

Institute for  
Interlaboratory Studies

# Results of Proficiency Test Toluene March 2023

**Organized by:** Institute for Interlaboratory Studies  
Spijkenisse, the Netherlands

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## 1 INTRODUCTION

Since 1999 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Toluene based on the latest version of ASTM D841 every year. During the annual proficiency testing program 2022/2023 it was decided to continue the round robin for the analysis of Toluene.

In this interlaboratory study 32 laboratories in 19 countries registered for participation, see appendix 2 for the number of participants per country. In this report the results of the Toluene proficiency test are presented and discussed. This report is also electronically available through the iis website [www.iisnl.com](http://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 Toluene in a 1 L bottle labelled #23021.

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 [www.iisnl.com](http://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 80 liters of Toluene was obtained from a local chemical supplier and was spiked with Benzene. After homogenization 60 amber glass bottles of 1 L were filled and labelled #23021.

The homogeneity of the subsamples was checked by determination of Density at 20 °C in accordance with ASTM D4052 on 8 stratified randomly selected subsamples.

	Density at 20 °C in kg/L
sample #23021-1	0.86685
sample #23021-2	0.86684
sample #23021-3	0.86685
sample #23021-4	0.86685
sample #23021-5	0.86686
sample #23021-6	0.86685
sample #23021-7	0.86685
sample #23021-8	0.86686

Table 1: homogeneity test results of subsamples #23021

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 20 °C in kg/L
r (observed)	0.00002
reference test method	ISO12185:96
0.3 x R (reference test method)	0.00015

Table 2: evaluation of the repeatability of subsamples #23021

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 1 L bottle of Toluene labelled #23021 was sent on February 1, 2023. 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 ANALYZES

The participants were requested to determine: Acid Wash Color, Appearance, Color Pt/Co, Copper Corrosion, Density at 20 °C, Distillation (IBP, 50% recovered, DP and Range), Purity by GC, Benzene, Nonaromatics, Total Impurities, Refractive Index at 25 °C and Water.

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/](http://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](http://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/](http://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 reanalyses). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the 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.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. Outliers are marked by  $D(0.01)$  for the Dixon's test, by  $G(0.01)$  or  $DG(0.01)$  for the Grubbs' test and by  $R(0.01)$  for the Rosner's test. Stragglers are marked by  $D(0.05)$  for the Dixon's test, by  $G(0.05)$  or  $DG(0.05)$  for the Grubbs' test and by  $R(0.05)$  for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

### 3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

### 3.3 Z-SCORES

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

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated according to:

$$z_{(\text{target})} = (\text{test result} - \text{average of PT}) / \text{target standard deviation}$$

The  $z_{(\text{target})}$  scores are listed in the test result tables in appendix 1.

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

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

## 4 EVALUATION

In this proficiency test some problems were encountered with the dispatch of the samples. Therefore, the reporting time on the data entry portal was extended with another week. Three participants reported test results after the extended reporting date and eight other participants did not report any test results. Not all participants were able to report all tests requested.

In total 24 participants reported 225 numerical test results. Observed were 8 outlying test results, which is 3.6%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

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

### 4.1 EVALUATION PER 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 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 3.

Unfortunately, a suitable reference test method, providing the precision data, is not available for all determinations. For these tests the calculated reproducibility was compared against the estimated reproducibility calculated with the Horwitz equation.

In the iis PT reports ASTM test methods are referred to with a number (e.g. D1209) and an added designation for the year that the test method was adopted or revised (e.g. D1209:05). When a method has been reapproved an "R" will be added and the year of approval (e.g. D1209:05R19).

Acid Wash Color: 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 D848:23.

Appearance: This determination was not problematic. All reporting participants agreed on a test result of Bright & Clear (Pass).

Color Pt/Co: 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 D5386:16 and ASTM D1209:05R19.

Copper Corrosion: This determination was not problematic. All reporting participants agreed on a result of 1/1a.

Density at 20 °C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ISO12185:96.

Distillation: This determination was not problematic. In total two statistical outliers were observed over three parameters. All calculated reproducibilities after rejection of the statistical outliers are in agreement with the requirements of ASTM D850:21 automated or manual mode, except for IBP manual mode.

Purity by GC: 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 D7504:21.

Benzene: This determination may be problematic. One statistical outlier was observed. In ASTM D7504:21 the given reproducibility is related to a much higher level of Benzene. Extrapolation of the literature reproducibility with the mean in this PT gives an unrealistic small reproducibility. Therefore, no z-scores are calculated.

Nonaromatics: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D7504:21.

Total Impurities: This determination may be problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the estimated reproducibility calculated with the Horwitz equation based on 3 components.



Refractive Index at 25 °C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D1218:21.

Water: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM E1064:16.

When the test results were evaluated separately for participants who used E1064 the calculated reproducibility is in full agreement with the requirements of ASTM E1064:16.

## 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 table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Acid Wash Color		17	0.7	0.7	2.0
Appearance		21	B&C (Pass)	n.a.	n.a.
Color Pt/Co		21	3.4	2.5	5.1
Copper Corrosion		15	1 (1a)	n.a.	n.a.
Density at 20 °C	kg/L	23	0.8669	0.0002	0.0005
Distillation, IBP	°C	20	110.2	0.5	0.6
Distillation, 50% recovered	°C	18	110.6	0.0	0.2
Distillation, DP	°C	19	110.7	0.2	0.5
Purity by GC	%M/M	21	99.960	0.008	0.013
Benzene	mg/kg	21	8.8	4.3	(1.2)
Nonaromatics	mg/kg	23	141	90	128
Total Impurities	mg/kg	18	394	150	124
Refractive Index at 25 °C		14	1.4940	0.0002	0.0005
Water	mg/kg	20	141	26	22

Table 3: reproducibilities of tests on sample #23021

For results between brackets no z-scores are calculated.

Without further statistical calculations it can be concluded that for many 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 COMPARISON OF THE PROFICIENCY TEST OF MARCH 2023 WITH PREVIOUS PTS

	March 2023	February 2022	February 2021	February 2020	February 2019
Number of reporting laboratories	24	31	40	26	35
Number of test results	225	276	405	253	284
Number of statistical outliers	8	12	10	16	14
Percentage of statistical outliers	3.6%	4.3%	2.5%	6.3%	4.9%

Table 4: comparison with previous proficiency tests

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

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

Parameter	March 2023	February 2022	February 2021	February 2020	February 2019
Acid Wash Color	++	+	++	++	++
Color Pt/Co	++	+	+	+	++
Density at 20 °C	++	++	++	++	++
Distillation	+	++	+	+	+
Purity by GC	+	-	+/-	--	-
Benzene	(--)	(--)	--	+/-	(--)
Nonaromatics	+	++	++	++	++
Total Impurities	-	-	+/-	+/-	n.e.
Refractive Index at 25 °C	++	+	-	-	+
Water	-	+	-	-	n.e.

Table 5: comparison of determinations to the reference test methods

For results between brackets no z-scores are calculated.

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

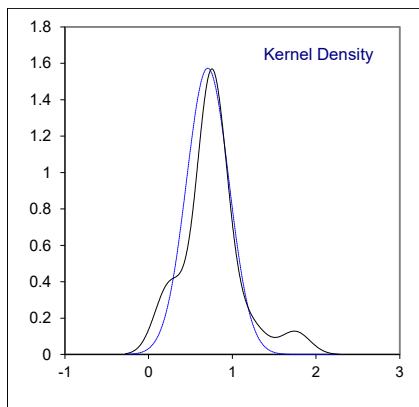
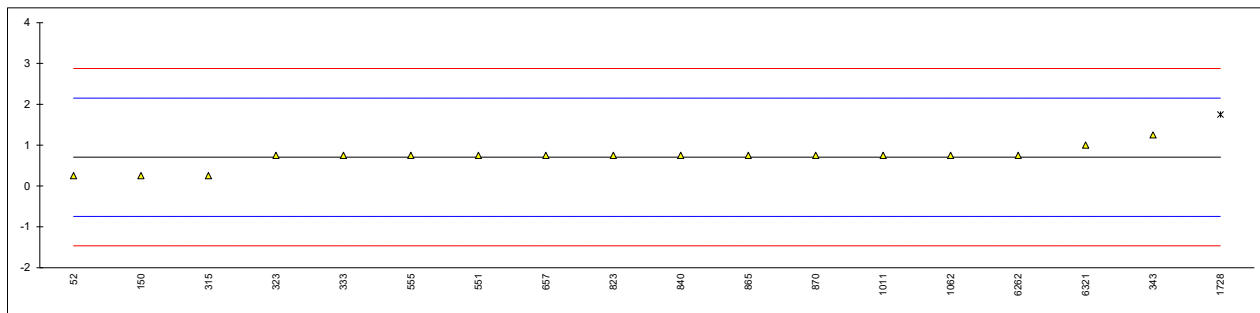
**APPENDIX 1**

**Determination of Acid Wash Color on sample #23021;**

lab	method	Reported test value	iis conversion *)	mark	z(targ)	remarks
52	D848	0+	0.25		-0.63	
150	D848	0+	0.25		-0.63	
171	D848	Pass	Pass		----	
315	D848	0+	0.25		-0.63	
323	D848	-1	0.75		0.06	
333	D848	1 - [X]	0.75		0.06	
334		----	----		----	
343	D848	1+	1.25		0.75	
372		----	----		----	
396		----	----		----	
445		----	----		----	
551	D848	1-	0.75		0.06	
555	D848	1-	0.75		0.06	
657	D848	1-	0.75		0.06	
823	D848	1-	0.75		0.06	
840	D848	1-	0.75		0.06	
855		----	----		----	
862		----	----		----	
864		----	----		----	
865	D848	1-	0.75		0.06	
866		----	----		----	
870	D848	1-	0.75		0.06	
902		----	----		----	
912		----	----		----	
913		----	----		----	
1011	D848	1-	0.75		0.06	
1062	D848	1-	0.75		0.06	
1728	D848	2-	1.75	G(0.05)	1.44	
1812		----	----		----	
6203		----	----		----	
6262	D848	1-	0.75		0.06	
6321	D848	1	1		0.41	

normality suspect  
n 17  
outliers 1  
mean (n) 0.706  
st.dev. (n) 0.2536  
R(calc.) 0.710  
st.dev.(D848:23) 0.7234  
R(D848:23) 2.025

\*) 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).

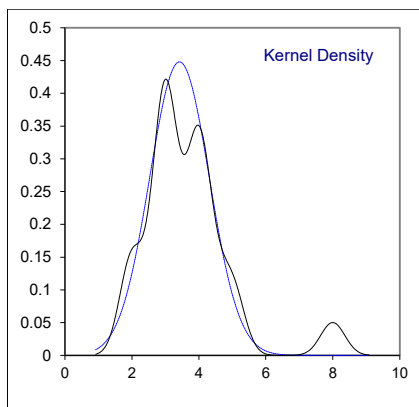
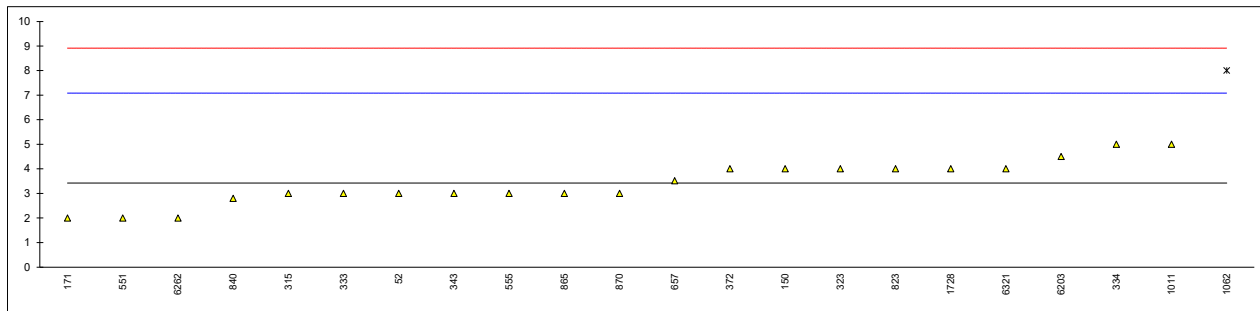


## Determination of Appearance on sample #23021;

lab	method	value	mark	z(targ)	remarks
52	E2680	Pass		----	
150	E2680	Pass		----	
171	E2680	Pass		----	
315	E2680	pass		----	
323	E2680	C&B		----	
333		----		----	
334	EN15769	clear and bright		----	
343	E2680	Pass		----	
372	E2680	pass		----	
396	Visual	Clear and Bright		----	
445		----		----	
551	E2680	Pass		----	
555	E2680	Pass		----	
657	Visual	Clear & Bright		----	
823	E2680	Pass		----	
840	E2680	Pass		----	
855		----		----	
862		----		----	
864		----		----	
865	E2680	Pass		----	
866		----		----	
870	E2680	Clear&Bright		----	
902		----		----	
912		----		----	
913		----		----	
1011	Visual	Bright and Clear		----	
1062	Visual	pass		----	
1728	Visual	CLEAR		----	
1812		----		----	
6203		----		----	
6262	Visual	clear and bright		----	
6321	Visual	Clear & Bright		----	
	n	21			
	mean (n)	Bright & Clear (Pass)			

Determination of Color Pt/Co on sample #23021;

lab	method	value	mark	z(targ)	remarks
52	D5386	3		-0.23	
150	D5386	4		0.32	
171	D5386	2		-0.78	
315	D5386	3		-0.23	
323	D5386	4		0.32	
333	D5386	3		-0.23	
334	D1209	5		0.86	
343	D5386	3		-0.23	
372	D5386	4		0.32	
396		----		----	
445		----		----	
551	D5386	2		-0.78	
555	D5386	3		-0.23	
657	D5386	3.52		0.05	
823	D5386	4		0.32	
840	D1209	2.8		-0.34	
855		----		----	
862		----		----	
864		----		----	
865	D5386	3		-0.23	
866		----		----	
870	D5386	3		-0.23	
902		----		----	
912		----		----	
913		----		----	
1011	D1209	5		0.86	
1062	D5386	8	R(0.01)	2.50	
1728	D1209	4		0.32	
1812		----		----	
6203	D1209	4.5		0.59	
6262	D5386	2.0		-0.78	
6321	D1209	4		0.32	
normality		OK			
n		21			
outliers		1			
mean (n)		3.42			
st.dev. (n)		0.890			
R(calc.)		2.49			
st.dev.(D5386:16)		1.832			
R(D5386:16)		5.13			
Compare					
R(D1209:05R19)		7			

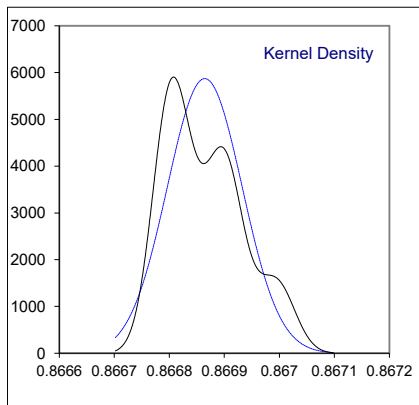
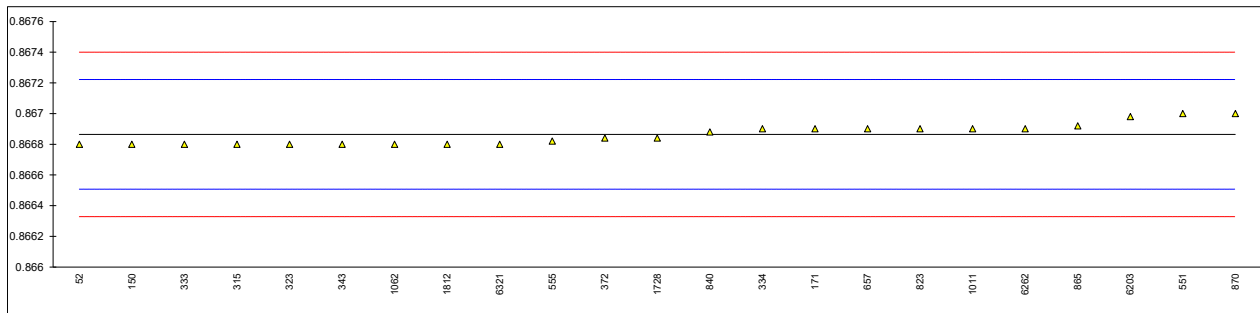


## Determination of Copper Corrosion on sample #23021;

lab	method	value	mark	z(targ)	remarks
52	D849	1a		----	
150	D849	1A		----	
171	D849	1a		----	
315	D849	1A		----	
323	D849	1A		----	
333		----		----	
334		----		----	
343		----		----	
372		----		----	
396		----		----	
445		----		----	
551	D849	1A		----	
555	D849	1a		----	
657	D849	1a		----	
823	D849	1a		----	
840	D849	1A		----	
855		----		----	
862		----		----	
864		----		----	
865	D849	1a		----	
866		----		----	
870	D849	1a		----	
902		----		----	
912		----		----	
913		----		----	
1011		----		----	
1062	D849	1A		----	
1728	D849	1A		----	
1812		----		----	
6203		----		----	
6262	D849	1		----	
6321		----		----	
	n	15			
	mean (n)	1 (1a)			

Determination of Density at 20 °C on sample #23021: results in kg/L

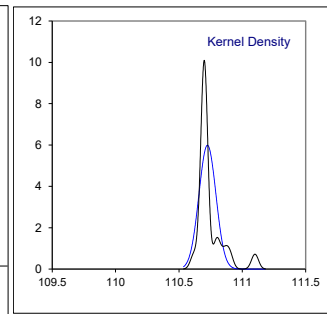
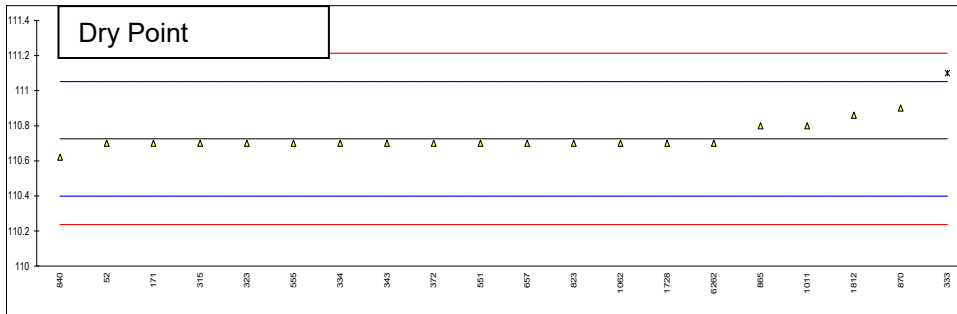
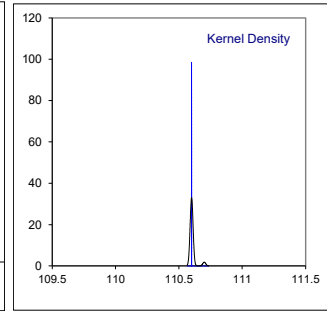
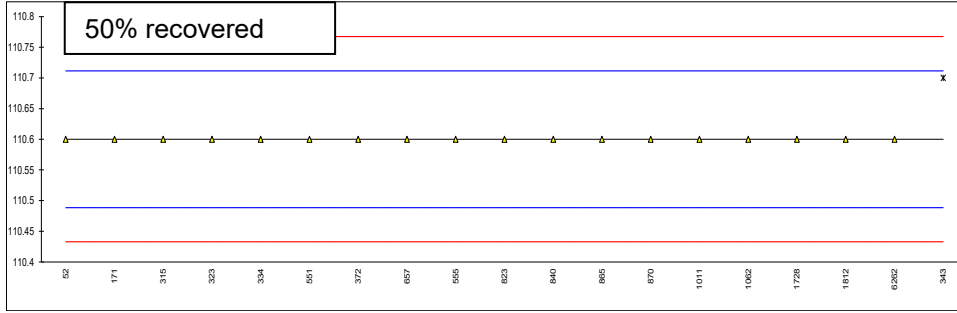
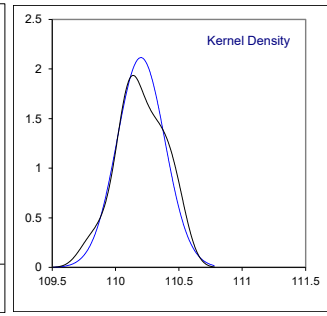
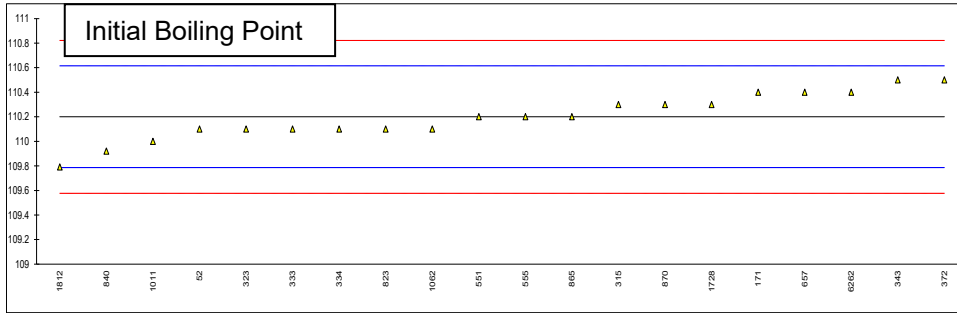
lab	method	value	mark	z(target)	remarks
52	D4052	0.8668		-0.36	
150	ISO12185	0.8668		-0.36	
171	ISO12185	0.8669		0.20	
315	D4052	0.8668		-0.36	
323	D4052	0.8668		-0.36	
333	D4052	0.8668		-0.36	
334	ISO12185	0.8669		0.20	
343	ISO12185	0.8668		-0.36	
372	ISO12185	0.86684		-0.14	
396		----		----	
445		----		----	
551	D4052	0.8670		0.76	
555	D4052	0.86682		-0.25	
657	D4052	0.8669		0.20	
823	ISO12185	0.8669		0.20	
840	D4052	0.86688		0.09	
855		----		----	
862		----		----	
864		----		----	
865	D4052	0.86692		0.31	
866		----		----	
870	D4052	0.8670		0.76	
902		----		----	
912		----		----	
913		----		----	
1011	D4052	0.8669		0.20	
1062	D4052	0.8668		-0.36	
1728	ISO12185	0.86684		-0.14	
1812	ISO12185	0.8668		-0.36	
6203	D4052	0.86698		0.65	
6262	D4052	0.8669		0.20	
6321	ISO12185	0.8668		-0.36	
normality		OK			
n		23			
outliers		0			
mean (n)		0.86686			
st.dev. (n)		0.000068			
R(calc.)		0.00019			
st.dev.(ISO12185:96)		0.000179			
R(ISO12185:96)		0.0005			



Determination of Distillation on sample #23021; results in °C

lab	method	IBP	mark	z(targ)	50% rec	mark	z(targ)	DP	mark	z(targ)	Range
52	D850-automated	110.1		-0.48	110.6		0.00	110.7		-0.16	0.6
150		----		----	----		----	----		----	----
171	D850-automated	110.4		0.96	110.6		0.00	110.7		-0.16	0.3
315	D850-automated	110.3		0.48	110.6		0.00	110.7		-0.16	0.4
323	D850-automated	110.1		-0.48	110.6		0.00	110.7		-0.16	0.6
333	D850-automated	110.1		-0.48	----		----	111.1	R(0.01)	2.30	1.1
334	D850-automated	110.1		-0.48	110.6		0.00	110.7		-0.16	1.1
343	D850-automated	110.5		1.44	110.7	G(0.01)	1.80	110.7		-0.16	0.2
372	D850-automated	110.5		1.44	110.6		0.00	110.7		-0.16	0.2
396		----		----	----		----	----		----	----
445		----		----	----		----	----		----	----
551	D850-automated	110.2		0.00	110.6		0.00	110.7		-0.16	0.5
555	D850-manual	110.2		0.00	110.6		0.00	110.7		-0.16	0.5
657	D850-automated	110.4		0.96	110.6		0.00	110.7		-0.16	0.3
823	D850-automated	110.1		-0.48	110.6		0.00	110.7		-0.16	0.6
840	D850-automated	109.92		-1.35	110.60		0.00	110.62		-0.65	0.70
855		----		----	----		----	----		----	----
862		----		----	----		----	----		----	----
864		----		----	----		----	----		----	----
865	D850-automated	110.2		0.00	110.6		0.00	110.8		0.46	0.6
866		----		----	----		----	----		----	----
870	D850-manual	110.3		0.48	110.6		0.00	110.9		1.07	0.6
902		----		----	----		----	----		----	----
912		----		----	----		----	----		----	----
913		----		----	----		----	----		----	----
1011	D850-automated	110.0		-0.97	110.6		0.00	110.8		0.46	0.8
1062	D850-automated	110.1		-0.48	110.6		0.00	110.7		-0.16	0.6
1728	D850-manual	110.3		0.48	110.6		0.00	110.7		-0.16	0.4
1812		109.79		-1.98	110.60		0.00	110.86		0.83	1.07
6203		----		----	----		----	----		----	----
6262	D850-automated	110.4		0.96	110.6		0.00	110.7		-0.16	0.3
6321		----		----	----		----	----		----	----
	normality	OK			suspect			not OK			
	n	20			18			19			
	outliers	0			1			1			
	mean (n)	110.20			110.60			110.73			
	st.dev. (n)	0.189			0.000			0.067			
	R(calc.)	0.53			0.00			0.19			
	st.dev.(D850-A:21)	0.208			0.056			0.163			
	R(D850-A:21)	0.58			0.16			0.46			
Compare	R(D850-M:21)	0.41			0.65			0.65			

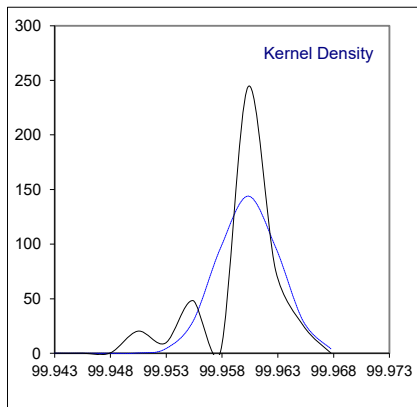
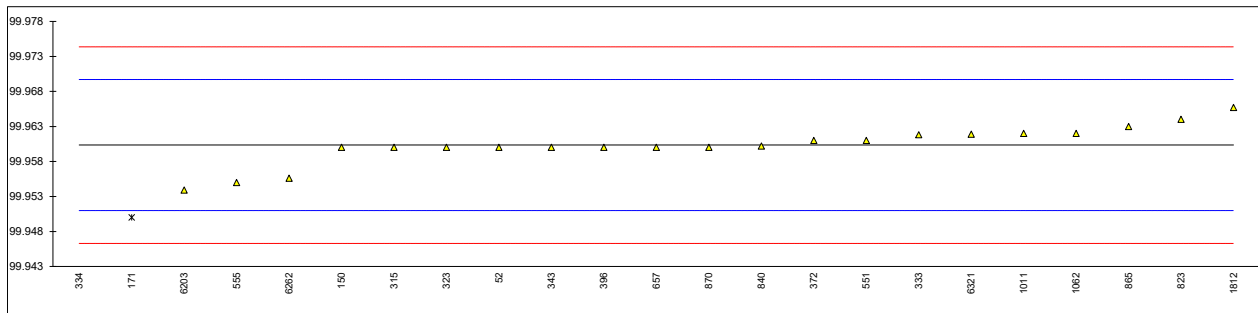




Determination of Purity by GC on sample #23021; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	D7504	99.96		-0.07	
150	D7504	99.96		-0.07	
171	D7504	99.95	C,R(0.05)	-2.21	first reported 99.97
315	D7504	99.96		-0.07	
323	D7504	99.96		-0.07	
333	D7504	99.9618		0.31	
334	D7504	99.34	R(0.01)	-132.56	
343	D2360	99.960		-0.07	
372	D7504	99.961		0.14	
396	D7504	99.96		-0.07	
445		----		----	
551	D7504	99.961		0.14	
555	D7504	99.955		-1.14	
657	D7504	99.96	C	-0.07	first reported 99.9469
823	D7504	99.9640		0.78	
840	D7504	99.9602		-0.03	
855		----		----	
862		----		----	
864		----		----	
865	D7504	99.963		0.57	
866		----		----	
870	D7504	99.960		-0.07	
902		----		----	
912		----		----	
913		----		----	
1011	D5917	99.962		0.35	
1062	D7504	99.962		0.35	
1728		----		----	
1812	D7504	99.9657		1.14	
6203	D7504	99.9539		-1.38	
6262	D7504	99.9556		-1.01	
6321	D2360	99.9619		0.33	

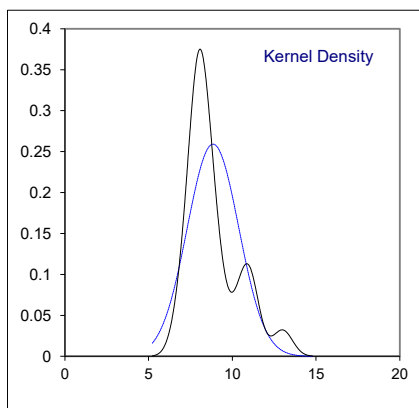
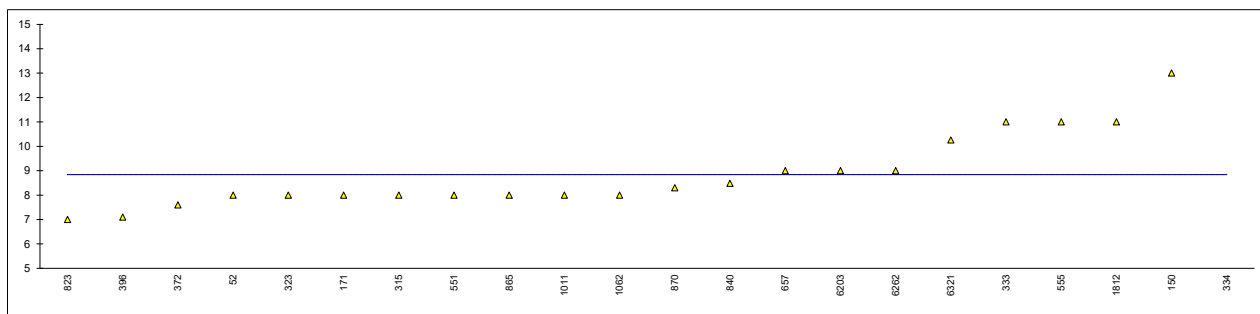
normality suspect  
n 21  
outliers 2  
mean (n) 99.96034  
st.dev. (n) 0.002770  
R(calc.) 0.00776  
st.dev.(D7504:21) 0.004680  
R(D7504:21) 0.01310



Determination of Benzene on sample #23021; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	D7504	8		----	
150	D7504	13	C	----	first reported 0.0013 mg/kg
171	D7504	8		----	
315	D7504	8		----	
323	D7504	8		----	
333	D7504	11		----	
334	D7504	6200	R(0.01)	----	
343	D2360	< 10		----	
372	D7504	7.6		----	
396	D7504	7.1		----	
445		----		----	
551	D7504	8		----	
555	D7504	11		----	
657	D7504	9		----	
823	D7504	7		----	
840	D7504	8.48		----	
855		----		----	
862		----		----	
864		----		----	
865	D7504	8		----	
866		----		----	
870	D7504	8.3		----	
902		----		----	
912		----		----	
913		----		----	
1011	D5917	8		----	
1062	D7504	8		----	
1728		----		----	
1812	D7504	11		----	
6203	D7504	9		----	
6262	D7504	9		----	
6321	D2360	10.255		----	

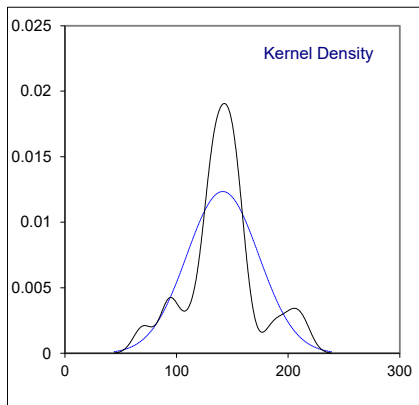
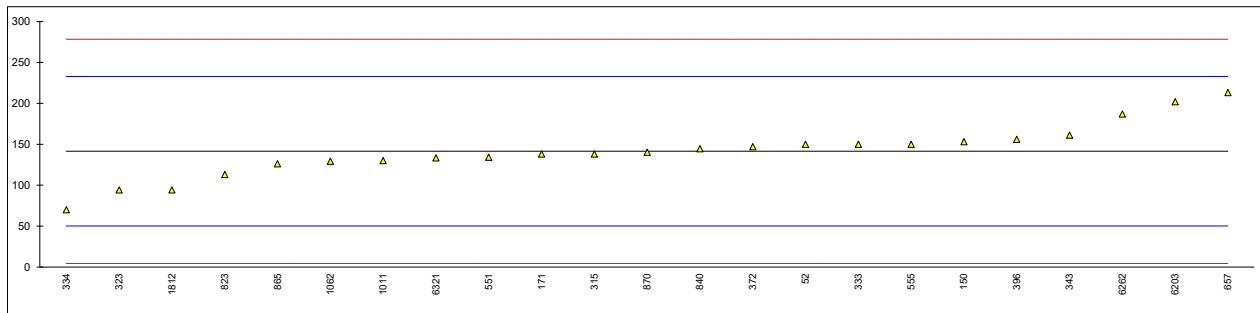
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n 21  
outliers 1  
mean (n) 8.845  
st.dev. (n) 1.5394  
R(calc.) 4.310  
st.dev.(D7504:21) (0.4371)  
R(D7504:21) (1.224)



Determination of Nonaromatics on sample #23021; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	D7504	150		0.19	
150	D7504	153	C	0.25	first reported 0.0153 mg/kg
171	D7504	138		-0.08	
315	D7504	138		-0.08	
323	D7504	94		-1.04	
333	D7504	150		0.19	
334	D7504	70		-1.56	
343	D2360	161		0.43	
372	D7504	147		0.12	
396	D7504	156	C	0.32	first reported 256
445		----		----	
551	D7504	134		-0.16	
555	D7504	150	C	0.19	first reported 217
657	D7504	213		1.57	
823	D7504	113		-0.62	
840	D7504	144.57		0.07	
855		----		----	
862		----		----	
864		----		----	
865	D7504	126		-0.34	
866		----		----	
870	D7504	140.2		-0.03	
902		----		----	
912		----		----	
913		----		----	
1011	D5917	130		-0.25	
1062	D7504	129		-0.27	
1728		----		----	
1812	D7504	94		-1.04	
6203	D7504	202		1.33	
6262	D7504	187		1.00	
6321	D2360	133.34		-0.18	

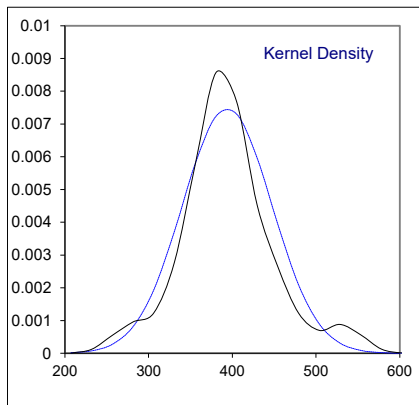
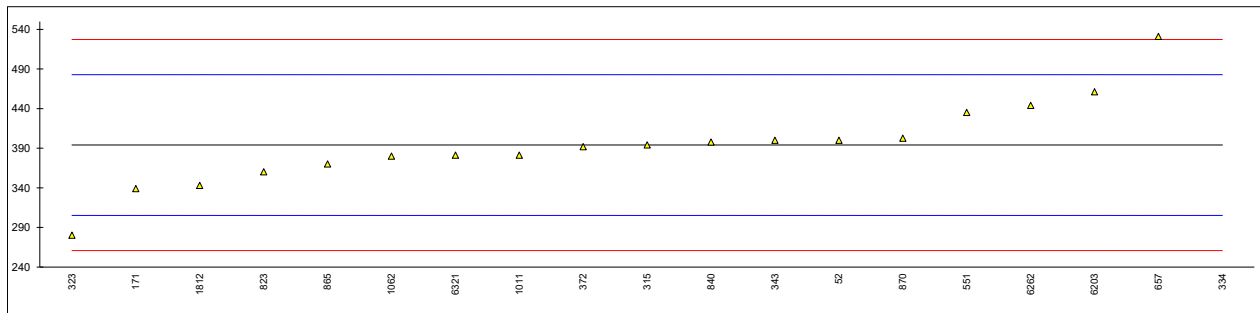
normality suspect  
n 23  
outliers 0  
mean (n) 141.44  
st.dev. (n) 32.289  
R(calc.) 90.41  
st.dev.(D7504:21) 45.657  
R(D7504:21) 127.84



Determination of Total Impurities on sample #23021; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	D7504	400		0.14	
150		----		----	
171	D7504	339		-1.24	
315	D7504	394		0.00	
323	D7504	280		-2.57	
333		----		----	
334	D7504	6440	G(0.01)	136.14	
343	D2360	400		0.14	
372	D7504	392		-0.04	
396		----		----	
445		----		----	
551	D7504	435		0.92	
555		----		----	
657	D7504	531		3.09	
823	D7504	360		-0.76	
840	D7504	397.74		0.09	
855		----		----	
862		----		----	
864		----		----	
865	D7504	370		-0.54	
866		----		----	
870	D7504	402.6		0.19	
902		----		----	
912		----		----	
913		----		----	
1011	D5917	381		-0.29	
1062	D7504	380		-0.31	
1728		----		----	
1812	D7504	343		-1.15	
6203	D7504	461		1.51	
6262	D7504	444		1.13	
6321	D2360	380.995		-0.29	

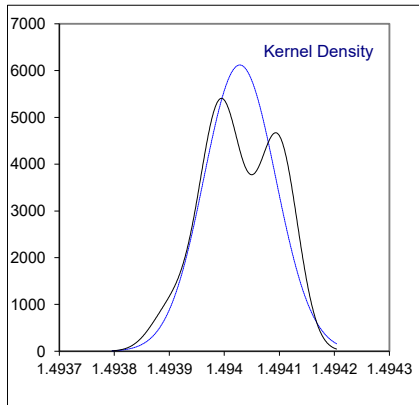
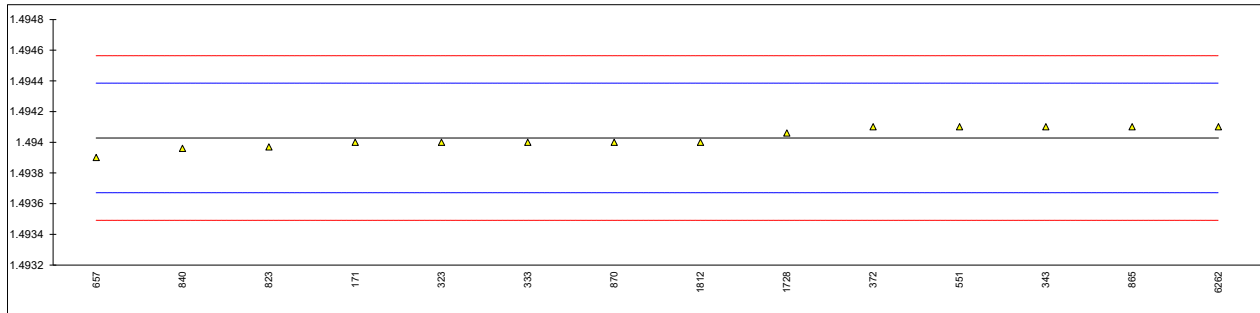
normality not OK  
n 18  
outliers 1  
mean (n) 393.96  
st.dev. (n) 53.408  
R(calc.) 149.54  
st.dev.(Horwitz 3 comp) 44.410  
R(Horwitz 3 comp) 124.35



Determination of Refractive Index at 25 °C on sample #23021;

lab	method	value	mark	z(targ)	remarks
52		----		----	
150		----		----	
171	D1218	1.4940		-0.16	
315		----		----	
323	D1218	1.4940		-0.16	
333	D1218	1.4940		-0.16	
334		----		----	
343	D1218	1.4941		0.40	
372	D1218	1.4941		0.40	
396		----		----	
445		----		----	
551	D1218	1.4941		0.40	
555		----		----	
657	D1218	1.49390		-0.72	
823	D1218	1.49397		-0.32	
840	D1218	1.49396		-0.38	
855		----		----	
862		----		----	
864		----		----	
865	D1218	1.4941		0.40	
866		----		----	
870	D1218	1.4940		-0.16	
902		----		----	
912		----		----	
913		----		----	
1011		----		----	
1062		----		----	
1728	D1218	1.49406		0.18	
1812	D1218	1.4940		-0.16	
6203		----		----	
6262	D1218	1.4941		0.40	
6321		----		----	

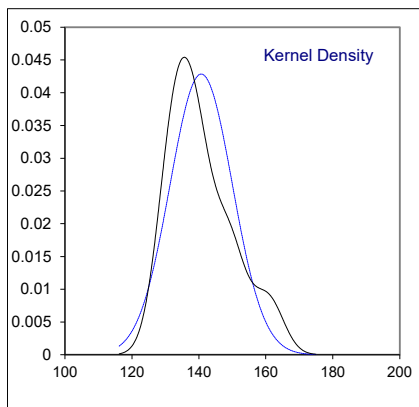
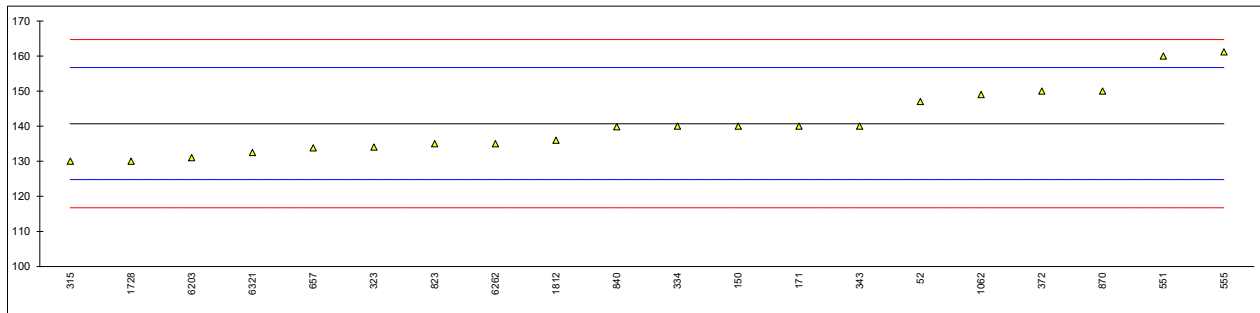
normality OK  
n 14  
outliers 0  
mean (n) 1.49403  
st.dev. (n) 0.000065  
R(calc.) 0.00018  
st.dev.(D1218:21) 0.000179  
R(D1218:21) 0.0005



Determination of Water on sample #23021; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	E1064	147		0.79	
150	E1064	140		-0.09	
171	E1064	140		-0.09	
315	D7375	130		-1.34	
323	E1064	134		-0.84	
333		----		----	
334	E1064	140		-0.09	
343	E1064	140		-0.09	
372	D6304	150		1.16	
396		----		----	
445		----		----	
551	E1064	160		2.41	
555	D4017	161.181		2.56	
657	E1064	133.8		-0.87	
823	E1064	135		-0.72	
840	E1064	139.8		-0.11	
855		----		----	
862		----		----	
864		----		----	
865		----		----	
866		----		----	
870	E1064	150		1.16	
902		----		----	
912		----		----	
913		----		----	
1011		----		----	
1062	D6304	149		1.04	
1728	E1064	130		-1.34	
1812		136		-0.59	
6203	ISO12937	131		-1.22	
6262	E1064	135		-0.72	
6321	E1064	132.5		-1.03	

normality	OK	<u>E1064 only:</u>
n	20	not OK
outliers	0	14
mean (n)	140.71	139.79
st.dev. (n)	9.308	7.986
R(calc.)	26.06	22.36
st.dev.(E1064:16)	7.991	7.938
R(E1064:16)	22.37	22.23



## **APPENDIX 2**

### **Number of participants per country**

3 labs 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  
1 lab in GERMANY  
2 labs in INDIA  
1 lab in ITALY  
1 lab in KOREA, Republic of  
1 lab in NETHERLANDS  
1 lab in PORTUGAL  
2 labs in ROMANIA  
1 lab in SINGAPORE  
1 lab in SPAIN  
1 lab in TURKEY  
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 test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?
SDS	= Safety Data Sheet

### Literature

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