

Institute for  
Interlaboratory Studies

## Results of Proficiency Test Toluene February 2022

**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 in accordance with the latest version of ASTM D841 every year. During the annual proficiency testing program 2021/2022 it was decided to continue the round robin for the analysis of Toluene.

In this interlaboratory study 39 laboratories in 21 different 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 1L bottle and labelled #22021.

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 100 liters of Toluene was obtained from a local chemical supplier. After homogenization 60 amber glass bottles of 1L were filled and labelled #22021. 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 #22021-1	0.86679
sample #22021-2	0.86679
sample #22021-3	0.86679
sample #22021-4	0.86679
sample #22021-5	0.86679
sample #22021-6	0.86679
sample #22021-7	0.86678
sample #22021-8	0.86679

Table 1: homogeneity test results of subsamples #22021

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

Table 2: evaluation of repeatability of subsamples #22021

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 sample Toluene labelled #22021 was sent on February 2, 2022. 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) test 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 of 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.

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

Some problems were encountered with the dispatch of the samples due to COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with another two weeks. One participant reported test results after the extended final reporting date and eight participants did not report any test results. Not all participants were able to report all tests requested.

In total 31 participants reported 276 numerical test results. Observed were 12 outlying test results, which is 4.3%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

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

### 4.1 EVALUATION PER 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 reported test results in appendix 1. The abbreviations, used in these tables, are explained in appendix 3.

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). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D1209:05(2019)). In the test results tables of appendix 1 only the method number (sub) and year of adoption or revision (e.g. D1209:05) will be used.

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.

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: This determination was not problematic. All reporting participants 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 and ASTM D1209:05(2019).

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

Density at 20°C: 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 ISO12185:96.

Distillation: This determination was not problematic. In total five 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.

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 D7504:21.

Benzene: This determination may be problematic. One statistical outlier was observed. In ASTM D7504:21 is the given reproducibility 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, it was decided not to calculate z-scores.

Nonaromatics: 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 D7504:21.



**Total Impurities:** This determination may be problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the estimated reproducibility calculated with the Horwitz equation based on 3 components.

**Refractive Index:** 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 D1218:21.

**Water:** 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 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 or as declared by the estimated target reproducibility calculated with the Horwitz equation and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility ( $2.8 \cdot$  standard deviation) and the target reproducibility derived from reference test methods (in casu ASTM and ISO test methods) or estimated using the Horwitz equation are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Acid Wash Color		23	0.6	1.1	2.0
Appearance		26	Pass (B&C)	n.a.	n.a.
Color Pt/Co		22	3.7	4.7	5.2
Copper Corrosion		18	1 (1a/1b)	n.a.	n.a.
Density at 20°C	kg/L	28	0.8668	0.0002	0.0005
Distillation, IBP	°C	21	110.2	0.3	0.6
Distillation, 50% recovered	°C	22	110.6	0.1	0.2
Distillation, DP	°C	23	110.7	0.2	0.5
Purity by GC	%M/M	29	99.939	0.018	0.013
Benzene	mg/kg	25	7.4	3.5	(1.0)
Nonaromatics	mg/kg	28	530	198	479
Total Impurities	mg/kg	27	613	233	181
Refractive Index at 25°C		16	1.4940	0.0003	0.0005
Water	mg/kg	23	125	17	20

Table 3: reproducibilities of tests on sample #22021

For results between brackets no z-scores are calculated

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 reference test methods. The tests that are problematic have been discussed in paragraph 4.1.

### 4.3 COMPARISON OF THE PROFICIENCY TEST OF FEBRUARY 2022 WITH PREVIOUS PTS

	February 2022	February 2021	February 2020	February 2019	March 2018
Number of reporting laboratories	31	40	26	35	36
Number of test results	276	405	253	284	267
Number of statistical outliers	12	10	16	14	10
Percentage of statistical outliers	4.3%	2.5%	6.3%	4.9%	3.8%

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 the following table.

Parameter	February 2022	February 2021	February 2020	February 2019	March 2018
Acid Wash Color (acid layer)	+	++	++	++	++
Color Pt/Co	+	+	+	++	++
Density at 20°C	++	++	++	++	++
Distillation	++	+	+	+	+/-
Purity	-	+/-	--	-	+
Benzene	(--)	--	+/-	(--)	n.e.
Nonaromatics	++	++	++	++	+/-
Total Impurities	-	+/-	+/-	n.e.	n.e.
Refractive Index at 25°C	+	-	-	+	+
Water	+	-	-	n.e.	n.e.

Table 5: comparison determinations against the reference test methods

Results between brackets no z-scores are calculated

The following performance categories were used:

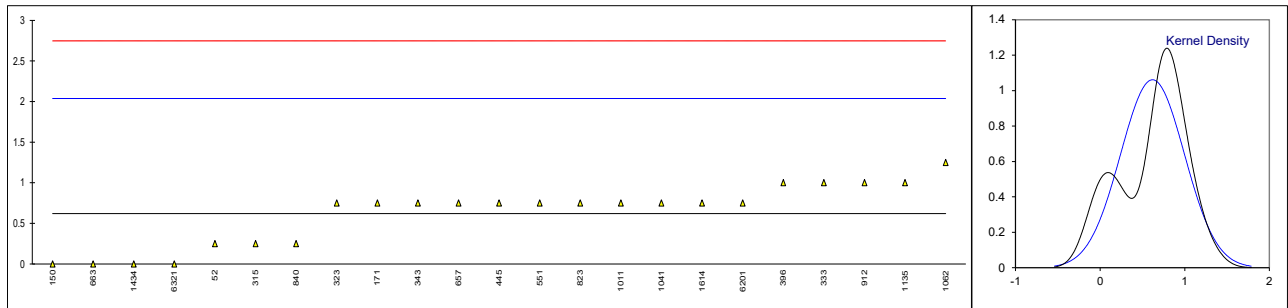
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance equals the reference test method
- : group performed worse than the reference test method
- : group performed much worse than the reference test method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

**APPENDIX 1**

**Determination of Acid Wash Color on sample #22021;**

lab	method	Reported test value	iis conversion *)	mark	z(targ)	remarks
52	D848	0+	0.25		-0.52	
150	D848	0	0		-0.87	
171	D848	1-	0.75		0.18	
315	D848	0+	0.25		-0.52	
323	D848	1-	0.75		0.18	
333	D848	1	1		0.54	
334		----	----		----	
343	D848	1-	0.75		0.18	
396	D848	1	1		0.54	
445	D848	1-	0.75		0.18	
551	D848	1-	0.75		0.18	
555		----	----		----	
657	D848	1-	0.75		0.18	
663	D848	0	0		-0.87	
823	D848	1-	0.75		0.18	
840	D848	0+	0.25		-0.52	
855		----	----		----	
862		----	----		----	
864		----	----		----	
865		----	----		----	
866		----	----		----	
870		----	----		----	
912	D848	1	1		0.54	
913		----	----		----	
1011	D848	1-	0.75		0.18	
1040		----	----		----	
1041	D848	1-	0.75		0.18	
1062	D848	1+	1.25		0.89	
1135	D848	1	1		0.54	
1320		----	----		----	
1434	D848	0	0		-0.87	
1530		----	----		----	
1538		----	----		----	
1614	D848	1-	0.75		0.18	
1707		----	----		----	
6201	D848	1-	0.75		0.18	
6262	D848	PASS	PASS		----	
6315		----	----		----	
6321	D848	0	0		-0.87	
normality			OK			
n			23			
outliers			0			
mean (n)			0.620			
st.dev. (n)			0.3759			
R(calc.)			1.053			
st.dev.(D848:18)			0.7094			
R(D848:18)			1.986			

\*) 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).



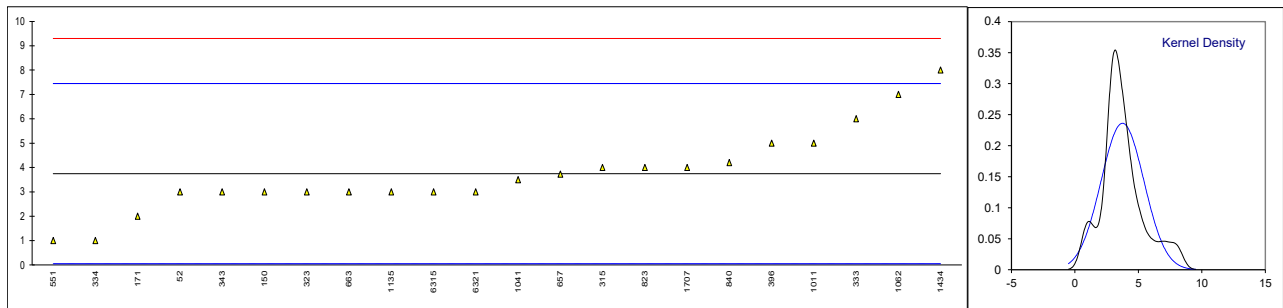
## Determination of Appearance on sample #22021;

lab	method	value	mark	z(targ)	remarks
52	E2680	Pass		----	
150	E2680	Pass		----	
171	E2680	Pass		----	
315	E2680	pass		----	
323	Visual	C&B		----	
333		----		----	
334	EN15769	clear and bright		----	
343	E2680	PASS		----	
396	Visual	Clear & Bright		----	
445	E2680	CBFSM [Pass]		----	
551	E2680	Pass		----	
555		----		----	
657	Visual	Clear & free from suspended solid		----	
663	Visual	B&C		----	
823	E2680	Pass		----	
840	E2680	Pass		----	
855		----		----	
862		----		----	
864		----		----	
865		----		----	
866		----		----	
870		----		----	
912	E2680	Pass		----	
913		----		----	
1011	Visual	Bright and Clear		----	
1040	Visual	clear & bright		----	
1041	Visual	CBFSM		----	
1062	Visual	B&C		----	
1135	D4176	CFSM		----	
1320		----		----	
1434	Visual	cleat liq		----	
1530	Visual	c&b		----	
1538		----		----	
1614	D4176	Clear Liquid Free of Sediments or haze		----	
1707		----		----	
6201	Visual	Br&Cl		----	
6262	Visual	PASS		----	
6315		----		----	
6321	D4176	Clear and free from suspended matter		----	
	n	26			
	mean	Pass (Bright&Clear)			

Determination of Color Pt/Co on sample #22021;

lab	method	value	mark	z(targ)	remarks
52	D5386	3		-0.40	
150	D5386	3		-0.40	
171	D5386	2		-0.94	
315	D5386	4		0.14	
323	D5386	3		-0.40	
333	D5386	6		1.22	
334	D1209	1		-1.48	
343	D5386	3		-0.40	
396	D5386	5		0.68	
445	D1209	<5.0		----	
551	D5386	1		-1.48	
555		----		----	
657	D5386	3.73		-0.01	
663	D5386	3		-0.40	
823	D5386	4		0.14	
840	D5386	4.2		0.24	
855		----		----	
862		----		----	
864		----		----	
865		----		----	
866		----		----	
870		----		----	
912	D5386	<30		----	
913		----		----	
1011	D1209	5		0.68	
1040		----		----	
1041	ISO6271	3.5		-0.13	
1062	D5386	7		1.76	
1135	D5386	3		-0.40	
1320		----		----	
1434	D1209	8		2.30	
1530	D1209	<3		----	
1538		----		----	
1614	D1209	<10		----	
1707	D5386	4		0.14	
6201	D5386	<5		----	
6262	D1209	<5		----	
6315	ISO6271	3		-0.40	
6321	D1209	3		-0.40	

normality suspect  
n 22  
outliers 0  
mean (n) 3.75  
st.dev. (n) 1.690  
R(calc.) 4.73  
st.dev.(D5386:16) 1.850  
R(D5386:16) 5.18  
Compare  
R(1209:05) 7

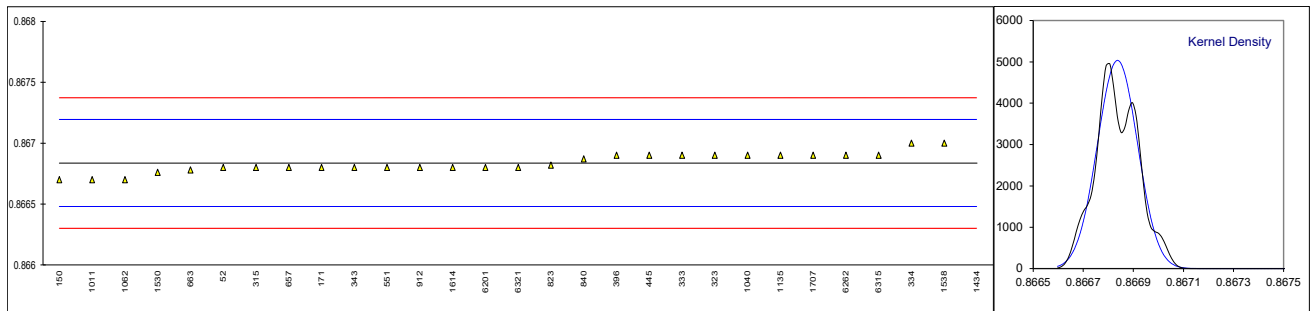


Determination of Copper Corrosion on sample #22021;

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		----		----	
396	D849	Pass		----	
445	D849	1a		----	
551	D849	1A		----	
555		----		----	
657	D849	1a		----	
663	D849	1a		----	
823	D849	1a		----	
840	D849	1A		----	
855		----		----	
862		----		----	
864		----		----	
865		----		----	
866		----		----	
870		----		----	
912		----		----	
913		----		----	
1011		----		----	
1040		----		----	
1041		----		----	
1062	D849	1B		----	
1135	D849	1A		----	
1320		----		----	
1434	D849	1a		----	
1530		----		----	
1538		----		----	
1614	D849	1a		----	
1707		----		----	
6201	D849	1a		----	
6262	D849	1A		----	
6315		----		----	
6321		----		----	
	n	18			
	mean (n)	1 (1a/1b)			

Determination of Density at 20°C on sample #22021: results in kg/L

lab	method	value	mark	z(targ)	remarks
52	D4052	0.8668		-0.21	
150	D4052	0.8667		-0.77	
171	D4052	0.8668		-0.21	
315	D4052	0.8668		-0.21	
323	D4052	0.8669		0.35	
333	D4052	0.8669		0.35	
334	ISO12185	0.8670		0.91	
343	ISO12185	0.8668		-0.21	
396	D4052	0.8669		0.35	
445	D4052	0.8669		0.35	
551	D4052	0.8668		-0.21	
555		----		----	
657	D4052	0.8668		-0.21	
663	D4052	0.86678		-0.32	
823	ISO12185	0.86682		-0.09	
840	D4052	0.86687		0.19	
855		----		----	
862		----		----	
864		----		----	
865		----		----	
866		----		----	
870		----		----	
912	D4052	0.8668		-0.21	
913		----		----	
1011	D4052	0.8667		-0.77	
1040	ISO12185	0.8669		0.35	
1041		----		----	
1062	D4052	0.8667		-0.77	
1135	ISO12185	0.8669		0.35	
1320		----		----	
1434	D4052	0.8703	R(0.01)	19.39	
1530	ISO12185	0.86676		-0.43	
1538	D4052	0.867		0.91	
1614	D4052	0.8668		-0.21	
1707	D4052	0.8669		0.35	
6201	ISO12185	0.8668		-0.21	
6262	D4052	0.8669		0.35	
6315	ISO12185	0.8669	C	0.35	First reported 0.8715
6321	ISO12185	0.8668		-0.21	
normality		OK			
n		28			
outliers		1			
mean (n)		0.86684			
st.dev. (n)		0.000079			
R(calc.)		0.00022			
st.dev.(ISO12185:96)		0.000179			
R(ISO12185:96)		0.0005			

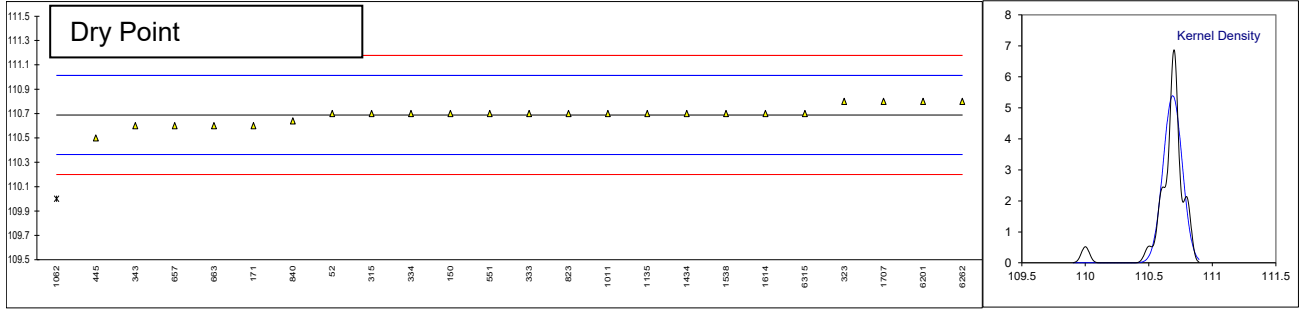
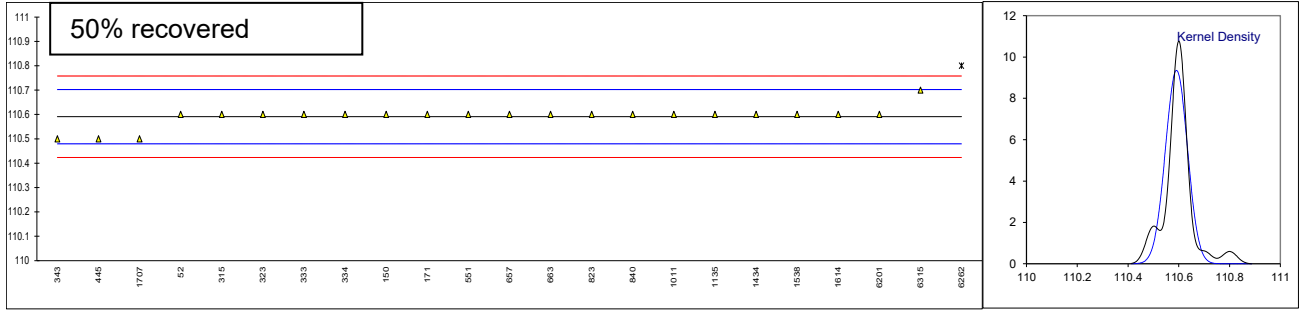
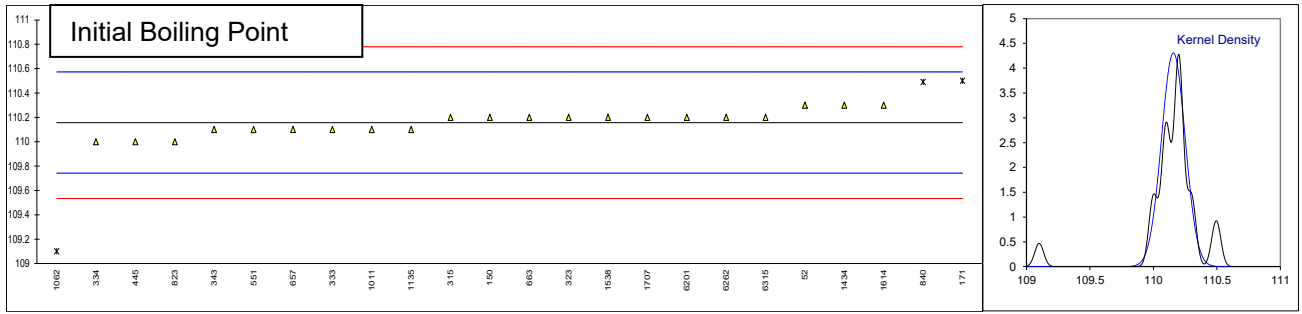


Determination of Distillation on sample #22021; results in °C

Lab	method	IBP	mark	z(targ)	50%rec	mark	z(targ)	DP	mark	z(targ)	range
52	D850-automated	110.3		0.69	110.6		0.16	110.7		0.07	0.4
150	D850-automated	110.2	C	0.21	110.6	C	0.16	110.7	C	0.07	0.5 C
171	D850-automated	110.5	R(5)	1.65	110.6		0.16	110.6		-0.54	0.1
315	D850-automated	110.2		0.21	110.6		0.16	110.7		0.07	0.5
323	D850-automated	110.2		0.21	110.6		0.16	110.8		0.68	0.6
333	D850-automated	110.1		-0.28	110.6		0.16	110.7		0.07	0.6
334	D850-automated	110.0		-0.76	110.6		0.16	110.7		0.07	0.7
343	D850-automated	110.1		-0.28	110.5		-1.63	110.6		-0.54	----
396		----		----	----		----	----		----	<1
445	D850-automated	110.0		-0.76	110.5		-1.63	110.5		-1.16	0.5
551	D850	110.1		-0.28	110.6		0.16	110.7		0.07	0.6
555		----		----	----		----	----		----	----
657	D850-automated	110.1		-0.28	110.6		0.16	110.6		-0.54	0.5
663	D850-automated	110.2		0.21	110.6		0.16	110.6		-0.54	0.4
823	D850-automated	110.0		-0.76	110.6		0.16	110.7		0.07	0.7
840	D850-automated	110.49	R(5)	1.60	110.60		0.16	110.64		-0.30	0.15
855		----		----	----		----	----		----	----
862		----		----	----		----	----		----	----
864		----		----	----		----	----		----	----
865		----		----	----		----	----		----	----
866		----		----	----		----	----		----	----
870		----		----	----		----	----		----	----
912		----		----	----		----	----		----	----
913		----		----	----		----	----		----	----
1011		110.1		-0.28	110.6		0.16	110.7		0.07	0.6
1040		----		----	----		----	----		----	----
1041		----		----	----		----	----		----	----
1062	D850-automated	109.1	R(1)	-5.09	----		----	110.0	R(1)	-4.23	0.9
1135	D850-automated	110.1		-0.28	110.6		0.16	110.7		0.07	0.6
1320		----		----	----		----	----		----	----
1434		110.3		0.69	110.6		0.16	110.7		0.07	0.4
1530		----		----	----		----	----		----	----
1538	D850-automated	110.2		0.21	110.6		0.16	110.7		0.07	----
1614	D850-automated	110.3		0.69	110.6		0.16	110.7		0.07	0.4
1707		110.2		0.21	110.5		-1.63	110.8		0.68	0.6
6201	D850-manual	110.2		0.21	110.6		0.16	110.8		0.68	0.6
6262	D850-automated	110.2		0.21	110.8	R(1)	3.75	110.8		0.68	0.6
6315	D850-automated	110.2		0.21	110.7		1.96	110.7		0.07	0.5
6321		----		----	----		----	----		----	----
	normality	OK			not OK			OK			
	n	21			22			23			
	outliers	3			1			1			
	mean (n)	110.16			110.59			110.69			
	st.dev. (n)	0.093			0.043			0.074			
	R(calc.)	0.26			0.12			0.21			
	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			

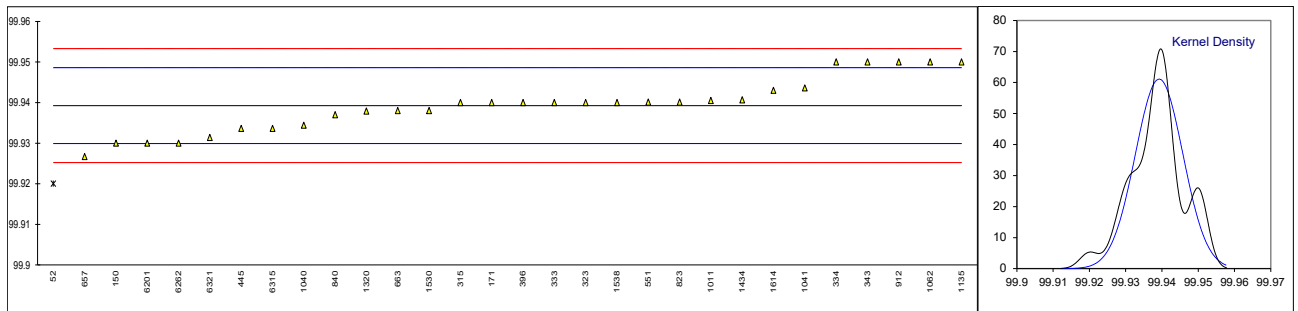
Lab 150: First reported respectively 109.2, 110.3, 111.5, 2.3





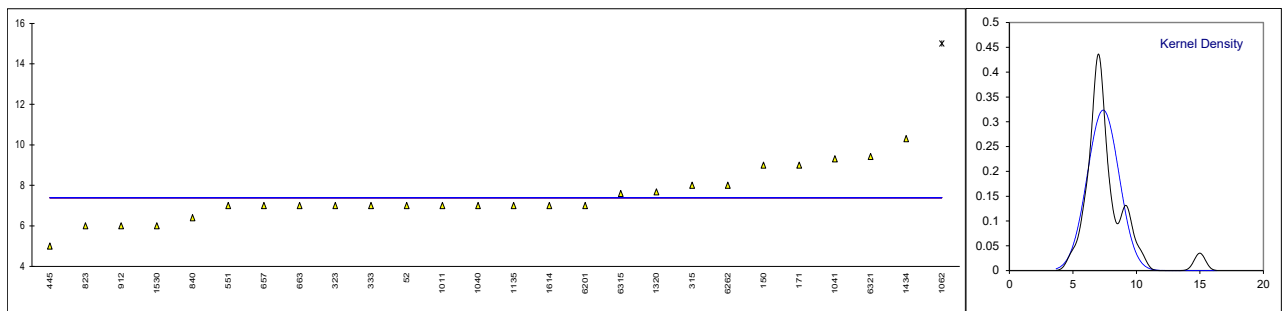
Determination of Purity by GC on sample #22021; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	D7504	99.92	R(0.05)	-4.11	
150	D7504	99.93		-1.97	
171	D7504	99.94		0.16	
315	D7504	99.94		0.16	
323	D7504	99.94		0.16	
333	D7504	99.94		0.16	
334	D7504	99.95		2.30	
343	D2360	99.950		2.30	
396	D7504	99.94		0.16	
445	D6526	99.9336		-1.21	
551	D7504	99.9401		0.18	
555		----		----	
657	D7504	99.9267		-2.68	
663	D7504	99.938		-0.26	
823	D2360	99.9401		0.18	
840	D7504	99.937		-0.48	
855		----		----	
862		----		----	
864		----		----	
865		----		----	
866		----		----	
870		----		----	
912	D7504	99.95		2.30	
913		----		----	
1011		99.9405		0.27	
1040	D7504	99.9344		-1.03	
1041		99.9436		0.93	
1062	D7504	99.95		2.30	
1135	D7504	99.95		2.30	
1320	D7504	99.9379		-0.29	
1434	D7504	99.94065		0.30	
1530	D7504	99.938		-0.26	
1538	D7504	99.94		0.16	
1614	D7504	99.943		0.80	
1707		----		----	
6201	D7504	99.93		-1.97	
6262	D7504	99.93	C	-1.97	First reported 99.9026
6315	D7504	99.9336		-1.21	
6321	D2360	99.9314		-1.68	
normality		OK			
n		29			
outliers		1			
mean (n)		99.93924			
st.dev. (n)		0.006525			
R(calc.)		0.01827			
st.dev.(D7504:21)		0.004679			
R(D7504:21)		0.01310			



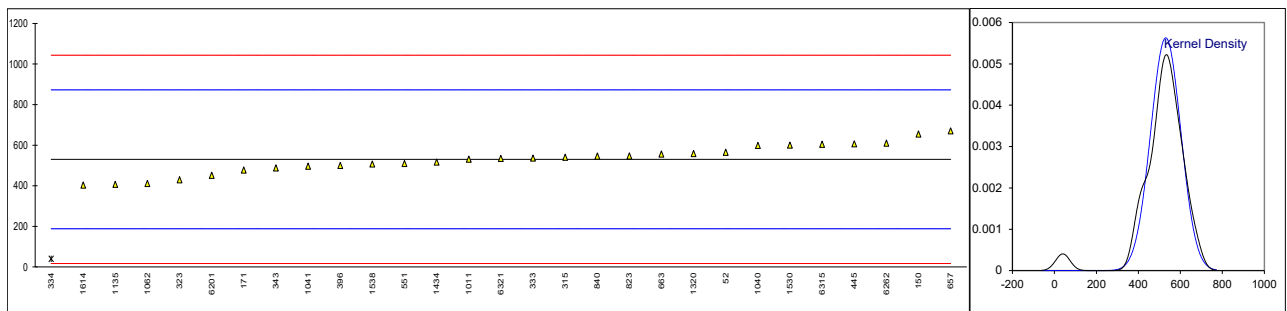
Determination of Benzene on sample #22021; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	D7504	7		----	
150	D7504	9	C	----	First reported 0.0009 mg/kg
171	D7504	9		----	
315	D7504	8		----	
323	D7504	7		----	
333	D7504	7		----	
334	D7504	<2		----	
343	D2360	<10		----	
396	D7504	<100		----	
445	D6526	5		----	
551	D7504	7		----	
555		----		----	
657	D7504	7		----	
663	D7504	7		----	
823	D2360	6		----	
840	D7504	6.4		----	
855		----		----	
862		----		----	
864		----		----	
865		----		----	
866		----		----	
870		----		----	
912	D7504	6		----	
913		----		----	
1011		7		----	
1040	D7504	7		----	
1041		9.31		----	
1062	D7504	15	R(0.01)	----	
1135	D7504	7		----	
1320	D7504	7.68		----	
1434	D7504	10.3	C	----	First reported 0.00103 mg/kg
1530	D6526	6		----	
1538		----		----	
1614	D7504	7		----	
1707		----		----	
6201	D7504	7		----	
6262	D7504	8		----	
6315	D7504	7.6		----	
6321	D2360	9.422		----	
	normality	OK		----	
	n	25			
	outliers	1			
	mean (n)	7.388			
	st.dev. (n)	1.2332			
	R(calc.)	3.453			
	st.dev.(D7504:21)	(0.3651)			
	R(D7504:21)	(1.022)			



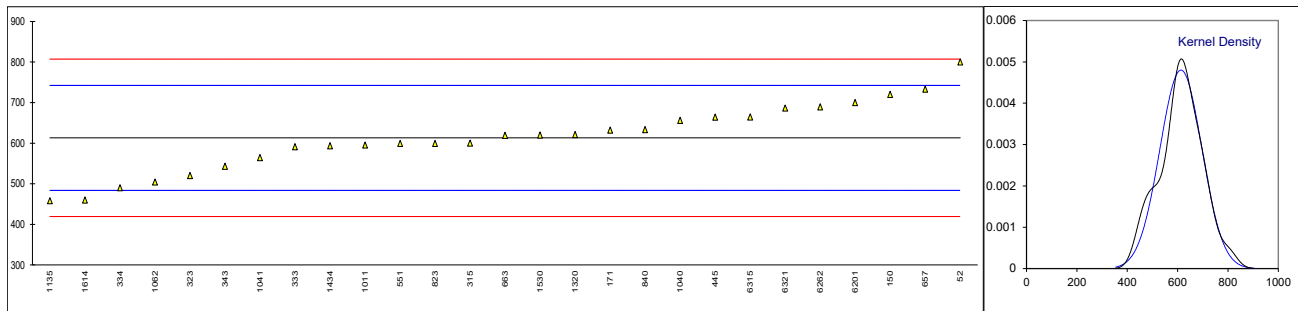
Determination of Nonaromatics on sample #22021; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	D7504	565		0.20	
150	D7504	655	C	0.73	First reported 0.0655 mg/kg
171	D7504	477		-0.31	
315	D7504	540		0.06	
323	D7504	429		-0.59	
333	D7504	536		0.03	
334	D7504	40	R(0.01)	-2.86	
343	D2360	487		-0.25	
396	D7504	500		-0.18	
445	D6526	606		0.44	
551	D7504	509		-0.12	
555		----		----	
657	D7504	670		0.82	
663	D7504	556.5		0.15	
823	D2360	547		0.10	
840	D7504	546.0		0.09	
855		----		----	
862		----		----	
864		----		----	
865		----		----	
866		----		----	
870		----		----	
912		----		----	
913		----		----	
1011		531		0.00	
1040	D7504	598		0.40	
1041		496.06		-0.20	
1062	D7504	411		-0.70	
1135	D7504	407		-0.72	
1320	D7504	558.23		0.16	
1434	D7504	516.4	C	-0.08	First reported 0.05164 mg/kg
1530	D7504	600		0.41	
1538	D7504	507		-0.14	
1614	D7504	403		-0.74	
1707		----		----	
6201	D7504	451		-0.46	
6262	D7504	610	C	0.47	First reported 771
6315	D7504	603.8		0.43	
6321	D2360	534.281		0.02	
normality		OK			
n		28			
outliers		1			
mean (n)		530.37			
st.dev. (n)		70.846			
R(calc.)		198.37			
st.dev.(D7504:21)		171.204			
R(D7504:21)		479.37			



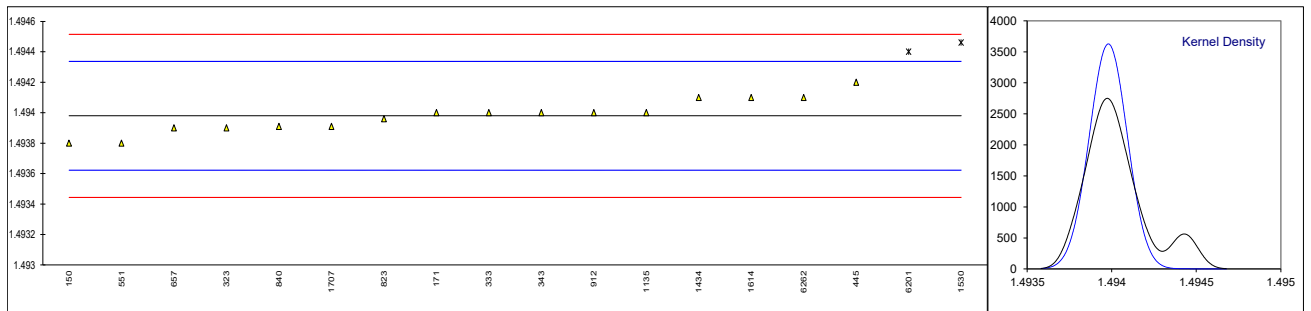
Determination of Total Impurities on sample #22021; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	D7504	800		2.89	
150	D7504	720	C	1.65	First reported 0.072 mg/kg
171	D7504	632		0.29	
315	D7504	600		-0.20	
323	D7504	520		-1.44	
333	D7504	591		-0.34	
334	D7504	490		-1.90	
343	D2360	543		-1.08	
396		----		----	
445	D6526	664		0.79	
551	D7504	599		-0.22	
555		----		----	
657	D7504	733	C	1.85	First reported 56
663	D7504	619		0.09	
823	D2360	599		-0.22	
840	D7504	633.3		0.31	
855		----		----	
862		----		----	
864		----		----	
865		----		----	
866		----		----	
870		----		----	
912		----		----	
913		----		----	
1011		595		-0.28	
1040	D7504	656		0.66	
1041		564.49		-0.75	
1062	D7504	504		-1.69	
1135	D7504	458		-2.40	
1320	D7504	620.98		0.12	
1434	D7504	593.5	C	-0.30	First reported 0.05935 mg/kg
1530	D7504	620		0.11	
1538		----		----	
1614	D7504	460		-2.37	
1707		----		----	
6201	D7504	700		1.34	
6262	D7504	689	C	1.17	First reported 974
6315	D7504	664.3		0.79	
6321	D2360	686.306		1.13	
normality		OK			
n		27			
outliers		0			
mean (n)		613.14			
st.dev. (n)		83.121			
R(calc.)		232.74			
st.dev.(Horwitz 3 comp)		64.666			
R(Horwitz 3 comp)		181.06			



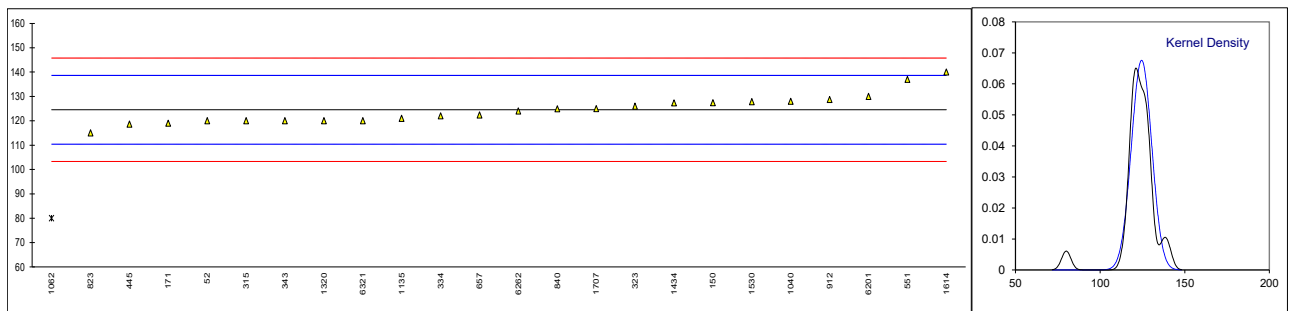
Determination of Refractive Index at 25°C on sample #22021;

lab	method	value	mark	z(targ)	remarks
52		----		----	
150	D1218	1.4938		-1.01	
171	D1218	1.4940		0.11	
315		----		----	
323	D1218	1.4939		-0.45	
333	D1218	1.4940		0.11	
334		----		----	
343	D1218	1.4940		0.11	
396		----		----	
445	D1218	1.4942		1.23	
551	D1218	1.4938		-1.01	
555		----		----	
657	D1218	1.49390		-0.45	
663		----		----	
823	D1218	1.49396		-0.11	
840	D1218	1.49391		-0.39	
855		----		----	
862		----		----	
864		----		----	
865		----		----	
866		----		----	
870		----		----	
912	D1218	1.4940		0.11	
913		----		----	
1011		----		----	
1040		----		----	
1041		----		----	
1062		----		----	
1135	D1218	1.4940		0.11	
1320		----		----	
1434	D1218	1.4941		0.67	
1530	D1218	1.49446	DG(0.05)	2.69	
1538		----		----	
1614	D1218	1.4941		0.67	
1707	D1218	1.49391	C	-0.39	First reported 1.4969
6201	D1218	1.4944	DG(0.05)	2.35	
6262	D1218	1.4941		0.67	
6315		----		----	
6321		----		----	
normality		OK			
n		16			
outliers		2			
mean (n)		1.49398			
st.dev. (n)		0.000110			
R(calc.)		0.00031			
st.dev.(D1218:21)		0.000179			
R(D1218:21)		0.0005			



Determination of Water on sample #22021; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	E1064	120		-0.64	
150	E1064	127.4		0.41	
171	E1064	119		-0.78	
315	D7375	120		-0.64	
323	E1064	126		0.21	
333		----		----	
334	E1064	122		-0.36	
343	E1064	120		-0.64	
396		----		----	
445	E1064	118.6		-0.84	
551	E1064	137		1.76	
555		----		----	
657	E1064	122.4		-0.30	
663		----		----	
823	E1064	115		-1.35	
840	E1064	124.9		0.05	
855		----		----	
862		----		----	
864		----		----	
865		----		----	
866		----		----	
870		----		----	
912	E1064	128.7		0.59	
913		----		----	
1011		----		----	
1040	DIN51777	128		0.49	
1041		----		----	
1062	D6304	80	R(0.01)	-6.30	
1135	E1064	121		-0.50	
1320	E203	120	C	-0.64	First reported 142
1434	D6304	127.3		0.39	
1530	E1064	127.8		0.46	
1538		----		----	
1614	E1064	140	C	2.19	First reported 150
1707	E1064	125		0.07	
6201	E1064	130		0.77	
6262	E1064	124		-0.07	
6315		----		----	
6321	E1064	120		-0.64	
	normality	not OK			
	n	23			
	outliers	1			
	mean (n)	124.53			
	st.dev. (n)	5.901			
	R(calc.)	16.52			
	st.dev.(E1064:16)	7.071			
	R(E1064:16)	19.80			



## APPENDIX 2

### Number of participants per country

4 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  
3 labs in INDIA  
1 lab in ISRAEL  
1 lab in ITALY  
1 lab in KOREA, Republic of  
2 labs in NETHERLANDS  
1 lab in POLAND  
1 lab in PORTUGAL  
1 lab in SINGAPORE  
1 lab in SLOVAKIA  
1 lab in SPAIN  
1 lab in THAILAND  
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) or D(1)	= outlier in Dixon's outlier test
D(0.05) or D(5)	= straggler in Dixon's outlier test
G(0.01) or G(1)	= outlier in Grubbs' outlier test
G(0.05) or G(5)	= straggler in Grubbs' outlier test
DG(0.01) or DG(1)	= outlier in Double Grubbs' outlier test
DG(0.05) or DG(5)	= straggler in Double Grubbs' outlier test
R(0.01) or R(1)	= outlier in Rosner's outlier test
R(0.05) or R(5)	= 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)
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