Results of Proficiency Test
Toluene
March 2018

Organised by: Institute for Interlaboratory Studies (iis)
Spijkenisse Netherlands

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1 INTRODUCTION

Since 1999, the Institute for Interlaboratory Studies (iis) organizes proficiency tests for the analysis of Toluene every year. During the annual proficiency testing program 2017/2018, 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 the interlaboratory study 38 laboratories in 21 different countries did register for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2018 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 analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send one sample of Toluene (1 litre bottle, labelled #18021). 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 organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

The necessary bulk material of Toluene was purchased from a local chemical supplier. The approximately 60 litre, after homogenisation, was divided over 58 brown glass bottles of 1 litre and labelled #18021. The homogeneity of the subsamples #18021 was checked by determination of Density at 20°C, according to ISO12185 on 8 stratified randomly selected samples.

Toluene	Density at 20°C in kg/L
sample #18021-1	0.86686
sample #18021-2	0.86686
sample #18021-3	0.86686
sample #18021-4	0.86687
sample #18021-5	0.86686
sample #18021-6	0.86687
sample #18021-7	0.86687
sample #18021-8	0.86687

Table 1: homogeneity test results of Toluene subsamples #18021

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)	0.00001
Reference test method	ISO12185:96
0.3*R (reference test method)	0.00015

Table 2: evaluation of repeatability of subsamples #18021

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 litre bottle of Toluene labelled #18021 was sent on February 14, 2018. 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 #18021: 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 calculations.

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

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. 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 reanalysis). 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 organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4). For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.

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

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. When the uncertainty passed the evaluation no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

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 standard. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. The Kernel Density Graph is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM 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:

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

4 EVALUATION

In this interlaboratory study, no problems were encountered with dispatch of the samples. Four participants reported the test results after the final reporting date and two other laboratories did not report any test results. Not all laboratories were able to perform all analyses requested.
Finally, in total 267 numerical test results were reported by 36 participants. Observed were 10 outlying results, which is 3.7% 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.

Unfortunately, a suitable standard test method, providing the precision data, is not available for all determinations. For the tests, that have no available precision data, the calculated reproducibility was compared against the reproducibility estimated from the Horwitz equation.

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 D848:14.
For the statistical analysis, a result expressed as y- or y+ was changed into a numerical value as follows: y- changed into y-0.25 and y+ into y+0.25.

Appearance: No analytical problems were observed. All labs 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. One statistical outlier was observed. However, the calculated reproducibility after rejection of the

statistical outlier is in agreement with the requirements of ASTM D5386:16 and of ASTM D1209:05e1(2011).

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

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 2 test results were excluded. However, all calculated reproducibilities after rejection of the suspect data are in agreement with the requirements of ASTM D850-Automated:16.
From the reported results of the 50% recovered, it appears that three participant probably did not correct the results for barometric pressure and thermometer inaccuracy as described in ASTM D850 (paragraph 11).

Purity: 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 D7504:18.

Benzene: This determination may be problematic at this low level of 4.1 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 outlier is in agreement with the requirements of ASTM D7504:18.

Refractive Index: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility 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 average results per sample, calculated reproducibilities and reproducibilities derived from literature reference test methods (in casu ASTM, ISO standards) are compared in the next tables.

Parameter	unit	n	average	2.8 *sd	R (lit.)
Acid Wash Color		30	0.7 (1-)	0.8	2.0
Appearance		31	Pass	n.a.	n.a.
Color Pt/Co		22	3.7	2.7	5.2
Copper Corrosion		24	1a	n.a.	n.a.
Density at 20°C	kg/L	32	0.8669	0.0002	0.0005
Distillation, IBP	°C	24	110.2	0.5	0.6
Distillation, 50% rec.	°C	22	110.6	0.1	0.2
Distillation, DP	°C	24	110.7	0.4	0.5
Purity	%M/M	31	99.984	0.011	0.013
Benzene	mg/kg	20	4.1	4.4	(0.6)*
Nonaromatics	mg/kg	28	80.4	76.8	72.6
Refractive Index at 25°C		22	1.4940	0.0004	0.0005

Table 3: reproducibilities on sample #18021

*) Reproducibility between brackets is based on a much higher level than present in sample #18021

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 MARCH 2018 WITH PREVIOUS PTS

	March 2018	March 2017	March 2016	Feb 2015	Feb 2014
Total Number of reporting labs	36 *)	67	59	51	58
Number of results reported	267	743	793	729	800
Statistical outliers	10	32	19	15	36
Percentage outliers	3.8%	4.3%	2.4%	2.1%	4.5%

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:

	March 2018	March 2017	March 2016	Feb 2015	Feb 2014
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.	n.e.

Table 5: comparison determinations on Toluene against the reference test methods

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

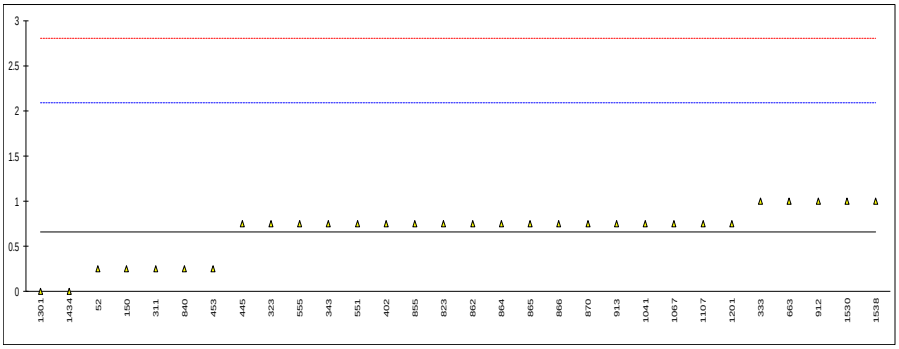
APPENDIX 1

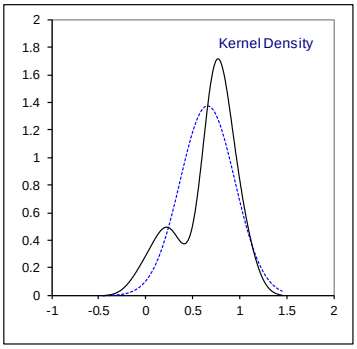
Determination of Acid Wash Color (acid layer) on sample #18021

lab	method	value	mark	z(targ)	remarks
52	D848	0+		-0.57	
150	D848	0+		-0.57	
171	D848	pass		-----	
311	D848	0+		-0.57	
323	D848	-1		0.13	
333	D848	1		0.48	
334		-----		-----	
343	D848	1-		0.13	
402	D848	1-		0.13	
445	D848	1-		0.13	
453	D848	0+		-0.57	
551	D848	1 -		0.13	
555	D848	1-		0.13	
663	D848	No. 1		0.48	
823	D848	1-		0.13	
840	D848	0+		-0.57	
855	D848	No.1-		0.13	
862	D848	NO.1-		0.13	
864	D848	No.1-		0.13	
865	D848	No.1-		0.13	
866	D848	NO.1-		0.13	
870	D848	No.1-		0.13	
912	D848	1		0.48	
913	D848	Less than 1.0		0.13	
1040		-----		-----	
1041	D848	1-		0.13	
1067	D848	1-		0.13	
1107	D848	1-		0.13	
1151		-----		-----	
1201	D848	1-		0.13	
1301	D848	Zero		-0.92	
1320		-----		-----	
1434	D848	0		-0.92	
1530	D848	1		0.48	
1538	D848	1		0.48	
1749		-----		-----	
1790		-----		-----	
6181		-----		-----	

normality	OK
n	30
outliers	0
mean (n)	0.66 (1-)
st.dev. (n)	0.290
R(calc.)	0.81
st.dev.(D848:14)	0.716
R(D848:14)	2.00

*) In the calculation of the mean, standard deviation, reproducibility and in 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).





Determination of Appearance on sample #18021

lab	method	value	mark	z(targ)	remarks
52	D4176	Pass		-----	
150	E2680	Pass		-----	
171	E2680	pass		-----	
311	INH-402	clear & free		-----	
323	E2680	pass		-----	
333		-----		-----	
334		-----		-----	
343	E2680	PASS		-----	
402	Visual	pass		-----	
445	D4176	C & B		-----	
453	Visual	Clear&Bright		-----	
551	D4176	Pass		-----	
555	Visual	Clear and free from impurities		-----	
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	CFSM		-----	
1040	Visual	clear, bright		-----	
1041	Visual	CBFSM		-----	
1067	E2680	Clear and Bright		-----	
1107	E2680	passes		-----	
1151		-----		-----	
1201	D4176	Br&Cl		-----	
1301	Visual	Clear and Bright		-----	
1320		-----		-----	
1434	Visual	clear liq		-----	
1530	D4176	pass		-----	
1538	Visual	C&B		-----	
1749		-----		-----	
1790		-----		-----	
6181		-----		-----	
	n	31			
	mean (n)	Pass (B&C)			

Abbreviations:

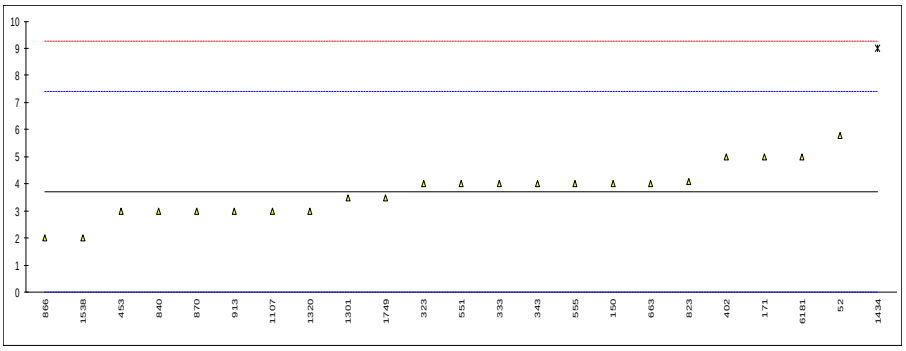
C&B / B&C = clear and bright / bright and clear
CFSM = clear and free from suspended matter
CBFSM = clear& bright and free from suspended matter

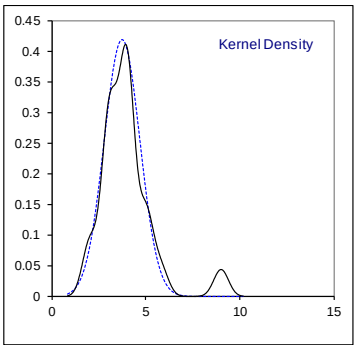
Determination of Color Pt/Co on sample #18021

lab	method	value	mark	z(targ)	remarks
52	D5386	5.8		1.12	
150	D5386	4		0.15	
171	D1209	5		0.69	
311	D1209	<5		-----	
323	D5386	4		0.15	
333	D5386	4		0.15	
334		-----		-----	
343	D5386	4		0.15	
402	D1209	5		0.69	
445	D1209	<5		-----	
453	D1209	3		-0.39	
551	D1209	4		0.15	
555	D5386	4		0.15	
663	D5386	4		0.15	
823	D5386	4.1		0.20	
840	D1209	3		-0.39	
855	D5386	<5		-----	
862	D1209	<5		-----	
864	D5386	<5		-----	
865	D1209	<5		-----	
866	D1209	2		-0.93	
870	D1209	3		-0.39	
912		-----		-----	
913	D5386	3		-0.39	
1040	ISO6271	<5		-----	
1041		-----		-----	
1067	D1209	< 5		-----	
1107	D5386	3		-0.39	
1151		-----		-----	
1201	D1209	<5		-----	
1301	D1209	3.5		-0.12	
1320	D1209	3		-0.39	
1434	D1209	9	R(0.01)	2.86	
1530	ISO6271	<5		-----	
1538	D1209	2		-0.93	
1749	D1209	3.5		-0.12	
1790		-----		-----	
6181	ISO6271	5		0.69	

	normality	OK
	n	22
	outliers	1
	mean (n)	3.72
	st.dev. (n)	0.953
	R(calc.)	2.67
	st.dev.(D5386:16)	1.848
	R(D5386:16)	5.18

Compare: R(D1209:05e1) = 7





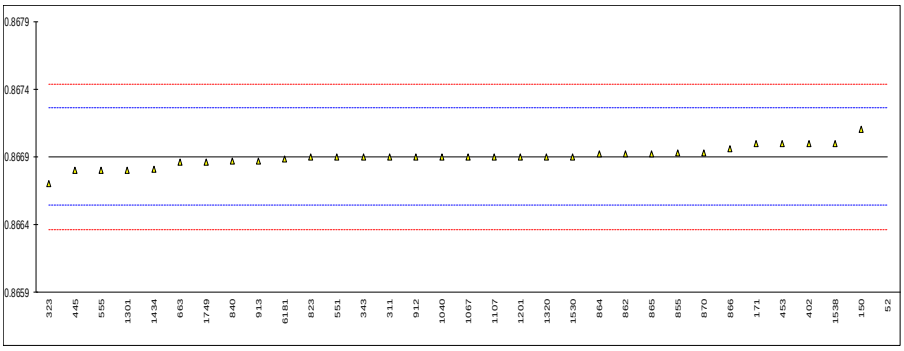
Determination of Copper Corrosion on sample #18021

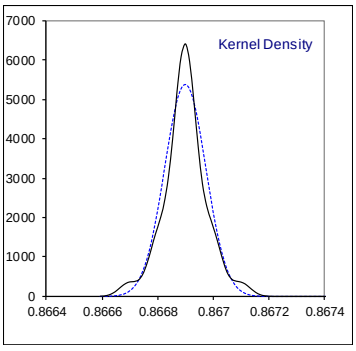
lab	method	value	mark	z(targ)	remarks
52	D849	1A		-----	
150	D849	1a		-----	
171	D849	1a		-----	
311	D849	1		-----	
323	D849	1A		-----	
333		-----		-----	
334		-----		-----	
343		-----		-----	
402		-----		-----	
445	D849	1a		-----	
453		-----		-----	
551	D849	1 a		-----	
555	D849	1a		-----	
663	D849	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		-----	
1040		-----		-----	
1041		-----		-----	
1067	D849	1A		-----	
1107		-----		-----	
1151		-----		-----	
1201	D849	1A		-----	
1301	D849	1A		-----	
1320		-----		-----	
1434	D849	1a		-----	
1530	D849	1a		-----	
1538		-----		-----	
1749		-----		-----	
1790		-----		-----	
6181		-----		-----	
	n	24			
	mean (n)	1a			

Determination of Density at 20°C on sample #18021: results in kg/L

lab	method	value	mark	z(targ)	remarks
52	D4052	0.8704	R(0.01)	19.59	
150	D4052	0.8671		1.11	
171	D4052	0.8670		0.55	
311	D4052	0.8669		-0.01	
323	D4052	0.8667		-1.13	
333		-----		-----	
334		-----		-----	
343	ISO12185	0.8669		-0.01	
402	ISO12185	0.8670		0.55	
445	D4052	0.8668		-0.57	
453	ISO12185	0.8670		0.55	
551	D4052	0.8669		-0.01	
555	D4052	0.8668		-0.57	
663	D4052	0.86686		-0.23	
823	ISO12185	0.86690		-0.01	
840	D4052	0.86687		-0.17	
855	ISO12185	0.86693		0.16	
862	D4052	0.86692		0.11	
864	D4052	0.86692		0.11	
865	D4052	0.86692	C	0.11	first reported: 866.92
866	D4052	0.86696		0.33	
870	D4052	0.86693		0.16	
912	D4052	0.8669		-0.01	
913	D4052	0.86687		-0.17	
1040	ISO12185	0.86690		-0.01	
1041		-----		-----	
1067	D4052	0.8669		-0.01	
1107	D4052	0.8669		-0.01	
1151		-----		-----	
1201	D4052	0.8669		-0.01	
1301	D4052	0.8668	C	-0.57	first reported: 866.8
1320	ISO12185	0.8669		-0.01	
1434	D4052	0.86681		-0.51	
1530	ISO12185	0.8669		-0.01	
1538	ISO3675	0.867		0.55	
1749	ISO12185	0.86686		-0.23	
1790		-----		-----	
6181	ISO12185	0.86688		-0.12	

normality	suspect
n	32
outliers	1
mean (n)	0.86690
st.dev. (n)	0.000074
R(calc.)	0.00021
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005



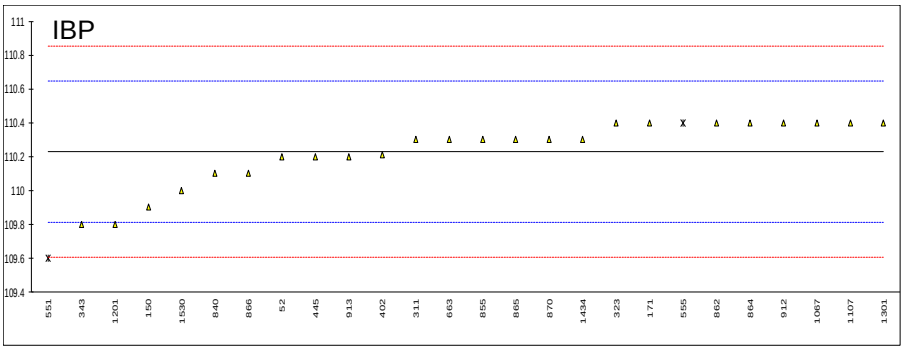


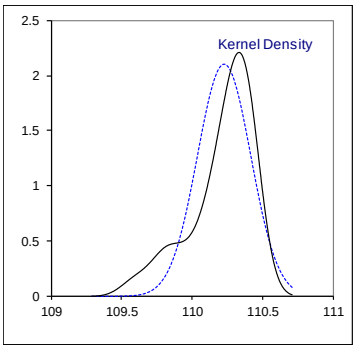
Determination of Distillation on sample #18021; results in °C

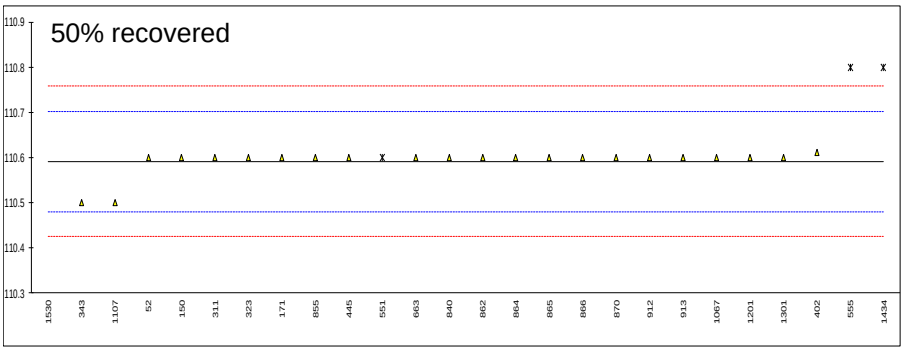
lab	method	IBP	mark	z(targ)	50% rec	mark	z(targ)	DP	mark	z(targ)
52	D850-automated	110.2		-0.14	110.6		0.16	110.7		-0.25
150	D850-automated	109.9	C	-1.59	110.6		0.16	110.7		-0.25
171	D850-automated	110.4		0.82	110.6		0.16	110.6		-0.86
311	D850-automated	110.3		0.34	110.6		0.16	110.8		0.37
323	D850-manual	110.4		0.82	110.6		0.16	110.8		0.37
333		-----		-----	-----		-----	-----		-----
334		-----		-----	-----		-----	-----		-----
343	D850-automated	109.8		-2.07	110.5		-1.64	110.5		-1.48
402	D850-manual	110.21		-0.09	110.61		0.33	110.71		-0.19
445	D850-manual	110.2		-0.14	110.6		0.16	111.2		2.82
453		-----		-----	-----		-----	-----		-----
551	D850-automated	109.6	R(0.05)	-3.03	110.6	ex	0.16	114.0	R(0.01)	20.02
555	D850	110.4	ex	0.82	110.8	R(0.01)	3.74	111.6	R(0.01)	5.28
663	D850-automated	110.30		0.34	110.60		0.16	110.70		-0.25
823		-----		-----	-----		-----	-----		-----
840	D850-automated	110.10		-0.62	110.60		0.16	110.66		-0.49
855	D850-manual	110.3		0.34	110.6		0.16	110.7		-0.25
862	D850-manual	110.4		0.82	110.6		0.16	110.7		-0.25
864	D850-automated	110.4		0.82	110.6		0.16	110.7		-0.25
865	D850-manual	110.3		0.34	110.6		0.16	110.7		-0.25
866	D850-manual	110.1		-0.62	110.6		0.16	110.8		0.37
870	D850-manual	110.3		0.34	110.6		0.16	110.7		-0.25
912	D850-manual	110.4		0.82	110.6		0.16	110.8		0.37
913	D850-manual	110.2		-0.14	110.6		0.16	110.8		0.37
1040		-----		-----	-----		-----	-----		-----
1041		-----		-----	-----		-----	-----		-----
1067	D850-manual	110.4		0.82	110.6		0.16	110.7		-0.25
1107	D850-automated	110.4		0.82	110.5		-1.64	110.7		-0.25
1151		-----		-----	-----		-----	-----		-----
1201	D850-automated	109.8		-2.07	110.6		0.16	110.8		0.37
1301	D850-manual	110.4		0.82	110.6		0.16	111.0		1.59
1320		-----		-----	-----		-----	-----		-----
1434	D850-automated	110.3		0.34	110.8	R(0.01)	3.74	110.9		0.98
1530		110.0		-1.11	110.2	R(0.01)	-7.02	110.4		-2.09
1538	D850-automated	-----		-----	-----		-----	-----		-----
1749		-----		-----	-----		-----	-----		-----
1790		-----		-----	-----		-----	-----		-----
6181		-----		-----	-----		-----	-----		-----
	normality	OK			not OK			not OK		
	n	24			22			24		
	outliers	1 (+1ex)			3 (+1ex)			2		
	mean (n)	110.23			110.59			110.74		
	st.dev. (n)	0.190			0.030			0.154		
	R(calc.)	0.53			0.08			0.43		
	st.dev.(D850-auto:16)	0.208			0.056			0.163		
	R(D850-auto:16)	0.58			0.16			0.46		

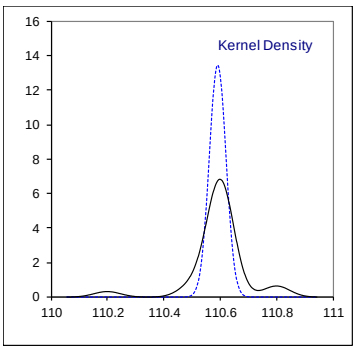
Lab 150 first reported for IBP: 109.6
Lab 551 and 555: two out of three test results were outliers, the other test result is excluded

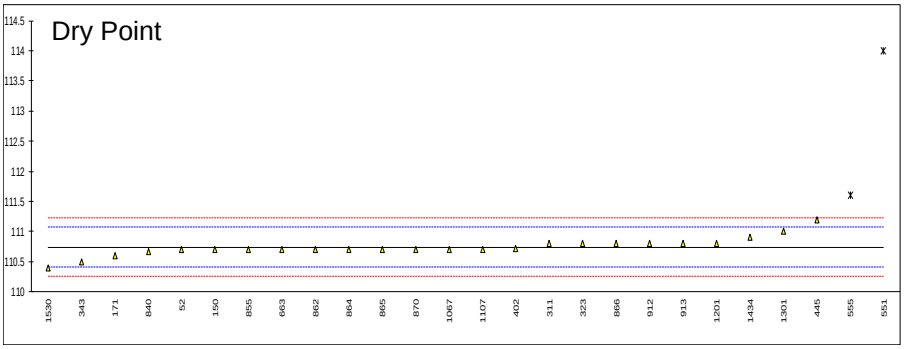
Theoretical mid-boiling point = 110.6°C

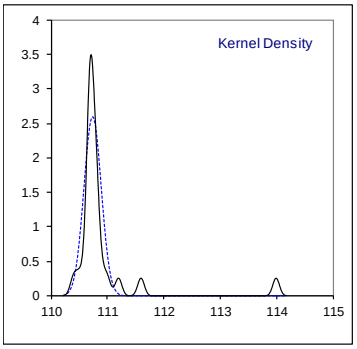

IBP
111
110.8
110.6
110.4
110.2
110
109.8
109.6
109.4
551 343 1201 150 1530 840 866 52 445 913 402 311 663 855 865 870 1434 323 171 595 862 864 912 1067 1107 1301


Kernel Density
2.5
2
1.5
1
0.5
0
109 109.5 110 110.5 111


50% recovered
110.9
110.8
110.7
110.6
110.5
110.4
110.3
1530 343 1107 52 150 311 323 171 855 445 551 663 840 862 864 865 866 870 912 913 1067 1201 1301 402 595 1434


Kernel Density
16
14
12
10
8
6
4
2
0
110 110.2 110.4 110.6 110.8 111

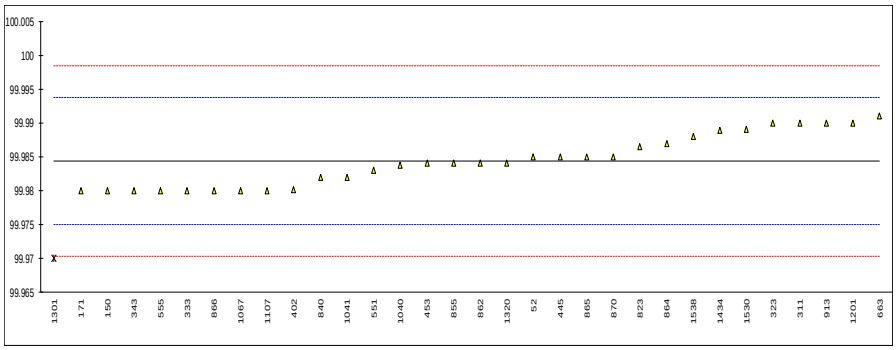

Dry Point
114.5
114
113.5
113
112.5
112
111.5
111
110.5
110
1530 343 171 840 52 150 855 663 862 864 865 870 1067 1107 402 311 323 866 912 913 1201 1434 1301 445 595 551

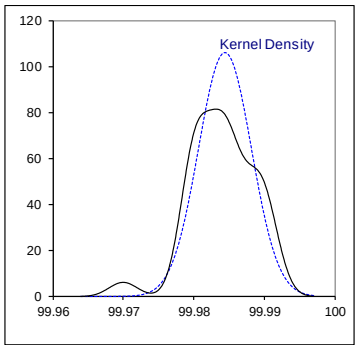

Kernel Density
4
3.5
3
2.5
2
1.5
1
0.5
0
110 111 112 113 114 115

Determination of Purity by GC on sample #18021; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	D7504	99.985		0.12	
150	D7504	99.98		-0.95	
171	D7504	99.98		-0.95	
311	D2360	99.99		1.19	
323	D7504	99.99		1.19	
333	D2360	99.98		-0.95	
334		-----		-----	
343	D2360	99.98		-0.95	
402	D7504	99.9802		-0.90	
445	D6526	99.985		0.12	
453	D2360	99.984		-0.09	
551	D2360	99.983		-0.31	
555	D7504	99.98		-0.95	
663	D7504	99.991		1.40	
823	D2360	99.9865		0.44	
840	D7504	99.982		-0.52	
855	D7504	99.984		-0.09	
862	D7504	99.984		-0.09	
864	D7504	99.987		0.55	
865	D7504	99.985		0.12	
866	D2360	99.98		-0.95	
870	D7504	99.985		0.12	
912		-----		-----	
913	D2360	99.99		1.19	
1040	D7504	99.9838		-0.14	
1041	In house	99.982	C	-0.52	first reported: 99.9410
1067	In house	99.98		-0.95	
1107	D7504	99.98		-0.95	
1151		-----		-----	
1201	D2360	99.99		1.19	
1301	D2360	99.97	C,R(0.05)	-3.08	first reported: 99.9
1320	D6526	99.984		-0.09	
1434	D4492	99.98891		0.96	
1530	D7504	99.989		0.98	
1538	D2360	99.988		0.76	
1749		-----		-----	
1790		-----		-----	
6181		-----		-----	

normality	OK
n	31
outliers	1
mean (n)	99.9844
st.dev. (n)	0.00375
R(calc.)	0.0105
st.dev.(D7504:18)	0.00468
R(D7504:18)	0.0131



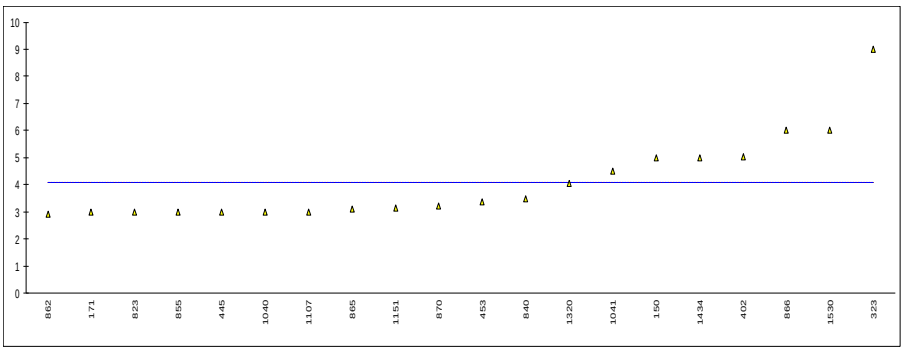


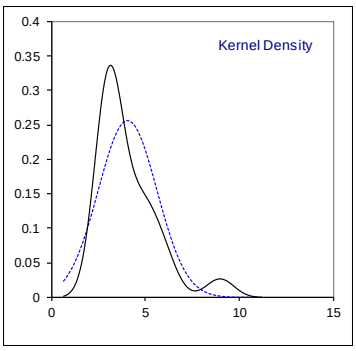
Determination of Benzene on sample #18021; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	D7504	<10		-----	
150	D7504	5		-----	
171	D7504	3		-----	
311	D2360	<10		-----	
323	D7504	9		-----	
333	D2360	<10		-----	
334		-----		-----	
343	D2360	<10		-----	
402	D7504	5.02		-----	
445	D6526	3		-----	
453	D2360	3.3595		-----	
551	D2360	< 5		-----	
555		-----		-----	
663	D7504	<1		-----	
823	D2360	3		-----	
840	D7504	3.5		-----	
855	D7504	3		-----	
862	D7504	2.9		-----	
864	D7504	<10		-----	
865	D7504	3.1		-----	
866	D2360	6.0		-----	
870	D7504	3.2		-----	
912		-----		-----	
913		-----		-----	
1040	D7504	3		-----	
1041	In house	4.5	C	-----	first reported: 22.9
1067	In house	< 10		-----	
1107	D7504	3		-----	
1151	In house	3.16		-----	
1201	D2360	<10		-----	
1301	D2360	<10		-----	
1320	D6526	4.05		-----	
1434	D4492	5.0		-----	
1530	D7504	6		-----	
1538	D2360	<10		-----	
1749		-----		-----	
1790		-----		-----	
6181		-----		-----	

	normality	not OK
	n	20
	outliers	0
	mean (n)	4.09
	st.dev. (n)	1.559
	R(calc.)	4.36
	st.dev.(D7504:18)	(0.202)
	R(D7504:18)	(0.57) *)

*) Reproducibility in ASTM D7504:18 is based on a Benzene level much higher than present in sample #18021, see also §4.1

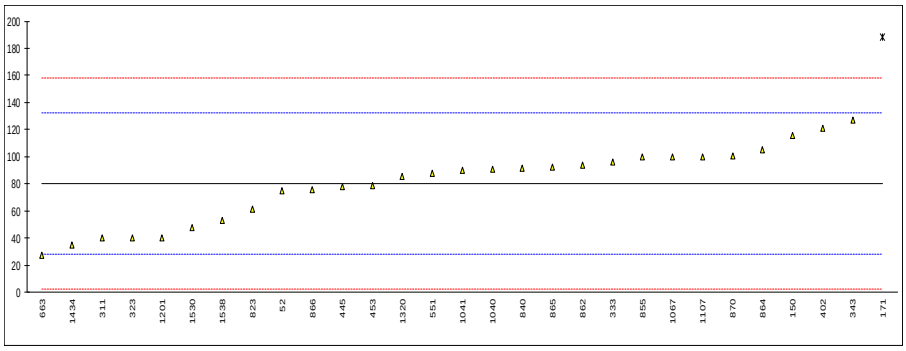


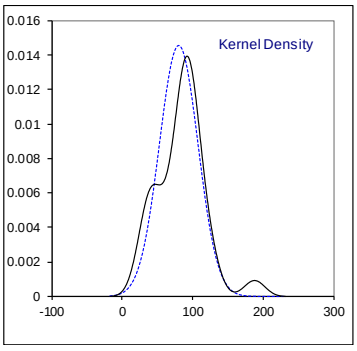


Determination of Nonaromatics on sample #18021; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	D7504	75		-0.21	
150	D7504	116		1.37	
171	D7504	188	R(0.05)	4.15	
311	D2360	40		-1.56	
323	D7504	40		-1.56	
333	D2360	96		0.60	
334		-----		-----	
343	D2360	127		1.80	
402	D7504	121.22		1.58	
445	D6526	78		-0.09	
453	D2360	78.881		-0.06	
551	D2360	88		0.29	
555		-----		-----	
663	D7504	27.0		-2.06	
823	D2360	61		-0.75	
840	D7504	91.6		0.43	
855	D7504	100		0.76	
862	D7504	94		0.53	
864	D7504	105		0.95	
865	D7504	92		0.45	
866	D2360	76.0		-0.17	
870	D7504	100.5		0.78	
912		-----		-----	
913		-----		-----	
1040	D7504	91		0.41	
1041	In house	90.4		0.39	
1067	In house	100		0.76	
1107	D7504	100		0.76	
1151		-----		-----	
1201	D2360	40		-1.56	
1301	D2360	<100		-----	
1320	D6526	85.55		0.20	
1434	D4492	34.9		-1.75	
1530	D7504	48		-1.25	
1538	D7504	53		-1.05	
1749		-----		-----	
1790		-----		-----	
6181		-----		-----	

normality	OK
n	28
outliers	1
mean (n)	80.36
st.dev. (n)	27.422
R(calc.)	76.78
st.dev.(D7504:18)	25.940
R(D7504:18)	72.63

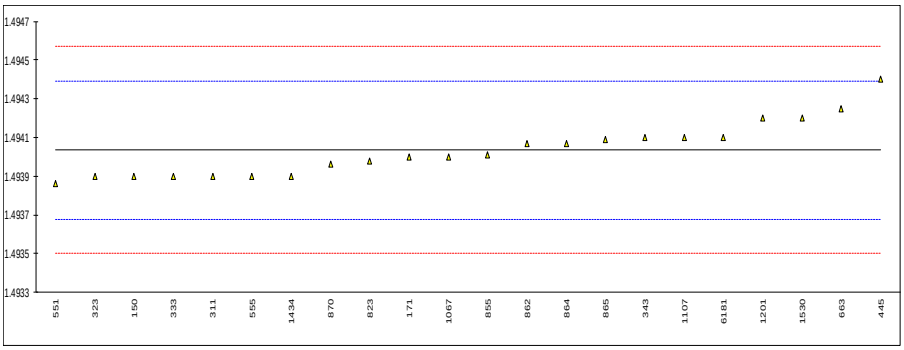


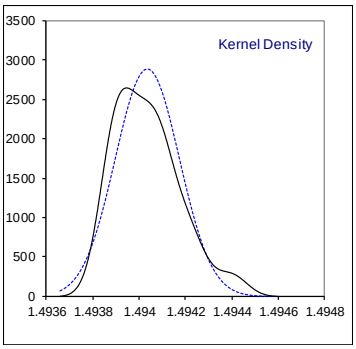


Determination of Refractive Index at 25 °C on sample #18021;

lab	method	value	mark	z(targ)	remarks
52		-----		-----	
150	D1218	1.4939		-0.76	
171	D1218	1.4940		-0.20	
311	D1218	1.4939		-0.76	
323	D1218	1.4939		-0.76	
333	D1218	1.4939	C	-0.76	first reported: 1.4968
334		-----		-----	
343	D1218	1.4941		0.36	
402		-----		-----	
445	D1218	1.4944		2.04	
453		-----		-----	
551	D1218	1.49386		-0.98	
555	D1218	1.4939		-0.76	
663	D1218	1.49425		1.20	
823	D1218	1.49398		-0.31	
840		-----		-----	
855	D1218	1.49401		-0.14	
862	D1218	1.49407		0.19	
864	D1218	1.49407		0.19	
865	D1218	1.49409		0.30	
866		-----		-----	
870	D1218	1.49396		-0.42	
912		-----		-----	
913		-----		-----	
1040		-----		-----	
1041		-----		-----	
1067	D1218	1.4940		-0.20	
1107	D1218	1.4941		0.36	
1151		-----		-----	
1201	D1218	1.4942		0.92	
1301		-----		-----	
1320		-----		-----	
1434	D1218	1.4939		-0.76	
1530	D1218	1.4942		0.92	
1538		-----		-----	
1749		-----		-----	
1790		-----		-----	
6181	D1045	1.49410		0.36	

normality	OK
n	22
outliers	0
mean (n)	1.49404
st.dev. (n)	0.000139
R(calc.)	0.00039
st.dev.(D1218:12)	0.000179
R(D1218:12)	0.0005





APPENDIX 2

Number of participants

3 labs in BELGIUM
2 labs in BRAZIL
1 lab in CANADA
6 labs in CHINA, People's Republic
2 labs in FRANCE
4 labs in GERMANY
2 labs in INDIA
1 lab in ISRAEL
1 lab in LATVIA
3 labs in NETHERLANDS
1 lab in POLAND
1 lab in ROMANIA
1 lab in SAUDI ARABIA
1 lab in SLOVAKIA
1 lab in SOUTH KOREA
1 lab in SPAIN
1 lab in THAILAND
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	= probably an error in calculations
U	= test result probably reported in a different unit
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, March 2017
2 W. Horwitz and R. Albert, J. AOAC Int., Vol. 79, 3, p. 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)