Results of Proficiency Test
AP & APEO in textile
February 2018

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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CONTENTS

1 INTRODUCTION

Alkylphenol ethoxylates (APEO), like Octylphenol ethoxylates (OPEO) and Nonylphenol ethoxylates (NPEO) have widely been used in manufacturing antioxidants, lubricating oil additives, laundry and dish detergents, emulsifiers, wetting agents in cosmetics, including hair products, defoaming agents and solubilizers. Human exposure to APEO can occur through various sources such as environmental, food or skin contact. Considering their toxicity on several animal species, minimization of exposure to APEO is recognized as important to the preservation of human health. APEO may degradate in the environment to the corresponding Octyl- and Nonylphenol (OP & NP). These alkylphenols (AP) have attracted attention due to its prevalence in the environment and its potential role as an endocrine disruptor and xenoestrogen, due to its ability to act with estrogen-like activity. The European Union has implemented sales and use restrictions on certain applications in which alkylphenols are used because of their alleged "toxicity, persistence, and the liability to bioaccumulate".

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 2017/2018 it was decided to continue with the PT for the analyses of AP and APEO content in textile. In this interlaboratory study, 96 laboratories in 24 different countries registered for participation. See appendix 4 for the number of participants per country.
In this report, the results of the 2018 proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send two different samples (labelled #18510 and #18511) which were positive (artificially fortified) 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 IEC/ISO17043:2010. 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 March 2017 (iis-protocol, version 3.4). 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, #18510 (pink cotton fabric) treated to be positive on OPEO and #18511 (red/navy patterned cotton/polyester) treated to be positive on NPEO were selected.

The batch for sample #18510 (pink cotton fabric) was used in a previous proficiency test on AP+APEO in textile (iis16A04, sample #16530). In iis16A04 the homogeneity of this batch was demonstrated sufficiently without doubt. Therefore, homogeneity of the subsamples #18510 was assumed.
From this batch, after mixing well, 117 subsamples of approx. 2.5 grams each were prepared and labelled #18510.

The batch for sample #18511 (red/navy patterned cotton/polyester) was prepared by a third party. The bulk sample was cut into pieces. From this batch, after mixing well, 120 subsamples of approx. 3 grams each were prepared and labelled #18511.
The homogeneity of eight stratified randomly selected samples was checked by determination of NPEO using an in-house test method. See the following table for the test results.

	NPEO in mg/kg
Sample #18511-1	78.9
Sample #18511-2	76.5
Sample #18511-3	80.8
Sample #18511-4	75.3
Sample #18511-5	75.2
Sample #18511-6	76.4
Sample #18511-7	76.6
Sample #18511-8	77.1

Table 1: homogeneity test results of subsamples #18511

From the above results of the homogeneity test, the repeatability was calculated and compared with 0.3 times the corresponding reproducibility of the reference method in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	NPEO in mg/kg
r (observed)	5.3
reference method	Horwitz (n=5)
0.3*R (reference method)	12.0

Table 2: evaluation of the repeatability of subsamples #18511

The repeatability of NPEO was in agreement with 0.3 times the target requirements. Therefore, homogeneity of the subsamples #18511 was assumed.

To each participating laboratory one sample labelled #18510 and one sample labelled #18511 were sent on February 5, 2018.

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 #18510 and #18511 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 calculations.

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 March 2017 (iis-protocol, version 3.4).

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 ISO 5725 the original test results per determination were submitted subsequently to Dixon's, Grubbs' and or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.
For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have significant consequences for the evaluation of the test results.

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

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. Outliers and other data, which were excluded from the

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

3.3 Z-SCORES

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

The target standard deviation was calculated from the target reproducibility (preferably taken from a standardized test method) by division with 2.8.

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:

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

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 serious problems occurred, although three participants reported the test results after closure of the data entry tool of this proficiency test (PT) and four participants did not report any results at all. In total 92 of the 96 participants reported 329 numerical test results. Observed in all reported results were 8 statistical outlying results, which is 2.4%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

ISO 18254, used by the majority of the participants, is the official test method for the determination of APEO in textiles. Regretfully ISO 18254 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). Therefore, the target requirements in this study were estimated using the Horwitz equation (for n=5).

4.1 EVALUATION PER SAMPLE AND PER COMPONENT

Not all original data sets proved to have a normal Gaussian distribution. These are referred to as “not OK” or “suspect”. The statistical evaluation of these data sets should be used with due care.
Most participants agreed about the absence of Octylphenol and Nonylphenol.

sample #18510

OPEO: The determination of this component may not be problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the estimated target reproducibility using the Horwitz equation for 5 components.

sum OP + NP + OPEO + NPEO:
The determination of this sum-component may not be problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated target reproducibility using the Horwitz equation for 5 components.

sample #18511

NPEO: The determination of this component may be problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not 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 may be problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated target reproducibility using the Horwitz equation for 5 components.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the estimated target reproducibilities and the reproducibilities as found for the group of participating laboratories.
The number of significant results, the average results, the calculated reproducibilities (2.8*standard deviation) and the target reproducibilities are compared in the next tables:

	unit	*n*	*Average*	*2.8 * sd*	*R (target)**
OPEO	mg/kg	89	63.12	28.41	33.88
sum OP + NP + OPEO + NPEO	mg/kg	74	63.85	26.77	34.22

Table 3: reproducibilities of tests on sample #18510

*) against the Horwitz equation (n=5)

	unit	*n*	*Average*	*2.8 * sd*	*R (target)**
NPEO	mg/kg	87	54.22	42.31	29.78
sum OP + NP + OPEO + NPEO	mg/kg	71	54.01	39.39	29.68

Table 4: reproducibilities of tests on sample #18511

*) against the Horwitz equation (n=5)

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF FEBRUARY 2018 WITH PREVIOUS PTS

	February 2018	March 2017	March 2016
Total Number of reporting labs	92	95	105
Number of results reported	329	378	412
Statistical outliers	8	9	13
Percentage outliers	2.4%	2.4%	3.2%

Table 5: 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 2018	March 2017	March 2016
Octylphenol (OP)	n.a.	n.a.	n.a.
Nonylphenol (NP)	n.a.	n.a.	n.a.
Octylphenol Ethoxylates (OPEO)	16%	15%	16%
Nonylphenol Ethoxylates (NPEO)	28%	18%	27%
sum OP + NP + OPEO + NPEO	15% / 26%	15% / 17%	16% / 25%

Table 6: comparison of uncertainties (relative in %)

4.4 EVALUATION ANALYTICAL DETAILS

For this PT, some analytical details were requested (see appendix 3). Questions like: Is your laboratory accredited in accordance with ISO/IEC17025 and some specific questions with regards to the analytical details of the test method used.
Based on the answers given by the participants the following can be summarized:
sixty-three 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 between 0.5 – 1.0 grams. Two mentioned to have use less material (0.5 gram) for intake and two have used more testing material for intake (>1 gram).
The majority of the group mentioned to have used Triton X-100 as calibration standard for sample #18510 and Igepal 630 as calibration standard for sample #18511.
All reporting participants mentioned to have used ultrasonic as technique to release/extract the analytes.
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 has no problem with the analysis of OPEO and NPEO in textile at the levels as present in this PT (respectively 63 and 54 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 two of the reporting laboratories would reject sample #18510 for containing too much OP + NP + OPEO + NPEO in total and that two of the reporting laboratories would reject sample #18511 for containing too much NPEO and/or too much OP + NP + OPEO + NPEO in total.
It is remarkable 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 7: 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.

For sample #18511 the average of the homogeneity test results is not in line with the average (consensus value) from the PT results. There are several reasons for this. First the goal of the homogeneity testing is very different from the goal of the evaluation of the reported PT results. In order to proof the homogeneity of the PT samples, a test method is selected with a high precision (smallest variation). The accuracy (trueness) of the test method is less relevant.

Secondly, the homogeneity testing is done by one laboratory only. The test results of this (ISO/IEC 17025 accredited) laboratory will have a bias (systematic deviation) depending on the test method used. The desire to detect small variations between the PT samples leads to the use of a sensitive test method with high precision, which may be a test method with significant bias. Also, each test result reported by the laboratories that participate in the PT will have a bias. However, some will have a positive bias and others a negative bias. These different biases compensate each other in the PT average (consensus value). Therefore, the PT consensus value may deviate from the average of the homogeneity test. At the same time, the accuracy of the PT consensus value is more reliable than the accuracy of the average of the results of the homogeneity test.

Sample #18510 was used in a previous proficiency test (iis16A04, sample #16530). It is concluded that the samples textile containing OPEO are stable for at least two years.

	unit	#18510			#16530		
		n	mean	R(calc)	n	mean	R(calc)
OPEO	mg/kg	89	63.1	28.4	102	65.6	29.6

Table 8: comparison of sample #18510 with #16530

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.

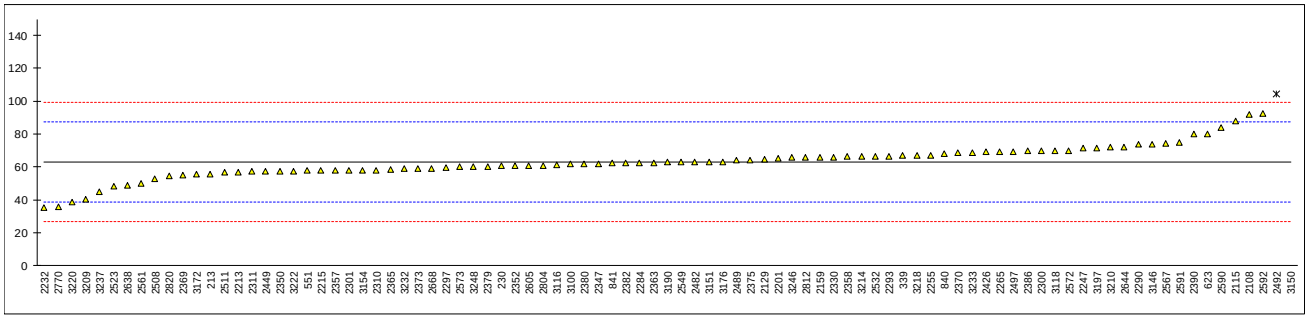
APPENDIX 1

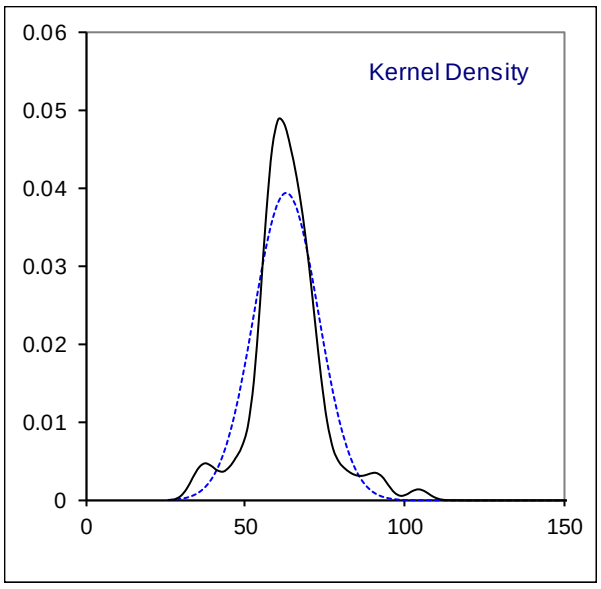
Determination of Octylphenol Ethoxylates (OPEO) on sample #18510; results in mg/kg

lab	Method	value	mark	z(targ)	remarks
213	In house	55.85		-0.60	
230	ISO18254-1	60.7		-0.20	
339	In house	66.8		0.30	
551	In house	57.78		-0.44	
623	ISO18254-1	79.90		1.39	
840	In house	68.20		0.42	
841	ISO18254	62.17		-0.08	
2108	In house	92.03		2.39	
2115	ISO18254-1	87.82		2.04	
2129	ISO/DIS 18254-1	64.58		0.12	
2159	ISO18254-1	66.0		0.24	
2201	GB/T23322	65.5		0.20	
2213	ISO18254-1	57		-0.51	
2215	In house	57.80		-0.44	
2232	In house	35.51		-2.28	
2247	ISO/DIS 18254-1	71.28		0.67	
2255	ISO/DIS 18254-1	67.22		0.34	
2265	ISO18254-1	69.4		0.52	
2284	ISO18254-1	62.6		-0.04	
2290	In house	73.52		0.86	
2293	In house	66.569		0.29	
2297	ISO/DIS 18254-1	59.82		-0.27	
2300	ISO/DIS 18254-1	69.82		0.55	
2301	ISO/DIS 18254-1	57.88		-0.43	
2310	ISO18254-1	58.05		-0.42	
2311	ISO18254-1	57.10		-0.50	
2330	ISO18254-1	66.08		0.24	
2347	ISO/DIS 18254-1	62		-0.09	
2350	ISO18254-1	57.42		-0.47	
2352	ISO18254-1	60.74		-0.20	
2357	ISO18254-1	57.8		-0.44	
2358	ISO18254-1	66.15		0.25	
2363	In house	62.7		-0.03	
2365	ISO18254-1	58.4		-0.39	
2369	ISO18254-1	55		-0.67	
2370	ISO18254	68.7		0.46	
2373	ISO/DIS 18254-1	58.97		-0.34	
2375	ISO18254-1	64.3		0.10	
2379	ISO18254-1	60.31		-0.23	
2380	ISO18254-1	61.70		-0.12	
2382	ISO/DIS 18254-1	62.2		-0.08	
2386	ISO18254-1	69.74		0.55	
2390	ISO18254-1	79.81		1.38	
2426	ISO18254	69.33		0.51	
2449	ISO18254-1	57.23		-0.49	
2452		-----		-----	
2482	ISO18254-1	63.08		0.00	
2489	ISO18254-1	64.1		0.08	
2492	In house	104.6	R(0.01)	3.43	
2495		-----		-----	
2497	ISO18254-1	69.41		0.52	
2508	D7485/D7742	52.85	C	-0.85	First reported 108.86
2511	ISO/DIS 18254-1	56.8		-0.52	
2523		48.216		-1.23	
2532	ISO18254-1	66.3		0.26	
2549	ISO/DIS 18254-1	62.8		-0.03	
2561	ISO18254-1	49.87		-1.09	
2567	ISO/DIS 18254-1	74.5		0.94	
2572	In house	70.08		0.58	
2573	ISO/DIS 18254-1	60.0		-0.26	
2590	ISO18254-1	84.087		1.73	
2591	In house	75.00		0.98	
2592	ISO18218-2	92.6		2.44	
2605	ISO18254-1	60.88		-0.18	
2629		-----		-----	
2638	In house	48.65		-1.20	
2644	ISO18254-1	72.34		0.76	
2668	ISO/DIS 18254-1	59.32		-0.31	
2713	In house	<50		-----	
2770	ISO18218-1	36		-2.24	
2804	ISO18254-1	60.95		-0.18	
2812	In house	65.92		0.23	
2820	ISO18218-1	54.32		-0.73	
3100	ISO18254-1	61.63		-0.12	

lab	Method	value	mark	z(targ)	remarks
3116	ISO18254-1	61.1		-0.17	
3118	In house	69.87		0.56	
3146	ISO18254-1	73.57		0.86	
3150	ISO/DIS 18254-1	505.1	C,R(0.01)	36.53	First reported 661.1
3151	In house	63.09		0.00	
3154	In house	57.89		-0.43	
3163		-----		-----	
3172	ISO18254-1	55.52		-0.63	
3176	In house	63.20		0.01	
3190	ISO18254-1	62.77		-0.03	
3197	ISO18254-1	71.3		0.68	
3209	In house	40.462		-1.87	
3210		72.26		0.76	
3214		66.29		0.26	
3218	ISO18254-1	66.8		0.30	
3220	ISO18254-1	38.53		-2.03	
3222	ISO18254-1	57.5		-0.46	
3232	ISO/DIS 18254-1	58.90		-0.35	
3233	In house	68.72		0.46	
3237	ISO18254-1	44.8		-1.51	
3246	In house	65.6		0.21	
3248	ISO18254-1	60		-0.26	

normality	suspect
n	89
outliers	2
mean (n)	63.116
st.dev. (n)	10.1461
R(calc.)	28.409
st.dev.(Horwitz (n=5))	12.1004
R(Horwitz (n=5))	33.881

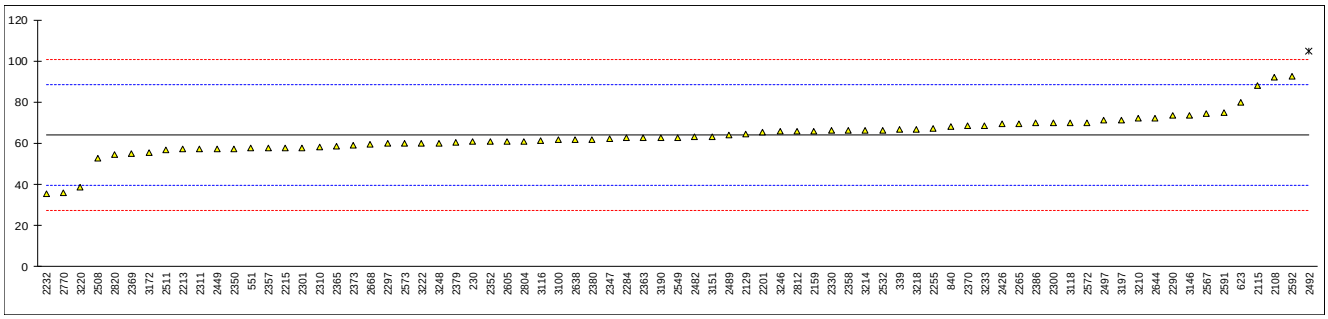


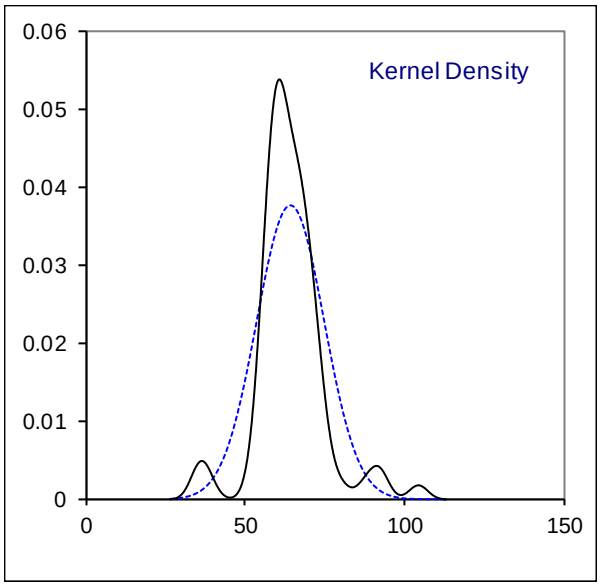


Determination of Total of OP, NP, OPEO and NPEO on sample #18510; results in mg/kg

lab	method	value	mark	z(targ)	remarks
213		-----		-----	
230	ISO18254-1	60.7		-0.26	
339	In house	66.8		0.24	
551	In house	57.78		-0.50	
623	ISO18254-1	79.90		1.31	
840	In house	68.20		0.36	
841		-----		-----	
2108	In house	92.03		2.31	
2115	ISO18254-1	87.82		1.96	
2129	ISO/DIS 18254-1	64.58		0.06	
2159	ISO18254-1	66.0		0.18	
2201	GB/T23322	65.5		0.13	
2213	ISO18254-1	57		-0.56	
2215	In house	57.80		-0.50	
2232	In house	35.51		-2.32	
2247		-----		-----	
2255	ISO/DIS 18254-1	67.22		0.28	
2265	ISO18254-1	69.4		0.45	
2284	ISO18254-1	62.6		-0.10	
2290	In house	73.52		0.79	
2293		-----		-----	
2297	ISO/DIS 18254-1	59.82		-0.33	
2300	ISO/DIS 18254-1	69.82		0.49	
2301	ISO/DIS 18254-1	57.88		-0.49	
2310	ISO18254-1	58.1		-0.47	
2311	ISO18254-1	57.10		-0.55	
2330	ISO18254-1	66.08		0.18	
2347	ISO/DIS 18254-1	62		-0.15	
2350	ISO18254-1	57.42		-0.53	
2352	ISO18254-1	60.74		-0.25	
2357	ISO18254-1	57.8		-0.50	
2358	ISO18254-1	66.15		0.19	
2363	In house	62.7		-0.09	
2365	ISO18254-1	58.4		-0.45	
2369	ISO18254-1	55		-0.72	
2370	ISO18254	68.7		0.40	
2373	ISO/DIS 18254-1	58.97		-0.40	
2375		-----		-----	
2379	ISO18254-1	60.31		-0.29	
2380	ISO18254-1	61.70		-0.18	
2382		-----		-----	
2386	ISO18254-1	69.74		0.48	
2390		-----		-----	
2426	ISO18254	69.33		0.45	
2449	ISO18254-1	57.23		-0.54	
2452		-----		-----	
2482	ISO18254-1	63.08		-0.06	
2489	ISO18254-1	64.1		0.02	
2492	In house	104.6	R(0.05)	3.33	
2495		-----		-----	
2497	ISO18254-1	71.284		0.61	
2508	D7485/D7742	52.85	C	-0.90	First reported 108.86
2511	ISO/DIS 18254-1	56.8		-0.58	
2523		-----		-----	
2532	ISO18254-1	66.3		0.20	
2549	ISO/DIS 18254-1	62.8		-0.09	
2561		-----		-----	
2567	ISO/DIS 18254-1	74.5		0.87	
2572	In house	70.08		0.51	
2573	ISO/DIS 18254-1	60.0		-0.32	
2590		-----		-----	
2591	In house	75.00		0.91	
2592	ISO18218-2	92.6		2.35	
2605	ISO18254-1	60.88		-0.24	
2629		-----		-----	
2638	In house	61.671		-0.18	
2644	ISO18254-1	72.34		0.69	
2668	ISO/DIS 18254-1	59.32		-0.37	
2713	In house	<50		-----	
2770	ISO18218-1	36		-2.28	
2804	ISO18254-1	60.95		-0.24	
2812	In house	65.92		0.17	
2820	ISO18218-1	54.32		-0.78	
3100	ISO18254-1	61.63		-0.18	

lab	method	value	mark	z(targ)	remarks
3116	ISO18254-1	61.1		-0.23	
3118	In house	69.87		0.49	
3146	ISO18254-1	73.57		0.80	
3150	ISO/DIS 18254-1	ND	C	-----	First reported 661.1
3151	In house	63.09		-0.06	
3154		-----		-----	
3163		-----		-----	
3172	ISO18254-1	55.52		-0.68	
3176	In house	-----		-----	
3190	ISO18254-1	62.77		-0.09	
3197	ISO18254-1	71.3		0.61	
3209		-----		-----	
3210	-----	72.26		0.69	
3214	-----	66.29		0.20	
3218	ISO18254-1	66.8		0.24	
3220	ISO18254-1	38.53		-2.07	
3222	ISO18254-1	60.0		-0.32	
3232		-----		-----	
3233	In house	68.72		0.40	
3237		-----		-----	
3246	In house	65.6		0.14	
3248	ISO18254-1	60		-0.32	
	normality	not OK			
	n	74			
	outliers	1			
	mean (n)	63.854			
	st.dev. (n)	9.5602			
	R(calc.)	26.769			
	st.dev.(Horwitz (n=5))	12.2204			
	R(Horwitz (n=5))	34.217			Compare R(ISO18254-1:16) = 17.536



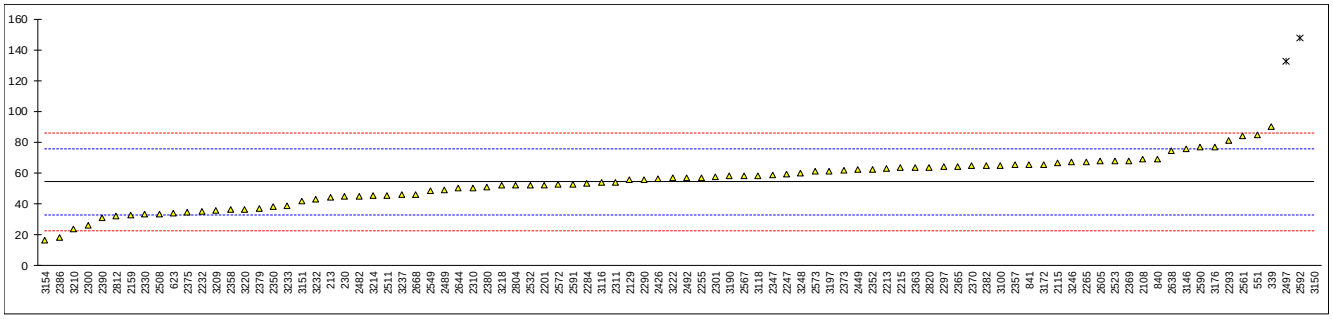


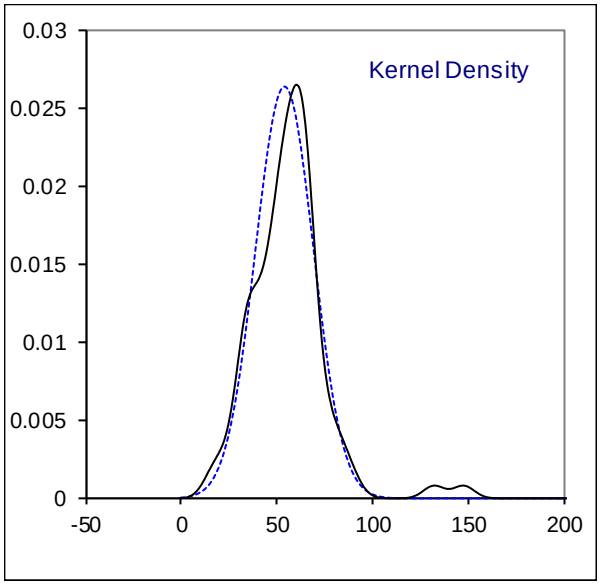
Determination of Nonylphenol Ethoxylates (NPEO) on sample #18511; results in mg/kg

lab	method	value	mark	z(targ)	remarks
213	In house	44.24		-0.94	
230	ISO18254-1	44.60		-0.90	
339	In house	90.2		3.38	
551	In house	84.98	C	2.89	First reported 104.23
623	ISO18254-1	34.08		-1.89	
840	In house	69.01		1.39	
841	ISO18254	65.24		1.04	
2108		68.96		1.39	
2115	ISO18254-1	66.31		1.14	
2129	ISO/DIS 18254-1	55.7		0.14	
2159	ISO18254-1	32.5		-2.04	
2201	GB/T23322	52.3		-0.18	
2213	ISO/DIS 18254-1	63		0.83	
2215	In house	63.61		0.88	
2232	In house	35.03		-1.80	
2247	ISO/DIS 18254-1	59.51		0.50	
2255	ISO18254-1	57.11		0.27	
2265	ISO18254-1	67.5		1.25	
2284	ISO18254-1	53.1		-0.11	
2290	In house	55.73		0.14	
2293	In house	81.407		2.56	
2297	ISO/DIS 18254-1	64.13		0.93	
2300	ISO/DIS 18254-1	26.28		-2.63	
2301	ISO/DIS 18254-1	57.27		0.29	
2310	ISO18254-1	50.56		-0.34	
2311	ISO18254-1	54.15		-0.01	
2330	ISO18254-1	33.43		-1.96	
2347	ISO/DIS 18254-1	59		0.45	
2350	ISO/DIS 18254-1	38.39		-1.49	
2352	ISO18254-1	62.42		0.77	
2357	ISO18254-1	65.2		1.03	
2358	ISO18254-1	36.35		-1.68	
2363	In house	63.7		0.89	
2365	ISO18254-1	64.2		0.94	
2369	ISO18254-1	68		1.30	
2370	ISO18254	64.7		0.99	
2373		62		0.73	
2375	ISO18254-1	34.7		-1.84	
2379	ISO18254-1	37.29		-1.59	
2380	ISO18254-1	50.6		-0.34	
2382	ISO/DIS 18254-1	64.9		1.00	
2386	ISO18254-1	18.18		-3.39	
2390	ISO18254-1	30.90		-2.19	
2426	ISO18254	56.46		0.21	
2449	ISO18254-1	62.31		0.76	
2452		-----		-----	
2482	ISO18254-1	44.88		-0.88	
2489	ISO18254-1	49		-0.49	
2492	In house	57.1		0.27	
2495		-----		-----	
2497	ISO18254-1	132.29	R(0.01)	7.34	
2508	D7485/D7742	33.54		-1.94	
2511	ISO/DIS 18254-1	45.3		-0.84	
2523		67.919		1.29	
2532	ISO18254-1	52.2		-0.19	
2549	ISO/DIS 18254-1	48.3		-0.56	
2561	ISO18254-1	83.84		2.78	
2567	ISO/DIS 18254-1	58.4		0.39	
2572	In house	52.45		-0.17	
2573	ISO/DIS 18254-1	61.0		0.64	
2590	ISO18254-1	76.926		2.13	
2591	In house	53.00		-0.12	
2592	ISO18218-2	147.8	R(0.01)	8.80	
2605	ISO18254-1	67.71		1.27	
2629		-----		-----	
2638	In house	74.37		1.89	
2644	ISO18254-1	50.53	C	-0.35	First reported 101.05
2668	ISO/DIS 18254-1	46.10		-0.76	
2713	In house	<50		-----	
2770	ISO18218-1	N.D.		-----	
2804	ISO18254-1	52.12		-0.20	
2812	In house	32.44		-2.05	
2820	ISO18218-1	63.84		0.90	
3100	ISO18254-1	65.08		1.02	

lab	method	value	mark	z(targ)	remarks
3116	ISO18254-1	53.8		-0.04	
3118		58.44		0.40	
3146	ISO18254-1	75.43		1.99	
3150	ISO/DIS 18254-1	448.1	C,R(0.01)	37.03	First reported test result as NP instead of NPEO
3151	In house	41.76		-1.17	
3154	In house	16.50		-3.55	
3163		-----		-----	
3172	ISO18254-1	65.66		1.08	
3176	In house	77.0		2.14	
3190	ISO18254-1	57.85		0.34	
3197	ISO18254-1	61.4		0.67	
3209	In house	35.521		-1.76	
3210		23.63		-2.88	
3214		45.29		-0.84	
3218	ISO18254-1	51.9		-0.22	
3220	ISO18254-1	36.65		-1.65	
3222	ISO18254-1	56.7		0.23	
3232	ISO/DIS 18254-1	42.95		-1.06	
3233	In house	38.86		-1.44	
3237	ISO18218-2	45.8		-0.79	
3246		67.0		1.20	
3248	ISO18254-1	60		0.54	

normality	OK
n	87
outliers	3
mean (n)	54.223
st.dev. (n)	15.1088
R(calc.)	42.305
st.dev.(Horwitz (n=5))	10.6358
R(Horwitz (n=5))	29.780

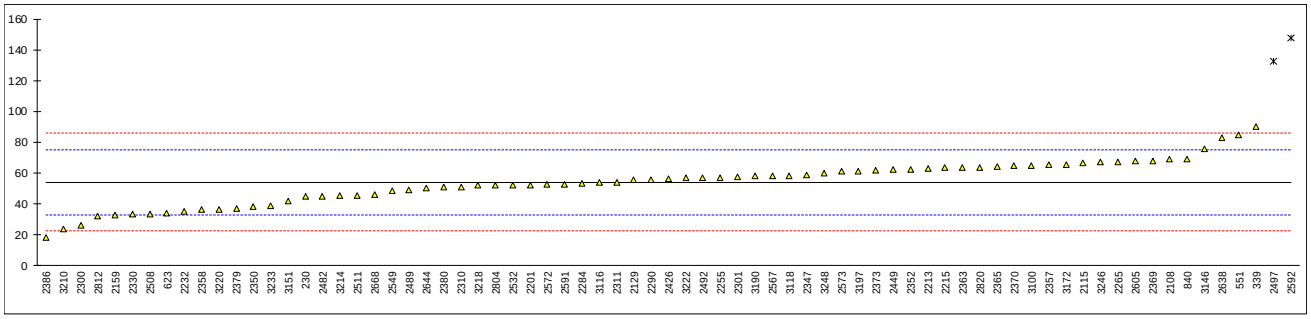


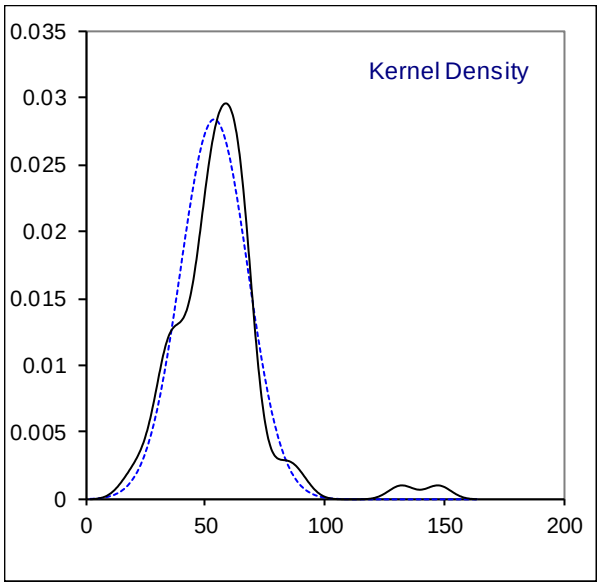


Determination of Total of OP, NP, OPEO and NPEO on sample #18511; results in mg/kg

lab	method	value	mark	z(targ)	remarks
213		-----		-----	
230	ISO18254-1	44.60		-0.89	
339	In house	90.2		3.41	
551	In house	84.98	C	2.92	First reported 104.23
623	ISO18254-1	34.08		-1.88	
840	In house	69.01		1.42	
841		-----		-----	
2108	----	68.96		1.41	
2115	ISO18254-1	66.31		1.16	
2129	ISO/DIS 18254-1	55.7		0.16	
2159	ISO18254-1	32.5		-2.03	
2201	GB/T23322	52.3		-0.16	
2213	ISO/DIS 18254-1	63		0.85	
2215	In house	63.61		0.91	
2232	In house	35.03		-1.79	
2247		-----		-----	
2255	ISO18254-1	57.11		0.29	
2265	ISO18254-1	67.5		1.27	
2284	ISO18254-1	53.1		-0.09	
2290	In house	55.73		0.16	
2293		-----		-----	
2297	ISO/DIS 18254-1	<10		<5.10	False negative test result?
2300	ISO/DIS 18254-1	26.28		-2.62	
2301	ISO/DIS 18254-1	57.27		0.31	
2310	ISO18254-1	50.6		-0.32	
2311	ISO18254-1	54.15		0.01	
2330	ISO18254-1	33.43		-1.94	
2347	ISO/DIS 18254-1	59		0.47	
2350	ISO/DIS 18254-1	38.39		-1.47	
2352	ISO18254-1	62.42		0.79	
2357	ISO18254-1	65.2		1.06	
2358	ISO18254-1	36.35		-1.67	
2363	In house	63.7		0.91	
2365	ISO18254-1	64.2		0.96	
2369	ISO18254-1	68		1.32	
2370	ISO18254	64.7		1.01	
2373	-----	62		0.75	
2375	ISO18254-1	-----		-----	
2379	ISO18254-1	37.29		-1.58	
2380	ISO18254-1	50.6		-0.32	
2382		-----		-----	
2386	ISO18254-1	18.18		-3.38	
2390		-----		-----	
2426	ISO18254	56.46		0.23	
2449	ISO18254-1	62.31		0.78	
2452		-----		-----	
2482	ISO18254-1	44.88		-0.86	
2489	ISO18254-1	49		-0.47	
2492	In house	57.1		0.29	
2495		-----		-----	
2497	ISO18254-1	132.367	R(0.01)	7.39	
2508	D7485/D7742	33.54		-1.93	
2511	ISO/DIS 18254-1	45.3		-0.82	
2523		-----		-----	
2532	ISO18254-1	52.2		-0.17	
2549	ISO/DIS 18254-1	48.3		-0.54	
2561		-----		-----	
2567	ISO/DIS 18254-1	58.4		0.41	
2572	In house	52.45		-0.15	
2573	ISO/DIS 18254-1	61.0		0.66	
2590		-----		-----	
2591	In house	53.00		-0.10	
2592	ISO18218-2	147.8	R(0.01)	8.85	
2605	ISO18254-1	67.71		1.29	
2629		-----		-----	
2638	In house	82.910		2.73	
2644	ISO18254-1	50.53	C	-0.33	First reported 101.05
2668	ISO/DIS 18254-1	46.10		-0.75	
2713	In house	<50		-----	
2770	ISO18218-1	N.D.		-----	
2804	ISO18254-1	52.12		-0.18	
2812	In house	32.44		-2.03	
2820	ISO18218-1	63.84		0.93	
3100	ISO18254-1	65.08		1.04	

lab	method	value	mark	z(targ)	remarks
3116	ISO18254-1	53.8		-0.02	
3118	----	58.44		0.42	
3146	ISO18254-1	75.43		2.02	
3150	ISO/DIS 18254-1	-----	W	-----	Test result withdrawn, reported 448.1
3151	In house	41.76		-1.16	
3154	In house	-----		-----	
3163		-----		-----	
3172	ISO18254-1	65.66		1.10	
3176	In house	-----		-----	
3190	ISO18254-1	57.85		0.36	
3197	ISO18254-1	61.4		0.70	
3209	In house	-----		-----	
3210	----	23.63		-2.87	
3214	----	45.29		-0.82	
3218	ISO18254-1	51.9		-0.20	
3220	ISO18254-1	36.65		-1.64	
3222	ISO18254-1	56.7		0.25	
3232		-----		-----	
3233	In house	38.86		-1.43	
3237	ISO18218-2	-----		-----	
3246	----	67.0		1.23	
3248	ISO18254-1	60		0.57	
	normality	OK			
	n	71			
	outliers	2			
	mean (n)	54.007			
	st.dev. (n)	14.0662			
	R(calc.)	39.385			
	st.dev.(Horwitz (n=5))	10.5998			
	R(Horwitz (n=5))	29.679			Compare R(ISO18254-1) = 14.891





APPENDIX 2

Summary of other reported components in sample #18510 and #18511

	#18510			#18511		
lab	**OP**	**NP**	**NPEO**	**OP**	**NP**	**OPEO**
213	-----	-----	-----	-----	-----	-----
230	-----	-----	-----	-----	-----	-----
339	<10	<10	<10	<10	<10	<10
551	ND	ND	ND	ND	ND	ND
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
841	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2108	-----	-----	-----	-----	-----	-----
2115	-----	-----	-----	-----	-----	-----
2129	<5	<5	<5	<5	<5	<5
2159	<10	<10	<10	<10	<10	<10
2201	<3	<3	<10	<3	<3	<10
2213	<10	<10	<10	<10	<10	<10
2215	ND	ND	ND	ND	ND	ND
2232	-----	-----	-----	-----	-----	-----
2247	nd	nd	nd	nd	nd	nd
2255	ND	ND	ND	ND	ND	ND
2265	-----	-----	< 10	-----	-----	< 10
2284	-----	-----	-----	-----	-----	-----
2290	<10	<10	<10	<10	<10	<10
2293	-----	-----	-----	-----	-----	-----
2297	<10	<10	<10	<10	<10	<10
2300	nd	nd	nd	nd	nd	nd
2301	ND	ND	ND	ND	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
2330	ND	ND	ND	ND	ND	ND
2347	<10	<10	<30	<10	<10	<30
2350	< 1.00	< 1.00	< 1.00	< 1.00	< 1.00	< 1.00
2352	0	0	0	0	0	0
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	<10mg/kg	<10mg/kg	<30mg/kg	<10mg/kg	<10mg/kg	<30mg/kg
2370	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2373	-----	-----	-----	-----	-----	-----
2375	-----	-----	-----	-----	-----	-----
2379	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2380	-----	-----	-----	-----	-----	-----
2382	-----	-----	-----	-----	-----	-----
2386	<5	<5	<10	<10	<10	<10
2390	ND	ND	ND	ND	ND	ND
2426	ND	ND	ND	ND	ND	ND
2449	-----	-----	-----	-----	-----	-----
2452	-----	-----	-----	-----	-----	-----
2482	-----	-----	-----	-----	-----	-----
2489	ND	ND	ND	ND	ND	ND
2492	-----	-----	-----	-----	-----	-----
2495	-----	-----	-----	-----	-----	-----
2497	0.0001	0.104	1.77	0.0001	0.077	0.0001
2508	< 5	< 5	< 10	< 5	< 5	< 10
2511	-----	-----	-----	-----	-----	-----
2523	-----	-----	N.D.	-----	-----	N.D.
2532	ND	ND	ND	ND	ND	ND
2549	<10	<10	<10	<10	<10	<10
2561	-----	-----	<10	-----	-----	<10
2567	<10	<10	<10	<10	<10	<10
2572	<10	<10	<10	<10	<10	<10
2573	ND	ND	ND	ND	ND	ND
2590	-----	-----	-----	-----	-----	-----
2591	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0
2592	-----	-----	-----	-----	-----	-----
2605	ND	ND	ND	ND	ND	ND
2629	-----	-----	-----	-----	-----	-----
2638	not detected	-----	-----	-----	8.54	-----
2644	-----	-----	-----	-----	-----	-----
2668	<10	<10	<10	<10	<10	<10
2713	<50	<50	<50	<50	<50	<50
2770	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
2804	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2812	< 10	< 10	< 10	< 10	< 10	< 10
2820	-----	-----	-----	-----	-----	-----

	#18510			#18511		
lab	OP	NP	NPEO	OP	NP	OPEO
3100	<3	<3	<10	<3	<3	<3
3116	-----	-----	-----	-----	-----	-----
3118	ND	ND	ND	ND	ND	ND
3146	<10	<10	<10	<10	<10	<10
3150	-----	-----	-----	-----	-----	-----
3151	-----	-----	-----	-----	-----	-----
3154	-----	-----	-----	-----	-----	-----
3163	-----	-----	-----	-----	-----	-----
3172	-----	-----	-----	-----	-----	-----
3176	-----	-----	-----	-----	-----	-----
3190	-----	-----	-----	-----	-----	-----
3197	ND	ND	ND	ND	ND	ND
3209	-----	-----	-----	-----	-----	-----
3210	<10	<10	<10	<10	<10	<10
3214	< 1 ppm	< 1 ppm	< 1 ppm	< 1 ppm	< 1 ppm	< 1 ppm
3218	<10	<10	<10	<10	<10	<10
3220	ND	ND	ND	ND	ND	ND
3222	N.D.	N.D.	2.5	N.D.	N.D.	N.D.
3232	-----	-----	n.d.	-----	-----	n.d.
3233	< 10	< 10	< 10	< 10	< 10	< 10
3237	-----	-----	-----	-----	-----	-----
3246	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3248	-----	-----	-----	-----	-----	-----

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 component(s)	sample intake (in grams)	Sample #18510: material(s) used for calibration	Sample #18510: isomers observed	Sample #18511: material(s) used for calibration	Sample #18511: isomers observed	release/extract the analyte(s)
213	No						Ultrasonic
230	---						---
339	No	1	Etalon Igepal 603 CO (CAS : 68412-54-4) Etalon triton X 100 (CAS : 9002-93-1) 4 n nonylphenol (CAS : 104-40-5) 4 octyl phenol (CAS : 1806-26-4) Nonyl phenol technical (CAS : 84852-15-3)		Etalon Igepal 603 CO (CAS : 68412-54-4) Etalon triton X 100 (CAS : 9002-93-1) 4 n nonylphenol (CAS : 104-40-5) 4 octyl phenol (CAS : 1806-26-4) Nonyl phenol technical (CAS : 84852-15-3)		Ultrasonic
551	Yes	1.0032	TRITON X-100 / CAS NUMBER 9002-93-1	-	IGEPAL CO-630 / CAS NUMBER 68412-54-4	-	Ultrasonic
623	Yes	1	Triton X-100 CAS: 9002-93-1	POE (9-10) Tert-octylphenol	IGEPAL(R) CO-630 CAS: 68412-54-4	POE (9-10) Nonylphenol	Ultrasonic
840	Yes	0.5					Ultrasonic
841	---						---
2108	Yes	0.5	Mix of OP-(0-2) EO and Mix of OP(3-20)EO (IPEGAL CA-520 and IPEGAL CA-720)	OP-1-20-EO	Mix of NP-(0-20) EO and Mix of NP-(3-20) EO (Imbentin-N/060 and IGEPAL CO-720	NP-1-20-EO	Ultrasonic
2115	Yes	0.5	varius	varius	varius	varius	Ultrasonic
2129	Yes	0,4	OPEO: Trition X-100 (CAS 9002-93-1, Sigma 542334); NPEO: Igepal Co-630 (CAS 68412-54-4, Sigma T9284)	OPEO: average grade of ethoxylation 9-10, NPEO: average grade of ethoxylation 9-10	OPEO: Trition X-100 (CAS 9002-93-1, Sigma 542334); NPEO: Igepal Co-630 (CAS 68412-54-4, Sigma T9284)	OPEO: average grade of ethoxylation 9-10, NPEO: average grade of ethoxylation 9-10	Ultrasonic
2159	No	1	NPEO CAS NO:68412-54-4 / OPEO CAS NO:9002-93-1		NPEO CAS NO:68412-54-4 / OPEO CAS NO:9002-93-1	Ultrasonic	
2201	Yes	1.0	OPEOs (CAS No. :9002-93-1)	/	NPEOs (CAS No. 68412-54-4)	/	Ultrasonic
2213	Yes	0.5					Ultrasonic
2215	Yes	1.0	Octylphenolethoxylates (Triton X-100), (OPEOs) CAS no. 9002-93-1 Sigma-Aldrich (Product No. T9284)	OPEO(2~16)	Nonylphenolethoxylates (IGEPAL CO-630), CAS no. 68412-54-4 Aldrich (Product No. 542334)	NPEO(2~18)	Ultrasonic
2232	No	1	Octylphenolethoxylates (Triton X-100), (OPEOs) CAS no. 9002-93-1	Octylphenolethoxylates (OPEOs[2-16])	Nonylphenolethoxylates (IGEPAL CO-630), (NPEOs) CAS no. 68412-54-4	Nonylphenolethoxylates (NPEOs[2-18])	Ultrasonic
2247	Yes	1	please see coments		please see coments		Ultrasonic
2255	Yes	0.501	Triton X-100 and 9002-93-1	NA	IGEPAL CO-630 and 68412-54-4	NA	Ultrasonic
2265	No	0.5	Triton X-100, CAS-Nr. 9002-93-1	OPEO 5-16	IGEPAL CO-630, CAS-Nr. 68412-54-4	NPEO 4-16	Ultrasonic
2284	Yes	0.5	Tritonx-100(9002-93-1)	OPEOs	Octoxynoi9(68412-54-4)	NPEOs	Ultrasonic
2290	---						---
2293	---						---
2297	Yes	1	OPEO:TritonX-100, CAS NO.:9002-93-1	sum of OP2EO,...OP16EO	NPEO:Igepal CO-630 CAS NO.:68412-54-4	sum of NP2EO,...NP16EO	Ultrasonic
2300	Yes	0.5	Triton X-100. (CAS no. 9002-93-1)		Tergitol (CAS no.127087-87-0)		Ultrasonic
2301	Yes	1					Ultrasonic

Lab	ISO/IEC17025 accredited for reported component(s)	sample intake (in grams)	Sample #18510: material(s) used for calibration	Sample #18510: isomers observed	Sample #18511: material(s) used for calibration	Sample #18511: isomers observed	release/extract the analyte(s)
2310	Yes	1	Triton X-100 and CAS NO -9002-93-1	Octyl phenol Ethoxylates	IGEPAL CO-630 and CAS NO - 68412-54-4	Nonyl phenol Ethoxylates.	Ultrasonic
2311	Yes	1	Triton X-100, CAS No:9002-93-1	-	IGEPAL CO-630, CAS No:68412-54-4	-	Ultrasonic
2330	Yes	0.50	Tration X-100 Cas no. 9002-93-1	OPEO-10	POE (9-10) nonylphenol Cas no. 9016-45-9	NPEO-(9-10)	Ultrasonic
2347	Yes						Ultrasonic
2350	Yes	1.0	Octylphenol ethoxylate, 9002-93-1	NPEO (2-16)	Nonylphenol ethoxylate, 68412-54-4	OPEO (2-16)	Ultrasonic
2352	Yes	1	9002-93-1	OPEO	68412-54-4	NPEO	Ultrasonic
2357	Yes	1	OPEO WITH n=9-10£¬CAS no 9002-93-1	None	NPEO WITH n=9-10£¬CAS no 68412-54-4	None	Ultrasonic
2358	Yes	0.5	Nonylphenol(25154-52-3), 4-nonylphenol(104-40-5), Octylphenol(27193-28-8), 4-octylphenol(1806-26-4)	2,4-Di-tert-butylphenol(96-76-4), 3,5-Di-tert-butylphenol(1138-52-9)	Nonylphenol(25154-52-3), 4-nonylphenol(104-40-5), Octylphenol(27193-28-8), 4-octylphenol(1806-26-4)	2,4-Di-tert-butylphenol(96-76-4), 3,5-Di-tert-butylphenol(1138-52-9)	Ultrasonic
2363	No	1	CAS No.:9002-93-1	OPEO(9-10)	CAS NO.:68412-54-4	NPEO(9-10)	Ultrasonic
2365	Yes	1	POE (9-10) tert-octylphenol,CAS NO.:9002-93-1	OPEO(2~16)	POE (9-10) nonylphenol,CAS NO.:68412-54-4	NPEO(2~16)	Ultrasonic
2369	---						---
2370	Yes	0.5022 / 0.5041	OP9-10EO(Triton X-100) ; CAS No. 9002-93-1	OPEO2 up to OPEO16	NP9-10EO(IGEPAL CO-630); CAS No. 68412-54-4	OPEO2 up to OPEO16	Ultrasonic
2373	Yes		NA	NA	NA	NA	Ultrasonic
2375	No	1	Triton X-100 CAS No:9002-93-1		IGEPAL CO-630 CAS No:68412-54-4		Ultrasonic
2379	No	1	CAS No.: 9002-93-1 : Triton TM X-100-BioXtra : OPEO 9-10	CAS No.: 68412-54-4 ; IQEPAL C0- Average Mn617, NPEO9-10	CAS No.: 68412-54-4 ; IQEPAL C0- Average Mn617, NPEO9-10		Ultrasonic
2380	Yes	#18510 1.0029 #18511-1.0028	OPEO (9-10) Octylphenol Ethoxylate Tritan X-100 CAS No 9002-93-1	9-10	NPEO (9-10) Nonyllphenol Ethoxylate IGEPAL CAS No 68412-54-4	9-10	Ultrasonic
2382	No	1	OPEO(9-10) and 9002-93-1	/	NPEO(9-10) and 68412-54-4	/	Ultrasonic
2386	Yes	0.5	Triton X CAS 9002-93-1 Sigma Aldrich	POE(9) OP CAS 9002-93-1	IGEPAL CO-630 CAS 68412-54-4 Sigma Aldrich	POE(9-10) NP CAS 68412-54-4	Ultrasonic
2390	Yes	#18510 0.5006 #18511 1.0001	NPEO 9-10 Cas#68412-54-4,OPEO9-10 Tert-OP Cas#9002-93-1,4-n-NP Cas#104-40-5,4-tert-OP Cas#140-66-9		NPEO 9-10 Cas#68412-54-4,OPEO9-10 Tert-OP Cas#9002-93-1,4-n-NP Cas#104-40-5,4-tert-OP Cas#140-66-9		Ultrasonic
2426	---						---
2449	Yes	1.0012					Ultrasonic
2452	---						---
2482	Yes	0.5	Triton X-100 (OPEOs) CAS-No. 9002-93-1; IGEPAL CO-630 (NPEOs) CAS-No. 68412-54-4	Triton X-100 (OPEOs) CAS-No. 9002-93-1; IGEPAL CO-630 (NPEOs) CAS-No. 68412-54-4	Ultrasonic		
2489	Yes	0.5001	TRITON X-100 (9002-93-1)	OPEOs	IGEPAL CO-630 (68412-54-4)	NPEOs	Ultrasonic

Lab	ISO/IEC17025 accredited for reported component(s)	sample intake (in grams)	Sample #18510: material(s) used for calibration	Sample #18510: isomers observed	Sample #18511: material(s) used for calibration	Sample #18511: isomers observed	release/extract the analyte(s)
2492	Yes	0.5	Trition X-45 & IGEPAL CA-720		Imbentin-N/060 & NP-(3-20)EO Chiron		Ultrasonic
2495	---						---
2497	Yes	1	multiple standard with multiple CAS numbers	multiple standard with multiple CAS numbers	multiple standard with multiple CAS numbers	multiple standard with multiple CAS numbers	Ultrasonic
2508	Yes	0.7	Igepal CO-720, CA-520, CA-720, Ibentin-N/060		Igepal CO-720, CA-520, CA-720, Ibentin-N/060		Ultrasonic
2511	---						---
2523	No	2.0000	SIGMA IGEPAL® CA-630 (9002-93-1) & SIGMA TRITON X-114 (9036-19-5)	SIGMA IGEPAL® CA-630 (9002-93-1) & SIGMA TRITON X-114 (9036-19-5)	Ultrasonic		
2532	Yes	0.5	Triton X-100 (CAS no - 9002-93-1)	Sum of OPEO 3 to 16	IGPAL CO - 630 (CAS No - 68412-54-4)	Sum of NPEO 3 to 18	Ultrasonic
2549	Yes	0.502	Commercial Name-TRITON X-100, CAS No. - 9002-93-1	OPEO	Commercial Name - IGEPAL-Co-630, CAS No. - 68412-54-4	NPEO	Ultrasonic
2561	Yes	1					Ultrasonic
2567	Yes	0.50	OPEOs: TritonX-100. CAS NO: 9002-93-1	N/A	NPEO: IGEPAL CO-630. CAS No: 68412-54-4	N/A	Ultrasonic
2572	---						---
2573	Yes	1	SIGMA Triton® X-100,	NO	SIGMA IGEPAL® CO-630	NO	Ultrasonic
2590	Yes	1	Triton X-100 from Sigma Aldrich (CAS 9002-93-1)	OPEO 2-16	IGEPAL CO-630 (CAS 68412-54-4)	NPEO 2-18	Ultrasonic
2591	No	1.0	Answer in comments		Answer in comments		Ultrasonic
2592	No	1.5	Ottilfenolo etossilato (CAS 9002-93-1)	Ottilfenolo etossilato (CAS 9002-93-1)	Nonilfenolo etossilato (CAS 9016-45-9, CAS 68412-54-4)	Nonilfenolo etossilato (CAS 9016-45-9, CAS 68412-54-4)	Ultrasonic
2605	Yes	0.700	9002-93-1		9016-45-9		Ultrasonic
2629	---						---
2638	No	0.8	4-tert octyl phenol CAS# 140-66-9, POE (12-13) tert Octyl phenol CAS # 9002-93-1	PEO(9-10) tert octyl phenol CAS# 9002-93-1	Nonyl phenol CAS# 25154-52-3, POE (9-10)nonyl phenol CAS# 9016-45-9, POE (4) nonyl phenol	nil	Ultrasonic
2644	No	1	triton x100	opeo 3-17	igepal co 630	npeo 3-18	Ultrasonic
2668	Yes	0.5	OPEO -Triton X-100 CAS No- 9002-93-1	2-16 (isomer 2 is having low response)	NPEO - IGEPAL CO-630 CAS no-68412-54-4	2-18 (isomer 2 is having low response)	Ultrasonic
2713	Yes	1.0027 / 1.0041	Triton X-100 (9002-93-1)	4-(1,1,3,3-Tetramethylbutyl)phenyl-polyethylene glycol	Igepal CO-630 (68412-54-4)	Polyoxyethylene (9)nonylphenylether, branched	Ultrasonic
2770	Yes	0.5	CAS no 127087-87-0,9002-93-1,140-66-9,25154-52-3		CAS no 127087-87-0,9002-93-1,140-66-9,25154-52-3	Ultrasonic	
2804	No	0.5	IGEPAL CO-630 CAS No 6841254-4 Triton X-100 CAS No 9002-93-1	NPEO C2-C18 OPEO C2-C16	IGEPAL CO-630 CAS No 6841254-4 Triton X-100 CAS No 9002-93-1	NPEO C2-C18 OPEO C2-C16	Ultrasonic
2812	No	1					Ultrasonic
2820	No	1	TRITON X-100 (CAS 9002-83-1)	OP3-20 (CAS 68412-54-4)	IGEPAL CO-630	NP3-20	Ultrasonic

Lab	ISO/IEC17025 accredited for reported component(s)	sample intake (in grams)	Sample #18510: material(s) used for calibration	Sample #18510: isomers observed	Sample #18511: material(s) used for calibration	Sample #18511: isomers observed	release/extract the analyte(s)
3100	Yes	1.008 / 1.012	Triton X-100(OPEOs) CAS no. 9002-93-1	not found	IGEPAL(CO-630), (NPEOs) CAS no. 68412-54-4	not found	Ultrasonic
3116	Yes	0.5	OPEOs (Sigma-Aldrich, 9002-93-1)		NPEOs (Aldrich, 68412-54-4)		Ultrasonic
3118	Yes	0.5					Ultrasonic
3146	Yes	1	Triton-X -100(B) cas: 9002-93-1		IGEPAL CO630 cas: 68412-54-4		Ultrasonic
3150	No	1	Tritin X-100 (9002-93-1) + IGEPAL CO-630 (68412-54-4)	OPEO2 - OPEO15 + NPEO2 - NPEO15	Tritin X-100 (9002-93-1) + IGEPAL CO-630 (68412-54-4)	OPEO2 - OPEO15 + NPEO2 - NPEO15	Ultrasonic
3151	Yes	0.25 / 0.5	Nonylphenol (techn.) (25154-52-3); Nonylphenol (84852-15-3); 4-n-Nonylphenol (104-40-5); Nonylphenol		Nonylphenol (techn.) (25154-52-3); Nonylphenol (84852-15-3); 4-n-Nonylphenol (104-40-5); Nonylphenol		Ultrasonic
3154	Yes						Ultrasonic
3163	---						---
3172	Yes	0.75	Triton X-100 - CAS: 9002-93-1		IGEPAL CO630 - CAS: 68412-54-4		Ultrasonic
3176	Yes	0.5	Triton X-100 (CAS no:9002-93-1)	Octylphenol Ethoxylates (OPEO)	IGEPAL (CAS no:68412-54-4)	Nonylphenol Ethoxylates (NPEO)	Ultrasonic
3190	Yes	1	Triton X-100 CAS No.: 9002-93-1	/	IGEPAL CO-630 CAS No.: 68412-54-4	/	Ultrasonic
3197	Yes	1	Triton X-100/CAS No:9002-93-1		IGEPAL CO-630/CAS No:68412-54-4		Ultrasonic
3209	Yes		OPEO: Sigma-Aldrich Triton X-100 CAS: 9002-93-1		NPEO: Sigma-Aldrich IGEPAL CO-630 CAS: 68412-54-4		Ultrasonic
3210	No	1	NPEO : IGEPAL-CO 630 (CAS : 68412-54-4) OPEO : triton X-100 (CAS : 9002-93-1)	NPEO : IGEPAL-CO 630 (CAS : 68412-54-4) OPEO : triton X-100 (CAS : 9002-93-1)	Ultrasonic		
3214	Yes	1	Triton X 100, CAS No. 9002-93-1	NO	IGEPAL CO-630, CAS No. 68412-54-4	NO	Ultrasonic
3218	Yes	1	CAS No.:9002-93-1 OPEO Triton X100	Octylphenolethoxylate, branched(n=10)	CAS No.:68412-54-4 NPEO (IGEPAL CO-630)	Nonylphenolethoxylate,branched(n=9)	Ultrasonic
3220	Yes	0.6	Triton(OPEO)CAS No.9002-93-1,IGEPAL(NPEO)CAS No.68412-54-4,OP CAS No.C15712000,NP CAS No.84852-15-3	-	Triton(OPEO)CAS No.9002-93-1,IGEPAL(NPEO)CAS No.68412-54-4,OP CAS No.C15712000,NP CAS No.84852-15-3	-	Ultrasonic
3222	Yes	1.00	OPEO CAS n.9002-93-1 / 9036-19-5 - NPEO CAS n.68412-54-4/127087-87-0	NPEO CAS n.68412-54-4/127087-87-0			Ultrasonic
3232	Yes	1	Triton X- 100 CAS No. 9002-93-1	OPEO	IGEPAL CO-630 CAS No. 68412-54-4	NPEO	Ultrasonic
3233	No	1	OPEO : TRITON X-100 [9002-93-1]	/	NPEO : IGEPAL CO-630 [68412-54-4]	/	Ultrasonic
3237	Yes	0.5	Triton X-100(OPEO) CAS NO: 9002-93-1		Igepal CO-630(NPEO) CAS NO: 68412-54-4		Ultrasonic
3246	Yes	0.5	TRITON X-100 CAS No.: 9002-93-1		IGEPAL CO-630 CAS No.: 68412-54-4		Ultrasonic
3248	No	1	CAS: 9002-93-1		CAS: 68412-54-4		Ultrasonic

APPENDIX 4

Number of participants per country

2 labs in	BANGLADESH
1 lab in	BRAZIL
2 labs in	CAMBODIA
3 labs in	FRANCE
10 labs in	GERMANY
1 lab in	GUATEMALA
5 labs in	HONG KONG
11 labs in	INDIA
3 labs in	INDONESIA
9 labs in	ITALY
1 lab in	KOREA
1 lab in	MAURITIUS
1 lab in	MOROCCO
19 labs in	P.R. of CHINA
4 labs in	PAKISTAN
1 lab in	SINGAPORE
1 lab in	SPAIN
3 labs in	TAIWAN R.O.C.
1 lab in	THAILAND
1 lab in	THE NETHERLANDS
2 labs in	TUNISIA
7 labs in	TURKEY
1 lab in	UNITED KINGDOM
6 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

Literature:

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3 Impacts of Environmental Standards and requirements in EU Countries. Aug 99
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12 M. Thompson and R. Wood. J. AOAC Int. 76. 926. (1993)
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16 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, T*echnometrics*, 25(2), 165-172, (1983)