

Results of Proficiency Test
Turbine Oil (used)
May 2019

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. G.A. Oosterlaken-Buijs
Correctors: ing. A.S. Noordman-de Neef & ing. L. Sweere
Report no.: iis19L05

September 2019

CONTENTS

1	INTRODUCTION	3
2	SET UP	3
2.1	ACCREDITATION	3
2.2	PROTOCOL	3
2.3	CONFIDENTIALITY STATEMENT	3
2.4	SAMPLES	4
2.5	STABILITY OF THE SAMPLES	4
2.6	ANALYSES	5
3	RESULTS	5
3.1	STATISTICS	5
3.2	GRAPHICS	6
3.3	Z-SCORES	7
4	EVALUATION	7
4.1	EVALUATION PER TEST	8
4.2	PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES	10
4.3	COMPARISON OF THE PROFICIENCY TEST OF MAY 2019 WITH PREVIOUS PTS	11

Appendices:

1.	Data, statistical and graphical results	12
2.	Number of participants per country	32
3.	Abbreviations and literature	33

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 2018/2019, it was decided to continue the round robin for the analysis of used Turbine Oil.

In this interlaboratory study 45 laboratories in 28 different countries registered for participation. See appendix 2 for the number of participants per country.

In this report, the results of the 2019 Turbine Oil (used) 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 Spijkensisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send one bottle of 1L labelled #19082 of used Turbine Oil.

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 Spijkensisse, 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

The necessary bulk material of about 60 liters of used Turbine Oil was obtained from a third party. After homogenisation 56 amber glass bottles of 1L were filled and labelled #19082. The homogeneity of the subsamples #19082 was checked by determination of Density at 15°C in accordance with ASTM D4052 on 8 stratified randomly selected samples.

	Density at 15°C in kg/L
Sample #19082-1	0.86398
Sample #19082-2	0.86399
Sample #19082-3	0.86399
Sample #19082-4	0.86398
Sample #19082-5	0.86397
Sample #19082-6	0.86397
Sample #19082-7	0.86397
Sample #19082-8	0.86397

Table 1: homogeneity test results of subsamples #19082

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

	Density at 15°C in kg/L
r(observed)	0.00002
reference test method	ASTM D1298:12b (2018)
0.3 x R(reference test method)	0.00045

Table 2: evaluation of the repeatability of the subsamples #19082

The calculated repeatability was in agreement with 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneity of the subsamples #19082 was assumed.

To each of the participating laboratories 1 liter of sample #19082 was sent on May 1, 2019.

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 ANALYSES

The participants were requested to determine on sample #19082: Acid Number (Total), 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, Steam Demulsibility Characteristics, Steam Water Separation Ability and Level of Contamination (counts/ ml and ISO4406 scale).

Also, some additional questions were asked about the determination of 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 appropriate reference test methods that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the 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 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. 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:

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

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

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

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

4 EVALUATION

In this interlaboratory study, no major problems were encountered with the dispatch of the samples. One participant reported test results after the final reporting date and three participants did not report any test result at all. Not all laboratories were able to report all analyses requested. In total 42 participants reported 600 test results. Observed were 13 outlying test results, which is 2.2% of the numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

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

4.1 EVALUATION PER TEST

In this section, the reported test results are discussed per test. The test methods, which were used by the various laboratories, were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data in appendix 1. The abbreviations, used in these tables, are listed 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.

Acid Number (Total): This determination may be problematic depending on mode of the test method used. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D664-A:18e2 for Buffer End Point (BEP) and Inflection Point (IP) and 60 mL of titration solvent used, but is not in agreement with the more strict requirements of ASTM D664-A:18e2 for BEP and IP and 125 mL of titration solvent used. Method ASTM D664 was updated in 2018. One of the major changes is the buffer used in the end point detection (pH 11 is changed into pH 10). Method ASTM D664 states in paragraph 13.1.2: "For all acid titrations on used oils, mark as an end point the point on the curve that corresponds to the meter reading for an aqueous basic buffer (pH 10)." Remarkably, of all the laboratories that reported a selection of the end point, around 65% reported to have used the Inflection Point instead of the Buffer End Point. When evaluating the test results using the BEP and the IP separately, the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D664-A:18e2 for BEP and IP and 60 mL of titration solvent used, but is not in agreement with the requirements of ASTM D664-A:18e2 for BEP and IP and 125 mL of titration solvent used. It appears that the volume of titration solvent used does have an influence on sample #19082 of the 2019 PT. However, on the sample of the 2018 PT this influence was not observed.

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. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D1298:12b(2017).

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 D93:16a, procedure A and B. When evaluated separately for procedure A and procedure B, the respective calculated reproducibilities are also in agreement with the respective reference test methods.

Insoluble Color Bodies: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D7843:18.

Kinematic Viscosity at 40°C: 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 D445:18.

Kinematic Viscosity at 100°C: 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 D445:18.

Viscosity Index: This determination was problematic. Two statistical outliers were observed and one other test result was excluded. However, the calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of ASTM D2270:10(2016). Two calculation errors were observed.

Oxidation Stability RPVOT: This determination was very problematic. No statistical outliers were observed. The variation in the reported test results was very large (527 – 1142), therefore no z-scores were calculated.

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. No statistical outliers were observed over 3 parameters. All calculated reproducibilities are in agreement with the requirements of ASTM D1401:18b.

Steam Demulsibility Characteristics: Only one participant reported a test result. Therefore, no significant conclusions were drawn.

Steam Water Separation Ability: Only two participants reported a test result. Therefore, no significant conclusions were drawn.

Level of Contamination: This determination was very problematic. No statistical outliers were observed, but six test results were excluded over 6 parameters (3 for counts/mL and 3 for scale number). The variation in the reported test results was large (e.g. for particle size $\geq 4 \mu\text{m}$ counts/mL from 325 – 49378.14) therefore no z-scores were calculated.

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

Parameter	unit	n	average	2.8 * sd	R(lit)
Acid Number (Total)	mg KOH/g	35	0.10	0.05	0.06
Color ASTM		31	4.0	0.6	1
Density at 15°C	kg/L	38	0.8639	0.0012	0.0015
Flash Point C.O.C.	°C	26	229.0	17.2	18
Flash Point PMcc	°C	34	210 .5	9.4	15.0
Insoluble Color Bodies		13	26.3	25.6	23.7
Kinematic Viscosity at 40°C	mm ² /s	38	37.702	0.427	0.308
Kinematic Viscosity at 100°C	mm ² /s	36	6.187	0.078	0.048
Viscosity Index		30	111.2	3.0	2
Oxidation Stability RPVOT	minutes	18	804	522	(184)
Water	mg/kg	32	32.1	23.8	135.4
Water Separability at 54°C, distilled water					
- Time \leq 3 mL emulsion	minutes	20	17.6	17.1	20
- Time 37 mL water	minutes	16	16.5	13.8	20
- Time to complete break	minutes	14	19.1	14.5	20
Level of Contamination					
- $\geq 4\mu\text{m}$ (c)	counts/mL	22	16027	44936	(18110)
- $\geq 6\mu\text{m}$ (c)	counts/mL	22	1530	5154	(1163)
- $\geq 14\mu\text{m}$ (c)	counts/mL	22	30	62	(40)
- $\geq 4\mu\text{m}$ (c)	ISO scale	27	20	7	(2)
- $\geq 6\mu\text{m}$ (c)	ISO scale	27	17	5	(1)
- $\geq 14\mu\text{m}$ (c)	ISO scale	27	12	3	(2)

Table 3: reproducibilities of tests of sample #19082

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF MAY 2019 WITH PREVIOUS PTs

	May 2019	May 2018	May 2017	May 2016	May 2015
Number of reporting labs	42	43	34	38	39
Number of test results	600	581	508	474	398
Number of statistical outliers	13	10	16	15	36
Percentage outliers	2.2%	1.7%	3.1%	3.2%	9.0%

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 2019	May 2018	May 2017	May 2016	May 2015
Acid Number (Total)	+	--	-	-	+
Color ASTM	+	+	+	+/-	+
Density at 15°C	+	+	+	++	++
Flash Point C.O.C.	+/-	-	+	(--)	n.e.
Flash Point PMcc	+	+	+	+/-	+/-
Insoluble Color Bodies	+/-	+	-	+/-	n.e.
Kinematic Viscosity at 40°C	-	+	-	++	++
Kinematic Viscosity at 100°C	-	+/-	--	+/-	+
Viscosity Index	-	(--)	--	--	--
Oxidation Stability RPVOT	(--)	-	-	++	n.e.
Water by KF	++	++	++	++	++
Water Separability	+	++	++	n.e.	+
Level of Cont. – counts/mL	(--)	-	--	-	--
Level of Cont. – scale no.	(--)	-	--	--	n.e.

Table 5: comparison determinations against the reference test methods

The following performance categories were used:

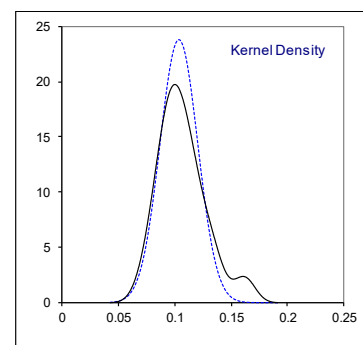
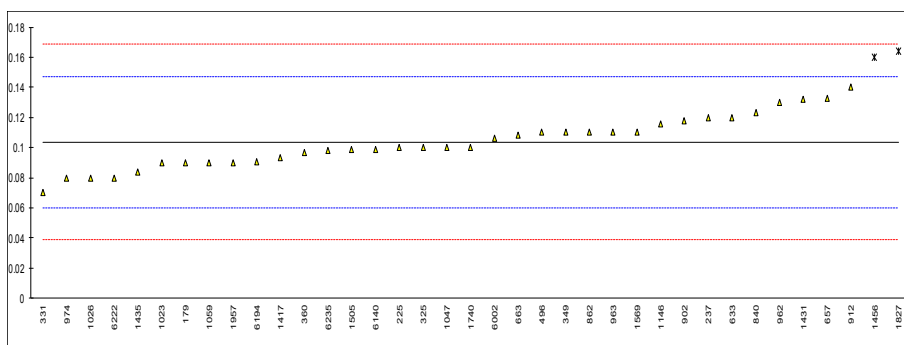
- ++: group performed much better than the reference test method
- + : group performed better than the reference test method
- +/-: group performance equals the reference test method
- : group performed worse than the reference test method
- : group performed much worse than the reference test method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

APPENDIX 1

Determination of Acid Number (Total) on sample #19082; results in mg KOH/g

lab	method	value	mark	z(targ)	end point determination	titration solvent vol.	remarks
178	INH-1118	<0.10		----	Inflection Point	---	
179	D664-A	0.09		-0.63	---	---	
225	D974	0.10		-0.17	---	---	
237	D664-A	0.12		0.76	Inflection Point	125 mL	
325	D664-A	0.10		-0.17	Buffer End Point (pH 10)	125 mL	
331	D664Mod.	0.07		-1.55	---	---	
349	D664-A	0.11		0.29	Inflection Point	125 mL	
360	D664-A	0.097		-0.31	Inflection Point	60 mL	
432		----		----	---	---	
451		----		----	---	---	
496	D664-A	0.11		0.29	Buffer End Point (pH 11)	60 mL	
541		----		----	---	---	
603		----		----	---	---	
614		----		----	---	---	
633	D664-A	0.12		0.76	Inflection Point	125 mL	
657	D664-B	0.133		1.36	Inflection Point	60 mL	
663	D664-A	0.108		0.20	Buffer End Point (pH 10)	60 mL	
840	D664-B	0.123		0.89	Buffer End Point (pH 10)	60 mL	
862	D664-A	0.11		0.29	Inflection Point	60 mL	
902	D664-A	0.118		0.66	Inflection Point	60 mL	
912	D664-A	0.14		1.68	---	---	
962	D974	0.13		1.22	---	---	
963	D664-A	0.11		0.29	Inflection Point	60 mL	
974	D664-A	0.08		-1.09	Inflection Point	125 mL	
1017		----		----	---	---	
1023	D8045Mod.	0.09		-0.63	Thermotitration	50 mL	
1026	D664-A	0.08		-1.09	Buffer End Point (pH 11)	60 mL	
1047	ISO6618	0.10		-0.17	Inflection Point	60 mL	
1059	ISO6619	0.09		-0.63	Buffer End Point (pH 11)	60 mL	
1146	D664-A	0.116		0.57	Buffer End Point (pH 11)	125 mL	
1417	D664-A	0.093		-0.49	Buffer End Point (pH 10)	60 mL	
1431	D664-A	0.132		1.31	Inflection Point	60 mL	
1435	D664-A	0.084		-0.91	---	100 mL	
1456	D974	0.16	DG(0.05)	2.60	---	125 mL	
1505	D664-A	0.099		-0.21	Inflection Point	60 mL	
1569	D664-A	0.11		0.29	Inflection Point	125 mL	
1740	D664-A	0.10		-0.17	---	---	
1827	D664-A	0.164	DG(0.05)	2.79	Inflection Point	125 mL	
1957	D664-A	0.09		-0.63	Buffer End Point (pH 10)	125 mL	
6002	D664-A	0.106		0.11	Buffer End Point (pH 10)	60 mL	
6016		----		----	---	---	
6140	D664-A	0.099		-0.21	Inflection Point	60 mL	
6194	D664-A	0.0904	C	-0.61	Inflection Point	60 mL	first reported 0.23355
6222	D664	0.08		-1.09	Inflection Point	60 mL	
6235	D664-A	0.098		-0.26	Inflection Point	60 mL	

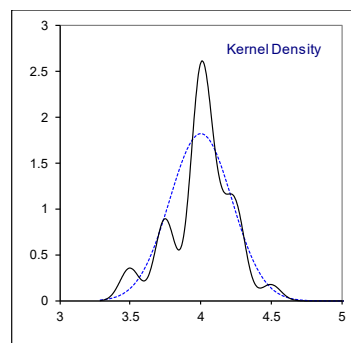
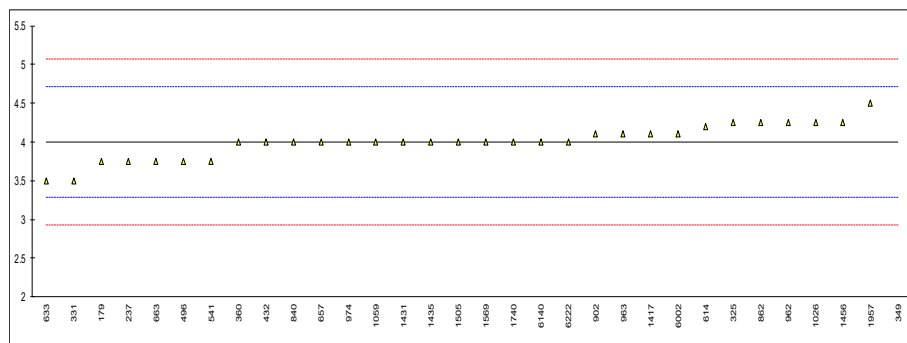
			<u>BEP (pH 10 and 11) only</u>	<u>Inflection Point only</u>
normality	OK		OK	OK
n	35		10	17
outliers	2		0	1
mean (n)	0.1036		0.1016	0.1063
st.dev. (n)	0.01678		0.0134	0.0156
R(calc.)	0.0470		0.0374	0.0436
st.dev.(D664-A:18e2)	0.02166	BEP (pH 10), 60mL	0.02125	---
R(D664-A:18e2)	0.0607	BEP (pH 10), 60mL	0.0595	---
compare				
R(D664-A:18e2)	0.0291	BEP (pH 10), 125mL	0.0286	---
R(D664-A:18e2)	0.0627	IP, 60mL	---	0.0640
R(D664-A:18e2)	0.0206	IP, 125mL	---	0.0211



Determination of Color ASTM on sample #19082;

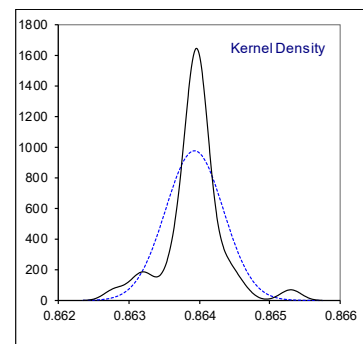
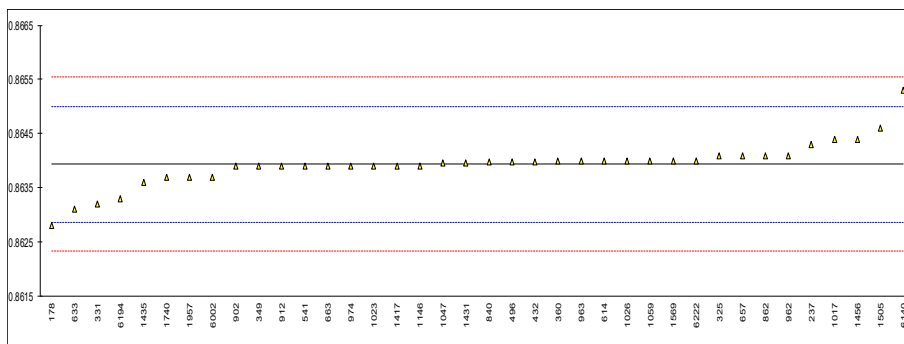
lab	method	reported test value	iis conversion *	mark	z(targ)	remarks
178		----	----		----	
179	D1500	L4.0	3.75		-0.71	
225		----	----		----	
237	D1500	L4.0	3.75		-0.71	
325	D6045	L4.5	4.25		0.69	
331	D1500	3.5	3.5		-1.41	
349	D6045	<6.5	6.25	R(0.01)	6.29	
360	D1500	4.0	4.0		-0.01	
432	D1500	4.0	4.0		-0.01	
451		----	----		----	
496	D1500	L4.0	3.75		-0.71	
541	D1500	L4.0	3.75		-0.71	
603		----	----		----	
614	D1500	4.2	4.2		0.55	
633	D1500	3.5	3.5		-1.41	
657	D1500	4.0	4.0		-0.01	
663	D1500	L4.0	3.75		-0.71	
840	D1500	4.0	4.0		-0.01	
862	D1500	L4.5	4.25		0.69	
902	D1500	4.1	4.1		0.27	
912		----	----		----	
962	D1500	L4.5	4.25		0.69	
963	D1500	4.1	4.1		0.27	
974	D1500	4.0	4.0		-0.01	
1017		----	----		----	
1023		----	----		----	
1026	D1500	L4.5	4.25		0.69	
1047		----	----		----	
1059	D1500	4.0	4.0		-0.01	
1146		----	----		----	
1417	D6045	4.1	4.1		0.27	
1431	D1500	4	4		-0.01	
1435	D1500	4.0	4.0		-0.01	
1456	D1500	L 4.5	4.25		0.69	
1505	D1500	4.0	4.0		-0.01	
1569	D1500	4.0	4.0		-0.01	
1740	D1500	4	4		-0.01	
1827		----	----		----	
1957	D1500	4.5	4.5		1.39	
6002	In house	4.1	4.1		0.27	
6016		----	----		----	
6140	D1500	4.0	4.0		-0.01	
6194		----	----		----	
6222	D1500	4.0	4.0		-0.01	
6235		----	----		----	
normality			OK			
n			31			
outliers			1			
mean (n)			4.00			
st.dev. (n)			0.220			
R(calc.)			0.62			
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 L4.0 into 3.75).



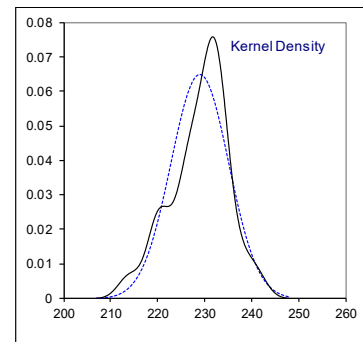
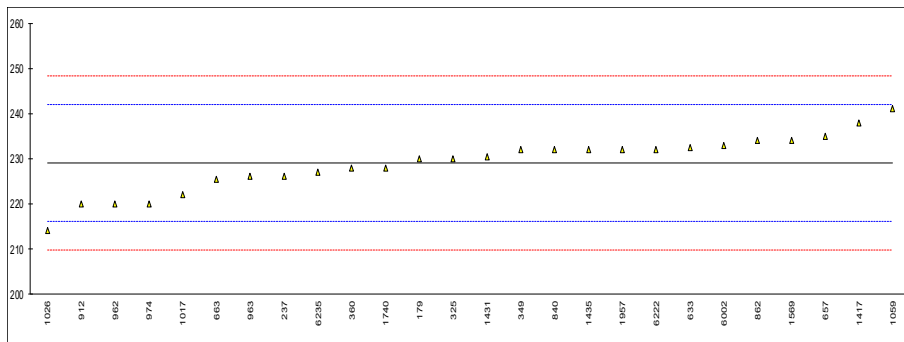
Determination of Density at 15°C on sample #19082; results in kg/L

lab	method	value	mark	z(targ)	remarks
178	D1298	0.8628		-2.11	
179		----		----	
225		----		----	
237	D4052	0.86430		0.69	
325	D4052	0.8641		0.31	
331	ISO12185	0.8632		-1.37	
349	D4052	0.8639		-0.06	
360	D4052	0.8640		0.13	
432	D4052	0.86398		0.09	
451		----		----	
496	D4052	0.86397		0.07	
541	D4052	0.86390		-0.06	
603		----		----	
614	D4052	0.8640		0.13	
633	D1298	0.8631		-1.55	
657	D4052	0.8641		0.31	
663	D4052	0.86390		-0.06	
840	D4052	0.86397		0.07	
862	D4052	0.8641		0.31	
902	D4052	0.8639		-0.06	
912	D1298	0.8639		-0.06	
962	D4052	0.8641		0.31	
963	D4052	0.8640		0.13	
974	D1298	0.8639		-0.06	
1017	D4052	0.8644		0.87	
1023	D4052	0.8639	C	-0.06	first reported 0.8639 kg/m ³
1026	D4052	0.8640		0.13	
1047	ISO12185	0.86395		0.03	
1059	ISO12185	0.8640		0.13	
1146	D4052	0.86391		-0.04	
1417	D4052	0.8639		-0.06	
1431	D4052	0.86396		0.05	
1435	D4052	0.8636		-0.62	
1456	D4052	0.8644		0.87	
1505	D7042	0.8646	C	1.25	first reported 0.8646 kg/m ³
1569	ISO12185	0.8640		0.13	
1740	D4052	0.8637		-0.43	
1827		----		----	
1957	D4052	0.8637		-0.43	
6002	ISO12185	0.8637	C	-0.43	first reported 0.8658
6016		----		----	
6140	D7042	0.8653	C	2.55	first reported 0.8653 kg/m ³
6194		0.8633	C	-1.18	first reported 0.8660
6222	D7042	0.864		0.13	
6235		----		----	
	normality	not OK			
	n	38			
	outliers	0			
	mean (n)	0.86393			
	st.dev. (n)	0.000410			
	R(calc.)	0.00115			
	st.dev.(D1298:12b)	0.000536			
	R(D1298:12b)	0.00150			
	compare				
	R(D4052:18a)	0.00050			



Determination of Flash Point C.O.C. on sample #19082; results in °C

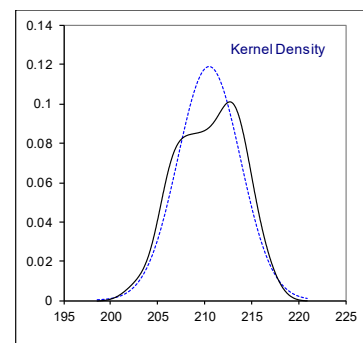
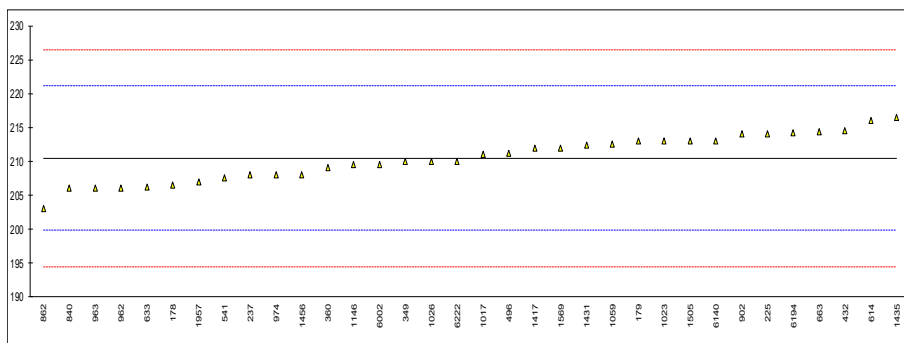
lab	method	value	mark	z(targ)	remarks
178		----		----	
179	D92	230		0.15	
225		----		----	
237	D92	226		-0.47	
325	D92	230		0.15	
331		----		----	
349	D92	232		0.46	
360	D92	228		-0.16	
432		----		----	
451		----		----	
496		----		----	
541		----		----	
603		----		----	
614		----		----	
633	D92	232.49		0.54	
657	D92	235		0.93	
663	D92	225.45		-0.55	
840	D92	232		0.46	
862	D92	234		0.78	
902		----		----	
912	D92	220		-1.40	
962	D92	220		-1.40	
963	D92	226		-0.47	
974	D92	220		-1.40	
1017	D92	222		-1.09	
1023		----		----	
1026	D92	214		-2.34	
1047		----		----	
1059	ISO2592	241		1.86	
1146		----		----	
1417	D92	238		1.40	
1431	D92	230.5		0.23	
1435	D92	232.0		0.46	
1456		----		----	
1505		----		----	
1569	D92	234.0		0.78	
1740	D92	228		-0.16	
1827		----		----	
1957	D92	232		0.46	
6002	ISO2592	233		0.62	
6016		----		----	
6140		----		----	
6194		----		----	
6222	ISO2592	232		0.46	
6235	ISO2592	227		-0.31	
	normality	OK			
	n	26			
	outliers	0			
	mean (n)	229.02			
	st.dev. (n)	6.138			
	R(calc.)	17.19			
	st.dev.(D92:18)	6.429			
	R(D92:18)	18			



Determination of Flash Point PMcc on sample #19082; results in °C

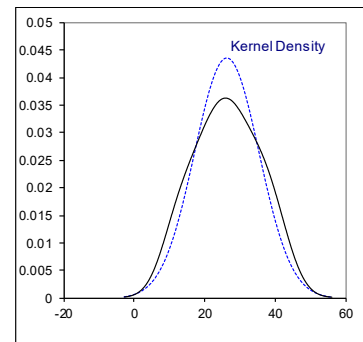
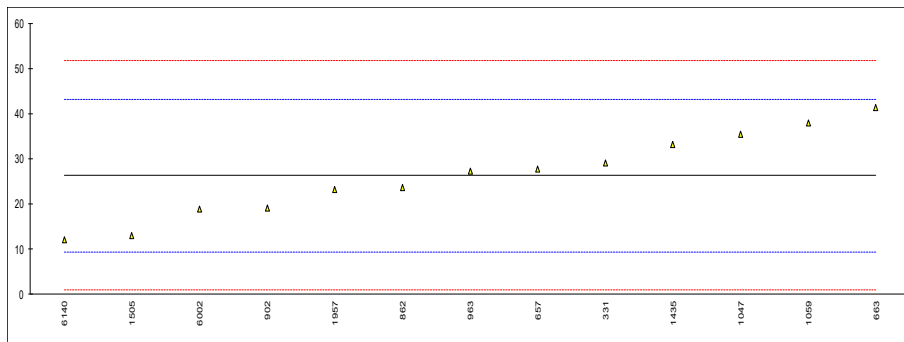
lab	method	value	mark	z(targ)	remarks
178	D93-A	206.5		-0.75	
179	D93	213.0		0.47	
225	D93-B	214.0		0.66	
237	D93-B	208		-0.47	
325		----		----	
331		----		----	
349	D93-B	210		-0.09	
360	D93-A	209.0		-0.28	
432	D93-A	214.5		0.75	
451		----		----	
496	D93-B	211.2		0.13	
541	D93-A	207.50		-0.56	
603		----		----	
614	D93-A	216		1.03	
633	D93-B	206.2		-0.80	
657		----		----	
663	D93-A	214.35		0.72	
840	D93-B	206.0		-0.84	
862	D93-A	203		-1.40	
902	D93-A	214		0.66	
912		----		----	
962	D93-A	206		-0.84	
963	D93-A	206		-0.84	
974	D93-A	208		-0.47	
1017	D93-A	211.0		0.09	
1023	D93-A	213		0.47	
1026	D93-A	210		-0.09	
1047		----		----	
1059	ISO2719-A	212.5		0.38	
1146	D93-A	209.5		-0.19	
1417	D93-A	212		0.28	
1431	D93-A	212.4		0.36	
1435	D93-A	216.5		1.13	
1456	D93-A	208.0		-0.47	
1505	D93-A	213		0.47	
1569	D93-A	212		0.28	
1740		----		----	
1827		----		----	
1957	D93-A	207		-0.65	
6002	ISO2719-A	209.5		-0.19	
6016		----		----	
6140	D93-A	213		0.47	
6194	D93-A	214.175		0.69	
6222	ISO2719-A	210		-0.09	
6235		----		----	

		<u>D93/ISO2719 method A only</u>	<u>D93/ISO2719 method B only</u>
normality	OK	OK	OK
n	34	27	6
outliers	0	0	0
mean (n)	210.49	210.68	209.23
st.dev. (n)	3.346	3.421	3.110
R(calc.)	9.37	9.58	8.71
st.dev.(D93-A:18)	5.338	5.342	---
R(D93-A:18)	14.95	14.96	---
compare			
R(D93-B:18)	10	---	10



Determination of Insoluble Color Bodies, membrane patch colorimetry on sample #19082;

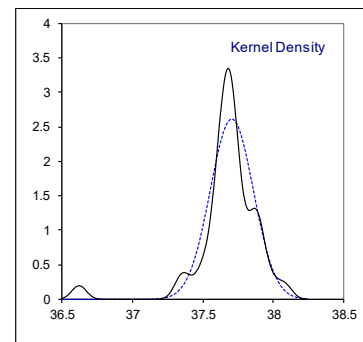
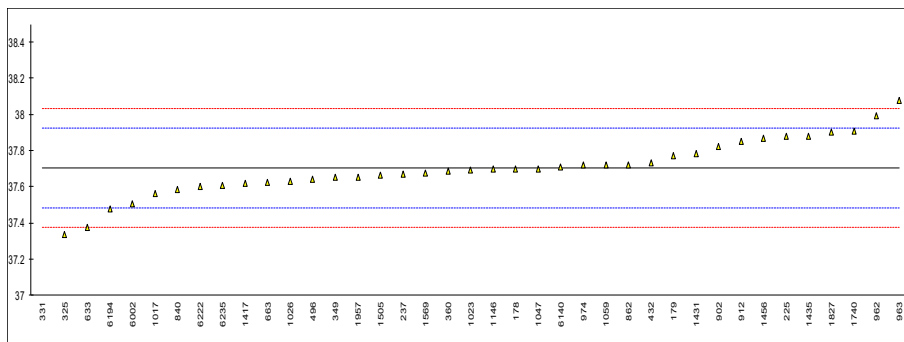
lab	method	value	mark	z(targ)	remarks
178		----		----	
179		----		----	
225		----		----	
237		----		----	
325		----		----	
331	D7843	29.1		0.33	
349		----		----	
360		----		----	
432		----		----	
451		----		----	
496		----		----	
541		----		----	
603		----		----	
614		----		----	
633		----		----	
657	D7843	27.8		0.18	
663	D7843	41.3		1.77	
840		----		----	
862	D7843	23.6		-0.32	
902	D7843	19.1		-0.85	
912		----		----	
962		----		----	
963	D7843	27.2		0.11	
974		----		----	
1017		----		----	
1023		----		----	
1026		----		----	
1047	D7843	35.44		1.08	
1059	D7843	37.8		1.36	
1146		----		----	
1417		----		----	
1431		----		----	
1435	D7843	33.2		0.82	
1456		----		----	
1505	D7843	13		-1.57	
1569		----		----	
1740		----		----	
1827		----		----	
1957	D7843	23.2		-0.36	
6002	D7843	18.9		-0.87	
6016		----		----	
6140	D7843	12		-1.69	
6194		----		----	
6222		----		----	
6235		----		----	
normality		OK			
n		13			
outliers		0			
mean (n)		26.28			
st.dev. (n)		9.148			
R(calc.)		25.61			
st.dev.(D7843:18)		8.472			
R(D7843:18)		23.72			



Determination of Kinematic Viscosity at 40°C on sample #19082; results in mm²/s

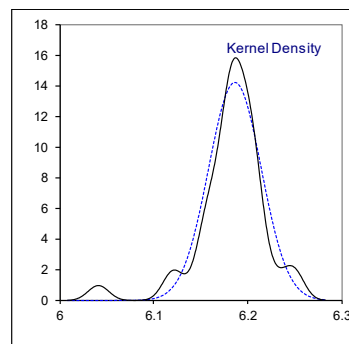
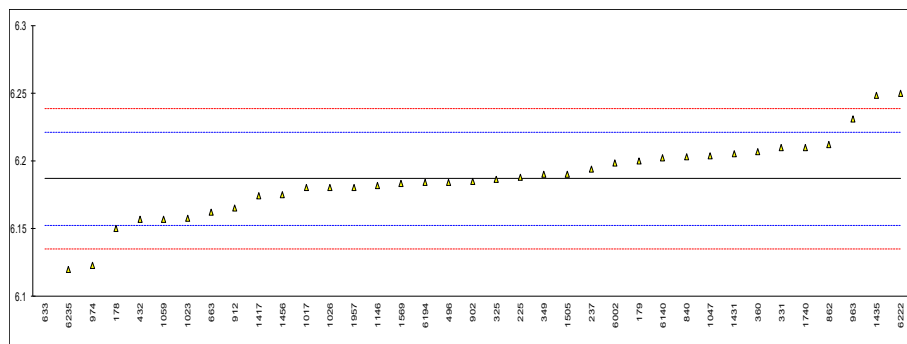
lab	method	value	mark	z(targ)	remarks
178	D445	37.7		-0.02	
179	D445	37.77		0.62	
225	D445	37.88		1.62	
237	D445	37.67115		-0.28	
325	D445	37.3366		-3.33	
331	D7279Mod.	36.62	R(0.01)	-9.84	
349	D445	37.65		-0.47	
360	ISO3104	37.684		-0.17	
432	D445	37.73		0.25	
451		----		----	
496	D445	37.644		-0.53	
541		----		----	
603		----		----	
614		----		----	
633	D7279 corr. to D445	37.375		-2.98	
657		----		----	
663	D445	37.622		-0.73	
840	D7042	37.586		-1.06	
862	D445	37.7224		0.18	
902	D445	37.82		1.07	
912	D445	37.85		1.34	
962	D445	37.99		2.62	
963	D445	38.08		3.44	
974	D445	37.72		0.16	
1017	D445	37.56		-1.29	
1023	D445	37.692		-0.09	
1026	D445	37.63		-0.66	
1047	ISO3104	37.70		-0.02	
1059	ISO3104	37.72		0.16	
1146	D445	37.696		-0.06	
1417	D445	37.62		-0.75	
1431	D7042	37.781		0.72	
1435	D7042	37.881		1.63	
1456	D445	37.87	C	1.53	first reported 38.8391
1505	D7042	37.664		-0.35	
1569	D445	37.675		-0.25	
1740	D445	37.91		1.89	
1827	D445	37.90		1.80	
1957	D7042	37.65		-0.47	
6002	ISO3104	37.508		-1.77	
6016		----		----	
6140	D7042	37.709		0.06	
6194	D445	37.47715		-2.05	
6222	D7042	37.60		-0.93	
6235	D445	37.610		-0.84	

normality OK
n 38
outliers 1
mean (n) 37.7022
st.dev. (n) 0.15251
R(calc.) 0.4270
st.dev.(D445:18) 0.10993
R(D445:18) 0.3078
compare
R(iis memo 1401) 0.6786



Determination of Kinematic Viscosity at 100°C on sample #19082; results in mm²/s

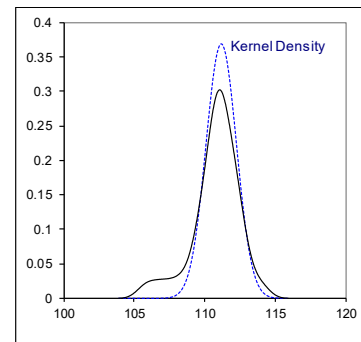
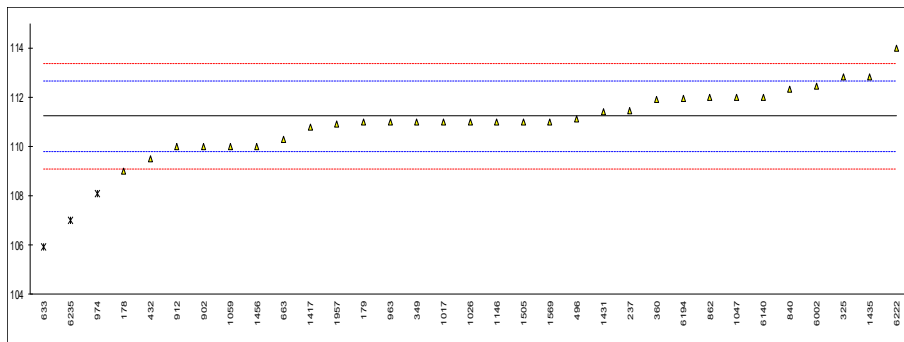
lab	method	value	mark	z(targ)	remarks
178	D445	6.15		-2.13	
179	D445	6.20		0.76	
225	D445	6.188		0.07	
237	D445	6.193596		0.39	
325	D445	6.186		-0.05	
331	D7279Mod.	6.21		1.34	
349	D445	6.190		0.18	
360	ISO3104	6.2067		1.15	
432	D445	6.157		-1.73	
451		----		----	
496	D445	6.1839		-0.17	
541		----		----	
603		----		----	
614		----		----	
633	D7279 corr. to D445	6.0416	R(0.01)	-8.40	
657		----		----	
663	D445	6.1620		-1.44	
840	D7042	6.2029		0.93	
862	D445	6.2117		1.44	
902	D445	6.185		-0.10	
912	D445	6.165		-1.26	
962		----		----	
963	D445	6.231		2.56	
974	D445	6.123		-3.69	
1017	D445	6.180		-0.39	
1023	D445	6.1579		-1.67	
1026	D445	6.18		-0.39	
1047	ISO3104	6.204		1.00	
1059	ISO3104	6.157		-1.73	
1146	D445	6.1814		-0.31	
1417	D445	6.174		-0.74	
1431	D7042	6.2049		1.05	
1435	D7042	6.2484		3.57	
1456	D445	6.175	C	-0.68	first reported 6.184
1505	D7042	6.190		0.18	
1569	D445	6.183		-0.22	
1740	D445	6.210		1.34	
1827		----		----	
1957	D7042	6.180		-0.39	
6002	ISO3104	6.1981		0.65	
6016		----		----	
6140	D7042	6.202		0.88	
6194	D445	6.18365		-0.18	
6222	D7042	6.25		3.66	
6235	D445	6.1199		-3.87	
normality		OK			
n		36			
outliers		1			
mean (n)		6.1868			
st.dev. (n)		0.02799			
R(calc.)		0.0784			
st.dev.(D445:18)		0.01728			
R(D445:18)		0.0484			
compare					
R(iis memo 1401)		0.1361			



Determination of Viscosity Index on sample #19082;

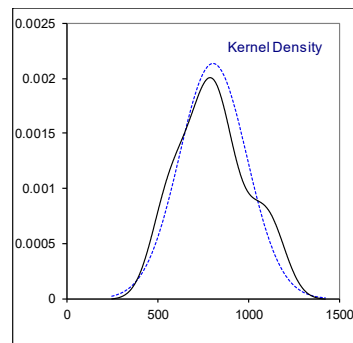
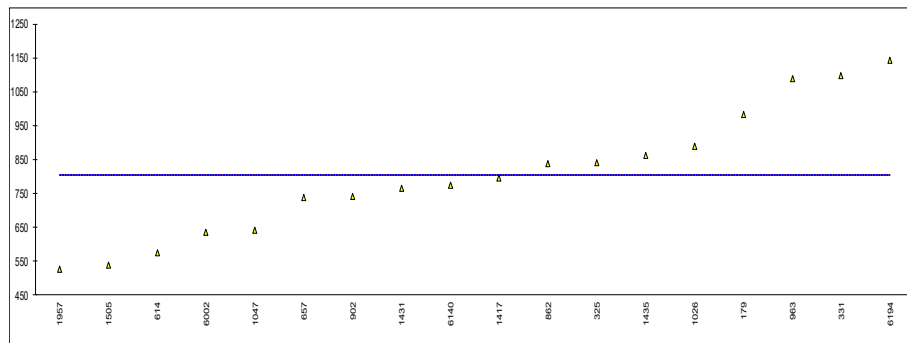
lab	method	value	mark	z(targ)	remarks
178	D2270	109		-3.11	
179	D2270	111		-0.31	
225		----		----	
237	D2270	111.445		0.31	
325	D2270	112.8		2.21	
331		----		----	
349	D2270	111		-0.31	
360	ISO2909	111.9		0.95	
432	D2270	109.5		-2.41	
451		----		----	
496	D2270	111.1		-0.17	
541		----		----	
603		----		----	
614		----		----	
633	D2270	105.9	ex	-7.45	excluded, outlier in Kinematic Viscosity 100°C
657		----		----	
663	D2270	110.28		-1.32	
840	D2270	112.3		1.51	
862	D2270	112		1.09	
902	D2270	110		-1.71	
912	D2270	110	E	-1.71	calculation error, iis calc = 109
962		----		----	
963	D2270	111		-0.31	
974	D2270	108.07	DG(0.05)	-4.41	
1017	D2270	111		-0.31	
1023		----		----	
1026	D2270	111		-0.31	
1047	ISO2909	112		1.09	
1059	ISO2909	110		-1.71	
1146	D2270	111		-0.31	
1417	D2270	110.8		-0.59	
1431	D2270	111.4		0.25	
1435	D2270	112.83		2.25	
1456	D2270	110	C	-1.71	first reported 105
1505	D2270	111		-0.31	
1569	D2270	111		-0.31	
1740		----		----	
1827		----		----	
1957	D2270	110.9		-0.45	
6002	ISO2909	112.46		1.73	
6016		----		----	
6140	D2270	112		1.09	
6194	D2270	111.956		1.03	
6222	D2270	114		3.89	
6235	ISO2909	107	E,DG(0.05)	-5.91	calculation error, iis calc = 108

normality OK
n 30
outliers 2 (+1 ex)
mean (n) 111.22
st.dev. (n) 1.080
R(calc.) 3.02
st.dev.(D2270:10) 0.714
R(D2270:10) 2



Determination of Oxidation Stability RPVOT on sample #19082; results in minutes

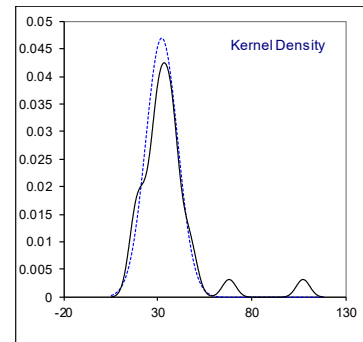
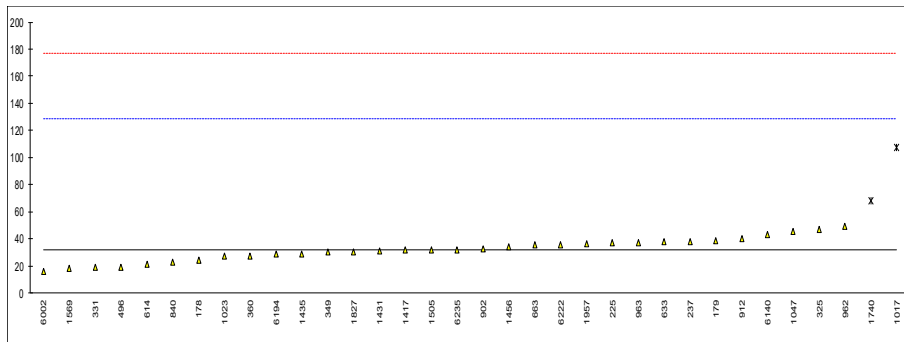
lab	method	value	mark	z(targ)	remarks
178		----		----	
179	D2272	983		----	
225		----		----	
237	D2272-A	>700		----	
325	D2272-A	841		----	
331	D2272-B	1096		----	
349		----		----	
360		----		----	
432		----		----	
451		----		----	
496		----		----	
541		----		----	
603		----		----	
614	D2272-A	573		----	
633		----		----	
657	D2272-B	738		----	
663		----		----	
840		----		----	
862	D2272-A	836		----	
902	D2272-A	741		----	
912		----		----	
962		----		----	
963	D2272-A	1087		----	
974		----		----	
1017		----		----	
1023		----		----	
1026	D2272-A	890		----	
1047	D2272-A	641		----	
1059	D2272-B	>1000		----	
1146		----		----	
1417	D2272-A	796		----	
1431	D2272-A	764		----	
1435	D2272-A	863		----	
1456		----		----	
1505	D2272-A	538		----	
1569	D2272-A	>1022		----	
1740		----		----	
1827		----		----	
1957	D2272-A	527		----	
6002	D2272-A	636		----	
6016		----		----	
6140	D2272-A	774		----	
6194	D2272-A	1142		----	
6222		----		----	
6235		----		----	
normality		OK			
n		18			
outliers		0			
mean (n)		803.7			
st.dev. (n)		186.52			
R(calc.)		522.3			
st.dev.(D2272:14a)		(65.62)			
R(D2272:14a)		(183.7)			



Determination of Water on sample #19082; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D6304-C	24		-0.17	
179	D6304-C	39		0.14	
225	D6304-A	37		0.10	
237	D6304-C	38.2		0.13	
325	D6304-C	47		0.31	
331	D6304Mod.	18.8		-0.28	
349	D6304-A	30		-0.04	
360	D6304-A	27.5		-0.10	
432		----		----	
451		----		----	
496	D6304-C	19		-0.27	
541		----		----	
603		----		----	
614	D6304-C	21		-0.23	
633	D6304-C	37.9		0.12	
657	D6304-C	<10		----	
663	D6304-C	35.75		0.08	
840	D6304-C	22.8		-0.19	
862		----		----	
902	D6304-A	32.4		0.01	
912	D6304-C	40		0.16	
962	D6304-A	49		0.35	
963	D6304-A	37		0.10	
974		----		----	
1017	D6304-C	107.4	R(0.01)	1.56	
1023	D6304-A	27		-0.11	
1026	D6304-C	<10		----	
1047	ISO12937	45.3		0.27	
1059	D6304-C	<30		----	
1146	D6304-C	<100		----	
1417	D6304-A	32		0.00	
1431	D6304-A	30.76		-0.03	
1435	D6304-A	29.0		-0.06	
1456	D6304-A	34.3		0.05	
1505	D6304-A	32		0.00	
1569	D6304-C	18		-0.29	
1740	D6304-C	68	R(0.01)	0.74	
1827	D6304-A	30.2		-0.04	
1957	D6304-A	36.5		0.09	
6002	ISO12937	16.19		-0.33	
6016		----		----	
6140	D6304-A	43		0.23	
6194	D6304-A	28.5436		-0.07	
6222	ISO12937	36		0.08	
6235	ISO12937	32		0.00	

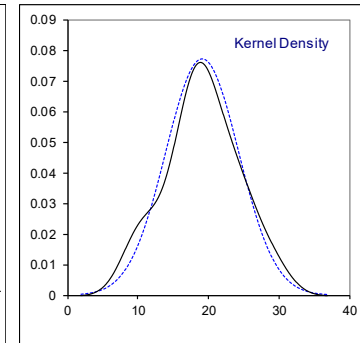
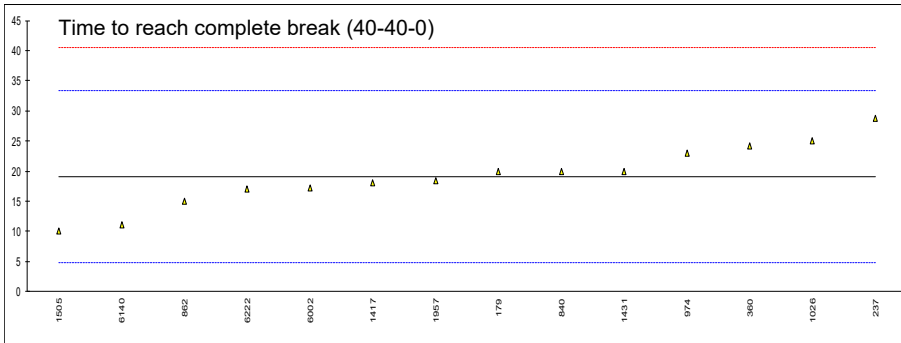
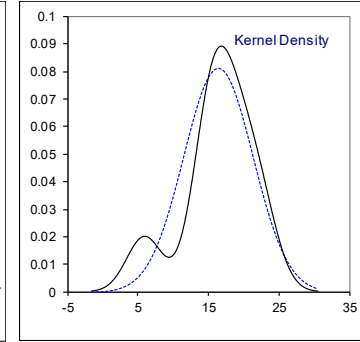
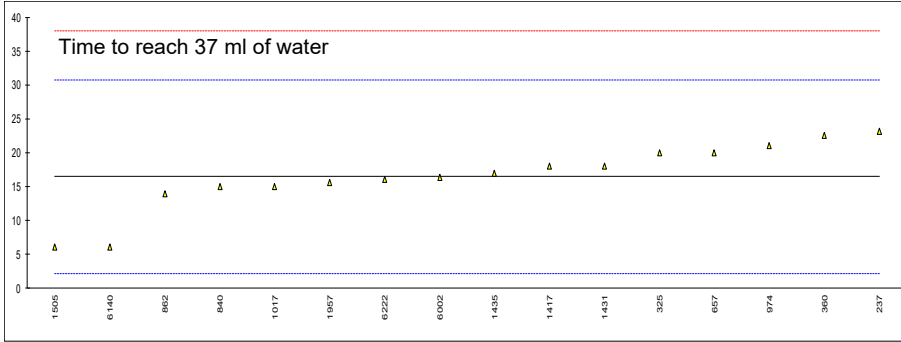
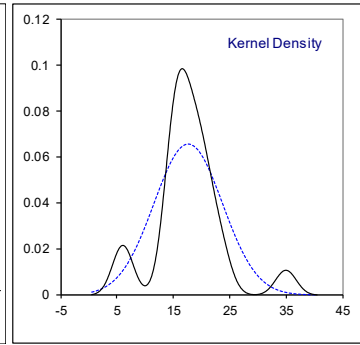
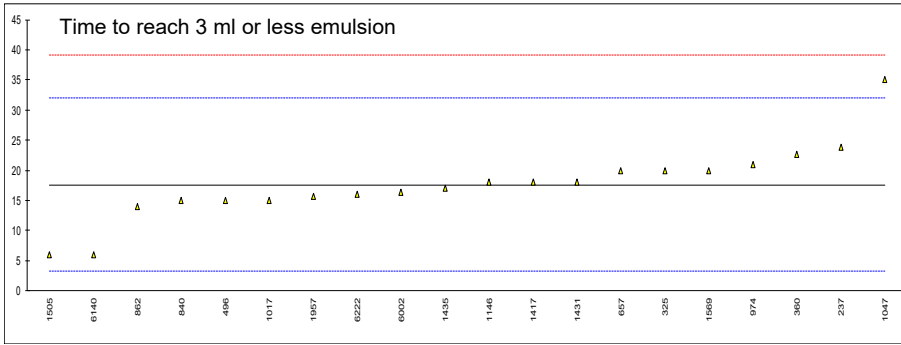
normality OK
n 32
outliers 2
mean (n) 32.10
st.dev. (n) 8.496
R(calc.) 23.79
st.dev.(D6304:16e1) 48.351
R(D6304:16e1) 135.38



Determination of Water Separability at 54°C, distilled water on sample #19082; results in minutes

lab method		time to reach 3 ml or less emulsion			time to reach 37 ml of water			time to reach complete break (40-40-0)			test aborted	time test aborted
		mark	z(targ)	mark	z(targ)	mark	z(targ)	mark	z(targ)			
178		----	----	----	----	----	----	----	----	----	----	
179	D1401	----	----	----	----	20	0.12	NO	----	----		
225		----	----	----	----	----	----	----	----	----	----	
237	D1401	23.8	0.86	23.1	0.93	28.8	1.36	NO	----	----		
325	D1401	20	0.33	20	0.49	----	----	YES	----	30		
331		----	----	----	----	----	----	----	----	----	----	
349		----	----	----	----	----	----	----	----	----	----	
360	ISO6614	22.6	0.70	22.6	0.86	24.2	0.71	NO	----	----		
432		----	----	----	----	----	----	----	----	----	----	
451		----	----	----	----	----	----	----	----	----	----	
496	D1401	15	-0.37	----	----	----	----	NO	----	----		
541		----	----	----	----	----	----	----	----	----	----	
603		----	----	----	----	----	----	----	----	----	----	
614		----	----	----	----	----	----	----	----	----	----	
633		----	----	----	----	----	----	----	----	----	----	
657	D1401	20	0.33	20	0.49	----	----	NO	----	20		
663		----	----	----	----	----	----	----	----	----	----	
840	D1401	15	-0.37	15	-0.21	20	0.12	NO	----	----		
862	D1401	14	-0.51	14	-0.35	15	-0.58	NO	----	15		
902		----	----	----	----	----	----	YES	----	25		
912		----	----	----	----	----	----	----	----	----	C	
962		----	----	----	----	----	----	----	----	----	----	
963		----	----	----	----	----	----	----	----	----	----	
974	D1401	21	0.47	21	0.63	23	0.54	----	----	----	----	
1017	D1401	15	-0.37	15	-0.21	----	----	YES	----	15		
1023		----	----	----	----	25	0.82	NO	----	----		
1026	D1401	----	----	----	----	----	----	YES	----	35		
1047	ISO6614	35	2.43	----	----	----	----	----	----	----	----	
1059		----	----	----	----	----	----	----	----	----	----	
1146	D1401	18	0.05	----	----	----	----	----	----	----	----	
1417	D1401	18	0.05	18	0.21	18	-0.16	NO	----	----		
1431	D1401	18	0.05	18	0.21	20	0.12	NO	----	----		
1435	ISO6614	17.0	-0.09	17.0	0.07	----	----	NO	----	----		
1456		----	----	----	----	----	----	----	----	----	----	
1505	D1401	6	-1.63	6	-1.47	10	-1.28	NO	----	----		
1569	D1401	20	0.33	----	----	----	----	----	----	----	----	
1740		----	----	----	----	----	----	----	----	----	----	
1827		----	----	----	----	----	----	----	----	----	----	
1957	D1401	15.73	-0.27	15.65	-0.12	18.43	-0.10	NO	----	----		
6002	ISO6614	16.4	-0.17	16.4	-0.01	17.25	-0.26	NO	----	17.25		
6016		----	----	----	----	----	----	----	----	----	----	
6140	D1401	6	-1.63	6	-1.47	11	-1.14	NO	----	----		
6194		----	----	----	----	----	----	----	----	----	----	
6222	D1401	16	-0.23	16	-0.07	17	-0.30	NO	----	----		
6235		----	----	----	----	----	----	----	----	----	----	
	normality	not OK			suspect			OK				
	n	20			16			14				
	outliers	0			0			0				
	mean (n)	17.63			16.48			19.12				
	st.dev. (n)	6.095			4.916			5.168				
	R(calc.)	17.06			13.76			14.47				
	st.dev.(D1401:18b)	7.143			7.143			7.143				
	R(D1401:18b)	20			20			20				

Lab 902 first reported 30



Determination of Water Separability at 54°C, distilled water on sample #19082; 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	D1401	40		39		1	
331		----		----		----	
349		----		----		----	
360	ISO6614	42		38		0	
432		----		----		----	
451		----		----		----	
496	D1401	41		36		3	
541		----		----		----	
603		----		----		----	
614		----		----		----	
633		----		----		----	
657	D1401	43		37		0	
663		----		----		----	
840		----		----		----	
862	D1401	40		40		0	
902		41	C	39	C	0	C
912		----		----		----	
962		----		----		----	
963		----		----		----	
974		----		----		----	
1017	D1401	43		37		0	
1023		----		----		----	
1026	D1401	40		40		0	
1047	ISO6614	42		37		1	
1059		----		----		----	
1146	D1401	40		37		3	
1417	D1401	40		40		0	
1431		----		----		----	
1435		----		----		----	
1456		----		----		----	
1505		----		----		----	
1569		----		----		----	
1740		----		----		----	
1827		----		----		----	
1957	D1401	40		40		0	
6002	ISO6614	40		40		0	
6016		----		----		----	
6140		----		----		----	
6194		----		----		----	
6222		----		----		----	
6235		----		----		----	

Lab 902 first reported 30, 5 and 45 respectively

Determination of Demulsibility Characteristics, Steam on sample #19082; results in s en mL

lab	method	time to reach 20ml of oil within 20 min	mark	volume oil after 20 min	mark	remarks
178		----		----		
179		----		----		
225		----		----		
237		----		----		
325		----		----		
331		----		----		
349		----		----		
360		----		----		
432		----		----		
451		----		----		
496		----		----		
541		----		----		
603		----		----		
614		----		----		
633		----		----		
657		----		----		
663		----		----		
840		----		----		
862		----		----		
902		----		----		
912		----		----		
962		----		----		
963		----		----		
974		----		----		
1017		----		----		
1023		----		----		
1026		----		----		
1047		----		----		
1059		----		----		
1146		----		----		
1417	IP19	570		20		
1431		----		----		
1435		----		----		
1456		----		----		
1505		----		----		
1569		----		----		
1740		----		----		
1827		----		----		
1957		----		----		
6002		----		----		
6016		----		----		
6140		----		----		
6194		----		----		
6222		----		----		
6235		----		----		

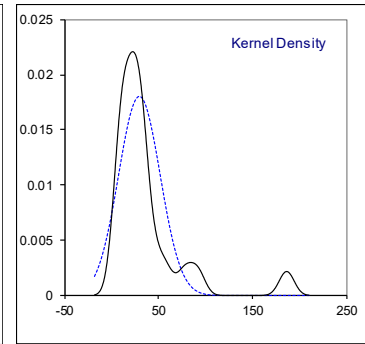
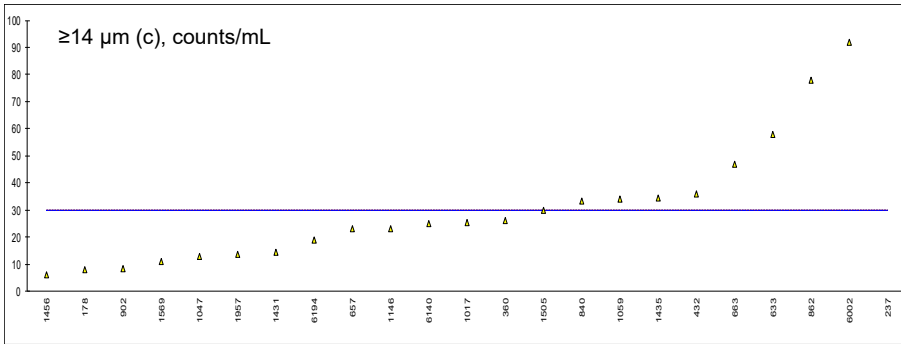
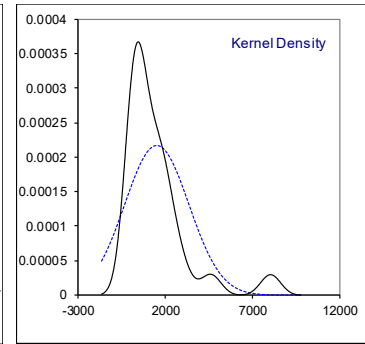
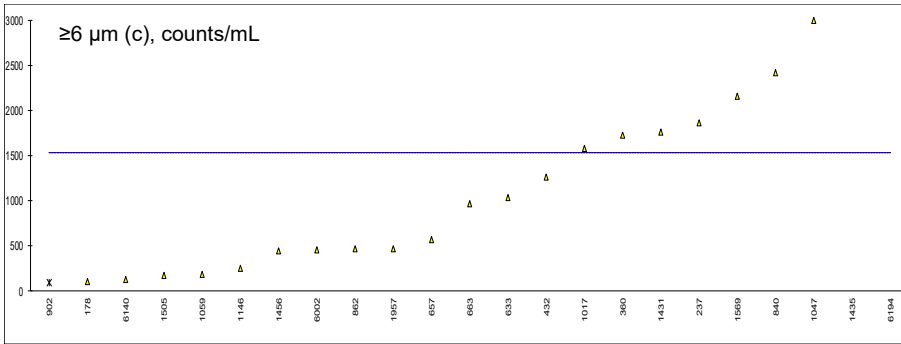
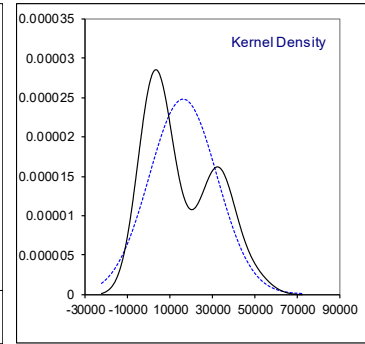
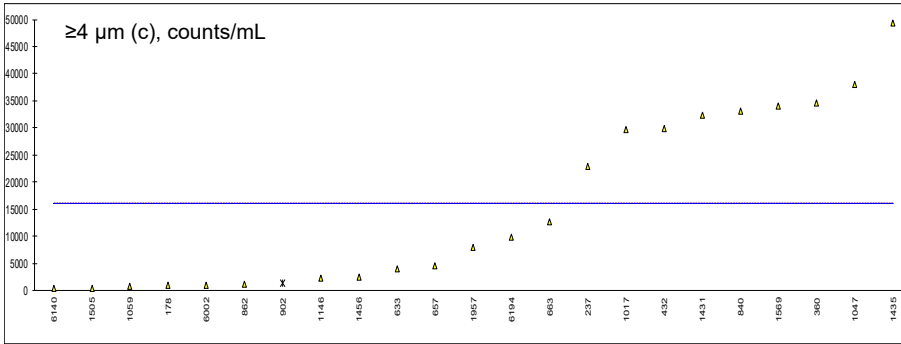
Determination of Water Separation Ability, Steam, time to reach the 55 mm measuring point (oil-water phase) on sample #19082; results in s

lab	method	value	mark	remarks
178		----		
179		----		
225		----		
237		----		
325		----		
331		----		
349		----		
360		----		
432		----		
451		----		
496	DIN51589	267		
541		----		
603		----		
614		----		
633		----		
657		----		
663		----		
840		----		
862		----		
902		----		
912		----		
962		----		
963		----		
974		----		
1017		----		
1023		----		
1026		----		
1047		----		
1059		----		
1146		----		
1417		----		
1431		----		
1435		----		
1456		----		
1505		----		
1569		----		
1740		----		
1827		----		
1957		----		
6002	In house	211		
6016		----		
6140		----		
6194		----		
6222		----		
6235		----		

Determination of Level of Contamination on sample #19082; 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	INH-1185	1026		----	106		----	8		----
179		----		----	----		----	----		----
225		----		----	----		----	----		----
237	ISO4407	22942.5		----	1865.6		----	186.6	ex,C	----
325		----		----	----		----	----		----
331		----		----	----		----	----		----
349		----		----	----		----	----		----
360	ISO4406	34668		----	1723		----	26		----
432	ISO11500	29813		----	1256		----	36		----
451		----		----	----		----	----		----
496		----		----	----		----	----		----
541		----		----	----		----	----		----
603		----		----	----		----	----		----
614		----		----	----		----	----		----
633	D7647	3933		----	1039		----	58		----
657	ISO4406	4557		----	567		----	23		----
663	D7647	12716		----	961		----	47		----
840	D7647	33079.8		----	2418.4	C	----	33.4		----
862	ISO4407	1111		----	462		----	78		----
902	D7647	1423	ex	----	96.7	ex	----	8.3		----
912		----		----	----		----	----		----
962		----		----	----		----	----		----
963		----		----	----		----	----		----
974		----		----	----		----	----		----
1017	ISO11500	29773.33		----	1573.45		----	25.30		----
1023		----		----	----		----	----		----
1026		----		----	----		----	----		----
1047		38077	C	----	2989	C	----	13	C	----
1059	D7647	830	C	----	186	C	----	34	C	----
1146	ISO11500	2304		----	252		----	23		----
1417		----		----	----		----	----		----
1431		32306.5		----	1761.1		----	14.4		----
1435		49378.14		----	4629.17		----	34.40		----
1456	ISO4406	2423.3		----	440.6		----	6		----
1505	D7647	440		----	170		----	30		----
1569		33991		----	2154		----	11		----
1740		----		----	----		----	----		----
1827		----		----	----		----	----		----
1957	ISO4407	8041.1		----	471.4		----	13.8		----
6002	D7596	1028.0		----	452.6		----	91.8		----
6016		----		----	----		----	----		----
6140	D7647	325		----	125		----	25		----
6194	ISO4407	9823.0		----	8054	C	----	19	C	----
6222		----		----	----		----	----		----
6235		----		----	----		----	----		----
	normality	OK			not OK			not OK		
	n	22			22			22		
	outliers	0 (+1 ex)			0 (+1 ex)			0 (+1 ex)		
	mean (n)	16026.7			1529.8			29.9		
	st.dev. (n)	16048.63			1840.64			22.09		
	R(calc.)	44936.2			5153.8			61.9		
	st.dev.(D7647:10)	(6467.90)			(415.24)			(14.43)		
	R(D7647:10)	(18110.1)			(1162.7)			(40.4)		

Lab 237 first reported 971.2, the test result for ≥14 µm was excluded for the test result in counts/mL did not match the ISO scale number
 Lab 840 first reported 3237.5
 Lab 902 the test results for ≥4 µm and 6 µm were excluded for the test results in counts/mL did not match the ISO scale number
 Lab 1047 first no test results reported (first reported as scale number)
 Lab 1059 first reported 563, 208 and 129 respectively
 Lab 6194 first reported 6554.0 and 4405.5 respectively



Determination of Level of Contamination on sample #19082; results in ISO 4406 scale numbers

lab	method	≥ 4 µm(c)	mark	z(targ)	≥ 6 µm(c)	mark	z(targ)	≥ 14 µm(c)	mark	z(targ)
178	INH-1185	17		----	14		----	10		----
179		----		----			----			----
225		----		----			----			----
237	ISO4406	22		----	18		----	14	ex,C	----
325		----		----			----			----
331		----		----			----			----
349		----		----			----			----
360	ISO4406	22		----	18		----	12		----
432	ISO4406	22		----	17		----	12		----
451		----		----			----			----
496		----		----			----			----
541		----		----			----			----
603		----		----			----			----
614	ISO4406	22		----	19		----	13		----
633	ISO4406	19		----	17		----	13		----
657	ISO4406	19		----	16		----	12		----
663	ISO4406	21		----	17		----	13		----
840	ISO4406	22		----	18	C	----	12		----
862	ISO4406	17		----	16		----	13		----
902	D7647	17	ex	----	13	ex	----	10		----
912		----		----			----			----
962		----		----			----			----
963		----		----			----			----
974		----		----			----			----
1017	ISO4406	22		----	18		----	12		----
1023	ISO4406	22		----	18		----	10		----
1026		21	C	----	19	C	----	15	C	----
1047	ISO4406	22	C	----	19	C	----	11	C	----
1059	ISO4406	17	C	----	15		----	12	C	----
1146		18		----	15		----	12		----
1417	ISO4406	24		----	18		----	12		----
1431		22		----	18		----	11		----
1435	ISO4406	23		----	19		----	12		----
1456	ISO4406	18		----	16		----	10		----
1505	ISO4406	16		----	15		----	12		----
1569	ISO4406	22		----	18		----	11		----
1740	ISO4406	22		----	17		----	11		----
1827		----		----			----			----
1957	ISO4406	20		----	16		----	11		----
6002	ISO4406	17		----	16		----	14		----
6016		----		----			----			----
6140		16		----	14		----	12		----
6194	ISO4406	20		----	20		----	11	C	----
6222		----		----			----			----
6235		----		----			----			----
	normality	OK			OK			OK		
	n	27			27			27		
	outliers	0 (+1 ex)			0 (+1 ex)			0 (+1 ex)		
	mean (n)	20.2			17.1			11.8		
	st.dev. (n)	2.39			1.62			1.21		
	R(calc.)	6.7			4.5			3.4		
	st.dev.(D7647:10)	(0.61)			(0.43)			(0.71)		
	R(D7647:10)	(1.7)			(1.2)			(2.0)		

Lab 237 first reported 17, the test result for ≥14 µm was excluded for the test result in ISO scale number did not match the counts/mL

Lab 840 first reported 19

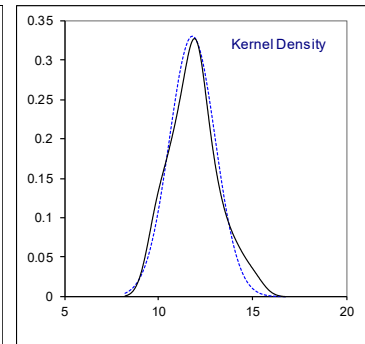
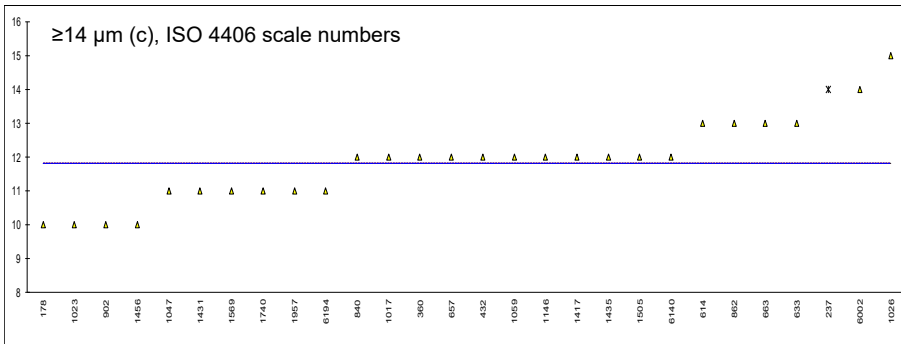
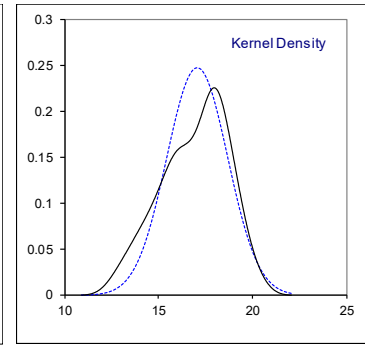
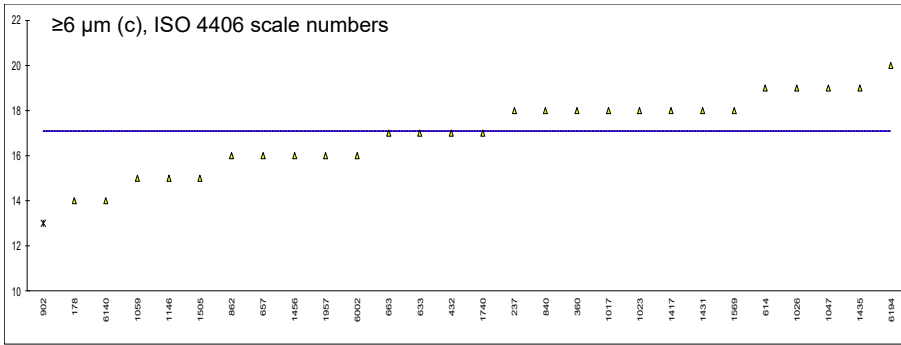
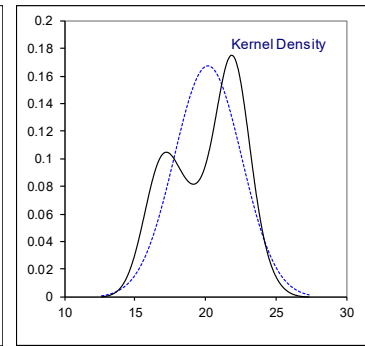
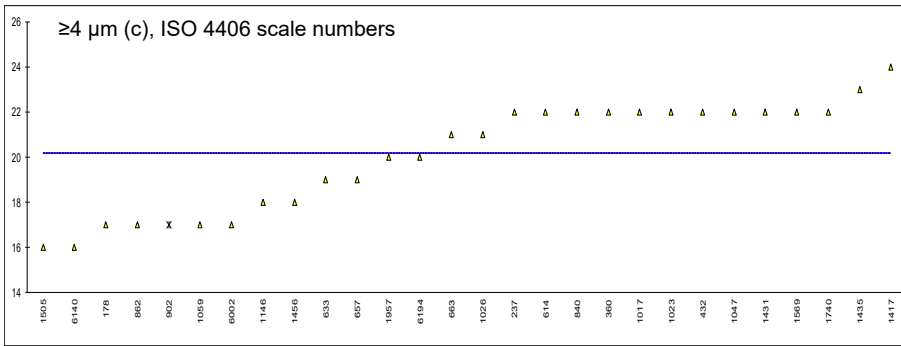
Lab 902 the test results for ≥4 µm and 6 µm were excluded for the test results in ISO scale number did not match the counts/mL

Lab 1026 first reported 22, 21 and 18 respectively

Lab 1047 first reported 38077, 2989 and 13 respectively (reported as counts/mL)

Lab 1059 first reported 16 and 14 respectively

Lab 6194 first reported 19



APPENDIX 2

Number of participants per country

1 lab in ARGENTINA
2 labs in AUSTRALIA
2 labs in AUSTRIA
3 labs in BELGIUM
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
3 labs in GREECE
1 lab in INDIA
1 lab in KAZAKHSTAN
4 labs in MALAYSIA
2 labs in NETHERLANDS
1 lab in NIGERIA
1 lab in NORWAY
1 lab in PHILIPPINES
3 labs in POLAND
2 labs in SAUDI ARABIA
1 lab in SINGAPORE
1 lab in SLOVENIA
2 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

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	= Material Safety Data Sheet

Literature:

- 1 iis Interlaboratory Studies, Protocol for the Organization, 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 IP 367:84
- 10 DIN 38402 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 2002, Analyst 2002, 127, 1359-1364
- 15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)
- 16 R.G. Visser and C. Nijssen-Wester, Estimation of reproducibility and measurement uncertainty of a viscosity test method from proficiency test data, Accred Qual Assur (2015) 20:125-129, DOI 10.1007/s00769-015-1110-y