Results of Proficiency Test
Turbine Oil (used)
May 2020

CONTENTS

Appendices:

1   INTRODUCTION

Since 2013 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for used Turbine Oil every year. During the annual proficiency testing program 2019/2020 it was decided to continue the round robin for the analysis of used Turbine Oil.

In this interlaboratory study 42 laboratories in 30 different countries registered for participation. See appendix 2 for the number of participants per country.
In this report the results of this proficiency test for used Turbine Oil 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 1-liter bottle labelled #20067.
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 60 liters of used Turbine Oil was obtained from a third party. After homogenization 52 amber glass bottles of 1L were filled and labelled #20067.
The homogeneity of the subsamples was checked by the determination of Density in accordance with ISO12185 on 8 stratified randomly selected subsamples.

	Density at 15°C in kg/L
Sample #20067-1	0.85472
Sample #20067-2	0.85471
Sample #20067-3	0.85471
Sample #20067-4	0.85471
Sample #20067-5	0.85471
Sample #20067-6	0.85471
Sample #20067-7	0.85470
Sample #20067-8	0.85471

Table 1: homogeneity test results of subsamples #20067

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 * R (reference test method)	0.00015

Table 2: evaluation of the repeatability of subsamples #20067

The calculated repeatability was 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 bottle of 1 liter of used Turbine Oil, labelled #20067, was sent on April 22, 2020. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of the 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 on sample #20067: Total Acid Number, Color ASTM, Density at 15°C, Flash Point (C.O.C. & PMcc), Insoluble Color Bodies, Kinematic Viscosity at 40°C and at 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 participants 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 original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5).
For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.

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

According to ISO5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

Finally, the reproducibilities were calculated from the standard deviations by multiplying 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 was projected over the Kernel Density Graph for reference.

3.3   Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM or ISO reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation 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. In some cases, a reproducibility based on former iis proficiency tests could be used.

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

The z-scores were calculated according to:

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

The $z_{(target)}$ 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:

$$|z| < 1 \quad \text{good}$$
$$1 < |z| < 2 \quad \text{satisfactory}$$
$$2 < |z| < 3 \quad \text{questionable}$$
$$3 < |z| \quad \text{unsatisfactory}$$

4   EVALUATION

In this interlaboratory study some problems were encountered with the dispatch of the samples due to the COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with another three weeks. After this period still seven participants did not report any test results. Not all participants were able to report test results for all requested tests.

In total 35 participants reported 440 numerical test results. Observed were 25 outlying test results, which is 5.7%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

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

4.1   EVALUATION PER TEST

In this section the reported test results are discussed per test. The test methods, which were used by the various laboratories, were taken into account for explaining the observed differences when possible and applicable. These 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 methods are referred to with a number e.g. D2270 and an added designation for the year that the 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 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 problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D664-A:18e2 regardless of which type of end point determination was used (Buffer End Point or Inflection Point) and what volume of titration solvent was used (60 mL or 125 mL).

Color ASTM: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with ASTM D1500:12(2017).

Density at 15°C: This determination was not problematic. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ISO12185:96 and ASTM D4052:18a.

Flash Point C.O.C.: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full 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 D93:19, procedure A. When evaluated separately for procedure A and procedure B, the respective calculated reproducibilities are in agreement with the requirements of the respective reference test methods for procedure A, but not for procedure B.

Insoluble Color Bodies: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D7843:18.

Kinematic Viscosity at 40°C: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D445:19a.

Kinematic Viscosity at 100°C: This determination was very problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ASTM D445:19a.

Viscosity Index: This determination was very problematic. No statistical outliers were observed but two test results were excluded. However, the calculated reproducibility after rejection of the suspect data is not at all in agreement with the requirements of ASTM D2270:10(2016).

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

Water: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D6304:16e1.

Water Separability at 54°C, distilled water: This determination was not problematic. Four statistical outliers were observed over 3 parameters. However, all calculated reproducibilities after rejection of the statistical outliers are in agreement with the requirements of ASTM D1401:19. Two participants reported deviating test results regarding the volume of the oil/water/emulsion phase.

Level of Contamination: This determination was very problematic. Six statistical outliers were observed and sixteen 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).

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Parameter	unit	n	average	2.8 * sd	R(lit)
Total Acid Number	mg KOH/g	29	0.09	0.08	0.05
Color ASTM		25	2.7	0.6	1
Density at 15°C	kg/L	29	0.8548	0.0005	0.0005
Flash Point C.O.C.	°C	21	235.7	17.0	18
Flash Point PMcc	°C	29	221.0	12.9	15.7
Insoluble Color Bodies		9	12.3	7.2	12
Kinematic Viscosity at 40°C	mm^2/s	32	38.733	0.580	0.323
Kinematic Viscosity at 100°C	mm^2/s	31	6.487	0.121	0.052
Viscosity Index		25	119.1	4.3	2
Oxidation Stability RPVOT	minutes	11	1765	1465	410

Parameter	unit	n	average	2.8 * sd	R(lit)
Water	mg/kg	26	33.5	49.6	138.9
Water Separability at 54°C, distilled water					
Time ≤ 3 mL emulsion	minutes	14	11.9	11.0	20
Time 37 mL water	minutes	12	10.7	6.5	20
Time to complete break	minutes	12	13.1	10.7	20
Level of Contamination					
≥ 4 µm (c)	counts/mL	12	6411	13751	7245
≥ 6 µm (c)	counts/mL	14	297	490	225
≥ 14 µm (c)	counts/mL	14	10	17	13
≥ 4 µm (c)	ISO scale	16	20	4	2
≥ 6 µm (c)	ISO scale	18	15	3	1
≥ 14 µm (c)	ISO scale	18	11	4	2

Table 3: reproducibilities of tests on sample #20067

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 tests that are problematic have been discussed in paragraph 4.1.

4.3   COMPARISON OF THE PROFICIENCY TEST OF MAY 2020 WITH PREVIOUS PTS

	May 2020	May 2019	May 2018	May 2017	May 2016
Number of reporting laboratories	35	42	43	34	38
Number of test results	440	600	581	508	474
Number of statistical outliers	25	13	10	16	15
Percentage of statistical outliers	5.7%	2.2%	1.7%	3.1%	3.2%

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 against the requirements of the respective reference test methods. The conclusions are given in the following table.

Determination	May 2020	May 2019	May 2018	May 2017	May 2016
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	-	-	+	-	++

Determination	May 2020	May 2019	May 2018	May 2017	May 2016
Kinematic Viscosity at 100°C	--	-	+/-	--	+/-
Viscosity Index	--	-	(--)	--	--
Oxidation Stability RPVOT	--	(--)	-	-	++
Water	++	++	++	++	++
Water Separability	+	+	++	++	n.e.
Level of Contamination	--	(--)	-	--	-

Table 5: comparison determinations against the reference test methods

For evaluations 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 Total Acid Number on sample #20067; results in mg KOH/g

lab	method	value	mark	z(targ)	end point determination	volume titration solv.	remarks
178	D664-A	<0.10		-----	Inflection Point	60 mL	
179	D664-A	0.03		-3.15	Inflection Point	60 mL	
225	D974	0.10		0.63	---	---	
237	D664-A	<0.1		-----	Inflection Point	125 mL	
325	D664-A	0.07		-0.99	Buffer End Point pH 10	125 mL	
331	D664Mod.	0.08		-0.45	Inflection Point	60 mL	
349	D664-A	0.09		0.09	Inflection Point	125 mL	
360	D664-A	0.082		-0.34	Inflection Point	60 mL	
432		-----		-----	---	---	
451	D664-A	0.03		-3.15	Buffer End Point pH 10	60 mL	
496	D664-A	0.07		-0.99	Buffer End Point pH 10	60 mL	
542	D974	0.67	R(0.01)	31.38	Buffer End Point pH 11	125 mL	
614	D664-A	0.05		-2.07	---	60 mL	
633		-----		-----	---	---	
657	D664-B	0.107		1.01	Inflection Point	60 mL	
663	D664-A	0.1209		1.76	Buffer End Point pH 10	60 mL	
840	D664-B	0.08		-0.45	Buffer End Point pH 10	60 mL	
862		-----		-----	---	---	
902	D664-A	0.09		0.09	Inflection Point	60 mL	
912		-----		-----	---	---	
962		-----		-----	---	---	
963		-----		-----	---	---	
974		-----		-----	---	---	
1017		-----		-----	---	---	
1023		0.07		-0.99	---	---	
1026	D664-A	0.07		-0.99	Buffer End Point pH 10	125 mL	
1059	ISO6619	0.12		1.71	Buffer End Point pH 11	60 mL	
1146	D664-A	0.102		0.74	Buffer End Point pH 11	125 mL	
1417	D664-A	0.043		-2.44	Buffer End Point pH 10	60 mL	
1431	D664-A	0.0748		-0.73	Inflection Point	60 mL	
1435	D664-A	0.063		-1.37	---	---	
1469		-----		-----	---	---	
1505	D664-A	0.161		3.92	Inflection Point	60 mL	
1569	D664-A	0.10	C	0.63	---	---	first reported 0.48
1599	D664-A	0.1		0.63	Inflection Point	125 mL	
1604	D664-A	0.1036		0.83	Inflection Point	60 mL	
1875	ISO6618	0.1085		1.09	Inflection Point	60 mL	
1941	ISO6619	0.095		0.36	Inflection Point	60 mL	
1957	D664-A	0.11		1.17	Buffer End Point pH 10	125 mL	
2124		-----		-----	---	---	
6016	D664-A	0.130		2.25	---	---	
6333	D8045	0.11		1.17	---	60 mL	

			BEP pH 10 and 11 only	Inflection Point only
normality	OK		OK	not OK
n	29		10	12
outliers	1		1	0
mean (n)	0.0883		0.0816	0.0935
st.dev. (n)	0.02948		0.03127	0.02990
R(calc.)	0.0825		0.0876	0.0837
st.dev.(D664-A:18e2)	0.01854	BEP pH10 60mL	0.01716	---
R(D664-A:18e2)	0.0519	BEP pH10 60mL	0.0480	---
Compare:				
R(D664-A:18e2)	0.0246	BEP pH10 125mL	0.0227	---
R(D664-A:18e2)	0.0550	IP 60mL	---	0.0576
R(D664-A:18e2)	0.0174	IP 125mL	---	0.0185
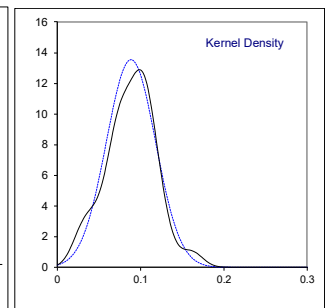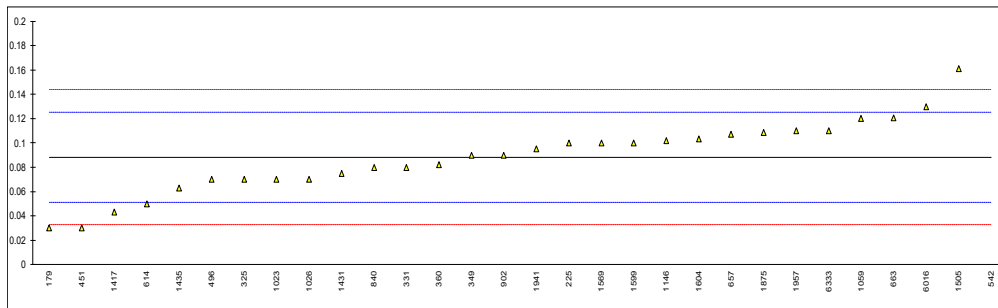
Determination of Color ASTM on sample #20067;

lab	method	reported test value	iis conversion *	mark	z(targ)	remarks
178		-----	-----		-----	
179	D1500	L2.5	2.25		-1.34	
225		-----	-----		-----	
237	D1500	L3.0	2.75		0.06	
325	D6045	L3	2.75		0.06	
331	D1500	2.5	2.5		-0.64	
349	D6045	3.8	3.8	R(0.01)	3.00	
360	D1500	2.5	2.5		-0.64	
432	D1500	L3,0	2.75		0.06	
451		-----	-----		-----	
496	D1500	2.5	2.5		-0.64	
542		-----	-----		-----	
614	D1500	L3.0	2.75		0.06	
633		-----	-----		-----	
657	D1500	3.0	3.0		0.76	
663	D1500	2.5	2.5		-0.64	
840	D6045	2.9	2.9		0.48	
862		-----	-----		-----	
902	D1500	2.6	2.6		-0.36	
912		-----	-----		-----	
962		-----	-----		-----	
963		-----	-----		-----	
974	D1500	L3.0	2.75		0.06	
1017		-----	-----		-----	
1023		-----	-----		-----	
1026	D1500	L3.0	2.75		0.06	
1059	D1500	L3,0	2.75		0.06	
1146		-----	-----		-----	
1417	D6045	2.7	2.7		-0.08	
1431	D1500	3.0	3.0		0.76	
1435	D1500	2.5	2.5		-0.64	
1469		-----	-----		-----	
1505	D1500	3.0	3.0		0.76	
1569	D1500	2.5	2.5		-0.64	
1599		-----	-----		-----	
1604	D1500	3	3		0.76	
1875		-----	-----		-----	
1941	ISO2049	3.0	3.0		0.76	
1957	D1500	3.0	3.0		0.76	
2124		-----	-----		-----	
6016	D1500	L3.0	2.75		0.06	
6333	D1500	L3.0	2.75		0.06	

normality	OK
n	25
outliers	1
mean (n)	2.73
st.dev. (n)	0.208
R(calc.)	0.58
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 L3.0 into 2.75).
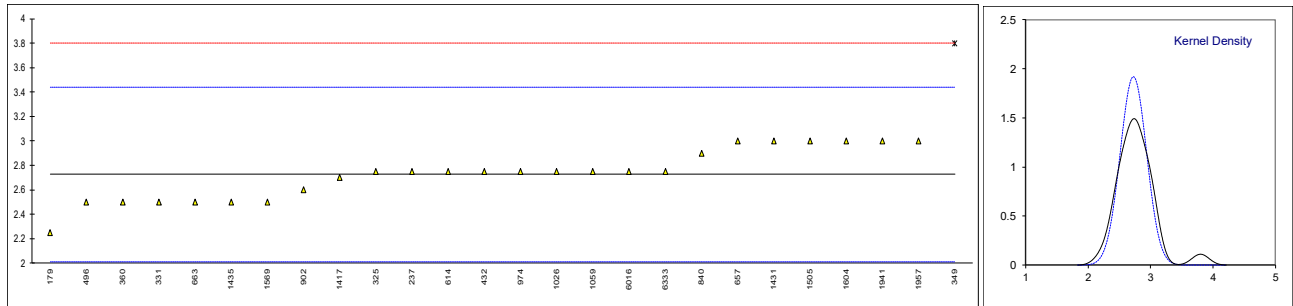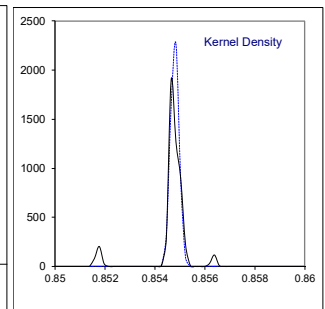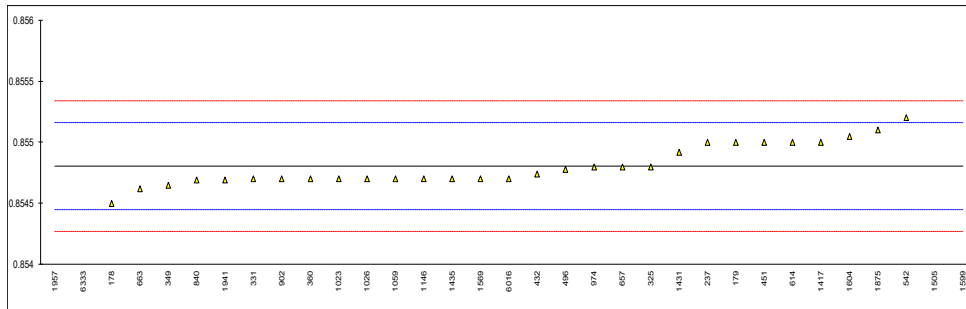
Determination of Density at 15°C on sample #20067; results in kg/L

lab	method	value	mark	z(targ)	remarks
178	D1298	0.8545		-1.71	
179	D4052	0.855		1.09	
225		-----		-----	
237	D4052	0.8550	C	1.09	first reported 858.0 kg/m³
325	D4052	0.8548		-0.03	
331	ISO12185	0.8547		-0.59	
349	D4052	0.85465		-0.87	
360	ISO12185	0.8547		-0.59	
432	D4052	0.85474		-0.36	
451	D4052	0.8550		1.09	
496	ISO12185	0.85478		-0.14	
542	D4052	0.8552		2.21	
614	D4052	0.8550		1.09	
633		-----		-----	
657	D4052	0.8548		-0.03	
663	D4052	0.85462		-1.03	
840	D4052	0.85469		-0.64	
862		-----		-----	
902	D4052	0.8547		-0.59	
912		-----		-----	
962		-----		-----	
963		-----		-----	
974	D4052	0.8548		-0.03	
1017		-----		-----	
1023	D4052	0.8547		-0.59	
1026	D4052	0.8547		-0.59	
1059	ISO12185	0.8547		-0.59	
1146	D4052	0.8547	C	-0.59	first reported 0,8628
1417	IP365	0.8550		1.09	
1431	D4052	0.85492		0.65	
1435	D4052	0.8547		-0.59	
1469		-----		-----	
1505	D7042	0.85636	R(0.01)	8.71	
1569	ISO12185	0.8547		-0.59	
1599	D4052	0.9	C,R(0.01)	253.09	first reported 9000 kg/m³
1604	D1298	0.85505		1.37	
1875	D7042	0.8551		1.65	
1941	D4052	0.85469		-0.64	
1957	D4052	0.8517	C,R(0.01)	-17.39	first reported 0.8540
2124		-----		-----	
6016	D4052	0.8547		-0.59	
6333	D7042	0.8518	R(0.01)	-16.83	

normality	OK
n	29
outliers	4
mean (n)	0.85480
st.dev. (n)	0.000169
R(calc.)	0.00047
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005
Compare:	
R(D4052:18a)	0.00050

Determination of Flash Point C.O.C. on sample #20067; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D92	232		-0.58	
225		-----		-----	
237	D92	236		0.04	
325	D92	236		0.04	
331	D92	242		0.98	
349	D92	242		0.98	
360	ISO2592	234		-0.27	
432	D92	228.0		-1.20	
451		-----		-----	
496	D92	230		-0.89	
542	D7094	>180		-----	
614		-----		-----	
633		-----		-----	
657	D92	232		-0.58	
663	D92	234.75		-0.15	
840	D92	234		-0.27	
862		-----		-----	
902		-----		-----	
912		-----		-----	
962		-----		-----	
963		-----		-----	
974	D92	230		-0.89	
1017		-----		-----	
1023		-----		-----	
1026	D92	234		-0.27	
1059	ISO2592	245		1.44	
1146		-----		-----	
1417	D92	246.0		1.60	
1431	D92	242.4		1.04	
1435	D92	228.0		-1.20	
1469		-----		-----	
1505	D92	238		0.35	
1569	D92	240		0.67	
1599		-----		-----	
1604		-----		-----	
1875		-----		-----	
1941	ISO2592	224		-1.82	
1957		-----		-----	
2124		-----		-----	
6016		-----		-----	
6333	D92	242		0.98	

normality	OK
n	21
outliers	0
mean (n)	235.72
st.dev. (n)	6.074
R(calc.)	17.01
st.dev.(D92:18)	6.429
R(D92:18)	18
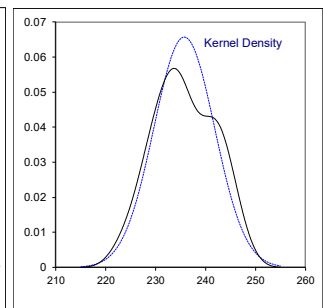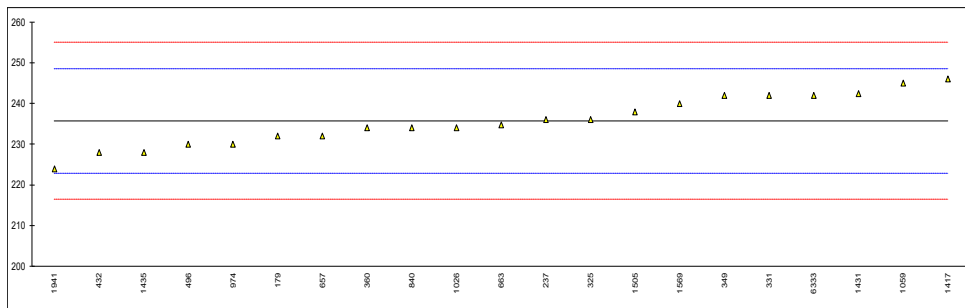
Determination of Flash Point PMcc on sample #20067; results in °C

lab	method	value	mark	z(targ)	remarks
178	D93-A	215.5		-0.99	
179	D93-A	232		1.96	
225	D93-B	224.0		0.53	
237	D93-B	222.0		0.17	
325	D93-A	223		0.35	
331		-----		-----	
349	D93-A	216		-0.90	
360	ISO2719-A	211.0		-1.79	
432	D93-A	221.0		-0.01	
451	D93-A	220		-0.18	
496		-----		-----	
542		-----		-----	
614	D93-A	222		0.17	
633		-----		-----	
657		-----		-----	
663	D93-A	219.72		-0.23	
840	D93-B	212.0		-1.61	
862		-----		-----	
902	D93-A	229.0		1.42	
912		-----		-----	
962		-----		-----	
963		-----		-----	
974	D93-A	216		-0.90	
1017		-----		-----	
1023	D93-A	223		0.35	
1026	D93-A	219.5		-0.27	
1059	ISO2719-A	225.5		0.80	
1146	D93-A	224.7		0.65	
1417	D93-A	219.0		-0.36	
1431	D93-A	221.4		0.07	
1435	D93-A	225.0		0.71	
1469		-----		-----	
1505	D3828	226.5		0.98	
1569	D93-A	222		0.17	
1599		-----		-----	
1604	D93-A	221		-0.01	
1875	ISO2719-B	222		0.17	
1941		-----		-----	
1957	D93-A	215		-1.08	
2124	D93-B	222.2		0.21	
6016	D7094	220		-0.18	
6333	D7094	220		-0.18	

				D93/ISO2719 Procedure A only	D93/ISO2719 Procedure B only
	normality	OK		OK	unknown
	n	29		21	5
	outliers	0		0	0
	mean (n)	221.04		221.02	220.44
	st.dev. (n)	4.613		4.849	4.792
	R(calc.)	12.92		13.58	13.42
	st.dev.(D93-A:19)	5.605		5.604	-----
	R(D93-A:19)	15.69		15.69	-----
Compare:					
	R(D93-B:19)	10		-----	10
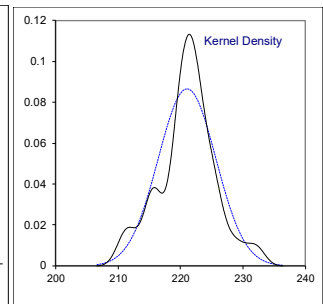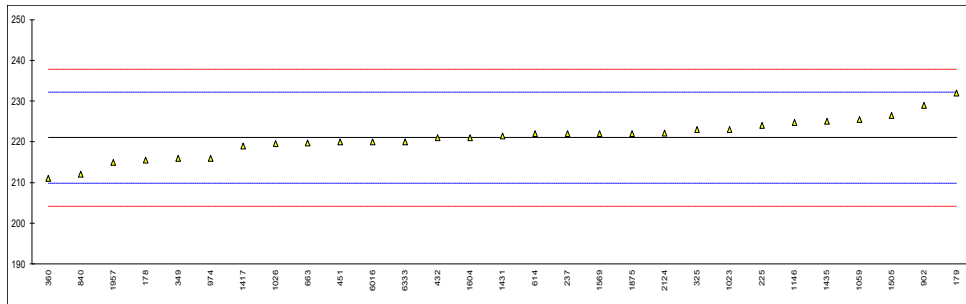
Determination of Insoluble Color Bodies, membrane patch colorimetry on sample #20067;

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
225		-----		-----	
237		-----		-----	
325		-----		-----	
331		-----		-----	
349		-----		-----	
360		-----		-----	
432		-----		-----	
451	D7843	13.1		0.18	
496		-----		-----	
542		-----		-----	
614		-----		-----	
633		-----		-----	
657	D7843	10.6		-0.40	
663	D7843	17.8		1.29	
840		-----		-----	
862		-----		-----	
902	D7843	25.4	DG(0.05)	3.07	
912		-----		-----	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1017		-----		-----	
1023		-----		-----	
1026		-----		-----	
1059	D7843	13.0		0.16	
1146		-----		-----	
1417		-----		-----	
1431		-----		-----	
1435	D7843	12.7		0.09	
1469		-----		-----	
1505	D7843	21	DG(0.05)	2.04	
1569	D7843	13		0.16	
1599		-----		-----	
1604		-----		-----	
1875	D7843	9.6		-0.64	
1941		-----		-----	
1957	D7843	9.1		-0.76	
2124		-----		-----	
6016		-----		-----	
6333	D7843	12		-0.08	

normality	not OK	
n	9	
outliers	2	
mean (n)	12.32	
st.dev. (n)	2.563	
R(calc.)	7.18	
st.dev.(D7843:18)	4.260	
R(D7843:18)	12	
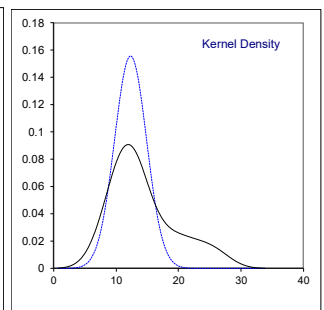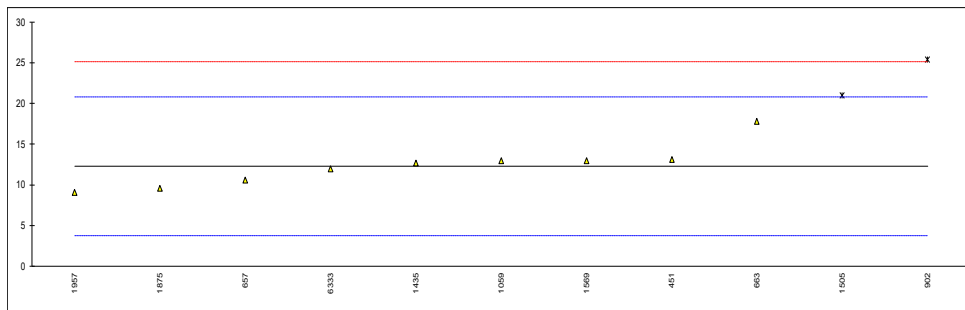
Determination of Kinematic Viscosity at 40°C on sample #20067; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178	D445	38.7		-0.29	
179	D445	38.57		-1.42	
225	D445	39.01		2.40	
237	D445	38.96		1.97	
325	D445	38.43		-2.63	
331	D7279Mod.	37.6	R(0.01)	-9.84	
349	D445	38.59		-1.25	
360	D445	38.734		0.00	
432	D445	38.76		0.23	
451	D7279	38.5		-2.03	
496	D445	38.760		0.23	
542	D7042	39.18		3.88	
614	D7042	38.648		-0.74	
633		-----		-----	
657	D7279	38.58		-1.33	
663	D445	38.5832		-1.30	
840	D7042	38.515		-1.90	
862		-----		-----	
902	D445	38.70		-0.29	
912		-----		-----	
962		-----		-----	
963		-----		-----	
974	D445	38.77	C	0.32	first reported 39.91
1017		-----		-----	
1023	D445	38.64		-0.81	
1026	D445	38.62		-0.98	
1059	ISO3104	38.81		0.66	
1146	D445	38.667		-0.58	
1417	D445	38.63		-0.90	
1431	D7042	38.880		1.27	
1435	D7042	38.903		1.47	
1469		-----		-----	
1505	D7042	38.683		-0.44	
1569	D445	38.405		-2.85	
1599	D445	39.03		2.58	
1604	D445	39.51	C,R(0.05)	6.74	first reported 37.510
1875	D7042	38.5045		-1.99	
1941	ISO3104	38.67		-0.55	
1957	D7042	39.22		4.23	
2124		-----		-----	
6016	D7042	39.077		2.98	
6333	D7042	38.74		0.06	

	normality	OK
	n	32
	outliers	2
	mean (n)	38.7334
	st.dev. (n)	0.20710
	R(calc.)	0.5799
	st.dev.(D445:19a)	0.11516
	R(D445:19a)	0.3225
Compare:		
	R (iis memo 1401)	0.6972
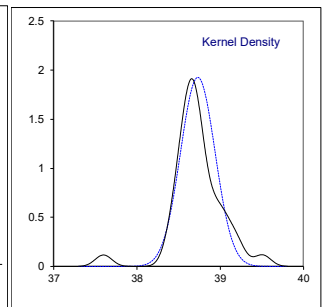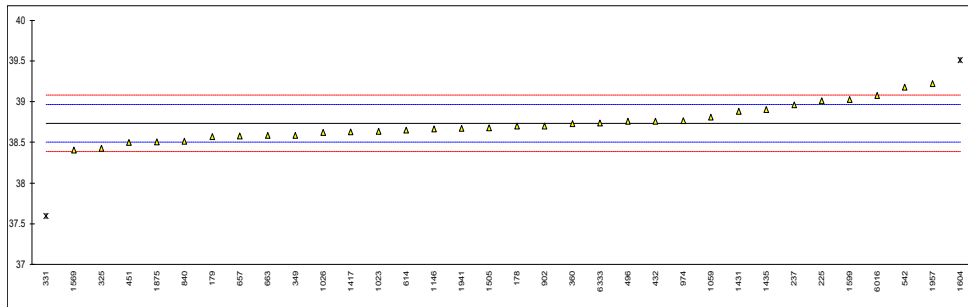
Determination of Kinematic Viscosity at 100°C on sample #20067; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178	D445	6.45		-1.97	
179	D445	6.46		-1.43	
225	D445	6.281	R(0.01)	-11.10	
237	D445	6.448		-2.08	
325	D445	6.512		1.38	
331	D7279Mod.	6.6		6.13	
349	D445	6.469		-0.94	
360	D445	6.4660		-1.11	
432	D445	6.486		-0.03	
451	D7279	6.46		-1.43	
496	D445	6.4662		-1.10	
542	D7042	6.818	R(0.01)	17.90	
614	D7042	6.4957		0.50	
633		-----		-----	
657	D7279	6.454		-1.76	
663	D445	6.4238		-3.39	
840	D7042	6.5173		1.66	
862		-----		-----	
902	D445	6.466		-1.11	
912		-----		-----	
962		-----		-----	
963		-----		-----	
974	D445	6.579	C	5.00	first reported 6.583
1017		-----		-----	
1023	D445	6.4506		-1.94	
1026	D445	6.47		-0.89	
1059	ISO3104	6.482		-0.24	
1146	D445	6.4622		-1.31	
1417	D445	6.479		-0.40	
1431	D7042	6.4942		0.42	
1435	D7042	6.5315		2.43	
1469		-----		-----	
1505	D7042	6.4792		-0.39	
1569	D445	6.453		-1.81	
1599	D445	5.7	R(0.01)	-42.48	
1604	D445	6.48	C	-0.35	first reported 6.207
1875	D7042	6.4937		0.39	
1941	ISO3104	6.433		-2.89	
1957	D7042	6.583	C	5.21	first reported 6.615
2124		-----		-----	
6016	D7042	6.553		3.59	
6333	D7042	6.484		-0.13	

	normality	not OK
	n	31
	outliers	3
	mean (n)	6.4865
	st.dev. (n)	0.04323
	R(calc.)	0.1211
	st.dev.(D445:19a)	0.01852
	R(D445:19a)	0.0518
Compare:		
	R(iis memo 1401)	0.1427
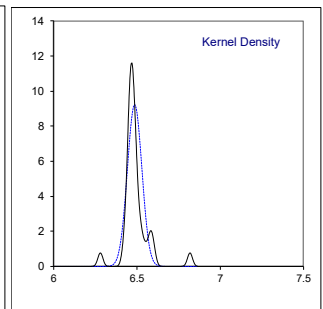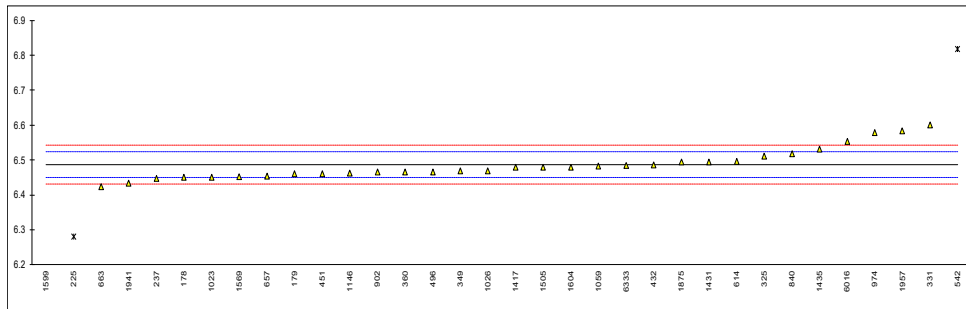
Determination of Viscosity Index on sample #20067;

lab	method	value	mark	z(targ)	remarks
178	D2270	118		-1.59	
179	D2270	119		-0.19	
225		-----		-----	
237	D2270	116.5		-3.69	
325	D2270	122		4.01	
331		-----		-----	
349	D2270	119		-0.19	
360	ISO2909	118.5		-0.89	
432	D2270	119.2		0.09	
451		-----		-----	
496	D2270	118.3		-1.17	
542	D2270	132	ex	18.01	excluded as outlier in Kinematic Viscosity 100°C
614		-----		-----	
633		-----		-----	
657		-----		-----	
663	D2270	117.25		-2.64	
840	D2270	121.9		3.87	
862		-----		-----	
902	D2270	118.6		-0.75	
912		-----		-----	
962		-----		-----	
963		-----		-----	
974	D2270	118	E	-1.59	calculation error, iis calculated 123
1017		-----		-----	
1023		-----		-----	
1026	D2270	119		-0.19	
1059	ISO2909	119		-0.19	
1146	D2270	119		-0.19	
1417	D2270	119.6		0.65	
1431	D2270	119.0		-0.19	
1435	D2270	120.6		2.05	
1469		-----		-----	
1505	D2270	119.3		0.23	
1569	D2270	120		1.21	
1599		-----		-----	
1604	D2270	115	ex,C	-5.79	excluded as outlier in Kinematic Viscosity 40°C / first reported 113
1875	ISO2909	116.45	E	-3.76	calculation error, iis calculated 121
1941	ISO2909	117.2		-2.71	
1957	D2270	122.3	E	4.43	calculation error, iis calculated 121
2124		-----		-----	
6016	D2270	120.7		2.19	
6333	D2270	120	E	1.21	calculation error, iis calculated 119

|	
normality	OK
n	25
outliers	0+ 2ex
mean (n)	119.14
st.dev. (n)	1.551
R(calc.)	4.34
st.dev.(D2270:10)	0.714
R(D2270:10)	2
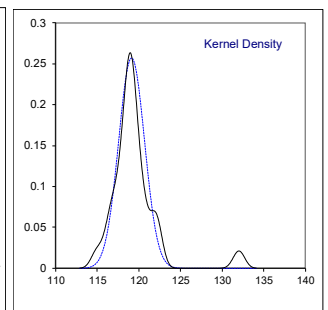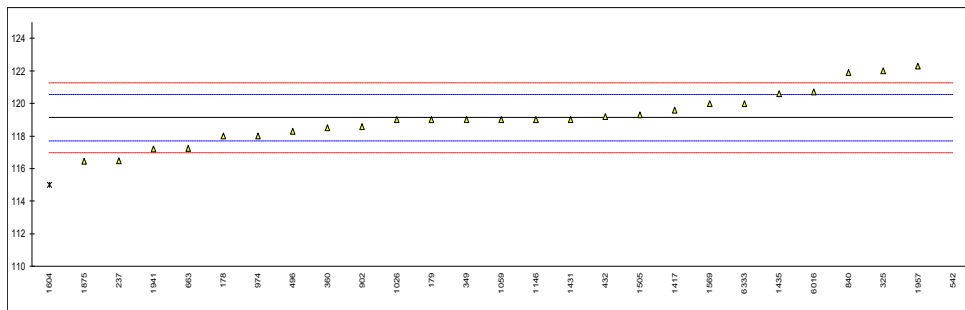
Determination of Oxidation Stability RPVOT on sample #20067; results in minutes

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
225		-----		-----	
237	D2272-A	>600		-----	
325	D2272-A	1854		0.61	
331		-----		-----	
349		-----		-----	
360		-----		-----	
432		-----		-----	
451	D2272-B	2722		6.54	
496		-----		-----	
542		-----		-----	
614	D2272-A	942		-5.62	
633		-----		-----	
657	D2272-B	1949		1.26	
663		-----		-----	
840		-----		-----	
862		-----		-----	
902	D2272-A	1587		-1.21	
912		-----		-----	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1017		-----		-----	
1023		-----		-----	
1026	D2272-A	1527		-1.62	
1059		-----		-----	
1146		-----		-----	
1417	D2272-A	1432		-2.27	
1431	D2272-B	2565		5.47	
1435	D2272-A	1968		1.39	
1469		-----		-----	
1505		-----		-----	
1569	D2272-A	>1022		-----	
1599		-----		-----	
1604		-----		-----	
1875		-----		-----	
1941	D2272-B	1464.5		-2.05	
1957		-----		-----	
2124		-----		-----	
6016	D2272-A	1402		-2.48	
6333		-----		-----	

normality	OK
n	11
outliers	0
mean (n)	1764.8
st.dev. (n)	523.21
R(calc.)	1465.0
st.dev.(D2272:14a)	146.38
R(D2272:14a)	409.9
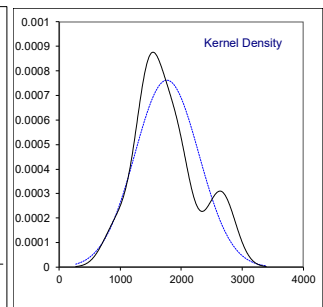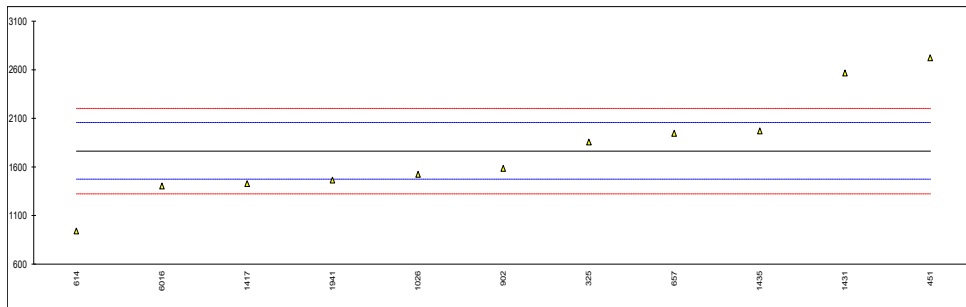
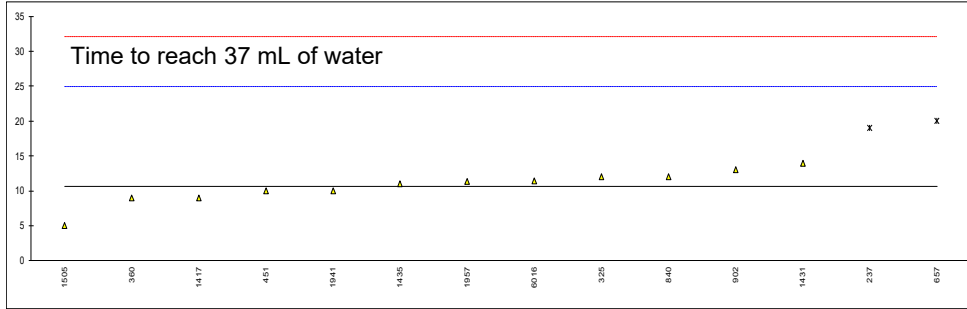## Determination of Water on sample #20067; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D6304-C	35		0.03	
179	D6304-C	29		-0.09	
225		-----		-----	
237	D6304-C	50		0.33	
325	D6304-C	24		-0.19	
331	D6304Mod.	27		-0.13	
349	D6304-A	25		-0.17	
360	ISO12937	26.8		-0.13	
432		-----		-----	
451	D6304-A	30		-0.07	
496	D6304-C	17		-0.33	
542	D6304	58		0.49	
614	D6304-C	44.5		0.22	
633		-----		-----	
657	D6304-C	<10		-----	
663	D6304-C	66.7		0.67	
840	D6304-C	28.2		-0.11	
862		-----		-----	
902	D6304-A	28.1		-0.11	
912		-----		-----	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1017		-----		-----	
1023	D6304-A	18		-0.31	
1026	D6304-C	27		-0.13	
1059	D6304-C	<30		-----	
1146		-----		-----	
1417	D6304-A	60		0.53	
1431	D6304-A	84.53		1.03	
1435	D6304-A	26		-0.15	
1469		-----		-----	
1505	D6304-A	100	R(0.05)	1.34	
1569	D6304-C	23		-0.21	
1599	D6304-A	16.4		-0.34	
1604	D6304-B	32.5		-0.02	
1875	ISO12937	242	C,R(0.01)	4.20	first reported 213
1941	D6304-C	26		-0.15	
1957	D6304-A	29.1		-0.09	
2124		-----		-----	
6016	D6304-A	37		0.07	
6333	D6304-C	2		-0.63	

normality	not OK	
n	26	
outliers	2	
mean (n)	33.49	
st.dev. (n)	17.707	
R(calc.)	49.58	
st.dev.(D6304:16e1)	49.601	
R(D6304:16e1)	138.88	
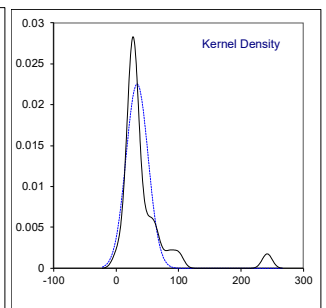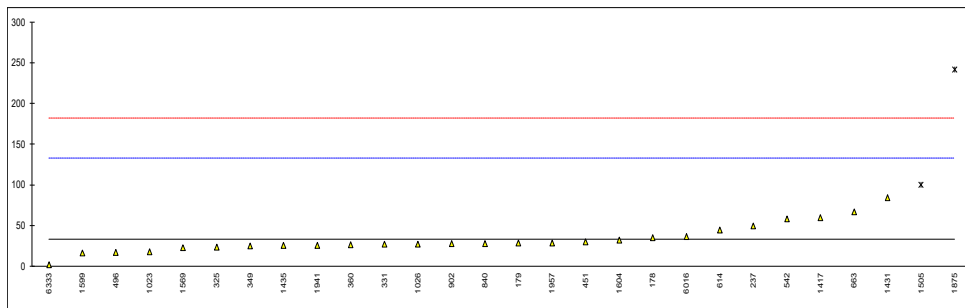
Determination of Water Separability at 54°C, distilled water on sample #20067; results in minutes

lab	method	3 mL or less emulsion	mark	z(targ)	37 mL of water	mark	z(targ)	complete break (40-40-0)	mark	z(targ)	test aborted	time test aborted
178		-----		-----	-----		-----	-----		-----	-----	-----
179	D1401	-----		-----	-----		-----	12		-0.15	NO	-----
225		-----		-----	-----		-----	-----		-----	-----	-----
237	D1401	18.81		0.97	19.03	DG(5)	1.17	23.16		1.41	NO	-----
325	D1401	12		0.01	12		0.19	13		-0.01	-----	-----
331		-----		-----	-----		-----	-----		-----	-----	-----
349		-----		-----	-----		-----	-----		-----	-----	-----
360	ISO6614	9.0		-0.41	9.0		-0.23	9.3		-0.53	NO	-----
432		-----		-----	-----		-----	-----		-----	-----	-----
451	D1401	10		-0.27	10		-0.09	11		-0.29	NO	-----
496	D1401	-----		-----	-----		-----	15		0.27	-----	-----
542		-----		-----	-----		-----	-----		-----	-----	-----
614		-----		-----	-----		-----	-----		-----	-----	-----
633		-----		-----	-----		-----	-----		-----	-----	-----
657	D1401	20		1.13	20	DG(5)	1.31	-----		-----	NO	-----
663		-----		-----	-----		-----	-----		-----	-----	-----
840	D1401	12		0.01	12		0.19	15		0.27	NO	15
862		-----		-----	-----		-----	-----		-----	-----	-----
902	D1401	13		0.15	13		0.33	30	DG(5)	2.37	YES	30
912		-----		-----	-----		-----	-----		-----	-----	-----
962		-----		-----	-----		-----	-----		-----	-----	-----
963		-----		-----	-----		-----	-----		-----	-----	-----
974		-----		-----	-----		-----	-----		-----	-----	-----
1017		-----		-----	-----		-----	-----		-----	-----	-----
1023		-----		-----	-----		-----	-----		-----	-----	-----
1026		-----		-----	-----		-----	15		0.27	NO	-----
1059		-----		-----	-----		-----	-----		-----	-----	-----
1146		-----		-----	-----		-----	-----		-----	YES	10
1417	D1401	9		-0.41	9		-0.23	9		-0.57	NO	-----
1431		-----		-----	14		0.47	25	DG(5)	1.67	NO	-----
1435	D1401	11		-0.13	11		0.05	-----		-----	NO	11
1469		-----		-----	-----		-----	-----		-----	-----	-----
1505	D1401	5		-0.97	5		-0.79	10		-0.43	-----	-----
1569	D1401	15		0.43	-----		-----	-----		-----	-----	-----
1599		-----		-----	-----		-----	-----		-----	-----	-----
1604		-----		-----	-----		-----	-----		-----	-----	-----
1875		-----		-----	-----		-----	-----		-----	-----	-----
1941	D1401	10		-0.27	10		-0.09	-----		-----	YES	15
1957	D1401	10.33		-0.22	11.37		0.10	12.52		-0.08	NO	-----
2124		-----		-----	-----		-----	-----		-----	-----	-----
6016	D1401	11.4		-0.07	11.4		0.11	12		-0.15	NO	-----
6333		-----		-----	-----		-----	-----		-----	YES	60

	normality	OK		not OK		not OK		
	n	14		12		12		
	outliers	0		2		2		
	mean (n)	11.90		10.65		13.08		
	st.dev. (n)	3.916		2.332		3.815		
	R(calc.)	10.97		6.53		10.68		
	st.dev.(D1401:19)	7.143		7.143		7.143		
	R(D1401:19)	20		20		20		

DG(5) = DG(0.05)

Time to reach 3 mL or less emulsion

Time to reach 37 mL of water

Time to reach complete break (40-40-0)
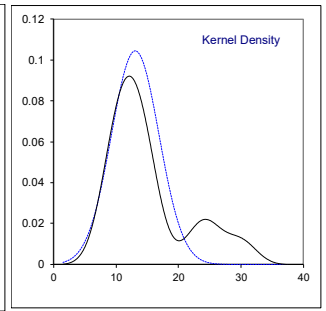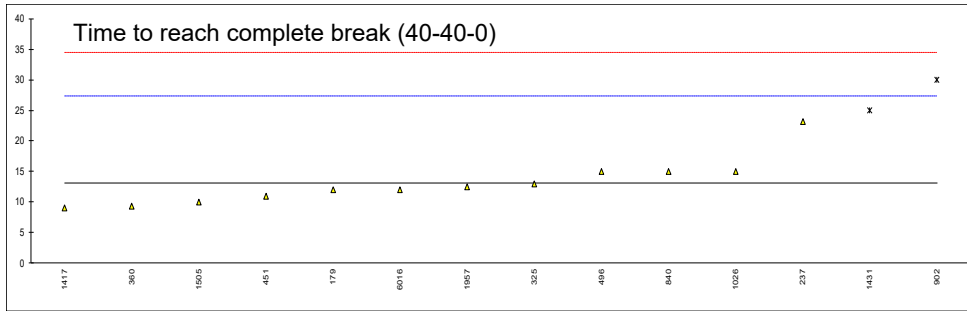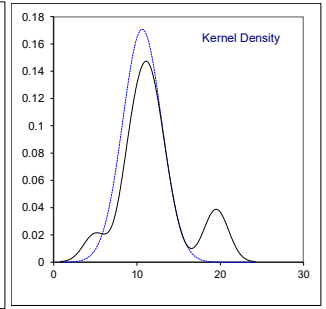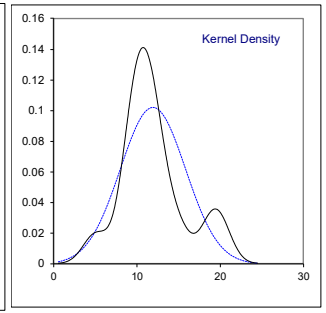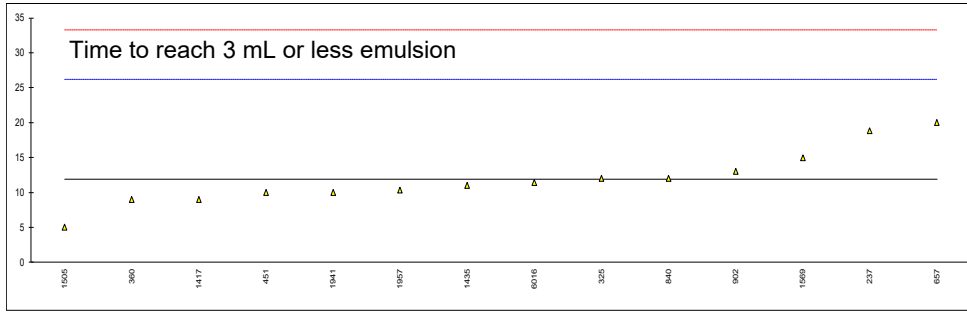
Determination of Water Separability at 54°C, distilled water on sample #20067; results in mL

lab	method	oil phase	mark	water phase	mark	emulsion phase	mark
178		-----		-----		-----	
179	D1401	40		40		0	
225		-----		-----		-----	
237	D1401	40		40		0	
325		-----		-----		-----	
331		-----		-----		-----	
349		-----		-----		-----	
360	ISO6614	42		38		0	
432		-----		-----		-----	
451	D1401	40		37		3	
496	D1401	42		38		0	
542		-----		-----		-----	
614		-----		-----		-----	
633		-----		-----		-----	
657	D1401	43		37		0	
663		-----		-----		-----	
840	D1401	40		40		0	
862		-----		-----		-----	
902	D1401	42		38		0	
912		-----		-----		-----	
962		-----		-----		-----	
963		-----		-----		-----	
974		-----		-----		-----	
1017		-----		-----		-----	
1023		-----		-----		-----	
1026		40		40		0	
1059		-----		-----		-----	
1146		35		24		21	
1417	D1401	40		40		0	
1431	D1401	43		37		0	
1435	D1401	40		37		3	
1469		-----		-----		-----	
1505		-----		-----		-----	
1569	D1401	40		40		0	
1599		-----		-----		-----	
1604		-----		-----		-----	
1875		-----		-----		-----	
1941	D1401	42		38		0	
1957	D1401	40		40		0	
2124		-----		-----		-----	
6016	D1401	40		40		0	
6333	D1401	40		28		12	

Determination of Level of Contamination on sample #20067; 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		-----		-----	-----		-----	-----		-----
360	ISO4406	6772.3		0.14	332.5		0.45	10.3		0.18
432		-----		-----	-----		-----	-----		-----
451	D7647	11746		2.06	368		0.89	1		-1.86
496	D7647	2026		-1.69	364		0.84	5		-0.98
542		-----		-----	-----		-----	-----		-----
614		-----		-----	-----		-----	-----		-----
633		-----		-----	-----		-----	-----		-----
657	ISO4406	5297		-0.43	232		-0.80	6		-0.76
663		-----		-----	-----		-----	-----		-----
840	D7647	18003.2		4.48	548.9		3.14	15.3		1.27
862		-----		-----	-----		-----	-----		-----
902	D7647	287.8	ex	-2.37	73.7	ex	-2.77	6.4	ex	-0.67
912		-----		-----	-----		-----	-----		-----
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
974		-----		-----	-----		-----	-----		-----
1017		-----		-----	-----		-----	-----		-----
1023		-----		-----	-----		-----	-----		-----
1026		-----		-----	-----		-----	-----		-----
1059		-----		-----	-----		-----	-----		-----
1146	ISO11500	1729.8		-1.81	138.13		-1.97	5.800		-0.80
1417		-----		-----	-----		-----	-----		-----
1431	ISO4407	10661		1.64	720		5.26	16		1.43
1435		18642	ex	4.73	1858	G(0.01)	19.40	72	G(0.01)	13.68
1469		-----		-----	-----		-----	-----		-----
1505	D7647	207.5	ex	-2.40	86.8		-2.61	15.9		1.41
1569		6035		-0.15	253		-0.54	4		-1.20
1599		4555		-0.72	239		-0.71	6		-0.76
1604	ISO4407	1602	C	-1.86	195		-1.26	11		0.33
1875		-----		-----	-----		-----	-----		-----
1941		16548.0	ex	3.92	4362.4	G(0.01)	50.52	144.9	G(0.01)	29.63
1957	ISO4407	5966.6		-0.17	285.9		-0.13	23.4		3.05
2124		-----		-----	-----		-----	-----		-----
6016	ISO4406	2540		-1.50	326		0.37	7		-0.54
6333	D7596	486	ex	-2.29	62		-2.91	6		-0.76
	normality	not OK			not OK			suspect		
	n	12			14			14		
	outliers	0 +5ex			2 +1ex			2 +1ex		
	mean (n)	6411.2			296.5			9.5		
	st.dev. (n)	4911.14			175.13			6.15		
	R(calc.)	13751.2			490.4			17.2		
	st.dev.(D7647:10)	2587.36			80.48			4.57		
	R(D7647:10)	7244.6			225.4			12.8		

Lab 902 test results excluded for ≥4 µm and ≥6 µm as test results in counts/mL and ISO4406 scale number did not match
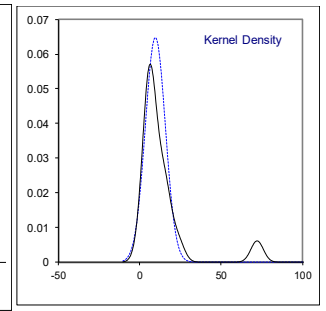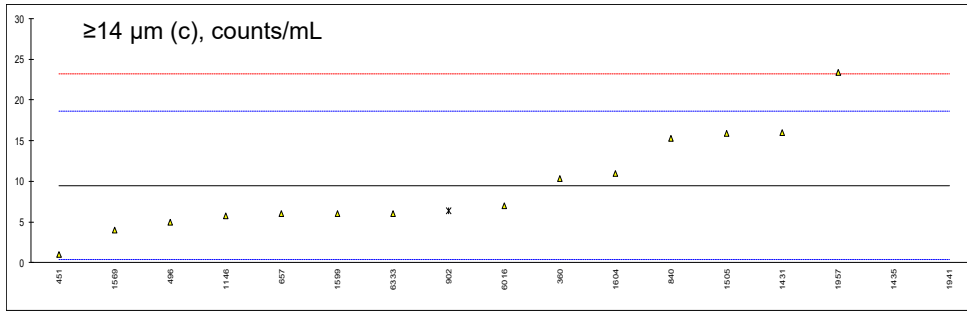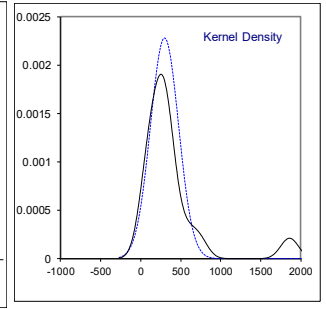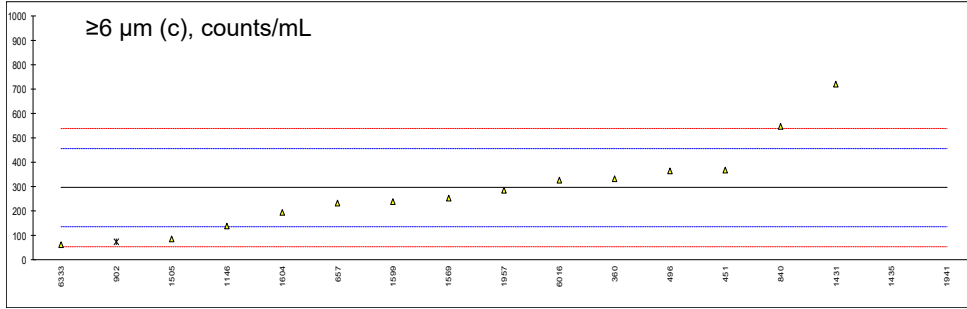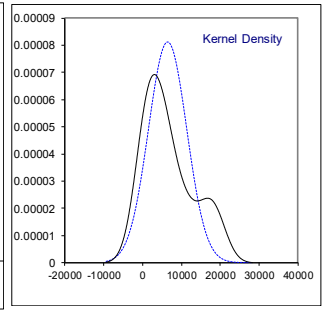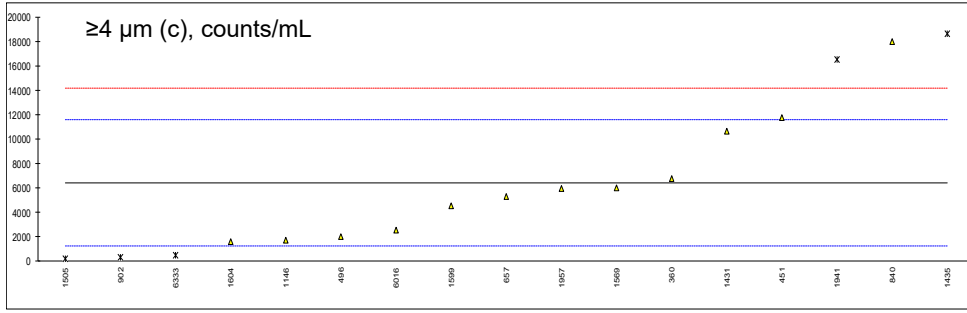Lab 902 test result excluded for ≥14 µm as related test results for ≥4 µm and ≥6 µm were excluded
Lab 1435 test result excluded for ≥4 µm as statistical outliers at related measurements for ≥6 µm and ≥14 µm
Lab 1505 test result excluded for ≥4 µm as statistical outlier in related conversion to ISO4406 scale number
Lab 1604 first reported 1178
Lab 1941 test result excluded for ≥4 µm as statistical outliers at related measurements for ≥6 µm and ≥14 µm
Lab 6333 test result excluded for ≥4 µm as statistical outlier in related conversion to ISO4406 scale number

≥4 µm (c), counts/mL

Kernel Density

≥6 µm (c), counts/mL

Kernel Density

≥14 µm (c), counts/mL

Kernel Density

Determination of Level of Contamination on sample #20067; 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		-----		-----	-----		-----	-----		-----
360	ISO4406	20		0.10	16		1.30	11		0.70
432		-----		-----	-----		-----	-----		-----
451	ISO4406	21		1.75	16		1.30	7		-4.90
496	ISO4406	18		-3.19	16		1.30	9		-2.10
542	ISO4406	21		1.75	16		1.30	13		3.50
614		-----		-----	-----		-----	-----		-----
633		-----		-----	-----		-----	-----		-----
657	ISO4406	20		0.10	15		-1.04	10		-0.70
663		-----		-----	-----		-----	-----		-----
840	ISO4406	21		1.75	16		1.30	11		0.70
862		-----		-----	-----		-----	-----		-----
902	ISO4406	17	ex,C	-4.84	15	ex,C	-1.04	10	ex	-0.70
912		-----		-----	-----		-----	-----		-----
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
974		-----		-----	-----		-----	-----		-----
1017		-----		-----	-----		-----	-----		-----
1023	ISO4406	20		0.10	15		-1.04	10		-0.70
1026	ISO4406	21		1.75	17		3.63	12		2.10
1059		-----		-----	-----		-----	-----		-----
1146		18		-3.19	14		-3.37	10		-0.70
1417	ISO4406	22		3.40	17		3.63	12		2.10
1431	ISO4406	21		1.75	17		3.63	11		0.70
1435		21	ex	1.75	18	ex	5.96	13	ex	3.50
1469		-----		-----	-----		-----	-----		-----
1505	ISO4406	15	DG(0.05)	-8.13	14		-3.37	11		0.70
1569		20		0.10	15		-1.04	9		-2.10
1599	ISO4406	19		-1.54	15		-1.04	10		-0.70
1604	ISO4406	18	C	-3.19	15		-1.04	11		0.70
1875		-----		-----	-----		-----	-----		-----
1941	ISO4406	21	ex	1.75	19	ex	8.30	14	ex	4.90
1957	ISO4406	20		0.10	15		-1.04	12		2.10
2124		-----		-----	-----		-----	-----		-----
6016	ISO4406	19		-1.54	16		1.30	10		-0.70
6333	D7647	16	DG(0.05)	-6.49	13		-5.70	10		-0.70
	normality	OK			OK			suspect		
	n	16			18			18		
	outliers	2 +3ex			0 +3ex			0 +3ex		
	mean (n)	19.9			15.4			10.5		
	st.dev. (n)	1.24			1.10			1.38		
	R(calc.)	3.5			3.1			3.9		
	st.dev.(D7647:10)	0.61			0.43			0.71		
	R(D7647:10)	1.7			1.2			2.0		

Lab 902 first reported 15 for ≥4 µm and 13 for ≥6 µm
Lab 902 test results excluded for ≥4 µm and ≥6 µm as test results in counts/mL and ISO4406 scale number did not match
Lab 902 test result excluded for ≥14 µm as related test results for ≥4 µm and ≥6 µm were excluded
Lab 1435 test results excluded for ≥6 and ≥14 µm as statistical outliers at related measurements for counts/mL
Lab 1435 test result excluded for ≥4 µm as related test results for ≥6 µm and ≥14 µm were excluded
Lab 1604 first reported 17
Lab 1941 test results excluded for ≥6 and ≥14 µm as statistical outliers at related measurements for counts/mL
Lab 1941 test result excluded for ≥4 µm as related test results for ≥6 µm and ≥14 µm were excluded

≥4 µm (c), ISO4406 scale numbers

≥6 µm (c), ISO4406 scale numbers

≥14 µm (c), ISO4406 scale numbers
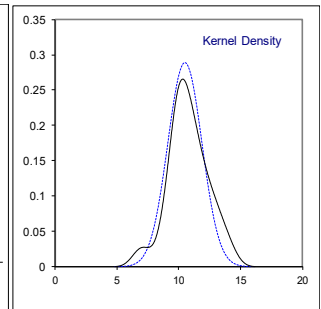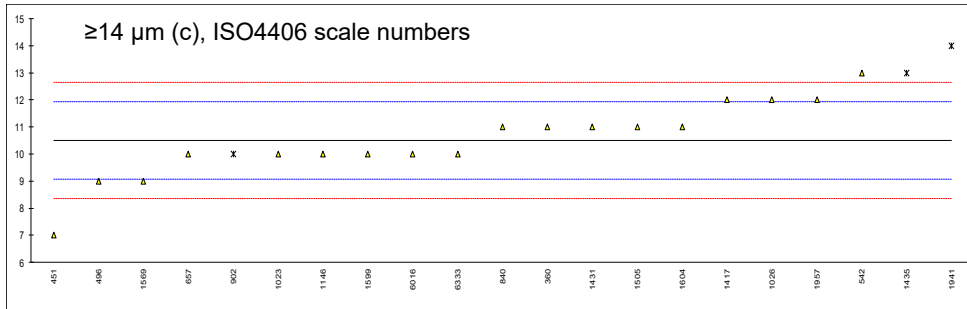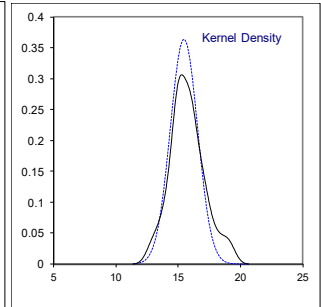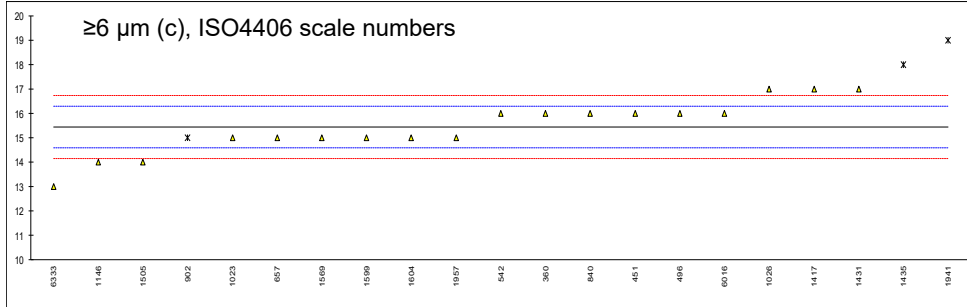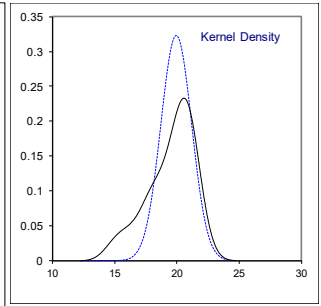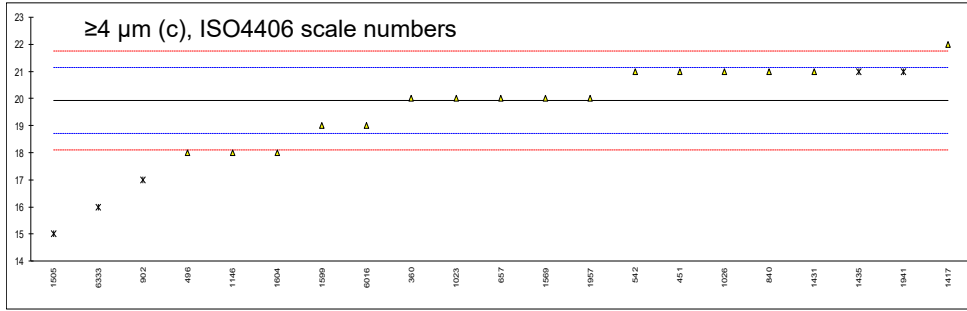
APPENDIX 2

Number of participants per country

```
1 lab in   ARGENTINA
2 labs in  AUSTRALIA
1 lab in   AUSTRIA
3 labs in  BELGIUM
1 lab in   BULGARIA
1 lab in   CHINA, People's Republic
1 lab in   COTE D'IVOIRE
1 lab in   EGYPT
1 lab in   FRANCE
2 labs in  GERMANY
2 labs in  GREECE
1 lab in   INDIA
1 lab in   ITALY
1 lab in   KAZAKHSTAN
1 lab in   MALAYSIA
2 labs in  NETHERLANDS
1 lab in   NIGERIA
1 lab in   NORWAY
1 lab in   PHILIPPINES
2 labs in  SAUDI ARABIA
1 lab in   SERBIA
1 lab in   SINGAPORE
1 lab in   SLOVENIA
4 labs in  SPAIN
1 lab in   THAILAND
1 lab in   TURKEY
1 lab in   UNITED ARAB EMIRATES
2 labs in  UNITED KINGDOM
2 labs in  UNITED STATES OF AMERICA
1 lab in   VIETNAM
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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
ex   = test result excluded from the statistical evaluation
W   = test result withdrawn on request of the participants
fr.   = first reported test result
n.a.   = not applicable
n.e.   = not evaluated
n.d.   = not detected
SDS   = Safety Data Sheet

Literature

1   iis Interlaboratory Studies, Protocol for the Organisation, Statistics and Evaluation, June 2018
2   ASTM E178:89
3   ASTM E1301:89
4   ISO5725:86
5   ISO5725, parts 1-6, 1994
6   ISO13528:05
7   M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
8   W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
9   IP367:84
10   DIN38402 T41/42
11   P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
12   J.N. Miller, Analyst, 118, 455, (1993)
13   Analytical Methods Committee, Technical brief, No 4, January 2001.
14   P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst 2002, 127, 1359-1364 (2002)
15   Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)
16   Horwitz, R. Albert, J. AOAC Int, 79, 3, 589, (1996)