Results of Proficiency Test
Bitumen
December 2021

CONTENTS

Appendices:

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 Paving Grade every year. During the annual proficiency testing program 2021/2022 it was decided to continue the round robin for the analysis of Bitumen.

In this interlaboratory study 52 laboratories in 29 different 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.

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 an ISO/IEC17025 accredited laboratory.
It was decided to send one sample of Bitumen grade 70/100 in a 2.5 liter can labelled #21255. 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 a regular basis by sending out questionnaires.

2.2   PROTOCOL

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

2.3   CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

A batch of 73 subsamples of Bitumen grade 70/100 in 2.5 liter cans was obtained from a local supplier and labelled #21255.

The homogeneity of the subsamples was checked by determination of Softening Point in accordance with EN1427 on 4 stratified randomly selected subsamples.

	Softening Point (Ring and Ball) in °C
sample #21255-1	51.4
sample #21255-2	51.4
sample #21255-3	51.2
sample #21255-4	51.2

Table 1: homogeneity test results of subsamples #21255

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

	Softening Point (Ring and Ball) in °C
r (observed)	0.3
reference test method	EN1427:15
0.3 x R (reference test method)	0.6

Table 2: evaluation of the repeatability of subsamples #21255

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

To each of the participating laboratories one 2.5 L can of sample #21255 was sent on November 3, 2021. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Bitumen stored 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 asked to determine on sample #21255: 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) and Solubility in Xylene.

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/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3   RESULTS

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

Directly after the deadline a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and 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:

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

The $z_{(target)}$ 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.
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 no problems were encountered with the dispatch of the samples. One participant did not report any test results. All other participants reported in time. Not all participants were able to report all analyzes requested.
Finally, 51 participants reported in total 348 numerical test results. Observed were 11 outlying test results, which is 3.2%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

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

4.1   EVALUATION PER TEST

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

Density at 25°C: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of EN15326:07.

Dynamic Viscosity at 60°C: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of EN12596:14.

Flash Point C.O.C.: This determination was very problematic. Two statistical outliers were observed. The calculated reproducibility is not at all in agreement with the requirements of ISO2592:17.

Fraass Breaking Point: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of EN12593:15.

Kinematic Viscosity at 135°C: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of EN12595:14 or ASTM D2170/2170M:18.

Penetration at 25°C: This determination may be problematic depending on the test method used. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of EN1426:15 but is in full agreement with the requirements of ASTM D5/5M:19a.
When the test results of test method EN1426 and ASTM D5/5M are evaluated separately, the reproducibility for the EN1426 data is still not in agreement, but the ASTM D5/5M data is in agreement with the respective method requirements.
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 practise, it will get abrasion and wear and should be replaced regularly.

Penetration Index: 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 EN12591:09.

RTFOT at 163°C: The determination on Change of Mass was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of EN12607-1:14.

The determination on Retained Penetration was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of EN12607-1:14.

The determination on Viscosity Ratio was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN12607-1:14.

The determination on Increase in Softening Point was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN12607-1:14.

Softening Point (Ring and Ball): This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN1427:15 and is also in agreement with the requirements of ASTM D36/36M:14e1.

Solubility in Xylene: This determination was very problematic. No statistical outliers were observed. Due to the large variation in the test results, it was decided not to calculate z-scores.

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 test methods (in casu ASTM, EN and ISO test methods) are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Density at 25°C	kg/m^3	34	1030.5	5.4	5
Dynamic Viscosity at 60°C	Pa.s	15	403	64	40
Flash Point C.O.C.	°C	24	312	42	18
Fraass Breaking Point	°C	18	-23.2	7.6	6
Kinematic Viscosity at 135°C	mm^2/s	19	669.4	86.9	60.2
Penetration at 25°C	0.1 mm	50	76.4	9.1	4.6
Penetration Index		22	0.18	0.52	0.5
RTFOT - Change of Mass	%	28	-0.17	0.25	0.20
RTFOT - Retained Penetration	%	27	62.4	10.2	10
RTFOT - Viscosity Ratio		7	5.4	1.0	1.1
RTFOT - Increase in Soft. Point	°C	26	10.0	3.1	4.0
Softening Point (Ring & Ball)	°C	49	51.2	2.0	2.0
Solubility in Xylene	%M/M	18	99.56	0.71	(0.15)

Table 3: reproducibilities of tests on sample #21255

Results between no z-scores were calculated

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

4.3   COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2021 WITH PREVIOUS PTS

	December 2021	December 2020	December 2019	December 2018	December 2017
Number of reporting laboratories	51	50	45	37	50
Number of test results	348	315	310	511	289
Number of statistical outliers	11	14	11	15	7
Percentage of statistical outliers	3.2%	4.4%	3.5%	2.9%	2.4%

Table 4: comparison with previous proficiency tests

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

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

Parameter	December 2021	December 2020	December 2019	December 2018		December 2017
Paving Grade	70/100	35/50	70/100	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	-	++	--	(--)	+/-	(--)
RTFOT - Retained Penetration	+/-	-	+/-	-	-	+
RTFOT - Viscosity Ratio	+/-	n.e.	(--)	+	+/-	--
RTFOT - Increase in Soft. Point	+	+/-	-	+/-	+/-	+
Softening Point (Ring and Ball)	+/-	-	+/-	+	+	-
Solubility in Xylene	(--)	(--)	(--)	+/-	+	n.e.

Table 5: comparison determinations against the reference test methods

Results between brackets should be used with care

In the table above 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 #21255; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
154	D70	1030		-0.29	
168	D70	1028		-1.41	
225		-----		-----	
328	EN15326	1030.0		-0.29	
333		-----		-----	
335	EN15326	1030		-0.29	
342	D70	1030		-0.29	
357	D70	1030.4		-0.06	
360	EN15326	1029.05		-0.82	
365		-----		-----	
396	ISO3838	1033.5		1.67	
398		-----		-----	
399	D70	1032.6		1.17	
444	D70	1027		-1.97	
447	D70	1030		-0.29	
604	D70	1031.1		0.33	
657	D70	1034		1.95	
781	EN15326	1028.3		-1.24	
865	D70	1030	C	-0.29	first reported 1.030 kg/m³
1011	D70	1373	C,R(0.01)	191.79	first reported 1.373 kg/m³
1026	EN15326	1031		0.27	
1040	Calc.	1029.3		-0.68	
1108	EN15326	1033.7		1.79	
1131	EN15326	1032		0.83	
1135	EN15326	1030.8		0.16	
1320		-----		-----	
1378	D70	1030.7		0.11	
1394		-----		-----	
1402	IP189/190	1030.4	C	-0.06	first reported 1.0304 kg/m³
1613	DIN51757	1031.0		0.27	
1710		-----		-----	
1724		-----		-----	
1730		-----		-----	
1741	EN15326	1031.396		0.50	
1833		-----		-----	
1849		-----		-----	
1852		-----		-----	
1857	D70	1025.5		-2.81	
1944	EN15326	1034.32		2.13	
1990	D70	1030.0		-0.29	
6054	D70	1031.1		0.33	
6076	EN15326	1031		0.27	
6228		-----		-----	
6229	EN15326	1033		1.39	
6359	EN15326	1030		-0.29	
6364	D70	1028.95		-0.87	
6404	EN15326	1031.06		0.31	
6407		-----		-----	
6419		-----		-----	
6420		-----		-----	
6429	EN15326	1028.2		-1.29	
6430		-----		-----	

	normality	OK
	n	34
	outliers	1
	mean (n)	1030.511
	st.dev. (n)	1.9330
	R(calc.)	5.412
	st.dev.(EN15326:07)	1.7857
	R(EN15326:07)	5
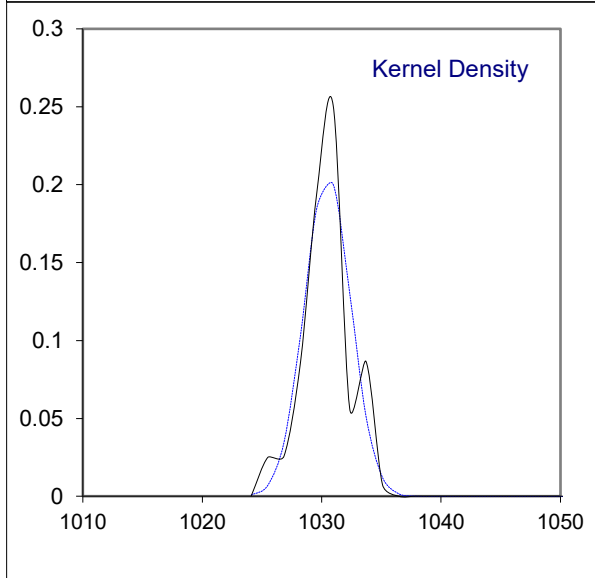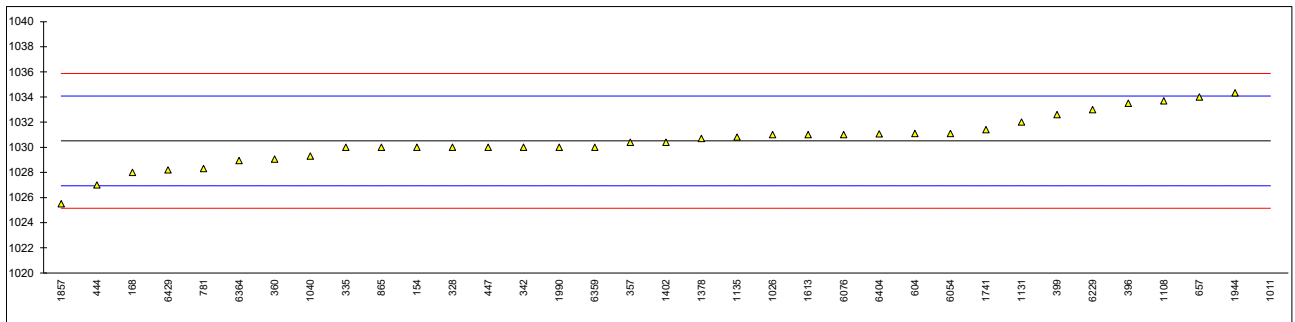
Determination of Dynamic Viscosity at 60°C on sample #21255; results in Pa.s

lab	method	value	mark	z(targ)	remarks
154	D2171	388		-1.05	
168		-----		-----	
225		-----		-----	
328		-----		-----	
333		-----		-----	
335		-----		-----	
342		-----		-----	
357	EN12596	399		-0.28	
360		-----		-----	
365		-----		-----	
396	EN12596	395.8		-0.51	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2171	427		1.66	
781	EN12596	386.0		-1.19	
865	D2171	346.5		-3.93	
1011		-----		-----	
1026	EN12596	430.5		1.91	
1040		-----		-----	
1108		-----		-----	
1131	EN12596	397.6		-0.38	
1135	EN12596	401		-0.14	
1320		-----		-----	
1378		-----		-----	
1394		-----		-----	
1402	EN12596	437		2.36	
1613		-----		-----	
1710		-----	W	-----	test result withdrawn, reported 607
1724		-----		-----	
1730		-----		-----	
1741	EN12596	408.71	C	0.39	first reported 321.85
1833		-----		-----	
1849		-----		-----	
1852		-----		-----	
1857	D2171	390.5		-0.87	
1944		-----		-----	
1990	D2171	402.5		-0.04	
6054		-----		-----	
6076		-----		-----	
6228		-----		-----	
6229		-----		-----	
6359	EN12596	430		1.87	
6364		-----		-----	
6404	EN12596	406	C	0.20	first reported 506
6407		-----		-----	
6419		-----		-----	
6420		-----		-----	
6429		-----	W	-----	test result withdrawn, reported 168.8
6430		-----		-----	

normality	suspect
n	15
outliers	0
mean (n)	403.07
st.dev. (n)	22.713
R(calc.)	63.60
st.dev.(EN12596:14)	14.396
R(EN12596:14)	40.31
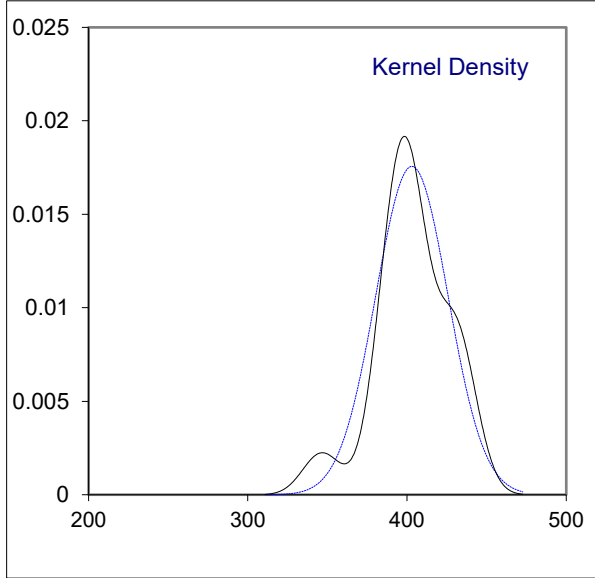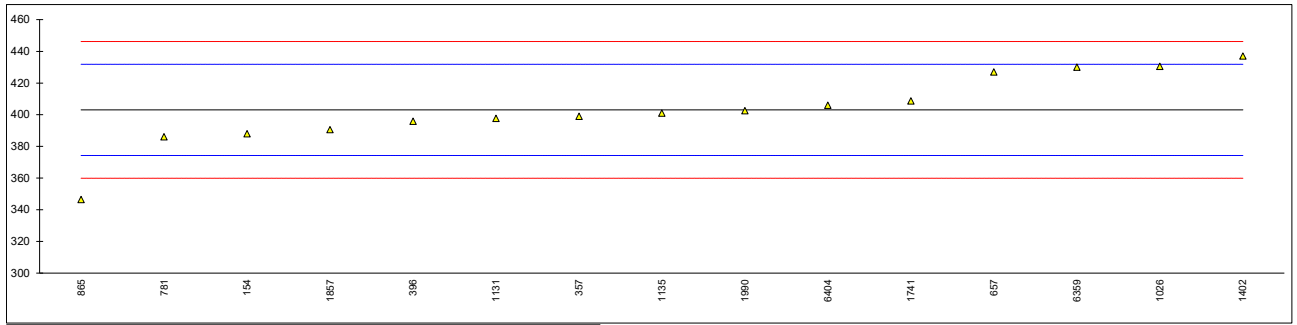
Determination of Flash Point C.O.C. on sample #21255; results in °C

lab	method	value	mark	z(targ)	remarks
154	D92 automated	334		3.42	
168	D92 automated	330		2.80	
225	D92 manual	306.0		-0.93	
328		-----		-----	
333		-----		-----	
335		-----		-----	
342		-----		-----	
357	ISO2592 automated	308		-0.62	
360	ISO2592 automated	290.7		-3.31	
365		-----		-----	
396	ISO2592 manual	300		-1.86	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604	D92 manual	310		-0.31	
657	D92 manual	368	R(0.05)	8.71	
781	ISO2592 automated	>300		-----	
865	D92 manual	306		-0.93	
1011	ISO2592 automated	>280		-----	
1026	ISO2592 manual	>320		-----	
1040	ISO2719	242.0	R(0.05)	-10.89	
1108		-----		-----	
1131	ISO2592 automated	308.0		-0.62	
1135	ISO2592 automated	314		0.31	
1320		-----		-----	
1378	D92 automated	307		-0.78	
1394		-----		-----	
1402	ISO2592 manual	>230		-----	
1613	D92 manual	332		3.11	
1710	ISO2592 automated	314		0.31	
1724	D92 manual	286		-4.04	
1730		-----		-----	
1741	ISO2592 automated	334.0		3.42	
1833	ISO2592 manual	296		-2.49	
1849	ISO2592 automated	295		-2.64	
1852		-----		-----	
1857	ISO2592 manual	310		-0.31	
1944	ISO2592 manual	316		0.62	
1990		-----		-----	
6054		-----		-----	
6076	ISO2592 automated	321		1.40	
6228		-----		-----	
6229	ISO2592 automated	326		2.18	
6359	ISO2592 automated	340		4.36	
6364		-----		-----	
6404	ISO2592 manual	314		0.31	
6407		-----		-----	
6419		-----		-----	
6420		-----		-----	
6429	ISO2592 automated	290		-3.42	
6430		-----		-----	

	normality	OK	
	n	24	
	outliers	2	
	mean (n)	311.99	
	st.dev. (n)	15.066	
	R(calc.)	42.18	
	st.dev.(ISO2592:17)	6.429	
	R(ISO2592:17)	18	
Compare			
	R(D92:18)	18	
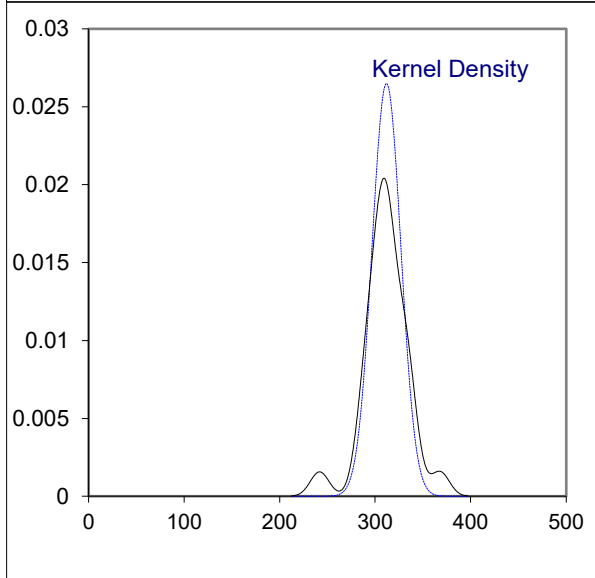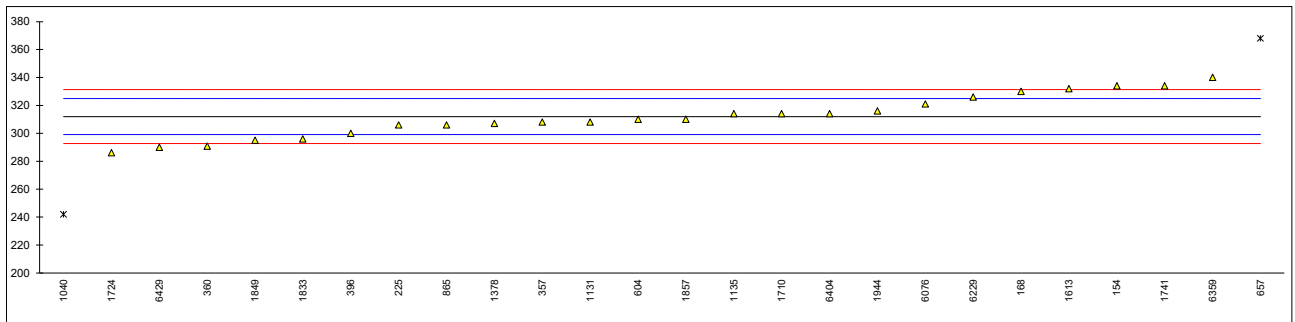
Determination of Fraass Breaking Point on sample #21255; results in °C

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225		-----		-----	
328		-----		-----	
333		-----		-----	
335		-----		-----	
342		-----		-----	
357	EN12593 automated	-24		-0.36	
360	EN12593 manual	-21.8		0.67	
365		-----		-----	
396	EN12593 automated	-17		2.91	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657		-----		-----	
781	EN12593 automated	-22		0.58	
865		-----		-----	
1011	EN12593 automated	<-20		-----	
1026		-----		-----	
1040		-----		-----	
1108		-----		-----	
1131		-----		-----	
1135	EN12593 automated	-23		0.11	
1320		-----		-----	
1378		-----		-----	
1394		-25		-0.82	
1402	EN12593 manual	-26		-1.29	
1613		-----		-----	
1710	EN12593 automated	-22		0.58	
1724		-----		-----	
1730		-----		-----	
1741	EN12593 automated	-27.0		-1.76	
1833		-----		-----	
1849		-----		-----	
1852	EN12593 automated	-22		0.58	
1857	EN12593 automated	-22.5		0.34	
1944	EN12593 manual	-21		1.04	
1990		-----		-----	
6054		-----		-----	
6076	EN12593 automated	-25		-0.82	
6228	EN12593 automated	-24.0		-0.36	
6229	EN12593 automated	-21		1.04	
6359	EN12593 automated	-21		1.04	
6364		-----		-----	
6404	EN12593 automated	-25		-0.82	
6407		-----		-----	
6419		-----		-----	
6420		-----		-----	
6429	EN12593 automated	-29		-2.69	
6430		-----		-----	

|		
normality	OK	
n	18	
outliers	0	
mean (n)	-23.24	
st.dev. (n)	2.730	
R(calc.)	7.64	
st.dev.(EN12593:15)	2.143	
R(EN12593:15)	6	
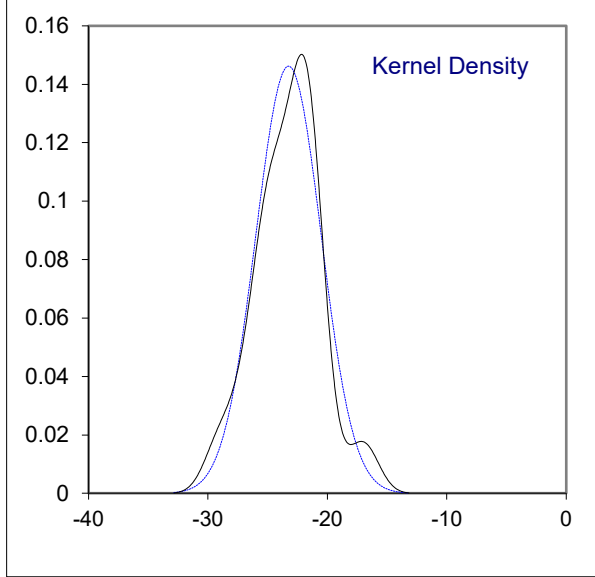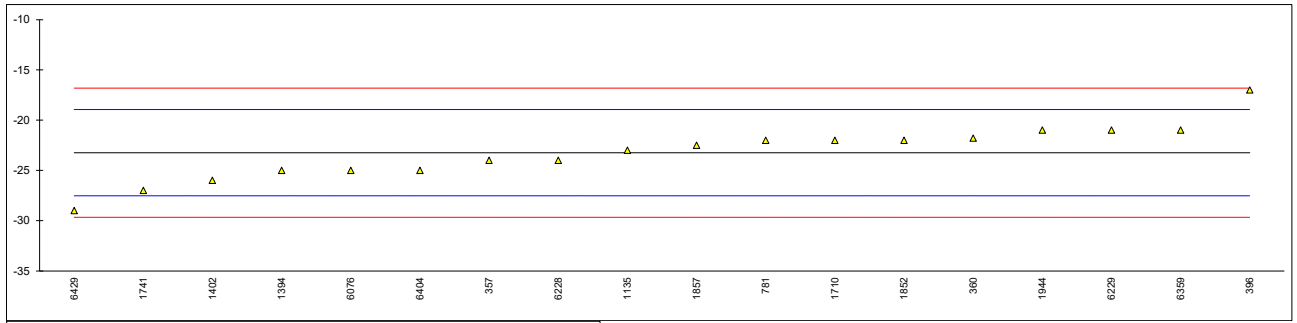
Determination of Kinematic Viscosity at 135°C on sample #21255; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
154	D2170	666		-0.16	
168		-----		-----	
225		-----		-----	
328		-----		-----	
333		-----		-----	
335		-----		-----	
342		-----		-----	
357	EN12595	622		-2.20	
360		-----		-----	
365		-----		-----	
396	EN12595	667.1		-0.11	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2170	648		-0.99	
781	EN12595	689.3		0.93	
865	D2170	676.9		0.35	
1011	EN12595	670		0.03	
1026	EN12595	643.1		-1.22	
1040		-----		-----	
1108		-----		-----	
1131	EN12595	693.8		1.13	
1135	EN12595	670		0.03	
1320		-----		-----	
1378		-----		-----	
1394		-----		-----	
1402	EN12595	653		-0.76	
1613	D2170	692.42		1.07	
1710	EN12595	680.1		0.50	
1724		-----		-----	
1730		-----		-----	
1741	EN12595	612.44		-2.65	
1833		-----		-----	
1849		-----		-----	
1852		-----		-----	
1857	EN12595	731.1		2.87	
1944		-----		-----	
1990	D2170	695.5		1.21	
6054		-----		-----	
6076		-----		-----	
6228		-----		-----	
6229	EN12595	652.4		-0.79	
6359	EN12595	634		-1.64	
6364		-----		-----	
6404	EN3104	721.1		2.40	
6407		-----		-----	
6419		-----		-----	
6420		-----		-----	
6429		-----		-----	
6430		-----		-----	

	normality	OK
	n	19
	outliers	0
	mean (n)	669.38
	st.dev. (n)	31.022
	R(calc.)	86.86
	st.dev.(EN12595:14)	21.516
	R(EN12595:14)	60.24
Compare		
	R(D2170/D2170M:18)	58.91
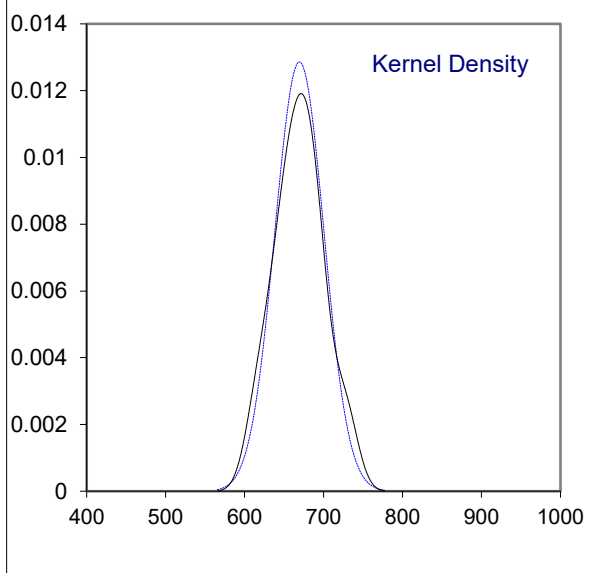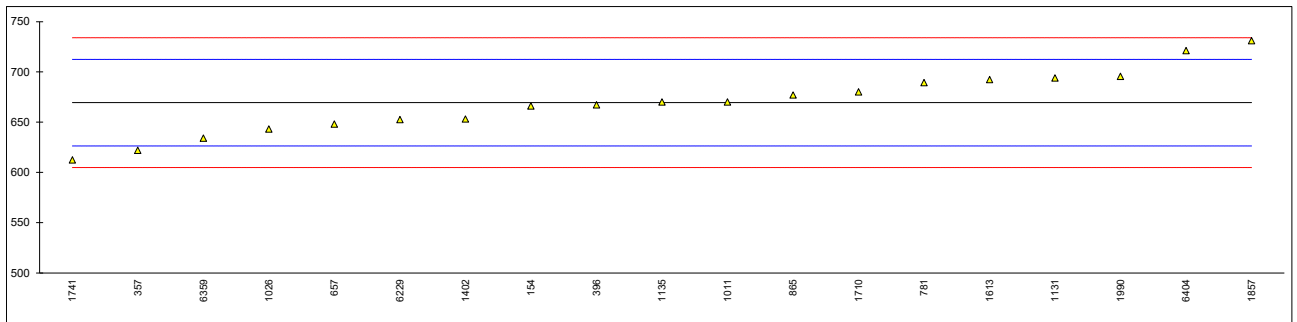
Determination of Penetration at 25°C on sample #21255; results in 0.1 mm

lab	method	value	mark	z(targ)	remarks
154	D5 manual	81		2.79	
168	D5 manual	79		1.57	
225	D5 manual	78.0		0.96	
328	EN1426 manual	77		0.35	
333	EN1426 automated	77		0.35	
335	EN1426 automated	80		2.18	
342	EN1426 manual	75		-0.87	
357	EN1426 automated	78		0.96	
360	EN1426 automated	70.8		-3.44	
365	EN1426 manual	75		-0.87	
396	EN1426 manual	75		-0.87	
398	EN1426 manual	77		0.35	
399	EN1426 manual	76		-0.26	
444	EN1426 manual	78.7		1.39	
447	EN1426 automated	84.7		5.05	
604	D5 manual	77		0.35	
657	D5 manual	80		2.18	
781	EN1426 automated	71		-3.31	
865	D5 manual	77.0		0.35	
1011	EN1426 automated	78		0.96	
1026	EN1426 automated	89	C,R(0.05)	7.68	first reported 90
1040	EN1426 automated	77.0		0.35	
1108	EN1426 automated	75		-0.87	
1131	EN1426 automated	78		0.96	
1135	EN1426 manual	76		-0.26	
1320	EN1426 manual	78		0.96	
1378	D5 automated	75		-0.87	
1394		75		-0.87	
1402	EN1426 manual	78		0.96	
1613	D5 automated	70.2		-3.80	
1710	EN1426 automated	70		-3.93	
1724	D5 manual	76		-0.26	
1730	EN1426 automated	71.5		-3.01	
1741	EN1426 automated	69.0		-4.54	
1833	EN1426 automated	78		0.96	
1849	EN1426 automated	80		2.18	
1852	EN1426 automated	76		-0.26	
1857	EN1426 manual	69.2	C	-4.41	first reported 64.2
1944	EN1426 automated	75		-0.87	
1990	D5 automated	76.0		-0.26	
6054	D5 automated	78.46		1.24	
6076	EN1426 automated	78		0.96	
6228	EN1426 automated	79.3		1.75	
6229	EN1426 automated	77		0.35	
6359	EN1426 automated	75		-0.87	
6364	D5 manual	74		-1.48	
6404	EN1426 automated	76		-0.26	
6407		-----		-----	
6419	EN1426 manual	79.1		1.63	
6420	D5 automated	74.6		-1.12	
6429	EN1426 manual	80.2		2.30	
6430	EN1426 automated	81.7		3.22	

				Only EN1426:	Only ASTM D5/5M:
	normality	OK		OK	OK
	n	50		37	13
	outliers	1		1	0
	mean (n)	76.429		76.357	76.635
	st.dev. (n)	3.2657		3.4336	2.8490
	R(calc.)	9.144		9.614	7.977
	st.dev.(EN1426:15)	1.6378		1.6362	-----
	R(EN1426:15)	4.586		4.581	-----
Compare					
	R(ASTM D5/D15M:19a)	9.400		-----	9.429
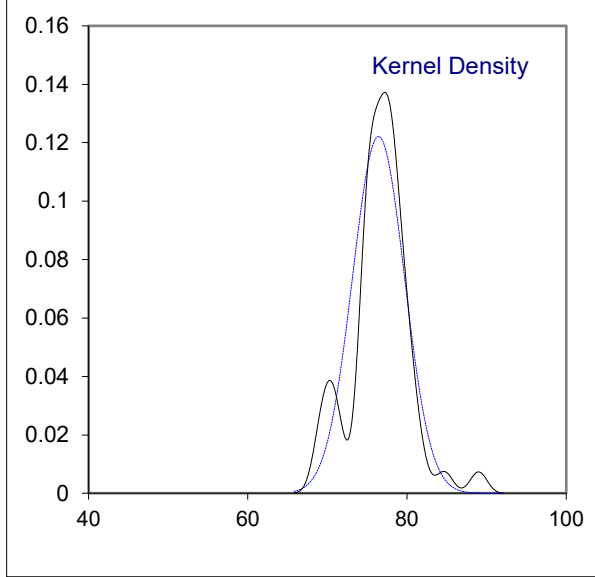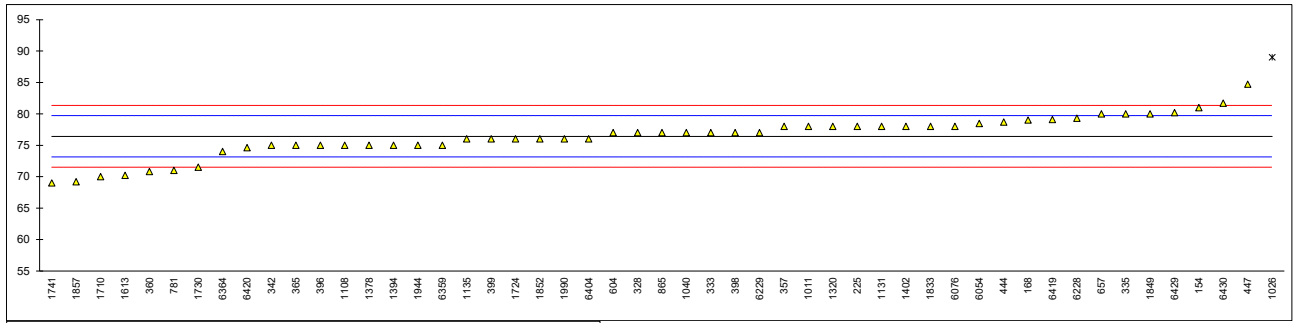
Determination of Penetration Index on sample #21255;

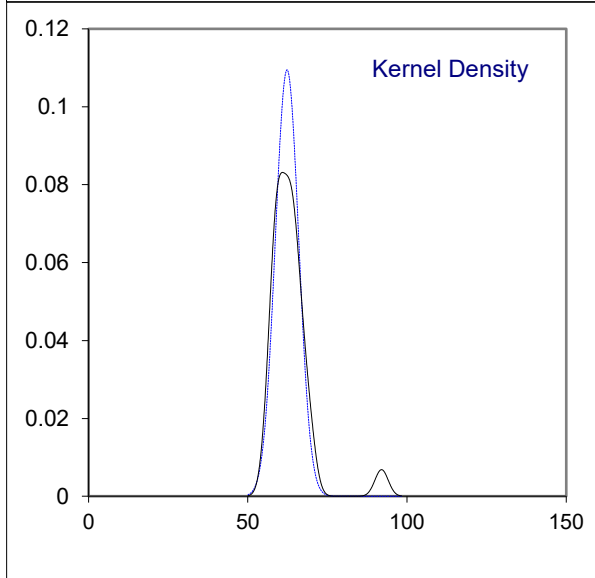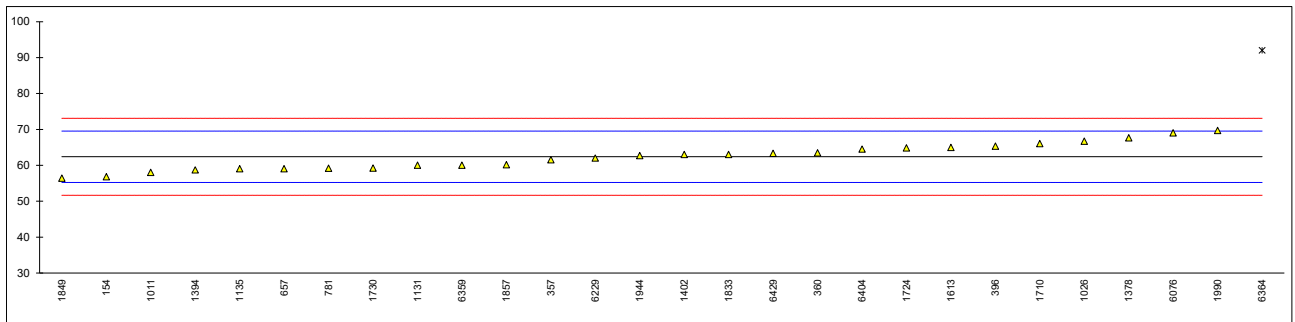| lab	method	value	mark	z(targ)	remarks
154	EN12591	0.5		1.81	
168		-----		-----	
225		-----		-----	
328		-----		-----	
333		-----		-----	
335		-----		-----	
342	EN12591	0.0		-0.99	
357	EN12591	0.24		0.36	
360		-----		-----	
365		-----		-----	
396	EN12591	0.2		0.13	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	Calculation	0.4		1.25	
781	EN12591	0.0		-0.99	
865		-----		-----	
1011	EN12591	0.3		0.69	
1026	EN12591	0.89	C,R(0.05)	4.00	first reported 0.92
1040	EN12591	-0.2		-2.11	
1108	EN12591	0.097		-0.44	
1131		-----		-----	
1135	EN12591	0.0		-0.99	
1320		-----		-----	
1378	EN12591	0.45		1.53	
1394		-----		-----	
1402	EN12591	0.2		0.13	
1613		-----		-----	
1710	EN12591	0.1		-0.43	
1724		-----		-----	
1730		-----		-----	
1741	EN12591	-0.06		-1.32	
1833		-----		-----	
1849		-----		-----	
1852	EN12591	0.16		-0.09	
1857	EN12591	-0.51	C,R(0.05)	-3.84	first reported -0.71
1944	EN12591	0.02		-0.87	
1990		-----		-----	
6054		-----		-----	
6076	EN12591	0.1		-0.43	
6228		-----		-----	
6229	EN12591	0.1		-0.43	
6359	EN12591	0.2		0.13	
6364		-----		-----	
6404	EN12591	0.5		1.81	
6407		-----		-----	
6419		-----		-----	
6420		-----		-----	
6429	EN12591	0.36		1.03	
6430	EN12591	0.21		0.19	
	normality	OK			
	n	22			
	outliers	2			
	mean (n)	0.176			
	st.dev. (n)	0.1864			
	R(calc.)	0.522			
	st.dev.(EN12591:09)	0.1786			
	R(EN12591:09)	0.5			
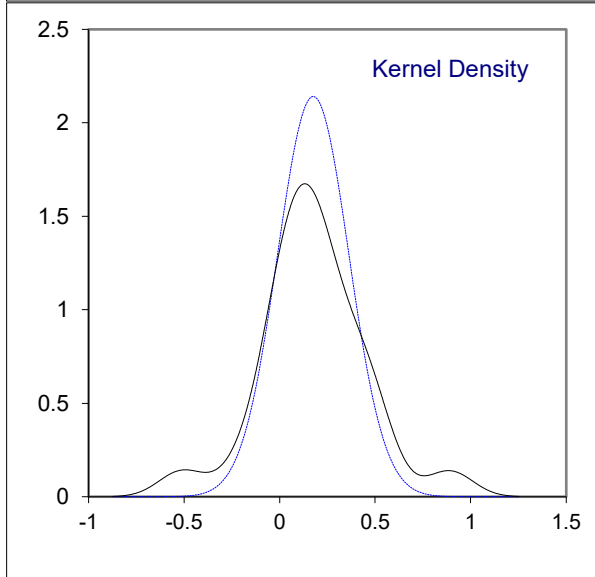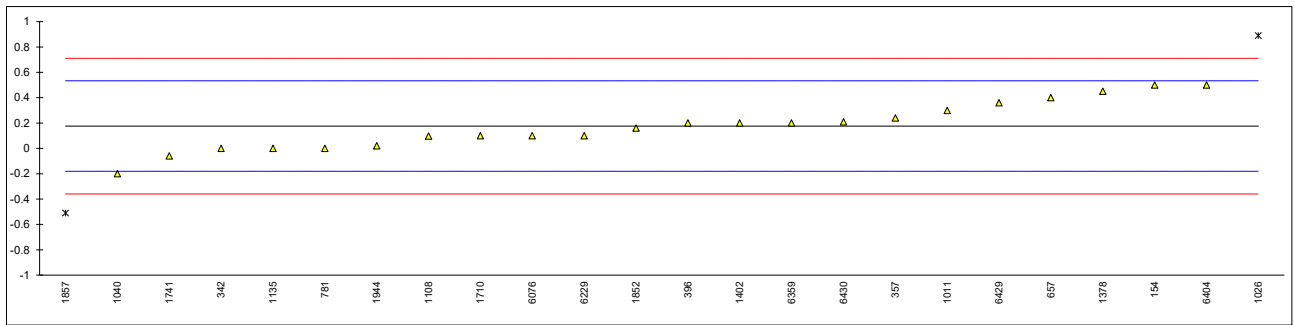
Determination of RTFOT at 163°C, Change of Mass on sample #21255; results in %

lab	method	value	mark	z(targ)	remarks
154	D2872	-0.250		-1.18	
168	D2872	-0.244		-1.09	
225		-----		-----	
328		-----		-----	
333		-----		-----	
335		-----		-----	
342		-----		-----	
357	EN12607-1	-0.11		0.78	
360	EN12607-1	-0.149		0.24	
365		-----		-----	
396	EN12607-1	-0.22		-0.76	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2872	0.02		2.60	
781	EN12607-1	-0.15		0.22	
865	D2872	-0.232		-0.92	
1011	EN12607-1	-0.26		-1.32	
1026	EN12607-1	-0.04		1.76	
1040		-----		-----	
1108		-----		-----	
1131		0.17	C,R(0.05)	4.70	first reported 0.24
1135	EN12607-1	-0.13		0.50	
1320		-----		-----	
1378	EN12607-1	-0.194		-0.39	
1394	GOST33140	-0.19		-0.34	first reported 0.19
1402	EN12607-1	-0.24		-1.04	
1613	D2872	-0.038		1.79	
1710	EN12607-1	-0.22		-0.76	
1724	D2872	-0.21		-0.62	
1730	EN12607-1	-0.192		-0.36	
1741	EN12607-1	-0.2758		-1.54	
1833	EN12607-1	-0.26		-1.32	
1849	EN12607-1	-0.27		-1.46	
1852		-----		-----	
1857	EN12607-1	-0.246		-1.12	
1944	EN12607-1	-0.058		1.51	
1990	D1754	-----		-----	
6054		-----		-----	
6076	EN12607-1	-0.16		0.08	
6228		-----		-----	
6229	EN12607-1	-0.14		0.36	
6359	EN12607-1	-0.19		-0.34	
6364		0.02	C	2.60	first reported 0.03025
6404	EN12607-1	-0.02		2.04	
6407		-----		-----	
6419		-----		-----	
6420		-----		-----	
6429	EN12607-1	0.25	R(0.05)	5.82	
6430		-----		-----	

|		
	normality	OK
	n	28
	outliers	2
	mean (n)	-0.1660
	st.dev. (n)	0.09035
	R(calc.)	0.2530
	st.dev.(EN12607-1:14)	0.07143
	R(EN12607-1:14)	0.20
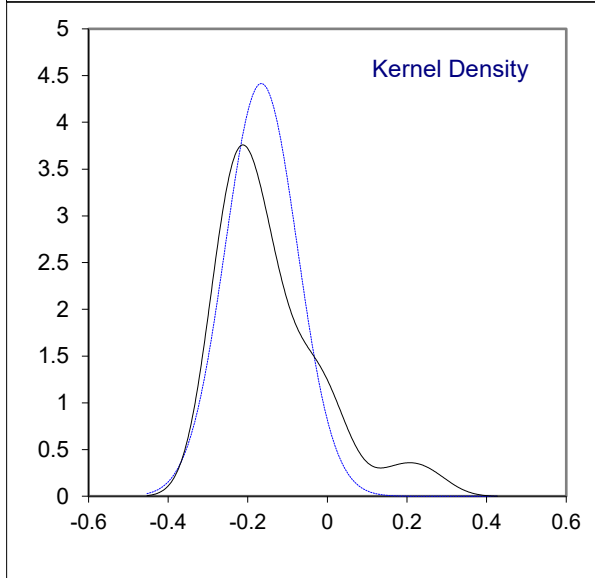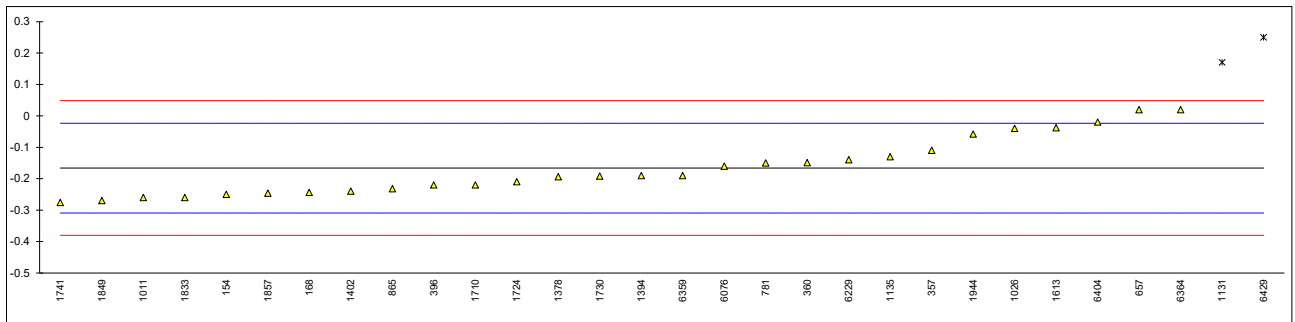
Determination of RTFOT at 163°C, Retained Penetration on sample #21255; results in %

lab	method	value	mark	z(targ)	remarks
154	D2872	56.79		-1.56	
168		-----		-----	
225		-----		-----	
328		-----		-----	
333		-----		-----	
335		-----		-----	
342		-----		-----	
357	EN12607-1	61.5		-0.24	
360	EN12607-1	63.4		0.29	
365		-----		-----	
396	EN12607-1	65.3		0.82	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2872	59		-0.94	
781	EN12607-1	59.15		-0.90	
865		-----		-----	
1011	EN12607-1	58		-1.22	
1026	EN12607-1	66.7		1.22	
1040		-----		-----	
1108		-----		-----	
1131		60		-0.66	
1135	EN12607-1	59		-0.94	
1320		-----		-----	
1378	EN12607-1	67.6		1.47	
1394		58.7		-1.02	
1402	EN12607-1	63		0.18	
1613	D2872	64.957		0.73	
1710	EN12607-1	66		1.02	
1724	D2872	64.8		0.68	
1730	EN12607-1	59.2		-0.88	
1741		-----		-----	
1833	EN12607-1	63		0.18	
1849	EN12607-1	56.4		-1.67	
1852		-----		-----	
1857	EN12607-1	60.1		-0.63	
1944	EN12607-1	62.66		0.08	
1990	D1754	69.7		2.06	
6054		-----		-----	
6076	EN12607-1	69		1.86	
6228		-----		-----	
6229	EN12607-1	62		-0.10	
6359	EN12607-1	60		-0.66	
6364		92	C,R(0.01)	8.30	first reported 91.9
6404	EN12607-1	64.47		0.59	
6407		-----		-----	
6419		-----		-----	
6420		-----		-----	
6429	EN12607-1	63.3		0.26	
6430		-----		-----	

	normality	OK	
	n	27	
	outliers	1	
	mean (n)	62.360	
	st.dev. (n)	3.6433	
	R(calc.)	10.201	
	st.dev.(EN12607-1:14)	3.5714	
	R(EN12607-1:14)	10	

Determination of RTFOT at 163°C, Viscosity Ratio on sample #21255

lab	method	value	mark	z(targ)	remarks
154	D2872	5.60		0.62	
168		-----		-----	
225		-----		-----	
328		-----		-----	
333		-----		-----	
335		-----		-----	
342		-----		-----	
357	EN12607-1	5.6		0.62	
360		-----		-----	
365		-----		-----	
396		-----		-----	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2872	4.76	C	-1.57	first reported 48
781	EN12607-1	4.97		-1.02	
865		-----		-----	
1011		-----		-----	
1026	EN12607-1	5.4		0.10	
1040		-----		-----	
1108		-----		-----	
1131		-----		-----	
1135	EN12607-1	5.5		0.36	
1320		-----		-----	
1378		-----		-----	
1394		-----		-----	
1402		-----		-----	
1613		-----		-----	
1710		-----		-----	
1724		-----		-----	
1730		-----		-----	
1741		-----		-----	
1833		-----		-----	
1849		-----		-----	
1852		-----		-----	
1857	EN12607-1	8.76	G(0.05)	8.87	
1944		-----		-----	
1990		-----		-----	
6054		-----		-----	
6076		-----		-----	
6228		-----		-----	
6229		-----		-----	
6359	EN12607-1	5.7		0.88	
6364		-----		-----	
6404	EN12607-1	3.6	G(0.05)	-4.60	
6407		-----		-----	
6419		-----		-----	
6420		-----		-----	
6429		-----	W	-----	Test result withdrawn, reported 1.55
6430		-----		-----	

normality	OK	
n	7	
outliers	2	
mean (n)	5.361	
st.dev. (n)	0.3569	
R(calc.)	0.999	
st.dev.(EN12607-1:14)	0.3830	
R(EN12607-1:14)	1.072	
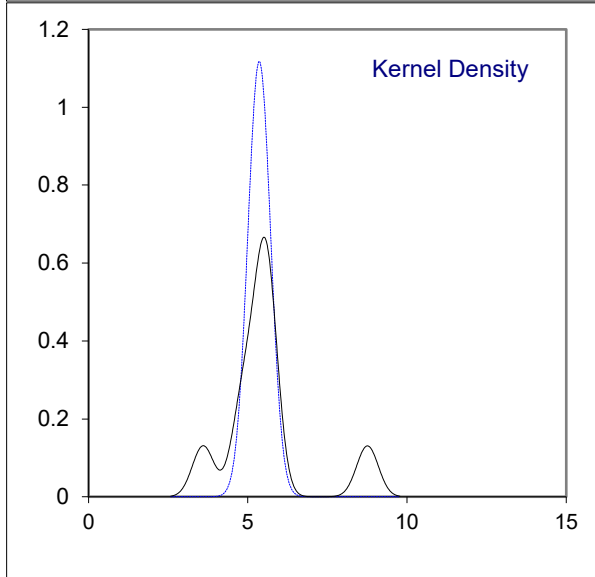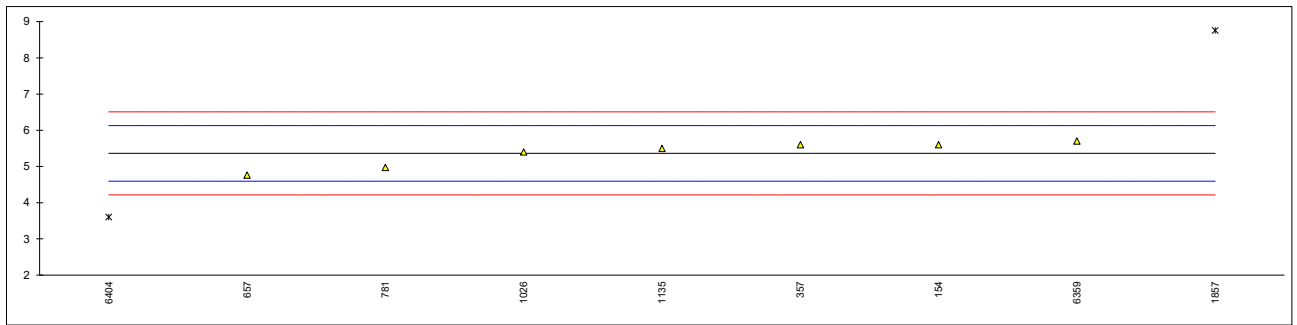
Determination of RTFOT at 163°C, Increase in Softening Point on sample #21255; results in °C
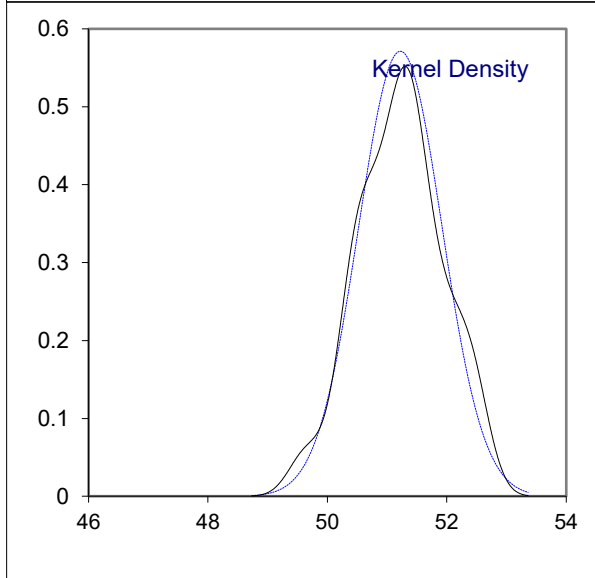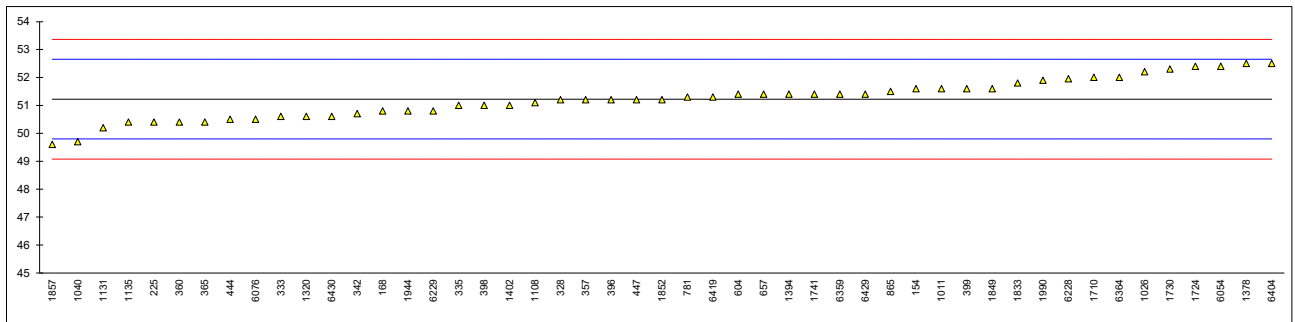
| lab	method	value	mark	z(targ)	remarks
154	D2872	11.1		0.75	
168		-----		-----	
225		-----		-----	
328		-----		-----	
333		-----		-----	
335		-----		-----	
342		-----		-----	
357	EN12607-1	9.2		-0.58	
360	EN12607-1	9.60		-0.30	
365		-----		-----	
396	EN12607-1	10.6		0.40	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2872	10		-0.02	
781	EN12607-1	9.6		-0.30	
865		-----		-----	
1011	EN12607-1	11.4		0.96	
1026	EN12607-1	11.0		0.68	
1040		-----		-----	
1108		-----		-----	
1131		10.4		0.26	
1135	EN12607-1	10.8		0.54	
1320		-----		-----	
1378	EN12607-1	8.2		-1.28	
1394		11.6		1.10	
1402	EN12607-1	9.6		-0.30	
1613	D2872	8.6		-1.00	
1710	EN12607-1	9.4		-0.44	
1724	D2872	8.2		-1.28	
1730	EN12607-1	9.4	C	-0.44	first reported 15.4
1741		-----		-----	
1833	EN12607-1	10.2		0.12	
1849	EN12607-1	9.4		-0.44	
1852		-----		-----	
1857	EN12607-1	12.9		2.01	
1944	EN12607-1	11.0		0.68	
1990		-----		-----	
6054		-----		-----	
6076	EN12607-1	9.3		-0.51	
6228		-----		-----	
6229	EN12607-1	9.2		-0.58	
6359	EN12607-1	10.8		0.54	
6364		-----		-----	
6404	EN12607-1	8.9		-0.79	
6407		-----		-----	
6419		-----		-----	
6420		-----		-----	
6429	EN12607-1	10.4		0.26	
6430		-----		-----	
	normality	OK			
	n	26			
	outliers	0			
	mean (n)	10.03			
	st.dev. (n)	1.115			
	R(calc.)	3.12			
	st.dev.(EN12607-1:14)	1.429			
	R(EN12607-1:14)	4.0			
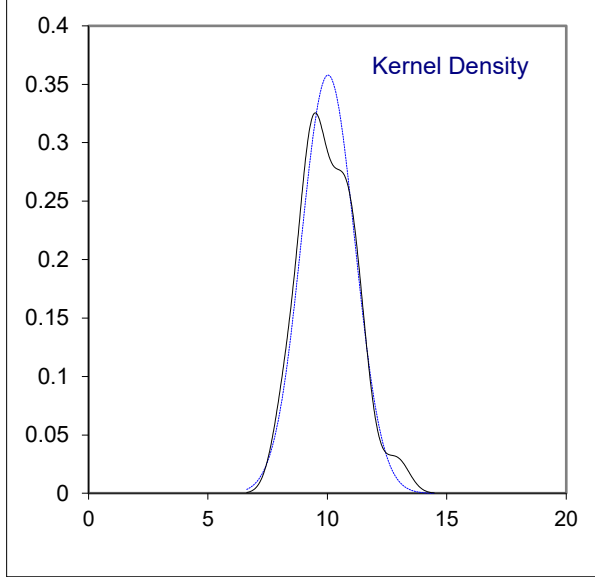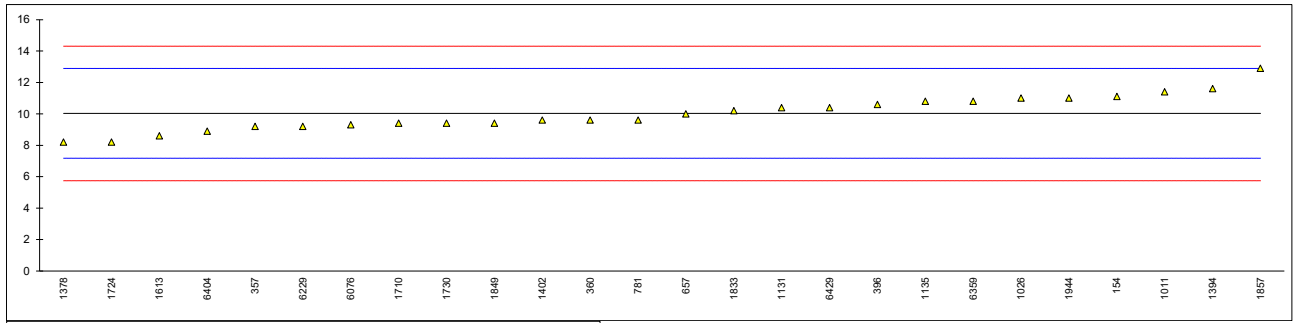
Determination of Softening Point (Ring and Ball) on sample #21255; results in °C

lab	method	value	mark	z(targ)	remarks
154	D36	51.6		0.53	
168	IP58	50.8		-0.59	
225	D36	50.4		-1.15	
328	EN1427	51.2		-0.03	
333	EN1427	50.6		-0.87	
335	EN1427	51.0		-0.31	
342	EN1427	50.7		-0.73	
357	EN1427	51.2		-0.03	
360	EN1427	50.4		-1.15	
365	EN1427	50.4		-1.15	
396	EN1427	51.2		-0.03	
398	EN1427	51.0		-0.31	
399	EN1427	51.6		0.53	
444	EN1427	50.5		-1.01	
447	EN1427	51.2		-0.03	
604	D36	51.4		0.25	
657	D36	51.4		0.25	
781	EN1427	51.3		0.11	
865	D36	51.5		0.39	
1011	EN1427	51.6		0.53	
1026	EN1427	52.2		1.37	
1040	EN1427	49.70		-2.13	
1108	EN1427	51.1		-0.17	
1131	EN1427	50.2		-1.43	
1135	EN1427	50.4		-1.15	
1320	EN1427	50.6		-0.87	
1378	D36	52.5		1.79	
1394		51.4		0.25	
1402	EN1427	51.0		-0.31	
1613		-----	W	-----	Test result withdrawn, reported 53.7
1710	EN1427	52		1.09	
1724	D36	52.4		1.65	
1730	EN1427	52.3		1.51	
1741	EN1427	51.40		0.25	
1833	EN1427	51.8		0.81	
1849	EN1427	51.6		0.53	
1852	EN1427	51.2		-0.03	
1857	EN1427	49.60		-2.27	
1944	EN1427	50.8		-0.59	
1990	D36	51.9		0.95	
6054	D36	52.4		1.65	
6076	EN1427	50.5		-1.01	
6228	EN1427	51.95		1.02	
6229	EN1427	50.8		-0.59	
6359	EN1427	51.4		0.25	
6364	D36	52.0		1.09	
6404	EN1427	52.5		1.79	
6407		-----		-----	
6419	EN1427	51.3		0.11	
6420		-----		-----	
6429	EN1427	51.4		0.25	
6430	EN1427	50.6		-0.87	

	normality	OK			
	n	49			
	outliers	0			
	mean (n)	51.223			
	st.dev. (n)	0.6984			
	R(calc.)	1.956			
	st.dev.(EN1427:15)	0.7143			
	R(EN1427:15)	2.0			
Compare					
	R(D36/D36M:14e1)	9.32	automated electronic thermometer		
	R(D36/D36M:14e1)	9.63	Mercury thermometer		

Determination of Solubility in Xylene on sample #21255; results in %M/M

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225		-----		-----	
328		-----		-----	
333		-----		-----	
335		-----		-----	
342		-----		-----	
357		-----		-----	
360	EN12592	99.67	C	-----	first reported 97.67
365		-----		-----	
396	EN12592	99.90		-----	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657		-----		-----	
781	EN12592	99.59		-----	
865	D2042	99.49		-----	
1011	EN12592	99.15		-----	
1026	EN12592	99.9		-----	
1040		-----		-----	
1108		-----		-----	
1131	EN12592	99.31		-----	
1135	EN12592	99.80		-----	
1320		-----		-----	
1378		-----		-----	
1394		-----		-----	
1402	EN12592	99.30		-----	
1613		-----		-----	
1710		-----		-----	
1724	EN12592	99.53		-----	
1730		-----		-----	
1741	EN12592	99.047		-----	
1833	EN12592	99.60		-----	
1849	EN12592	99.65		-----	
1852		-----		-----	
1857	EN12592	99.400		-----	
1944	EN12592	99.53		-----	
1990		-----		-----	
6054		-----		-----	
6076		-----		-----	
6228		-----		-----	
6229	EN12592	99.48		-----	
6359	EN12592	99.8		-----	
6364		-----		-----	
6404	EN12592	99.918		-----	
6407		-----		-----	
6419		-----		-----	
6420		-----		-----	
6429		-----		-----	
6430		-----		-----	

	normality	OK
	n	18
	outliers	0
	mean (n)	99.559
	st.dev. (n)	0.2541
	R(calc.)	0.711
	st.dev.(EN12592:14)	(0.0536)
	R(EN12592:14)	(0.15)
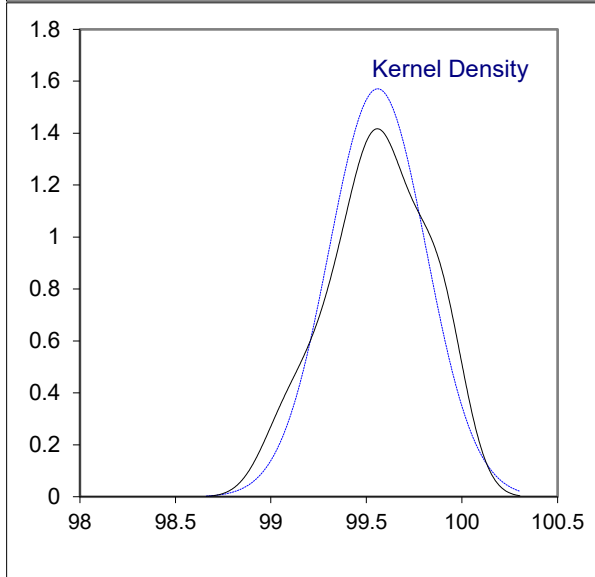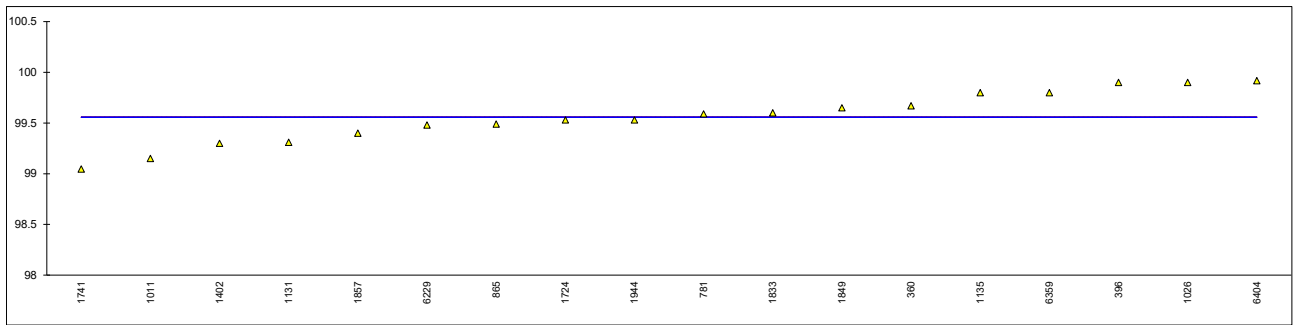
APPENDIX 2

Number of participants per country

1 lab in AUSTRIA
1 lab in BELGIUM
2 labs in BULGARIA
1 lab in CHINA, People's Republic
2 labs in COTE D'IVOIRE
1 lab in EGYPT
1 lab in FINLAND
3 labs in FRANCE
2 labs in GERMANY
3 labs in GREECE
1 lab in HUNGARY
1 lab in IRELAND
4 labs in ITALY
1 lab in JORDAN
1 lab in KENYA
1 lab in LITHUANIA
2 labs in MALAYSIA
5 labs in NETHERLANDS
1 lab in POLAND
1 lab in PORTUGAL
1 lab in ROMANIA
3 labs in RUSSIAN FEDERATION
1 lab in SERBIA
1 lab in SINGAPORE
1 lab in SLOVAKIA
1 lab in SPAIN
3 labs in TURKEY
4 labs in UNITED KINGDOM
2 labs 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

1   iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
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7   P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
8   J.N. Miller, Analyst, 118, 455, (1993)
9   Analytical Methods Committee, Technical Brief, No 4, January 2001
10   P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)
11   W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
12   Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)