Results of Proficiency Test
AP & APEO in textile
March 2020

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. APEO may degrade in the environment to the corresponding Octyl- and Nonylphenols (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 in textile every year. During the annual proficiency testing program 2019/2020, it was decided to continue with the PT for the analyzes of AP and APEO in textile.

In this interlaboratory study 106 laboratories in 25 different countries registered for participation. See appendix 4 for the number of participants per country.
In this report the results of this proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2   SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send two different textile samples of 3 grams each, labelled #20530 and #20531 respectively. The samples were positive on OPEO or NPEO. The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1   QUALITY SYSTEM

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

2.2   PROTOCOL

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

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

A batch of violet viscose was selected which was made positive on OPEO by a third-party laboratory. A part of this batch was cut into small pieces. After homogenization the batch was divided over 150 subsamples in small bags of 3 grams each and labelled #20530.
The homogeneity of the subsamples was checked by determination of OPEO using an in-house test method on eight stratified randomly selected subsamples.

	OPEO in mg/kg
Sample #20530-1	156.00
Sample #20530-2	159.62
Sample #20530-3	150.48
Sample #20530-4	158.13
Sample #20530-5	162.89
Sample #20530-6	159.30
Sample #20530-7	155.85
Sample #20530-8	156.97

Table 1: homogeneity test results of subsamples #20530

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

	OPEO in mg/kg
r (observed)	10.13
reference method	Horwitz (n=5)
0.3 * R (reference method)	22.09

Table 2: evaluation of the repeatability of subsamples #20530

The calculated repeatability was in agreement with 0.3 times the estimated reproducibility of the reference method. Therefore, homogeneity of the subsamples #20530 was assumed.

A batch of blue cotton was selected which was made positive on NPEO by iis. A part of this batch was cut into small pieces. After homogenization the batch was divided over 150 subsamples in small bags of 3 grams each and labelled #20531. The homogeneity of the subsamples was checked by determination of NPEO using an in-house test method on eight stratified randomly selected subsamples.

	NPEO in mg/kg
Sample #20531-1	103.45
Sample #20531-2	100.72
Sample #20531-3	101.27
Sample #20531-4	106.80
Sample #20531-5	110.63
Sample #20531-6	107.44
Sample #20531-7	104.06
Sample #20531-8	109.15

Table 3: homogeneity test results of subsamples #20531

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

	NPEO in mg/kg
r (observed)	10.16
reference method	Horwitz (n=5)
0.3 * R (reference method)	15.72

Table 4: evaluation of the repeatability of subsamples #20531

The calculated repeatability was in agreement with 0.3 times the estimated reproducibility of the reference method. Therefore, homogeneity of the subsamples #20531 was assumed.

To each participating laboratory, one sample labelled #20530 and one sample labelled #20531 were sent on February 12, 2020.

2.5 ANALYZES

The participants were requested to determine on samples #20530 and #20531 the concentrations of Octylphenol (OP), Nonylphenol (NP), Octylphenol Ethoxylates (OPEO), Nonylphenol Ethoxylates (NPEO) and the Total of OP, NP, OPEO + NPEO. It was requested, to ensure homogeneity, to not use less than 0.5 grams per determination. Also, some analytical 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 but report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3   RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per determination in appendix 1 and 2 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 Organization, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5).

For the statistical evaluation, the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<...' or '>...' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the results of the statistical evaluation should be used with due care.

In accordance to ISO5725 the original test results per determination were submitted subsequently to Dixon's, Grubbs' and or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

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

3.2 GRAPHICS

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

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

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

3.3 Z-SCORES

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

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency tests could be used.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated in accordance with:

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

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 problems occurred with the dispatch of the samples. However, five laboratories informed iis that they were not able to report test results due to the measures taken to contain the Covid-19 pandemic in their countries. Five other participants did not report any results at all. In total 96 participants reported 347 numerical test results. Observed in all reported results were 17 outlying results, which is 4.9%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

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, see also paragraph 3.1.

4.1   EVALUATION PER SAMPLE AND PER COMPONENT

In this section the reported test results are discussed per sample and per component. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These methods are also in the table together with the original data. The abbreviations, used in these tables, are explained in appendix 5.

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

sample #20530

OPEO:

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

Total OP + NP + OPEO + NPEO:

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

sample #20531

NPEO:

The determination of this component was problematic. Six 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.

Most participants agreed about the absence of Octylphenol and Nonylphenol in this sample and therefore the z-scores are not calculated. See appendix 2 for reported test results.

Total OP + NP + OPEO + NPEO:

The determination of this sum-component was problematic. Four 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.

Most participants agreed about the absence of Octylphenol and Nonylphenol in this sample and therefore the z-scores are not calculated. See appendix 2 for reported test results.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Components	unit	n	average	2.8 * sd	R(target)
OPEO	mg/kg	92	181.2	85.6	83.0
Total OP + NP + OPEO + NPEO	mg/kg	74	179.3	78.1	82.3

Table 5: reproducibilities of components on sample #20530

Components	unit	n	average	2.8 * sd	R(target)
NPEO	mg/kg	90	126.7	97.1	62.3
Total OP + NP + OPEO + NPEO	mg/kg	74	127.9	101.0	61.7

Table 6: reproducibilities of components on sample #20531

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

4.3   COMPARISON OF THE PROFICIENCY TEST OF MARCH 2020 WITH PREVIOUS PTS

	March 2020	February 2019	February 2018	March 2017	March 2016
Number of reporting laboratories	96	105	92	95	105
Number of test results	347	366	329	378	412
Number of statistical outliers	17	21	8	9	13
Percentage of statistical outliers	4.9%	5.7%	2.4%	2.4%	3.2%

Table 7: comparison with previous proficiency test

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

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

	March 2020	February 2019	February 2018	March 2017	March 2016	Horwitz 500-1500 mg/kg
Octylphenol Ethoxylates (OPEO)	17%	10%	16%	15%	16%	18-12%
Nonylphenol Ethoxylates (NPEO)	27%	13%	28%	18%	27%	18-12%
Total OP + NP + OPEO + NPEO	16-28%	11%	15-26%	15-17%	16-25%	18-12%

Table 8: development of uncertainties over the years

The uncertainties observed in this PT are comparable to the uncertainties observed in previous PTs. Especially for the determination of NPEO the uncertainty is large in comparison to the estimated requirements mentioned in the target.

4.4   EVALUATION OF THE ANALYTICAL DETAILS

For this PT, some analytical details were requested (see appendix 3). Based on the answers given by the participants the following can be summarized:
Seventy-four of the registered participants mentioned that they are accredited for determination of AP+APEO in textile. Eighty-six participants mentioned that they have used a test portion of 0.5 or 1.0 grams. One mentioned to have used less material (0.1 gram) for intake, one mentioned to have used more testing material for intake (1.5 gram). One participant did not mention a weight, but measurements only (5mm x 5mm).
All participants that mentioned the technique to release/extract the analytes as ultrasonic. All mentioned to have used methanol for release/extraction. The majority of the group mentioned to use the following conditions: 60 minutes at 70°C.

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

5   DISCUSSION

In this proficiency test for the determination of AP and APEO in textile the majority of the participants had no problems with the analysis of OPEO in textile, but did have problems with the determination of NPEO in textile at the levels as present in this PT.

When the test results of this interlaboratory study were compared to the OEKO-TEX and Blue Sign requirements and the EU (REACH) regulations on Textiles (see table 9), it is noticed that all, but one of the reporting laboratories would reject sample #20530 for containing too much OPEO and the total of OP + NP + OPEO + NPEO and that thirteen of the reporting laboratories would reject sample #20531 for containing too much NPEO and/or too much of the total of OP + NP + OPEO + NPEO.

It is observed that not all participants reported a value for the total of 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
Total OP + NP	10 mg/kg	---	---
Every single APEO	---	100 mg/kg *)	---
NPEO	---	---	100 mg/kg
Total OP + NP + OPEO + NPEO	100 mg/kg	---	---

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

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

6   CONCLUSION

Although, it can be concluded that the majority of the participants has no problem with the determination of some APEO 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 #20530; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210		-----		-----	
230	ISO18254-1	479.24	C,R(0.01)	10.05	first reported: 23.96
339	In house	309	R(0.01)	4.31	remark participant: this is 4-tert-octyl-phenol ethoxylates
551	In house	244.0099		2.12	
623	ISO18254-1	176.67		-0.15	
840	ISO18254-1	186.0		0.16	
841	ISO18254-1	186.71		0.19	
2115		-----		-----	
2121		-----		-----	
2129	ISO18254-1	170.5		-0.36	
2132	In house	146.70		-1.16	
2201	ISO/DIS 18254-1	161.9		-0.65	
2213	ISO18254-1	185.5		0.14	
2217	ISO/DIS 18254-1	194.005		0.43	
2241	ISO18254-1	201.3		0.68	
2255	ISO/DIS 18254-1	185.7		0.15	
2265	ISO18254-1	155		-0.88	
2286	ISO/DIS 18254-1	258.6		2.61	
2287	ISO18254-1	178.04	C	-0.11	first reported: 374.25
2289	ISO18254-1	160		-0.72	
2290	ISO18254-1	163.8		-0.59	
2293		-----		-----	
2297	ISO18218-1	168.3		-0.44	
2300		213.76		1.10	
2301	ISO18254-1	174.90	C	-0.21	first reported: 17.49
2310	ISO18254-1	183		0.06	
2311	ISO18254-1	176.799		-0.15	
2320	ISO18254-1	214.29		1.12	
2330	ISO18254-1	111.80		-2.34	
2347	ISO18254-1	182		0.03	
2350	ISO18254-1	265.36	C	2.84	first reported; 281.32
2352	ISO18254-1	181.31		0.00	
2357	ISO/DIS 18254-1	181.0		-0.01	
2358	ISO/DIS 18254-1	162.04		-0.65	
2363	ISO/DIS 18254-1	181.7		0.02	
2365	ISO/DIS 18254-1	177.16		-0.14	
2366	ISO/DIS 18254-1	178.2		-0.10	
2370	ISO18254-1	186		0.16	
2374	In house	180.02		-0.04	
2375	ISO18254-1	184		0.09	
2378	ISO18254-1	181.97		0.03	
2379	ISO18254-1	194.49		0.45	
2380	ISO/DIS 18254-1	180.7		-0.02	
2382	ISO18254-1	182.3		0.04	
2386	ISO18254-1	160.99		-0.68	
2390	ISO18254-1	210.97		1.00	
2410	ISO18254-1	149.08		-1.08	
2425	ISO18218-1	188.02		0.23	
2426	ISO18254-1	193.22		0.40	
2449		-----		-----	
2456	ISO/DIS 18254-1	135.93		-1.53	
2459	ISO18254-1	150		-1.05	
2462	ISO18254-1	160		-0.72	
2489	ISO/DIS 18254-1	190.2		0.30	
2494	ISO18254-1	1881.1	C,R(0.01)	57.35	first reported: 431.42
2508	ASTM D7485/D7742	131.82		-1.67	
2511	ISO18254-1	187.95		0.23	
2514	ISO18254-1	188.17		0.23	
2532	ISO18254-1	182.85		0.06	
2549	ISO18254-1	182.5		0.04	
2560	ISO18254-1	186.8		0.19	
2561	ISO/DIS 18254-1	269.98		2.99	
2567	ISO18254-1	192.1		0.37	
2573	ISO18254-1	176.3		-0.17	
2582		-----		-----	
2590	ISO/DIS 18254-1	137.139		-1.49	
2591	In house	149.540		-1.07	
2605	GB/T23972	152.51		-0.97	
2614	In house	181.23		0.00	
2618	ISO/DIS 18254-1	185.26		0.14	
2629	ISO18254-1	236.4		1.86	
2638	In house	98.6		-2.79	
2644		-----		-----	
2668	ISO18254-1	180.24		-0.03	

lab	method	value	mark	z(targ)	remarks
2678		-----		-----	
2726	ISO/DIS 18254-1	226.13		1.52	
2741	ISO18254-1	191.12		0.33	
2743	ISO18254-1	183.0		0.06	
2766	ISO/DIS 18254-1	190.0		0.30	
2789	ISO18254-1	228.42		1.59	
2798	ISO18254-1	172		-0.31	
2804	ISO18254-1	167		-0.48	
2812	ISO18254-1	204		0.77	
2864	CNS15579	139.75		-1.40	
2912		-----		-----	
3100	ISO18254-1	150.8		-1.03	
3116	ISO/DIS 18254-1	158		-0.78	
3118	In house	152.49		-0.97	
3149	ISO18254-1	260		2.66	
3154	In house	624.05	C,R(0.01)	14.94	first reported: 560.22
3160	ISO18254-1	212.3		1.05	
3172	ISO/DIS 18254-1	196.5		0.52	
3176	ISO/DIS 18254-1	183.2	C	0.07	first reported: 283.60
3182	ISO/DIS 18254-1	173.17		-0.27	
3185	ISO18254-1	153.85		-0.92	
3197	ISO18254-1	196.6		0.52	
3200		-----		-----	
3210	In house	197.87		0.56	
3214	ISO18254-1	152.9		-0.96	
3218	ISO/DIS 18254-1	155.03		-0.88	
3220	ISO/DIS 18254-1	153.36		-0.94	
3222	ISO18254-1	230.7		1.67	
3232	ISO/DIS 18254-1	145.052		-1.22	
3237	ISO18254-1	217		1.21	
3248	In house	153		-0.95	
3250	ISO18254-1	175.34		-0.20	

normality	suspect			
n	92			
outliers	4			
mean (n)	181.216			
st.dev. (n)	30.5728	RSD = 17%		
R(calc.)	85.604			
st.dev.(Horwitz (n=5))	29.6422			
R(Horwitz (n=5))	82.998		compare R(ISO18254-1:16) = 49.768	
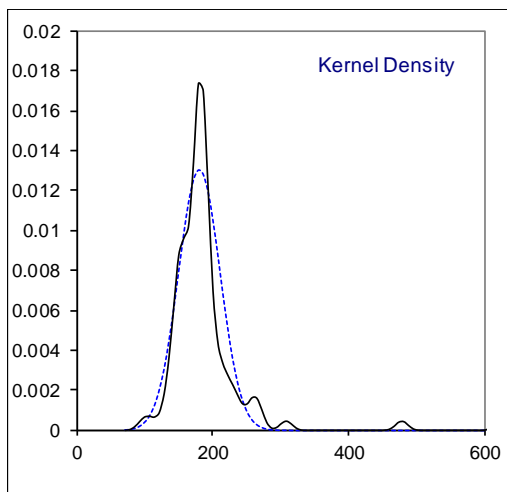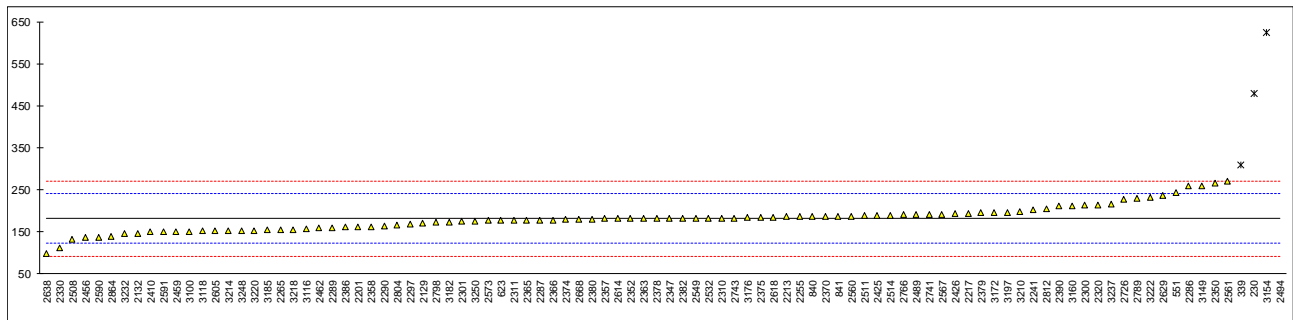
Determination of Total of OP, NP, OPEO and NPEO on sample #20530; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210		-----		-----	
230	ISO18254-1	479.24	C,R(0.01)	10.21	first reported: 23.96
339		-----		-----	
551	In house	244.0099		2.20	
623	ISO18254-1	176.67		-0.09	
840	ISO18254-1	186.0		0.23	
841	ISO18254-1	186.71		0.25	
2115		-----		-----	
2121		-----		-----	
2129	ISO18254-1	170.5		-0.30	
2132	In house	146.70		-1.11	
2201	ISO/DIS 18254-1	161.9		-0.59	
2213	ISO18254-1	185.5		0.21	
2217	ISO/DIS 18254-1	194.005		0.50	
2241	ISO18254-1	201.3		0.75	
2255	ISO/DIS 18254-1	185.7		0.22	
2265	ISO18254-1	155		-0.83	
2286	ISO/DIS 18254-1	258.6		2.70	
2287	ISO18254-1	178.04	C	-0.04	first reported: 374.25
2289	ISO18254-1	160		-0.66	
2290	ISO18254-1	163.8		-0.53	
2293		-----		-----	
2297	ISO18218-1	168.3		-0.37	
2300		213.76		1.17	
2301	ISO18254-1	174.90	C	-0.15	first reported: 17.49
2310	ISO18254-1	183		0.13	
2311	ISO18254-1	176.799		-0.09	
2320	ISO18254-1	214.29		1.19	
2330	ISO18254-1	111.80		-2.30	
2347	ISO18254-1	182		0.09	
2350	ISO18254-1	265.36	C	2.93	first reported: 281.32
2352	ISO18254-1	181.31		0.07	
2357	ISO/DIS 18254-1	181.0		0.06	
2358	ISO/DIS 18254-1	162.04		-0.59	
2363	ISO/DIS 18254-1	181.7		0.08	
2365	ISO/DIS 18254-1	177.16		-0.07	
2366	ISO/DIS 18254-1	178.2		-0.04	
2370	ISO18254-1	186		0.23	
2374	In house	180.02		0.02	
2375	ISO18254-1	184		0.16	
2378	ISO18254-1	181.97		0.09	
2379		-----		-----	
2380	ISO/DIS 18254-1	180.7		0.05	
2382	ISO18254-1	182.3		0.10	
2386	ISO18254-1	160.99		-0.62	
2390	ISO18254-1	210.97		1.08	
2410	ISO18254-1	149.08		-1.03	
2425	ISO18218-1	188.02		0.30	
2426	ISO18254-1	ND		-----	
2449		-----		-----	
2456	ISO/DIS 18254-1	135.93		-1.48	
2459	ISO18254-1	150		-1.00	
2462		-----		-----	
2489	ISO/DIS 18254-1	190.2		0.37	
2494	ISO18254-1	1881.1	C,R(0.01)	57.93	first reported: 431.42
2508		-----		-----	
2511	ISO18254-1	187.95		0.29	
2514	ISO18254-1	188.17		0.30	
2532	ISO18254-1	182.85		0.12	
2549	ISO18254-1	182.5		0.11	
2560	ISO18254-1	186.8		0.25	
2561		-----		-----	
2567	ISO18254-1	192.1		0.44	
2573	ISO18254-1	176.3		-0.10	
2582		-----		-----	
2590	ISO/DIS 18254-1	137.139		-1.44	
2591	In house	149.540		-1.01	
2605	GB/T23972	152.51		-0.91	
2614	In house	181.23		0.07	
2618		-----		-----	
2629		-----		-----	
2638	In house	98.6		-2.75	
2644		-----		-----	
2668	ISO18254-1	180.24		0.03	

lab	method	value	mark	z(targ)	remarks
2678		-----		-----	
2726		-----		-----	
2741	ISO18254-1	191.12		0.40	
2743	ISO18254-1	209.7		1.03	
2766	ISO/DIS 18254-1	190		0.36	
2789	ISO18254-1	228.42		1.67	
2798		-----		-----	
2804	ISO18254-1	167		-0.42	
2812		-----		-----	
2864		-----		-----	
2912		-----		-----	
3100	ISO18254-1	150.8		-0.97	
3116		-----		-----	
3118	In house	152.49		-0.91	
3149		-----		-----	
3154	In house	624.05	C,R(0.01)	15.14	first reported: 560.22
3160	ISO18254-1	212.3		1.12	
3172	ISO/DIS 18254-1	196.5		0.58	
3176		-----		-----	
3182	ISO/DIS 18254-1	173.17		-0.21	
3185	ISO18254-1	153.85		-0.87	
3197	ISO18254-1	196.6		0.59	
3200		-----		-----	
3210		-----		-----	
3214	ISO18254-1	152.9		-0.90	
3218		-----		-----	
3220	ISO/DIS 18254-1	153.36		-0.88	
3222	ISO18254-1	230.7		1.75	
3232		-----		-----	
3237		-----		-----	
3248	In house	153		-0.90	
3250	ISO18254-1	175.34		-0.14	

	normality	not OK			
	n	74			
	outliers	3			
	mean (n)	179.316			
	st.dev. (n)	27.8954	RSD = 16%		
	R(calc.)	78.107			
	st.dev.(Horwitz (n=5))	29.3779			
	R(Horwitz (n=5))	82.258		compare R(ISO18254-1:16) = 49.214	
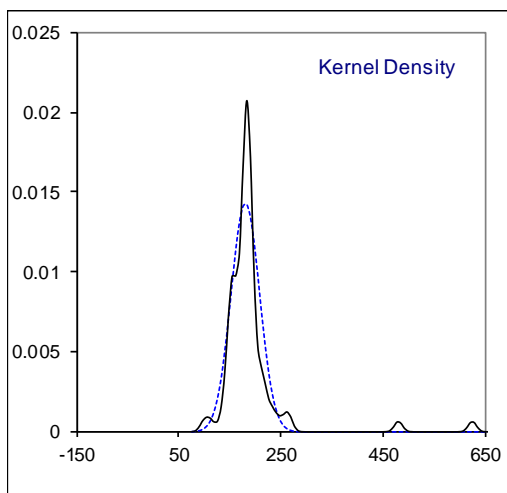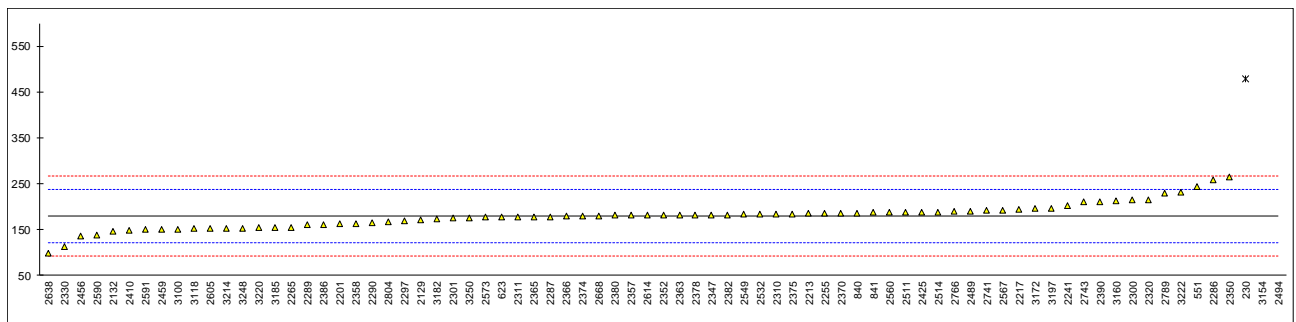
Determination of Nonylphenol Ethoxylates (NPEO) on sample #20531; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210		-----		-----	
230	ISO18218-1	721.21	C,R(0.01)	27.18	first reported: 36.06
339	In house	162		1.61	remark participant: this is 4-nonyl-phenol ethoxylates
551	In house	160.8377		1.56	
623	ISO18254-1	126.25		-0.02	
840	ISO18254-1	126.0		-0.03	
841	ISO18254-1	128.71		0.09	
2115		-----		-----	
2121		-----		-----	
2129	ISO18254-1	113.0		-0.63	
2132	In house	125.86		-0.04	
2201	ISO/DIS 18254-1	109.3		-0.80	
2213	ISO18254-1	118		-0.40	
2217	ISO/DIS 18254-1	136.5		0.45	
2241	ISO18254-1	64.3		-2.85	
2255	ISO/DIS 18254-1	96.3		-1.39	
2265	ISO18254-1	84		-1.95	
2286	ISO/DIS 18254-1	224.7		4.48	
2287	ISO18254-1	188.4		2.82	
2289	ISO18254-1	107		-0.90	
2290	ISO18254-1	116.9		-0.45	
2293		-----		-----	
2297	ISO18218-1	119.8		-0.32	
2300		1019	C,R(0.01)	40.79	first reported: 3541.39
2301	ISO18254-1	90.19	C	-1.67	first reported: 9.02
2310	ISO18254-1	140		0.61	
2311	ISO18254-1	143.59		0.77	
2320	ISO18254-1	197.87		3.25	
2330	ISO18254-1	93.22	C	-1.53	first reported: 42.47
2347	ISO18254-1	118		-0.40	
2350	ISO18254-1	246.72	C	5.49	
2352	ISO18254-1	119.49		-0.33	first reported: 270.52
2357	ISO/DIS 18254-1	120.0		-0.31	
2358	ISO/DIS 18254-1	125.92		-0.04	
2363	ISO/DIS 18254-1	116.9		-0.45	
2365	ISO/DIS 18254-1	124.54		-0.10	
2366	ISO/DIS 18254-1	119.6		-0.33	
2370	ISO18254-1	108		-0.86	
2374	GB/T23322	118.63		-0.37	
2375	ISO18254-1	142		0.70	
2378	ISO18254-1	118.52		-0.38	
2379	ISO18254-1	156.92		1.38	
2380	ISO/DIS 18254-1	108.6		-0.83	
2382	ISO18254-1	120.3		-0.29	
2386	ISO18254-1	81.18		-2.08	
2390	ISO18254-1	103.36		-1.07	
2410	ISO18254-1	114.32		-0.57	
2425	ISO18218-1	115.84		-0.50	
2426	ISO18254-1	100.24		-1.21	
2449		-----		-----	
2456	ISO/DIS 18254-1	194.9		3.12	
2459	ISO18254-1	120		-0.31	
2462	ISO18254-1	110		-0.76	
2489	ISO/DIS 18254-1	105.8		-0.96	
2494	ISO18254-1	1605.7	C,R(0.01)	67.61	first reported: 280.9
2508	ASTM D7485/D7742	121.32		-0.25	
2511	ISO18254-1	104.02		-1.04	
2514	ISO18254-1	98.07		-1.31	
2532	ISO18254-1	101.77		-1.14	
2549	ISO18254-1	135.4		0.40	
2560	ISO18254-1	114.36		-0.57	
2561	ISO/DIS 18254-1	333.26	R(0.01)	9.44	
2567	ISO18254-1	98.0		-1.31	
2573	ISO18254-1	119.8		-0.32	
2582		-----		-----	
2590	ISO/DIS 18254-1	81.930		-2.05	
2591		175.840		2.25	
2605	GB/T23972	112.32		-0.66	
2614	In house	108.52		-0.83	
2618	ISO/DIS 18254-1	112.15		-0.67	
2629	ISO18254-1	160.1		1.53	
2638	In house	152.59		1.18	
2644		-----		-----	
2668	ISO18254-1	117.28		-0.43	

lab	method	value	mark	z(targ)	remarks
2678		-----		-----	
2726	ISO/DIS 18254-1	102.25		-1.12	
2741	ISO18254-1	118.19		-0.39	
2743	ISO18254-1	170.4		2.00	
2766	ISO/DIS 18254-1	108.4		-0.84	
2789	ISO18254-1	158.98		1.47	
2798	ISO18254-1	114		-0.58	
2804	ISO18254-1	143		0.74	
2812	ISO18254-1	160	C	1.52	first reported: 300
2864	CNS15579	33.04	C	-4.28	first reported: 26.72
2912		-----		-----	
3100	ISO18254-1	121.8		-0.23	
3116	ISO/DIS 18254-1	130		0.15	
3118	In house	90.89		-1.64	
3149	ISO18254-1	574	C,R(0.01)	20.45	first reported: 351
3154	In house	571.38	C,R(0.01)	20.33	first reported: 510.78
3160	ISO18254-1	234.0		4.90	
3172	ISO/DIS 18254-1	119.4		-0.33	
3176	ISO/DIS 18254-1	161.4	C	1.59	first reported: 256.40
3182	ISO/DIS 18254-1	110.61		-0.74	
3185	ISO18254-1	115.21		-0.53	
3197	ISO18254-1	102.5		-1.11	
3200		-----		-----	
3210		143.56		0.77	
3214	ISO18254-1	116.3		-0.48	
3218	ISO/DIS 18254-1	115.05		-0.53	
3220	ISO/DIS 18254-1	95.08		-1.45	
3222	ISO18254-1	201.3		3.41	
3232	ISO/DIS 18254-1	109.038		-0.81	
3237	ISO18254-1	175	C	2.21	first reported: 304
3248	In house	145		0.84	
3250	ISO18254-1	115.04		-0.53	

|				
normality	not OK			
n	90			
outliers	6			
mean (n)	126.727			
st.dev. (n)	34.6792	RSD = 27%		
R(calc.)	97.102			
st.dev.(Horwitz (n=5))	21.8756			
R(Horwitz (n=5))	61.252	compare R(ISO18254-1:16) = 34.803		
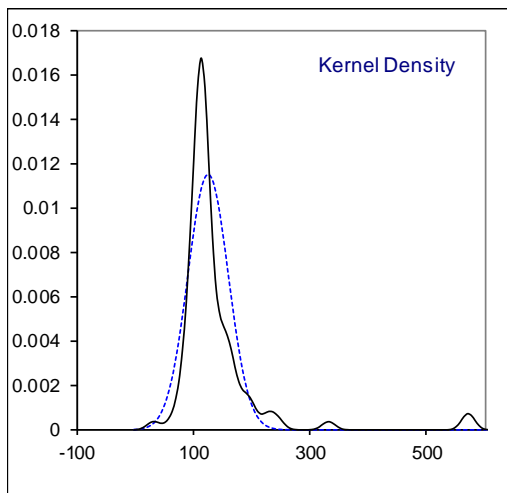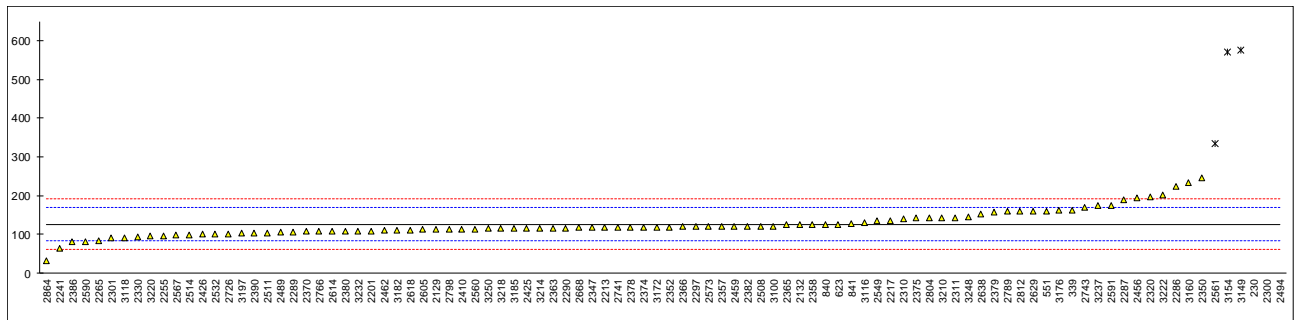
Determination of Total of OP, NP, OPEO and NPEO on sample #20531; results in mg/kg

lab	method	value	mark	z(targ)	remarks
210		-----		-----	
230	ISO18218-1	721.21	C,R(0.01)	26.91	first reported: 36.06
339		-----		-----	
551	In house	160.8377		1.49	
623	ISO18254-1	126.25		-0.08	
840	ISO18254-1	126.0		-0.09	
841	ISO18254-1	128.71		0.04	
2115		-----		-----	
2121		-----		-----	
2129	ISO18254-1	113.0		-0.68	
2132	In house	125.86		-0.09	
2201	ISO/DIS 18254-1	109.3		-0.84	
2213	ISO18254-1	118		-0.45	
2217	ISO/DIS 18254-1	136.5		0.39	
2241	ISO18254-1	64.3		-2.89	
2255	ISO/DIS 18254-1	96.3		-1.43	
2265	ISO18254-1	84		-1.99	
2286	ISO/DIS 18254-1	224.7		4.39	
2287	ISO18254-1	188.4		2.74	
2289	ISO18254-1	107		-0.95	
2290	ISO18254-1	116.9		-0.50	
2293		-----		-----	
2297	ISO18218-1	119.8		-0.37	
2300		1019	C,R(0.01)	40.41	first reported: 3541.39
2301	ISO18254-1	90.19	C	-1.71	first reported: 9.02
2310	ISO18254-1	140		0.55	
2311	ISO18254-1	143.59		0.71	
2320	ISO18254-1	197.87		3.17	
2330	ISO18254-1	93.22	C	-1.57	first reported: 42.47
2347	ISO18254-1	118		-0.45	
2350	ISO18254-1	246.72	C	5.39	first reported: 270.52
2352	ISO18254-1	119.49		-0.38	
2357	ISO/DIS 18254-1	120.0		-0.36	
2358	ISO/DIS 18254-1	125.92		-0.09	
2363	ISO/DIS 18254-1	116.9		-0.50	
2365	ISO/DIS 18254-1	124.54		-0.15	
2366	ISO/DIS 18254-1	119.6		-0.38	
2370	ISO18254-1	108		-0.90	
2374	GB/T23322	118.63		-0.42	
2375	ISO18254-1	142		0.64	
2378	ISO18254-1	118.52		-0.43	
2379		-----		-----	
2380	ISO/DIS 18254-1	108.6		-0.88	
2382	ISO18254-1	120.3		-0.35	
2386	ISO18254-1	81.18		-2.12	
2390	ISO18254-1	103.36		-1.11	
2410	ISO18254-1	114.32		-0.62	
2425	ISO18218-1	115.84		-0.55	
2426	ISO18254-1	100.24		-1.26	
2449		-----		-----	
2456	ISO/DIS 18254-1	194.9		3.04	
2459	ISO18254-1	120		-0.36	
2462		-----		-----	
2489	ISO/DIS 18254-1	105.8		-1.00	
2494	ISO18254-1	1605.7	C,R(0.01)	67.02	first reported: 280.9
2508		-----		-----	
2511	ISO18254-1	104.02		-1.08	
2514	ISO18254-1	98.07		-1.35	
2532	ISO18254-1	101.77		-1.19	
2549	ISO18254-1	135.4		0.34	
2560	ISO18254-1	114.36		-0.61	
2561		-----		-----	
2567	ISO18254-1	98.0		-1.36	
2573	ISO18254-1	119.8		-0.37	
2582		-----		-----	
2590	ISO/DIS 18254-1	81.930		-2.09	
2591		175.840		2.17	
2605	GB/T23972	112.32		-0.71	
2614	In house	108.52		-0.88	
2618		-----		-----	
2629		-----		-----	
2638	In house	183.888		2.54	
2644		-----		-----	
2668	ISO18254-1	117.28		-0.48	

lab	method	value	mark	z(targ)	remarks
2678		-----		-----	
2726		-----		-----	
2741	ISO18254-1	118.19		-0.44	
2743	ISO18254-1	192.7		2.94	
2766	ISO/DIS 18254-1	108.4		-0.89	
2789	ISO18254-1	158.98		1.41	
2798		-----		-----	
2804	ISO18254-1	143		0.68	
2812		-----		-----	
2864		-----		-----	
2912		-----		-----	
3100	ISO18254-1	121.8		-0.28	
3116		-----		-----	
3118	In house	90.89		-1.68	
3149		-----		-----	
3154	In house	586.18	C,R(0.01)	20.78	first reported: 525.58
3160	ISO18254-1	234.0		4.81	
3172	ISO/DIS 18254-1	119.4		-0.39	
3176		-----		-----	
3182	ISO/DIS 18254-1	110.61		-0.78	
3185	ISO18254-1	115.21		-0.58	
3197	ISO18254-1	<10		<-5.35	possible a false negative test result?
3200		-----		-----	
3210		-----		-----	
3214	ISO18254-1	116.3		-0.53	
3218		-----		-----	
3220	ISO/DIS 18254-1	95.08		-1.49	
3222	ISO18254-1	201.3		3.33	
3232		-----		-----	
3237	ISO18254-1	175	C	2.14	first reported: 304
3248	In house	145		0.77	
3250	ISO18254-1	115.04		-0.58	

normality	not OK			
n	74			
outliers	4			
mean (n)	127.915			
st.dev. (n)	36.0756	RSD = 28%		
R(calc.)	101.012			
st.dev.(Horwitz (n=5))	22.0497			
R(Horwitz (n=5))	61.739		compare R(ISO18254-1:16) = 35.130	

Kernel Density
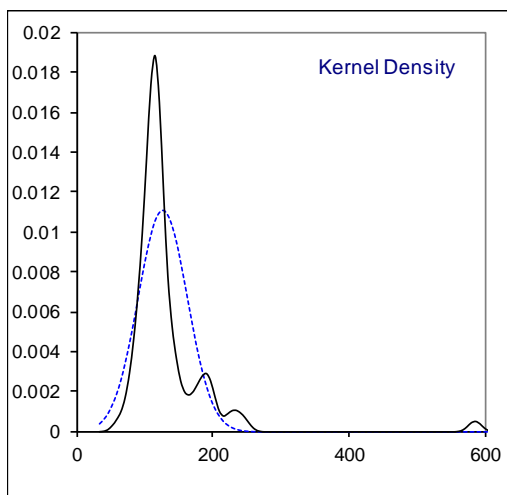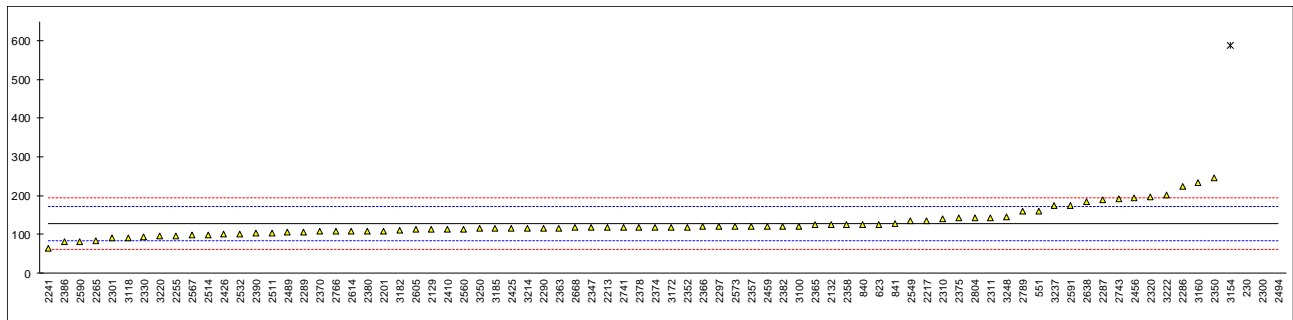
APPENDIX 2

Summary of other reported components in sample #20530 and #20531

lab	#20530 OP	NP	NPEO	#20531 OP	NP	OPEO
210	-----	-----	-----	-----	-----	-----
230	-----	-----	-----	-----	-----	-----
339	<10	<10	<10	<10	<10	<10
551	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
623	ND	ND	ND	ND	ND	ND
840	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
841	ND	ND	ND	ND	ND	ND
2115	-----	-----	-----	-----	-----	-----
2121	-----	-----	-----	-----	-----	-----
2129	<5	<5	<5	<5	<5	<5
2132	<10	<10	<10	<10	<10	<10
2201	ND	ND	ND	ND	ND	ND
2213	<10	<10	ND	<10	<10	<10
2217	-----	-----	-----	-----	-----	-----
2241	<10	<10	<10	<10	<10	<10
2255	n.d	n.d	n.d	n.d	n.d	n.d
2265	< 3	< 3	< 20	< 3	< 3	< 20
2286	<3	<3	<10	<3	<3	<10
2287	-----	-----	-----	-----	-----	-----
2289	<10	<10	<10	<10	<10	<10
2290	<10	<10	<10	<10	<10	<10
2293	-----	-----	-----	-----	-----	-----
2297	nd	nd	nd	nd	nd	nd
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
2320	N.D[<3]	N.D[<3]	N.D[<10]	N.D[<3]	N.D[<3]	N.D[<10]
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	-----	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----	-----
2358	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
2363	<10	<10	<10	<10	<10	<10
2365	<10	<10	<10	<10	<10	<10
2366	<50	<50	<50	<50	<50	<50
2370	< 1	< 1	< 1	< 1	< 1	< 1
2374	ND	ND	ND	ND	ND	ND
2375	-----	-----	-----	-----	-----	-----
2378	-----	-----	-----	-----	-----	-----
2379	Not detected	Not detected	1.29	Not detected	Not detected	0.89
2380	<3.0	<3.0	<3.0	<3.0	<3.0	<1.0
2382	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0
2386	< 5	<5	< 10	< 5	< 5	< 10
2390	nd	ND	ND	ND	ND	ND
2410	-----	-----	-----	-----	-----	-----
2425	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2426	ND	ND	ND	ND	ND	ND
2449	-----	-----	-----	-----	-----	-----
2456	-----	-----	-----	-----	-----	-----
2459	ND	ND	ND	ND	ND	ND
2462	-----	-----	-----	-----	-----	-----
2489	ND	ND	ND	ND	ND	ND
2494	ND	ND	ND	ND	ND	ND
2508	-----	-----	-----	-----	-----	-----
2511	-----	-----	-----	-----	-----	-----
2514	-----	-----	-----	-----	-----	-----
2532	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2549	ND	ND	ND	ND	ND	ND
2560	ND	ND	ND	ND	ND	ND
2561	-----	-----	<10	-----	-----	<10
2567	<10	<10	<10	<10	<10	<10
2573	ND	ND	ND	ND	ND	ND
2582	-----	-----	-----	-----	-----	-----
2590	-----	-----	-----	-----	-----	-----
2591	<10.0	<10.0	<10.0	<10.0	<10.0	<10.0
2605	ND	ND	ND	ND	ND	ND
2614	Not detected	Not detected	Not detected	Not Detected	Not Detected	Not Detected
2618	-----	-----	-----	-----	-----	-----
2629	< 1 mg/kg	< 1 mg/kg	< 10 mg/kg	< 1 mg/kg	< 1 mg/kg	< 10 mg/kg
2638	-----	-----	-----	-----	31.298	-----
2644	-----	-----	-----	-----	-----	-----

lab	#20530			#20531		
	OP	NP	NPEO	OP	NP	OPEO
2668	Not detected	Not detected	Not detected	Not detected	Not detected	Not detected
2678	-----	-----	-----	-----	-----	-----
2726	-----	-----	-----	-----	-----	-----
2741	<3	<3	<3	<3	<3	<3
2743	12.9	0.2	1.3	0.5	21.5	-----
2766	ND	ND	ND	ND	ND	ND
2789	<1	<1	<10	<1	<1	<10
2798	-----	-----	-----	-----	-----	-----
2804	<5	<5	<10	<5	<5	<10
2812	-----	-----	-----	-----	-----	-----
2864	-----	-----	-----	-----	-----	-----
2912	-----	-----	-----	-----	-----	-----
3100	<3	<3	<10	<3	<3	<10
3116	-----	-----	-----	-----	-----	-----
3118	ND	ND	ND	ND	ND	ND
3149	-----	-----	-----	-----	-----	-----
3154	-----	-----	-----	-----	-----	14.80
3160	<10	<10	<10	<10	<10	<10
3172	< 10	< 10	< 10	< 10	< 10	< 10
3176	-----	-----	-----	-----	-----	-----
3182	<3	<3	<3	<3	<3	<3
3185	<10	<10	<10	<10	<10	<10
3197	<10	<10	<10	<10	<10	<10
3200	-----	-----	-----	-----	-----	-----
3210	<20	<20	<20	<20	<20	<20
3214	<1	<1	<1	<1	<1	<1
3218	-----	-----	-----	-----	-----	-----
3220	ND	ND	ND	ND	ND	ND
3222	-----	-----	-----	-----	-----	-----
3232	-----	-----	-----	-----	-----	-----
3237	-----	-----	-----	-----	-----	-----
3248	-----	-----	-----	-----	-----	-----
3250	-----	-----	-----	-----	-----	-----

Abbreviations of components:

OP =   Octylphenol

NP =   Nonylphenol

OPEO =   Octylphenol Ethoxylates

NPEO =   Nonylphenol Ethoxylates

APPENDIX 3

Analytical Details

Lab	ISO/IEC17025 accredited for reported components(s)	sample intake (grams)	releasing/extraction technique	release solvent	extraction time (min)	extraction temperature (°C)
210	---		---			
230	Yes	0.5 g	Ultrasonic	Methanol	60	60
339	No		---			
551	Yes	1.0	Ultrasonic	Methanol	60	70
623	---	1 gram	---	Methanol	60 minutes	70°C
840	Yes	0.5g	Ultrasonic	MEOH	60 minutes	70°C
841	Yes	0.5G	Ultrasonic	AP: THF/CAN, APEO:MeOH	60 MIN	70
2115	---		---			
2121	---		---			
2129	Yes	0,5 g	Ultrasonic	MeOH	60 min	70°C
2132	No	0.5g	Ultrasonic	Methanol	60 minutes	70°C
2201	Yes	1 gram	Ultrasonic	Methanol	60min	70°C
2213	Yes	1 gm	Ultrasonic	Methanol	60 mins	70
2217	Yes	0.5 g	Ultrasonic	MeOH	60 min	70
2241	Yes	0.5g	Ultrasonic	Methanol	60min	70°C
2255	Yes	0.5	Ultrasonic	Methanol	60	70
2265	Yes	0,5 g	Ultrasonic	Methanol	60min	70°C
2286	No	1g	Ultrasonic	Methaol	60min	70°C
2287	No	1.0 g	Ultrasonic	Methanol	60 min	70°C
2289	Yes	1g	Ultrasonic	methanol	60mins	70°C
2290	Yes		---			
2293	---		---			
2297	Yes	1.0	Ultrasonic	methanol	60	70
2300	---		---			
2301	No	1 gram	Ultrasonic	Methanol	60 min	70 C
2310	Yes	1 gram	Ultrasonic	Methanol	60	70
2311	Yes	1	Ultrasonic	Methanol	60	70
2320	Yes	1grams	Ultrasonic	Methanol	60minutes	70°C
2330	Yes	1 g	Ultrasonic	MeOH	60 min	70 ± 2°C
2347	Yes	0.5g	Ultrasonic	methanol	60min	70°C
2350	No	1 g	Ultrasonic	Methanol, THF/ACN=1:2	60 minutes	70°C
2352	Yes	1g	Ultrasonic	Methanol	60min	70°C
2357	---		---			
2358	Yes	0.5 grams	Ultrasonic	Methanol	60 mins	70°C
2363	Yes	1g	Ultrasonic	MeOH	60	70°C
2365	Yes	5mm*5mm	Ultrasonic	methanol	60min	70°C
2366	No	0.5	Ultrasonic	methanol	60	70°C
2370	Yes	0.5 g	Ultrasonic	Methanol	60 min	70°C
2374	No	1g	Ultrasonic	20mL methanol	1 h	70°C
2375	Yes	0,5 g	Ultrasonic	Methanol	60 min	70°C
2378	Yes	1g	Ultrasonic	Methanol	60	70
2379	Yes	0.5 g	Ultrasonic	Methanol	60 minutes	70°C
2380	Yes	1.00 g	Ultrasonic	Methanol	60 Minutes	70 °C
2382	Yes	1.0g	Ultrasonic	methanol	60min±5min	70°C±2°C
2386	Yes	0,5	Ultrasonic	Methanol	60 min	70 °C
2390	Yes	1 gram	Ultrasonic	Methanol	60min	70°C
2410	Yes	0.5 g	Ultrasonic	Methanol	60 min	70°C
2425	Yes	1.0 gm	Ultrasonic	Methanol	60± 5 minutes	(70± 5)°C
2426	Yes	0.5 g	Ultrasonic	Methanol	60 minutes	70°C
2449	---		---			
2456	Yes	1 g	Ultrasonic	Methanol, 20 ml	60	70
2459	No	1.0 g	Ultrasonic	Methanol	60	70
2462	---		---			
2489	Yes	20530: 1.0010g/20531: 1.0015g	Ultrasonic	Methanol/Water	60 Minutes	70°C
2494	No	1 Gram	Ultrasonic	Methanol	60 minutes	70
2508	Yes	0.5	Ultrasonic	MEOH	60	60
2511	No	1 g	Ultrasonic	Methanol	60	60
2514	Yes	0.5130 g	Ultrasonic	Methanol	60	70
2532	Yes	0.5 grams	Ultrasonic	Methanol	60 minutes	70 °C
2549	Yes	0.5 grams	Ultrasonic	Methanol	60 mins	70°C
2560	Yes	1 gm	Ultrasonic	Methanol	60 min	70
2561	Yes	1.0	Ultrasonic	methanol	60	70
2567	Yes	0.5	Ultrasonic	Methanol	60	70
2573	---	1g	Ultrasonic	Methanol	60minutes	70°C
2582	---		---			
2590	Yes	1g	Ultrasonic	meoH	60 min	60°C
2591	No	1.00 grams	Ultrasonic	Methanol	60 min	40ºC
2605	Yes	0.500	Ultrasonic	Methanol	30	70°C

Lab	ISO/IEC17025 accredited for reported components(s)	sample intake (grams)	releasing/extraction technique	release solvent	extraction time (min)	extraction temperature (°C)
2614	Yes	1.0008grams	Ultrasonic	methanol	60mins	70°C
2618	Yes	1 gm	Ultrasonic	Methanol	60 minute	70 °C
2629	Yes	1.0g	Ultrasonic	Methanol (MeOH)	60	70
2638	No	1 gm	Ultrasonic	methanol	60 min	room temperature
2644	---		---			
2668	Yes	0.5 gms	Ultrasonic	Methanol	60 Min	70
2678	---		---			
2726	Yes	each sample 1g	Ultrasonic	methanol	60 minutes	70C
2741	Yes	0.5	Ultrasonic	Methanol	60	70
2743	Yes	1 grams	Ultrasonic	MeOH	60 minutes	70°C
2766	Yes	1.0 GMS	Ultrasonic	METHANOL	60MIN	65 -70°C
2789	Yes	1	Ultrasonic	Methanol	60	70
2798	Yes	0.5g	Ultrasonic	MEOH	60min	70°C
2804	Yes	0.5	Ultrasonic	Methanol	60	70
2812	Yes	1 gram	Ultrasonic	methanol	60 minutes	70
2864	Yes	0.5	Ultrasonic	Methanol	60	70
2912	---		---			
3100	Yes	Sample #20530:0.490 grams Sample #20531:0.495 grams	Ultrasonic	Methanol	60 minutes	70°C
3116	Yes	1 gram	Ultrasonic	Methanol	60 minutes	70°C
3118	Yes	0.5 g (±0.01 g)	Ultrasonic	methanol	60 minutes	70° C
3149	Yes	aboud 1g	Ultrasonic	methanol	aboud 1h	around 70°C
3154	Yes		Ultrasonic			
3160	No	1 gr	Ultrasonic	MeOH	60 minutes	70ºC
3172	Yes	1.5	Ultrasonic	Methanol	60	70
3176	Yes	1,0 g	Ultrasonic	Methanol	60 minutes	70°C
3182	No	0.1 gram	Ultrasonic	Methanol 20 mL when finished extract add water 5 mL	60	70
3185	Yes	1g	Ultrasonic	Methanol	60 mins	70℃
3197	Yes	0,5 grams	Ultrasonic	Metanol	60 minutes	70 C
3200	---		---			
3210	No	1	Ultrasonic	Methanol	60	70
3214	Yes	1 g	Ultrasonic	MeOH	1 hr	70°C
3218	Yes	0.5g	Ultrasonic	Methanol	60min	70°C
3220	Yes	1gm	Ultrasonic	Methanol	60 min	70°C
3222	Yes	0.5g	Ultrasonic	Methanol	60 minutes	70 °C
3232	Yes	1	Ultrasonic	Methanol	30	room temperature
3237	Yes	0,5g	Ultrasonic	methonol	60min.	60°C
3248	Yes	0.5	Ultrasonic	Methanol	60	70
3250	---		---			
210	---		---			

APPENDIX 4

Number of participants per country

  6 labs in BANGLADESH

  1 lab in BRAZIL

  3 labs in CAMBODIA

  3 labs in FRANCE

  6 labs in GERMANY

  1 lab in GUATEMALA

  5 labs in HONG KONG

  1 lab in HUNGARY

 12 labs in INDIA

  4 labs in INDONESIA

  8 labs in ITALY

  2 labs in JAPAN

  1 lab in MAURITIUS

  1 lab in MOROCCO

 21 labs in P.R. of CHINA

  5 labs in PAKISTAN

  2 labs in SOUTH KOREA

  3 labs in SPAIN

  2 labs in SRI LANKA

  3 labs in TAIWAN R.O.C.

  2 labs in THAILAND

  2 labs in TUNISIA

  6 labs in TURKEY

  1 lab in UNITED KINGDOM

  5 labs in VIETNAM

APPENDIX 5

Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= possibly an error in calculations
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported

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