Institute for
Interlaboratory Studies

Results of Proficiency Test
Bitumen
November 2022

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2014 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Bitumen in accordance with the latest version of EN12591 Paving Grade every year. During the annual proficiency testing program 2022/2023 it was decided to continue the round robin for the analysis of Bitumen.

In this interlaboratory study 47 laboratories in 27 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 35/50 in a 2.5 liter can labelled #22240.
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 70 subsamples of Bitumen grade 35/50 in 2.5 L metal cans was obtained from a local supplier and labelled #22240.
The homogeneity of the subsamples was checked by determination of Penetration at 25 °C in accordance with EN1426 and Softening Point in accordance with EN1427 on 4 stratified randomly selected subsamples.

	Softening Point (Ring and Ball) in °C	Penetration in 0.1 mm
sample #22240-1	54.4	44
sample #22240-2	54.4	44
sample #22240-3	55.0	44
sample #22240-4	54.4	45

Table 1: homogeneity test results of subsamples #22240

From the above test results the relative standard deviations (RSD) were calculated and compared with 0.3 times the corresponding average relative standard deviation obtained from two iis PTs with grade 35/50 Bitumen conducted in 2018 and 2020 in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Softening Point (Ring and Ball)	Penetration
RSD% (observed)	0.5	1.1
reference method	iis PTs	iis PTs
0.3 x RSD% (reference method)	0.4	2.0

Table 2: evaluation of the repeatability of subsamples #22240

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

To each of the participating laboratories one 2.5 L can of sample #22240 was sent on November 8, 2022. 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 requested to determine: Density at 25 °C, Dynamic Viscosity at 60 °C, Flash Point C.O.C., Fraass Breaking Point, Kinematic Viscosity at 135 °C, Penetration at 25 °C, Penetration Index, RTFOT at 163 °C (Change of Mass, Retained Penetration, Viscosity Ratio and Increase in Softening Point), Softening Point (Ring and Ball), Solubility in Xylene and Ductility.

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

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. 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 some problems were encountered with the dispatch of the samples. Six participants reported test results after the final reporting date and two other participants did not report any test results. Not all participants were able to report all tests requested. Finally, 45 participants reported in total 318 numerical test results. Observed were 15 outlying test results, which is 4.7%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

All data sets proved to have a normal Gaussian distribution.

4.1 EVALUATION PER TEST

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

In the iis PT reports ASTM test methods are referred to with a number (e.g. D36) and an added designation for the year that the test method was adopted or revised (e.g. D36:14). When a method has been reapproved an “R” will be added and the year of approval (e.g. D36:14R20).

Density at 25 °C: 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 EN15326:07+A1:09.

Dynamic Viscosity at 60 °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 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 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 D2170:18.

Penetration at 25 °C: This determination may be problematic depending on the test method used. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of EN1426:15 but is in full agreement with the requirements of ASTM D5:20.
When the test results of test method EN1426 and ASTM D5 are evaluated separately, the reproducibility for the EN1426 test results is still not in agreement but it is in agreement for ASTM D5 test results.
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. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of EN12591:09.

RTFOT at 163 °C - Change of Mass: The determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN12607-1:14.

RTFOT at 163 °C - Retained Penetration: The 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 EN12607-1:14.

RTFOT at 163 °C - Viscosity Ratio: The 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 EN12607-1:14.

RTFOT at 163 °C on Increase in Softening Point: The determination 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. Five statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN1427:15 and ASTM D36:14R20.

Solubility in Xylene: 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 EN12595:14.

Ductility: This determination was not problematic. All reporting participants, except one, agreed on a test result >100 cm.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Parameter	unit	n	average	2.8 * sd	R(lit)
Density at 25 °C	kg/m^3	28	1044.2	5.4	5
Dynamic Viscosity at 60 °C	Pa.s	12	709	73	71
Flash Point C.O.C.	°C	23	315	28	18
Fraass Breaking Point	°C	19	-11.3	7.7	6
Kinematic Viscosity at 135 °C	mm^2/s	17	747.6	79.8	67.3
Penetration at 25 °C	0.1 mm	45	44.3	5.5	3
Penetration Index		20	-0.49	0.35	0.5
RTFOT - Change of Mass	%	26	-0.15	0.20	0.2
RTFOT - Retained Penetration	%	24	65.2	9.7	10
RTFOT - Viscosity Ratio		9	3.2	0.8	0.6
RTFOT - Increase in Soft. Point	°C	25	7.1	3.0	4
Softening Point (Ring & Ball)	°C	39	54.1	1.6	2

Parameter	unit	n	average	2.8 * sd	R(lit)
Solubility in Xylene	%M/M	16	99.87	0.16	0.15
Ductility	cm	10	>100	n.a.	n.a.

Table 3: reproducibilities of tests on sample #22240

Without further statistical calculations it can be concluded that for many tests there is a good compliance of the group of participants with the reference test methods. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2022 WITH PREVIOUS PTS

	November 2022	December 2021	December 2020	December 2019	December 2018
Number of reporting laboratories	45	51	50	45	37
Number of test results	318	348	315	310	511
Number of statistical outliers	15	11	14	11	15
Percentage of statistical outliers	4.7%	3.2%	4.4%	3.5%	2.9%

Table 4: comparison with previous proficiency tests

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

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

Parameter	November 2022	December 2021	December 2020	December 2019	December 2018	
Paving Grade	35/50	70/100	35/50	70/100	70/100	35/50
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	+/-	(--)	(--)	(--)	+/-	+

Table 5: comparison determinations to the reference test methods

For results between brackets no z-scores are calculated

In the table above the following performance categories were used:

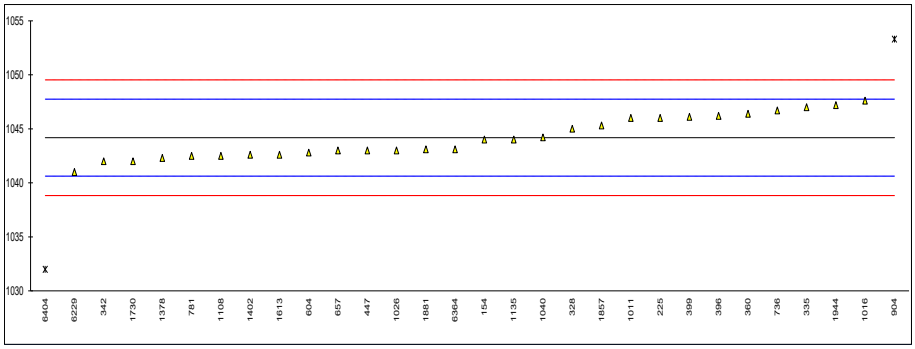
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test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

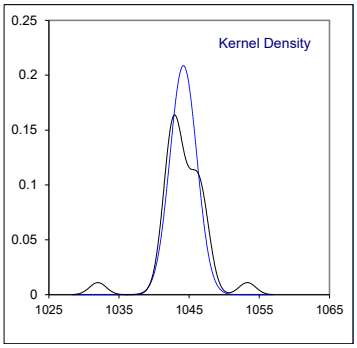
APPENDIX 1

Determination of Density at 25 °C on sample #22240; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
154	D70	1044	C	-0.10	first reported: 1.044 kg/m^3
225	D70	1046		1.02	
328	EN15326	1045	C	0.46	first reported: 1.045 kg/m^3
333		-----		-----	
335	EN15326	1047	C	1.58	first reported: 1.047 kg/m^3
342	D70	1042		-1.22	
357		-----		-----	
360	EN15326	1046.40		1.24	
365		-----		-----	
396	EN15326	1046.2		1.13	
398		-----		-----	
399	D70	1046.1		1.07	
444		-----		-----	
447	D70	1043		-0.66	
604	D70	1042.8	C	-0.78	first reported: 1049.2
657	D70	1043		-0.66	
736	EN15326	1046.7		1.41	
781	EN15326	1042.5		-0.94	
865		-----		-----	
904	D70	1053.3	R(0.01)	5.10	
1011	D70	1046	C	1.02	first reported: 1.046 kg/m^3
1016	EN15326	1047.6	C	1.91	first reported: 1.0476 kg/m^3
1026	EN15326	1043		-0.66	
1040	ISO3838	1044.2		0.01	
1108	EN15326	1042.5	C	-0.94	first reported: 1.0425 kg/m^3
1135	EN15326	1044		-0.10	
1378	EN15326	1042.3		-1.06	
1402	ISO3838	1042.6		-0.89	
1613	DIN51757	1042.6		-0.89	
1631		-----		-----	
1724		-----		-----	
1730	EN15326	1042.0	C	-1.22	first reported: 1.0420 kg/m^3
1833		-----		-----	
1849		-----		-----	
1852		-----		-----	
1857	D70	1045.3		0.62	
1881	EN15326	1043.1		-0.61	
1944	EN15326	1047.2		1.69	
1990		-----		-----	
6037		-----		-----	
6076		-----		-----	
6228		-----		-----	
6229	EN15326	1041		-1.78	
6364	D70	1043.1		-0.61	
6404	ISO12185	1032	R(0.01)	-6.82	
6419		-----		-----	
6474		-----		-----	

normality	OK
n	28
outliers	2
mean (n)	1044.186
st.dev. (n)	1.9113
R(calc.)	5.352
st.dev.(EN15326:07+A1:09)	1.786
R(EN15326: 07+A1:09)	5

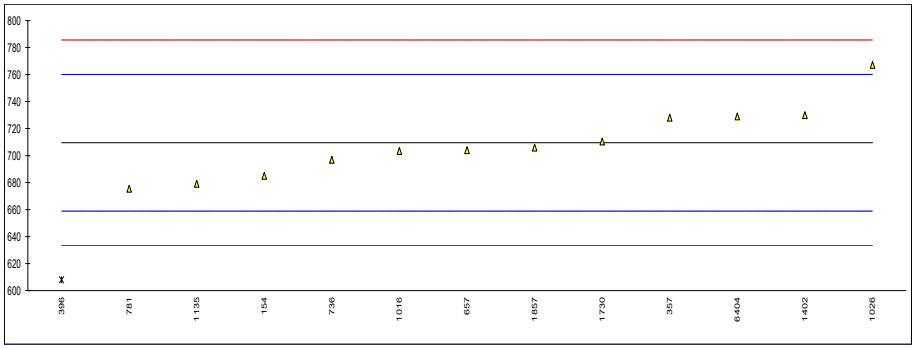


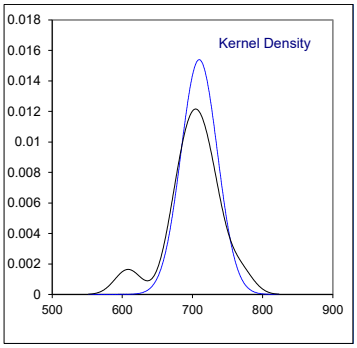


Determination of Dynamic Viscosity at 60 °C on sample #22240; results in Pa.s

lab	method	value	mark	z(targ)	remarks
154	D2171	685		-0.97	
225		-----		-----	
328		-----		-----	
333		-----		-----	
335		-----		-----	
342		-----		-----	
357	EN12596	728		0.73	
360		-----		-----	
365		-----		-----	
396	EN12596	608	G(0.05)	-4.01	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2171	704		-0.22	
736	EN12596	696.8		-0.50	
781	EN12596	675.4		-1.35	
865		-----		-----	
904		-----		-----	
1011		-----		-----	
1016	EN12596	703.36		-0.24	
1026		767		2.27	
1040		-----		-----	
1108		-----		-----	
1135	EN12596	679		-1.20	
1378		-----		-----	
1402	EN12596	730		0.81	
1613		-----		-----	
1631		-----		-----	
1724		-----		-----	
1730	EN12596	710.4		0.04	
1833		-----		-----	
1849		-----		-----	
1852		-----		-----	
1857	D2171	705.9		-0.14	
1881		-----		-----	
1944		-----		-----	
1990		-----		-----	
6037		-----		-----	
6076		-----		-----	
6228		-----		-----	
6229		-----		-----	
6364		-----		-----	
6404	EN12596	729		0.77	
6419		-----		-----	
6474		-----		-----	

normality	OK
n	12
outliers	1
mean (n)	709.49
st.dev. (n)	25.911
R(calc.)	72.55
st.dev.(EN12596:14)	25.339
R(EN12596:14)	70.95

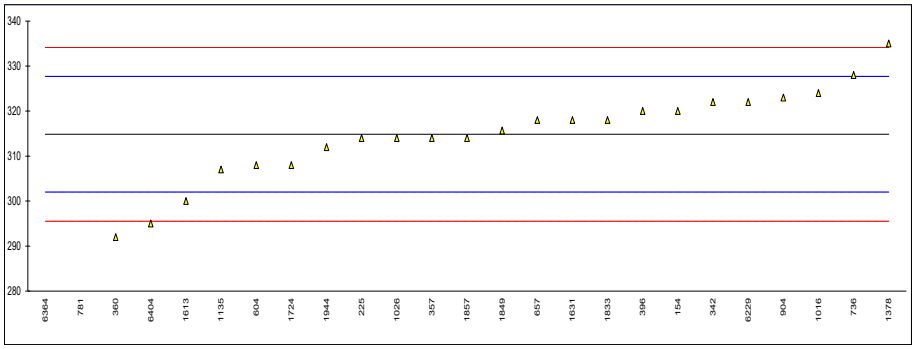


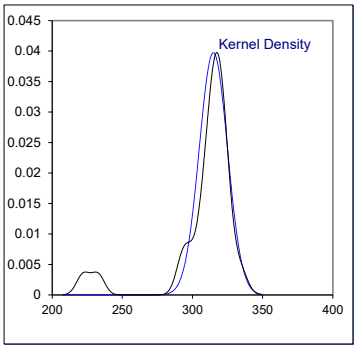


Determination of Flash Point C.O.C. on sample #22240; results in °C

lab	method	value	mark	z(targ)	remarks
154	D92 automated	320		0.80	
225	D92 manual	314.0		-0.13	
328		-----		-----	
333		-----		-----	
335		-----		-----	
342	ISO2592 automated	322		1.11	
357	ISO2592 automated	314		-0.13	
360	ISO2592 automated	292.0		-3.56	
365		-----		-----	
396	ISO2592	320		0.80	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604	D92 manual	308		-1.07	
657	D92 manual	318		0.49	
736	ISO2592 automated	328		2.04	
781	ISO2592	232.5	R(0.01)	-12.81	
865		-----		-----	
904	ISO2592	323		1.27	
1011	ISO2592 automated	>280		-----	
1016	ISO2592 automated	324		1.42	
1026	ISO2592 manual	314		-0.13	
1040		-----		-----	
1108		-----		-----	
1135	ISO2592 automated	307.0		-1.22	
1378	ISO2592 automated	335		3.13	
1402	ISO2592 manual	>300		-----	
1613	D92 manual	300	C	-2.31	first reported: 250
1631	ISO2592 automated	318		0.49	
1724	D92 manual	308		-1.07	
1730		-----		-----	
1833	ISO2592 automated	318		0.49	
1849	ISO2592 automated	315.7		0.13	
1852		-----		-----	
1857	D92 manual	314		-0.13	
1881		-----		-----	
1944	ISO2592 manual	312		-0.44	
1990		-----		-----	
6037		-----		-----	
6076		-----		-----	
6228		-----		-----	
6229	ISO2592 automated	322		1.11	
6364	D92 automated	222	C,R(0.01)	-14.44	first reported:238.0
6404	ISO2592 manual	295		-3.09	
6419		-----		-----	
6474		-----		-----	

	normality	OK
	n	23
	outliers	2
	mean (n)	314.86
	st.dev. (n)	10.041
	R(calc.)	28.11
	st.dev.(ISO2592:17)	6.429
	R(ISO2592:17)	18
compare		
	R(D92:18)	18

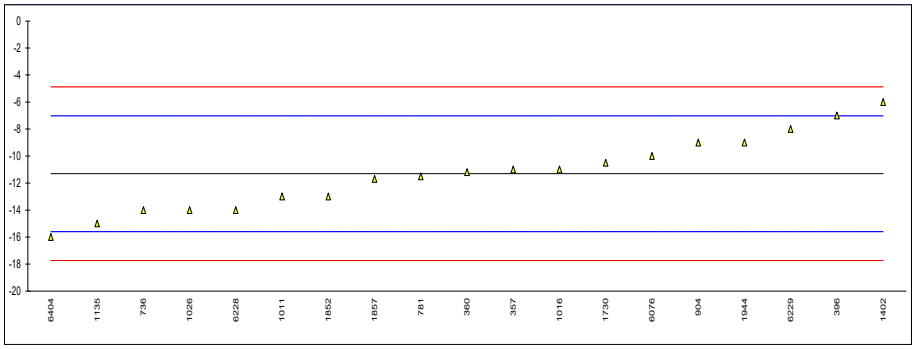


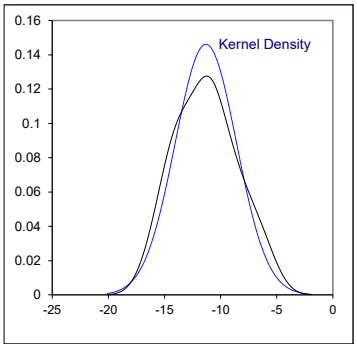


Determination of Fraass Breaking Point on sample #22240; results in °C

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
225		-----		-----	
328		-----		-----	
333		-----		-----	
335		-----		-----	
342		-----		-----	
357	EN12593 automated	-11		0.14	
360	EN12593 automated	-11.2		0.05	
365		-----		-----	
396	EN12593	-7		2.01	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657		-----		-----	
736	EN12593 automated	-14		-1.26	
781	EN12593 automated	-11.5		-0.09	
865		-----		-----	
904	EN12593 manual	-9		1.08	
1011	EN12593 automated	-13		-0.79	
1016	EN12593 automated	-11		0.14	
1026		-14		-1.26	
1040		-----		-----	
1108		-----		-----	
1135	EN12593 automated	-15		-1.72	
1378		-----		-----	
1402	EN12593 manual	-6		2.48	
1613		-----		-----	
1631		-----		-----	
1724		-----		-----	
1730	EN12593 automated	-10.5		0.38	
1833		-----		-----	
1849		-----		-----	
1852	EN12593 automated	-13		-0.79	
1857	EN12593 automated	-11.7		-0.18	
1881		-----		-----	
1944	EN12593 automated	-9		1.08	
1990		-----		-----	
6037		-----		-----	
6076	EN12593 automated	-10		0.61	
6228	EN12593 automated	-14		-1.26	
6229	EN12593 automated	-8		1.54	
6364		-----		-----	
6404	EN12593 automated	-16		-2.19	
6419		-----		-----	
6474		-----		-----	

normality	OK
n	19
outliers	0
mean (n)	-11.31
st.dev. (n)	2.730
R(calc.)	7.65
st.dev.(EN12593:15)	2.14
R(EN12593:15)	6

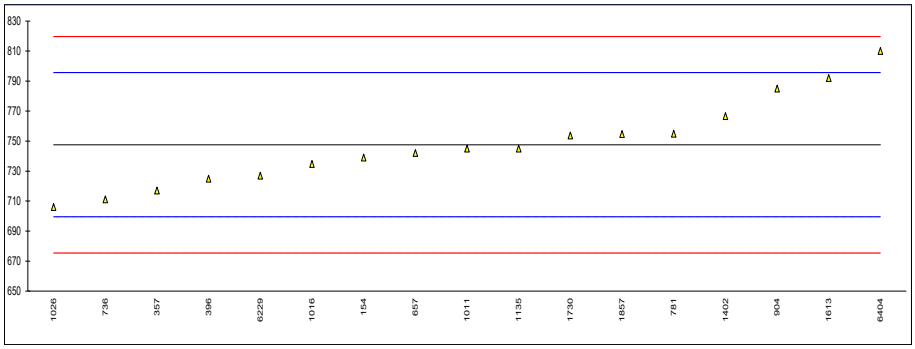


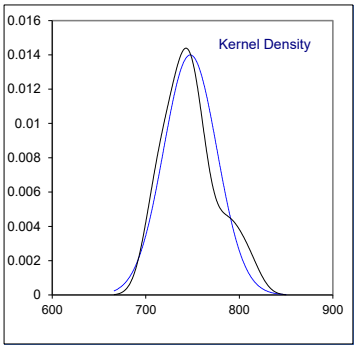


Determination of Kinematic Viscosity at 135 °C on sample #22240; results in mm²/s

lab	method	value	mark	z(targ)	remarks
154	D2170	739		-0.36	
225		-----		-----	
328		-----		-----	
333		-----		-----	
335		-----		-----	
342		-----		-----	
357	EN12595	717		-1.27	
360		-----		-----	
365		-----		-----	
396	EN12595	725		-0.94	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2170	742	C	-0.23	first reported: 597
736	EN12595	711.1		-1.52	
781	EN12595	754.9		0.31	
865		-----		-----	
904	EN12595	785		1.56	
1011	EN12595	745		-0.11	
1016	EN12595	734.7		-0.54	
1026	EN12595	706		-1.73	
1040		-----		-----	
1108		-----		-----	
1135	EN12595	745		-0.11	
1378		-----		-----	
1402	EN12595	766.6		0.79	
1613	D2170	792		1.85	
1631		-----		-----	
1724		-----		-----	
1730	EN12595	753.7		0.26	
1833		-----		-----	
1849		-----		-----	
1852		-----		-----	
1857	D445	754.7		0.30	
1881		-----		-----	
1944		-----		-----	
1990		-----		-----	
6037		-----		-----	
6076		-----		-----	
6228		-----		-----	
6229	EN12595	726.9		-0.86	
6364		-----		-----	
6404		810		2.60	
6419		-----		-----	
6474		-----		-----	

	normality	OK
	n	17
	outliers	0
	mean (n)	747.56
	st.dev. (n)	28.493
	R(calc.)	79.78
	st.dev.(EN12595:14)	24.029
	R(EN12595:14)	67.28
compare		
	R(D2170:18)	65.79

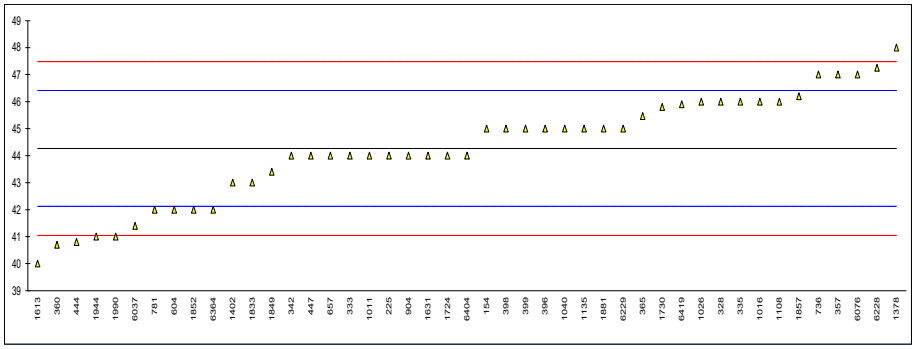


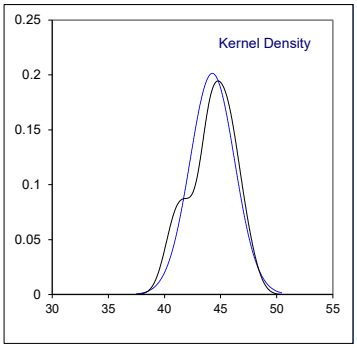


Determination of Penetration at 25 °C on sample #22240; results in 0.1 mm

lab	method	value	mark	z(targ)	remarks
154	D5 manual	45		0.69	
225	D5 manual	44.0		-0.25	
328	EN1426 manual	46		1.62	
333	EN1426 manual	44		-0.25	
335	EN1426 automated	46		1.62	
342	EN1426 manual	44		-0.25	
357	EN1426 automated	47		2.55	
360	EN1426 automated	40.7		-3.33	
365	EN1426	45.46		1.12	
396	EN1426	45		0.69	
398	EN1426 manual	45		0.69	
399	EN1426 manual	45		0.69	
444	EN1426 manual	40.8		-3.23	
447	EN1426 automated	44		-0.25	
604	D5 manual	42		-2.11	
657	D5 manual	44		-0.25	
736	EN1426 automated	47		2.55	
781	EN1426 automated	42		-2.11	
865		-----		-----	
904	EN1426	44		-0.25	
1011	EN1426 automated	44		-0.25	
1016	EN1426 automated	46		1.62	
1026	D5 automated	46		1.62	
1040	EN1426 automated	45.0		0.69	
1108	EN1426 automated	46		1.62	
1135	EN1426 automated	45		0.69	
1378	EN1426 automated	48		3.49	
1402	EN1426 manual	43		-1.18	
1613	D5 automated	40.0		-3.98	
1631	EN1426 automated	44		-0.25	
1724	D5 manual	44		-0.25	
1730	EN1426 automated	45.8		1.43	
1833	EN1426 automated	43		-1.18	
1849	EN1426 automated	43.4		-0.81	
1852	EN1426 automated	42		-2.11	
1857	EN1426 manual	46.2		1.81	
1881	EN1426 automated	45.0		0.69	
1944	EN1426 automated	41		-3.05	
1990	D5 manual	41		-3.05	
6037	EN1426 manual	41.4		-2.67	
6076	EN1426 automated	47		2.55	
6228	EN1426 automated	47.25		2.79	
6229	EN1426 automated	45		0.69	
6364	D5 manual	42	C	-2.11	first reported: 37
6404	EN1426 manual	44		-0.25	
6419	EN1426 manual	45.9		1.53	
6474		-----		-----	

			Only EN1426	Only D5
	normality	OK	OK	OK
	n	45	36	9
	outliers	0	0	0
	mean (n)	44.265	44.553	43.111
	st.dev. (n)	1.9799	1.9020	1.9650
	R(calc.)	5.544	5.326	5.502
	st.dev.(EN1426:15)	1.0714	1.0714	-----
	R(EN1426:15)	3	3	-----
compare				
	R(D5:20)	7.075	-----	7.075

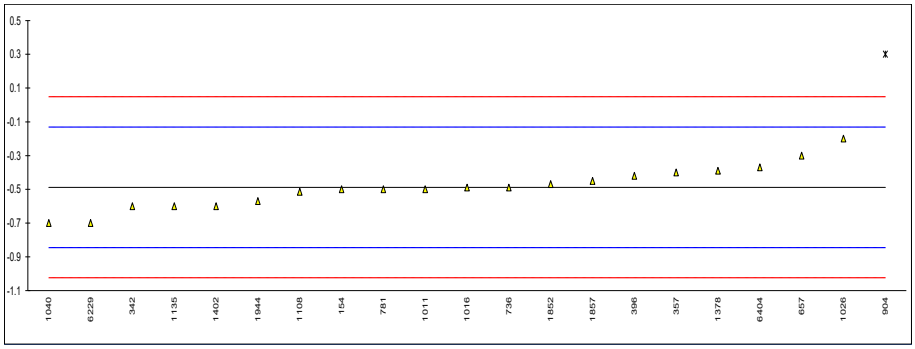


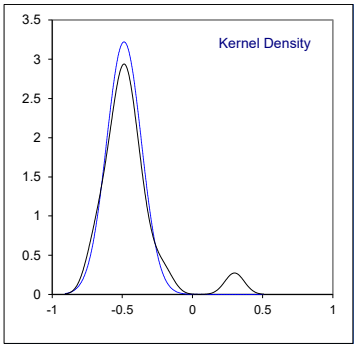


Determination of Penetration Index on sample #22240;

lab	method	value	mark	z(targ)	remarks
154	EN12591	-0.5		-0.07	
225		-----		-----	
328		-----		-----	
333		-----		-----	
335		-----		-----	
342	EN12591	-0.6		-0.63	
357	EN12591	-0.4		0.49	
360		-----		-----	
365		-----		-----	
396	EN12591	-0.42		0.38	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	Calculation	-0.3		1.05	
736	EN12591	-0.49		-0.01	
781	EN12591	-0.5		-0.07	
865		-----		-----	
904	EN12591	0.3	R(0.01)	4.41	
1011	EN12591	-0.5		-0.07	
1016	EN12591	-0.49		-0.01	
1026	EN12591	-0.2		1.61	
1040	EN12591	-0.7		-1.19	
1108	EN12591	-0.515		-0.15	
1135	EN12591	-0.6		-0.63	
1378	EN12591	-0.39		0.55	
1402		-0.6		-0.63	
1613		-----		-----	
1631		-----		-----	
1724		-----		-----	
1730		-----		-----	
1833		-----		-----	
1849		-----		-----	
1852	EN12591	-0.47		0.10	
1857	EN12591	-0.45		0.21	
1881		-----		-----	
1944	EN12591	-0.570		-0.46	
1990		-----		-----	
6037		-----		-----	
6076		-----		-----	
6228		-----		-----	
6229	EN12591	-0.7		-1.19	
6364		-----		-----	
6404	EN12591	-0.37		0.66	
6419		-----		-----	
6474		-----		-----	

normality	OK
n	20
outliers	1
mean (n)	-0.488
st.dev. (n)	0.1238
R(calc.)	0.347
st.dev.(EN12591:09)	0.1786
R(EN12591:09)	0.5

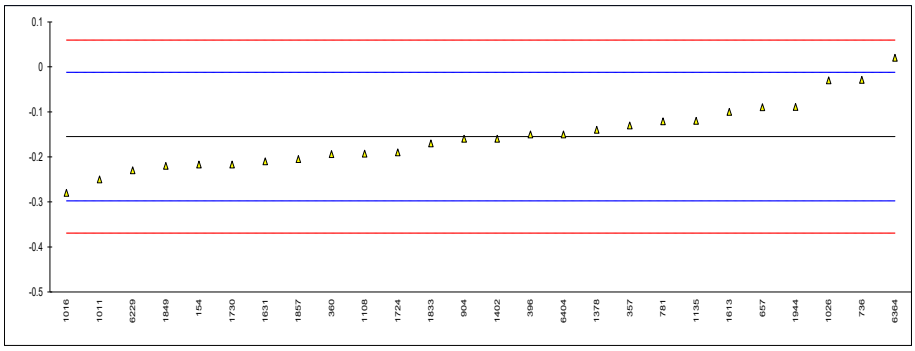


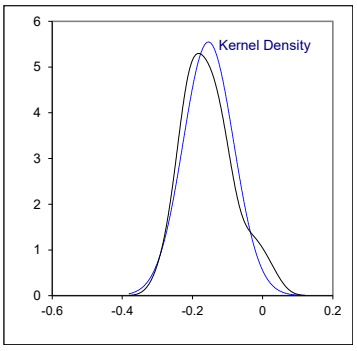


Determination of RTFOT at 163 °C, Change of Mass on sample #22240; results in %

lab	method	value	mark	z(targ)	remarks
154	D2872	-0.217		-0.87	
225		-----		-----	
328		-----		-----	
333		-----		-----	
335		-----		-----	
342		-----		-----	
357	EN12607-1	-0.13		0.35	
360	EN12607-1	-0.194		-0.55	
365		-----		-----	
396	EN12607-1	-0.15		0.07	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2872	-0.09		0.91	
736	EN12607-1	-0.029		1.76	
781	EN12607-1	-0.121		0.47	
865		-----		-----	
904	EN12607-1	-0.16		-0.07	
1011	EN12607-1	-0.25		-1.33	
1016	EN12607-1	-0.28		-1.75	
1026		-0.03		1.75	
1040		-----		-----	
1108	EN12607-1	-0.193		-0.53	
1135	EN12607-1	-0.12		0.49	
1378	EN12607-1	-0.140	C	0.21	first reported: 0.140
1402	EN12607-1	-0.16		-0.07	
1613	D2872	-0.1		0.77	
1631	EN12607-1	-0.21		-0.77	
1724		-0.19		-0.49	
1730	EN12607-1	-0.217		-0.87	
1833		-0.17		-0.21	
1849	EN12607-1	-0.22		-0.91	
1852		-----		-----	
1857	EN12607-1	-0.205		-0.70	
1881		-----		-----	
1944	EN12607-1	-0.089	C	0.92	first reported: -0.389
1990		-----		-----	
6037		-----		-----	
6076		-----		-----	
6228		-----		-----	
6229	EN12607-1	-0.23		-1.05	
6364		0.02	C	2.45	first reported: 0.0198
6404	EN12607-1	-0.15		0.07	
6419		-----		-----	
6474		-----		-----	

normality	OK
n	26
outliers	0
mean (n)	-0.1548
st.dev. (n)	0.07189
R(calc.)	0.2013
st.dev.(EN12607-1:14)	0.07143
R(EN12607-1:14)	0.2

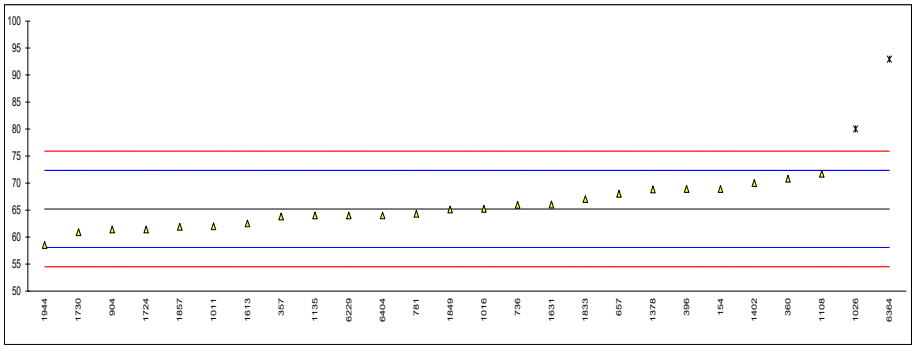


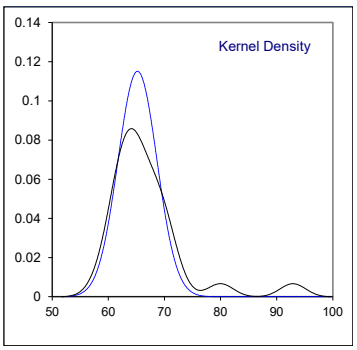


Determination of RTFOT at 163 °C, Retained Penetration on sample #22240; results in %

lab	method	value	mark	z(targ)	remarks
154	D2872	68.9		1.03	
225		-----		-----	
328		-----		-----	
333		-----		-----	
335		-----		-----	
342		-----		-----	
357	EN12607-1	63.8		-0.40	
360	EN12607-1	70.8		1.56	
365		-----		-----	
396	EN12607-1	68.89		1.03	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2872	68		0.78	
736	EN12607-1	65.96		0.21	
781	EN12607-1	64.29		-0.26	
865		-----		-----	
904	EN12607-1	61.4		-1.07	
1011	EN12607-1	62		-0.90	
1016	EN12607-1	65.2174		0.00	
1026		80	R(0.01)	4.14	
1040		-----		-----	
1108	EN12607-1	71.7		1.82	
1135	EN12607-1	64		-0.34	
1378	EN12607-1	68.8		1.00	
1402	EN12607-1	70		1.34	
1613	D2872	62.5		-0.76	
1631	EN12607-1	66		0.22	
1724		61.4		-1.07	
1730	EN12607-1	60.9		-1.21	
1833		67		0.50	
1849	EN12607-1	65.1		-0.03	
1852		-----		-----	
1857	EN12607-1	61.9		-0.93	
1881		-----		-----	
1944	EN12607-1	58.53		-1.87	
1990		-----		-----	
6037		-----		-----	
6076		-----		-----	
6228		-----		-----	
6229	EN12607-1	64		-0.34	
6364		92.9	C,R(0.01)	7.75	first reported: 94.56
6404	EN12607-1	64		-0.34	
6419		-----		-----	
6474		-----		-----	

normality	OK
n	24
outliers	2
mean (n)	65.212
st.dev. (n)	3.4679
R(calc.)	9.710
st.dev.(EN12607-1:14)	3.5714
R(EN12607-1:14)	10

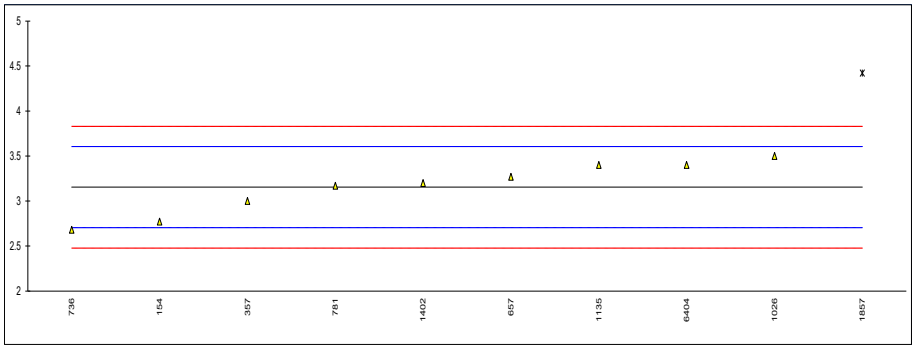


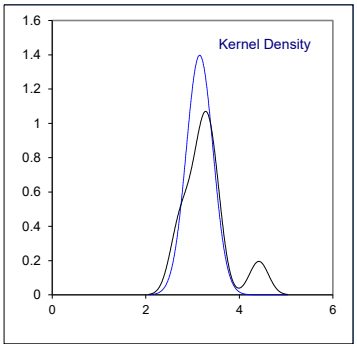


Determination of RTFOT at 163 °C, Viscosity Ratio on sample #22240

lab	method	value	mark	z(targ)	remarks
154	D2872	2.77	C	-1.71	first reported: 2.766
225		-----		-----	
328		-----		-----	
333		-----		-----	
335		-----		-----	
342		-----		-----	
357	EN12607-1	3.0		-0.69	
360		-----		-----	
365		-----		-----	
396		-----		-----	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2872	3.27		0.51	
736	EN12607-1	2.68		-2.11	
781	EN12607-1	3.17		0.07	
865		-----		-----	
904		-----		-----	
1011		-----		-----	
1016		-----		-----	
1026		3.5		1.53	
1040		-----		-----	
1108		-----		-----	
1135	EN12607-1	3.4		1.09	
1378		-----		-----	
1402	EN12607-1	3.2		0.20	
1613		-----		-----	
1631		-----		-----	
1724		-----		-----	
1730		-----		-----	
1833		-----		-----	
1849		-----		-----	
1852		-----		-----	
1857	EN12607-1	4.42	C,G(0.05)	5.62	first reported: 0.227
1881		-----		-----	
1944		-----		-----	
1990		-----		-----	
6037		-----		-----	
6076		-----		-----	
6228		-----		-----	
6229		-----		-----	
6364		-----		-----	
6404	EN12607-1	3.4		1.09	
6419		-----		-----	
6474		-----		-----	

normality	OK
n	9
outliers	1
mean (n)	3.154
st.dev. (n)	0.2856
R(calc.)	0.800
st.dev.(EN12607-1:14)	0.2253
R(EN12607-1:14)	0.6319

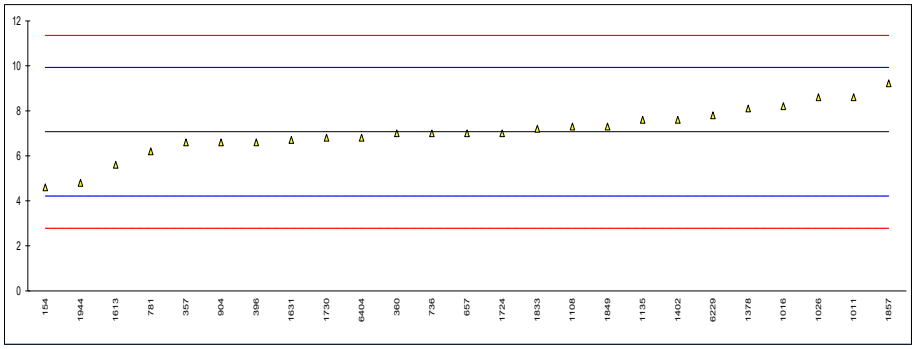


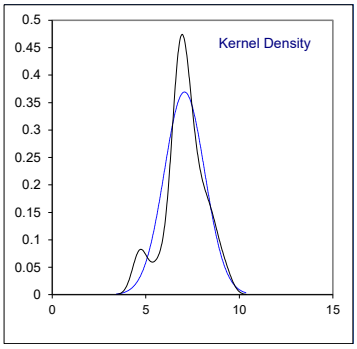


Determination of RTFOT at 163 °C, Increase in Softening Point on sample #22240; results in °C

lab	method	value	mark	z(targ)	remarks
154	D2872	4.6	C	-1.73	first reported: 4.8
225		-----		-----	
328		-----		-----	
333		-----		-----	
335		-----		-----	
342		-----		-----	
357	EN12607-1	6.6		-0.33	
360	EN12607-1	7.00		-0.05	
365		-----		-----	
396	EN12607-1	6.6		-0.33	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2872	7.0		-0.05	
736	EN12607-1	7.0		-0.05	
781	EN12607-1	6.2		-0.61	
865		-----		-----	
904	EN12607-1	6.6		-0.33	
1011	EN12607-1	8.6		1.07	
1016	EN12607-1	8.2		0.79	
1026		8.6		1.07	
1040		-----		-----	
1108	EN12607-1	7.3		0.16	
1135	EN12607-1	7.6		0.37	
1378	EN12607-1	8.1		0.72	
1402	EN12607-1	7.6		0.37	
1613	D2872	5.6		-1.03	
1631	EN12607-1	6.7		-0.26	
1724		7		-0.05	
1730	EN12607-1	6.8		-0.19	
1833		7.2		0.09	
1849	EN12607-1	7.3		0.16	
1852		-----		-----	
1857	EN12607-1	9.22		1.50	
1881		-----		-----	
1944	EN12607-1	4.8	C	-1.59	first reported: 9.8
1990		-----		-----	
6037		-----		-----	
6076		-----		-----	
6228		-----		-----	
6229	EN12607-1	7.8		0.51	
6364		-----		-----	
6404	EN12607-1	6.8		-0.19	
6419		-----		-----	
6474		-----		-----	

normality	OK
n	25
outliers	0
mean (n)	7.07
st.dev. (n)	1.080
R(calc.)	3.02
st.dev.(EN12607-1:14)	1.429
R(EN12607-1:14)	4





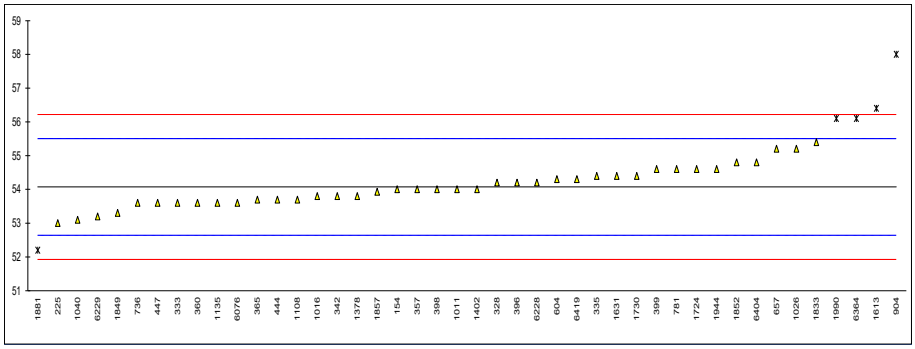
Determination of Softening Point (Ring and Ball) on sample #22240; results in °C

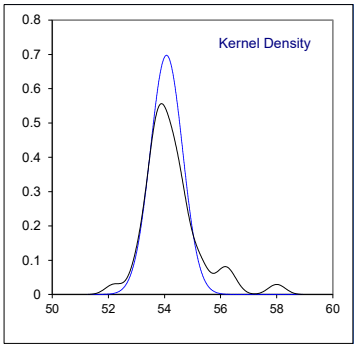
lab	method	value	mark	z(targ)	remarks
154	D36	54.0	C	-0.10	first reported: 58.8
225	D36	53.0		-1.50	
328	EN1427	54.2		0.18	
333	EN1427	53.6		-0.66	
335	EN1427	54.4		0.46	
342	EN1427	53.8		-0.38	
357	EN1427	54		-0.10	
360	EN1427	53.6		-0.66	
365	EN1427	53.7		-0.52	
396	EN1427	54.2		0.18	
398	EN1427	54.0		-0.10	
399	EN1427	54.6		0.74	
444	EN1427	53.7		-0.52	
447	EN1427	53.6		-0.66	
604	D36	54.3		0.32	
657	D36	55.2		1.58	
736	EN1427	53.6		-0.66	
781	EN1427	54.6		0.74	
865		-----		-----	
904	EN1427	58.0	R(0.01)	5.50	
1011	EN1427	54.0		-0.10	
1016	EN1427	53.8		-0.38	
1026	EN1427	55.2		1.58	
1040	EN1427	53.10		-1.36	
1108	EN1427	53.7		-0.52	
1135	EN1427	53.6		-0.66	
1378	EN1427	53.8		-0.38	
1402	EN1427	54.0		-0.10	
1613	D36	56.4	R(0.05)	3.26	
1631	EN1427	54.4		0.46	
1724	D36	54.6		0.74	
1730	EN1427	54.4		0.46	
1833	EN1427	55.4		1.86	
1849	EN1427	53.3		-1.08	
1852	EN1427	54.8		1.02	
1857	EN1427	53.93		-0.20	
1881	EN1427	52.20	R(0.05)	-2.62	
1944	EN1427	54.6		0.74	
1990	D36	56.1	R(0.05)	2.84	
6037		-----		-----	
6076	EN1427	53.6		-0.66	
6228	EN1427	54.2		0.18	
6229	EN1427	53.2		-1.22	
6364	D36	56.1	R(0.05)	2.84	
6404	EN1427	54.8		1.02	
6419	EN1427	54.3		0.32	
6474		-----		-----	

	normality	OK
	n	39
	outliers	5
	mean (n)	54.073
	st.dev. (n)	0.5719
	R(calc.)	1.601
	st.dev.(EN1427:15)	0.7143
	R(EN1427:15)	2
compare		
	R(D36:14R20)	5.47
	R(D36:14R20)	5.15

automated electronic thermometer

mercury thermometer

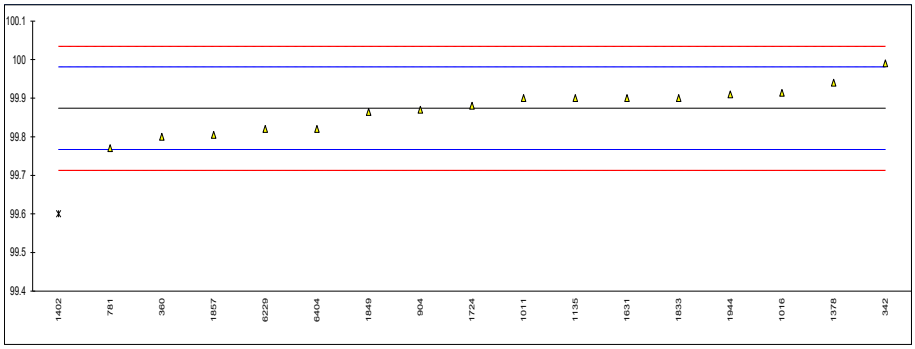


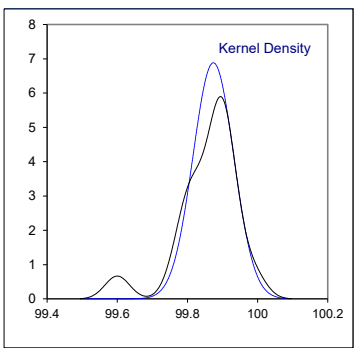


Determination of Solubility in Xylene on sample #22240; results in %M/M

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
225		-----		-----	
328		-----		-----	
333		-----		-----	
335		-----		-----	
342	EN12592	99.99		2.17	
357		-----		-----	
360	EN12592	99.80		-1.38	
365		-----		-----	
396		-----		-----	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657		-----		-----	
736		-----		-----	
781	EN12592	99.77		-1.94	
865		-----		-----	
904	EN12592	99.87		-0.07	
1011	EN12592	99.90		0.49	
1016	EN12592	99.9135		0.74	
1026		-----		-----	
1040		-----		-----	
1108		-----		-----	
1135	EN12592	99.90		0.49	
1378	EN12592	99.94	C	1.23	first reported: 99.6
1402	EN12592	99.60	G(0.01)	-5.11	
1613		-----		-----	
1631	EN12592	99.9		0.49	
1724	EN12592	99.88		0.11	
1730		-----		-----	
1833	EN12592	99.90		0.49	
1849	EN12592	99.864		-0.19	
1852		-----		-----	
1857	EN12592	99.805		-1.29	
1881		-----		-----	
1944	EN12592	99.91	C	0.67	first reported 99.31
1990		-----		-----	
6037		-----		-----	
6076		-----		-----	
6228		-----		-----	
6229	EN12592	99.82		-1.01	
6364		-----		-----	
6404	EN12592	99.82		-1.01	
6419		-----		-----	
6474		-----		-----	

normality	OK
n	16
outliers	1
mean (n)	99.874
st.dev. (n)	0.0580
R(calc.)	0.162
st.dev.(EN12592:14)	0.0536
R(EN12592:14)	0.15





Determination of Ductility on sample #22240; results in cm

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
225		-----		-----	
328		-----		-----	
333		-----		-----	
335		-----		-----	
342		-----		-----	
357		-----		-----	
360		-----		-----	
365		-----		-----	
396	D113	150+		-----	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D113	150+		-----	
736	D113	150+		-----	
781	D113	>100		-----	
865		-----		-----	
904	D113	>100		-----	
1011		-----		-----	
1016		-----		-----	
1026	EN13589	16.4		-----	possible false negative test result?
1040		-----		-----	
1108		-----		-----	
1135	D113	>150		-----	
1378		-----		-----	
1402		-----		-----	
1613	D113	150		-----	
1631		-----		-----	
1724		-----		-----	
1730		-----		-----	
1833		-----		-----	
1849		-----		-----	
1852		-----		-----	
1857		-----		-----	
1881		-----		-----	
1944		-----		-----	
1990		-----		-----	
6037		-----		-----	
6076		-----		-----	
6228		-----		-----	
6229	D113	150+		-----	
6364	D113	>100		-----	
6404	EN13589	> 150		-----	
6419		-----		-----	
6474		-----		-----	
	n	10			
	mean (n)	>100			

APPENDIX 2

Number of participants per country

1 lab in ALBANIA
1 lab in AUSTRIA
1 lab in BELGIUM
1 lab in BULGARIA
1 lab in CHINA, People's Republic
1 lab in COTE D'IVOIRE
1 lab in FINLAND
3 labs in FRANCE
2 labs in GERMANY
3 labs in GREECE
1 lab in IRELAND
4 labs in ITALY
1 lab in JORDAN
1 lab in KAZAKHSTAN
1 lab in KENYA
1 lab in LITHUANIA
2 labs in MALAYSIA
4 labs in NETHERLANDS
1 lab in PORTUGAL
1 lab in ROMANIA
2 labs in RUSSIAN FEDERATION
1 lab in SAUDI ARABIA
1 lab in SINGAPORE
1 lab in SPAIN
5 labs in TURKEY
4 labs in UNITED KINGDOM
1 lab in UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations

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

Literature

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7 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
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