Results of Proficiency Test
AP & APEO in textile
February 2019

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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CONTENTS

1 INTRODUCTION

Since 2016, the Institute for Interlaboratory Studies organizes a proficiency test (PT) for the determination of AP and APEO content in textile. During the annual proficiency testing program 2018/2019, it was decided to continue with the PT for the analyzes of AP and APEO content in textile.
In this interlaboratory study, 107 laboratories in 25 different countries registered for participation. See appendix 4 for the number of participants 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 analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send two different textile samples of 3 grams each, labelled #19510 and #19511. The samples were positive on OPEO or NPEO. The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

Two different textile samples were selected. Sample #19510 (fabric with black and white pattern) is a recall batch from the market, found to be positive on NPEO and sample #19511 (yellow cotton) has been treated to be positive on OPEO.

The bulk textile for sample #19510 was cut into pieces. From this batch, after mixing well, 119 subsamples of approx. 3 grams each were prepared and labelled #19510. The homogeneity of the subsamples was checked by determination of NPEO using an in-house test method on seven stratified randomly selected samples.

	NPEO in mg/kg
Sample #19510-1	897
Sample #19510-2	966
Sample #19510-3	881
Sample #19510-4	925
Sample #19510-5	986
Sample #19510-6	959
Sample #19510-7	943

Table 1: homogeneity test results of subsamples #19510

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

	NPEO in mg/kg
r (observed)	106
reference method	Horwitz (n=5)
0.3 * R (reference method)	101

Table 2: evaluation of the repeatability of subsamples #19510

The calculated repeatability of NPEO was in agreement with 0.3 times the corresponding estimated reproducibility of the reference method. Therefore, homogeneity of the subsamples #19510 was assumed.

The bulk textile for sample #19511 was cut into pieces. From this batch, after mixing well, 120 subsamples of approx. 3 grams each were prepared and labelled #19511. The homogeneity of the subsamples was checked by determination of OPEO using an in-house test method on eight stratified randomly selected samples.

	OPEO in mg/kg
Sample #19511-1	68.6
Sample #19511-2	69.5
Sample #19511-3	67.5
Sample #19511-4	71.8
Sample #19511-5	73.2
Sample #19511-6	68.3
Sample #19511-7	67.8
Sample #19511-8	64.5

Table 3: homogeneity test results of subsamples #19511

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

	NPEO in mg/kg
r (observed)	7.5
reference method	Horwitz (n=5)
0.3 * R (reference method)	11.0

Table 4: evaluation of the repeatability of subsamples #19511

The calculated repeatability of OPEO was in agreement with 0.3 times the corresponding estimated reproducibility of the reference method. Therefore, homogeneity of the subsamples #19511 was assumed.

To each participating laboratory, one sample labelled #19510 and one sample labelled #19511 were sent on February 13, 2019.

2.5 ANALYSES

The participants were requested to determine the concentrations of OP, NP, OPEO, NPEO and total OP, NP, OPEO + NPEO on both samples #19510 and #19511, applying the analysis procedure that is routinely used in the laboratory. Also, some method details were 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 more, but report as much significant figures as possible. It was also requested not to report ‘less than’ test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test 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 test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are represented by the code numbers.

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

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report ‘iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation’ of June 2018 (iis-protocol, version 3.5).
For the statistical evaluation, the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as ‘<...’ or ‘>...’ were not used in the statistical evaluation.
First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either ‘unknown’, ‘OK’, ‘suspect’ or ‘not OK’. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the results of the statistical evaluation should be used with due care.
In accordance to ISO5725 the original test results per determination were submitted subsequently to Dixon’s, Grubbs’ and or Rosner’s outlier tests. Outliers are marked by D(0.01) for the Dixon’s test, by G(0.01) or DG(0.01) for the Grubbs’ test and by R(0.01) for the Rosner’s test. Stragglers are marked by D(0.05) for the Dixon’s test, by G(0.05) or DG(0.05) for the Grubbs’ test and by R(0.05) for the Rosner’s test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

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

3.2 GRAPHICS

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

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

Furthermore, Kernel Density Graphs were made. 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 variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency tests could be 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 in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated in accordance with:

$z_{(target)}$ = (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

During the execution of this proficiency test no problems occurred with the dispatch of the samples. Five participants reported the test results after the final reporting date and two participants did not report any results at all. In total 105 participants reported 366 numerical test results. Observed in all reported results were 21 statistical outlying results, which is 5.7%. 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 table together with the original data. The abbreviations, used in these tables, are listed in appendix 5.

ISO18254-1, used by the majority of the participants, is the official test method for the determination of APEO in textiles. Regretfully ISO18254-1 does not mention reproducibilities for OP, NP, OPEO or NPEO, but only for APEO at a level of 954 mg/kg (R=262 mg/kg). Because the samples did not contain the same concentrations of APEO as the concentration used in ISO18254-1, the target requirements in this study were estimated using the Horwitz equation for 5 components (n=5). The above mentioned target of ISO18254-1 was mentioned in appendix 1 for comparison.

sample #19510

NPEO: The determination of this component was not problematic at the level of 1252 mg/kg. Five statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated target reproducibility using the Horwitz equation for 5 components.

sum OP + NP + OPEO + NPEO:

The determination of this sum-component was not problematic at the level of 1264 mg/kg. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated target reproducibility using the Horwitz equation for 5 components.

sample #19511

OPEO: The determination of this component was not problematic at the level of 71 mg/kg. Six statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated target reproducibility using the Horwitz equation for 5 components.

sum OP + NP + OPEO + NPEO:

The determination of this sum-component was not problematic at the level of 74 mg/kg. Six statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated target reproducibility using the Horwitz equation for 5 components.

Most participants agreed about the absence of Octylphenol and Nonylphenol (see appendix 2 for reported test results).

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made as declared by the estimated target reproducibility using the Horwitz equation and the reproducibility as found for the group of participating laboratories. The number of significant results, the average result, the calculated reproducibility (2.8 * standard deviation) and the estimated target reproducibility are presented in the next tables.

Components	unit	n	average	2.8 * sd	R (target)
NPEO	mg/kg	100	1252	452	429
sum OP + NP + OPEO + NPEO	mg/kg	74	1264	398	432

Table 5: reproducibilities of components on sample #19510

Components	unit	n	average	2.8 * sd	R (target)
OPEO	mg/kg	99	71.3	20.8	37.6
sum OP + NP + OPEO + NPEO	mg/kg	72	73.6	22.6	38.6

Table 6: reproducibilities of components on sample #19511

Without further statistical calculations, it can be concluded that the group of participating laboratories has no problem with the analysis of APEO in textile at the investigated levels.

4.3 COMPARISON OF THE PROFICIENCY TEST OF FEBRUARY 2019 WITH PREVIOUS PTS

	February 2019	February 2018	March 2017	March 2016
Number of reporting labs	105	92	95	105
Number of results reported	366	329	378	412
Number of statistical outliers	21	8	9	13
Percentage outliers	5.7%	2.4%	2.4%	3.2%

Table 7: comparison with previous proficiency test

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

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

	February 2019	February 2018	March 2017	March 2016	Horwitz 500-1500 mg/kg
Octylphenol (OP)	n.a.	n.a.	n.a.	n.a.	-
Nonylphenol (NP)	n.a.	n.a.	n.a.	n.a.	-
Octylphenol Ethoxylates (OPEO)	10%	16%	15%	16%	18-12%
Nonylphenol Ethoxylates (NPEO)	13%	28%	18%	27%	18-12%
sum OP + NP + OPEO + NPEO	11%	15%-26%	15%-17%	16%-25%	18-12%

Table 8: comparison of uncertainties (relative in %)

4.4 EVALUATION 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:
Eighty-seven of the registered participants mentioned that they are accredited for determination of AP+APEO in textile. Seventy-five participants mentioned that they have used a test portion of 0.5 or 1.0 grams. Two mentioned to have used less material (0.5 gram) for intake and four have used more testing material for intake (>2 gram).
The majority of the group mentioned to have used ultrasonic as technique to release/extract the analytes, three mentioned to use Soxhlet. All but one mentioned to have used methanol for release/extraction. The majority of the group mentioned to use the following conditions: 60 minutes at 70°C.

When evaluating the above differences in the execution of the test, no clear correlation was found between these test conditions.

5 DISCUSSION

In this proficiency test for the determination of AP and APEO content in textile the majority of the participants had no problems with the analysis of NPEO and OPEO in textile at the levels as present in this PT (respectively 1252 and 71 mg/kg).

When the results of this interlaboratory study were compared to the OEKO-TEX requirements and the EU (REACH) regulations on Textiles (table 7), it is noticed that all of the reporting laboratories would reject sample #19510 for containing too much NPEO and OP + NP + OPEO + NPEO in total and that four of the reporting laboratories would reject sample #19511 for containing too much OPEO and/or too much OP + NP + OPEO + NPEO in total.

It is observed that not all participants reported a value for the sum-parameter OP + NP + OPEO + NPEO. This parameter is listed in the OEKO-TEX criteria.

	OEKO-TEX	Blue Sign BSSL v6.0	EU 2016/26
NP	---	10 mg/kg	100 mg/kg
sum OP + NP	10 mg/kg	---	---
Every single APEO	---	100 mg/kg *)	---
NPEO	---	---	100 mg/kg
sum OP + NP + OPEO + NPEO	100 mg/kg	---	---

Table 9: Ecolabelling Standards and EU regulatory limits for Textiles in EU

*) When above 10 mg/kg; source of contamination has to be identified and phased out.

6 CONCLUSION

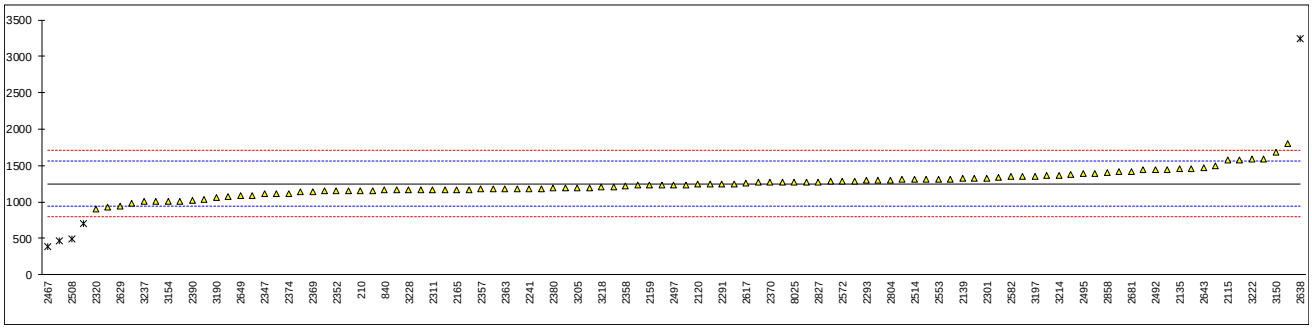
Although, it can be concluded that the majority of the participants has no problem with the determination of OPEO and NPEO in the samples of this PT, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary.
Therefore, participation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

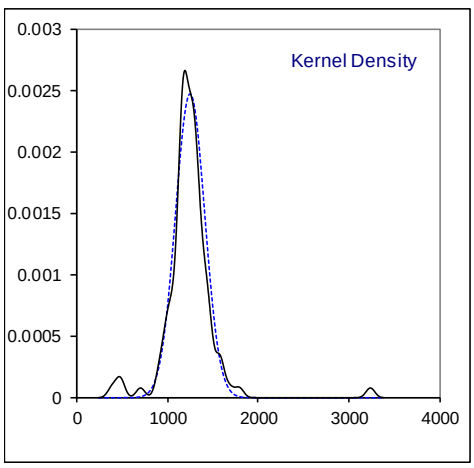
APPENDIX 1

Determination of Nonylphenol Ethoxylates (NPEO) on sample #19510; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	In house	1154.87		-0.63	
230	ISO18254-1	1312		0.39	
339	In house	1235.7		-0.10	
551	In house	1170.66		-0.53	
623	ISO18254-1	1358.79		0.70	
840	In house	1166.0		-0.56	
2108	ISO18254-1	1795.75		3.55	
2115	ISO18254-1	1573.38		2.10	
2120	ISO18254-1	1239		-0.08	
2129	ISO/DIS 18254-1	1450	C	1.30	first reported: 1711
2135		1456		1.33	
2138	ISO18254-1	1031		-1.44	
2139	ISO18254-1	1322.7		0.46	
2159	In house	1230.8		-0.14	
2165	ISO18254-1	1172.6		-0.52	
2201	ISO18254-1	1247.8		-0.03	
2232	In house	1290.52		0.25	
2241	ISO18254-1	1181.5		-0.46	
2247	ISO18254-1	1375.08		0.81	
2250	ISO/DIS 18254-1	1393.5		0.93	
2255	ISO/DIS 18254-1	1307.12		0.36	
2265	ISO18254-1	1084.9		-1.09	
2285		1267.688		0.10	
2289	ISO/DIS 18254-1	1203		-0.32	
2290	ISO18254-1	1339.7		0.57	
2291	GB/T23322	1249.2		-0.02	
2293	ISO/DIS 18254-1	1300.00		0.32	
2295	ISO18254-1	975		-1.81	
2301	ISO18254-1	1328.80		0.50	
2310	ISO18254-1	1230		-0.14	
2311	ISO18254-1	1169.88		-0.53	
2320	ISO18254-1	901.78		-2.29	
2330	ISO18254-1	1003.75		-1.62	
2347	ISO18254-1	1109		-0.93	
2350	ISO18254-1	1490.25		1.56	
2352	ISO18254-1	1148		-0.68	
2357	ISO/DIS 18254-1	1174.8		-0.50	
2358	ISO/DIS 18254-1	1219.11		-0.21	
2363	In house	1178		-0.48	
2365	ISO18254-1	1238.06		-0.09	
2369	ISO18254-1	1142		-0.72	
2370	ISO18254-1	1270		0.12	
2372	ISO18218-1	1177		-0.49	
2374	In house	1119.18		-0.87	
2375	ISO18254-1	1190		-0.40	
2378	ISO18254-1	1071.5		-1.18	
2379	ISO18254-1	1172.63		-0.52	
2380	ISO/DIS 18254-1	1187.0		-0.42	
2382	ISO18254-1	1148.0		-0.68	
2386	ISO18254-1	1439		1.22	
2390	ISO18254-1	1027.31		-1.47	
2410	ISO/DIS 18254-1	1460		1.36	
2415	ISO/DIS 18254-1	1007.4		-1.60	
2426	ISO18254-1	1414.68		1.06	
2432		-----		-----	
2467	In house	386.88	C,R(0.01)	-5.65	first reported: 341.04
2492	In house	1441.69		1.24	
2495	ISO18254-1	1387.7		0.89	
2497	ISO18218-1	1237.58		-0.09	
2508		486.34	C,R(0.01)	-5.00	first reported: 567.47
2514	ISO/DIS 18254-1	1309.07		0.37	
2549	ISO18254-1	1277.4		0.17	
2553	In house	1310.20		0.38	
2561	ISO18254-1	1181.250		-0.46	
2567	ISO18254-1	1310		0.38	
2572	ISO18254-1	1283.2		0.21	
2582	ISO18254-1	1344.80		0.61	
2590	ISO18254-1	1133.997		-0.77	
2605	GB/T23972	1159.51		-0.60	
2617	GB/T23322	1260.26		0.06	
2629	ISO18254-1	936.566		-2.06	
2638	In house	3244.32	R(0.01)	13.02	
2643	ISO18254-1	1470.34		1.43	

lab	method	value	mark	z(targ)	remarks
2644	ISO18254-1	1300		0.32	
2649	ISO/DIS 18254-1	1080.96		-1.12	
2681	ISO18254-1	1415.47		1.07	
2737	ISO/DIS 18254-1	1167.05		-0.55	
2773	ISO18254-1	1282.2		0.20	
2789		-----		-----	
2791	ISO18254-1	1354.41		0.67	
2804	ISO18254-1	1300		0.32	
2812	ISO18218-1	1277		0.17	
2827	ISO18254-1	1278.41		0.17	
2858	ISO/DIS 18254-1	1409.81		1.03	
2870	ISO18254-1	1578		2.13	
2877	ISO18254-1	470.9947	R(0.01)	-5.10	
3100	ISO18254-1	1113.28		-0.90	
3116	ISO18254-1	1169		-0.54	
3146	ISO18254-1	1588		2.20	
3150	ISO18254-1	1688		2.85	
3154	In house	1007.00		-1.60	
3160	ISO18218-2	1188.81		-0.41	
3172	ISO18254-1	925		-2.13	
3182	ISO/DIS 18254-1	1249.27		-0.02	
3190	ISO/DIS 18254-1	1058.3		-1.26	
3197	ISO18254-1	1355.2		0.68	
3205	In house	1189		-0.41	
3210	ISO18254-1	1148.71		-0.67	
3214	ISO/DIS 18254-1	1359.04		0.70	
3218	ISO18254-1	1200.00		-0.34	
3220	ISO18254-1	703.03	C,R(0.05)	-3.58	first reported: 712.96
3222	ISO18254-1	1583.81		2.17	
3228	ISO/DIS 18254-1	1168.2		-0.55	
3237	ISO18254-1	1002.5		-1.63	
3248	In house	1184		-0.44	
3250	ISO18254-1	1326.61		0.49	
8025	In house	1277		0.17	
	normality	OK			
	n	100			
	outliers	5			
	mean (n)	1251.690			
	st.dev. (n)	161.3185	RSD = 13%		
	R(calc.)	451.692			
	st.dev.(Horwitz (n=5))	153.0672			
	R(Horwitz (n=5))	428.588			compare R(ISO18254-1:16) = 343.755

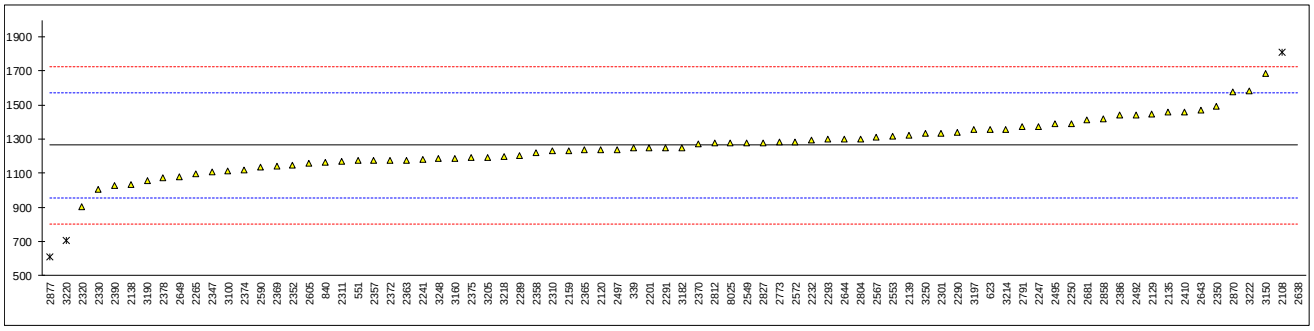


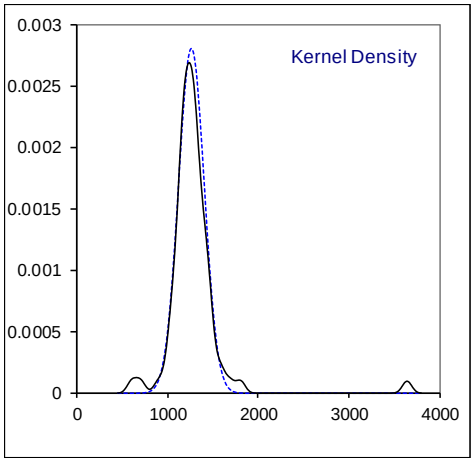


Determination of Total of OP, NP, OPEO and NPEO on sample #19510; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210		-----		-----	
230		-----		-----	
339	In house	1246.6		-0.11	
551	In house	1173.65		-0.59	
623	ISO18254-1	1358.79		0.61	
840	In house	1166.0		-0.64	
2108	ISO18254-1	1807.48	R(0.05)	3.52	
2115		-----		-----	
2120	ISO18254-1	1239		-0.16	
2129	ISO/DIS 18254-1	1450	C	1.20	first reported: 1711
2135		1457.5		1.25	
2138	ISO18254-1	1031		-1.51	
2139	ISO18254-1	1322.7		0.38	
2159	In house	1230.8		-0.22	
2165		-----		-----	
2201	ISO18254-1	1247.8		-0.11	
2232	In house	1295.69		0.20	
2241	ISO18254-1	1181.5		-0.54	
2247	ISO18254-1	1375.08		0.72	
2250	ISO/DIS 18254-1	1393.5		0.84	
2255		-----		-----	
2265	ISO18254-1	1098.55		-1.07	
2285		-----		-----	
2289	ISO/DIS 18254-1	1203		-0.40	
2290	ISO18254-1	1339.7		0.49	
2291	GB/T23322	1249.2		-0.10	
2293	ISO/DIS 18254-1	1300.00		0.23	
2295		-----		-----	
2301	ISO18254-1	1333.20		0.45	
2310	ISO18254-1	1230		-0.22	
2311	ISO18254-1	1169.88		-0.61	
2320	ISO18254-1	901.78		-2.35	
2330	ISO18254-1	1005.72		-1.68	
2347	ISO18254-1	1109		-1.01	
2350	ISO18254-1	1490.25		1.46	
2352	ISO18254-1	1148		-0.75	
2357	ISO/DIS 18254-1	1174.8		-0.58	
2358	ISO/DIS 18254-1	1219.11		-0.29	
2363	In house	1178		-0.56	
2365	ISO18254-1	1238.06		-0.17	
2369	ISO18254-1	1142		-0.79	
2370	ISO18254-1	1270		0.04	
2372	ISO18218-1	1177		-0.57	
2374	In house	1119.18		-0.94	
2375	ISO18254-1	1190		-0.48	
2378	ISO18254-1	1071.5		-1.25	
2379		-----		-----	
2380		-----		-----	
2382		-----		-----	
2386	ISO18254-1	1439		1.13	
2390	ISO18254-1	1027.31		-1.54	
2410	ISO/DIS 18254-1	1460		1.27	
2415		-----		-----	
2426		-----		-----	
2432		-----		-----	
2467		-----		-----	
2492	In house	1441.69		1.15	
2495	ISO18254-1	1388.58		0.80	
2497	ISO18218-1	1239.781		-0.16	
2508		-----		-----	
2514		-----		-----	
2549	ISO18254-1	1277.4		0.08	
2553	In house	1315.4		0.33	
2561		-----		-----	
2567	ISO18254-1	1310		0.30	
2572	ISO18254-1	1283.2		0.12	
2582		-----		-----	
2590	ISO18254-1	1137.092		-0.82	
2605	GB/T23972	1159.51		-0.68	
2617		-----		-----	
2629		-----		-----	
2638	In house	3648.5	R(0.01)	15.44	
2643	ISO18254-1	1470.34		1.33	

lab	method	value	mark	z(targ)	remarks
2644	ISO18254-1	1300		0.23	
2649	ISO/DIS 18254-1	1080.96		-1.19	
2681	ISO18254-1	1415.47		0.98	
2737		-----		-----	
2773	ISO18254-1	1282.2		0.12	
2789		-----		-----	
2791	ISO18254-1	1373.24		0.71	
2804	ISO18254-1	1300		0.23	
2812	ISO18218-1	1277		0.08	
2827	ISO18254-1	1278.41		0.09	
2858	ISO/DIS 18254-1	1416.91		0.99	
2870	ISO18254-1	1578		2.03	
2877	ISO18254-1	605.9772	R(0.05)	-4.26	
3100	ISO18254-1	1113.28		-0.98	
3116		-----		-----	
3146		-----		-----	
3150	ISO18254-1	1688		2.74	
3154		-----		-----	
3160	ISO18218-2	1188.81		-0.49	
3172		-----		-----	
3182	ISO/DIS 18254-1	1249.27		-0.10	
3190	ISO/DIS 18254-1	1058.3		-1.33	
3197	ISO18254-1	1355.2		0.59	
3205	In house	1191.4		-0.47	
3210		-----		-----	
3214	ISO/DIS 18254-1	1359.04		0.61	
3218	ISO18254-1	1200.00		-0.42	
3220	ISO18254-1	703.03	C,R(0.05)	-3.64	first reported: 712.96
3222	ISO18254-1	1583.81		2.07	
3228		-----		-----	
3237		-----		-----	
3248	In house	1184		-0.52	
3250	ISO18254-1	1331.95		0.44	
8025	In house	1277		0.08	
	normality	OK			
	n	74			
	outliers	4			
	mean (n)	1264.312			
	st.dev. (n)	141.9847	RSD = 11%		
	R(calc.)	397.557			
	st.dev.(Horwitz (n=5))	154.3774			
	R(Horwitz (n=5))	432.257			compare R(ISO18254-1:16) = 347.222

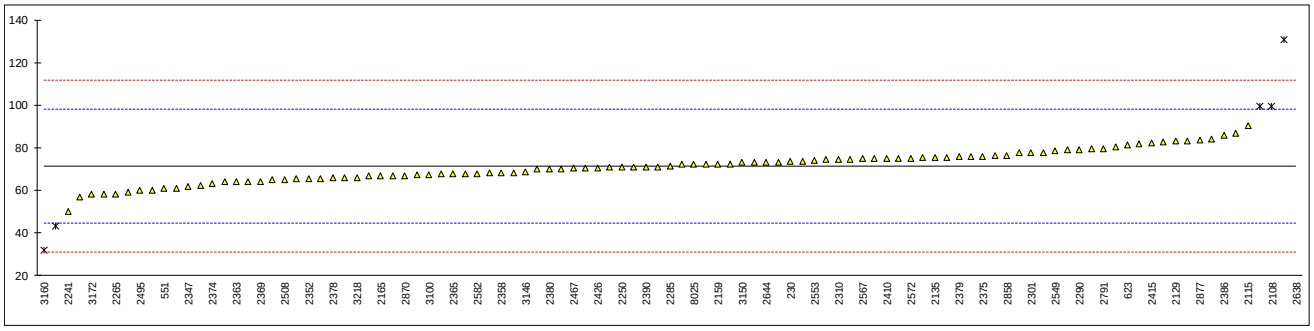


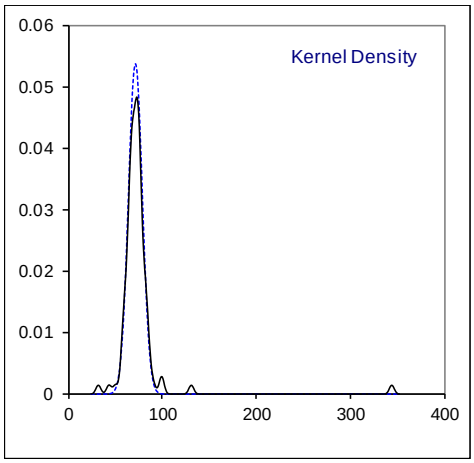


Determination of Octylphenol Ethoxylates (OPEO) on sample #19511; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210	In house	72.45		0.09	
230	ISO18254-1	73.7		0.18	
339	In house	67.9		-0.25	
551	In house	60.74		-0.78	
623	ISO18254-1	81.25		0.74	
840	In house	75.9		0.34	
2108	ISO18254-1	99.28	R(0.05)	2.09	
2115	ISO18254-1	90.48		1.43	
2120	ISO18218-1	66		-0.39	
2129	ISO18254-1	83.1		0.88	
2135		75.4		0.31	
2138	ISO18254-1	68		-0.24	
2139	ISO18254-1	68.3		-0.22	
2159	ISO18254-1	72.4		0.08	
2165	ISO18254-1	66.9		-0.33	
2201		67.8		-0.26	
2232	In house	70.643356		-0.05	
2241	ISO18254-1	50.1		-1.58	
2247	ISO18254-1	62.35		-0.66	
2250	ISO/DIS 18254-1	70.86		-0.03	
2255	ISO/DIS 18254-1	76.1		0.36	
2265	ISO18254-1	58.2		-0.97	
2285		71.226		0.00	
2289	ISO/DIS 18254-1	64		-0.54	
2290	ISO18254-1	79.23		0.59	
2291		75.0		0.28	
2293	ISO/DIS 18254-1	83.12	C	0.88	first reported as NPEO
2295	ISO18254-1	57		-1.06	
2301	ISO18254-1	77.60		0.47	
2310	ISO18254-1	74.47		0.24	
2311	ISO18254-1	74.32		0.23	
2320	ISO18254-1	58.865		-0.92	
2330	ISO18254-1	66.92		-0.32	
2347	In house	62		-0.69	
2350	ISO18254-1	83.85		0.94	
2352	ISO18254-1	65.5		-0.43	
2357	ISO/DIS 18254-1	64.8		-0.48	
2358	ISO/DIS 18254-1	68.27		-0.22	
2363	In house	64		-0.54	
2365	ISO18254-1	67.85		-0.26	
2369	ISO18254-1	64		-0.54	
2370	ISO18254-1	70.1		-0.09	
2372	ISO18218-1	72.95		0.13	
2374	In house	63.31		-0.59	
2375	ISO18254-1	76		0.35	
2378	ISO18254-1	66		-0.39	
2379	ISO18254-1	75.67		0.33	
2380	ISO/DIS 18254-1	70.0		-0.09	
2382	ISO18254-1	64.0		-0.54	
2386	ISO18254-1	86		1.10	
2390	ISO18254-1	70.96		-0.02	
2410	ISO18254-1	75		0.28	
2415	ISO/DIS 18254-1	82.3		0.82	
2426	ISO18254-1	70.63		-0.05	
2432		-----		-----	
2467	In house	70.36		-0.07	
2492	In house	86.55	C	1.14	first reported: 94.58
2495	ISO18254-1	59.81		-0.85	
2497	ISO18218-1	130.931	R(0.01)	4.45	
2508		64.98		-0.47	
2514	ISO/DIS 18254-1	73.08		0.13	
2549	ISO18254-1	78.5		0.54	
2553	In house	74.2		0.22	
2561	ISO18254-1	66.691		-0.34	
2567	ISO18254-1	74.74		0.26	
2572	ISO18254-1	75.12		0.29	
2582	ISO18254-1	67.90		-0.25	
2590	ISO18254-1	80.604		0.70	
2605	GB/T23972	65.60		-0.42	
2617	GB/T23322	69.82		-0.11	
2629	ISO18254-1	67.23		-0.30	
2638	In house	344.89	R(0.01)	20.39	
2643	ISO18254-1	77.68		0.48	

lab	method	value	mark	z(targ)	remarks
2644	ISO18254-1	72.95		0.13	
2649	ISO/DIS 18254-1	60.00		-0.84	
2681	ISO18254-1	81.74		0.78	
2737	ISO/DIS 18254-1	61.04		-0.76	
2773	ISO18254-1	79.26		0.60	
2789		-----		-----	
2791	ISO18254-1	79.46		0.61	
2804	ISO/DIS 18254-1	70.9		-0.03	
2812	ISO18218-1	72		0.05	
2827	ISO18254-1	77.53		0.47	
2858	ISO/DIS 18254-1	76.16		0.36	
2870	ISO18254-1	67		-0.32	
2877	ISO18254-1	83.4615		0.91	
3100	ISO18254-1	67.39		-0.29	
3116	ISO18254-1	70.38		-0.07	
3146	ISO18254-1	68.505		-0.21	
3150	ISO18254-1	72.93	C	0.12	first reported: 96.33
3154	In house	72.28		0.08	
3160	ISO18218-2	31.72	R(0.01)	-2.95	
3172		58.0		-0.99	
3182	ISO/DIS 18254-1	74.58		0.25	
3190	ISO/DIS 18254-1	58.1		-0.98	
3197	ISO18254-1	78.9		0.57	
3205	In house	73.8		0.19	
3210		75.229	C	0.29	first reported: 175.229
3214	ISO/DIS 18254-1	75.01		0.28	
3218	ISO18254-1	66.10		-0.39	
3220	ISO/DIS 18254-1	99.25	C,R(0.05)	2.09	first reported; 105.93
3222	ISO18254-1	82.79		0.86	
3228	ISO/DIS 18254-1	65.4		-0.44	
3237	ISO18254-1	43.02	R(0.05)	-2.11	
3248	In house	71		-0.02	
3250	ISO18254-1	75.62		0.32	
8025	In house	72		0.05	
	normality	OK			
	n	99			
	outliers	6			
	mean (n)	71.272			
	st.dev. (n)	7.4121	RSD = 10%		
	R(calc.)	20.754			
	st.dev.(Horwitz (n=5))	13.4162			
	R(Horwitz (n=5))	37.565			compare R(ISO18254-1:16) = 19.574

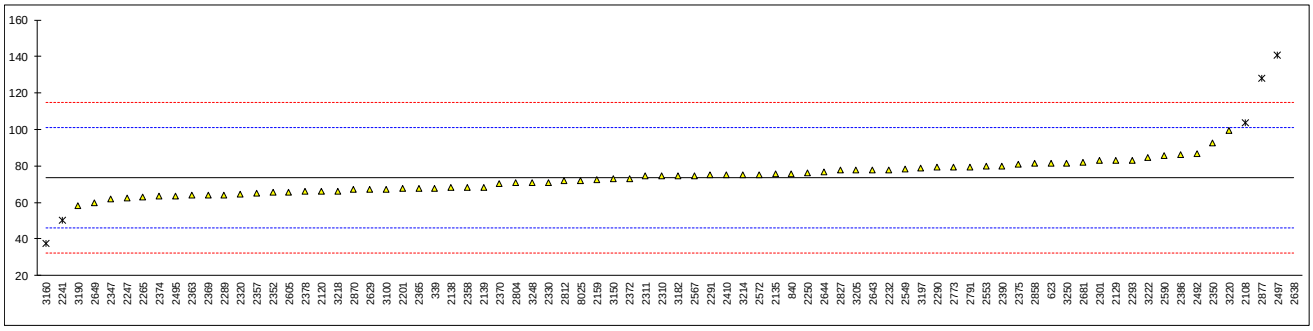


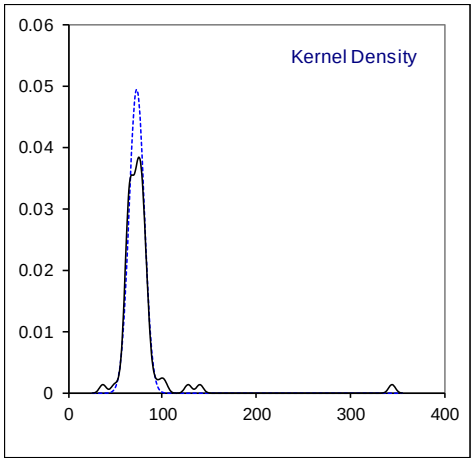


Determination of Total of OP, NP, OPEO and NPEO on sample #19511; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210		-----		-----	
230		-----		-----	
339	In house	67.9		-0.41	
551		-----		-----	
623	ISO18254-1	81.25		0.56	
840	In house	75.9		0.17	
2108	ISO18254-1	103.47	R(0.05)	2.17	
2115		-----		-----	
2120	ISO18218-1	66		-0.55	
2129	ISO18254-1	83.1		0.69	
2135		75.4		0.13	
2138	ISO18254-1	68		-0.40	
2139	ISO18254-1	68.3		-0.38	
2159	ISO18254-1	72.4		-0.08	
2165		-----		-----	
2201		67.8		-0.42	
2232	In house	77.774356	C	0.31	first reported as NPEO
2241	ISO18254-1	50.1	R(0.05)	-1.70	
2247	ISO18254-1	62.35		-0.81	
2250	ISO/DIS 18254-1	75.95		0.17	
2255		-----		-----	
2265	ISO18254-1	62.75		-0.78	
2285		-----		-----	
2289	ISO/DIS 18254-1	64		-0.69	
2290	ISO18254-1	79.23		0.41	
2291		75.0		0.10	
2293	ISO/DIS 18254-1	83.12		0.69	
2295		-----		-----	
2301	ISO18254-1	83.10		0.69	
2310	ISO18254-1	74.5		0.07	
2311	ISO18254-1	74.32		0.05	
2320	ISO18254-1	64.633		-0.65	
2330	ISO18254-1	71.04		-0.18	
2347	In house	62		-0.84	
2350	ISO18254-1	92.78		1.39	
2352	ISO18254-1	65.5		-0.59	
2357	ISO/DIS 18254-1	64.8		-0.64	
2358	ISO/DIS 18254-1	68.27		-0.38	
2363	In house	64		-0.69	
2365	ISO18254-1	67.85		-0.41	
2369	ISO18254-1	64		-0.69	
2370	ISO18254-1	70.1		-0.25	
2372	ISO18218-1	72.95		-0.04	
2374	In house	63.31		-0.74	
2375	ISO18254-1	81		0.54	
2378	ISO18254-1	66		-0.55	
2379		-----		-----	
2380		-----		-----	
2382		-----		-----	
2386	ISO18254-1	86		0.90	
2390	ISO18254-1	79.88		0.46	
2410	ISO18254-1	75		0.10	
2415		-----		-----	
2426		-----		-----	
2432		-----		-----	
2467		-----		-----	
2492	In house	86.55	C	0.94	first reported: 94.58
2495	ISO18254-1	63.49		-0.73	
2497	ISO18218-1	140.682	R(0.01)	4.87	
2508		-----		-----	
2514		-----		-----	
2549	ISO18254-1	78.5		0.36	
2553	In house	79.8		0.45	
2561		-----		-----	
2567	ISO18254-1	74.74		0.09	
2572	ISO18254-1	75.12		0.11	
2582		-----		-----	
2590	ISO18254-1	85.864		0.89	
2605	GB/T23972	65.60		-0.58	
2617		-----		-----	
2629	ISO18254-1	67.23		-0.46	
2638	In house	344.89	R(0.01)	19.69	
2643	ISO18254-1	77.68		0.30	

lab	method	value	mark	z(targ)	remarks
2644	ISO18254-1	76.79		0.23	
2649	ISO/DIS 18254-1	60.00		-0.98	
2681	ISO18254-1	81.74		0.59	
2737		-----		-----	
2773	ISO18254-1	79.26		0.41	
2789		-----		-----	
2791	ISO18254-1	79.46		0.43	
2804	ISO/DIS 18254-1	70.9		-0.19	
2812	ISO18218-1	72		-0.11	
2827	ISO18254-1	77.53		0.29	
2858	ISO/DIS 18254-1	81.24		0.56	
2870	ISO18254-1	67		-0.48	
2877	ISO18254-1	128.2411	R(0.01)	3.97	
3100	ISO18254-1	67.39		-0.45	
3116		-----		-----	
3146		-----		-----	
3150	ISO18254-1	72.93	C	-0.05	first reported: 96.33
3154		-----		-----	
3160	ISO18218-2	37.58	R(0.05)	-2.61	
3172		-----		-----	
3182	ISO/DIS 18254-1	74.58		0.07	
3190	ISO/DIS 18254-1	58.1		-1.12	
3197	ISO18254-1	78.9		0.39	
3205	In house	77.6		0.29	
3210		-----		-----	
3214	ISO/DIS 18254-1	75.01		0.10	
3218	ISO18254-1	66.10		-0.54	
3220	ISO/DIS 18254-1	99.25	C	1.86	first reported: 105.93
3222	ISO18254-1	84.76		0.81	
3228		-----		-----	
3237		-----		-----	
3248	In house	71		-0.19	
3250	ISO18254-1	81.42		0.57	
8025	In house	72		-0.11	
	normality	OK			
	n	72			
	outliers	6			
	mean (n)	73.567			
	st.dev. (n)	8.0665	RSD = 11%		
	R(calc.)	22.586			
	st.dev.(Horwitz (n=5))	13.7823			
	R(Horwitz (n=5))	38.590			compare R(ISO18254-1:16) = 20.204





APPENDIX 2

Summary of other reported components in sample #19510 and #19511

	#19510			#19511		
lab	OP	NP	OPEO	OP	NP	NPEO
210	-----	-----	-----	-----	-----	-----
230	-----	-----	-----	-----	-----	-----
339	<10	10.9	<10	<10	<10	<10
551	N.D.	2.99	N.D.	N.D.	2.20	1.14
623	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
840	not detected	not detected	not detected	not detected	not detected	not detected
2108	-----	-----	11.74	4.19	-----	-----
2115	-----	-----	9.28	-----	-----	1.40
2120	< 1,25	< 1,25	< 5	< 1,25	< 1,25	< 5
2129	<10	<20	<10	<10	<10	<10
2135	-----	1.5	-----	-----	-----	-----
2138	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2139	<10	<10	<30	<10	<10	<30
2159	<5	<5	<5	<5	<5	<5
2165	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2201	ND<10	ND<10	ND<10	<10	<10	<10
2232	-----	5.1759	-----	7.13102	-----	-----
2241	<10	<10	<10	<10	<10	<10
2247	ND	ND	ND	ND	ND	ND
2250	<3	<3	<10	5.09	<3	<10
2255	< 10	<10	<10	<10	<10	<10
2265	< 2	2.05	11.6	4.55	< 2	< 20
2285	-----	-----	-----	-----	-----	-----
2289	<10	<10	<10	<10	<10	<10
2290	<10	<10	<10	<10	<10	<10
2291	<10	<10	<10	<10	<10	<10
2293	-----	-----	-----	-----	-----	-----
2295	-----	-----	-----	-----	-----	-----
2301	ND	4.40	ND	5.50	ND	ND
2310	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2311	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2320	N.D[<3]	N.D[<3]	N.D[<10]	5.768	N.D[<3]	N.D[<10]
2330	ND	ND	1.97	4.12	ND	ND
2347	<10	<10	<30	<10	<10	<30
2350	< 1.00	< 1.00	< 1.00	4.92	< 1.00	4.01
2352	-----	-----	-----	-----	-----	-----
2357	ND	ND	ND	ND	ND	ND
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2363	ND	ND	ND	ND	ND	ND
2365	<10	<10	<30	<10	<10	<30
2369	<10	<10	<30	<10	<10	<30
2370	n.d.	n.d.	n.d.	n.d	n.d	n.d
2372	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2374	-----	-----	-----	-----	-----	-----
2375	-----	-----	-----	<10	-----	-----
2378	-----	-----	-----	-----	-----	-----
2379	Not detected	Not detected	Not detected	Not detected	Not detected	1.26
2380	<10	<10	<30	<10	<10	<30
2382	-----	-----	-----	-----	-----	-----
2386	<5	<5	<10	<5	<5	<10
2390	-----	-----	-----	8.92	-----	-----
2410	-----	-----	-----	-----	-----	-----
2415	-----	-----	-----	-----	-----	-----
2426	-----	-----	-----	-----	-----	-----
2432	-----	-----	-----	-----	-----	-----
2467	-----	-----	6.22	-----	-----	-----
2492	-----	-----	-----	-----	-----	-----
2495	<0.5	0.88	<0.5	3.68	<0.5	<0.5
2497	-----	2.021	-----	4.322	-----	5.429
2508	-----	-----	-----	-----	-----	-----
2514	-----	-----	-----	-----	-----	-----
2549	ND	ND	ND	ND	ND	ND
2553	ND	5.2	ND	5.6	ND	ND
2561	-----	-----	6.777	-----	-----	2.652
2567	<10	<10	<10	<10	<10	<10
2572	<10	<10	<10	<10	<10	<10
2582	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2590	-----	3.095	-----	5.260	-----	-----
2605	ND	ND	ND	ND	ND	ND
2617	-----	-----	0	-----	-----	0
2629	ND	ND	ND	ND	ND	ND
2638	n.d	404.18	n.d	n.d	n.d	n.d
2643	-----	-----	-----	-----	-----	-----

	#19510			#19511		
lab	OP	NP	OPEO	OP	NP	NPEO
2644	-----	-----	-----	1.82	-----	2.02
2649	-----	-----	-----	-----	-----	-----
2681	<10	<10	<20	<10	<10	<20
2737	ND	ND	ND	ND	ND	ND
2773	ND	ND	ND	ND	ND	ND
2789	-----	-----	-----	-----	-----	-----
2791	N.D	18.83	N.D	N.D	N.D	N.D
2804	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
2812	-----	-----	-----	-----	-----	-----
2827	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2858	n.d	n.d	7.10	5.08	n.d	n.d
2870	-----	-----	-----	-----	-----	-----
2877	<5	93.4560	41.5265	38.5544	<5	6.2252
3100	<10.00	<10.00	<10.00	<10.00	<10.00	<10.00
3116	-----	-----	-----	-----	-----	-----
3146	-----	-----	-----	-----	-----	-----
3150	-----	-----	-----	-----	-----	-----
3154	-----	-----	-----	-----	-----	-----
3160	<5	<10	<30	5.86	<10	<30
3172	-----	-----	-----	-----	-----	-----
3182	<3	<3	<3	<3	<3	<3
3190	<10	<10	<10	<10	<10	<10
3197	<10	<10	<10	<10	<10	<10
3205	<2	2.4	<10	3.8	<2	<5
3210	<100	<100	<20	<100	<100	<20
3214	<10	<10	<10	<10	<10	<10
3218	＜10	＜10	<10	＜10	＜10	＜10
3220	ND	ND	ND	ND	ND	ND
3222	-----	-----	-----	-----	-----	1.97
3228	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3237	-----	-----	-----	-----	-----	-----
3248	-----	-----	-----	-----	-----	-----
3250	-----	5.34	-----	5.80	-----	-----
8025	<5	<5	<5	<5	<5	<5

Abbreviations of components:

OP = Octylphenol

NP = Nonylphenol

OPEO = Octylphenol Ethoxylates

NPEO = Nonylphenol Ethoxylates

APPENDIX 3

Details of the methods used by the participants

Lab	ISO/IEC17025 accredited for reported components(s)	sample intake (grams)	releasing/extraction technique	release solvent	extraction time (min)	extraction temperature (°C)
210	No	-----	Ultrasonic	Methanol	60	70
230	-----	-----	-----	-----	-----	-----
339	No	1	Ultrasonic	Methanol	60	70
551	No	1.0	Ultrasonic	Methanol	60	70
623	Yes	1	Ultrasonic	Methanol	60	70
840	Yes	0.5	Ultrasonic	MEOH	60	70
2108	Yes	0.5	-----	-----	-----	-----
2115	Yes	0.5	Ultrasonic	Methanol	60	60
2120	No	0.10 / 0.25	Ultrasonic	methanol	60	70
2129	Yes	0.5	Ultrasonic	Methanol	60	70
2135	Yes	0.5	Ultrasonic	Methanol	60	70
2138	Yes	1	Ultrasonic	Methanol	60	70
2139	Yes	1	Ultrasonic	Methanol	60	70
2159	Yes	1	Ultrasonic	Methanol	60	70
2165	Yes	1.0	Ultrasonic	Methanol	60	70
2201	Yes	1	Soxhlet	Methanol.	60	70
2232	Yes	1	Ultrasonic	Methanol	60	70
2241	Yes	0.5	Ultrasonic	methanol	60	70
2247	Yes	1.0	Ultrasonic	Methonal	60	70
2250	Yes	2.5	Ultrasonic	methanol	60	70
2255	Yes	0.5	Ultrasonic	Methanol	60	70
2265	Yes	0.5	Ultrasonic	Methanol	60	70
2285	Yes	1.00	Soxhlet	methyl alcohol	300	100
2289	Yes	1	Ultrasonic	methanol	60	70
2290	-----	-----	-----	-----	-----	-----
2291	Yes	1.00	Ultrasonic	methanol	60	70
2293	Yes	1	Ultrasonic	Methanol/ water	60	70
2295	Yes	1	Ultrasonic	Methanol	60	70
2301	Yes	1	Ultrasonic	Methanol	60	70
2310	Yes	1	Ultrasonic	Methanol	60	70
2311	Yes	1	Ultrasonic	Methanol	60	70
2320	Yes	1	Ultrasonic	Methanol	60	70
2330	Yes	0.5	Ultrasonic	Methanol	60	70
2347	Yes	1.0	Ultrasonic	methanol	60	60
2350	Yes	0.5 / 1.0	Ultrasonic	Methanol	60	70
2352	Yes	1	Ultrasonic	methanol	60	70
2357	Yes	1	Ultrasonic	methanol	60	70
2358	Yes	1.0	Ultrasonic	Methanol	60	70
2363	No	1	Ultrasonic	methanol	60	70
2365	Yes	1.0	Ultrasonic	Methyl alcohol	60	70
2369	Yes	0.5	Ultrasonic	methanol	60	70
2370	Yes	0.5	Ultrasonic	Methanol	60	70
2372	No	0.5	Ultrasonic	MeOH	60	70
2374	-----	-----	-----	-----	-----	-----
2375	Yes	0.5	Ultrasonic	Methanol	60	70
2378	No	1	Ultrasonic	methanol	60	70
2379	Yes		Ultrasonic	MeOH	60	70
2380	Yes	1.0	Ultrasonic	Methanol	60	70
2382	Yes	1	Ultrasonic	Methanol	60	70
2386	Yes	0.5	Ultrasonic	Methanol	60	70
2390	Yes	1	Ultrasonic	Methanol	60	70
2410	Yes	0.5	Ultrasonic	Methanol	60	70
2415	Yes	0.5	Ultrasonic	Methanol	60	70
2426	Yes	0.50	Ultrasonic	Methanol, water	60	70
2432	-----	-----	-----	-----	-----	-----
2467	No	0.5	Ultrasonic	Methanol	60	70
2492	Yes	0.5	Ultrasonic	Methanol	60	60
2495	Yes	0.5	Ultrasonic	Methanol	60	70
2497	Yes	1	Ultrasonic	methanol	60	60
2508	Yes	0.5	Ultrasonic	MEOH	60	40
2514	Yes	0.50	Ultrasonic	Methanol solvent	60	70
2549	Yes	0.5	Ultrasonic	Methanol	60	70
2553	Yes	1	Ultrasonic	Methanol	60	70
2561	Yes	1	Ultrasonic	Methanol	60	70
2567	Yes	0.5	Ultrasonic	Methanol	60	70
2572	Yes	-----	-----	-----	-----	-----
2582	Yes	1.00	Ultrasonic	Methanol	60	70
2590	Yes	0.5	Ultrasonic	MeOH	60	70
2605	Yes	1.000	Ultrasonic	Methanol	60	70
2617	Yes	1.00	Soxhlet	methanol	180	95
2629	Yes	1.0	Ultrasonic	Methanol	60	70
2638	No	1	Ultrasonic	methanol	60	room temperature

Lab	ISO/IEC17025 accredited for reported components(s)	sample intake (grams)	releasing/extraction technique	release solvent	extraction time (min)	extraction temperature (°C)
2643	Yes	1	Ultrasonic	methanol	60	70
2644	Yes	0.5	Ultrasonic	methanol	60	70
2649	Yes	0.5	Ultrasonic	Methanol	60	70
2681	Yes	1	Ultrasonic	methanol	60	70
2737	Yes	1	Ultrasonic	methanol	60	70
2773	Yes	0.50	Ultrasonic	methanol	60	70
2789	-----	-----	-----	-----	-----	-----
2791	Yes	0.5	Ultrasonic	Methanol	60	70
2804	Yes	1	Ultrasonic	Methanol	60	70
2812	No	1	Ultrasonic	Methanol	60	70
2827	Yes	1	Ultrasonic	Methanol	60	70
2858	Yes	1	Ultrasonic	Methanol	60	60
2870	Yes	1	Ultrasonic	Methanol	60	60
2877	No	1	Ultrasonic	Methanol	60	70
3100	Yes	1	Ultrasonic	methanol	60	70
3116	Yes	1	Ultrasonic	Methanol	60	70
3146	Yes	0.5	Ultrasonic	Methanol	60	70
3150	No	0.5	Ultrasonic	Methanol	60	70
3154	-----	-----	-----	-----	-----	-----
3160	No	2.5	Ultrasonic	Acetonitrile	60	50
3172	Yes	2	Ultrasonic	Methanol	60	70
3182	No	0.5	Ultrasonic	Methanol, water	60	70
3190	Yes	1	Ultrasonic	methanol	60	70
3197	Yes	0.5	Ultrasonic	Methanol	60	70
3205	Yes	0.5	Ultrasonic	Methanol	60	-----
3210	Yes	0.5	Ultrasonic	Methanol	60	70
3214	Yes	1	Ultrasonic	Methanol	60	70
3218	Yes	3	Ultrasonic	methanol	60	70
3220	Yes	1	Ultrasonic	Methanol	60	70
3222	Yes	1	Ultrasonic	methyl alcohol	60	70
3228	Yes	0.5	Ultrasonic	methanol	60	70
3237	Yes	0.5	Ultrasonic	Methanol	60	70
3248	Yes	1	Ultrasonic	Methanol	60	70
3250	Yes	1	Ultrasonic	Methanol (CH3OH)	60	70
8025	Yes	1	Ultrasonic	Methanol	60	40

APPENDIX 4

Number of participants per country

5 labs in	BANGLADESH
1 lab in	BRAZIL
3 labs in	CAMBODIA
2 labs in	FRANCE
11 labs in	GERMANY
1 lab in	GUATEMALA
5 labs in	HONG KONG
10 labs in	INDIA
2 labs in	INDONESIA
7 labs in	ITALY
5 labs in	KOREA
1 lab in	MAURITIUS
1 lab in	MOROCCO
24 labs in	P.R. of CHINA
3 labs in	PAKISTAN
1 lab in	PORTUGAL
1 lab in	ROMANIA
1 lab in	SINGAPORE
2 labs in	SPAIN
3 labs in	SRI LANKA
3 labs in	TAIWAN R.O.C.
2 labs in	THAILAND
7 labs in	TURKEY
1 lab in	UNITED KINGDOM
5 labs in	VIETNAM

APPENDIX 5

Abbreviations:

C = final 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
E = probably an error in calculations
W = test result withdrawn on request of participant
ex = test result excluded from statistical evaluation
n.a. = not applicable
n.e. = not evaluated
n.d. = not detected
fr. = first reported

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