Results of Proficiency Test
Toluene
February 2019

Organised by: Institute for Interlaboratory Studies
Spijkenisse Netherlands

Author: ing. C.M. Nijssen-Wester
Correctors: ing. A.S. Noordman-de Neef & ing. G.A. Oosterlaken-Buijs
Report: iis19C05

May 2019

CONTENTS

1 INTRODUCTION

Since 1999, the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Toluene every year. During the annual proficiency testing program 2018/2019, it was decided to continue the round robins for the analysis of Toluene in accordance with the latest applicable version of the specification for Toluene: ASTM D841.
In this interlaboratory study 36 laboratories in 22 different countries registered for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2019 proficiency test for Toluene 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 tests (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send one sample of one liter of Toluene, labelled #19021. 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/IEC 17043:2010 (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 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

The necessary bulk material of Toluene was purchased from a local chemical supplier. The approximately 60 liter, after homogenisation, was divided over 59 amber glass bottles of 1 liter and labelled #19021. The homogeneity of the subsamples #19021 was checked by determination of Density at 20°C, according to ASTM D4052 on 8 stratified randomly selected samples.

Toluene	Density at 20°C in kg/L
sample #19021-1	0.86680
sample #19021-2	0.86680
sample #19021-3	0.86678
sample #19021-4	0.86682
sample #19021-5	0.86680
sample #19021-6	0.86683
sample #19021-7	0.86683
sample #19021-8	0.86684

Table 1: homogeneity test results of subsamples #19021

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

	Density at 20°C in kg/L
r (observed)	0.00006
reference test method	ISO12185:96
0.3*R (reference test method)	0.00015

Table 2: evaluation of repeatability of subsamples #19021

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

To each of the participating laboratories one 1L bottle of Toluene labelled #19021 was sent on February 6, 2019. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Toluene packed in amber glass bottles was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYSES

The participants were requested to determine on the Toluene sample #19021: Acid Wash Color, Appearance, Color Pt/Co, Copper Corrosion, Density at 20°C, Distillation (IBP, 50% recovered, DP), Purity, Benzene, Nonaromatics and Refractive Index at 25°C.

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 appropriate reference test methods that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendix 1 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 test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5).

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

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

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

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. 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 these 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 analysis results are plotted. The corresponding laboratory numbers are on the X-axis.

The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected 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. The Kernel Density Graph is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 z-scores

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

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. In some cases, a reproducibility based on former iis proficiency tests could be used.

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 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:

	$\lvert z\rvert < 1$	good
$1 <$	$\lvert z\rvert < 2$	satisfactory
$2 <$	$\lvert z\rvert < 3$	questionable
$3 <$	$\lvert z\rvert$	unsatisfactory

4 Evaluation

In this interlaboratory study, no problems were encountered with the dispatch of the samples. Three participants reported the test results after the final reporting date and one other laboratory did not report any test results. Not all laboratories were able to perform all analyses requested.
Finally, in total 284 numerical test results were reported by 35 participants. Observed were 14 outlying results, which is 4.9% of the total of numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

Not all original data sets proved to have a normal Gaussian distribution. These are referred to as “not OK” or “suspect”. The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1 EVALUATION PER TEST

In this section, the reported test results are discussed per test. The test methods, which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the original data. The abbreviations, used in these tables, are listed in appendix 3.

In the iis PT reports, ASTM methods are referred to with a number (e.g. D1218) and an added designation for the year that the method was adopted or revised (e.g. D1218:12). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D1218:12(2016)). In the results tables of appendix 1 only the method number and year of adoption or revision e.g. D1218:12 will be used.

Acid Wash Color: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D848:18.

Appearance: No problems were observed. All participants, but one, agreed about the appearance of the sample, which was bright, clear and free of suspended matter (Pass).

Color Pt/Co: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5386:16 or ASTM D1209:05(2015).

Copper Corrosion: No problems have been observed. All participants agreed on a result of 1a (Pass).

Density at 20°C: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ISO12185:96.

Distillation: This determination may be problematic for a number of laboratories. In total six statistical outliers were observed and one other test result was excluded. However, all calculated reproducibilities after rejection of the suspect data are in agreement with the requirements of ASTM D850-automated:18.
From the reported results of the 50% recovered, it appears that four participants probably did not correct the results for barometric pressure and thermometer inaccuracy as described in ASTM D850 (paragraph 11).

Purity: 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 D7504:18.

Benzene: This determination may be problematic at this low level of 4.4 mg/kg Benzene. Considering that the reproducibility of ASTM D7504:18 is based on a much higher level of Benzene, no z-scores were calculated.

Nonaromatics: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D7504:18.

Refractive Index: 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 D1218:12(2016).

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average result, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from literature reference test methods (in casu ASTM, ISO test methods) are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R (lit.)
Acid Wash Color		29	0.7 (1-)	0.7	2.0
Appearance		30	Pass	n.a.	n.a.
Color Pt/Co		26	4.7	2.3	5.3
Copper Corrosion		24	1a	n.a.	n.a.
Density at 20°C	kg/L	33	0.8668	0.0002	0.0005
Distillation, IBP	°C	27	110.2	0.6	0.6
Distillation, 50% rec.	°C	25	110.6	0.1	0.2
Distillation, DP	°C	28	110.7	0.4	0.5
Purity	%M/M	29	99.945	0.022	0.013
Benzene	mg/kg	21	4.4	2.5	(0.7)*
Nonaromatics	mg/kg	29	415.8	173.5	375.8
Refractive Index at 25°C		22	1.4940	0.0003	0.0005

Table 3: reproducibilities of tests on sample #19021

*) Reproducibility between brackets is based on a much higher level than present in sample #19021

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF FEBRUARY 2019 WITH PREVIOUS PTS

	February 2019	March 2018	March 2017	March 2016	February 2015
Total Number of reporting labs	35	36 *)	67	59	51
Number of results reported	284	267	743	793	729
Number of statistical outliers	14	10	32	19	15
Percentage outliers	4.9%	3.8%	4.3%	2.4%	2.1%

Table 4: comparison with previous proficiency tests
*) from March 2018 the Toluene results are reported separately from Benzene, hence the lower number of reporting laboratories.

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

The performance of the determinations of the proficiency tests was compared against the requirements of the respective reference test methods. The conclusions are given the following table.

	February 2019	March 2018	March 2017	March 2016	February 2015
Acid Wash Color	++	++	+	++	++
Appearance	n.e.	n.e.	n.e.	n.e.	n.e.
Color Pt/Co	++	++	+	++	+
Copper Corrosion	n.e.	n.e.	n.e.	n.e.	n.e.
Density at 20°C	++	++	++	++	++
Distillation	+	+/-	+/-	+	+
Purity	-	+	n.e.	+	+
Benzene	(--)	n.e.	--	+/-	+
Nonaromatics	++	+/-	+	++	+
Refractive Index at 25°C	+	+	+	-	n.e.

Table 5: comparison determinations of sample #19021 against the reference test methods
*) Reproducibility between brackets is based on a much higher level than present in sample

The performance of the determinations against the requirements of the respective reference test methods is listed in the above table. 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

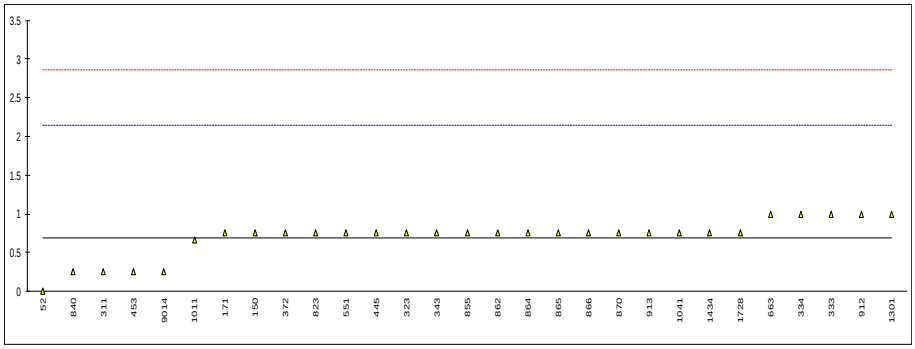
APPENDIX 1

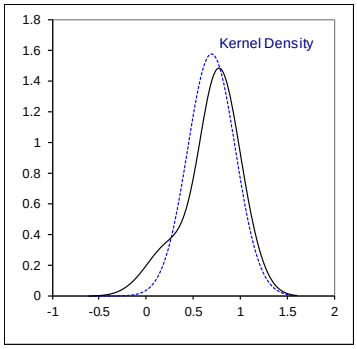
Determination of Acid Wash Color (acid layer) on sample #19021;

lab	method	value	mark	z(targ)	remarks
52	D848	0		-0.96	
150	D848	1-		0.08	
171	D848	1-		0.08	
311	D848	0+		-0.62	
323	D848	-1		0.08	
333	D848	1		0.42	
334	D848	1		0.42	
343	D848	1-		0.08	
372	D848	1-		0.08	
445	D848	1-		0.08	
453	D848	0+		-0.62	
551	D848	1-		0.08	
555		-----		-----	
663	D848	No. 1		0.42	
823	D848	1-		0.08	
840	D848	0+		-0.62	
855	D848	NO1-		0.08	
862	D848	NO.1-		0.08	
864	D848	No.1-		0.08	
865	D848	No.1-		0.08	
866	D848	NO.1-		0.08	
870	D848	No.1-		0.08	
912	D848	1		0.42	
913	D848	1-		0.08	
1011	D848	0.67		-0.04	
1040		-----		-----	
1041	D848	1-		0.08	
1151		-----		-----	
1301	D848	1		0.42	
1434	D848	<1		0.08	
1530		-----		-----	
1728	D848	1-		0.08	
1783		-----		-----	
1812		-----		-----	
6203		-----		-----	
9014	D848	0+		-0.62	

normality	suspect
n	29
outliers	0
mean (n)	0.70 (1-)
st.dev. (n)	0.253
R(calc.)	0.71
st.dev.(D848:18)	0.722
R(D848:18) **)	2.02

*) In the calculation of the mean, standard deviation, reproducibility and for the graphs, a reported value of ‘y-‘, ‘-y’ or ‘<y’ is changed into y-0.25 (for example 1- into 0.75) and ‘y+’ is changed into y+0.25 (for example 0+ into 0.25).
**) The precision data of Benzene is used





Determination of Appearance on sample #19021;

lab	method	value	mark	z(targ)	remarks
52	E2680	Pass		-----	
150	E2680	clear and bright		-----	
171	E2680	Pass		-----	
311	E2680	pass		-----	
323	E2680	clear & bright		-----	
333		-----		-----	
334	EN15769	Clear & Bright		-----	
343	E2680	pass		-----	
372	E2680	Pass		-----	
445	D4176	CFFSM		-----	
453	D4176	Fail/Sediment		-----	
551	Visual	PASS		-----	
555		-----		-----	
663	Visual	Bright & Clear		-----	
823	E2680	Pass		-----	
840	E2680	Pass		-----	
855	E2680	PASS		-----	
862	E2680	Pass		-----	
864	D4176	Pass		-----	
865	E2680	pass		-----	
866	E2680	Pass		-----	
870	E2680	Pass		-----	
912	E2680	PASS		-----	
913	E2680	Pass		-----	
1011	Visual	Bright and Clear		-----	
1040	Visual	Clear and bright		-----	
1041	Visual	CBFSM		-----	
1151		-----		-----	
1301	D4176	Clear and bright		-----	
1434	Visual	Clear Liq		-----	
1530	Visual	c & b		-----	
1728	Visual	CLEAR		-----	
1783	Visual	Clear and Bright		-----	
1812		-----		-----	
6203		-----		-----	
9014	E2680	CLEAR&BRIGHT		-----	
	n	30			
	mean (n)	Pass (B&C)			

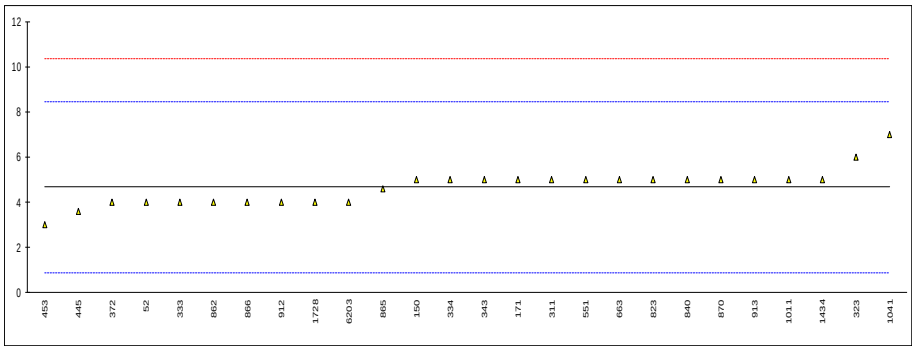
Abbreviations:
C&B / B&C = clear and bright / bright and clear
CFFSM = clear and free from suspended matter
CBFSM = clear and bright and free from suspended matter

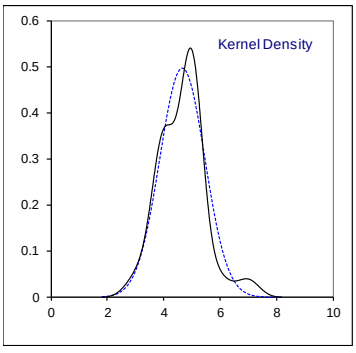
Determination of Color Pt/Co on sample #19021;

lab	method	value	mark	z(targ)	remarks
52	D5386	4		-0.35	
150	D5386	5		0.18	
171	D1209	5		0.18	
311	D1209	5		0.18	
323	D5386	6		0.70	
333	D5386	4		-0.35	
334	D1209	5		0.18	
343	D5386	5		0.18	
372	D5386	4		-0.35	
445	D1209	3.6		-0.56	
453	D1209	3		-0.87	
551	D5386	5		0.18	
555		-----		-----	
663	D5386	5		0.18	
823	D5386	5		0.18	
840	D1209	5		0.18	
855	D5386	<5		-----	
862	D5386	4		-0.35	
864	D1209	<5		-----	
865	D5386	4.6		-0.03	
866	D1209	4		-0.35	
870	D1209	5		0.18	
912	D5386	4		-0.35	
913	D5386	5		0.18	
1011	D1209	5		0.18	
1040	ISO6271	<5		-----	
1041	ISO6271	7.0		1.23	
1151		-----		-----	
1301	D1209	LT 5		-----	
1434	D1209	5		0.18	
1530	D1209	< 3		-----	
1728	D1209	4		-0.35	
1783	D156	>+30		-----	Reported in a different color scale
1812		-----		-----	
6203	D1209	4.0		-0.35	
9014		-----		-----	

normality	suspect
n	26
outliers	0
mean (n)	4.66
st.dev. (n)	0.804
R(calc.)	2.25
st.dev.(D5386:16)	1.900
R(D5386:16)	5.32

Compare: R(D1209:05) = 7





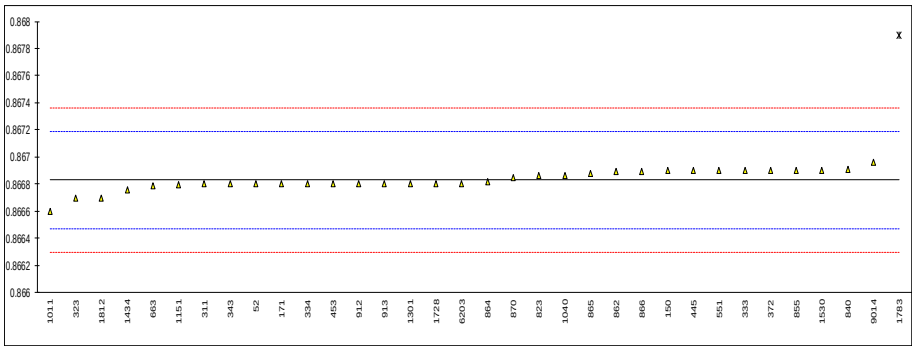
Determination of Copper Corrosion on sample #19021;

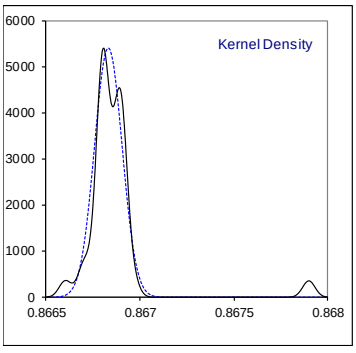
lab	method	value	mark	z(targ)	remarks
52	D849	1a		-----	
150	D849	1a		-----	
171	D849	1a		-----	
311	D849	1a		-----	
323	D849	1a		-----	
333		-----		-----	
334	D849	pass		-----	
343		-----		-----	
372	D849	1a		-----	
445	D849	1a		-----	
453		-----		-----	
551	D130	1A		-----	
555		-----		-----	
663	D849	1a		-----	
823	D849	1a		-----	
840	D849	1A		-----	
855	D849	1A		-----	
862	D849	1a		-----	
864	D849	1a		-----	
865	D849	1a		-----	
866	D849	1a		-----	
870	D849	1a		-----	
912	D849	1A		-----	
913	D849	1a		-----	
1011	D849	1a		-----	
1040		-----		-----	
1041		-----		-----	
1151		-----		-----	
1301	D849	1a		-----	
1434	D849	1a		-----	
1530		-----		-----	
1728	D849	1A		-----	
1783		-----		-----	
1812		-----		-----	
6203		-----		-----	
9014		-----		-----	
	n	24			
	mean (n)	1a (Pass)			

Determination of Density at 20°C on sample #19021: results in kg/L

lab	method	value	mark	z(targ)	remarks
52	D4052	0.8668		-0.16	
150	D4052	0.8669		0.40	
171	D4052	0.8668		-0.16	
311	D4052	0.8668		-0.16	
323	ISO12185	0.8667		-0.72	
333	ISO12185	0.8669		0.40	
334	ISO12185	0.8668		-0.16	
343	ISO12185	0.8668		-0.16	
372	D4052	0.8669		0.40	
445	D4052	0.8669		0.40	
453	ISO12185	0.8668		-0.16	
551	D4052	0.8669		0.40	
555		-----		-----	
663	D4052	0.86679		-0.22	
823	ISO12185	0.86686		0.17	
840	D4052	0.86691		0.45	
855	ISO12185	0.8669		0.40	
862	D4052	0.86689		0.34	
864	D4052	0.86682		-0.05	
865	D4052	0.86688		0.28	
866	D4052	0.86689		0.34	
870	D4052	0.86685		0.12	
912	D4052	0.8668		-0.16	
913	D4052	0.8668		-0.16	
1011	D4052	0.8666		-1.28	
1040	ISO12185	0.86686		0.17	
1041		-----		-----	
1151	D4052	0.8667975		-0.18	
1301	D4052	0.8668		-0.16	
1434	D4052	0.86676		-0.39	
1530	ISO12185	0.86690		0.40	
1728	ISO12185	0.86680		-0.16	
1783	D4052	0.8679	R(0.01)	6.00	
1812	ISO12185	0.8667		-0.72	
6203	ISO12185	0.8668		-0.16	
9014	D4052	0.86696		0.73	

normality	suspect
n	33
outliers	1
mean (n)	0.86683
st.dev. (n)	0.000074
R(calc.)	0.00021
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005



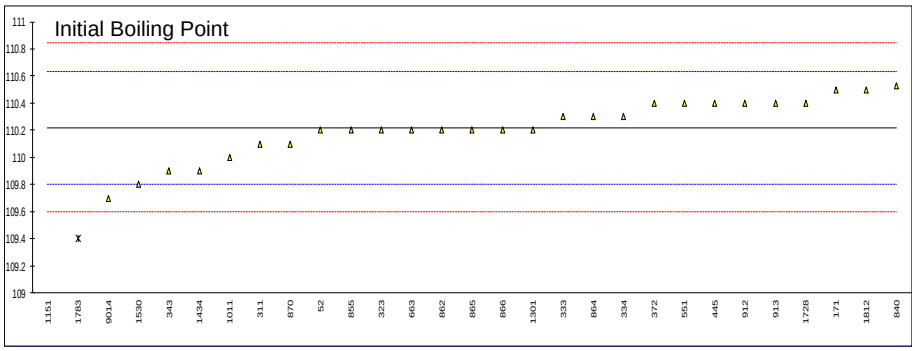


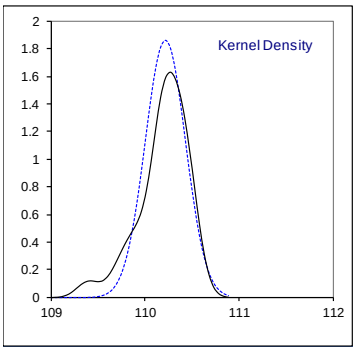
Determination of Distillation on sample #19021; results in °C

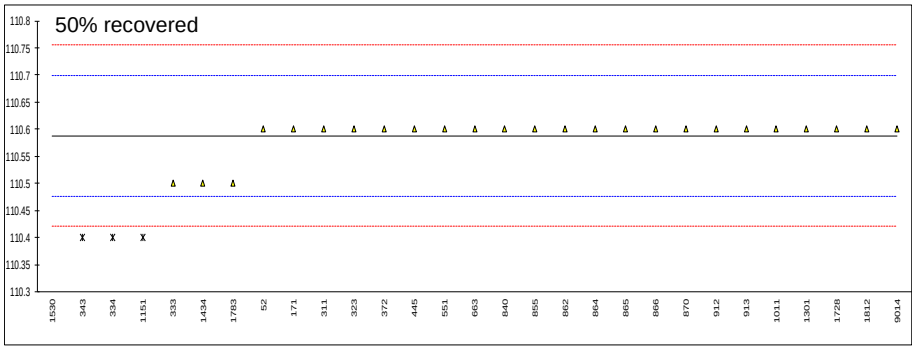
lab	method	IBP	mark	z(targ)	50% rec	mark	z(targ)	DP	mark	z(targ)	range
52	D850-automated	110.2		-0.09	110.6		0.22	110.7		-0.20	0.5
150	D850-automated	-----		-----	-----		-----	-----		-----	-----
171	D850-automated	110.5		1.35	110.6		0.22	110.7		-0.20	0.2
311	D850-automated	110.1		-0.58	110.6		0.22	110.7		-0.20	0.6
323	D850-manual	110.2		-0.09	110.6		0.22	110.7		-0.20	0.5
333	D850-automated	110.3		0.39	110.5		-1.58	110.7		-0.20	0.4
334	D850-automated	110.3		0.39	110.4	R(0.01)	-3.37	110.5		-1.43	< 145
343	D850-automated	109.9		-1.54	110.4	R(0.01)	-3.37	110.4		-2.04	0.5
372	D850-automated	110.4		0.87	110.6		0.22	110.8		0.41	0.4
445	D850-manual	110.4		0.87	110.6		0.22	110.8		0.41	0.4
453		-----		-----	-----		-----	-----		-----	-----
551	D850	110.4		0.87	110.6		0.22	110.8		0.41	0.4
555		-----		-----	-----		-----	-----		-----	-----
663	D850-automated	110.2		-0.09	110.6		0.22	110.6		-0.81	0.4
823		-----		-----	-----		-----	-----		-----	-----
840	D850-automated	110.53		1.50	110.60		0.22	110.61		-0.75	0.1
855	D850-manual	110.2		-0.09	110.6		0.22	110.8		0.41	0.6
862	D850-manual	110.2		-0.09	110.6		0.22	110.8		0.41	0.6
864	D850-automated	110.3		0.39	110.6		0.22	110.8		0.41	0.5
865	D850-manual	110.2		-0.09	110.6		0.22	110.8		0.41	0.6
866	D850-manual	110.2		-0.09	110.6		0.22	110.8		0.41	0.6
870	D850-manual	110.1		-0.58	110.6		0.22	110.8		0.41	0.7
912		110.4		0.87	110.6		0.22	110.8		0.41	0.4
913	D850-manual	110.4		0.87	110.6		0.22	111.0		1.64	0.6
1011	D850-automated	110.0		-1.06	110.6		0.22	110.8		0.41	-----
1040		-----		-----	-----		-----	-----		-----	-----
1041		-----		-----	-----		-----	-----		-----	-----
1151		98.15	R(0.01)	-58.17	110.4	R(0.01)	-3.37	110.55	ex	-1.12	0.2
1301	D850-manual	110.2		-0.09	110.6		0.22	111.0		1.64	0.8
1434	D850-automated	109.9		-1.54	110.5		-1.58	110.7		-0.20	0.8
1530		109.8		-2.02	109.9	R(0.01)	-12.35	110.6		-0.81	0.8
1728	D850-manual	110.4		0.87	110.6		0.22	110.8		0.41	0.4
1783	D1078	109.4	R(0.05)	-3.95	110.5		-1.58	110.6		-0.81	-----
1812		110.50		1.35	110.60		0.22	110.70		-0.20	0.2
6203		-----		-----	-----		-----	-----		-----	-----
9014	D850-automated	109.7		-2.50	110.6		0.22	110.7		-0.20	1.0
	normality	OK			not OK			suspect			
	n	27			25			28			
	outliers	2			4			0(+1ex)			
	mean (n)	110.22			110.59			110.73			
	st.dev. (n)	0.215			0.033			0.127			
	R(calc.)	0.60			0.09			0.36			
	st.dev.(D850-A:18)	0.208			0.056			0.163			
	R(D850-A:18)	0.58			0.16			0.46			
	compare										
	R(D850-M:18)	0.41			0.65			0.65			

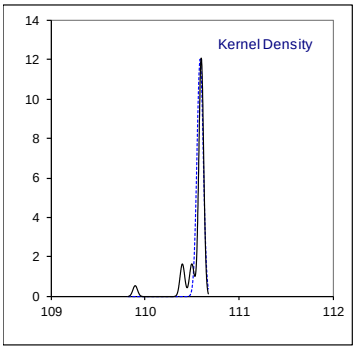
Lab 1151: two out of three results were outliers, therefore the other test result is excluded.

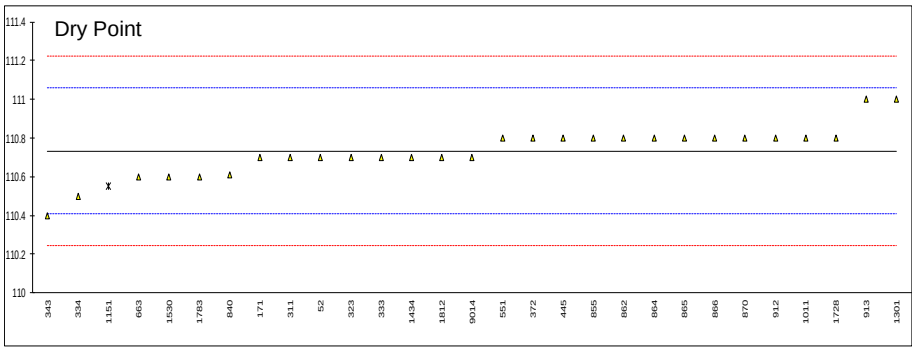
Theoretical mid-boiling point = 110.6°C

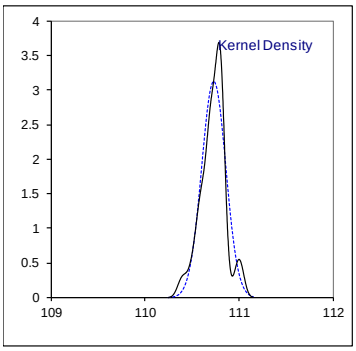

Initial Boiling Point
111
110.8
110.6
110.4
110.2
110
109.8
109.6
109.4
109.2
109
1151 1783 9014 1530 343 1434 1011 311 870 52 855 323 663 862 865 866 1301 333 864 334 372 551 445 912 913 1728 171 1812 840


Kernel Density
2
1.8
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
109 110 111 112


50% recovered
110.8
110.75
110.7
110.65
110.6
110.55
110.5
110.45
110.4
110.35
110.3
1530 343 334 1151 333 1434 1783 52 171 311 323 372 445 551 663 840 855 862 864 865 866 870 912 913 1011 1301 1728 1812 9014


Kernel Density
14
12
10
8
6
4
2
0
109 110 111 112

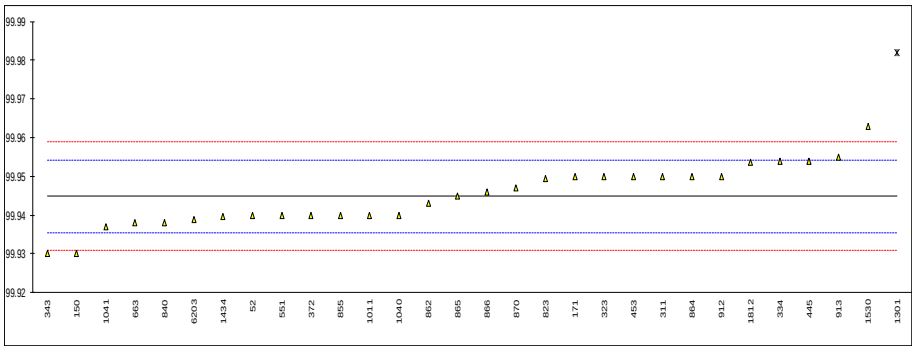

Dry Point
111.4
111.2
111
110.8
110.6
110.4
110.2
110
343 334 1151 663 1530 1783 840 171 311 52 323 333 1434 1812 9014 551 372 445 855 862 864 865 866 870 912 1011 1728 913 1301

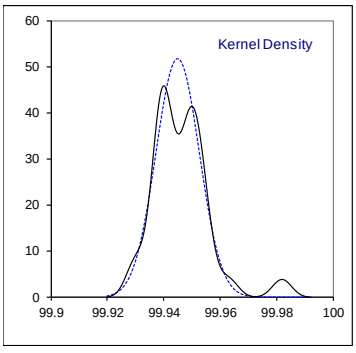

Kernel Density
4
3.5
3
2.5
2
1.5
1
0.5
0
109 110 111 112

Determination of Purity by GC on sample #19021; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	D7504	99.94		-1.04	
150	D7504	99.93		-3.18	
171	D7504	99.95		1.09	
311	D7504	99.95		1.09	
323	D7504	99.95		1.09	
333		-----		-----	
334	D2360	99.954		1.95	
343	D2360	99.93		-3.18	
372	D7504	99.94		-1.04	
445	D6526	99.954		1.95	
453	D2360	99.95		1.09	
551	D2360	99.94		-1.04	
555		-----		-----	
663	D7504	99.938		-1.47	
823	D2360	99.9495		0.99	
840	D7504	99.938		-1.47	
855	D7504	99.94		-1.04	
862	D7504	99.943		-0.40	
864	D7504	99.95		1.09	
865	D7504	99.945		0.03	
866	D7504	99.946		0.24	
870	D7504	99.947		0.45	
912	D2360	99.95		1.09	
913	D7504	99.955		2.16	
1011	D5917	99.94		-1.04	
1040	D6526	99.9400		-1.04	
1041	In house	99.9371		-1.66	
1151		-----		-----	
1301	D7504	99.982	C,R(0.01)	7.93	first reported: 99.989
1434	D4492	99.93963		-1.12	
1530	D6526	99.963		3.87	
1728		-----		-----	
1783		-----		-----	
1812		99.9537		1.89	
6203	D7504	99.9387		-1.32	
9014		-----		-----	

normality	OK
n	29
outliers	1
mean (n)	99.9449
st.dev. (n)	0.00772
R(calc.)	0.0216
st.dev.(D7504:18)	0.00468
R(D7504:18)	0.0131



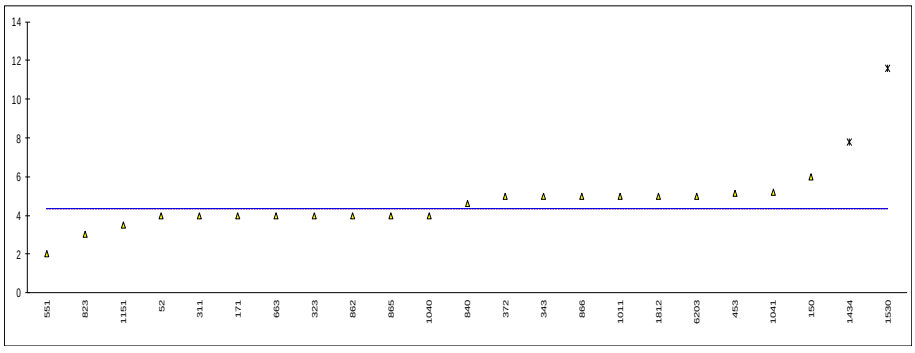


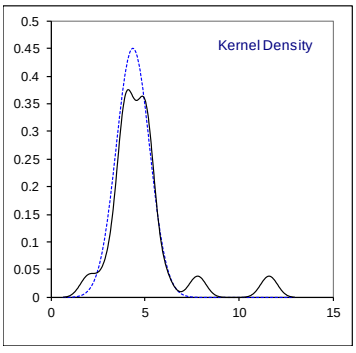
Determination of Benzene on sample #19021; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	D7504	4		-----	
150	D7504	6		-----	
171	D7504	4		-----	
311	D7504	4		-----	
323	D7504	4		-----	
333		-----		-----	
334	D2360	<10	C	-----	first reported: <0.001 mg/kg
343	D2360	5		-----	
372	D7504	5		-----	
445	D6526	<5	C	-----	first reported: <0.0005 mg/kg
453	D2360	5.166		-----	
551	D2360	2		-----	
555		-----		-----	
663	D7504	4.0		-----	
823	D2360	3		-----	
840	D7504	4.6		-----	
855	D7504	<10		-----	
862	D7504	4		-----	
864	D7504	<10		-----	
865	D7504	4		-----	
866	D7504	5		-----	
870	D7504	<10		-----	
912	D2360	<10		-----	
913	D7504	<5	C	-----	first reported: 20
1011	D5917	5		-----	
1040	D6526	4	C	-----	first reported: 0.0004 mg/kg
1041	In house	5.2		-----	
1151	In house	3.52		-----	
1301	D7504	<1		-----	
1434	D4492	7.8	C,G(0.05)	-----	first reported: 0.00078 mg/kg
1530	D6526	11.6	G(0.01)	-----	
1728		-----		-----	
1783		-----		-----	
1812		5		-----	
6203	D7504	5		-----	
9014		-----		-----	

normality	suspect
n	21
outliers	2
mean (n)	4.36
st.dev. (n)	0.887
R(calc.)	2.48
st.dev.(D7504:18)	(0.238)
R(D7504:18)	(0.67)

Reproducibility in ASTM D7508:18 is based on a Benzene level much higher than present in sample #19021, see also §4.1.

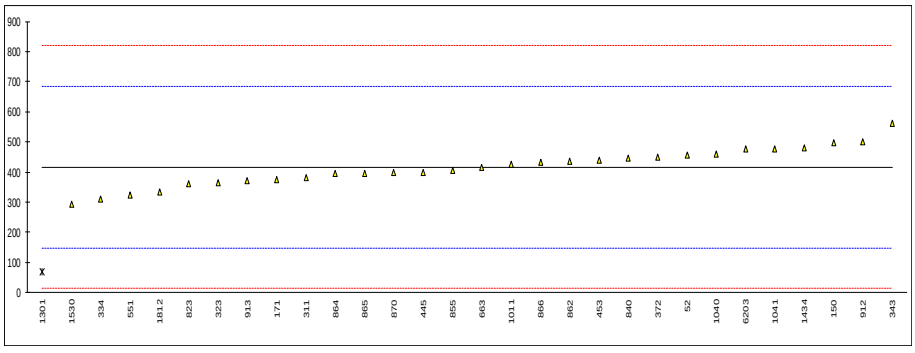


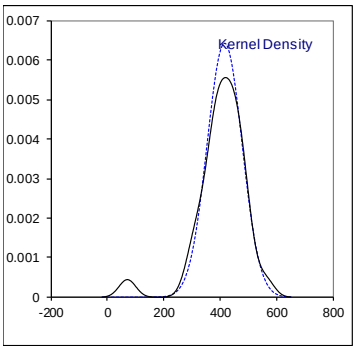


Determination of Nonaromatics on sample #19021; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	D7504	455		0.29	
150	D7504	498		0.61	
171	D7504	375		-0.30	
311	D7504	380		-0.27	
323	D7504	363		-0.39	
333		-----		-----	
334	D2360	310	C	-0.79	first reported: 0.031 mg/kg
343	D2360	561.8		1.09	
372	D7504	450		0.25	
445	D6526	400	C	-0.12	first reported: 0.04 mg/kg
453	D2360	439.597		0.18	
551	D2360	324		-0.68	
555		-----		-----	
663	D7504	416		0.00	
823	D2360	362		-0.40	
840	D7504	446.8		0.23	
855	D7504	405		-0.08	
862	D7504	436		0.15	
864	D7504	395		-0.15	
865	D7504	396		-0.15	
866	D7504	433		0.13	
870	D7504	398		-0.13	
912	D2360	500		0.63	
913	D7504	370		-0.34	
1011	D5917	424		0.06	
1040	D6526	460	C	0.33	first reported: 0.046 mg/kg
1041	In house	477		0.46	
1151		-----		-----	
1301	D7504	70	C,R(0.01)	-2.58	first reported <1
1434	D4492	480.4	C	0.48	first reported: 0.04804 mg/kg
1530	D6526	291.6		-0.93	
1728		-----		-----	
1783		-----		-----	
1812		335		-0.60	
6203	D7504	476		0.45	
9014		-----		-----	

normality	OK
n	29
outliers	1
mean (n)	415.80
st.dev. (n)	61.954
R(calc.)	173.47
st.dev.(D7504:18)	134.221
R(D7504:18)	375.82

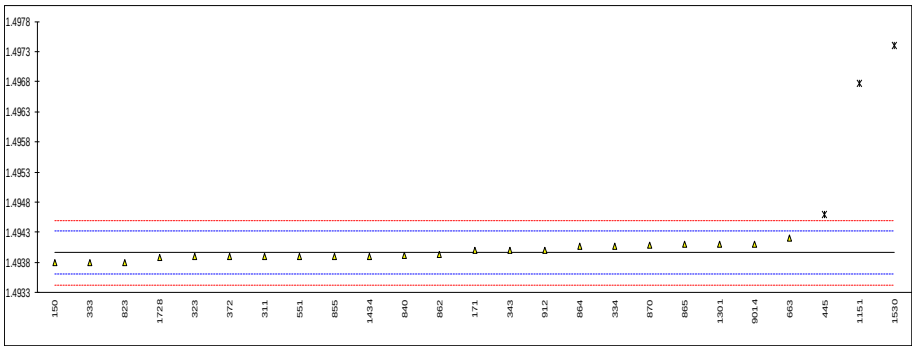


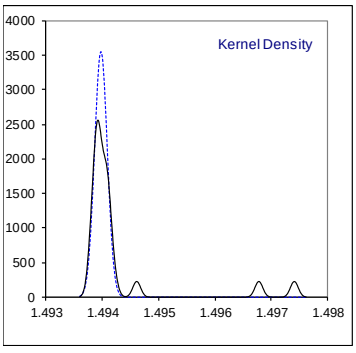


Determination of Refractive Index at 25°C on sample #19021;

lab	method	value	mark	z(targ)	remarks
52		-----		-----	
150	D1218	1.4938		-0.92	
171	D1218	1.4940		0.20	
311	D1218	1.4939		-0.36	
323	D1218	1.49389		-0.42	
333	D1218	1.4938		-0.92	
334	D1218	1.49407		0.59	
343	D1218	1.4940		0.20	
372	D1218	1.4939		-0.36	
445	D1218	1.4946	R(0.01)	3.56	
453		-----		-----	
551	D1218	1.4939		-0.36	
555		-----		-----	
663	D1218	1.4942		1.32	
823	D1218	1.49380		-0.92	
840	D1218	1.49392		-0.25	
855	D1218	1.4939		-0.36	
862	D1218	1.49393		-0.20	
864	D1218	1.49406		0.53	
865	D1218	1.4941		0.76	
866		-----		-----	
870	D1218	1.49408		0.64	
912	D1218	1.4940		0.20	
913		-----		-----	
1011		-----		-----	
1040		-----		-----	
1041		-----		-----	
1151	D1218	1.49677	R(0.01)	15.71	
1301	D1218	1.4941		0.76	
1434	D1218	1.4939	C	-0.36	first reported: 1.4967 (at 15°C)
1530	D1218	1.4974	R(0.01)	19.24	
1728	D1218	1.49388		-0.48	
1783		-----		-----	
1812		-----		-----	
6203		-----		-----	
9014	D1218	1.4941		0.76	

normality	OK
n	22
outliers	3
mean (n)	1.49397
st.dev. (n)	0.000112
R(calc.)	0.00031
st.dev.(D1218:12)	0.000179
R(D1218:12)	0.0005





APPENDIX 2

Number of participants

1 lab in AUSTRALIA
1 lab in BELGIUM
2 labs in BRAZIL
1 lab in CANADA
6 labs in CHINA, People's Republic
1 lab in ESTONIA
2 labs in FRANCE
4 labs in GERMANY
2 labs in INDIA
1 lab in ISRAEL
1 lab in NETHERLANDS
1 lab in PORTUGAL
2 labs in ROMANIA
1 lab in SAUDI ARABIA
1 lab in SOUTH KOREA
1 lab in SPAIN
1 lab in THAILAND
1 lab in TURKEY
1 lab in UNITED ARAB EMIRATES
2 labs in UNITED KINGDOM
2 labs in UNITED STATES OF AMERICA
1 lab in VIETNAM

APPENDIX 3

Abbreviations:

C	= final test result after checking of first reported suspect 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	= possibly an error in calculations
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
SDS	= Safety Data Sheet

Literature:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2 W. Horwitz and R. Albert, J. AOAC Int., 79, 3, 589, (1996)
3 ASTM E178:02
4 ASTM E1301:03
5 ISO13528:05
6 ISO 5725:86
7 ISO 5725, parts 1-6, 1994
8 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
9 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
10 IP 367:84
11 DIN 38402 T41/42
12 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
13 J.N. Miller, Analyst, 118, 455, (1993)
14 Analytical Methods Committee Technical brief, No 4, January 2001.
15 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry 2002, 127, 1359-1364 (2002)
16 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)