Institute for
Interlaboratory Studies

Results of Proficiency Test
Fuel Oil Chemical Species
July 2023

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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CONTENTS

1 INTRODUCTION

Since 1994 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Fuel Oil based on the latest version of ISO8217 and ASTM D396 every year and twice a year since 2016.
In ISO8217 paragraph 5.2 is stated that “the Fuel Oil should be free from any material (..) that causes the fuel to be unacceptable for use (..)”. ASTM D7845 provides a list of possible Chemical Species that should not be present in Fuel Oil and how to measure them. On request of several participants it was decided to organize a round robin especially for the analysis of Fuel Oil Chemical Species in 2023 for the first time.

In this interlaboratory study 13 laboratories in 10 countries registered for participation, see appendix 3 for the number of participants per country. In this report the results of this Fuel Oil Chemical Species proficiency tests 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 one sample of Fuel Oil for the determination of Chemical Species in an amber glass bottle of 50 mL labelled #23107.
The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. 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 site www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

A batch of approximately 5 liters of Fuel Oil containing a number of Chemical Species was provided by a third-party laboratory. After heating to 50 °C and homogenization 38 amber glass bottles of 50 mL were filled and labelled #23107.
The homogeneity of the subsamples was checked by determination of Density at 15 °C in accordance with ISO12185 on 8 stratified randomly selected subsamples.

	Density at 15 °C in kg/m^3
sample #23107-1	1017.7
sample #23107-2	1017.7
sample #23107-3	1017.6
sample #23107-4	1017.7
sample #23107-5	1017.6
sample #23107-6	1017.6
sample #23107-7	1017.6
sample #23107-8	1017.6

Table 1: homogeneity test results of subsamples #23107

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

	Density at 15 °C in kg/m^3
r (observed)	0.1
reference test method	ISO12185:96
0.3 x R (reference test method)	0.5

Table 2: evaluation of the repeatability of subsamples #23107

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

To each of the participating laboratories one 50 mL bottle of Fuel Oil labelled #23107 was sent on June 14, 2023. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Fuel Oil Chemical Species packed in the amber glass was checked. The material was found to be sufficiently stable for the period of the proficiency test.

2.6 ANALYZES

The participants were requested to determine: n-Butyl alcohol, Cyclohexanol, n-Butyl ether, n-Butyl acrylate, Styrene, alpha-Pinene, Phenol, alpha-Methyl styrene, beta-Pinene, 4-Methyl styrene, trans-B-Methyl styrene, 3-Methyl styrene, 2-Methyl styrene, Dicyclopentadiene, Limonene, Indene, 1-Phenyl ethanol, para,alpha-Dimethyl styrene, 2,5-Dimethyl styrene, 2,4-Dimethyl styrene, 2-Phenyl ethanol, 2-Ethyl phenol, 2,4-Dimethyl phenol, 4-Ethyl phenol, 2-Phenoxy-1-propanol, 2-Phenoxy ethanol, 4-iso-Propylphenol, 1-Phenoxy-2-Propanol and Styrene glycol.

It was explicitly requested to treat the sample as if it was a routine sample 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/. 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/. 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.

This 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 a 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 of appendix 1.

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

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

4 EVALUATION

In this proficiency test some problems were encountered with the dispatch of the samples and/or perform the testing. Therefore, the reporting time on the data entry portal was extended with another two weeks. Two participants reported test results after the extended reporting date and all other participants reported the test results in time. Not all participants did report all components requested.
In total 13 participants reported 124 numerical test results. Observed were 8 outlying test results, which is 6.5%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

Not all data sets proved to have a normal Gaussian distribution. The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1 EVALUATION PER COMPONENT

In this section the reported test results are discussed 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 4.

In the iis PT reports ASTM test methods are referred to with a number (e.g. D7845) and an added designation for the year that the test method was adopted or revised (e.g. D7845:20).

Unfortunately, test method ASTM D7845:20 only mentions a temporary standard deviation for the repeatability for one concentration for every component (see table 6 of the test method ASTM D7845:20). As a rule of thumb, the reproducibility is three times the repeatability. Therefore, the reproducibility for a component was calculated as follows: the repeatability of the component was divided by the concentration mentioned in table 6 of ASTM D7845:20, then multiplied by the mean concentration of the component in this PT and finally multiplied by 3.

Styrene: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D7845:20.

Phenol: This determination was problematic. No statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D7845:20.

4-Methyl styrene: This determination was problematic. No statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D7845:20.

trans-B-Methyl styrene: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D7845:20.

3-Methyl styrene: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D7845:20.

2-Methyl styrene: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D7845:20.

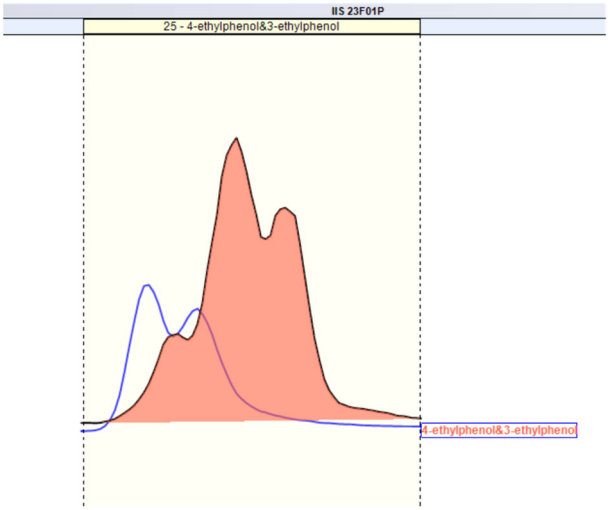
Indene: This determination was problematic. No statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D7845:20.

2,5-Dimethyl styrene: This determination was problematic. No statistical evaluation was performed because the variation is high with a bimodal distribution. Seven participants reported a test result below the detection limit, while five participants reported a test result well above the detection limit. This component may be difficult to determine.

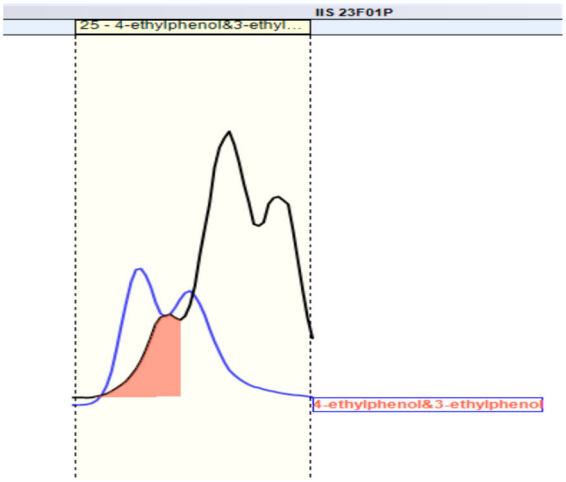
2-Ethyl phenol: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D7845:20.

2,4-Dimethyl phenol: This determination was not problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D7845:20.

4-Ethyl phenol: This determination was problematic. No statistical evaluation was performed because the variation is high with a bimodal distribution. Seven participants reported a test result below the detection limit, while five participants reported a test result that is very high compared to the detection limit.
iis requested only this component, while the method describes that the sum of 3-Ethyl phenol and 4-Ethyl phenol is determined because 4-Ethyl phenol co-elutes with 3-Ethyl phenol (see D7845:20 table 1). In the next PT the sum of these two analytes will be requested.
As this is a new PT for iis, we contacted an expert laboratory to help us explain the determination of these two components.
Looking at the chromatogram (next picture, blue line is reference sample, black line is PT sample), two peaks can be found near the expected peaks of 3-Ethyl phenol and 4-Ethyl phenol. When the peaks which lie next to and over the reference peaks are determined as a whole, the test result would be around 500 ppm.



When only the first peak (which lies in the reference peak area) is determined, the test result would be around 50 ppm.



The expert laboratory is of the opinion that these two components are not present and/or below the detection limit because the two peaks of the sample are not of the same retention time as the reference sample.
Since the method already states that these two components co-elute and that other peaks are present in the same area, each laboratory may make a different decision about whether these components are present or not. Therefore, iis has decided not to evaluate the test results for these two components.

4-iso-Propylphenol: This determination was problematic. No statistical evaluation was performed because the variation is high with a bimodal distribution. Seven participants reported a test result below the detection limit, while five participants reported a test result well above the detection limit. This component may be difficult to determine.

The participants agreed on a concentration near or below the limit of detection for all other components mentioned in paragraph 2.6. Therefore, no z-scores are calculated for these components. The reported test results are given in appendix 2.

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.

Parameter	unit	n	average	2.8 * sd	R(lit)
Styrene	mg/kg	10	101.3	25.9	31.0
Phenol	mg/kg	13	389.0	359.2	325.4
4-Methyl styrene	mg/kg	11	84.5	98.0	48.0
trans-B-Methyl styrene	mg/kg	11	139.8	53.2	121.5
3-Methyl styrene	mg/kg	10	101.5	98.4	54.0
2-Methyl styrene	mg/kg	11	116.1	112.2	209.7
Indene	mg/kg	13	550.2	504.3	232.2
2,5-Dimethyl styrene	mg/kg	n.e.	n.e.	n.e.	n.e.
2-Ethyl phenol	mg/kg	11	133.0	124.4	119.7
2,4-Dimethyl phenol	mg/kg	8	144.4	184.8	175.5
4-Ethyl phenol	mg/kg	n.e.	n.e.	n.e.	n.e.
4-iso-Propylphenol	mg/kg	n.e.	n.e.	n.e.	n.e.

Table 3: reproducibilities of tests on sample #23107

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

4.3 OVERVIEW OF THE PROFICIENCY TEST OF JULY 2023

	July 2023
Number of reporting laboratories	13
Number of test results	124
Number of statistical outliers	8
Percentage of statistical outliers	6.5%

Table 4: overview of the proficiency tests

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

The performance of the determinations of the proficiency test was compared to the requirements of the reference test method. The conclusions are given in the following table.

Parameter	July 2023
Styrene	+
Phenol	-
4-Methyl styrene	--
trans-B-Methyl styrene	++
3-Methyl styrene	-
2-Methyl styrene	+
Indene	--
2-Ethyl phenol	+/-
2,4-Dimethyl phenol	+/-

Table 5: comparison of determinations to the reference test method

The following performance categories were used:

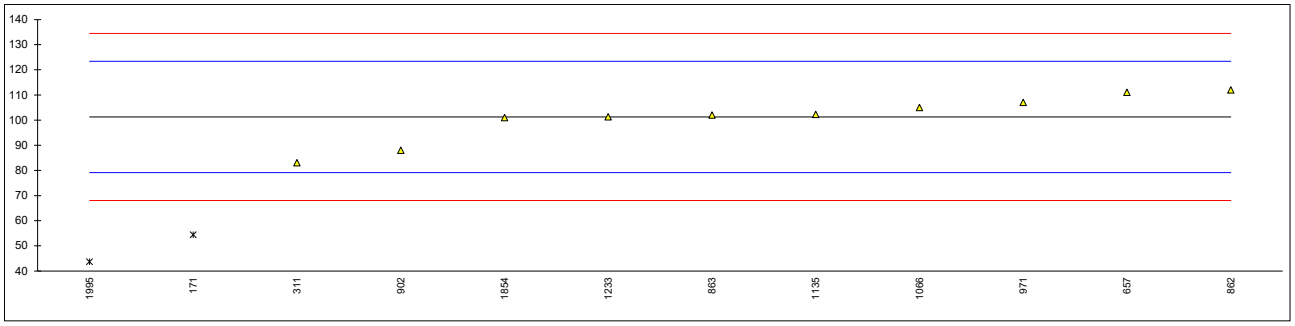
++ : group performed much better than the reference test method
+ : group performed better than the reference test method
+/- : group performance equals the reference test method
- : group performed worse than the reference test method
-- : group performed much worse than the reference test method
n.e. : not evaluated

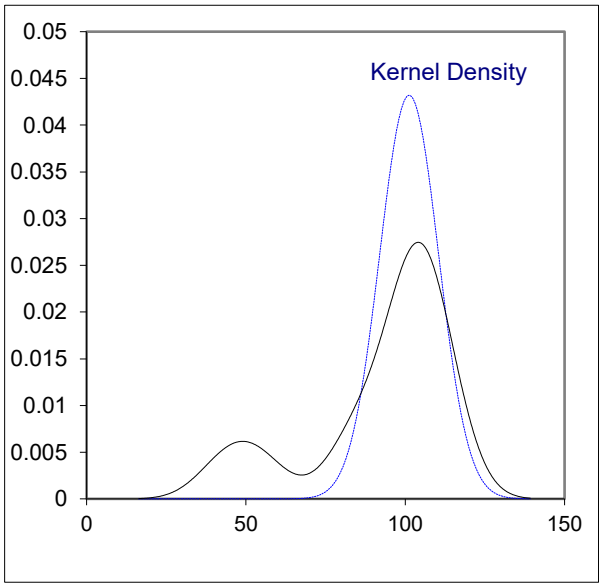
APPENDIX 1

Determination of Styrene on sample #23107; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D7845	54.35	DG(0.01)	-4.24	
311	D7845	83		-1.65	
323	D7845	Co-elution		-----	
657	D7845	111		0.88	
862	D7845	112		0.97	
863	D7845	102		0.07	
902	D7845	88		-1.20	
971	D7845	107	C	0.52	first reported: 167
1066	D7845	105		0.34	
1135	D7845	102.23		0.09	
1233	D7845	101.31		0.01	
1854	D7845	101	C	-0.02	first reported: 364
1995	D7845	43.69	C,DG(0.01)	-5.20	first reported: 30.7124

normality	OK
n	10
outliers	2
mean (n)	101.25
st.dev. (n)	9.237
R(calc.)	25.86
st.dev.(D7845:20)	11.069
R(D7845:20)	30.99

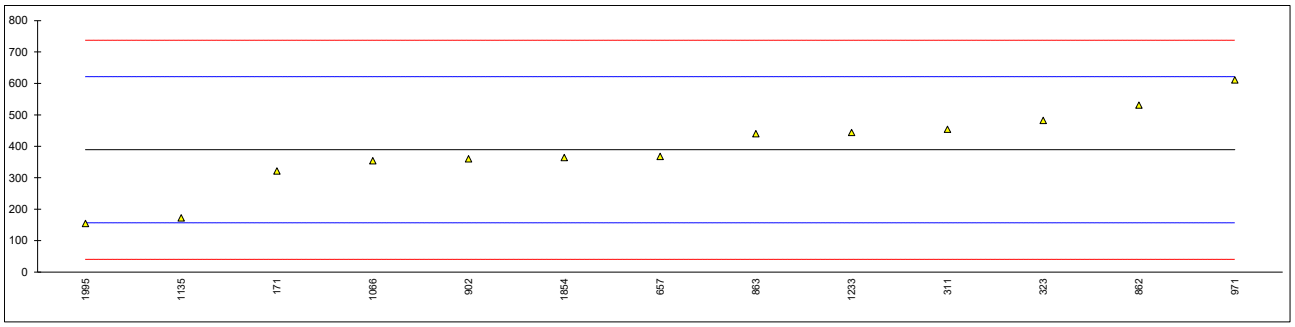


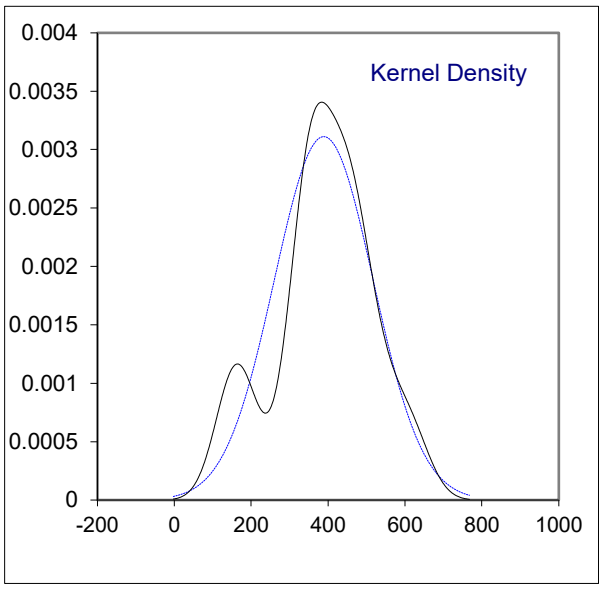


Determination of Phenol on sample #23107; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D7845	321.68		-0.58	
311	D7845	454		0.56	
323	D7845	482		0.80	
657	D7845	368		-0.18	
862	D7845	531		1.22	
863	D7845	440		0.44	
902	D7845	360		-0.25	
971	D7845	611		1.91	
1066	D7845	354		-0.30	
1135	D7845	172.18		-1.87	
1233	D7845	444.028		0.47	
1854	D7845	364		-0.21	
1995	D7845	154.8581		-2.01	

normality	OK
n	13
outliers	0
mean (n)	388.98
st.dev. (n)	128.273
R(calc.)	359.17
st.dev.(D7845:20)	116.210
R(D7845:20)	325.39

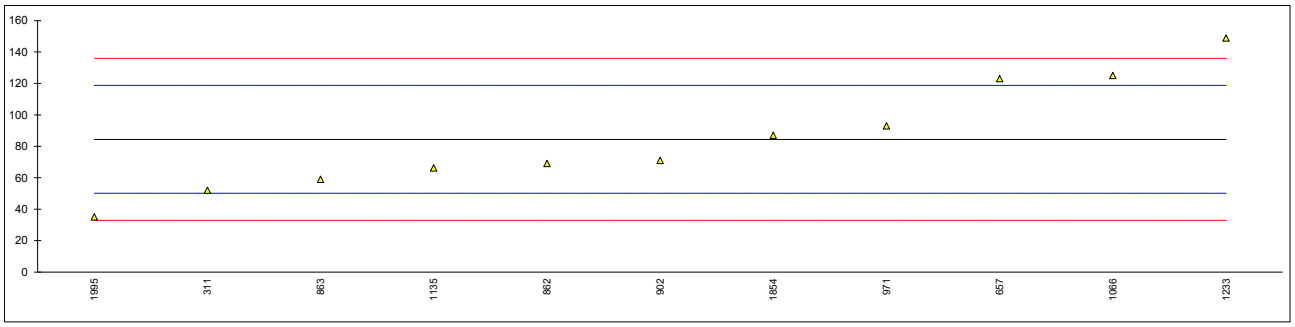


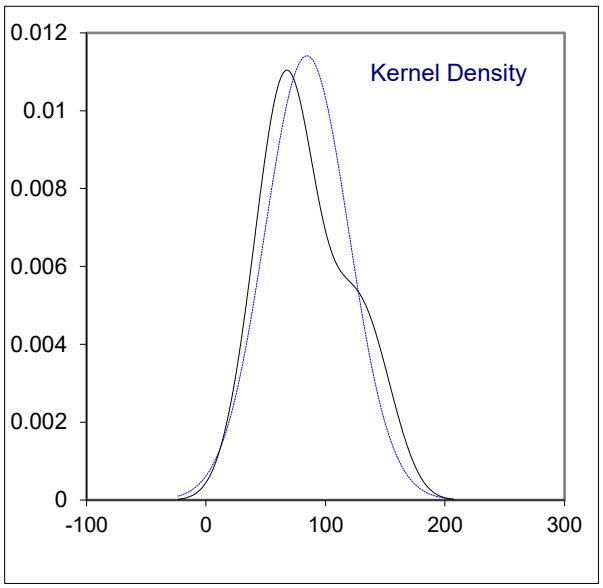


Determination of 4-Methyl styrene on sample #23107; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D7845	<10	f-?	<-4.34	possibly a false negative test result?
311	D7845	52		-1.89	
323	D7845	Co-elution		-----	
657	D7845	123	C	2.25	first reported: <10
862	D7845	69		-0.90	
863	D7845	59		-1.48	
902	D7845	71		-0.79	
971	D7845	93	C	0.50	first reported: 293
1066	D7845	125		2.36	
1135	D7845	66.24		-1.06	
1233	D7845	148.7882		3.75	
1854	D7845	87		0.15	
1995	D7845	35.09	C	-2.88	first reported: 26.5935

normality	OK
n	11
outliers	0
mean (n)	84.47
st.dev. (n)	34.982
R(calc.)	97.95
st.dev.(D7845:20)	17.153
R(D7845:20)	48.03

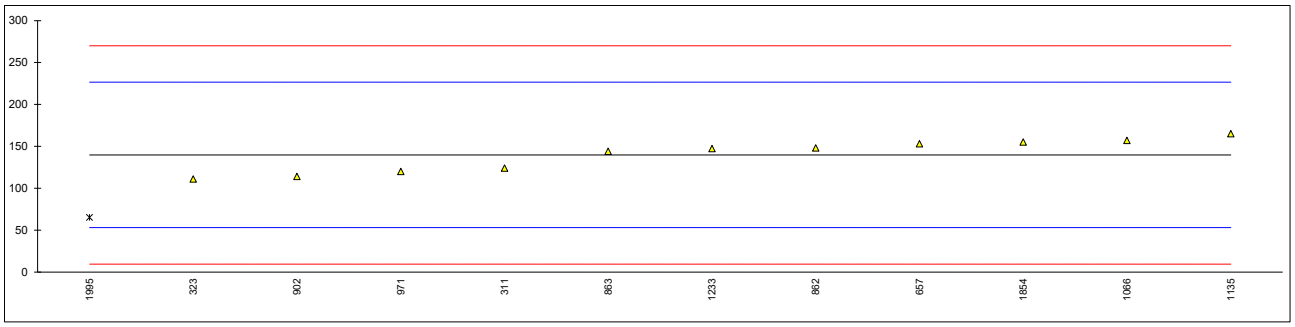


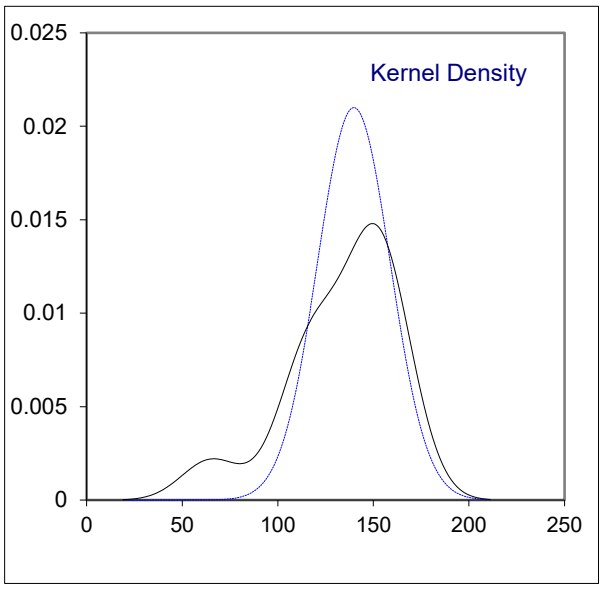


Determination of trans-B-Methyl styrene on sample #23107; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D7845	<10	f-?	<-3.00	possibly a false negative test result?
311	D7845	124		-0.36	
323	D7845	111		-0.66	
657	D7845	153	C	0.30	first reported: <10
862	D7845	148		0.19	
863	D7845	144		0.10	
902	D7845	114		-0.60	
971	D7845	120	C	-0.46	first reported: <10
1066	D7845	157		0.40	
1135	D7845	165	C	0.58	first reported: 341.1
1233	D7845	147.1986		0.17	
1854	D7845	155	C	0.35	first reported: 23
1995	D7845	65.23	C,G(0.05)	-1.72	first reported: 51.6829

normality	OK
n	11
outliers	1
mean (n)	139.84
st.dev. (n)	19.007
R(calc.)	53.22
st.dev.(D7845:20)	43.399
R(D7845:20)	121.52

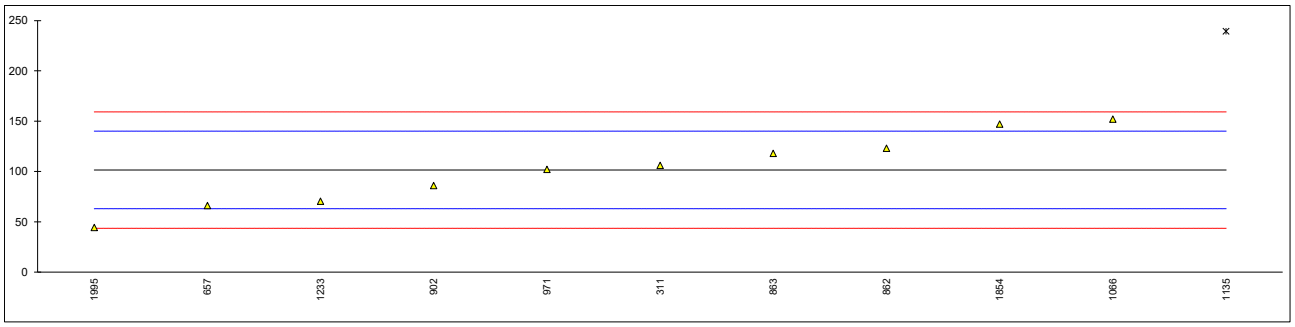


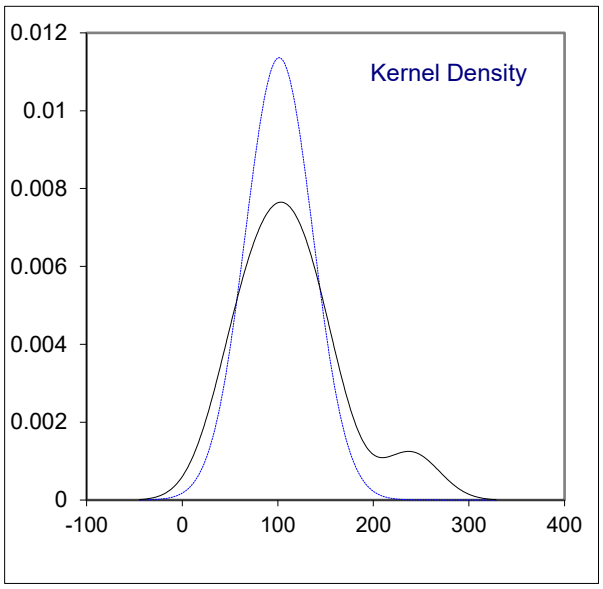


Determination of 3-Methyl styrene on sample #23107; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D7845	<10	f-?	<-4.75	possibly a false negative test result?
311	D7845	106		0.23	
323	D7845	Co-elution		-----	
657	D7845	66	C	-1.84	first reported: 214
862	D7845	123		1.12	
863	D7845	118		0.86	
902	D7845	86		-0.80	
971	D7845	102	C	0.03	first reported: <20
1066	D7845	152		2.62	
1135	D7845	239.28	D(0.05)	7.15	
1233	D7845	70.468		-1.61	
1854	D7845	147		2.36	
1995	D7845	44.33	C	-2.96	first reported: 31.2317

normality	OK
n	10
outliers	1
mean (n)	101.48
st.dev. (n)	35.131
R(calc.)	98.37
st.dev.(D7845:20)	19.279
R(D7845:20)	53.98

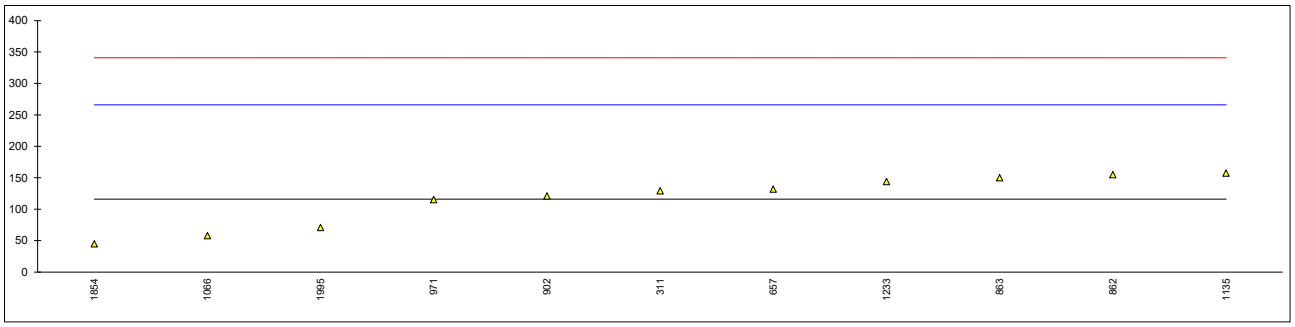


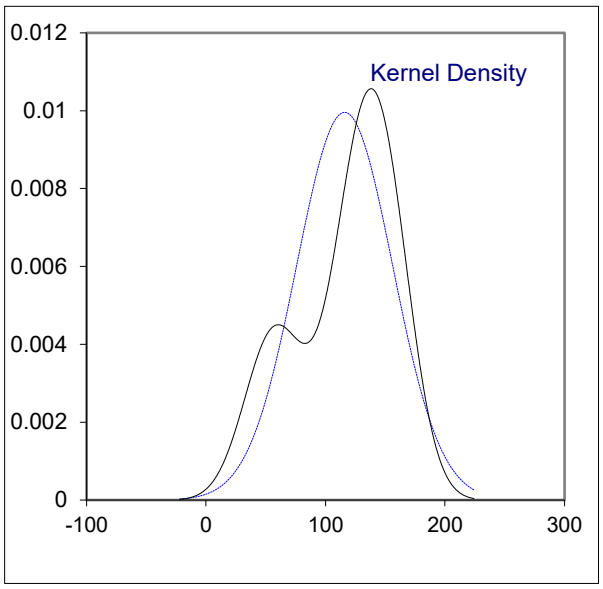


Determination of 2-Methyl styrene on sample #23107; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D7845	<10		-----	
311	D7845	129		0.17	
323	D7845	Co-elution		-----	
657	D7845	132		0.21	
862	D7845	155		0.52	
863	D7845	150		0.45	
902	D7845	121		0.07	
971	D7845	115	C	-0.01	first reported: 1150
1066	D7845	58		-0.78	
1135	D7845	157.39		0.55	
1233	D7845	143.751		0.37	
1854	D7845	45	C	-0.95	first reported: 31
1995	D7845	70.73	C	-0.61	first reported: 21.4631

normality	OK
n	11
outliers	0
mean (n)	116.08
st.dev. (n)	40.082
R(calc.)	112.23
st.dev.(D7845:20)	74.905
R(D7845:20)	209.73

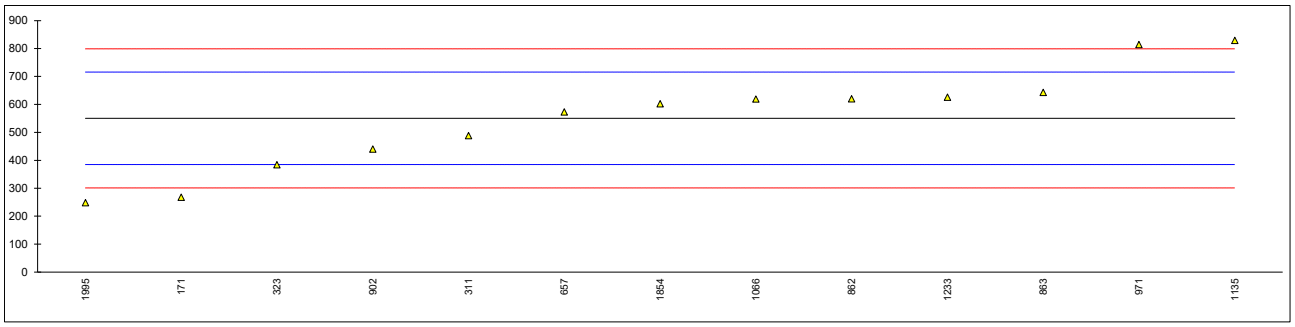


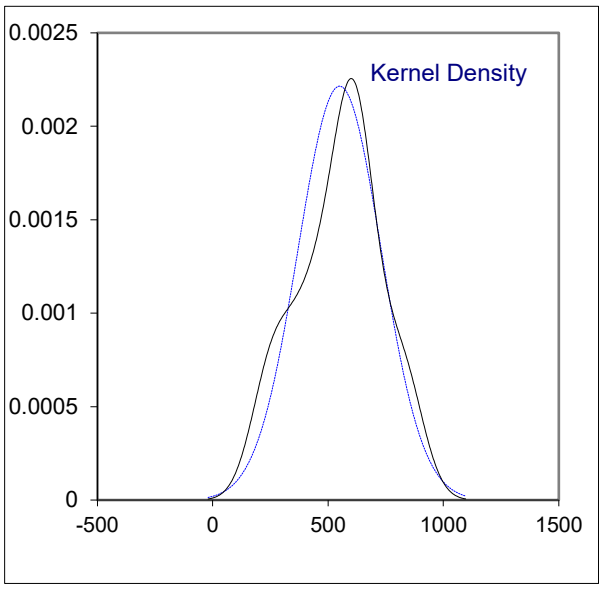


Determination of Indene on sample #23107; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D7845	267.62		-3.41	
311	D7845	488		-0.75	
323	D7845	384		-2.00	
657	D7845	573		0.27	
862	D7845	620		0.84	
863	D7845	643		1.12	
902	D7845	440		-1.33	
971	D7845	814		3.18	
1066	D7845	619		0.83	
1135	D7845	829.21		3.36	
1233	D7845	625.3972		0.91	
1854	D7845	602		0.62	
1995	D7845	248.0	C	-3.65	first reported: 193.6836

normality	OK
n	13
outliers	0
mean (n)	550.25
st.dev. (n)	180.121
R(calc.)	504.34
st.dev.(D7845:20)	82.918
R(D7845:20)	232.17





Determination of 2,5-Dimethyl styrene on sample #23107; results in mg/kg

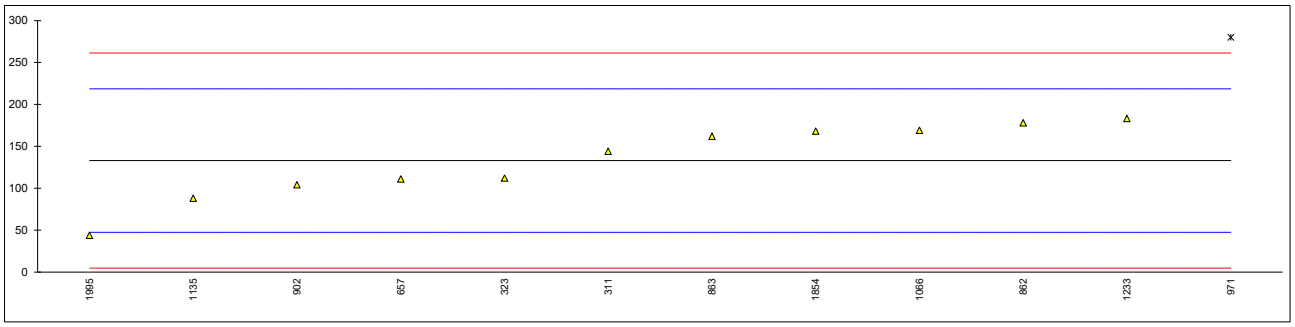
lab	method	value	mark	z(targ)	remarks
171	D7845	<20		-----	
311	D7845	<20		-----	
323	D7845	Co-elution		-----	
657	D7845	<20		-----	
862	D7845	33		-----	
863	D7845	29		-----	
902	D7845	<20	C	-----	first reported: 435
971	D7845	119		-----	
1066	D7845	40		-----	
1135	D7845	<20		-----	
1233	D7845	56.4872		-----	
1854	D7845	<20		-----	
1995	D7845	0.0000		-----	

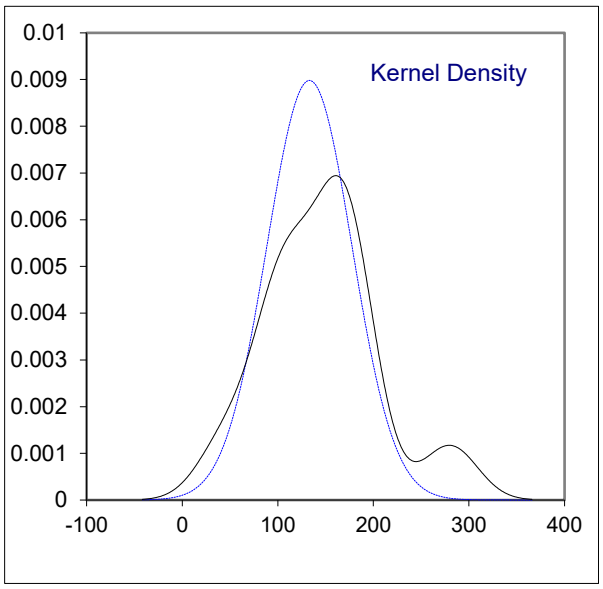
Remark:
No statistical evaluation was performed because the variation is high with a bimodal distribution. Seven participants reported a test result below the detection limit, while five participants reported a test result well above the detection limit. See also paragraph 4.1.

Determination of 2-Ethyl phenol on sample #23107; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D7845	<50		-----	
311	D7845	144		0.26	
323	D7845	112		-0.49	
657	D7845	111		-0.51	
862	D7845	178		1.05	
863	D7845	162		0.68	
902	D7845	104		-0.68	
971	D7845	280	D(0.05)	3.44	
1066	D7845	169		0.84	
1135	D7845	88.15		-1.05	
1233	D7845	183.1544		1.17	
1854	D7845	168		0.82	
1995	D7845	43.9228		-2.08	

normality	OK
n	11
outliers	1
mean (n)	133.02
st.dev. (n)	44.425
R(calc.)	124.39
st.dev.(D7845:20)	42.767
R(D7845:20)	119.75

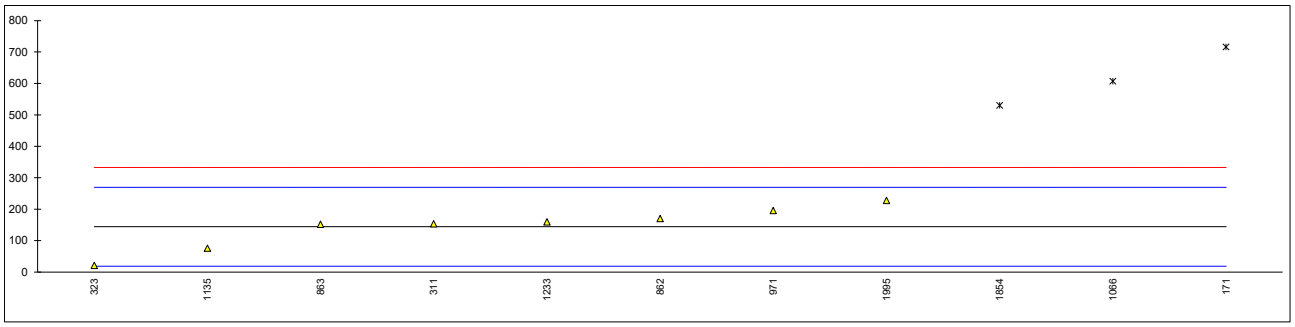


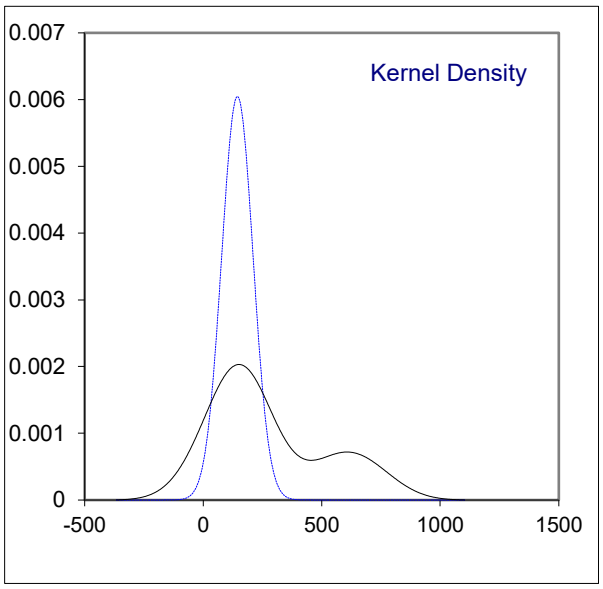


Determination of 2,4-Dimethyl phenol on sample #23107; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D7845	716	C,G(0.05)	9.12	first reported: 0
311	D7845	153		0.14	
323	D7845	21		-1.97	
657	D7845	<20		-----	
862	D7845	170		0.41	
863	D7845	152		0.12	
902	D7845	<20		-----	
971	D7845	196		0.82	
1066	D7845	607	G(0.05)	7.38	
1135	D7845	76	C	-1.09	first reported: 442.20
1233	D7845	159.3748		0.24	
1854	D7845	530	C,G(0.05)	6.15	first reported: 600
1995	D7845	227.61	C	1.33	first reported: 0.0000

normality	OK
n	8
outliers	3
mean (n)	144.37
st.dev. (n)	66.013
R(calc.)	184.84
st.dev.(D7845:20)	62.693
R(D7845:20)	175.54





Determination of 4-Ethyl phenol on sample #23107; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D7845	543.67		-----	
311	D7845	<50		-----	
323	D7845	<20		-----	
657	D7845	<20		-----	
862	D7845	<20		-----	
863	D7845	<20		-----	
902	D7845	<20		-----	
971	D7845	216	C	-----	first reported: 1160
1066	D7845	870		-----	
1135	D7845	513.13		-----	
1233	D7845	81.0908		-----	
1854	D7845	854		-----	remark lab: 4- + 3-Ethylphenol
1995	D7845	Not Detected		-----	

Remark:
No statistical evaluation was performed because the variation is high with a bimodal distribution. Seven participants reported a test result below the detection limit, while five participants reported a test result that is very high compared to the detection limit..
In the method ASTM D7845 the component of 4-Ethyl phenol is requested as part of the sum with 3-Ethyl phenol. See also paragraph 4.1.

Determination of 4-iso-Propylphenol on sample #23107; results in mg/kg

lab	method	value	mark	z(targ)	remarks
171	D7845	<50		-----	
311	D7845	61		-----	
323	D7845	<50		-----	
657	D7845	<50		-----	
862	D7845	87		-----	
863	D7845	74		-----	
902	D7845	<50		-----	
971	D7845	64	C	-----	first reported: 164
1066	D7845	<50		-----	
1135	D7845	<50		-----	
1233	D7845	not detected		-----	
1854	D7845	74		-----	
1995	D7845	98.96	C	-----	first reported: 16.29

Remark:
No statistical evaluation was performed because the variation is high with a bimodal distribution. Seven participants reported a test result below the detection limit, while five participants reported a test result well above the detection limit. See also paragraph 4.1.

APPENDIX 2 Other reported components on sample #23107; results in mg/kg

lab	n-Butyl alcohol	Cyclo hexanol	n-Butyl ether	n-Butyl acrylate	alpha-Pinene	alpha-Methyl styrene	beta-Pinene	Dicyclo pentadiene	Limonene
171	<10	<10	<10	<10	<10	<10	<10	<10	<10
311	<10	<10	<10	<10	<10	<10	<10	<10	<10
323	<10	-----	-----	<10	<10	24	<10	<10	<10
657	<10	<10	<10	<10	<10	<10 C	<10	<10	<10
862	<10	<10	<10	<10	<10	<10	<10	<10	<10
863	<10	<10	<10	<10	<10	<10	<10	<10	<10
902	14	<10	<10	<10	<10	<10	<10	<10	<10
971	<10	<10	<10	<10	<10	<10	<10	<10	32
1066	<10	39	<10	<10	<10	<10	<10	<10	<10
1135	<10	<10	<10	<10	<10	<10	<10	<10	<10
1233	not detected	not detected	not detected	49.4608	not detected	42.563	not detected	not detected	not detected
1854	<10	<10	<10	<10	<10	<10	<10	<10	<10
1995	12.38 C	not detected	3.3422	86.12 C	4.5811	11.5388	6.5186	0.0000	0.0000

lab	1-Phenyl ethanol	para, alpha-Dimethyl styrene	2,4-Dimethyl styrene	2-Phenyl ethanol	2-Phenoxy-1-propanol	2-Phenoxy ethanol	1-Phenoxy-2-Propanol	Styrene glycol
171	<20	<20	<20	<20	<50	<50	<20	<50
311	<20	<20	<20	<20	<50	<50	<20	<20
323	Co-elution	-----	Co-elution	-----	<50	<50	<50	-----
657	<20	<20	<20	<20 C	<50	<50	<50	<20
862	<20	<20	42	<20	<50	<50	<20	<50
863	<20	<20	35	<20	<50	<50	<50	<50
902	<20	28	<20	<20	<50	<50	<20	<50
971	<20	<20	<20	<50	<50	<20	<20	<50
1066	<20	687	40	<20	<50	<50	<50	<50
1135	<20	<20	<20	<20	<50	<50	<20	<50
1233	not detected	218.7164	66.1096	not detected	not detected	not detected	not detected	<50
1854	<20	39	<20	<20	<20	<50	<50	<50
1995	11.5095	26.3841	0.0000	1.7 C	<50	10.4148	14.6325	13.5905

Lab 657 first reported for alpha-Methyl styrene 29 and for 2-Phenyl Ethanol 168.
Lab 1995 first reported for n-Butyl alcohol 61.3046, for n-Butyl acrylate 44.5053 and of 2-Phenyl ethanol 66.9943

APPENDIX 3

Number of participants per country

2 labs in BELGIUM
2 labs in CHINA, People's Republic
1 lab in EGYPT
1 lab in GREECE
2 labs in NETHERLANDS
1 lab in SINGAPORE
1 lab in TURKEY
1 lab in UNITED ARAB EMIRATES
1 lab in UNITED KINGDOM
1 lab in UNITED STATES OF AMERICA

APPENDIX 4

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)/G5	= 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?
SDS	= Safety Data Sheet

Literature

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
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7 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
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12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)