# Results of Proficiency Test
# AP & APEO in textile
# March 2017

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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. Human exposure to APEO can occur through various sources such as environmental, food or skin contact. Considering their toxicity on several animal species, minimization of exposure to APEO is recognized as important to the preservation of human health. APEO may degradate in the environment to the corresponding Octyl- and Nonylphenol (OP & NP). These alkylphenols (AP) have attracted attention due to its prevalence in the environment and its potential role as an endocrine disruptor and xenoestrogen, due to its ability to act with estrogen-like activity. The European Union has implemented sales and use restrictions on certain applications in which alkylphenols are used because of their alleged "toxicity, persistence, and the liability to bioaccumulate".

In 2016 Institute for Interlaboratory Studies organised a new proficiency test (PT) for the determination of AP and APEO content in textile on request of several participants. During the annual proficiency testing program 2016/2017 it was decided to continue with the PT for the analyses of AP and APEO content in textile. In this interlaboratory study, 99 laboratories in 28 different countries registered for participation. See appendix 5 for the number of participants per country.

In this report, the results of the 2017 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 organiser of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to use two different samples (labelled #17530 and #17531) which were positive (artificially fortified) on OPEO or NPEO. The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

### 2.1    QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on IEC/ISO17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

### 2.2    PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

### 2.3   CONFIDENTIALITY STATEMENT

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

### 2.4   SAMPLES

Two different batches of textile were prepared by a third party. The first bulk sample, a cotton fabric artificially fortified with OPEO by means of Triton X-100 (CAS 9002-93-1), was cut into pieces. Out of this batch, after mixing well, 120 subsamples of approx. 3 grams each were prepared and labelled #17530.
The homogeneity of the stratified randomly selected samples was checked by determination of OPEO using an in-house test method by an accredited third party laboratory. See the following table for the test results.

|                    | OPEO in mg/kg |
|--------------------|---------------|
| Sample #17530-1    | 78.21         |
| Sample #17530-2    | 74.33         |
| Sample #17530-3    | 73.81         |
| Sample #17530-4    | 79.14         |
| Sample #17530-5    | 77.89         |
| Sample #17530-6    | 82.59         |
| Sample #17530-7    | 77.14         |
| Sample #17530-8    | 75.23         |

Table 1: homogeneity test results of subsamples #17530

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

|                          | OPEO in mg/kg  |
|--------------------------|----------------|
| r (observed)             | 8.05           |
| reference method         | Horwitz (n=5)  |
| 0.3*R (reference method) | 12.07          |

Table 2: evaluation of the repeatability of subsamples #17530

The second bulk sample, a cotton fabric artificially fortified with NPEO by means of Tergitol NP-10 (CAS 127087-87-0), was cut into pieces. Out of this batch, after mixing well, 120 subsamples of approx. 3 grams each were prepared and labelled #17531.
The homogeneity of 7 stratified randomly selected samples was checked by determination of NPEO using an in-house test method by an accredited third party laboratory. See the following table for the test results.

|                  | NPEO in mg/kg |
|------------------|---------------|
| Sample #17531-1  | 145.9         |
| Sample #17531-2  | 134.4         |
| Sample #17531-3  | 133.8         |
| Sample #17531-4  | 151.0         |
| Sample #17531-5  | 148.6         |
| Sample #17531-6  | 141.5         |
| Sample #17531-7  | 150.3         |

Table 3: homogeneity test results of subsamples #17531

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

|                             | NPEO in mg/kg  |
|-----------------------------|----------------|
| r (observed)                | 20.3           |
| reference method            | Horwitz (n=5)  |
| 0.3*R (reference method)    | 20.4           |

Table 4: evaluation of the repeatability of subsamples #17531

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

To each participating laboratory one sample of approx. 3 grams, labelled #17530 and one sample of approx. 3 grams, labelled #17531 were sent on March 8, 2017.

## 2.5  ANALYSES

The participants were requested to determine the concentrations of OP, NP,OPEO, NPEO and total OP, NP, OPEO + NPEO on both samples #17530 and #17531 applying the analysis procedure that is routinely used in the laboratory. Also some method details were requested to be reported.

It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the test results more, but report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical calculations.

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

## 3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are represented by the code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyses). 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 organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4).

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

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

In accordance to ISO 5725 the original test results per determination were submitted subsequently to Dixon's, Grubbs' and or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have significant consequences for the evaluation of the test results.

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

## 3.2 GRAPHICS

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

## 3.3 Z-SCORES

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

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

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

The z-scores were calculated in accordance with:

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

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

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

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

## 4    EVALUATION

During the execution of this proficiency test no serious problems occurred, although one participants reported the test results after closure of the data entry tool of this proficiency test (PT) and four participants did not report any results at all. In total 95 of the 99 participants reported 378 numerical test results. Observed in all reported results were 9 statistical outlying results, which is 2.4%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

ISO 18254, used by the majority of the participants, is the official test method for the determination of APEO in textiles. A few of the participating laboratories used ISO18218-1, a test method developed for the determination of APEO in <u>leather</u>. The methods are almost identical, except for the ratio grams of sample:extraction liquid, which is 1:20 for ISO 18254 and 1:10 for ISO 18218-1. Regretfully ISO 18254 does not mention reproducibilities for OP, NP, OPEO or NPEO, but only for APEO at a level of 954 mg/kg (R=262 mg/kg). ISO 18218-1 and ISO 18218-2 do not have any precision data mentioned. Therefore, the target requirements in this study were estimated using the Horwitz equation (for n=5).

### 4.1    EVALUATION PER SAMPLE AND PER COMPONENT

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

**sample #17530**

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

sum OP + NP + OPEO + NPEO:

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

**sample #17531**

NPEO:           The determination of this component may not be problematic. One statistical
                outlier was detected. However, the calculated reproducibility after rejection of
                the statistical outlier is in full agreement with the estimated target
                reproducibility using the Horwitz equation for 5 components.

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

### 4.2   PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

|                            | unit  | n  | Average | 2.8 * sd | R (target)* |
|----------------------------|-------|----|---------|----------|-------------|
| OPEO                       | mg/kg | 92 | 108.9   | 46.0     | 53.9        |
| sum OP + NP + OPEO + NPEO  | mg/kg | 71 | 110.2   | 47.4     | 54.4        |

Table 5: reproducibilities of tests on sample #17530

*)   against the strict Horwitz equation

|                            | unit  | n  | Average | 2.8 * sd | R (target)* |
|----------------------------|-------|----|---------|----------|-------------|
| NPEO                       | mg/kg | 92 | 137.3   | 67.3     | 65.5        |
| sum OP + NP + OPEO + NPEO  | mg/kg | 71 | 138.8   | 64.8     | 66.2        |

Table 6: reproducibilities of tests on sample #17531

*)   against the strict Horwitz equation

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

### 4.3   COMPARISON OF THE PROFICIENCY TEST OF MARCH 2017 WITH PREVIOUS PT

|                               | March 2017 | March 2016 |
|-------------------------------|------------|------------|
| Total Number of reporting labs | 95         | 105        |
| Number of results reported     | 378        | 412        |
| Statistical outliers           | 9          | 13         |
| Percentage outliers            | 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 2017 | March 2016 |
|------------------------------------|------------|------------|
| Octylphenol (OP)                   | n.a.       | n.a.       |
| Nonylphenol (NP)                   | n.a.       | n.a.       |
| Octylphenol Ethoxylates (OPEO)     | 15%        | 16%        |
| Nonylphenol Ethoxylates (NPEO)     | 18%        | 27%        |
| sum OP + NP + OPEO + NPEO          | 15% / 17%  | 16% / 25%  |

Table 8: comparison of uncertainties (relative in %)

## 5    DISCUSSION

In this proficiency test for the determination of AP and APEO content in textile the majority of the participants has no problem with the analysis of OPEO and NPEO in textile at the levels as present in this PT (respectively 109 and 137 mg/kg).

When the results of this interlaboratory study were compared to the OEKO-TEX requirements and the EU (REACH) regulations on Textiles (table 9), it is noticed that about 75% of the reporting laboratories would reject sample #17530 for containing too much OP + NP + OPEO + NPEO in total and that about 90% of the reporting laboratories would reject sample #17531 for containing too much NPEO and/or too much OP + NP + OPEO + NPEO in total.
For samples #17530 and #17531 two laboratories detected more than 10 mg/kg OP+NP. For sample #17531 one laboratory detected more than 100 mg/kg OPEO (note: OPEO was possibly mixed up with NPEO). One laboratory detected OPEO in sample #17530 and NPEO in sample #17531 but reported n.d. as result for the 'sum OP + NP + OPEO + NPEO'.

|                           | OEKO-TEX  | EU 2016/26 |
|---------------------------|-----------|------------|
| NP                        | ---       | 100 mg/kg  |
| sum OP + NP               | 10 mg/kg  | ---        |
| NPEO                      | ---       | 100 mg/kg  |
| sum OP + NP + OPEO + NPEO | 100 mg/kg | ---        |

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

For sample #17530 the average of the homogeneity test results is not in line with the average (consensus value) from the PT results. There are several reasons for this.
First the goal of the homogeneity testing is very different from the goal of the evaluation of the reported PT results. In order to proof the homogeneity of the PT samples, a test method is selected with a high precision (smallest variation). The accuracy (trueness) of the test method is less relevant.
Secondly, the homogeneity testing is done by one laboratory only. The test results of this (ISO/IEC 17025 accredited) laboratory will have a bias (systematic deviation) depending on the test method used. The desire to detect small variations between the PT samples leads to the use of a sensitive test method with high precision, which may be a test method with significant bias.

Also each test result reported by the laboratories that participate in the PT will have a bias. However, some will have a positive bias and others a negative bias. These different biases compensate each other in the PT average (consensus value). Therefore the PT consensus value may deviate from the average of the homogeneity test. At the same time the accuracy of the PT consensus value is more reliable than the accuracy of the average of the results of the homogeneity test.

## 6      CONCLUSION

Although, it can be concluded that the majority of the participants has no problem with the determination of OPEO and NPEO in the samples of this PT, each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary.
Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.
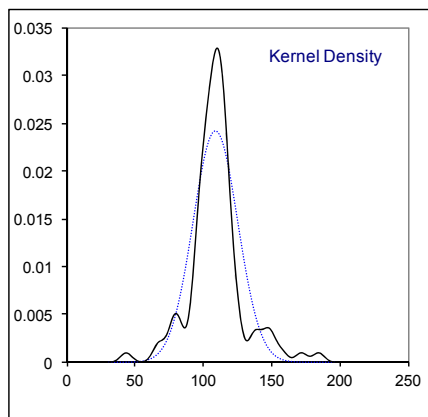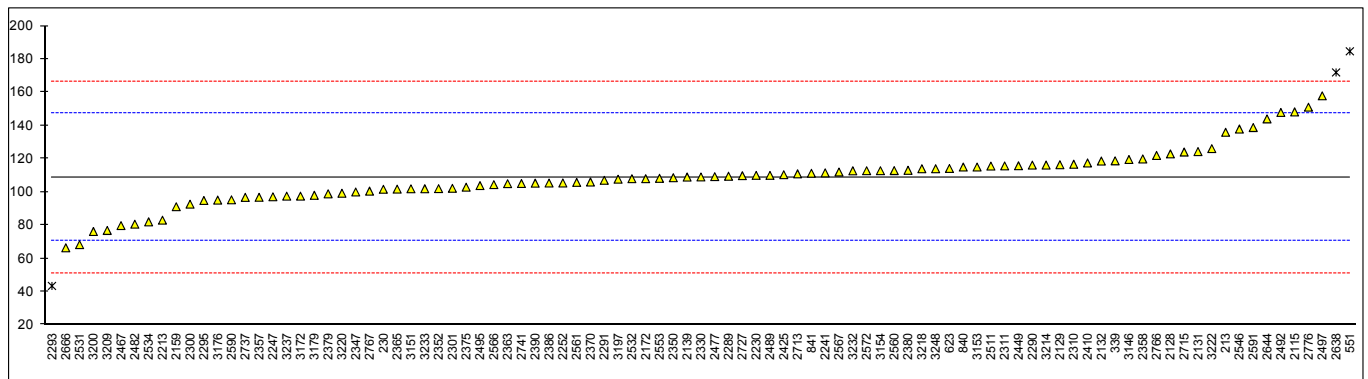
## APPENDIX 1

Determination of Octylphenol Ethoxylates (OPEO) on sample #17530; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|------|----------------|------------|-----------|---------|------------------------------|
| 110 | | ----- | | ----- | |
| 213 | In house | 135.96 | | 1.41 | |
| 230 | ISO18218-2 | 101.6 | | -0.38 | |
| 339 | In house | 118.75 | | 0.51 | |
| 551 | In house | 184.7141565 | R(0.05) | 3.94 | |
| 623 | ISO/DIS 18254-1 | 114.14 | | 0.27 | |
| 840 | In house | 115 | | 0.32 | |
| 841 | ISO18254-1 | 111.25 | | 0.12 | |
| 2115 | ISO18254-1 | 148.23 | | 2.04 | |
| 2128 | ISO18254-1 | 122.9 | | 0.73 | |
| 2129 | ISO18218-1 | 116.4 | | 0.39 | |
| 2131 | In house | 124.32805 | | 0.80 | |
| 2132 | ISO18254-1 | 118.62 | | 0.51 | |
| 2139 | ISO18254-1 | 108.9833 | | 0.00 | |
| 2159 | ISO/DIS 18254-1 | 91.12 | | -0.92 | |
| 2172 | ISO18254-1 | 108 | | -0.05 | |
| 2213 | ISO/DIS 18254-1 | 83 | | -1.35 | |
| 2230 | ISO18254-1 | 110 | | 0.06 | |
| 2241 | ISO18254-1 | 111.5 | | 0.13 | |
| 2247 | ISO/DIS 18254-1 | 97.109 | | -0.61 | |
| 2252 | ISO18254-1 | 105.432 | | -0.18 | |
| 2265 | | ----- | | ----- | |
| 2289 | ISO18254-1 | 109.47 | | 0.03 | |
| 2290 | ISO18254-1 | 116.2 | | 0.38 | |
| 2291 | ISO18218-1 | 107 | | -0.10 | |
| 2293 | ISO18254-1 | 43.398 | R(0.05) | -3.41 | |
| 2295 | ISO/DIS 18254-1 | 94.865 | | -0.73 | |
| 2300 | ISO18254-1 | 92.6611 | | -0.84 | |
| 2301 | | 102.2 | | -0.35 | |
| 2310 | ISO18254-1 | 116.7 | | 0.41 | |
| 2311 | ISO18254-1 | 115.7 | | 0.35 | |
| 2330 | ISO18218-1 | 109.0479 | | 0.01 | |
| 2347 | ISO18254-1 | 100 | | -0.46 | |
| 2350 | In house | 108.596 | | -0.02 | |
| 2352 | ISO18254-1 | 102.0 | | -0.36 | |
| 2357 | ISO18254-1 | 96.8 | | -0.63 | |
| 2358 | ISO18254-1 | 119.9 | | 0.57 | |
| 2363 | ISO18254-1 | 105 | | -0.20 | |
| 2365 | ISO18254-1 | 101.7 | | -0.37 | |
| 2369 | | ----- | | ----- | |
| 2370 | ISO18254 | 106 | | -0.15 | |
| 2375 | ISO18254-1 | 102.9 | | -0.31 | |
| 2379 | ISO18254-1 | 98.8885 | | -0.52 | |
| 2380 | In house | 113.0554 | | 0.22 | |
| 2386 | In house | 105.4 | | -0.18 | |
| 2390 | ISO18254-1 | 105.311 | | -0.19 | |
| 2410 | ISO/DIS 18254-1 | 117.47 | | 0.45 | |
| 2425 | In house | 110.4 | | 0.08 | |
| 2449 | GB/T23322 | 115.7634 | | 0.36 | |
| 2467 | In house | 79.76 | | -1.52 | |
| 2477 | ISO18218-2 | 109.22 | | 0.02 | |
| 2482 | ISO18254-1 | 80.6 | | -1.47 | |
| 2489 | ISO18254-1 | 110 | | 0.06 | |
| 2492 | In house | 147.9 | | 2.03 | |
| 2495 | ISO/DIS 18254-1 | 103.84 | | -0.26 | |
| 2497 | ISO18254-1 | 157.89 | C | 2.55 | First reported 236.921 |
| 2511 | ISO18254-1 | 115.59 | | 0.35 | |
| 2531 | ISO18218-2 | 68.2524 | | -2.11 | |
| 2532 | ISO/DIS 18254-1 | 107.99 | | -0.05 | |
| 2534 | ISO/DIS 18254-1 | 82 | | -1.40 | |
| 2546 | ISO18254-1 | 137.9 | | 1.51 | |
| 2553 | ISO18254-1 | 108.21 | | -0.04 | |
| 2560 | In house | 112.87 | | 0.21 | |
| 2561 | | 105.75 | C | -0.16 | First reported as sample #17531 |
| 2566 | In house | 104.4 | | -0.23 | |
| 2567 | ISO/DIS 18254-1 | 112 | | 0.16 | |
| 2572 | ISO18254-1 | 112.8 | | 0.20 | |
| 2590 | ISO/DIS 18254-1 | 95.2820 | | -0.71 | |
| 2591 | In house | 138.80 | | 1.55 | |
| 2638 | In house | 171.99 | C,R(0.05) | 3.28 | First reported 227.1 |
| 2644 | ISO18254-1 | 144 | C | 1.82 | First reported 214 |
| 2666 | ISO18218-2 | 66.3734 | | -2.21 | |
| 2713 | In house | 110.91 | | 0.10 | |
| 2715 | In house | 124.0478 | | 0.79 | |

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 2727 | ISO18218-1 | 109.82 | | 0.05 | |
| 2737 | ISO18254-1 | 96.76 | | -0.63 | |
| 2741 | ISO18254-1 | 105.1 | | -0.20 | |
| 2766 | ISO18254-1 | 122 | | 0.68 | |
| 2767 | ISO/DIS 18254-1 | 100.5 | | -0.44 | |
| 2776 | ISO18254-1 | 151.05 | | 2.19 | |
| 3146 | ISO/DIS 18254-1 | 119.5691 | | 0.55 | |
| 3151 | ISO18254-1 | 101.9 | | -0.36 | |
| 3153 | ISO/DIS 18254-1 | 115 | | 0.32 | |
| 3154 | In house | 112.8 | | 0.20 | |
| 3172 | ISO/DIS 18254-1 | 97.5 | | -0.59 | |
| 3176 | In house | 95.10 | | -0.72 | |
| 3179 | In house/ISO18254-1 | 98 | | -0.57 | |
| 3197 | ISO18254-1 | 107.7 | | -0.06 | |
| 3200 | ISO18254-1 | 76.2 | | -1.70 | |
| 3209 | In house | 76.82 | | -1.67 | |
| 3210 | | ----- | | ----- | |
| 3214 | ISO18218-1 | 116.21 | | 0.38 | |
| 3218 | ISO18254-1 | 114 | | 0.26 | |
| 3220 | ISO18254-1 | 99.24 | | -0.50 | |
| 3222 | ISO18254-1 | 126.10 | | 0.89 | |
| 3232 | ISO/DIS 18254-1 | 112.76 | | 0.20 | |
| 3233 | In house | 101.9447 | | -0.36 | |
| 3237 | ISO/DIS 18254-1 | 97.4982 | | -0.59 | |
| 3248 | In house | 114 | | 0.26 | |

| | | | |
|---|---|---|---|
| | normality | suspect | |
| | n | 92 | |
| | outliers | 3 | |
| | mean (n) | 108.906 | |
| | st.dev. (n) | 16.4354 | |
| | R(calc.) | 46.019 | |
| | R(Horwitz (n=5)) | 53.853 | |





Kernel Density

Determination of Total of OP, NP, OPEO and NPEO on sample #17530; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 110 | | ----- | | ----- | |
| 213 | In house | ----- | | ----- | |
| 230 | ISO18218-2 | 101.6 | | -0.44 | |
| 339 | In house | ----- | | ----- | |
| 551 | In house | 189.4840155 | R(0.01) | 4.08 | |
| 623 | ISO/DIS 18254-1 | 114.14 | | 0.20 | |
| 840 | In house | 115 | | 0.25 | |
| 841 | ISO18254-1 | ----- | | ----- | |
| 2115 | ISO18254-1 | 148.23 | | 1.96 | |
| 2128 | ISO18254-1 | 122.9 | | 0.65 | |
| 2129 | ISO18218-1 | 116.4 | | 0.32 | |
| 2131 | In house | 124.32805 | | 0.73 | |
| 2132 | ISO18254-1 | 118.62 | | 0.43 | |
| 2139 | ISO18254-1 | 108.9833 | | -0.06 | |
| 2159 | ISO/DIS 18254-1 | 91.12 | | -0.98 | |
| 2172 | ISO18254-1 | ----- | | ----- | |
| 2213 | ISO/DIS 18254-1 | 83 | | -1.40 | |
| 2230 | ISO18254-1 | ----- | | ----- | |
| 2241 | ISO18254-1 | ----- | | ----- | |
| 2247 | ISO/DIS 18254-1 | 97.109 | | -0.68 | |
| 2252 | ISO18254-1 | 105.432 | | -0.25 | |
| 2265 | | ----- | | ----- | |
| 2289 | ISO18254-1 | 109.47 | | -0.04 | |
| 2290 | ISO18254-1 | 116.2 | | 0.31 | |
| 2291 | ISO18218-1 | 107 | | -0.17 | |
| 2293 | ISO18254-1 | 43.398 | R(0.05) | -3.44 | |
| 2295 | ISO/DIS 18254-1 | | | ----- | |
| 2300 | ISO18254-1 | 92.6611 | | -0.90 | |
| 2301 | | 102.2 | | -0.41 | |
| 2310 | ISO18254-1 | 116.7 | | 0.33 | |
| 2311 | ISO18254-1 | 115.7 | | 0.28 | |
| 2330 | ISO18218-1 | 109.0479 | | -0.06 | |
| 2347 | ISO18254-1 | 100 | | -0.53 | |
| 2350 | In house | 108.596 | | -0.08 | |
| 2352 | ISO18254-1 | 102.0 | | -0.42 | |
| 2357 | ISO18254-1 | 96.8 | | -0.69 | |
| 2358 | ISO18254-1 | 119.9 | | 0.50 | |
| 2363 | ISO18254-1 | 105 | | -0.27 | |
| 2365 | ISO18254-1 | 101.7 | | -0.44 | |
| 2369 | | ----- | | ----- | |
| 2370 | ISO18254 | 106 | | -0.22 | |
| 2375 | ISO18254-1 | ----- | | ----- | |
| 2379 | ISO18254-1 | 98.8885 | | -0.58 | |
| 2380 | In house | 113.0554 | | 0.15 | |
| 2386 | In house | 105.4 | | -0.25 | |
| 2390 | ISO18254-1 | 105.311 | | -0.25 | |
| 2410 | ISO/DIS 18254-1 | 117.47 | | 0.37 | |
| 2425 | In house | 110.4 | | 0.01 | |
| 2449 | GB/T23322 | ----- | | ----- | |
| 2467 | In house | ----- | | ----- | |
| 2477 | ISO18218-2 | 109.22 | | -0.05 | |
| 2482 | ISO18254-1 | ----- | | ----- | |
| 2489 | ISO18254-1 | 110 | | -0.01 | |
| 2492 | In house | 147.9 | | 1.94 | |
| 2495 | ISO/DIS 18254-1 | 103.84 | | -0.33 | |
| 2497 | ISO18254-1 | 164.42 | C | 2.79 | First reported 245.659 |
| 2511 | ISO18254-1 | 115.59 | | 0.28 | |
| 2531 | ISO18218-2 | 68.2524 | | -2.16 | |
| 2532 | ISO/DIS 18254-1 | 107.99 | | -0.12 | |
| 2534 | ISO/DIS 18254-1 | 82 | | -1.45 | |
| 2546 | ISO18254-1 | 139.1 | | 1.49 | |
| 2553 | ISO18254-1 | 108.21 | | -0.10 | |
| 2560 | In house | n.d. | | ----- | False negative test result? |
| 2561 | | ----- | | ----- | |
| 2566 | In house | 104.4 | | -0.30 | |
| 2567 | ISO/DIS 18254-1 | 112 | | 0.09 | |
| 2572 | ISO18254-1 | 112.8 | | 0.13 | |
| 2590 | ISO/DIS 18254-1 | 95.2820 | | -0.77 | |
| 2591 | In house | 138.80 | | 1.47 | |
| 2638 | In house | ----- | | ----- | |
| 2644 | ISO18254-1 | 144 | C | 1.74 | First reported 214 |
| 2666 | ISO18218-2 | 66.3734 | | -2.26 | |
| 2713 | In house | 110.91 | | 0.03 | |
| 2715 | In house | 124.0478 | | 0.71 | |
| 2727 | ISO18218-1 | ----- | | ----- | |

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 2737 | ISO18254-1 | ----- | | ----- | |
| 2741 | ISO18254-1 | 105.1 | | -0.26 | |
| 2766 | ISO18254-1 | 202 | C,R(0.01) | 4.72 | First reported 211 |
| 2767 | ISO/DIS 18254-1 | 100.5 | | -0.50 | |
| 2776 | ISO18254-1 | 151.98 | | 2.15 | |
| 3146 | ISO/DIS 18254-1 | 119.5691 | | 0.48 | |
| 3151 | ISO18254-1 | 101.9 | | -0.43 | |
| 3153 | ISO/DIS 18254-1 | 115 | | 0.25 | |
| 3154 | In house | ----- | | ----- | |
| 3172 | ISO/DIS 18254-1 | 97.5 | | -0.66 | |
| 3176 | In house | 95.10 | | -0.78 | |
| 3179 | In house/ISO18254-1 | 98 | | -0.63 | |
| 3197 | ISO18254-1 | ----- | | ----- | |
| 3200 | ISO18254-1 | ----- | | ----- | |
| 3209 | In house | ----- | | ----- | |
| 3210 | | ----- | | ----- | |
| 3214 | ISO18218-1 | 116.21 | | 0.31 | |
| 3218 | ISO18254-1 | 114 | | 0.19 | |
| 3220 | ISO18254-1 | 99.24 | | -0.57 | |
| 3222 | ISO18254-1 | 127.29 | | 0.88 | |
| 3232 | ISO/DIS 18254-1 | ----- | | ----- | |
| 3233 | In house | 101.9447 | | -0.43 | |
| 3237 | ISO/DIS 18254-1 | 97.4982 | | -0.66 | |
| 3248 | In house | 114 | | 0.19 | |

|  |  |
|--|--|
| normality | not OK |
| n | 71 |
| outliers | 3 |
| mean (n) | 110.230 |
| st.dev. (n) | 16.9374 |
| R(calc.) | 47.425 |
| R(Horwitz (n=5)) | 54.408 |





Kernel Density
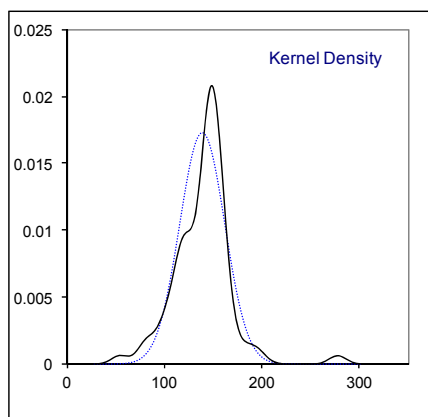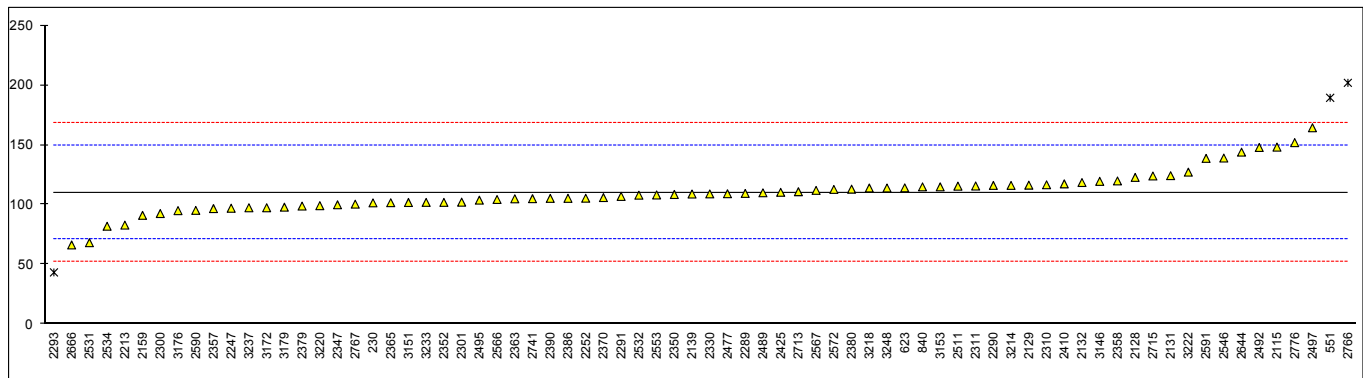
Determination of Nonylphenol Ethoxylates (NPEO) on sample #17531; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 110 | | ----- | | ----- | |
| 213 | In house | 169.16 | | 1.36 | |
| 230 | ISO18218-2 | 158.9 | | 0.92 | |
| 339 | In house | 113.08 | | -1.03 | |
| 551 | In house | 102.363898 | | -1.49 | |
| 623 | ISO/DIS 18254-1 | 157.60 | | 0.87 | |
| 840 | In house | 145 | | 0.33 | |
| 841 | ISO18254-1 | 129.81 | | -0.32 | |
| 2115 | ISO18254-1 | 160.33 | | 0.99 | |
| 2128 | ISO18254-1 | 119.2 | | -0.77 | |
| 2129 | ISO18218-1 | 197.8 | | 2.59 | |
| 2131 | In house | 160.3394 | | 0.99 | |
| 2132 | ISO18254-1 | 142.48 | | 0.22 | |
| 2139 | ISO18254-1 | 143.8456 | | 0.28 | |
| 2159 | ISO/DIS 18254-1 | 163.51 | | 1.12 | |
| 2172 | ISO18254-1 | 135 | | -0.10 | |
| 2213 | ISO/DIS 18254-1 | 155 | | 0.76 | |
| 2230 | ISO18254-1 | 146 | | 0.37 | |
| 2241 | ISO18254-1 | 137.0 | | -0.01 | |
| 2247 | ISO/DIS 18254-1 | 143.912 | | 0.28 | |
| 2252 | ISO18254-1 | 148.351 | | 0.47 | |
| 2265 | | ----- | | ----- | |
| 2289 | ISO18254-1 | 139.04 | | 0.08 | |
| 2290 | ISO18254-1 | 145.1 | | 0.34 | |
| 2291 | ISO18218-1 | 143 | | 0.25 | |
| 2293 | ISO18254-1 | 54.155 | R(0.05) | -3.55 | |
| 2295 | ISO/DIS 18254-1 | 117.17 | | -0.86 | |
| 2300 | ISO18254-1 | 82.1098 | | -2.36 | |
| 2301 | | 114.2 | | -0.98 | |
| 2310 | ISO18254-1 | 152.8 | | 0.66 | |
| 2311 | ISO18254-1 | 155.1 | | 0.76 | |
| 2330 | ISO18218-1 | 148.7320 | | 0.49 | |
| 2347 | ISO18254-1 | 138 | | 0.03 | |
| 2350 | In house | 152.176 | | 0.64 | |
| 2352 | ISO18254-1 | 147.0 | | 0.42 | |
| 2357 | ISO18254-1 | 150.2 | | 0.55 | |
| 2358 | ISO18254-1 | 145.6 | | 0.36 | |
| 2363 | ISO18254-1 | 153 | | 0.67 | |
| 2365 | ISO18254-1 | 141.2 | | 0.17 | |
| 2369 | | ----- | | ----- | |
| 2370 | ISO18254 | 149 | | 0.50 | |
| 2375 | ISO18254-1 | 140.8 | | 0.15 | |
| 2379 | ISO18254-1 | 125.8403 | | -0.49 | |
| 2380 | In house | 141.3572 | | 0.18 | |
| 2386 | In house | 113.2 | | -1.03 | |
| 2390 | ISO18254-1 | 151.494 | | 0.61 | |
| 2410 | ISO/DIS 18254-1 | 155.54 | | 0.78 | |
| 2425 | In house | 129.4 | | -0.34 | |
| 2449 | GB/T23322 | ----- | | ----- | |
| 2467 | In house | 115.79 | | -0.92 | |
| 2477 | ISO18218-2 | 164.07 | | 1.15 | |
| 2482 | ISO18254-1 | 140.7 | | 0.15 | |
| 2489 | ISO18254-1 | 152 | | 0.63 | |
| 2492 | In house | 100.0 | | -1.59 | |
| 2495 | ISO/DIS 18254-1 | 134.59 | | -0.11 | |
| 2497 | ISO18254-1 | 180.678 | | 1.85 | |
| 2511 | ISO18254-1 | 110.53 | | -1.14 | |
| 2531 | ISO18218-2 | 99.0968 | | -1.63 | |
| 2532 | ISO/DIS 18254-1 | 143.99 | | 0.29 | |
| 2534 | ISO/DIS 18254-1 | 170 | | 1.40 | |
| 2546 | ISO18254-1 | 186.7 | | 2.11 | |
| 2553 | ISO18254-1 | 119.32 | | -0.77 | |
| 2560 | In house | 96.24 | | -1.75 | |
| 2561 | | 203.3 | C | 2.82 | First reported as sample #17530 |
| 2566 | In house | 114 | | -0.99 | |
| 2567 | ISO/DIS 18254-1 | 151 | | 0.59 | |
| 2572 | ISO18254-1 | 140.2 | | 0.13 | |
| 2590 | ISO/DIS 18254-1 | < L.O.Q. | | ----- | Possibly mixed up with OPEO? |
| 2591 | In house | 155.20 | | 0.77 | |
| 2638 | In house | 94.42 | | -1.83 | |
| 2644 | ISO18254-1 | 117.5 | | -0.84 | |
| 2666 | ISO18218-2 | 120.1648 | | -0.73 | |
| 2713 | In house | 150.03 | | 0.55 | |
| 2715 | In house | 148.9167 | | 0.50 | |
| 2727 | ISO18218-1 | 168.95 | | 1.35 | |

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 2737 | ISO18254-1 | 127.73 | | -0.41 | |
| 2741 | ISO18254-1 | 151.1 | | 0.59 | |
| 2766 | ISO18254-1 | 104 | | -1.42 | |
| 2767 | ISO/DIS 18254-1 | 150.3 | | 0.56 | |
| 2776 | ISO18254-1 | 112.18 | | -1.07 | |
| 3146 | ISO/DIS 18254-1 | 161.3783 | | 1.03 | |
| 3151 | ISO18254-1 | 121.66 | | -0.67 | |
| 3153 | ISO/DIS 18254-1 | 140 | | 0.12 | |
| 3154 | In house | 106.5 | | -1.31 | |
| 3172 | ISO/DIS 18254-1 | 122.5 | | -0.63 | |
| 3176 | In house | 123.20 | | -0.60 | |
| 3179 | In house/ISO18254-1 | 102 | | -1.51 | |
| 3197 | ISO18254-1 | 137.1 | | -0.01 | |
| 3200 | ISO18254-1 | 150.2 | C | 0.55 | First reported 38.4 |
| 3209 | In house | 92.2 | C | -1.92 | First reported 46.10 |
| 3210 | | ----- | | ----- | |
| 3214 | ISO18218-1 | 142.39 | | 0.22 | |
| 3218 | ISO18254-1 | 127 | | -0.44 | |
| 3220 | ISO18254-1 | 122.19 | | -0.64 | |
| 3222 | ISO18254-1 | 76.40 | | -2.60 | |
| 3232 | ISO/DIS 18254-1 | 144.94 | | 0.33 | |
| 3233 | In house | 147.5328 | | 0.44 | |
| 3237 | ISO/DIS 18254-1 | 97.9432 | | -1.68 | |
| 3248 | In house | 130 | | -0.31 | |

| | | | |
|---|---|---|---|
| | normality | OK | |
| | n | 92 | |
| | outliers | 1 | |
| | mean (n) | 137.256 | |
| | st.dev. (n) | 24.0361 | |
| | R(calc.) | 67.301 | |
| | R(Horwitz (n=5)) | 65.549 | |
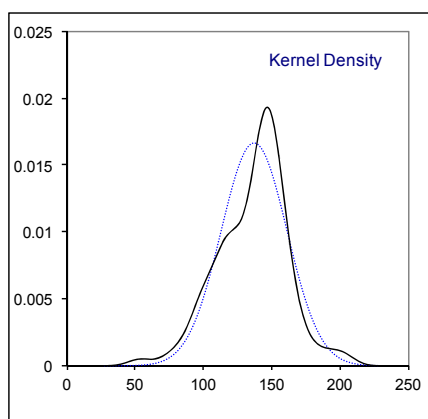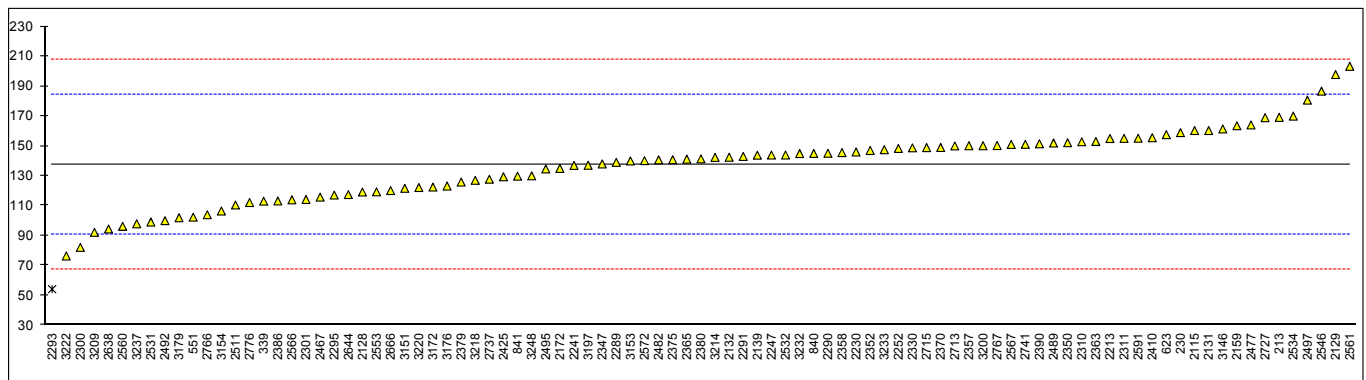




Kernel Density

## Determination of Total of OP, NP, OPEO and NPEO on sample #17531; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 110 | | ----- | | ----- | |
| 213 | In house | ----- | | ----- | |
| 230 | ISO18218-2 | 158.9 | | 0.85 | |
| 339 | In house | ----- | | ----- | |
| 551 | In house | 102.363898 | | -1.54 | |
| 623 | ISO/DIS 18254-1 | 157.60 | | 0.80 | |
| 840 | In house | 145 | | 0.26 | |
| 841 | ISO18254-1 | ----- | | ----- | |
| 2115 | ISO18254-1 | 160.33 | | 0.91 | |
| 2128 | ISO18254-1 | 119.2 | | -0.83 | |
| 2129 | ISO18218-1 | 197.8 | | 2.50 | |
| 2131 | In house | 160.3394 | | 0.91 | |
| 2132 | ISO18254-1 | 142.48 | | 0.16 | |
| 2139 | ISO18254-1 | 143.8456 | | 0.22 | |
| 2159 | ISO/DIS 18254-1 | 163.51 | | 1.05 | |
| 2172 | ISO18254-1 | ----- | | ----- | |
| 2213 | ISO/DIS 18254-1 | 155 | | 0.69 | |
| 2230 | ISO18254-1 | ----- | | ----- | |
| 2241 | ISO18254-1 | ----- | | ----- | |
| 2247 | ISO/DIS 18254-1 | 143.912 | | 0.22 | |
| 2252 | ISO18254-1 | 148.351 | | 0.41 | |
| 2265 | | ----- | | ----- | |
| 2289 | ISO18254-1 | 139.04 | | 0.01 | |
| 2290 | ISO18254-1 | 145.1 | | 0.27 | |
| 2291 | ISO18218-1 | 143 | | 0.18 | |
| 2293 | ISO18254-1 | 54.155 | R(0.05) | -3.58 | |
| 2295 | ISO/DIS 18254-1 | ----- | | ----- | |
| 2300 | ISO18254-1 | 82.1098 | | -2.40 | |
| 2301 | | 114.2 | | -1.04 | |
| 2310 | ISO18254-1 | 152.8 | | 0.59 | |
| 2311 | ISO18254-1 | 155.1 | | 0.69 | |
| 2330 | ISO18218-1 | 148.732 | | 0.42 | |
| 2347 | ISO18254-1 | 138 | | -0.03 | |
| 2350 | In house | 152.176 | | 0.57 | |
| 2352 | ISO18254-1 | 147.0 | | 0.35 | |
| 2357 | ISO18254-1 | 150.2 | | 0.48 | |
| 2358 | ISO18254-1 | 145.6 | | 0.29 | |
| 2363 | ISO18254-1 | 153 | | 0.60 | |
| 2365 | ISO18254-1 | 141.2 | | 0.10 | |
| 2369 | | ----- | | ----- | |
| 2370 | ISO18254 | 149 | | 0.43 | |
| 2375 | ISO18254-1 | ----- | | ----- | |
| 2379 | ISO18254-1 | 125.8403 | | -0.55 | |
| 2380 | In house | 141.3572 | | 0.11 | |
| 2386 | In house | 113.2 | | -1.08 | |
| 2390 | ISO18254-1 | 151.494 | | 0.54 | |
| 2410 | ISO/DIS 18254-1 | 155.54 | | 0.71 | |
| 2425 | In house | 129.4 | | -0.40 | |
| 2449 | GB/T23322 | ----- | | ----- | |
| 2467 | In house | ----- | | ----- | |
| 2477 | ISO18218-2 | 164.07 | | 1.07 | |
| 2482 | ISO18254-1 | ----- | | ----- | |
| 2489 | ISO18254-1 | 152 | | 0.56 | |
| 2492 | In house | 100.0 | | -1.64 | |
| 2495 | ISO/DIS 18254-1 | 134.59 | | -0.18 | |
| 2497 | ISO18254-1 | 180.678 | | 1.77 | |
| 2511 | ISO18254-1 | 110.53 | | -1.19 | |
| 2531 | ISO18218-2 | 99.0968 | | -1.68 | |
| 2532 | ISO/DIS 18254-1 | 143.99 | | 0.22 | |
| 2534 | ISO/DIS 18254-1 | 170 | | 1.32 | |
| 2546 | ISO18254-1 | 188.95 | | 2.12 | |
| 2553 | ISO18254-1 | 119.32 | | -0.82 | |
| 2560 | In house | n.d. | | ----- | False negative test result? |
| 2561 | | ----- | | ----- | |
| 2566 | In house | 114 | | -1.05 | |
| 2567 | ISO/DIS 18254-1 | 151 | | 0.52 | |
| 2572 | ISO18254-1 | 140.2 | | 0.06 | |
| 2590 | ISO/DIS 18254-1 | 86.7470 | | -2.20 | |
| 2591 | In house | 155.20 | | 0.70 | |
| 2638 | In house | ----- | | ----- | |
| 2644 | ISO18254-1 | 118 | | -0.88 | |
| 2666 | ISO18218-2 | 120.1648 | | -0.79 | |
| 2713 | In house | 150.03 | | 0.48 | |
| 2715 | In house | 148.92 | | 0.43 | |
| 2727 | ISO18218-1 | ----- | | ----- | |

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 2737 | ISO18254-1 | ----- | | ----- | |
| 2741 | ISO18254-1 | 151.1 | | 0.52 | |
| 2766 | ISO18254-1 | 278 | C,R(0.01) | 5.89 | First reported 284 |
| 2767 | ISO/DIS 18254-1 | 150.3 | | 0.49 | |
| 2776 | ISO18254-1 | 113.27 | | -1.08 | |
| 3146 | ISO/DIS 18254-1 | 161.3783 | | 0.96 | |
| 3151 | ISO18254-1 | 121.66 | | -0.72 | |
| 3153 | ISO/DIS 18254-1 | 140 | | 0.05 | |
| 3154 | In house | ----- | | ----- | |
| 3172 | ISO/DIS 18254-1 | 122.5 | | -0.69 | |
| 3176 | In house | 123.20 | | -0.66 | |
| 3179 | In house/ISO18254-1 | 102 | | -1.56 | |
| 3197 | ISO18254-1 | ----- | | ----- | |
| 3200 | ISO18254-1 | ----- | | ----- | |
| 3209 | In house | ----- | | ----- | |
| 3210 | | ----- | | ----- | |
| 3214 | ISO18218-1 | 142.39 | | 0.15 | |
| 3218 | ISO18254-1 | 127 | | -0.50 | |
| 3220 | ISO18254-1 | 122.19 | | -0.70 | |
| 3222 | ISO18254-1 | 77.72 | | -2.58 | |
| 3232 | ISO/DIS 18254-1 | ----- | | ----- | |
| 3233 | In house | 147.5328 | | 0.37 | |
| 3237 | ISO/DIS 18254-1 | ----- | | ----- | |
| 3248 | In house | 130 | | -0.37 | |

| | | |
|---|---|---|
| | normality | OK |
| | n | 71 |
| | outliers | 2 |
| | mean (n) | 138.753 |
| | st.dev. (n) | 23.1471 |
| | R(calc.) | 64.812 |
| | R(Horwitz (n=5)) | 66.155 |





Kernel Density
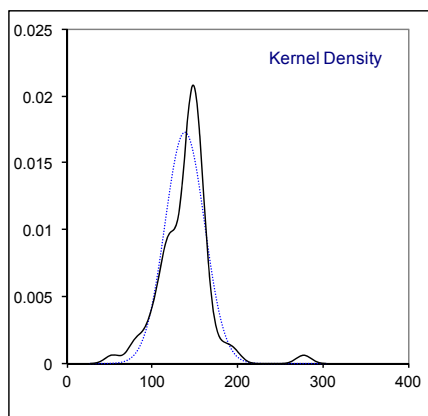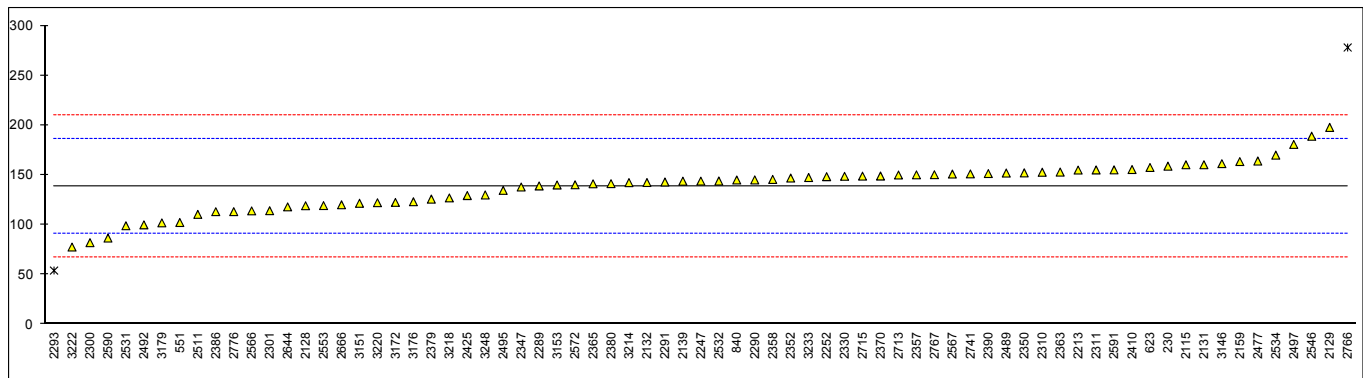
## APPENDIX 2

Summary of other reported components in sample #17530

| lab | Components | remarks |
|-----|-----------|---------|
| 551 | 4.769859 mg/kg NPEO | |
| 2497 | 8.738 mg/kg NPEO | |
| 2546 | 1.2 mg/kg NPEO | |
| 2766 | 80 mg/kg OP | False positive test result OP? First reported 89 mg/kg OP |
| 2776 | 0.67 mg/kg OP, 0.26 mg/kg NP | |
| 3154 | 21.38 mg/kg NP | False positive test result NP? |
| 3222 | 1.19 mg/kg NPEO | |

Summary of other reported components in sample #17531

| lab | Components | remarks |
|-----|-----------|---------|
| 2546 | 2.25 mg/kg OPEO | |
| 2590 | 137.2 mg/kg OPEO | Possibly mixed up with NPEO. First reported 86.7470 mg/kg OPEO |
| 2766 | 174 mg/kg OP | False positive test result OP? First reported 180 mg/kg OP |
| 2776 | 0.75 mg/kg OP, 0.34 mg/kg NP | |
| 3154 | 16.32 mg/kg NP | False positive test result NP? |
| 3222 | 1.32 mg/kg OPEO | |

## Abbreviations of components:

OP =        Octylphenol

NP =        Nonylphenol

OPEO =  Octylphenol Ethoxylates

NPEO =  Nonylphenol Ethoxylates

## APPENDIX 3

Other reported components in sample #17530 & #17531; results in mg/kg (see abbreviations in appendix 2)

| Lab | sample #17530 | | | sample #17531 | | |
|---|---|---|---|---|---|---|
| | OP | NP | NPEO | OP | NP | OPEO |
| 110 | ----- | ----- | ----- | ----- | ----- | ----- |
| 213 | ----- | ----- | ----- | ----- | ----- | ----- |
| 230 | ----- | ----- | ----- | ----- | ----- | ----- |
| 339 | <50 | <50 | <50 | <50 | <50 | <50 |
| 551 | n.d. | n.d. | 4.769859 | n.d. | n.d. | n.d. |
| 623 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 840 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 841 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2115 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2128 | < 5 | < 5 | < 10 | < 5 | < 5 | < 10 |
| 2129 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2131 | 0 | 0 | 0 | 0 | 0 | 0 |
| 2132 | <10 | <10 | <10 | <10 | <10 | <10 |
| 2139 | <3 | <3 | <30 | <3 | <3 | <30 |
| 2159 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2172 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2213 | <10 | <10 | <10 | <10 | <10 | <10 |
| 2230 | <10 | <10 | <50 | <10 | <10 | <50 |
| 2241 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2247 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2252 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2265 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2289 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2290 | < 10 | < 10 | < 10 | < 10 | < 10 | < 10 |
| 2291 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2293 | n.d. | BRL | BRL | n.d. | BRL | n.d. |
| 2295 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2300 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2301 | 0 | 0 | 0 | 0 | 0 | 0 |
| 2310 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2311 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2330 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2347 | <30 | <30 | <30 | <30 | <30 | <30 |
| 2350 | 0 | 0 | 0 | 0 | 0 | 0 |
| 2352 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2357 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2358 | <10 | <10 | <30 | <10 | <10 | <10 |
| 2363 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2365 | <10 | <10 | <30 | <10 | <10 | <30 |
| 2369 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2370 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2375 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2379 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2380 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2386 | <10 | <10 | <10 | <10 | <10 | <10 |
| 2390 | n.d. | n.d | n.d. | n.d. | n.d. | n.d. |
| 2410 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2425 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2449 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2467 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2477 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2482 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2489 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2492 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2495 | <2 | <2 | <2 | <2 | <2 | <2 |
| 2497 | ----- | ----- | 8.738 | ----- | ----- | ----- |
| 2511 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2531 | 0 | 0 | 0 | 0 | 0 | 0 |
| 2532 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2534 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2546 | 0 | 0 | 1.2 | 0 | 0 | 2.25 |
| 2553 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2560 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2561 | ----- | ----- | ----- * | ----- | ----- | ----- * |
| 2566 | n.d | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2567 | <10 | <10 | <10 | <10 | <10 | <10 |
| 2572 | <10 | <10 | <10 | <10 | <10 | <10 |
| 2590 | < L.O.Q. | < L.O.Q. | < L.O.Q. | < L.O.Q. | < L.O.Q. | 137.2 |
| 2591 | <10 | <10 | <10 | <10 | <10 | <10 |
| 2638 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2644 | ----- | ----- | ----- | ----- | ----- | ----- |

| | sample #17530 | | | sample #17531 | | |
|---|---|---|---|---|---|---|
| **Lab** | **OP** | **NP** | **NPEO** | **OP** | **NP** | **OPEO** |
| 2666 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2713 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2715 | ----- | ----- | -------- | -------- | -------- | -------- |
| 2727 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2737 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2741 | <3 | <3 | <3 | <3 | <3 | <3 |
| 2766 | 80 | ----- | ----- | 174 | ----- | ----- |
| 2767 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 2776 | 0.67 | 0.26 | n.d. | 0.75 | 0.34 | n.d. |
| 3146 | ----- | ----- | ----- | ----- | ----- | ----- |
| 3151 | <3 | <3 | <10 | <3 | <3 | <10 |
| 3153 | <10 | <10 | <10 | <10 | <10 | <10 |
| 3154 | ----- | 21.38 | ----- | ----- | 16.32 | ----- |
| 3172 | < 1 | < 1 | < 1 | < 1 | < 1 | < 1 |
| 3176 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 3179 | <5 | <5 | <20 | <5 | <5 | <20 |
| 3197 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 3200 | <10 | <10 | <10 | <10 | <10 | <10 |
| 3209 | ----- | ----- | ----- | ----- | ----- | ----- |
| 3210 | ----- | ----- | ----- | ----- | ----- | ----- |
| 3214 | <1 | <1 | <1 | <1 | <1 | <1 |
| 3218 | <10 | <10 | <10 | <10 | <10 | <10 |
| 3220 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 3222 | ----- | ----- | 1.19 | ----- | ----- | 1.32 |
| 3232 | ----- | ----- | n.d. | ----- | ----- | n.d |
| 3233 | n.d. | n.d. | n.d. | n.d. | n.d. | n.d. |
| 3237 | ----- | ----- | ----- | ----- | ----- | ----- |
| 3248 | ----- | ----- | ----- | ----- | ----- | ----- |

*) lab 2561: first reported NPEO = 203.300 on sample #17530, first reported OPEO = 105.75 on sample #17531

## APPENDIX 4

Details of the methods used by the participants

| Lab | Brief summary of the method used | Component(s) used for quantification |
|---|---|---|
| 110 | | |
| 213 | In house method, The extraction has been done in ultrasonic with methanol at 70°C for 60 min | OPEO 9-10 for sample #17530 / NPEO9-10 for sample #17531 |
| 230 | 1 g of sample, 20 ml of methanol, sonicate at 70 C for 60 min | OPEO 9-10, NPEO 9-10 |
| 339 | In house methode : 1g + 20mL MeOH 　¨US 60min 70 ‹C | 4-Nonylphenol-Ethoxylate: CAS: 68412-54-4 Igepal Co-520: CAS: 68412-54-4 Igepal Co-630: CAS: 68412-54-4 POE (12) Nonylphenol POE (14) Nonylphenol POE (20) Nonylphenol 4-Tert-Octylphenol (Op): CAS: 140-66-9 Nonylphenol: CAS: 84852-15-3 4-n-Nonylphenol: CAS: 104-40-5 |
| 551 | | |
| 623 | | OPEO (9-10) and NPEO (9-10) |
| 840 | | |
| 841 | | |
| 2115 | official method | various |
| 2128 | DIN ISO 18254-1 | |
| 2129 | EN ISO 18218-1:2015-11 (modified); extraction in ultrasonic bath for 60 min at 70 °C with methanol | nNP: 84852-15-3; nOP: 1806-26-4; t-OP: 140669; Triton X-100: 9002931; Igepal Co-630: 68412544 |
| 2131 | Oeko-Tex method | CAS No. 104-40-5;CAS No. 1806-26-4; CAS No. 68412-54-4; CAS No. 9036-19-5 |
| 2132 | Scan mode follow ISO 18254 | NPEO: CAS 9016-45-9; OPEO: CAS 9002-93-1 |
| 2139 | ISO18254-1 | OP:1806-26-4,NP:104-40-5,OPEO:9002-93-1,NPEO:68412-54-4 |
| 2159 | APEO in textile by HPLC-MS (AFIRM, PR EN ISO 18254)& AP in textile by GC-MS(AFIRM ISO MODIFIED CADS) | OP-CAS no:27193-28-8; NP CAS no:84852-15-3; NPEO CAS no:68412-54-4; OPEO CAS no:9002-93-1 |
| 2172 | | |
| 2213 | ISO 18254-1 | |
| 2230 | Followed ISO 18254-1, Solvent extraction and analyzed by LC-MS and GC-MS. | OPEO CAS 9002-93-1, NPEO CAS 68412-54-4 |
| 2241 | 0.5g sample 20ml methanol 70 degree c for 60 mins | NPEO CAS No.68412-54-4 OPEO CAS No.9002-93-1 |
| 2247 | iso/dis 18254 -1,1gm spl+20 ml Meoh sonicated at 70 digree for 1 hr,filter followed LCMS analalysis. | Triton cas no 9002-93-1:IGPAL CO-630 cas ni:68412-54-4, OP (4 n- oct 1806-26-4:np cas no 104-40-5 , |
| 2252 | Textiles-Method for the detection and determination of alkylphenolethoxylates APEO-Part1: Method using HPLC-MS | 9016-45-9 for NPEO;9002-93-1 for OPEO |
| 2265 | | |
| 2289 | ISO 18254-1:2016 The sample is extracted with methanol using ultrasound and determined by HPLC-MS. | X-100/CAS 9002-93-1;CO-630NPEO/CAS 68412-54-4 |
| 2290 | | |
| 2291 | ISO 18218-1 | NPEO£¨9-10£©CAS 68412-54-4£¬OPEO(9-10)CAS 9002-93-1,NP CAS 84852-15-3,OP CAS 140-66-9 |
| 2293 | ISO18254-1(1g sample, extracted 20 mL methanol/ACN 1:1; 60min at 70°C in ultrasonic bath plus 5mLH2O | OPEOs; Triton X-100 CAS 9002-93-1; NPEOs; IGEPAL CO-630 CAS 68412-54-4; 4-n-Octylphenol CAS 1806-26-4; 4-tert-Octylphenol CAS 140-66-9; 4-n-Nonylphenol CAS 104-40-5; Nonylphenol; mixture of ring and chain isomers CAS 84852-15-3 |
| 2295 | ISO/DIS 18254-1, methanol extraction | APEO CAS:9002-93-1, NPEO CAS:68412-54-4 |
| 2300 | ISO 18254-1 | 4-Nonylphenol Ethoxylate(CAS NO 68412-54-4) & 4-Octyllphenol Ethoxylate( CAS NO 26636-32-8) |
| 2301 | AFIRM, Methanol extraction and analysis by (LC-MS for APEO and GCMS for AP) | OPEO CAS no. 9002-93-1 , NPEO CAS no. 68412-54-4 , n-OP CAS no. 1806-26-4, NPs CAS no. 1806-26-4 |
| 2310 | ISO 18254-1: 2016. Extraction using Methanol/Analysis by LC-MS | NPEO: IGEPAL CO-630(CAS No.68412-54-4) OPEO: Triton X-100(CAS No.9002-93-1) |
| 2311 | ISO18254-1, 1gm sample in 20ml Methanol. Sonicated at 70°C for 1hours, analysis by LCMS | NPEO (CAS number: 68412-54-4 ), OPEO (CAS number: 9002-93-1) |
| 2330 | ISO 18254-1:2016 | POE(9-10) Test-octylphenol ; CAS 9002-93-1, POE(9-10) nonylphenol ; CAS 9016-45-9 |
| 2347 | ISO 18254-1:2016 | |
| 2350 | In house method | NP(25154-52-3), OP(140-66-9), NPEO(68412-54-4), OPEO(9002-93-1) |
| 2352 | ISO18254-1:2016 | OPEO9 CAS NO.:9002-93-1; NPEO9 CAS NO.:127087-87-0 |
| 2357 | ISO 18254-1:2016 | OPEO£º9002-93-1£¬NPEO£º68412-54-4 |
| 2358 | ISO 18254-1 | |
| 2363 | ISO 18254-1 | NPEO 9& OPEO 9 |
| 2365 | The cutted sample was extracted with methanol using ultrasond. the analysis was performed by LC-MS | OPEO:CAS no. 9002-93-1; NPEO: CAS no: 684231-54-4; NP: 25154-52-3/104-40-5, OP: 140-66-9/180-26-4 |

| Lab | Brief summary of the method used | Component(s) used for quantification |
|---|---|---|
| 2369 | | |
| 2370 | Method used: ISO18254 Q1.Cut the textile sample into pieces of approximately 5 mm ¡Ñ 5 mm and mix them homogeneously. Prepare approximately 1 g of the cut textile, weigh it to the nearest 10 mg, and then place it into the glass container (extraction vessel). Pipette 20 ml of methanol into the glass container (extraction vessel). Place the glass container (extraction vessel) into an ultrasonic bath at 70 ¢XC for (60 ¡Ó 5) min. Afterwards, let the extract cool down to room temperature. Filter about 1 ml of the extraction solution into a HPLC vial using a disposable syringe equipped with a membrane filter. | 1.OP9-10EO(Triton X-100):9002-93-1 2.NP9-10EO(IGEPAL CO-630):68412-54-4 |
| 2375 | ISO 18254-1 | 9to10-NPEO10 |
| 2379 | ISO 18254-1:2016 | NPEO 9-10 CAS NO.:68412-54-4 , OPEO 9-10 CAS NO.:9002-93-1 |
| 2380 | In house | NPEO = POE (9-10) Nonylphenol, OPEO = POE (9-10) tert-Octylphenol |
| 2386 | Ultrasonic 70°C 60min Methanol | IGEPAL CO-630 / Triton x100 |
| 2390 | ISO 18254-1 : 2016 | NPEO 9-10, OPEO 9-10 |
| 2410 | ISO DIS 18254 | NPEO 68412-54-4, OPEO 9002-93-1 |
| 2425 | In house method, 0.5 gm sample extraction with Methanol and quantification by UPLC-DAD-MS | OPEOs (Triton-X-100), CAS: 9002-93-1 and NPEO (IGEPAL-CO-630), CAS: 68412-54-4 |
| 2449 | GB/T23322 | APEO |
| 2467 | Determination of APEOs and APs in Textiles and Plastics-The extraction of APEOs and APs is done wi | OPEO: CAS: 9002-93-1; NPEO : CAS 68412-54-4 |
| 2477 | ISO 18218-2 Leather-Determination of ethoxylated alkylphenols part 2: indirect method. | 4n-nonylphenol (4n-NP, CAS no. 104-40-5) |
| 2482 | | |
| 2489 | ISO 18254-1, 0.5g sample+20 ml methanol, 70°C, sonication for 1 hour, filter the solution, analysis | Triton x 100 (CAS NO: 9002-93-1)/IGEPAL CO-630 (CAS NO: 68412-54-4) |
| 2492 | In-house Method | |
| 2495 | ISO/DIS 18254-1 extraction with methanol and analysis with LC/MS/MS | OPEO: Triton X (CAS 9002-93-1); NPEO: IGEPAL CO-630 (CAS 68412-54-4) |
| 2497 | | |
| 2511 | ISO 18254-1 | for sample #17530: 68412-54-4, for sample #17531: 9002-93-1 |
| 2531 | Annex B - HPLC | cas 84852–15–3, cas 140–66–9, cas 9016– 45–9, cas 9002– 93–1 |
| 2532 | 1g sample extracted with 20ml Methanol using ultrasound @ 70°C .added 5 ml water and filtered | Triton X100 CAS No. 9002-93-1 IGEPAL CO630 -CAS No 68412-54-4 Nonylphenol - CAS 84852-15-3 Octyl Phenol - CAS No 27193-28-8 |
| 2534 | Method ISO 18254-1:2016 | Triton X-100 CAS n. 9002-93-1, Igepal CO-630 CAS n. 68412-54-4 for ethoxylates; The components for the quantification of NP/OP are: 4-nonylphenol (mix of isomers) CAS 84852-15-3, 4-tert-octylphenol CAS n. 140-66-9 |
| 2546 | ISO 18254-1:2016 | NPEO CAS#68412-54-4 OPEO CAS#9002-93-1 OP CAS#140-66-9 NP CAS#84852-15-3 |
| 2553 | Cut the textile or leather sample into pieces with 5 mm x 5 mm and mix homogeneously. Weigh 1 g into a 40 mL reaction vial. Pipette 20 mL methanol to the reaction vial Place the vial into an ultrasonic bath at 70oC for 60 minutes. For NPEOs and OPEOs analysis,filter the extract through a 0.45ƒÝm nylon filter disc into an injection vial for LC-MS analysis. Inject Vial for LC-MS analysis. For APs analysis, Pipette 1 mL sample extract and spike 10 ƒÝL of 100 mg/L Anthracene -d10 internal standard stock solution into injection vial, mix well and reserve for GC-MS analysis. MDL: 0.2 mg/L Reporting limits: APEO- 50 mg/kg AP ¡V 10 mg/kg | |
| 2560 | In house method; sonicate at 70°C for 60 min  Detection Limit = 3 mg/kg | CAS NOs of the components: 25154-52-3; 1806-26-4; 27193-28-8; 68412-54-4; 9002-93-1; 104-40-5 |
| 2561 | | |
| 2566 | INHOUSE METHOD | NPEO CAS no. 68412-54-4 and OPEO CAS no. 9002-93-1 |
| 2567 | Method: ISO 18254; Extraction solvent: methanol, Ultrasonic extraction 1h 70°C, analysed by LCMSMS. | OPEOs: TritonX-100 (9002-93-1), NPEO: IGEPAL CO-630 (68412-54-4). |
| 2572 | | |
| 2590 | ISO/DIS 18254-1 (extraction with MeOH into an ultrasonic bath at 70°C for 60 min) | NPEOs (CAS no. 68412-54-4) - OPEOs (CAS 9002-93-1) |
| 2591 | In house method based on ISO18218-1 with some modifications on procedure and instrumental conditions | Triton-X-100 (9002-93-1); 4-Octylphenol-ethoxylate(mono-, di-, tri-) (26636-32-8) N-40 Alternative (4-nonylphenyl-polyethylene) (9016-45-9) 4-Nonylphenol-ethoxylate(mono-, di-, tri-) (68412-54-4) 4-Octylphenol (1806-26-4) 4-tert-octylphenol (140-66-9) Nonoxynol-9 (26027-38-3) P-(1,1,3,3-Tetramethylbuyl)phenol (27193-28-8) Nonylphenol |

| Lab | Brief summary of the method used | Component(s) used for quantification |
|---|---|---|
| | | (25154-52-3) 4-n-nonylphenol (104-40-5) Nonylphenol isomers (601-53-00-8) |
| 2638 | 0.5 gm sample/ 10 methanol. sonicate for 60 min at room temperature. filter and analyse on HPLC-DAD | 9-10 NPEO. CAS # 9016-45-9 |
| 2644 | extraction with US at 70°C for 1 hour, bring to room temperature filter with 0.2 ptfe, HPLC-MS | triton x100 for opeo and igepal co-630 for npeo |
| 2666 | ISO 18218-2:2015 | OP(CAS 140-66-9/27193-28-8);NP(CAS 84852-15-3/25154-52-3);OPEO(CAS 9002-93-1);NPEO(CAS 68412-54-4) |
| 2713 | In house method: 1 g sample is extracted by 20 ml methanol in ultrasonic bath | NPEO:68412-54-4; NP:84852-15-3; OPEO: 9002-93-1 ; OP: 1806-26-4 |
| 2715 | 20 mL of methanol was added to 1.0 g of the sample and the Ultrasonic extraction was carried out for 60 min. The extracts were evaporated to dryness and then re-dissolved with 1 mLof methanol. The extraction solutions were filtered through a 0.45¦lm filter prior to chromatographic analysis | Octyphenol ethoxylate (OPEO, CAS 9002-93-1), Nonylphenol ethoxylate (NPEO, CAS 009016-45-9) |
| 2727 | ISO18218-1; EXTRACTION IN METHANOL 1H SONICATION AT 70ºC | IGEPAL CO-630 CAS: 68412-54-4; TRITON X-100 CAS: 9002-93-1 |
| 2737 | ISO 18254-1 | NPEO 68412-54-4; OPEO 9002-93-1 |
| 2741 | | |
| 2766 | ISO18254-1 for APEO and ISO18218-2 for AP, extraction in methanol by sonication and analysis by LCMS | Triton X-100 for OPEO and Tergetol NP-9 for NPEO, we used pure OP, TOP and NP for AP. |
| 2767 | ISO 18254-1 ( Methanol- Acetonitrile extraction and determination by HPLC-MS ) | OPEO ( Cas number : 9002-93-1 ) , NPEO ( Cas number : 68412-54-4 ) |
| 2776 | ISO18254-1 | NP(25154-52-3);OP(140-66-9);NPEO(9016-45-9);OPEO(9002-93-1) |
| 3146 | 18254-1 | OPEOs; Triton X-100 CAS: 9002-93-1; NPEOs; IGEPAL CO-630 CAS: 68412-54-4 |
| 3151 | 1 g sample was extracted with 20 mL Methanol; AP analysis with GC-MS and APEOs with LC-MS | Nonylphenol (techn.) (25154-52-3); Nonylphenol (84852-15-3); 4-n-Nonylphenol (104-40-5); Nonylphenol |
| 3153 | ISO 18254-1 | OPEO Triton X-100 CAS No. 9002-93-1; NPEO IGEPAL CO-360 CAS No. 68412-54-4 |
| 3154 | | |
| 3172 | | |
| 3176 | in house method | 1806-26-4 ,68412-54-4 ,9002-93-1, 104-40-5 |
| 3179 | OP+NP: In-House, GC-MS, after solvent extraction;;;APEO: ISO 18254-1 | NP: 25154-52-3/OP:1806-26-4 and 14-66-9//APEO: according to standard |
| 3197 | 1 g sample ; 20 mL methanol ; 70C ultrasonic bath for 60 minutes | NPEO (IGEPAL CO630) Cas No: 68412-54-4 / OPEO (TRITON X-100) Cas No: 9002-93-1 |
| 3200 | MeOH 70℃ ultrasonic 60min,LC-MSMS analysis | NPEO (68412-54-4),OPEO (9036-19-5) |
| 3209 | 0.5g sample in 10ml MEOH, 70 degree ultrasonic 1h | NPEOs CAS NO.9016-45-9;OPEOs CAS NO.9036-19-5 |
| 3210 | | |
| 3214 | Solvent extraction and determined by LC/MS | NP(25154-52-3), OP(140-66-9), NPEO(68412-54-4), OPEO(9002-93-1) |
| 3218 | 1.0g sample into the tube, add 20ml MeOH, then place the tube into ultrasonic bath at 70¡æ for 60 mi | NPEO(n-9):68412-54-4,OPEO(n-10):9002-93-1 |
| 3220 | ISO 18254-1(1gm sample extracted in 25ml Methanol sonicate at 70°C for 1 hr & analyzed by LC-MS/MS   ND-Not detected   Detection Limit- 2mg/kg   NP/OP : BASED ON ASTM D 7065 (GC - MS) | NPEO-IGEPAL CO-630 (CAS No. 68412-54-4), OPEO-Triton X-100(CAS No. 9002-93-1)    NP-CAS No. 84852-15-3 OP-CAS No. 140-66-9 |
| 3222 | Extraction with methanol at 40°C- quantitative determination : LC MS/MS | OPEO CAS nr. 9002-93-1 / 9036-19-5 - NPEO CAS nr. 68412-54-4 / 127087-87-0 |
| 3232 | 1.0 g of sample in 20 ml of Methanol,Keep it for ultra-sonication for 60min at ambient temperatures. | Triton x-100,CAS NO:9002-93-1 and IGEPAL CO-630,CAS NO:68412-54-4 |
| 3233 | In house method : extraction with methanol in ultrasonic bath (40°C-1H) | OPEO : Triton X100 [9002-93-1] / NPEO : Igepal CO-630 [68412-54-4] |
| 3237 | Methanol extraction+Water bath @70 C+ LC MS MS | NPEO (68412-54-4) OPEO(9002-93-1) |
| 3248 | | |

## APPENDIX 5

## Number of participants per country

  3 labs in  BANGLADESH

  1 lab in  BRAZIL

  2 labs in  CAMBODIA, Kingdom of

  3 labs in  FRANCE

  9 labs in  GERMANY

  1 lab in  GUATEMALA

  5 labs in  HONG KONG

11 labs in  INDIA

  2 labs in  INDONESIA

11 labs in  ITALY

  3 labs in  KOREA

  1 lab in  MAURITIUS

  1 lab in  MEXICO

  1 lab in  MOROCCO

17 labs in  P.R. of CHINA

  3 labs in  PAKISTAN

  1 lab in  PORTUGAL

  1 lab in  ROMANIA

  1 lab in  SPAIN

  1 lab in  SRI LANKA

  1 lab in  SWITZERLAND

  3 labs in  TAIWAN R.O.C.

  1 lab in  THAILAND

  1 lab in  TUNISIA

  8 labs in  TURKEY

  1 lab in  U.S.A.

  1 lab in  UNITED KINGDOM

  5 labs in  VIETNAM

## APPENDIX 6

**Abbreviations**:

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

**Literature**:

| | |
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