

Results of Proficiency Test  
Turbine Oil  
May 2014

Organised by: Institute for Interlaboratory Studies  
Spijkenisse, the Netherlands

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## 1 INTRODUCTION

Since 2013, the Institute for Interlaboratory Studies organises every year a proficiency test for Turbine Oil. In the annual proficiency testing program 2013/2014, it was decided to continue the proficiency test for the analyses of Turbine Oil. In this interlaboratory study 30 laboratories in 20 different countries have participated. See appendix 2 for the number of participants per country. In this report, the results of the 2014 Turbine Oil proficiency test are presented and discussed. This report is also electronically available through the iis internet site [www.iisnl.com](http://www.iisnl.com).

## 2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, The Netherlands, was the organizer of this proficiency test. It was decided to send one bottle of 1L (labelled #14056) of used Turbine Oil that was provided by a third party. The analyses for fit-for-use and homogeneity were subcontracted. Participants were requested to report rounded and unrounded results. The unrounded 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, since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). 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 Organization, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3), which can be downloaded from [www.iisnl.com](http://www.iisnl.com).

### 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 was provided by a third party. The 60 litre bulk material was transferred after homogenizing into 60 brown glass bottles of 1 litre (labelled #14056). The homogeneity of the subsamples #14056 was checked by determination of Density @15°C in accordance with ASTM D4052:11 on 8 stratified randomly selected samples.

	Density @ 15 °C in kg/m <sup>3</sup>
Sample #14056-1	994.86
Sample #14056-2	994.89
Sample #14056-3	994.90
Sample #14056-4	994.90
Sample #14056-5	994.88
Sample #14056-6	994.89
Sample #14056-7	994.89
Sample #14056-8	994.89

Table 1: homogeneity test results of subsamples #14056

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

	Density @ 15 °C in kg/m <sup>3</sup>
r (sample #14056)	0.04
reference test	ASTM 1298:12b
0.3 x R(reference test)	0.45

Table 2: evaluation of the repeatability of the subsamples #14056

The calculated repeatability is less than 0.3 times the corresponding reproducibility of the reference method. Therefore, homogeneity of the subsamples #14056 was assumed.

To each of the participating laboratories, one sample of 1 L in a brown glass bottle (labelled #14056) was sent on April 16, 2014.

## 2.5 ANALYSES

The participants were requested to determine on sample #14056: Total Acidity, Base Number, Acid Number, Color ASTM, Density @ 15°C, Flash Point PMcc, Kinematic Viscosity @ 40°C and @ 100°C, Viscosity Index, Level of contamination, Water by KF, Water separability and Foaming Characteristics (at the end of the 5 min blowing time and at the end of the 10 min settling time).

To get comparable results a detailed report form, on which the units were prescribed as well as some of the required standards and a letter of instructions were prepared and made available for download on the iis website ([www.iisnl.com](http://www.iisnl.com)).

A SDS and a form to confirm receipt of the samples were added to the sample package.

### 3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered. The original data are tabulated per determination in Appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder fax was sent to those laboratories that had not reported results at that moment.

Shortly after the deadline, the available results were screened for suspect data. A 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 results. Additional or corrected results are used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

#### 3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' (iis-protocol, version 3.3) of April 2014.

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. 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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.

According to ISO 5725 the original results per determination were submitted to Dixon's and/or 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 (ref. 15). 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. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

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 analysis results are plotted. The corresponding laboratory numbers are under 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 standard. Outliers and other data, which were excluded from the calculations, are represented as a "x". Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. The Kernel Density is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3; nos.12 and 13). 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 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:

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

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.

Therefore, the usual interpretation of z-scores is as follows:

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

## 4 EVALUATION

In this interlaboratory study, some problems were encountered with the dispatch of the samples to laboratories in Austria, Nigeria and Togo. Eleven participants reported after the final reporting date and one participant did not report any test results at all. Not all laboratories were able to report all analyses requested. In total 29 participants reported 301 test results. Observed were 10 outlying results, which is 3.3% of the numerical 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 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. D2086) and an added designation for the year that the method was adopted or revised (e.g. D2086:08). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D2086:08(2013)). In the results tables of Appendix 1 only the method number and year of adoption or revision e.g. D2086:08 will be used.

Total Acidity: This determination may be problematic, depending on the test method used. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D974:12. One participant used another test method (ASTM D664). When the ASTM D974 results are evaluated separately, the calculated reproducibility is in agreement with the requirements of ASTM D974:12.

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 D664:11a.

Base Number: This determination may be very problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the estimated reproducibility requirements of ASTM D2896:11. It should be noted that the precision data of ASTM D2896:11 are based on acid numbers ranging from 5 – 27 mg KOH/g.

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

iis also calculated a value for all results that were reported as 'less than' for example L4.0 or <4.0. This test method uses color standards with values that are 0.5 points apart, ranging from 0.5 – 8.0. Since the color is determined by comparing the color of the sample to these standards, it is assumed when for example L4.0 is reported, the actual colour lies between 3.5 and 4.0. iis calculated this value as 3.75 (4.0 minus 0.25). After converting these values, a statistical evaluation was done on this larger group of data (n=18). This resulted in a similar consensus value and a slightly smaller spread as compared to the original group with 10 numerical data.

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

Flash Point PMcc: This determination was problematic. One statistical outlier was observed and two laboratories were excluded from the statistical evaluation for using an Open Cup Flash Point method. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of ASTM D93:13e1, procedure B.

Kin.Visco.@ 40°C: This determination was not problematic. Three statistical outliers were observed. The reproducibility for used oils is not present in ASTM D445:12 (see §17.3). Therefore the target reproducibility is calculated from the reproducibilities found in iis PT's on used oils like hydraulic oil and lubricating oil (see appendix 3, ref. 16). The calculated reproducibility after rejection of the statistical outliers is in full agreement with the average reproducibility found for used oils in previous iis PTs.

Kin.Visco.@ 100°C: This determination was not problematic. One statistical outlier was observed. The reproducibility for used oils is not present in ASTM D445:12 (see §17.3). Therefore the target reproducibility is calculated from the reproducibilities found in iis PT's on used oils like hydraulic oil and lubricating oil (see appendix 3, ref. 16). The calculated reproducibility after rejection of the statistical outlier is in full agreement with the average reproducibility found for used oils in previous iis PTs.

Viscosity Index This determination was very problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is not at all in agreement with ASTM D2270:10e1.



Also iis calculated the Viscosity Index from the test results reported for the kinematic viscosities at 40°C and 100°C. These calculated viscosity indices were compared to the reported viscosity indices.

Seven reported test results were the same as the calculated test results. Twelve reported test results were rounded to nearest whole number or to the nearest even number, as is described in the method. This means half of the participants submitted rounded results, which might explain (part of) the higher spread found in this test. This is the reason iis prefers to do the statistical evaluation with unrounded results. Four participants did not calculate the viscosity index correctly, one submitted a completely different value and one participant corrected the value for the viscosity index without revising also the viscosities at 40°C and 100°C. Four participants used Stabinger viscosity and one used Houillon viscosity to calculate the viscosity index.

A separate statistical evaluation was done on the group of (iis) calculated viscosity indices without the values of the laboratories that had outlying test results in the Viscosity D445 tests or had used Stabinger or Houillon viscosities. The calculated reproducibility after rejection of the seven suspect data is smaller, but still not in agreement with ASTM D2270:10e1.

Level of Cont:

No statistical conclusions were drawn as the reported test results are categories. However, it is observed that the reported categories for contamination for each of the three levels vary over a large range. For  $\geq 4\mu\text{m}$ , 14-20 (80/160 – 5000/10000 particles/ml), for  $\geq 6\mu\text{m}$ , 12-19 (20/10 – 2500/5000 particles/ml) and for  $\geq 14\mu\text{m}$ , 8-16 (1.3/2.5 – 320/640 particles/ml).

For the next PT iis will request the participants to report the amount of particles per ml, in order to be able to perform a valid statistical evaluation.

Water:

This determination was not problematic. One statistical outlier was observed. However the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ASTM D6304:07.

Water separability: This determination for this oil was very problematic. Various test results were reported, ranging from no emulsion to 35 ml emulsion, 40 to 10 ml oil and 40 to 12 ml water. ASTM D1401 determines different stages of the water separability, for example the time needed to reach 3 mL or less of emulsion, the time needed to reach 37 mL of water and the time needed to reach a complete break of 0 mL emulsion. Also the method states that the test may be aborted after 30 minutes. It appears that some participants did. So in the next PT, iis will be more specific on the report form about which stage of water separability should be reported.

Five participants reached the stage with 3 mL or less emulsion, the

corresponding times have been statistically evaluated. No outliers were observed. The calculated reproducibility is almost in agreement with the requirements of ASTM D1401:12.

Foaming Characteristics: The Foaming characteristics can be divided into three groups, Sequence I, II and III. Two test results per sequence were reported: foaming tendency after a 5 minute blowing period and foaming stability after a 10 minute settling period. The determinations for foaming tendency were problematic. No outliers were observed. The calculated reproducibilities for the foaming tendency after rejection of the statistical outliers for all three sequences are not in agreement with the requirements of ASTM D892:13. For the foam stability, the reporting participants submitted zero for all three sequences. No statistical conclusions could be drawn for foam stability.

## 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and the reproducibility as found for the group of participating laboratories that participated. The average results, calculated reproducibilities and reproducibilities derived from literature standards (in casu ASTM and IP standards), are compared in the next table.

Parameter	unit	n	Average	2.8 * sd	R(lit)
Total Acidity	mg KOH/g	9	0.157	0.102	0.080
Total Acid Number	mg KOH/g	20	0.14	0.17	0.06
Total Base Number	mg KOH/g	10	1.24	2.89	(0.09)
Color ASTM		10	4.3	0.7	1.0
Density @ 15 °C	kg/m <sup>3</sup>	24	994.6	1.5	1.5
Flash Point PMcc	°C	18	256.9	14.7	10.0
Kinematic Viscosity @ 40 °C	mm <sup>2</sup> /s	26	25.013	0.327	0.450
Kinematic Viscosity @ 100 °C	mm <sup>2</sup> /s	27	5.051	0.116	0.111
Viscosity Index		24	132.2	7.2	2.0
Level of Contamination ≥4µm	ISO scale	18	16	n.a.	n.a.
Level of Contamination ≥6µm	ISO scale	18	15	n.a.	n.a.
Level of Contamination ≥14µm	ISO scale	18	11	n.a.	n.a.
Water by KF	mg/kg	17	675.06	202.15	841.93
Water Separability, o-w-e (t)	min	12	30	n.a.	n.a.
Foaming Charac. Seq I, 5 min	ml	12	163	317	85
Foaming Charac. Seq II, 5 min	ml	11	31	39	28
Foaming Charac. Seq III, 5 min	ml	11	204	369	104
Foaming Charac. Seq I, 10 min	ml	10	0	n.a.	n.a.
Foaming Charac. Seq II, 10 min	ml	9	0	n.a.	n.a.
Foaming Charac. Seq III, 10min	ml	9	0	n.a.	n.a.

Table 3: reproducibilities of results of sample #14056

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 standards. The tests that are problematic have been discussed in paragraph 4.1.

### 4.3 COMPARISON OF THE PROFICIENCY TEST OF MAY 2014 WITH PREVIOUS PTS

	May 2014	May 2013
Number of reporting labs	29	27
Number of results reported	301	336
Statistical outliers	10	14
Percentage outliers	3.3%	4.2%

Table 4: comparison with previous proficiency tests

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

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

Determination	May 2014	May 2013
Total Acidity	-	+/-
Total Acid Number	--	-
Total Base Number	--	--
Color ASTM	++	-
Density @ 15 °C	+/-	--
Flash Point PMcc	-	-
Kinematic Viscosity @ 40 °C	++	+
Kinematic Viscosity @ 100 °C	+/-	+
Viscosity Index	--	--
Level of Contamination	n.e.	n.e.
Water by KF	++	++
Water Separability, o-w-e (t)	n.e.	+
Foaming Charac. Seq I, 5 min	--	-
Foaming Charac. Seq II, 5 min	-	-
Foaming Charac. Seq III, 5 min	--	-
Foaming Charac. Seq I, 10 min	n.e.	n.e.
Foaming Charac. Seq II, 10 min	n.e.	n.e.
Foaming Charac. Seq III, 10min	n.e.	n.e.

Table 5: comparison determinations against the standard

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

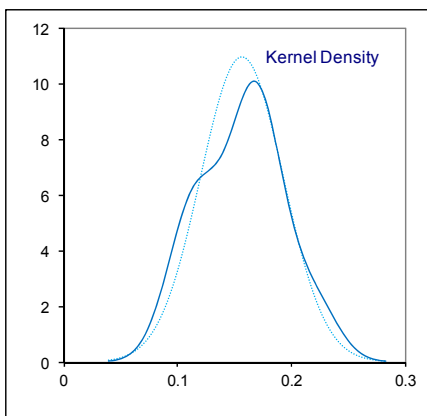
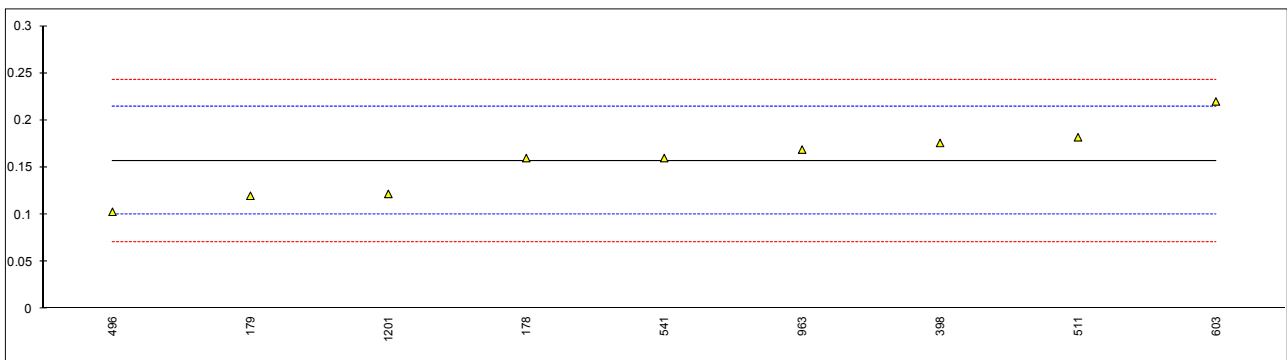
- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- : group performed worse than the standard
- : group performed much worse than the standard
- n.e.: not evaluated

**APPENDIX 1**

Determination of Total Acidity on sample #14056; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
178	D974	0.16		0.11	
179	D974	0.12		-1.29	
228		----		----	
331		----		----	
349		----		----	
398	D974	0.176		0.67	
432		----		----	
451		----		----	
495		----		----	
496	D974	0.103		-1.89	
511	D974	0.182		0.88	
541	D974	0.16		0.11	
603	D664	0.22		2.21	
657		----		----	
902		----		----	
963	D974	0.169		0.42	
1013		----		----	
1134		----		----	
1146		----		----	
1174		----		----	
1201	D974	0.122		-1.22	
1423		----		----	
1431		----		----	
1435		----		----	
1461		----		----	
1578		----		----	
1599		----		----	
1604		----		----	
1660		----		----	
1827		----		----	

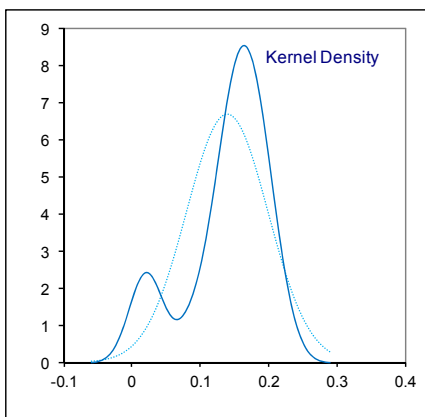
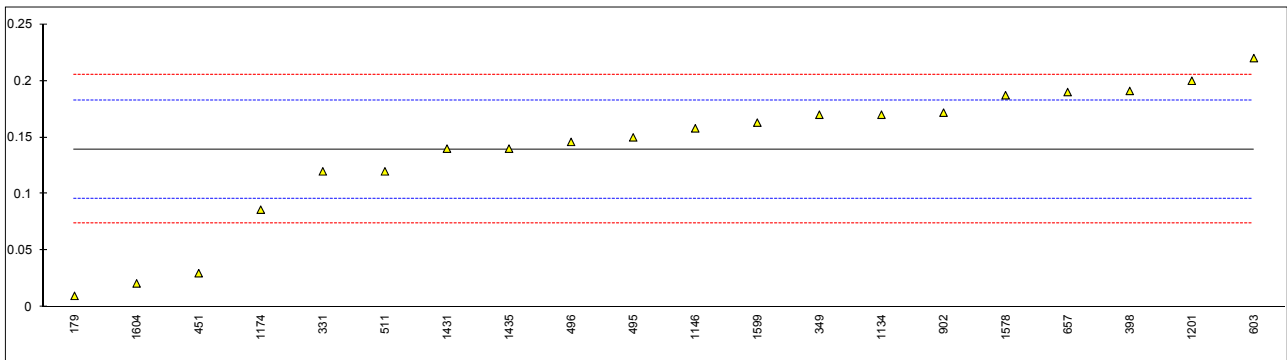
normality	OK	<u>Only ASTM D974</u>
n	9	OK
outliers	0	8
mean (n)	0.1569	0.1490
st.dev. (n)	0.03645	0.02963
R(calc.)	0.1021	0.0830
R(D974:12)	0.0800	0.0800



Determination of Acid Number on sample #14056; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
178		----		----	
179	D664	0.01		-5.91	
228		----		----	
331	D664	0.12		-0.88	
349	D664	0.17		1.41	
398	D664	0.191		2.37	
432		----		----	
451	D664-A	0.03		-4.99	
495	D664-A	0.15		0.49	
496	D664-A	0.146		0.31	
511	D664-A	0.120		-0.88	
541		----		----	
603	D664-A	0.22		3.69	
657	D664-A	0.19		2.32	
902	D664-A	0.1718		1.49	
963		----		----	
1013		----		----	
1134	IP177	0.17		1.41	
1146	D664-A	0.158		0.86	
1174	INH-1752	0.0861		-2.43	
1201	D664-A	0.20		2.78	
1423		----		----	
1431	D664-A	0.14		0.04	
1435	D664-A	0.14		0.04	
1461		----		----	
1578	ISO6618	0.1872		2.19	
1599	D664-A	0.163		1.09	
1604	D664-A	0.021		-5.40	
1660		----		----	
1827		----		----	

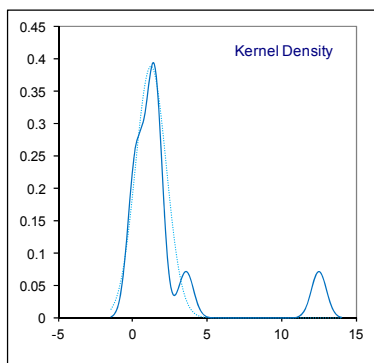
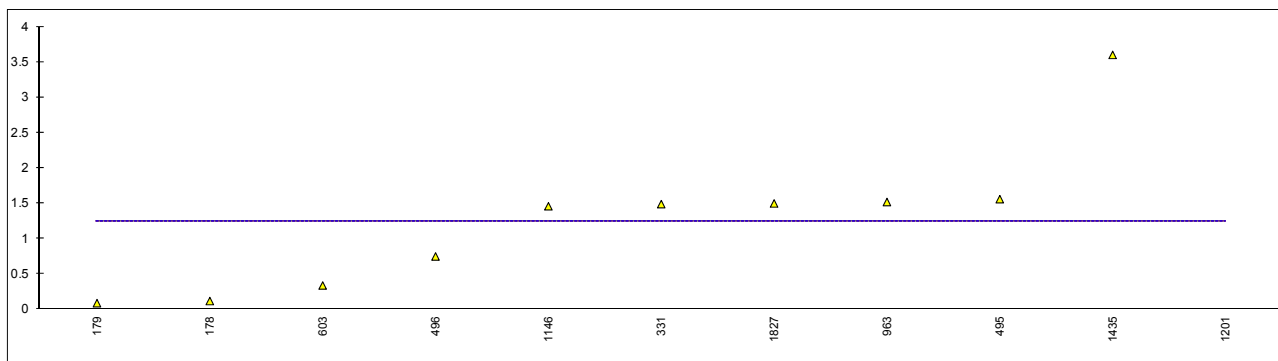
normality OK  
n 20  
outliers 0  
mean (n) 0.14  
st.dev. (n) 0.060  
R(calc.) 0.17  
R(D664:11a) 0.06



Determination of Base Number on sample #14056; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
178	D4739Mod.	0.12		----	
179	D2896-B	0.09		----	
228		----		----	
331	D2896-B	1.49		----	
349		----		----	
398	D2896-B	<0.1		----	
432		----		----	
451		----		----	
495	D2896-A	1.56		----	
496	D2896-B	0.75		----	
511		----		----	
541	D2896-B	<0.5		----	
603	D2896-A	0.34		----	
657		----		----	
902		----		----	
963	D2896-A	1.52		----	
1013		----		----	
1134		----		----	
1146	D2896-A	1.46		----	
1174		----		----	
1201	D2896-A	12.52	G(0.01)	----	False positive result?
1423		----		----	
1431		----		----	
1435	D2896	3.60		----	
1461		----		----	
1578		----		----	
1599		----		----	
1604		----		----	
1660		----		----	
1827	D2896-B	1.5	C	----	Probably unit error, reported: 0.0015

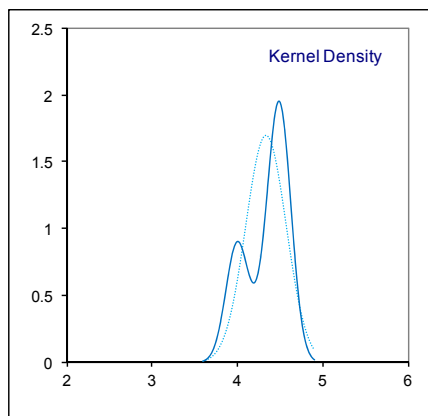
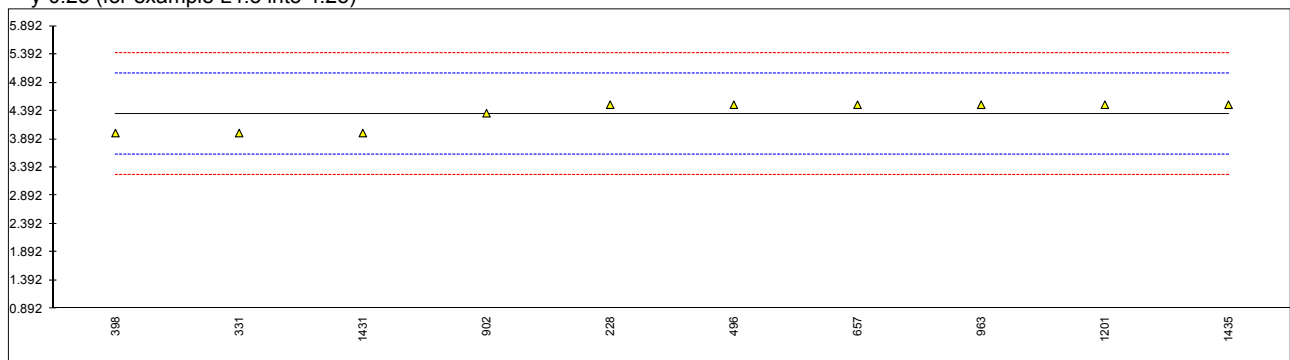
			<u>Only proc. A</u>	<u>Only proc. B</u>	
normality	not OK		Unknown	Unknown	
n	10		4	4	
outliers	1		1	0	
mean (n)	1.243		1.220	0.957	
st.dev. (n)	1.0304		0.5881	0.6766	
R(calc.)	2.885		1.647	1.895	
R(D2896:11)	(0.087)		(0.087)	(0.087)	Range ASTM D2896 = 5 – 27 mg KOH/g



Determination of Color ASTM on sample #14056

lab	method	value	mark	z(targ)	value calc.	mark	remarks
178		----		----	----		
179	D1500	L4.0			3.75		
228	D1500	4.5		0.46	4.5		
331	D1500	4.0		-0.94	4.0		
349		----		----	----		
398	D1500	4.0		-0.94	4.0		
432	D1500	L4.5			4.25		
451		----		----	----		
495	D1500	L4.5			4.25		
496	D1500	4.5		0.46	4.5		
511		----		----	----		
541	D1500	L4.5			4.25		
603	D1500	L4.5			4.25		
657	D1500	4.5		0.46	4.5		
902	D1500	4.35		0.04	4.35		
963	D1500	4.5		0.46	4.5		
1013	D1500	L4.5			4.25		
1134	D1500	>4.0			4.25		
1146		----		----	----		
1174	ISO2049	L4.5			4.25		
1201	D1500	4.5		0.46	4.5		
1423		----		----	----		
1431	D1500	4		-0.94	4		
1435	D1500	4.5		0.46	4.5		
1461		----		----	----		
1578		----		----	----		
1599		----		----	----		
1604		----		----	----		
1660		----		----	----		
1827		----		----	----		
normality		OK			OK		
n		10			18		
outliers		0			0		
mean (n)		4.34			4.27 (L4.5)		
st.dev. (n)		0.236			0.219		
R(calc.)		0.66			0.61		
R(D1500:12)		1.00			1.00		

\*) In the calculation of the mean, standard deviation and the reproducibility of this column, a reported value of 'L y' is changed into y-0.25 (for example L4.5 into 4.25)

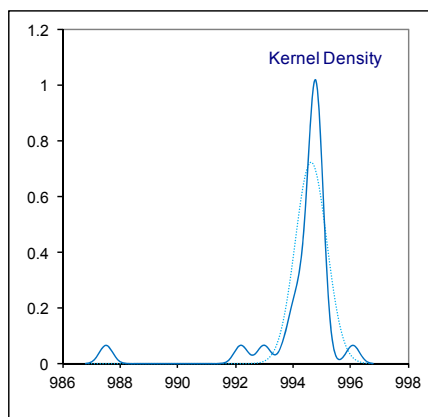
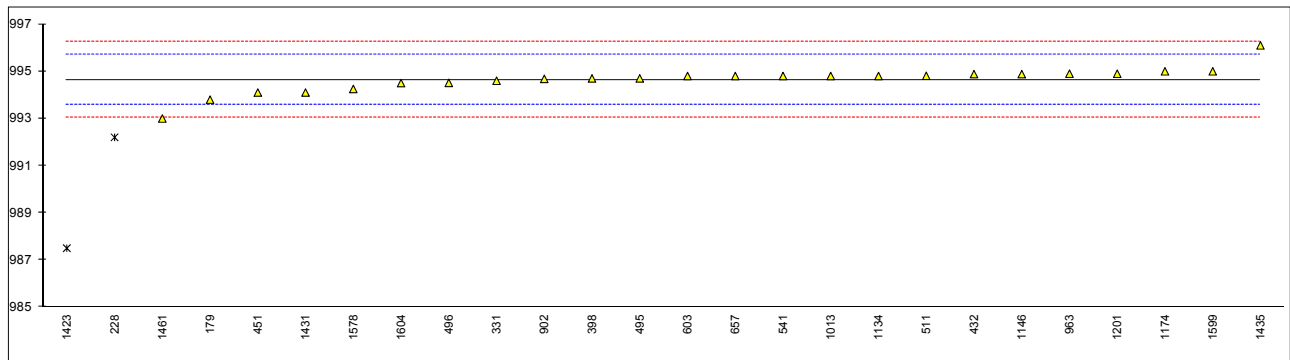




Determination of Density @ 15°C on sample #14056; results in kg/m<sup>3</sup>

lab	method	value	mark	z(targ)	remarks
178		----		----	
179	D1298	993.8		-1.57	
228	D1298	992.2	R(0.01)	-4.56	
331	ISO12185	994.6		-0.08	
349		----		----	
398	D4052	994.7		0.11	
432	ISO12185	994.88		0.44	
451	D4052	994.1	C	-1.01	Probably unit error, reported: 0.9941
495	D4052	994.7		0.11	
496	D1298	994.51		-0.25	
511	D1298	994.81		0.31	
541	D4052	994.8		0.29	
603	D4052	994.8		0.29	
657	D4052	994.8		0.29	
902	D4052	994.68		0.07	
963	D4052	994.9		0.48	
1013	D4052	994.8	C	0.29	Probably unit error, reported: 0.9948
1134	IP363	994.8		0.29	
1146	D4052	994.88		0.44	
1174	ISO3675	995.0		0.67	
1201	D1298	994.9		0.48	
1423	D1298	987.5	C,R(0.01)	-13.33	Probably unit error, reported: 0.9875
1431	D4052	994.1		-1.01	
1435	D4052	996.1	C	2.72	Probably unit error, reported: 0.9961
1461	ISO3675	993.0		-3.07	
1578	ISO3675	994.25		-0.73	
1599	D5002	995	C	0.67	First reported: 0.995
1604	D1298	994.5		-0.27	
1660		----		----	
1827		----		----	

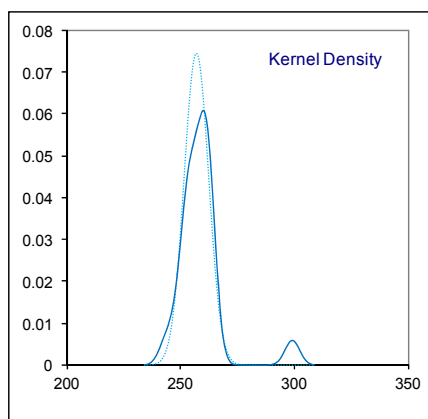
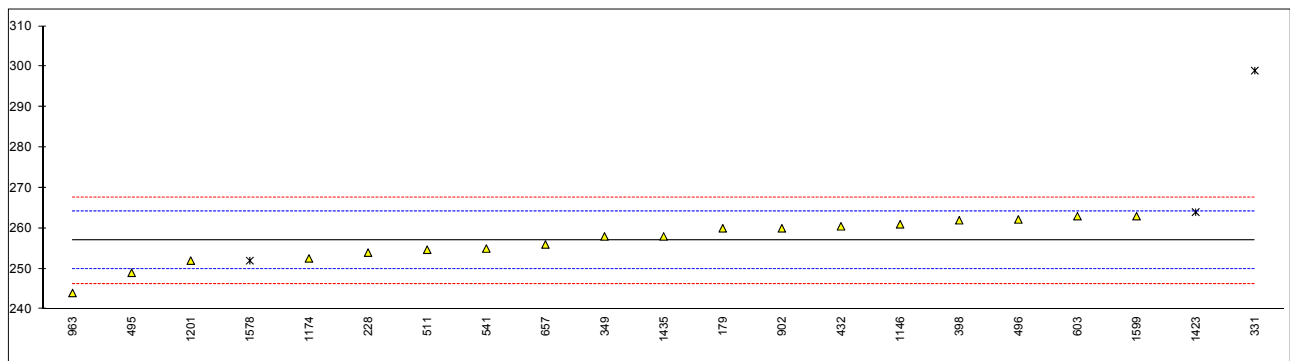
normality not OK  
n 24  
outliers 2  
mean (n) 994.64  
st.dev. (n) 0.551  
R(calc.) 1.54  
R(D1298:12b) 1.50



Determination of Flash Point PMcc on sample #14056; results in °C

lab	method	value	mark	z(targ)	remarks
178		----		----	
179	D93	260		0.86	
228	D93	254.0		-0.82	
331	D93	299.0	G(0.01)	11.78	
349	D93	258.0		0.30	
398	D93	262		1.42	
432	D93	260.5		1.00	
451		----		----	
495	D93	249.0		-2.22	
496	D93	262.2		1.47	
511	D93	254.7		-0.63	
541	D7094	255.0		-0.54	
603	D3828	263.0		1.70	
657	D93	256.0		-0.26	
902	D93	260.0		0.86	
963	D93	244		-3.62	
1013		----		----	
1134	D93	>110		----	
1146	in house	261.0		1.14	
1174	ISO2719	252.55	C	-1.23	First reported: 234.57
1201	D93	252.0		-1.38	
1423	D92	264.0	ex	1.98	See §4.1
1431		----		----	
1435	D93	258.0		0.30	
1461		----		----	
1578	ISO2592	252	ex, C	-1.38	See §4.1, first reported: 277.5
1599	D93	263		1.70	
1604		----		----	
1660		----		----	
1827		----		----	

normality OK  
n 18  
outliers 1 (+2 ex)  
mean (n) 256.94  
st.dev. (n) 5.254  
R(calc.) 14.71  
R(D93:13e1) 10.00

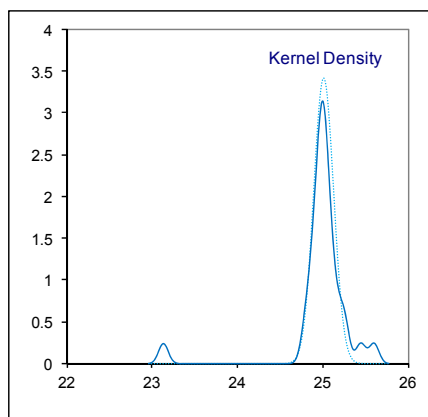
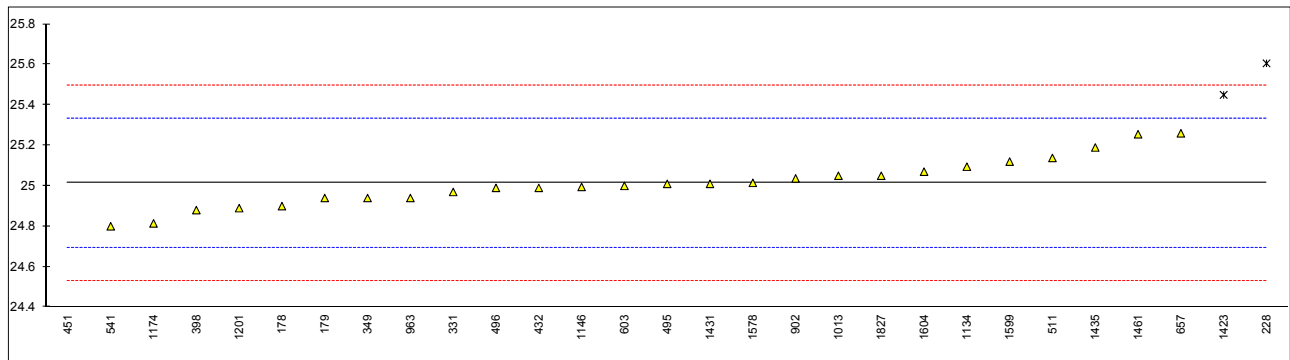


Determination of Kinematic Viscosity @ 40°C on sample #14056; results in mm<sup>2</sup>/s

lab	method	value	mark	z(targ)	remarks
178	D445	24.9		-0.71	
179	D445	24.94		-0.46	
228	D445	25.606	R(0.05)	3.69	
331	D7279	24.97		-0.27	
349	D445	24.94		-0.46	
398	D445	24.880		-0.83	
432	D445	24.99		-0.15	
451	D7279	23.13	R(0.01)	-11.71	
495	D445	25.01		-0.02	
496	D445	24.990		-0.15	
511	D445	25.138		0.77	
541	D445	24.80		-1.33	
603	D7042	25.00		-0.08	
657	D445	25.26		1.53	
902	D445	25.037		0.15	
963	D445	24.94		-0.46	
1013	D7042	25.05		0.23	
1134	IP71	25.095		0.51	
1146	D445	24.995		-0.11	
1174	ISO3104	24.81416		-1.24	
1201	D445	24.89		-0.77	
1423	D445	25.45	R(0.05)	2.71	
1431	D7042	25.01		-0.02	
1435	D7042	25.19		1.10	
1461	ISO3104	25.2552		1.50	
1578	ISO3104	25.01496		0.01	
1599	D445	25.12		0.66	
1604	D445	25.07		0.35	
1660		----		----	
1827	D445	25.05		0.23	

normality OK  
n 26  
outliers 3  
mean (n) 25.0134  
st.dev. (n) 0.11683  
R(calc.) 0.3271  
R(iis) 0.4502

R calculated from iis reports on used oils for ASTM D445: 1.8% of mean

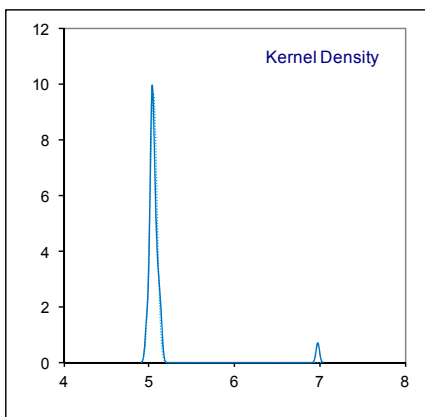
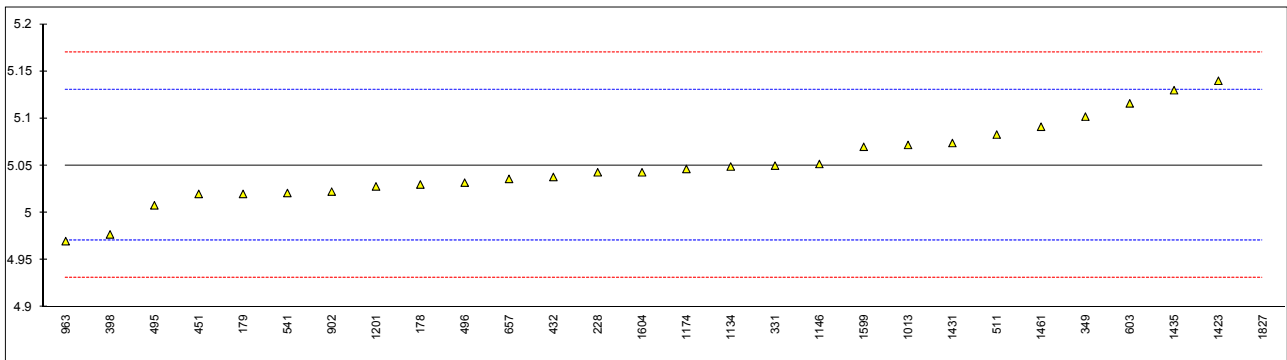


Determination of Kinematic Viscosity @ 100°C on sample #14056; results in mm<sup>2</sup>/s

lab	method	value	mark	z(targ)	remarks
178	D445	5.03		-0.52	
179	D445	5.02		-0.77	
228	D445	5.043		-0.19	
331	D7279	5.05		-0.01	
349	D445	5.102		1.30	
398	D445	4.9771		-1.85	
432	D445	5.038		-0.32	
451	D7279	5.02		-0.77	
495	D445	5.008		-1.07	
496	D445	5.0320		-0.47	
511	D445	5.083		0.82	
541	D445	5.021		-0.74	
603	D7042	5.116		1.65	
657	D445	5.036		-0.37	
902	D445	5.0225		-0.71	
963	D445	4.970		-2.03	
1013	D7042	5.072		0.54	
1134	IP71	5.0491		-0.04	
1146	D445	5.0518		0.03	
1174	ISO3104	5.04648		-0.10	
1201	D445	5.028		-0.57	
1423	D445	5.14		2.25	
1431	D7042	5.074		0.59	
1435	D7042	5.13		2.00	
1461	ISO3104	5.0913		1.03	
1578		----		----	
1599	D445	5.07		0.49	
1604	D445	5.043		-0.19	
1660		----		----	
1827	D445	6.975	R(0.01)	48.50	

normality OK  
n 27  
outliers 1  
mean (n) 5.0505  
st.dev. (n) 0.04130  
R(calc.) 0.1156  
R(iis) 0.1111

R calculated from iis reports on used oils for ASTM D445: 2.2% of mean

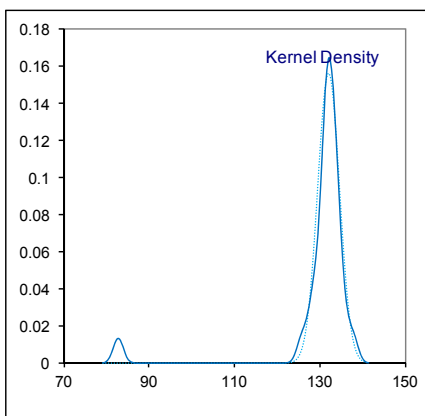
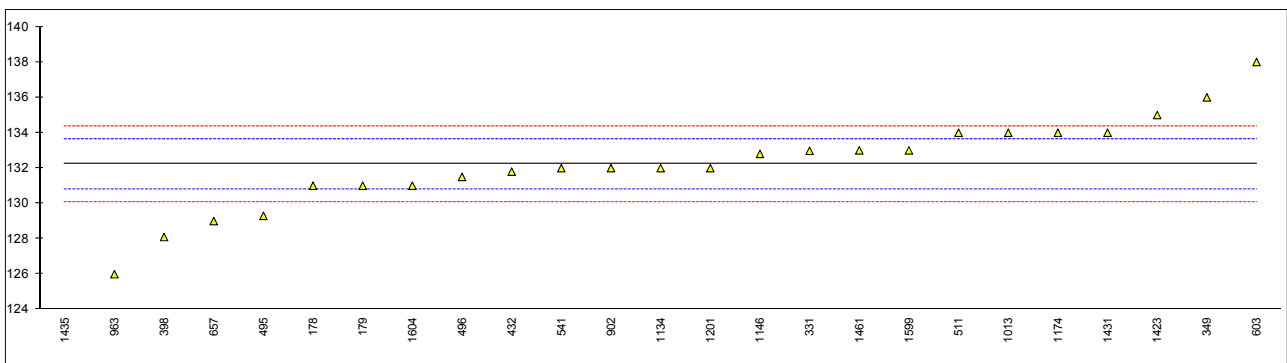


Determination of Viscosity Index from Kinematic Viscosity @ 40&100°C on sample #14056

lab	method	value	mark	z(targ)	calc. iis	mark	remarks
178	D2270	131		-1.72	132.07		
179	D2270	131		-1.72	130.90		
228		----		----	----		
331	INH-2270	132.98		1.05	132.94	ex	Based on D7279
349	D2270	136	C	5.28	137.28	ex	First reported: 137
398	D2270	128.1		-5.78	128.13		
432	D2270	131.8		-0.60	131.81		
451		----		----	----		
495	D2270	129.3		-4.10	129.28		
496	D2270	131.5		-1.02	131.35		
511	D2270	134		2.48	133.85		
541	D2270	132		-0.32	132.35		
603	D2270	138		8.08	137.75	ex	Based on D7042
657	D2270	129		-4.52	129.05		
902	D2270	132.0		-0.32	130.15		
963	D2270	126		-8.72	127.00		
1013	D2270	134		2.48	133.86	ex	Based on D7042
1134	IP226	132		-0.32	131.65		
1146	D2270	132.8		0.80	132.84		
1174	ISO2909	134		2.48	134.21		
1201	D2270	132		-0.32	132.01		
1423	in house	135		3.88	135.15	ex	Outlier in D445 40°C
1431	D2270	134		2.48	134.41	ex	Based on D7042
1435	D2270	82.94	R(0.01)	-69.00	136.94	ex	Based on D7042
1461	ISO2909	133		1.08	133.34		
1578		----		----	----		
1599	D2270	133		1.08	133.02		
1604	D2270	131		-1.72	131.42		
1660		----		----	----		
1827		----		----	----		

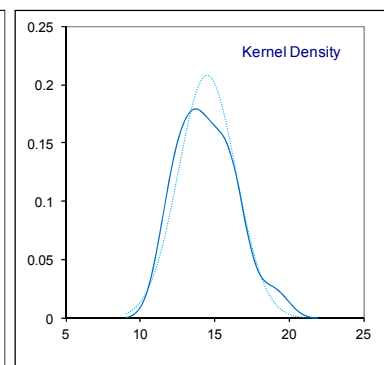
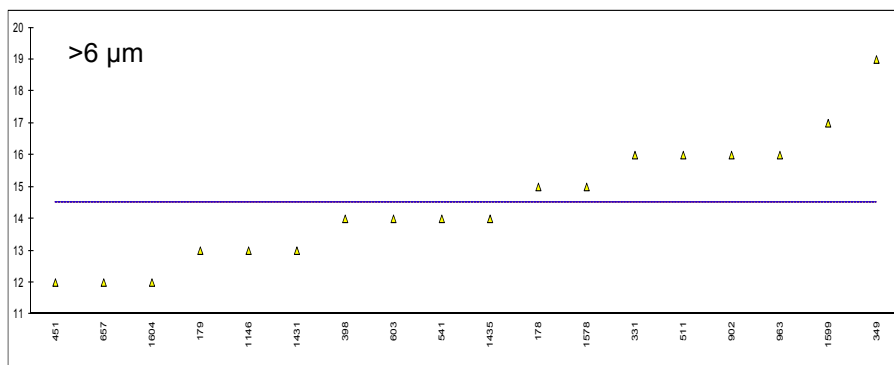
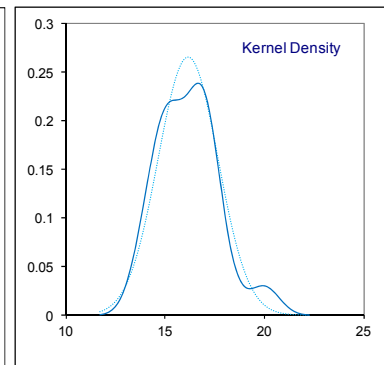
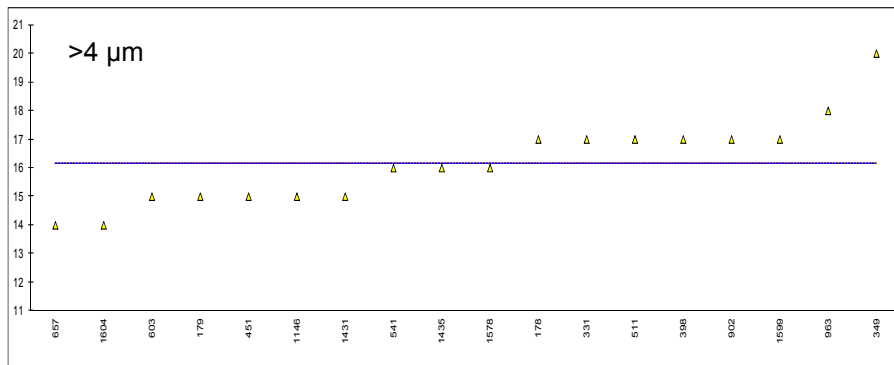
  

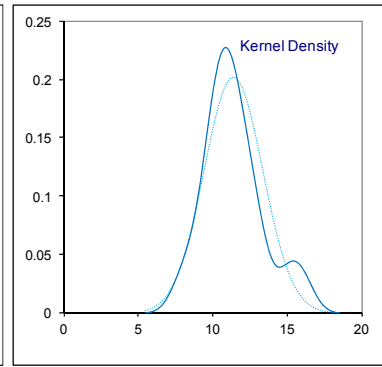
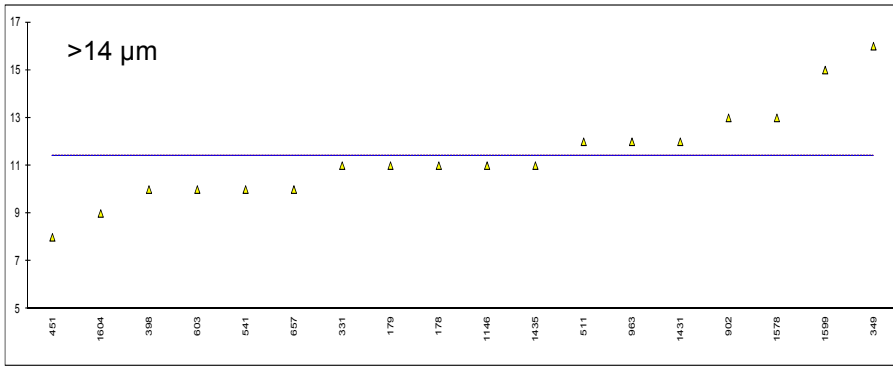
normality	suspect	VI based on D445/ISO3104/IP71 valid data
n	24	OK
outliers	1	18
mean (n)	132.23	0 (+7ex)
st.dev. (n)	2.555	131.36
R(calc.)	7.15	1.973
R(D2270:10e1)	2.00	5.52
		2.00



Determination of Level of contamination on sample #14056; results in ISO scale

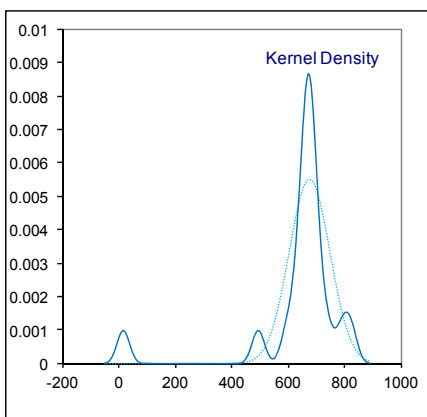
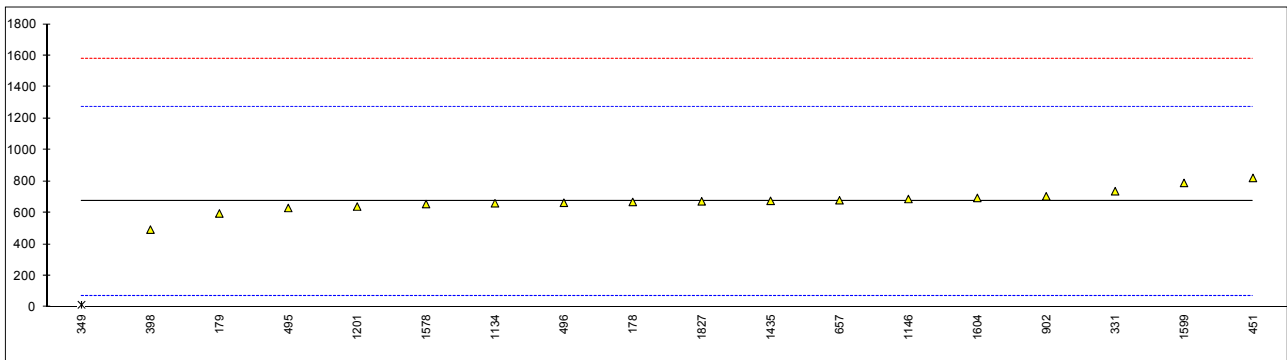
lab	method	≥4µm (c)	≥6µm (c)	≥14µm (c)	mark	z(targ)	remarks
178	INH-1185	17	15	11		----	
179	ISO4406	15	13	11		----	
228		----	----	----		----	
331	ISO4406	17	16	11		----	
349	ISO4406	20	19	16		----	
398	ISO4406	17	14	10		----	
432		----	----	----		----	
451	ISO4406	15	12	8		----	
495		----	----	----		----	
496		----	----	----		----	
511	ISO4406	17	16	12		----	
541	ISO4406	16	14	10		----	
603	ISO4406	15	14	10		----	
657	ISO4406/7	14	12	10		----	
902	ISO4406	17	16	13		----	
963	ISO440/7	18	16	12		----	
1013		----	----	----		----	
1134		----	----	----		----	
1146	ISO4406/7	15	13	11		----	
1174		----	----	----		----	
1201		----	----	----		----	
1423		----	----	----		----	
1431	ISO4406/7	15	13	12		----	
1435	ISO4406	16	14	11		----	
1461		----	----	----		----	
1578	ISO4406	16	15	13		----	
1599	ISO4406/9	17	17	15		----	
1604	ISO4406	14	12	9		----	
1660		----	----	----		----	
1827		----	----	----		----	
normality		n.a.	n.a.	n.a.			
n		18	18	18			
outliers		n.a.	n.a.	n.a.			
mean (n)		16.2	14.5	11.4			
st.dev. (n)		n.a.	n.a.	n.a.			
R(calc.)		n.a.	n.a.	n.a.			
R(ISO4406)		Unknown	Unknown	Unknown			





Determination of Water by KF on sample #14056; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D6304	669		-0.02	
179	D6304	596		-0.26	
228		----		----	
331	D6304	737.3		0.21	
349	D6304C	15	C,G(0.01)	-2.20	First reported: 35
398	D6304E	493.2		-0.60	
432		----		----	
451	D6304C	822		0.49	
495	D6304C	631		-0.15	
496	D6304C	664		-0.04	
511		----		----	
541		----		----	
603		----		----	
657	D6304C	680		0.02	
902	D6304A	705.33		0.10	
963		----		----	
1013		----		----	
1134	IP438	660.8		-0.05	
1146	D6304C	688		0.04	
1174		----		----	
1201	D6304C	640		-0.12	
1423		----		----	
1431		----		----	
1435	D1744	676		0.00	
1461		----		----	
1578	ISO12937	655.775		-0.06	
1599	D4377	790		0.38	
1604	D6304C	694.5		0.06	
1660		----		----	
1827	D6304A	673.1		-0.01	
normality		not OK			
n		17			
outliers		1			
mean (n)		675.059			
st.dev. (n)		72.1957			
R(calc.)		202.148			
R(D6304:07)		841.934			



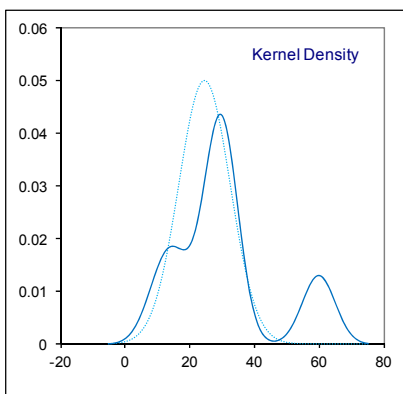
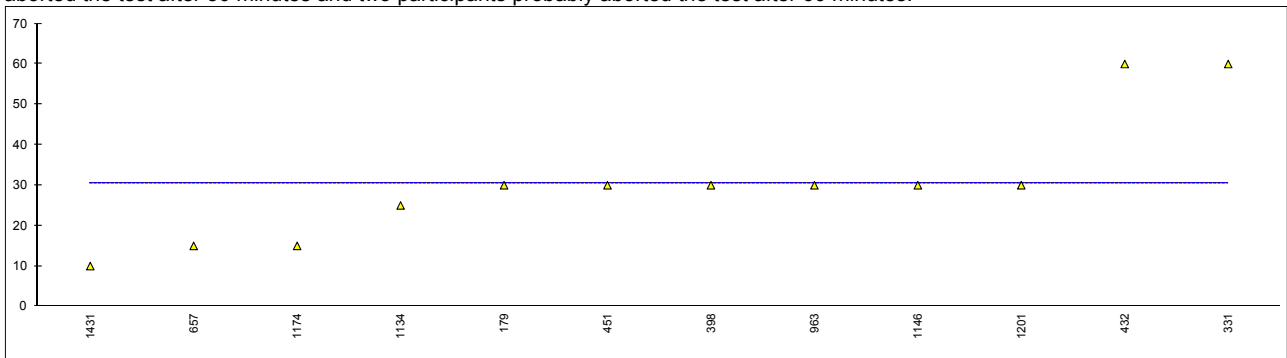


Determination of Water Separability @ 54°C on sample #14056; oil-water-em. in ml / time in min.

lab	method	ml o-w-e	time	mark	z(targ)	remarks
178		----	----		----	
179	D1401	32-37-11	30		----	
228		----	----		----	
331	D1401	31-35-14	60		----	
349		----	----		----	
398	D1401	10-23-47	30		----	
432	D1401	35-37-8	60		----	
451	D1401	36-12-32	30		----	
495		----	----		----	
496		----	----		----	
511		----	----		----	
541		----	----		----	
603		----	----		----	
657	D1401	40-38-2	15		----	(A)
902		----	----		----	
963	D1401	40-40-0	30		----	(A)
1013		----	----		----	
1134	D1401	39-40-1	25		----	(A)
1146	D1401	22-30-28	30		----	
1174	ISO6614	38-40-2	15		----	(A)
1201	D1401	25-20-35	30		----	
1423		----	----		----	
1431	D1401	40-40-0	10		----	(A)
1435		----	----		----	
1461		----	----		----	
1578		----	----		----	
1599		----	----		----	
1604		----	----		----	
1660		----	----		----	
1827		----	----		----	

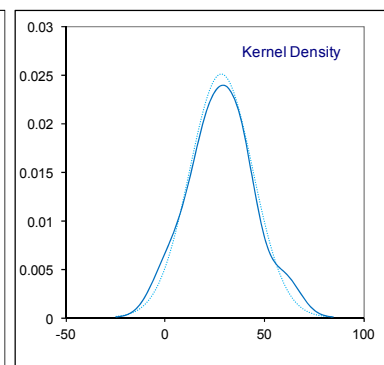
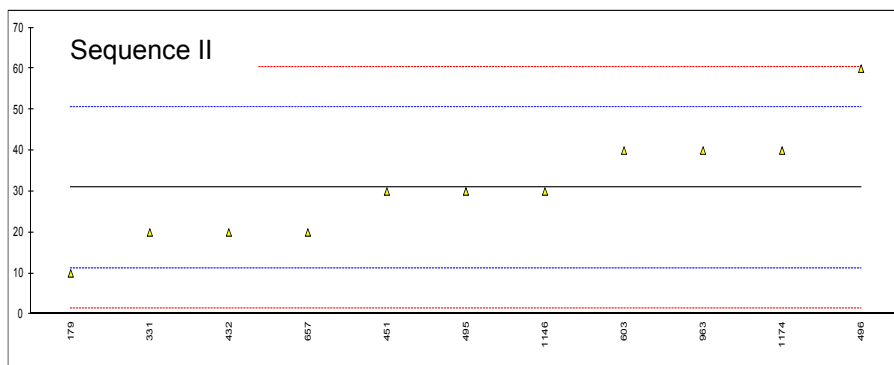
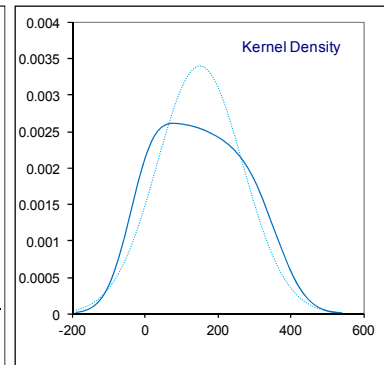
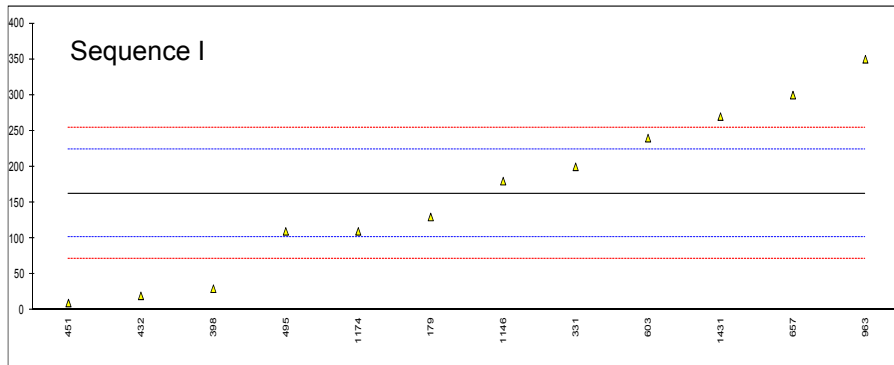
			<u>Only emulsion &lt; 3ml (A)</u>
normality		Suspect	Unknown
n		12	5
outliers		0	0
mean (n)		30.4	19.0
st.dev. (n)		n.a.	8.22
R(calc.)		n.a.	23.0
R(D1401:12)		n.a.	20.0

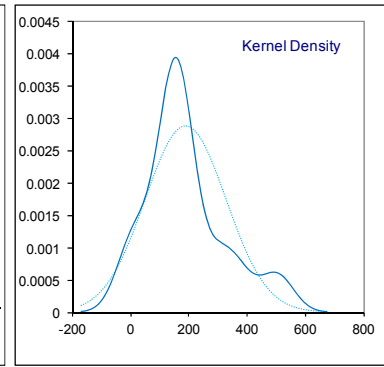
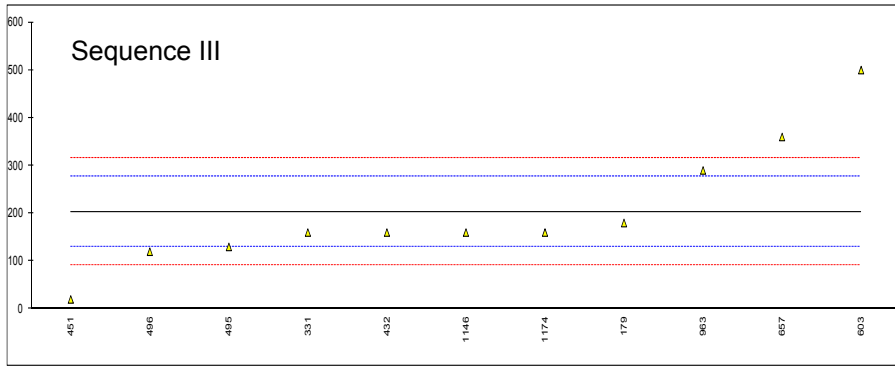
ASTM D1401 states that the test may be aborted after 30 minutes at 54°C and after 60 minutes at 82°C. Other participants probably aborted the test after 30 minutes and two participants probably aborted the test after 60 minutes.



Determination of Foaming Characteristics, as received; Sequence I, II and III at the end of 5 min blowing period on sample #14056; results in ml

Lab	method	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
178										
179	D892	130		-1.07	10		-2.13	180		-0.63
228										
331	D892	200		1.23	20		-1.11	160		-1.17
349										
398	D892	30		-4.34						
432	D892	20		-4.67	20		-1.11	160		-1.17
451	D892	10		-5.00	30		-0.09	20		-4.93
495	D892	110		-1.72	30		-0.09	130		-1.97
496					60		2.96	120		-2.24
511										
541										
603	D892	240		2.54	40		0.93	500		7.95
657	D892	300		4.51	20		-1.11	360		4.19
902										
963	D892	350		6.15	40		0.93	290		2.32
1013										
1134										
1146	D892	180		0.57	30		-0.09	160		-1.17
1174	D892	110		-1.72	40		0.93	160		-1.17
1201										
1423										
1431	D892	270		3.52						
1435										
1461										
1578										
1599										
1604										
1660										
1827										
normality		OK			OK			not OK		
n		12			11			11		
outliers		0			0			0		
mean (n)		162.5			30.9			203.6		
st.dev. (n)		113.23			13.75			131.93		
R(calc.)		317.0			38.5			369.4		
R(D892:13)		85.4			27.5			104.4		





Determination of Foaming Characteristics, as received; Sequence I, II and III at the end of 10 min settling period on sample #14056; results in ml

Lab	method	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
178		----		----	----		----	----		----
179		----		----	----		----	----		----
228		----		----	----		----	----		----
331		----		----	----		----	----		----
349		----		----	----		----	----		----
398	D892	0		----	----		----	----		----
432	D892	0		----	0		----	0		----
451	D892	0		----	0		----	0		----
495	D892	0		----	0		----	0		----
496		----		----	0		----	0		----
511		----		----	----		----	----		----
541		----		----	----		----	----		----
603	D892	0		----	0		----	0		----
657	D892	0		----	0		----	0		----
902		----		----	----		----	----		----
963		----		----	----		----	----		----
1013		----		----	----		----	----		----
1134		----		----	----		----	----		----
1146	D892	0		----	0		----	0		----
1174	D892	0		----	0		----	0		----
1201		----		----	----		----	----		----
1423		----		----	----		----	----		----
1431	D892	0		----	----		----	----		----
1435	ISO 6247	0		----	0		----	0		----
1461		----		----	----		----	----		----
1578		----		----	----		----	----		----
1599		----		----	----		----	----		----
1604		----		----	----		----	----		----
1660		----		----	----		----	----		----
1827		----		----	----		----	----		----
	normality	n.a.			n.a.			n.a.		
	n	10			9			9		
	outliers	n.a.			n.a.			n.a.		
	mean (n)	0			0			0		
	st.dev. (n)	n.a.			n.a.			n.a.		
	R(calc.)	n.a.			n.a.			n.a.		
	R(D892:13)	n.a.			n.a.			n.a.		

## APPENDIX 2

### Number of participants per country

1 lab in ARGENTINA  
1 lab in AUSTRIA  
1 lab in BELGIUM  
3 labs in BULGARIA  
1 lab in FRANCE  
2 labs in GERMANY  
1 lab in GREECE  
2 labs in ITALY  
2 labs in MALAYSIA  
2 labs in NETHERLANDS  
1 lab in NIGERIA  
1 lab in PERU  
1 lab in PORTUGAL  
1 lab in SAUDI ARABIA  
1 lab in SINGAPORE  
3 labs in SPAIN  
1 lab in TOGO  
1 lab in TURKEY  
2 labs in UNITED KINGDOM  
2 labs in UNITED STATES OF AMERICA

**APPENDIX 3****Abbreviations:**

C	= final result after checking of first reported suspect 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 outlier test
R(0.05)	= straggler in Rosner outlier test
ex	= excluded from calculations
U	= reported in different unit
W	= result withdrawn on request of the participants
fr.	= first reported
S	= scope of the reported method is not applicable
n.a.	= not applicable
n.e.	= not evaluated
SDS	= Material Safety Data Sheet
R(iis)	= reproducibility based on the reproducibilities found in previous iis PT's for this test

**Literature:**

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