



Institute for  
Interlaboratory Studies

# Results of Proficiency Test Bitumen November 2023

**Organized by:** Institute for Interlaboratory Studies  
Spijkenisse, the Netherlands

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## 1 INTRODUCTION

Since 2014 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Bitumen in accordance with the latest version of EN12591 every year. During the annual proficiency testing program of 2023 it was decided to continue the round robin for the analysis of Bitumen.

In this interlaboratory study 48 laboratories in 30 countries registered for participation, see appendix 2 for the number of participants per country. In this report the results of the Bitumen proficiency test are presented and discussed. This report is also electronically available through the iis website [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 (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to a laboratory that has performed the tests in accordance with for ISO/IEC17043 relevant requirements of ISO/IEC17025.

It was decided to send one sample of Bitumen grade 35/50 in a 2.5 liter can labelled #23255. The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

### 2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

### 2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website [www.iisnl.com](http://www.iisnl.com), from the FAQ page.

### 2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

## 2.4 SAMPLES

A batch of 60 subsamples of Bitumen grade 35/50 in 2.5 L metal cans was obtained from a local supplier and labelled #23255.

The homogeneity of the subsamples was checked by determination of Penetration at 25 °C in accordance with EN1426 on 5 stratified randomly selected subsamples.

	Penetration in 0.1 mm
sample #23255-1	39
sample #23255-2	38
sample #23255-3	39
sample #23255-4	39
sample #23255-5	39

Table 1: homogeneity test results of subsamples #23255

From the above test results the relative standard deviation (RSD) was calculated and compared with 0.3 times the average relative standard deviation obtained from three iis PTs with grade 35/50 Bitumen conducted from 2018 - 2022 in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Penetration
RSD% (observed)	1.2
reference method	iis PTs
0.3 x RSD% (reference method)	1.8

Table 2: evaluation of the relative standard deviation of subsamples #23255

The calculated relative standard deviation is in agreement with 0.3 times the average relative standard deviation obtained from the previous iis PTs. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one 2.5 L can of sample #23255 was sent on November 8, 2023. An SDS was added to the sample package.

## 2.5 STABILITY OF THE SAMPLES

The stability of Bitumen in the metal cans was checked. The material has been found sufficiently stable for the period of the proficiency test.

## 2.6 ANALYZES

The participants were requested to determine: Density at 25 °C, Dynamic Viscosity at 60 °C, Flash Point C.O.C., Fraass Breaking Point, Kinematic Viscosity at 135 °C, Penetration at 25 °C, Penetration Index, RTFOT at 163 °C (Change of Mass, Retained Penetration, Viscosity Ratio and Increase in Softening Point), Softening Point (Ring and Ball), Solubility in Xylene and Ductility.

It was explicitly requested to treat the sample as if it was a routine sample and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal [www.kpmd.co.uk/sgs-iis/](http://www.kpmd.co.uk/sgs-iis/). The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website [www.iisnl.com](http://www.iisnl.com).

### 3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal [www.kpmd.co.uk/sgs-iis/](http://www.kpmd.co.uk/sgs-iis/). The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

#### 3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<... ' or '>... ' were not used in the statistical evaluation.

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

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. Outliers are marked by  $D(0.01)$  for the Dixon's test, by  $G(0.01)$  or  $DG(0.01)$  for the Grubbs' test and by  $R(0.01)$  for the Rosner's test. Stragglers are marked by  $D(0.05)$  for the Dixon's test, by  $G(0.05)$  or  $DG(0.05)$  for the Grubbs' test and by  $R(0.05)$  for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

### 3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

### 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 (derived from e.g. ISO or ASTM test methods), the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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

The z-scores were calculated according to:

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

The  $z_{(\text{target})}$  scores are listed in the test 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 proficiency test some problems were encountered with the dispatch of the samples. Two participants reported test results after the final reporting date and three other participants did not report any test results. Not all participants were able to report all tests requested.

In total 45 participants reported 307 numerical test results. Observed were 14 outlying test results, which is 4.6%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

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

### 4.1 EVALUATION PER TEST

In this section the reported test results are discussed per test. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 3.

In the iis PT reports ASTM test methods are referred to with a number (e.g. D36/D36M) and an added designation for the year that the test method was adopted or revised (e.g. D36/D36M:14).

When a method has been reapproved an "R" will be added and the year of approval (e.g. D36/D36M:14R20).

Density at 25 °C: The group of participants met the target requirements. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN15326:07+A1:09.

Dynamic Viscosity at 60 °C: The group of participants had difficulty to meet the target requirements. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of EN12596:23.

Flash Point C.O.C.: The group of participants had difficulty to meet the target requirements. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ISO2592:17 nor ASTM D92:18.

Fraass Breaking Point: The group of participants met the target requirements. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN12593:15.

Kinematic Viscosity at 135 °C: The group of participants met the target requirements. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN12595:23 or ASTM D2170:22.

Penetration at 25 °C: The group of participants may have had difficulty to meet the target requirements depending on the procedure used. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of EN1426:15 but is in agreement with the requirements of ASTM D5:20. When evaluated separately the calculated reproducibility after rejection of the statistical outlier for EN1426 is still not in agreement with the requirements of EN1426:15 but the calculated reproducibility after rejection of the statistical outlier for ASTM D5 is in agreement with ASTM D5:20. Different factors could cause this large variation, such as preparation, temperature and needle. During the measurement, the temperature should be kept at 25 °C, by immersing the sample in enough water of this temperature. For measurements outside of the waterbath, a transfer dish of 350 ml should be used. Deviations from this temperature will have influence on the penetration. Another factor is the tip of the needle used. This tip should keep the same dimensions/surface through out testing in time. In practice, it will get abrasion and wear and should be replaced regularly.



Penetration Index: The group of participants had difficulty to meet the target requirements. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of EN12591:09.

RTFOT at 163 °C - Change of Mass: The group of participants met the target requirements. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN12607-1:14.

RTFOT at 163 °C - Retained Penetration: The group of participants had difficulty to meet the target requirements. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of EN12607-1:14.

RTFOT at 163 °C - Viscosity Ratio: The group of participants had difficulty to meet the target requirements. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of EN12607-1:14.

RTFOT at 163 °C on Increase in Softening Point: The group of participants had difficulty to meet the target requirements. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of EN12607-1:14.

Softening Point (Ring and Ball): The group of participants met the target requirements. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN1427:15 and ASTM D36/D36M:14R20.

Solubility in Xylene: The group of participants had difficulty to meet the target requirements. No statistical outliers were observed. It was decided not to calculate z-scores due to the large difference between the calculated and reference reproducibility.

Ductility: The reporting participants agreed on a test result >100 cm.

#### 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Parameter	unit	n	average	2.8 * sd	R(lit)
Density at 25 °C	kg/m <sup>3</sup>	21	1041.5	4.1	5
Dynamic Viscosity at 60 °C	Pa.s	13	1019.8	148.1	102.0
Flash Point C.O.C.	°C	21	318	37	18
Fraass Breaking Point	°C	15	-10.0	6.6	6

Parameter	unit	n	average	2.8 * sd	R(lit)
Kinematic Viscosity at 135 °C	mm <sup>2</sup> /s	17	959.9	85.7	86.4
Penetration at 25 °C	0.1 mm	43	38.9	4.8	3
Penetration Index		18	-0.30	0.66	0.5
RTFOT - Change of Mass	%	25	-0.16	0.12	0.2
RTFOT - Retained Penetration	%	25	65.2	15.2	10
RTFOT - Viscosity Ratio		8	3.8	1.9	0.8
RTFOT - Increase in Soft. Point	°C	23	7.6	5.0	4
Softening Point (Ring & Ball)	°C	42	56.4	1.9	2
Solubility in Xylene	%M/M	16	99.57	0.66	(0.15)
Ductility	cm	11	>100	n.a.	n.a.

Table 3: reproducibilities of tests on sample #23255

For results between brackets no z-scores are calculated.

Without further statistical calculations it can be concluded that for many tests there is not a good compliance of the group of participants with the reference test methods. The problematic tests have been discussed in paragraph 4.1.

#### 4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2023 WITH PREVIOUS PTS

	November 2023	November 2022	December 2021	December 2020	December 2019
Number of reporting laboratories	45	45	51	50	45
Number of test results	307	318	348	315	310
Number of statistical outliers	14	15	11	14	11
Percentage of statistical outliers	4.6%	4.7%	3.2%	4.4%	3.5%

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 reference test methods. The conclusions are given in the following table.

Parameter	November 2023	November 2022	December 2021	December 2020	December 2019
Paving Grade	35/50	35/50	70/100	35/50	70/100
Density at 25 °C	+	+/-	+/-	-	-
Dynamic Viscosity at 60 °C	-	+/-	-	--	-
Flash Point C.O.C.	--	-	--	(--)	-
Fraass Breaking Point	+/-	-	-	-	+/-
Kinematic Viscosity at 135 °C	+/-	-	-	--	-
Penetration at 25 °C	-	-	-	--	-
Penetration Index	-	+	+/-	+/-	-
RTFOT - Change of Mass	+	+/-	-	++	--

Parameter	November 2023	November 2022	December 2021	December 2020	December 2019
RTFOT - Retained Penetration	-	+/-	+/-	-	+/-
RTFOT - Viscosity Ratio	--	-	+/-	n.e.	(--)
RTFOT - Increase in Soft. Point	-	+	+	+/-	-
Softening Point (Ring and Ball)	+/-	+	+/-	-	+/-
Solubility in Xylene	(--)	+/-	(--)	(--)	(--)

Table 5: comparison of determinations to the reference test methods

For results between brackets no z-scores are calculated.

The following performance categories were used:

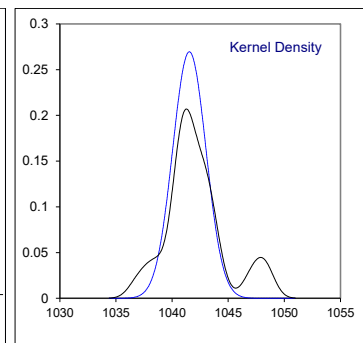
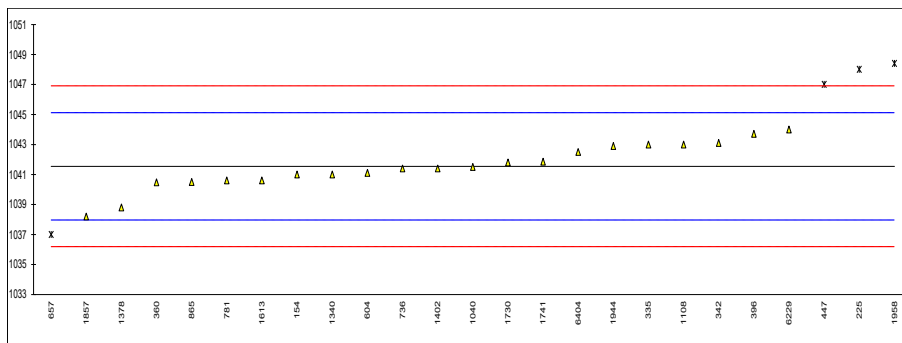
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance equals the reference test method
- : group performed worse than the reference test method
- : group performed much worse than the reference test method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

**APPENDIX 1**

**Determination of Density at 25 °C on sample #23255; results in kg/m<sup>3</sup>**

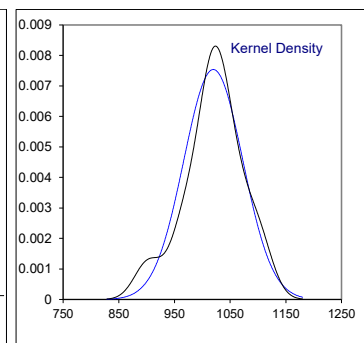
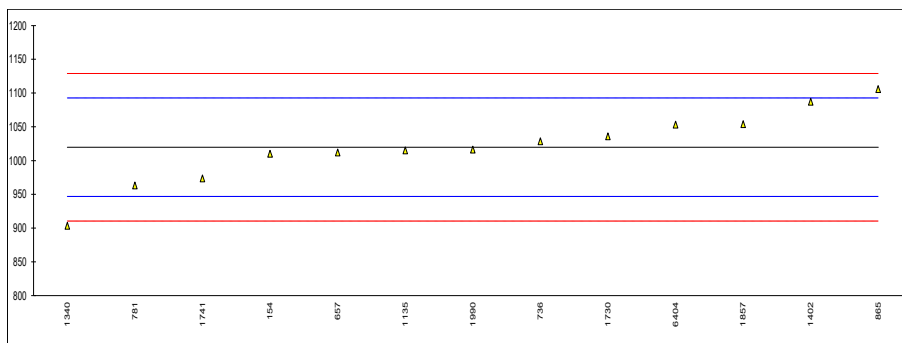
lab	method	value	mark	z(targ)	remarks
154	D70	1041		-0.30	
225	D70	1048	R(0.05)	3.62	
328		----		----	
333		----		----	
335	EN15326	1043	C	0.82	first reported 1.043 kg/m <sup>3</sup>
342	D70	1043.1	C	0.87	first reported 1060
360	EN15326	1040.47		-0.60	
365		----		----	
396	D70	1043.7		1.21	
398		----		----	
399		----		----	
444		----		----	
447	D70	1047	R(0.05)	3.06	
604	D70	1041.1		-0.25	
657	D70	1037	R(0.05)	-2.54	
736	EN15326	1041.4		-0.08	
781	EN15326	1040.6		-0.53	
865	D70/D70M	1040.5	C	-0.58	reported 1.0405 kg/m <sup>3</sup>
1011		----		----	
1026		----		----	
1040	ISO12185	1041.5		-0.02	
1108	EN15326	1043.0	C	0.82	first reported 1035.8
1135		----		----	
1320		----		----	
1340	EN15326	1041		-0.30	
1378	EN15326	1038.8		-1.54	
1399		----		----	
1402	ISO3838	1041.4		-0.08	
1613	DIN51757	1040.6		-0.53	
1631		----		----	
1724		----		----	
1730	EN15326	1041.8	C	0.14	first reported 1.0418 kg/m <sup>3</sup>
1741	EN15326	1041.86		0.18	
1833		----		----	
1849		----		----	
1852		----		----	
1857	D70	1038.2		-1.87	
1881		----		----	
1944	EN15326	1042.9		0.76	
1958	D70	1048.4	C,R(0.05)	3.84	reported 1.0484 kg/m <sup>3</sup>
1990		----	W	----	test result withdrawn, reported 1.0341 kg/m <sup>3</sup>
6228		----		----	
6229	EN15326	1044		1.38	
6404	EN15326	1042.5	C	0.54	first reported 1.0425 kg/m <sup>3</sup>
6419		----		----	
6420		----		----	
6474		----		----	
6561		----		----	

normality OK  
n 21  
outliers 4  
mean (n) 1041.544  
st.dev. (n) 1.4804  
R(calc.) 4.145  
st.dev.(EN15326:07+A1:09) 1.7857  
R(EN15326:07+A1:09) 5



Determination of Dynamic Viscosity at 60 °C on sample #23255; results in Pa.s

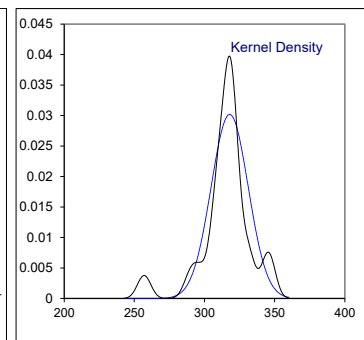
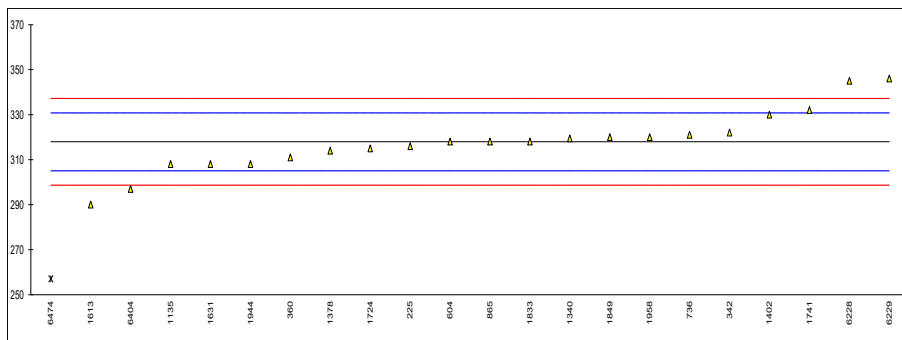
lab	method	value	mark	z(targ)	remarks
154	D2171	1010		-0.27	
225		----		----	
328		----		----	
333		----		----	
335		----		----	
342		----		----	
360		----		----	
365		----		----	
396		----		----	
398		----		----	
399		----		----	
444		----		----	
447		----		----	
604		----		----	
657	D2171	1012		-0.21	
736	EN12596	1028.5		0.24	
781	EN12596	963.29		-1.55	
865	D2171/D2171M	1105.8		2.36	
1011		----		----	
1026		----		----	
1040		----		----	
1108		----		----	
1135	EN12596	1015		-0.13	
1320		----		----	
1340	EN12596	903.38		-3.20	
1378		----		----	
1399		----		----	
1402	EN12596	1087		1.84	
1613		----		----	
1631		----		----	
1724		----		----	
1730	EN12596	1036		0.44	
1741	EN12596	973.6		-1.27	
1833		----		----	
1849		----		----	
1852		----		----	
1857	D2171	1053.9		0.94	
1881		----		----	
1944		----		----	
1958		----		----	
1990	D2171	1016.23		-0.10	
6228		----		----	
6229		----		----	
6404	EN12596	1053	C	0.91	first reported 1153
6419		----		----	
6420		----		----	
6474		----		----	
6561		----		----	
normality		OK			
n		13			
outliers		0			
mean (n)		1019.82			
st.dev. (n)		52.906			
R(calc.)		148.14			
st.dev.(EN12596:23)		36.422			
R(EN12596:23)		101.98			



Determination of Flash Point C.O.C. on sample #23255; results in °C

lab	method	value	mark	z(targ)	remarks
154		----		----	
225	D92 manual	316.0		-0.30	
328		----		----	
333		----		----	
335		----		----	
342	ISO2592 automated	322		0.63	
360	ISO2592 automated	311.0		-1.08	
365		----		----	
396		----		----	
398		----		----	
399		----		----	
444		----		----	
447		----		----	
604	D92 manual	318		0.01	
657		----		----	
736	ISO2592 automated	321		0.48	
781	ISO2592 automated	more 290		----	
865	D92 manual	318		0.01	
1011	ISO2592 automated	>280		----	
1026		----		----	
1040		----		----	
1108		----		----	
1135	ISO2592 automated	308.0		-1.54	
1320		----		----	
1340	ISO2592 automated	319.5		0.24	
1378	ISO2592 automated	314		-0.61	
1399		----		----	
1402	ISO2592 automated	330		1.88	
1613	D92 manual	290	C	-4.34	first reported 250
1631	ISO2592 automated	308		-1.54	
1724	D92 manual	315	C	-0.46	first reported 268
1730		----		----	
1741	ISO2592 automated	332.0		2.19	
1833		318		0.01	
1849	ISO2592 automated	320		0.32	
1852		----		----	
1857	ISO2592 manual	more 260		----	
1881		----		----	
1944	ISO2592 manual	308		-1.54	
1958	D92 manual	320		0.32	
1990		----	W	----	test result withdrawn, reported 151.5
6228	ISO2592 automated	345		4.21	
6229	ISO2592 automated	346		4.37	
6404	ISO2592 manual	297		-3.26	
6419		----		----	
6420		----		----	
6474	D92 automated	257	R(0.01)	-9.48	
6561		----		----	

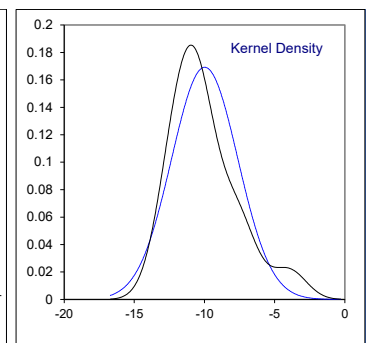
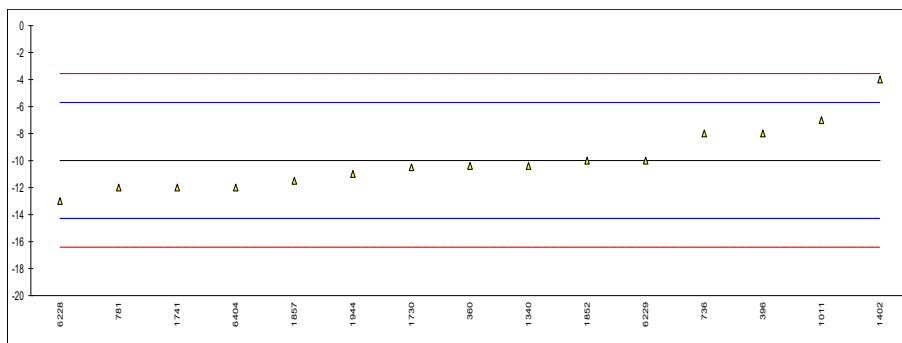
normality suspect  
n 21  
outliers 1  
mean (n) 317.93  
st.dev. (n) 13.212  
R(calc.) 36.99  
st.dev.(ISO2592:17) 6.429  
R(ISO2592:17) 18  
compare  
R(D92:18) 18



Determination of Fraass Breaking Point on sample #23255; results in °C

lab	method	value	mark	z(targ)	remarks
154		----		----	
225		----		----	
328		----		----	
333		----		----	
335		----		----	
342		----		----	
360	EN12593 automated	-10.4		-0.19	
365		----		----	
396	EN12593	-8		0.93	
398		----		----	
399		----		----	
444		----		----	
447		----		----	
604		----		----	
657		----		----	
736	EN12593 automated	-8		0.93	
781	EN12593 automated	-12		-0.94	
865		----		----	
1011	EN12593 manual	-7		1.39	
1026		----		----	
1040		----		----	
1108		----		----	
1135		----		----	
1320		----		----	
1340	EN12593 automated	-10.4		-0.19	
1378		----		----	
1399		----		----	
1402	EN12593 manual	-4		2.79	
1613		----		----	
1631		----		----	
1724		----		----	
1730	EN12593 automated	-10.5		-0.24	
1741	EN12593 automated	-12.0		-0.94	
1833		----		----	
1849		----		----	
1852	EN12593 automated	-10		-0.01	
1857	EN12593 automated	-11.5		-0.71	
1881		----		----	
1944	EN12593 automated	-11		-0.47	
1958		----		----	
1990		----		----	
6228	EN12593 automated	-13		-1.41	
6229	EN12593 automated	-10		-0.01	
6404	EN12593 automated	-12		-0.94	
6419		----		----	
6420		----		----	
6474		----		----	
6561		----		----	

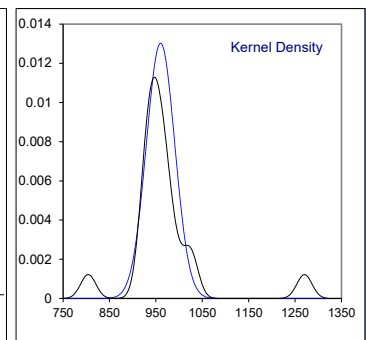
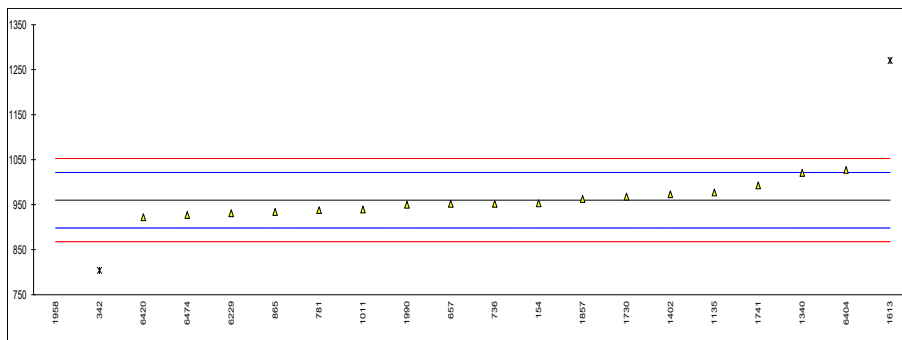
normality not OK  
n 15  
outliers 0  
mean (n) -9.99  
st.dev. (n) 2.358  
R(calc.) 6.60  
st.dev.(EN12593:15) 2.143  
R(EN12593:15) 6



Determination of Kinematic Viscosity at 135 °C on sample #23255; results in mm<sup>2</sup>/s

lab	method	value	mark	z(targ)	remarks
154	D2170	953		-0.22	
225		----		----	
328		----		----	
333		----		----	
335		----		----	
342	EN12595	804	G(0.01)	-5.05	
360		----		----	
365		----		----	
396		----		----	
398		----		----	
399		----		----	
444		----		----	
447		----		----	
604		----		----	
657	D2170	952		-0.26	
736	EN12595	952.1		-0.25	
781	EN12595	937.98		-0.71	
865	D2170/D2170M	934.0		-0.84	
1011	EN12595	939		-0.68	
1026		----		----	
1040		----		----	
1108		----		----	
1135	EN12595	977		0.56	
1320		----		----	
1340	EN12595	1020.5		1.96	
1378		----		----	
1399		----		----	
1402	EN12595	973		0.43	
1613	D2170	1270	C,G(0.01)	10.05	first reported 1080
1631		----		----	
1724		----		----	
1730	EN12595	968		0.26	
1741	EN12595	992.4		1.05	
1833		----		----	
1849		----		----	
1852		----		----	
1857	D2170	962.5		0.09	
1881		----		----	
1944		----		----	
1958	D2170	440	G(0.01)	-16.85	
1990	D2170	949.7		-0.33	
6228		----		----	
6229	EN12595	930.85		-0.94	
6404	ISO3104	1027		2.18	
6419		----		----	
6420	D2170	921.84		-1.23	
6474	D2170	927		-1.07	
6561		----		----	

normality suspect  
n 17  
outliers 3  
mean (n) 959.87  
st.dev. (n) 30.624  
R(calc.) 85.75  
st.dev.(EN12595:23) 30.853  
R(EN12595:23) 86.39  
compare  
R(D2170:22) 92.15

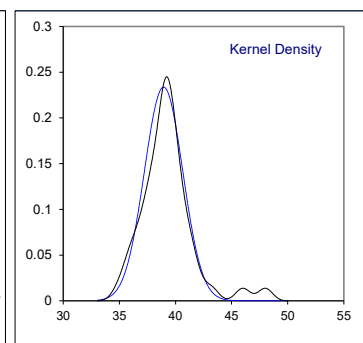
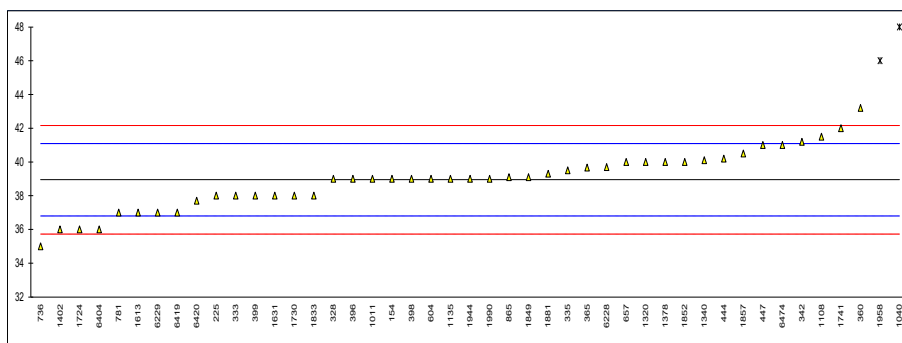




Determination of Penetration at 25 °C on sample #23255; results in 0.1 mm

lab	method	value	mark	z(targ)	remarks
154	D5 manual	39		0.05	
225	D5 manual	38.0		-0.88	
328	EN1426 manual	39		0.05	
333	EN1426 manual	38		-0.88	
335	EN1426 automated	39.5		0.52	
342	EN1426 manual	41.2		2.10	
360	EN1426 automated	43.2		3.97	
365	EN1426 manual	39.667		0.67	
396	EN1426	39		0.05	
398	EN1426 manual	39		0.05	
399	EN1426 manual	38		-0.88	
444	EN1426 automated	40.2	C	1.17	first reported 45.1
447	EN1426 automated	41		1.92	
604	D5 manual	39		0.05	
657	D5 manual	40		0.98	
736	EN1426 manual	35		-3.68	
781	EN1426 automated	37		-1.82	
865	D5/D5M manual	39.1		0.14	
1011	EN1426 automated	39		0.05	
1026		----		----	
1040	EN1426 automated	48.0	R(0.01)	8.45	
1108	EN1426 automated	41.5		2.38	
1135	EN1426 automated	39		0.05	
1320	EN1426 manual	40		0.98	
1340	EN1426 manual	40.1		1.08	
1378	EN1426 automated	40		0.98	
1399		----		----	
1402	EN1426 manual	36		-2.75	
1613	D5 automated	37.0		-1.82	
1631	EN1426 automated	38		-0.88	
1724	D5 manual	36		-2.75	
1730	EN1426 automated	38		-0.88	
1741	EN1426 automated	42.0		2.85	
1833	EN1426 automated	38		-0.88	
1849	EN1426 automated	39.1		0.14	
1852	EN1426 automated	40		0.98	
1857	EN1426 manual	40.5		1.45	
1881	EN1426 automated	39.3		0.33	
1944	EN1426 automated	39		0.05	
1958	D5 manual	46	R(0.01)	6.58	
1990	D5 automated	39		0.05	
6228	EN1426 automated	39.7		0.70	
6229	EN1426 automated	37		-1.82	
6404	EN1426 manual	36		-2.75	
6419	EN1426 automated	37		-1.82	
6420	D5 automated	37.7		-1.16	
6474	D5 automated	41		1.92	
6561		----		----	

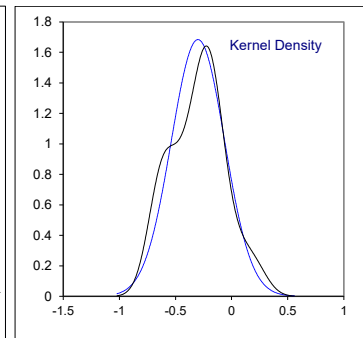
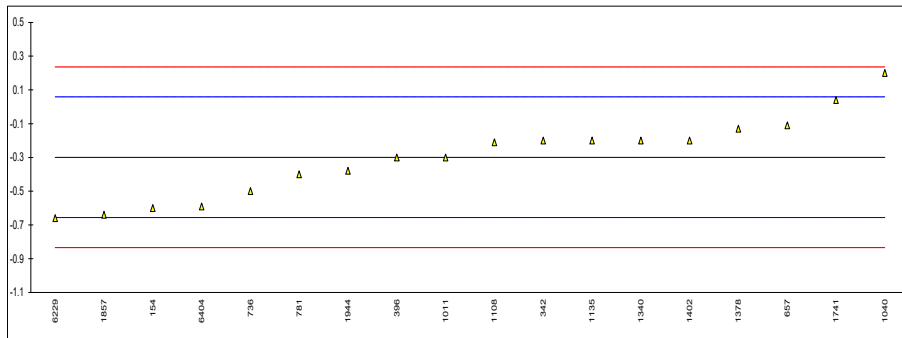
		<u>EN1426 only</u>	<u>D5 only</u>
normality	OK	OK	OK
n	43	33	10
outliers	2	1	1
mean (n)	38.948	39.060	38.580
st.dev. (n)	1.7078	1.7836	1.4505
R(calc.)	4.782	4.994	4.061
st.dev.(EN1426:15)	1.0714	1.0714	---
R(EN1426:15)	3	3	---
compare			
R(D5:20)	7	---	7



Determination of Penetration Index on sample #23255;

lab	method	value	mark	z(targ)	remarks
154	EN12591	-0.6		-1.69	
225		----		----	
328		----		----	
333		----		----	
335		----		----	
342	EN12591	-0.2		0.55	
360		----		----	
365		----		----	
396	EN12591	-0.3		-0.01	
398		----		----	
399		----		----	
444		----		----	
447		----		----	
604		----		----	
657	Calculation	-0.11		1.06	
736	EN12591	-0.5		-1.13	
781	EN12591	-0.4		-0.57	
865		----		----	
1011	EN12591	-0.3		-0.01	
1026		----		----	
1040	EN12591	0.2		2.79	
1108	EN12591	-0.21		0.50	
1135	EN12591	-0.2		0.55	
1320		----		----	
1340	EN12591	-0.20		0.55	
1378	EN12591	-0.13		0.95	
1399		----		----	
1402		-0.2		0.55	
1613		----		----	
1631		----		----	
1724		----		----	
1730		----		----	
1741	EN12591	0.04		1.90	
1833		----		----	
1849		----		----	
1852		----		----	
1857	EN12591	-0.64	E	-1.91	*)
1881		----		----	
1944	EN12591	-0.38		-0.45	
1958		----		----	
1990		----		----	
6228		----		----	
6229	EN12591	-0.66		-2.02	
6404	EN12591	-0.59		-1.63	
6419		----		----	
6420		----		----	
6474		----		----	
6561		----		----	
normality		OK			
n		18			
outliers		0			
mean (n)		-0.299			
st.dev. (n)		0.2369			
R(calc.)		0.663			
st.dev.(EN12591:09)		0.1786			
R(EN12591:09)		0.5			

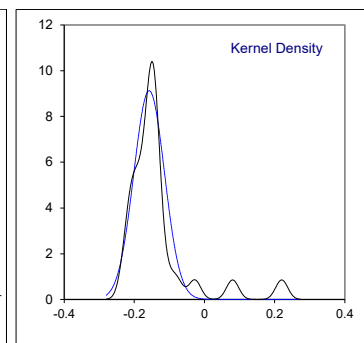
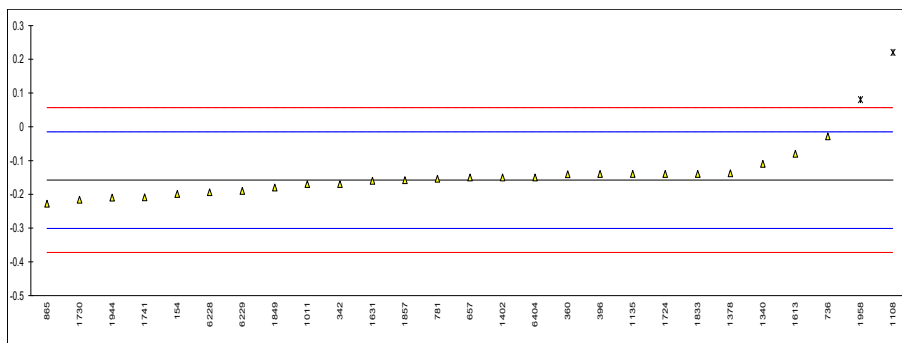
Lab 1857: calculation difference as Softening Point was corrected without correction of Penetration Index, iis calculated -0.21



Determination of RTFOT at 163 °C, Change of Mass on sample #23255; results in %

lab	method	value	mark	z(targ)	remarks
154	D2872	-0.199		-0.58	
225		----		----	
328		----		----	
333		----		----	
335		----		----	
342	EN12607-1	-0.17		-0.17	
360	EN12607-1	-0.141		0.24	
365		----		----	
396	EN12607-1	-0.14		0.25	
398		----		----	
399		----		----	
444		----		----	
447		----		----	
604		----		----	
657	D2872	-0.15		0.11	
736	EN12607-1	-0.028		1.82	
781	EN12607-1	-0.154		0.05	
865	D2872	-0.228		-0.98	
1011	EN12607-1	-0.17		-0.17	
1026		----		----	
1040		----		----	
1108	EN12607-1	0.22	C,R(0.01)	5.29	first reported 0.094
1135	EN12607-1	-0.14		0.25	
1320		----		----	
1340	EN12607-1	-0.11		0.67	
1378	EN12607-1	-0.138		0.28	
1399		----		----	
1402	EN12607-1	-0.15		0.11	
1613	D2872	-0.08	C	1.09	first reported 0.08
1631	EN12607-1	-0.16		-0.03	
1724		-0.14		0.25	
1730	EN12607-1	-0.216		-0.81	
1741	EN12607-1	-0.209		-0.72	
1833		-0.14		0.25	
1849	EN12607-1	-0.18		-0.31	
1852		----		----	
1857	EN12607-1	-0.158		0.00	
1881		----		----	
1944	EN12607-1	-0.21		-0.73	
1958	D2872	0.08	R(0.01)	3.33	
1990		----	W	----	test result withdrawn, reported 48.6
6228	EN12607-1	-0.194		-0.51	
6229	EN12607-1	-0.19		-0.45	
6404	EN12607-1	-0.15	C	0.11	first reported 0.15
6419		----		----	
6420		----		----	
6474		----		----	
6561		----		----	

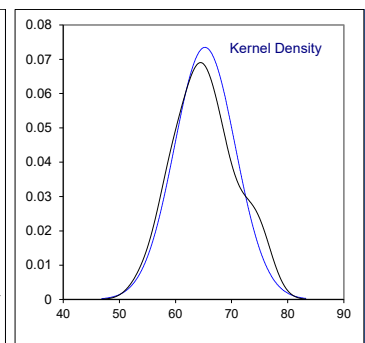
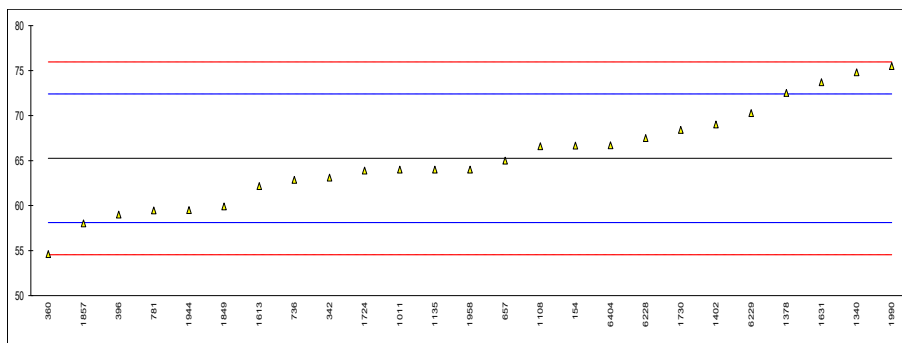
normality not OK  
n 25  
outliers 2  
mean (n) -0.158  
st.dev. (n) 0.0437  
R(calc.) 0.1224  
st.dev.(EN12607-1:14) 0.0714  
R(EN12607-1:14) 0.2



Determination of RTFOT at 163 °C, Retained Penetration on sample #23255; results in %

lab	method	value	mark	z(targ)	remarks
154	D2872	66.67		0.40	
225		----		----	
328		----		----	
333		----		----	
335		----		----	
342	EN12607-1	63.1		-0.60	
360	EN12607-1	54.6		-2.98	
365		----		----	
396	EN12607-1	59.0		-1.75	
398		----		----	
399		----		----	
444		----		----	
447		----		----	
604		----		----	
657	D2872	65		-0.07	
736	EN12607-1	62.86		-0.67	
781	EN12607-1	59.46		-1.62	
865		----		----	
1011	EN12607-1	64		-0.35	
1026		----		----	
1040		----		----	
1108	EN12607-1	66.59		0.38	
1135	EN12607-1	64		-0.35	
1320		----		----	
1340	EN12607-1	74.81		2.68	
1378	EN12607-1	72.5		2.03	
1399		----		----	
1402	EN12607-1	69		1.05	
1613	D2872	62.16		-0.86	
1631	EN12607-1	73.7		2.37	
1724		63.89		-0.38	
1730	EN12607-1	68.4		0.88	
1741		----		----	
1833		----		----	
1849	EN12607-1	59.9		-1.50	
1852		----		----	
1857	EN12607-1	58.02		-2.02	
1881		----		----	
1944	EN12607-1	59.5		-1.61	
1958	D2872	64		-0.35	
1990	D1754	75.5		2.87	
6228	EN12607-1	67.5		0.63	
6229	EN12607-1	70.27		1.41	
6404	EN12607-1	66.7		0.41	
6419		----		----	
6420		----		----	
6474		----		----	
6561		----		----	

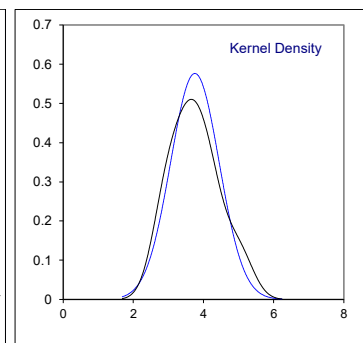
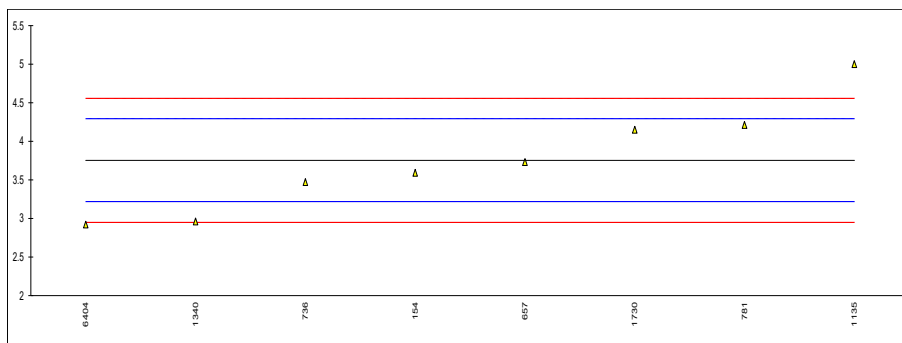
normality OK  
n 25  
outliers 0  
mean (n) 65.245  
st.dev. (n) 5.4277  
R(calc.) 15.197  
st.dev.(EN12607-1:14) 3.5714  
R(EN12607-1:14) 10



Determination of RTFOT at 163 °C, Viscosity Ratio on sample #23255

lab	method	value	mark	z(targ)	remarks
154	D2872	3.59		-0.61	
225		----		----	
328		----		----	
333		----		----	
335		----		----	
342		----		----	
360		----		----	
365		----		----	
396		----		----	
398		----		----	
399		----		----	
444		----		----	
447		----		----	
604		----		----	
657	D2872	3.73		-0.09	
736	EN12607-1	3.47		-1.06	
781	EN12607-1	4.21		1.70	
865		----		----	
1011		----		----	
1026		----		----	
1040		----		----	
1108		----		----	
1135	EN12607-1	5.0		4.65	
1320		----		----	
1340	EN12607-1	2.96		-2.96	
1378		----		----	
1399		----		----	
1402		----		----	
1613		----		----	
1631		----		----	
1724		----		----	
1730	EN12607-1	4.15		1.48	
1741		----		----	
1833		----		----	
1849		----		----	
1852		----		----	
1857		----		----	
1881		----		----	
1944		----		----	
1958		----		----	
1990		----		----	
6228		----		----	
6229		----		----	
6404	EN12607-1	2.92		-3.11	
6419		----		----	
6420		----		----	
6474		----		----	
6561		----		----	

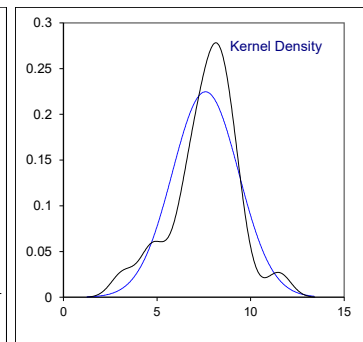
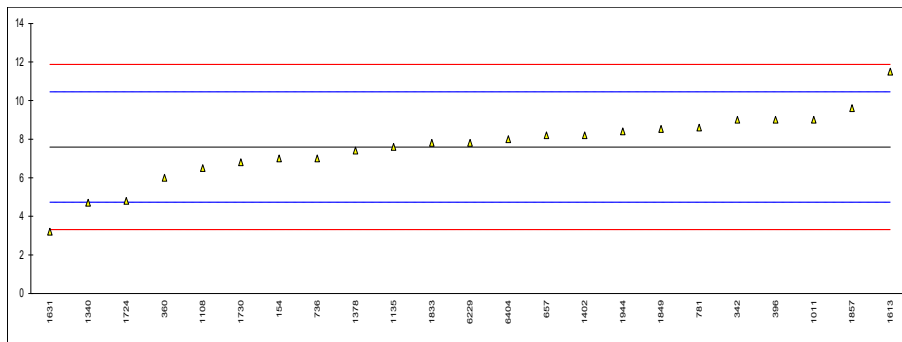
normality unknown  
n 8  
outliers 0  
mean (n) 3.754  
st.dev. (n) 0.6919  
R(calc.) 1.937  
st.dev.(EN12607-1:14) 0.2681  
R(EN12607-1:14) 0.751



Determination of RTFOT at 163 °C, Increase in Softening Point on sample #23255; results in °C

lab	method	value	mark	z(targ)	remarks
154	D2872	7.0		-0.41	
225		----		----	
328		----		----	
333		----		----	
335		----		----	
342	EN12607-1	9		0.99	
360	EN12607-1	6.00		-1.11	
365		----		----	
396	EN12607-1	9.0		0.99	
398		----		----	
399		----		----	
444		----		----	
447		----		----	
604		----		----	
657	D2872	8.2		0.43	
736	EN12607-1	7.0		-0.41	
781	EN12607-1	8.6		0.71	
865		----		----	
1011	EN12607-1	9.0		0.99	
1026		----		----	
1040		----		----	
1108	EN12607-1	6.5		-0.76	
1135	EN12607-1	7.6		0.01	
1320		----		----	
1340	EN12607-1	4.7		-2.02	
1378	EN12607-1	7.4		-0.13	
1399		----		----	
1402	EN12607-1	8.2		0.43	
1613	D2872	11.5		2.74	
1631	EN12607-1	3.2	C	-3.07	first reported 60.8
1724		4.8		-1.95	
1730	EN12607-1	6.8		-0.55	
1741		----		----	
1833		7.8		0.15	
1849	EN12607-1	8.53		0.66	
1852		----		----	
1857	EN12607-1	9.6		1.41	
1881		----		----	
1944	EN12607-1	8.4		0.57	
1958		----		----	
1990		----		----	
6228		----		----	
6229	EN12607-1	7.8		0.15	
6404	EN12607-1	8		0.29	
6419		----		----	
6420		----		----	
6474		----		----	
6561		----		----	

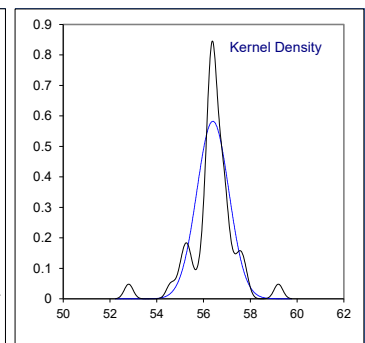
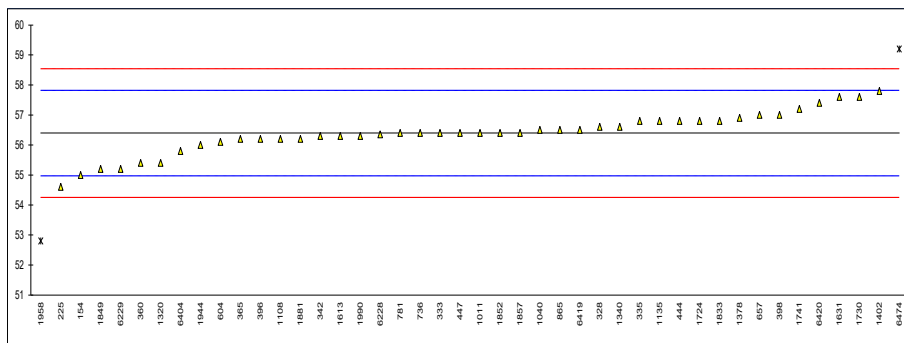
normality suspect  
n 23  
outliers 0  
mean (n) 7.59  
st.dev. (n) 1.775  
R(calc.) 4.97  
st.dev.(EN12607-1:14) 1.429  
R(EN12607-1:14) 4.0



Determination of Softening Point (Ring and Ball) on sample #23255; results in °C

lab	method	value	mark	z(target)	remarks
154	D36	55	C	-1.96	first reported 62
225	D36	54.6		-2.52	
328	EN1427	56.6		0.28	
333	EN1427	56.4		0.00	
335	EN1427	56.8		0.56	
342	EN1427	56.3		-0.14	
360	EN1427	55.4		-1.40	
365	EN1427	56.2		-0.28	
396	EN1427	56.2		-0.28	
398	EN1427	57		0.84	
399		----		----	
444	EN1427	56.8		0.56	
447	EN1427	56.4		0.00	
604	D36	56.1		-0.42	
657	D36	57.0		0.84	
736	EN1427	56.4		0.00	
781	EN1427	56.4		0.00	
865	D36/D36M	56.5		0.14	
1011	EN1427	56.4		0.00	
1026		----		----	
1040	EN1427	56.5		0.14	
1108	EN1427	56.2		-0.28	
1135	EN1427	56.8		0.56	
1320	EN1427	55.4		-1.40	
1340	EN1427	56.6		0.28	
1378	EN1427	56.9		0.70	
1399		----		----	
1402	EN1427	57.8		1.96	
1613	D36	56.3		-0.14	
1631	EN1427	57.6		1.68	
1724	D36	56.8		0.56	
1730	EN1427	57.6		1.68	
1741	EN1427	57.20		1.12	
1833	EN1427	56.8		0.56	
1849	EN1427	55.2		-1.68	
1852	EN1427	56.4		0.00	
1857	EN1427	56.4	C	0.00	first reported 54.4
1881	EN1427	56.20		-0.28	
1944	EN1427	56.0		-0.56	
1958	D36	52.8	R(0.01)	-5.04	
1990	D36	56.3		-0.14	
6228	EN1427	56.35		-0.07	
6229	EN1427	55.2		-1.68	
6404	EN1427	55.8		-0.84	
6419	EN1427	56.5		0.14	
6420	D36	57.4		1.40	
6474	D3461	59.2	R(0.01)	3.92	
6561		----		----	

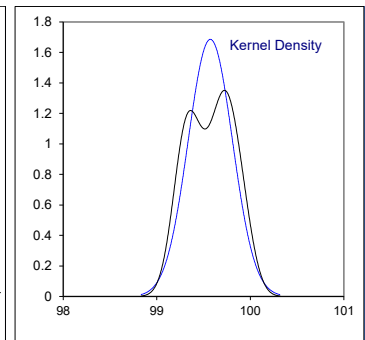
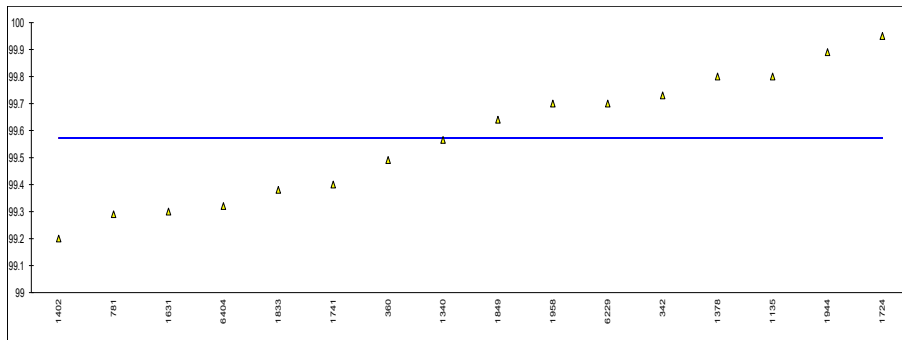
normality OK  
 n 42  
 outliers 2  
 mean (n) 56.40  
 st.dev. (n) 0.685  
 R(calc.) 1.92  
 st.dev.(EN1427:15) 0.714  
 R(EN1427:15) 2.0  
 compare  
 R(D36/D36M:14R20) 5.47 automated electronic thermometer  
 R(D36/D36M:14R20) 5.15 mercury thermometer



Determination of Solubility in Xylene on sample #23255; results in %M/M

lab	method	value	mark	z(targ)	remarks
154		----		----	
225		----		----	
328		----		----	
333		----		----	
335		----		----	
342	EN12592	99.73		----	
360	EN12592	99.491		----	
365		----		----	
396		----		----	
398		----		----	
399		----		----	
444		----		----	
447		----		----	
604		----		----	
657		----		----	
736		----		----	
781	EN12592	99.29		----	
865		----		----	
1011		----		----	
1026		----		----	
1040		----		----	
1108		----		----	
1135	EN12592	99.80		----	
1320		----		----	
1340	EN12592	99.565		----	
1378	EN12592	99.8		----	
1399		----		----	
1402	EN12592	99.20		----	
1613		----		----	
1631	EN12592	99.30		----	
1724	EN12592	99.95		----	
1730		----		----	
1741	EN12592	99.400		----	
1833	EN12592	99.38		----	
1849	EN12592	99.64		----	
1852		----		----	
1857		----		----	
1881		----		----	
1944	EN12592	99.89		----	
1958	D2042	99.7		----	
1990		----		----	
6228		----		----	
6229	EN12592	99.70		----	
6404	EN12592	99.32		----	
6419		----		----	
6420		----		----	
6474		----		----	
6561		----		----	

normality OK  
n 16  
outliers 0  
mean (n) 99.57  
st.dev. (n) 0.236  
R(calc.) 0.66  
st.dev.(EN12592:14) (0.054)  
R(EN12592:14) (0.150)





## Determination of Ductility on sample #23255; results in cm

lab	method	value	mark	z(targ)	remarks
154	D113	>120		----	
225		----		----	
328		----		----	
333		----		----	
335		----		----	
342		----		----	
360		----		----	
365		----		----	
396		----		----	
398		----		----	
399		----		----	
444		----		----	
447		----		----	
604		----		----	
657	D113	150+		----	
736	D113	150+		----	
781	D113	>100		----	
865	D113	>100		----	
1011		----		----	
1026		----		----	
1040		----		----	
1108		----		----	
1135		----		----	
1320		----		----	
1340		----		----	
1378		----		----	
1399		----		----	
1402		----		----	
1613	D113	>100		----	
1631		----		----	
1724		----		----	
1730		----		----	
1741		----		----	
1833		----		----	
1849		----		----	
1852		----		----	
1857		----		----	
1881		----		----	
1944	D113	150		----	
1958	D113	>100		----	
1990		----		----	
6228		----		----	
6229	D113	150		----	
6404	EN13589	115		----	
6419		----		----	
6420		----		----	
6474	D113	116		----	
6561		----		----	
	n	11			
	mean (n)	>100			

## APPENDIX 2

### Number of participants per country

1 lab in AUSTRIA  
1 lab in BELGIUM  
1 lab in BOSNIA and HERZEGOVINA  
1 lab in BULGARIA  
1 lab in CHINA, People's Republic  
1 lab in COTE D'IVOIRE  
1 lab in EGYPT  
3 labs in FRANCE  
2 labs in GERMANY  
3 labs in GREECE  
1 lab in IRELAND  
4 labs in ITALY  
1 lab in JORDAN  
1 lab in KAZAKHSTAN  
1 lab in LITHUANIA  
2 labs in MALAYSIA  
2 labs in NETHERLANDS  
1 lab in PORTUGAL  
1 lab in ROMANIA  
2 labs in RUSSIAN FEDERATION  
1 lab in SAUDI ARABIA  
1 lab in SERBIA  
1 lab in SINGAPORE  
1 lab in SLOVAKIA  
1 lab in SOUTH AFRICA  
1 lab in SPAIN  
4 labs in TURKIYE  
2 labs in UNITED ARAB EMIRATES  
4 labs in UNITED KINGDOM  
1 lab in UNITED STATES OF AMERICA

## APPENDIX 3

### Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?
SDS	= Safety Data Sheet

### Literature

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- 12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)

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