

Results of Proficiency Test  
Base 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 Base Oil. In the annual proficiency testing program 2013/2014, it was decided to continue the proficiency test for the analyses of Base Oil. In this interlaboratory study 45 laboratories in 26 different countries have participated. See appendix 2 for the number of participants per country. In this report, the results of the 2014 Base 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 #14055) of Base Oil that was purchased from a local supplier. 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 (R007), 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 obtained from a local supplier. The 200 litre bulk material (HVI-650) was homogenized and part of this bulk was transferred into 62 brown glass bottles of 1 litre (labelled #14055). The homogeneity of the subsamples #14055 was checked by determination of Density @15°C in accordance with ASTM D4052:11 and Kinematic Viscosity 40°C in accordance with ASTM D445:12 on 8 stratified randomly selected samples.

	Density @ 15 °C in kg/L	Kin. Vis. @ 40°C in mm <sup>2</sup> /s
Sample #14055-1	0.86713	21.05
Sample #14055-2	0.86713	21.05
Sample #14055-3	0.86713	21.07
Sample #14055-4	0.86713	21.05
Sample #14055-5	0.86713	21.04
Sample #14055-6	0.86713	21.04
Sample #14055-7	0.86713	21.04
Sample #14055-8	0.86713	21.04

Table 1: homogeneity test results of subsamples #14055

From the above test results, the repeatabilities were 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/L	Kin. Vis. @ 40°C in mm <sup>2</sup> /s
r (sample #14055)	0.00000	0.03
reference test	ASTM D4052:11	ASTM D445:12
0.3 x R(reference test)	0.00015	0.04

Table 2: evaluation of the repeatabilities of the subsamples #14055

The calculated repeatabilities were less than 0.3 times the corresponding reproducibilities of the reference test methods. Therefore, homogeneity of the subsample #14055 was assumed.

To each of the participating laboratories, one sample of 1 L in a brown glass bottle (labelled #14055) was sent on April 16, 2014.

## 2.5 ANALYSES

The participants were requested to determine on sample #14055: Acid Number (Total), Air-release time @25°C, Color ASTM, Conradson Carbon Residue, Ramsbottom Carbon Residue, Density @ 15°C, Flash Point COC, Kinematic Viscosity @ 40°C and @ 100°C, Viscosity Stabinger @ 40°C and @100°C, Viscosity Index, Evaporation loss by Noack test, Pour Point, Rust prevention (proc. B), Sulphur, Water and Water Separability @ 54°C.

To get comparable results, a detailed report form, on which the units were prescribed as well as some of the required standards, was prepared. This form together with a letter of instructions were 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 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 the appendix 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' of April 2014 (iis-protocol, version 3.3). 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 (1986 and 1994, lit.8 and 9) 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.13 and 14). 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, no problems were encountered with the dispatch of the samples. Five participants reported the test results after the final reporting date and two participants did not report any test results at all. Not all laboratories were able to report all analyses requested. In total 43 participants reported 408 test results. Observed were 19 outlying results, which is 4.7% 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.

### 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. D6892) and an added designation for the year that the method was adopted or revised (e.g. D6892:03). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D6892:03(2014)). In the results tables of Appendix 1 only the method number and year of adoption or revision e.g. D6892:03 will be used.

Acid Number (total): This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D974:12.

Air-release time: Only seven test results were reported. The determination may be problematic. The calculated reproducibility is not in agreement with the requirements of ASTM D3427:12.

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

iis also calculated a value for all results that were reported as 'less than' for example 1.0 or <1.0. This test method uses color standards with values that are 0.5 points apart, ranging from 0.5 – 8.0. Since this color test is determined by comparing the color of the sample to these standards, it is assumed when for example L1.0 is reported, the actual colour lies between 0.5 and 1.0. iis calculated this value as 0.75 (1.0 minus 0.25).

After converting these values, a statistical evaluation was done on this larger group of data (n=40). This resulted in a similar consensus value, but a smaller spread as compared to the original group with 18 numerical data.

- Conradson CR: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D4530:11. The application range of this method is 0.1% - 30%.
- Ramsbottom CR: Only five test results were reported. The determination may be problematic. The calculated reproducibility is not in agreement with the requirements of ASTM D524:10.
- 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 agreement with the requirements of ASTM D4052:11.
- Flash Point COC: This determination was not problematic. No statistical outliers were observed, although the test results of two participants were excluded for performing a Closed Cup test (ASTM D93 and ASTM D3828). The calculated reproducibility after rejection of the suspect data is in agreement with ASTM D92:12b.
- Kin.Visco.@ 40°C: This determination was problematic. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D445:12.
- Kin.Visco.@ 100°C: This determination was problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D445:12.
- Visco. Stabinger at 40°C This determination was very problematic. One statistical outlier was observed. However, the calculated reproducibility is not at all in agreement with ASTM D7042:12a.
- Visco. Stabinger at 100°C This determination was very problematic. No statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outlier is not at all in agreement with ASTM D7042:12a.
- Viscosity Index: This determination was very problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in not at all 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 test results were compared to the reported test results.

Thirteen reported test results were the same as the calculated test results. Eighteen 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. Three participants used Stabinger viscosity to calculate the viscosity index. Although this is allowed by the method (ASTM D2270), the differences reported for this PT sample for both kinematic and Stabinger viscosities by single laboratories are significant and thus will have an impact on the viscosity index result.

Two participants corrected the value for viscosity without revising also the viscosity index. Calculations on the corrected and uncorrected viscosity values showed that even if only one decimal figure is added to the value of viscosity, it could cause the viscosity index to change more than one point. Rounding of the viscosity values before calculating the viscosity index appears to have a great impact. This could be another cause of the high spread in viscosity index found.

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

It may be concluded that using less rounded results of the kinematic viscosities for the calculation of the viscosity index and reporting less rounded results for viscosity index might improve the performance of the group.

Evaporation loss: 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 D5800B:10.

Two participants reported the method CEC L-040-93B, which is equivalent to ASTM D5800, except this method uses a known correction factor. One participant reported DIN51581, which is equivalent to ASTM D5800A. This value can be converted according to the method to a ASTM D5800B result. The statistical evaluation was performed after conversion of these three results to ASTM D5800B.

The spread found in this PT is much higher than the requirements of ASTM D5800B and CEC L-040093B, but similar to ASTM D5800A. It is remarkable that a group of laboratories using an automated method like ASTM D5800B would have almost the same large spread as the spread of the manual method (ASTM D5800A).

- Pour Point: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with (strict) requirements of ASTM D6892:03(2014). The large spread may (partly) be explained by rounding of the test results up to 3 degrees.
- Rust prevention: Regretfully, only four participants reported a test result. Three reported the presence of rust (Fail), while one participant reported the test as "Pass".
- Sulphur: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of both ASTM D2622:10 and ASTM D4294:10.
- Water: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D6304:07.
- Water separability The determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D1401:12.

## 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 standards), are compared in the next table.

Parameter	unit	n	Average	2.8 * sd	R(lit)
Total Acid Number	mg KOH/g	21	0.014	0.038	0.040
Air-release time @25°C	min	7	2.6	3.0	2.1
Color ASTM		18	0.8	0.8	1.0
Conradson Carbon Residue	%M/M	11	0.008	0.020	0.141
Ramsbottom Carbon Residue	%M/M	5	0.060	0.084	0.029
Density @ 15 °C	kg/L	41	0.8671	0.0004	0.0005
Flash Point COC	°C	34	207.3	17.7	18.0
Kinematic Viscosity @ 40 °C	mm <sup>2</sup> /s	36	21.11	0.20	0.14
Kinematic Viscosity @ 100 °C	mm <sup>2</sup> /s	37	4.153	0.037	0.027
Stabinger Viscosity @ 40 °C	mm <sup>2</sup> /s	11	21.19	0.38	0.12
Stabinger Viscosity @ 100 °C	mm <sup>2</sup> /s	10	4.173	0.120	0.027
Viscosity Index		39	95.9	5.2	2.0
Evaporation loss by Noack	%M/M	11	26.44	4.29	1.34
Pour Point	°C	30	-12.8	4.1	3.6
Rust Prevention (proc. B)		4	n.a.	n.a.	n.a.
Sulphur	%M/M	32	0.696	0.044	0.051
Water	mg/kg	28	57.6	47.9	192.2
Water Separability @ 54°C	min	12	6	7	20

Table 3: reproducibilities of results of sample #14055

Without further statistical calculations it can be concluded that for a number of tests there is a not 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 PT'S

	May 2014	May 2013
Number of reporting labs	43	28
Number of results reported	408	260
Statistical outliers	19	17
Percentage outliers	4.7%	6.5%

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 Acid Number	+/-	+/-
Air-release time @25°C	-	n.e.
Color ASTM	+	++
Conradson Carbon Residue	++	-
Ramsbottom Carbon Residue	--	n.e.
Density @ 15 °C	++	--
Flash Point COC	+/-	+
Kinematic Viscosity @ 40 °C	--	--
Kinematic Viscosity @ 100 °C	--	-
Stabinger Viscosity @ 40 °C	--	--
Stabinger Viscosity @ 100 °C	--	--
Viscosity Index	--	+
Evaporation loss by Noack	--	--
Pour Point	-	-
Rust Prevention	n.e.	n.e.
Sulphur	+	+/-
Water	++	+
Water Separability @ 54°C	++	++

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

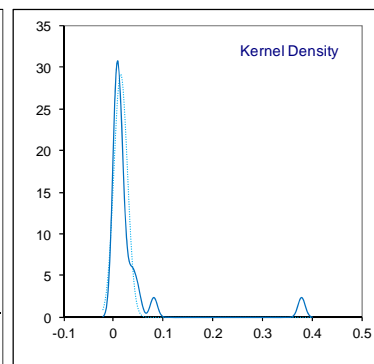
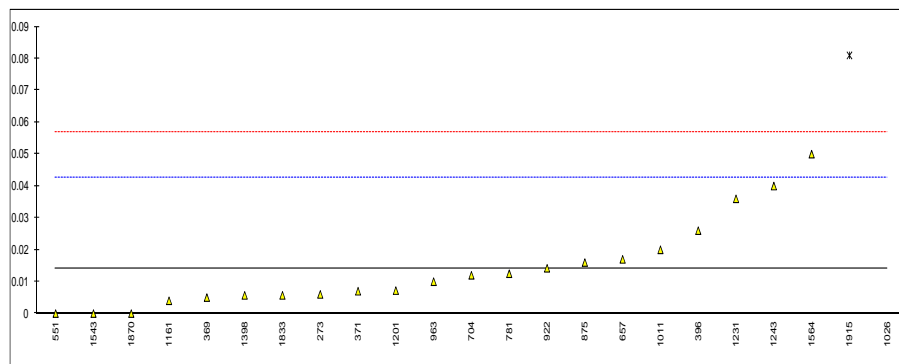
**APPENDIX 1**

Determination of Acid Number (Total) on sample #14055; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
273	D974	0.006		-0.56	
311	D974	<0.01		----	
315		----		----	
337		----		----	
357	D664	<0.05		----	
369	D974	0.005		-0.63	
371	D974	0.007		-0.49	
396	D974	0.026		0.84	
432		----		----	
485		----		----	
494	D664	<0.05		----	
495	D974	<0.0003		----	
496	D974	<0.02		----	
541	D974	<0.1		----	
551	D974	0.00		-0.98	
601		----		----	
603	D664	<0.05		----	
657	D974	0.017		0.21	
704	D974	0.012		-0.14	
781	D974	0.0125		-0.11	
875	D664	0.016		0.14	
902	D974	<0.02		----	
922	D974	0.0142		0.01	
963	D974	0.010		-0.28	
966		----		----	
1011	D974	0.020		0.42	
1013		----		----	
1026	D974	0.38	R(0.01)	25.62	
1161	D664	0.004		-0.70	
1201	D974	0.0072		-0.48	
1231	D664	0.036		1.54	
1243	D974	0.04		1.82	
1349		----		----	
1394		----		----	
1398	INH-5985	0.0057		-0.58	
1423		----		----	
1461		----		----	
1543	D664	0.00		-0.98	
1564	D664	0.05		2.52	
1706		----		----	
1793		----		----	
1833	D974	0.0057		-0.58	
1870	D664	0.00		-0.98	
1877		----		----	
1915	D974	0.081	R(0.01)	4.69	

Only ASTM D974 data

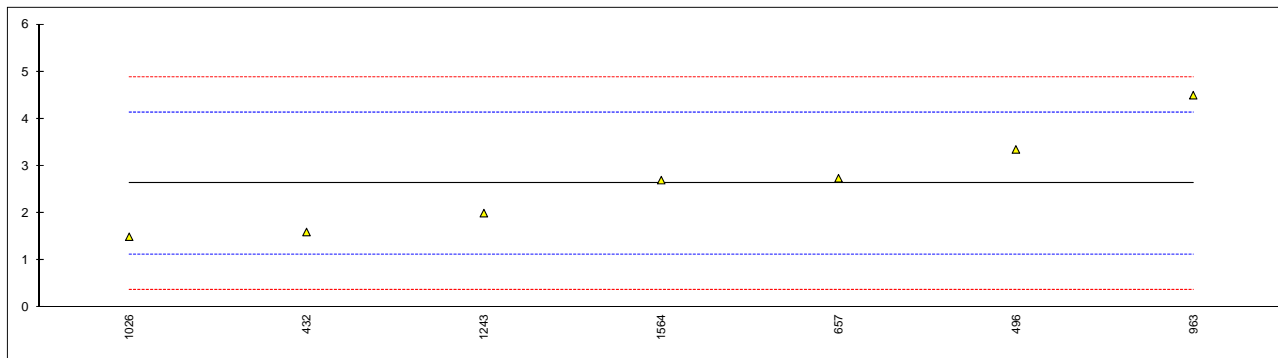
normality	not OK	not OK
n	21	14
outliers	2	2
mean (n)	0.0140	0.0130
st.dev. (n)	0.01367	0.01030
R(calc.)	0.0383	0.0288
R(D974:12)	0.0400	0.0400



Determination of Air-release time @ 25°C on sample #14055; results in min

lab	method	value	mark	z(targ)	remarks
273		----		----	
311		----		----	
315		----		----	
337		----		----	
357		----		----	
369		----		----	
371		----		----	
396		----		----	
432	ISO9120	1.6		-1.36	
485		----		----	
494		----		----	
495		----		----	
496	D3427	3.35		0.96	
541		----		----	
551		----		----	
601		----		----	
603		----		----	
657	D3427	2.74		0.15	
704		----		----	
781		----		----	
875		----		----	
902		----		----	
922		----		----	
963	D3427	4.5		2.49	
966		----		----	
1011		----		----	
1013		----		----	
1026	D3427	1.5		-1.50	
1161		----		----	
1201		----		----	
1231		----		----	
1243	D3427	2.0		-0.83	
1349		----		----	
1394		----		----	
1398		----		----	
1423		----		----	
1461		----		----	
1543		----		----	
1564	D3427	2.7		0.10	
1706		----		----	
1793		----		----	
1833		----		----	
1870		----		----	
1877		----		----	
1915		----		----	

normality unknown No Kernel Density graph available  
n 7  
outliers 0  
mean (n) 2.63  
st.dev. (n) 1.062  
R(calc.) 2.97  
R(D3427:12) 2.11

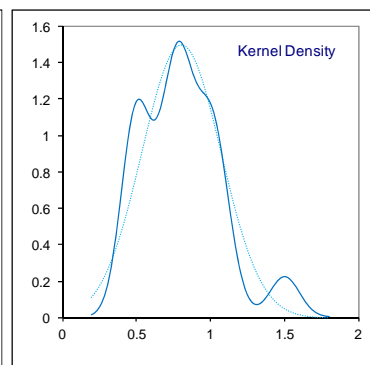
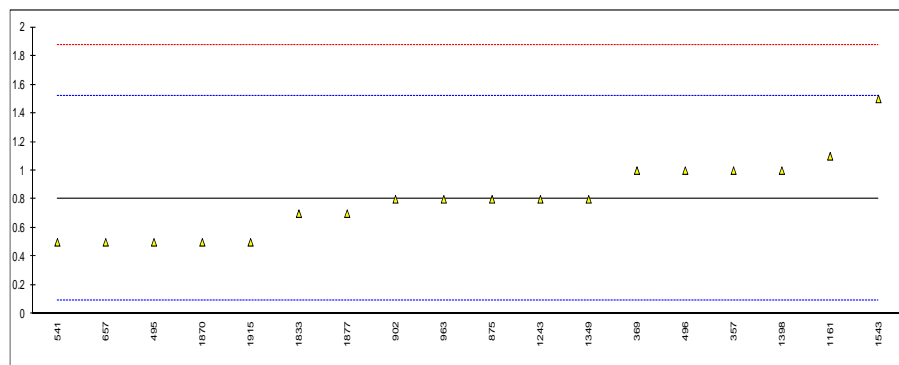


Determination of Color on sample #14055

lab	method	value	mark	z(targ)	value calc.	mark	remarks
273	D1500	L1.0		----	0.75		
311	D1500	L1.0		----	0.75		
315	D1500	L1.0		----	0.75		
337				----			
357	D1500	1.0		0.54	1.0		
369	D1500	1.0		0.54	1.0		
371	D1500	L1.0		----	0.75		
396	D1500	L1.0		----	0.75		
432	D1500	L1.0		----	0.75		
485				----			
494	D1500	L0.5		----	0.25		
495	D1500	0.5		-0.86	0.5		
496	D1500	1.0		0.54	1.0		
541	D1500	0.5		-0.86	0.5		
551	D1500	L0.5		----	0.25		
601	D1500	L1.0		----	0.75		
603	D1500	L1.0		----	0.75		
657	D1500	0.5		-0.86	0.5		
704	D1500	L1.0		----	0.75		
781	D1500	<1.0		----	0.75		
875	D6045	0.8		-0.02	0.8		
902	D1500	0.8		-0.02	0.8		
922	D1500	L1.0		----	0.75		
963	D1500	0.8		-0.02	0.8		
966				----			
1011	D1500	L0.5		----	0.25		
1013	D1500	L1.0		----	0.75		
1026	D1500	L1.0		----	0.75		
1161	D6045	1.1		0.82	1.1		
1201	D1500	L1.0		----	0.75		
1231	D1500	L1.0		----	0.75		
1243	D1500	0.8		-0.02	0.8		
1349	D1500	0.8		-0.02	0.8		
1394	D1500	<1		----	0.75		
1398	D1500	1.0		0.54	1.0		
1423	D1500	L1.0		----	0.75		
1461				----			
1543	D1500	1.5		1.94	1.5		
1564	D1500	L1		----	0.75		
1706	D1500	L1.0		----	0.75		
1793				----			
1833	D1500	0.7		-0.30	0.7		
1870	D1500	0.5		-0.86	0.5		
1877	D6045	0.7		-0.30	0.7		
1915	D1500	0.5		-0.86	0.5		

normality	suspect	not OK
n	18	40
outliers	0	0
mean (n)	0.81	0.74 (L1.0)
st.dev. (n)	0.267	0.227
R(calc.)	0.75	0.64
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 L1.0 into 0.75)

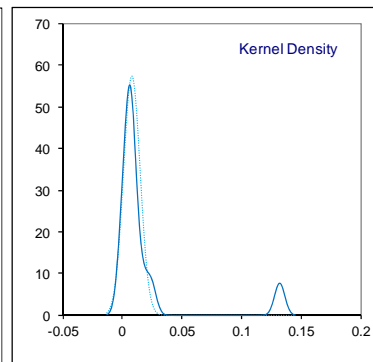
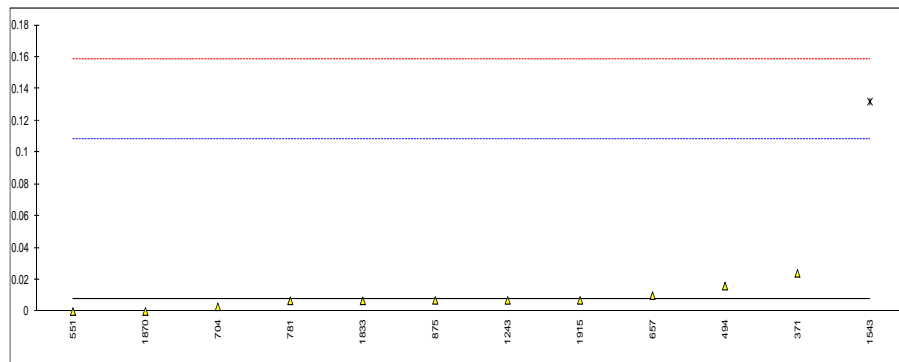


Determination of Conradson Carbon Residue on sample #14055; results in %M/M

lab	method	value	mark	z(targ)	remarks
273	D4530	<0.01		----	
311	D4530	<0.10		----	
315		----		----	
337		----		----	
357		----		----	
369	D4530	<0.01		----	
371	D189	0.024		0.32	
396		----		----	
432		----		----	
485		----		----	
494	D4530	0.016		0.16	
495	D4530	<0.01		----	
496	D4530	<0.03		----	
541		----		----	
551	D4530	0.0		-0.16	
601		----		----	
603	D4530	<0.01		----	
657	D4530	0.01		0.04	
704	D4530	0.003		-0.10	
781	D4530	0.0067		-0.02	
875	D4530	0.007		-0.02	
902		----		----	
922	D4530	<0.01		----	
963		----		----	
966		----		----	
1011		----		----	
1013		----		----	
1026		----		----	
1161		----		----	
1201	D4530	<0.10		----	
1231		----		----	
1243	D4530	0.007		-0.02	
1349		----		----	
1394		----		----	
1398		----		----	
1423		----		----	
1461		----		----	
1543	D4530	0.1320	G(0.01)	2.47	False positive test result?
1564		----		----	
1706		----		----	
1793		----		----	
1833	D4530	0.0067		-0.02	
1870	D4530	0.00		-0.16	
1877		----		----	
1915	D189	0.007		-0.02	

normality not OK  
n 11  
outliers 1  
mean (n) 0.0079  
st.dev. (n) 0.00695  
R(calc.) 0.0195  
R(D4530:11) 0.1408

Application range of ASTM D4530: 0.1% – 30%

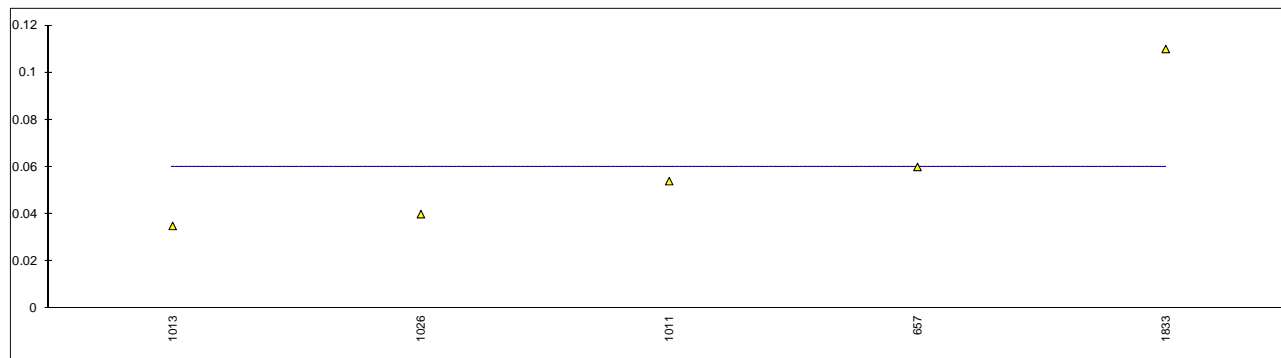




Determination of Ramsbottom Carbon Residue on sample #14055; results in %M/M

lab	method	value	mark	z(targ)	remarks
273		----		----	
311		----		----	
315		----		----	
337		----		----	
357		----		----	
369		----		----	
371		----		----	
396		----		----	
432		----		----	
485		----		----	
494		----		----	
495		----		----	
496		----		----	
541		----		----	
551		----		----	
601		----		----	
603		----		----	
657	D524	0.06		----	
704		----		----	
781		----		----	
875		----		----	
902		----		----	
922		----		----	
963		----		----	
966		----		----	
1011	D524	0.054		----	
1013	D524	0.035		----	
1026	D524	0.04		----	
1161		----		----	
1201		----		----	
1231		----		----	
1243		----		----	
1349		----		----	
1394		----		----	
1398		----		----	
1423		----		----	
1461		----		----	
1543		----		----	
1564		----		----	
1706		----		----	
1793		----		----	
1833	D524	0.11		----	
1870		----		----	
1877		----		----	
1915		----		----	

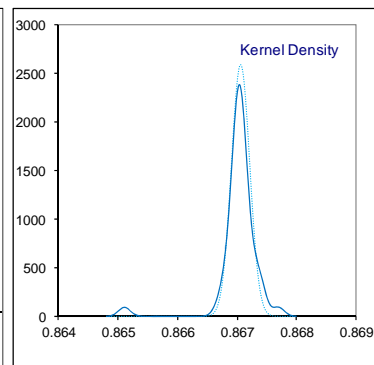
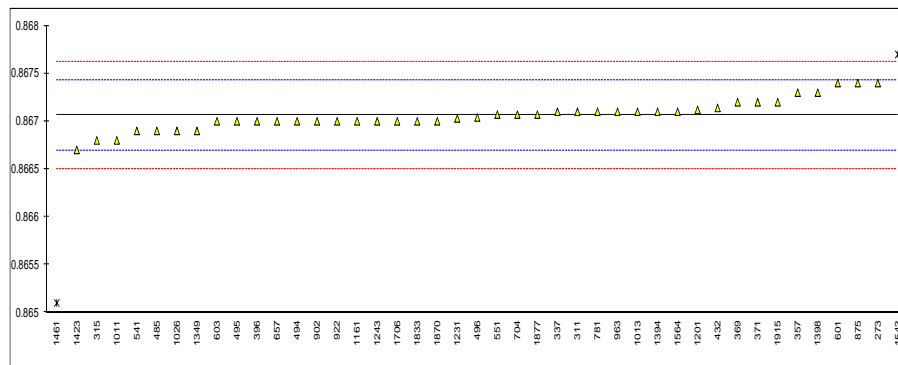
normality unknown No Kernel Density graph available  
n 5  
outliers 0  
mean (n) 0.0598  
st.dev. (n) 0.02984  
R(calc.) 0.0835  
R(D524:10) 0.0292



Determination of Density @ 15°C on sample #14055; results in kg/L

lab	method	value	mark	z(targ)	remarks
273	D4052	0.8674		1.82	
311	D4052	0.8671		0.21	
315	D4052	0.8668		-1.41	
337	D4052	0.8671		0.21	
357	D4052	0.8673		1.28	
369	D4052	0.8672		0.74	
371	D4052	0.8672		0.74	
396	D4052	0.8670		-0.33	
432	D4052	0.86714		0.42	
485	D4052	0.8669		-0.87	
494	D4052	0.8670		-0.33	
495	D4052	0.8670		-0.33	
496	D4052	0.86704		-0.12	
541	D4052	0.8669		-0.87	
551	D4052	0.86707		0.04	
601	D4052	0.8674	C	1.82	Probably unit error, reported 867.4
603	D4052	0.8670		-0.33	
657	D4052	0.8670		-0.33	
704	D4052	0.86707		0.04	
781	D4052	0.8671		0.21	
875	D4052	0.8674		1.82	
902	D4052	0.8670		-0.33	
922	D4052	0.8670		-0.33	
963	D4052	0.8671		0.21	
966		----		----	
1011	D4052	0.8668		-1.41	
1013	D4052	0.8671		0.21	
1026	D4052	0.8669		-0.87	
1161	ISO12185	0.867	C	-0.33	First reported: 866.0 (ISO 3675)
1201	D4052	0.86712		0.31	
1231	D4052	0.86703		-0.17	
1243	D4052	0.867		-0.33	
1349	IP365	0.8669	C	-0.87	First reported: 866.9
1394	D4052	0.8671	C	0.21	First reported: 867.1
1398	D4052	0.8673		1.28	
1423	D1298	0.8667		-1.95	
1461	ISO3675	0.8651	C, R(0.01)	-10.56	Probably unit error, reported 865.1
1543	D4052	0.8677	R(0.01)	3.44	
1564	D4052	0.8671		0.21	
1706	ISO12185	0.8670		-0.33	
1793		----		----	
1833	D4052	0.8670		-0.33	
1870	D4052	0.8670		-0.33	
1877	D4052	0.86707		0.04	
1915	D4052	0.8672	C	0.74	First reported 867.2

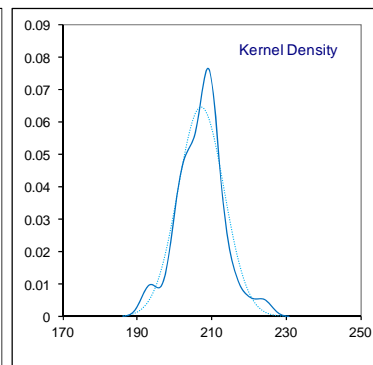
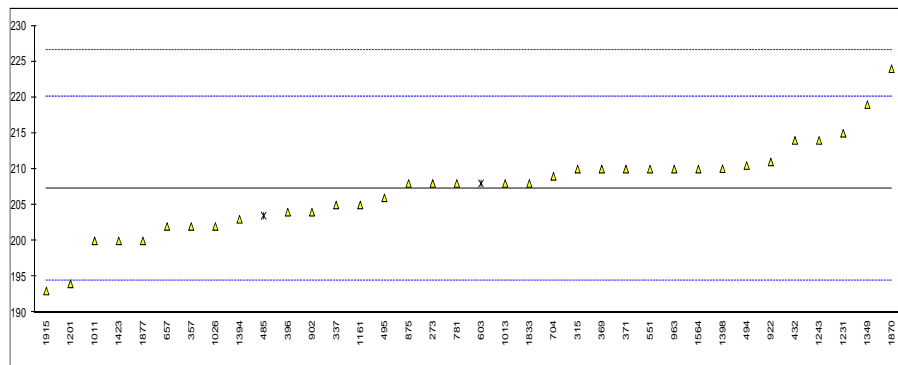
normality OK  
n 41  
outliers 2  
mean (n) 0.86706  
st.dev. (n) 0.000154  
R(calc.) 0.00043  
R(D4052:11) 0.00052



Determination of Flash Point C.O.C. on sample #14055; results in °C

lab	method	value	mark	z(targ)	remarks
273	D92	208		0.12	
311		-----		-----	
315	D92	210		0.43	
337	D92	205		-0.35	
357	D92	202		-0.82	
369	D92	210.0		0.43	
371	D92	210		0.43	
396	D92	204		-0.51	
432	D92	214		1.05	
485	D93	203.5	ex	-0.58	See §4.1
494	D92	210.5		0.51	
495	D92	206.0		-0.19	
496		-----		-----	
541		-----		-----	
551	D92	210		0.43	
601		-----		-----	
603	D3828	208.0	ex	0.12	See §4.1
657	D92	202		-0.82	
704	D92	209.0		0.27	
781	D92	208		0.12	
875	D92	208		0.12	
902	D92	204		-0.51	
922	D92	211		0.58	
963	D92	210		0.43	
966		-----		-----	
1011	D92	200		-1.13	
1013	D92	208		0.12	
1026	D92	202		-0.82	
1161	ISO2592	205.0		-0.35	
1201	D92	194.0		-2.06	
1231	D92	215		1.21	
1243	D92	214.0		1.05	
1349	D92	219.0		1.83	
1394	D92	203		-0.66	
1398	D92	210.05		0.44	
1423	D92	200.0		-1.13	
1461		-----		-----	
1543		-----		-----	
1564	D92	210		0.43	
1706		-----		-----	
1793		-----		-----	
1833	D92	208		0.12	
1870	D92	224		2.61	
1877	D92	200.0		-1.13	
1915	D92	193		-2.22	

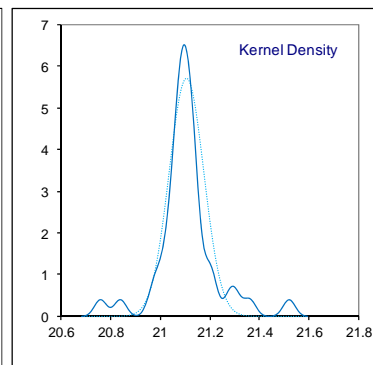
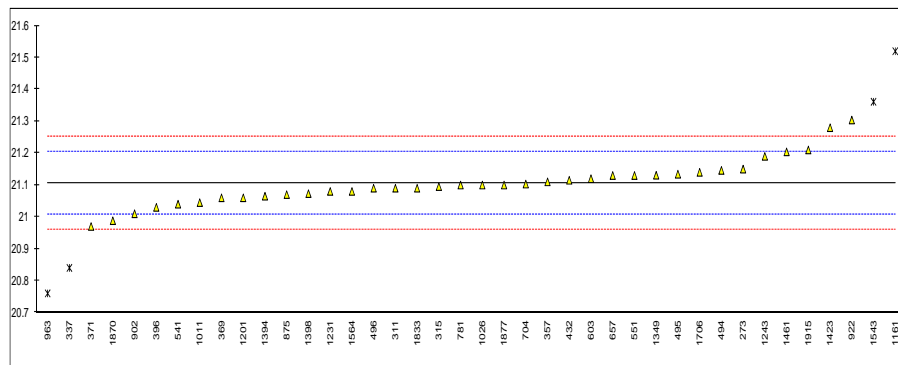
normality OK  
n 34 (+2 ex)  
outliers 0  
mean (n) 207.25  
st.dev. (n) 6.333  
R(calc.) 17.73  
R(D92:12b) 18.00



Determination of Kinematic Viscosity @ 40°C on sample #14055; results in mm<sup>2</sup>/s

lab	method	value	mark	z(targ)	remarks
273	D445	21.15		0.89	
311	D445	21.09		-0.34	
315	D445	21.095		-0.23	
337	D445	20.84	C,R(0.05)	-5.44	First reported: 21.78
357	D445	21.11		0.07	
369	D445	21.06		-0.95	
371	D445	20.97		-2.79	
396	D445	21.03		-1.56	
432	D445	21.115		0.17	
485		-----			
494	D445	21.146		0.81	
495	D445	21.134		0.56	
496	D445	21.090		-0.34	
541	D445	21.04		-1.36	
551	D445	21.13		0.48	
601		-----			
603	D445	21.12		0.28	
657	D445	21.13		0.48	
704	D445	21.103		-0.07	
781	D445	21.10		-0.13	
875	D445	21.07		-0.74	
902	D445	21.01		-1.97	
922	D445	21.304		4.03	
963	D445	20.76	R(0.05)	-7.07	
966		-----			
1011	D445	21.045		-1.25	
1013		-----			
1026	D445	21.10		-0.13	
1161	ISO3104	21.52	C,R(0.05)	8.44	First reported: 20.81
1201	D445	21.06		-0.95	
1231	D445	21.08		-0.54	
1243	D445	21.19		1.70	
1349	D445	21.1308		0.50	
1394	D445	21.065		-0.85	
1398	D445	21.073		-0.68	
1423	D445	21.28		3.54	
1461	ISO3104	21.2041		1.99	
1543	D445	21.36130	R(0.05)	5.20	
1564	D445	21.08		-0.54	
1706	ISO3104	21.14		0.68	
1793		-----			
1833	D445	21.09		-0.34	
1870	D445	20.9882		-2.41	
1877	D445	21.10		-0.13	
1915	D445	21.21		2.11	

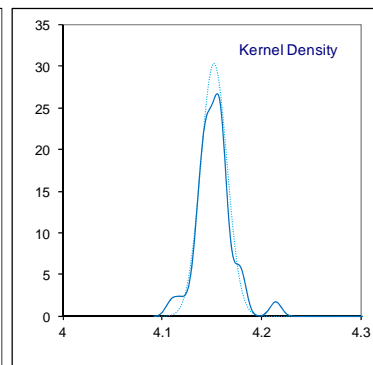
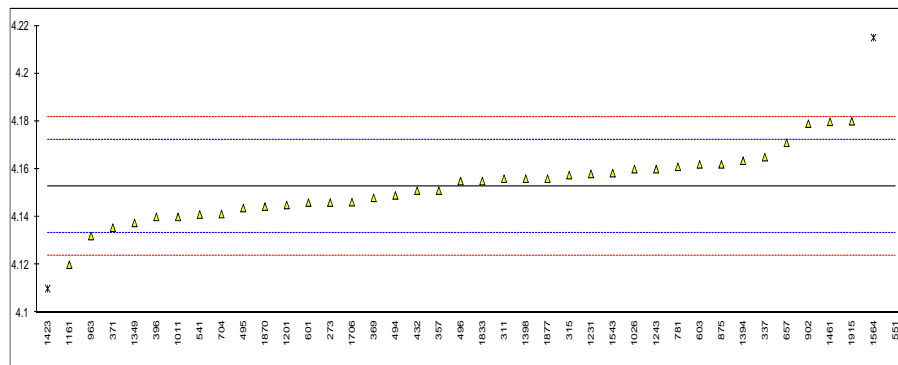
normality suspect  
n 36  
outliers 4  
mean (n) 21.106  
st.dev. (n) 0.0698  
R(calc.) 0.195  
R(D445:12) 0.137



Determination of Kinematic Viscosity @ 100°C on sample #14055; results in mm<sup>2</sup>/s

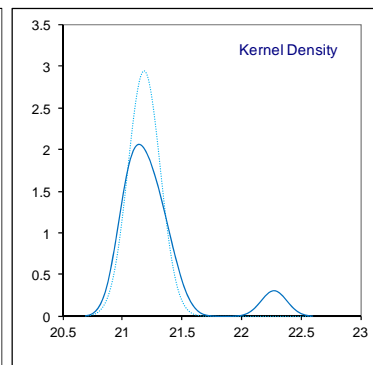
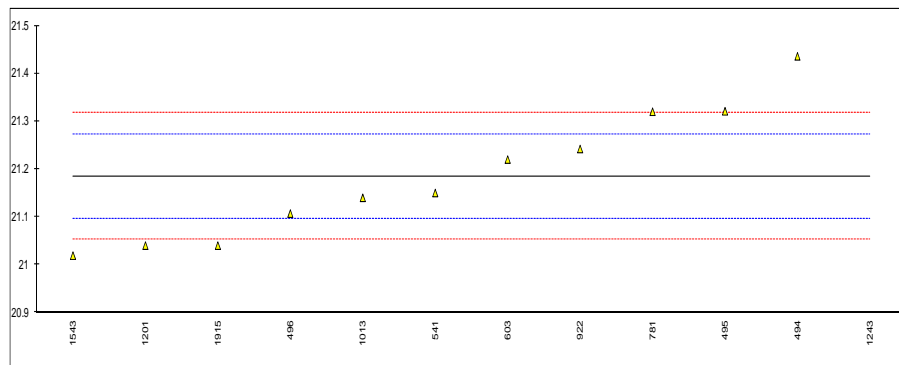
lab	method	value	mark	z(targ)	remarks
273	D445	4.146		-0.70	
311	D445	4.156		0.34	
315	D445	4.1575		0.49	
337	D445	4.165		1.27	
357	D445	4.151		-0.18	
369	D445	4.148		-0.49	
371	D445	4.1355		-1.79	
396	D445	4.140		-1.32	
432	D445	4.1510		-0.18	
485		----		----	
494	D445	4.149		-0.39	
495	D445	4.1437		-0.94	
496	D445	4.1550		0.23	
541	D445	4.141		-1.22	
551	D445	4.62117	R(0.01)	48.59	
601	D445	4.146		-0.70	
603	D445	4.162		0.96	
657	D445	4.171		1.89	
704	D445	4.1412		-1.20	
781	D445	4.161		0.85	
875	D445	4.162		0.96	
902	D445	4.179		2.72	
922		----		----	
963	D445	4.132		-2.15	
966		----		----	
1011	D445	4.140		-1.32	
1013		----		----	
1026	D445	4.16		0.75	
1161	ISO3104	4.12		-3.40	
1201	D445	4.145		-0.81	
1231	D445	4.158		0.54	
1243	D445	4.16		0.75	
1349	D445	4.1375		-1.58	
1394	D445	4.1635		1.11	
1398	D445	4.1560		0.34	
1423	D445	4.11	C,R(0.01)	-4.44	First reported: 4.1
1461	ISO3104	4.1798		2.80	
1543	D445	4.158327		0.58	
1564	D445	4.215	R(0.01)	6.46	
1706	ISO3104	4.1462		-0.68	
1793		----		----	
1833	D445	4.155		0.23	
1870	D445	4.1443		-0.88	
1877	D445	4.156		0.34	
1915	D445	4.18		2.82	

normality OK  
n 37  
outliers 3  
mean (n) 4.1528  
st.dev. (n) 0.01318  
R(calc.) 0.0369  
R(D445:12) 0.0270



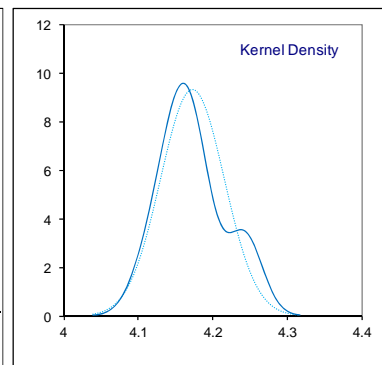
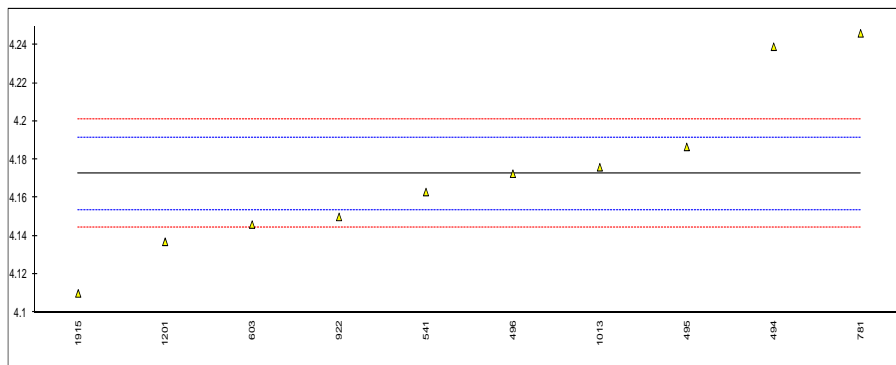
Determination of Viscosity Stabinger @ 40 °C on sample #14055; results in mm<sup>2</sup>/s

lab	method	value	mark	z(targ)	remarks
273		----		----	
311		----		----	
315		----		----	
337		----		----	
357		----		----	
369		----		----	
371		----		----	
396		----		----	
432		----		----	
485		----		----	
494	D7042	21.436		5.68	
495	D7042	21.321		3.08	
496	D7042	21.107		-1.77	
541	D7042	21.15		-0.79	
551		----		----	
601		----		----	
603	D7042	21.22		0.79	
657		----		----	
704		----		----	
781	D7042	21.32		3.06	
875		----		----	
902		----		----	
922	D7042	21.242		1.29	
963		----		----	
966		----		----	
1011		----		----	
1013	D7042	21.14		-1.02	
1026		----		----	
1161		----		----	
1201	D7042	21.04		-3.28	
1231		----		----	
1243	D7042	22.275	G(0.01)	24.67	
1349		----		----	
1394		----		----	
1398		----		----	
1423		----		----	
1461		----		----	
1543	D7042	21.019		-3.76	
1564		----		----	
1706		----		----	
1793		----		----	
1833		----		----	
1870		----		----	
1877		----		----	
1915	D7042	21.04		-3.28	
normality		OK			
n		11			
outliers		1			
mean (n)		21.185			
st.dev. (n)		0.1353			
R(calc.)		0.379			
R(D7042:12a)		0.124			



Determination of Viscosity Stabinger @ 100 °C on sample #14055; results in mm<sup>2</sup>/s

lab	method	value	mark	z(targ)	remarks
273		----		----	
311		----		----	
315		----		----	
337		----		----	
357		----		----	
369		----		----	
371		----		----	
396		----		----	
432		----		----	
485		----		----	
494	D7042	4.239		7.01	
495	D7042	4.1866		1.48	
496	D7042	4.1726		0.00	
541	D7042	4.163		-1.02	
551		----		----	
601		----		----	
603	D7042	4.146		-2.81	
657		----		----	
704		----		----	
781	D7042	4.246		7.75	
875		----		----	
902		----		----	
922	D7042	4.150		-2.39	
963		----		----	
966		----		----	
1011		----		----	
1013	D7042	4.176		0.36	
1026		----		----	
1161		----		----	
1201	D7042	4.137		-3.76	
1231		----		----	
1243		----		----	
1349		----		----	
1394		----		----	
1398		----		----	
1423		----		----	
1461		----		----	
1543		----		----	
1564		----		----	
1706		----		----	
1793		----		----	
1833		----		----	
1870		----		----	
1877		----		----	
1915	D7042	4.11		-6.62	
normality		OK			
n		10			
outliers		0			
mean (n)		4.1726			
st.dev. (n)		0.04282			
R(calc.)		0.1199			
R(D7042:12a)		0.0265			

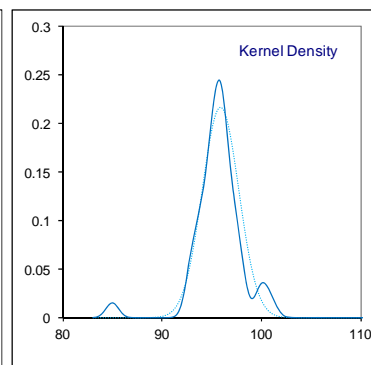
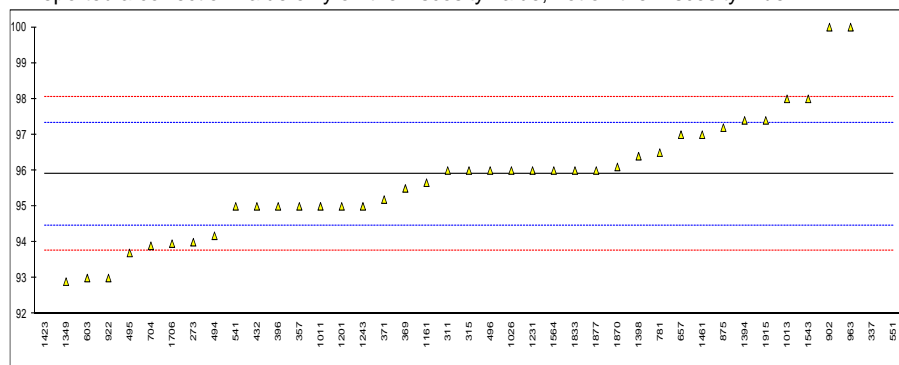


Determination of Viscosity index on sample #14055

lab	method	value	mark	z(targ)	calc. iis	mark	remarks
273	D2270	94		-2.66	93.77		
311	D2270	96		0.14	96.08		
315	D2270	96		0.14	96.20		
337	D2270	101	C	7.14	100.80	ex	First reported: 96, outlier in D445 40°C
357	D2270	95		-1.26	95.08		
369	D2270	95.5		-0.56	95.47		
371	D2270	95.19		-0.99	95.19		
396	D2270	95		-1.26	94.85		
432	D2270	95.0		-1.26	95.01		
485		----		----	----		
494	D2270	94.18		-2.40	94.24		
495	D2270	93.7		-3.08	93.71		
496	D2270	96.0		0.14	95.94		
541	D2270	95		-1.26	94.83		
551	D2270	136	R(0.01)	56.14	139.20	ex	Outlier in D445 100°C, iis calc. different VI
601		----		----	----		
603	D2270	93		-4.06	92.66	ex	VI based on D7042, iis calc 96.41 from D445 visc.
657	D2270	97		1.54	97.45		
704	D2270	93.9		-2.80	93.86		
781	D2270	96.5		0.84	96.59		
875	D2270	97.196		1.82	97.19		
902	D2270	100		5.74	100.30	R(0.05)	
922	D2270	93.0		-4.06	92.87	ex	VI based on D7042.
963	D2270	100		5.74	98.06	ex	Outlier in D445 40°C, iis calc. different VI
966		----		----	----		
1011	D2270	95		-1.26	94.61		
1013	D2270	98		2.94	97.96	ex	VI based on D7042.
1026	D2270	96		0.14	96.46		
1161	D2270	95.66		-0.33	84.19	ex	Outlier in D445 40°C, iis calc. different VI*
1201	D2270	95		-1.26	95.06		
1231	D2270	96		0.14	96.50		
1243	D2270	95		-1.26	95.04		
1349	D2270	92.9		-4.20	92.91		
1394	D2270	97.4		2.10	97.47		
1398	D2270	96.4		0.70	96.34		
1423	D2270	85	R(0.01)	-15.26	86.61	ex	Outlier in D445 100°C, iis calc. different VI*
1461	ISO2909	97		1.54	97.46		
1543	D2270	98		2.94	92.13	ex	Outlier in D445 40°C, iis calc. different VI
1564	D2270	96		0.14	102.70	ex	Outlier in D445 100°C, iis calc. different VI
1706	ISO2909	93.96		-2.71	93.96		
1793		----		----	----		
1833	D2270	96		0.14	95.94		
1870	D2270	96.10	C	0.28	96.10		
1877	D2270	96		0.14	95.92		
1915	D2270	97.4		2.10	97.39		

		<u>VI based on D445 valid data</u>
normality	suspect	OK
n	39	30
outliers	2	1 (+10 ex)
mean (n)	95.90	95.55
st.dev. (n)	1.844	1.254
R(calc.)	5.16	3.51
R(D2270:10e1)	2.00	2.00

\* Reported a correction value only on the viscosity value, not on the Viscosity Index



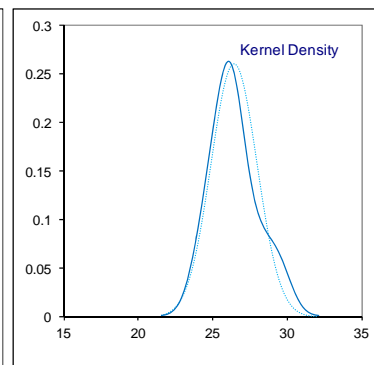
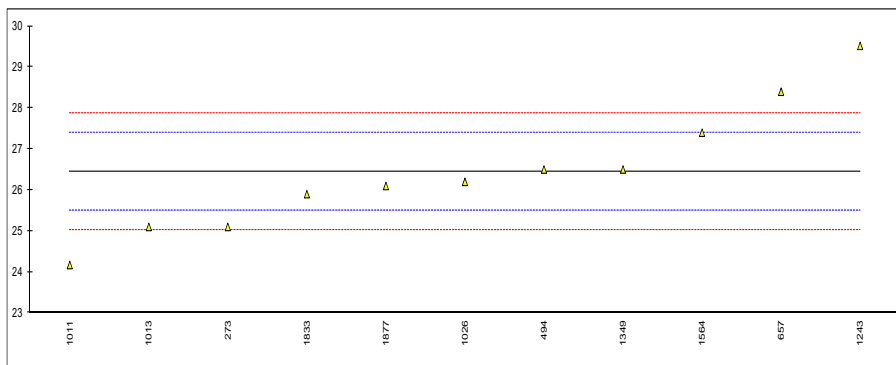


Determination of Evaporation loss by Noack test on sample #14055; results in %M/M

lab	method	value	mark	z(targ)	remarks
273	D5800-B	25.1		-2.82	
311		----		----	
315		----		----	
337		----		----	
357		----		----	
369		----		----	
371		----		----	
396		----		----	
432		----		----	
485		----		----	
494	D5800-B	26.5		0.12	
495		----		----	
496		----		----	
541		----		----	
551		----		----	
601		----		----	
603		----		----	
657	D5800-B	28.4		4.10	
704		----		----	
781		----		----	
875		----		----	
902		----		----	
922		----		----	
963		----		----	
966		----		----	
1011	D5800-B	24.17		-4.76	
1013	D5800-B	25.1		-2.82	
1026	CEC L-40-93-B	26.2	C	-0.51	Reported result: 25.4 (CEC), equivalent to (25.4/0.970 =) 26.2 in D5800B
1161		----		----	
1201		----		----	
1231		----		----	
1243	D5800	29.52		6.44	
1349	D5800-B	26.5		0.12	
1394		----		----	
1398		----		----	
1423		----		----	
1461		----		----	
1543		----		----	
1564	DIN51581	27.4	C	2.00	Reported result: 28.5 (DIN), equivalent to (28.5*0.962 =) 27.4 in D5800B
1706		----		----	
1793		----		----	
1833	D5800B	25.9		-1.14	
1870		----		----	
1877	CEC L-40-93-B	26.1	C	-0.72	Reported result: 25.3 (CEC), equivalent to (25.3/0.970 =) 26.1 in D5800B
1915		----		----	

normality OK  
n 11  
outliers 0  
mean (n) 26.444  
st.dev. (n) 1.5318  
R(calc.) 4.289  
R(D5800B:10) 1.337

Compare R(CEC L-040-93:11): 1.08 (range 6-20%)  
Compare R(ASTM D5800A:10): 4.84

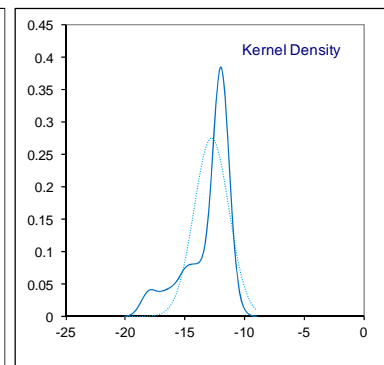
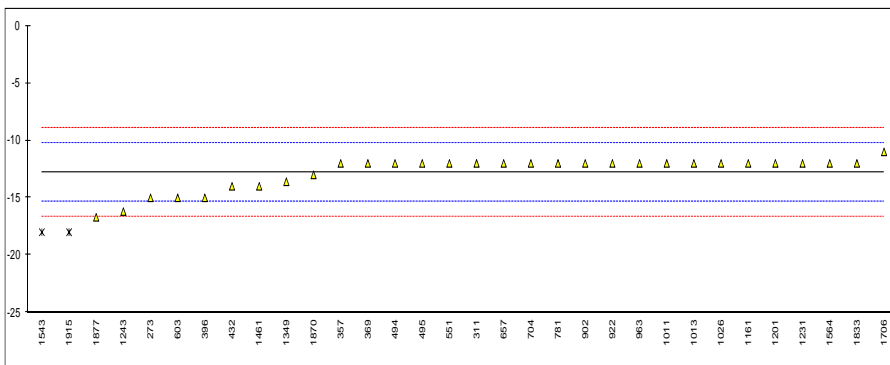


Determination of Pour Point on sample #14055; results in °C

lab	method	value	mark	z(target)	remarks
273	D6892	-15		-1.72	
311	D5950	-12		0.61	
315		----		----	
337		----		----	
357	D5950	-12		0.61	
369	D97	-12		0.61	
371		----		----	
396	D97	-15		-1.72	
432	D97	-14		-0.95	
485		----		----	
494	D6892	-12		0.61	
495	D6892	-12		0.61	
496		----		----	
541		----		----	
551	D97	-12		0.61	
601		----		----	
603	D97	-15		-1.72	
657	D5950	-12		0.61	
704	D97	-12		0.61	
781	D97	-12		0.61	
875		----		----	
902	D6892	-12		0.61	
922	D97	-12		0.61	
963	D97	-12		0.61	
966		----		----	
1011	D6892	-12		0.61	
1013	D6892	-12		0.61	
1026	D97	-12		0.61	
1161	ISO3016	-12		0.61	
1201	D6892	-12		0.61	
1231	D5950	-12		0.61	
1243	D6892	-16.2		-2.66	
1349	D6892	-13.6		-0.64	
1394		----		----	
1398		----		----	
1423		----		----	
1461	ISO3016	-14		-0.95	
1543	D97	-18	R(0.05)	-4.06	
1564	D5950	-12		0.61	
1706	ISO3016	-11.0		1.39	
1793		----		----	
1833	D5950	-12		0.61	
1870	D97	-13		-0.17	
1877	D7346	-16.7		-3.05	
1915	D97	-18	R(0.05)	-4.06	

normality suspect  
n 30  
outliers 2  
mean (n) -12.78  
st.dev. (n) 1.449  
R(calc.) 4.06  
R(D6892:03) 3.60

Compare R(D97:12): 8.0 or R(D5950:14): 6.1



Determination of Rust prevention (proc.B) on sample #14055

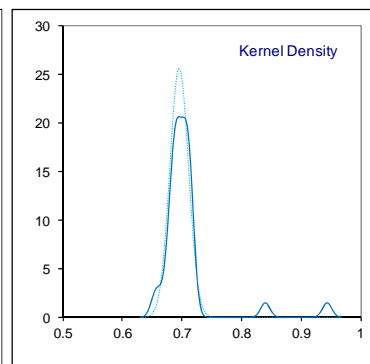
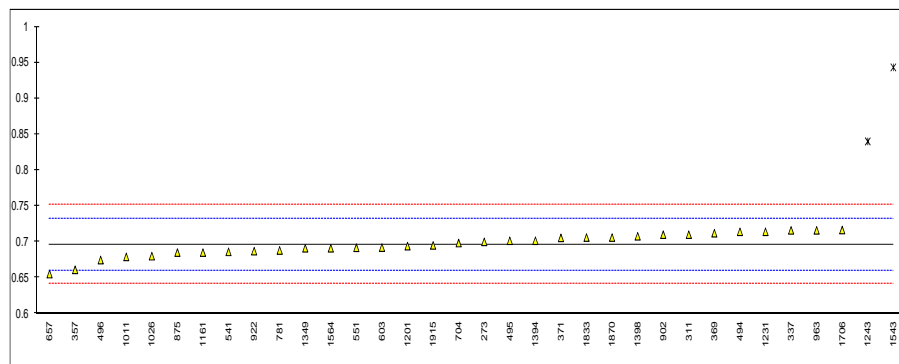
lab	method	value	mark	z(targ)	remarks
273		----		----	
311		----		----	
315	D665	Fail		----	
337		----		----	
357		----		----	
369		----		----	
371		----		----	
396		----		----	
432		----		----	
485		----		----	
494		----		----	
495		----		----	
496		----		----	
541		----		----	
551		----		----	
601		----		----	
603		----		----	
657		----		----	
704		----		----	
781		----		----	
875		----		----	
902		----		----	
922		----		----	
963	D665	Pass		----	
966		----		----	
1011		----		----	
1013	D665	Fail		----	
1026		----		----	
1161		----		----	
1201		----		----	
1231		----		----	
1243		----		----	
1349		----		----	
1394		----		----	
1398		----		----	
1423		----		----	
1461		----		----	
1543		----		----	
1564		----		----	
1706		----		----	
1793		----		----	
1833		----		----	
1870		----		----	
1877		----		----	
1915	D665	Fail		----	
	normality	n.a.			
	n	4			
	outliers	n.a.			
	mean (n)	n.a.			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D665:12)	n.a.			

Determination of Sulphur on sample #14055; results in %M/M

lab	method	value	mark	z(targ)	Remarks
273	D4294	0.70	C	0.22	First reported: 0.52
311	D2622	0.710		0.77	
315		-----			
337	D2622	0.716		1.09	
357	D4294	0.661		-1.91	
369	D2622	0.712		0.88	
371	D4294	0.7055		0.52	
396		-----			
432		-----			
485		-----			
494	ISO8754	0.714		0.98	
495	D2622	0.7015		0.30	
496	D2622	0.6746		-1.16	
541	D4294	0.686		-0.54	
551	D4294	0.69176		-0.23	
601		-----			
603	D4294	0.692		-0.22	
657	D5453	0.655	C	-2.23	First reported: 0.529
704	D4294	0.6983		0.13	
781	D4294	0.688		-0.43	
875	D2622	0.685		-0.60	
902	D2622	0.71		0.77	
922	D4294	0.687		-0.49	
963	D4294	0.716		1.09	
966		-----			
1011	IP336	0.679		-0.92	
1013		-----			
1026	D2622	0.68		-0.87	
1161	ISO8754	0.685		-0.60	
1201	ISO8754	0.694		-0.11	
1231	D4294	0.714		0.98	
1243	D2622	0.84	R(0.01)	7.85	
1349	IP336	0.691		-0.27	
1394	D4294	0.7015		0.30	
1398	D2622	0.7077		0.64	
1423		-----			
1461		-----			
1543	D4294	0.9433	R(0.01)	13.49	
1564	D5453	0.691		-0.27	
1706	in house	0.7165		1.12	
1793		-----			
1833	IP336	0.7060		0.55	
1870	ISO8754	0.706		0.55	
1877		-----			
1915	D4294	0.695	C	-0.05	First reported: 0.47

normality OK  
n 32  
outliers 2  
mean (n) 0.6959  
st.dev. (n) 0.01556  
R(calc.) 0.0436  
R(D2622:10) 0.0514

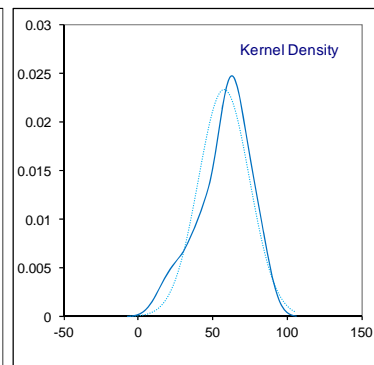
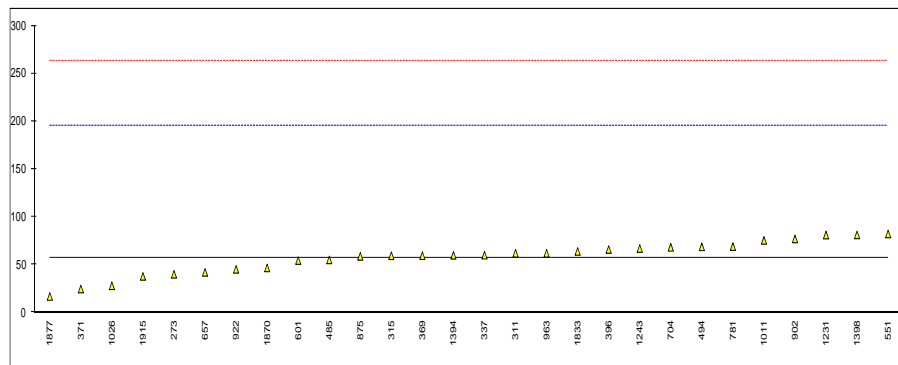
Compare R(D4294:10): 0.0575



Determination of Water on sample #14055; results in mg/kg

lab	method	value	mark	z(targ)	remarks
273	D6304	40		-0.26	
311	D6304	62		0.06	
315	D6304	59.45		0.03	
337	D6304	60		0.04	
357		----		----	
369	D6304	59.5		0.03	
371	D6304	24.6		-0.48	
396	D6304	65.9		0.12	
432		----		----	
485	D6304	55		-0.04	
494	D6304	68.7		0.16	
495	D6304	<30		----	
496	D6304	<10		----	
541		----		----	
551	D6304	82.2		0.36	
601	D6304	54.4		-0.05	
603		----		----	
657	D6304	42		-0.23	
704	D6304	68.2		0.15	
781	D6304	69		0.17	
875	D6304	58.797		0.02	
902	D6304	77		0.28	
922	D6304	45.2		-0.18	
963	D6304	62		0.06	
966		----		----	
1011	D6304	75.5		0.26	
1013		----		----	
1026	D6304	28		-0.43	
1161		----		----	
1201	D6304	<30		----	
1231	D6304	81.05		0.34	
1243	D6304	67		0.14	
1349		----		----	
1394	D6304	59.9		0.03	
1398	D6304	81.15		0.34	
1423		----		----	
1461		----		----	
1543		----		----	
1564		----		----	
1706		----		----	
1793		----		----	
1833	D6304	63.9209		0.09	
1870	D6304	46.63		-0.16	
1877	D6304	16.8		-0.59	
1915	D1533	37.8		-0.29	

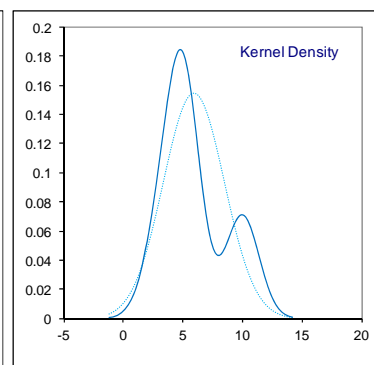
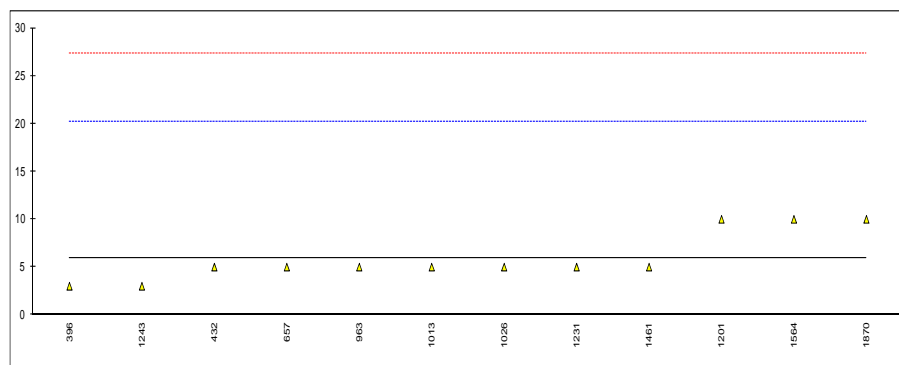
normality OK  
n 28  
outliers 0  
mean (n) 57.56  
st.dev. (n) 17.098  
R(calc.) 47.88  
R(D6304:07) 192.20



Determination of Water Separability @ 54°C on sample #14055; results in minutes

lab	method	ml oil-water-emulsion	time	mark	z(targ)	remarks
273		----	----		----	
311		----	----		----	
315		----	----		----	
337		----	----		----	
357		----	----		----	
369		----	----		----	
371		----	----		----	
396	D1401	40-40-0	3		-0.41	
432	D1401	40-40-0	5		-0.13	
485		----	----		----	
494	D1401	40-39-0	----		----	
495		----	----		----	
496		----	----		----	
541		----	----		----	
551		----	----		----	
601		----	----		----	
603		----	----		----	
657	D1401	40-40-0	5		-0.13	
704		----	----		----	
781		----	----		----	
875		----	----		----	
902		----	----		----	
922		----	----		----	
963	D1401	40-40-0	5		-0.13	
966		----	----		----	
1011		----	----		----	
1013	D1401	40-40-0	5		-0.13	
1026	D1401	40-40-0	5		-0.13	
1161		----	----		----	
1201	D1401	38-39.5-2.5	10		0.57	
1231	D1401	40-40-0	5		-0.13	
1243	D1401	40-40-0	3		-0.41	
1349		----	----		----	
1394		----	----		----	
1398		----	----		----	
1423		----	----		----	
1461	ISO6614	40-40-0	5		-0.13	
1543	D1401	40-40-0	----		----	
1564	D1401	40-40-0	10		0.57	
1706		----	----		----	
1793		----	----		----	
1833		----	----		----	
1870	D1401	40/40/0	10		0.57	
1877		----	----		----	
1915		----	----		----	

normality OK  
n 12  
outliers 0  
mean (n) 5.9  
st.dev. (n) 2.57  
R(calc.) 7.2  
R(D1401:12) 20



## APPENDIX 2

### Number of participants per country

1 lab in ARGENTINA  
2 labs in AUSTRIA  
1 lab in BELGIUM  
1 lab in BRAZIL  
1 lab in BULGARIA  
1 lab in FINLAND  
1 lab in FRANCE  
4 labs in GERMANY  
1 lab in ITALY  
2 labs in LATVIA  
2 labs in MALAYSIA  
4 labs in NETHERLANDS  
1 lab in NIGERIA  
1 lab in PAKISTAN  
1 lab in POLAND  
2 labs in PORTUGAL  
5 labs in RUSSIAN FEDERATION  
3 labs in SAUDI ARABIA  
1 lab in SINGAPORE  
1 lab in SOUTH AFRICA  
2 labs in SPAIN  
1 lab in SUDAN  
1 lab in THAILAND  
3 labs in TURKEY  
1 lab in UKRAINE  
1 lab in UNITED KINGDOM

## 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

### Literature:

- 1 iis Interlaboratory Studies, Protocol for the Organization, Statistics and Evaluation, April 2014
- 2 ASTM E178:08
- 3 ISO 5725-86
- 4 ISO 5725, parts 1-6, 1994
- 5 ISO13528:05
- 6 ISO17043:2010
- 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, First reported Z. Anal. Chem, 331, 513, (1988)
- 12 J.N. Miller, Analyst, 118, 455, (1993)
- 13 Analytical Methods Committee Technical brief, No4 January 2001.
- 14 The Royal Society of Chemistry 2002, Analyst 2002, 127 pages 1359-1364, P.J. Lowthian and M. Thompson (see <http://www.rsc.org/suppdata/an/b2/b205600n/>).
- 15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), pp. 165-172, (1983)