

Results of Proficiency Test
Turbine Oil (used)
May 2018

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2013, the Institute for Interlaboratory Studies (iis) organizes a proficiency test for used Turbine Oil. During the annual proficiency testing program 2017/2018, it was decided to continue the round robin for the analysis of used Turbine Oil. In this interlaboratory study 43 laboratories in 31 different countries registered for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2018 used Turbine Oil proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, The Netherlands, was the organizer of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send one bottle of 1L (labelled #18077) 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 Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC 17043:2010 (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 these proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organization, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4). 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 used Turbine Oil was obtained from a third party. The 60 litre bulk material was homogenized and 49 amber glass bottles of 1 litre were filled and labelled #18077. The homogeneity of the subsamples #18077 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/m ³
Sample #18077-1	959.98
Sample #18077-2	960.00
Sample #18077-3	959.98
Sample #18077-4	960.05
Sample #18077-5	959.99
Sample #18077-6	960.03
Sample #18077-7	959.99
Sample #18077-8	960.04

Table 1: homogeneity test results of subsamples #18077

From the above test results, the repeatability was calculated and compared with 0.3 times the corresponding reproducibility in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Density at 15°C in kg/m ³
r(observed)	0.08
reference test method	ASTM D1298:12b(2017)
0.3 x R(reference test method)	0.45

Table 2: evaluation of the repeatabilities of the subsamples #18077

The calculated repeatability was in agreement with 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneity of the subsamples #18077 was assumed.

To each of the participating laboratories, one sample of 1 L amber glass bottle (labelled #18077) was sent on April 25, 2018.

2.5 STABILITY OF THE SAMPLES

The stability of the Turbine Oil (used), packed in the 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 #18077: Acid Number, Color ASTM, Density at 15°C, Flash Point (COC & PMcc), Insoluble Color Bodies, Kinematic Viscosity at 40°C and at 100°C, Viscosity Index, Oxidation Stability RPVOT, Water by KF, Water separability, Steam Demulsibility Characteristics, Steam Water Separation Ability and Level of contamination (parts per ml and ISO Class).

Also, some additional questions were requested about Acid Number (method ASTM D664).

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 results, but report as much significant figures as possible. It was also requested not to report 'less than' 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 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 reanalyses). 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 March 2017 (iis-protocol, version 3.4).

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 ISO 5725 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 visualise 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.

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 problems were encountered with the dispatch of the samples to laboratories. All participating laboratories reported test results, but not all laboratories were able to report all analyses requested. In total 43 participants reported 581 test results. Observed were 10 outlying results, which is 1.7% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER TEST

In this section, the results are discussed per test. The methods, which are used by the various laboratories, are taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the original data. 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. D1500) and an added designation for the year that the method was adopted or revised (e.g. D1500:12). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D1500:12(2017)). In the results tables of Appendix 1 only the method number and year of adoption or revision e.g. D1500:12 will be used.

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.

Acid Number: 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 D664:17a, method A (both Buffer End Point pH 11 and the less strict Inflection Point). 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 11).” Remarkably, of all the laboratories that reported a selection of the end point, around fifty percent reported to have used the Inflection Point instead of the Buffer End Point (BEP). When evaluating the results using the Inflection Point and the BEP separately, the calculated reproducibility for using a determination with BEP is still not in agreement with the requirements of ASTM D664:17a, method A (BEP pH 11), but the calculated reproducibility for using a determination with Inflection Point is in agreement with the less strict requirements of ASTM D664:17a, method A (Inflection Point). The volume of titration solvent used did not appear to have an influence.

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

Density at 15°C: This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D1298:12b(2017).

Flash Point COC: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D92:16b.

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. When evaluated separately for procedure A and procedure B, the respective calculated reproducibilities are also in agreement with the respective methods.

Insoluble Color Bodies: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D7843:16.

Kin.Visco.at 40°C: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated reproducibility based on the reproducibilities as observed in a large number of iis PTs on used oils (lit. 16). The kinematic viscosity is lower than the range that is mentioned in ASTM D445:17a (25-125 mm²/s), therefore the strict requirements of ASTM D445:17a were not used.

Kin.Visco.at 100°C: This determination was problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is not in agreement with the estimated reproducibility based on the reproducibilities as observed in a large number of iis PTs on used oils (lit. 16). The kinematic viscosity is lower than the range that is mentioned in ASTM D445:17a (6-16 mm²/s), therefore the strict requirements of ASTM D445:17a were not used.

Viscosity Index This determination was problematic. One statistical outlier was observed and one other test result was excluded. Since the performance of the kinematic viscosity test at 40°C and 100°C was calculated using the target reproducibility based on iis historical data (lit. 16) and not against the very strict requirements of ASTM D445, the larger variation present in these data sets will give more variation in the results of the Viscosity Index calculation. Therefore, no z-scores were calculated. From the test results reported for kinematic viscosity at 40°C and at 100°C, iis calculated the Viscosity Indices and compared the results with the reported Viscosity Indices. Four participants did not calculate the VI index correctly. A separate statistical evaluation was done on the group of (iis) calculated viscosity indices. No improvement was found for the resulting calculated reproducibility of the group.

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

Water by KF: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D6304:16e1.

Water separability: This determination was not problematic. No statistical outliers were observed. All reporting laboratories, except one, reported a complete break of (40-40-0) or emulsion less than 3 ml. The calculated reproducibilities for time to reach 3 ml or less emulsion, time to reach 37 ml of water and time to reach complete break (40-40-0) are all in agreement with the requirements of ASTM D1401:18a.

Steam Demulsibility Char.: Only three participants reported a result between 76 and 120 seconds. No z-scores were calculated.

Steam Water Separation Ability.: Only one participant reported a result of 79 seconds. No z-scores were calculated.

Level of Contamination: This determination was problematic. No statistical outliers were observed and eight test results were excluded. The calculated reproducibilities for number of particles in counts/ml for $\geq 4\mu\text{m}$, $\geq 6\mu\text{m}$ and $\geq 14\mu\text{m}$ after rejection of the suspect data are not in agreement with the requirements of ASTM D7647:10(2018). This determination was also problematic for the test results reported in ISO scale numbers. Three statistical outliers were observed and eight other test results were excluded. The calculated reproducibilities for number of particles in ISO scale numbers $\geq 6\mu\text{m}$ and $\geq 14\mu\text{m}$ after rejection of the suspect data are not in agreement with the requirements of ASTM D7647:10(2018). The calculated reproducibility in ISO scale numbers for $\geq 4\mu\text{m}$ is in good 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 that participated. The average results, calculated reproducibilities and reproducibilities derived from reference test methods (for example ASTM or IP standards), are compared in the next table.

parameter	unit	n	average	2.8 * sd	R(lit)
Acid Number, total	mg KOH/g	37	0.19	0.19	0.09
Color ASTM		17	3.6	0.6	1
Density at 15°C	kg/m ³	39	959.9	0.8	1.5
Flash Point COC	°C	29	224	24	18
Flash Point PMcc	°C	37	188	11	13
Insoluble Color Bodies		11	15.9	7.3	9.2
Kinematic Viscosity at 40°C	mm ² /s	37	12.78	0.15	0.23
Kinematic Viscosity at 100°C	mm ² /s	36	3.20	0.08	0.07
Viscosity Index		31	116	12	(2)
Oxidation Stability RPVOT	minutes	16	802	305	183
Water by KF	mg/kg	34	933	320	1022
Water Separability at 54°C					
- Time to reach 3 ml or less emulsion	minutes	16	6	5	20
- Time to reach 37 of water	minutes	15	6	5	20
- Time to reach complete break	minutes	14	7	7	20
Demulsibility Characteristics, Steam	s	3	76-120	n.e	n.e.
Water Separation Ability, Steam	s	1	79	n.e.	n.e.
Level of Contamination ≥4µm (c)	counts/ml	22	20235	28165	22866
Level of Contamination ≥6µm (c)	counts/ml	24	4561	6908	3466
Level of Contamination ≥14µm (c)	counts/ml	21	176	302	238
Level of Contamination ≥4µm (c)	ISO scale	22	22	2	2
Level of Contamination ≥6µm (c)	ISO scale	27	19	3	1
Level of Contamination ≥14µm (c)	ISO scale	24	15	3	2

Table 3: reproducibilities of tests of sample #18077

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 tests that are problematic have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF MAY 2018 WITH PREVIOUS PTS

	May 2018	May 2017	May 2016	May 2015	May 2014
Number of reporting labs	43	34	38	39	29
Number of results reported	581	508	474	398	301
Statistical outliers	10	16	15	36	10
Percentage outliers	1.7%	3.1%	3.2%	9.0%	3.3%

Table 4: comparison with previous proficiency tests

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

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

Determination	May 2018	May 2017	May 2016	May 2015	May 2014
Acid Number, total	--	-	-	+	--
Color ASTM	+	+	+/-	+	++
Density at 15°C	+	+	++	++	+/-
Flash Point COC	-	+	(--)	n.e.	n.e.
Flash Point PMcc	+	+	+/-	+/-	-
Insoluble Color Bodies	+	-	+/-	n.e.	n.e.
Kinematic Viscosity at 40°C	+	-	++	++	++
Kinematic Viscosity at 100°C	+/-	--	+/-	+	+/-
Viscosity Index	(--)	--	--	--	--
Oxidation Stability RPVOT	-	-	++	n.e.	n.e.
Water by KF	++	++	++	++	++
Water Separability	++	++	n.e.	+	n.e.
Level of Contamination $\geq 4\mu\text{m}$	-	-	--	--	n.e.
Level of Contamination $\geq 6\mu\text{m}$	--	--	--	--	n.e.
Level of Contamination $\geq 14\mu\text{m}$	-	-	-	--	n.e.

Table 5: comparison determinations against the standard

The performance of the determinations against the requirements of the respective reference test methods is listed in the above table. The following performance categories were used:

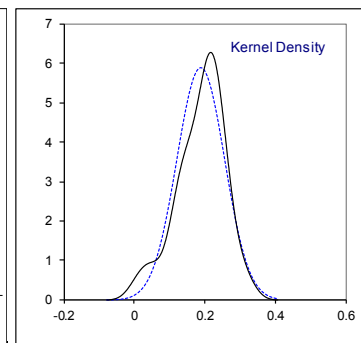
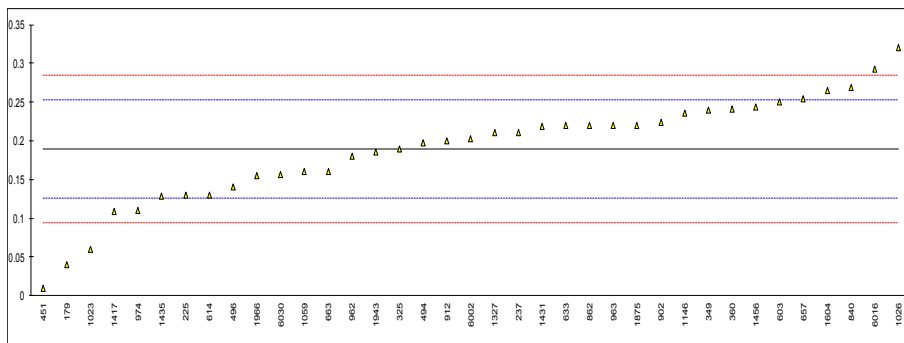
- ++: group performed much better than the reference test method
- + : group performed better than the reference test method
- +/-: group performance equals the reference test method
- : group performed worse than the reference test method
- : group performed much worse than the reference test method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

APPENDIX 1

Determination of Acid Number (total) on sample #18077; results in mg KOH/g

lab	method	value	mark	z(targ)	determination of end point	volume of titration solvent
178	D664Mod.	<0.10		----	----	----
179	D664-A	0.04		-4.70	Buffer End Point (pH 11)	60 mL
225	D974	0.13		-1.86	----	----
237	D664-A	0.211		0.69	----	----
325	D664-A	0.19		0.03	Buffer End Point (pH 11)	125 mL
331		----		----	----	----
349	D664-A	0.24		1.61	Inflection Point	125 mL
360	D664-A	0.241		1.64	Inflection Point	60 mL
432		----		----	----	----
451	In house	0.01		-5.64	----	----
494	D664-A	0.197		0.25	Inflection Point	125 mL
496	D664-A	0.14		-1.55	Buffer End Point (pH 11)	60 mL
541		----		----	----	----
603	D664-A	0.2498		1.91	Inflection Point	125 mL
614	D974	0.13		-1.86	----	60 mL
633	D664-A	0.22		0.98	Inflection Point	125 mL
657	D664-A	0.254		2.05	----	----
663	D664-A	0.1605		-0.90	Buffer End Point (pH 11)	125 mL
840	D664-A	0.269		2.52	Inflection Point	125 mL
862	D664-A	0.22		0.98	----	----
902	D664-A	0.224		1.10	Buffer End Point (pH 11)	60 mL
912	D664-A	0.20		0.34	----	----
962	D974	0.18		-0.29	----	----
963	D664-A	0.22		0.98	Inflection Point	60 mL
974	D664-A	0.11		-2.49	----	60 mL
1017		----		----	----	----
1023	In house	0.06		-4.07	----	----
1026	D664-A	0.32		4.13	----	----
1059	ISO6619	0.16		-0.92	Buffer End Point (pH 11)	60 mL
1146	D664-A	0.235		1.45	Buffer End Point (pH 11)	125 mL
1327	D664-B	0.21		0.66	Buffer End Point (pH 11)	60 mL
1417	D664-A	0.109		-2.52	Buffer End Point (pH 11)	125 mL
1431	D664-A	0.2183		0.92	Inflection Point	60 mL
1435	D664-A	0.129		-1.89	Buffer End Point (pH 11)	----
1456	D974	0.2433		1.71	----	125 mL
1604	D664-A	0.2646		2.38	----	----
1875	ISO6618	0.22		0.98	Inflection Point	60 mL
1943	ISO6618	0.185		-0.13	----	----
1966	ISO6618	0.1548		-1.08	----	----
6002	D664-A	0.202		0.41	Inflection Point	60 mL
6016	D664-A	0.292		3.24	Inflection Point	60 mL
6030	GOST5985	0.1560		-1.04	----	----
6190		----		----	----	----

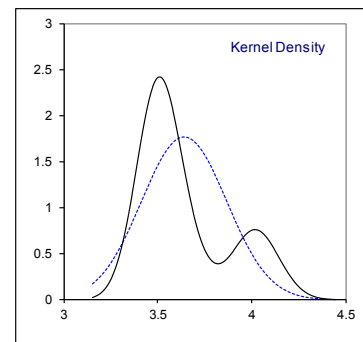
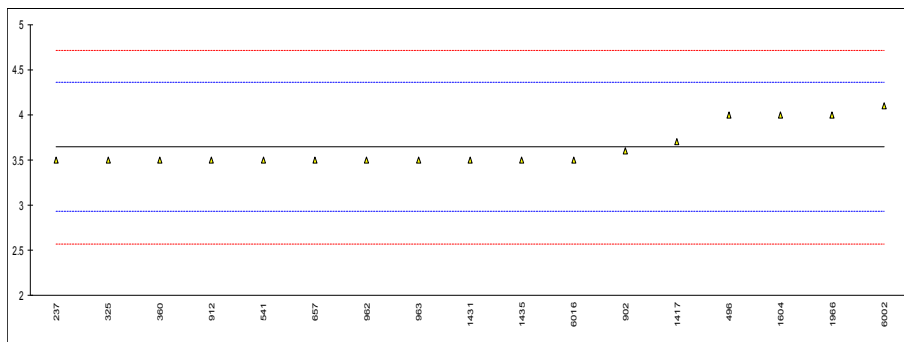
		<u>BEP (pH 11) only</u>	<u>Inflection Point only</u>
normality	OK	OK	OK
n	37	10	11
outliers	0	0	0
mean (n)	0.1891	0.1598	0.2336
st.dev. (n)	0.06788	0.05914	0.02855
R(calc.)	0.1901	0.1656	0.0799
st.dev.(D664-A:17a, BEP (pH 11) 60mL)	0.03173	0.02692	----
R(D664-A:17a, BEP (pH 11) 60mL)	0.0888	0.0754	----
compare			
st.dev.(D664-A:17a, Inflection - 60 mL)	0.03666	----	0.04359
R(D664-A:17a, Inflection - 60 mL)	0.1026	----	0.1221



Determination of Color ASTM on sample #18077;

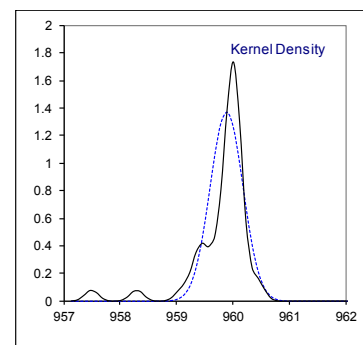
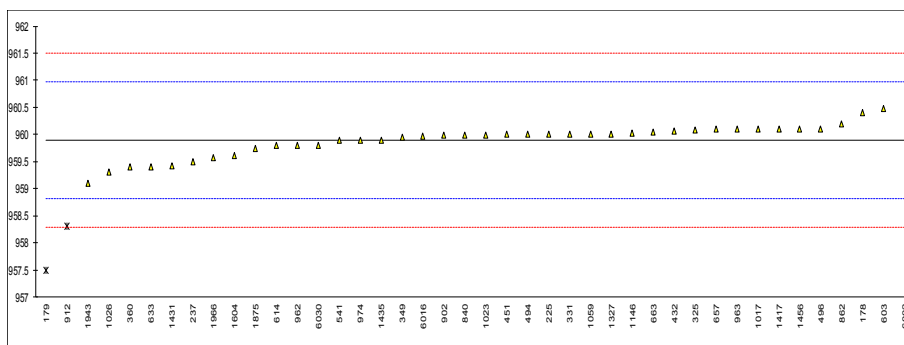
lab	method	value	mark	z(targ)	calc. value*)	remarks
178		----		----		
179	D1500	L3.5		----	3.25	
225		----		----		
237	D1500	3.5		-0.40	3.5	
325	D6045	3.5		-0.40	3.5	
331		----		----		
349	D6045	L4,0		----	3.75	
360	D1500	3.5		-0.40	3.5	
432	D1500	L0,5		----	0.25	R(0.01) possible false negative test result?
451		----		----		
494	D1500	L3,5		----	3.25	
496	D1500	4.0		1.00	4.0	
541	D1500	3.5		-0.40	3.5	
603	D1500	L4.0		----	3.75	
614	D1500	<4.0		----	3.75	
633	D1500	L3.5		----	3.25	
657	D1500	3.5		-0.40	3.5	
663	D1500	L4.0		----	3.75	
840	D1500	L4.0		----	3.75	
862	D1500	L0.5		----	0.25	R(0.01) possible false negative test result?
902	D1500	3.6		-0.12	3.6	
912	D1500	3.5		-0.40	3.5	
962	D1500	3.5		-0.40	3.5	
963	D1500	3.5		-0.40	3.5	
974	D1500	L4.0		----	3.75	
1017		----		----		
1023		----		----		
1026	D1500	L4.0		----	3.75	
1059	D1500	L4,0		----	3.75	
1146		----		----		
1327	D1500	<3.5		----	3.25	
1417	D6045	3.7		0.16	3.7	
1431	D1500	3.5		-0.40	3.5	
1435	D1500	3.5		-0.40	3.5	
1456	D1500	L4.0		----	3.75	
1604	D1500	4.0		1.00	4.0	
1875		----		----		
1943		----		----		
1966	D1500	4.0		1.00	4.0	
6002	D1500	4.1		1.28	4.1	
6016	D1500	3.5		-0.40	3.5	
6030		----		----		
6190		----		----		
normality		suspect			OK	
n		17			30	
outliers		0			2	
mean (n)		3.64			3.62	
st.dev. (n)		0.227			0.230	
R(calc.)		0.63			0.64	
st.dev.(D1500:12)		0.357			0.357	
R(D1500:12)		1			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).



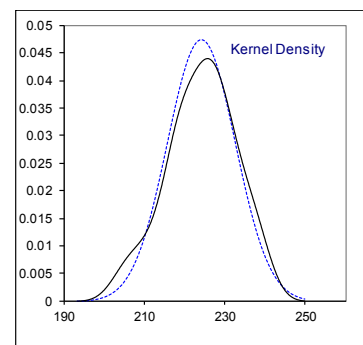
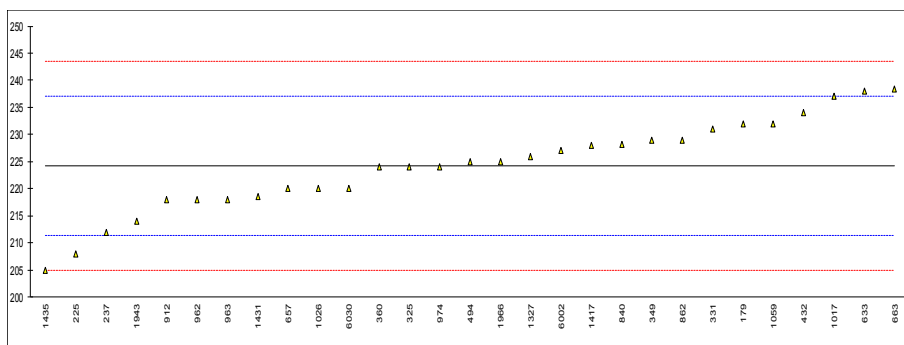
Determination of Density at 15°C on sample #18077; results in kg/m³

lab	method	value	mark	z(targ)	remarks
178	D1298	960.4		0.94	
179	D1298	957.5	C,R(0.01)	-4.47	first reported: 863.4
225	D4052	960.0		0.19	
237	D4052	959.5		-0.74	
325	D4052	960.09		0.36	
331	ISO12185	960.0		0.19	
349	D4052	959.95		0.10	
360	ISO12185	959.4		-0.93	
432	D4052	960.06		0.31	
451	D4052	960		0.19	
494	D4052	960.0		0.19	
496	D4052	960.11		0.40	
541	D4052	959.90		0.01	
603	D4052	960.47		1.07	
614	D4052	959.8		-0.18	
633	D1298	959.41		-0.91	
657	D4052	960.10		0.38	
663	D4052	960.05		0.29	
840	D4052	959.99		0.17	
862	D4052	960.2		0.57	
902	D4052	959.99		0.17	
912	D1298	958.3	R(0.01)	-2.98	
962	D4052	959.8		-0.18	
963	D4052	960.1		0.38	
974	D4052	959.9		0.01	
1017	D4052	960.1		0.38	
1023	D4052	959.99		0.17	
1026	D4052	959.3		-1.11	
1059	ISO12185	960.0		0.19	
1146	D4052	960.03		0.25	
1327	D4052	960.0	C	0.19	reported: 0.9600 kg/m ³
1417	D4052	960.1		0.38	
1431	D4052	959.42		-0.89	
1435	D4052	959.9		0.01	
1456	D4052	960.1		0.38	
1604	D1298	959.61	C	-0.53	reported: 0.9561 kg/m ³
1875	ISO12185	959.75	C	-0.27	first reported: 956.95
1943	ISO3675	959.1		-1.49	
1966	D1298	959.58		-0.59	
6002	ISO3675	985	R(0.01)	46.86	
6016	D4052	959.97		0.14	
6030	GOST3900	959.8		-0.18	
6190		----		----	
normality		OK			
n		39			
outliers		3			
mean (n)		959.90			
st.dev. (n)		0.291			
R(calc.)		0.82			
st.dev.(D1298:12b)		0.536			
R(D1298:12b)		1.5			



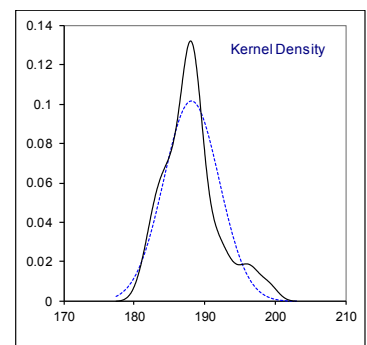
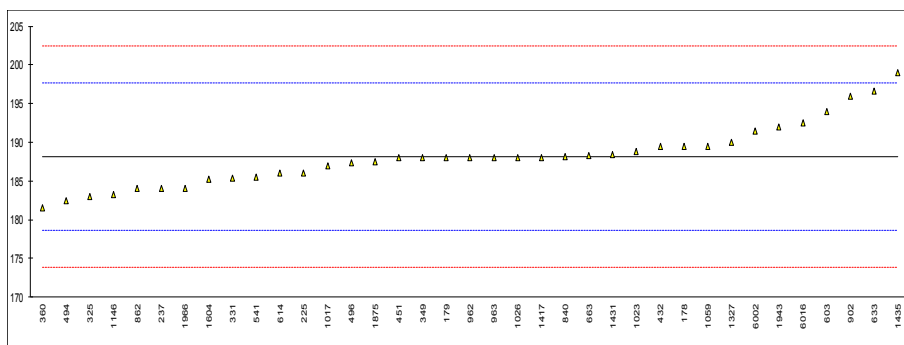
Determination of Flash Point COC on sample #18077; results in °C

lab	method	value	mark	z(targ)	remarks
178		----		----	
179	D92	232		1.21	
225	D92	208.0		-2.53	
237	D92	212		-1.90	
325	D92	224		-0.04	
331	D92	231.0		1.05	
349	D92	229		0.74	
360	D92	224		-0.04	
432	D92	234		1.52	
451		----		----	
494	D92	225		0.12	
496		----		----	
541		----		----	
603		----		----	
614		----		----	
633	D92	238		2.14	
657	D92	220		-0.66	
663	D92	238.3		2.19	
840	D92	228.1		0.60	
862	D92	229		0.74	
902		----		----	
912	D92	218		-0.97	
962	D92	218		-0.97	
963	D92	218		-0.97	
974	D92	224		-0.04	
1017	D92	237		1.99	
1023		----		----	
1026	D92	220		-0.66	
1059	ISO2592	232		1.21	
1146		----		----	
1327	D92	226		0.27	
1417	D92	228		0.59	
1431	D92	218.5		-0.89	
1435	D92	205.0		-2.99	
1456		----		----	
1604		----		----	
1875		----		----	
1943	ISO2592	214		-1.59	
1966	D92	225		0.12	
6002	ISO2592	227		0.43	
6016		----		----	
6030	GOST4333	220		-0.66	
6190		----		----	
normality		OK			
n		29			
outliers		0			
mean (n)		224.24			
st.dev. (n)		8.430			
R(calc.)		23.60			
st.dev.(D92:16b)		6.429			
R(D92:16b)		18			



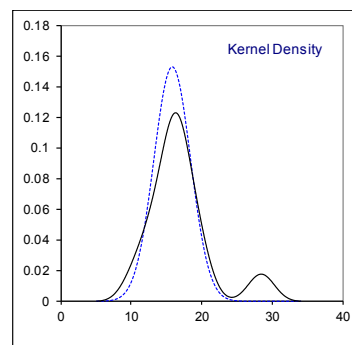
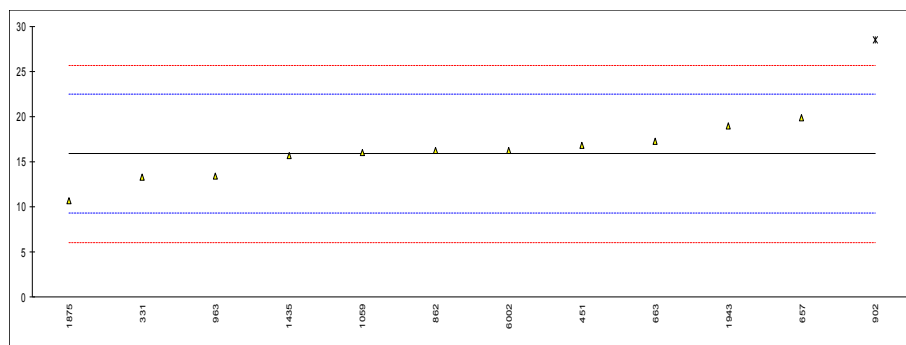
Determination of Flash Point PMcc on sample #18077; results in °C

lab	method	value	mark	z(targ)	remarks
178	D93-A	189.5		0.28	
179	D93-A	188.0		-0.03	
225	D93-B	186.0		-0.45	
237	D93-B	184.0		-0.87	
325	D93-A	183		-1.08	
331	D93-B	185.3		-0.60	
349	D93-A	188		-0.03	
360	D93-B	181.5		-1.39	
432	D93-A	189.5		0.28	
451	D93-A	188		-0.03	
494	D93-B	182.5		-1.18	
496	D93-A	187.3		-0.18	
541	D93-A	185.50		-0.56	
603	D93-A	194		1.23	
614	D93-A	186		-0.45	
633	D93-A	196.58		1.77	
657		----		----	
663	D93-A	188.30		0.03	
840	D93-B	188.1		-0.01	
862	D93-A	184		-0.87	
902	D93-A	196.0		1.64	
912		----		----	
962	D93-A	188		-0.03	
963	D93-A	188		-0.03	
974		----		----	
1017	D93-A	187.0		-0.24	
1023	D93-A	188.8		0.14	
1026	ISO2719-A	188		-0.03	
1059	ISO2719-A	189.5		0.28	
1146	D93-A	183.2		-1.04	
1327	D93-A	190.0		0.39	
1417	D93-A	188		-0.03	
1431	D93-A	188.4		0.05	
1435	D93-A	199.0		2.27	
1456		----		----	
1604	D93	185.2		-0.62	
1875	ISO2719-B	187.5		-0.14	
1943	ISO2719-A	192		0.81	
1966	D93-A	184		-0.87	
6002	D93-A	191.5		0.70	
6016	D93-A	192.5		0.91	
6030		----		----	
6190		----		----	
	normality	OK		Only D93/ISO2719 meth. A	Only D93/ISO2719 meth. B
	n	37		OK	OK
	outliers	0		29	7
	mean (n)	188.15		0	0
	st.dev. (n)	3.921		189.02	184.99
	R(calc.)	10.98		3.856	2.465
	st.dev.(D93-A:16a)	4.771		10.80	6.90
	R(D93-A:16a)	13.36	Comp. R(D93-B:16a) = 10	4.793	3.571
				13.42	10



Determination of Insoluble Color Bodies, membrane patch colorimetry on sample #18077;

lab	method	value	mark	z(targ)	remarks
178		----		----	
179		----		----	
225		----		----	
237		----		----	
325		----		----	
331	D7843	13.3		-0.78	
349		----		----	
360		----		----	
432		----		----	
451	D7843	16.8		0.29	
494		----		----	
496		----		----	
541		----		----	
603		----		----	
614		----		----	
633		----		----	
657	D7843	19.83		1.21	
663	D7843	17.3		0.44	
840		----		----	
862	D7843	16.2		0.10	
902	D7843	28.5	G(0.05)	3.86	
912		----		----	
962		----		----	
963	D7843	13.4		-0.75	
974		----		----	
1017		----		----	
1023		----		----	
1026		----		----	
1059	D7843	16		0.04	
1146		----		----	
1327		----		----	
1417		----		----	
1431		----		----	
1435	D7843	15.7		-0.05	
1456		----		----	
1604		----		----	
1875	D7843	10.7		-1.57	
1943	D7843	18.958		0.95	
1966		----		----	
6002	D7843	16.24		0.12	
6016		----		----	
6030		----		----	
6190		----		----	
normality		OK			
n		11			
outliers		1			
mean (n)		15.86			
st.dev. (n)		2.605			
R(calc.)		7.29			
st.dev.(D7843:16)		3.278			
R(D7843:16)		9.18			

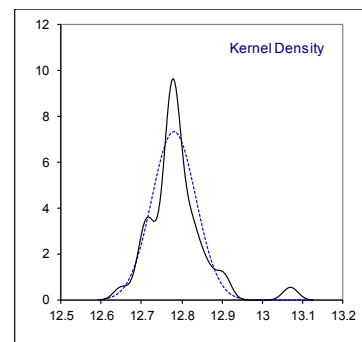
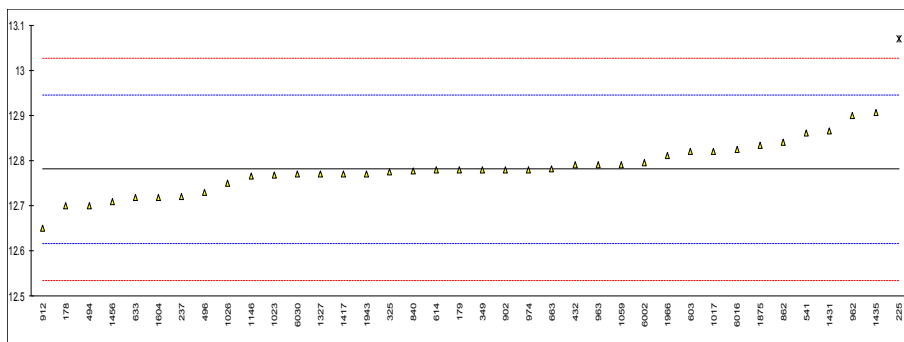


Determination of Kinematic Viscosity at 40°C on sample #18077; results in mm²/s

lab	method	value	mark	z(targ)	remarks
178	D445	12.7		-0.98	
179	D445	12.78		-0.01	
225	D445	13.07	R(0.01)	3.52	
237	D445	12.72		-0.74	
325	D445	12.774		-0.08	
331		----		----	
349	D445	12.78	C	-0.01	first reported:12.58
360		----		----	
432	D445	12.79		0.11	
451		----		----	
494	D445	12.701		-0.97	
496	D445	12.729		-0.63	
541	D7042	12.860		0.97	
603	D7042	12.820		0.48	
614	D445	12.78		-0.01	
633	D7279 corr. to D445	12.717		-0.77	
657		----		----	
663	D445	12.782		0.02	
840	D7042	12.776		-0.06	
862	D445	12.84		0.72	
902	D445	12.78		-0.01	
912	D445	12.65		-1.59	
962	D445	12.90		1.45	
963	D445	12.79		0.11	
974	D445	12.78		-0.01	
1017	D445	12.82		0.48	
1023	D445	12.769		-0.14	
1026	D445	12.75		-0.37	
1059	ISO3104	12.79		0.11	
1146	D445	12.766		-0.18	
1327	D445	12.77		-0.13	
1417	D445	12.77		-0.13	
1431	D7042	12.866		1.04	
1435	D7042	12.906		1.53	
1456	D7042	12.71		-0.86	
1604	D445	12.717		-0.77	
1875	D7042	12.833		0.64	
1943	ISO3104	12.77		-0.13	
1966	D445	12.81		0.36	
6002	ISO3104	12.794		0.16	
6016	D7042	12.825		0.54	
6030	GOST33	12.7697		-0.13	
6190		----		----	

normality OK
n 37
outliers 1
mean (n) 12.781
st.dev. (n) 0.0546
R(calc.) 0.153
st.dev.(iis, lit. 16) 0.0822
R(iis, lit. 16) 0.230

Compare R(D445:17) = 0.048, range 25 to125 mm²/s

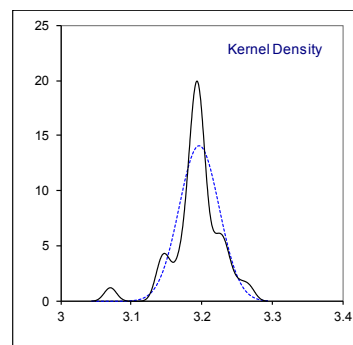
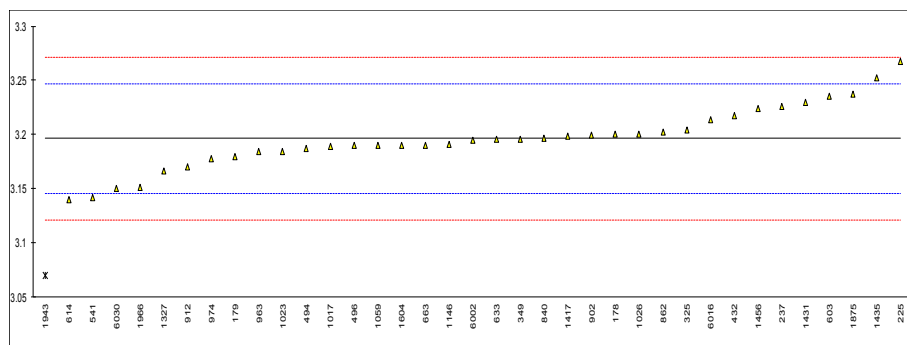


Determination of Kinematic Viscosity at 100°C on sample #18077; results in mm²/s

lab	method	value	mark	z(targ)	remarks
178	D445	3.20		0.15	
179	D445	3.180		-0.64	
225	D445	3.267		2.82	
237	D445	3.226		1.19	
325	D445	3.204		0.31	
331		----		----	
349	D445	3.196		0.00	
360		----		----	
432	D445	3.217		0.83	
451		----		----	
494	D445	3.1876		-0.34	
496	D445	3.1896		-0.26	
541	D7042	3.1417		-2.17	
603	D7042	3.2357		1.58	
614	D445	3.14		-2.23	
633	D445	3.1958		-0.01	
657		----		----	
663	D445	3.1903		-0.23	
840	D7042	3.1963		0.01	
862	D445	3.202		0.23	
902	D445	3.199		0.11	
912	D445	3.170		-1.04	
962		----		----	
963	D445	3.184		-0.48	
974	D445	3.178		-0.72	
1017	D445	3.189		-0.28	
1023	D445	3.184		-0.48	
1026	D445	3.20		0.15	
1059	ISO3104	3.190		-0.24	
1146	D445	3.1907		-0.22	
1327	D445	3.166		-1.20	
1417	D445	3.198		0.07	
1431	D7042	3.2292		1.32	
1435	D7042	3.2527		2.25	
1456	D7042	3.224		1.11	
1604	D445	3.190		-0.24	
1875	D7042	3.237		1.63	
1943	ISO3104	3.07	C,R(0.01)	-5.02	first reported: 3.11
1966	D445	3.151		-1.80	
6002	ISO3104	3.195		-0.04	
6016	D7042	3.2139		0.71	
6030	GOST33	3.150		-1.84	
6190		----		----	

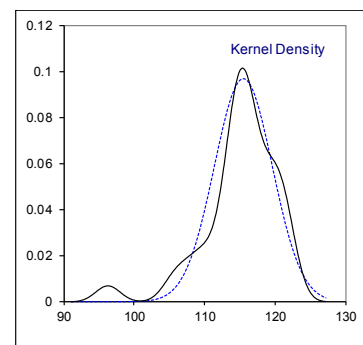
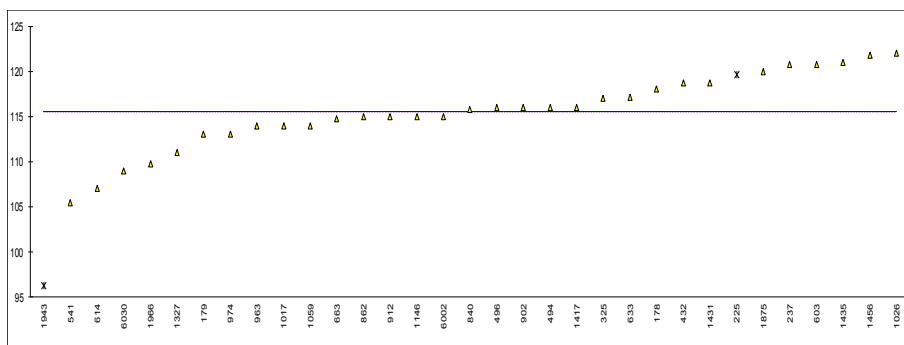
normality OK
n 36
outliers 1
mean (n) 3.196
st.dev. (n) 0.0284
R(calc.) 0.079
st.dev.(iis, lit. 16) 0.0251
R(iis, lit. 16) 0.070

Compare R(D445:17) = 0.018, range 6 to 16 mm²/s



Determination of Viscosity Index on sample #18077;

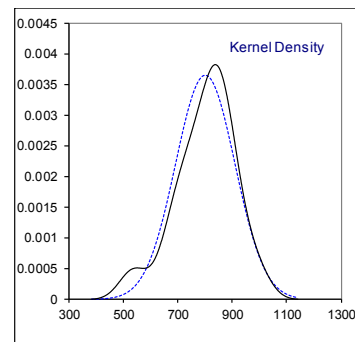
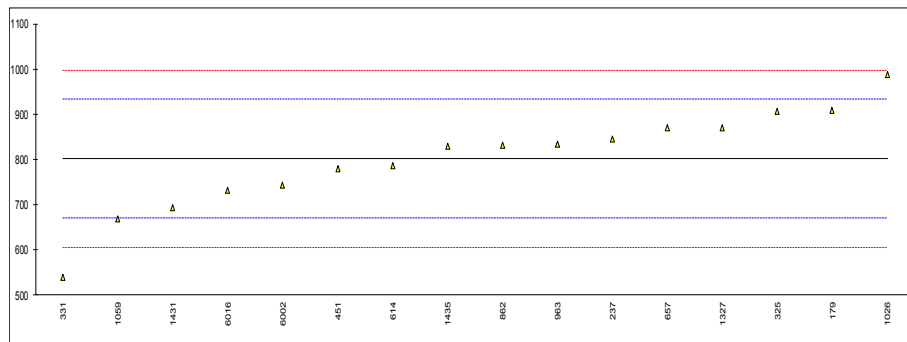
lab	method	value	mark	z(targ)	iis calc.	remarks
178	D2270	118		----	118.21	
179	D2270	113		----	113.19	
225	D2270	119.6	ex	----	119.54	ex
237	D2270	120.7	E	----	121.79	excluded, outlier in kin. visc. 40°C
325	D2270	117.0		----	117.02	calculation error
331		----		----	----	
349		----		----	115.64	
360		----		----	----	
432	D2270	118.7		----	118.65	
451		----		----	----	
494	D2270	116		----	116.27	
496	D2270	116		----	115.89	
541	D2270	105.5		----	105.47	
603	D2270	120.8		----	120.81	
614	D2270	107		----	107.05	
633	D2270	117.1		----	117.14	
657		----		----	----	
663	D2270	114.7		----	114.72	
840	D2270	115.8		----	115.79	
862	D2270	115		----	115.13	
902	D2270	116		----	116.10	
912	D2270	115		----	114.78	
962		----		----	----	
963	D2270	114		----	113.57	
974	D2270	113		----	112.88	
1017	D2270	114		----	113.62	
1023		----		----	114.07	
1026	D2270	122	E	----	116.99	calculation error
1059	ISO2909	114		----	114.48	
1146	D2270	115		----	115.17	
1327	D2270	111		----	111.28	
1417	D2270	116		----	116.19	
1431	D2270	118.7		----	118.69	
1435	D2270	121		----	121.32	
1456	D2270	121.72		----	121.72	
1604		----		----	116.25	
1875	ISO2909	120	E	----	120.70	calculation error
1943	ISO2909	96.3	C,E,R(0.01)	----	95.66	R(0.01) first reported: 101.53, calculation error
1966	D2270	109.71	E	----	108.04	calculation error
6002	ISO2909	115		----	115.15	
6016		----		----	117.32	
6030	GOST25371	109		----	108.82	
6190		----		----	----	
	normality	OK			OK	
	n	31			35	
	outliers	1 (+1ex)			1 (+1ex)	
	mean (n)	115.50			115.43	
	st.dev. (n)	4.116			3.917	
	R(calc.)	11.53			10.97	
	st.dev.(D2270:10)	(0.714)			(0.714)	
	R(D2270:10)	(2)			(2)	



Determination of Oxidation Stability RPVOT on sample #18077; results in minutes

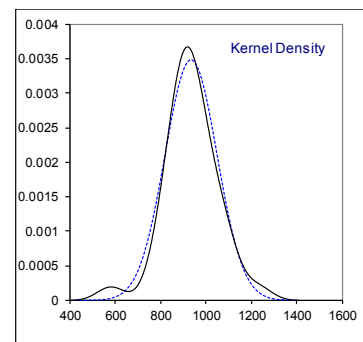
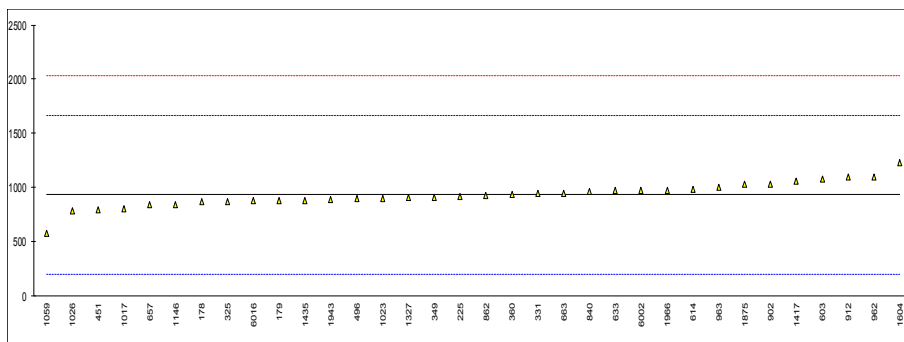
lab	method	value	mark	z(targ)	remarks
178		----		----	
179	D2272	909		1.64	
225		----		----	
237	D2272	845		0.66	
325	D2272	907		1.61	
331	D2272	538		-4.03	
349		----		----	
360		----		----	
432		----		----	
451	D2272	780		-0.33	
494		----		----	
496		----		----	
541		----		----	
603		----		----	
614	D2272	786		-0.24	
633		----		----	
657	D2272	869		1.03	
663		----		----	
840		----		----	
862	D2272	832		0.47	
902		----		----	
912		----		----	
962		----		----	
963	D2272	833		0.48	
974		----		----	
1017		----		----	
1023		----		----	
1026	D2272	987		2.83	
1059	D2272	669		-2.02	
1146		----		----	
1327	D2272	869		1.03	
1417		----		----	
1431	D2272	694		-1.64	
1435	D2272	830		0.44	
1456		----		----	
1604		----		----	
1875		----		----	
1943		----		----	
1966		----		----	
6002	D2272	744		-0.88	
6016	D2272	731.75		-1.07	
6030		----		----	
6190		----		----	

normality suspect
n 16
outliers 0
mean (n) 801.5
st.dev. (n) 109.01
R(calc.) 305.2
st.dev.(D2272:14a) 65.44
R(D2272:14a) 183.2



Determination of Water by KF on sample #18077; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D6304-C	867		-0.18	
179	D6304-C	880		-0.15	
225	D6304-A	914.44		-0.05	
237		----		----	
325	D6304-C	874		-0.16	
331	In house	948.0		0.04	
349	D6304-A	913		-0.05	
360	D6304-A	938.8		0.02	
432		----		----	
451	D6304-C	798		-0.37	
494		----		----	
496	D6304-A	900		-0.09	
541		----		----	
603	D6304-C	1077.7		0.40	
614	D4928	988		0.15	
633	D6304-C	970		0.10	
657	D6304-C	840		-0.25	
663	D6304-C	949.45		0.04	
840	D6304-C	963.4		0.08	
862	D6304-C	929		-0.01	
902	D6304-A	1033		0.27	
912	D6304-C	1100		0.46	
962	D6304-A	1101		0.46	
963	D6304-A	1001		0.19	
974		----		----	
1017	D6304-C	803.25		-0.36	
1023	D6304-A	900.6		-0.09	
1026	D6304-A	788		-0.40	
1059	D6304Mod.	580		-0.97	
1146	D6304-C	845		-0.24	
1327	D6304-C	912		-0.06	
1417	D6304-A	1060		0.35	
1431		----		----	
1435	D6304-A	883.1		-0.14	
1456		----		----	
1604	D6304	1228.2		0.81	
1875	ISO12937	1030		0.27	
1943	EN60814	887.9		-0.12	
1966	EN60814	972.87		0.11	
6002	ISO12937	970.23		0.10	
6016	D6304-A	877		-0.15	
6030		----		----	
6190		----		----	
	normality	not OK			
	n	34			
	outliers	0			
	mean (n)	933.06			
	st.dev. (n)	114.404			
	R(calc.)	320.33			
	st.dev.(D6304:16e1)	365.140			
	R(D6304:16e1)	1022.39			

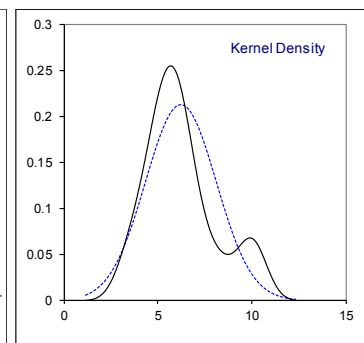
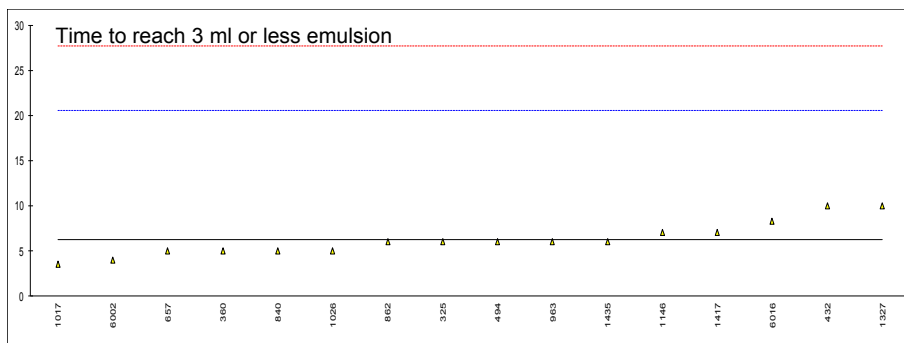


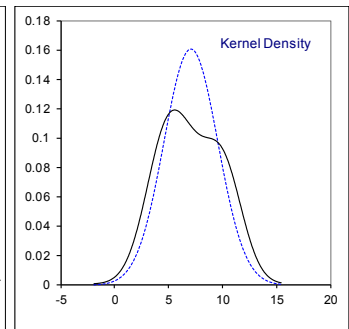
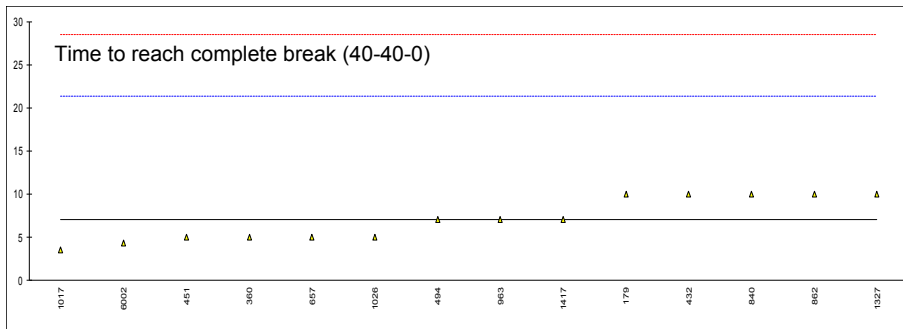
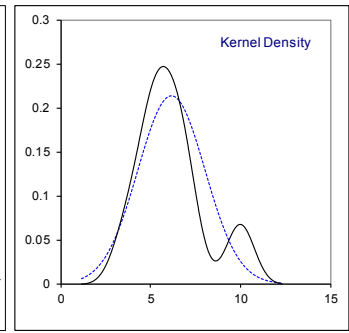
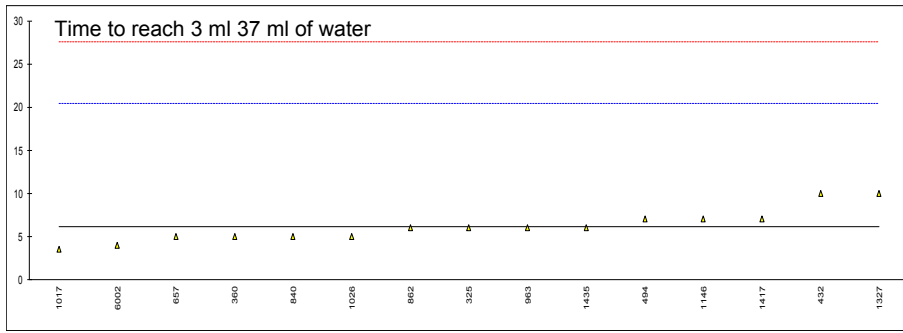
Determination of Water Separability at 54°C, distilled water on sample #18077; results in minutes

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	
		emulsion	mark	z(targ)	water	mark	z(targ)	(40-40-0)	mark	z(targ)			
178		----		----	----		----	----		----		----	
179	D1401	----		----	----		----	10		0.41		NO	----
225		----		----	----		----	----		----		----	----
237		----		----	----		----	----		----		YES	31.00
325	D1401	6		-0.03	6		-0.02	----		----		YES	30
331		----		----	----		----	----		----		----	----
349		----		----	----		----	----		----		----	----
360	D1401	5		-0.17	5		-0.16	5		-0.29		NO	----
432	D1401	10		0.53	10		0.54	10		0.41		NO	----
451	D1401	----		----	----		----	5		-0.29		----	----
494	D1401	6		-0.03	7		0.12	7		-0.01		NO	----
496		----		----	----		----	----		----		----	----
541		----		----	----		----	----		----		----	----
603		----		----	----		----	----		----		----	----
614		----		----	----		----	----		----		----	----
633		----		----	----		----	----		----		----	----
657	D1401	5		-0.17	5		-0.16	5		-0.29		----	----
663		----		----	----		----	----		----		----	----
840	D1401	5		-0.17	5		-0.16	10		0.41		NO	----
862	D1401	6		-0.03	6		-0.02	10		0.41		NO	10
902		----		----	----		----	----		----		----	----
912		----		----	----		----	----		----		----	----
962		----		----	----		----	----		----		----	----
963	D1401	6		-0.03	6		-0.02	7		-0.01		NO	----
974		----		----	----		----	----		----		----	----
1017	D1401	3.5		-0.38	3.5		-0.37	3.5		-0.50		NO	----
1023		----		----	----		----	----		----		----	----
1026	D1401	5		-0.17	5		-0.16	5		-0.29		NO	----
1059		----		----	----		----	----		----		----	----
1146	D1401	7		0.11	7		0.12	----		----		----	----
1327	D1401	10		0.53	10		0.54	10		0.41		NO	----
1417	D1401	7		0.11	7		0.12	7		-0.01		NO	----
1431		----		----	----		----	----		----		----	----
1435	ISO6614	6.01		-0.03	6.01		-0.02	----		----		----	----
1456		----		----	----		----	----		----		----	----
1604		----		----	----		----	----		----		----	----
1875		----		----	----		----	----		----		----	----
1943		----		----	----		----	----		----		----	----
1966		----		----	----		----	----		----		----	----
6002	ISO6614	4.0		-0.31	4.0		-0.30	4.3		-0.39		NO	----
6016	D1401	8.32		0.29	----		----	----		----		NO	----
6030		----		----	----		----	----		----		----	----
6190		----		----	----		----	----		----		----	----

normality	OK	OK	OK
n	16	15	14
outliers	0	0	0
mean (n)	6.24	6.17	7.06
st.dev. (n)	1.875	1.868	2.488
R(calc.)	5.25	5.23	6.97
st.dev.(D1401)	7.143	7.143	7.143
R(D1401)*	20	20	20

*) R(D1401:18a)





Determination of Water Separability at 54°C, distilled water on sample #18077; results in ml (cont.)

lab	method	volume oil phase	volume water phase	volume emulsion phase	test OK/NOK	remark
178		----	----	----		
179	D1401	40	40	0	OK	reported 10 min. to break
225		----	----	----		
237		39	35	6	OK	reported abort time of 31 min.
325	D1401	41	39	0	OK	reported abort time of 30 min.
331		----	----	----		
349		----	----	----		
360	D1401	43	37	0	OK	reported 5 min. to break
432	D1401	40	40	0	OK	reported 10 min. to break
451	D1401	40	40	0	OK	reported 5 min. to break
494	D1401	40	40	0	OK	reported 7 min. to break
496		----	----	----		
541		----	----	----		
603		----	----	----		
614		----	----	----		
633		----	----	----		
657	D1401	----	----	----	OK	reported 5 min. to break
663		----	----	----		
840	D1401	----	----	----	OK	reported 10 min. to break
862	D1401	40	40	0	OK	reported 10 min. to break
902		----	----	----		
912		----	----	----		
962		----	----	----		
963	D1401	40	40	0	OK	reported 7 min. to break
974		----	----	----		
1017	D1401	40	40	0	OK	reported 3.5 min. to break
1023		----	----	----		
1026	D1401	40	40	0	OK	reported 5 min. to break
1059		----	----	----		
1146	D1401	40	37	3	NOK*	did not report an abort time
1327	D1401	----	----	----	OK	reported 10 min. to break
1417	D1401	40	40	0	OK	reported 7 min. to break
1431		----	----	----		
1435	ISO6614	----	----	----	NOK*	did not report a complete break or abort time
1456		----	----	----		
1604		----	----	----		
1875		----	----	----		
1943		----	----	----		
1966		----	----	----		
6002	ISO6614	----	----	----	OK	reported 4.3 min. to break
6016	D1401	----	----	----	NOK*	did not report a complete break or abort time
6030		----	----	----		
6190		----	----	----		

* according to ASTM D1401 the amount of ml oil, ml water and ml emulsion should be reported as well as either the time the different volume requirements are met or when these are not reached, the time when the test is aborted (usually 30 minutes for tests at 54°C)

Determination of Demulsibility Characteristics, Steam on sample #18077; results in s en mL

lab	method	time to reach 20ml of oil within 20 min	mark	z(targ)	volume oil after 20 min	mark	z(targ)	remarks
178		----		----			----	
179		----		----			----	
225		----		----			----	
237		----		----			----	
325		----		----			----	
331		----		----			----	
349		----		----			----	
360		----		----			----	
432		----		----			----	
451	IP19	120		----			----	
494		----		----			----	
496		----		----			----	
541		----		----			----	
603		----		----			----	
614		----		----			----	
633		----		----			----	
657		----		----			----	
663		----		----			----	
840		----		----			----	
862		----		----			----	
902		----		----			----	
912		----		----			----	
962		----		----			----	
963		----		----			----	
974		----		----			----	
1017		----		----			----	
1023		----		----			----	
1026		----		----			----	
1059		----		----			----	
1146		----		----			----	
1327		----		----			----	
1417	IP19	120		----	20		----	
1431		----		----			----	
1435		----		----			----	
1456		----		----			----	
1604		----		----			----	
1875		----		----			----	
1943		----		----			----	
1966		----		----			----	
6002	PN-C04110	76		----	20		----	
6016		----		----			----	
6030		----		----			----	
6190		----		----			----	
n		3			2			
mean (n)		76-120			20			

Determination of Water separation ability, Steam, time to reach the 55 mm measuring point (oil-water phase) on sample #18077; results in s

lab	method	value	mark	z(targ)	remarks
178		----		----	
179		----		----	
225		----		----	
237		----		----	
325		----		----	
331		----		----	
349		----		----	
360		----		----	
432		----		----	
451		----		----	
494		----		----	
496	DIN51589	79		----	
541		----		----	
603		----		----	
614		----		----	
633		----		----	
657		----		----	
663		----		----	
840		----		----	
862		----		----	
902		----		----	
912		----		----	
962		----		----	
963		----		----	
974		----		----	
1017		----		----	
1023		----		----	
1026		----		----	
1059		----		----	
1146		----		----	
1327		----		----	
1417		----		----	
1431		----		----	
1435		----		----	
1456		----		----	
1604		----		----	
1875		----		----	
1943		----		----	
1966		----		----	
6002		----		----	
6016		----		----	
6030		----		----	
6190		----		----	

Determination of Level of Contamination on sample #18077; results in counts per ml

lab	method	≥ 4 µm(c)	mark	z(targ)	≥ 6 µm(c)	mark	z(targ)	≥ 14 µm(c)	mark	z(targ)
178	INH-1184	15301		-0.60	2355		-1.78	13	ex	-1.92
179	D7647	21824	C	0.19	3831	C	-0.59	123	ex, C	-0.63
225		----		----	----		----	----		----
237		----		----	----		----	----		----
325		----		----	----		----	----		----
331		----		----	----		----	----		----
349		----		----	----		----	----		----
360	ISO4406	25578		0.65	4813		0.20	169		-0.09
432		----		----	----		----	----		----
451	ISO11500	23905		0.45	5247		0.55	55		-1.43
494		----		----	----		----	----		----
496	D7647	25610		0.66	5891		1.07	238		0.72
541		----		----	----		----	----		----
603		----		----	----		----	----		----
614	ISO4407	24332		0.50	6724		1.75	23		-1.80
633	D7647	32847		1.54	5121		0.45	110		-0.78
657	ISO4406	3953		-1.99	1826		-2.21	220		0.51
663	D7647	4787		-1.89	1856		-2.18	177		0.01
840		23455.1		0.39	6254.4		1.37	256.3		0.94
862	ISO11500	23253		0.37	4110		-0.36	122		-0.64
902	D7647	13668		-0.80	3339		-0.99	76.9		-1.17
912		----		----	----		----	----		----
962		----		----	----		----	----		----
963	D7647	5075		-1.86	1456		-2.51	37		-1.64
974		----		----	----		----	----		----
1017	ISO11500	34879.99		1.79	9308.45		3.84	350.49		2.04
1023		----		----	----		----	----		----
1026		----		----	----		----	----		----
1059	D7647	1922	ex	-2.24	3596		-0.78	84		-1.09
1146	ISO11500	2580	ex	-2.16	571	ex	-3.22	20	ex	-1.84
1327	D7647	23230		0.37	4508		-0.04	237		0.71
1417		----		----	----		----	----		----
1431	ISO4407	26342.28		0.75	6043.32		1.20	245.60		0.81
1435		31557.10		1.39	8062.60		2.83	320.83		1.70
1456	ISO4406	36259		1.96	10883.5		5.11	439.4		3.09
1604	D7647	16623		-0.44	3552		-0.81	113		-0.75
1875		----		----	----		----	----		----
1943		29766.54	ex	1.17	3075.14		-1.20	194.77	ex	0.21
1966		----		----	----		----	----		----
6002	D7596	1975.8		-2.24	802.2		-3.04	120.1		-0.66
6016	D7596	12586		-0.94	2973		-1.28	140		-0.43
6030		----		----	----		----	----		----
6190	ISO11500	18138.06		-0.26	3828.06		-0.59	171.70		-0.06
	normality	OK			OK			OK		
	n	22			24			21		
	outliers	0 (+3ex)			0(+1ex)			0(+4ex)		
	mean (n)	20235.4			4560.65			176.49		
	st.dev. (n)	10058.99			2467.310			107.930		
	R(calc.)	28165.2			6908.47			302.20		
	st.dev.(D7647:10)	8166.44			1237.891			85.094		
	R(D7647:10)	22866.0			3466.10			238.26		

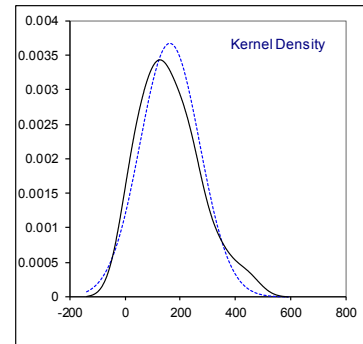
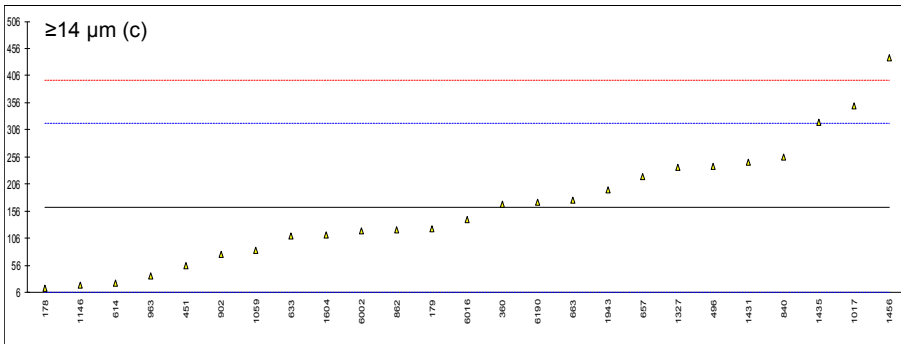
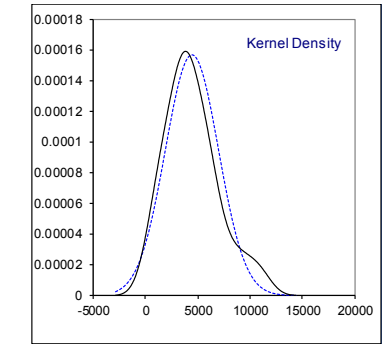
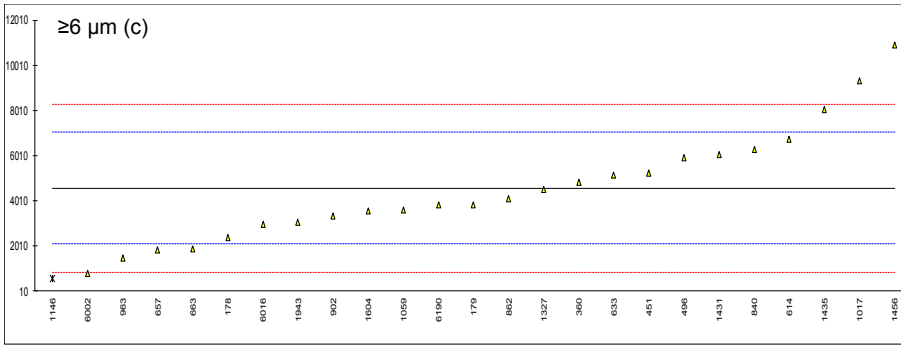
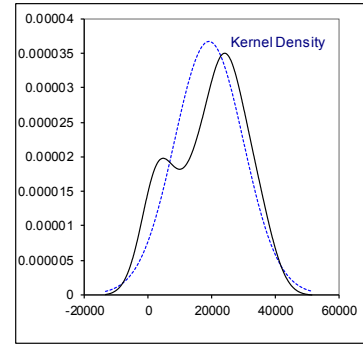
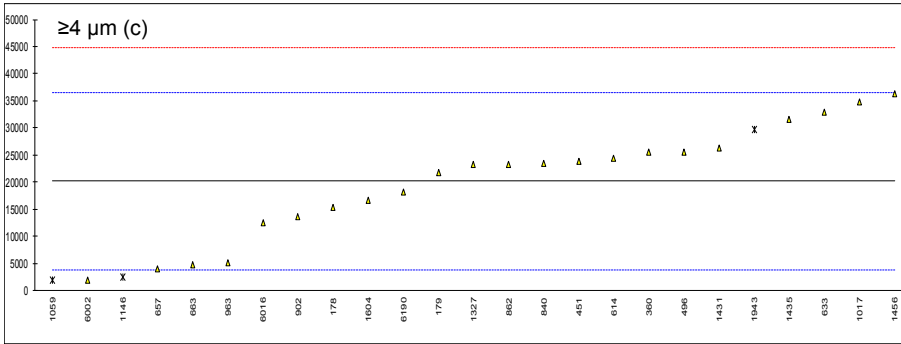
Lab 178 the test result for ≥14 µm was excluded for the test result in counts/ml did not match the ISO scale number

Lab 179 first reported for ≥4 µm: 2409460, for for ≥6 µm: 343604 and for for ≥14 µm: 17417, the test result for ≥14 µm was excluded for the test result in counts/ml did not match the ISO scale number

Lab 1059 the test result for ≥4 µm was excluded for the test result in counts/ml did not match the ISO scale number

Lab 1146 all test results were excluded for the test result in counts/ml did not match the ISO scale numbers

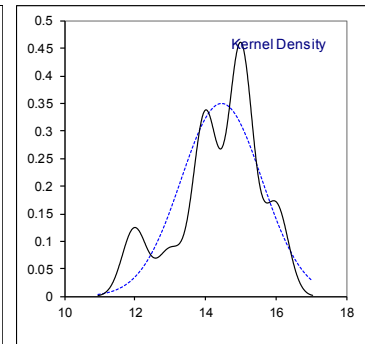
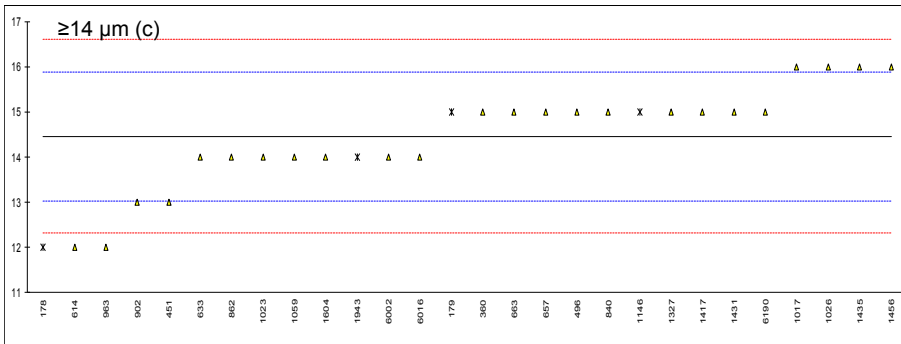
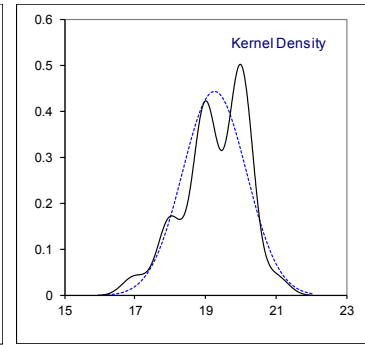
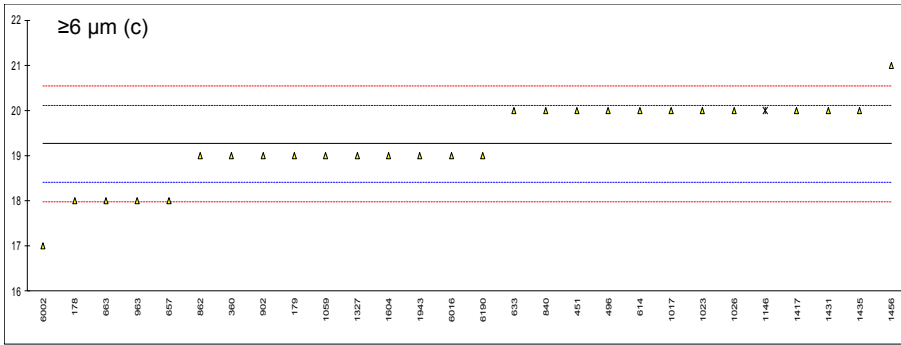
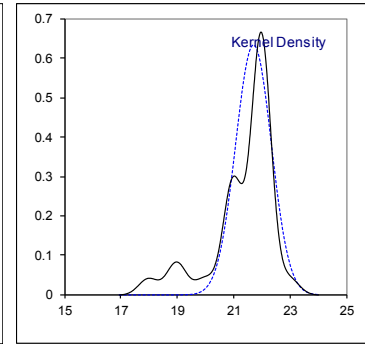
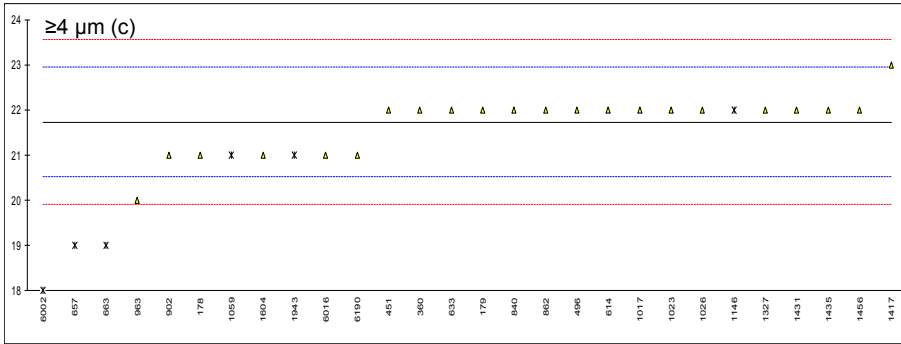
Lab 1943 the test results for ≥4 µm and ≥14 µm were excluded for the test result in counts/ml did not match the ISO scale numbers



Determination of Level of Contamination on sample #18077; 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	ISO4406	21		-1.20	18		-2.94	12	ex	-3.44
179	ISO4406	22		0.45	19		-0.60	15	ex	0.76
225		----		----	----		----	----		----
237		----		----	----		----	----		----
325		----		----	----		----	----		----
331		----		----	----		----	----		----
349		----		----	----		----	----		----
360	ISO4406	22		0.45	19		-0.60	15		0.76
432		----		----	----		----	----		----
451	ISO4406	22		0.45	20		1.73	13		-2.04
494		----		----	----		----	----		----
496	ISO4406	22		0.45	20		1.73	15		0.76
541		----		----	----		----	----		----
603		----		----	----		----	----		----
614	ISO4406	22		0.45	20		1.73	12		-3.44
633	ISO4406	22		0.45	20		1.73	14		-0.64
657	ISO4406	19	C, R(0.01)	-4.49	18	C	-2.94	15	C	0.76
663	ISO4406	19	R(0.01)	-4.49	18		-2.94	15		0.76
840		22		0.45	20		1.73	15		0.76
862	ISO11500	22		0.45	19		-0.60	14		-0.64
902	ISO4406	21		-1.20	19		-0.60	13		-2.04
912		----		----	----		----	----		----
962		----		----	----		----	----		----
963	ISO4406	20		-2.84	18		-2.94	12		-3.44
974		----		----	----		----	----		----
1017	ISO11500	22		0.45	20		1.73	16		2.16
1023	ISO4406	22		0.45	20		1.73	14		-0.64
1026	ISO4406	22	C	0.45	20		1.73	16	C	2.16
1059	ISO4406	21	ex	-1.20	19		-0.60	14		-0.64
1146	ISO4406	22	ex	0.45	20	ex	1.73	15	ex	0.76
1327	ISO4406	22		0.45	19		-0.60	15		0.76
1417	ISO4406	23		2.10	20		1.73	15		0.76
1431	ISO4406	22		0.45	20		1.73	15		0.76
1435	ISO4406	22		0.45	20		1.73	16		2.16
1456	ISO4406	22		0.45	21		4.06	16		2.16
1604	D7647	21		-1.20	19		-0.60	14		-0.64
1875		----		----	----		----	----		----
1943	ISO4406	21	ex	-1.20	19		-0.60	14	ex	-0.64
1966		----		----	----		----	----		----
6002	ISO4406	18	R(0.01)	-6.14	17		-5.27	14		-0.64
6016	ISO4406	21		-1.20	19		-0.60	14		-0.64
6030		----		----	----		----	----		----
6190	ISO4406	21		-1.20	19		-0.60	15		0.76
	normality	suspect			OK			OK		
	n	22			27			24		
	outliers	3 (+3ex)			0 (+1ex)			0 (+4ex)		
	mean (n)	21.7			19.3			14.5		
	st.dev. (n)	0.63			0.90			1.14		
	R(calc.)	1.8			2.5			3.2		
	st.dev.(D7647:10)	0.61			0.43			0.71		
	R(D7647:10)	1.7			1.2			2		

Lab 178 the test result for ≥14 µm was excluded for the test result in counts/ml did not match the ISO scale number
 Lab 179 the test result for ≥14 µm was excluded for the test result in counts/ml did not match the ISO scale number
 Lab 657 first reported for ≥4 µm: 3953, for for ≥6 µm: 1826 and for for ≥14 µm: 220
 Lab 1026 first reported for ≥4 µm: 21, for for ≥6 µm: 18 and for for ≥14 µm: 9
 Lab 1059 the test result for ≥4 µm was excluded for the test result in counts/ml did not match the ISO scale number
 Lab 1146 all test results were excluded for the test result in counts/ml did not match the ISO scale numbers
 Lab 1943 the test results for ≥4 µm and ≥14 µm were excluded for the test result in counts/ml did not match the ISO 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
2 labs in CHINA, People's Republic
1 lab in COTE D'IVOIRE
1 lab in FRANCE
2 labs in GEORGIA
3 labs in GERMANY
1 lab in GREECE
1 lab in INDIA
1 lab in KAZAKHSTAN
1 lab in LATVIA
1 lab in MALAYSIA
2 labs in NETHERLANDS
1 lab in NIGERIA
1 lab in NORWAY
1 lab in PHILIPPINES
1 lab in POLAND
1 lab in ROMANIA
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
U	= reported test result in a different unit
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, March 2017
- 2 ASTM E178:89
- 3 ASTM E1301:89
- 4 ISO 5725:86
- 5 ISO 5725, 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