Results of Proficiency Test
APEO in textile
March 2016

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: dr. R.G. Visser &
Corrector: ing. C.M. Nijssen-Wester & ing. R.J. Starink
Report: iis16A04

May 2016

CONTENTS

Appendices:

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".
On request of several participants, the Institute for Interlaboratory Studies decided to organise an interlaboratory study for the determination of AP and APEO content in textile in the 2015/2016 PT program.
In this first interlaboratory study organized in March 2016, 107 laboratories from 23 different countries did register for participation (See appendix 3). In this report, the results of the 2016 proficiency test are presented and discussed. This report is also electronically available through the iis web site www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies in Spijkenisse was the organiser of the proficiency test. It was decided to use two different samples which were positive on OPEO or NPEO. Sample analyses for fit-for-use and homogeneity testing were subcontracted to an accredited laboratory. 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 organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol is electronically available through the iis web site 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 batches of textile were prepared by a third party. The first bulk sample, a cotton fabric artificially fortified with OPEO by means of Triton X-100 (CAS 9002-93-1), was cut into pieces. Out of this batch, after mixing well, 118 subsamples of approx. 3 grams each were prepared and labelled #16530.
The homogeneity of 8 stratified randomly selected samples was checked by determination of OPEO by an accredited third party laboratory. The determination is performed in accordance with an in-house test method for OPEO. See the following table for the test results.

	OPEO in mg/kg
Sample #16530-1	68.37
Sample #16530-2	69.66
Sample #16530-3	70.35
Sample #16530-4	71.06
Sample #16530-5	70.57
Sample #16530-6	71.70
Sample #16530-7	70.08
Sample #16530-8	70.33

Table 1: homogeneity test results of subsample #16530

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:

	OPEO in mg/kg
r (samples #16530)	2.76
Reference	Horwitz (n=4)
0.3 x R (reference)	4.98

Table 2: evaluation of the repeatability of subsamples #16530.

The second bulk sample, a cotton fabric artificially fortified with NPEO by means of Triton X-57 (CAS 127087-87-0), was cut into pieces. Out of this batch, after mixing well, 118 subsamples of approx. 3 grams each were prepared and labelled #16531.
The homogeneity of 8 stratified randomly selected samples was checked by determination of NPEO by an accredited third party laboratory. The determination is performed in accordance with an in-house test method for OPEO. See the following table for the test results.

	NPEO in mg/kg
Sample #16531-1	133.9
Sample #16531-2	147.0
Sample #16531-3	127.8
Sample #16531-4	147.5
Sample #16531-5	124.1
Sample #16531-6	130.5
Sample #16531-7	123.3
Sample #16531-8	144.3

Table 3: homogeneity test results of subsample #16531

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 (samples #16531)	28.3
Reference	Horwitz (n=4)
0.3 x R (reference)	27.3

Table 4: evaluation of the repeatability of subsamples #16531.

The repeatabilities of OPEO and NPEO were in agreement with 0.3 times the target requirements. Therefore, homogeneity of the subsamples was assumed.

To each participating laboratory one sample of approx. 3 grams, labelled #16530 and one sample of approx. 3 grams, labelled #16531 were sent on March 9, 2016.

2.5 ANALYSES

The participants were asked to determine the concentrations of OP, NP,OPEO, NPEO and total AP + APEO on both samples #16530 and #16531 applying the analysis procedure that is routinely used in the laboratory.
To get comparable results a detailed report form, on which the units were prescribed as well as the reference test methods and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. A form to confirm receipt of the samples and a letter of instructions were added to the samples.

3 RESULTS

During five weeks after sample despatch, the results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per 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. 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

Statistical calculations were performed as described in the report ’iis Interlaboratory Studies: Protocol for the Organization, Statistics and Evaluation’ of 2014 (iis-protocol, version 3.3).
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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.

According to ISO 5725 the original test results per determination were submitted to Dixon’s and/or 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 visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. The Kernel Density Graph is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the spread of 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 eleven participants reported the test results after the final reporting date and two participants did not report any results at all. In total 105 of the 107 participants reported 412 numerical results. Observed in all reported results were 13 statistical outlying results, which is 3.2%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.
ISO 18254, used by many participants, is the official test method for the determination of APEO in textiles. However, a number of participating laboratories used ISO18218-1, a test method developed for the determination of APEO in leather. The methods are almost identical, except for the ratio grams of sample:extraction liquid, which is 1:20 for ISO 18254 and 1:10 for ISO 18218-1. Regretfully ISO 18254 does not mention reproducibilities for OP, NP, OPEO or NPEO, but only for the sum AP + APEO content at a level of 954 mg/kg (R=26%). ISO 18218-1 and ISO 18218-2 do not have any precision mentioned. Therefore, the target requirements in this study were estimated using the Horwitz equation (for n=4).

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. All participants agreed about the absence of Octylphenol and Nonylphenol, except one participant that may have mixed up test results or misidentified components.

sample #16530

OPEO: The determination of this component was not problematic. Three statistical outliers were detected. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the target reproducibility estimated from the Horwitz equation for 4 components.

sum AP+APEO: The determination of this parameter was not problematic. Two statistical outliers were detected and the calculated reproducibility after rejection of the statistical outliers is in full agreement with the target reproducibility estimated from the Horwitz equation for 4 components.

sample #16531

NPEO: The determination of this component was problematic. Five statistical outliers were detected and the calculated reproducibility after rejection of the statistical outliers is not in agreement with the target reproducibility estimated from the Horwitz equation for 4 components.

sum AP+ APEO: The determination of this parameter was problematic. Two statistical outliers were detected and the calculated reproducibility after rejection of the statistical outliers is not in agreement with the target reproducibility estimated from the Horwitz equation for 4 components.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the estimated target reproducibilities (see 4.1) and the reproducibilities as found for the group of participating laboratories.
The number of significant results, the average results, the calculated reproducibilities (standard deviation*2.8) and the target reproducibilities are compared in the next tables:

	unit	*n*	*Average*	*2.8 * sd*	*R (target)*
OPEO	mg/kg	102	65.6	29.6	31.3
sum AP + APEO	mg/kg	71	66.3	29.6	31.6

Table 5: reproducibilities on textile sample #16530

	unit	*n*	*Average*	*2.8 * sd*	*R (target)*
NPEO	mg/kg	100	162	124	68
sum AP + APEO	mg/kg	69	156	109	65

Table 6: reproducibilities on textile sample #16531

Without further statistical calculations, it can be concluded that the total group of participating laboratories may have no difficulties with the analysis of OPEO, but that the analysis of NPEO appears to be more difficult. See also the discussion in paragraphs 4.1 and 6.

5 DISCUSSION

It is not clear why the determination of NPEO was more difficult than the determination of OPEO. The details as reported by the participating laboratories do not give an answer to this. The expected influence of the various calibrants used by the participating laboratories is not detected. Also deviations from the test method (e.g. extraction at 40°C as done by several laboratories) do not show any correlation with the reported test results.

In this proficiency test for the determination of phenols in textile the majority of the participants was able to detect and quantify correctly OPEO in sample #16530 and NPEO in sample #16531.

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 the majority of the reporting laboratories would accept the sample #16530 and reject sample #16531 for containing too much APEO. For sample #16530 only two laboratories would conclude differently as they detected more than 100 mg/kg OPEO, while for sample #16531 seven laboratories would conclude differently as they detected less than 100 mg/kg NPEO.

	OEKO-TEX	*EU 2016/26*
NP	---	100 mg/kg
sum OP + NP	10 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

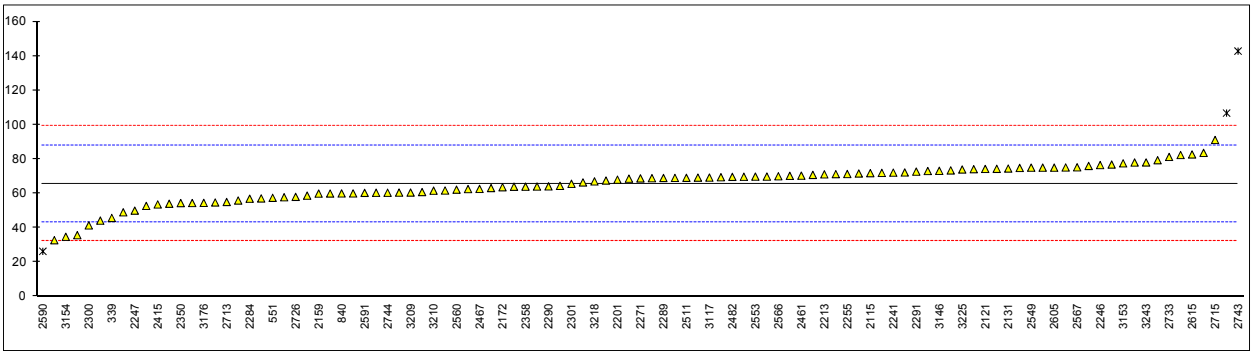
APPENDIX 1

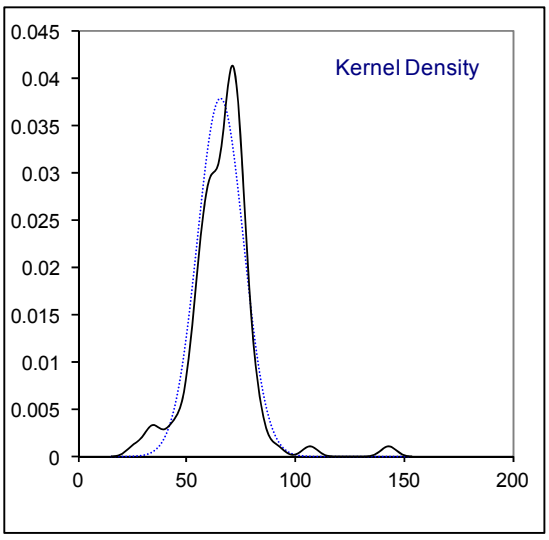
Determination of Octylphenol Ethoxylates (OPEO) on sample #16530; results in mg/kg

lab	method	value	mark	z(targ)	remarks
213	In house	63.9		-0.16	
230	ISO18218-1	61.7		-0.35	
339	In house	45.731		-1.78	
551	In house	57.4451		-0.73	
623	ISO/FDIS18254	60.38		-0.47	
840	ISO18218-1	60		-0.50	
2108	In house	106.75	R(0.05)	3.67	
2115	ISO18218-1	71.81		0.55	
2121	In house	74.24		0.77	
2129	ISO18218-1	67.5		0.17	
2131	In house	74.5		0.79	
2135	ISO18254Draft	73.4		0.69	
2139	ISO18218-1	52.73		-1.15	
2159	In house	59.90		-0.51	
2172	ISO/DIS18254	63.6165		-0.18	
2201	In house	68		0.21	
2213	ISO18218-1	71.1		0.49	
2215	In house	60.5		-0.46	
2241	In house	72.1		0.58	
2246	In house	76.5		0.97	
2247	In house	50.0		-1.40	
2255	In house	71.3		0.51	
2271	ISO/FDIS18254	68.74		0.28	
2272	ISO18254	76		0.93	
2284	ISO18218-1	56.92		-0.78	
2285	ISO/FDIS18254	79.3315		1.22	
2289	ISO18218-1	69		0.30	
2290	ISO18218-1	64.1		-0.14	
2291	ISO18218-1	72.7		0.63	
2293	ISO/DIS18254Draft	74.125		0.76	
2295	ISO18254	60		-0.50	
2297	ISO18218-1	62.53		-0.28	
2300	ISO18218-2	41.4		-2.17	
2301		65.5987		0.00	
2310	In house	75.11		0.85	
2311	In house	70.244		0.41	
2320	ISO18218-1	57.7751		-0.70	
2330	In house	83.6		1.61	
2350	ISO18218-1	54.4		-1.00	
2358	In house	63.9458		-0.15	
2370	In house	54.74		-0.97	
2375	In house	64.4034		-0.11	
2380	In house	55.88		-0.87	
2386	ISO18218-1	68.87		0.29	
2389	In house	64		-0.15	
2390	ISO18254	59.9641		-0.51	
2415	In house	53.6		-1.08	
2461	GB/T23322	70.3376		0.42	
2467	In house	62.63		-0.27	
2482	ISO/DIS18254	69.63		0.36	
2486	ISO18254	70.789		0.46	
2489	In house	75		0.84	
2492	In house	68.6		0.26	
2495	ISO/DIS18254Draft	82.345		1.49	
2497	In house	74.83		0.82	
2508	ISO18218-1	32.79		-2.94	
2511	ISO18218-1	69.09		0.31	
2532	ISO18254Draft	78		1.10	
2536	In house	76.82		1.00	
2549	ISO/DIS18254Mod	74.99		0.84	
2553	In house	69.78		0.37	
2560	ISO18218-1	62.2		-0.31	
2566	In house	70		0.39	
2567	In house	75.2		0.85	
2590	ISO/DIS18254	26.3	C,R(0.05)	-3.52	first reported 30.1025
2591	In house	60.32		-0.48	
2592	ISO18218-2	35.70		-2.68	
2605	ISO18218-1	75.0		0.84	
2614	In house	72.2		0.59	
2615	ISO18254	82.6902		1.52	
2618	ISO18218-1	63.12		-0.23	
2644		-----		-----	
2649	In house	57.09		-0.76	
2665	In house	44.17		-1.92	
2668	ISO/DIS18254	74.32		0.78	
2671	In house	71.23		0.50	

2713	In house	55.01		-0.95	
2715	GB/T23322	91.103	C	2.28	first reported 9.1103
2723		-----		-----	
2726	ISO/FDIS18254	57.998		-0.68	
2733	GB/T23322	81.2320		1.39	
2737	ISO18218-1	69.03		0.30	
2743	In house	142.8	C,R(0.01)	6.90	first reported <1
2744	ISO/DIS18254	60.4		-0.47	
3100	In house	71.9342		0.56	
3117	OEKO-TEX	69.2125		0.32	
3118	In house	73.04		0.66	
3146	ISO18254	73.153		0.67	
3149	ISO18218-2	60.8		-0.43	
3151	In house	69.4400		0.34	
3153	ISO18218-1	77.5		1.06	
3154	In house	34.75		-2.76	
3172	ISO18218-1	58.7		-0.62	
3176	In house	54.5		-1.00	
3185	In house	69.2		0.32	
3190	ISO/DIS18254	71.6		0.53	
3197	ISO/DIS18254	69.7		0.36	
3200	ISO18218-1	69.8		0.37	
3209	ISO18218-1	60.5025		-0.46	
3210	ISO/DIS18254	61.6150		-0.36	
3218	ISO/DIS18254	67.0		0.12	
3220	CEN N 1057	49		-1.49	
3225	In house	73.939		0.74	
3233	In house	54.4111		-1.00	
3237	In house	66.40		0.07	
3243	ISO18218-1	78.0294		1.11	
3246	ISO18218-1	54.00		-1.04	

normality	suspect
n	102
outliers	3
mean (n)	65.639
st.dev. (n)	10.5555
R(calc.)	29.555
R(Horwitz (n=4))	31.330



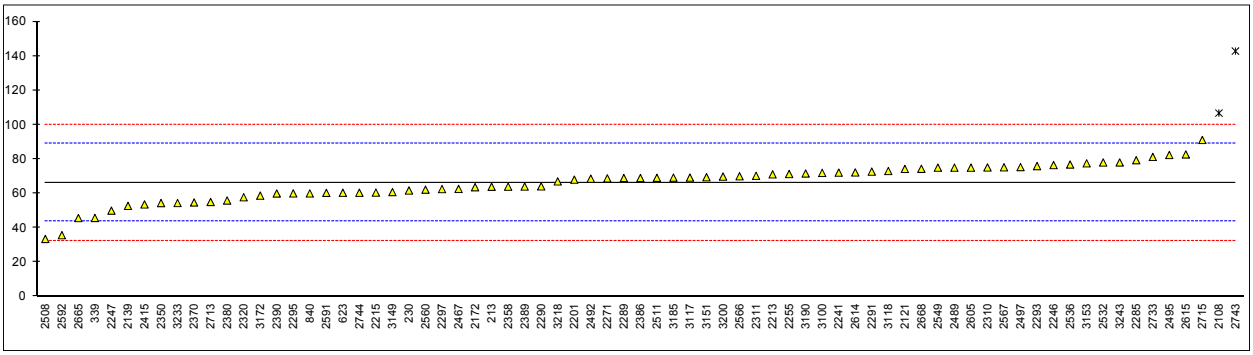


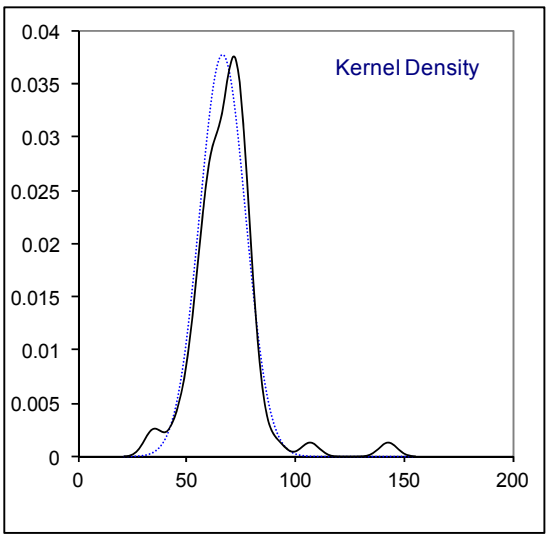
Determination of sum of OP, NP, OPEO and NPEO on sample #16530; results in mg/kg

lab	method	value	mark	z(targ)	remarks
213	In house	63.9		-0.22	
230	ISO18218-1	61.7		-0.41	
339	In house	45.731		-1.83	
551		-----		-----	
623	ISO/FDIS18254	60.38		-0.53	
840	ISO18218-1	60		-0.56	
2108	In house	106.75	R(0.05)	3.58	
2115		-----		-----	
2121	In house	74.24		0.70	
2129		-----		-----	
2131		-----		-----	
2135		-----		-----	
2139	ISO18218-1	52.73		-1.21	
2159		-----		-----	
2172	ISO/DIS18254	63.6165		-0.24	
2201	In house	68		0.15	
2213	ISO18218-1	71.1		0.42	
2215	In house	60.5		-0.52	
2241	In house	72.1		0.51	
2246	In house	76.5		0.90	
2247	In house	50.0		-1.45	
2255	In house	71.3		0.44	
2271	ISO/FDIS18254	68.74		0.21	
2272		-----		-----	
2284		-----		-----	
2285	ISO/FDIS18254	79.3315		1.15	
2289	ISO18218-1	69		0.23	
2290	ISO18218-1	64.1		-0.20	
2291	ISO18218-1	72.7		0.56	
2293	ISO/DIS18254Draft	75.909		0.85	
2295	ISO18254	60		-0.56	
2297	ISO18218-1	62.53		-0.34	
2300		-----		-----	
2301		-----		-----	
2310	In house	75.11		0.78	
2311	In house	70.244		0.35	
2320	ISO18218-1	57.7751		-0.76	
2330		-----		-----	
2350	ISO18218-1	54.4		-1.06	
2358	In house	63.9458		-0.21	
2370	In house	54.74		-1.03	
2375		-----		-----	
2380	In house	55.88		-0.93	
2386	ISO18218-1	69.00	C	0.23	first reported 293.70
2389	In house	64		-0.21	
2390	ISO18254	59.9641		-0.57	
2415	In house	53.6		-1.13	
2461		-----		-----	
2467	In house	62.63		-0.33	
2482		-----		-----	
2486		-----		-----	
2489	In house	75		0.77	
2492	In house	68.6		0.20	
2495	ISO/DIS18254Draft	82.345		1.42	
2497	In house	75.34		0.80	
2508	ISO18218-1	33.50		-2.91	
2511	ISO18218-1	69.09		0.24	
2532	ISO18254Draft	78		1.03	
2536	In house	76.82		0.93	
2549	ISO/DIS18254Mod	74.99		0.77	
2553		-----		-----	
2560	ISO18218-1	62.2		-0.37	
2566	In house	70		0.32	
2567	In house	75.2		0.78	
2590		-----		-----	
2591	In house	60.32		-0.53	
2592	ISO18218-2	35.70		-2.72	
2605	ISO18218-1	75.0		0.77	
2614	In house	72.2		0.52	
2615	ISO18254	82.6902		1.45	
2618		-----		-----	
2644		-----		-----	
2649		-----		-----	
2665	In house	45.65		-1.83	
2668	ISO/DIS18254	74.32		0.71	
2671		-----		-----	

2713	In house	55.01		-1.00	
2715	GB/T23322	91.103	C	2.19	first reported 9.1103
2723		-----		-----	
2726		-----		-----	
2733	GB/T23322	81.2320		1.32	
2737		-----		-----	
2743	In house	142.8	R(0.01)	6.77	
2744	ISO/DIS18254	60.4		-0.53	
3100	In house	71.9342		0.49	
3117	OEKO-TEX	69.2125		0.25	
3118	In house	73.04		0.59	
3146		-----		-----	
3149	ISO18218-2	60.8		-0.49	
3151	In house	69.4400		0.27	
3153	ISO18218-1	77.5		0.99	
3154		-----		-----	
3172	ISO18218-1	58.7	C	-0.68	first reported <10
3176		-----		-----	
3185	In house	69.2		0.25	
3190	ISO/DIS18254	71.6		0.47	
3197		-----		-----	
3200	ISO18218-1	69.8		0.31	
3209		-----		-----	
3210		-----		-----	
3218	ISO/DIS18254	67.0		0.06	
3220	In house	ND		-----	false negative test result?
3225	In house	NA		-----	
3233	In house	54.4111		-1.06	
3237		-----		-----	
3243	ISO18218-1	78.0294		1.03	
3246		-----		-----	

normality	suspect
n	71
outliers	2
mean (n)	66.349
st.dev. (n)	10.5835
R(calc.)	29.634
R(Horwitz (n=4))	31.618

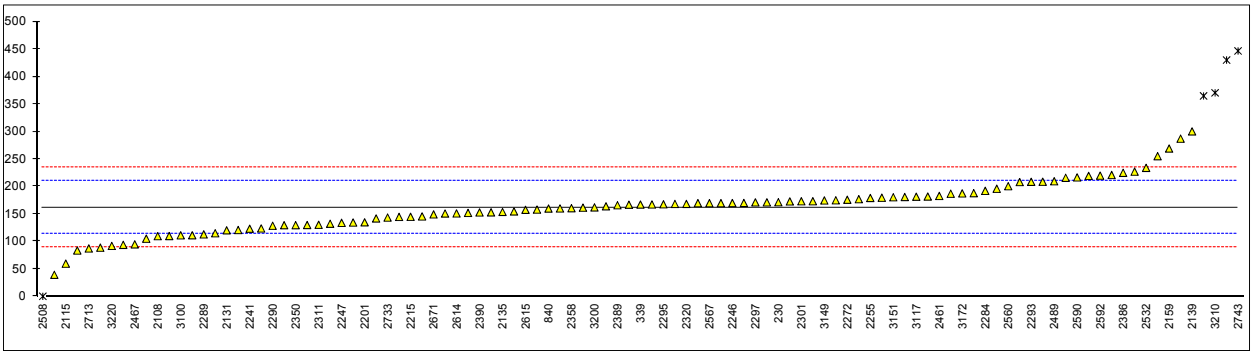


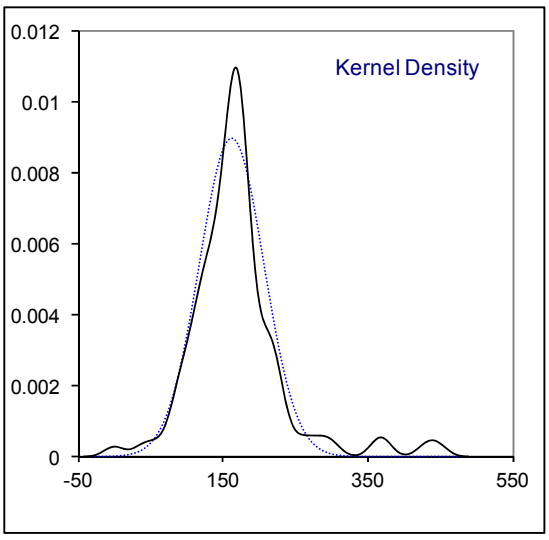


Determination of Nonylphenol Ethoxylates (NPEO) on sample #16531; results in mg/kg

lab	method	value	mark	z(targ)	remarks
213	In house	142.0		-0.84	
230	ISO18218-1	171.5		0.39	
339	In house	167.303		0.21	
551	In house	181.8419		0.82	
623	ISO/FDIS18254	134.64		-1.14	
840	ISO18218-1	160		-0.09	
2108	In house	109.77		-2.17	
2115	ISO18218-1	59.79		-4.24	
2121	In house	164.11		0.08	
2129	ISO18218-1	151		-0.46	
2131	In house	120.4		-1.73	
2135	ISO18254Draft	153.9		-0.34	
2139	ISO18218-1	300.33		5.73	
2159	In house	269.2	C	4.44	first reported 321.30
2172	ISO/DIS18254	172.925		0.45	
2201	In house	135		-1.13	
2213	ISO18218-1	170.2		0.33	
2215	In house	145		-0.71	
2241	In house	123.3		-1.61	
2246	In house	170		0.33	
2247	In house	134.0		-1.17	
2255	In house	179.2		0.71	
2271	ISO/FDIS18254	186.95		1.03	
2272	ISO18254	176		0.57	
2284	ISO18218-1	192.17		1.24	
2285	ISO/FDIS18254	169.756		0.32	
2289	ISO18218-1	113		-2.04	
2290	ISO18218-1	128.7	C	-1.39	first reported 257.3
2291	ISO18218-1	120.9		-1.71	
2293	ISO/DIS18254Draft	208.625	C	1.93	first reported 240.875
2295	ISO18254	167.7	C	0.23	first reported 127
2297	ISO18218-1	171.2		0.38	
2300	ISO18218-2	144.74		-0.72	
2301	In house	173.22369		0.46	
2310	In house	153.1		-0.37	
2311	In house	130.446		-1.31	
2320	ISO18218-1	168.4545		0.26	
2330	In house	160.2		-0.08	
2350	ISO18218-1	129.8		-1.34	
2358	In house	160.6428		-0.06	
2370	In house	167		0.20	
2375	In house	171.3		0.38	
2380	In house	145.7		-0.68	
2386	ISO18218-1	225	C	2.61	first reported this test result for sample #16530
2389	In house	166		0.16	
2390	ISO18254	152.945		-0.38	
2415	In house	196	C	1.40	first reported 249.35
2461	GB/T23322	182.9291		0.86	
2467	In house	94.86		-2.79	
2482	ISO/DIS18254	430.05	R(0.01)	11.11	
2486	ISO18254	169.8		0.32	
2489	In house	209.6	C	1.97	first reported 320
2492	In house	94.0		-2.82	
2495	ISO/DIS18254Draft	88.880		-3.04	
2497	In house	123.74		-1.59	
2508	ISO18218-1	0.37	R(0.05)	-6.71	
2511	ISO18218-1	175.00		0.53	
2532	ISO18254Draft	234		2.98	
2536	In house	179.71		0.73	
2549	ISO/DIS18254Mod	220.99		2.44	
2553	In house	154.78		-0.31	
2560	ISO18218-1	200.7		1.60	
2566	In house	152		-0.42	
2567	In house	169.77		0.32	
2590	ISO/DIS18254	216.6013		2.26	
2591	In house	83.68		-3.25	
2592	ISO18218-2	219.8		2.39	
2605	ISO18218-1	110.0		-2.16	
2614	In house	151.1		-0.46	
2615	ISO18254	157.6646		-0.19	
2618	ISO18218-1	208.74		1.93	
2644		-----		-----	
2649	In house	167.39		0.22	
2665	In house	132.4		-1.23	
2668	ISO/DIS18254	219.32		2.37	
2671	In house	149.35		-0.53	

2713	In house	87.40		-3.10	
2715	GB/T23322	227.1242		2.69	
2723		-----		-----	
2726	ISO/FDIS18254	287.121		5.18	
2733	GB/T23322	143.6513		-0.77	
2737	ISO18218-1	215.95		2.23	
2743	In house	446.6	C,R(0.01)	11.79	first reported <1
2744	ISO/DIS18254	161.6		-0.02	
3100	In house	111.3488		-2.11	
3117	OEKO-TEX	181.5432		0.80	
3118	In house	173.33		0.46	
3146	ISO18254	111.376		-2.10	
3149	ISO18218-2	174.7		0.52	
3151	In house	180.5536		0.76	
3153	ISO18218-1	158.1		-0.17	
3154	In house	255.25		3.86	
3172	ISO18218-1	187.6		1.06	
3176	In house	129.7		-1.34	
3185	In house	115.1		-1.95	
3190	ISO/DIS18254	130.0		-1.33	
3197	ISO/DIS18254	177.0		0.62	
3200	ISO18218-1	162.1		0.00	
3209	ISO18218-1	168.3324		0.26	
3210	ISO/DIS18254	370.4215	R(0.01)	8.63	
3218	ISO/DIS18254	105.2		-2.36	
3220	CEN N 1057	92	C	-2.91	first reported 76
3225	In house	208.153		1.91	
3233	In house	39.4851	C	-5.08	first reported 37.1899
3237	In house	364.80	R(0.01)	8.40	
3243	ISO18218-1	181.0204		0.78	
3246	ISO18218-1	188.00		1.07	
					IGEPAL CO-630 used as calibrant as per ISO18254
	normality	suspect			suspect
	n	100			35
	outliers	5			3
	mean (n)	162.145			166.392
	st.dev. (n)	44.3978			48.3240
	R(calc.)	124.314			135.307
	R(Horwitz (n=4))	67.544			69.044



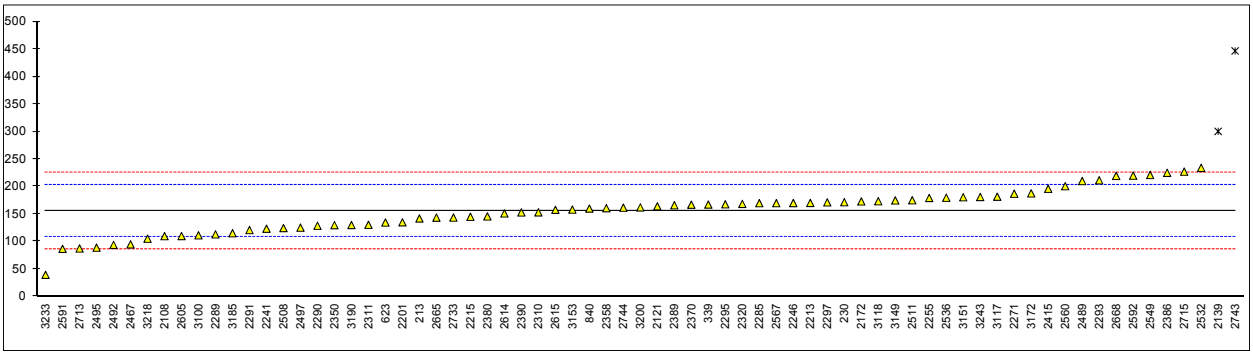


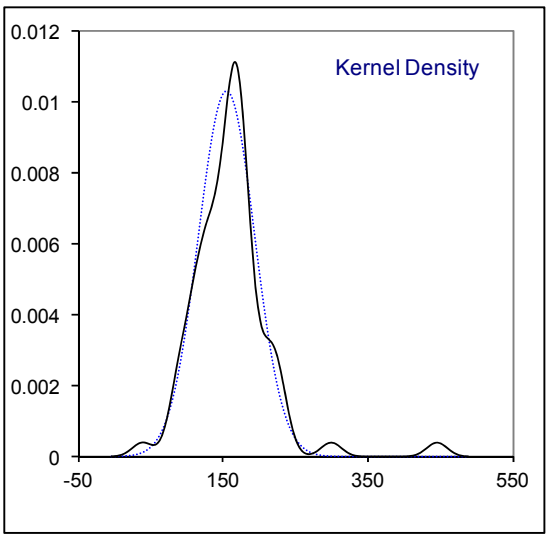
Determination of sum of OP, NP, OPEO and NPEO on sample #16531; results in mg/kg

lab	method	value	mark	z(targ)	remarks
213	In house	142.0		-0.58	
230	ISO18218-1	171.5		0.68	
339	In house	167.303		0.50	
551		-----		-----	
623	ISO/FDIS18254	134.64		-0.90	
840	ISO18218-1	160		0.19	
2108	In house	109.77		-1.97	
2115		-----		-----	
2121	In house	164.11		0.37	
2129		-----		-----	
2131		-----		-----	
2135		-----		-----	
2139	ISO18218-1	300.33	R(0.05)	6.21	
2159		-----		-----	
2172	ISO/DIS18254	172.925		0.74	
2201	In house	135		-0.88	
2213	ISO18218-1	170.2		0.63	
2215	In house	145		-0.45	
2241	In house	123.3		-1.39	
2246	In house	170		0.62	
2247		-----		-----	
2255	In house	179.2		1.01	
2271	ISO/FDIS18254	186.95		1.35	
2272		-----		-----	
2284		-----		-----	
2285	ISO/FDIS18254	169.756		0.61	
2289	ISO18218-1	113		-1.83	
2290	ISO18218-1	128.7	C	-1.15	first reported 257.3
2291	ISO18218-1	120.9		-1.49	
2293	ISO/DIS18254Draft	211.4	C	2.40	first reported 243.650
2295	ISO18254	167.7	C	0.52	first reported 127
2297	ISO18218-1	171.2		0.67	
2300		-----		-----	
2301		-----		-----	
2310	In house	153.1		-0.11	
2311	In house	130.446		-1.08	
2320	ISO18218-1	168.4545		0.55	
2330		-----		-----	
2350	ISO18218-1	129.8		-1.11	
2358	In house	160.6428		0.22	
2370	In house	167		0.49	
2375		-----		-----	
2380	In house	145.7		-0.42	
2386	ISO18218-1	225	C	2.98	first reported for sample #16530
2389		166		0.45	
2390	ISO18254	152.945		-0.11	
2415	In house	196	C	1.74	first reported 249.35
2461		-----		-----	
2467	In house	94.86		-2.61	
2482		-----		-----	
2486		-----		-----	
2489	In house	210	C	2.34	first reported 320
2492	In house	94.0		-2.64	
2495	ISO/DIS18254Draft	88.880		-2.86	
2497	In house	125.27		-1.30	
2508	ISO18218-1	124.53		-1.33	
2511	ISO18218-1	175.00		0.83	
2532	ISO18254Draft	234	C	3.37	first reported n.d.
2536	In house	179.71		1.04	
2549	ISO/DIS18254Mod	220.99		2.81	
2553		-----		-----	
2560	ISO18218-1	200.7		1.94	
2566	In house	n.d.		-----	false negative test result?
2567	In house	169.77		0.61	
2590		-----		-----	
2591	In house	86.68		-2.96	
2592	ISO18218-2	219.8		2.76	
2605	ISO18218-1	110.0		-1.96	
2614	In house	151.1		-0.19	
2615	ISO18254	157.6646		0.09	
2618		-----		-----	
2644		-----		-----	
2649		-----		-----	
2665	In house	143.5		-0.52	
2668	ISO/DIS18254	219.32		2.74	
2671		-----		-----	

2713	In house	87.40		-2.93	
2715	GB/T23322	227.1242		3.07	
2723		-----		-----	
2726		-----		-----	
2733	GB/T23322	143.6513		-0.51	
2737		-----		-----	
2743	In house	446.6	R(0.01)	12.49	
2744	ISO/DIS18254	161.6		0.26	
3100	In house	111.3488		-1.90	
3117	OEKO-TEX	181.5432		1.11	
3118	In house	173.33		0.76	
3146		-----		-----	
3149	ISO18218-2	174.7		0.82	
3151	In house	180.5536		1.07	
3153	ISO18218-1	158.1		0.11	
3154		-----		-----	
3172	ISO18218-1	187.6	C	1.37	first reported <10
3176		-----		-----	
3185	In house	115.1		-1.74	
3190	ISO/DIS18254	130.0		-1.10	
3197		-----		-----	
3200	ISO18218-1	162.1		0.28	
3209		-----		-----	
3210		-----		-----	
3218	ISO/DIS18254	105.2		-2.16	
3220	In house	ND		-----	false negative test result?
3225	In house	NA		-----	
3233	In house	39.4851	C	-4.98	first reported 37.1899
3237		-----		-----	
3243	ISO18218-1	181.0204		1.09	
3246		-----		-----	

normality	OK
n	69
outliers	2
mean (n)	155.584
st.dev. (n)	38.7816
R(calc.)	108.589
R(Horwitz (n=4))	65.215





APPENDIX 2

Summary of other reported components in sample #16530 & #16531; results in mg/kg

lab	amount of component	remarks
2508	124.16 mg/kg OPEO in sample #16531,	possibly mixed up with NPEO?

Summary of not detected components in sample #16530& #16531; results in mg/kg

	sample #16530			sample #16531		
Lab	OP	NP	NPEO	OP	NP	OPEO
213	0	0	0	0	0	0
230	-----	-----	-----	-----	-----	-----
339	< 10	< 10	< 10	< 10	< 50	< 10
551	-----	-----	-----	-----	-----	-----
623	ND	ND	ND	ND	ND	ND
840	ND	ND	ND	ND`	ND	ND
2108	-----	-----	-----	-----	-----	-----
2115	-----	-----	-----	-----	-----	-----
2121	0	0	0	0	0	0
2129	-----	-----	-----	-----	-----	-----
2131	-----	-----	-----	-----	-----	-----
2135	-----	-----	-----	-----	-----	-----
2139	< 5	< 5	< 30	< 5	< 5	< 30
2159	-----	-----	-----	-----	-----	-----
2172	<10	<10	<50	<10	<10	<50
2201	<3	<3	<10	<3	<3	<10
2213	<10	<10	<10	<10	<10	<10
2215	<10	<10	<30	<10	<10	<30
2241	-----	-----	-----	-----	-----	-----
2246	<10	<10	<10	<10	<10	<10
2247	-----	-----	-----	-----	-----	-----
2255	nd	nd	nd	nd	nd	nd
2271	<10	<10	<10	<10	<10	<10
2272	<10	<10	<10	<10	<10	<10
2284	-----	-----	-----	-----	-----	-----
2285	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
2289	ND	ND	ND	ND	ND	ND
2290	<10	<10	<10	<10	<10	<10
2291	nd	nd	nd	nd	nd	nd
2293	n.d.	n.d.	1.784	n.d.	n.d.	2.775
2295	ND	ND	ND	ND	ND	ND
2297	<10	<10	<30	<10	<10	<30
2300	-----	-----	-----	-----	-----	-----
2301	-----	-----	-----	-----	-----	-----
2310	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2311	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2320	N.D	N.D	N.D	N.D	N.D	N.D
2330	-----	-----	<30	-----	-----	<30
2350	<3	<3	<30	<3	<3	<30
2358	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
2370	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2375	-----	-----	-----	-----	-----	-----
2380	>2	>2	>1	>2	>2	>1
2386	<10	<10	<10	<10	<10	<10
2389	n.d	n.d	n.d	n.d	n.d	n.d
2390	ND	ND	ND	ND	ND	ND
2415	ND	ND	ND	ND	ND	ND
2461	-----	-----	-----	-----	-----	-----
2467	-----	-----	-----	-----	-----	-----
2482	-----	-----	<10	-----	-----	<10
2486	-----	-----	<30	-----	-----	<30
2489	ND	ND	ND	ND	ND	ND
2492	-----	-----	-----	-----	-----	-----
2495	<2	<2	<2	<2	<2	<2
2497	0.001	0.001	0.001	0.001	0.001	1.53
2508	0.0	0.0	0.71	0.0	0.0	124.16
2511	-----	-----	-----	-----	-----	-----
2532	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2536	0.00	0.00	0.00	0.00	0.00	0.00
2549	ND	ND	ND	ND	ND	ND
2553	-----	-----	-----	-----	-----	-----
2560	<2	<2	<2	<2	<2	<2

2566	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2567	ND	ND	ND	ND	ND	ND
2590	-----	-----	-----	-----	-----	-----
2591	<10	<10	<10	<10	<10	<10
2592	-----	-----	-----	-----	-----	-----
2605	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2614	<5	<5	<30	<5	<5	<30
2615	-----	-----	<5	-----	-----	<5
2618	-----	-----	-----	-----	-----	-----
2644	-----	-----	-----	-----	-----	-----
2649	-----	-----	-----	-----	-----	-----
2665	0.022	0.022	1.156	0.028	0.318	10.73
2668	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2671	-----	-----	-----	-----	-----	-----
2713	-----	-----	-----	-----	-----	-----
2715	-----	-----	0.0000	---	---	0.0000
2723	-----	-----	-----	-----	-----	-----
2726	-----	-----	-----	-----	-----	-----
2733	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2737	-----	-----	-----	-----	-----	-----
2743	<1	<1	<1	<1	<1	<1
2744	-----	-----	-----	-----	-----	-----
3100	<10	<10	<10	<10	<10	<10
3117	-----	-----	-----	-----	-----	-----
3118	<10	<10	<10	<10	<10	<10
3146	-----	-----	-----	-----	-----	-----
3149	< 3	< 3	< 5	< 3	< 3	< 5
3151	0	0	0	0	0	0
3153	<10	<10	<10	<10	<10	<10
3154	-----	-----	-----	-----	-----	-----
3172	<10	<10	<10	<10	<10	<10
3176	-----	-----	-----	-----	-----	-----
3185	ND	ND	ND	ND	ND	ND
3190	-----	-----	-----	-----	-----	-----
3197	ND	ND	ND	ND	ND	ND
3200	0	0	0	0	0	0
3209	-----	-----	-----	-----	-----	-----
3210	-----	-----	<50	-----	-----	<50
3218	0.0	0.0	0.0	0.0	0.0	0.0
3220	ND	ND	ND	ND	ND	ND
3225	NA	NA	ND	NA	NA	ND
3233	ND	ND	ND	<4	<4	<10
3237	-----	-----	-----	-----	-----	-----
3243	-----	-----	-----	-----	-----	-----
3246	-----	-----	-----	-----	-----	-----

APPENDIX 2

Details of the methods used by the participants

lab	brief summary of the method.	component(s) used for the quantification
213		OPEO 9-10 AND NPEO 04
230	extracted by ultrasonication by methanol at 70°C for 60 minutes.	POE (9-10), POE(4)
339	Extraction in methanol, ultrasonic bath at 70°C for 60 minutes, filtration, analysis by LC-MS	Triton X-100, Triton X-102, NPEO (4), NPEO (9-10), NPEO (12), NPEO(14), NPEO (20)
551		
623		#136530: OPEO 9-10 CAS: 9002-93-1 ; #13651: NPEO 4 CAS: 68412-54-4
840	Solvent extraction analysis by LC-MS	OP7-8EO CAS 9036-19-5, NP4EO CAS 68412-54-4
2108	MeOH extraction; ultrasonic bath; LC-MS/MS	mixture contains: Imbentin-N/060 and IGEPAL CO-720 for NPEOs and Triton X-45 and IGEPAL CA-720 for O
2115		68412-54-4
2121	extraction with methanol in ultrasonic bath 1h at 40°C	Triton X100 / Triton X114 / IGEPAL CA720 / IGEPAL CO720 / IGEPAL CO520 / IGEPAL CO630
2129		NPEO: Igepal CO630 (CAS: 68412-54-4); OPEO: Triton X-100 (CAS: 9002-93-1)
2131		Igepal CA 520 and 720, Igepal CO 720, Imbetin N/060
2135		IGEPAL CO-630 (Nonylphenolethoxylat n 9) CAS: 68412-54-4;Triton X-100 (Octylphenolethoxylat n 12)
2139		OP:1806-26-4, NP:104-40-5 , OPEO:9002-93-1, NPEO:68412-54-4
2159		Triton X-100 (9002-93-1) and IGEPALCO-630 (68412-54-4)
2172		NPEO:CAS no. 68412-54-4 ,OPEO CAS no. 9002-93-1
2201	MeOH 70 ℃ ultrasonic 60min	OPEO (9 to 10) CAS:9002-93-1 NPEO(9 to 10) CAS:68412-54-4
2213	Approximately 1 g sample+10 ml methanol+70 degree for 1 hour sonication	Quantification by SIM mode. Case No NPEO:9002-93-1, OPEO:68412-54-4
2215	1g sample+20ml methanol,ultrasonic extract 70°C for60min	Octylphenolethoxylates CAS no. 9002-93-1 Nonylphenolethoxylates (IGEPAL CO-630),CAS no. 68412-54-4
2241	weigh 0.5g sample,then extract with 20 methanol at 40°Cfor 30 mins, analyse by LCMS	Triton X-100 (OPEO) ,CAS No.9002-93-1; Triton N-57 (NPEO), CAS No.127087-87-0
2246		Triton X-100, POE (9,10) -CAS No.: 9016-45-9, POE (4) - CAS No.: 68412-54-4
2247	solvent extraction followed by LCMSMS	NPGO: IGEPAL CAS 68412-54-4; OPEO: Triton X100 CAS 9002-93-1
2255	1 gm samples, 20 ml MeOH , sonicate for 70C at 30 minutes	OPEOs , CAS # 9220-93-1 & NPOEs , CAS # 68412-54-4
2271		NPEO:68412-54-4(IGEPAL CO-630), OPEO: 9002-93-1 (Triton (x-100)
2272		CAS 9002-93-1
2284		OPEO: 9002-93-1; NPEO: 68412-54-4
2285		OPEN, Triton X-100, CAS No.:9002-93-1; NPEO,CO-630,CAS No.:68412-54-4
2289	weigh 1.0g sample, add 10 ml methol, extract an hour in 70°C ultrasonic bath	Trition X-100 CAS 9002-93-1 IGEPAL CO-630 CAS 68412-54-4
2290		Triton X-100 (9002-93-1) and IGEPAL CO-630 (68412-54-4)
2291		degree of polymerization 9-10
2293	extraction in methanol ultrasonic bath 60 min at 70°C/ LCMSMS	OPEOs; Triton X-100 CAS 9002-93-1; NPEOs; IGEPAL CO-630 CAS 68412-54-4; NP CAS8852-15-3
2295		NPEO IGAPAL CO-630 CAS NO:68412-54-4 OPEO-TRITON X100 CAS NO:9002-93-1
2297		NPEOn with n = 9 – 10, CAS No. 68412-54-4 OPEOn with n = 9 – 10, CAS No. 9002-93-1
2300	Extraction in methanol and analysis on LCMS	Tergital Types NP-9 (CAS No.:- 127087-87-0) Triton X-114 (CAS No.:- 9036-19-5)
2301	AFIRM	OPEOs CAS 9002-93-1, NPEOs CAS 68412-54-4
2310	1.0g sample+add 20 ml methanol+sonicate sample at 70ºC for 60 mins+inject in LCMS	OPEO(CAS NO:9002-93-1), NPEO
2311	1gm sample in 20ml Methanol. Sonicated at 70°C for 1 hour analyzed by LCMS.	OPEO: 9002-93-1; NPEO 4 ethoxylate
2320	Methanol extract(20ml MeOH,sonicate 60min at 70°C) & analysis by HPLC-MS	4-Nonylphenol-ethoxylate(penta-),CAS# 68412-54-4 POE(9 to 10) tert-Octylphenol cat- (NG-S341)

2330		--
2350		NPEO 68412-54-4 OPEO 9002-93-1
2358	(1) 1g smpl into tube (2) pipette 20ml methanol and seal it (3) 70C for 60 mins (4) Cool (5) Filter	NPEO: Sigma, IGEPAL CO-630 CAS: 68412-54-4 OPEO: Sigma, Triton X-100 CAS: 9002-93-1
2370	Solvent extraction: (1) Weigh 1.0 g sample into a screw capped culture tube. (2) Pipette 20 ml methanol into the tube and seal it with cap. (3)Place the tube into the ultrasonic bath and sonicate at 70°C for 60 minutes. (4) Cool the tube to room temperature. (5) Filter the solution with 0.45 µm PTFE syringe filter into 1.5 ml amber vial before injection into HPLC/MS	#16530 (1) OP9-10EO (2) CAS number :9002-93-1; #16531 (1) OP9-10EO (2) CAS number :68412-54-4
2375	1g sample 20ml methanol,70C 60min ultrasonic	OPEO CAS.No.:9002-93-1, NPEO CAS No.:68412-54-4
2380		OPEO 9-10 & NPEO 4
2386		TritonX-100, Triton N-57
2389		OPEO (9-10) CAS No. 9002-93-1 & POE (4) NPEO CAS No. 68412-54-4
2390	1g sample in 20 ml methonol. Sonicate for 60 min at 70 C. Cool filter run.	POE (9-10) tert-octylphenol Cat:NG-S341-1G, Lot : 3221100 POE(4) nonyl phenol Cat:NG-S347-1G, Lot : 2754700
2415		OPEO(9002-93-1) & NPEO (68412-54-4)
2461	HPLC	We use standard substance of OPEO and NPEO.
2467	determination by LC MS MS	Triton X-100, CAS 9002-93-1; IGEPAL CO-630, CAS 68412-54-4
2482		OPEO: Triton X-100, CAS: 9002-93-1 NPEO: IGEPAL CO-630, CAS: 68412-54-4
2486	1 g sample extracted with 20 ml Methanol at sonicator at 70 C for 60 minutes	Octylphenolethoxylates (OPEOs);CAS no. 9002-93-1, Nonylphenolethoxylates (NPEOs);CAS no. 68412-54-4
2489		TRITON X-100 (CAS NO: 9002-93-1)/ IGEPAL CO 630 (CAS NO: 68412-54-4)
2492	in house mthod	NPEO(1-20) & OPEO(1-20) & NP & OP
2495	extraction with methanol/ACN in ultrasonic bath, 60min 70°C	OPEOs: Triton X-100 CAS n. 9002-93-1 NPEOs: IGEPAL CO-630 CAS n. 68412-54-4
2497	methanol extraction for APEOS - toluene extraction for AP - LC-MSMS an	9016-45-9 / 26027-38-3 / 37205-87-1 / 68412-54-4 / 127087-87-0
2508		IGEPAL CA-720, IGEPAL CO-720, Imbantin-N/060
2511	ultrasonic extraction with methanol at 70 C for 1 hour	9002-92-1// 68412-54-4
2532	sample extracted with Methanol using ultrasound and filtered .methanol extract is analysed by LCMSMS	Triton X-100 (OPEOs CAS No 9002-93-1) - Sigma make IGEPAL CO-630 (NPEOs CAS- 68412-54-4) Sigma
2536		Octylphenolethoxylates(Triton X-100)CN-9002-93-1 Nonylphenolethoxylates(IGEPAL CO-630)CN-68412-54-4
2549	„«1.Prepare approximately 1 g of the cut textile, weigh it to the nearest 10 mg and then place it into the glass container (extraction vessel). „ «2.Pipette 20 ml of methanol into the glass container (extraction vessel). „ «3.Place the glass container (extraction vessel) into an ultrasonic bath at 70°C for (60 +/- 5) min. «4.Afterwards, let the extract cool down to room temperature. „ «5.Filter about 1 ml of the extraction solution into a LC vial using a disposable syringe equipped with a membrane filter	(OPEOs) CAS no. 9002-93-1, (Triton X-100), NPEO (IGEPAL CO-6),CAS no. 68412-54-4
2553		NPEO-68412-54-4
2560	Approximately 1 g of the leather sample is weighed accurately to 10	OPEO with n= 9-10 (Triton-X-100) (CAS 9002-93-1) NPEO n= 9-10 (IGEPAL CO-630) (CAS-68412-54-4)
2566		OPEO: CAS 9002-93-1; NPEO: CAS 68412-54
2567		-
2590	Extraction with methanol (ultrasonich bath), filtered and injected	OPEO Triton X-100 Sigma Aldrich, CAS no. 9002-93-1 NPEO Igepal CO-630 Sigma Aldrich, CAS no. 68412-54-4
2591		Triton-X-100 (9002-93-1); 4-Octylphenol-ethoxylate(mono-, di-, tri-) (26636-32-8) N-40 Alternative (4-nonylphenyl-polyethylene) (9016-45-9) 4-Nonylphenol-ethoxylate(mono-, di-, tri-) (68412-54-4) 4-Octylphenol (1806-26-4) 4-tert-octylphenol (140-66-9) Nonoxynol-9 (26027-38-3) P-(1,1,3,3-Tetramethylbuyl)phenol (27193-28-8) Nonylphenol (25154-52-3) 4-n-nonylphenol (104-40-5) Nonylphenol isomers (601-53-00-8
2592		Ottilfenolo etossilato (CAS 9002-93-1) Nonilfenolo etossilato (CAS 9016-45-9, CAS 68412-54-4)

2605	1g,20mL methanol,70°C,ultrasonic 60mins	NPEO(n=9),9016-45-9;OPEO(n=10),9002-93-1
2614	1 g of sample + 20 ml of methanoladded to the flask and sonicat for 60 mins at 70 Deg C, Inject inLC	NPEO, CAS No.68412-54-4 OPEO CAS No.9002-93-1
2615	weigh sample, and then place it into the glass container. Pipette 20 ml of methanol .ultrasonic bath extraction at 70°C for 60 min. Filter the extraction solution with a 0.22um membrane filter. APEO contents were tested by HPLC-FLD.	OPEO£º9002-93-1 NPEO£º68412-54-4
2618		NPEO (2-18)
2644		
2649	extracted with methanol at 40oC and sonicate for 60 minutes./analyzed by LCMS/GCMS	IGEPAL-Cas no:68412-54-4;Triton X-100-Cas no:9002-93-1;NP-Cas no:104-40-5;OP-Cas no: 1806-26-4n
2665	extraction with hexane, derivatization with MSTFA, determination by GC-MSD	CAS-Numbers.: 140-66-9; 25154-52-3; 1806-26-4;ý104-40-5;9002-93-1; 127087-87-0
2668	„«1.Prepare approximately 1 g of the cut textile, weigh it to the nearest 10 mg and then place it into the glass container (extraction vessel). «2.Pipette 20 ml of methanol into the glass container (extraction vessel). «3.Place the glass container (extraction vessel) into an ultrasonic bath at 70°C for (60 +/- 5) min «4.Afterwards, let the extract cool down to room temperature. „ «5.Filter about 1 ml of the extraction solution into a LC vial using a disposable syringe equipped with a membrane filter.	(Triton X-100),(OPEOs) CAS no. 9002-93-1 (NPEOs) CAS no. 68412-54-4,(IGEPAL CO-630)
2671	solvent extraction followed by LC MS and GC MS analyis	CAS no. 9002-93-1,CAS no. 68412-54-4,CAS no. 1806-26-4,CAS no. 84852-15-3,CAS no. 140-66-9
2713	Extraction with methanol	NPEO 68412-54-4; NP 84852-15-3; OPEO 9002-93-1; OP 1806-26-4
2715	Approximately 1 g of the sample is weighed accurately to 10 mg.150 ml methanol is added. The samples were extracted with methanol by Soxhlet Extraction for 3h.Remove the organic solvent by rotary evaporator at approximately 70 °C. Redissolve the residues in 2 ml of methanol and filter through a polyamide membrane. Aliquot of the extraction solution is transferred into an HPLC Vial. The aliquot is just ready for the HPLC with fluorescence detector analysis.	9016-45-9 9002-93-1
2723		
2726		Triton X-100, (OPEOs) CAS no. 9002-93-1 and IGEPAL CO-630, (NPEOs) CAS no. 68412-54-4
2733	Methanol extraction followed by LC-MS; OP and NP: Acetonitrile extraction followed by GC-MS.	Quantification: 5.4.4, CAS No.: 9002-93-1(OPEO), 68412-54-4(NPEO)
2737		9002-93-1 for OPEO, 68412-54-4 for NPEO
2743	Extraction with methanol in ultrasonic bath and LC/MS/MS for APEOs determination and GC/MS for APs.	NPEO (CAS 009016-45-9) OPEO (CAS 009002-93-1) NP (CAS 104-40-5);OP (CAS 140-66-9)
2744		(Triton X-100 Cas no:900293-1)(Nonidet P 40 substitute cas no:9016-45-9)
3100	1g smaple is extracted by 20ml Methanol ,and the extraction is analyzed by LC-MS	OPEO 9002-93-1 NPEO 68412-54-4
3117	OEKO-TEX Testing Procedures£¨2015	OPEO(9-10) NPEO(9-10)
3118	100% methanol extraction.	OPEO's Triton X-100 cas no : 9002-93-1 NPEO's IGEPAL CO-630 cas no : 68412-54-4
3146	extraction solvent: methanol, Ultrasonic extraction 1h 70°C	IGEPAL CO-630 CAS: 68412-54-4; Triton X-100 CAS:9002-93-1
3149		NPEO: Ethylan 77, CAS 9016-45-9, OPEO: Triton X 100, CAS 9002-93-1
3151	extraction with methanol, GCMS/LCMS detection	Octylphenolethoxylate CAS 9002-93-1 Nonylphenolethoxylate CAS 68412-54-4
3153		OPEO-->Triton X-100, NPEO-->Igepal CO-520
3154		OPEO CAS: 9002-93-1 / NPEO CAS: 68412-54-4
3172	1g+10ml Methanol, 70°C 1h in ultrasoinic bath Analysis method: HPLC-MS, SCAN Mode	9002-93-1:TritonTM X-100; 68412-54-4: IGEPAL CO-630. All component present in std used for quant
3176		NPEO (104-35-8), OPEO (9002-93-1)
3185	In house method.Weigh 0.5g ,add 20mL MeOH ,put in ultrasonic bath for 30 mins at 70¡æ	NPEO:68412-54-4,OPEO:9002-93-1
3190	1g+20mL methanol, place the vial in an ultrasonic bath for 60 min at 70°C.	OPEO: 9002-93-1; NPEO:68412-54-4

3197		Igepal CO-630 (NPEO) 68412-54-4 Triton X-100 (OPEO) 9002-93-1
3200	extracted with methanol in ultra bath and analyzed by UPLC-MSMS	Octylphenol ethoxylate, OPEOn with n = 9 ¨C 10,CAS No. 9002-93-1 Nonylphenol ethoxylate, NPEOn with
3209		OPEO: Sigma-Aldrich Triton X-100 CAS: 9002-93-1 NPEO: Sigma-Aldrich IGEPAL CO-630 CAS: 68412-54-4
3210	ultrasonic extraction with methanol LC/QQQ analysis	TRITON X-100 CAS number :9002-93-1 (OPEO) IGEPAL CO-630 (NPEO)
3218		No
3220	Extraction in Methanol and Detection & Quantification by LC/MSD & GC/MSD.	For NPEO- Tergitol® Type NP-9 (127087-87-0) For OPEO- TritonTM X-100 BioXtra (9002-93-1)
3225	solvent extraction at 70C for 1 hour	Triton X-100 for OPEO (CAS: 9002-93-1) IGEPAL CO-630 for NPEO (CAS:68412-54-4)
3233	extraction with MeOH in ultrasonic bath at 40°C during 1H. LCMS for APEO and GCMS for AP	OPEO [9002-93-1] NPEO [68412-54-4]
3237	Methanol extraction at 40 C for 1 hour-LCMS-MS analyse	OPEO Triton X-100 NPEO IGEPAL CO-630
3243	HPLC/DAD detection, extraction with Methanol 70°C, 1 hour, ultrasonic	Triton N 57 (NPEO), Triton X 100 (OPEO)
3246		OPEO TRITON X-100 (CAS: 9002-93-1) NPEO IGEPAL CO 630 (CAS: 68412-54-4)

APPENDIX 3

Number of participants per country

7 labs in	BANGLADESH
1 lab in	BRAZIL
2 labs in	CAMBODIA
4 labs in	FRANCE
12 labs in	GERMANY
1 lab in	GUATEMALA
5 labs in	HONG KONG
13 labs in	INDIA
3 labs in	INDONESIA
8 labs in	ITALY
2 labs in	KOREA
1 lab in	MAURITIUS
1 lab in	MOROCCO
24 labs in	P.R. of CHINA
2 labs in	PAKISTAN
1 lab in	ROMANIA
1 lab in	SPAIN
2 labs in	SRI LANKA
2 labs in	SWITZERLAND
1 lab in	TAIWAN R.O.C.
1 lab in	TUNISIA
9 labs in	TURKEY
4 labs in	VIETNAM

APPENDIX 4

Abbreviations:

C = final result after checking of first reported suspect result
D(0.01) = outlier in Dixon's outlier test
D(0.05) = straggler in Dixon's outlier test
G(0.01) = outlier in Grubbs' outlier test
G(0.05) = straggler in Grubbs' outlier test
DG(0.01) = outlier in Double Grubbs' outlier test
DG(0.05) = straggler in Double Grubbs' outlier test
R(0.01) = outlier in Rosner outlier test
R(0.05) = straggler in Rosner outlier test
n.a. = not applicable
n.d. = not detected

Literature:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, April 2014
2 Öko-Tex Standard 100: January 2016
3 Öko-Tex customer information letter, January 2016
4 Impacts of Environmental Standards and requirements in EU Countries. Aug 99
5 Horwitz. Journal of AOAC International Vol. 79 No.3. 1996
6 P.L. Davies. Fr Z. Anal. Chem. 351. 513. (1988)
7 W.J. Conover. Practical; Nonparametric Statistics. J. Wiley&Sons. NY. p.302. (1971)
8 ISO 5725. (1986)
9 ISO 5725. parts 1-6. (1994)
10 ISO18254:2016
11 ISO18218-1:2015
12 ISO13528-05
13 M. Thompson and R. Wood. J. AOAC Int. 76. 926. (1993)
14 Analytical Methods Committee Technical brief, No4 January 2001.
15 The Royal Society of Chemistry 2002, Analyst 2002, 127 page 1359-1364, P.J. Lowthian and M. Thompson
16 Official Journal of the European Communities 2016/26
17 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), pp. 165-172, (1983)