Results of Proficiency Test
Bitumen
December 2019

Organised 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 Bitumen every year. During the annual proficiency testing program 2019/2020, it was decided to continue the round robin for the analysis of Bitumen based on the scope of the latest specification of EN12591 Paving Grade.
In this interlaboratory study 48 laboratories in 27 different countries registered for participation. See appendix 2 for the number of participants per country.
In this report the results of the 2019 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, 1x 2.5 L can of sample #19270 grade 70/100. 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

In this proficiency test one batch of Bitumen grade 70/100 was obtained from a third party. Approximately 65 subsamples of Bitumen grade 70/100 were supplied in 2.5 L cans and labelled #19270. The homogeneity of the subsamples #19270 was checked by determination of Softening Point in accordance with EN1427 on 4 stratified randomly selected samples.

	Softening Point (Ring and Ball) in °C
Sample #19270-1	46.4
Sample #19270-2	47.2
Sample #19270-3	46.8
Sample #19270-4	46.6

Table 1: homogeneity test results of subsamples #19270

From the above test results the repeatability was calculated and compared with the repeatability 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)	0.96
reference test method	EN1427:15
r (reference test method)	1.0

Table 2: evaluation of the repeatability of subsamples #19270

The calculated repeatability was in agreement with the repeatability 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 #19270 was sent on November 20, 2019. 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 #19270: 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 & 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 appropriate reference test methods that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the 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 reanalysis). Additional or corrected test results are used for data analyis and the original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

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

According to ISO5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

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

3.2 GRAPHICS

In order to 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 was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. EN reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation of 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. In some cases, a reproducibility based on former iis proficiency tests could be used.

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 major problems were encountered with the dispatch of the samples.
Three participants did not report any test results at all and one participant reported test results after the final reporting date. Not all participants were able to report all analyzes requested. Finally, 45 participants reported in total 310 numerical test results. Observed were 11 statistically outlying test results, which is 3.5%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

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

4.1 EVALUATION PER TEST

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

Sample #19270

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

Dynamic Viscosity at 60°C: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of EN12596:14.

Flash Point C.O.C.: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ISO2592:17 or ASTM D92:18.

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

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

Penetration at 25°C: This determination was problematic depending on the test method used. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of EN1426:15, but is in agreement with the less strict 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 sufficient 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 problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of EN12591:09.

RTFOT at 163°C: Four prarameters were determined: Change of Mass, Retained Penetration, Viscosity Ratio and Increase in Softening Point.

The determination on Change of Mass was problematic. Three 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. However, the calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of EN12607-1:14.

For the determination on Viscosity Ratio few participants (seven) reported test results. No statistical outliers were observed. However, due to the large variation in the test results it was decided not to calculate z-scores.

The determination on Increase in Softening Point 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.

Softening Point (Ring & Ball): This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of EN1427:15 and in agreement with the requirements of ASTM D36/36M:14e1.

Solubility in Xylene: Seventeen participants reported test results. No statistical outliers were observed. However, 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 relevant reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average result, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from literature reference test methods (in casu EN and ASTM standards) are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Density at 25°C	kg/m^3	30	1026.3	5.7	5
Dynamic Viscosity at 60°C	Pa.s	16	216.9	36.6	21.7
Flash Point C.O.C.	°C	24	292	24	18
Fraass Breaking Point	°C	13	-17.7	5.6	6
Kinematic Viscosity at 135°C	mm^2/s	16	390.7	43.2	23.4
Penetration at 25°C	0.1 mm	45	83.7	9.4	5.0
Penetration Index		23	-0.65	0.74	0.5
RTFOT - Change of Mass	%	22	-0.72	0.41	0.20
RTFOT - Retained Penetration	%	22	49.8	10.3	10
RTFOT - Viscosity Ratio		7	6.35	8.09	(1.27)
RTFOT - Increase in Soft. Point	°C	21	11.5	2.8	2.0
Softening Point (Ring & Ball)	°C	43	47.3	2.0	2.0
Solubility in Xylene	%M/M	17	99.62	1.00	(0.15)

Table 3: performance evaluation sample #19270

Results between brackets should be used with care

Without further statistical calculations it can be concluded that for a number of tests there is no good compliance of the group of participating laboratories with the relevant reference test methods. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2019 WITH PREVIOUS PTS

	December 2019	December 2018	December 2017	December 2016	December 2015
Number of reporting laboratories	45	37	50	51	35
Number of test results	310	511	289	318	388
Number of statistical outliers	11	15	7	8	30
Percentage statistical outliers	3.5%	2.9%	2.4%	2.5%	7.7%

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

Parameter	2019	2018		2017	2016	2015	
	#19270	#18260	#18261	#17260	#16275	#15255	#15256
Paving Grade	70/100	70/100	35/50	70/100	50/70	70/100	50/70
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.	n.e.
RTFOT - Increase in Soft. Point	-	+/-	+/-	+	+	+	+/-
Softening Point (Ring and Ball)	+/-	+	+	-	-	+	+
Solubility in Xylene	(--)	+/-	+	n.e.	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:

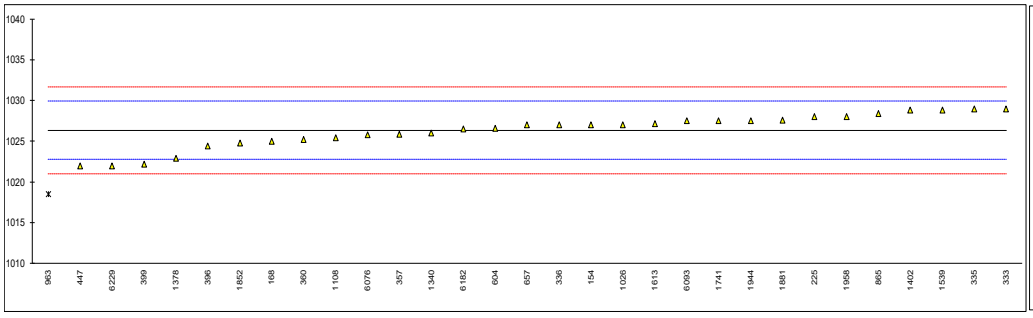
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test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

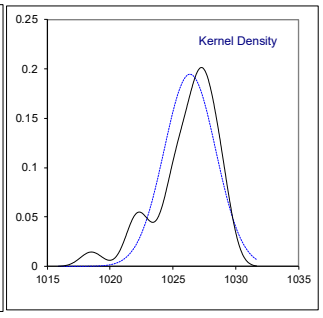
APPENDIX 1

Determination of Density at 25°C on sample #19270; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
154	D70	1027		0.37	
168	D70	1025		-0.75	
225	D70	1028		0.93	
333	EN15326	1029		1.49	
335	EN15326	1029		1.49	
336	EN15326	1027	C	0.37	first reported 1.027 kg/m^3
353		-----		-----	
357	D70	1025.9		-0.24	
360	EN15326	1025.21		-0.63	
396	ISO3838	1024.4		-1.08	
398		-----		-----	
399	D70	1022.2		-2.32	
444		-----		-----	
447	D70	1022		-2.43	
604	D70	1026.6		0.15	
657	D70	1027		0.37	
865	JTG E20/T0603	1028.4	C	1.16	first reported 1.0284 kg/m^3
963	D70	1018.5	C,R(0.05)	-4.39	first reported 1.0185 kg/m^3
1011		-----		-----	
1026		1027		0.37	
1040		-----		-----	
1047		-----		-----	
1108	EN15326	1025.4		-0.52	
1340	EN15326	1026		-0.19	
1378	D70	1022.9		-1.92	
1385		-----		-----	
1402	ISO3838	1028.8		1.38	
1539	EN15326	1028.8		1.38	
1613	DIN51757	1027.2		0.48	
1631		-----		-----	
1724		-----		-----	
1730		-----		-----	
1741	EN15326	1027.537		0.67	
1833		-----		-----	
1849		-----		-----	
1852		1024.8		-0.86	
1881	D70	1027.6		0.71	
1944	EN15326	1027.54		0.67	
1958	ASTM	1028		0.93	
6054		-----		-----	
6076	EN15326	1025.8		-0.30	
6093	EN15326	1027.5		0.65	
6094		-----		-----	
6152		-----		-----	
6182	D70	1026.5		0.09	
6228		-----		-----	
6229	EN15326	1022.0	C	-2.43	first reported 1.022 kg/m^3
6277		-----		-----	

normality	OK
n	30
outliers	1
mean (n)	1026.34
st.dev. (n)	2.049
R(calc.)	5.74
st.dev.(EN15326:07)	1.786
R(EN15326:07)	5

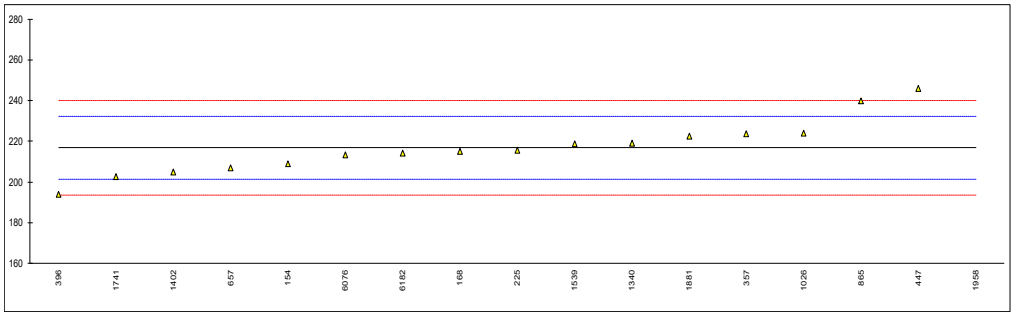


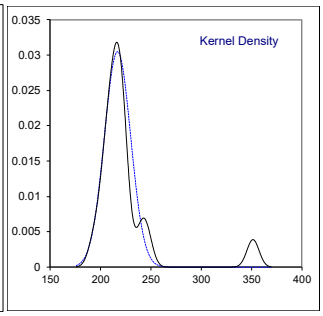


Determination of Dynamic Viscosity at 60°C on sample #19270; results in Pa.s

lab	method	value	mark	z(targ)	remarks
154	D2171	209		-1.02	
168	D2171	215		-0.24	
225	EN12596	215.7		-0.15	
333		-----		-----	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357	EN12596	223.7		0.88	
360		-----		-----	
396	EN12596	194		-2.95	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447	EN13302	246		3.76	
604		-----		-----	
657	D2171	207		-1.27	
865	JTG E20/T0620	240.0		2.99	
963		-----		-----	
1011		-----		-----	
1026	EN12596	224		0.92	
1040		-----		-----	
1047		-----		-----	
1108		-----		-----	
1340	EN12596	219		0.28	
1378		-----		-----	
1385		-----		-----	
1402	EN12596	205		-1.53	
1539	EN12596	218.7		0.24	
1613		-----		-----	
1631		-----		-----	
1724		-----		-----	
1730		-----		-----	
1741	EN12596	202.7		-1.83	
1833		-----		-----	
1849		-----		-----	
1852		-----		-----	
1881	EN12596	222.5		0.73	
1944		-----		-----	
1958	D4402	351.6	C,G(0.01)	17.40	first reported 270.8
6054		-----		-----	
6076	EN13302	213.308		-0.46	
6093		-----		-----	
6094		-----		-----	
6152		-----		-----	
6182	D2171	214.3		-0.33	
6228		-----		-----	
6229		-----		-----	
6277		-----		-----	

normality	OK
n	16
outliers	1
mean (n)	216.87
st.dev. (n)	13.087
R(calc.)	36.64
st.dev.(EN12596:14)	7.745
R(EN12596:14)	21.69

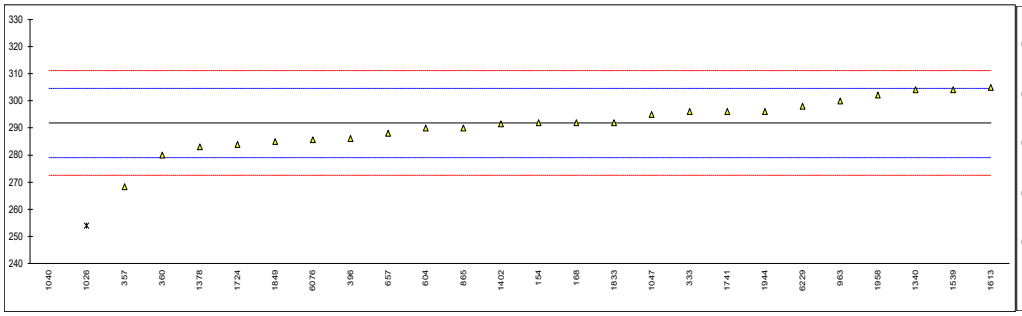


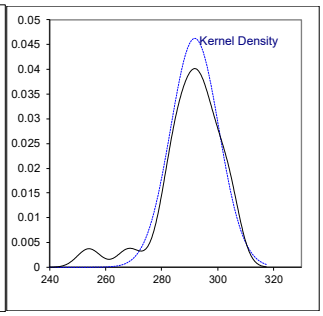


Determination of Flash Point C.O.C. on sample #19270; results in °C

lab	method	mode	value	mark	z(targ)	remarks
154	D92	Automated	292		0.03	
168	D92	Automated	292		0.03	
225		-----	-----		-----	
333	ISO2592	-----	296		0.65	
335		-----	-----		-----	
336		-----	-----		-----	
353		-----	-----		-----	
357	ISO2592	Automated	268.4		-3.64	
360	ISO2592	-----	280		-1.84	
396	ISO2592	Manual	286		-0.90	
398		-----	-----		-----	
399		-----	-----		-----	
444		-----	-----		-----	
447		-----	-----		-----	
604	D92	Manual	290		-0.28	
657	D92	Manual	288		-0.59	
865	JTG E20/T0611	Manual	290		-0.28	
963	D92	-----	300		1.27	
1011		-----	-----		-----	
1026	ISO2592	Manual	254	R(0.01)	-5.88	
1040	ISO2719	-----	208.0	R(0.01)	-13.04	
1047	ISO2592	Manual	295		0.50	
1108		-----	-----		-----	
1340	ISO2592	Automated	304		1.90	
1378	D92	-----	283		-1.37	
1385		-----	-----		-----	
1402	ISO2592	Manual	291.5		-0.05	
1539	ISO2592	Manual	304		1.90	
1613	D92	Manual	305		2.05	
1631		-----	-----		-----	
1724	ISO2592	-----	284		-1.22	
1730		-----	-----		-----	
1741	ISO2592	-----	296.0		0.65	
1833	ISO2592	-----	292		0.03	
1849	ISO2592	-----	285		-1.06	
1852		-----	-----		-----	
1881		-----	-----		-----	
1944	ISO2592	Manual	296		0.65	
1958	D92	Manual	302		1.58	
6054		-----	-----		-----	
6076	ISO2592	Automated	285.7		-0.95	
6093		-----	-----		-----	
6094		-----	-----		-----	
6152		-----	-----		-----	
6182		-----	-----		-----	
6228		-----	-----		-----	
6229	ISO2592	Automated	298		0.96	
6277		-----	-----		-----	

	normality	OK
	n	24
	outliers	2
	mean (n)	291.82
	st.dev. (n)	8.618
	R(calc.)	24.13
	st.dev.(ISO2592:17)	6.429
	R(ISO2592:17)	18
Compare		
	R(D92:18)	18

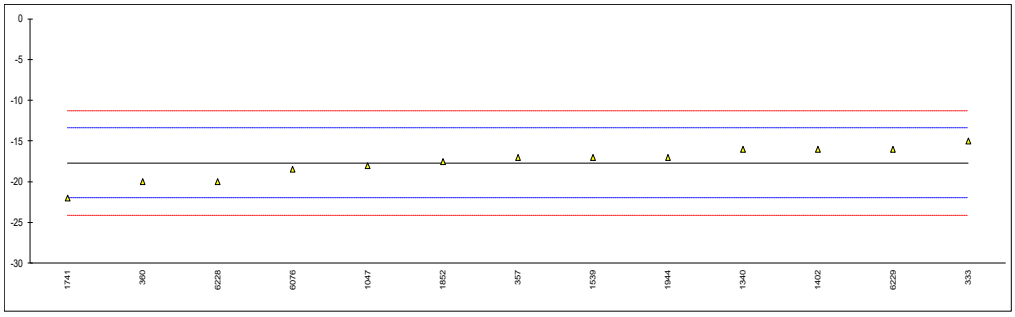


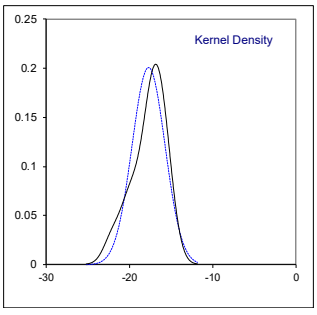


Determination of Fraass Breaking Point on sample #19270; results in °C

lab	method	mode	value	mark	z(targ)	remarks
154		-----	-----		-----	
168		-----	-----		-----	
225		-----	-----		-----	
333	EN12593	-----	-15		1.26	
335		-----	-----		-----	
336		-----	-----		-----	
353		-----	-----		-----	
357	EN12593	Automated	-17		0.32	
360	EN12593	-----	-20		-1.08	
396		-----	-----		-----	
398		-----	-----		-----	
399		-----	-----		-----	
444		-----	-----		-----	
447		-----	-----		-----	
604		-----	-----		-----	
657		-----	-----		-----	
865		-----	-----		-----	
963		-----	-----		-----	
1011		-----	-----		-----	
1026		-----	-----		-----	
1040		-----	-----		-----	
1047	EN12593	Manual	-18		-0.14	
1108		-----	-----		-----	
1340	EN12593	Automated	-16		0.79	
1378		-----	-----		-----	
1385		-----	-----		-----	
1402	EN12593	Manual	-16		0.79	
1539	EN12593	Automated	-17		0.32	
1613		-----	-----		-----	
1631		-----	-----		-----	
1724		-----	-----		-----	
1730		-----	-----		-----	
1741	EN12593	-----	-22.0		-2.01	
1833		-----	-----		-----	
1849		-----	-----		-----	
1852	EN12593	-----	-17.5		0.09	
1881		-----	-----		-----	
1944	EN12593	Manual	-17		0.32	
1958		-----	-----		-----	
6054		-----	-----		-----	
6076	EN12593	Automated	-18.5	C	-0.38	reported -18 / -19
6093		-----	-----		-----	
6094		-----	-----		-----	
6152		-----	-----		-----	
6182		-----	-----		-----	
6228	EN12593	-----	-20		-1.08	
6229	EN12593	Manual	-16		0.79	
6277		-----	-----		-----	

normality	OK
n	13
outliers	0
mean (n)	-17.69
st.dev. (n)	1.985
R(calc.)	5.56
st.dev.(EN12593:15)	2.143
R(EN12593:15)	6

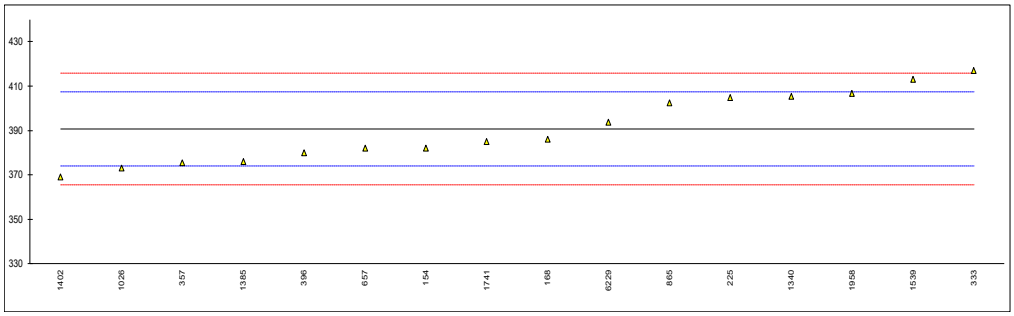


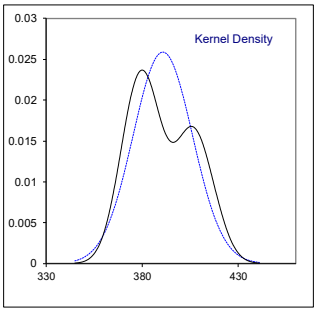


Determination of Kinematic Viscosity at 135°C on sample #19270; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
154	D2170	382		-1.04	
168	D2170	386		-0.57	
225	EN12595	404.9		1.69	
333	EN12595	417		3.14	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357	EN12595	375.6		-1.81	
360		-----		-----	
396	EN12595	380		-1.28	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2170	382		-1.04	
865	JTG E20/T0619	402.5		1.41	
963		-----		-----	
1011		-----		-----	
1026	EN12595	373		-2.12	
1040		-----		-----	
1047		-----		-----	
1108		-----		-----	
1340	EN12595	405.4		1.75	
1378		-----		-----	
1385	EN12595	375.9		-1.77	
1402	EN12595	369		-2.60	
1539	EN12595	413.0		2.66	
1613		-----		-----	
1631		-----		-----	
1724		-----		-----	
1730		-----		-----	
1741	EN12595	384.9		-0.70	
1833		-----		-----	
1849		-----		-----	
1852		-----		-----	
1881		-----		-----	
1944		-----		-----	
1958	D445	406.7		1.91	
6054		-----		-----	
6076		-----		-----	
6093		-----		-----	
6094		-----		-----	
6152		-----		-----	
6182		-----		-----	
6228		-----		-----	
6229	EN12595	393.8		0.37	
6277		-----		-----	

			EN12595 only:
	normality	OK	OK
	n	16	11
	outliers	0	0
	mean (n)	390.73	390.23
	st.dev. (n)	15.415	17.301
	R(calc.)	43.16	48.44
	st.dev.(EN12595:14)	8.373	8.362
	R(EN12595:14)	23.44	23.41
Compare			
	R(D2170/D2170M:18)	34.38	

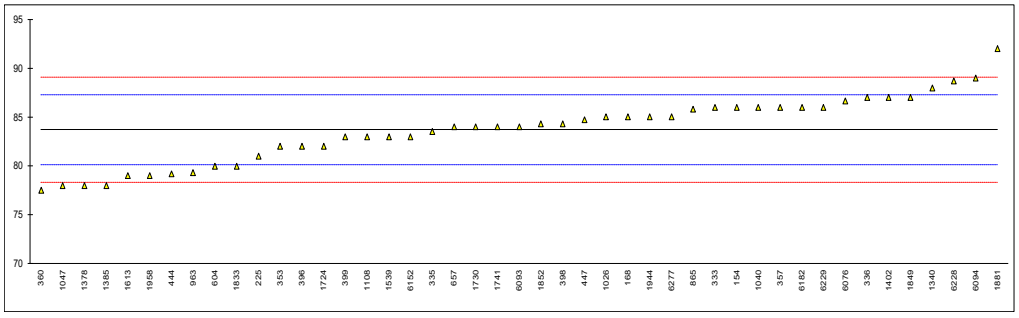


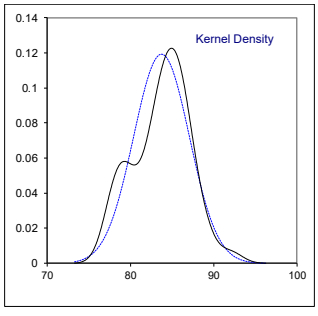


Determination of Penetration at 25°C on sample #19270; results in 0.1 mm

lab	method	mode	value	mark	z(targ)	remarks
154	D5	Manual	86		1.28	
168	D5	Manual	85		0.72	
225	D5	Manual	81		-1.51	
333	EN1426		86		1.28	
335	EN1426	Automated	83.5		-0.12	
336	EN1426		87		1.83	
353	EN1426		82		-0.95	
357	EN1426	Automated	86		1.28	
360	EN1426		77.5		-3.46	
396	EN1426	Manual	82		-0.95	
398	EN1426	Manual	84.33		0.35	
399	EN1426	Manual	83		-0.40	
444	EN1426		79.2		-2.51	
447	EN1426	Automated	84.7		0.55	
604	D5	Manual	80		-2.07	
657	D5	Manual	84		0.16	
865	JTG E20/T0604	Manual	85.8		1.16	
963	D5		79.3		-2.46	
1011			-----		-----	
1026	EN1426	Manual	85		0.72	
1040	EN1426	Automated	86.0		1.28	
1047	EN1426	Manual	78		-3.18	
1108	EN1426	Automated	83.0		-0.40	
1340	EN1426	Automated	88		2.39	
1378	D5		78		-3.18	
1385	EN1426	Automated	78		-3.18	
1402	EN1426	Manual	87		1.83	
1539	EN1426	Manual	83		-0.40	
1613	D5	Automated	79		-2.63	
1631			-----		-----	
1724	EN1426		82		-0.95	
1730	EN1426		84		0.16	
1741	EN1426		84.0		0.16	
1833	EN1426		80		-2.07	
1849	EN1426	Automated	87		1.83	
1852	EN1426	Automated	84.3		0.33	
1881	EN1426	Automated	92.0		4.62	
1944	EN1426	Automated	85		0.72	
1958	D5	Manual	79		-2.63	
6054			-----		-----	
6076	EN1426	Automated	86.67	C	1.65	reported 87 / 87 / 86
6093	EN1426	Automated	84		0.16	
6094	EN1426	Automated	89		2.95	
6152	EN1426	Automated	83		-0.40	
6182	D5		86		1.28	
6228	EN1426	Automated	88.67		2.76	
6229	EN1426	Automated	86		1.28	
6277	D5	Manual	85		0.72	

			EN1426 only:	D5/D5M only:
	normality	OK	OK	OK
	n	45	33	11
	outliers	0	0	0
	mean (n)	83.71	84.21	82.03
	st.dev. (n)	3.343	3.296	3.167
	R(calc.)	9.36	9.23	8.87
	st.dev.(EN1426:15)	1.794	1.804	-----
	R(EN1426:15)	5.02	5.05	-----
Compare				
	R(D5/5M:19a)	10.43	-----	10.19

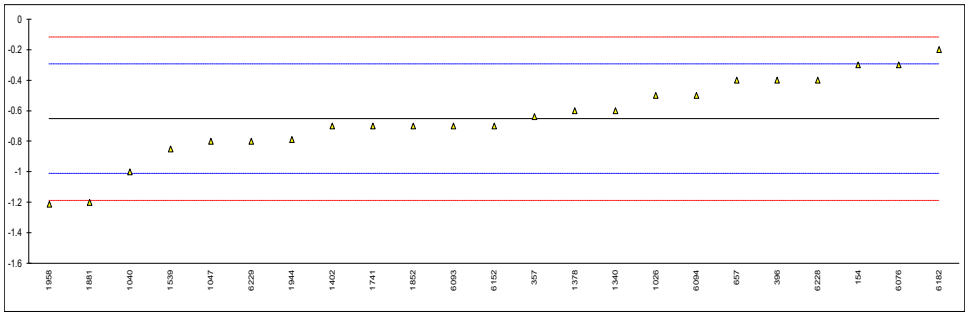


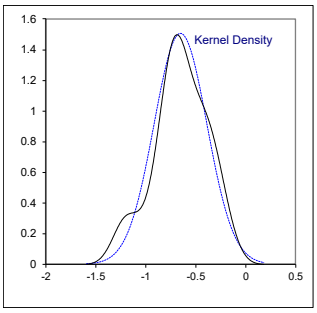


Determination of Penetration Index on sample #19270;

lab	method	value	mark	z(targ)	remarks
154	EN12591	-0.3		1.97	
168		-----		-----	
225		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357	EN12591	-0.64		0.07	
360		-----		-----	
396	EN12591	-0.40		1.41	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	Calculation	-0.4		1.41	
865		-----		-----	
963		-----		-----	
1011		-----		-----	
1026	EN12591	-0.5		0.85	
1040	EN12591	-1.0		-1.95	
1047	EN12591	-0.8		-0.83	
1108		-----		-----	
1340	EN12591	-0.6		0.29	
1378	EN12591	-0.6		0.29	
1385		-----		-----	
1402	EN12591	-0.7	E	-0.27	iis calculated -0.78
1539	EN12591	-0.85		-1.11	
1613		-----		-----	
1631		-----		-----	
1724		-----		-----	
1730		-----		-----	
1741	EN12591	-0.70		-0.27	
1833		-----		-----	
1849		-----		-----	
1852	EN12591	-0.70		-0.27	
1881	EN12591	-1.2	E	-3.07	iis calculated -0.86
1944	EN12591	-0.788		-0.76	
1958	EN12591	-1.21		-3.13	
6054		-----		-----	
6076	EN12591	-0.3		1.97	
6093	EN12591	-0.7		-0.27	
6094	EN12591	-0.5		0.85	
6152	EN12591	-0.7		-0.27	
6182	EN12591	-0.2	E	2.53	iis calculated -0.29
6228	EN12591	-0.4		1.41	
6229	EN12591	-0.8		-0.83	
6277		-----		-----	

normality	OK
n	23
outliers	0
mean (n)	-0.652
st.dev. (n)	0.2646
R(calc.)	0.741
st.dev.(EN12591:09)	0.1786
R(EN12591:09)	0.5

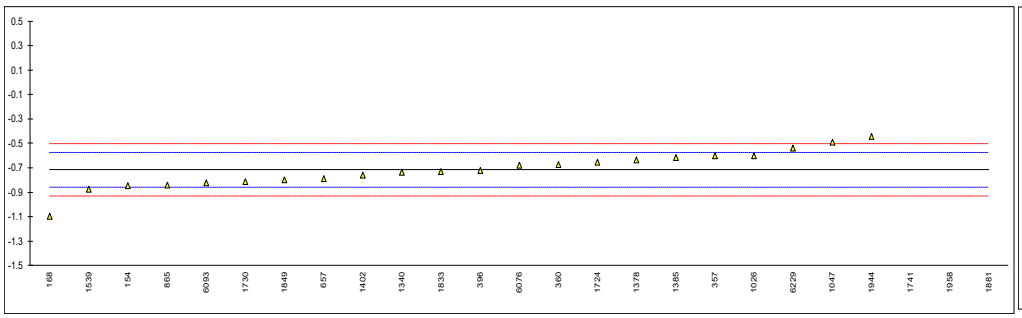


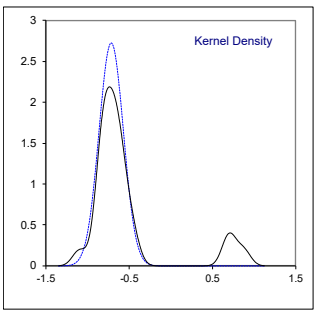


Determination of RTFOT at 163°C, Change of Mass on sample #19270; results in %

lab	method	value	mark	z(targ)	remarks
154	D2872	-0.847		-1.83	
168	D2872	-1.098		-5.34	
225		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357	EN12607-1	-0.601		1.61	
360	EN12607-1	-0.675		0.58	
396	EN12607-1	-0.72		-0.05	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2872	-0.79		-1.03	
865	JTG E20/T0610	-0.842		-1.76	
963		-----		-----	
1011		-----		-----	
1026	EN12607-1	-0.60		1.63	
1040		-----		-----	
1047	EN12607-1	-0.49		3.17	
1108		-----		-----	
1340	EN12607-1	-0.73655		-0.28	
1378	EN12607-1	-0.635		1.14	
1385	EN12607-1	-0.614		1.43	
1402	EN12607-1	-0.76		-0.61	
1539	EN12607-1	-0.874		-2.21	
1613		-----		-----	
1631		-----		-----	
1724	EN12607-1	-0.652		0.90	
1730	EN12607-1	-0.814		-1.37	
1741	EN12607-1	0.69	C,R(0.01)	19.69	first reported 0.760
1833	EN12607-1	-0.73		-0.19	
1849	EN12607-1	-0.8		-1.17	
1852		-----		-----	
1881	EN12607-1	0.875	C,R(0.05)	22.28	first reported 0.78
1944	EN12607-1	-0.44		3.87	
1958	D2872	0.71	C,R(0.05)	19.97	first reported 28.03
6054		-----		-----	
6076	EN12607-1	-0.68		0.51	
6093	EN12607-1	-0.82		-1.45	
6094		-----		-----	
6152		-----		-----	
6182		-----		-----	
6228		-----		-----	
6229	EN12607-1	-0.54		2.47	
6277		-----		-----	

normality	suspect
n	22
outliers	3
mean (n)	-0.7163
st.dev. (n)	0.14612
R(calc.)	0.4091
st.dev.(EN12607-1:14)	0.07143
R(EN12607-1:14)	0.20

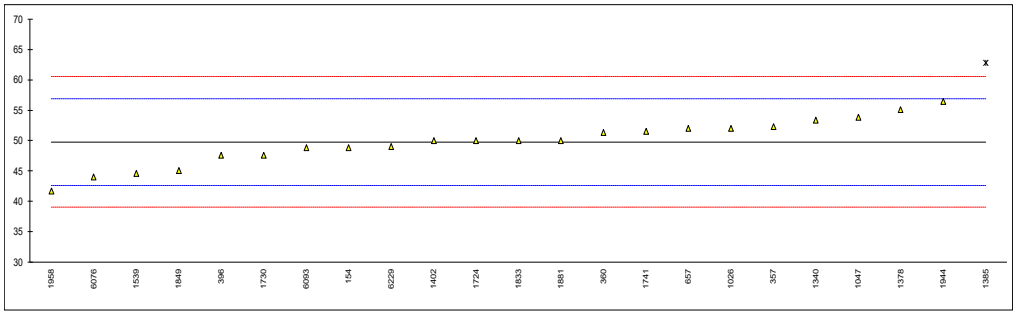


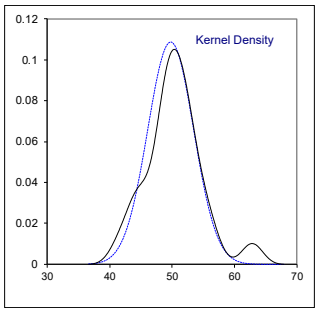


Determination of RTFOT at 163°C, Retained Penetration on sample #19270; results in %

lab	method	value	mark	z(targ)	remarks
154	D2872	48.84		-0.26	
168		-----		-----	
225		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357	EN12607-1	52.3		0.71	
360	EN12607-1	51.3		0.43	
396	EN12607-1	47.57		-0.62	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2872	52		0.62	
865		-----		-----	
963		-----		-----	
1011		-----		-----	
1026	EN12607-1	52		0.62	
1040		-----		-----	
1047	EN12607-1	53.8		1.13	
1108		-----		-----	
1340	EN12607-1	53.40		1.02	
1378	EN12607-1	55.1		1.49	
1385	EN12607-1	62.8	R(0.05)	3.65	
1402	EN12607-1	50		0.06	
1539	EN12607-1	44.6		-1.45	
1613		-----		-----	
1631		-----		-----	
1724	EN12607-1	50		0.06	
1730	EN12607-1	47.6		-0.61	
1741	EN12607-1	51.50		0.48	
1833	EN12607-1	50		0.06	
1849	EN12607-1	45.1		-1.31	
1852		-----		-----	
1881	EN12607-1	50		0.06	
1944	EN12607-1	56.4		1.86	
1958	D2872	41.7	C	-2.26	first reported 39.0
6054		-----		-----	
6076	EN12607-1	44	C	-1.62	reported 43 / 44 / 45
6093	EN12607-1	48.8		-0.27	
6094		-----		-----	
6152		-----		-----	
6182		-----		-----	
6228		-----		-----	
6229	EN12607-1	49		-0.22	
6277		-----		-----	

normality	OK
n	22
outliers	1
mean (n)	49.77
st.dev. (n)	3.668
R(calc.)	10.27
st.dev.(EN12607-1:14)	3.571
R(EN12607-1:14)	10

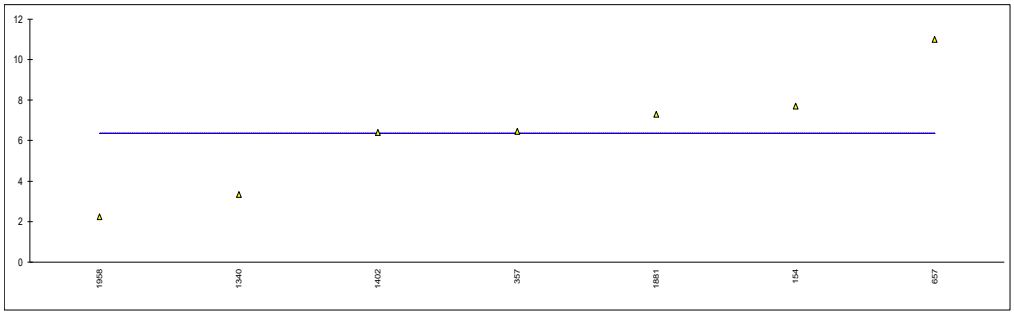


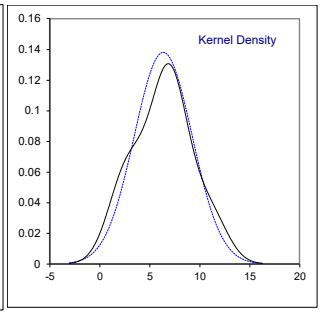


Determination of RTFOT at 163°C, Viscosity Ratio on sample #19270;

lab	method	value	mark	z(targ)	remarks
154	D2872	7.70		-----	
168		-----		-----	
225		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357	EN12607-1	6.46		-----	
360		-----		-----	
396		-----		-----	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2872	10.99		-----	
865		-----		-----	
963		-----		-----	
1011		-----		-----	
1026		-----		-----	
1040		-----		-----	
1047		-----		-----	
1108		-----		-----	
1340	EN12607-1	3.357		-----	
1378		-----		-----	
1385		-----		-----	
1402	EN12607-1	6.41		-----	
1539		-----		-----	
1613		-----		-----	
1631		-----		-----	
1724		-----		-----	
1730		-----		-----	
1741		-----		-----	
1833		-----		-----	
1849		-----		-----	
1852		-----		-----	
1881	EN12607-1	7.3		-----	
1944		-----		-----	
1958	D2872	2.24		-----	
6054		-----		-----	
6076		-----		-----	
6093		-----		-----	
6094		-----		-----	
6152		-----		-----	
6182		-----		-----	
6228		-----		-----	
6229		-----		-----	
6277		-----		-----	

normality	unknown
n	7
outliers	0
mean (n)	6.351
st.dev. (n)	2.8902
R(calc.)	8.093
st.dev.(EN12607-1:14)	(0.4536)
R(EN12607-1:14)	(1.270)

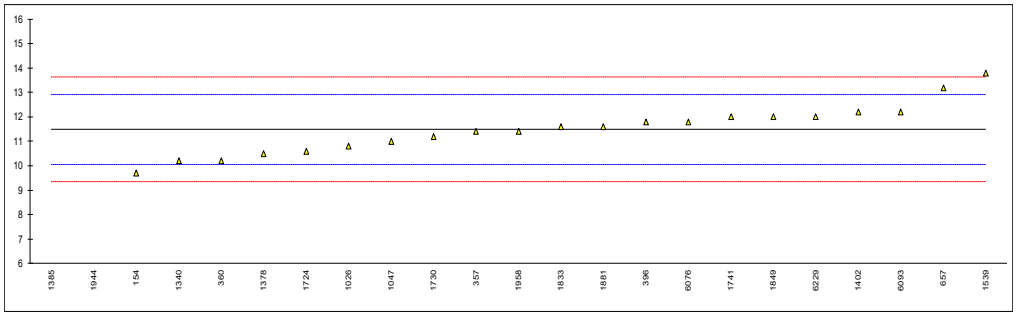


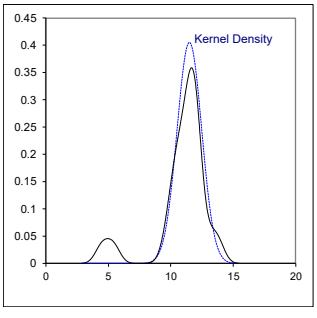


Determination of RTFOT at 163°C, Increase in Softening Point on sample #19270; results in °C

lab	method	value	mark	z(targ)	remarks
154	D2872	9.7		-2.50	
168		-----		-----	
225		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357	EN12607-1	11.4		-0.12	
360	EN12607-1	10.20		-1.80	
396	EN12607-1	11.8		0.44	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2872	13.2		2.40	
865		-----		-----	
963		-----		-----	
1011		-----		-----	
1026	EN12607-1	10.8		-0.96	
1040		-----		-----	
1047	EN12607-1	11.0		-0.68	
1108		-----		-----	
1340	EN12607-1	10.2		-1.80	
1378	EN12607-1	10.5		-1.38	
1385	EN12607-1	4.5	R(0.01)	-9.78	
1402	EN12607-1	12.2		1.00	
1539	EN12607-1	13.8		3.24	
1613		-----		-----	
1631		-----		-----	
1724	EN12607-1	10.6		-1.24	
1730	EN12607-1	11.2		-0.40	
1741	EN12607-1	12.0		0.72	
1833	EN12607-1	11.6		0.16	
1849	EN12607-1	12.0		0.72	
1852		-----		-----	
1881	EN12607-1	11.6	C	0.16	first reported 11.2
1944	EN12607-1	5.4	C,R(0.01)	-8.52	first reported 8.4
1958	D2872	11.4		-0.12	
6054		-----		-----	
6076	EN12607-1	11.8		0.44	
6093	EN12607-1	12.2		1.00	
6094		-----		-----	
6152		-----		-----	
6182		-----		-----	
6228		-----		-----	
6229	EN12607-1	12		0.72	
6277		-----		-----	

normality	OK
n	21
outliers	2
mean (n)	11.49
st.dev. (n)	0.985
R(calc.)	2.76
st.dev.(EN12607-1:14)	0.714
R(EN12607-1:14)	2.0

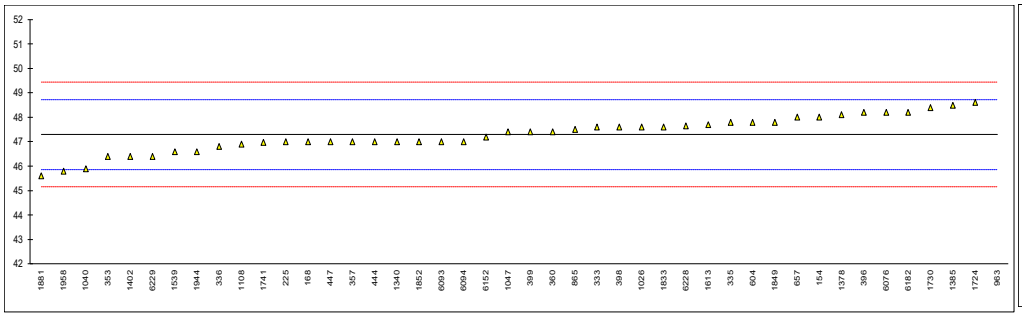


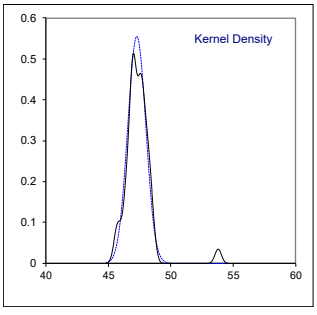


Determination of Softening Point (Ring & Ball) on sample #19270; results in °C

lab	method	value	mark	z(targ)	remarks
154	D36	48		0.99	
168	D36	47		-0.41	
225	D36	47.0		-0.41	
333	EN1427	47.6		0.43	
335	EN1427	47.8		0.71	
336	EN1427	46.8		-0.69	
353	EN1427	46.4		-1.25	
357	EN1427	47.0		-0.41	
360	EN1427	47.4		0.15	
396	EN1427	48.2		1.27	
398	EN1427	47.6		0.43	
399	EN1427	47.4		0.15	
444	EN1427	47.0		-0.41	
447	EN1427	47.0		-0.41	
604	D36	47.8		0.71	
657	D36	48.0		0.99	
865	JTG E20/T0606	47.5		0.29	
963	D36	53.8	C,R(0.01)	9.11	first reported 43.8
1011		-----		-----	
1026	EN1427	47.6		0.43	
1040	EN1427	45.90		-1.95	
1047	EN1427	47.4		0.15	
1108	EN1427	46.9		-0.55	
1340	EN1427	47.0		-0.41	
1378	D36	48.1		1.13	
1385	EN1427	48.5		1.69	
1402	EN1427	46.4		-1.25	
1539	EN1427	46.6		-0.97	
1613	D36	47.7		0.57	
1631		-----		-----	
1724	EN1427	48.6		1.83	
1730	EN1427	48.4		1.55	
1741	EN1427	46.97		-0.45	
1833	EN1427	47.6		0.43	
1849	EN1427	47.8		0.71	
1852	EN1427	47.0		-0.41	
1881	EN1427	45.6	C	-2.37	first reported 44.6
1944	EN1427	46.6		-0.97	
1958	D36	45.8		-2.09	
6054		-----		-----	
6076	EN1427	48.2	C	1.27	reported 48.2 / 48.2
6093	EN1427	47.0		-0.41	
6094	EN1427	47.0		-0.41	
6152	EN1427	47.2		-0.13	
6182	IP58	48.2		1.27	
6228	EN1427	47.65		0.50	
6229	EN1427	46.4		-1.25	
6277		-----		-----	

	normality	OK
	n	43
	outliers	1
	mean (n)	47.293
	st.dev. (n)	0.7188
	R(calc.)	2.013
	st.dev.(EN1427:15)	0.7143
	R(EN1427:15)	2.0
Compare		
	R(D36/36M:14e1)	9.32 Automated electronic thermometer
	R(D36/36M:14e1)	9.63 Mercury thermometer

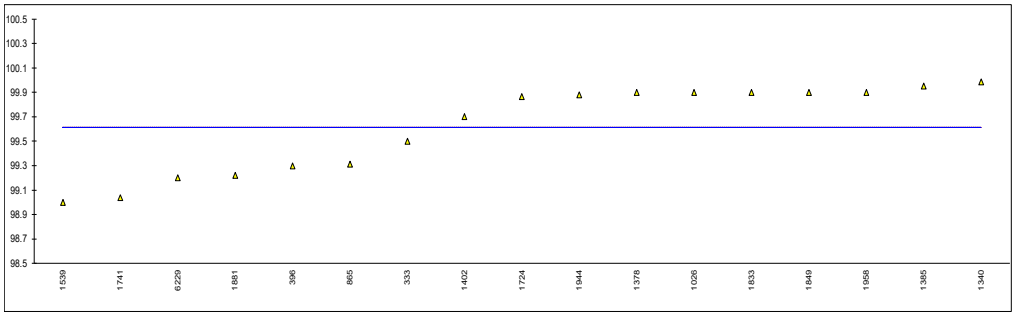


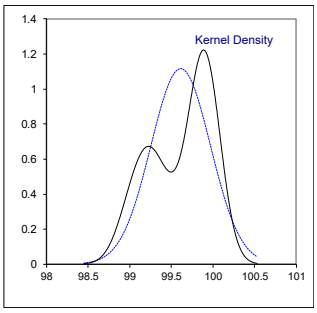


Determination of Solubility in Xylene on sample #19270; results in %M/M

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225		-----		-----	
333	EN12592	99.50		-----	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357		-----		-----	
360		-----		-----	
396	EN12592	99.30		-----	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657		-----		-----	
865	JTG E20/T0607	99.31		-----	
963		-----		-----	
1011		-----		-----	
1026	EN12592	99.9		-----	
1040		-----		-----	
1047		-----		-----	
1108		-----		-----	
1340	EN12592	99.985		-----	solubility in Toluene
1378	EN12592	99.9		-----	
1385	EN12592	99.95		-----	
1402	EN12592	99.70		-----	
1539	EN12592	99.0		-----	
1613		-----		-----	
1631		-----		-----	
1724	EN12592	99.8655		-----	
1730		-----		-----	
1741	EN12592	99.037		-----	
1833	EN12592	99.90		-----	
1849	EN12592	99.90		-----	
1852		-----		-----	
1881	EN12592	99.22		-----	
1944	EN12592	99.88		-----	
1958	D2042	99.9		-----	
6054		-----		-----	
6076		-----		-----	
6093		-----		-----	
6094		-----		-----	
6152		-----		-----	
6182		-----		-----	
6228		-----		-----	
6229	EN12592	99.2		-----	
6277		-----		-----	

normality	OK
n	17
outliers	0
mean (n)	99.615
st.dev. (n)	0.3572
R(calc.)	1.000
st.dev.(EN12592:14)	(0.0536)
R(EN12592:14)	(0.15)





APPENDIX 2

Number of participants per country

1 lab in	BOSNIA and HERZEGOVINA
1 lab 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	HONG KONG
1 lab in	IRELAND
4 labs in	ITALY
1 lab in	JORDAN
1 lab in	LEBANON
1 lab in	LITHUANIA
1 lab in	MALAYSIA
6 labs in	NETHERLANDS
2 labs in	POLAND
1 lab in	PORTUGAL
1 lab in	ROMANIA
1 lab in	SAUDI ARABIA
1 lab in	SERBIA
1 lab in	SINGAPORE
4 labs in	TURKEY
1 lab in	UNITED ARAB EMIRATES
3 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 = possibly an error in calculations
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
SDS = Safety Data Sheet

Literature

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