

**Results of Proficiency Test
Phthalates in PVC
February 2007**

Organised by: Institute for Interlaboratory Studies
Spijkensisse, the Netherlands

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1 INTRODUCTION

Phthalates act as softeners and are commonly used as plasticizers in PVC. Phthalates may migrate fairly easily from PVC because they are not chemically bonded with PVC.

Because phthalates have negative effects on health and the environment, regulations have been set up.

In ASTM F963:03a (standard consumer safety specification on toy safety) § 4.3.8 is stated: "Pacifiers, rattles and teeters shall not intentionally contain DEHP (DOP)".

In Europe, the Commissioner for Industrial Affairs of the EC is responsible for toy regulations. The manufacture and import of toys is regulated by the European Union's Toy Directive (88/378), with in addition the general product safety, which is covered by EU directive 2001/95. These regulations govern conditions related to toys intended for children under 36 months of age (this group often suck or chew on toys and phthalates migrate easily). The regulations for migration are covered by EU directive 98/485, which described the maximum tolerance extractable quantity of migrated phthalates from toys for children under 36 months of age.

Beside the migration tolerance, manufactures are not allowed to bring toys into the EC market which are (partly) made of soft PVC and contain more than 0.1 %M/M of one of the following phthalates:

• di-isononylphthalate (DINP)	CASno. 28553-12-0	EINECS no. 249-079-5
• bis(2-ethylhexyl)phthalate (DEHP) ¹⁾	CASno. 117-81-7	EINECS no. 204-211-0
• di-n-octylphthalate (DNOP)	CASno. 117-84-0	EINECS no. 204-214-7
• di-isodecylphthalate (DIDP)	CASno. 26761-40-0	EINECS no. 247-977-1
• benzylbutylphthalate (BBP)	CASno. 85-68-7	EINECS no. 201-622-7
• dibutylphthalate (DBP)	CASno. 84-74-2	EINECS no. 201-557-4

Further more on request of several participants the following phthalates were added to the test scope of this PT:

• dipropylphthalate (DPrP)	CASno. 131-16-8	EINECS no. 205-015-8
• dipentylphthalate (DPP)	CASno. 131-18-0	EINECS no. 205-017-9
• dicyclohexylphthalate (DCHP)	CASno. 84-61-7	EINECS no. 201-545-9
• diethylphthalate (DEP)	CASno. 84-66-2	EINECS no. 201-550-6
• diheptylphthalate (DHP)	CASno. 3648-21-3	EINECS no. 222-885-4

¹⁾ DEHP is also known as di-iso-octylphthalate (DOP).

The determination of phthalates in PVC is known to give problems with the comparability of laboratory results. However, no appropriate PVC reference materials are available.

As an alternative, participation in a proficiency test may enable laboratories to check this comparability. Therefore, a proficiency test (laboratory-evaluating interlaboratory study) for the determination of total and individual phthalates in PVC was again organized by the Institute for Interlaboratory Studies in February 2007.

In the present international interlaboratory study of February 2007, 70 laboratories in 23 different countries have participated. See appendix 3 for a list of the number of participating laboratories per country. In this report the results of the proficiency test are presented and discussed.

2 SET UP

The Institute for Interlaboratory Studies (i.i.s.) in Spijkensisse, The Netherlands, was the organizer of this proficiency test. It was decided to send two different samples containing phthalates. Participants were requested to report results with one extra significant figure. These results with an extra figure are preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkensisse, the Netherlands, has implemented a quality system based on ISO-guide 43 and ILAC-G13:2000. This ensures 100% confidentiality of participant's data. Also customer's satisfaction is measured on a regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organization was the one as described for proficiency testing in the report 'i.i.s. Interlaboratory Studies: Protocol for the Organization, Statistics and Evaluation' of November 2003 (i.i.s.-protocol, version 3.0). The statistics were extended with methods from ISO13582:2005. The uncertainty in the assigned value (u (mean)), target reproducibilities (called Horwitz') and z' -scores were calculated, see also § 3.3.

2.3 SAMPLES

Two samples were prepared from two different bulk materials. The first bulk material (#0712) was blue-grey/white coloured PVC child toy. The second bulk material (#0713) was black coloured PVC floating beach toy. Both bulk materials were obtained from the local Dutch market. The bulk materials of #0712 and #0713 were cut into pieces and thoroughly mixed and subsequently distributed over plastic bags at random. The homogeneity of the subsamples was checked by determination of the total phthalates content by extraction of 4 stratified random selected subsamples using an in house method.

	DEHP in %M/M	
	Subsamples of 0712	Subsamples of 0713
Sample 1	40.7	35.4
Sample 2	38.3	37.7
Sample 3	39.8	36.1
Sample 4	39.9	37.6

Table 1: results of the homogeneity test on the subsamples 0712 and 0713

From the results of the homogeneity tests, the repeatability was calculated:

	DEHP in %M/M #0712	DEHP in %M/M #0713
r (observed)	2.4	2.8

Table 2: repeatability of DEHP content of the subsamples 0712 and 0713

For the determination of DEHP content an in-house extraction/GC-MS method was used. The calculated repeatability is in good agreement with the usual repeatability of the laboratory that performed the homogeneity tests. Therefore, homogeneity of subsamples 0712 and 0713 was assumed.

To each of the participating laboratories 2 samples were sent on February 14, 2007.

2.4 ANALYSIS

The participants were requested to determine eleven individual phthalates (DINP, DBP, BBP, DIDP, DNOP, DEHP, DprP, DPP, DCHP, DEP and DHP), other (OP) and total phthalates (TP) of each sample (0712 and 0713). They were explicitly asked to treat the samples as if they were routine samples and to report the analytical results using the indicated units on the report form and not to round the results, but report as much significant figures as possible. They were also asked not to report 'less than' results which are above the detection limit, because such results can not be used for meaningful statistical calculations.

To get comparable results a detailed report form, on which the units were prescribed, was sent together with each set of samples. Also a letter of instructions was added to the package.

The laboratories were asked to complete the report form with the requested details of the methods used.

3 RESULTS

During four weeks after sample despatch the results of the individual laboratories were received. The original data are tabulated per sample in the appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after deadline, a reminder fax was sent to those laboratories that did not report results at that moment.

Shortly after the deadline the available results were screened for suspect data. A 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 results. Additional or corrected results are used for the data analysis and the original results are placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

Statistical calculations were performed as described in the report 'i.i.s. Interlaboratory Studies: Protocol for the Organization, Statistics and Evaluation' of November 2003 (i.i.s.-protocol, version 3.0).

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test. After removal of outliers this check was repeated. All data sets proved to have a normal distribution.

In accordance with ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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 under the X-axis. The average of the reported data is presented by a straight line. The reproducibility limits of the selected standard, calculated as mean \pm target reproducibility, are presented by two striped lines parallel to the average line. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms.

(See appendix 4, nr.17 and 18)

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 spread of this interlaboratory study.

The standard uncertainty (u_x) was calculated from the proficiency test standard deviation in accordance with ISO13528, paragraph 5.6.2.

$$u_x = 1.23 * (\text{st.dev } (n)) / \sqrt{n}$$

In this proficiency test the standard uncertainty (u_x) of the assigned value is very large in comparison with the standard deviation for proficiency testing.

In ISO13528 is stated that if:

$$u_x \geq 0.3 * \text{standard deviation for proficiency testing}$$

the uncertainty of the assigned value is not negligible and need to be included in the interpretation of the results of the proficiency test. As this was the case in this study, z'-scores were calculated instead of the usual z-scores.

The z'-scores were calculated in accordance with ISO13528 paragraph 7.6.1:

$$z'_{(\text{target})} = (\text{result} - \text{mean of PT}) / \sqrt{((\text{standard deviation PT})^2 + (u_x)^2)}$$

The z'_(target) scores are listed in the result tables in appendix 1.

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

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

4 EVALUATION

In this interlaboratory study some problems were encountered during the execution. Two participants did not receive the samples on time and three participants did not send in any results. Finally 67 of the 70 participating laboratories did send in 394 numerical results. Observed were 31 outlying results, which is 7.9%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER SAMPLE

In this section the results are discussed per sample.

For comparison of the results of this Interlaboratory Study, the requirements from a standardised method like ASTM D3421:75, "Extraction and analysis of plasticizers mixtures from vinyl chloride plastics" should be used. Unfortunately, this method was discontinued in 1987, although it is still referred in ASTM F963:03 § 4.3.8 and no other validated determinations are published yet. However, in this method, the scope of the reproducibility requirements is not mentioned. The spreads found in this proficiency test are compared with the spreads estimated from the Horwitz equation. For all phthalates the Horwitz equation for one component is used, except for DINP, DIDP and Total Phthalates.

In most cases the uncertainty in the consensus value was very large and therefore the Horwitz spread was combined with the uncertainty to calculate the target reproducibility (see also § 3.3.) by the Horwitz' equation.

Some of the participants did not report a value for the “Total Phthalates”; therefore also a manual summation was made by i.i.s. for evaluation purposes.

Sample 0712: For this sample all participants reported positive on DEHP and detected low concentrations of DINP (except for 7 participants). Some of the reporting participants detected also low concentrations of DBP and DIDP. The levels of DBP and DIDP are between 0.006-0.094 %M/M (<0.1 %M/M). The calculated reproducibilities are, after rejection of the statistical outliers, not at all in agreement for DINP and DEHP with the target reproducibility (Horwitz'). For Total Phthalates “as reported”, eight results were outside the estimated reproducibility limits. The calculated reproducibility is, after rejection of the statistical outlier, not at all in agreement with the requirements estimated from the Horwitz' equation. When the calculated reproducibility for the Total Phthalates “after manual summation” was evaluated separately, the spread is somewhat larger. The calculated reproducibility is still not in agreement with the requirements estimated from the Horwitz' equation. Most participants reported a 'less than'-result or 'not detected' for the other phthalates.

Sample 0713: For this sample all participants reported positive on DEHP and detected low concentrations of DINP. Some of the reporting participants detected also low concentrations of DBP. The levels of DINP and DBP are between 0.036-0.098 %M/M (<0.1%M/M). The calculated reproducibilities are, after rejection of the statistical outliers, not at all in agreement for DINP and DEHP with the target reproducibility (Horwitz'). For total phthalates “as reported” eleven results were outside the estimated reproducibility limits and the calculated reproducibility is, after rejection of the statistical outliers, not in agreement with the requirements estimated from the Horwitz' equation. When the calculated reproducibility for the total Phthalates “after manual summation” was evaluated separately, the spread is somewhat smaller. The calculated reproducibility of total phthalates “after manual summation” is again not in agreement with the requirements estimated from the Horwitz' equation. Most participants reported a 'less than'-result or 'not detected' for the other phthalates.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibilities as found for the group of participating laboratories and the estimated target reproducibilities (Horwitz') are compared in the next tables:

Parameter	Unit	n	Average	2.8 * sd	R (target)
DINP	%M/M	37	0.405	0.765	0.220
DBP	%M/M	12	0.0063	0.0177	0.0065
DIDP	%M/M	36	0.0940	0.3859	0.0805
DEHP	%M/M	67	28.884	16.981	3.211
Total Phthalates as reported	%M/M	34	30.091	12.926	6.642
Total Phthalates *)	%M/M	65	30.176	14.342	6.454

Table 3: sample 0712

Parameter	Unit	n	Average	2.8 * sd	R (target)
DINP	%M/M	43	0.0983	0.1027	0.0506
DBP	%M/M	11	0.0036	0.0094	0.0036
DEHP	%M/M	67	26.689	14.190	2.805
Total Phthalates as reported	%M/M	36	25.293	16.374	6.211
Total Phthalates *)	%M/M	67	26.790	13.967	5.875

Table 4: sample 0713

*) = after manually summation of all reported results

4.3 COMPARISON WITH PREVIOUS INTERLABORATORY STUDIES

	February 2007	March 2006	March 2005	March 2004	March 2003
Number of reporting labs	67	51	38	39	26
Number of results	394	329	303	171	190
Statistical outliers	31	17	15	15	6
Percentage outliers	7.9%	5.2%	5.0%	8.8%	3.2%

Table 5: comparison with previous proficiency tests

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

The relative large spreads, which were found in the results of the phthalates in PVC samples during the present round, are in line compared with the spreads as observed in previous rounds (see table 6). Only the relative spreads for components found which were higher than 1.5% are taken into account. For components with an average less than 1.5%, the relative spreads were larger than 100%.

Parameter	February 2007	March 2006	March 2005	March 2004	March 2003
DINP	104% / 189%	76%	--	--	--
DBP	--	54%	54%	--	--
DEHP	53% / 59%	49% / 39%	59% / 49%	50% / 52%	24% / 27%
DIBP	--	38%	28%	--	--

Table 6: Relative reproducibilities of detected phthalates for last five years

It is hard to find any improvement over the years, perhaps the concentration of the components and the deviating composition of the group may be the cause of this.

5 CONCLUSIONS

The determination of phthalates in PVC seems again problematic like in previous years. The reported details of the methods, which were used by the participants, are listed in appendix 2. The technique to release the phthalates used by the participants was rather diverse, although a lot of participants used a method, which was related to ASTM D3421-75. However, for detecting and quantifying more common techniques were used: GC/MS, GC/FID and HPLC/DAD/MS.

The samples that were used in this proficiency test were relatively difficult, because of the high concentrations for DEHP.

In sample 0712, most participants detected DINP with a level of 0.4 %M/M, but 7 participants did reported a < results.

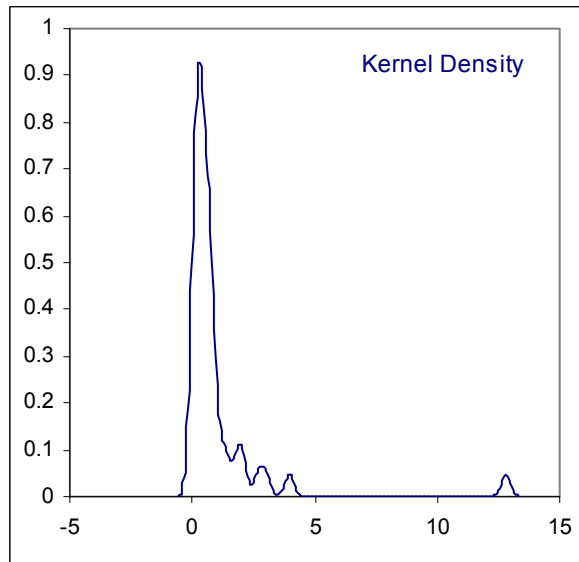
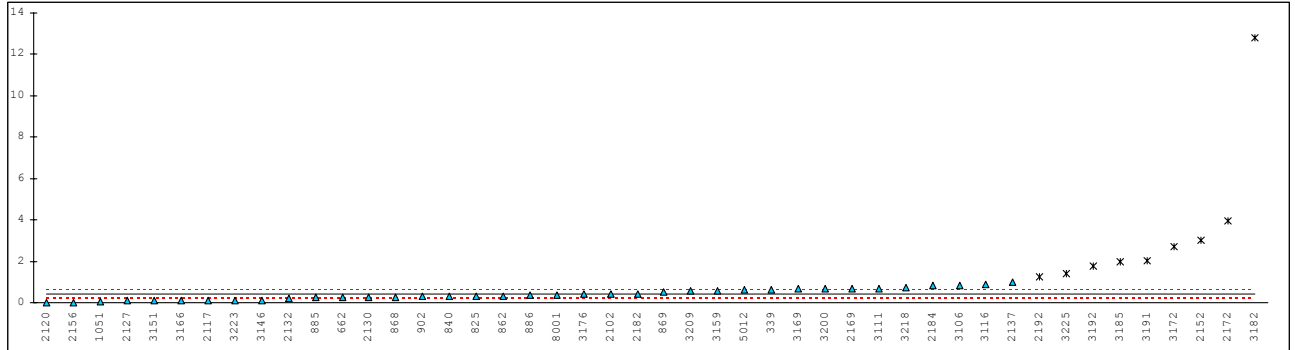
Finally, from the details of the analyses provided by the participants (see appendix 2) it is clear that the participants that reported values which are close to the mean values use a chlorinated solvent like Dichloromethane or Chloroform and used prolonged extraction at higher temperatures (e.g. Soxhlet) as mentioned in ASTM D3421 and Chromatographia No.784.

APPENDIX 1

Determination of DINP on sample 0712; results in %M/M

lab	method	value	mark	Z'(targ)	remarks
310	INHOUSE	<0.1		<-3.89	False negative
339	INHOUSE	0.62		2.74	
662	INHOUSE	0.25		-1.98	
825	EPA8061	0.315		-1.15	
840	D3421	0.3		-1.34	
852		----		----	
862	D3421	0.32		-1.08	
868	INHOUSE	0.28		-1.59	
869	D3421	0.53		1.59	
885	INHOUSE	0.235	C	-2.17	First reported 3.258
886	INHOUSE	0.34		-0.83	
902	D3421	0.29		-1.47	
917		----		----	
1051	D3421	0.045		-4.59	
1087		----		----	
2102	INHOUSE	0.416		0.14	
2117	INHOUSE	0.11		-3.76	
2120	EN14372	0		-5.16	
2127	INHOUSE	0.096		-3.94	
2129	INHOUSE	<0.01		<-5.04	False negative
2130	INHOUSE	0.26		-1.85	
2131		----		----	
2132	INHOUSE	0.210		-2.49	
2137	INHOUSE	1.005	C	7.65	First reported 1.592
2139		----		----	
2152	INHOUSE	3.01	C,G(0.05)	33.20	First reported 2.2
2156	EPA3550C	0.0010		-5.15	
2169	INHOUSE	0.687		3.59	
2172	INHOUSE	3.95	G(0.01)	45.18	
2173		----		----	
2182	EN14372	0.42		0.19	
2184	INHOUSE	0.821		5.30	
2190	INHOUSE	<0.03		<-4.78	False negative
2192	INHOUSE	1.263	DG(0.05)	10.93	
3106	EN14372	0.839		5.53	
3107		----		----	Results withdrawn, first reported 2.0
3109	INHOUSE	<0.1		<-3.89	False negative
3111	INHOUSE	0.7		3.76	
3116	D3421	0.90		6.31	
3134	INHOUSE	<0.0210		<-4.90	False negative
3146	INHOUSE	0.13		-3.51	
3147		----		----	
3150		----		----	
3151	INHOUSE	0.098		-3.91	
3153		----		----	
3154	INHOUSE	ND		----	
3159	INHOUSE	0.571		2.11	
3161		----		----	
3165	INHOUSE	<0.1		<-3.89	False negative
3166	INHOUSE	0.1		-3.89	
3169	INHOUSE	0.6604		3.25	
3172	INHOUSE	2.7	G(0.01)	29.25	
3173		----		----	
3176	EN14372	0.396	C	-0.12	First reported 0.02
3182	EN14372	12.8	G(0.01)	157.99	
3184		----		----	
3185	EN14372	1.98	DG(0.01)	20.07	
3191	INHOUSE	2.05	DG(0.01)	20.97	
3192	INHOUSE	1.75	G(0.05)	17.14	
3199	INHOUSE	<0.005		<-5.10	False negative
3200	D3421	0.686		3.58	
3209	INHOUSE	0.548	C	1.82	First reported 0.580
3217		----		----	
3218	EN14372	0.73		4.14	
3219		----		----	
3220		----		----	
3223	INHOUSE	0.12		-3.63	
3225	INHOUSE	1.3880	DG(0.05)	12.53	
5012	INHOUSE	0.60		2.48	
8001	D3421	0.36		-0.58	

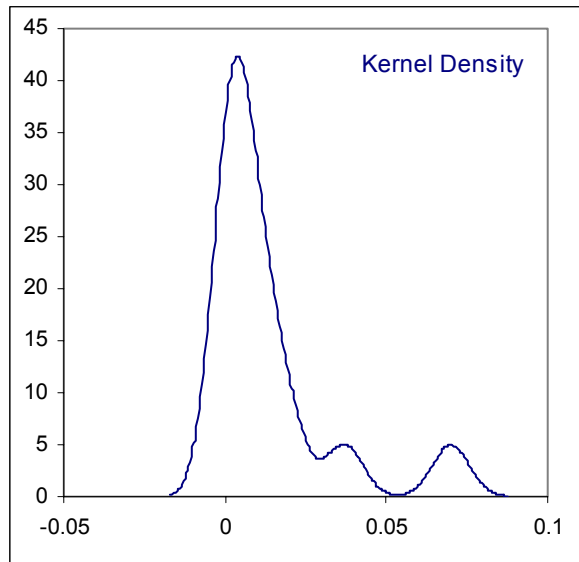
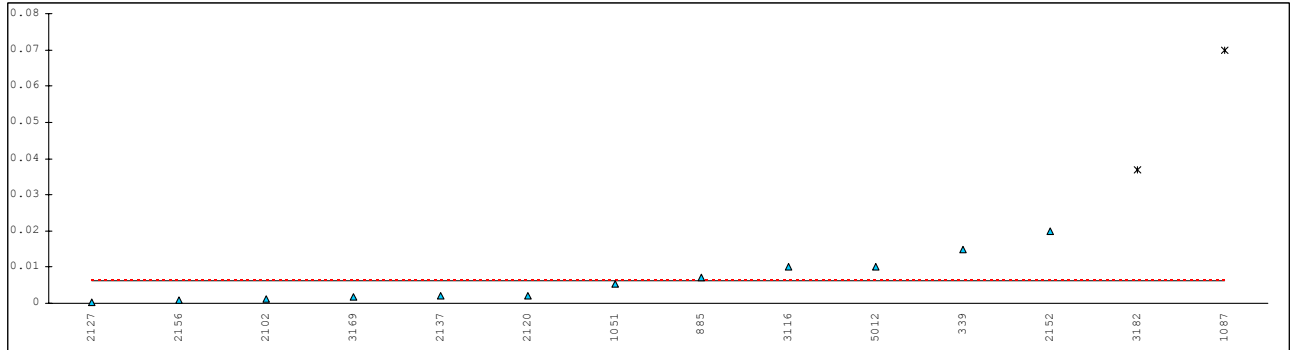
normality	OK
n	37
outliers	9
mean (n)	0.405
st.dev. (n)	0.2733
R(calc.)	0.765
R(Horwitz')	0.220
U(mean)	0.055



Determination of DBP on sample 0712; results in %M/M

lab	method	value	mark	Z(targ)	remarks
310	INHOUSE	<0.1		----	
339	INHOUSE	0.015		----	
662	INHOUSE	ND		----	
825	EPA8061	<0.0005		----	
840	D3421	ND		----	
852		----		----	
862	D3421	<0.003		----	
868	INHOUSE	<0.005		----	
869	D3421	ND		----	
885	INHOUSE	0.00723		----	
886		----		----	
902	INHOUSE	ND		----	
917	INHOUSE	ND		----	
1051	D3421	0.0054		----	
1087	INHOUSE	0.07	G(0.01)	----	
2102	INHOUSE	0.0011		----	
2117		----		----	
2120	EN14372	0.002		----	
2127	INHOUSE	0.0003		----	
2129	INHOUSE	<0.01		----	
2130		----		----	
2131		----		----	
2132	INHOUSE	<0.005		----	
2137	INHOUSE	0.002		----	
2139		----		----	
2152	INHOUSE	0.02		----	
2156	EPA3550C	0.0010		----	
2169	INHOUSE	<0.01		----	
2172	INHOUSE	ND		----	
2173		----		----	
2182	EN14372	<0.01		----	
2184	INHOUSE	ND		----	
2190	INHOUSE	<0.01		----	
2192	INHOUSE	<0.003		----	
3106	EN14372	<0.01		----	
3107	EN14372	<0.01		----	
3109	INHOUSE	<0.1		----	
3111		----		----	
3116	D3421	0.01		----	
3134	INHOUSE	<0.1		----	
3146	INHOUSE	<0.1		----	
3147		----		----	
3150		----		----	
3151	INHOUSE	<0.005		----	
3153		----		----	
3154	INHOUSE	ND		----	
3159	INHOUSE	<0.005		----	
3161		----		----	
3165	INHOUSE	<0.05		----	
3166		----		----	
3169	INHOUSE	0.0019		----	
3172	INHOUSE	<0.005		----	
3173		----		----	
3176		----		----	
3182	EN14372	0.037	G(0.01)	----	
3184	D3421	<0.01		----	
3185	EN14372	<0.01		----	
3191	INHOUSE	<0.01		----	
3192	INHOUSE	ND		----	
3199	INHOUSE	<0.005		----	
3200	D3421	<0.001		----	
3209	INHOUSE	<0.005		----	
3217		----		----	
3218		----		----	
3219		----		----	
3220		----		----	
3223	INHOUSE	<0.01		----	
3225	INHOUSE	<0.005		----	
5012	INHOUSE	0.01		----	
8001	INHOUSE	ND		----	

normality	not OK
n	12
outliers	2
mean (n)	0.0063
st.dev. (n)	0.00632
R(calc.)	0.0177
R(Horwitz')	0.0065
U(mean)	0.0022

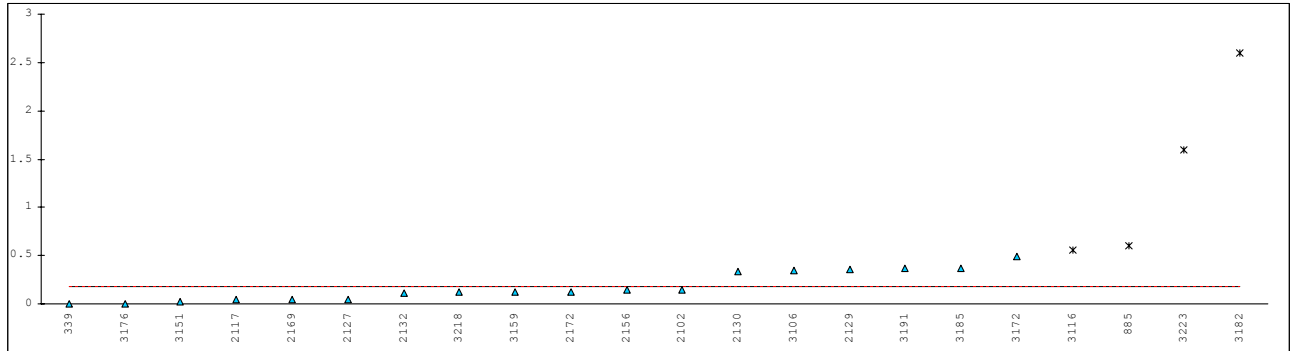


Determination of DIDP on sample 0712; results in %M/M

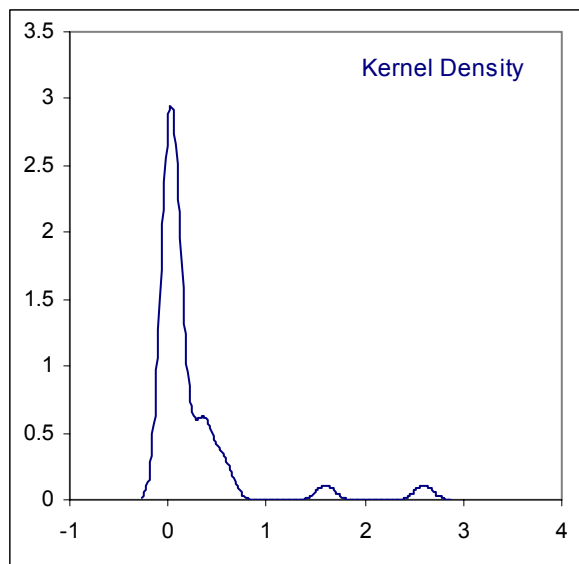
lab	method	value	mark	Z'(targ)*	remarks
310	INHOUSE	<0.1		----	
339	INHOUSE	0		----	
662	INHOUSE	ND		----	
825	EPA8061	<0.002		----	
840	D3421	ND		----	
852		----		----	
862	D3421	<0.01		----	
868	INHOUSE	<0.010		----	
869	INHOUSE	ND		----	
885	INHOUSE	0.607		----	
886		----		----	
902	INHOUSE	ND		----	
917	INHOUSE	ND		----	
1051	D3421	<0.005		----	
1087		----		----	
2102	INHOUSE	0.145		----	
2117	INHOUSE	0.04		----	
2120		----		----	
2127	INHOUSE	0.044		----	
2129	INHOUSE	0.36		----	
2130	INHOUSE	0.33		----	
2131		----		----	
2132	INHOUSE	0.107		----	
2137	INHOUSE	<0.001		----	
2139		----		----	
2152		----		----	
2156	EPA3550C	0.143		----	
2169	INHOUSE	0.0412		----	
2172	INHOUSE	0.124		----	
2173		----		----	
2182	EN14372	<0.01		----	
2184	INHOUSE	ND		----	
2190	INHOUSE	<0.03		----	
2192	INHOUSE	<0.01		----	
3106	EN14372	0.346		----	
3107	INHOUSE	ND		----	
3109	INHOUSE	<0.1		----	
3111		----		----	
3116	D3421	0.56		----	
3134	INHOUSE	<0.0010		----	
3146	INHOUSE	<0.01		----	
3147		----		----	
3150		----		----	
3151	INHOUSE	0.023		----	
3153		----		----	
3154	INHOUSE	ND		----	
3159	INHOUSE	0.121		----	
3161		----		----	
3165	INHOUSE	<0.1		----	
3166		----		----	
3169	INHOUSE	<0.01		----	
3172	INHOUSE	0.49		----	
3173		----		----	
3176	EN14372	0.0036		----	
3182	EN14372	2.6	G(0.01)	----	False positive
3184		----		----	
3185	EN14372	0.37		----	
3191	INHOUSE	0.37		----	
3192	INHOUSE	ND		----	
3199	INHOUSE	<0.005		----	
3200	D3421	<0.001		----	
3209	INHOUSE	<0.005		----	
3217		----		----	
3218	EN14372	0.12		----	
3219		----		----	
3220		----		----	
3223	INHOUSE	1.60	G(0.01)	----	False positive
3225	INHOUSE	<0.005		----	
5012		----		----	
8001	INHOUSE	ND		----	

	<u>All results (*):</u>
normality	not OK
n	36
outliers	4
mean (n)	0.0940
st.dev. (n)	0.13782
R(calc.)	0.3859
R(Horwitz)	0.0911
U(mean)	0.0283

*) In the calculation of the mean, standard deviation, the reproducibility and below graph, a reported value of <x is changed in x/2 (for example <1 into 0.5).



All results (*)

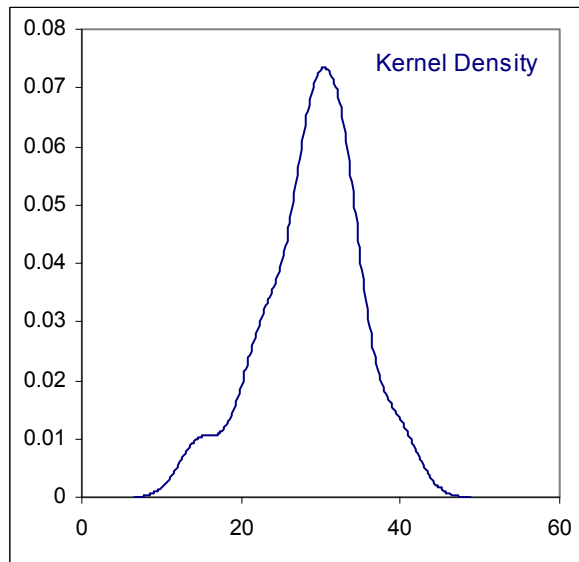
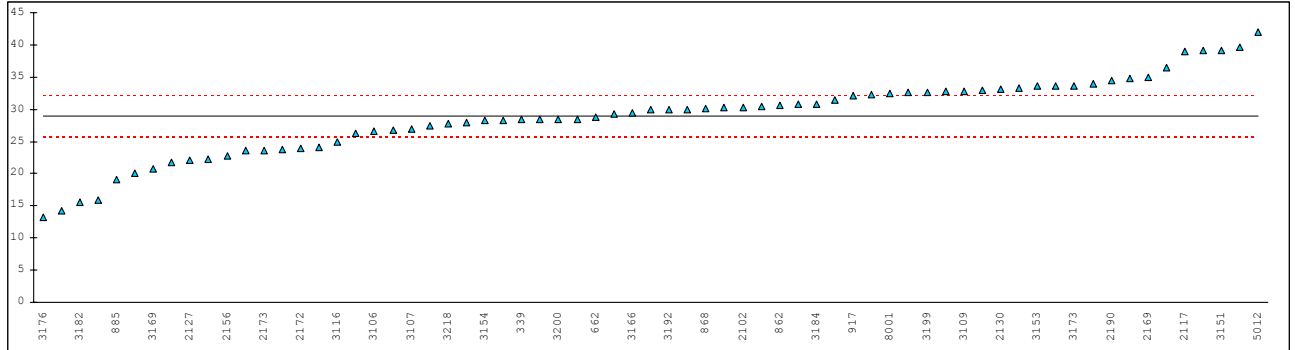


All results (*)

Determination of DEHP on sample 0712; results in %M/M

lab	method	value	mark	Z'(targ)	remarks
310	INHOUSE	39.7		9.43	
339	INHOUSE	28.40		-0.42	
662	INHOUSE	28.75		-0.12	
825	EPA8061	28.5		-0.33	
840	D3421	27.46		-1.24	
852	D3421	33		3.59	
862	D3421	30.60		1.50	
868	INHOUSE	30.10		1.06	
869	D3421	29.94		0.92	
885	INHOUSE	19.1		-8.53	
886	INHOUSE	33.6		4.11	
902	D3421	30.7		1.58	
917	INHOUSE	32.2		2.89	
1051	D3421	30.5		1.41	
1087	INHOUSE	34.73		5.10	
2102	INHOUSE	30.30		1.23	
2117	INHOUSE	38.9		8.73	
2120	EN14372	36.4		6.55	
2127	INHOUSE	22		-6.00	
2129	INHOUSE	28.32		-0.49	
2130	INHOUSE	33.2		3.76	
2131	INHOUSE	26.20		-2.34	
2132	D3421	33.22		3.78	
2137	INHOUSE	22.216		-5.81	
2139		----		----	
2152	INHOUSE	28.40		-0.42	
2156	EPA3550C	22.83		-5.28	
2169	INHOUSE	35		5.33	
2172	INHOUSE	23.9		-4.35	
2173	INHOUSE	23.6		-4.61	
2182	EN14372	28		-0.77	
2184	INHOUSE	26.8		-1.82	
2190	INHOUSE	34.5		4.90	
2192	INHOUSE	39.11		8.92	
3106	EN14372	26.63		-1.96	
3107	EN14372	27		-1.64	
3109	INHOUSE	32.8		3.41	
3111	INHOUSE	14.2		-12.80	
3116	D3421	25	C	-3.39	First reported 17.31
3134	INHOUSE	32.24		2.93	
3146	INHOUSE	21.8		-6.18	
3147	INHOUSE	34.0		4.46	
3150	INHOUSE	32.8		3.41	
3151	INHOUSE	39.2		8.99	
3153	D3421	33.6		4.11	
3154	INHOUSE	28.31		-0.50	
3159	INHOUSE	23.7		-4.52	
3161		----		----	
3165	INHOUSE	29.9		0.89	
3166	INHOUSE	29.4		0.45	
3169	INHOUSE	20.79		-7.06	
3172	INHOUSE	32.6		3.24	
3173	INHOUSE	33.7		4.20	
3176	EN14372	13.28	C	-13.60	First reported 9.649
3182	EN14372	15.5		-11.67	
3184	D3421	30.79		1.66	
3185	EN14372	29.3		0.36	
3191	INHOUSE	30.2		1.15	
3192	INHOUSE	29.93		0.91	
3199	INHOUSE	32.65		3.28	
3200	D3421	28.4		-0.42	
3209	INHOUSE	24.051	C	-4.21	First reported 8.240
3217	INHOUSE	31.46		2.25	
3218	EN14372	27.84		-0.91	
3219		----		----	
3220	INHOUSE	20		-7.75	
3223	INHOUSE	15.93		-11.29	
3225	INHOUSE	23.5207		-4.68	
5012	INHOUSE	42.00		11.44	
8001	D3421	32.5		3.15	

normality OK
 n 67
 outliers 0
 mean (n) 28.884
 st.dev. (n) 6.0647
 R(calc.) 16.981
 R(Horwitz') 3.211
 U(mean) 0.911

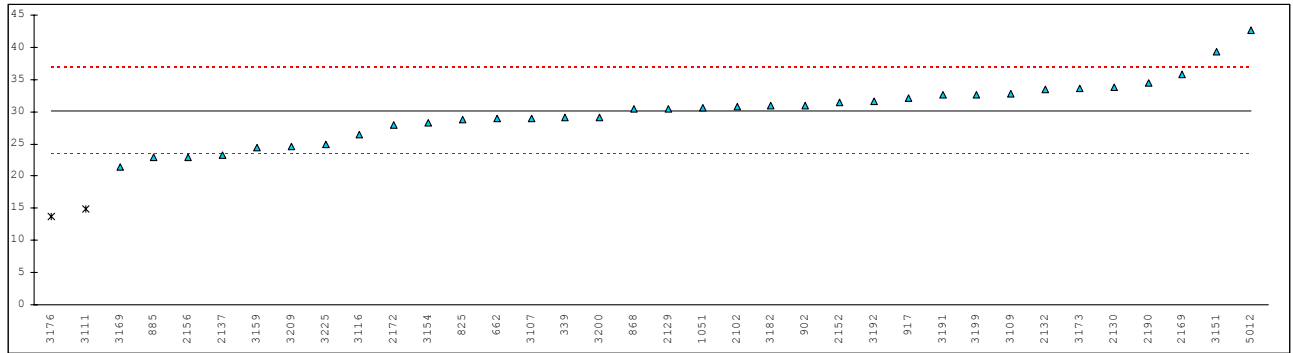


Determination of Total Phthalate on sample 0712; results in %M/M

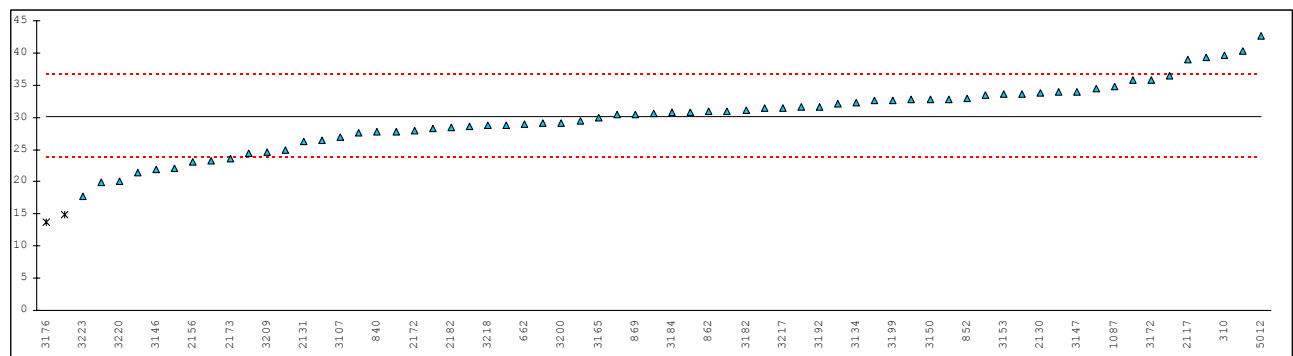
lab	method	value	mark	Z'(targ)	value *)	mark	Z'(targ)
310		----		----	39.7		4.13
339	INHOUSE	29.035		-0.45	29.04		-0.49
662	INHOUSE	29.00		-0.46	29.00		-0.51
825	EPA8061	28.8		-0.54	28.815		-0.59
840		----		----	27.76		-1.05
852		----		----	33		1.23
862		----		----	30.92		0.32
868	INHOUSE	30.38		0.12	30.38		0.09
869		----		----	30.47		0.13
885	INHOUSE	22.98		-3.00	19.9555		-4.43
886		----		----	33.94		1.63
902	D3421	30.99		0.38	30.99		0.35
917	INHOUSE	32.2		0.89	32.2		0.88
1051	D3421	30.55		0.19	30.5504		0.16
1087		----		----	34.8		2.01
2102	INHOUSE	30.86		0.32	30.8621		0.30
2117		----		----	39.05		3.85
2120		----		----	36.402		2.70
2127		----		----	22.1403		-3.49
2129	INHOUSE	30.39		0.13	28.68		-0.65
2130	INHOUSE	33.8		1.56	33.79		1.57
2131		----		----	26.2		-1.72
2132	INHOUSE	33.54		1.45	33.537		1.46
2137	INHOUSE	23.223	C	-2.90	23.223		-3.02
2139		----		----	----		----
2152	INHOUSE	31.43	C	0.56	31.43		0.54
2156	EPA3550C	23.00		-2.99	23.007		-3.11
2169	INHOUSE	35.8		2.41	35.7		2.41
2172	INHOUSE	28.0		-0.88	27.974		-0.96
2173		----		----	23.6		-2.85
2182		----		----	28.42		-0.76
2184		----		----	27.621		-1.11
2190	INHOUSE	34.5		1.86	34.5		1.88
2192		----		----	40.373		4.42
3106		----		----	27.815		-1.02
3107	EN14372	29.0		-0.46	27		-1.38
3109	INHOUSE	32.8		1.14	32.8		1.14
3111	INHOUSE	14.9	DG(0.05)	-6.40	14.9	DG(0.05)	-6.63
3116	D3421	26.51	C	-1.51	26.51		-1.59
3134		----		----	32.24		0.90
3146		----		----	21.93		-3.58
3147		----		----	34		1.66
3150		----		----	32.8		1.14
3151	INHOUSE	39.33		3.89	39.331		3.97
3153		----		----	33.6		1.49
3154	INHOUSE	28.31		-0.75	28.31		-0.81
3159	INHOUSE	24.4		-2.40	24.392		-2.51
3161		----		----	----		----
3165		----		----	29.9		-0.12
3166		----		----	29.5		-0.29
3169	INHOUSE	21.45		-3.64	21.4523		-3.78
3172		----		----	35.79		2.44
3173	INHOUSE	33.7		1.52	33.7		1.53
3176	EN14372	13.679	C,DG(0.05)	-6.92	13.6796	DG(0.05)	-7.16
3182	EN14372	30.98		0.37	31.03421		0.37
3184		----		----	30.79		0.27
3185		----		----	31.65		0.64
3191	INHOUSE	32.6		1.06	32.62		1.06
3192	INHOUSE	31.68		0.67	31.68		0.65
3199	INHOUSE	32.65		1.08	32.65		1.07
3200	D3421	29.086		-0.42	29.086		-0.47
3209	INHOUSE	24.599	C	-2.32	24.599		-2.42
3217		----		----	31.46		0.56
3218		----		----	28.69		-0.64
3219		----		----	----		----
3220		----		----	20		-4.41
3223		----		----	17.65		-5.43
3225	INHOUSE	24.9087		-2.18	24.9087		-2.29
5012	INHOUSE	42.623		5.28	42.623		5.40
8001		----		----	32.86		1.16

normality	OK	<u>All results (*):</u>
n	34	OK
outliers	2	65
mean (n)	30.091	2
st.dev. (n)	4.6164	30.176
R(calc.)	12.926	5.1220
R(Horwitz')	6.642	14.342
U(mean)	0.974	6.454
		0.781

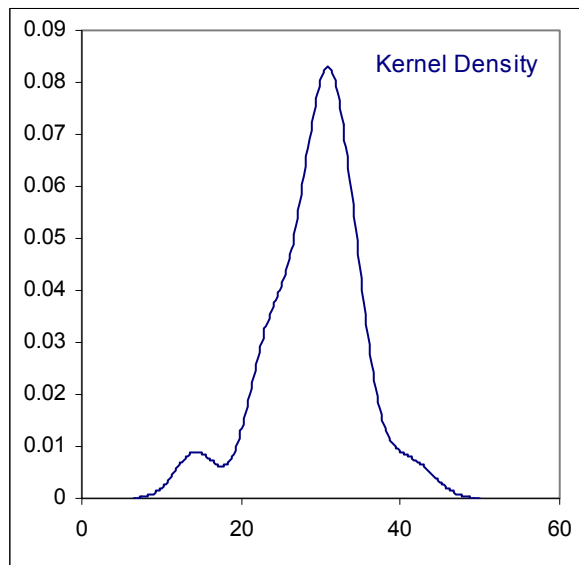
*) After manual summation of all reported results



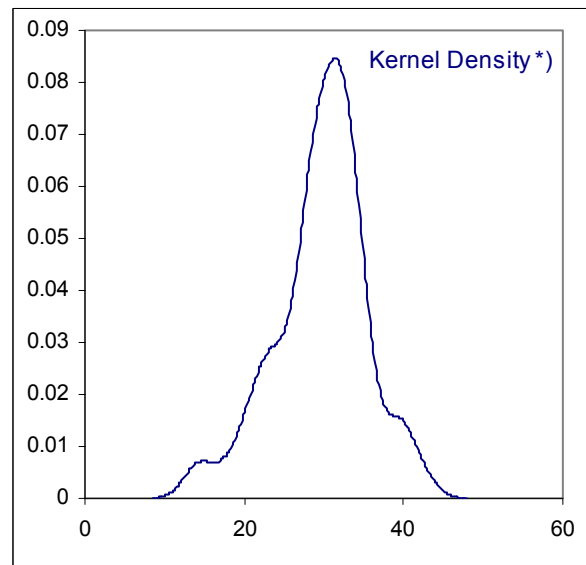
Original reported



After manual summation of all reported results



Original reported

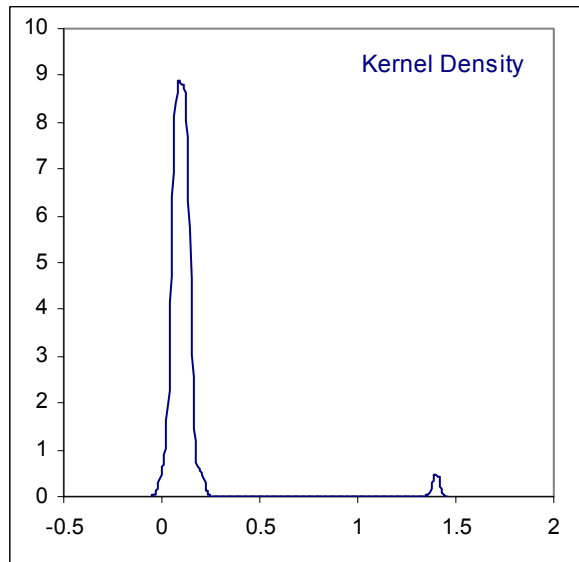
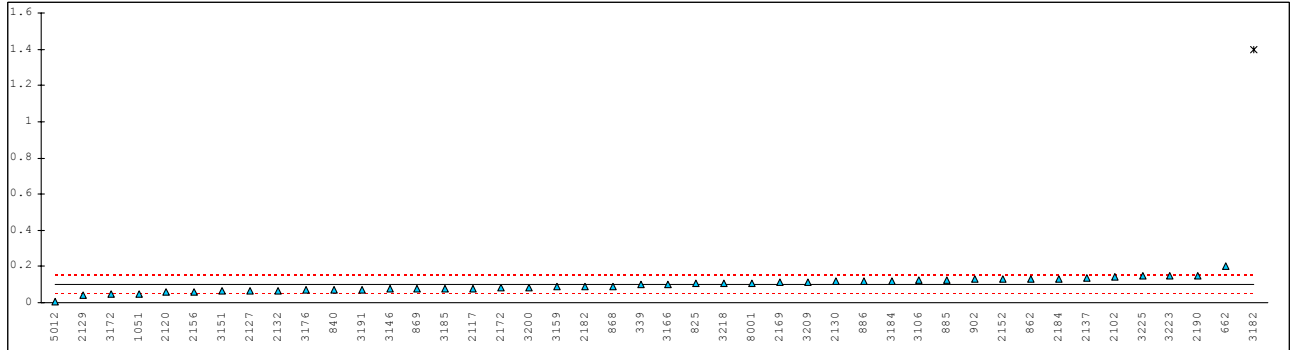


After manual summation of all reported results

Determination of DINP on sample 0713; results in %M/M

lab	method	value	mark	Z'(targ)	remarks
310	INHOUSE	<0.1		----	
339	INHOUSE	0.10		0.10	
662	INHOUSE	0.20		5.63	
825	EPA8061	0.106		0.43	
840	D3421	0.07		-1.56	
852		----		----	
862	D3421	0.13		1.75	
868	INHOUSE	0.09		-0.46	
869	D3421	0.08		-1.01	
885	INHOUSE	0.124		1.42	
886	INHOUSE	0.12		1.20	
902	D3421	0.13		1.75	
917		----		----	
1051	D3421	0.05		-2.67	
1087		----		----	
2102	INHOUSE	0.140		2.31	
2117	INHOUSE	0.08		-1.01	
2120	EN14372	0.06		-2.12	
2127	INHOUSE	0.066		-1.78	
2129	INHOUSE	0.04		-3.22	
2130	INHOUSE	0.12		1.20	
2131		----		----	
2132	INHOUSE	0.067		-1.73	
2137	INHOUSE	0.137		2.14	
2139		----		----	
2152	INHOUSE	0.13		1.75	
2156	EPA3550C	0.0600	C	-2.12	First reported 0.0010
2169	INHOUSE	0.111		0.70	
2172	INHOUSE	0.0808		-0.97	
2173		----		----	
2182	EN14372	0.09		-0.46	
2184	INHOUSE	0.131		1.81	
2190	INHOUSE	0.15		2.86	
2192	INHOUSE	<0.01		----	
3106	EN14372	0.122		1.31	
3107	EN14372	ND		----	
3109	INHOUSE	<0.1		----	
3111		----		----	
3116	D3421	<0.01		----	
3134	INHOUSE	<0.1		----	
3146	INHOUSE	0.08		-1.01	
3147		----		----	
3150		----		----	
3151	INHOUSE	0.063		-1.95	
3153		----		----	
3154	INHOUSE	ND		----	
3159	INHOUSE	0.090		-0.46	
3161		----		----	
3165	INHOUSE	<0.1		----	
3166	INHOUSE	0.1		0.10	
3169	INHOUSE	<0.01		----	
3172	INHOUSE	0.05		-2.67	
3173		----		----	
3176	EN14372	0.069		-1.62	
3182	EN14372	1.4	G(0.01)	72.00	
3184	D3421	0.121		1.26	
3185	EN14372	0.08		-1.01	
3191	INHOUSE	0.07		-1.56	
3192	INHOUSE	ND		----	
3199	INHOUSE	<0.01		----	
3200	D3421	0.086		-0.68	
3209	INHOUSE	0.113	C	0.81	First reported 0.115
3217		----		----	
3218	EN14372	0.11		0.65	
3219		----		----	
3220		----		----	
3223	INHOUSE	0.15		2.86	
3225	INHOUSE	0.1458		2.63	
5012	INHOUSE	0.003		-5.27	
8001	D3421	0.11		0.65	

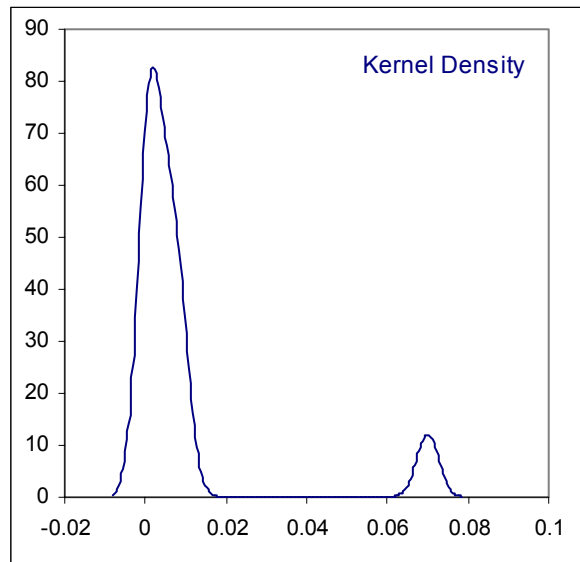
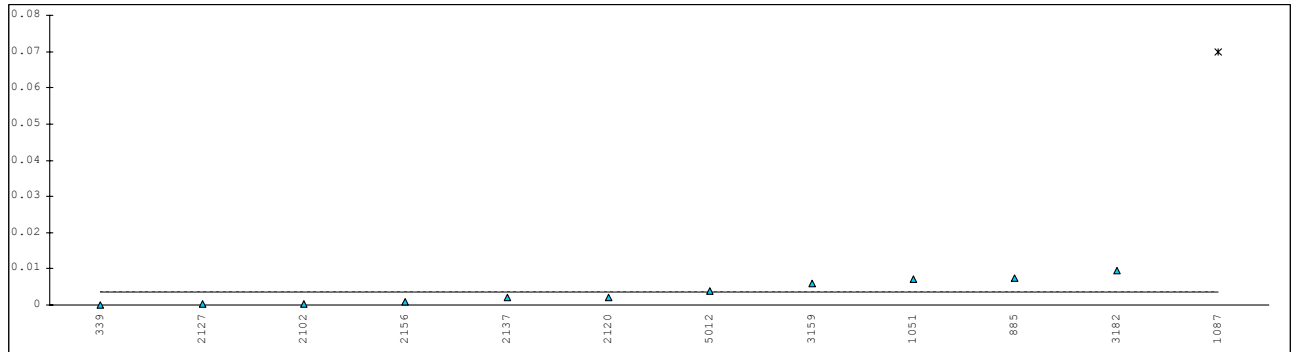
normality OK
 n 43
 outliers 1
 mean (n) 0.0983
 st.dev. (n) 0.03667
 R(calc.) 0.1027
 R(Horwitz') 0.0506
 U(mean) 0.0069



Determination of DBP on sample 0713; results in %M/M

lab	method	value	mark	Z'(targ)	remarks
310	INHOUSE	<0.1		----	
339	INHOUSE	0		----	
662	INHOUSE	ND		----	
825	EPA8061	<0.0005		----	
840	D3421	ND		----	
852		----		----	
862	D3421	<0.003		----	
868	INHOUSE	<0.005		----	
869	INHOUSE	ND		----	
885	INHOUSE	0.0074		----	
886		----		----	
902	INHOUSE	ND		----	
917	INHOUSE	ND		----	
1051	D3421	0.007		----	
1087	INHOUSE	0.07	G(0.01)	----	
2102	INHOUSE	0.00039		----	
2117		----		----	
2120	EN14372	0.002		----	
2127	INHOUSE	0.0003		----	
2129	INHOUSE	<0.01		----	
2130		----		----	
2131		----		----	
2132	INHOUSE	<0.005		----	
2137	INHOUSE	0.002		----	
2139		----		----	
2152		----		----	
2156	EPA3550C	0.0010		----	
2169	INHOUSE	<0.01		----	
2172	INHOUSE	ND		----	
2173		----		----	
2182	EN14372	<0.01		----	
2184	INHOUSE	ND		----	
2190	INHOUSE	<0.01		----	
2192	INHOUSE	<0.003		----	
3106	EN14372	<0.01		----	
3107	INHOUSE	ND		----	
3109	INHOUSE	<0.1		----	
3111		----		----	
3116	D3421	<0.01		----	
3134	INHOUSE	<0.1		----	
3146	INHOUSE	<0.01		----	
3147		----		----	
3150		----		----	
3151	INHOUSE	<0.005		----	
3153		----		----	
3154	INHOUSE	ND		----	
3159	INHOUSE	0.006		----	
3161		----		----	
3165	INHOUSE	<0.05		----	
3166		----		----	
3169	INHOUSE	<0.001		----	
3172	INHOUSE	<0.005		----	
3173		----		----	
3176		----		----	
3182	EN14372	0.0094		----	
3184	D3421	<0.01		----	
3185	EN14372	<0.01		----	
3191	INHOUSE	<0.01		----	
3192	INHOUSE	ND		----	
3199	INHOUSE	<0.005		----	
3200	D3421	<0.001		----	
3209	INHOUSE	<0.005		----	
3217		----		----	
3218		----		----	
3219		----		----	
3220		----		----	
3223	INHOUSE	<0.01		----	
3225	INHOUSE	<0.005		----	
5012	INHOUSE	0.004		----	
8001	INHOUSE	ND		----	

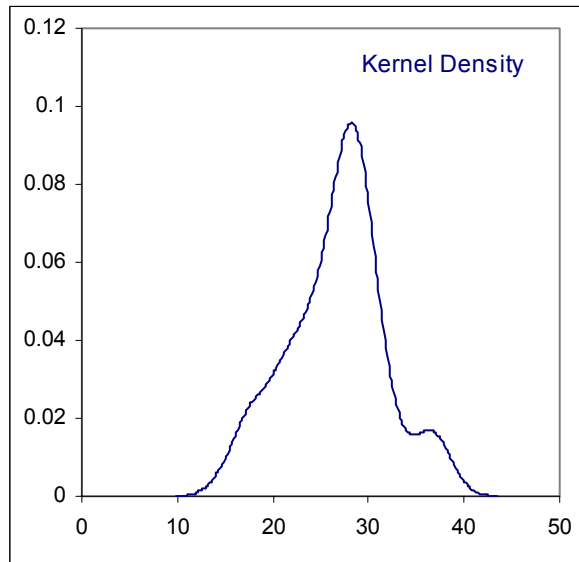
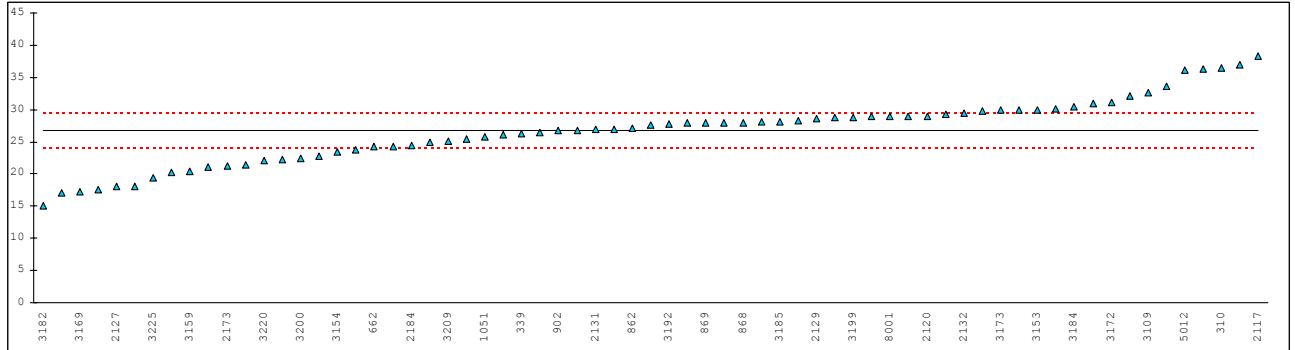
normality	OK
n	11
outliers	1
mean (n)	0.0036
st.dev. (n)	0.00334
R(calc.)	0.0094
R(Horwitz')	0.0036
U(mean)	0.0012



Determination of DEHP on sample 0713; results in %M/M

lab	method	value	mark	Z'(targ)	remarks
310	INHOUSE	36.4		9.69	
339	INHOUSE	26.20		-0.49	
662	INHOUSE	24.20		-2.48	
825	EPA8061	27.0		0.31	
840	D3421	27.96		1.27	
852	D3421	31		4.30	
862	D3421	27.18		0.49	
868	INHOUSE	28.00		1.31	
869	D3421	27.91		1.22	
885	INHOUSE	17.5		-9.17	
886	INHOUSE	28.8		2.11	
902	D3421	26.7		0.01	
917	INHOUSE	27.9		1.21	
1051	D3421	25.7		-0.99	
1087	INHOUSE	26.16		-0.53	
2102	INHOUSE	24.33		-2.35	
2117	INHOUSE	38.3		11.59	
2120	EN14372	29		2.31	
2127	INHOUSE	18		-8.67	
2129	INHOUSE	28.58		1.89	
2130	INHOUSE	30.1		3.40	
2131	INHOUSE	27.00	C	0.31	First reported 40.50
2132	D3421	29.38		2.69	
2137	INHOUSE	26.355	C	-0.33	First reported 6.955
2139		----		----	
2152	INHOUSE	27.6		0.91	
2156	EPA3550C	21.45		-5.23	
2169	INHOUSE	29.8		3.10	
2172	INHOUSE	25		-1.69	
2173	INHOUSE	21.3		-5.38	
2182	EN14372	29		2.31	
2184	INHOUSE	24.5		-2.19	
2190	INHOUSE	33.6		6.90	
2192	INHOUSE	36.22		9.51	
3106	EN14372	25.49		-1.20	
3107	EN14372	17		-9.67	
3109	INHOUSE	32.6		5.90	
3111	INHOUSE	18.1		-8.57	
3116	D3421	21	C	-5.68	First reported 12.23
3134	INHOUSE	29.22		2.53	
3146	INHOUSE	20.2		-6.48	
3147	INHOUSE	30.0		3.30	
3150	INHOUSE	32.2		5.50	
3151	INHOUSE	37.0		10.29	
3153	D3421	30.0		3.30	
3154	INHOUSE	23.45		-3.23	
3159	INHOUSE	20.4		-6.28	
3161		----		----	
3165	INHOUSE	28.1		1.41	
3166	INHOUSE	26.8		0.11	
3169	INHOUSE	17.15		-9.52	
3172	INHOUSE	31.1		4.40	
3173	INHOUSE	29.9		3.20	
3176	EN14372	22.32	C	-4.36	First reported 15.247
3182	EN14372	15.0		-11.67	
3184	D3421	30.51		3.81	
3185	EN14372	28.1		1.41	
3191	INHOUSE	28.3		1.61	
3192	INHOUSE	27.75		1.06	
3199	INHOUSE	28.83		2.14	
3200	D3421	22.5		-4.18	
3209	INHOUSE	25.114	C	-1.57	First reported 6.950
3217	INHOUSE	28.91		2.22	
3218	EN14372	23.81		-2.87	
3219		----		----	
3220	INHOUSE	22		-4.68	
3223	INHOUSE	22.67		-4.01	
3225	INHOUSE	19.3890		-7.29	
5012	INHOUSE	36.15		9.44	
8001	D3421	29		2.31	

normality	OK
n	67
outliers	0
mean (n)	26.689
st.dev. (n)	5.0678
R(calc.)	14.190
R(Horwitz')	2.805
U(mean)	0.762

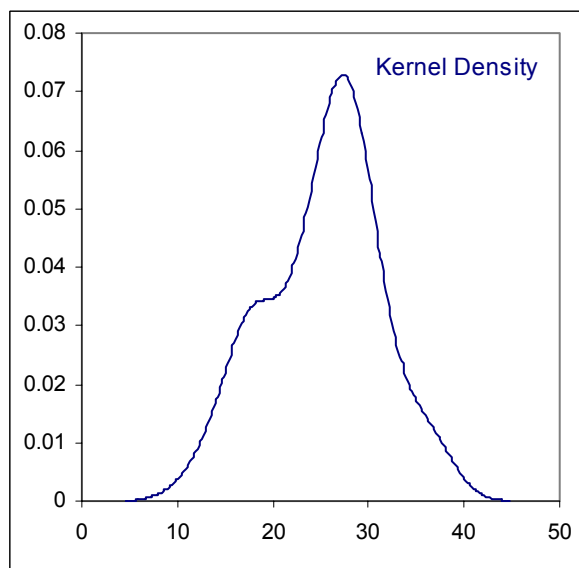
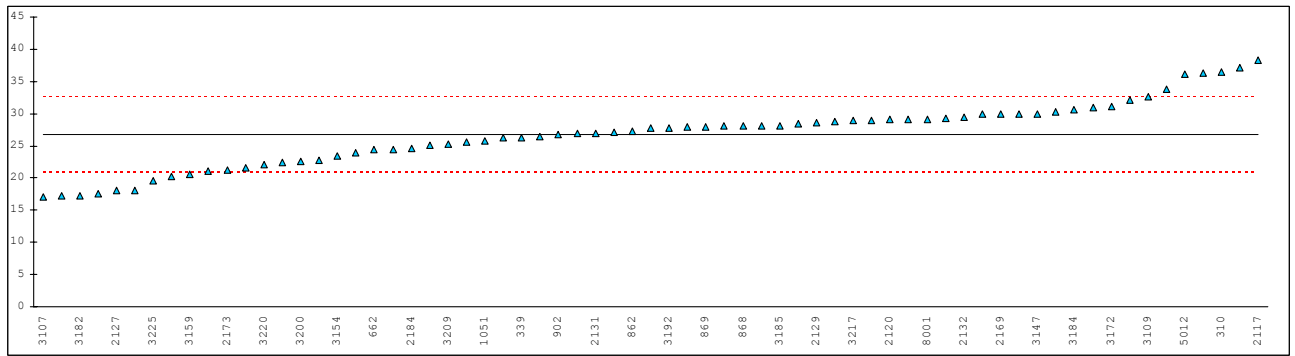
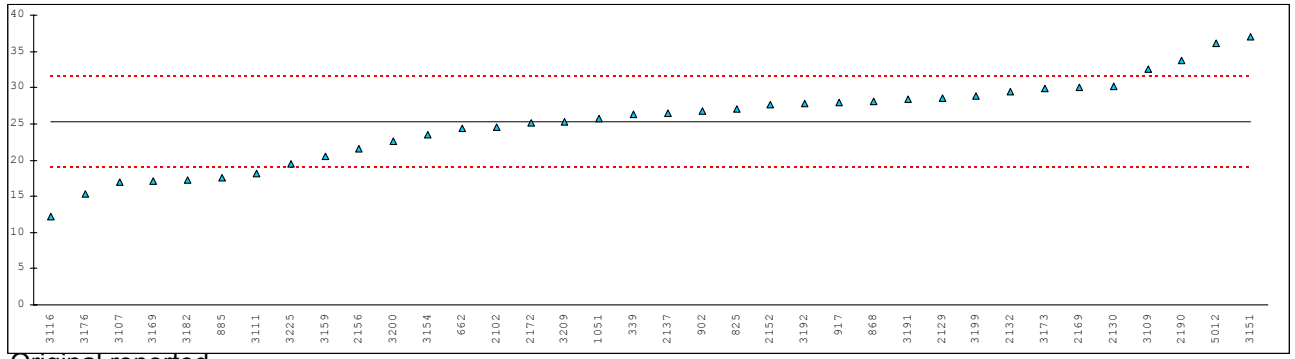


Determination of Total Phthalate on sample 0713; results in %M/M

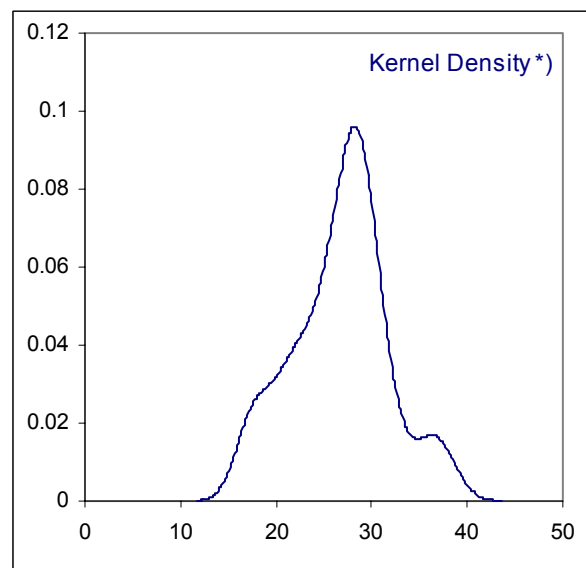
lab	method	value	mark	Z'(targ)	value *)	mark	Z'(targ)	remarks
310		----		----	36.4		4.58	
339	INHOUSE	26.30		0.45	26.30		-0.23	
662	INHOUSE	24.40		-0.40	24.40		-1.14	
825	EPA8061	27.1		0.81	27.106		0.15	
840		----		----	28.03		0.59	
852		----		----	31		2.01	
862		----		----	27.31		0.25	
868	INHOUSE	28.09		1.26	28.09		0.62	
869		----		----	27.99		0.57	
885	INHOUSE	17.6		-3.47	17.6352		-4.36	
886		----		----	28.92		1.02	
902	D3421	26.83		0.69	26.83		0.02	
917	INHOUSE	27.9		1.18	27.9		0.53	
1051	D3421	25.757		0.21	25.757		-0.49	
1087		----		----	26.23		-0.27	
2102	INHOUSE	24.47		-0.37	24.47039		-1.11	
2117		----		----	38.38		5.52	
2120		----		----	29.062		1.08	
2127		----		----	18.0663		-4.16	
2129	INHOUSE	28.62		1.50	28.62		0.87	
2130	INHOUSE	30.2		2.21	30.22		1.63	
2131		----		----	27		0.10	
2132	INHOUSE	29.45		1.87	29.447		1.27	
2137	INHOUSE	26.494	C	0.54	26.494	C	-0.14	First reported 7.094
2139		----		----	----		----	
2152	INHOUSE	27.73		1.10	27.73		0.45	
2156	EPA3550C	21.55	C	-1.69	21.559	C	-2.49	First reported 21.49
2169	INHOUSE	30.0		2.12	29.9		1.49	
2172	INHOUSE	25.1		-0.09	25.0808		-0.81	
2173		----		----	21.3		-2.62	
2182		----		----	29.09		1.10	
2184		----		----	24.631		-1.03	
2190	INHOUSE	33.8		3.84	33.75		3.32	
2192		----		----	36.22		4.49	
3106		----		----	25.612		-0.56	
3107	EN14372	17.02		-3.73	17.02		-4.66	
3109	INHOUSE	32.6		3.29	32.6		2.77	
3111	INHOUSE	18.1		-3.24	18.1		-4.14	
3116	D3421	12.26		-5.88	21.03		-2.75	
3134		----		----	29.221		1.16	
3146		----		----	20.28		-3.10	
3147		----		----	30		1.53	
3150		----		----	32.2		2.58	
3151	INHOUSE	37.07		5.31	37.069		4.90	
3153		----		----	30		1.53	
3154	INHOUSE	23.45		-0.83	23.45		-1.59	
3159	INHOUSE	20.5		-2.16	20.503		-3.00	
3161		----		----	----		----	
3165		----		----	28.1		0.62	
3166		----		----	26.9		0.05	
3169	INHOUSE	17.15		-3.67	17.15		-4.59	
3172		----		----	31.15		2.08	
3173	INHOUSE	29.9		2.08	29.9		1.48	
3176	EN14372	15.316		-4.50	22.389		-2.10	
3182	INHOUSE	17.29		-3.61	17.28832		-4.53	
3184		----		----	30.631		1.83	
3185		----		----	28.18		0.66	
3191	INHOUSE	28.4		1.40	28.37		0.75	
3192	INHOUSE	27.75		1.11	27.75		0.46	
3199	INHOUSE	28.83		1.59	28.83		0.97	
3200	D3421	22.586		-1.22	22.586		-2.00	
3209	INHOUSE	25.227	C	-0.03	25.227	C	-0.74	First reported 7.065
3217		----		----	28.91		1.01	
3218		----		----	23.92		-1.37	
3219		----		----	----		----	
3220		----		----	22		-2.28	
3223		----		----	22.82		-1.89	
3225	INHOUSE	19.5348		-2.60	19.5348		-3.46	
5012	INHOUSE	36.161		4.90	36.161		4.47	
8001		----		----	29.11		1.11	

normality	OK	<u>All results (*):</u>
n	36	OK
outliers	0	67
mean (n)	25.293	26.790
st.dev. (n)	5.8479	4.9880
R(calc.)	16.374	13.967
R(Horwitz')	6.211	5.875
U(mean)	1.199	0.750

*) After manual summation of all reported results



Original reported



After manual summation of all reported results

Determination of other Phthalates not listed before on sample 0712; results in %M/M

lab	method	BBP	mark	DNOP	mark	DPrP	mark	DPP	mark
310	INHOUSE	<0.1		<0.1		----		----	
339	INHOUSE	0		0		----		----	
662	INHOUSE	ND		ND		ND		ND	
825	EPA8061	<0.0005		<0.0005		----		----	
840	D3421	ND		ND		----		----	
852		----		----		----		----	
862	D3421	<0.003		<0.003		----		----	
868	INHOUSE	<0.005		<0.005		<0.005		<0.005	
869	INHOUSE	ND		ND		----		----	
885	INHOUSE	<0.003		<0.003		0.00627		<0.003	
886		----		----		----		----	
902	INHOUSE	ND		ND		----		----	
917	INHOUSE	ND		ND		----		----	
1051	D3421	<0.005		<0.005		<0.005		<0.005	
1087		----		----		----		----	
2102	INHOUSE	ND		ND		DT		ND	
2117		----		----		----		----	
2120		----		----		----		----	
2127	INHOUSE	<0.0001		<0.0001		----		----	
2129	INHOUSE	<0.01		<0.01		----		----	
2130		----		----		----		----	
2131		----		----		----		----	
2132	INHOUSE	<0.005		<0.005		<0.005		<0.005	
2137	INHOUSE	<0.001		<0.001		<0.001		<0.001	
2139		----		----		----		----	
2152		----		----		----		----	
2156	EPA3550C	0.0010		0.0010		0.0010		0.0010	
2169	INHOUSE	<0.01		<0.01		<0.01		<0.01	
2172	INHOUSE	ND		ND		ND		ND	
2173		----		----		----		----	
2182	EN14372	<0.01		<0.01		<0.01		<0.01	
2184	INHOUSE	ND		ND		----		----	
2190	INHOUSE	<0.01		<0.01		----		----	
2192	INHOUSE	<0.003		<0.003		----		----	
3106	EN14372	<0.01		<0.01		<0.01		<0.01	
3107	INHOUSE	ND		ND		ND		ND	
3109	INHOUSE	<0.1		<0.1		----		----	
3111		----		----		----		----	
3116	D3421	<0.01		<0.01		<0.01		<0.01	
3134	INHOUSE	<0.1		<0.1		----		----	
3146	INHOUSE	<0.01		<0.01		<0.01		----	
3147		----		----		----		----	
3150		----		----		----		----	
3151	INHOUSE	<0.005		<0.005		<0.005		<0.005	
3153	INHOUSE	ND		----		----		----	
3154		----		ND		ND		ND	
3159	INHOUSE	<0.005		<0.005		<0.005		<0.005	
3161		----		----		----		----	
3165	INHOUSE	<0.05		<0.05		<0.05		<0.05	
3166		----		----		----		----	
3169	INHOUSE	<0.001		<0.001		<0.001		<0.001	
3172	INHOUSE	<0.005		<0.005		----		----	
3173		----		----		----		----	
3176		----		----		----		----	
3182	EN14372	0.00048		0.067		0.00049		0.0052	
3184	D3421	<0.01		<0.01		<0.01		<0.01	
3185	EN14372	<0.01		<0.01		<0.01		<0.01	
3191	INHOUSE	<0.01		<0.01		<0.01		<0.01	
3192	INHOUSE	ND		ND		----		----	
3199	INHOUSE	<0.005		<0.005		<0.01		<0.01	
3200	D3421	<0.001		<0.001		----		----	
3209	INHOUSE	<0.005		<0.005		<0.005		<0.005	
3217		----		----		----		----	
3218		----		----		----		----	
3219		----		----		----		----	
3220		----		----		----		----	
3223	INHOUSE	<0.01		<0.01		----		----	
3225	INHOUSE	<0.005		<0.005		<0.005		<0.005	
5012		----		----		----		----	
8001	INHOUSE	ND		ND		----		----	

normality	n.a.	n.a.	n.a.	n.a.
n	3	3	3	2
outliers	0	0	0	0
mean (n)	n.a.	n.a.	n.a.	n.a.
st.dev. (n)	n.a.	n.a.	n.a.	n.a.
R(calc.)	n.a.	n.a.	n.a.	n.a.
R(lit)	Unknown	Unknown	Unknown	Unknown

Determination of other Phthalates not listed before on sample 0712; results in %M/M (continued)

lab	method	DCHP	mark	DEP	mark	DHP	mark	OP	mark
310		----		----		----		----	
339		----		----		----		----	
662	INHOUSE	ND		ND		----		ND	
825		----		----		----		----	
840		----		----		----		----	
852		----		----		----		----	
862		----		----		----		----	
868	INHOUSE	<0.005		<0.005		<0.005		<0.005	
869		----		----		----		----	
885	INHOUSE	<0.003		<0.003		<0.003		ND	
886		----		----		----		----	
902		----		----		----		----	
917		----		ND		----		----	
1051	D3421	<0.005		<0.005		<0.005		----	
1087		----		----		----		----	
2102	INHOUSE	ND		ND		ND		----	
2117		----		----		----		----	
2120		----		----		----		----	
2127		----		<0.0001		----		----	
2129		----		----		----		----	
2130		----		----		----		----	
2131		----		----		----		----	
2132	INHOUSE	<0.005		<0.005		<0.005		----	
2137	INHOUSE	<0.001		<0.001		<0.001		<0.001	
2139		----		----		----		----	
2152		----		----		----		----	
2156	EPA3550C	0.0010		0.0010		0.0010		0.025	
2169	INHOUSE	<0.01		<0.01		<0.01		<0.01	
2172	INHOUSE	ND		ND		ND		----	
2173		----		----		----		----	
2182	EN14372	<0.01		<0.01		<0.01		----	
2184		----		----		----		----	
2190		----		----		----		----	
2192		----		----		----		----	
3106	EN14372	<0.01		<0.01		<0.01		----	
3107	INHOUSE	ND		ND		ND		ND	
3109		----		<0.1		----		----	
3111		----		----		----		----	
3116	D3421	0.04		<0.01		<0.01		----	
3134		----		----		----		----	
3146		----		<0.01		----		----	
3147		----		----		----		----	
3150		----		----		----		----	
3151	INHOUSE	<0.005		<0.005		<0.005		0.01	
3153		----		----		----		----	
3154	INHOUSE	ND		ND		ND		ND	
3159	INHOUSE	<0.005		<0.005		<0.005		NIL	
3161		----		----		----		----	
3165	INHOUSE	<0.05		<0.05		----		----	
3166		----		----		----		----	
3169	INHOUSE	<0.001		<0.001		<0.001		----	
3172		----		----		----		----	
3173		----		----		----		----	
3176		----		----		----		----	
3182	EN14372	0.018		0.00085		0.0049		0.00029	
3184	D3421	<0.01		<0.01		<0.01		----	
3185	EN14372	<0.01		<0.01		<0.01		----	
3191	INHOUSE	<0.01		<0.01		<0.01		----	
3192	INHOUSE	ND		ND		----		----	
3199	INHOUSE	<0.01		<0.01		<0.01		<0.01	
3200		----		----		----		----	
3209	INHOUSE	<0.005		<0.005		<0.005		<0.005	
3217		----		----		----		----	
3218		----		----		----		----	
3219		----		----		----		----	
3220		----		----		----		----	
3223		----		----		----		----	
3225	INHOUSE	<0.005		<0.005		<0.005		<0.005	
5012		----		----		----		0.013	
8001		----		----		----		----	

normality	n.a.	n.a.	n.a.	n.a.
n	3	2	2	4
outliers	0	0	0	0
mean (n)	n.a.	n.a.	n.a.	n.a.
st.dev. (n)	n.a.	n.a.	n.a.	n.a.
R(calc.)	n.a.	n.a.	n.a.	n.a.
R(lit)	Unknown	Unknown	Unknown	Unknown

Determination of other Phthalates not listed before on sample 0713; results in %M/M

lab	method	BBP	mark	DIDP	mark	DNOP	mark	DPrP	mark	DPP	mark
310	INHOUSE	<0.1		<0.1		<0.1		----		----	
339	INHOUSE	0		0		0		----		----	
662	INHOUSE	ND		ND		ND		ND		ND	
825	EPA8061	<0.0005		<0.002		<0.0005		----		----	
840	D3421	ND		ND		ND		----		----	
852		----		----		----		----		----	
862	D3421	<0.003		<0.01		<0.003		----		----	
868	INHOUSE	<0.005		<0.010		<0.005		<0.005		<0.005	
869	INHOUSE	ND		ND		ND		----		----	
885	INHOUSE	<0.003		<0.003		<0.003		0.0038		<0.003	
886		----		----		----		----		----	
902	INHOUSE	ND		ND		ND		----		----	
917	INHOUSE	ND		ND		ND		----		----	
1051	D3421	<0.005		<0.005		<0.005		<0.005		<0.005	
1087		----		----		----		----		----	
2102	INHOUSE	ND		ND		ND		DT		ND	
2117		----		----		----		----		----	
2120		----		----		----		----		----	
2127	INHOUSE	<0.0001		<0.001		<0.0001		----		----	
2129	INHOUSE	<0.01		<0.01		<0.01		----		----	
2130		----		----		----		----		----	
2131		----		----		----		----		----	
2132	INHOUSE	<0.005		<0.005		<0.005		<0.005		<0.005	
2137	INHOUSE	<0.001		<0.001		<0.001		<0.001		<0.001	
2139		----		----		----		----		----	
2152		----		----		----		----		----	
2156	EPA3550C	0.040		0.0010		0.0010		0.0010		0.0010	
2169	INHOUSE	<0.01		<0.01		<0.01		<0.01		<0.01	
2172	INHOUSE	ND		ND		ND		ND		ND	
2173		----		----		----		----		----	
2182	EN14372	<0.01		<0.01		<0.01		<0.01		<0.01	
2184	INHOUSE	ND		ND		ND		----		----	
2190	INHOUSE	<0.01		<0.03		<0.01		----		----	
2192	INHOUSE	<0.003		<0.01		<0.003		----		----	
3106	EN14372	<0.01		<0.01		<0.01		<0.01		<0.01	
3107	EN14372	0.02		ND		ND		ND		ND	
3109	INHOUSE	<0.1		<0.1		<0.1		----		----	
3111		----		----		----		----		----	
3116	D3421	<0.01		<0.01		<0.01		<0.01		<0.01	
3134	INHOUSE	0.0010		<0.1		<0.1		----		----	
3146	INHOUSE	<0.01		<0.01		<0.01		<0.01		----	
3147		----		----		----		----		----	
3150		----		----		----		----		----	
3151	INHOUSE	<0.005		<0.005		<0.005		<0.005		<0.005	
3153		----		----		----		----		----	
3154	INHOUSE	ND		ND		ND		ND		ND	
3159	INHOUSE	<0.005		0.007		<0.005		<0.005		<0.005	
3161		----		----		----		----		----	
3165	INHOUSE	<0.05		<0.1		<0.05		<0.05		<0.05	
3166		----		----		----		----		----	
3169	INHOUSE	<0.001		<0.01		<0.001		<0.001		<0.001	
3172	INHOUSE	<0.005		<0.005		<0.005		----		----	
3173		----		----		----		----		----	
3176		----		----		----		----		----	
3182	EN14372	0.25		0.6		0.00055		0.0016		0.0041	
3184	D3421	<0.01		<0.01		<0.01		<0.01		<0.01	
3185	EN14372	<0.01		<0.01		<0.01		<0.01		<0.01	
3191	INHOUSE	<0.01		<0.01		<0.01		<0.01		<0.01	
3192	INHOUSE	ND		ND		ND		----		----	
3199	INHOUSE	<0.005		<0.005		<0.005		<0.01		<0.01	
3200	D3421	<0.001		<0.001		<0.001		----		----	
3209	INHOUSE	<0.005		<0.005		<0.005		<0.005		<0.005	
3217		----		----		----		----		----	
3218		----		----		----		----		----	
3219		----		----		----		----		----	
3220		----		----		----		----		----	
3223	INHOUSE	<0.01		<0.01		<0.01		----		----	
3225	INHOUSE	<0.005		<0.005		<0.005		<0.005		<0.005	
5012		----		----		----		----		----	
8001	INHOUSE	ND		ND		ND		----		----	

normality	n.a.	n.a.	n.a.	n.a.	n.a.
n	5	4	3	3	2
outliers	0	0	0	0	0
mean (n)	n.a.	n.a.	n.a.	n.a.	n.a.
st.dev. (n)	n.a.	n.a.	n.a.	n.a.	n.a.
R(calc.)	n.a.	n.a.	n.a.	n.a.	n.a.
R(lit)	Unknown	Unknown	Unknown	Unknown	Unknown

Determination of other Phthalates not listed before on sample 0713; results in %M/M (continued)

lab	method	DCHP	mark	DEP	mark	DHP	mark	OP	mark
310		----		----		----		----	
339		----		----		----		----	
662	INHOUSE	ND		ND		----		ND	
825		----		----		----		----	
840		----		----		----		----	
852		----		----		----		----	
862		----		----		----		----	
868	INHOUSE	<0.005		<0.005		<0.005		<0.005	
869		----		----		----		----	
885	INHOUSE	<0.003		<0.003		<0.003		ND	
886		----		----		----		----	
902		----		----		----		----	
917		----		ND		----		----	
1051	D3421	<0.005		<0.005		<0.005		----	
1087		----		----		----		----	
2102	INHOUSE	ND		ND		ND		----	
2117		----		----		----		----	
2120		----		----		----		----	
2127		----		<0.0001		----		----	
2129		----		----		----		----	
2130		----		----		----		----	
2131		----		----		----		----	
2132	INHOUSE	<0.005		<0.005		<0.005		----	
2137	INHOUSE	<0.001		<0.001		<0.001		<0.001	
2139		----		----		----		----	
2152		----		----		----		----	
2156	EPA3550C	0.0010		0.0010		0.0010		0.0010	
2169	INHOUSE	<0.01		<0.01		<0.01		<0.01	
2172	INHOUSE	ND		ND		ND		----	
2173		----		----		----		----	
2182	EN14372	<0.01		<0.01		<0.01		----	
2184		----		----		----		----	
2190		----		----		----		----	
2192		----		----		----		----	
3106	EN14372	<0.01		<0.01		<0.01		----	
3107	INHOUSE	ND		ND		ND		ND	
3109		----		<0.1		----		----	
3111		----		----		----		----	
3116	D3421	0.03		<0.01		<0.01		----	
3134		----		----		----		----	
3146		----		<0.01		----		----	
3147		----		----		----		----	
3150		----		----		----		----	
3151	INHOUSE	<0.005		<0.005		<0.005		0.006	
3153		----		----		----		----	
3154	INHOUSE	ND		ND		ND		ND	
3159	INHOUSE	<0.005		<0.005		<0.005		NIL	
3161		----		----		----		----	
3165	INHOUSE	<0.05		<0.05		----		----	
3166		----		----		----		----	
3169	INHOUSE	<0.001		<0.001		<0.001		----	
3172		----		----		----		----	
3173		----		----		----		----	
3176		----		----		----		----	
3182	EN14372	0.015		0.00097		0.0057		0.0010	
3184	D3421	<0.01		<0.01		<0.01		----	
3185	EN14372	<0.01		<0.01		<0.01		----	
3191	INHOUSE	<0.01		<0.01		<0.01		----	
3192	INHOUSE	ND		ND		----		----	
3199	INHOUSE	<0.01		<0.01		<0.01		<0.01	
3200		----		----		----		----	
3209	INHOUSE	<0.005		<0.005		<0.005		<0.005	
3217		----		----		----		----	
3218		----		----		----		----	
3219		----		----		----		----	
3220		----		----		----		----	
3223		----		----		----		----	
3225	INHOUSE	<0.005		<0.005		<0.005		<0.005	
5012		----		----		----		0.004	
8001		----		----		----		----	

normality	n.a.	n.a.	n.a.	n.a.
n	3	2	2	4
outliers	0	0	0	0
mean (n)	n.a.	n.a.	n.a.	n.a.
st.dev. (n)	n.a.	n.a.	n.a.	n.a.
R(calc.)	n.a.	n.a.	n.a.	n.a.
R(lit)	Unknown	Unknown	Unknown	Unknown

APPENDIX 2

Method information

lab	Analysis method	Technique to release/extract	Technique to detect and quantify	Calibration Used	Corr. for recovery
310	INHOUSE	Solvent extraction THF, precipitation in n-Hexane	GC/MS	Int. std.	No
339	INHOUSE	Soxhlet extraction, methanol: chloroform (1:2)	GC/MS	Int. std.	No
662	INHOUSE	Soxhlet extraction	GC/MSD	Ext. std.	No
825	EPA 8061	Soxhlet extraction	GC/MSD	Ext. std.	No
840	ASTM D3421	Soxhlet extraction	GC/MSD	Ext. std.	No
852	ASTM D3421	Soxhlet extraction, dichloromethane: methanol (1:1)	GC/FID	Ext. std.	No
862	ASTM D3421	Soxhlet extraction, dichloromethane: methanol (1:1), 6 hrs	GC/MSD	Ext. std.	No
868	INHOUSE	Soxhlet extraction	GC/MS	Ext. std.	No
869	ASTM D3421	Soxhlet extraction	GC/MS	Ext. std.	No
885	--	--	--	--	--
886	Chromatographia Vol 47. No 784	Soxhlet extraction with 100 ml dichloromethane	GC/MS	Ext. std.	No
902	ASTM D3421	Soxhlet extraction, dichloromethane: methanol	GC/MS	Ext. std.	No
917	INHOUSE	Soxhlet extraction with dichloromethane	GC/MS	Ext. std.	No
1051	ASTM D3421	Soxhlet extraction	GC/MS	Ext. std.	No
1087	INHOUSE	dissolve in tetrahydrofurane, after mecipitated with hexane and filtered, the filtrate immediately injected	GC/MS	Std. add.	No
2102	INHOUSE	Solvent extraction THF, precipitation in n-Hexane	GC/MS	Ext. std.	No
2117	INHOUSE	Soxhlet extraction with tetrabutylmethylether	GC/MSD	Int. std.	No
2120	EN14372	Soxhlet extraction, solvent diethyl ether, 6hrs	GC/MS	Int. std.	Yes
2127	INHOUSE	Ultrasonic with n-Hexane	GC/MS	Int. std.	Yes
2129	INHOUSE	Ultrasonic extraction in THF	GC/MS	Ext. std.	Yes
2130	INHOUSE	50 mg solved in THF (10ml), fill up with ACN to 100ml	HPLC/DAD/MS	Ext. std.	No
2131	INHOUSE	ASE	GC/MS	Int. std.	No
2132	INHOUSE	INHOUSE	GC/MSD/FID	Int. std.	No
2137	INHOUSE	Soxhlet extraction	GC/MSD	Ext. std.	No
2152	INHOUSE	Soxhlet extraction, methanol: chloroform (1:2) 16hours	GC/MS	Ext. std.	No
2156	USEPA 3550C	Ultrasonic extraction	GC/MS	Ext. std.	No
2169	INHOUSE	Soxhlet extraction	GC/MS - LC/MS	Ext. std.	No
2172	INHOUSE	Liquid extraction with ultrasonic bath	LC/ MS	Ext. std.	No
2173	INHOUSE	Ultrasonic extraction with chloroform	GC/MS	Ext. std.	No
2182	EN14372	Diethyl ether Extraction	GC/MS	Ext. std.	No
2184	INHOUSE	Tert-Butylmethylether	GC/MS	Int. std.	No
2190	INHOUSE	Extraction under pressure and temperature with a system of accelerated extraction by solvents	GC/MS	Int. std.	No
2192	INHOUSE	Soxhlet extraction 16hrs	GC/MS	Ext. std.	No
3106	EN14372	Soxhlet extraction, diethyl ether,6hrs	GC/MSD	Int. std.	No

3107	EN14372	Soxhlet extraction, diethyl ether,6hrs	GC/MS	Ext. std.	No
3109	INHOUSE	Diluted in THF, precipitated with methanol	GC/FID	Int. std.	No
3111	INHOUSE	Extraction in dichloromethane, precipitation with methanol	GC/MS	Int. std.	No
3116	ASTM D3421	Soxhlet extraction	GC/MS	Ext. std.	No
3134	INHOUSE	Dissolution in THF, precipitation in methanol	HPLC/UV diode array	Ext. std.	No
3146	INHOUSE	Ultrasonic/Soxhlet	GC/MS	Int. std.	No
3147	INHOUSE	Ether extraction	GC/FID - GC/MS	Int. std.	No
3150	INHOUSE	Ultrasonic extraction with Acetone, 30 min	GC/MS	Ext. std.	No
3151	INHOUSE	Extraction with THF (10 ml), ultrasonic 30 min 40°C, precipitated with ACN	GC/MS - LC/MS; LC/DAD	Ext. std.	No
3153	ASTM D3421	Soxhlet extraction, methanol: chloroform (1:2)	GC/FID - GC/MS	Ext. std.	No
3154	INHOUSE	Soxhlet extraction with dichloromethane, 2hrs	GC/MSD	Ext. std.	No
3159	INHOUSE	Solvent extraction	GC/MS - LC/MS	Ext. std.	Yes
3165	INHOUSE	Extraction with THF, precipitation in ethanol, diluted with n-hexane	GC/MS	Ext. std.	No
3166	INHOUSE	Extraction with dichloromethane	GC/MS	Int. std.	No
3169	INHOUSE	ASTM D3421:1975	GC/MS	Int. std.	No
3172	INHOUSE	Extraction with THF, precipitated with ACN	HPLC/DAD-MS	Ext. std.	No
3173	INHOUSE	Extraction with Methyl-ethyl-ketone	GC/FID	Int. std.	Yes
3176	EN14372	Extraction	GC/MSD	Ext. std.	No
3182	EN14372	Soxhlet extraction	GC/MSD	Ext. std.	Yes
3184	ASTM D3421	Soxhlet extraction	GC/MS	Ext. std.	No
3185	EN14372	Soxhlet extraction	GC/MS	Ext. std.	No
3191	INHOUSE	Solvent extraction with dichloromethane, 2 hours	GC/MS	Ext. std.	No
3192	INHOUSE	Extraction with ether in ultrasonic bath	GC/MS	Int. std.	No
3199	INHOUSE	THF, Sonicate 30 min, precipitate with ACN	LC/MS - GC/MS	Ext. std.	No
3200	ASTM D3421	Extraction with dichloromethane in ultrasonic bath	GC/MS	Int. std.	No
3209	INHOUSE	Chloroform extraction	GC/MS	Ext. std.	Yes
3217	INHOUSE	Swell in dimethylformamid, extraction with ethyl acetate	GC/MS	Int. std.	No
3218	INHOUSE	Soxhlet extraction with ethyl ether	GC/FID - GC/MS	Ext. std.	No
3220	INHOUSE	Dissolution in THF and water, extraction with hexane	GC/MS	Ext. std.	No
3223	INHOUSE	Extraction with THF, precipitation with ACN	GC/MSD	Ext. std.	No
3225	INHOUSE	Ultrasonic extraction	GC/MSD	Ext. std.	No
5012	INHOUSE	Soxhlet extraction	GC/MS	Ext. std.	No
8001	ASTM D3421	Soxhlet extraction with dichloromethane: methanol (1:1)	GC/MSD	Ext. std.	No

APPENDIX 3**List of number participating laboratories**

Name	Country
1 laboratory in	AUSTRIA
1 laboratory in	BELGIUM
1 laboratory in	DENMARK
2 laboratories in	FRANCE
13 laboratories in	GERMANY
1 laboratory in	GREECE
11 laboratories in	HONG KONG
2 laboratories in	INDIA
2 laboratories in	ITALY
1 laboratory in	JAPAN
3 laboratories in	KOREA
2 laboratories in	MALAYSIA
13 laboratories in	P.R. of CHINA
1 laboratory in	PORTUGAL
3 laboratories in	SWITZERLAND
2 laboratories in	TAIWAN R.O.C.
2 laboratories in	THAILAND
3 laboratories in	THE NETHERLANDS
2 laboratories in	TURKEY
2 laboratories in	U.S.A.
1 laboratory in	UNITED KINGDOM
1 laboratory in	VIETNAM

APPENDIX 4

Abbreviations:

C	= final result after checking of first reported suspect 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
n.a.	= not applicable
ND	= not detected
DT	= detected not quantified

Literature:

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- 2 ASTM D3421:75: "extraction and analysis of plasticizer mixtures from vinyl chloride plastics".
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- 10 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
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