Institute for
Interlaboratory Studies

Results of Proficiency Test
AP and APEO in Textile
March 2022

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Alkylphenol Ethoxylates (APEO), like Octylphenol Ethoxylates (OPEO) and Nonylphenol Ethoxylates (NPEO) have widely been used in manufacturing antioxidants, lubricating oil additives, laundry and dish detergents, emulsifiers, wetting agents in cosmetics, including hair products, defoaming agents and solubilizers. APEO may degrade in the environment to the corresponding Octyl- and Nonylphenols (OP & NP). These alkylphenols (AP) have attracted attention due to its prevalence in the environment and its potential role as an endocrine disruptor and xenoestrogen, due to its ability to act with oestrogen-like activity. The European Union has implemented sales and use restrictions on certain applications in which alkylphenols are used because of their alleged "toxicity, persistence, and the liability to bioaccumulate".

Since 2016 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of AP and APEO in Textile every year. During the annual proficiency testing program 2021/2022 it was decided to continue the proficiency test (PT) for the analysis of AP and APEO in textile.

In this interlaboratory study 95 laboratories in 25 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 positive on AP and/or APEO of 3 grams each and labelled #22515 and #22516 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 yellow cotton was selected which was made positive on NP and NPEO by a third-party laboratory. This batch was cut into small pieces. After homogenization 120 small plastic bags were filled with approximately 3 grams each and labelled #22515.
The homogeneity of the subsamples was checked by determination of NP and NPEO using an in-house test method on eight stratified randomly selected subsamples.

	NP in mg/kg	NPEO in mg/kg
sample #22515-1	18.11	29.25
sample #22515-2	17.89	28.26
sample #22515-3	18.84	29.02
sample #22515-4	18.41	29.19
sample #22515-5	17.13	28.07
sample #22515-6	18.89	28.24
sample #22515-7	16.72	29.28
sample #22515-8	18.38	29.13

Table 1: homogeneity test results of subsamples #22515

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

	NP in mg/kg	NPEO in mg/kg
r (observed)	2.17	1.45
reference method	iis memo 2203	iis memo 2203
0.3 x R (reference method)	2.58	4.11

Table 2: evaluation of repeatabilities of subsamples #22515, see also paragraph 4.1 for more details

The calculated repeatabilities are in agreement with 0.3 times the corresponding reproducibility of the reference method. Therefore, homogeneity of the subsamples was assumed.

For the second sample a batch of black viscose/elastane was selected which was made positive on OPEO and NPEO by a third-party laboratory. This batch was cut into small pieces. After homogenization 120 small plastic bags were filled with approximately 3 grams each and labelled #22516.
The homogeneity of the subsamples was checked by determination of OPEO and NPEO using an in-house test method on eight stratified randomly selected subsamples.

	OPEO in mg/kg	NPEO in mg/kg
sample #22516-1	75.73	167.38
sample #22516-2	68.99	156.61
sample #22516-3	70.00	153.76
sample #22516-4	70.20	154.85
sample #22516-5	70.40	155.64
sample #22516-6	65.63	145.34
sample #22516-7	71.43	155.24
sample #22516-8	66.60	143.70

Table 3: homogeneity test results of subsamples #22516

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

	OPEO in mg/kg	NPEO in mg/kg
r (observed)	8.64	20.41
reference method	iis memo 2203	iis memo 2203
0.3 x R (reference method)	9.98	22.00

Table 4: evaluation of the repeatabilities of subsamples #22516, see also paragraph 4.1 for more details

The calculated repeatabilities are in agreement with 0.3 times the corresponding reproducibility of the reference method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one textile sample labelled #22515 and one textile sample labelled #22516 were sent on February 09, 2022.

2.5 ANALYZES

The participants were requested to determine on samples #22515 and #22516 the concentrations of Octylphenol (OP), Nonylphenol (NP), Octylphenol Ethoxylates (OPEO), Nonylphenol Ethoxylates (NPEO) and the Total of OP, NP, OPEO and NPEO. It was requested to report if the laboratory was accredited for the requested components that were determined and to report some analytical details. To ensure homogeneity it was requested to not use less than 0.5 grams per determination.

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. Eighteen participants reported test results after the final reporting date and three other participants did not report any test results. Not all participants were able to report all tests requested.
In total 92 participants reported 526 numerical test results. Observed were 28 outlying test results, which is 5.3%. 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. OekoTex®). As alternative for the evaluation of the quality of the AP and APEO test results iis had used an estimated target reproducibility calculated with the Horwitz equation based on 5 components.
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 #22515

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

NPEO: This determination was problematic for a number of participants. Eight statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full agreement with the target reproducibility as derived from iis memo 2203.

Total OP + NP + OPEO + NPEO: This determination was problematic for a number of participants. Seven statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full agreement with the target reproducibility as derived from iis memo 2203.

Most participants agreed that the levels for the components OP 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.

sample #22516

OPEO: This determination was problematic for a number of participants. Six statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the target reproducibility as derived from iis memo 2203.

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.

Total OP + NP + OPEO + NPEO: This determination was not problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the target reproducibility as derived from iis memo 2203.

Most participants agreed that the levels for the components OP and NP 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 previous iis PTs are presented in the next tables.

Component	unit	n	average	2.8 * sd	R(target)
NP	mg/kg	80	12.7	7.2	6.0
NPEO	mg/kg	83	22.9	11.8	10.9
Total OP + NP + OPEO + NPEO	mg/kg	69	34.5	15.3	16.4

Table 5: reproducibilities of components on sample #22515

Component	unit	n	average	2.8 * sd	R(target)
OPEO	mg/kg	85	63.6	23.1	30.3
NPEO	mg/kg	90	138.7	38.6	66.0
Total OP + NP + OPEO + NPEO	mg/kg	73	203.5	52.8	96.8

Table 6: reproducibilities of components on sample #22516

Without further statistical calculations it can be concluded that for almost all tests there is a good compliance of the group of participants with the reference method. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF MARCH 2022 WITH PREVIOUS PTS

	March 2022	March 2021	March 2020	February 2019	February 2018
Number of reporting laboratories	92	100	96	105	92
Number of test results	526	444	347	366	329
Number of statistical outliers	28	15	17	21	8
Percentage of statistical outliers	5.3%	3.4%	4.9%	5.7%	2.4%

Table 7: comparison with previous proficiency tests

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

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

Component	March 2022	March 2021	March 2020	February 2019	2018 - 2016	Target
Octylphenol (OP)	n.e.	12%	n.e.	n.e.	n.e.	17%
Nonylphenol (NP)	20%	n.e.	n.e.	n.e.	n.e.	17%
Octylphenol Ethoxylates (OPEO)	13%	12%	17%	10%	15-16%	17%
Nonylphenol Ethoxylates (NPEO)	10-18%	15%	27%	13%	18-28%	17%
Total OP + NP + OPEO + NPEO	9-16%	12%	16-28%	11%	15-26%	17%

Table 8: development of uncertainties over the years

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

4.4 EVALUATION OF THE ANALYTICAL DETAILS

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

- 89% mentioned that they are ISO/IEC17025 accredited to determine the reported components.
- 58% further cut the samples prior to analysis and 42% used the samples as received.
- 28% around 0.5 grams and 69% around 1 gram for the sample intake.
- 98% 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 88% used an extraction/release temperature of 70°C, 12% used a lower temperature.

It appeared that the effect of the analytical details on the determination of the components is small and not statistically significant.

5 DISCUSSION

Almost all reporting participants were able to detect Nonylphenol (NP) and Nonylphenol Ethoxylates (NPEO) in sample #22515 and Octylphenol Ethoxylates (OPEO) and Nonylphenol Ethoxylates (NPEO) in sample #22516.

When the test results of this interlaboratory study were compared to the OEKO-TEX®, Bluesign® requirements and the EU (REACH) regulations on Textiles (see table 9), it is noticed that not all participants would make identical decisions about the acceptability of the samples for the determined components.
Compared to the Bluesign® system substances list (BSSL) eleven reporting laboratories would have accepted sample #22515, while all other laboratories would have rejected the sample for containing too much NP. Compared to OEKO-TEX® all laboratories would have accepted the sample based on the component NP.

All laboratories would have accepted sample #22515 based on the component NPEO and the total of OP + NP + OPEO + NPEO.
For sample #22516 it is noticed that all the reporting laboratories would reject the sample for containing too much NPEO, except one laboratory. Four of the reporting laboratories would reject the sample for containing too much OPEO, while all other laboratories would have accepted the sample. All laboratories would have rejected sample #22516 based on the total of OP + NP + OPEO + NPEO.

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

	OEKO-TEX®	Bluesign® BSSL v11.0	EU 2016/26
NP	---	10 mg/kg	100 mg/kg
Total OP + NP	10 mg/kg	---	---
Every single APEO	---	100 mg/kg *)	---
NPEO	---	---	100 mg/kg
Total OP + NP + OPEO + NPEO	100 mg/kg	---	---

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

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

6 CONCLUSION

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

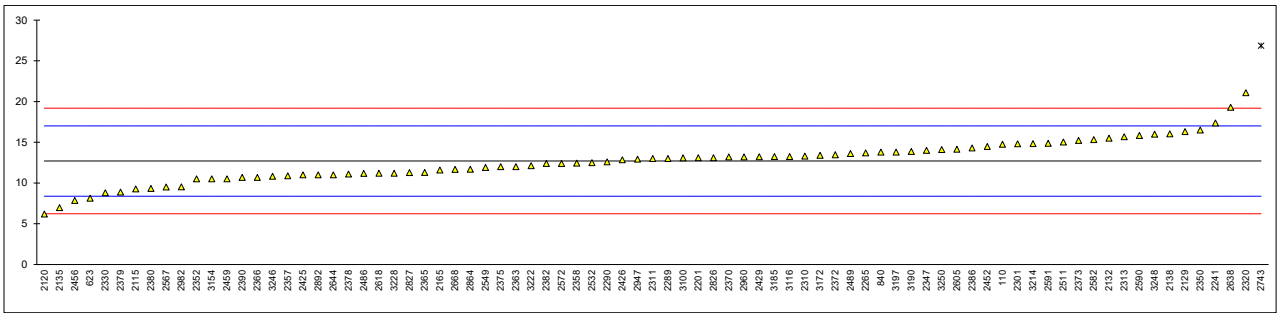
APPENDIX 1

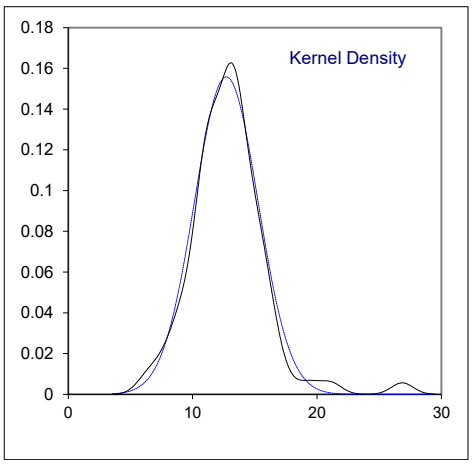
Determination of Nonylphenol (NP) on sample #22515; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO18254-1	14.74		0.94	
551		-----		-----	
623	ISO18254-1	8.13	C	-2.12	First reported 6.07
840		13.8		0.51	
2115	ISO18254-1	9.28		-1.58	
2120	ISO21084	6.2		-3.01	
2129		16.3		1.67	
2132	ISO18254-2	15.5		1.30	
2135	ISO18254	6.98		-2.65	
2138	ISO/DIS 18254-1	16.03		1.54	
2165	ISO18254-1	11.6		-0.51	
2201	GB/T23322	13.1		0.18	
2241	ISO18254-1	17.360		2.16	
2265	ISO18254-1	13.7		0.46	
2289	ISO18254-1	13		0.14	
2290	ISO/DIS 18254-1	12.6		-0.05	
2293		-----		-----	
2301	ISO18254-1	14.81		0.98	
2310	ISO18254-1	13.3		0.28	
2311	ISO18254-1	13		0.14	
2313	ISO18254-1	15.68		1.38	
2320	ISO21084	21.09		3.88	
2330	ISO18254-1	8.805		-1.80	
2347	ISO18254-1	14		0.60	
2350	ISO18254-1	16.5	C	1.76	First reported 28.13
2352	ISO18254-1	10.5		-1.02	
2357	ISO21084	10.89		-0.84	
2358	In house	12.43		-0.13	
2363	ISO18254-1	12		-0.33	
2365	ISO18254-1	11.3		-0.65	
2366	ISO18254-1	10.7		-0.93	
2370	ISO18254-1	13.2		0.23	
2372	ISO/DIS 18254-1	13.47		0.36	
2373	ISO18254-1	15.21		1.16	
2375	ISO18254-1	12		-0.33	
2378	GB/T23322	11.1		-0.74	
2379	ISO18254-1	8.880		-1.77	
2380	ISO18254-1	9.32		-1.57	
2382	ISO18254-1	12.4		-0.14	
2386	ISO18254-1	14.3		0.74	
2390	ISO18254-1	10.69		-0.93	
2425	In house	11.0		-0.79	
2426	ISO18254-1	12.85		0.07	
2429	ISO18254-1	13.23		0.24	
2449		-----		-----	
2452	ISO18254-1	14.47		0.82	
2456	ISO21084	7.85		-2.25	
2459	ISO21084	10.501	C	-1.02	First reported 10.73
2486	ISO18254-1	11.189		-0.70	
2489	ISO18254-1	13.6		0.42	
2511	ISO18254	15.021		1.07	
2532	ISO/DIS 18254-1	12.5		-0.09	
2549	ISO/DIS 18254-1	11.91		-0.37	
2561		-----		-----	
2567	ISO18254-1	9.5		-1.48	
2572	ISO/DIS 18254-1	12.4		-0.14	
2582	In house	15.32		1.21	
2590	ISO/DIS 18254-1	15.846		1.46	
2591	In house	14.87		1.00	
2605	GB/T23972	14.134		0.66	
2618	ISO18254-1	11.2		-0.70	
2638	In house	19.29		3.05	
2643		-----		-----	
2644	ISO21084 / ISO18254-1	11.01		-0.78	
2649		-----		-----	
2668	ISO18254-1	11.67		-0.48	
2678	ISO18254-1	<3		<-4.49	Possibly a false negative test result?
2719		-----		-----	
2743	ISO21084	26.85	C,R(0.01)	6.55	First reported 32.97
2798		-----		-----	
2826	ISO18254-1	13.1		0.18	
2827	ISO/DIS 18254-1	11.29		-0.65	
2864	In house	11.69		-0.47	
2892	ISO/DIS 18254-1	11.000		-0.79	

lab	method	value	mark	z(targ)	remarks
2947	ISO18254Mod.	12.93		0.11	
2960	ISO18254-1	13.2		0.23	
2982	ISO/DIS 18254-1	9.54		-1.46	
3100	ISO/DIS 18254-1	13.08		0.17	
3116	ISO/DIS 18254-1	13.25		0.25	
3118		-----		-----	
3154	In house	10.5	C	-1.02	First reported 3.9
3172	ISO18254-1	13.40		0.32	
3176		-----		-----	
3185	ISO18254-1	13.24		0.25	
3190	ISO18254-1	13.87		0.54	
3197	ISO18254-1	13.8	C	0.51	First reported <10
3210	In house	<20		-----	
3214	ISO/DIS 18254-1	14.83		0.99	
3222	ISO18254-1	12.12		-0.27	
3228	ISO18254-1	11.2		-0.70	
3230	In house	not analyzed		-----	
3237		-----		-----	
3246	ISO21084 / ISO18254-1	10.82		-0.87	
3248	In house	16		1.53	
3250		14.1		0.65	

	normality	suspect	
	n	80	
	outliers	1	
	mean (n)	12.703	
	st.dev. (n)	2.5601	RSD= 20%
	R(calc.)	7.168	
	st.dev.(iis memo 2203)	2.1595	
	R(iis memo 2203)	6.046	
Compare			
	R(ISO18254-1:16)	3.489	



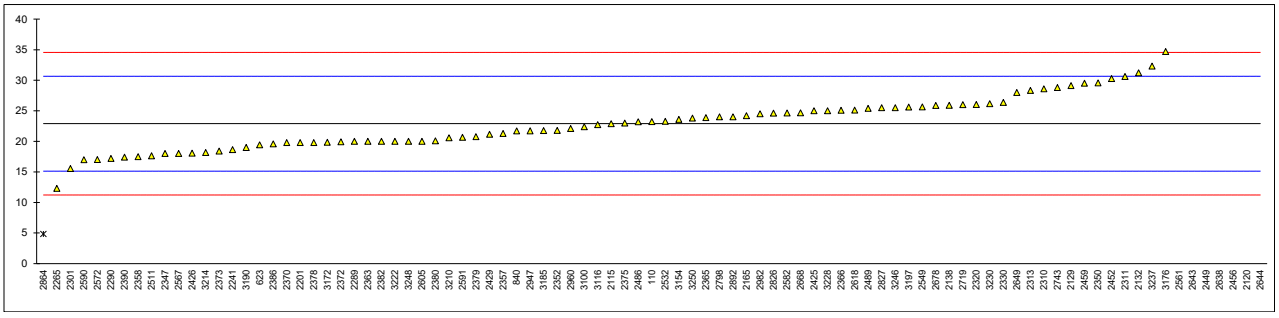


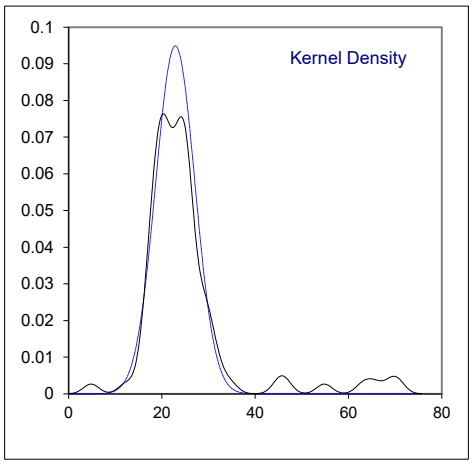
Determination of Nonylphenol Ethoxylates (NPEO) on sample #22515; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO18254-1	23.22		0.09	
551		-----		-----	
623	ISO18254-1	19.44	C	-0.89	First reported 11.205
840		21.7		-0.30	
2115	ISO18254-1	22.9		0.00	
2120	ISO18254-1	69	C,R(0.01)	11.85	First reported 50
2129		29.1		1.60	
2132	ISO18254-2	31.2		2.14	
2135		-----		-----	
2138	ISO/DIS 18254-1	25.90		0.77	
2165	ISO18254-1	24.2		0.34	
2201	GB/T23322	19.8		-0.79	
2241	ISO18254-1	18.644		-1.09	
2265	ISO18254-1	12.3		-2.72	
2289	ISO18254-1	20		-0.74	
2290	ISO/DIS 18254-1	17.2		-1.46	
2293		-----		-----	
2301	ISO18254-1	15.55		-1.89	
2310	ISO18254-1	28.6		1.47	
2311	ISO18254-1	30.61		1.99	
2313	ISO18254-1	28.33		1.40	
2320	ISO18254-1	26.04		0.81	
2330	ISO18254-1	26.37		0.90	
2347	ISO18254-1	18		-1.26	
2350	ISO18254-1	29.58		1.72	
2352	ISO18254-1	21.8		-0.28	
2357	ISO18254-1	21.28		-0.41	
2358	In house	17.50		-1.38	
2363	ISO18254-1	20		-0.74	
2365	ISO18254-1	23.9		0.26	
2366	ISO18254-1	25.1		0.57	
2370	ISO18254-1	19.8		-0.79	
2372	ISO/DIS 18254-1	19.92		-0.76	
2373	ISO18254-1	18.42		-1.15	
2375	ISO18254-1	23		0.03	
2378	GB/T23322	19.8		-0.79	
2379	ISO18254-1	20.767		-0.54	
2380	ISO18254-1	20.08		-0.72	
2382	ISO18254-1	20.0		-0.74	
2386	ISO18254-1	19.6		-0.84	
2390	ISO18254-1	17.40		-1.41	
2425	In house	25.0		0.54	
2426	ISO18254-1	18.06		-1.24	
2429	ISO18254-1	21.16		-0.44	
2449	ISO18254	54.8	C,R(0.01)	8.20	First reported 157.6
2452	ISO18254-1	30.26	C	1.90	First reported not detected
2456	ISO18254-1	65.6	R(0.01)	10.98	
2459	ISO18254-1	29.50	C	1.70	First reported 26.58
2486	ISO18254-1	23.197		0.08	
2489	ISO18254-1	25.4		0.65	
2511	ISO18254	17.64		-1.35	
2532	ISO/DIS 18254-1	23.3		0.11	
2549	ISO/DIS 18254-1	25.65		0.71	
2561		45.09	R(0.01)	5.71	
2567	ISO18254-1	18		-1.26	
2572	ISO/DIS 18254-1	17.0		-1.51	
2582	In house	24.65		0.45	
2590	ISO/DIS 18254-1	16.989		-1.52	
2591	In house	20.66		-0.57	
2605	GB/T23972	20.021		-0.74	
2618	ISO18254-1	25.11		0.57	
2638	In house	63.16	R(0.01)	10.35	
2643	ISO18254-1	46.39	C,R(0.01)	6.04	First reported 61.57
2644	ISO18254-1	70.66	R(0.01)	12.28	
2649	In house	28.00		1.31	
2668	ISO18254-1	24.67		0.46	
2678	ISO18254-1	25.870		0.77	
2719		26	C	0.80	First reported 53
2743	ISO18254-1	28.80		1.52	
2798	ISO18254-1	24		0.29	
2826	ISO18254-1	24.6		0.44	
2827	ISO/DIS 18254-1	25.50		0.67	
2864	In house	4.85	C,R(0.01)	-4.64	First reported 5.73
2892	ISO/DIS 18254-1	24.000		0.29	

lab	method	value	mark	z(targ)	remarks
2947	ISO18254Mod.	21.73	C	-0.30	First reported 40.85
2960	ISO18254-1	22.1		-0.20	
2982	ISO/DIS 18254-1	24.52		0.42	
3100	ISO/DIS 18254-1	22.39		-0.13	
3116	ISO/DIS 18254-1	22.73		-0.04	
3118		-----		-----	
3154	In house	23.6		0.18	
3172	ISO18254-1	19.85		-0.78	
3176	ISO/DIS 18254-1	34.70		3.04	
3185	ISO18254-1	21.76		-0.29	
3190	ISO18254-1	18.99		-1.00	
3197	ISO18254-1	25.6		0.70	
3210	In house	20.59		-0.59	
3214	ISO/DIS 18254-1	18.16		-1.21	
3222	ISO18254-1	20.00	C	-0.74	First reported 48.07
3228	ISO18254-1	25.0		0.54	
3230	In house	26.14877		0.84	
3237	ISO18254-1	32.31		2.42	
3246	ISO18254-1	25.50		0.67	
3248	In house	20		-0.74	
3250		23.8		0.23	

	normality	OK	
	n	83	
	outliers	8	
	mean (n)	22.886	
	st.dev. (n)	4.2041	RSD=18%
	R(calc.)	11.771	
	st.dev.(iis memo 2203)	3.8907	
	R(iis memo 2203)	10.894	
Compare			
	R(ISO18254-1:16)	6.285	



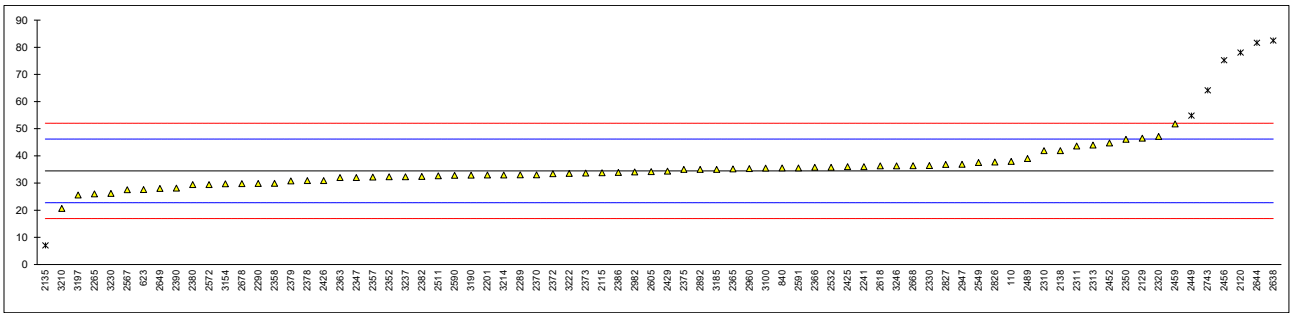


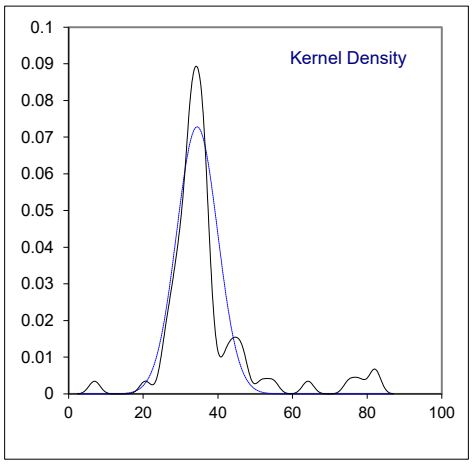
Determination of Total of OP, NP, OPEO and NPEO on sample #22515; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO18254-1	37.96		0.59	
551		-----		-----	
623	ISO18254-1	27.57	C	-1.18	First reported 18.675
840		35.5		0.17	
2115	ISO18254-1	33.77		-0.12	
2120	ISO21081 / ISO18254-1	78	C,E,R(0.01)	7.43	First reported 59.2 / iis calc. 75
2129		46.5		2.05	
2132	ISO18254-2	NA		-----	
2135	ISO18254	6.98	R(0.01)	-4.69	
2138	ISO/DIS 18254-1	41.93		1.27	
2165		-----		-----	
2201	GB/T23322	32.9		-0.27	
2241	ISO18254-1	36.004		0.26	
2265	ISO18254-1	26.0	C	-1.45	First reported 16.0
2289	ISO18254-1	33		-0.25	
2290	ISO/DIS 18254-1	29.8		-0.80	
2293		-----		-----	
2301		-----		-----	
2310	ISO18254-1	41.9		1.27	
2311	ISO18254-1	43.61		1.56	
2313	ISO18254-1	44.01		1.63	
2320	ISO21084 / ISO18254-1	47.13		2.16	
2330	ISO18254-1	36.38		0.33	
2347	ISO18254-1	32		-0.42	
2350	ISO18254-1	46.1	C	1.98	First reported 57.71
2352	ISO18254-1	32.3		-0.37	
2357		32.17		-0.39	
2358	In house	29.93		-0.78	
2363	ISO18254-1	32		-0.42	
2365	ISO18254-1	35.2		0.12	
2366	ISO18254-1	35.8		0.23	
2370	ISO18254-1	33.0		-0.25	
2372	ISO/DIS 18254-1	33.39		-0.19	
2373	ISO18254-1	33.63		-0.14	
2375	ISO18254-1	35		0.09	
2378	GB/T23322	30.9		-0.61	
2379	ISO18254-1	30.729		-0.64	
2380	ISO18254-1	29.40		-0.87	
2382	ISO18254-1	32.4		-0.35	
2386	ISO18254-1	33.9		-0.10	
2390	ISO18254-1	28.09		-1.09	
2425	In house	36.0		0.26	
2426	ISO18254-1	30.91		-0.61	
2429	ISO18254-1	34.39		-0.01	
2449	ISO18254	54.8	C,R(0.05)	3.47	First reported 285.2
2452	ISO18254-1	44.73	C	1.75	First reported 14.47
2456	ISO18254-1	75.2	R(0.01)	6.95	
2459	ISO21084 / ISO18254-1	51.751	C	2.95	First reported 74.06
2486		-----		-----	
2489	ISO18254-1	39		0.77	
2511	ISO18254	32.661		-0.31	
2532	ISO/DIS 18254-1	35.8		0.23	
2549	ISO/DIS 18254-1	37.56		0.53	
2561		-----		-----	
2567	ISO18254-1	27.5		-1.19	
2572	ISO/DIS 18254-1	29.4		-0.87	
2582	In house	Not determined		-----	
2590	ISO/DIS 18254-1	32.8		-0.29	
2591	In house	35.53		0.18	
2605	GB/T23972	34.155		-0.05	
2618	ISO18254-1	36.31		0.31	
2638	In house	82.45	R(0.01)	8.19	
2643		-----		-----	
2644	ISO21084 / ISO18254-1	81.67	R(0.01)	8.05	
2649	In house	28.00		-1.10	
2668	ISO18254-1	36.34		0.32	
2678	ISO18254-1	29.750		-0.81	
2719		-----		-----	
2743	ISO21084 / ISO18254-1	64.17	C,E,R(0.01)	5.07	First reported 63.35 / iis calc. 57.23
2798		-----		-----	
2826	ISO18254-1	37.7		0.55	
2827	ISO/DIS 18254-1	36.79		0.40	
2864		-----		-----	
2892	ISO/DIS 18254-1	35.000		0.09	

lab	method	value	mark	z(targ)	remarks
2947	ISO18254Mod.	36.89	C	0.41	First reported 56.01
2960	ISO18254-1	35.3		0.14	
2982	ISO/DIS 18254-1	34.06		-0.07	
3100	ISO/DIS 18254-1	35.47		0.17	
3116		-----		-----	
3118		-----		-----	
3154	In house	29.7	C,E	-0.81	First reported 29.8 / iis calc. 36.3
3172		-----		-----	
3176		-----		-----	
3185	ISO18254-1	35.00		0.09	
3190	ISO18254-1	32.86		-0.28	
3197	ISO18254-1	25.6	E	-1.51	iis calc. 39.4, after NP correction Total was not updated
3210	In house	20.59		-2.37	
3214	ISO/DIS 18254-1	32.99		-0.25	
3222	ISO18254-1	33.52	C	-0.16	First reported 61.59
3228		-----		-----	
3230	In house	26.14877		-1.42	
3237	ISO18254-1	32.31		-0.37	
3246	ISO21084 / ISO18254-1	36.32		0.31	
3248		-----		-----	
3250		-----		-----	

	normality	suspect	
	n	69	
	outliers	7	
	mean (n)	34.474	
	st.dev. (n)	5.4811	RSD=16%
	R(calc.)	15.347	
	st.dev.(iis memo 2203)	5.8607	
	R(iis memo 2203)	16.410	
Compare			
	R(ISO18254-1:16)	9.468	



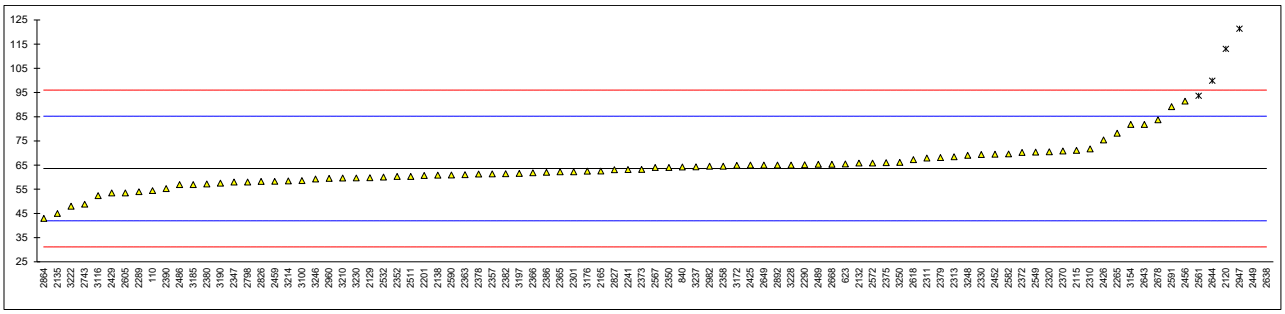


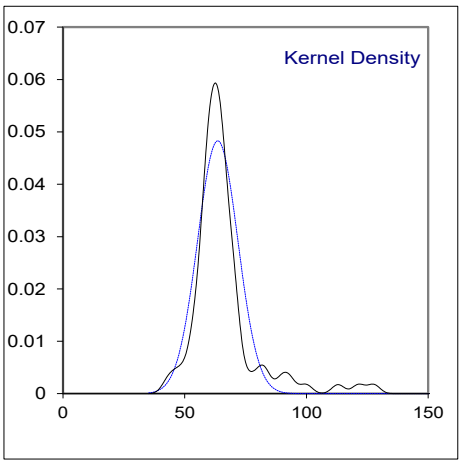
Determination of Octylphenol Ethoxylates (OPEO) on sample #22516; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO18254-1	54.43		-0.85	
551		-----		-----	
623	ISO18254-1	65.45	C	0.17	First reported 94.52
840	ISO18254-1	64.2		0.06	
2115	ISO18254-1	71.1		0.69	
2120	ISO18254-1	113	C,R(0.01)	4.57	First reported 103
2129		59.8		-0.35	
2132	ISO18254-2	65.8		0.20	
2135	ISO18254	45.0		-1.72	
2138	ISO18254-1	60.82		-0.26	
2165	ISO18254-1	62.5		-0.10	
2201	GB/T23322	60.7		-0.27	
2241	ISO18254-1	63.174		-0.04	
2265	ISO18254-1	78.2		1.35	
2289	ISO18254-1	54		-0.89	
2290	ISO/DIS 18254-1	65.1		0.14	
2293		-----		-----	
2301	ISO18254-1	62.24		-0.13	
2310	ISO18254-1	71.7		0.75	
2311	ISO18254-1	67.92		0.40	
2313	ISO18254-1	68.44		0.45	
2320	ISO18254-1	70.47		0.64	
2330	ISO18254-1	69.37		0.53	
2347	ISO18254-1	58		-0.52	
2350	ISO18254-1	64.02		0.04	
2352	ISO18254-1	60.3		-0.31	
2357	ISO18254-1	61.35		-0.21	
2358	In house	64.55		0.09	
2363	ISO18254-1	61		-0.24	
2365	ISO18254-1	62.2		-0.13	
2366	ISO18254-1	61.8		-0.17	
2370	ISO18254-1	70.8		0.67	
2372	ISO/DIS 18254-1	70.21		0.61	
2373	ISO18254-1	63.21		-0.04	
2375	ISO18254-1	66		0.22	
2378	GB/T23322	61.3		-0.21	
2379	ISO18254-1	68.075		0.41	
2380	ISO/DIS 18254-1	57.17		-0.59	
2382	ISO18254-1	61.4		-0.20	
2386	ISO18254-1	62		-0.15	
2390	ISO18254-1	55.23		-0.77	
2425	In house	65.0		0.13	
2426	ISO18254-1	75.42		1.09	
2429	ISO18254-1	53.48		-0.94	
2449	ISO18254	127.6	C,R(0.01)	5.92	First reported for sample #22515
2452	ISO18254-1	69.45		0.54	
2456	ISO18254-1	91.4		2.57	
2459	ISO/DIS 18254-1	58.29		-0.49	
2486	ISO18254-1	56.909		-0.62	
2489	ISO18254-1	65.3		0.16	
2511	ISO18254	60.31		-0.30	
2532	ISO/DIS 18254-1	60.0		-0.33	
2549	ISO/DIS 18254-1	70.32	C	0.62	First reported not detected
2561		93.65	R(0.05)	2.78	
2567	ISO18254-1	64		0.04	
2572	ISO/DIS 18254-1	65.8		0.20	
2582	In house	69.60		0.55	
2590	ISO18254-1	60.871		-0.25	
2591	In house	89.18		2.37	
2605	GB/T23972	53.481		-0.94	
2618	ISO18254-1	67.21		0.33	
2638	In house	193.86	C,R(0.01)	12.05	First reported not detected
2643	ISO18254-1	81.79	C	1.68	First reported 96.09
2644	ISO21084 / ISO18254-1	99.85	R(0.05)	3.35	
2649	In house	65.00		0.13	
2668	ISO18254-1	65.30		0.16	
2678	ISO18254-1	83.720		1.86	
2719		-----		-----	
2743	ISO18254-1	48.83		-1.37	
2798	ISO18254-1	58		-0.52	
2826	ISO18254-1	58.2		-0.50	
2827	ISO/DIS 18254-1	63.11		-0.05	
2864	In house	42.92		-1.91	
2892	ISO18254-1	65.000		0.13	

lab	method	value	mark	z(targ)	remarks
2947	ISO18254Mod.	121.39	C,R(0.01)	5.34	First reported 89.95
2960	ISO18254-1	59.5		-0.38	
2982	ISO/DIS 18254-1	64.53		0.09	
3100	ISO/DIS 18254-1	58.55		-0.47	
3116	ISO/DIS 18254-1	52.36		-1.04	
3118		-----		-----	
3154	In house	81.76	C	1.68	First reported 125.4
3172	ISO18254-1	64.9		0.12	
3176	ISO18254-1	62.44		-0.11	
3185	ISO18254-1	56.94		-0.62	
3190	ISO18254-1	57.54		-0.56	
3197	ISO18254-1	61.5		-0.19	
3210	In house	59.62		-0.37	
3214	ISO18254-1	58.47		-0.47	
3222	ISO/DIS 18254-1	48	C	-1.44	First reported 101.4
3228	ISO18254-1	65.0		0.13	
3230	In house	59.67243		-0.36	
3237	ISO18254-1	64.23		0.06	
3246	ISO18254-1	59.25		-0.40	
3248	In house	69		0.50	
3250		66.1		0.23	

	normality	not OK	
	n	85	
	outliers	6	
	mean (n)	63.603	
	st.dev. (n)	8.2658	RSD=13%
	R(calc.)	23.144	
	st.dev.(iis memo 2203)	10.8126	
	R(iis memo 2203)	30.275	
Compare			
	R(ISO18254-1:16)	17.468	



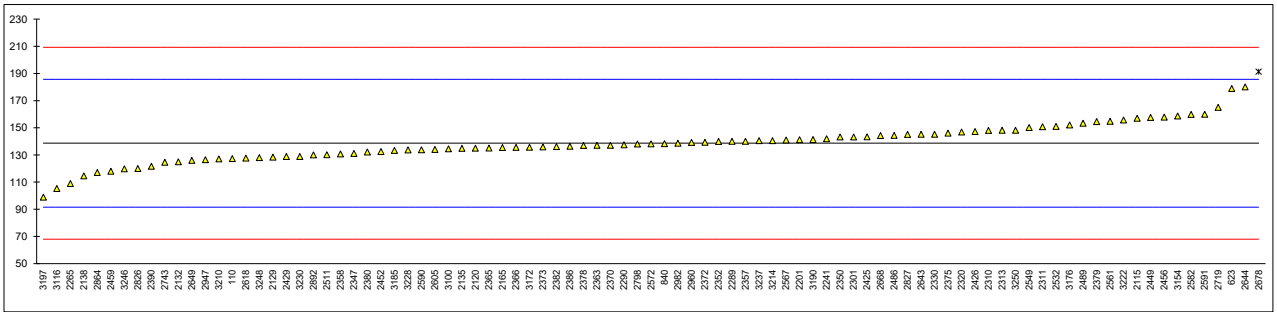


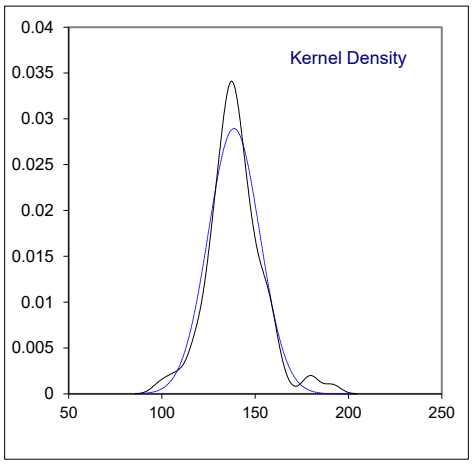
Determination of Nonylphenol Ethoxylates (NPEO) on sample #22516; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO18254-1	127.3		-0.48	
551		-----		-----	
623	ISO18254-1	178.89		1.71	
840	ISO18254-1	138.3		-0.02	
2115	ISO18254-1	157		0.78	
2120	ISO18254-1	135		-0.16	
2129		128.3		-0.44	
2132	ISO18254-2	125		-0.58	
2135	ISO18254	134.9		-0.16	
2138	ISO18254-1	114.61		-1.02	
2165	ISO18254-1	135.4		-0.14	
2201	GB/T23322	141.2		0.11	
2241	ISO18254-1	141.943		0.14	
2265	ISO18254-1	108.9		-1.26	
2289	ISO18254-1	140		0.06	
2290	ISO/DIS 18254-1	137.4		-0.05	
2293		-----		-----	
2301	ISO18254-1	143.2		0.19	
2310	ISO18254-1	148		0.40	
2311	ISO18254-1	150.8		0.52	
2313	ISO18254-1	148.20		0.40	
2320	ISO18254-1	146.93		0.35	
2330	ISO18254-1	145.265		0.28	
2347	ISO18254-1	131		-0.32	
2350	ISO18254-1	143.18		0.19	
2352	ISO18254-1	139.9		0.05	
2357	ISO18254-1	140.1		0.06	
2358	In house	130.68		-0.34	
2363	ISO18254-1	137		-0.07	
2365	ISO18254-1	135.1		-0.15	
2366	ISO18254-1	135.6		-0.13	
2370	ISO18254-1	137		-0.07	
2372	ISO/DIS 18254-1	139.3		0.03	
2373	ISO18254-1	135.91		-0.12	
2375	ISO18254-1	146		0.31	
2378	GB/T23322	136.9		-0.07	
2379	ISO18254-1	154.496		0.67	
2380	ISO/DIS 18254-1	132.14		-0.28	
2382	ISO18254-1	136.1		-0.11	
2386	ISO18254-1	136.3		-0.10	
2390	ISO18254-1	121.61		-0.72	
2425	In house	143.3		0.20	
2426	ISO18254-1	147.23		0.36	
2429	ISO18254-1	128.80		-0.42	
2449	ISO18254	157.6	C	0.80	First reported 54.8
2452	ISO18254-1	132.46		-0.26	
2456	ISO18254-1	157.8		0.81	
2459	ISO/DIS 18254-1	117.97		-0.88	
2486	ISO18254-1	144.37		0.24	
2489	ISO18254-1	153.3		0.62	
2511	ISO18254	130.095		-0.36	
2532	ISO/DIS 18254-1	151.0		0.52	
2549	ISO/DIS 18254-1	150.12		0.49	
2561		154.79		0.68	
2567	ISO18254-1	141		0.10	
2572	ISO/DIS 18254-1	138.1		-0.02	
2582	In house	159.80		0.90	
2590	ISO18254-1	133.682		-0.21	
2591	In house	159.92		0.90	
2605	GB/T23972	134.044		-0.20	
2618	ISO18254-1	127.64		-0.47	
2638	In house	not detected		-----	
2643	ISO18254-1	145.26	C	0.28	First reported 206.04
2644	ISO18254-1	180.20		1.76	
2649	In house	126.00		-0.54	
2668	ISO18254-1	144.31		0.24	
2678	ISO18254-1	191.270	R(0.05)	2.23	
2719		165		1.12	
2743	ISO18254-1	124.54		-0.60	
2798	ISO18254-1	138		-0.03	
2826	ISO18254-1	120		-0.79	
2827	ISO/DIS 18254-1	145.01		0.27	
2864	In house	117.01		-0.92	
2892	ISO18254-1	130.000		-0.37	

lab	method	value	mark	z(targ)	remarks
2947	ISO18254Mod.	126.44		-0.52	
2960	ISO18254-1	139.2		0.02	
2982	ISO/DIS 18254-1	138.63		0.00	
3100	ISO/DIS 18254-1	134.47		-0.18	
3116	ISO/DIS 18254-1	105.28		-1.42	
3118		-----		-----	
3154	In house	158.7		0.85	
3172	ISO18254-1	135.65		-0.13	
3176	ISO18254-1	152.00		0.57	
3185	ISO18254-1	133.33		-0.23	
3190	ISO18254-1	141.25		0.11	
3197	ISO18254-1	98.9		-1.69	
3210	In house	127.10		-0.49	
3214	ISO18254-1	140.51		0.08	
3222	ISO/DIS 18254-1	155.71		0.72	
3228	ISO18254-1	133.6		-0.21	
3230	In house	128.8325		-0.42	
3237	ISO18254-1	140.45		0.08	
3246	ISO18254-1	119.8		-0.80	
3248	In house	128		-0.45	
3250		148.2		0.40	

	normality	suspect	
	n	90	
	outliers	1	
	mean (n)	138.658	
	st.dev. (n)	13.7803	RSD=10%
	R(calc.)	38.585	
	st.dev.(iis memo 2203)	23.5719	
	R(iis memo 2203)	66.001	
Compare			
	R(ISO18254-1:16)	38.080	



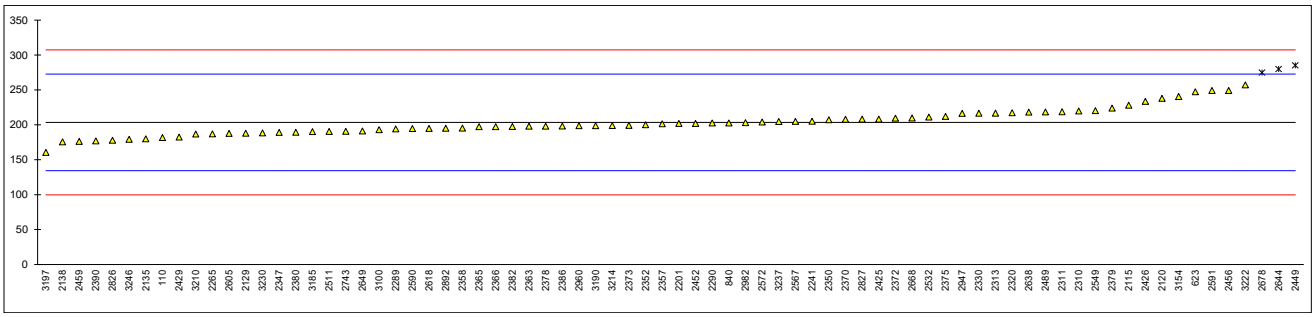


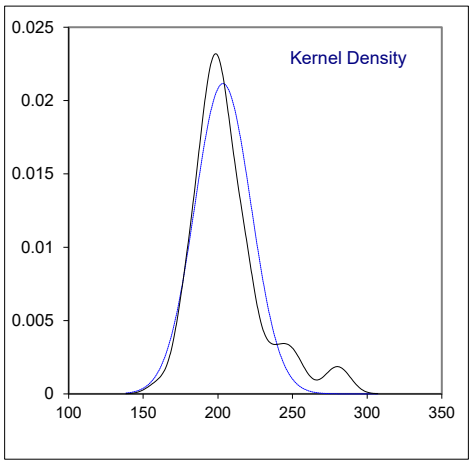
Determination of Total of OP, NP, OPEO and NPEO on sample #22516; results in mg/kg

lab	method	value	mark	z(targ)	remarks
110	ISO18254-1	181.73		-0.63	
551		-----		-----	
623	ISO18254-1	247.28	C	1.27	First reported 276.35
840	ISO18254-1	202.5		-0.03	
2115	ISO18254-1	228.1		0.71	
2120	ISO18254-1	238	E	1.00	iis cal. 248, after OPEO correction Total was not updated
2129		188.1		-0.44	
2132	ISO18254-2	NA		-----	
2135	ISO18254	179.9		-0.68	
2138	ISO18254-1	175.43		-0.81	
2165		-----		-----	
2201	GB/T23322	201.9		-0.05	
2241	ISO18254-1	205.117		0.05	
2265	ISO18254-1	187.1		-0.47	
2289	ISO18254-1	194		-0.27	
2290	ISO/DIS 18254-1	202.5		-0.03	
2293		-----		-----	
2301		-----		-----	
2310	ISO18254-1	219.7		0.47	
2311	ISO18254-1	218.72		0.44	
2313	ISO18254-1	216.64		0.38	
2320	ISO21084 / ISO18254-1	217.40		0.40	
2330	ISO18254-1	216.43		0.37	
2347	ISO18254-1	189		-0.42	
2350	ISO18254-1	207.20		0.11	
2352	ISO18254-1	200.2		-0.09	
2357	ISO18254-1	201.45		-0.06	
2358	In house	195.23		-0.24	
2363	ISO18254-1	198		-0.16	
2365	ISO18254-1	197.3		-0.18	
2366	ISO18254-1	197.4		-0.18	
2370	ISO18254-1	207.8		0.13	
2372	ISO/DIS 18254-1	209.51		0.17	
2373	ISO18254-1	199.12		-0.13	
2375	ISO18254-1	212		0.25	
2378	GB/T23322	198.2		-0.15	
2379	ISO18254-1	224.044		0.60	
2380	ISO/DIS 18254-1	189.31		-0.41	
2382	ISO18254-1	197.5		-0.17	
2386	ISO18254-1	198.3		-0.15	
2390	ISO18254-1	176.84		-0.77	
2425	In house	208.3		0.14	
2426	ISO18254-1	233.54	E	0.87	iis cal. 222.65
2429	ISO18254-1	182.28		-0.61	
2449	ISO18254	285.2	C,E,R(0.05)	2.36	First reported 71.4 / iis cal. 301.8
2452	ISO18254-1	201.91		-0.04	
2456	ISO18254-1	249.2		1.32	
2459	ISO/DIS 18254-1	176.26		-0.79	
2486		-----		-----	
2489	ISO18254-1	218.6		0.44	
2511	ISO18254	190.405		-0.38	
2532	ISO/DIS 18254-1	211		0.22	
2549	ISO/DIS 18254-1	220.44		0.49	
2561		-----		-----	
2567	ISO18254-1	205		0.04	
2572	ISO/DIS 18254-1	203.9		0.01	
2582	In house	Not determined		-----	
2590	ISO18254-1	194.553		-0.26	
2591	In house	249.10		1.32	
2605	GB/T23972	187.525		-0.46	
2618	ISO18254-1	194.85		-0.25	
2638	In house	218.06	C	0.42	First reported 257.76
2643		-----		-----	
2644	ISO21084 / ISO18254-1	280.05	R(0.05)	2.21	
2649	In house	191.00		-0.36	
2668	ISO18254-1	209.61		0.18	
2678	ISO18254-1	274.990	R(0.05)	2.07	
2719		-----		-----	
2743	ISO21084 / ISO18254-1	190.72	E	-0.37	iis calc. 181.66, after NP correction Total was not updated
2798		-----		-----	
2826	ISO18254-1	178		-0.74	
2827	ISO/DIS 18254-1	208.12		0.13	
2864		-----		-----	
2892	ISO18254-1	195.000		-0.24	

lab	method	value	mark	z(targ)	remarks
2947	ISO18254Mod.	216.39	E	0.37	iis cal. 247.83. After OPEO correct. Total was not updated
2960	ISO18254-1	198.7		-0.14	
2982	ISO/DIS 18254-1	203.16		-0.01	
3100	ISO/DIS 18254-1	193.02		-0.30	
3116		-----		-----	
3118		-----		-----	
3154	In house	240.5	C	1.07	First reported 284.2
3172		-----		-----	
3176		-----		-----	
3185	ISO18254-1	190.27		-0.38	
3190	ISO18254-1	198.79		-0.14	
3197	ISO18254-1	160.4		-1.24	
3210	In house	186.72		-0.48	
3214	ISO18254-1	198.98		-0.13	
3222	ISO/DIS 18254-1	257.11	E	1.55	iis cal. 203.71. After OPEO correct. Total was not updated
3228		-----		-----	
3230	In house	188.50493		-0.43	
3237	ISO18254-1	204.68		0.04	
3246	ISO21084 / ISO18254-1	179.05		-0.71	
3248		-----		-----	
3250		-----		-----	

	normality	OK	
	n	73	
	outliers	3	
	mean (n)	203.460	
	st.dev. (n)	18.8655	RSD=9%
	R(calc.)	52.823	
	st.dev.(iis memo 2203)	34.5882	
	R(iis memo 2203)	96.847	
Compare			
	R(ISO18254-1:16)	55.877	





APPENDIX 2

Summary of other reported components in sample #22515 and #221516; results in mg/kg

Abbreviations of components

OP = Octylphenol
OPEO = Octylphenol Ethyloxylates
NP = Nonylphenol

	#22515				#22516		
lab	**OP**		**OPEO**		**OP**		**NP**
110	not detected		not detected		not detected		not detected
551	n		-----		-----		-----
623	Not Detected	C	not detected		1.13		1.81
840	not determined		not determined		not determined		not determined
2115	N		1.59		-----		-----
2120	< 0.5		< 0.5		< 5		< 5
2129	not detected		1.19		not detected		not detected
2132	<10		<10		<10		<10
2135	N		-----		-----		-----
2138	ND		ND		ND		ND
2165	not detected		not detected		not detected		not detected
2201	<10		<10		<10		<10
2241	<10		<10		<10		<10
2265	< 2		< 10		< 2		< 2
2289	N		-----		-----		-----
2290	<10		<10		<10		<10
2293	N		-----		-----		-----
2301	N		-----		-----		-----
2310	Not Detected		Not Detected		Not Detected		Not Detected
2311	Not Detected		<3		Not Detected		Not Detected
2313	NOT DETECTED		NOT DETECTED		NOT DETECTED		NOT DETECTED
2320	Not Detected		Not Detected		Not Detected		Not Detected
2330	Not detected		1.205		Not detected		1.795
2347	<10		<10		<10		<10
2350	< 3.00		< 1.00		< 3.00		< 3.00
2352	N		-----		-----		-----
2357	N		-----		-----		-----
2358	not detected		not detected		not detected		not detected
2363	<3		<3		<3		<3
2365	<10		<20		<10		<10
2366	<10		<20		<10		<10
2370	<1		<1		<1		<1
2372	<1		<1		<1		<1
2373	not detected		not detected		not detected		not detected
2375	N		-----		-----		-----
2378	<0.25		<0.5		<0.25		<0.5
2379	Not detected		1.082		Not detected		1.473
2380	<3		<1		<3		<3
2382	<10		<10		<10		<10
2386	<5		<10		<5		<5
2390	not detected		not detected		not detected		not detected
2425	Not detected		Not detected		Not detected		Not detected
2426	Not Detected		Not Detected		Not Detected		Not Detected
2429	N		-----		-----		-----
2449	N		-----	C	-----		16.6
2452	not detected		not detected		not detected		not detected
2456	N		1.7		not detected		not detected
2459	11.750	C	ND		ND		ND
2486	n		-----		-----		-----
2489	Not Detected		Not Detected		Not Detected		Not Detected
2511	n		-----		-----		-----
2532	Not detected		Not detected		Not Detected		Not Detected
2549	Not Detected		Not Detected		Not Detected	C	Not Detected
2561	n		not detected		-----		-----
2567	<10		<10		<10		<10
2572	<10		<10		<10		<10
2582	Not determined		Not determined		Not determined		Not determined
2590	n		-----		-----		-----
2591	not detected		not detected		not detected		not detected
2605	<10.000		<10.000		<10.000		<10.000
2618	not detected		not detected		not detected		not detected
2638	not detected		not detected		24.2		not detected
2643	n		-----		-----		-----
2644	n		-----		-----		-----
2649	n		-----		-----		-----

	#22515		#22516		
lab	OP	OPEO	OP	NP	
2668	Not detected	Not detected	Not detected	Not detected	
2678	<3	3.880	<3	<3	
2719	n	-----	-----	-----	
2743	not detected	1.58	2.34	5.95	C
2798	n	-----	-----	-----	
2826	not detected	not detected	not detected	not detected	
2827	Not Detected	Not Detected	Not Detected	Not Detected	
2864	n	1.39	-----	-----	
2892	n	-----	-----	-----	
2947	not detected	2.23	not detected	<LOQ [2mg/kg]	
2960	not detected	not detected	not detected	not detected	
2982	not detected	not detected	not detected	not detected	
3100	＜10	＜10	＜10	＜10	
3116	n	-----	-----	-----	
3118	n	-----	-----	-----	
3154	n	2.2	-----	-----	
3172	< 1	< 1	< 1	< 1	
3176	n	-----	-----	-----	
3185	Not detected (<10)	Not detected (<10)	Not detected (<10)	Not detected (<10)	
3190	<10	<10	<10	<10	
3197	<10	<10	<10	<10	
3210	<20	<20	<20	<20	
3214	N.D.	N.D.	N.D.	N.D.	
3222	n	1.40	-----	-----	
3228	not detected	not detected	not detected	not detected	
3230	not analyzed	not detected	not analyzed	not analyzed	
3237	n	-----	-----	-----	
3246	Not detected	Not detected	Not detected	Not detected	
3248	n	-----	-----	-----	
3250	n	-----	-----	-----	

Lab 623 first reported 1.40
Lab 2449 first reported for sample #22516, reported 127.6
Lab 2459 first reported 36.75
Lab 2549 first reported 70.32
Lab 2743 first reported 15.01

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)
110	Yes	Further cut	1	Ultrasonic	methanol	60	70
551	---	---		---			
623	Yes	Further cut	1	Ultrasonic	methanol	60	70
840	Yes	Further cut	0.5g	Ultrasonic	Methanol	60 minutes	70°C
2115	Yes	Used as received	0.5 g	Ultrasonic	MeOH	60 min	60°C
2120	Yes	Used as received	1 g	Ultrasonic	Methanol	60 min	70 ºC
2129	Yes	Used as received	1.0 g	Ultrasonic	MeOH	60 min	70°C
2132	No	Used as received	1 gram	Ultrasonic	Methanol	60 minutes	70°C
2135	Yes	Used as received		Ultrasonic	Methanol	60	60
2138	Yes	Used as received	about 0.5 g	Ultrasonic	methanol	60 minutes	70 C
2165	Yes	Further cut	1	Ultrasonic	Methanol	60	70
2201	Yes	Further cut	1g	Ultrasonic	Methanol	60 min	70°C
2241	Yes	Further cut	0.5g	Ultrasonic	methanol	60 min	70°C
2265	Yes	Used as received	1g	Ultrasonic	Methanol	60	60
2289	Yes	Further cut	1.0g	Ultrasonic	Methanol	60mins	70°C
2290	Yes	---		---			
2293	---	---		---			
2301	Yes	Used as received	1 gram	Ultrasonic	Methanol	60 min	70c
2310	Yes	Used as received	1 gram	Ultrasonic	Methanol	60 minutes	70°C
2311	Yes	Further cut	1	Ultrasonic	Methanol	60	70
2313	No	Further cut	1.0g	Ultrasonic	METHANOL	1.0hr	70
2320	Yes	Further cut	0.5g	Ultrasonic	Methanol	60min	70°C
2330	Yes	Further cut #22515 used as received; #22516	1 g	Ultrasonic	Methanol	60 mins	70 ± 2 °C
2347	Yes	further cut	0.5g	Ultrasonic	methanol	60min	70℃
2350	Yes	Further cut	1 g	Ultrasonic	Methanol	60 minutes	70 °C
2352	Yes	Further cut	1g	Ultrasonic	Mathanol	60min	70°C
2357	---	---		---			
2358	Yes	Further cut	1.0 g	Ultrasonic	Methanol	60 minutes	70°C
2363	Yes	Further cut	19	Ultrasonic	MeOH	60 mins	70°C
2365	Yes	Further cut	1.0g	Ultrasonic	methanol	60min	70℃
2366	Yes	Used as received	0.5	Ultrasonic	Methanol	60	70
2370	Yes	Further cut	0.5 g	Ultrasonic	10 mL	60 min	70°C
2372	No	Further cut	1g	Ultrasonic	MeOH	60	70
2373	Yes	Used as received	1.0g	Ultrasonic	Methanol	1 hour	60°C
2375	Yes	Further cut	0.5g	Ultrasonic	Methanol	60 min	70°C
2378	Yes	Used as received	1G	Ultrasonic	methanol	60	70
2379	Yes	Further cut	0.5 g	Ultrasonic	Methanol	60 minutes	70 C
2380	Yes	Used as received	1.0 g	Ultrasonic	Methanol	60 Minute	70 °C
2382	Yes	Further cut	1G	Ultrasonic	Methanol	60 min	70°C
2386	Yes	Further cut	0,5 g	Ultrasonic	Methanol	60 min	70 °C
2390	Yes	Further cut	1g	Ultrasonic	methanol	60	70
2425	Yes	Further cut	1.0g	Ultrasonic	Methanol	60 Minutes	70°C
2426	Yes	Further cut	0.5g	Ultrasonic	Methanol	60minutes	70°C
2429	Yes	Further cut	0.5g	Ultrasonic	Methanol	60min	70°C
2449	Yes	Further cut	1.0 gram	Ultrasonic			
2452	Yes	Used as received	1	Ultrasonic	Methanol	60	70
2456	Yes	Used as received	All sample dispatched was used	Ultrasonic	Methanol	60	70
2459	Yes	Used as received	1.0 gm	Ultrasonic	Methanol	60 mints	60C
2486	Yes	Used as received	1 g	Ultrasonic	Methanol	60 minutes	70°C
2489	Yes	Further cut	1.0085g/ 1.0024g	Ultrasonic	Methanol/Water	60 minutes	70°C
2511	No	Further cut		Ultrasonic	Methanol	60 min	70°C
2532	No	Further cut	1 gram	Ultrasonic	Methanol	60 minutes	70 °C
2549	Yes	Used as received	1 gm	Ultrasonic	Methanol	60 minutes	70°C
2561	Yes	Further cut	1 g	Ultrasonic	methanol	60	70
2567	Yes	Further cut	1 gm	Ultrasonic	Methanol	60	70
2572	Yes	---	#22515-1.0002g; #22516-	---			
2582	Yes	Further cut	1.0011g	Ultrasonic	Methanol	60 Minutes	70°C
2590	Yes	Used as received	1g	Ultrasonic	MeOH	60 min	70°C
2591	No	Further cut	1.0 gram	Ultrasonic	Methanol	60 min	40ºC
2605	Yes	Further cut	1.000g	Ultrasonic	Methanol	30min	70°C
2618	No	Used as received	1.0 gm	Ultrasonic	Methanol	60 minutes	70 °C

lab	ISO/IEC17025 accredited	sample preparation	sample intake (g)	release/ extraction technique	release/ extraction solvent	extraction time (min)	extraction temperature (°C)
2638	No	Further cut	1 grams	Ultrasonic	Methanol	60 minutes	room temp.
2643	Yes	Further cut	1 g	Ultrasonic	Methanol	1h	70
2644	Yes	Used as received	0.5 g	Ultrasonic	Methanol	60 min	70°C
2649	Yes	Used as received	2.5 g	Ultrasonic	MeOH	60 Min	70 °C
2668	Yes	Further cut	0.5 g	Ultrasonic	Methanol	60 minutes	70°C
2678	No	Further cut	1 g	Ultrasonic	Methanol	60	70°C
2719	Yes	Used as received	0.5g	Ultrasonic	methanol	60	70
2743	No	Used as received	1g	Ultrasonic	Methanol	60min	70°C
2798	Yes	Used as received	0.5g	Ultrasonic	MeOH	60mins	70
2826	Yes	Used as received	1g	Ultrasonic	Methanol	60mins	
2827	Yes	Further cut	0.5g	Ultrasonic	Methanol	60 mins	70 °C
2864	Yes	Further cut		Ultrasonic	MEOH	60 min	60
2892	Yes	Further cut	1g	---	MeOH	60	40
2947	Yes	Used as received	0.5	Ultrasonic Mechanical	Methanol	60	60
2960	Yes	Used as received	0.5g	Shaking	methanol	1h	70
2982	Yes	Used as received	1.002g	Stirrer	Methanol	1.0 Hour	70°C
3100	Yes	Further cut	1.0g	Ultrasonic	Methanol	60mins	70℃
3116	Yes	Used as received	1 gram	Ultrasonic	Methanol	60 mins	70°C
3118	---	---		---			
3154	Yes	Used as received		Ultrasonic	Methanol	60	70
3172	Yes	---		---			
3176	Yes	Used as received	1	Ultrasonic	MeOH	60 min	70
3185	Yes	Further cut	1g	Ultrasonic	Methanol	60min	70°C
3190	Yes	Further cut	1g	Ultrasonic	methanol	60min	70
3197	Yes	Further cut	0.5 g	Ultrasonic	Methanol	60 min.	70 C
3210	Yes	Further cut	1	Ultrasonic	Methanol	60	70
3214	Yes	Further cut	1g	Ultrasonic	Methanol	60 mins	70C
3222	Yes	Further cut	0.5	Ultrasonic	Methanol	60	70
3228	Yes	Further cut	0.5	Ultrasonic	Methanol	60	70
3230	Yes	Further cut	1.0g	Ultrasonic	Methanol	60 minutes	70°C
3237	Yes	Used as received	0.5	Ultrasonic	Methanol	60	70
3246	Yes	Used as received	1.0 g	Ultrasonic	Methanol	60 min	70°C
3248	Yes	Used as received	1.0g	Ultrasonic	Methanol	60 minutes	70°C
3250	Yes	Used as received	1g	Ultrasonic	methanol	60min	70°C

APPENDIX 4

Number of participants per country

1 lab in	AUSTRIA
6 labs in	BANGLADESH
1 lab in	BRAZIL
3 labs in	CAMBODIA
1 lab in	FRANCE
5 labs in	GERMANY
1 lab in	GUATEMALA
5 labs in	HONG KONG
8 labs in	INDIA
3 labs in	INDONESIA
7 labs in	ITALY
3 labs in	KOREA, Republic of
1 lab in	MAURITIUS
21 labs in	P.R. of CHINA
5 labs in	PAKISTAN
1 lab in	PORTUGAL
1 lab in	SPAIN
2 labs in	SRI LANKA
4 labs in	TAIWAN
1 lab in	THAILAND
3 labs in	TUNISIA
4 labs in	TURKEY
1 lab in	U.S.A.
1 lab in	UNITED KINGDOM
6 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?

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