Results of Proficiency Test
AP and APEO in Textile
March 2021

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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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 oestrogen-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 (iis) organizes a proficiency scheme for the analysis of AP and APEO in Textile every year. During the annual proficiency testing program 2020/2021 it was decided to continue the proficiency test (PT) for the analysis of AP and APEO in textile.

In this interlaboratory study 101 laboratories in 23 different countries registered for participation. See appendix 4 for the number of participants per country. In this report the results of the AP and APEO in textile 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 #21525 and #21526. The samples were positive on OP, 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

For the first sample a batch of brown cotton was selected which was made positive on OP and OPEO by a third-party laboratory. This batch was cut into small pieces. After homogenization the batch was divided over 120 subsamples in small bags of approximately 3 grams each and labelled #21525.
The homogeneity of the subsamples was checked by determination of OP and OPEO using an in-house test method on eight stratified randomly selected subsamples.

	OP in mg/kg	OPEO in mg/kg
Sample #21525-1	40.41	116.7
Sample #21525-2	41.49	115.1
Sample #21525-3	40.15	107.6
Sample #21525-4	36.96	108.5
Sample #21525-5	39.98	110.8
Sample #21525-6	41.95	115.2
Sample #21525-7	42.79	116.5
Sample #21525-8	41.88	113.0

Table 1: homogeneity test results of subsamples #21525

From the above test results the repeatabilities were calculated and compared with 0.3 times the estimated reproducibilities calculated with the Horwitz equation in agreement with the procedure of ISO13528, Annex B2 in the next table.

	OP in mg/kg	OPEO in mg/kg
r (observed)	5.06	10.0
reference method	Horwitz (n=5)	Horwitz (n=5)
0.3 x R (reference method)	7.00	16.7

Table 2: evaluation of repeatabilities of subsamples #21525

The calculated repeatabilities are in agreement with 0.3 times the estimated reproducibilities calculated with the Horwitz equation. Therefore, homogeneity of the subsamples was assumed.

For the second sample a batch of beige/gray cotton was selected which was made positive on NPEO by a third-party laboratory. This batch was cut into small pieces. After homogenization the batch was divided over 120 subsamples in small bags of approximately 3 grams each and labelled #21526.
The homogeneity of the subsamples was checked by determination of NPEO using an in-house test method on seven stratified randomly selected subsamples.

	NPEO in mg/kg
Sample #21526-1	145.8
Sample #21526-2	140.9
Sample #21526-3	137.4
Sample #21526-4	129.5
Sample #21526-5	147.4
Sample #21526-6	147.6
Sample #21526-7	145.6

Table 3: homogeneity test results of subsamples #21526

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

	NPEO in mg/kg
r (observed)	18.7
reference method	Horwitz (n=5)
0.3 x R (reference method)	20.2

Table 4: evaluation of the repeatability of subsamples #21526

The calculated repeatability is in agreement with 0.3 times the estimated reproducibility calculated with the Horwitz equation. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one textile sample labelled #21525 and one textile sample labelled #21526 were sent on February 10, 2021.

2.5 ANALYZES

The participants were requested to determine on samples #21525 and #21526 the concentrations of Octylphenol (OP), Nonylphenol (NP), Octylphenol Ethoxylates (OPEO), Nonylphenol Ethoxylates (NPEO) and the Total of OP, NP, OPEO and NPEO.
To ensure homogeneity it was requested 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 (when applicable) 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 presented by their 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 data analysis and the original test results are placed under 'Remarks' in the result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report ‘iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation’ of June 2018 (iis-protocol, version 3.5).
For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as ‘<…’ or ‘>…’ were not used in the statistical evaluation.

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

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. 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 test 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. This 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 (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

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, e.g. ISO reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the 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, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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 according to:

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

The $z_{(target)}$ scores are listed in the test result tables in appendix 1.

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

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

4 EVALUATION

In this proficiency test some problems were encountered with the dispatch of the samples. Fourteen participants reported test results after the final reporting date and one other participant did not report any test results. Not all participants were able to report all tests requested.
In total 100 participants reported 444 numerical test results. Observed were 15 outlying test results, which is 3.4%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

Not all 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 test methods are also in the tables 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 #21525

OP: This determination was not problematic. Five statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated reproducibility calculated with the Horwitz equation for 5 components.

OPEO: This determination was not problematic. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated reproducibility calculated with the Horwitz equation for 5 components.

Total OP + NP + OPEO + NPEO: The determination of this sum-component was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated reproducibility calculated with the Horwitz equation for 5 components.

Most participants agreed that the levels for the components NP and NPEO in this sample were near or below the detection limit and therefore no z-scores were calculated. See appendix 2 for the reported test results.

sample #21526

NPEO: This determination was not problematic. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated reproducibility calculated with the Horwitz equation for 5 components.

Total OP + NP + OPEO + NPEO: The determination of this sum-component was not evaluated separately because only one component (NPEO) was detected.

Most participants agreed that the levels for the components OP, NP and OPEO in this sample were near or below the detection limit and therefore no z-scores were calculated. See appendix 2 for the reported test results.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the estimated target reproducibility calculated with 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 target reproducibility estimated using the Horwitz equation are presented in the next tables.

Component	unit	n	average	2.8 * sd	R(target)
OP	mg/kg	83	43.4	14.5	24.6
OPEO	mg/kg	96	110.6	38.0	54.5
Total OP + NP + OPEO + NPEO	mg/kg	76	151.9	51.7	71.5

Table 5: reproducibilities of components on sample #21525

Component	unit	n	average	2.8 * sd	R(target)
NPEO	mg/kg	96	130.3	55.0	62.7

Table 6: reproducibilities of components on sample #21526

Without further statistical calculations, it can be concluded that for all tests there is a good compliance of the group of participants with the reference method.

4.3 COMPARISON OF THE PROFICIENCY TEST OF MARCH 2021 WITH PREVIOUS PTS

	March 2021	March 2020	February 2019	February 2018	March 2017
Number of reporting laboratories	100	96	105	92	95
Number of test results	444	347	366	329	378
Number of statistical outliers	15	17	21	8	9
Percentage of statistical outliers	3.4%	4.9%	5.7%	2.4%	2.4%

Table 7: comparison with previous proficiency tests

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, in the next table.

	March 2021	March 2020	February 2019	February 2018	2017-2016	Horwitz 500-1500 mg/kg
Octylphenol (OP)	12%	n.e.	n.e.	n.e.	n.e.	n.e
Octylphenol Ethoxylates (OPEO)	12%	17%	10%	16%	15-16%	18-12%
Nonylphenol Ethoxylates (NPEO)	15%	27%	13%	28%	18-27%	18-12%
Total OP + NP + OPEO + NPEO	12%	16-28%	11%	15-26%	15-25%	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.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

The participants were asked to provide some analytical details which are listed in appendix 3. Based on the reported answers the following can be summarized:

- 89% mentioned that they are ISO/IEC17025 accredited to determine the reported components.
- 60% further cut the samples prior to analysis, 37% used the samples as received and 3% further grinded the sample or did other type of preparation
- 95% used between 0.5 - 1 grams of sample intake; 37% around 0.5 grams and 58% around 1 grams.
- 97% used Ultrasonic technique to extract/release the components from the samples.
- 98% used Methanol as extraction solvent.
- Almost all participants used an extraction/release time of 60 minutes and 93% used an extraction/release temperature of 70°C, 7% used a lower temperature.

The effect of sample preparation prior to analysis and amount of sample intake on the determinations of Octylphenol (OP) in sample #21525 was further investigated in the next table.

Analytical Details	unit	n	average	RSD
Used as received	mg/kg	28	42.27	15%
Further cut	mg/kg	47	43.97	10%
around 1 g sample intake	mg/kg	41	43.23	10%
around 0.5 g sample intake	mg/kg	30	44.03	13%

Table 9: effect of sample preparation and amount of sample intake on OP in textile sample #21525

While the effect of the analytical details on the determination of OP is not statistically significant it seems that further cutting the sample prior to analysis yield a slightly higher OP level with less variation.

5 DISCUSSION

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 10), it is noticed that all, but four of the reporting laboratories would reject sample #21525 for containing too much OP, all but seventeen laboratories would reject sample #21525 for containing too much OPEO and all but one laboratory would reject sample #21525 for containing too much of the total of OP + NP + OPEO + NPEO.
For sample #21526 it is noticed that all, but two of the reporting laboratories would reject the sample for containing too much NPEO.

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

	OEKO-TEX	Blue Sign BSSL v11.0	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 10: 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.

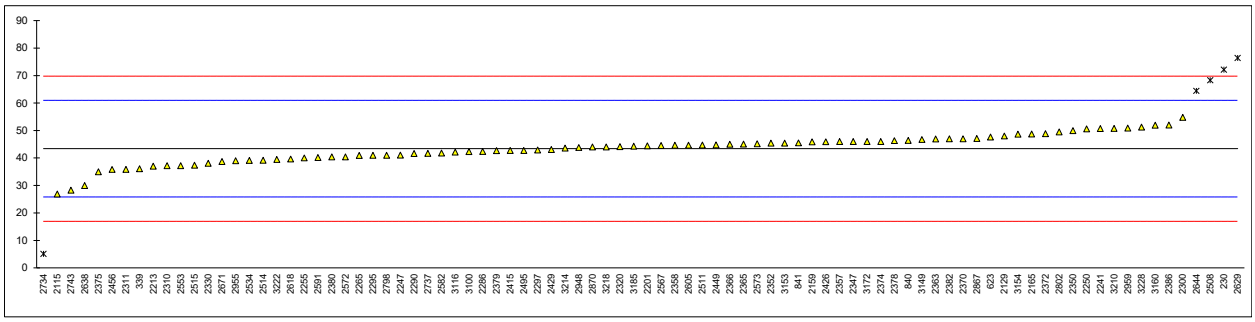
APPENDIX 1

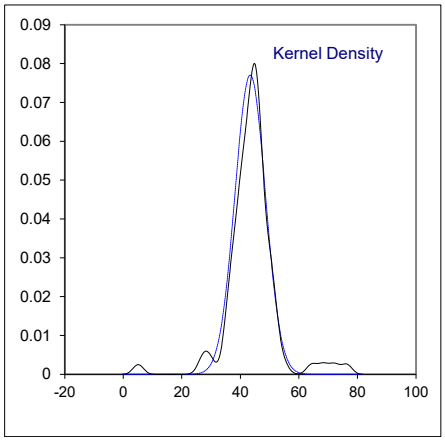
Determination of Octylphenol (OP) on sample #21525; results in mg/kg

lab	method	value	mark	z(targ)	remarks
230	ISO18254-1	72.1721	C,R(0.01)	3.27	first reported Not Detected
339	ISO18254-1Mod.	36.083		-0.83	
551		-----		-----	
623	ISO/DIS 18254-1	47.62	C	0.48	first reported not detected
840	In house	46.40		0.34	
841	ISO18254-1	45.495		0.24	
2115	ISO21084	26.87		-1.88	
2129	ISO/DIS 18254-2	48		0.52	
2159	ISO18254-1	45.83		0.28	
2165	ISO18254-1	48.7		0.60	
2201	ISO18254-1	44.40		0.11	
2213	ISO18254-1	37		-0.73	
2241	ISO18254-1	50.72		0.83	
2247	ISO18254-1	41.04		-0.27	
2250	In house	50.6	C	0.82	first reported 62.04
2255	ISO/DIS 18254-1	40.0		-0.39	
2265	ISO18254-1	40.95		-0.28	
2286	ISO18254-1	42.36		-0.12	
2290	ISO18254-1	41.6		-0.20	
2293		-----		-----	
2295	ISO/DIS 18254-1	41		-0.27	
2297	ISO18254-1	42.9		-0.06	
2300	ISO18254-1	54.81		1.30	
2310	ISO18254-1	37.2		-0.70	
2311	ISO18254-1	35.882		-0.85	
2320	ISO18254-1	44.1228		0.08	
2330	ISO/DIS 18254-1	38.055		-0.61	
2347	ISO/DIS 18254-1	46		0.30	
2350	ISO18254-1	49.92		0.74	
2352	ISO18254-1	45.4		0.23	
2357	ISO18254-1	46.0		0.30	
2358	ISO18254-1	44.66		0.14	
2363	ISO18254-1	46.92		0.40	
2365	ISO/DIS 18254-1	45.1		0.19	
2366	ISO18254-1	45		0.18	
2370	ISO18254-1	47.0		0.41	
2372	ISO/DIS 18254-1	48.86		0.62	
2374	ISO18254-1	46.02		0.30	
2375	ISO18254-1	35		-0.95	
2378	ISO18254-1	46.3		0.33	
2379	ISO18254-1	42.664		-0.08	
2380	ISO18254-1	40.38		-0.34	
2382	ISO18254-1	47.0		0.41	
2386	ISO18254-1	52.01		0.98	
2390	ISO/DIS 18254-1	Not Detected	C	-----	first reported 14.47
2415	ISO18254-1	42.71		-0.08	
2426	ISO18254-1	45.915		0.29	
2429	ISO/DIS 18254-1	43.1		-0.03	
2449	ISO21084	44.8		0.16	
2452		-----		-----	
2456	ISO21084	35.8		-0.86	
2495	ISO18254-1	42.75		-0.07	
2508		68.32	R(0.01)	2.83	
2511	ISO18254-1	44.7		0.15	
2514	ISO/DIS 18254-1	39.19		-0.48	
2515	ISO18254-1	37.37		-0.68	
2534	ISO21084	39.1		-0.49	
2536	ISO21084	Not Detected		-----	
2553	ISO/DIS 18254-1	37.20		-0.70	
2561		-----		-----	
2567	ISO18254-1	44.5		0.13	
2572	ISO/DIS 18254-1	40.4		-0.34	
2573	ISO/DIS 18254-1	45.2		0.21	
2582	In house	41.822		-0.18	
2590		-----		-----	
2591	In house	40.15		-0.37	
2605	GB/T23972	44.66		0.14	
2618	ISO/DIS 18254-1	39.6	C	-0.43	first reported not detected
2629	ISO21084	76.4	R(0.01)	3.75	
2638	In house	29.97		-1.52	
2643		-----		-----	
2644	ISO21084	64.4	R(0.05)	2.39	
2671	ISO18254-1	38.71		-0.53	

lab	method	value	mark	z(targ)	remarks
2678	ISO21084	not detected		-----	
2734	ISO/DIS 18254-1	5.127	C,R(0.01)	-4.35	first reported 10.254
2737	ISO21084	41.638		-0.20	
2743	ISO18254-1	28.285	C	-1.72	first reported 17.468
2789		-----		-----	
2798	ISO18254-1	41		-0.27	
2802	ISO18254-1	49.5		0.69	
2864		-----		-----	
2867	ISO18254-1	47.1		0.42	
2870	ISO18254-1	44		0.07	
2948	ISO21084	43.78		0.04	
2955	ISO/DIS 18254-1	39.0		-0.50	
2959	ISO18254-1	50.87		0.85	
3100	GB/T23322	42.312		-0.12	
3116	ISO18254-1	42.18		-0.14	
3149	ISO/DIS 18254-1	46.7		0.38	
3153		45.4		0.23	
3154	In house	48.68	C	0.60	first reported 16.46
3160	ISO18254-1	51.95		0.97	
3172	ISO18254-1	46		0.30	
3176		-----		-----	
3185	ISO18254-1	44.31		0.10	
3210	In house	50.76		0.84	
3214	ISO18254-1	43.61		0.03	
3218	ISO18254-1	44.00		0.07	
3222	ISO21084	39.47		-0.45	
3228	ISO/DIS 18254-1	51.2		0.89	
3237		-----		-----	

	normality	suspect	
	n	83	
	outliers	5	
	mean (n)	43.389	
	st.dev. (n)	5.1780	RSD=12%
	R(calc.)	14.498	
	st.dev.(Horwitz (n=5))	8.8010	
	R(Horwitz (n=5))	24.643	
Compare			
	R(ISO18254-1:16)	11.916	

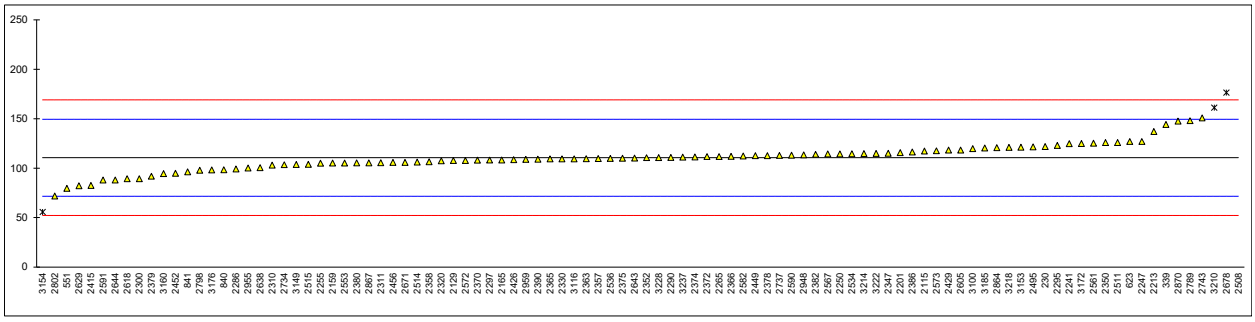


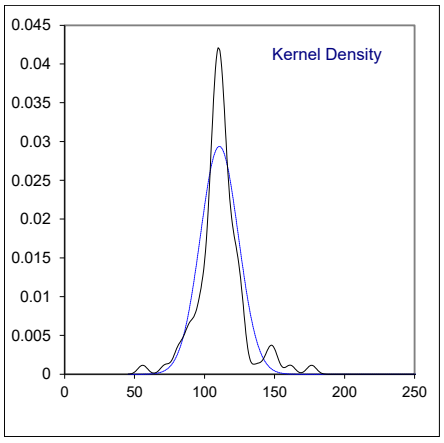


Determination of Octylphenol Ethoxylates (OPEO) on sample #21525; results in mg/kg

lab	method	value	mark	z(targ)	remarks
230	ISO18254-1	121.7864271		0.58	
339	ISO18254-1Mod.	144.08		1.72	
551	ISO18254-1	79.59		-1.59	
623	ISO/DIS 18254-1	126.930		0.84	
840	In house	98.30		-0.63	
841	ISO18254-1	96.18		-0.74	
2115	ISO18254	117.33		0.35	
2129	ISO/DIS 18254-2	107.8		-0.14	
2159	ISO18254-1	105.17		-0.28	
2165	ISO18254-1	108.3		-0.12	
2201	ISO18254-1	115.75		0.27	
2213	ISO18254-1	137		1.36	
2241	ISO18254-1	124.80		0.73	
2247	ISO18254-1	126.96		0.84	
2250	In house	114.43		0.20	
2255	ISO/DIS 18254-1	105.10		-0.28	
2265	ISO18254-1	111.75		0.06	
2286	ISO18254-1	99.15		-0.59	
2290	ISO18254-1	110.9		0.02	
2293		-----		-----	
2295	ISO/DIS 18254-1	123		0.64	
2297	ISO18254-1	108.2		-0.12	
2300	ISO18254-1	89.32		-1.09	
2310	ISO18254-1	103		-0.39	
2311	ISO18254-1	105.46		-0.26	
2320	ISO18254-1	107.5024		-0.16	
2330	ISO/DIS 18254-1	109.475		-0.06	
2347	ISO/DIS 18254-1	115		0.23	
2350	ISO18254-1	125.88		0.79	
2352	ISO18254-1	110.6		0.00	
2357	ISO18254-1	109.8		-0.04	
2358	ISO18254-1	106.45		-0.21	
2363	ISO18254-1	109.6		-0.05	
2365	ISO/DIS 18254-1	109.3		-0.06	
2366	ISO18254-1	112		0.07	
2370	ISO18254-1	108		-0.13	
2372	ISO/DIS 18254-1	111.7		0.06	
2374	ISO18254-1	111.23		0.03	
2375	ISO18254-1	110		-0.03	
2378	ISO18254-1	112.6		0.10	
2379	ISO18254-1	91.776		-0.96	
2380	ISO18254-1	105.3		-0.27	
2382	ISO18254-1	114.0		0.18	
2386	ISO18254-1	116.33		0.30	
2390	ISO/DIS 18254-1	109.05		-0.08	
2415	ISO18254-1	82.51		-1.44	
2426	ISO18254-1	108.654		-0.10	
2429	ISO/DIS 18254-1	118.2		0.39	
2449	ISO21084	112.51		0.10	
2452	ISO18254-1	94.864		-0.81	
2456	ISO18254-1	105.7		-0.25	
2495	ISO18254-1	121.50		0.56	
2508		362.33	R(0.01)	12.92	
2511	ISO18254-1	126		0.79	
2514	ISO/DIS 18254-1	106.19		-0.22	
2515	ISO18254-1	103.92		-0.34	
2534	ISO18254-1	114.6		0.21	
2536	ISO18254-1	109.86		-0.04	
2553	ISO/DIS 18254-1	105.24		-0.27	
2561	ISO18254-2	125.213		0.75	
2567	ISO18254-1	114.4		0.20	
2572	ISO/DIS 18254-1	107.8		-0.14	
2573	ISO/DIS 18254-1	117.6		0.36	
2582	In house	112.3411		0.09	
2590	ISO/DIS 18254-1	113.093		0.13	
2591	In house	87.98		-1.16	
2605	GB/T23972	118.24		0.39	
2618	ISO/DIS 18254-1	89.27		-1.09	
2629	ISO18254-1	82.14		-1.46	
2638	In house	100.43		-0.52	
2643	ISO18254-1	110.19		-0.02	
2644	ISO18254-1	88.0		-1.16	
2671	ISO18254-1	105.82		-0.24	
2678	ISO18254-1	176.40	R(0.01)	3.38	

lab	method	value	mark	z(targ)	remarks
2734	ISO/DIS 18254-1	103.466		-0.36	
2737	ISO21084	112.794		0.11	
2743	ISO18254-1	150.863		2.07	
2789	ISO18254-1	148.0		1.92	
2798	ISO18254-1	98		-0.64	
2802	ISO18254-1	71.8		-1.99	
2864	ISO18254-1	120.66		0.52	
2867	ISO18254-1	105.3		-0.27	
2870	ISO18254-1	147.5		1.90	
2948		113.48		0.15	
2955	ISO/DIS 18254-1	100.2		-0.53	
2959	ISO18254-1	108.9		-0.09	
3100	GB/T23322	119.630		0.47	
3116	ISO18254-1	109.5		-0.05	
3149	ISO/DIS 18254-1	103.7		-0.35	
3153		121.3		0.55	
3154	In house	55.79	C,R(0.05)	-2.81	first reported 62.67
3160	ISO18254-1	94.49		-0.82	
3172	ISO18254-1	125		0.74	
3176	In house	98.20		-0.63	
3185	ISO18254-1	120.36		0.50	
3210	In house	161.17	R(0.05)	2.60	
3214	ISO18254-1	114.66		0.21	
3218	ISO18254-1	121.00		0.54	
3222	ISO18254-1	114.83		0.22	
3228	ISO/DIS 18254-1	110.8		0.01	
3237	ISO18254-1	111.12		0.03	
	normality	suspect			
	n	96			
	outliers	4			
	mean (n)	110.559			
	st.dev. (n)	13.5830	RSD=12%		
	R(calc.)	38.032			
	st.dev.(Horwitz (n=5))	19.4809			
	R(Horwitz (n=5))	54.546			
Compare					
	R(ISO18254-1:16)	30.363			



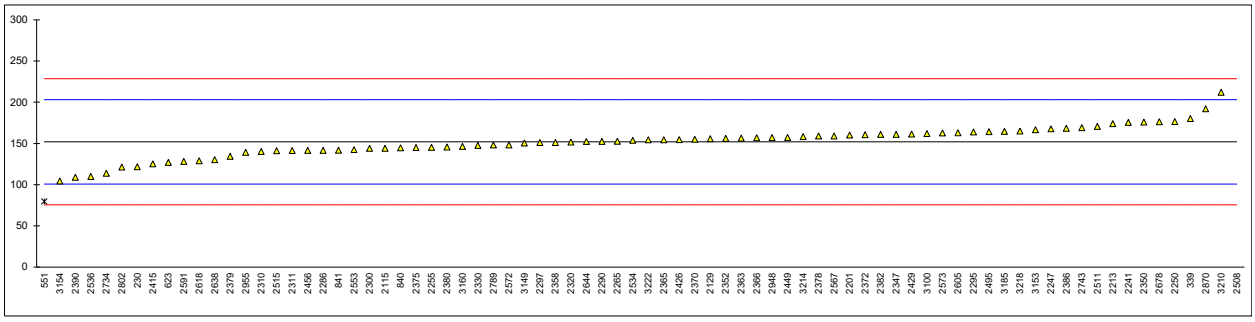


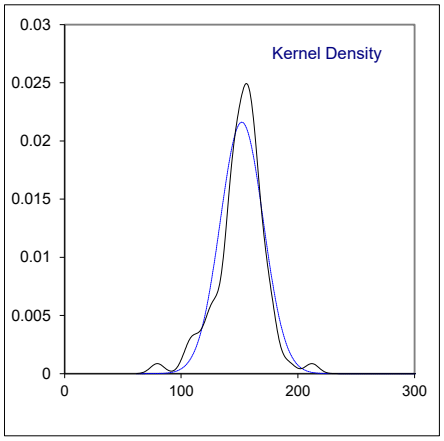
Determination of Total of OP, NP, OPEO and NPEO on sample #21525; results in mg/kg

lab	method	value	mark	z(targ)	remarks
230	ISO18254-1	121.7864271	E	-1.18	iis calc. 194.0, after OP correction the Total was not updated
339	ISO18254-1Mod.	180.16		1.11	
551	ISO18254-1	79.59	R(0.05)	-2.83	
623	ISO/DIS 18254-1	126.930	E	-0.98	iis calc. 174.6, after OP correction the Total was not updated
840	In house	144.70		-0.28	
841	ISO18254-1	141.675		-0.40	
2115		144.2		-0.30	
2129	ISO/DIS 18254-2	155.8		0.15	
2159		-----		-----	
2165		-----		-----	
2201	ISO18254-1	160.15		0.32	
2213	ISO18254-1	174		0.86	
2241	ISO18254-1	175.52		0.92	
2247	ISO18254-1	167.73		0.62	
2250	In house	176.47	E	0.96	iis calculated 165.0
2255	ISO/DIS 18254-1	145.10		-0.27	
2265	ISO18254-1	152.7		0.03	
2286	ISO18254-1	141.51		-0.41	
2290	ISO18254-1	152.5		0.02	
2293		-----		-----	
2295	ISO/DIS 18254-1	164		0.47	
2297	ISO18254-1	151.1		-0.03	
2300	ISO18254-1	144.13		-0.31	
2310	ISO18254-1	140.2		-0.46	
2311	ISO18254-1	141.342		-0.41	
2320	ISO18254-1	151.6252		-0.01	
2330	ISO/DIS 18254-1	147.530		-0.17	
2347	ISO/DIS 18254-1	161		0.36	
2350	ISO18254-1	175.80		0.94	
2352	ISO18254-1	156.0		0.16	
2357		-----		-----	
2358	ISO18254-1	151.11		-0.03	
2363	ISO18254-1	156.52		0.18	
2365	ISO/DIS 18254-1	154.4		0.10	
2366	ISO18254-1	157		0.20	
2370	ISO18254-1	155		0.12	
2372	ISO/DIS 18254-1	160.56		0.34	
2374		-----		-----	
2375		145		-0.27	
2378	ISO18254-1	158.9		0.27	
2379	ISO18254-1	134.440		-0.69	
2380	ISO18254-1	145.68		-0.24	
2382	ISO18254-1	161.0		0.36	
2386	ISO18254-1	168.34		0.64	
2390	ISO/DIS 18254-1	109.05	C	-1.68	first reported 123.52
2415	ISO18254-1	125.22		-1.05	
2426	ISO18254-1	154.569		0.10	
2429	ISO/DIS 18254-1	161.3		0.37	
2449		157.3		0.21	
2452		-----		-----	
2456	ISO21084/ISO18254-1	141.5		-0.41	
2495	ISO18254-1	164.25		0.48	
2508		430.65	R(0.01)	10.92	
2511	ISO18254-1	170.7		0.74	
2514		-----		-----	
2515	ISO18254-1	141.29		-0.42	
2534	ISO18254-1	153.7		0.07	
2536	ISO18254-1	109.86		-1.65	
2553	ISO/DIS 18254-1	142.44		-0.37	
2561		-----		-----	
2567	ISO18254-1	158.9		0.27	
2572	ISO/DIS 18254-1	148.2		-0.15	
2573	ISO/DIS 18254-1	162.8		0.43	
2582		-----		-----	
2590		-----		-----	
2591	In house	128.13		-0.93	
2605	GB/T23972	162.90		0.43	
2618	ISO/DIS 18254-1	128.87	C	-0.90	first reported 89.27
2629		-----		-----	
2638	In house	130.4		-0.84	
2643		-----		-----	
2644	ISO21084/ISO18254-1	152.4		0.02	
2671		-----		-----	
2678		176.40		0.96	

lab	method	value	mark	z(targ)	remarks
2734	ISO/DIS 18254-1	113.8	E	-1.49	iis calc. 108.6, after OP correction the Total was not updated
2737		-----		-----	
2743	ISO18254-1	169.192	E	0.68	iis calc. 179.1, after OP correction the Total was not updated
2789	ISO18254-1	148.0		-0.15	
2798		-----		-----	
2802	ISO18254-1	121.3		-1.20	
2864		-----		-----	
2867		-----		-----	
2870	ISO18254-1	192		1.57	
2948		157.126		0.20	
2955	ISO/DIS 18254-1	139.2		-0.50	
2959		-----		-----	
3100	GB/T23322	161.942		0.39	
3116		-----		-----	
3149	ISO/DIS 18254-1	150.4		-0.06	
3153		166.7		0.58	
3154	In house	104.47	C	-1.86	first reported 79.13
3160	ISO18254-1	146.43		-0.22	
3172		-----		-----	
3176		-----		-----	
3185	ISO18254-1	164.67		0.50	
3210	In house	211.93		2.35	
3214	ISO18254-1	158.27		0.25	
3218	ISO18254-1	165.00		0.51	
3222	ISO18254-1	154.30		0.09	
3228		-----		-----	
3237		-----		-----	

	normality	suspect
	n	76
	outliers	2
	mean (n)	151.928
	st.dev. (n)	18.4631
	R(calc.)	51.697
	st.dev.(Horwitz (n=5))	25.5196
	R(Horwitz (n=5))	71.455
Compare		
	R(ISO18254-1:16)	41.724



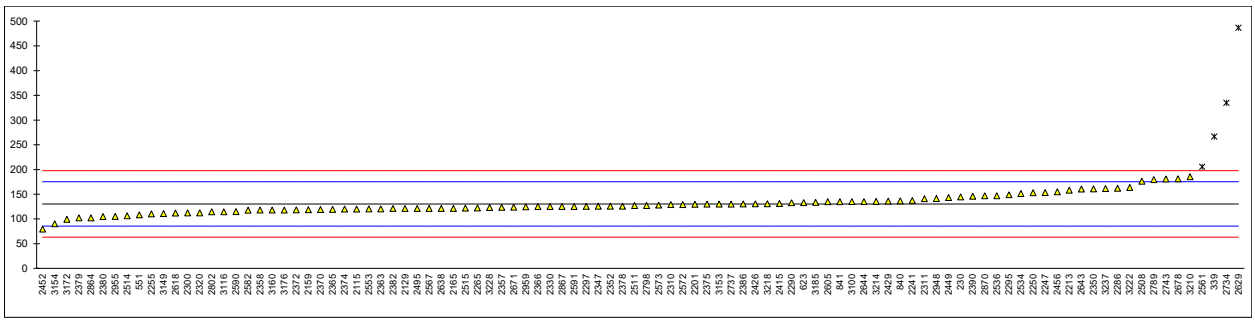


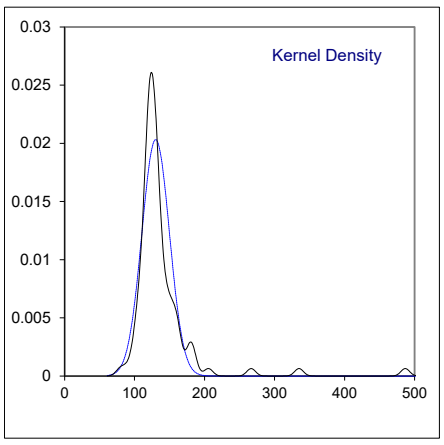
Determination of Nonylphenol Ethoxylates (NPEO) on sample #21526; results in mg/kg

lab	method	value	mark	z(targ)	Total OP,NP, OPEO,NPEO		remarks
230	ISO18254-1	144.813154		0.65	144.813154		
339		266.69	R(0.01)	6.09	266.69		
551	ISO18254-1	108.41		-0.98	108.41		
623	ISO/DIS 18254-1	133.200		0.13	133.200		
840	In house	136.4		0.27	136.4		
841	ISO18254-1	134.9		0.21	134.9		
2115	ISO18254	120.12		-0.45	121.97		
2129		121.2		-0.41	121.2		
2159	ISO18254-1	118.71		-0.52	-----		
2165	ISO18254-1	121.5		-0.39	-----		
2201	ISO18254-1	129.67		-0.03	129.67		
2213	ISO18254-1	158		1.24	158		
2241	ISO18254-1	137.34		0.31	137.34		
2247	ISO18254-1	153.26		1.03	153.26		
2250	In house	152.99		1.01	152.99		
2255	ISO/DIS 18254-1	110.21		-0.90	110.21		
2265	ISO18254-1	122.25		-0.36	122.25		
2286	ISO18254-1	162.00		1.42	162		
2290	ISO18254-1	132.7		0.11	132.7		
2293		-----		-----	-----		
2295	ISO/DIS 18254-1	149		0.84	149		
2297	ISO/DIS 18254-1	125.3		-0.22	125.3		
2300	ISO18254-1	112.24		-0.81	112.24		
2310	ISO18254-1	129		-0.06	129		
2311	ISO18254-1	141.08		0.48	141.08		
2320	ISO18254-1	112.4094		-0.80	112.4094		
2330	ISO/DIS 18254-1	125.045		-0.23	125.045		
2347	ISO/DIS 18254-1	126		-0.19	126		
2350	ISO18254-1	160.86		1.37	160.86		
2352	ISO18254-1	126.0		-0.19	126.0		
2357	ISO18254-1	123.4		-0.31	-----		
2358		117.86		-0.55	117.86		
2363	ISO18254-1	120.2		-0.45	120.2		
2365	ISO/DIS 18254-1	119.4		-0.49	119.4		
2366	ISO18254-1	125		-0.24	125		
2370	ISO18254-1	119		-0.50	119		
2372	ISO/DIS 18254-1	118.29		-0.54	118.29		
2374	ISO18254-1	120.03		-0.46	-----		
2375	ISO18254-1	130		-0.01	130		
2378	ISO18254-1	126.0		-0.19	126.0		
2379	ISO18254-1	101.957		-1.26	101.957		
2380	ISO18254-1	104.89		-1.13	104.89		
2382	ISO18254-1	121.0		-0.41	121.0		
2386	ISO18254-1	130.29		0.00	130.29		
2390	ISO/DIS 18254-1	145.94		0.70	145.94	C	first reported not detected
2415	ISO18254-1	131.30		0.05	-----		
2426	ISO18254-1	130.446		0.01	130.446		
2429		136.2		0.26	136.2		
2449	ISO21084	143.43		0.59	143.3		
2452	ISO18254-1	79.736		-2.26	-----		
2456	ISO18254-1	155.0		1.10	156.85		
2495	ISO18254-1	121.25		-0.40	122.40		
2508		176.16		2.05	213.82		
2511	ISO18254-1	127		-0.15	127		
2514	ISO/DIS 18254-1	106.21		-1.08	-----		
2515	ISO18254-1	121.95		-0.37	121.95		
2534	ISO18254-1	151.2		0.93	151.2		
2536	ISO18254-1	147.06		0.75	147.06		
2553	ISO/DIS 18254-1	120.18		-0.45	120.18		
2561	ISO18254-2	205.647	R(0.05)	3.36	-----		
2567	ISO18254-1	121.4		-0.40	121.4		
2572	ISO/DIS 18254-1	129.2		-0.05	129.2		
2573	ISO/DIS 18254-1	127.9		-0.11	127.9		
2582	In house	117.7482		-0.56	-----		
2590	ISO/DIS 18254-1	114.903		-0.69	114.903		
2591	In house	125.22		-0.23	125.22		
2605	GB/T23972	134.77		0.20	134.77		
2618	ISO/DIS 18254-1	111.95		-0.82	111.95		
2629	ISO18254-1	486.7	R(0.01)	15.91	-----		
2638	In house	121.41		-0.40	159.14		
2643	ISO18254-1	160.31		1.34	-----		
2644	ISO18254-1	135.0		0.21	135		
2671	ISO18254-1	123.84		-0.29	-----		

lab	method	value	mark	z(targ)	Total OP,NP, OPEO,NPEO		remarks
2678	ISO18254-1	181.21		2.27	181.21		
2734	ISO/DIS 18254-1	335.0	C,R(0.01)	9.14	335.0	C	first reported 218.325 and 218.3
2737	ISO21084	130.038	C	-0.01	-----		first reported as OPEO
2743	ISO/DIS 18254-1	180.951	C	2.26	185.660	C	first reported 195.038 and 200.222
2789	ISO18254-1	179.7		2.21	179.7		
2798	ISO18254-1	127		-0.15	-----		
2802	ISO18254-1	114.7	C	-0.70	77.8	E	first reported 77.8 / iis calc. 114.7
2864	ISO18254-1	102.01		-1.26	-----		
2867	ISO18254-1	125.2		-0.23	-----		
2870	ISO18254-1	147		0.75	147		
2948	ISO18254-1	141.347		0.49	141.347		
2955	ISO/DIS 18254-1	105.3		-1.12	105.3		
2959	ISO18254-1	124.6		-0.25	-----		
3100	GB/T23322	134.911		0.21	134.911		
3116	ISO18254-1	114.7		-0.70	-----		
3149	ISO/DIS 18254-1	111	C	-0.86	111	C	first reported 200.5 and 200.5
3153		130.0		-0.01	130.0		
3154	In house	90.23		-1.79	90.23		
3160	ISO18254-1	117.86		-0.55	117.9		
3172		99.32	C	-1.38	-----		first reported 169
3176	In house	118.0		-0.55	-----		
3185	ISO18254-1	133.70		0.15	133.70		
3210	In house	185.28		2.46	185.28		
3214	ISO18254-1	135.755		0.24	135.755		
3218	ISO18254-1	130.69		0.02	130.69		
3222	ISO18254-1	163.73		1.49	166.70		
3228	ISO/DIS 18254-1	123.1		-0.32	-----		
3237	ISO18254-1	161.6		1.40	-----		

	normality	OK	
	n	96	
	outliers	4	
	mean (n)	130.287	
	st.dev. (n)	19.6454	RSD=15%
	R(calc.)	55.007	
	st.dev.(Horwitz (n=5))	22.3966	
	R(Horwitz (n=5))	62.711	
Compare			
	R(ISO18254-1:16)	35.781	





APPENDIX 2

Summary of other reported components in sample #21525 and #21526; results in mg/kg

Abbreviations of components

OP = Octylphenol
NP = Nonylphenol
OPEO = Octylphenol Ethoxylates
NPEO = Nonylphenol Ethoxylates

	#21525		#21526		
lab	**NP**	**NPEO**	**OP**	**NP**	**OPEO**
230	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
339	<10	<10	<10	<10	<10
551	-----	-----	-----	-----	-----
623	not detected	not detected	not detected	not detected	not detected
840	not detected	not detected	not detected	not detected	not detected
841	<3	<1	<3	<3	<1
2115	-----	-----	-----	-----	1.85
2129	<10	<10	<10	<10	<10
2159	<5	<30	<5	<5	<30
2165	not detected	not detected	not detected	not detected	not detected
2201	not detected	not detected	not detected	not detected	not detected
2213	<10	<10	<10	<10	<10
2241	<10	<10	<10	<10	<10
2247	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2250	-----	-----	-----	-----	-----
2255	n.d	n.d	n.d	n.d	n.d
2265	< 3	< 20	< 3	< 3	< 20
2286	ND	ND	ND	ND	ND
2290	<10	<10	<10	<10	<10
2293	-----	-----	-----	-----	-----
2295	-----	-----	-----	-----	-----
2297	ND	ND	ND	ND	ND
2300	ND	ND	ND	ND	ND
2310	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2311	Not Detected	Not Detected	Not detected	Not detected	Not detected
2320	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2330	Not detected	Not detected	Not detected	Not detected	Not detected
2347	<10	<30	<10	<10	<30
2350	< 3.00	< 1.00	< 3.00	< 3.00	< 1.00
2352	-----	-----	-----	-----	-----
2357	-----	-----	-----	-----	-----
2358	n.d..	n.d..	n.d..	n.d..	n.d..
2363	<10	<10	<10	<10	<10
2365	<10	<30	<10	<10	<30
2366	<10	<30	<10	<10	<30
2370	<1	<1	<1	<1	<1
2372	not detected	not detected	not detected	not detected	not detected
2374	-----	-----	-----	-----	-----
2375	-----	-----	-----	-----	-----
2378	-----	-----	-----	-----	-----
2379	not detected	not detected	not detected	not detected	not detected
2380	<3	<1	<3	<3	<1
2382	<10.0	<10.0	<10.0	<10.0	<10.0
2386	<5	<10	<5	<5	<10
2390	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2415	-----	-----	-----	-----	-----
2426	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2429	ND	ND	ND	ND	ND
2449	-----	-----	-----	-----	-----
2452	-----	Not Detected	-----	-----	Not Detected
2456	not detected	not detected	not detected	not detected	1.85
2495	<0.5	<0.5	0.65	<0.5	0.50
2508	<1.0	<10.0	<1.0	<1.0	37.66
2511	-----	-----	-----	-----	-----
2514	-----	-----	-----	-----	-----
2515	ND	ND	ND	ND	ND
2534	not detected	not detected	not detected	not detected	not detected
2536	Not Detected	Not Detected	Not detected	Not detected	Not detected
2553	Not detected	Not detected	Not detected	Not detected	Not detected
2561	-----	less than 10	-----	-----	<10
2567	<10	<10	<10	<10	<10
2572	< 10	< 10	< 10	< 10	< 10
2573	not detected	not detected	not detected	not detected	not detected

	#21525		#21526		
lab	NP	NPEO	OP	NP	OPEO
2582	Not detected	Not detected	Not detected	Not detected	Not detected
2590	-----	-----	-----	-----	-----
2591	not detected	not detected	not detected	not detected	not detected
2605	<3	<10	<3	<3	<10
2618	not detected	not detected	not detected	not detected	not detected
2629	<10	343.7	48.4	<10	<30
2638	not detected	not detected	not detected	37.73	not detected
2643	-----	-----	-----	-----	-----
2644	-----	-----	-----	-----	-----
2671	-----	-----	-----	-----	-----
2678	not detected	not detected	not detected	not detected	not detected
2734	not detected	not detected	not detected	not detected	not detected
2737	-----	-----	-----	-----	Not detected
2743	not detected	0.861	not detected	3.719	1.465
2789	-----	-----	-----	-----	-----
2798	-----	-----	-----	-----	-----
2802	-----	-----	-----	-----	-----
2864	-----	-----	-----	-----	-----
2867	not detected	not detected	not detected	not detected	not detected
2870	-----	-----	-----	-----	-----
2948	Not Detected	Not Detected	Not Detected	Not Detected	Not Detected
2955	n.d	n.d	n.d	n.d	n.d
2959	-----	-----	-----	-----	-----
3100	<10	<10	<10	<10	<10
3116	-----	-----	-----	-----	-----
3149	-----	-----	-----	-----	-----
3153	<10	<10	<10	<10	<10
3154	-----	-----	-----	-----	-----
3160	not detected	not detected	not detected	not detected	not detected
3172	< 1	< 1	< 1	< 1	< 1
3176	-----	-----	-----	-----	-----
3185	not detected[<10]	not detected[<10]	not detected[<10]	not detected[<10]	not detected[<10]
3210	<20	<20	<20	<20	<20
3214	-----	-----	-----	-----	-----
3218	<10	<10	<10	<10	<10
3222	-----	-----	-----	-----	2.97
3228	not detected	not detected	not detected	not detected	not detected
3237	-----	-----	-----	-----	-----

APPENDIX 3

Analytical Details

lab	ISO/IEC17025 accredited	sample preparation	sample intake (g)	release/ extraction technique	release/ extraction solvent	extraction time (min)	extraction temperature (°C)
230	Yes	Further cut	1.0	Ultrasonic	Methanol	60	70
339	No	Used as received	1	Ultrasonic	Methanol	60	70
551	No	Used as received	1	Ultrasonic	Methanol	60	70
623	Yes	Further cut	0.5	Ultrasonic	MeOH	60	70
840	Yes	Further cut	0.5	Ultrasonic	Methanol	60	70
841	Yes	Further cut	1	Ultrasonic	Methanol	60	70
2115	Yes	Used as received	0.5	Ultrasonic	Methanol	60	70
2129	Yes	Used as received	1	Ultrasonic	Methanol	60	70
2159	Yes	Further cut	1	Ultrasonic	meOH	60	70
2165	Yes	Further cut	1	Ultrasonic	methanol	60	70
2201	Yes	Used as received	1	Ultrasonic	Methanol	60	70
2213	Yes	Used as received	0.3	Ultrasonic	Methanol	60	70
2241	Yes	Further cut	0.5	Ultrasonic	Methanol	60	70
2247	Yes	Used as received	1-2	Ultrasonic	Methanol	60.0	70
2250	Yes	Used as received	0.5	Ultrasonic	Methanol	60	70
2255	Yes	Used as received	0.5	Ultrasonic	Methanol	60	60
2265	Yes	Further cut	0.5	Ultrasonic	MeOH	60	70
2286	Yes	Further cut	0.5	Ultrasonic	Methanol	60	70
2290	Yes	---	---	---	---	---	---
2293	---	---	---	---	---	---	---
2295	Yes	Further cut	1	Ultrasonic	Methanol	60	70
2297	Yes	Used as received	1	Ultrasonic	methanol	60	40
2300	Yes	Further cut	0.5	Ultrasonic	Methanol	60	70
2310	Yes	Used as received	1	Ultrasonic	Methanol	60	70
2311	Yes	Used as received	0.5	Ultrasonic	Methanol	60	70
2320	Yes	Further cut	0.5	Ultrasonic	Methanol	60	70
2330	Yes	Further cut	1	Ultrasonic	Methanol	60	70 ± 2
2347	Yes	---	0.5	Ultrasonic	MeOH	60	70
2350	Yes	Further cut	1	Ultrasonic	Methyl alcohol	60	70
2352	Yes	Further cut	1.0	Ultrasonic	methanol	60	70
2357	---	---	---	---	---	---	---
2358	Yes	Further cut	1.0	Ultrasonic	Methanol	60	70
2363	Yes	Further cut	1	Ultrasonic	methonal	60	70
2365	Yes	Further cut	1	Ultrasonic	methanol	60	70
2366	Yes	Further cut	0.5	Soxhlet	methanol	60	70
2370	Yes	Further grinded	0.5	Ultrasonic	10 mL	60	70
2372	No	Used as received	1	Ultrasonic	MeOH	60	70
2374	Yes	Further cut	0.5	Ultrasonic	methanol	60	70
2375	Yes	Further cut	0.5	---	Methanol	60	70
2378	Yes	Further cut	1.0	Other	methanol	60	70
2379	Yes	Further cut	0.5	Ultrasonic	Methanol	60	70
2380	Yes	Further cut	1.00	Ultrasonic	Methanol	60	70
2382	Yes	Further cut	1.0	Ultrasonic	CH3OH	60	70
2386	Yes	Further cut	0.5	Ultrasonic	Methanole	60	70
2390	Yes	Further cut	1	Ultrasonic	Methanol	60	70
2415	Yes	Used as received	0.5	Ultrasonic	Methanol	60	70
2426	Yes	Further cut	0.5	Ultrasonic	Methanol 20 mL	60	70
2429	Yes	Further cut	1.0	Ultrasonic	Methonal 20mL	60	70
2449	Yes	Further cut	---	Ultrasonic	Methanol	60	70
2452	---	---	---	---	---	---	---
2456	Yes	Used as received	2.5	Ultrasonic	Methanol	60	70
2495	Yes	Used as received	1	Ultrasonic	MeOH	60	70
2508	Yes	Used as received	0.5	Ultrasonic	MEOH	60	70
2511	---	---	---	---	---	---	---
2514	Yes	Further cut	0.5-0.6	Ultrasonic	Methanol:Water=20:5	60	70
2515	Yes	Further cut	1	Ultrasonic	Methanol	60	70
2534	No	Further cut	1 +/- 0.02	Ultrasonic	methanol	60	70
2536	Yes	Further cut	1.0012	Ultrasonic	Methanol	60	70
2553	Yes	Further cut	1	Ultrasonic	Methanol	60	70
2561	Yes	Further cut	0.9-1.1	Ultrasonic	methanol	60	70
2567	Yes	Other	1	Soxhlet	Methanol	60	70
2572	---	---	---	---	---	---	---
2573	No	Used as received	0.5	Ultrasonic	Methanol	60	70
2582	Yes	Further cut	1.00	Ultrasonic	Methanol	60	70
2590	Yes	Used as received	1	Ultrasonic	MeOH	60	70
2591	No	Further cut	1.00	Ultrasonic	Methanol	60	40
2605	Yes	Further cut	1.000	Ultrasonic	methanol	60	70
2618	Yes	Used as received	1.0	Ultrasonic	Methanol	60	70
2629	Yes	Further cut	1.0	Ultrasonic	Methanol	60	70
2638	No	Further cut	1	Ultrasonic	Methanol	60	Room temp.
2643	Yes	Further cut	0.5	Ultrasonic	Methanol	60	70
2644	Yes	Used as received	---	Ultrasonic	Methanol	60	70

lab	ISO/IEC17025 accredited	sample preparation	sample intake (g)	release/ extraction technique	release/ extraction solvent	extraction time (min)	extraction temperature (°C)
2671	Yes	Further cut	1.0	Ultrasonic	Methanol	60	70
2678	No	Further cut	1	Ultrasonic	Methanol	60	70
2734	Yes	Used as received	1.0	Ultrasonic	methanol	60	70
2737	Yes	Further cut	1	Ultrasonic	methanol	60	70
2743	Yes	Used as received	0.7	Ultrasonic	Methanol	60	70
2789	Yes	Used as received	1	Ultrasonic	methanol	60	70
2798	Yes	Further grinded	1	Ultrasonic	MeOH	60	70
2802	No	Further cut	1	Ultrasonic	Methanol	60	70
2864	Yes	Used as received	0.5	Ultrasonic	MEOH	60	70
2867	Yes	Used as received	0.5	Ultrasonic	Methanol	60	70
2870	Yes	Further cut	1	Ultrasonic	Methanol	60	60
2948	Yes	Used as received	1.013	Ultrasonic	Methanol	60	70
2955	Yes	Used as received	0.5	Ultrasonic	Methanol	60	70
2959	No	Used as received	---	---	---	---	---
3100	Yes	Further cut	1	Ultrasonic	MeoH	60	70
3116	Yes	Used as received	1	Ultrasonic	Methanol	60	70
3149	Yes	Used as received	0.5	Ultrasonic	methanol	60	70
3153	Yes	Further cut	0.5	Ultrasonic	Methanol	60	70
3154	Yes	Used as received	0.5	Ultrasonic	MeOH	60	70
3160	No	Used as received	1	Ultrasonic	Methanol	60	70
3172	Yes	Further cut	0.3	Ultrasonic	Methanol	60	70
3176	Yes	Further cut	1	Ultrasonic	MeOH	60	50
3185	Yes	Further cut	1	Ultrasonic	methanol	60	70
3210	Yes	Used as received	1	Ultrasonic	Methanol	70	60
3214	Yes	Further cut	0.5	Ultrasonic	MeOH	60	70
3218	Yes	Further cut	0.5	Ultrasonic	methanol	60	70
3222	Yes	Used as received	0.5	Ultrasonic	methanol	60	70
3228	Yes	Further cut	2	Ultrasonic	methanol	60	70
3237	Yes	Further cut	0.5	Ultrasonic	metonol	60	70

APPENDIX 4

Number of participants per country

6 labs in	BANGLADESH
1 lab in	BRAZIL
2 labs in	CAMBODIA
2 labs in	FRANCE
7 labs in	GERMANY
1 lab in	GUATEMALA
3 labs in	HONG KONG
7 labs in	INDIA
1 lab in	INDONESIA
10 labs in	ITALY
1 lab in	JAPAN
1 lab in	MAURITIUS
24 labs in	P.R. of CHINA
5 labs in	PAKISTAN
3 labs in	SOUTH KOREA
3 labs in	SPAIN
3 labs in	SRI LANKA
4 labs in	TAIWAN
1 lab in	THAILAND
3 labs in	TUNISIA
5 labs in	TURKEY
1 lab in	UNITED KINGDOM
7 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	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported

Literature

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