

**Results of Proficiency Test
Turbine Oil (used)
May 2021**

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2013 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of used Turbine Oil every year. During the annual proficiency testing program 2020/2021 it was decided to continue the round robin for the analysis of used Turbine Oil.

In this interlaboratory study 46 laboratories in 33 different countries registered for participation. See appendix 2 for the number of participants per country. In this report the results of the used Turbine Oil proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send one sample of used Turbine Oil in a one liter bottle labelled #21067. 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, 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 75 liters of used Turbine Oil was obtained from a local supplier. After homogenization 58 amber glass bottles of 1L were filled and labelled #21067. The homogeneity of the subsamples was checked by determination of Density at 15°C in accordance with ISO12185 on 8 stratified randomly selected subsamples.

	Density at 15°C in kg/L
Sample #21067-1	0.87314
Sample #21067-2	0.87314
Sample #21067-3	0.87313
Sample #21067-4	0.87314
Sample #21067-5	0.87314
Sample #21067-6	0.87314
Sample #21067-7	0.87314
Sample #21067-8	0.87313

Table 1: homogeneity test results of subsamples #21067

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

	Density at 15°C in kg/L
r (observed)	0.00001
reference test method	ISO12185:96
0.3 x R (reference test method)	0.00015

Table 2: evaluation of the repeatability of subsamples #21067

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 of used Turbine Oil labelled #21067 was sent on April 21, 2021. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of used Turbine Oil 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: Total Acid Number, Color ASTM, Density at 15°C, Flash Point (C.O.C. and PMcc), Insoluble Color Bodies, Kinematic Viscosity at 40°C and 100°C, Viscosity Index, Oxidation Stability RPVOT, Water, Water Separability at 54°C

distilled water and Level of Contamination (counts/mL and ISO4406 scale). Some extra information was asked about the determination of Total Acid Number.

It was explicitly requested to treat the sample as if it was a routine sample and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

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

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the 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, e.g. ISO reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in

this interlaboratory study.

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. Four participants reported test results after the final reporting date and one other participant was not able to report any test results.

In total 45 participants reported 547 numerical test results. Observed were 24 outlying test results, which is 4.4%. 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. 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. D2270) and an added designation for the year that the test method was adopted or revised (e.g. D2270:10). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D2270:10(2016)). In the test results tables of appendix 1 only the method number and year of adoption or revision (e.g. D2270:10) will be used.

Total Acid Number: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of ASTM D664-A:18e2 for the end point determination Buffer End Point 60mL and Inflection Point 60mL, but is not in agreement for these end point modes at 125mL. Remarkably, two participants still have used pH 11 for BEP instead of pH 10. In test method ASTM D664:18e2 pH 10 is mentioned.

Color ASTM: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with ASTM D1500:12(2017).

Density at 15°C: This determination was problematic for a number of laboratories. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ISO12185:96.

Flash Point C.O.C.: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D92:18.

Flash Point PMcc: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D93:20 procedure A. Only three participants used procedure B.

Insoluble Color Bodies: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D7843:21.

Kinematic Viscosity at 40°C: This determination was not problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D445:21.

Kinematic Viscosity at 100°C: This determination was problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D445:21.

Viscosity Index: This determination was problematic. One statistical outlier was observed and one other test result was excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of ASTM D2270:10(2016).

Oxidation Stability RPVOT: This determination was very problematic. No statistical outliers were observed. The calculated reproducibility is not at all in agreement with the requirements of ASTM D2272:14a.

Water: This determination was problematic. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D6304:20 procedure C and procedure A, but is in agreement with procedure B.

Water Separability at 54°C, distilled water: This determination was not problematic. One statistical outlier was observed over 3 parameters. All calculated reproducibilities after rejection of the statistical outlier are in agreement with the requirements of ASTM D1401:19.

Level of Contamination: This determination was very problematic. Six statistical outliers were observed and fourteen other test results were excluded over 6 parameters. The calculated reproducibilities for the determinations in counts/mL and ISO4406 scale numbers after rejection of the suspect data are not (at all) in agreement with the requirements of ASTM D7647:10(2018), except for counts/mL at $\geq 14 \mu\text{m}$ which is in agreement.

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 ($2.8 \cdot$ standard deviation) and the target reproducibility derived from literature reference test methods (in casu ASTM and ISO test methods) are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Total Acid Number	mg KOH/g	42	0.16	0.09	0.09
Color ASTM		36	5.1	1.2	1
Density at 15°C	kg/L	37	0.87313	0.00045	0.0005
Flash Point C.O.C.	°C	29	234.5	21.1	18
Flash Point PMcc	°C	29	218.9	12.8	15.5
Insoluble Color Bodies		13	14.7	12.5	13.9
Kinematic Viscosity at 40°C	mm ² /s	40	46.31	0.38	0.44
Kinematic Viscosity at 100°C	mm ² /s	40	6.908	0.108	0.057
Viscosity Index		32	104.7	3.6	2
Oxidation Stability RPVOT	minutes	14	510	232	116
Water	mg/kg	34	31.5	32.8	17.6
Water Separability at 54°C, distilled water					
Time \leq 3 mL emulsion	minutes	19	13.5	4.3	20
Time 37 mL water	minutes	17	13.5	4.5	20
Time to complete break	minutes	17	14.1	5.2	20
Level of Contamination					
$\geq 4 \mu\text{m}$ (c)	counts/mL	18	4363	9317	4930
$\geq 6 \mu\text{m}$ (c)	counts/mL	18	502	937	381
$\geq 14 \mu\text{m}$ (c)	counts/mL	16	22	25	30

Parameter	unit	n	average	2.8 * sd	R(lit)
≥ 4 μm (c)	ISO scale	25	19	3	2
≥ 6 μm (c)	ISO scale	25	16	3	1
≥ 14 μm (c)	ISO scale	23	11	3	2

Table 3: reproducibilities of tests on sample #21067

Without further statistical calculations, it can be concluded that for a number of 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 MAY 2021 WITH PREVIOUS PTS

	May 2021	May 2020	May 2019	May 2018	May 2017
Number of reporting laboratories	45	35	42	43	34
Number of test results	547	440	600	581	508
Number of statistical outliers	24	25	13	10	16
Percentage of statistical outliers	4.4%	5.7%	2.2%	1.7%	3.1%

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.

Determination	May 2021	May 2020	May 2019	May 2018	May 2017
Total Acid Number	+/-	-	+	--	-
Color ASTM	-	+	+	+	+
Density at 15°C	+/-	+/-	+	+	+
Flash Point C.O.C.	-	+/-	+/-	-	+
Flash Point PMcc	+	+	+	+	+
Insoluble Color Bodies	+	+	+/-	+	-
Kinematic Viscosity at 40°C	+	-	-	+	-
Kinematic Viscosity at 100°C	-	--	-	+/-	--
Viscosity Index	-	--	-	(--)	--
Oxidation Stability RPVOT	--	--	(--)	-	-
Water	-	++	++	++	++
Water Separability	++	+	+	++	++
Level of Contamination	--	--	(--)	-	--

Table 5: comparison determinations against the reference test methods / () means no z-scores were calculated

The following performance categories were used:

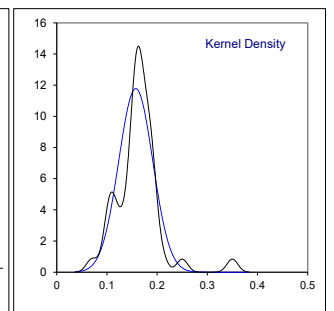
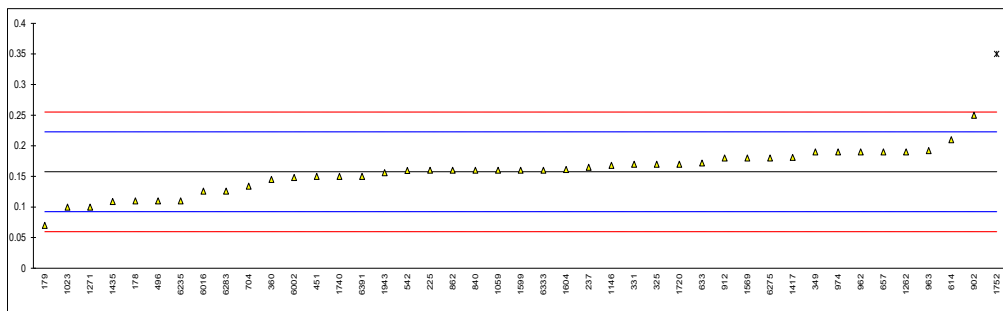
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance equals the reference test method
- : group performed worse than the reference test method
- : group performed much worse than the reference test method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

APPENDIX 1

Determination of Total Acid Number on sample #21067; results in mg KOH/g

lab	method	value	mark	z(targ)	end point determination	volume titration solv.	remarks
178	D664	0.11		-1.46	Inflection Point	60 mL	
179	D664-A	0.07		-2.68	Inflection Point	60 mL	
225	D664-B	0.16		0.08	---	---	
237	D664-A	0.165		0.23	Inflection Point	125 mL	
325	D664-A	0.17	C	0.39	Buffer End Point pH 10	125 mL	first reported 0.07
331	D664Mod.	0.17		0.39	Inflection Point	60 mL	
349	D664-A	0.19		1.00	Buffer End Point pH 10	125 mL	
360	ISO6618	0.145		-0.38	---	---	
432		----		----	---	---	
451	D664-A	0.15		-0.23	Buffer End Point pH 10	60 mL	
496	D664-A	0.11		-1.46	Buffer End Point pH 10	60 mL	
542	D974	0.1597		0.07	Buffer End Point pH 11	125 mL	
614	D664-A	0.21		1.61	---	60 mL	
633	D664-A	0.1718		0.44	Inflection Point	125 mL	
657	D664-A	0.190		1.00	Inflection Point	60 mL	
663		----		----	---	---	
704	D664-A	0.134		-0.72	Buffer End Point pH 10	125 mL	
840	D664-B	0.16		0.08	Buffer End Point pH 10	60 mL	
862	D664-A	0.16		0.08	Inflection Point	60 mL	
902	D664-A	0.25		2.84	Inflection Point	60 mL	
912	D664-A	0.18		0.69	---	---	
962	D664-A	0.19		1.00	---	---	
963	D664-A	0.192		1.06	Inflection Point	60 mL	
974	D664-A	0.19		1.00	Inflection Point	125 mL	
1023	D8045	0.10		-1.76	---	---	
1059	ISO6619	0.16		0.08	Buffer End Point pH 11	60 mL	
1146	D664-A	0.168		0.32	Buffer End Point pH 10	125 mL	
1262	ISO6618	0.19		1.00	---	---	
1271	D974	0.10		-1.76	---	---	
1414		----		----	---	---	
1417	D664-A	0.181		0.72	Buffer End Point pH 10	60 mL	
1435	D664-A	0.109		-1.49	Buffer End Point pH 10	---	
1569	D664-A	0.18		0.69	Inflection Point	125 mL	
1599	D664-A	0.16		0.08	---	---	
1604	D664	0.1612		0.12	Inflection Point	128 mL	
1720	D974	0.170		0.39	---	---	
1740	D664-A	0.15		-0.23	Inflection Point	60 mL	
1752	D664-A	0.35	C,R(0.01)	5.91	Buffer End Point pH 10	60 mL	first reported 0.28
1943	ISO6618	0.156		-0.04	Inflection Point	50 mL	
6002	D664-A	0.148		-0.29	Inflection Point	60 mL	
6016	D664-A	0.126		-0.96	Inflection Point	60 mL	
6235	D664-A	0.110		-1.46	Inflection Point	60 mL	
6275	D974	0.18		0.69	---	---	
6283	D664-A	0.126		-0.96	Buffer End Point pH 10	125 mL	
6333	D8045	0.16		0.08	---	60 mL	
6391	ISO6618	0.15		-0.23	---	---	

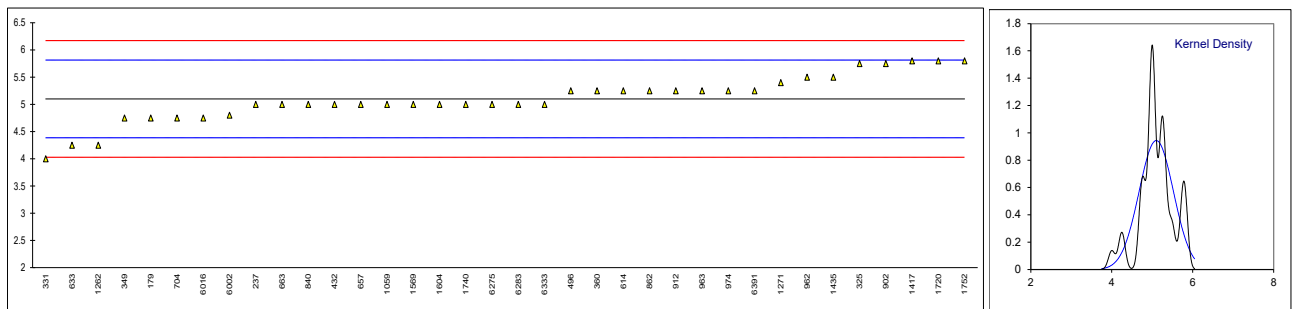
				<u>Buffer End Point only</u>	<u>Inflection Point only</u>
	normality	OK		OK	suspect
	n	42		12	17
	outliers	1		1	0
	mean (n)	0.1574		0.1515	0.1588
	st.dev. (n)	0.03389		0.02640	0.04037
	R(calc.)	0.0949		0.0739	0.1130
	st.dev.(D664-A:18e2)	0.03259		0.03138	---
	R(D664-A:18e2)	0.0912	BEP pH10	60mL	0.0879
Compare:					
	R(D664-A:18e2)	0.0453	BEP pH10	125mL	0.0434
	R(D664-A:18e2)	0.0883	IP	60mL	0.0890
	R(D664-A:18e2)	0.0318	IP	125mL	0.0321



Determination of Color ASTM on sample #21067;

lab	method	reported test result	iis conversion *	mark	z(targ)	remarks
178		----	----		----	
179	D1500	L5.0	4.75		-0.98	
225		----	----		----	
237	D1500	5.0	5.0		-0.28	
325	D6045	L6.0	5.75		1.82	
331	D1500	4	4		-3.08	
349	D1500	<5.0	4.75	C	-0.98	first reported 6.4
360	ISO2049	L5.5	5.25		0.42	
432	D1500	5.0	5.0		-0.28	
451		----	----		----	
496	D1500	5.25	5.25		0.42	
542		----	----		----	
614	D1500	<5.5	5.25		0.42	
633	D1500	L4.5	4.25		-2.38	
657	D1500	5.0	5.0		-0.28	
663	D1500	5.0	5.0		-0.28	
704	D1500	L5.0	4.75		-0.98	
840	D1500	5.0	5.0		-0.28	
862	D1500	L5.5	5.25		0.42	
902	D1500	L6,0	5.75		1.82	
912	D1500	<5.5	5.25		0.42	
962	D1500	5.5	5.5		1.12	
963	D1500	L5.5	5.25		0.42	
974	D1500	L5.5	5.25		0.42	
1023		----	----		----	
1059	D1500	5.0	5.0		-0.28	
1146		----	----		----	
1262	ISO2049	L 4.5	4.25		-2.38	
1271	D6045	5.4	5.4		0.84	
1414		----	----		----	
1417	D6045	5.8	5.8		1.96	
1435	D1500	5.5	5.5		1.12	
1569	D1500	5.0	5.0		-0.28	
1599		----	----		----	
1604	D1500	5	5		-0.28	
1720	D1500	5.8	5.8		1.96	
1740	D1500	5	5		-0.28	
1752	D6045	5.8	5.8		1.96	
1943		----	----		----	
6002	D1500	4.8	4.8		-0.84	
6016	D1500	<5	4.75		-0.98	
6235		----	----		----	
6275	D1500	5.0	5.0		-0.28	
6283	D1500	5.0	5.0		-0.28	
6333	D1500	5.0	5.0		-0.28	
6391	ISO2049	L5.5	5.25		0.42	
normality			OK			
n			36			
outliers			0			
mean (n)			5.10			
st.dev. (n)			0.423			
R(calc.)			1.18			
st.dev.(D1500:12)			0.357			
R(D1500:12)			1			

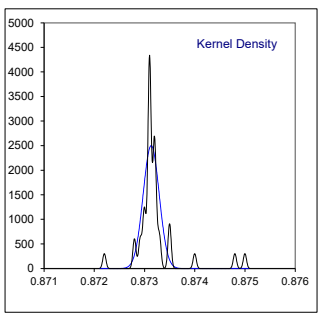
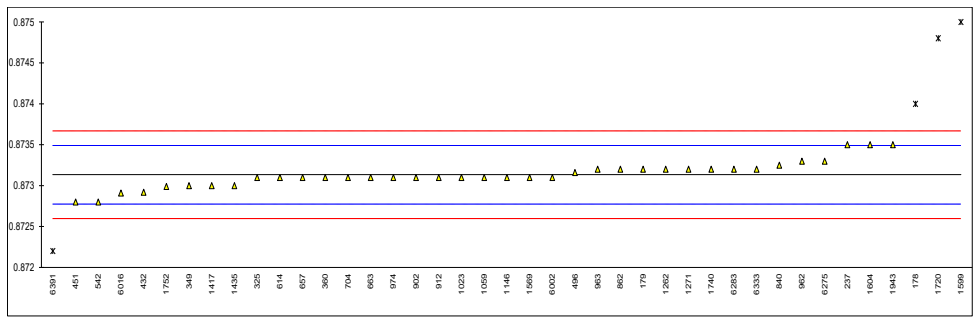
* In the calculation of the mean, standard deviation and the reproducibility in this column, a reported value of 'L y' is changed tot y-0.25 (for example L5.5 into 5.25).



Determination of Density at 15°C on sample #21067; results in kg/L

lab	method	value	mark	z(target)	remarks
178	D1298	0.8740	R(0.01)	4.85	
179	D4052	0.8732		0.37	
225		-----		-----	
237	D4052	0.8735		2.05	
325	D4052	0.8731		-0.19	
331		-----		-----	
349	D4052	0.8730		-0.75	
360	ISO12185	0.8731		-0.19	
432	D4052	0.87292		-1.19	
451	D4052	0.8728		-1.87	
496	ISO12185	0.87316		0.15	
542	D4052	0.8728		-1.87	
614	D4052	0.8731		-0.19	
633		-----		-----	
657	D4052	0.8731		-0.19	
663	D4052	0.87310		-0.19	
704	D4052	0.8731		-0.19	
840	D4052	0.87325		0.65	
862	D4052	0.8732		0.37	
902	D4052	0.8731		-0.19	
912	ISO12185	0.87310		-0.19	
962	ISO12185	0.8733		0.93	
963	D4052	0.8732		0.37	
974	D4052	0.8731		-0.19	
1023	D4052	0.8731	C	-0.19	first reported 873.1 kg/L
1059	ISO12185	0.8731		-0.19	
1146	D4052	0.8731		-0.19	
1262	ISO3675	0.8732	C	0.37	first reported 873.2 kg/L
1271	D4052	0.8732		0.37	
1414		-----		-----	
1417	IP365	0.8730	C	-0.75	first reported 871.3 kg/m ³
1435	D4052	0.8730		-0.75	
1569	ISO12185	0.8731		-0.19	
1599	D4052	0.875	C,R(0.01)	10.45	first reported 0.874
1604	D1298	0.8735		2.05	
1720	D4052	0.8748	C,R(0.01)	9.33	first reported 875.8 kg/m ³
1740	D4052	0.8732		0.37	
1752	D4052	0.87299		-0.80	
1943	ISO3675	0.8735		2.05	
6002	ISO3675	0.8731		-0.19	
6016	D4052	0.87291		-1.25	
6235		-----		-----	
6275	D1298	0.8733		0.93	
6283	D7042	0.8732		0.37	
6333	D7042	0.8732		0.37	
6391	ISO3675	0.8722	C,R(0.01)	-5.23	first reported 872.3 kg/m ³

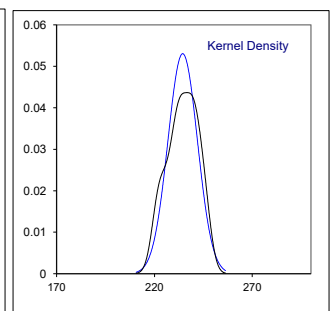
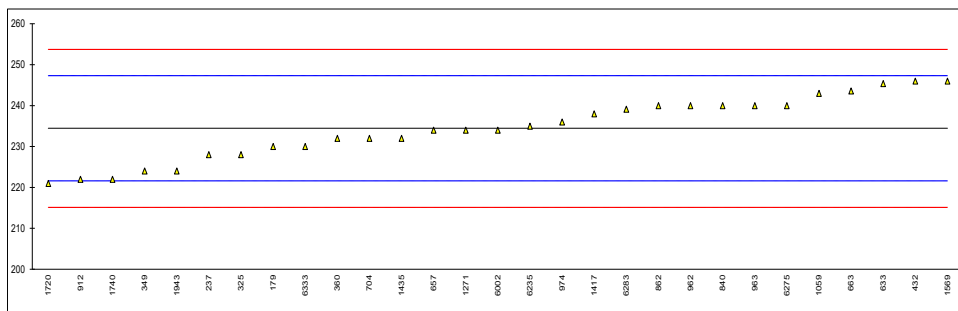
normality suspect
n 37
outliers 4
mean (n) 0.87313
st.dev. (n) 0.000159
R(calc.) 0.00045
st.dev.(ISO12185:96) 0.000179
R(ISO12185:96) 0.0005



Determination of Flash Point C.O.C. on sample #21067; results in °C

lab	method	value	mark	z(targ)	remarks
178		----		----	
179	D92	230		-0.69	
225		----		----	
237	D92	228		-1.00	
325	D92	228		-1.00	
331		----		----	
349	D92	224	C	-1.63	first reported 212
360	D92	232		-0.38	
432	D92	246		1.80	
451		----		----	
496		----		----	
542		----		----	
614		----		----	
633	D92	245.4		1.70	
657	D92	234		-0.07	
663	D92	243.6		1.42	
704	D92	232		-0.38	
840	D92	240		0.86	
862	D92	240		0.86	
902		----		----	
912	D92	222		-1.94	
962	D92	240.0		0.86	
963	D92	240.0		0.86	
974	D92	236		0.24	
1023		----		----	
1059	ISO2592	243		1.33	
1146		----		----	
1262		----		----	
1271	ISO2592	234		-0.07	
1414		----		----	
1417	D92	238		0.55	
1435	D92	232.0		-0.38	
1569	D92	246		1.80	
1599		----		----	
1604		----		----	
1720	D92	221.0		-2.09	
1740	D92	222		-1.94	
1752		----		----	
1943	ISO2592	224		-1.63	
6002	ISO2592	234.0		-0.07	
6016		----		----	
6235	ISO2592	235		0.09	
6275	D92	240		0.86	
6283	D92	239.1		0.72	
6333	D92	230		-0.69	
6391		----		----	

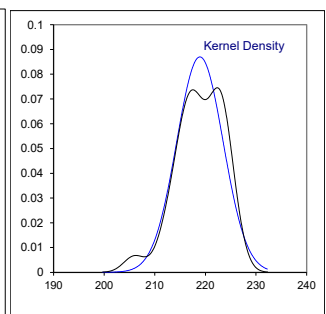
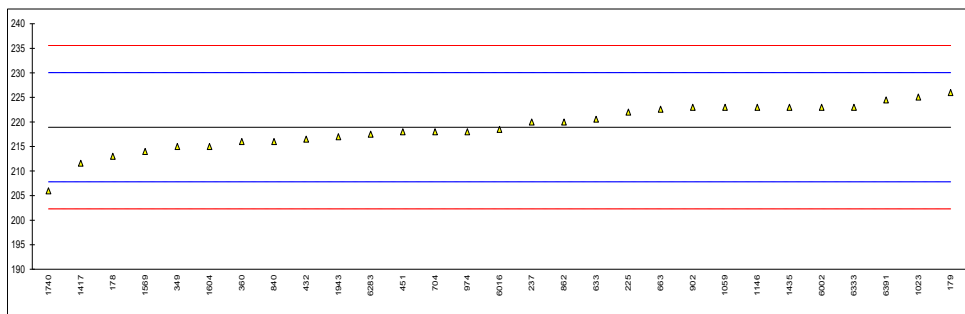
normality OK
n 29
outliers 0
mean (n) 234.45
st.dev. (n) 7.524
R(calc.) 21.07
st.dev.(D92:18) 6.429
R(D92:18) 18



Determination of Flash Point PMcc on sample #21067; results in °C

lab	method	value	mark	z(targ)	remarks
178	D93-A	213		-1.07	
179	D93-A	226.0		1.27	
225	D93-B	222.0		0.55	
237	D93-B	220.0		0.19	
325		----		----	
331		----		----	
349	D93-A	215		-0.71	
360	D93-A	216.0		-0.53	
432	D93-A	216.5		-0.44	
451	D93-A	218		-0.17	
496		----		----	
542	D7094	> 185		----	
614		----		----	
633	D93-A	220.56		0.29	
657		----		----	
663	D93-A	222.6		0.66	
704	D93-A	218.0		-0.17	
840	D93-B	216.0		-0.53	
862	D93-A	220		0.19	
902	D93-A	223.0		0.73	
912		----		----	
962		----		----	
963		----		----	
974	D93-A	218.0		-0.17	
1023	D93-A	225.1		1.11	
1059	ISO2719-A	223.0		0.73	
1146	D93-A	223.0		0.73	
1262		----		----	
1271		----		----	
1414		----		----	
1417	D93-A	211.6		-1.32	
1435	D93-A	223.0		0.73	
1569	D93-A	214		-0.89	
1599		----		----	
1604	D93-A	215		-0.71	
1720		----		----	
1740	D93-A	206		-2.33	
1752		----		----	
1943	ISO2719-A	217		-0.35	
6002	ISO2719-A	223.0		0.73	
6016	D93-A	218.5		-0.08	
6235		----		----	
6275		----		----	
6283	D93-A	217.5		-0.26	
6333	D7094	223		0.73	
6391	ISO2719-A	224.5		1.00	

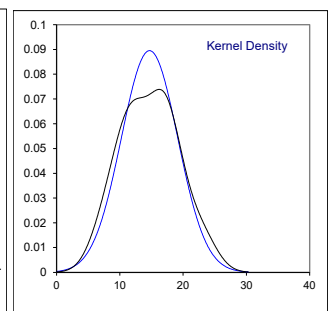
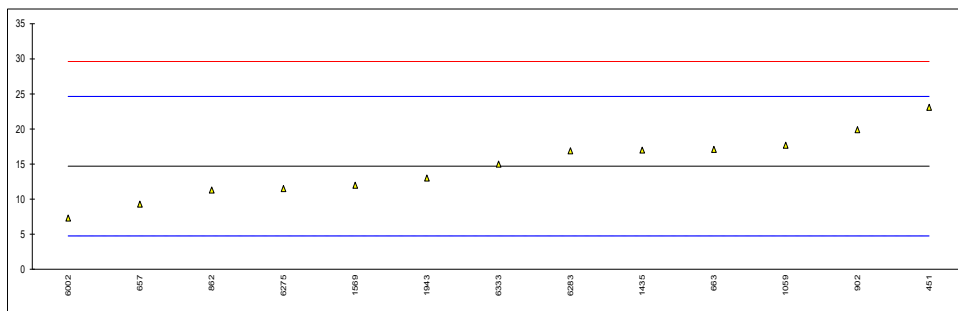
normality OK
n 29
outliers 0
mean (n) 218.93
st.dev. (n) 4.587
R(calc.) 12.84
st.dev.(D93-A:20) 5.551
R(D93-A:20) 15.54



Determination of Insoluble Color Bodies, membrane patch colorimetry on sample #21067;

lab	method	value	mark	z(targ)	remarks
178		----		----	
179	D7843	Normal		----	
225		----		----	
237		----		----	
325		----		----	
331		----		----	
349		----		----	
360		----		----	
432		----		----	
451	D7843	23.1		1.69	
496		----		----	
542		----		----	
614		----		----	
633		----		----	
657	D7843	9.3		-1.08	
663	D7843	17.1		0.48	
704		----		----	
840		----		----	
862	D7843	11.3		-0.68	
902	D7843	19.9		1.04	
912		----		----	
962		----		----	
963		----		----	
974		----		----	
1023		----		----	
1059	D7843	17.7		0.60	
1146		----		----	
1262		----		----	
1271		----		----	
1414		----		----	
1417		----		----	
1435	D7843	17.0		0.46	
1569	D7843	12		-0.54	
1599		----		----	
1604		----		----	
1720		----		----	
1740		----		----	
1752		----		----	
1943	D7843	12.999		-0.34	
6002	D7843	7.3		-1.49	
6016		----		----	
6235		----		----	
6275	D7843	11.5		-0.64	
6283	D7843	16.9		0.44	
6333	D7843	15		0.06	
6391		----		----	

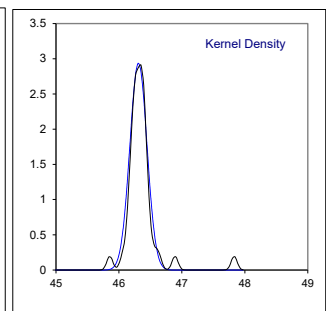
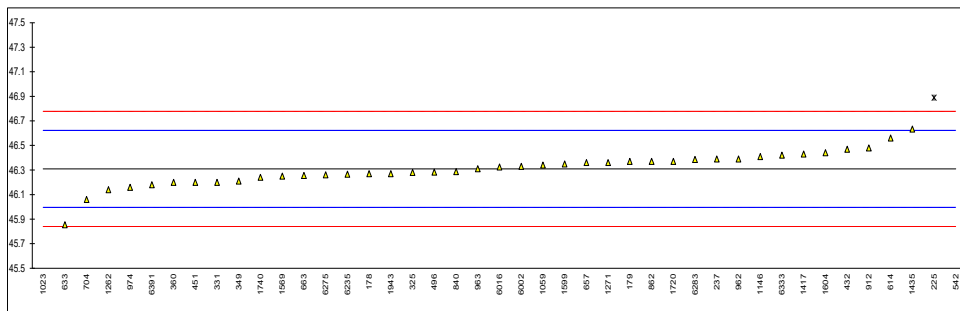
normality OK
n 13
outliers 0
mean (n) 14.70
st.dev. (n) 4.458
R(calc.) 12.48
st.dev.(D7843:21) 4.978
R(D7843:21) 13.94



Determination of Kinematic Viscosity at 40°C on sample #21067; results in mm²/s

lab	method	value	mark	z(targ)	remarks
178	D445	46.27		-0.25	
179	D445	46.37		0.39	
225	D445	46.89	R(0.01)	3.71	
237	D445	46.39		0.52	
325	D445	46.28		-0.19	
331	D7279Mod.	46.2		-0.70	
349	D445	46.21		-0.63	
360	ISO3104	46.20		-0.70	
432	D445	46.47		1.03	
451	D7279 corrected to D445	46.2		-0.70	
496	D445	46.283		-0.17	
542	D7042	47.8294	R(0.01)	9.71	
614	D7042	46.56		1.60	
633	D7279 corrected to D445	45.854		-2.90	
657	D7279	46.36		0.33	
663	D445	46.256		-0.34	
704	D445	46.060	C	-1.59	first reported 45.685
840	D7042	46.286		-0.15	
862	D445	46.37		0.39	
902		----		----	
912	D445	46.48	C	1.09	first reported 46.82
962	D445	46.39		0.52	
963	D445	46.31		0.01	
974	D445	46.16		-0.95	
1023	D445	37.225	R(0.01)	-57.99	
1059	ISO3104	46.34	C	0.20	first reported 6.888
1146	D445	46.41		0.64	
1262	ISO3104	46.14		-1.08	
1271	ISO3104	46.36		0.33	
1414		----		----	
1417	D445	46.43		0.77	
1435	D7042	46.633		2.07	
1569	D445	46.25		-0.38	
1599	D445	46.35		0.26	
1604	D445	46.44		0.84	
1720	D445	46.37		0.39	
1740	D445	46.24		-0.44	
1752		----		----	
1943	ISO3104	46.27		-0.25	
6002	ISO3104	46.33		0.13	
6016	D7042	46.325		0.10	
6235	D445	46.266		-0.27	
6275	D445	46.26		-0.31	
6283	D7042	46.386		0.49	
6333	D7042	46.422		0.72	
6391	ISO3104	46.18		-0.82	

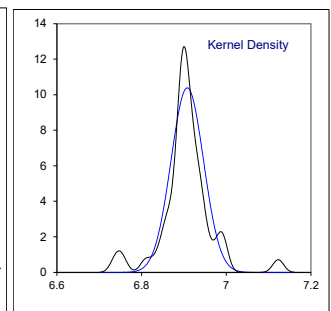
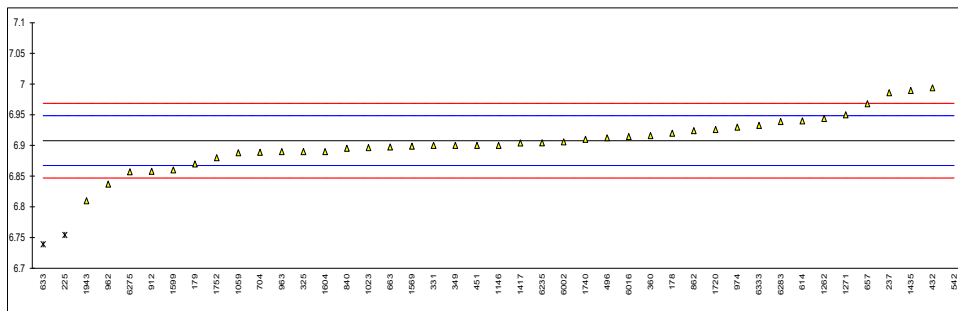
normality not OK
n 40
outliers 3
mean (n) 46.3090
st.dev. (n) 0.13577
R(calc.) 0.3802
st.dev.(D445:21) 0.15664
R(D445:21) 0.4386



Determination of Kinematic Viscosity at 100°C on sample #21067; results in mm²/s

lab	method	value	mark	z(targ)	remarks
178	D445	6.92		0.59	
179	D445	6.87		-1.87	
225	D445	6.754	R(0.05)	-7.58	
237	D445	6.986		3.84	
325	D445	6.890		-0.88	
331	D7279Mod.	6.9		-0.39	
349	D445	6.900		-0.39	
360	ISO3104	6.916		0.40	
432	D445	6.994		4.24	
451	D7279 corrected to D445	6.90		-0.39	
496	D445	6.9125		0.22	
542	D7042	7.1228	R(0.01)	10.58	
614	D7042	6.940		1.58	
633	D7279 corrected to D445	6.7391	R(0.05)	-8.32	
657	D7279	6.968		2.96	
663	D445	6.8975		-0.51	
704	D445	6.889		-0.93	
840	D7042	6.8953		-0.62	
862	D445	6.924		0.79	
902		-----		-----	
912	D445	6.858		-2.46	
962	D445	6.837		-3.49	
963	D445	6.890		-0.88	
974	D445	6.930		1.09	
1023	D445	6.8965		-0.56	
1059	ISO3104	6.888	C	-0.98	first reported 46.34
1146	D445	6.900		-0.39	
1262	ISO3104	6.944		1.78	
1271	ISO3104	6.95		2.07	
1414		-----		-----	
1417	D445	6.904		-0.19	
1435	D7042	6.9896		4.02	
1569	D445	6.899		-0.44	
1599	D445	6.86		-2.36	
1604	D445	6.89		-0.88	
1720	D445	6.926		0.89	
1740	D445	6.910		0.10	
1752	D7279 corrected to D445	6.880		-1.38	
1943	ISO3104	6.81		-4.82	
6002	ISO3104	6.906		-0.10	
6016	D7042	6.9145		0.32	
6235	D445	6.9042		-0.18	
6275	D445	6.857		-2.51	
6283	D7042	6.9391		1.53	
6333	D7042	6.9328		1.22	
6391		-----		-----	

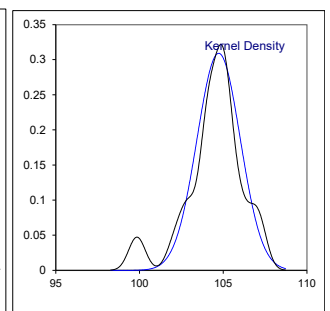
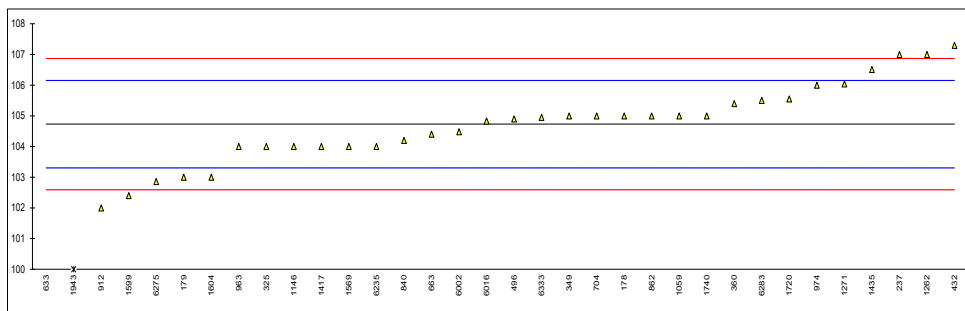
normality OK
n 40
outliers 3
mean (n) 6.9079
st.dev. (n) 0.03841
R(calc.) 0.1075
st.dev.(D445:21) 0.02030
R(D445:21) 0.0568



Determination of Viscosity Index on sample #21067;

lab	method	value	mark	z(targ)	remarks
178	D2270	105		0.38	
179	D2270	103		-2.42	
225		----		----	
237	D2270	107		3.18	
325	D2270	104		-1.02	
331		----		----	
349	D2270	105		0.38	
360	ISO2909	105.4		0.94	
432	D2270	107.3		3.60	
451		----		----	
496	D2270	104.9		0.24	
542		----		----	
614		----		----	
633	D2270	99.7	ex	-7.04	test result excluded as statistical outlier in Kin. Viscosity 100°C
657		----		----	
663	D2270	104.4		-0.46	
704	D2270	105	C	0.38	first reported 106
840	D2270	104.2		-0.74	
862	D2270	105		0.38	
902		----		----	
912	D2270	102	C	-3.82	first reported 101
962		----		----	
963	D2270	104		-1.02	
974	D2270	106		1.78	
1023		----		----	
1059	ISO2909	105	E	0.38	iis calculated 104
1146	D2270	104		-1.02	
1262	ISO2909	107		3.18	
1271	ISO2909	106.04	C	1.84	first reported 95
1414		----		----	
1417	D2270	104		-1.02	
1435	D2270	106.51		2.49	
1569	D2270	104		-1.02	
1599	D2270	102.4		-3.26	
1604	D2270	103		-2.42	
1720	D2270	105.55		1.15	
1740	D2270	105		0.38	
1752		----		----	
1943	ISO2909	100	C,R(0.05),E	-6.62	first reported 101 / iis calculated 101
6002	ISO2909	104.48		-0.35	
6016	D2270	104.83		0.14	
6235	ISO2909	104	E	-1.02	iis calculated 105
6275	D2270	102.86		-2.62	
6283	D2270	105.5		1.08	
6333	D2270	104.95		0.31	
6391		----		----	

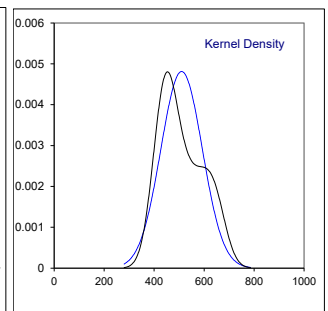
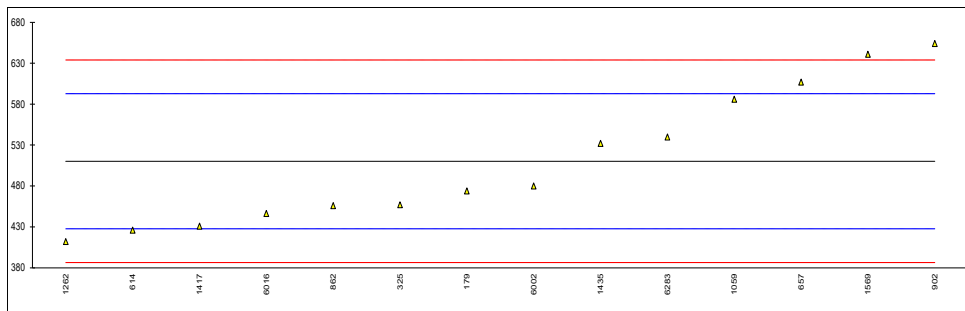
normality OK
n 32
outliers 1 +1ex
mean (n) 104.73
st.dev. (n) 1.291
R(calc.) 3.61
st.dev.(D2270:10) 0.714
R(D2270:10) 2



Determination of Oxidation Stability RPVOT on sample #21067; results in minutes

lab	method	value	mark	z(targ)	remarks
178		----		----	
179	D2272-A	474		-0.88	
225		----		----	
237		----		----	
325	D2272-B	457		-1.29	
331		----		----	
349		----		----	
360		----		----	
432		----		----	
451		----		----	
496		----		----	
542		----		----	
614	D2272-A	426		-2.04	
633		----		----	
657	D2272-B	607		2.35	
663		----		----	
704		----		----	
840		----		----	
862	D2272-A	456		-1.31	
902	D2272-A	654		3.48	
912		----		----	
962		----		----	
963		----		----	
974		----		----	
1023		----		----	
1059	D2272-B	586		1.84	
1146		----		----	
1262	D2272-A	412		-2.38	
1271		----		----	
1414		----		----	
1417	D2272-A	431	C	-1.92	first reported 739.5
1435	D2272-A	532		0.53	
1569	D2272-A	641		3.17	
1599		----		----	
1604		----		----	
1720		----		----	
1740		----		----	
1752		----		----	
1943		----		----	
6002	D2272-B	480		-0.73	
6016	D2272-A	446.5		-1.54	
6235		----		----	
6275		----		----	
6283	D2272-A	540		0.72	
6333		----		----	
6391		----		----	

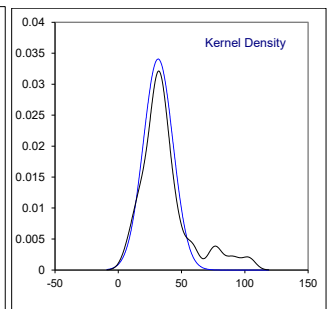
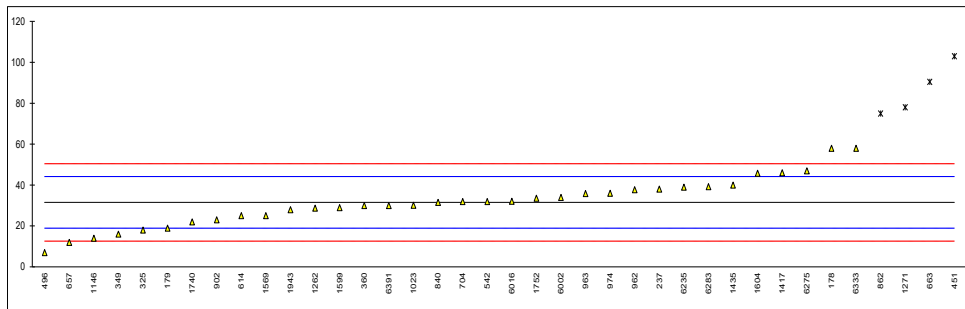
	procedure A only	procedure B only
normality	OK	unknown
n	14	4
outliers	0	0
mean (n)	510.2	532.5
st.dev. (n)	82.89	74.99
R(calc.)	232.1	210.0
st.dev.(D2272:14a)	41.28	43.12
R(D2272:14a)	115.6	120.7



Determination of Water on sample #21067; results in mg/kg

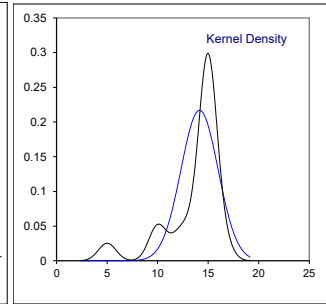
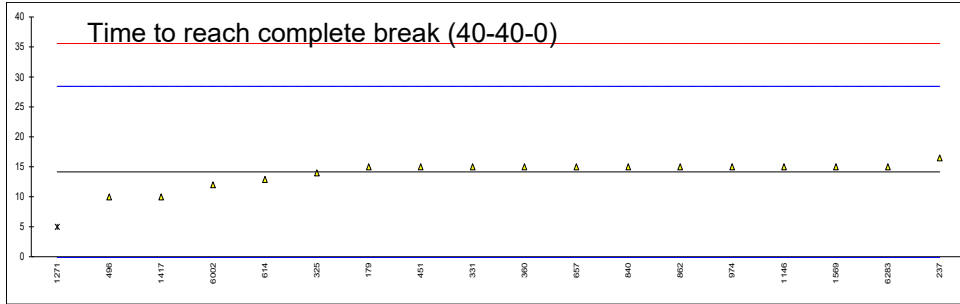
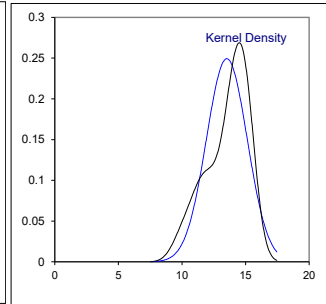
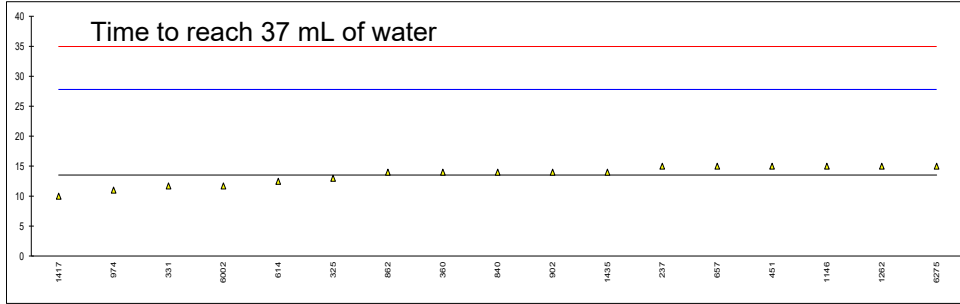
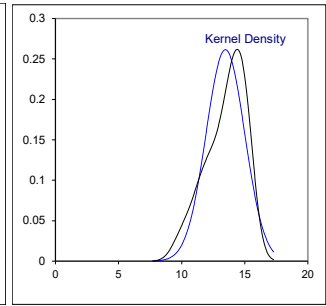
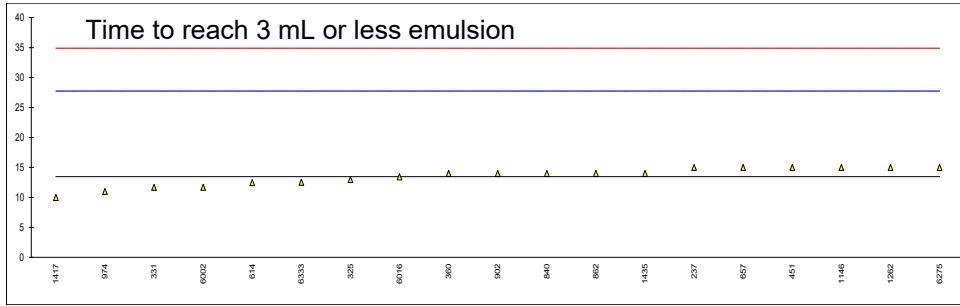
lab	method	value	mark	z(targ)	remarks
178	D6304-C	58		4.20	
179	D6304-C	19		-1.99	
225		----		----	
237	D6304-C	38		1.03	
325	D6304-C	18		-2.15	
331		----		----	
349	D6304-A	16		-2.47	
360	D6304-A	30.0		-0.24	
432		----		----	
451	D6304-B	103	C,R(0.05)	11.35	first reported 206
496	D6304-C	7		-3.90	
542	D6304-A	31.98		0.07	
614	D6304-B	25		-1.04	
633		----		----	
657	D6304-C	12		-3.10	
663	D6304-C	90.45	R(0.05)	9.36	
704	D6304-A	31.97		0.07	
840	D6304-B	31.5		-0.01	
862	D6304-B	75	R(0.05)	6.90	
902	D6304-C	23		-1.36	
912		----		----	
962	D6304-C	37.74		0.98	
963	D6304-A	35.8		0.68	
974	D6304-A	36		0.71	
1023	D6304-A	30.1		-0.23	
1059	D6304-B	<30		----	
1146	D6304-C	14		-2.79	
1262	ISO760	28.7		-0.45	
1271	ISO12937	78	R(0.05)	7.38	
1414		----		----	
1417	D6304-A	46		2.30	
1435	D6304-A	40		1.34	
1569	D6304-C	25		-1.04	
1599	D6304-A	29		-0.40	
1604	D6304	45.8		2.26	
1720		----		----	
1740	D6304-B	22		-1.52	
1752	D6304-A	33.6		0.33	
1943	EN60814	27.91		-0.58	
6002	In house	33.97		0.39	
6016	D6304-A	32.11		0.09	
6235	ISO12937	39		1.18	
6275	D6304-A	47		2.46	
6283	EN60814	39.2		1.22	
6333	D6304-C	58		4.20	
6391	ISO12937	30		-0.24	

normality OK
 n 34
 outliers 4
 mean (n) 31.54
 st.dev. (n) 11.709
 R(calc.) 32.79
 st.dev.(D6304-C:20) 6.297
 R(D6304-C:20) 17.63 range 20 - 360 mg/kg
 Compare
 R(D6304-A:20) 26.67 range 20 - 25000 mg/kg
 R(D6304-B:20) 96.58 range 30 - 2100 mg/kg



Determination of Water Separability at 54°C, distilled water on sample #21067; results in minutes

lab	method	3 mL or less emulsion	z(targ)	37 mL of water	z(targ)	complete break (40-40-0)	z(targ)	test aborted	time aborted
178		----	----	----	----	----	----	----	----
179	D1401	----	----	----	----	15	0.12	----	----
225		----	----	----	----	----	----	----	----
237	D1401	15.0	0.21	15.0	0.21	16.5	0.33	No	----
325	D1401	13	-0.07	13	-0.07	14	-0.02	----	----
331	ISO6614	11.7	-0.25	11.7	-0.26	15	0.12	No	15
349		----	----	----	----	----	----	----	----
360	D1401	14.0	0.07	14.0	0.07	15.0	0.12	No	----
432		----	----	----	----	----	----	----	----
451	D1401	15	0.21	15	0.21	15	0.12	No	----
496	D1401	----	----	----	----	10	-0.58	No	----
542		----	----	----	----	----	----	----	----
614	D1401	12.5	-0.14	12.5	-0.14	12.9	-0.17	No	----
633		----	----	----	----	----	----	----	----
657	D1401	15	0.21	15	0.21	15	0.12	No	----
663		----	----	----	----	----	----	----	----
704		----	----	----	----	----	----	----	----
840	D1401	14	0.07	14	0.07	15	0.12	Yes	15
862	D1401	14	0.07	14	0.07	15	0.12	No	15
902	D1401	14	0.07	14	0.07	----	----	No	30
912		----	----	----	----	----	----	----	----
962		----	----	----	----	----	----	----	----
963		----	----	----	----	----	----	----	----
974	D1401	11	-0.35	11	-0.35	15	0.12	No	----
1023		----	----	----	----	----	----	----	----
1059		----	----	----	----	----	----	----	----
1146	D1401	15	0.21	15	0.21	15	0.12	Yes	15
1262	ISO6614	15	0.21	15	0.21	----	----	Yes	40
1271	ISO6614	----	----	----	----	5	G(0.01) -1.28	----	----
1414		----	----	----	----	----	----	----	----
1417	D1401	10	-0.49	10	-0.49	10	-0.58	No	----
1435	D1401	14	0.07	14	0.07	----	----	No	----
1569	D1401	----	----	----	----	15	0.12	No	----
1599		----	----	----	----	----	----	----	----
1604		----	----	----	----	----	----	----	----
1720		----	----	----	----	----	----	----	----
1740		----	----	----	----	----	----	----	----
1752		----	----	----	----	----	----	----	----
1943		----	----	----	----	----	----	----	----
6002	ISO6614	11.7	-0.25	11.7	-0.26	12	-0.30	No	----
6016	D1401	13.46	0.00	----	----	----	----	No	----
6235		----	----	----	----	----	----	----	----
6275	D1401	15	0.21	15	0.21	----	----	Yes	30
6283	D1401	----	----	----	----	15	0.12	Yes	15
6333	D1401	12.53	-0.13	----	----	----	----	Yes	15
6391		----	----	----	----	----	----	----	----
	normality	OK		OK		suspect			
	n	19		17		17			
	outliers	0		0		1			
	mean (n)	13.47		13.52		14.14			
	st.dev. (n)	1.526		1.600		1.839			
	R(calc.)	4.27		4.48		5.15			
	st.dev.(D1401:19)	7.143		7.143		7.143			
	R(D1401:19)	20		20		20			



Determination of Water Separability at 54°C, distilled water on sample #21067; results in mL

lab	method	volume oil phase	mark	volume water phase	mark	volume emulsion phase	mark
178		----		----		----	
179	D1401	40		40		0	
225		----		----		----	
237	D1401	40		40		0	
325		----		----		----	
331	ISO6614	40		40		0	
349		----		----		----	
360	D1401	43		37		0	
432		----		----		----	
451	D1401	40		40		0	
496		----		----		----	
542		----		----		----	
614	D1401	40		40		0	
633		----		----		----	
657	D1401	40		40		0	
663		----		----		----	
704		----		----		----	
840	D1401	40		40		0	
862	D1401	40		40		0	
902	D1401	42		38		0	
912		----		----		----	
962		----		----		----	
963		----		----		----	
974	D1401	40		40		0	
1023		----		----		----	
1059		----		----		----	
1146	D1401	40		40		0	
1262	ISO6614	41		39		0	
1271		----		----		----	
1414		----		----		----	
1417	D1401	40		40		0	
1435		----		----		----	
1569	D1401	42		38		0	
1599		----		----		----	
1604		----		----		----	
1720		----		----		----	
1740		----		----		----	
1752		----		----		----	
1943		----		----		----	
6002	ISO6614	40		40		0	
6016		----		----		----	
6235		----		----		----	
6275	D1401	42		38		0	
6283	D1401	40		40		0	
6333	D1401	44		36		0	
6391		----		----		----	

Determination of Level of Contamination on sample #21067; results in counts/mL

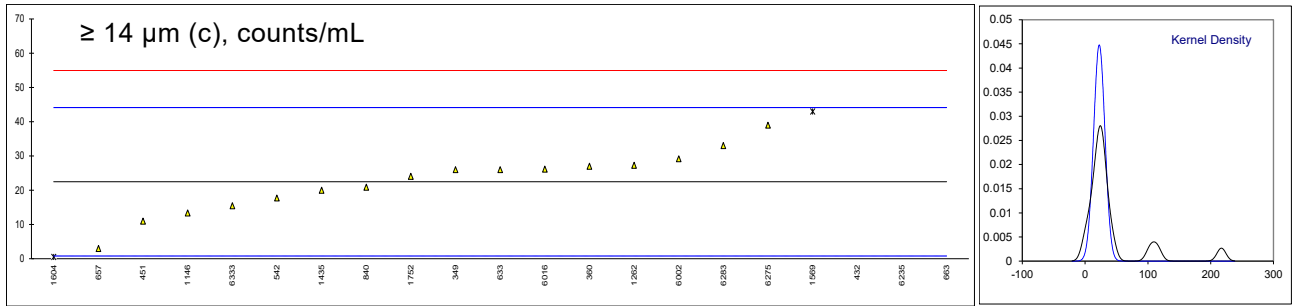
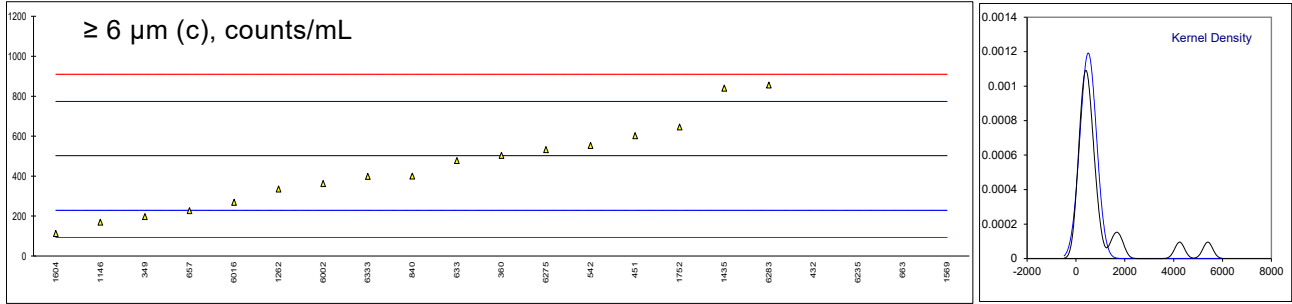
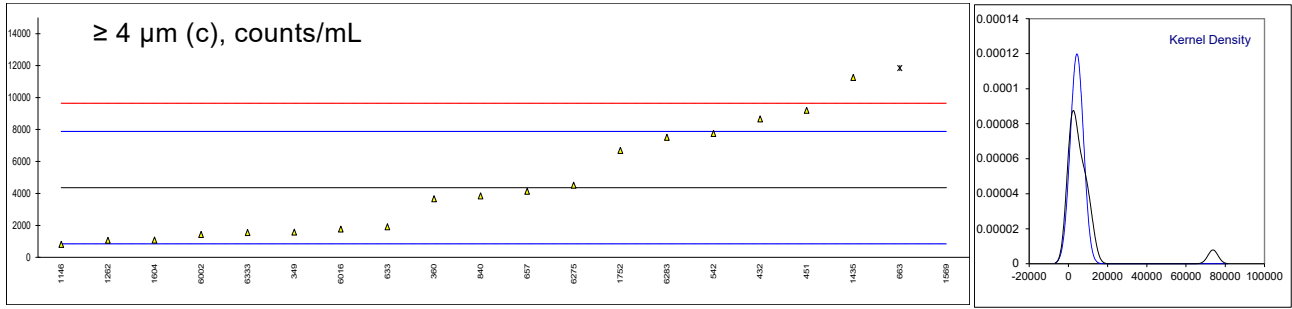
lab	method	≥ 4 µm (c)	mark	z(targ)	≥ 6 µm (c)	mark	z(targ)	≥ 14 µm (c)	mark	z(targ)
178		----		----			----			----
179		----		----			----			----
225		----		----			----			----
237		----		----			----			----
325		----		----			----			----
331		----		----			----			----
349	ISO4406	1585		-1.58	198		-2.23	26		0.33
360	ISO4406	3670		-0.39	504		0.02	27		0.42
432	ISO11500	8662		2.44	1539		7.62	104	G(0.01)	7.53
451	ISO11500	9201		2.75	603		0.74	11		-1.06
496		----		----			----			----
542	ISO11500	7762.13		1.93	554.07		0.38	17.73		-0.44
614		----		----			----			----
633	D7647	1915		-1.39	478		-0.17	26		0.33
657	ISO4406	4145		-0.12	227		-2.02	3		-1.80
663	D7647	11848	ex	4.25	4245	G(0.01)	27.49	217	G(0.01)	17.96
704		----		----			----			----
840	D7647	3851.3		-0.29	400.8		-0.74	20.9		-0.14
862		----		----			----			----
902		----		----			----			----
912		----		----			----			----
962		----		----			----			----
963		----		----			----			----
974		----		----			----			----
1023		----		----			----			----
1059		----		----			----			----
1146	ISO11500	815.00		-2.01	169.93		-2.44	13.40		-0.84
1262	ISO11500	1067.9		-1.87	335.8		-1.22	27.3		0.45
1271		----		----			----			----
1414		----		----			----			----
1417		----		----			----			----
1435	D7647	11261		3.92	841		2.49	20		-0.23
1569		73732	C,ex	39.40	5400	C,ex	35.97	43	C,ex	1.90
1599		----		----			----			----
1604	ISO4406	1093		-1.86	113		-2.85	0.5	ex	-2.03
1720		----		----			----			----
1740		----		----			----			----
1752		6694.2		1.32	646.4		1.06	24.1		0.15
1943		----		----			----			----
6002	D7647	1442.0		-1.66	362.9		-1.02	29.2		0.62
6016	D7596	1769		-1.47	270		-1.70	26.2		0.35
6235	ISO4407	----		----	1805.03	C,G(0.05)	9.57	114.92	C,G(0.05)	8.54
6275	ISO11500	4519		0.09	533		0.23	39		1.53
6283	ISO4407	7519.00		1.79	856.20		2.60	33.07		0.98
6333	D7596	1557.7		-1.59	399.52		-0.75	15.47		-0.65
6391		----		----			----			----
	normality	OK			not OK			OK		
	n	18			18			16		
	outliers	0 +2ex			2 +1ex			3 +2ex		
	mean (n)	4362.7			501.8			22.5		
	st.dev. (n)	3327.36			334.51			8.90		
	R(calc.)	9316.6			936.6			24.9		
	st.dev.(D7647:10)	1760.68			136.19			10.83		
	R(D7647:10)	4929.9			381.3			30.3		

Lab 663 test result excluded at ≥ 4 µm because of two statistical outliers at related measurements for counts/mL

Lab 1569 first reported as scale number, test results excluded as test results in counts/mL and ISO4406 scale number did not match

Lab 1604 test result excluded at ≥ 14 µm because of statistical outlier at related measurement for ISO4406 scale number

Lab 6235 first reported 828.84 at ≥ 6 µm and 58.47 at ≥ 14 µm



Determination of Level of Contamination on sample #21067; results in ISO4406 scale numbers

lab	method	≥ 4 µm (c)	mark	z(targ)	≥ 6 µm (c)	mark	z(targ)	≥ 14 µm (c)	mark	z(targ)
178		----		----	----		----	----		----
179		----		----	----		----	----		----
225		----		----	----		----	----		----
237		----		----	----		----	----		----
325		----		----	----		----	----		----
331		----		----	----		----	----		----
349	ISO4406	18		-1.38	15		-2.05	12		1.03
360	ISO4406	19		0.26	16		0.28	12		1.03
432	ISO4406	20		1.91	18		4.95	14	ex	3.83
451	ISO4406	20		1.91	16		0.28	11		-0.37
496		----		----	----		----	----		----
542	ISO4406	20		1.91	16		0.28	11		-0.37
614	ISO4406	19		0.26	16		0.28	12		1.03
633	ISO4406	18		-1.38	16		0.28	12		1.03
657	ISO4406	19		0.26	15		-2.05	9		-3.17
663	ISO4406	21	ex	3.56	19	ex	7.28	15	ex	5.23
704	ISO4406	19		0.26	16		0.28	13		2.43
840	D7647	19		0.26	16		0.28	12		1.03
862		----		----	----		----	----		----
902		----		----	----		----	----		----
912		----		----	----		----	----		----
962		----		----	----		----	----		----
963		----		----	----		----	----		----
974		----		----	----		----	----		----
1023	ISO4406	20		1.91	16		0.28	10		-1.77
1059		----		----	----		----	----		----
1146		----		----	----		----	----		----
1262	ISO4406	17		-3.03	16		0.28	12		1.03
1271	ISO4406	18		-1.38	15		-2.05	10		-1.77
1414		----		----	----		----	----		----
1417	ISO4406	19	C	0.26	18		4.95	11		-0.37
1435	ISO4406	21		3.56	17		2.61	11		-0.37
1569	ISO4406	20	C,ex	1.91	16	C,ex	0.28	9	C,ex	-3.17
1599		18		-1.38	14		-4.39	9		-3.17
1604	ISO4406	17		-3.03	14		-4.39	6	G(0.05)	-7.37
1720		----		----	----		----	----		----
1740	ISO4406	18		-1.38	15		-2.05	11		-0.37
1752	ISO4406	20		1.91	17		2.61	12		1.03
1943	ISO4406	19		0.26	15		-2.05	10		-1.77
6002	D7647	18		-1.38	16		0.28	12		1.03
6016	ISO4406	18		-1.38	15		-2.05	12		1.03
6235	ISO4406	----		----	18	C,ex	4.95	14	C,ex	3.83
6275	ISO4406	19		0.26	16		0.28	12		1.03
6283	ISO4406	20		1.91	17		2.61	12		1.03
6333	ISO4406	18		-1.38	16		0.28	11		-0.37
6391		----		----	----		----	----		----
normality		OK			OK			OK		
n		25			25			23		
outliers		0 +2ex			0 +3ex			1 +4ex		
mean (n)		18.8			15.9			11.3		
st.dev. (n)		1.03			1.01			1.05		
R(calc.)		2.9			2.8			3.0		
st.dev.(D7647:10)		0.61			0.43			0.71		
R(D7647:10)		1.7			1.2			2.0		

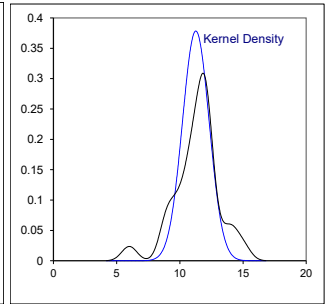
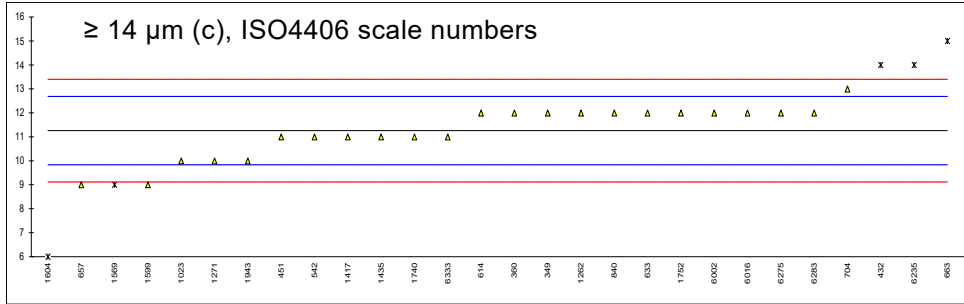
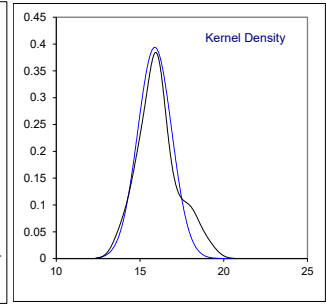
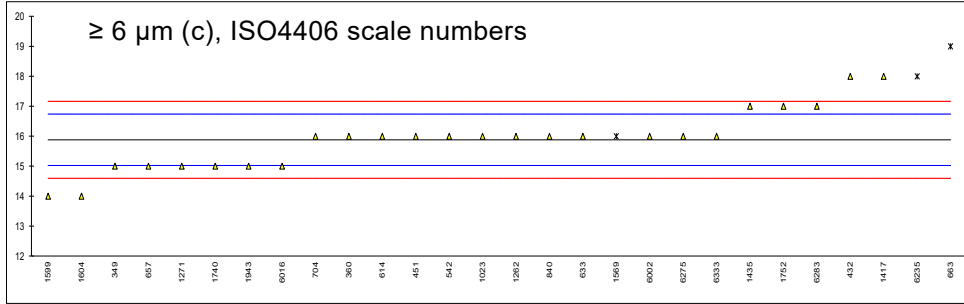
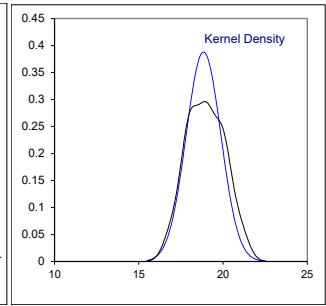
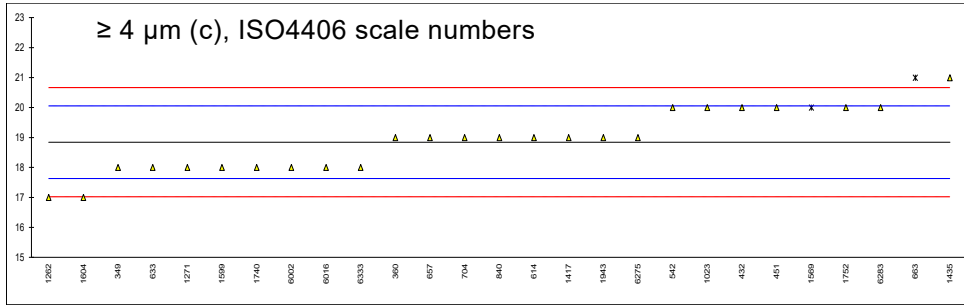
Lab 432 test result excluded at ≥ 14 µm because of statistical outlier at related measurements for counts/mL

Lab 663 test results excluded as statistical outliers and excluded test result at related measurements for counts/mL

Lab 1417 first reported 22

Lab 1569 first reported as counts/mL, test results excluded as test results in counts/mL and ISO4406 scale number did not match

Lab 6235 first reported 17 and 13 respectively, test results excluded as statistical outliers at related measurements for counts/mL



APPENDIX 2

Number of participants per country

1 lab in ARGENTINA
1 lab in AUSTRALIA
1 lab in AUSTRIA
2 labs in BELGIUM
1 lab in BOSNIA and HERZEGOVINA
2 labs in BULGARIA
1 lab in CHINA, People's Republic
1 lab in COTE D'IVOIRE
1 lab in FRANCE
1 lab in GERMANY
1 lab in GREECE
1 lab in INDIA
1 lab in INDONESIA
2 labs in ITALY
1 lab in KAZAKHSTAN
1 lab in LATVIA
1 lab in NETHERLANDS
1 lab in NIGERIA
1 lab in NORWAY
1 lab in PHILIPPINES
4 labs in POLAND
3 labs in SAUDI ARABIA
1 lab in SINGAPORE
1 lab in SLOVENIA
4 labs in SPAIN
1 lab in SUDAN
1 lab in THAILAND
1 lab in TURKEY
1 lab in UKRAINE
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 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)