Institute for
Interlaboratory Studies

# Results of Proficiency Test
# AP and APEO in Textile
# March 2023


Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. C.M. Nijssen-Wester
Correctors: ing. R.J. Starink & Mrs. E.R. Montenij-Bos
Approved by: ing. A.S. Noordman-de Neef



Report: iis23T31

April 2023

**CONTENTS**

# 1 INTRODUCTION

Alkylphenol Ethoxylates (APEO), like Octylphenol Ethoxylates (OPEO) and Nonylphenol Ethoxylates (NPEO) have widely been used in manufacturing antioxidants, lubricating oil additives, laundry and dish detergents, emulsifiers, wetting agents in cosmetics, including hair products, defoaming agents and solubilizers. 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 determination of AP and APEO in Textile every year. During the annual proficiency testing program 2022/2023 it was decided to continue the proficiency test (PT) for the analysis of AP and APEO in textile.

In this interlaboratory study 90 laboratories in 22 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 #23515 and #23516 respectively.
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 blue cotton was selected which was made positive on OPEO by a third-party laboratory. This batch was cut into small pieces. After homogenization 115 small plastic bags were filled with approximately 3 grams each and labelled #23515.
The batch for sample #23515 was used in a previous proficiency test on AP and APEO in Textile (as sample #17530 in iis17A04). Therefore, homogeneity of the subsamples was assumed.

For the second sample a batch of beige cotton was selected which was made positive on NPEO by a third-party laboratory. This batch was cut into small pieces. After homogenization 115 small plastic bags were filled with approximately 3 grams each and labelled #23516.
The batch for sample #23516 was used in a previous proficiency test on AP and APEO in Textile (as sample #21526 in iis21T01). Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories two textile samples labelled #23515 and #23516 respectively were sent on February 8, 2023.

## 2.5 ANALYZES

The participants were requested to determine the total concentrations of the following components: Octylphenol (OP), Nonylphenol (NP), Octylphenol Ethoxylates (OPEO), Nonylphenol Ethoxylates (NPEO) and the sum of OP, NP, OPEO and NPEO on both samples #23515 and #23516.
To ensure homogeneity it was requested to not use less than 0.5 grams per determination. It was also requested to report if the laboratory was accredited for the determined components and to report some analytical details.

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 appendices 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 appendices 1 and 2. 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 (derived from e.g. ISO or ASTM test methods), 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 no problems were encountered with the dispatch of the samples. Six participants reported test results after the final reporting date and five other participants did not report any test results. Not all participants were able to report all components requested. In total 85 participants reported 170 numerical test results. Observed were 2 outlying test results, which is 1.2%. 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 in appendix 1. The abbreviations, used in these tables, are explained in appendix 5.

For the determination of AP and APEO in textile test method ISO18254-1, used by the majority of the participants, is considered to be the official test method. Regretfully, only one general precision statement is given for APEO at one concentration and it is not mentioned which APEO is used. In Table A.2 of ISO18254-1 the reproducibility is 262 mg/kg at 954 mg/kg APEO. This concentration is much higher than the APEO or AP found in the iis PTs. Furthermore, the concentration is also far above the rejection limit of known environmental standards (e.g. OEKO-TEX®).

In 2022 iis decided to use the iis PT data gathered from 2016 to 2021 to estimate a more realistic target reproducibility for the evaluation of the quality of the test results for the determination of AP and APEO in Textile. Furthermore, it was decided to use the same target reproducibility for all individual AP and APEO. The average relative standard deviation over all iis PTs of APEO and AP in Textile is 17%. This investigation is summarized in iis memo 2203. For comparison the target of ISO18254-1 is also mentioned in appendix 1.

**sample #23515**

Total OPEO: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the target reproducibility as derived from iis memo 2203.

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

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

**sample #23516**

Total NPEO: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the target reproducibility as derived from iis memo 2203.

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

The participants agreed on the levels for the components OP, NP and OPEO in this sample were near or below the detection limit and therefore no z-scores are 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 reference test method 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 derived from reference methods are presented in the next tables.

| Component | unit | n | average | 2.8 * sd | R(target) |
|---|---|---|---|---|---|
| Total OPEO | mg/kg | 84 | 107.2 | 28.2 | 51.0 |

Table 1: reproducibility of components on sample #23515

| Component | unit | n | average | 2.8 * sd | R(target) |
|---|---|---|---|---|---|
| Total NPEO | mg/kg | 84 | 129.0 | 45.4 | 61.4 |

Table 2: reproducibility of components on sample #23516

Without further statistical calculations it can be concluded that for both components there is a good compliance of the group of participants with the target method.

### 4.3 COMPARISON OF THE PROFICIENCY TEST OF MARCH 2023 WITH PREVIOUS PTS

| | March 2023 | March 2022 | March 2021 | March 2020 | February 2019 |
|---|---|---|---|---|---|
| Number of reporting laboratories | 85 | 92 | 100 | 96 | 105 |
| Number of test results | 170 | 526 | 444 | 347 | 366 |
| Number of statistical outliers | 2 | 28 | 15 | 17 | 21 |
| Percentage of statistical outliers | 1.2% | 5.3% | 3.4% | 4.9% | 5.7% |

Table 3: 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.

| Component | March 2023 | March 2022 | March 2021 | March 2020 | 2019 - 2016 | Target |
|---|---|---|---|---|---|---|
| Total Octylphenol (OP) | -- | -- | 12% | -- | -- | 17% |
| Total Nonylphenol (NP) | -- | 20% | -- | -- | -- | 17% |
| Total Octylphenol Ethoxylates (OPEO) | 9% | 13% | 12% | 17% | 10-16% | 17% |
| Total Nonylphenol Ethoxylates (NPEO) | 13% | 10-18% | 15% | 27% | 13-28% | 17% |
| Sum of OP + NP + OPEO + NPEO | -- | 9-16% | 12% | 16-28% | 11-26% | 17% |

Table 4: development of uncertainties over the years

The uncertainties observed in this PT are comparable to the uncertainties observed in previous iis PTs.

### 4.4 EVALUATION OF THE ANALYTICAL DETAILS

For this PT some analytical details were requested. The answers are given in appendix 3. Based on the answers given by the participants the following can be summarized:

- About 90% mentioned that they are ISO/IEC17025 accredited to determine the reported components.
- About 75% further cut the samples prior to analysis and about 25% used the samples as received.
- About 40% around 0.5 grams and about 50% around 1 gram for the sample intake.
- All reporting participants used Ultrasonic technique to extract/release the components from the samples.
- All reporting participants used Methanol as extraction solvent.
- Almost all participants used an extraction/release time of 60 minutes and used an extraction/release temperature of 70°C.

As the majority of the group follow the same analytical procedures no separate statistical analysis has been performed.

## 5 DISCUSSION

All reporting participants were able to detect Octylphenol Ethoxylates (OPEO) in sample #23515 and Nonylphenol Ethoxylates (NPEO) in sample #23516.

When the test results of this interlaboratory study were compared to the OEKO-TEX® 100, the Bluesign® system and EU regulation 2016/26 amending Reach as regards to NPEO on Textiles (see table 5) it is noticed that not all participants would have made identical decisions about the acceptability of the samples for the determined components. Considering the limits for OPEO and NPEO for sample #23515 eighteen participants would have accepted the sample whereas sixty-seven participants would have rejected the sample for containing too much OPEO. For sample #23516 all most all participants would have accepted the sample, one participant would have rejected this sample for containing too much NPEO.

| | OEKO-TEX®100 Annex 4 in mg/kg | OEKO-TEX®100 Annex 6 in mg/kg | Bluesign® RSL v13.0 in mg/kg | EU 2016/26 in mg/kg |
|---|---|---|---|---|
| NP | -- | -- | 10 | -- |
| OP | -- | -- | 10 | -- |
| NPEO | -- | -- | 100 *) | 100 |
| OPEO | -- | -- | 100 *) | -- |
| OP + NP | 10 | 5 | -- | -- |
| OP+NP+OPEO+NPEO | 100 | 50 | -- | -- |

Table 5: Ecolabelling Standards and EU regulatory limits for Textiles in EU

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

Sample #23515 was used before in proficiency test iis17A04 as sample #17530. It is observed that the average concentrations of sample #23515 is in line with the previous PT, see next table. The observed reproducibility (expressed as 2.8 * sd) is much better in 2023 iis PT.

| Component | unit | #23515 | | | #17530 | | |
|---|---|---|---|---|---|---|---|
| | | n | average | 2.8 * sd | n | average | 2.8 * sd |
| Total OPEO | mg/kg | 84 | 107.2 | 28.2 | 92 | 108.9 | 46.0 |

Table 6: comparison of sample #23515 with sample #17530

Sample #23516 was used before in proficiency test iis21T01 as sample #21526. It is observed that the average concentrations of sample #23516 is in line with the previous PT, see next table. Again the observed reproducibility (expressed as 2.8 * sd) is much better in 2023 iis PT.

| Component | unit | #23516 | | | #21526 | | |
|---|---|---|---|---|---|---|---|
| | | n | average | 2.8 * sd | n | average | 2.8 * sd |
| Total NPEO | mg/kg | 84 | 129.0 | 45.4 | 96 | 130.3 | 55.0 |

Table 7: comparison of sample #23516 with sample #21526

## 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.

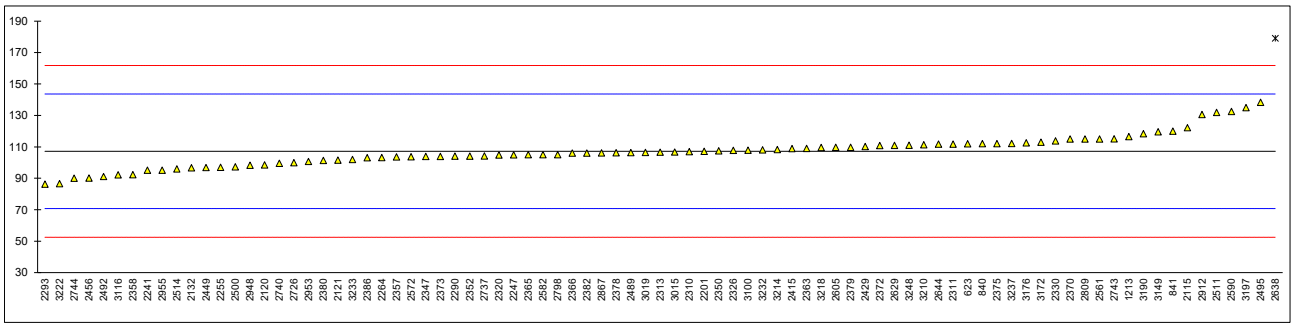
**APPENDIX 1**

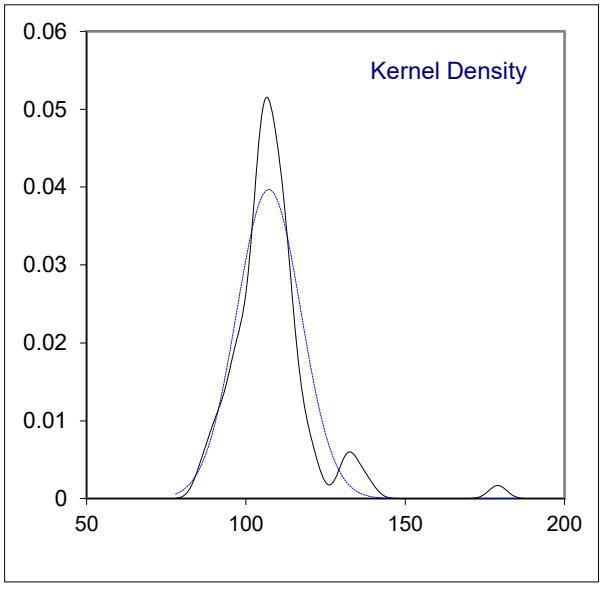
Determination of Total Octylphenol Ethoxylates (OPEO) on sample #23515; results in mg/kg

| lab | method | value | mark | z(targ) | Total NP+OP+OPEO+NPEO | | remarks |
|---|---|---|---|---|---|---|---|
| 551 | | ----- | | ----- | ----- | | |
| 623 | ISO18254-1 | 111.88 | | 0.26 | 111.88 | | |
| 840 | ISO18254-1 | 112 | | 0.26 | 112 | | |
| 841 | ISO18254-1 | 119.92 | | 0.70 | 119.92 | | |
| 1213 | | 116.50 | | 0.51 | not detected | | |
| 2115 | ISO18254-1 | 122.2 | | 0.82 | 122.2 | | |
| 2120 | ISO18254-1 | 98.5 | | -0.48 | 98.5 | | |
| 2121 | In house | 101.6 | C | -0.31 | 101.6 | C | first reported: 49.87 / 49.87 |
| 2132 | ISO/DIS 18254-1 | 96.6 | | -0.58 | NA | | |
| 2201 | ISO18254-1 | 107.2 | | 0.00 | 107.2 | | |
| 2241 | ISO18254-1 | 95.162 | | -0.66 | 95.162 | | |
| 2247 | ISO18254-1 | 104.98 | | -0.12 | 104.98 | | |
| 2255 | ISO/DIS 18254-1 | 97.0 | | -0.56 | 97.0 | | |
| 2264 | ISO18254-1 | 103.2 | | -0.22 | 103.2 | | |
| 2265 | | ----- | | ----- | ----- | | |
| 2290 | ISO18254-1 | 104.1 | | -0.17 | 104.1 | | |
| 2293 | ISO18254-1 | 86.29 | | -1.15 | 86.29 | | |
| 2310 | ISO18254-1 | 107 | | -0.01 | 107 | | |
| 2311 | ISO18254-1 | 111.7 | | 0.25 | 111.7 | | |
| 2313 | ISO18254-1 | 106.58 | | -0.03 | 106.58 | | |
| 2320 | ISO18254-1 | 104.8 | | -0.13 | 104.8 | | |
| 2326 | ISO/DIS 18254-1 | 107.76 | | 0.03 | 107.76 | | |
| 2330 | ISO18254-1 | 113.78 | | 0.36 | 113.78 | | |
| 2347 | ISO/DIS 18254-1 | 103.9 | | -0.18 | 103.9 | | |
| 2350 | ISO18254-1 | 107.50 | | 0.02 | 107.50 | | |
| 2352 | ISO18254-1 | 104.1 | | -0.17 | 104.1 | | |
| 2357 | ISO/DIS 18254-1 | 103.6 | | -0.20 | 103.6 | | |
| 2358 | ISO18254-1 | 92.29 | | -0.82 | 92.29 | | |
| 2363 | ISO18254-1 | 109 | | 0.10 | 109 | | |
| 2365 | ISO/DIS 18254-1 | 104.99 | | -0.12 | 104.99 | | |
| 2366 | ISO18254-1 | 106 | | -0.06 | 106 | | |
| 2370 | ISO18254-1 | 115 | | 0.43 | 115 | | |
| 2372 | ISO18254-1 | 110.804 | | 0.20 | 110.804 | | |
| 2373 | ISO18254-1 | 103.91 | | -0.18 | 103.91 | | |
| 2375 | ISO18254-1 | 112 | | 0.26 | 112 | | |
| 2378 | ISO18254-1 | 106.2 | | -0.05 | 106.2 | | |
| 2379 | ISO18254-1 | 109.727 | | 0.14 | 109.727 | | |
| 2380 | ISO18254-1 | 101.32 | | -0.32 | 101.32 | | |
| 2382 | ISO18254-1 | 106.0 | | -0.06 | 106.0 | | |
| 2386 | ISO/DIS 18254-1 | 103.14 | | -0.22 | ----- | | |
| 2415 | ISO18254-1 | 108.91 | | 0.09 | 108.91 | | |
| 2429 | ISO18254-1 | 110.21 | | 0.17 | ----- | | |
| 2449 | ISO18254-1 | 96.84 | | -0.57 | ----- | | |
| 2456 | ISO18254-1 | 90.1 | | -0.94 | 91.1 | | |
| 2489 | ISO/DIS 18254-1 | 106.3 | | -0.05 | 106.3 | | |
| 2492 | ISO18254-1 | 91.1 | | -0.88 | 91.1 | | |
| 2495 | ISO18254-1 | 138.3 | | 1.71 | 138.3 | | |
| 2500 | ISO18254-1 | 97.26 | | -0.54 | 97.26 | | |
| 2511 | ISO18254-1 | 131.9 | | 1.36 | 131.9 | | |
| 2514 | ISO/DIS 18254-1 | 95.97 | | -0.62 | 95.97 | | |
| 2561 | ISO/DIS 18254-1 | 115.01 | | 0.43 | ----- | | |
| 2572 | ISO/DIS 18254-1 | 103.7 | | -0.19 | 103.7 | | |
| 2582 | ISO18254-1 | 104.99 | | -0.12 | 104.99 | | |
| 2590 | ISO18254-1 | 132.464 | | 1.39 | 132.464 | | |
| 2605 | | 109.68 | | 0.14 | 109.68 | | |
| 2629 | ISO18254-1 | 110.97 | | 0.21 | 110.97 | | |
| 2638 | In house | 179.04 | R(0.01) | 3.94 | 179.04 | | |
| 2644 | ISO/DIS 18254-1 | 111.67 | | 0.25 | 111.67 | | |
| 2665 | | ----- | | ----- | ----- | | |
| 2678 | | ----- | | ----- | ----- | | |
| 2726 | In house | 99.9 | | -0.40 | ----- | | |
| 2737 | GB/T23322 | 104.1188 | | -0.17 | ----- | | |
| 2740 | ISO18254-1/ISO21084 | 99.5 | | -0.42 | 100.7 | | |
| 2743 | ISO18254-1 | 115.09 | | 0.43 | 117.60 | | |
| 2744 | ISO18254-1 | 90 | | -0.94 | 90 | | |
| 2798 | ISO18254-1 | 105 | | -0.12 | ----- | | |
| 2809 | ISO18254-1 | 115 | | 0.43 | ----- | | |
| 2867 | ISO18254-1 | 106.1 | | -0.06 | 106.1 | | |
| 2912 | ISO18254-1 | 130.54 | | 1.28 | 130.54 | | |
| 2948 | ISO18254-1 | 98.25 | | -0.49 | 98.25 | | |
| 2953 | ISO18254-1 | 100.64 | | -0.36 | 102.07 | | |
| 2955 | ISO18254-1 | 95.2 | | -0.66 | 95.2 | | |
| 2977 | | ----- | | ----- | ----- | | |
| 3015 | ISO18254-1 | 106.7 | | -0.03 | 106.7 | | |

| lab | method | value | mark | z(targ) | Total NP+OP+OPEO+NPEO | remarks |
|---|---|---|---|---|---|---|
| 3019 | ISO18254-1 | 106.42 | | -0.04 | 106.42 | |
| 3100 | ISO/DIS 18254-1 | 107.80 | | 0.03 | 107.80 | |
| 3116 | ISO18254-1 | 92.2 | | -0.82 | ----- | |
| 3149 | | 119.5 | | 0.68 | 119.5 | |
| 3172 | ISO18254-1 | 112.93 | | 0.32 | ----- | |
| 3176 | ISO18254-1 | 112.6 | | 0.30 | ----- | |
| 3190 | ISO/DIS 18254-1 | 118.36 | | 0.61 | 118.36 | |
| 3197 | ISO/DIS 18254-1 | 134.9 | | 1.52 | 134.9 | |
| 3210 | | 111.36 | | 0.23 | ----- | |
| 3214 | ISO18254-1 | 108.20 | | 0.06 | 108.20 | |
| 3218 | ISO18254-1 | 109.56 | | 0.13 | 109.56 | |
| 3222 | ISO18254-1 | 86.54 | | -1.13 | 86.54 | |
| 3232 | ISO18254-1 | 108 | | 0.05 | ----- | |
| 3233 | In house | 101.98 | | -0.29 | 101.98 | |
| 3237 | ISO/DIS 18254-1 | 112.06 | | 0.27 | ----- | |
| 3248 | ISO/DIS 18254-1 | 111 | | 0.21 | 111 | |

| | | | |
|---|---|---|---|
| | normality | suspect | |
| | n | 84 | |
| | outliers | 1 | |
| | mean (n) | 107.179 | |
| | st.dev. (n) | 10.0571 | RSD = 9.4% |
| | R(calc.) | 28.160 | |
| | st.dev.(iis memo 2203 ) | 18.2205 | |
| | R(iis memo 2203 ) | 51.017 | |
| compare | | | |
| | R(ISO18254-1:16) | 29.435 | |



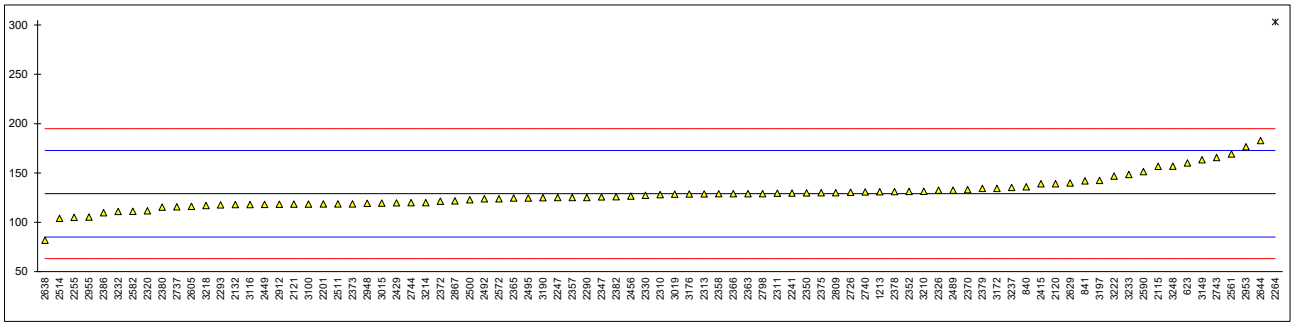



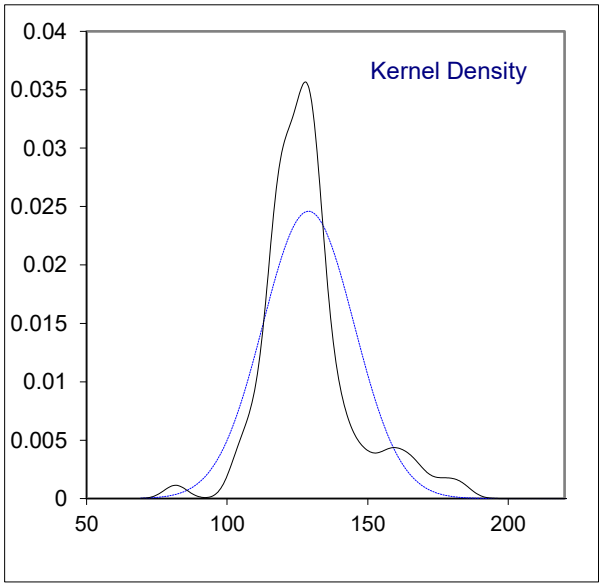
Determination of Total Nonylphenol Ethoxylates (NPEO) on sample #23516; results in mg/kg

| lab | method | value | mark | z(targ) | Total NP+OP+OPEO+NPEO | | remarks |
|---|---|---|---|---|---|---|---|
| 551 | | ----- | | ----- | ----- | | |
| 623 | ISO18254-1 | 160.19 | | 1.42 | 160.19 | | |
| 840 | | 136 | | 0.32 | 136 | | |
| 841 | ISO18254-1 | 141.99 | | 0.59 | 141.99 | | |
| 1213 | ISO18245-1/ISO21084 | 130.97 | | 0.09 | ND | | |
| 2115 | ISO18254-1 | 156.8 | | 1.27 | 156.8 | | |
| 2120 | ISO18254-1 | 139 | | 0.46 | 139 | | |
| 2121 | In house | 118.38 | | -0.48 | 118.38 | | |
| 2132 | ISO/DIS 18254-1 | 118 | | -0.50 | NA | | |
| 2201 | ISO18254-1 | 118.6 | | -0.47 | 118.6 | | |
| 2241 | ISO18254-1 | 129.705 | | 0.03 | 129.705 | | |
| 2247 | ISO18254-1 | 124.99 | | -0.18 | 124.99 | | |
| 2255 | ISO/DIS 18254-1 | 105.0 | | -1.09 | 105.0 | | |
| 2264 | ISO/DIS 18254-1 | 303.0 | C,R(0.01) | 7.93 | 303.0 | C | first reported: 357.0 / 357.0 |
| 2265 | | ----- | | ----- | ----- | | |
| 2290 | ISO18254-1 | 125.1 | | -0.18 | 125.1 | | |
| 2293 | ISO18254-1 | 117.62 | | -0.52 | 117.62 | | |
| 2310 | ISO18254-1 | 128 | | -0.05 | 128 | | |
| 2311 | ISO18254-1 | 129.53 | | 0.02 | 129.53 | | |
| 2313 | ISO18254-1 | 128.67 | | -0.02 | 128.67 | | |
| 2320 | | 111.6 | | -0.79 | 111.6 | | |
| 2326 | ISO/DIS 18254-1 | 132.55 | | 0.16 | 132.55 | | |
| 2330 | ISO18254-1 | 127.30 | | -0.08 | 127.30 | | |
| 2347 | ISO/DIS 18254-1 | 125.7 | | -0.15 | 125.7 | | |
| 2350 | ISO18254-1 | 129.75 | | 0.03 | 129.75 | | |
| 2352 | ISO18254-1 | 131.2 | | 0.10 | 131.2 | | |
| 2357 | ISO/DIS 18254-1 | 125.1 | | -0.18 | 125.1 | | |
| 2358 | ISO18254-1 | 128.87 | | -0.01 | 128.87 | | |
| 2363 | ISO18254-1 | 129 | | 0.00 | 129 | | |
| 2365 | ISO/DIS 18254-1 | 124.6 | | -0.20 | 124.6 | | |
| 2366 | ISO18254-1 | 129 | | 0.00 | 129 | | |
| 2370 | ISO18254-1 | 133 | | 0.18 | 133 | | |
| 2372 | ISO18254-2 | 121.283 | | -0.35 | 121.283 | | |
| 2373 | ISO18254-1 | 118.62 | | -0.47 | 118.62 | | |
| 2375 | ISO18254-1 | 130 | | 0.05 | 130 | | |
| 2378 | ISO/DIS 18254-1 | 131.1 | | 0.10 | 131.1 | | |
| 2379 | ISO18254-1 | 134.244 | | 0.24 | 134.244 | | |
| 2380 | ISO18254-1 | 115.31 | | -0.62 | 115.31 | | |
| 2382 | ISO18254-1 | 126.0 | | -0.14 | 126.0 | | |
| 2386 | ISO/DIS 18254-1 | 109.67 | | -0.88 | ----- | | |
| 2415 | ISO18254-1 | 139.0 | | 0.46 | 139 | | |
| 2429 | ISO18254-1 | 119.64 | | -0.43 | ----- | | |
| 2449 | ISO18254-1 | 118.144 | | -0.50 | ----- | | |
| 2456 | ISO18254-1 | 126.6 | | -0.11 | 128.7 | | |
| 2489 | ISO/DIS 18254-1 | 132.6 | | 0.16 | 132.6 | | |
| 2492 | ISO18254-1 | 123.8 | | -0.24 | 123.8 | | |
| 2495 | ISO18254-1 | 124.64 | | -0.20 | 126.4 | | |
| 2500 | ISO18254-1 | 122.9 | | -0.28 | 122.9 | | |
| 2511 | ISO18254-1 | 118.6 | | -0.47 | 118.6 | | |
| 2514 | ISO/DIS 18254-1 | 103.99 | | -1.14 | 103.99 | | |
| 2561 | ISO/DIS 18254-1 | 169.35 | | 1.84 | ----- | | |
| 2572 | ISO/DIS 18254-1 | 123.8 | | -0.24 | 123.8 | | |
| 2582 | ISO18254-1 | 111.24 | | -0.81 | 111.24 | | |
| 2590 | ISO18254-1 | 151.4 | C | 1.02 | 151.4 | C | first reported: 192.192 / 192.192 |
| 2605 | GB/T23972 | 116.15 | | -0.59 | 116.15 | | |
| 2629 | ISO18254-1 | 139.79 | | 0.49 | 139.79 | | |
| 2638 | In house | 81.72 | | -2.16 | 81.72 | | |
| 2644 | ISO/DIS 18254-1 | 182.87 | | 2.46 | 182.87 | | |
| 2665 | | ----- | | ----- | ----- | | |
| 2678 | | ----- | | ----- | ----- | | |
| 2726 | In house | 130.5 | | 0.07 | ----- | | |
| 2737 | GB/T23322 | 115.7077 | | -0.61 | ----- | | |
| 2740 | ISO18245-1/ISO21084 | 130.7 | | 0.08 | 131.5 | | |
| 2743 | ISO/DIS 18254-1 | 165.51 | | 1.66 | 166.90 | | |
| 2744 | ISO18254-1 | 120 | C | -0.41 | 120 | C | first reported: 86 / 86 |
| 2798 | ISO18254-1 | 129 | | 0.00 | ----- | | |
| 2809 | ISO18254-1 | 130 | | 0.05 | ----- | | |
| 2867 | ISO18254-1 | 121.7 | | -0.33 | 121.7 | | |
| 2912 | ISO18254-1 | 118.26 | | -0.49 | 120.31 | | |
| 2948 | ISO18254-1 | 119.25 | | -0.44 | 119.25 | | |
| 2953 | ISO18254-1 | 176.51 | | 2.17 | 179.41 | | |
| 2955 | ISO18254-1 | 105.3 | | -1.08 | 105.3 | | |
| 2977 | | ----- | | ----- | ----- | | |
| 3015 | ISO18254-1 | 119.3 | | -0.44 | 119.3 | | |

| lab | method | value | mark | z(targ) | Total NP+OP+OPEO+NPEO | remarks |
|---|---|---|---|---|---|---|
| 3019 | ISO18254-1 | 128.44 | | -0.03 | 128.44 | |
| 3100 | GB/T23322 | 118.42 | | -0.48 | 118.42 | |
| 3116 | ISO18254-1 | 118 | | -0.50 | ----- | |
| 3149 | | 163.5 | | 1.57 | 163.5 | |
| 3172 | ISO18254-1 | 134.35 | | 0.24 | ----- | |
| 3176 | ISO/DIS 18254-1 | 128.6 | | -0.02 | ----- | |
| 3190 | ISO/DIS 18254-1 | 124.82 | | -0.19 | 124.82 | |
| 3197 | ISO18254-1 | 142.5 | | 0.62 | 142.5 | |
| 3210 | | 131.405 | | 0.11 | ----- | |
| 3214 | ISO18254-1 | 120.03 | | -0.41 | 120.03 | |
| 3218 | ISO18254-1 | 117.09 | | -0.54 | 117.09 | |
| 3222 | ISO18254-1 | 146.78 | | 0.81 | 147.78 | |
| 3232 | ISO18254-1 | 111 | | -0.82 | ----- | |
| 3233 | In house | 148.44 | | 0.89 | 148.44 | |
| 3237 | ISO/DIS 18254-1 | 135.43 | | 0.29 | ----- | |
| 3248 | ISO/DIS 18254-1 | 157 | | 1.28 | 157 | |

| | | | |
|---|---|---|---|
| | normality | not OK | |
| | n | 84 | |
| | outliers | 1 | |
| | mean (n) | 129.003 | |
| | st.dev. (n) | 16.2186 | (RSD = 13%) |
| | R(calc.) | 45.412 | |
| | st.dev.(iis memo 2203 ) | 21.9304 | |
| | R(iis memo 2203 ) | 61.405 | |
| compare | | | |
| | R(ISO18254-1:16) | 35.428 | |



## APPENDIX 2

Summary of other reported components in sample #23515 and #23516; results in mg/kg

**Abbreviations of components**

OP = Total Octylphenol
NP = Total Nonylphenol
OPEO = Total Octylphenol Ethyloxylates
NPEO = Total Nonylphenol Ethyloxylates

| | #23515 | | | #23516 | | |
|---|---|---|---|---|---|---|
| lab | OP | NP | NPEO | OP | NP | OPEO |
| 551 | ----- | ----- | ----- | ----- | ----- | ----- |
| 623 | Not Detection | Not Detection | Not Detection | Not detected | Not detected | Not detected |
| 840 | not detected | not detected | not detected | not detected | not detected | not detected |
| 841 | not detected | not detected | not detected | not detected | not detected | not detected |
| 1213 | ND | ND | ND | ND | ND | ND |
| 2115 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2120 | < 5 | < 5 | < 5 | < 5 | < 5 | < 5 |
| 2121 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2132 | <10 | <10 | <10 | <10 | <10 | <10 |
| 2201 | <10 | <10 | <10 | <10 | <10 | <10 |
| 2241 | < 10 | < 10 | < 10 | < 10 | < 10 | < 10 |
| 2247 | Not detected | Not detected | Not detected | Not detected | Not detected | Not detected |
| 2255 | Not Detected | Not Detected | Not Detected | Not Detected | Not Detected | Not Detected |
| 2264 | not detected | not detected | not detected | not detected | not detected | not detected |
| 2265 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2290 | <10 | <10 | <10 | <10 | <10 | <10 |
| 2293 | 0 | 0 | 0 | 0 | 0 | 0 |
| 2310 | not detected | not detected | not detected | not detected | not detected | not detected |
| 2311 | Not Detected | Not Detected | Not Detected | Not Detected | Not Detected | Not Detected |
| 2313 | NOT DETECTED | NOT DETECTED | NOT DETECTED | NOT DETECTED | NOT DETECTED | NOT DETECTED |
| 2320 | Not Detected | Not Detected | Not Detected | Not Detected | Not Detected | Not Detected |
| 2326 | ND | ND | ND | ND | ND | ND |
| 2330 | Not Detected | Not Detected | Not Detected | Not Detected | Not Detected | Not Detected |
| 2347 | <10 | <10 | <30 | <10 | <10 | <30 |
| 2350 | < 3.00 | < 3.00 | < 1.00 | < 3.00 | < 3.00 | < 1.00 |
| 2352 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2357 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2358 | not detected | not detected | not detected | not detected | not detected | not detected |
| 2363 | <10 | <10 | <30 | <10 | <10 | <30 |
| 2365 | ＜10 | ＜10 | ＜20 | ＜10 | ＜10 | ＜20 |
| 2366 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2370 | <1 | <1 | <1 | <1 | <1 | <1 |
| 2372 | not detected | not detected | not detected | not detected | not detected | not detected |
| 2373 | not detected | not detected | not detected | not detected | not detected | not detected |
| 2375 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2378 | ＜10 | ＜10 | ＜30 | ＜10 | ＜10 | ＜30 |
| 2379 | Not detected | Not detected | Not detected | Not detected | Not detected | Not detected |
| 2380 | <3 | <3 | <20 | <3 | <3 | <20 |
| 2382 | ＜10.0 | ＜10.0 | ＜20.0 | ＜10.0 | ＜10.0 | ＜20.0 |
| 2386 | < 3 | < 3 | < 10 | < 3 | < 3 | < 10 |
| 2415 | not detected | not detected | not detected | not detected | not detected | not detected |
| 2429 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2449 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2456 | not detectable | not detectable | 1.0 | not detectable | not detectable | 2.1 |
| 2489 | Not Detected | Not Detected | Not Detected | Not Detected | Not Detected | Not Detected |
| 2492 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2495 | <0.5 | <0.5 | <0.5 | <0.5 | <0.5 | 1.76 |
| 2500 | not detected | not detected | not detected | not detected | not detected | not detected |
| 2511 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2514 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2561 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2572 | <10 | <10 | <10 | <10 | <10 | <10 |
| 2582 | Not Detected | Not Detected | Not Detected | Not Detected | Not Detected | Not Detected |
| 2590 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2605 | <10.00 | <10.00 | <10.00 | <10.00 | <10.00 | <10.00 |
| 2629 | < 10 mg/kg | < 10 mg/kg | < 30 mg/kg | < 10 mg/kg | < 10 mg/kg | < 30 mg/kg |
| 2638 | not detected | not detected | not detected | not detected | not detected | not detected |
| 2644 | not detected | not detected | not detected | not detected | not detected | not detected |
| 2665 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2678 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2726 | not detected | not detected | not detected | not detected | not detected | not detected |
| 2737 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2740 | 0.68 | 0.47 | < 1,3 | < 0,22 | 0.77 | < 1,5 |

| | #23515 | | | #23516 | | |
|---|---|---|---|---|---|---|
| lab | OP | NP | NPEO | OP | NP | OPEO |
| 2743 | 2.51 | not detected | not detected | not detected | 1.39 | not detected |
| 2744 | not detected | not detected | not detected | not detected | not detected | not detected |
| 2798 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2809 | ----- | ----- | ----- | ----- | ----- | ----- |
| 2867 | not detected | not detected | not detected | not detected | not detected | not detected |
| 2912 | ----- | ----- | ----- | ----- | ----- | 2.05 |
| 2948 | Not Detected | Not Detected | Not Detected | Not Detected | Not Detected | Not Detected |
| 2953 | ----- | ----- | 1.43 | ----- | ----- | 2.9 |
| 2955 | not detected | not detected | not detected | not detected | not detected | not detected |
| 2977 | ----- | ----- | ----- | ----- | ----- | ----- |
| 3015 | <10 | <10 | <10 | <10 | <10 | <10 |
| 3019 | nd | nd | nd | nd | nd | nd |
| 3100 | not detected | not detected | not detected | not detected | not detected | not detected |
| 3116 | <5 | <5 | <30 | <5 | <5 | <30 |
| 3149 | ----- | ----- | ----- | ----- | ----- | ----- |
| 3172 | < 1 | < 1 | < 1 | < 1 | < 1 | < 1 |
| 3176 | ----- | ----- | ----- | ----- | ----- | ----- |
| 3190 | <10 | <10 | <10 | <10 | <10 | <10 |
| 3197 | <10 | <10 | <10 | <10 | <10 | <10 |
| 3210 | <20 | <20 | <20 | <20 | <20 | <20 |
| 3214 | N.D. | N.D. | N.D. | N.D. | N.D. | N.D. |
| 3218 | not detected | not detected | not detected | not detected | not detected | not detected |
| 3222 | not detected | not detected | not detected | not detected | not detected | 1.0 |
| 3232 | ----- | ----- | not detected | ----- | ----- | not detected |
| 3233 | < 8 | < 4 | < 4 | < 8 | < 4 | < 4 |
| 3237 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |
| 3248 | not detected | not detected | not detected | not detected | not detected | not detected |

## 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) |
|---|---|---|---|---|---|---|---|
| 551 | --- | --- | | --- | | | |
| 623 | Yes | Further cut | 1 | Ultrasonic | methanol | 60 | 60 |
| 840 | Yes | Further cut | 0.5g | Ultrasonic | Methanol | 60 minutes | 70°C |
| 841 | Yes | Further cut | 0.9 g | Ultrasonic | methanol | 60 minutes | 70 |
| 1213 | Yes | Further cut | 0.5g | Ultrasonic | MeOH | 1hour | 70 |
| 2115 | Yes | Used as received | 0.5 g | Ultrasonic | MeOH | 60 min | 60°C |
| 2120 | Yes | Used as received | | Ultrasonic | MeOH | 60 min | 70ºC |
| 2121 | No | Used as received | 1g | Ultrasonic | Methanol | 60 minutes | 60°c +/- 5°C |
| 2132 | No | Further cut | 1g | Ultrasonic | Methanol | 60 minutes | 70°C |
| 2201 | Yes | Further cut | 1.0 g | Ultrasonic | Methanol | 60min | 70 degrees |
| 2241 | Yes | Further cut | 0.5g | Ultrasonic | Methanol | 60mins | 70° |
| 2247 | Yes | Further cut | 1.5gm | Ultrasonic | Methanol | 60.0 | 70.0 |
| 2255 | Yes | Further cut | 0.5 | Ultrasonic | Methanol | 60 | 70 |
| 2264 | No | Further cut | 1 gram | Ultrasonic | methanol | 60 minutes | 70°C |
| 2265 | --- | --- | | --- | | | |
| 2290 | Yes | --- | | --- | | | |
| 2293 | Yes | Further cut | 1 gram | Ultrasonic | Methanol | 60 minutes | 70°C |
| 2310 | Yes | Further cut | 1 | Ultrasonic | Methanol | 60 | 70 |
| 2311 | Yes | Further cut | 1 | Ultrasonic | Methanol | 60 | 70 |
| 2313 | Yes | Further cut | 1.0g | Ultrasonic | Methanol | 60 min | 70 |
| 2320 | Yes | Further cut | 0.5g | Ultrasonic | Methanol | 60 min | 70˚C |
| 2326 | Yes | Further cut | 1 gm | Ultrasonic | Methanol | 60 min | 70 C |
| 2330 | Yes | Further cut | 0.5 g | Ultrasonic | Methanol | 60 min | 70 C |
| 2347 | No | Further cut | 1g | Ultrasonic | Methanol | 60min | 70℃ |
| 2350 | Yes | Further cut | 1 g | Ultrasonic | Methanol | 60 minutes | 70 °C |
| 2352 | Yes | Further cut | 0.5g | Ultrasonic | Methanol | 60min | 70°C |
| 2357 | --- | --- | | --- | | | |
| 2358 | Yes | Further cut | 1.0g | Ultrasonic | Methanol | 60 | 70 |
| 2363 | Yes | Further cut | 1g | Ultrasonic | MEOH | 1h | 70℃ |
| 2365 | Yes | Further cut | 0.5g | Ultrasonic | methanol | 60min | 70℃ |
| 2366 | Yes | Further cut | 0.5g | Ultrasonic | meoh | 60min | 70°C |
| 2370 | Yes | Further cut | 0.5 g | Ultrasonic | 10 mL | 60 min | 70°C |
| 2372 | No | Further cut | 1 g | Ultrasonic | MeOH | 60 | 70 |
| 2373 | Yes | Further cut | 0.5g | Ultrasonic | Methanol | 60min | 70℃ |
| 2375 | Yes | Further cut | 0,5 gram | Ultrasonic | Methanol | 60 | 70 |
| 2378 | Yes | Further cut | 2g | Ultrasonic | Methanol | 60min | 70°C |
| 2379 | Yes | Further cut | 0.5 | Ultrasonic | MeOH | 60 minutes | 70 C |
| 2380 | Yes | Further cut | 1.0 g | Ultrasonic | Methanol | 60 Minute | 70 °C |
| 2382 | Yes | Further cut | 1.0g | Ultrasonic | Methanol | 1h | 70℃ |
| 2386 | Yes | Further cut | 0.5 g | Ultrasonic | Methanol | 60 min | 70°C |
| 2415 | Yes | Used as received | 0.5 gram | Ultrasonic | Methanol (MeOH) | 60 min | 70 |
| 2429 | Yes | Further cut | 0.5 | --- | MeOH | 60 | 70 |
| 2449 | Yes | Further cut | 0.5 gram | Ultrasonic | Methanol | 01 hour | 70 C |
| 2456 | Yes | Used as received | | Ultrasonic | Methanol | 1 h | 70°C |
| 2489 | Yes | Further cut | 1.0011g/ 1.0038g | Ultrasonic | methanol/water | 60 minutes | 70 degrees |
| 2492 | Yes | Used as received | 0.5g | Ultrasonic | Methanol | 60 mins | 70°C |
| 2495 | Yes | Used as received | 0.5g | Ultrasonic | Methanol | 60 minutes | 70 °C |
| 2500 | Yes | Used as received | 1.0002g | Ultrasonic | Methanol | 60min | 70°C |
| 2511 | No | Further cut | | Ultrasonic | Methanol | 60 min | 70 °C |
| 2514 | Yes | Further cut | 1 gram | Ultrasonic | Methanol: Water=80:20 | 60 | 70 |
| 2561 | Yes | Used as received | 1g | Ultrasonic | Methanol | 60 | 70 |
| 2572 | Yes | --- | | --- | | | |
| 2582 | Yes | Further cut | 1.0019g; 1.0023g | Ultrasonic | Methanol | 60min | 70°C |
| 2590 | Yes | Used as received | 1g | Ultrasonic | MeOH | 60 min | 70°C |
| 2605 | Yes | Used as received | | Ultrasonic | Methanol | 30 | 70 |
| 2629 | Yes | Further cut | 1.0 gram | Ultrasonic | Methanol | 60 | 70 |
| 2638 | No | Further cut | 1 gram | Ultrasonic | Methanol | 60 minutes | room temperature |
| 2644 | Yes | Used as received | 0.5 | Ultrasonic | methanol | 60 | 70 |
| 2665 | --- | --- | | --- | | | |
| 2678 | --- | --- | | --- | | | |
| 2726 | Yes | Further cut | 1g and 0.5 g | Ultrasonic | Methanol | 60 min | 70 C |
| 2737 | Yes | Further cut | 1g | Ultrasonic | methanol | 60min | 70°C |
| 2740 | Yes | Further cut | 0,8 g & 0,7 g | Ultrasonic | methanol | 60 min | 60 °C |
| 2743 | Yes | Used as received | 1g | Ultrasonic | Methanol | 60min | 70°C |
| 2744 | Yes | Further cut | 0,5 g | Ultrasonic | Methanol | 1 hour | 70 |
| 2798 | Yes | Used as received | 0.5g | Ultrasonic | MeOH | 60mins | 70°C |

| lab | ISO/IEC17025 accredited | sample preparation | sample intake (g) | release/ extraction technique | release/ extraction solvent | extraction time (min) | extraction temperature (°C) |
|---|---|---|---|---|---|---|---|
| 2809 | Yes | Further cut | 0.5 | Ultrasonic | MeOH | 60 | 70 |
| 2867 | Yes | Further cut | 0.5g | Ultrasonic | methanol | 1h | 70℃ |
| 2912 | Yes | Used as received | 0.5 g | Ultrasonic | methanol | 60 | 70 |
| 2948 | --- | --- | 1.0 | --- | Methanol | 60 | 70 |
| 2953 | Yes | Further cut | 1 | Ultrasonic | Methanol | 60 | 70 |
| 2955 | Yes | Further cut | 0.5 | Ultrasonic | Methanol : Water | 60 | 70 |
| 2977 | --- | --- | | --- | | | |
| 3015 | Yes | Used as received | 1.0 | Ultrasonic | methanol | 60 | 70 |
| 3019 | No | Further cut | 1 | Ultrasonic | Methanol | 60 | 70 |
| 3100 | Yes | Used as received | 1.0g | Ultrasonic | Methanol | 60min | 70℃ |
| 3116 | Yes | Used as received | 0.5g | Ultrasonic | MeOH | 60 | 70 |
| 3149 | --- | --- | | --- | | | |
| 3172 | Yes | --- | | --- | | | |
| 3176 | Yes | Further cut | 1 g | Ultrasonic | MeOH | 60 minute | 70 C |
| 3190 | Yes | Further cut | 1g | Ultrasonic | methanol | 60min | 70℃ |
| 3197 | Yes | Further cut | 0,5 | Ultrasonic | Methanol | 60 min. | 70 |
| 3210 | No | Used as received | 1 | Ultrasonic | Methanol | 60 | 70 |
| 3214 | Yes | Further cut | 1g | Ultrasonic | methanol | 60 mins | 70°C |
| 3218 | Yes | Further cut | 0.5g | Ultrasonic | methanol | 60min | 70 °C |
| 3222 | Yes | Further cut | 1 g | Ultrasonic | Methanol | 60 min | 60°C |
| 3232 | Yes | Used as received | 0.55g | Ultrasonic | Methanol | 30 minutes | 70 0C |
| 3233 | No | Used as received | 1g | Ultrasonic | methanol | 60 min | 70°C |
| 3237 | Yes | Further cut | 0,5 gr | Ultrasonic | Methanol | 60 minutes | 70 C |
| 3248 | Yes | Used as received | 1.0 | Ultrasonic | Methanol | 1 | 70 |

## APPENDIX 4

### Number of participants per country

4 labs in BANGLADESH
1 lab in BRAZIL
1 lab in CAMBODIA
3 labs in FRANCE
5 labs in GERMANY
1 lab in GUATEMALA
5 labs in HONG KONG
7 labs in INDIA
1 lab in INDONESIA
11 labs in ITALY
1 lab in KOREA, Republic of
21 labs in P.R. of CHINA
4 labs in PAKISTAN
1 lab in PERU
1 lab in PORTUGAL
2 labs in SRI LANKA
3 labs in TAIWAN
1 lab in THAILAND
2 labs in TUNISIA
6 labs in TURKEY
1 lab in UNITED KINGDOM
8 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 |
| f+? | = possibly a false positive test result? |
| f-? | = possibly a false negative test result? |

### Literature


1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2 ISO5725:86
3 ISO5725 parts 1-6:94
4 ISO13528:05
5 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
6 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
7 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
8 J.N. Miller, Analyst, 118, 455, (1993)
9 Analytical Methods Committee, Technical Brief, No 4, January 2001
10 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)
11 W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)
13 iis memo 2203: Reproducibility of AP and APEO in Textile in iis PTs