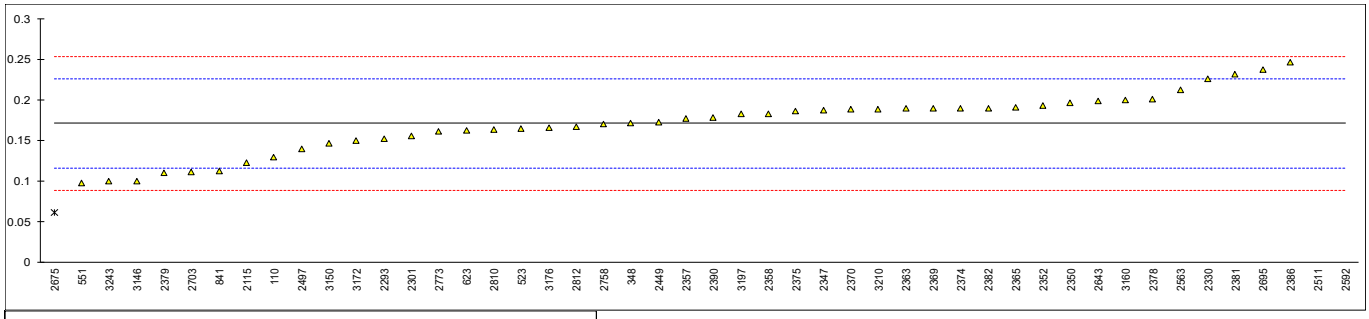
Although it can be concluded that most of the participants have no problem with the determination on Phthalates in Leather in this PT, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary.
Therefore, participation on a 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 DIDP – Diisodecylphthalate on sample #19543; results in %M/M

lab	method	value	mark	z(targ)	remarks
110	In house	0.1297		-1.50	
348	CPSC-CH-C1001-09.4	0.1708		0.00	
362		-----		-----	
523		0.1644	C	-0.23	First reported <0.0025
551	In house	0.0974	C	-2.69	First reported 0.359
623	ISO14389	0.162		-0.32	
841	CPSC-CH-C1001-09.4	0.112714		-2.13	Reported 1127.14 %M/M
2115	ISO/TS 16181	0.123		-1.75	
2293	CPSC-CH-C1001-09.3	0.152		-0.69	
2301	In house	0.155		-0.58	
2330	ISO14389	0.2263		2.03	
2347	ISO/TS 16181	0.187		0.59	
2350	CPSC-CH-C1001-09.4	0.196		0.92	
2352	ISO/TS 16181	0.1930		0.81	
2357	ISO/TS 16181	0.1773		0.24	
2358	ISO/TS 16181	0.18238		0.42	
2363	ISO/TS 16181	0.190		0.70	
2365	ISO/TS 16181	0.19115		0.74	
2369	ISO/TS 16181	0.190		0.70	
2370	CPSC-CH-C1001-09.4	0.188		0.63	
2374	In house	0.190		0.70	
2375	ISO/TS 16181	0.186		0.56	
2378	ISO/TS 16181	0.2010		1.11	
2379	ISO/TS 16181	0.110		-2.22	
2381		0.2319		2.24	
2382	ISO/TS 16181	0.190		0.70	
2386	ISO/TS 16181	0.246		2.75	
2390	ISO14389	0.178	C	0.26	First reported 178.99 %M/M
2449		0.173		0.08	
2455		-----		-----	
2497	ISO/TS 16181	0.1401		-1.12	
2511	ISO/TS 16181	0.425	R(0.01)	9.30	
2563	ISO/TS 16181	0.212		1.51	
2590		-----		-----	
2592	ISO/TS 16181	0.92	C,R(0.01)	27.42	First reported <0.004
2643	CPSC-CH-C1001-09.3	0.199		1.03	
2656		-----		-----	
2675	ISO14389	0.061	C,R(0.05)	-4.02	First reported 0.958
2695	ISO/TS 16181	0.2366	C	2.41	First reported 0.4731
2703	ISO14389	0.111		-2.19	
2705		-----		-----	
2734	ISO/TS 16181	nd		-----	
2756		-----		-----	
2758	ISO14389	0.170		-0.03	
2773	ISO/TS 16181	0.161		-0.36	
2810	ISO/TS 16181	0.16391		-0.25	
2812	ISO/TS 16181	0.167		-0.14	
2883		-----		-----	
3146	In house	0.10		-2.59	
3150	In house	0.14635		-0.89	
3154		-----		-----	
3160	ISO/TS 16181	0.20		1.07	
3172	ISO/TS 16181	0.1496		-0.78	
3176	ISO/TS 16181	0.1652		-0.20	
3197	ISO/TS 16181	0.1822		0.42	
3210	In house	0.1881		0.63	
3243	In house	0.0998		-2.60	
					Only ISO/TS16181 data *):
	normality	OK			suspect
	n	45			20
	outliers	3			1
	mean (n)	0.17080			0.18213
	st.dev. (n)	0.036459	RSD = 21%		0.032856 RSD = 18%
	R(calc.)	0.10209			0.09200
	st.dev.(iis memo 1701)	0.027328			0.029141
	R(iis memo 1701)	0.07652			0.08160
Compare					
	R(ISO/TS16181:11)	0.04964			0.05293

*) Only data of participants that followed ISO/TS16181 to the letter, with a minimum sample intake of 0.5 gram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0.25
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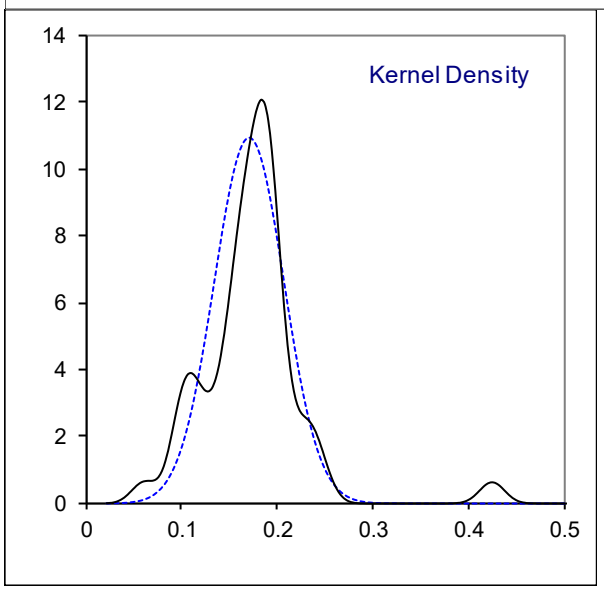
Kernel Density
14
12
10
8
6
4
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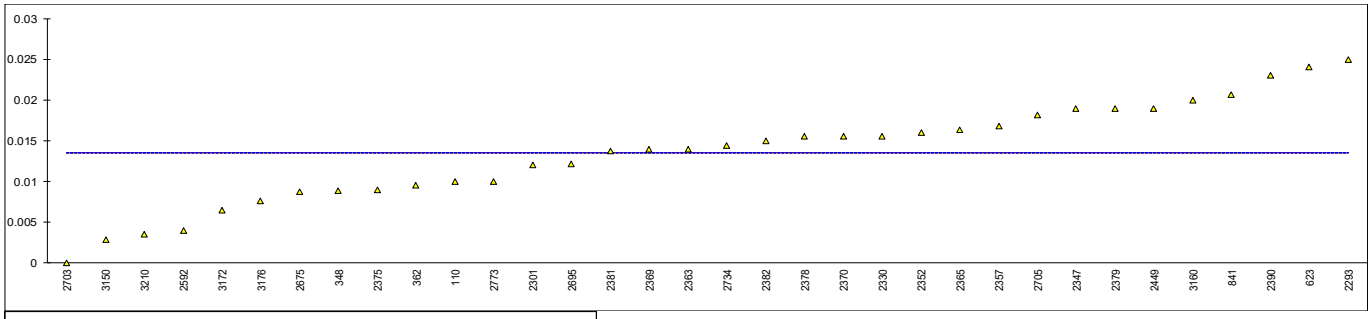
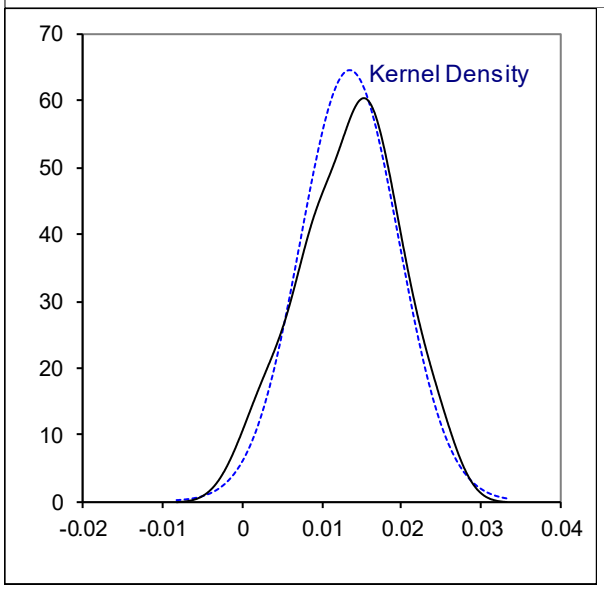
0 0.1 0.2 0.3 0.4 0.5

Determination of DMP – Dimethylphthalate on sample #19543; results in %M/M

lab	method	value	mark	z(targ)	remarks
110	In house	0.01	C	-----	First reported N.D.
348	CPSC-CH-C1001-09.4	0.0089		-----	
362	ISO/TS 16181	0.0095		-----	
523		-----		-----	
551		-----		-----	
623	ISO14389	0.024		-----	
841	CPSC-CH-C1001-09.4	0.020662	C	-----	First reported 206.62 %M/M
2115		-----		-----	
2293	CPSC-CH-C1001-09.3	0.025		-----	
2301	In house	0.012		-----	
2330	ISO14389	0.0155		-----	
2347	ISO/TS 16181	0.019		-----	
2350		-----		-----	
2352	ISO/TS 16181	0.0160		-----	
2357	ISO/TS 16181	0.0168		-----	
2358	ISO/TS 16181	N/A		-----	
2363	ISO/TS 16181	0.014		-----	
2365	ISO/TS 16181	0.01631		-----	
2369	ISO/TS 16181	0.014		-----	
2370	CPSC-CH-C1001-09.4	0.0155		-----	
2374		-----		-----	
2375	ISO/TS 16181	0.009		-----	
2378	ISO/TS 16181	0.0155		-----	
2379	ISO/TS 16181	0.019		-----	
2381		0.0137		-----	
2382	ISO/TS 16181	0.015		-----	
2386	ISO/TS 16181	<0,005		-----	
2390	ISO14389	0.023	C	-----	First reported 229.77 %M/M
2449		0.019		-----	
2455		-----		-----	
2497		-----		-----	
2511		-----		-----	
2563	ISO/TS 16181	ND		-----	
2590		-----		-----	
2592	ISO/TS 16181	0.004		-----	
2643		-----		-----	
2656		-----		-----	
2675	ISO14389	0.0088		-----	
2695	ISO/TS 16181	0.0121	C	-----	First reported 0.0243
2703	ISO14389	0		-----	
2705	In house	0.0182		-----	
2734	ISO/TS 16181	0.0144		-----	
2756		-----		-----	
2758		-----		-----	
2773	ISO/TS 16181	0.01		-----	
2810		-----		-----	
2812		-----		-----	
2883		-----		-----	
3146		-----		-----	
3150	In house	0.0028		-----	
3154		-----		-----	
3160	ISO/TS 16181	0.02		-----	
3172	ISO/TS 16181	0.0065	C	-----	First reported <0.005
3176	ISO/TS 16181	0.0076		-----	
3197		-----		-----	
3210	In house	0.0035		-----	
3243	In house	n.d.		-----	
					Only ISO/TS16181 data *):
	normality	OK			suspect
	n	34			14
	outliers	0			0
	mean (n)	0.01351			0.01440
	st.dev. (n)	0.006176	RSD = 46%		0.003330 RSD = 23%
	R(calc.)	0.01729			0.00933
	st.dev.(iis memo 1701)	(0.002161)			(0.002304)
	R(iis memo 1701)	(0.00605)			(0.00645)
Compare					
	R(ISO/TS16181:11)	(0.00393)			(0.00419)

*) Only data of participants that followed ISO/TS16181 to the letter, with a minimum sample intake of 0.5 gram

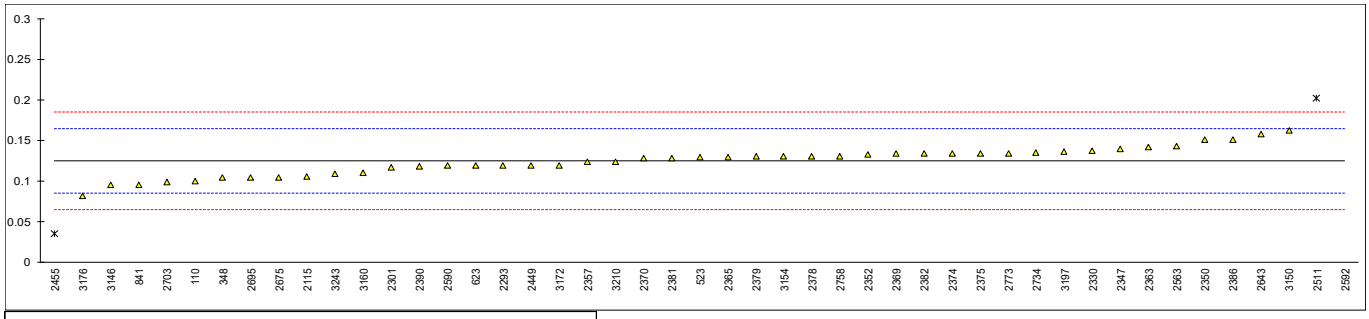

0.03
0.025
0.02
0.015
0.01
0.005
0
2703
3150
3210
2592
3172
3176
2675
348
2375
362
110
2773
2301
2695
2381
2369
2363
2734
2382
2378
2370
2330
2352
2365
2357
2705
2347
2379
2449
3160
841
2390
623
2293

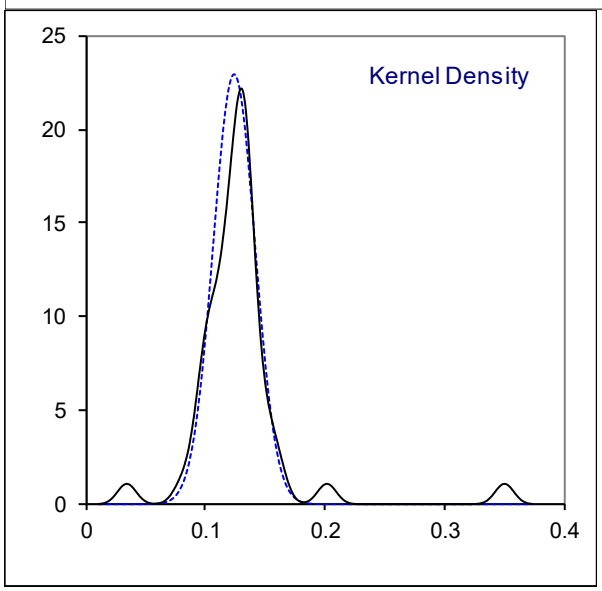

Kernel Density
70
60
50
40
30
20
10
0
-0.02
-0.01
0
0.01
0.02
0.03
0.04

Determination of DNHP – Di-n-hexylphthalate on sample #19543; results in %M/M

lab	method	value	mark	z(targ)	remarks
110	In house	0.0998		-1.25	
348	CPSC-CH-C1001-09.4	0.1044		-1.02	
362		-----		-----	
523		0.1291		0.22	
551		-----		-----	
623	ISO14389	0.119		-0.28	
841	CPSC-CH-C1001-09.4	0.095688	C	-1.45	First reported 956,88 %M/M
2115	ISO/TS 16181	0.106		-0.94	
2293	CPSC-CH-C1001-09.3	0.119		-0.28	
2301	In house	0.117		-0.38	
2330	ISO14389	0.1375		0.64	
2347	ISO/TS 16181	0.140		0.77	
2350	CPSC-CH-C1001-09.4	0.151		1.32	
2352	ISO/TS 16181	0.1330		0.42	
2357	ISO/TS 16181	0.1235		-0.06	
2358	ISO/TS 16181	N/A		-----	
2363	ISO/TS 16181	0.142		0.87	
2365	ISO/TS 16181	0.12999		0.27	
2369	ISO/TS 16181	0.134		0.47	
2370	CPSC-CH-C1001-09.4	0.128		0.17	
2374	In house	0.134		0.47	
2375	ISO/TS 16181	0.134		0.47	
2378	ISO/TS 16181	0.1310		0.32	
2379	ISO/TS 16181	0.130		0.27	
2381		0.1285		0.19	
2382	ISO/TS 16181	0.134		0.47	
2386	ISO/TS 16181	0.151		1.32	
2390	ISO14389	0.118	C	-0.33	First reported 1176.5 %M/M
2449		0.119		-0.28	
2455	CPSC-CH-C1001-09.4	0.03522	R(0.01)	-4.48	
2497		-----		-----	
2511	ISO/TS 16181	0.202	R(0.01)	3.88	
2563	ISO/TS 16181	0.143		0.92	
2590	ISO/TS 16181	0.11881		-0.29	
2592	ISO/TS 16181	0.35	C,R(0.01)	11.30	First reported <0.004
2643	CPSC-CH-C1001-09.3	0.158		1.67	
2656		-----		-----	
2675	ISO14389	0.105	C	-0.99	First reported 0.073
2695	ISO/TS 16181	0.1048	C	-1.00	First reported 0.2096
2703	ISO14389	0.099		-1.29	
2705		-----		-----	
2734	ISO/TS 16181	0.1352	C	0.53	First reported 0.0676
2756		-----		-----	
2758	ISO14389	0.131		0.32	
2773	ISO/TS 16181	0.134		0.47	
2810		-----		-----	
2812		-----		-----	
2883		-----		-----	
3146	In house	0.095		-1.49	
3150	In house	0.1622		1.88	
3154	In house	0.130		0.27	
3160	ISO/TS 16181	0.11		-0.74	
3172	ISO/TS 16181	0.1196		-0.25	
3176	ISO/TS 16181	0.0818		-2.15	
3197	ISO/TS 16181	0.1359		0.56	
3210	In house	0.1238		-0.04	
3243	In house	0.1095		-0.76	
					Only ISO/TS16181 data *):
	normality	OK			OK
	n	44			18
	outliers	3			1
	mean (n)	0.12466			0.12946
	st.dev. (n)	0.017373	RSD = 14%		0.012516 RSD = 10%
	R(calc.)	0.04864			0.03504
	st.dev.(iis memo 1701)	0.019946			0.020713
	R(iis memo 1701)	0.05585			0.05800
Compare					
	R(ISO/TS16181:11)	0.03623			0.03762

*) Only data of participants that followed ISO/TS16181 to the letter, with a minimum sample intake of 0.5 gram

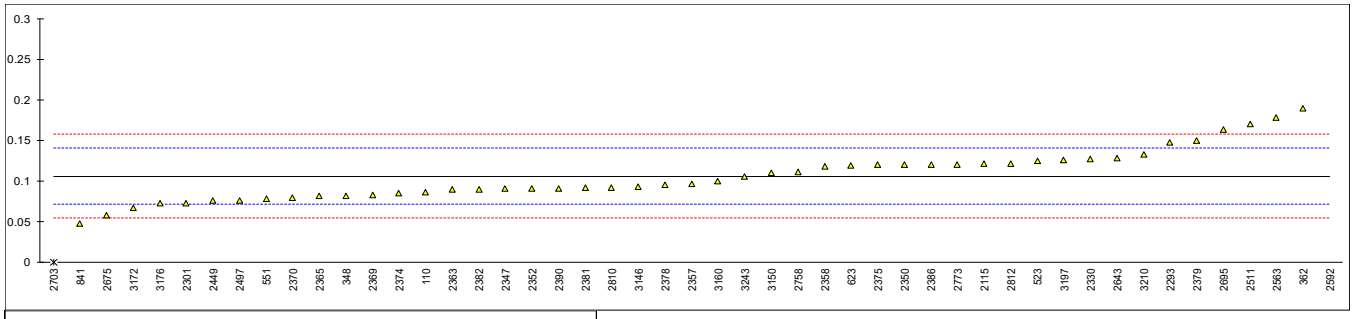

0.3
0.25
0.2
0.15
0.1
0.05
0
2455 3176 3146 841 2703 110 348 2685 2675 2115 3243 3160 2301 2390 2590 623 2293 2449 3172 2357 3210 2370 2381 523 2365 2379 3154 2378 2758 2352 2369 2382 2374 2375 2773 2734 3197 2330 2347 2363 2563 2350 2386 2643 3150 2511 2592

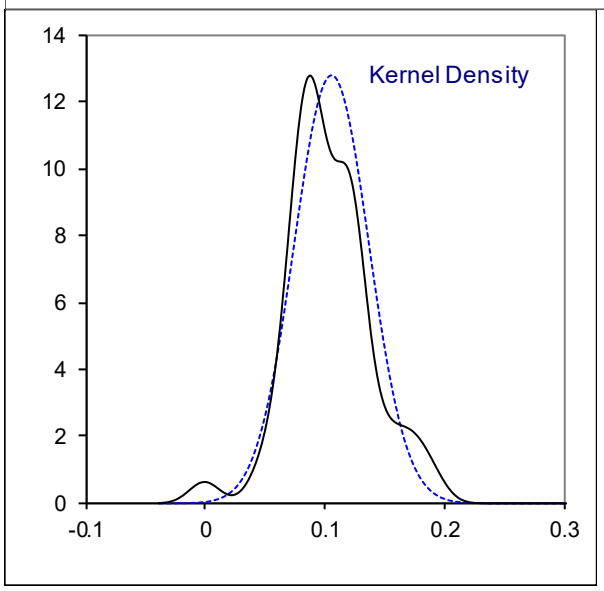

Kernel Density
25
20
15
10
5
0
0
0.1
0.2
0.3
0.4

Determination of DINP – Diisononylphthalate on sample #19544; results in %M/M

lab	method	value	mark	z(targ)	remarks
110	In house	0.0859		-1.19	
348	CPSC-CH1001-09.4	0.0813		-1.46	
362	ISO/TS 16181	0.19	C	4.94	First reported 0.774
523		0.1251		1.12	
551	In house	0.0787	C	-1.62	First reported 0.0194
623	ISO14389	0.119		0.76	
841	CPSC-CH-C1001-09.4	0.047453	C	-3.46	First reported 474.35 %M/M
2115	ISO/TS 16181	0.121		0.87	
2293	CPSC-CH-C1001-09.3	0.147		2.41	
2301	In house	0.073		-1.95	
2330	ISO14389	0.1267		1.21	
2347	ISO/TS 16181	0.091		-0.89	
2350	CPSC-CH-C1001-09.4	0.120		0.82	
2352	ISO/TS 16181	0.0910		-0.89	
2357	ISO/TS 16181	0.0966		-0.56	
2358	ISO/TS 16181	0.11810		0.70	
2363	ISO/TS 16181	0.090		-0.95	
2365	ISO/TS 16181	0.08130		-1.46	
2369	ISO/TS 16181	0.083		-1.36	
2370	CPSC-CH-C1001-09.4	0.0793		-1.58	
2374	In house	0.085		-1.25	
2375	ISO/TS 16181	0.120		0.82	
2378	ISO/TS 16181	0.0950		-0.66	
2379	ISO/TS 16181	0.150		2.58	
2381		0.0915		-0.86	
2382	ISO/TS 16181	0.09		-0.95	
2386	ISO/TS 16181	0.120		0.82	
2390	ISO14389	0.091	C	-0.89	First reported 907.9 %M/M
2449		0.076		-1.78	
2455		-----		-----	
2497	ISO/TS 16181	0.0761		-1.77	
2511	ISO/TS 16181	0.170		3.76	
2563	ISO/TS 16181	0.178		4.23	
2590		-----		-----	
2592	ISO/TS 16181	0.57	C,R(0.01)	27.31	First reported <0.004
2643	CPSC-CH-C1001-09.3	0.128		1.29	
2656		-----		-----	
2675	ISO14389	0.058	C	-2.83	First reported 3.023
2695	ISO/TS 16181	0.1636		3.38	
2703	ISO14389	0	ex	-6.25	Test result exclude as zero is not a real test result
2705		-----		-----	
2734	ISO/TS 16181	nd		-----	
2756		-----		-----	
2758	ISO14389	0.111		0.29	
2773	ISO/TS 16181	0.12		0.82	
2810	ISO/TS 16181	0.09161		-0.86	
2812	ISO/TS 16181	0.122		0.93	
2883		-----		-----	
3146	In house	0.093		-0.77	
3150	In house	0.1102		0.24	
3154		-----		-----	
3160	ISO/TS 16181	0.10		-0.36	
3172	ISO/TS 16181	0.0666		-2.33	
3176	ISO/TS 16181	0.0723		-1.99	
3197	ISO/TS 16181	0.1260		1.17	
3210	In house	0.1333		1.60	
3243	In house	0.1053		-0.05	
					Only ISO/TS16181 data *):
	normality	OK			OK
	n	47			21
	outliers	1 (+1ex)			0
	mean (n)	0.10615			0.11987
	st.dev. (n)	0.031169	RSD = 29%		0.032784 RSD = 27%
	R(calc.)	0.08727			0.09179
	st.dev.(iis memo 1701)	0.016984			0.019179
	R(iis memo 1701)	0.04755			0.05370
Compare					
	R(ISO/TS16181:11)	0.03085			0.03484

*) Only data of participants that followed ISO/TS16181 to the letter, with a minimum sample intake of 0.5 gram

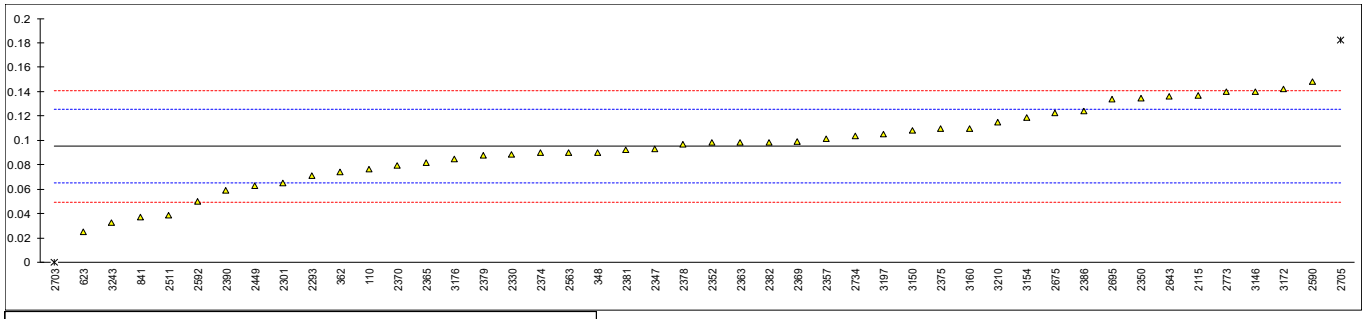

0.3
0.25
0.2
0.15
0.1
0.05
0
2703
841
2675
3172
3176
2301
2449
2497
551
2370
2365
348
2369
2374
110
2363
2382
2347
2352
2390
2381
2810
3146
2378
2357
3160
3243
3150
2758
2358
623
2375
2350
2386
2773
2115
2812
523
3197
2330
2643
3210
2293
2379
2695
2511
2563
362
2592


Kernel Density
14
12
10
8
6
4
2
0
-0.1
0
0.1
0.2
0.3

Determination of DMP – Dimethylphthalate on sample #19544; results in %M/M

lab	method	value	mark	z(targ)	remarks
110	In house	0.0767	C	-1.22	First reported ND
348	CPSC-CH1001-09.4	0.0904		-0.32	
362	ISO/TS 16181	0.074		-1.40	
523		-----		-----	
551		-----		-----	
623	ISO14389	0.025		-4.61	
841	CPSC-CH-C1001-09.4	0.036815	C	-3.84	First reported 368.15 %M/M
2115	ISO/TS 16181	0.137		2.74	
2293	CPSC-CH-C1001-09.3	0.071		-1.59	
2301	In house	0.065		-1.99	
2330	ISO14389	0.0884		-0.45	
2347	ISO/TS 16181	0.093		-0.15	
2350	CPSC-CH-C1001-09.4	0.135		2.60	
2352	ISO/TS 16181	0.0980		0.18	
2357	ISO/TS 16181	0.1016		0.41	
2358	ISO/TS 16181	N/A		-----	
2363	ISO/TS 16181	0.098		0.18	
2365	ISO/TS 16181	0.08146		-0.91	
2369	ISO/TS 16181	0.099		0.24	
2370	CPSC-CH-C1001-09.4	0.0797		-1.02	
2374	In house	0.090		-0.35	
2375	ISO/TS 16181	0.110		0.96	
2378	ISO/TS 16181	0.0970		0.11	
2379	ISO/TS 16181	0.088		-0.48	
2381		0.0924		-0.19	
2382	ISO/TS 16181	0.098		0.18	
2386	ISO/TS 16181	0.124		1.88	
2390	ISO14389	0.0593	C	-2.36	First reported 593 %M/M
2449		0.063		-2.12	
2455		-----		-----	
2497		-----		-----	
2511	ISO/TS 16181	0.039		-3.69	
2563	ISO/TS 16181	0.09		-0.35	
2590	ISO/TS 16181	0.14861		3.50	
2592	ISO/TS 16181	0.05	C	-2.97	First reported 0.004
2643	CPSC-CH-C1001-09.3	0.136		2.67	
2656		-----		-----	
2675	ISO14389	0.1227		1.80	
2695	ISO/TS 16181	0.1340		2.54	
2703	ISO14389	0	ex	-6.25	Test results excluded as zero is not a real test result
2705	In house	0.18208	R(0.05)	5.69	
2734	ISO/TS 16181	0.1038		0.56	
2756		-----		-----	
2758		-----		-----	
2773	ISO/TS 16181	0.14		2.93	
2810		-----		-----	
2812		-----		-----	
2883		-----		-----	
3146	In house	0.14		2.93	
3150	In house	0.1084		0.86	
3154	In house	0.119		1.56	
3160	ISO/TS 16181	0.11		0.96	
3172	ISO/TS 16181	0.1419		3.06	
3176	ISO/TS 16181	0.0847		-0.69	
3197	ISO/TS 16181	0.1050		0.64	
3210	In house	0.115		1.29	
3243	In house	0.0328		-4.10	
					Only ISO/TS16181 data *):
	normality	OK			suspect
	n	44			20
	outliers	1 (+1 excl)			0
	mean (n)	0.09529			0.10382
	st.dev. (n)	0.031153	RSD = 33%		0.025152 RSD = 24%
	R(calc.)	0.08723			0.07043
	st.dev.(iis memo 1701)	0.015246			0.016612
	R(iis memo 1701)	0.04269			0.04651
Compare					
	R(ISO/TS16181:2011)	0.02769			0.03018

*) Only data of participants that followed ISO/TS16181 to the letter, with a minimum sample intake of 0.5 gram


0.2
0.18
0.16
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
2703
623
3243
841
2511
2592
2390
2449
2301
2293
362
110
2370
2365
3176
2379
2330
2374
2563
348
2381
2347
2378
2352
2363
2382
2369
2357
2734
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3150
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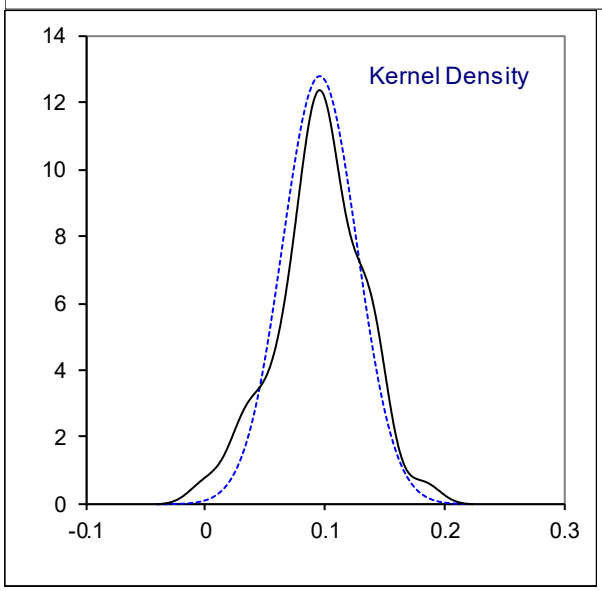
2350
2643
2115
2773
3146
3172
2590
2705


Kernel Density
14
12
10
8
6
4
2
0
-0.1
0
0.1
0.2
0.3

APPENDIX 2

Summary of other Phthalates in sample #19543: results in %M/M

BBP = Benzylbutylphthalate
DEHP = Bis-2-ethylhexylphthalate
DBP = Dibutylphthalate
DINP = Diisononylphthalate
DNOP = Di-n-Octylphthalate
DCHP = Dicyclohexylphthalate
DEP = Diethylphthalate

Lab	BBP	DEHP	DBP	DINP	DNOP	DCHP	DEP
110	ND	ND	ND	ND	ND	ND	ND
348	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
362	-----	-----	-----	-----	-----	-----	-----
523	<0.0025	<0.0025	<0.0025	<0.0025	<0.0025	<0.0025	-----
551	N.D.	0.0029	N.D.	0.0152	N.D.	N.D.	N.D.
623	n.d.	n.d.	n.d.	0.008	n.d.	n.d.	n.d.
841	ND	ND	ND	0.006412	ND	ND	ND
2115	-----	-----	-----	0.0028	-----	-----	-----
2293	<0.009	<0.009	<0.009	<0.009	<0.009	<0.009	<0.009
2301	ND	ND	ND	ND	ND	ND	ND
2330	ND	ND	ND	0.0122	ND	ND	ND
2347	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2350	-----	-----	-----	0.00970	-----	-----	-----
2352	-----	-----	-----	-----	-----	-----	-----
2357	ND	ND	ND	ND	ND	ND	ND
2358	n.d.	n.d.	n.d.	n.d.	n.d.	N/A	N/A
2363	ND	ND	ND	ND	ND	ND	ND
2365	<0.003	<0.003	<0.003	<0.010	<0.003	<0.003	<0.003
2369	<0.003	<0.003	<0.003	<0.01	<0.003	<0.003	<0.003
2370	< 0.0050	< 0.0050	< 0.0050	< 0.0050	< 0.0050	< 0.0050	< 0.0050
2374	-----	-----	-----	-----	-----	-----	-----
2375	-----	-----	-----	-----	-----	-----	-----
2378	-----	-----	-----	-----	-----	-----	-----
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2381	-----	-----	-----	-----	-----	-----	-----
2382	-----	-----	-----	-----	-----	-----	-----
2386	<0,005	<0,005	<0,005	0.011	<0,005	<0,005	<0,005
2390	-----	-----	-----	-----	-----	-----	-----
2449	-----	-----	-----	-----	-----	-----	-----
2455	-----	-----	-----	-----	-----	0.01168	-----
2497	-----	-----	-----	0.0031	-----	-----	0.1263
2511	-----	-----	-----	-----	-----	-----	-----
2563	ND	ND	ND	ND	ND	ND	ND
2590	-----	-----	-----	-----	-----	-----	-----
2592	< 0.004	< 0.004	0.0048	< 0.004	0.00788	< 0.004	<0.004
2643	-----	-----	-----	-----	-----	-----	-----
2656	-----	-----	-----	-----	-----	-----	-----
2675	-----	-----	0.0032	-----	-----	-----	0.0018
2695	-----	-----	-----	0.0168	-----	-----	-----
2703	0	0	0	0	0	0	0
2705	-----	0.0080	-----	-----	-----	-----	0.0069
2734	nd	nd	nd	nd	nd	nd	nd
2756	-----	-----	-----	-----	-----	-----	-----
2758	0	0	0	0.010	0	0	0
2773	ND	ND	ND	ND	ND	ND	ND
2810	-----	-----	-----	0.00492	-----	-----	-----
2812	-----	-----	-----	-----	-----	-----	-----
2883	-----	-----	-----	-----	-----	-----	-----
3146	-----	-----	-----	0.005	-----	-----	-----
3150	-----	0.0006	0.00028	0.0062	-----	-----	0.0001
3154	-----	-----	-----	-----	-----	-----	-----
3160	-----	-----	-----	0.01	-----	-----	-----
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	<0.002	<0.002	<0.002	0.0112	<0.002	<0.002	<0.002
3243	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

Summary of other Phthalates in sample #19543: results in %M/M - continued

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

Lab	DIBP	DPHP	DNPP	DUP	other
110	ND	-----	ND	-----	ND
348	<0.005	-----	<0.005	-----	-----
362	0.0054	-----	-----	-----	-----
523	<0.0025	-----	<0.0025	-----	-----
551	0.0020	N.D.	N.D.	-----	-----
623	n.d.	n.d.	n.d.	n.d.	n.d.
841	ND	ND	ND	ND	-----
2115	-----	-----	-----	-----	-----
2293	<0.009	<0.009	<0.009	<0.009	<0.009
2301	ND	ND	ND	ND	-----
2330	ND	ND	ND	ND	-----
2347	<0.005	<0.005	<0.005	<0.005	<0.005
2350	-----	-----	-----	-----	-----
2352	-----	-----	-----	-----	-----
2357	ND	ND	ND	ND	ND
2358	n.d.	N/A	N/A	N/A	N/A
2363	ND	ND	ND	ND	NA
2365	<0.003	<0.010	<0.003	<0.003	-----
2369	<0.003	<0.003	<0.003	<0.003	-----
2370	< 0.0050	< 0.0050	< 0.0050	< 0.0050	< 0.0050
2374	-----	-----	-----	-----	-----
2375	-----	-----	-----	-----	-----
2378	-----	-----	-----	-----	-----
2379	Not detected	Not detected	Not detected	Not detected	Not tested
2381	-----	-----	-----	-----	-----
2382	-----	-----	-----	-----	-----
2386	<0,005	<0,005	<0,005	<0,005	<0,005
2390	-----	-----	-----	-----	-----
2449	-----	-----	-----	-----	-----
2455	-----	-----	-----	-----	-----
2497	0.0017	-----	-----	-----	-----
2511	-----	-----	-----	-----	-----
2563	ND	-----	ND	-----	-----
2590	-----	-----	-----	-----	-----
2592	0.0068	< 0,004	< 0,004	< 0,004	0.1659
2643	-----	-----	-----	-----	-----
2656	-----	-----	-----	-----	-----
2675	0.0062	-----	-----	-----	0.0064
2695	-----	-----	-----	-----	-----
2703	0.002	0	0	0	0
2705	-----	-----	-----	-----	-----
2734	nd	nd	nd	nd	nd
2756	-----	-----	-----	-----	-----
2758	0.002	-----	-----	0	-----
2773	ND	ND	ND	ND	ND
2810	-----	-----	-----	-----	-----
2812	-----	-----	-----	-----	-----
2883	-----	-----	-----	-----	-----
3146	-----	-----	-----	-----	-----
3150	0.0018	-----	-----	-----	-----
3154	-----	-----	-----	-----	-----
3160	-----	-----	-----	-----	-----
3172	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
3176	-----	-----	-----	-----	-----
3197	ND	ND	ND	ND	-----
3210	0.0021	-----	<0.002	<0.002	-----
3243	n.d.	n.d.	n.d.	n.d.	n.d.

Summary of other Phthalates in sample #19544: results in %M/M

BBP = Benzylbutylphthalate
DEHP = Bis-2-ethylhexylphthalate
DBP = Dibutylphthalate
DIDP = Diisodecylphthalate
DNOP = Di-n-Octylphthalate
DCHP = Di-cyclohexylphthalate
DEP = Diethylphthalate

Lab	BBP	DEHP	DBP	DIDP	DNOP	DCHP	DEP
110	ND	ND	ND	0.0327	ND	ND	ND
348	<0.005	<0.005	<0.005	0.0204	<0.005	<0.005	<0.005
362	-----	-----	-----	-----	-----	-----	-----
523	<0.0025	<0.0025	<0.0025	<0.0025	<0.0025	<0.0025	-----
551	N.D.	N.D.	N.D.	0.0229	N.D.	N.D.	N.D.
623	n.d.	n.d.	n.d.	0.015	n.d.	n.d.	n.d.
841	ND	ND	ND	ND	ND	ND	ND
2115	-----	-----	-----	0.011	-----	-----	-----
2293	<0.009	<0.009	<0.009	<0.009	<0.009	<0.009	<0.009
2301	ND	ND	ND	ND	ND	ND	ND
2330	ND	ND	ND	0.0204	ND	ND	ND
2347	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2350	-----	-----	-----	-----	-----	-----	-----
2352	-----	-----	-----	-----	-----	-----	-----
2357	ND	ND	ND	ND	ND	ND	ND
2358	n.d.	n.d.	n.d.	0.01961	n.d.	N/A	N/A
2363	ND	ND	ND	ND	ND	ND	ND
2365	<0.003	<0.003	<0.003	<0.010	<0.003	<0.003	<0.003
2369	<0.003	<0.003	<0.003	<0.01	<0.003	<0.003	<0.003
2370	< 0.0050	< 0.0050	< 0.0050	< 0.0050	< 0.0050	< 0.0050	< 0.0050
2374	-----	-----	-----	-----	-----	-----	-----
2375	-----	-----	-----	0.015	-----	-----	-----
2378	-----	-----	-----	-----	-----	-----	-----
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2381	-----	-----	-----	-----	-----	-----	-----
2382	-----	-----	-----	-----	-----	-----	-----
2386	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
2390	-----	-----	-----	-----	-----	-----	-----
2449	-----	-----	-----	-----	-----	-----	-----
2455	-----	-----	-----	-----	-----	0.01206	-----
2497	-----	-----	-----	0.0289	-----	-----	-----
2511	-----	-----	-----	-----	-----	-----	-----
2563	ND	ND	ND	ND	ND	ND	ND
2590	-----	-----	-----	-----	-----	-----	-----
2592	< 0.004	< 0.004	0.0048	< 0.004	0.0074	< 0.004	<0.004
2643	-----	-----	-----	-----	-----	-----	-----
2656	-----	-----	-----	-----	-----	-----	-----
2675	-----	-----	-----	-----	-----	-----	0.0019
2695	-----	-----	-----	0.0265	-----	-----	-----
2703	0	0	0	0	0	0	0
2705	-----	0.0059	-----	-----	-----	-----	0.0060
2734	nd	nd	nd	nd	nd	nd	nd
2756	-----	-----	-----	-----	-----	-----	-----
2758	0	0	0	0.031	0	0	0
2773	ND	ND	ND	0.027	ND	ND	ND
2810	-----	-----	-----	0.01946	-----	-----	-----
2812	-----	-----	-----	-----	-----	-----	-----
2883	-----	-----	-----	-----	-----	-----	-----
3146	-----	-----	-----	0.018	-----	-----	-----
3150	-----	0.0007	-----	0.0229	-----	-----	0.0002
3154	-----	-----	-----	-----	-----	-----	-----
3160	-----	-----	-----	0.03	-----	-----	-----
3172	< 0.005	< 0.005	< 0.005	0.0080	< 0.005	< 0.005	< 0.005
3176	-----	-----	-----	-----	-----	-----	-----
3197	ND	ND	ND	ND	ND	ND	ND
3210	<0.002	<0.002	<0.002	<0.005	<0.002	<0.002	<0.002
3243	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

Summary of other Phthalates in sample #19544: results in %M/M - continued

DNHP = Di-n-hexylphthalate
DIBP = Diisobutylphthalate
DPHP = Di(2-propylheptyl)phthalate
DNPP = Di-n-Pentylphthalate
DUP = Diundecylphthalate
Other = other Phthalates

Lab	DNHP	DIBP	DPHP	DNPP	DUP	other
110	ND	ND	-----	ND	-----	ND
348	<0.005	<0.005	-----	<0.005	-----	-----
362	-----	0.0047	-----	-----	-----	-----
523	<0.0025	<0.0025	-----	<0.0025	-----	-----
551	-----	N.D.	N.D.	N.D.	-----	-----
623	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
841	0.003266	ND	ND	ND	ND	-----
2115	-----	-----	-----	-----	-----	-----
2293	<0.009	<0.009	<0.009	<0.009	<0.009	<0.009
2301	ND	ND	ND	ND	ND	-----
2330	ND	ND	ND	ND	ND	-----
2347	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
2350	-----	-----	-----	-----	-----	-----
2352	-----	-----	-----	-----	-----	-----
2357	ND	ND	ND	ND	ND	ND
2358	N/A	n.d.	N/A	N/A	N/A	N/A
2363	ND	ND	ND	ND	ND	ND
2365	<0.003	<0.003	<0.010	<0.003	<0.003	-----
2369	<0.003	<0.003	<0.003	<0.003	<0.003	-----
2370	< 0.0050	< 0.0050	< 0.0050	< 0.0050	< 0.0050	< 0.0050
2374	-----	-----	-----	-----	-----	-----
2375	-----	-----	-----	-----	-----	-----
2378	-----	-----	-----	-----	-----	-----
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not tested
2381	-----	-----	-----	-----	-----	-----
2382	-----	-----	-----	-----	-----	-----
2386	<0,005	<0,005	<0,005	<0,005	<0,005	<0,005
2390	-----	-----	-----	-----	-----	-----
2449	-----	-----	-----	-----	-----	-----
2455	0.01583	-----	-----	-----	-----	-----
2497	-----	-----	-----	-----	-----	-----
2511	-----	-----	-----	-----	-----	-----
2563	ND	ND	-----	ND	-----	-----
2590	-----	-----	-----	-----	-----	-----
2592	< 0.004	0.00516	< 0.004	< 0.004	< 0.004	0.1414
2643	-----	-----	-----	-----	-----	-----
2656	-----	-----	-----	-----	-----	-----
2675	-----	-----	-----	-----	-----	-----
2695	-----	-----	-----	-----	-----	-----
2703	0	0.001	0	0	0	0
2705	-----	-----	-----	-----	-----	-----
2734	nd	nd	nd	nd	nd	nd
2756	-----	-----	-----	-----	-----	-----
2758	0	0	-----	-----	0	-----
2773	ND	ND	ND	ND	ND	ND
2810	-----	-----	-----	-----	-----	-----
2812	-----	-----	-----	-----	-----	-----
2883	-----	-----	-----	-----	-----	-----
3146	-----	-----	-----	-----	-----	-----
3150	0.0007	0.0003	-----	-----	-----	-----
3154	-----	-----	-----	-----	-----	-----
3160	-----	-----	-----	-----	-----	-----
3172	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
3176	-----	-----	-----	-----	-----	-----
3197	ND	ND	ND	ND	ND	-----
3210	<0.002	<0.002	-----	<0.002	<0.002	-----
3243	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)
110	Yes	0.05	THF/Hexane	120	~22
348	No	0.5	Tetrahydrofuran	180	60±5
362	No	1.0	Hexane:Acetone 80:20	60	50
523	No	2.0 g	Hexane/Acetone	30	50
551	No	0.5	Hexano/Acetona	60	50
623	Yes	0.1 g	Tetrahydrofuran (THF)	60	60
841	Yes	0.1	THF/Hexane	180	60
2115	No	0.5	acetone/hexane	60	50
2293	Yes	0.050	Tetrahidrofurane (5 mL) and hexane (10 mL)	120	Room T
2301	Yes	0.3	THF	60	40
2330	No	0.5	THF:Hexane (Ratio 1:2)	60	60
2347	No				
2350	Yes	0.5	THF+ACN	60	60
2352	Yes	0.5	Hexane:Acetone=4:1	60	50
2357	Yes	0.5	n-Hexane:Acetone=8:2	60	50
2358	Yes	1	8:2 n-hexane:Acetone-20 ml n-hexane make up to 25 ml	60	50
2363	Yes	1	Mixture of n-hexane/acetone=80:20	60	60
2365	Yes	2	n-hexane:acetone(v/v)=4:1	60	50
2369	Yes	0.5	normal hexane:acetone=4:1	60	50
2370	Yes	0.1	THF : Hexane = 10 : 20 mL	30	Room T
2374	Yes	0.1	THF+hexane	60	60
2375	Yes	0.5	Hexane : Acetone (1:1)	60	50
2378	Yes	0.5	Hexane:acetone 4:1	60	50
2379	No	0.5	Hexane : Acetone (80:20)	60	50
2381	Yes	1	n-Hexane,Tetrahydrofuran(THF)	60	60
2382	Yes	0.5	Acetone/n-hexane=1:4	60	50
2386	Yes	0,5	Acetone/n-Hexane	60	70
2390	Yes	0.5	Tetrahydofurane and n-hexane	60	60
2449	Yes	0.3	ACN/THF	40	30
2455	---				
2497	Yes	1	hexane/acetone=80/20	60	50
2511	No	500mg	THF/CAN	1 hours	Ambient
2563	Yes	0.5	Tetrahydrofurane	60	60
2590	Yes	1.25	hexane/acetone mixture and after hexane	1 h	50
2592	---				
2643	Yes	0.3	THF + Hexane	60	70
2656	---				
2675	No	0,5	THF	60	60
2695	Yes	2	acetone / exane 20:80 mix	1h	50
2703	Yes	0.1	THF	60	60
2705	No	0.276	Hexane/Aceton	15	160
2734	Yes	1	Acetone/Hexane 20:80	60	50
2756	---				
2758	No	0.3	THF / Hexane	60	60
2773	Yes	1.0	hexane/acetone	1 hour	50
2810	Yes	2,00	n-hexane 80%/acetone 20%	60	50
2812	Yes	2	asetone (%20) hexane (%80)	1 hour	50
2883	---				
3146	Yes	0,5	Tetrahydrofurane / Acetonitrile	60	70
3150	No	2,0	THF/hexane	60	60
3154	Yes	0,3	n-hexane / acetone	60	60
3160	No	1	Acetone/n-hexane (20:80 %v/v)	60	50
3172	---				
3176	Yes				
3197	Yes	2	ACN/n-hexane	60	50
3210	Yes	1	Hexane/acetone (80/20)	60	50
3243	No	0.5	dichlormethane	30	Room T

APPENDIX 4

Number of participating laboratories per country

1 lab in BANGLADESH
1 lab in BRAZIL
1 lab in BULGARIA
1 lab in CAMBODIA
1 lab in ETHIOPIA
2 labs in FRANCE
7 labs in GERMANY
1 lab in GUATEMALA
1 lab in HONG KONG
1 lab in INDIA
2 labs in INDONESIA
8 labs in ITALY
2 labs in KOREA
1 lab in LUXEMBOURG
1 lab in MEXICO
9 labs in P.R. of CHINA
2 labs in PAKISTAN
2 labs in SPAIN
1 lab in SWITZERLAND
1 lab in TAIWAN R.O.C.
1 lab in THAILAND
1 lab in TUNISIA
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
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

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