Results of Proficiency Test
Bitumen
December 2015

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Bitumen is a highly viscous, black and sticky form of petroleum. In the United States, bitumen is often referred to as asphalt. In this report we will use the word 'bitumen' for the substance that is the bottom product of the vacuum distillation step in oil refining. This bitumen can be used in road pavement as a binder for the sand and stones that build this pavement. But it is also used, among other applications for waterproofing products, like sealing of roofs and it can be a part of printing inks.
At the request of several participants, the Institute of Interlaboratory Studies decided to organise an interlaboratory study for bitumen in 2014. During the annual proficiency testing program 2015/2016, it was decided to continue the round robin for the analysis of Bitumen.
In this interlaboratory study 36 laboratories in 22 different countries have participated. See appendix 2 for the number of participants per country. In this report, the results of the 2015 interlaboratory study on Bitumen are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

2 SET-UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted to a third party laboratory. It was decided to send two containers of 1 litre bitumen (labelled #15255 and #15256), one is a 70/100 grade, the other a 50/70 grade bitumen. Participants were requested to report rounded and unrounded results. The unrounded 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/IEC 17043:2010. 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 organisation 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 April 2014 (iis-protocol, version 3.3). This protocol can be downloaded from the iis website http://www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

In this proficiency test two different samples were prepared. Both bitumen samples were supplied by a third party laboratory.

The first batch is a straight-run bitumen, 70/100 grade. This batch was heated, filled into 50 one litre metal cans and labelled (#15255). The homogeneity of the subsamples #15255 was checked by determination of penetration (EN 1426) and softening point (EN 1427) on four randomly stratified samples.

	Penetration at 25°C in 0.1 mm	Softening Point in °C
Sample #15255-1	84	46.4
Sample #15255-2	83	46.2
Sample #15255-3	83	46.4
Sample #15255-4	83	46.2

Table 1 : homogeneity test results of subsamples #15255

From the test results of table 1, the repeatabilities were calculated and compared with the repeatability of the method and 0.3 times the corresponding target reproducibility in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Penetration at 25°C in 0.1 mm	Softening Point in °C
r (observed)	1.4	0.3
Reference method	EN1426:15	EN1427:15
r (Reference method)	3.3	1.0
0.3*R (Ref. method)	1.5	0.6

Table 2: repeatabilities of subsamples #15255

The calculated repeatabilities for Penetration and Softening Point were in full agreement with 0.3 times the reproducibility and the repeatability of the respective reference methods. Therefore, homogeneity of the subsamples of #15255 was assumed.

The second batch is another straight-run bitumen, 50/70 grade. This batch was heated, filled into 50 one litre metal cans and labelled (#15256). The homogeneity of the subsamples #15256 was checked by determination of penetration (EN 1426) and softening point (EN 1427) at the beginning and the end of the straight run.

	Penetration at 25°C in 0.1 mm	Softening Point in °C
Sample #15256-1	56	50.2
Sample #15256-2	56	50.2
Sample #15256-3	56	49.8
Sample #15256-4	55	49.8

Table 3: homogeneity test results of subsamples #15256

From the test results of table 3, the repeatabilities were calculated and compared with the repeatability of the method and 0.3 times the corresponding target reproducibility in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Penetration at 25°C in 0.1 mm	Softening Point in °C
r (Observed)	1.4	0.65
Reference method	EN1426:15	EN1427:15
r (Reference method)	2.2	1.0
0.3*R (Ref. method)	1.0	0.6

Table 4: repeatabilities of subsamples #15256

The calculated repeatabilities for Penetration and Softening Point were in full agreement with 0.3 times the reproducibility and the repeatability of the respective reference methods. Therefore, homogeneity of the subsamples of #15256 was assumed.

To each of the participating laboratories a 1 litre metal can, labelled #15255 and a 1 litre metal can, labelled #15256 were sent on November 25, 2015.

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 ANALYSES

The participants were asked to determine on both samples: Density at 25°C, Dynamic Shear Rheometer Modulus G* and Phase Angle, Dynamic Viscosity at 60°C, Flash Point COC, Fraass Breaking Point, Kinematic Viscosity at 135°C, Penetration at 25°C, Penetration Index, RTFOT at 163°C (Change of Mass, Retained Penetration, Viscosity Ratio and Increase in Softening Point), Softening Point (Ring and Ball) and Solubility in Xylene.

To get comparable results a detailed report form, on which the units were prescribed as well as the required standards and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis/. A SDS and a form to confirm receipt of the samples were added to the sample package.

3 RESULTS

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

Directly after deadline, a reminder was sent to those laboratories that did not report results at that moment. Shortly after the deadline, the available results were screened for suspect data. A result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the results. Additional or corrected results are used for the data analysis and the original results are placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of January 2014 (iis-protocol, version 3.3).

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

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

According to ISO 5725 the original results per determination were submitted to Dixon's and/or Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test (ref. 15). Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

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

3.2 GRAPHICS

In order to visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are under the X-axis.
The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. Outliers and other data, which were excluded from the calculations, are represented as a 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 (see appendix 3, nos.13-14). Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the spread of this interlaboratory study. The target standard deviation was calculated from the literature reproducibility by division with 2.8.

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

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

The $z_{(target)}$ scores are listed in the 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:

	$\lvert z\rvert < 1$	good
$1 <$	$\lvert z\rvert < 2$	satisfactory
$2 <$	$\lvert z\rvert < 3$	questionable
$3 <$	$\lvert z\rvert$	unsatisfactory

4 EVALUATION

In this proficiency test no problems were encountered with the dispatch of the samples.
One participant did not report any test results and all other laboratories reported the test results before the data entry website was closed for this round. Not all laboratories were able to report all analyses requested.
Finally, 35 participants reported in total 388 numerical results. Observed were 30 statistically outlying results, which is 7.7%. 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 SAMPLE AND PER TEST

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

Sample #15255:

Density: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of EN15326:2007.

Dynamic Shear Rheometer: Two properties were requested: Shear Modulus G* and Phase Angle. Unfortunately no results were reported by the participants on any of the two properties.

Dynamic Viscosity: This determination was very problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements of EN12596:2014.

Flash Point COC: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ISO2592:2000.
Because of the low number of reported results, it is not possible to evaluate the effect of using a manual or automatic method.
Surprisingly, the observed reproducibility of the group of laboratories for Flash Point on sample #15256 (Bitumen 50/70) was smaller, although the flash point is in the same range.

Fraass Breaking Point: 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 EN12593:2015.

Kinematic Viscosity: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of EN12595:2014, but almost in agreement with the less strict requirements of ASTM D2170/2170M:2010.

Penetration: This determination may be very problematic depending on the method used. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the strict requirements of EN1426:2015. However, the calculated reproducibility is just in agreement with the requirements of ASTM D5/5M:2013.
When the test results of test method EN1426 (n=22) and ASTM D5/D5M (n=6) are evaluated separately, the reproducibilities are still not in agreement with their respective method requirements.

Different factors could cause this large dispersion, 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.
For the less soft bitumens the surface area can be a factor. When the bitumen is poured in a cup, air bubbles may remain trapped just below the surface. When the penetration needle hits such a bubble, it will not measure penetration of the bitumen, but penetration of the bubble. With heat (like a flame) the bubbles can be removed.

Penetration Index: This determination was not problematic for the laboratories that reported this parameter. One statistical outlier was observed and two results appeared to have an error in calculation. Two results were excluded, because the test result of the Penetration and/or the Softening Point of these laboratories was an outlier. However, the calculated reproducibility is in good agreement with the requirements of EN12591:2009.

The Penetration Index was calculated by iis for all the laboratories, which reported Penetration and Softening Point.
The values for Penetration Index, calculated by iis, were also statistically evaluated. Five calculated test results were excluded as these laboratories had outliers in the Penetration and/or Softening Point test results. The calculated reproducibility was just not in agreement with the requirements of EN12591:2009. Since the calculated reproducibility of the determination of Penetration is not meeting the requirements of its method, it can be expected that the spread in the calculated Penetration Index is large too.

RTFOT: This is a Rolling Thin Film Oven Test (RTFOT, EN12607-1 or ASTM D2872). It is a bitumen ageing test, in which rotating glass vessels with a coating of bitumen is used.
Four properties were determined after the RTFOT ageing test: Change of Mass, Retained Penetration, Viscosity Ratio and Increase in Softening Point.

The determination on Change of Mass was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of EN12607-1:2014.

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 agreement with the requirements of EN12607-1:2014.

Only two participants reported a test result for Viscosity Ratio. Both results were very different, it is most likely that one of the reported results is not a ratio, but a value for viscosity. Therefore no significant conclusions can be drawn.

The determination on Increase in Softening Point was not problematic. Two outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of EN12607-1:2014.

Softening Point: This determination was not problematic. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN1427:2015.

Solubility in Xylene: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of EN12592:2014.

Sample #15256:

Density: This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of EN15326:2007.

Dynamic Shear Rheometer: Two properties were requested: Shear Modulus G* and Phase Angle. Unfortunately no results were reported by the participants on any of the two properties.

Dynamic Viscosity: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of EN12596:2014.

Flash Point COC: 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 ISO2592:2000.

Fraass Breaking Point: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of EN12593:2015.
A possible cause for the large spread could be that both manual determinations and automated equipment was used. Because of the low number of reported results, it is not possible to evaluate the effect of using a manual or automatic method.
Surprisingly, the observed reproducibility of the group of laboratories for the Fraass Breaking Point on sample #15255 (Bitumen 70/100) was smaller than for sample #15256.

Kinematic Viscosity: This determination was very problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements of EN12595:2014 or the requirements of ASTM D2170/2170M:2010.

Penetration: This determination may be very problematic depending on the method used. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the strict requirements of EN1426:2007 and only just in agreement with the less strict requirements of ASTM D5/5M:2013.

When the test results of test method EN1426 (n=20) are evaluated separately, the spread remains large. When the same is done for the test results of ASTM D5/5M (n=6), the spread is within the requirements of the method.
Possible causes for the large spread have already been explained in the discussion of the penetration of sample #15255.

Penetration Index: This determination was not problematic. No statistical outliers were observed and one test result appeared to have an error in calculation. The calculated reproducibility is in full agreement with the requirements of EN12591:2009.

The Penetration Index was calculated by iis for all the laboratories, which reported Penetration and Softening Point.
The values for Penetration Index, calculated by iis, were also statistically evaluated. The calculated reproducibility was again in full agreement with the requirements of EN12591:2009.

RTFOT: This is a Rolling Thin Film Oven Test (RTFOT, EN12607-1 or ASTM D2872). It is a bitumen ageing test, in which rotating glass vessels with a coating of bitumen is used.
Four properties were determined after the RTFOT ageing test: Change of Mass, Retained Penetration, Viscosity Ratio and Increase in Softening Point.

The determination on Change of Mass was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN12607-1:2014.

The determination on Retained Penetration was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of EN12607-1:2014.

Only two participants reported a test result for Viscosity Ratio. Both result very different, it is most likely that one of the reported results is not a ratio, but a value for viscosity. Therefore no significant conclusions can be drawn.

The determination on Increase in Softening Point was not problematic. One outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of EN12607-1:2014.

Softening Point: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN1427:2015.

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 EN12592:2014.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and the reproducibility as found for the group of participating laboratories that participated. The average results of the evaluated parameters, calculated reproducibilities and reproducibilities, derived from literature standards (in casu ASTM, EN, ISO and IP standards) are compared in the next tables.

Parameters #15255	Unit	n	average	2.8 * sd	R (lit)
Density at 25°C	kg/m^3	18	1028.4	4.6	5.0
Dynamic Viscosity at 60°C	Pa.s	7	161.9	42.2	16.2
Flash Point COC	°C	13	360	27	17
Fraass Breaking Point	°C	6	-17	4	6
Kinematic Viscosity at 135°C	mm^2/s	11	394.9	39.4	23.7
Penetration at 25°C	0.1 mm	31	82.3	9.9	4.9
Penetration Index		7	-1.08	0.21	0.50
RTFOT - Change of Mass	%	13	0.069	0.050	0.200
RTFOT - Retained Penetration	%	12	63.9	8.1	10.0
RTFOT - Viscosity Ratio		2	n.a.	n.a.	n.a.
RTFOT - Increase in Soft. Point	°C	11	4.4	1.3	2.0
Softening Point (Ring and Ball)	°C	28	46.0	1.5	2.0
Solubility in Xylene	% M/M	8	99.96	0.09	0.15

Table 5: summary of test results on Bitumen 70/100 grade, sample #15255

Parameters #15256	Unit	n	average	2.8 * sd	R (lit)
Density at 25°C	kg/m^3	15	1032.7	4.9	5.0
Dynamic Viscosity at 60°C	Pa.s	7	278.9	20.2	27.9
Flash Point COC	°C	12	359	15	17
Fraass Breaking Point	°C	7	-13	11	6
Kinematic Viscosity at 135°C	mm^2/s	10	508.6	80.8	30.5
Penetration at 25°C	0.1 mm	28	56.3	7.7	3.4
Penetration Index		9	-1.14	0.41	0.50
RTFOT - Change of Mass	%	15	0.058	0.078	0.200
RTFOT - Retained Penetration	%	13	64.3	16.6	10.0
RTFOT - Viscosity Ratio		2	n.a.	n.a.	n.a.
RTFOT - Increase in Soft. Point	°C	13	4.7	2.1	2.0
Softening Point (Ring and Ball)	°C	27	49.2	1.8	2.0
Solubility in Xylene	% M/M	7	99.93	0.10	0.15

Table 6: summary of test results on Bitumen 50/70 grade, sample #15256

Without further statistical calculations it can be concluded that for a number tests there is a good compliance of the group of participating laboratories with the relevant standards. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2015 WITH PREVIOUS PTS

	2015	2014
Number of reporting labs	35	36
Number of results reported	388	464
Number of statistical outliers	30	18
Percentage statistical outliers	7.7%	3.9%

Table 7: comparison with previous proficiency tests

The performance of the determinations against the requirements of the respective standards is listed in the table below.

Parameters	2015		2014	
	#15255	#15256	#14260	#14261
Density at 25°C	+	+/-	+/-	-
Dynamic Viscosity at 60°C	--	+	+/-	+
Flash Point COC	-	+	-	--
Fraass Breaking Point	+	-	-	--
Kinematic Viscosity at 135°C	-	--	--	+/-
Penetration at 25°C	--	--	--	--
Penetration Index	++	+	--	+
RTFOT at 163°C Change of Mass	++	++	++	++
RTFOT at 163°C Retained Penetration	+	-	-	-
RTFOT at 163°C Viscosity Ratio	n.e.	n.e.	(++)	n.e.
RTFOT at 163°C Increase in Soft. Point	+	+/-	+/-	+
Softening Point (Ring and Ball)	+	+	--	--
Solubility in Xylene	+	+	+/-	+

Table 8: comparison determinations against the standard

The following performance categories were used:

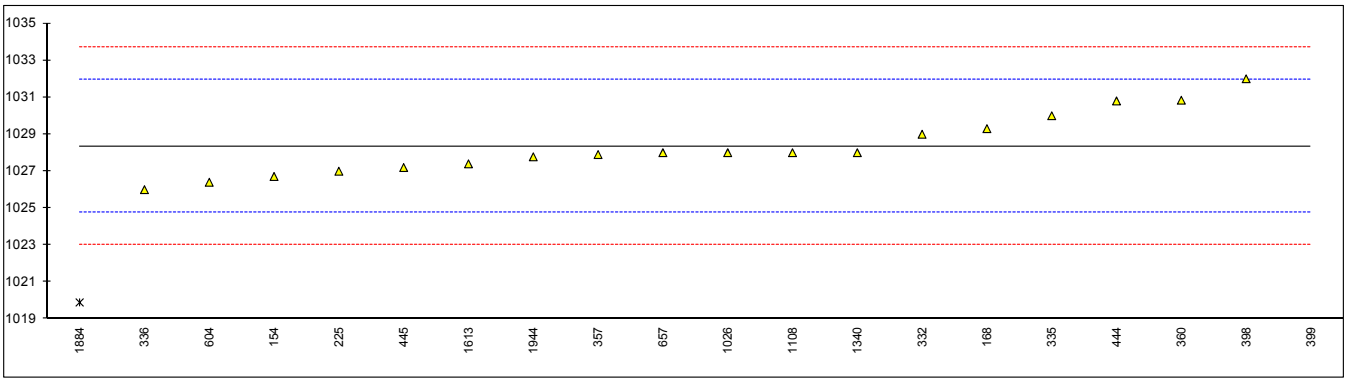
++: group performed much better than the standard
+ : group performed better than the standard
+/-: group performance equals the standard
- : group performed worse than the standard
-- : group performed much worse than the standard
n.e.: not evaluated

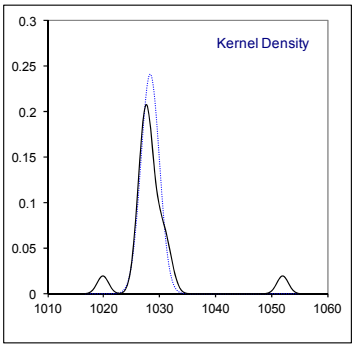
APPENDIX 1

Determination of Density at 25°C on sample #15255; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
154	D70	1026.7143		-0.92	
168	D70	1029.3	C	0.53	reported 1.0293 kg/m^3
225	D70	1027		-0.76	
332	EN15326	1029.0		0.36	
333		-----		-----	
335	EN15326	1030	C	0.92	first reported: 1.03 kg/m^3
336	EN15326	1026	C	-1.32	first reported: 1.026 kg/m^3
337		-----		-----	
353		-----		-----	
357	EN15326	1027.9		-0.25	
360	ISO3838	1030.84	C	1.39	first reported: 1.03084 kg/m^3
398	D70	1032		2.04	
399	D70	1052.0	R(0.01)	13.24	
440		-----		-----	
444	D70	1030.8	C	1.37	first reported: 1036.9
445	D70	1027.2		-0.65	
604	D70	1026.4		-1.09	
657	D70	1028		-0.20	
962		-----		-----	
1011		-----		-----	
1026	EN15326	1028		-0.20	
1040		-----		-----	
1108	EN15326	1028		-0.20	
1340	EN15326	1028		-0.20	
1543		-----		-----	
1613	DIN51757	1027.4		-0.53	also reported density of 970.31 at 120°C
1724		-----		-----	
1730		-----		-----	
1833		-----		-----	
1842		-----		-----	
1849		-----		-----	
1852		-----		-----	
1884	D70	1019.9	R(0.01)	-4.73	
1944	EN15326	1027.78	C	-0.32	first reported: 1032.75
6022		-----		-----	
6023		-----		-----	

normality	OK
n	18
outliers	2
mean (n)	1028.35
st.dev. (n)	1.656
R(calc.)	4.64
R(EN15326:07)	5.00

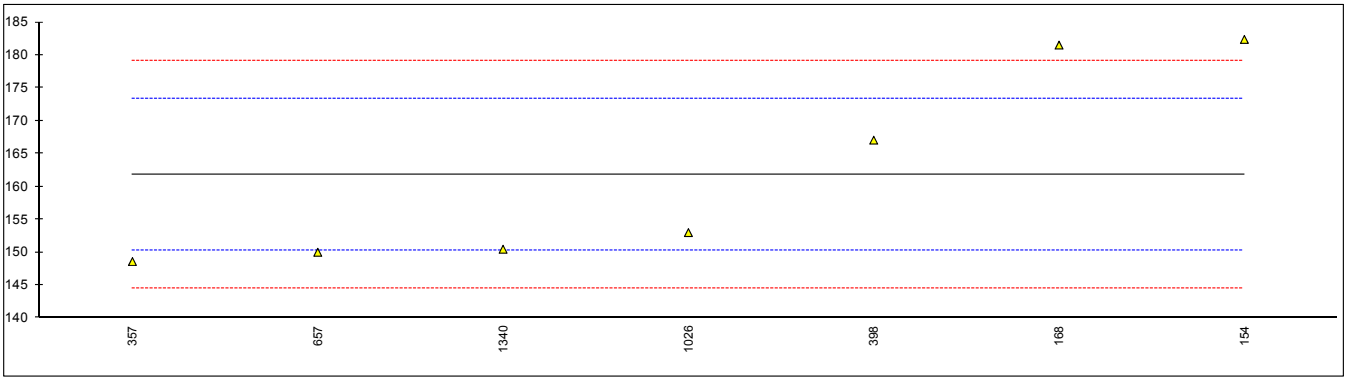




Determination of Dynamic Viscosity at 60°C on sample #15255; results in Pa.s

lab	method	value	mark	z(targ)	remarks
154	D2171	182.4		3.55	
168	D2171	181.53		3.40	
225		-----		-----	
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
337		-----		-----	
353		-----		-----	
357	EN12596	148.6		-2.29	
360		-----		-----	
398	EN12596	167.065		0.90	
399		-----		-----	
440		-----		-----	
444		-----		-----	
445		-----		-----	
604		-----		-----	
657	D2171	150		-2.05	
962		-----		-----	
1011		-----		-----	
1026	EN12596	153		-1.53	
1040		-----		-----	
1108		-----		-----	
1340	EN12596	150.47		-1.97	
1543		-----		-----	
1613		-----		-----	
1724		-----		-----	
1730		-----		-----	
1833		-----		-----	
1842		-----		-----	
1849		-----		-----	
1852		-----		-----	
1884		-----		-----	
1944		-----		-----	
6022		-----		-----	
6023		-----		-----	

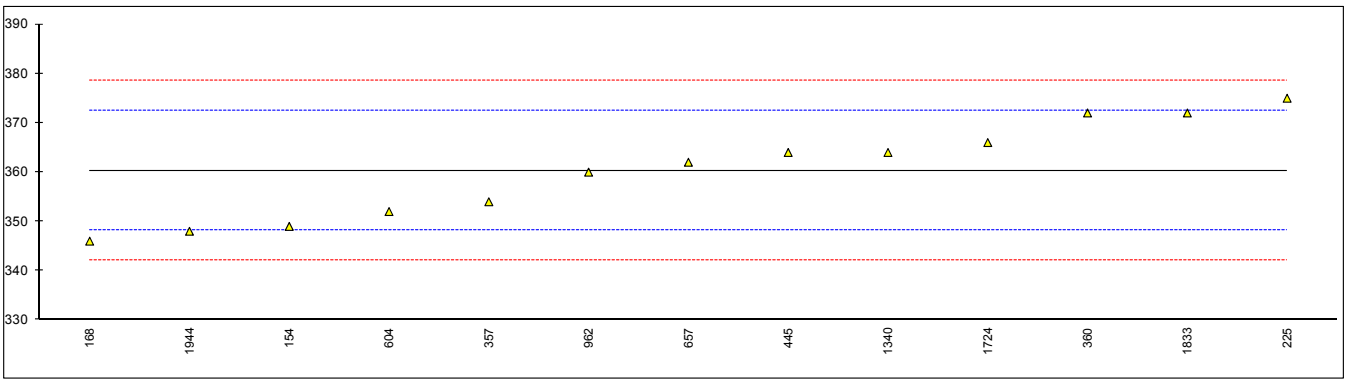
normality	unknown
n	7
outliers	0
mean (n)	161.87
st.dev. (n)	15.059
R(calc.)	42.16
R(EN12596:14)	16.19

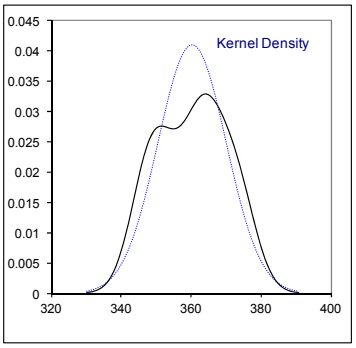


Determination of Flash Point COC on sample #15255; results in °C

lab	method	mode	value	mark	z(targ)	remarks
154	D92	Manual	349		-1.86	
168	D92		346		-2.36	
225	D92		375		2.42	
332			-----		-----	
333			-----		-----	
335			-----		-----	
336			-----		-----	
337			-----		-----	
353			-----		-----	
357	ISO2592	Manual	354		-1.04	
360	ISO2592		372.0		1.93	
398			-----		-----	
399			-----		-----	
440			-----		-----	
444			-----		-----	
445	D92	Manual	364.0		0.61	
604	D92		352		-1.37	
657	D92	Manual	362		0.28	
962	D92		360		-0.05	
1011	ISO2592		>280		-----	
1026	ISO2592	Manual	> 320		-----	
1040			-----		-----	
1108			-----		-----	
1340	ISO2592	Automated	364		0.61	
1543			-----		-----	
1613			-----		-----	
1724	D92		366		0.94	
1730			-----		-----	
1833	ISO2592		372		1.93	
1842			-----		-----	
1849			-----	W	-----	first reported: 314 (ISO 2592)
1852			-----		-----	
1884			-----		-----	
1944	ISO2592	Manual	348		-2.03	
6022			-----		-----	
6023			-----		-----	

normality	OK	
n	13	
outliers	0	
mean (n)	360.3	
st.dev. (n)	9.77	
R(calc.)	27.3	
R(ISO2592:00)	17.0	Compare R(D92:12b) = 18.0

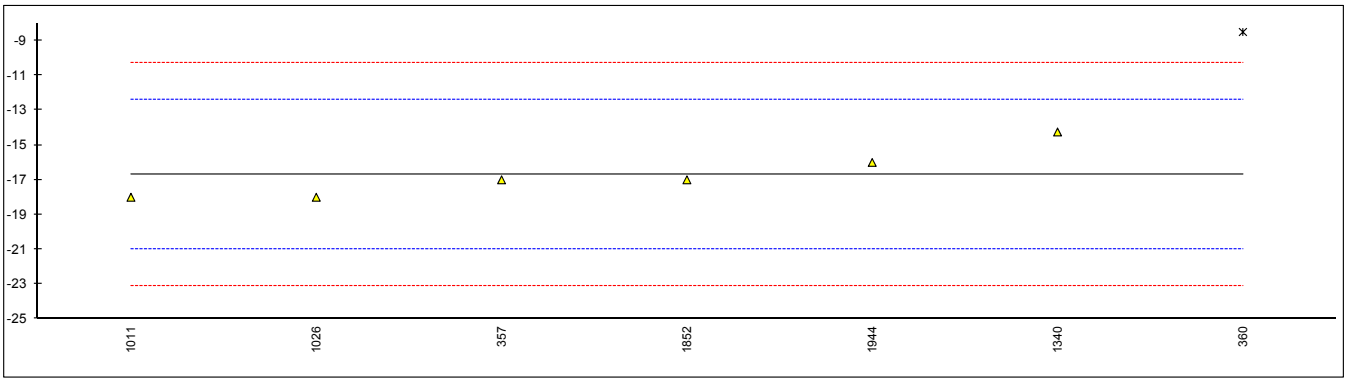




Determination of Fraass Breaking point on sample #15255; results in °C

lab	method	mode	value	mark	z(targ)	remarks
154			-----		-----	
168			-----		-----	
225			-----		-----	
332			-----		-----	
333			-----		-----	
335			-----		-----	
336			-----		-----	
337			-----		-----	
353			-----		-----	
357	EN12593	Manual	-17		-0.14	
360	EN12593	Automated	-8.5	C,D(0.05)	3.83	first reported: -6.5
398			-----		-----	
399			-----		-----	
440			-----		-----	
444			-----		-----	
445			-----		-----	
604			-----		-----	
657			-----		-----	
962			-----		-----	
1011	EN12593	Manual	-18		-0.60	
1026	EN12593	Automated	-18		-0.60	
1040			-----		-----	
1108			-----		-----	
1340	EN12593	Automated	-14.25		1.15	
1543			-----		-----	
1613			-----		-----	
1724			-----		-----	
1730			-----		-----	
1833			-----		-----	
1842			-----		-----	
1849			-----		-----	
1852	EN12593		-17		-0.14	
1884			-----		-----	
1944	EN12593	Manual	-16		0.33	
6022			-----		-----	
6023			-----		-----	

normality	unknown
n	6
outliers	1
mean (n)	-16.7
st.dev. (n)	1.42
R(calc.)	4.0
R(EN12593:15)	6.0

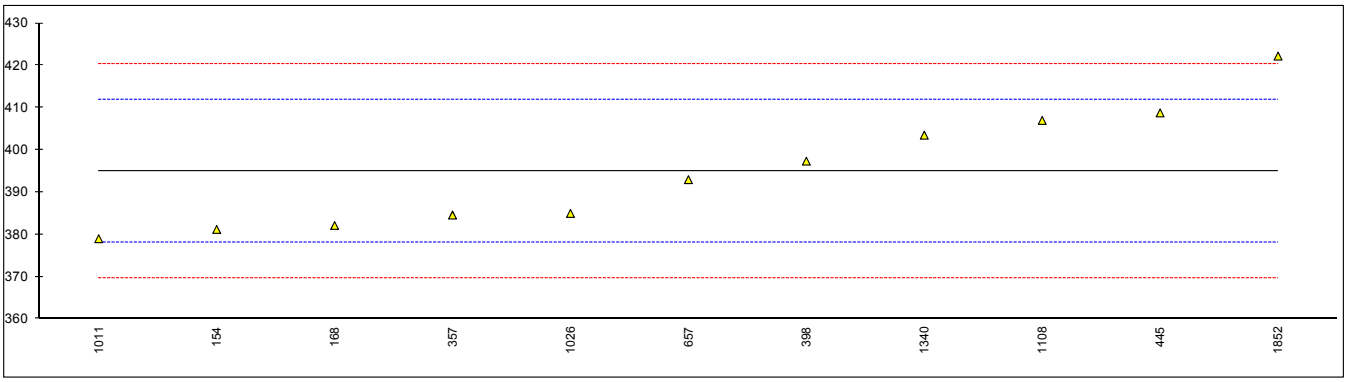


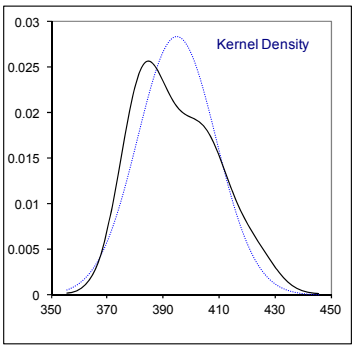
Determination of Kinematic Viscosity at 135°C on sample #15255; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
154	D2170	381.2		-1.62	
168	D2170	382.13		-1.51	
225		-----		-----	
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
337		-----		-----	
353		-----		-----	
357	EN12595	384.6		-1.22	
360		-----		-----	
398	EN12595	397.365		0.29	
399		-----		-----	
440		-----		-----	
444		-----		-----	
445	D2170	408.8		1.64	
604		-----		-----	
657	D2170	393		-0.22	
962		-----		-----	
1011	EN12595	379		-1.88	
1026	EN12595	385		-1.17	
1040		-----		-----	
1108	EN12595	407.0		1.43	
1340	EN12595	403.52		1.02	
1543		-----		-----	
1613		-----		-----	
1724		-----		-----	
1730		-----		-----	
1833		-----		-----	
1842		-----		-----	
1849		-----		-----	
1852	EN12595	422.251		3.23	
1884		-----		-----	
1944		-----		-----	
6022		-----		-----	
6023		-----		-----	

normality	OK
n	11
outliers	0
mean (n)	394.90
st.dev. (n)	14.069
R(calc.)	39.39
R(EN12595:14)	23.69

Compare R(D2170/D2170M:10) = 34.75

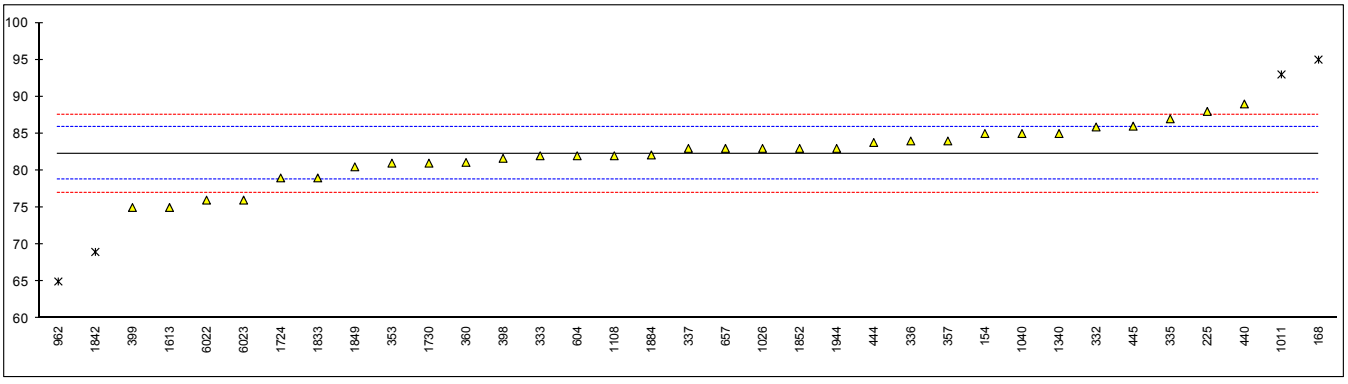


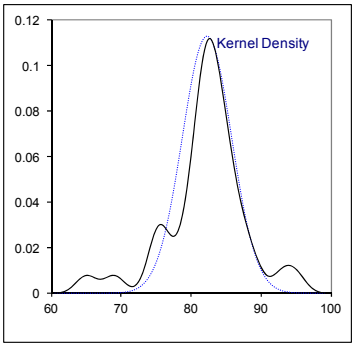


Determination of Penetration at 25°C on sample #15255; results in 0.1 mm

lab	method	value	mark	z(targ)	remarks
154	D5	85		1.54	
168	D5	95	DG(0.05)	7.21	
225	D5	88		3.24	
332	EN1426	85.9		2.05	
333	EN1426	82		-0.17	
335	EN1426	87		2.67	
336	EN1426	84		0.97	
337	EN1426	83	C	0.40	first reported: 56
353	EN1426	81		-0.73	
357	EN1426	84		0.97	
360	EN1426	81.1		-0.68	
398	EN1426	81.66		-0.36	
399	EN1426	75		-4.14	
440	IP49	89.0		3.80	
444	IP49	83.8		0.86	
445	IP49	86		2.10	
604	D5	82		-0.17	
657	D5	83		0.40	
962	D5	65	DG(0.05)	-9.81	
1011	EN1426	93	DG(0.05)	6.07	
1026	EN1426	83		0.40	
1040	EN1426	85.0		1.54	
1108	EN1426	82		-0.17	
1340	EN1426	85		1.54	
1543		-----		-----	
1613	D5	75	C	-4.14	first reported: 57
1724	D5	79		-1.87	
1730	EN1426	81		-0.73	
1833	EN1426	79		-1.87	
1842	IP49	69	DG(0.05)	-7.54	
1849	EN1426	80.5		-1.02	
1852	EN1426	83		0.40	
1884	EN1426	82.1		-0.11	
1944	EN1426	83		0.40	
6022	EN1426	76	C	-3.57	first reported: 75, 76, 77, 77, 75, 76
6023	EN1426	76	C	-3.57	first reported: 75, 76, 77, 76, 74, 75

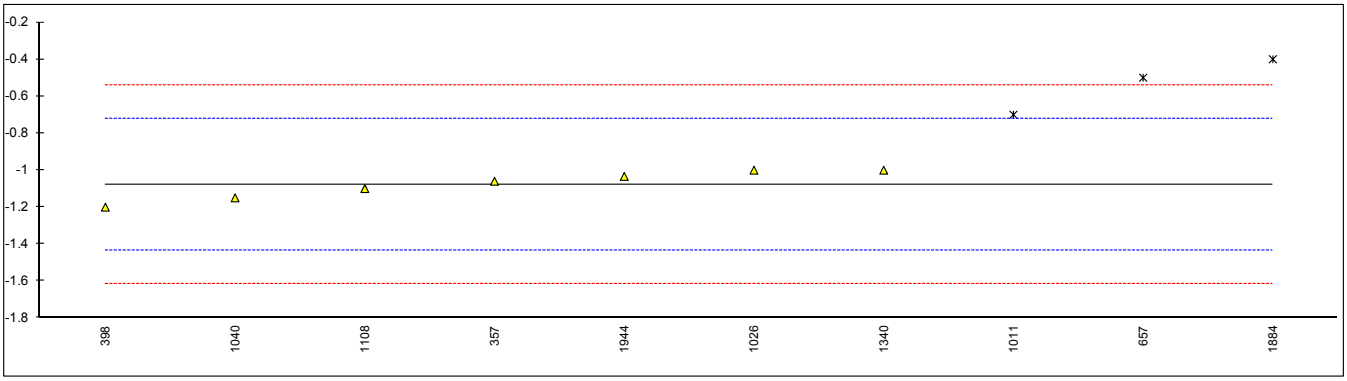
			EN1426 only	D5 only
normality	OK		OK	unknown
n	31		22	6
outliers	4		1	2
mean (n)	82.29		81.83	82.00
st.dev. (n)	3.535		3.123	4.561
R(calc.)	9.90		8.74	12.772
R(EN1426:15)	4.94	Compare R(D5/D5M:13) = 10.33	4.91	10.19

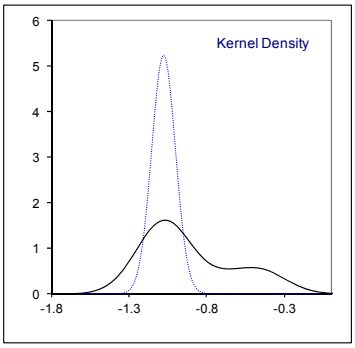




Determination of Penetration Index on sample #15255;

lab	method	value	mark	z(targ)	iis calc.	remarks
154		-----		-----	-----	
168		-----		-----	-----	
225		-----		-----	-0.869	
332		-----		-----	-1.060	
333		-----		-----	-0.890	
335		-----		-----	-0.902	
336		-----		-----	-1.001	
337		-----		-----	-1.034	
353		-----		-----	-1.162	
357	EN12591	-1.06		0.10	-1.061	
360		-----		-----	-1.301	
398	EN12591	-1.2	E	-0.68	-1.261	error in calculation
399		-----		-----	-1.308	
440		-----		-----	-0.507	
444		-----		-----	-0.799	
445		-----		-----	-0.208	(excluded in calc. Pen. Index, outlier in Softening Point)
604		-----		-----	-1.173	
657	Calc.	-0.5	D(0.01),E	3.24	-0.738	error in calculation
962		-----		-----	-----	
1011	EN12591	-0.7	ex	2.12	-0.707	excluded for outlier in Pen. and Soft. Point
1026	EN12591	-1.0		0.44	-0.975	
1040	EN12591	-1.15		-0.40	-1.150	
1108	EN12591	-1.1		-0.12	-1.128	
1340	EN12591	-1.0		0.44	-1.028	
1543		-----		-----	-----	
1613		-----		-----	-1.367	
1724		-----		-----	-1.051	
1730		-----		-----	-0.924	
1833		-----		-----	-1.472	
1842		-----		-----	-0.629	(excluded in calc. Pen. Index, outlier in Pen. and Soft. Point)
1849		-----		-----	-1.179	
1852		-----		-----	-1.095	
1884	EN12591	-0.4	ex	3.80	-0.369	excluded for outlier in Softening Point
1944	EN12591	-1.034		0.24	-1.034	
6022		-----		-----	-1.603	
6023		-----		-----	-1.944	(excluded in calc. Pen. Index, outlier in Softening Point)
	normality	OK			OK	
	n	7			27	
	outliers	1 (+2ex)			0 (+5 ex)	
	mean (n)	-1.078			-1.077	
	st.dev. (n)	0.0764			0.2288	
	R(calc.)	0.214			0.641	
	R(EN12591:09)	0.500			0.500	

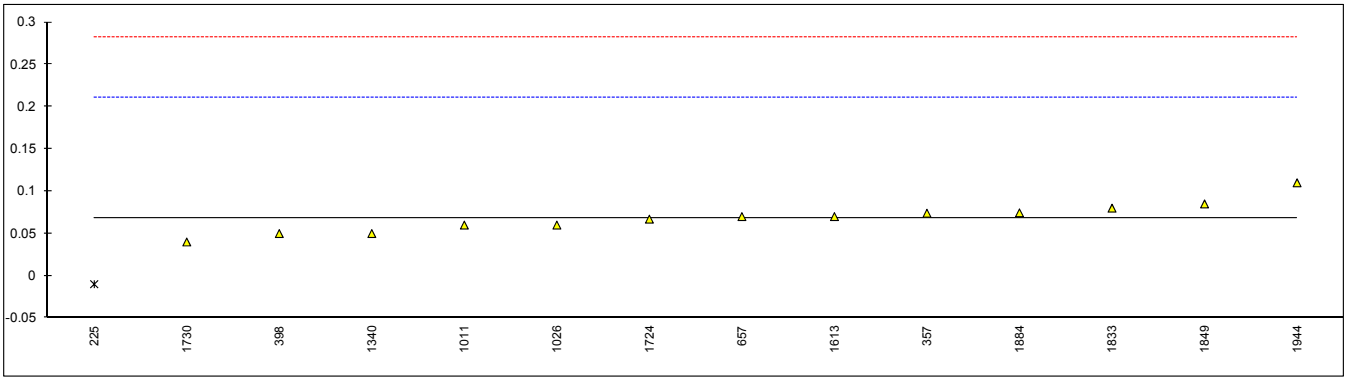


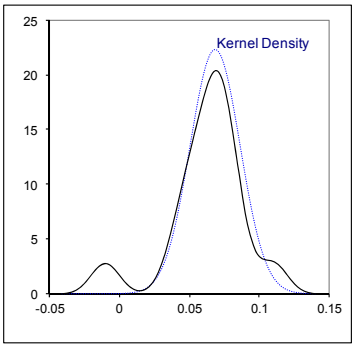


Determination of RTFOT at 163°C, Change of Mass on sample #15255; results in %

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225	EN12607-1	-0.01	G(0.05)	-1.10	
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
337		-----		-----	
353		-----		-----	
357	EN12607-1	0.074		0.08	
360		-----		-----	
398	EN12607-1	0.05		-0.26	
399		-----		-----	
440		-----		-----	
444		-----		-----	
445		-----		-----	
604		-----		-----	
657	D2872	0.07		0.02	
962		-----		-----	
1011	EN12607-1	0.06		-0.12	
1026	EN12607-1	0.06		-0.12	
1040		-----		-----	
1108		-----		-----	
1340	EN12607-1	0.05		-0.26	
1543		-----		-----	
1613	D2872	0.07		0.02	
1724	D2872	0.067		-0.02	
1730	EN12607-1	0.04		-0.40	
1833	EN12607-1	0.08		0.16	
1842		-----		-----	
1849	EN12607-1	0.085		0.23	
1852		-----		-----	
1884	EN12607-1	0.0745		0.08	
1944	EN12607-1	0.11		0.58	
6022		-----		-----	
6023		-----		-----	

normality	suspect
n	13
outliers	1
mean (n)	0.0685
st.dev. (n)	0.01789
R(calc.)	0.0501
R(EN12607-1:14)	0.2000

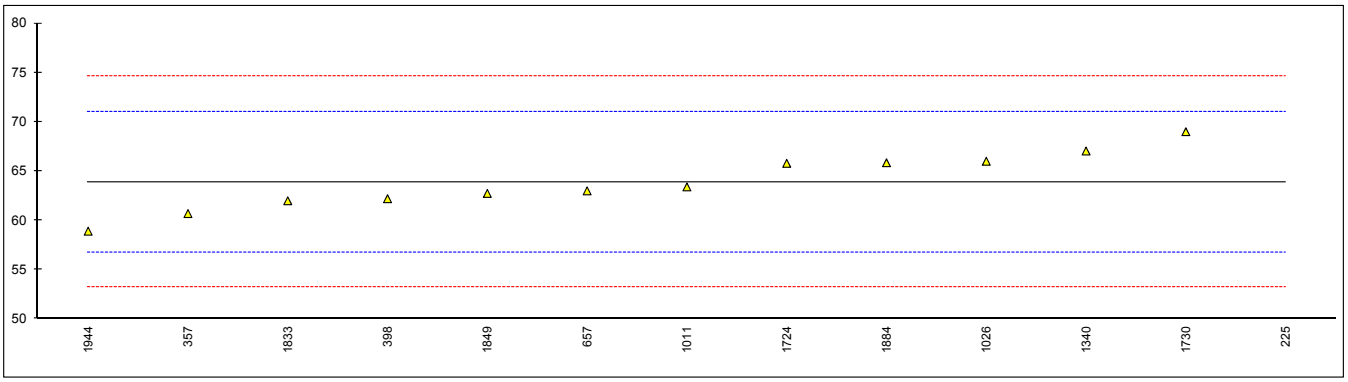


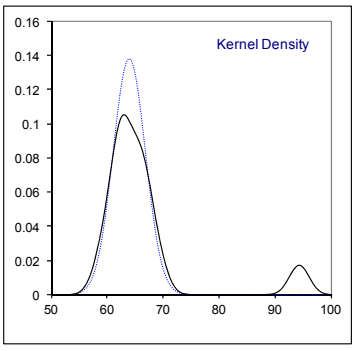


Determination of RTFOT at 163°C, Retained Penetration on sample #15255; results in %

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225	EN12607-1	94.32	G(0.01)	8.52	
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
337		-----		-----	
353		-----		-----	
357	EN12607-1	60.7		-0.89	
360		-----		-----	
398	EN12607-1	62.2		-0.47	
399		-----		-----	
440		-----		-----	
444		-----		-----	
445		-----		-----	
604		-----		-----	
657	D2872	63		-0.25	
962		-----		-----	
1011	EN12607-1	63.4		-0.14	
1026	EN12607-1	66		0.59	
1040		-----		-----	
1108		-----		-----	
1340	EN12607-1	67.05		0.89	
1543		-----		-----	
1613		-----		-----	
1724	D2872	65.8		0.54	
1730	EN12607-1	69		1.43	
1833	EN12607-1	62		-0.53	
1842		-----		-----	
1849	EN12607-1	62.74		-0.32	
1852		-----		-----	
1884	EN12607-1	65.85		0.55	
1944	EN12607-1	58.91		-1.39	
6022		-----		-----	
6023		-----		-----	

normality	OK
n	12
outliers	1
mean (n)	63.89
st.dev. (n)	2.889
R(calc.)	8.09
R(EN12607-1:14)	10.00





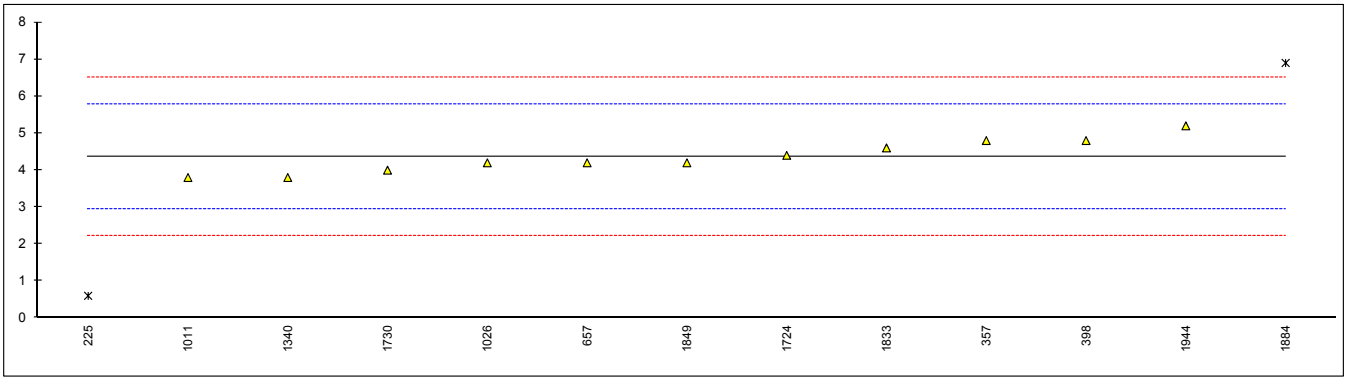
Determination of RTFOT at 163°C, Viscosity Ratio on sample #15255;

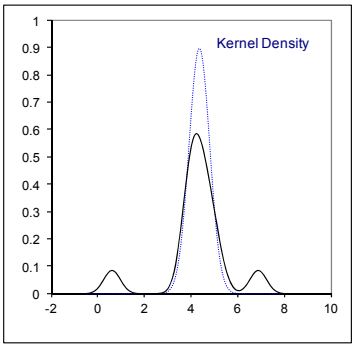
lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225		-----		-----	
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
337		-----		-----	
353		-----		-----	
357	EN12607-1	2.24		-----	
360		-----		-----	
398		-----		-----	
399		-----		-----	
440		-----		-----	
444		-----		-----	
445		-----		-----	
604		-----		-----	
657	D2872	205		-----	Possibly not a ratio, but the viscosity result?
962		-----		-----	
1011		-----		-----	
1026		-----		-----	
1040		-----		-----	
1108		-----		-----	
1340		-----		-----	
1543		-----		-----	
1613		-----		-----	
1724		-----		-----	
1730		-----		-----	
1833		-----		-----	
1842		-----		-----	
1849		-----		-----	
1852		-----		-----	
1884		-----		-----	
1944		-----		-----	
6022		-----		-----	
6023		-----		-----	
	n	2			

Determination of RTFOT at 163°C, Increase in Softening Point on sample #15255; results in °C

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225	EN12607-1	0.6	G(0.05)	-5.27	
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
337		-----		-----	
353		-----		-----	
357	EN12607-1	4.8		0.61	
360		-----		-----	
398	EN12607-1	4.8		0.61	
399		-----		-----	
440		-----		-----	
444		-----		-----	
445		-----		-----	
604		-----		-----	
657	D2872	4.2		-0.23	
962		-----		-----	
1011	EN12607-1	3.8		-0.79	
1026	EN12607-1	4.2		-0.23	
1040		-----		-----	
1108		-----		-----	
1340	EN12607-1	3.8		-0.79	
1543		-----		-----	
1613		-----		-----	
1724	D2872	4.4		0.05	
1730	EN12607-1	4.0		-0.51	
1833	EN12607-1	4.6		0.33	
1842		-----		-----	
1849	EN12607-1	4.2		-0.23	
1852		-----		-----	
1884	EN12607-1	6.9	G(0.01)	3.55	
1944	EN12607-1	5.2		1.17	
6022		-----		-----	
6023		-----		-----	

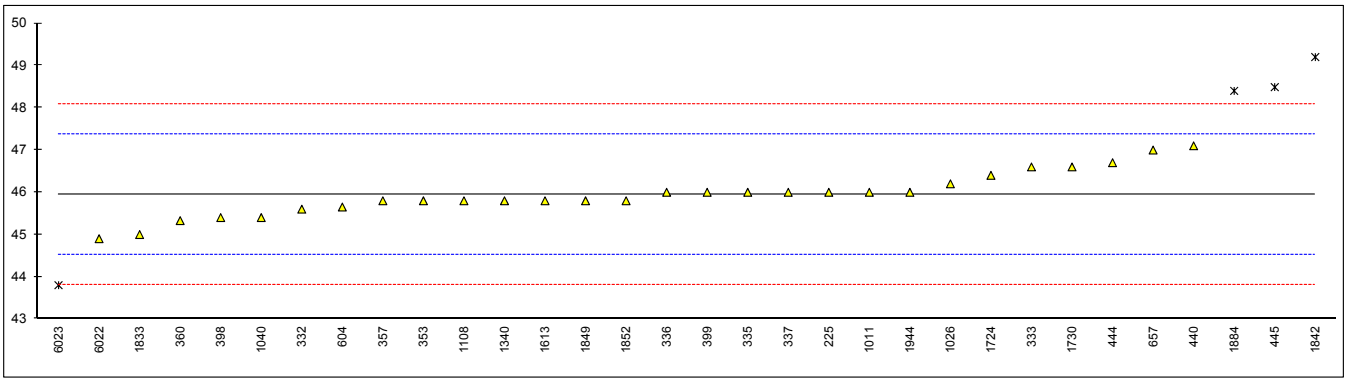
normality	OK
n	11
outliers	2
mean (n)	4.36
st.dev. (n)	0.446
R(calc.)	1.25
R(EN12607-1:14)	2.00

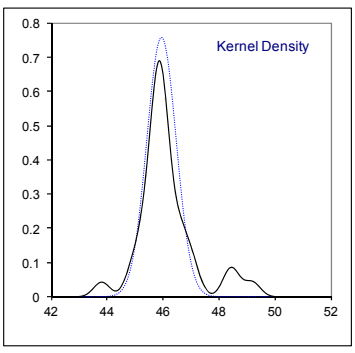




Determination of Softening Point (Ring & Ball) on sample #15255; results in °C

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225	EN1427	46.0		0.08	
332	EN1427	45.6		-0.48	
333	EN1427	46.6		0.92	
335	EN1427	46.0		0.08	
336	EN1427	46.0		0.08	
337	EN1427	46.0	C	0.08	first reported: 49.4
353	EN1427	45.8		-0.20	
357	EN1427	45.8		-0.20	
360	EN1427	45.33		-0.86	
398	EN1427	45.4		-0.76	
399	EN1427	46.0		0.08	
440	IP58	47.1	C	1.62	first reported: 49.1
444	IP58	46.7		1.06	
445	IP58	48.49	R(0.01)	3.56	
604	D36	45.65		-0.41	
657	D36	47.0	C	1.48	first reported: 51.2
962		-----		-----	
1011	EN1427	46.0		0.08	
1026	EN1427	46.2		0.36	
1040	EN1427	45.4		-0.76	
1108	EN1427	45.8		-0.20	
1340	EN1427	45.8		-0.20	
1543		-----		-----	
1613	D36	45.8		-0.20	
1724	D36	46.4		0.64	
1730	EN1427	46.6		0.92	
1833	EN1427	45.0		-1.32	
1842	IP58	49.2	R(0.01)	4.56	
1849	EN1427	45.8		-0.20	
1852	EN1427	45.8		-0.20	
1884	EN1427	48.4	C,R(0.01)	3.44	first reported: 48.325
1944	EN1427	46		0.08	
6022	EN1427	44.9	C	-1.46	first reported: 44.8 / 45.2 / 44.6 / 45.0
6023	EN1427	43.8	C,R(0.01)	-3.00	first reported: 43.2 / 43.6 / 44.0 / 44.2
	normality	OK			
	n	28			
	outliers	4			
	mean (n)	45.95			
	st.dev. (n)	0.526			
	R(calc.)	1.47			
	R(EN1427:15)	2.00			Compare R(ASTM D36/D36M:14e1) = 9.63

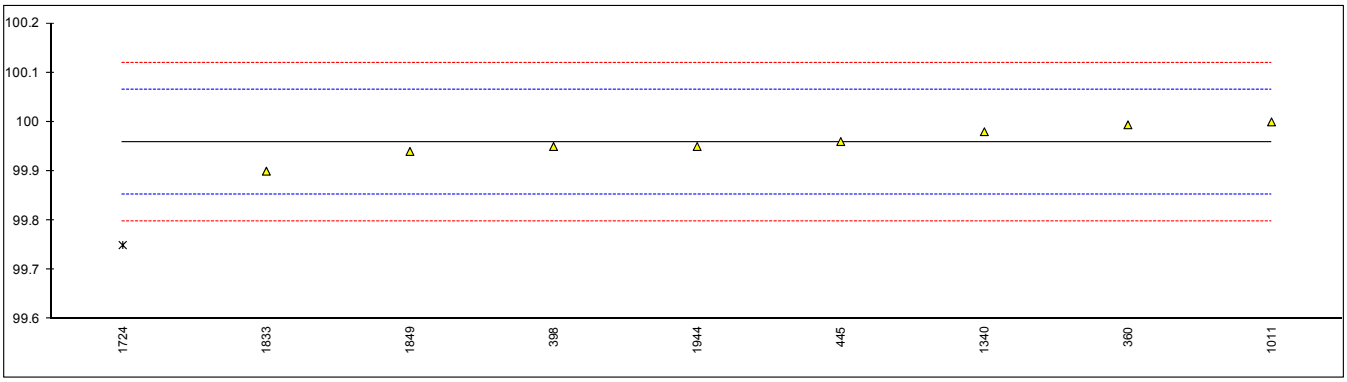


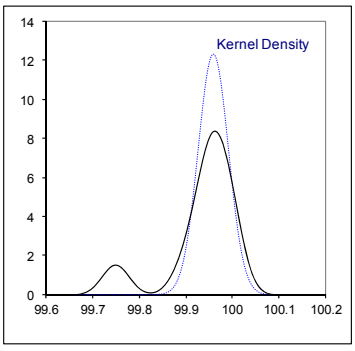


Determination of Solubility in Xylene on sample #15255; results in %M/M

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225		-----		-----	
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
337		-----		-----	
353		-----		-----	
357		-----		-----	
360	EN12592	99.994		0.65	
398	EN12592	99.95		-0.17	
399		-----		-----	
440		-----		-----	
444		-----		-----	
445	IP47	99.96		0.01	
604		-----		-----	
657		-----		-----	
962		-----		-----	
1011	EN12592	100.0		0.76	
1026	EN12592	> 99.0		-----	
1040		-----		-----	
1108		-----		-----	
1340	EN12592	99.98		0.39	
1543		-----		-----	
1613		-----		-----	
1724	EN12592	99.75	D(0.05)	-3.91	
1730		-----		-----	
1833	EN12592	99.90		-1.11	
1842		-----		-----	
1849	EN12592	99.94		-0.36	
1852		-----		-----	
1884		-----		-----	
1944	EN12592	99.95	C	-0.17	first reported: 99.34
6022		-----		-----	
6023		-----		-----	

normality	OK
n	8
outliers	1
mean (n)	99.959
st.dev. (n)	0.0324
R(calc.)	0.091
R(EN12592:14)	0.150

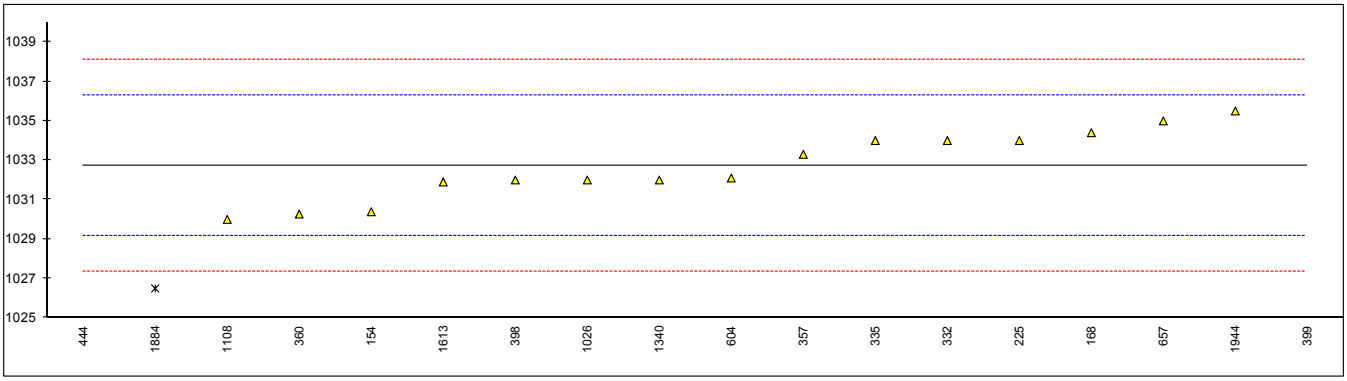


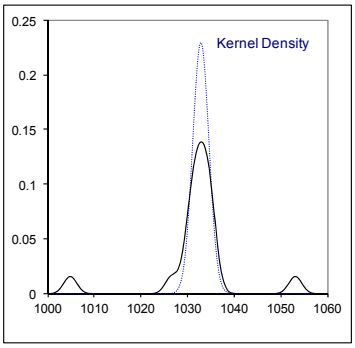


Determination of Density at 25°C on sample #15256; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
154	D70	1030.3872		-1.31	
168	D70	1034.4	C	0.94	reported: 1.0344 kg/m^3
225	D70	1034		0.71	
332	EN15326	1034.0		0.71	
333		-----		-----	
335	EN15326	1034	C	0.71	first reported: 1.034 kg/m^3
336		-----		-----	
337		-----		-----	
353		-----		-----	
357	EN15326	1033.3		0.32	
360	ISO3838	1030.27	C	-1.37	first reported: 1.03027 kg/m^3
398	D70	1032		-0.41	
399	D70	1053.0	G(0.01)	11.35	
440		-----		-----	
444	D70	1004.7	C,G(0.01)	-15.69	first reported: 1010.8
445		-----		-----	
604	D70	1032.1		-0.35	
657	D70	1035		1.27	
962		-----		-----	
1011		-----		-----	
1026	EN15326	1032		-0.41	
1040		-----		-----	
1108	EN15326	1030		-1.53	
1340	EN15326	1032		-0.41	
1543		-----		-----	
1613	DIN51757	1031.9		-0.46	also reported density of 975.14 kg/m3 at 120°C
1724		-----		-----	
1730		-----		-----	
1833		-----		-----	
1842		-----		-----	
1849		-----		-----	
1852		-----		-----	
1884	D70	1026.5	G(0.05)	-3.49	
1944	EN15326	1035.50		1.55	
6022		-----		-----	
6023		-----		-----	

normality	OK
n	15
outliers	3
mean (n)	1032.72
st.dev. (n)	1.741
R(calc.)	4.88
R(EN15326:07)	5.00

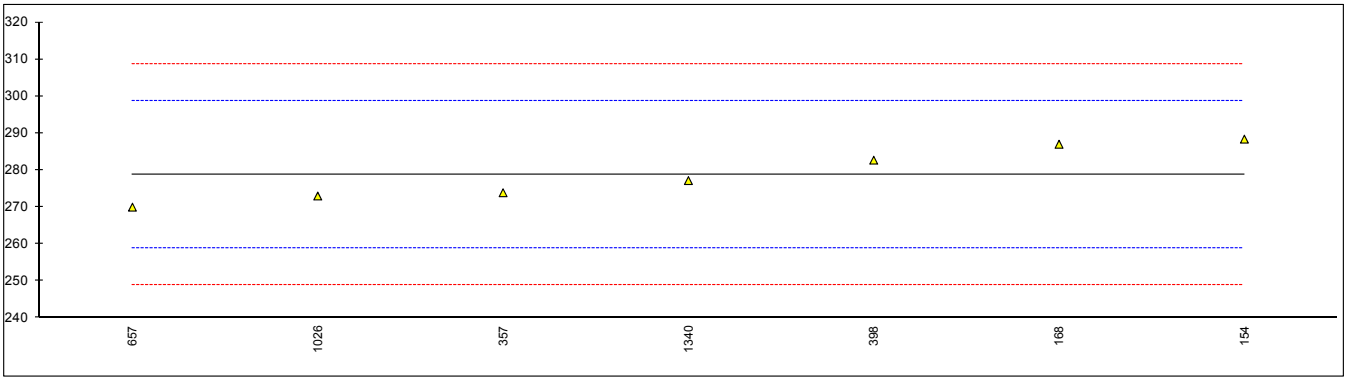




Determination of Dynamic Viscosity at 60°C on sample #15256; results in Pa.s

lab	method	value	mark	z(targ)	remarks
154	D2171	288.4		0.96	
168	D2171	287.0		0.82	
225		-----		-----	
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
337		-----		-----	
353		-----		-----	
357	EN12596	273.9		-0.50	
360		-----		-----	
398	EN12596	282.7		0.38	
399		-----		-----	
440		-----		-----	
444		-----		-----	
445		-----		-----	
604		-----		-----	
657	D2171	270		-0.89	
962		-----		-----	
1011		-----		-----	
1026	EN12596	273		-0.59	
1040		-----		-----	
1108		-----		-----	
1340	EN12596	277.175		-0.17	
1543		-----		-----	
1613		-----		-----	
1724		-----		-----	
1730		-----		-----	
1833		-----		-----	
1842		-----		-----	
1849		-----		-----	
1852		-----		-----	
1884		-----		-----	
1944		-----		-----	
6022		-----		-----	
6023		-----		-----	

normality	unknown
n	7
outliers	0
mean (n)	278.88
st.dev. (n)	7.214
R(calc.)	20.20
R(EN12596:14)	27.89

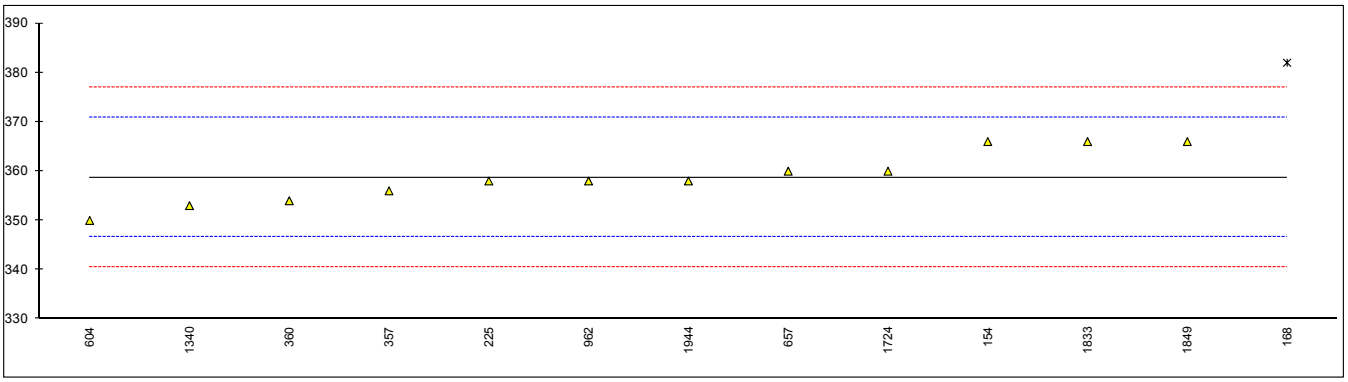


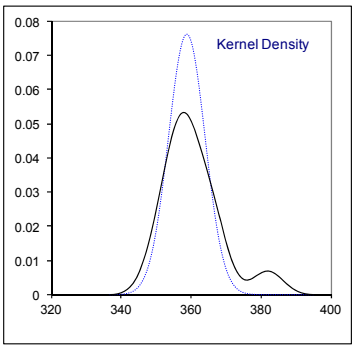
Determination of Flash Point COC on sample #15256; results in °C

lab	method	mode	value	mark	z(targ)	remarks
154	D92	Manual	366	C	1.19	first reported: 326
168	D92		382	G(0.05)	3.83	
225	D92		358		-0.12	
332			-----		-----	
333			-----		-----	
335			-----		-----	
336			-----		-----	
337			-----		-----	
353			-----		-----	
357	ISO2592	Manual	356		-0.45	
360	ISO2592		354.0		-0.78	
398			-----		-----	
399			-----		-----	
440			-----		-----	
444			-----		-----	
445		Manual	-----		-----	
604	D92		350		-1.44	
657	D92	Manual	360		0.21	
962	D92		358		-0.12	
1011	ISO2592	Manual	>280		-----	
1026	ISO2592	Manual	>320		-----	
1040			-----		-----	
1108			-----		-----	
1340	ISO2592	Automated	353		-0.95	
1543			-----		-----	
1613			-----		-----	
1724	D92		360		0.21	
1730			-----		-----	
1833	ISO2592		366		1.19	
1842			-----		-----	
1849	ISO2592		366		1.19	
1852			-----		-----	
1884			-----		-----	
1944	ISO2592	Manual	358		-0.12	
6022			-----		-----	
6023			-----		-----	

normality	OK
n	12
outliers	1
mean (n)	358.8
st.dev. (n)	5.24
R(calc.)	14.7
R(ISO2592:00)	17.0

Compare R(D92:12b) = 18.0

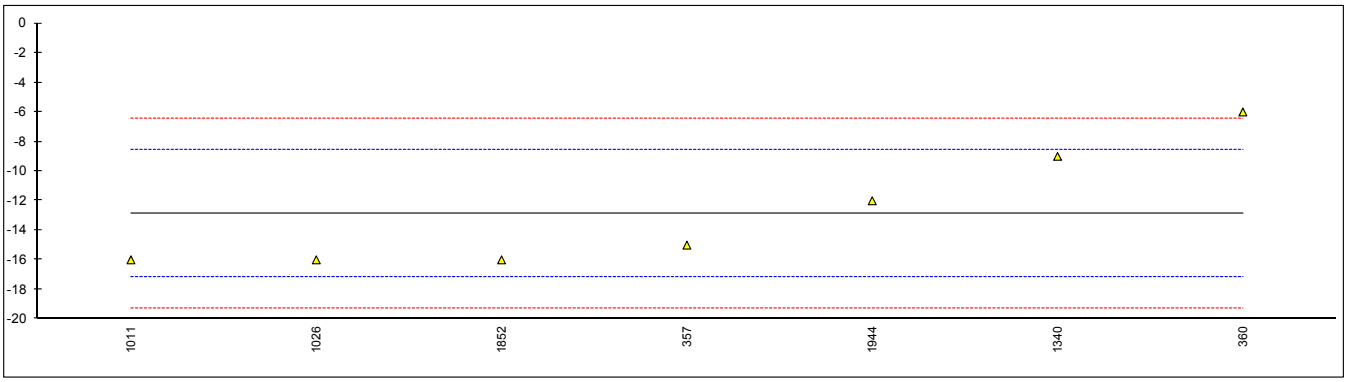




Determination of Fraass Breaking point on sample #15256; results in °C

lab	method	mode	value	mark	z(targ)	remarks
154			-----		-----	
168			-----		-----	
225			-----		-----	
332			-----		-----	
333			-----		-----	
335			-----		-----	
336			-----		-----	
337			-----		-----	
353			-----		-----	
357	EN12593	Manual	-15		-1.00	
360	EN12593	Automated	-6.0		3.20	
398			-----		-----	
399			-----		-----	
440			-----		-----	
444			-----		-----	
445			-----		-----	
604			-----		-----	
657			-----		-----	
962			-----		-----	
1011	EN12593	Manual	-16		-1.47	
1026	EN12593	Automated	-16		-1.47	
1040			-----		-----	
1108			-----		-----	
1340	EN12593	Automated	-9		1.80	
1543			-----		-----	
1613			-----		-----	
1724			-----		-----	
1730			-----		-----	
1833			-----		-----	
1842			-----		-----	
1849			-----		-----	
1852	EN12593		-16		-1.47	
1884			-----		-----	
1944	EN12593	Manual	-12		0.40	
6022			-----		-----	
6023			-----		-----	

normality	unknown
n	7
outliers	0
mean (n)	-12.9
st.dev. (n)	4.02
R(calc.)	11.2
R(EN12593:15)	6.0

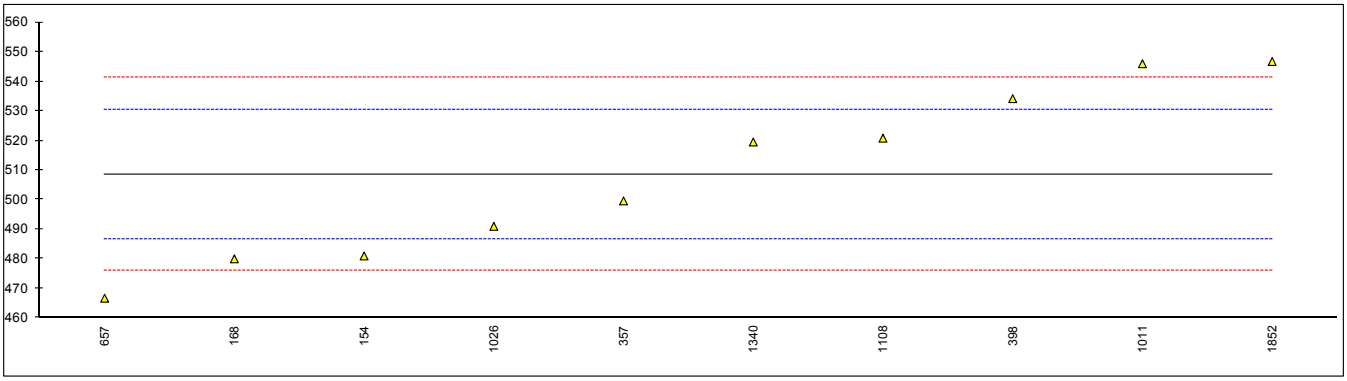


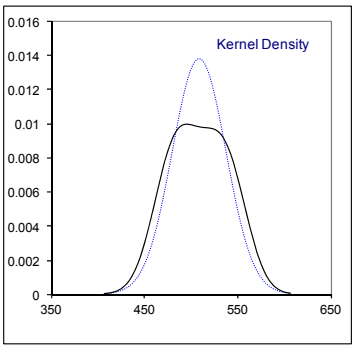
Determination of Kinematic Viscosity at 135°C on sample #15256; results in mm²/s

lab	method	value	mark	z(targ)	remarks
154	D2170	481.0		-2.53	
168	D2170	480.01		-2.62	
225		-----		-----	
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
337		-----		-----	
353		-----		-----	
357	EN12595	499.6		-0.82	
360		-----		-----	
398	EN12595	534.2		2.35	
399		-----		-----	
440		-----		-----	
444		-----		-----	
445		-----		-----	
604		-----		-----	
657	D2170	466.71		-3.84	
962		-----		-----	
1011	EN12595	546		3.44	
1026	EN12595	491		-1.61	
1040		-----		-----	
1108	EN12595	520.8		1.12	
1340	EN12595	519.515		1.01	
1543		-----		-----	
1613		-----		-----	
1724		-----		-----	
1730		-----		-----	
1833		-----		-----	
1842		-----		-----	
1849		-----		-----	
1852	EN12595	546.753		3.50	
1884		-----		-----	
1944		-----		-----	
6022		-----		-----	
6023		-----		-----	

normality	OK
n	10
outliers	0
mean (n)	508.56
st.dev. (n)	28.872
R(calc.)	80.84
R(EN12595:14)	30.51

Compare R(D2170/D2170M:10) = 44.75

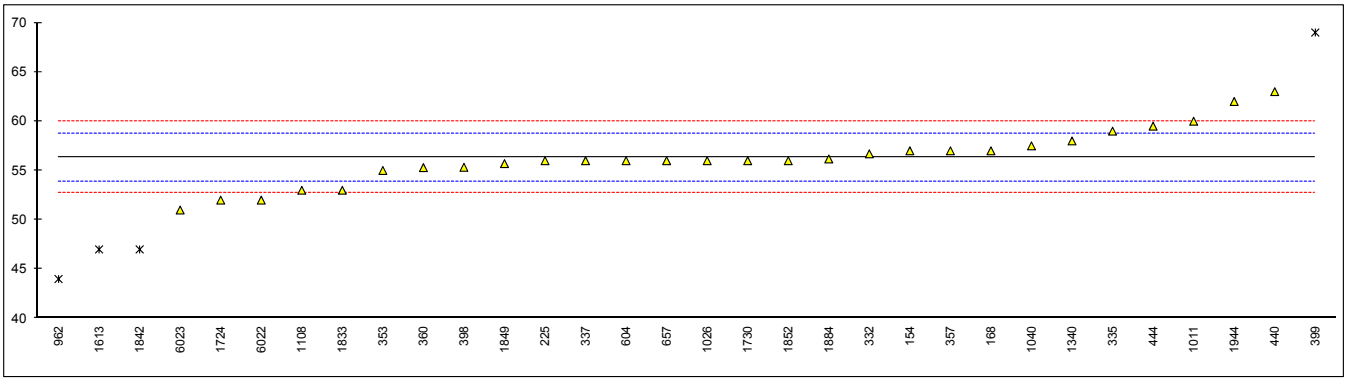


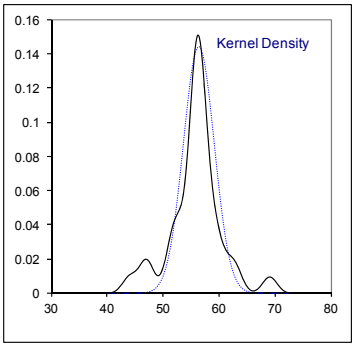


Determination of Penetration at 25°C on sample #15256; results in 0.1 mm

lab	method	value	mark	z(targ)	remarks
154	D5	57		0.56	
168	D5	57		0.56	
225	D5	56		-0.27	
332	EN1426	56.7		0.31	
333		-----		-----	
335	EN1426	59		2.23	
336		-----		-----	
337	EN1426	56	C	-0.27	first reported: 83
353	EN1426	55		-1.11	
357	EN1426	57		0.56	
360	EN1426	55.3		-0.86	
398	EN1426	55.33		-0.83	
399	EN1426	69	G(0.05)	10.58	
440	IP49	63		5.57	
444	IP49	59.5		2.65	
445		-----		-----	
604	D5	56		-0.27	
657	D5	56		-0.27	
962	D5	44	G(0.01)	-10.29	
1011	EN1426	60		3.06	
1026	EN1426	56		-0.27	
1040	EN1426	57.5		0.98	
1108	EN1426	53		-2.78	
1340	EN1426	58		1.40	
1543		-----		-----	
1613	D5	47	DG(0.05)	-7.79	
1724	D5	52		-3.61	
1730	EN1426	56		-0.27	
1833	EN1426	53		-2.78	
1842	IP49	47	DG(0.05)	-7.79	
1849	EN1426	55.7		-0.52	
1852	EN1426	56		-0.27	
1884	EN1426	56.17		-0.13	
1944	EN1426	62		4.73	
6022	EN1426	52	C	-3.61	first reported: 53, 52, 51, 52, 51, 52
6023	EN1426	51	C	-4.45	first reported: 49, 50, 51, 51, 52, 53

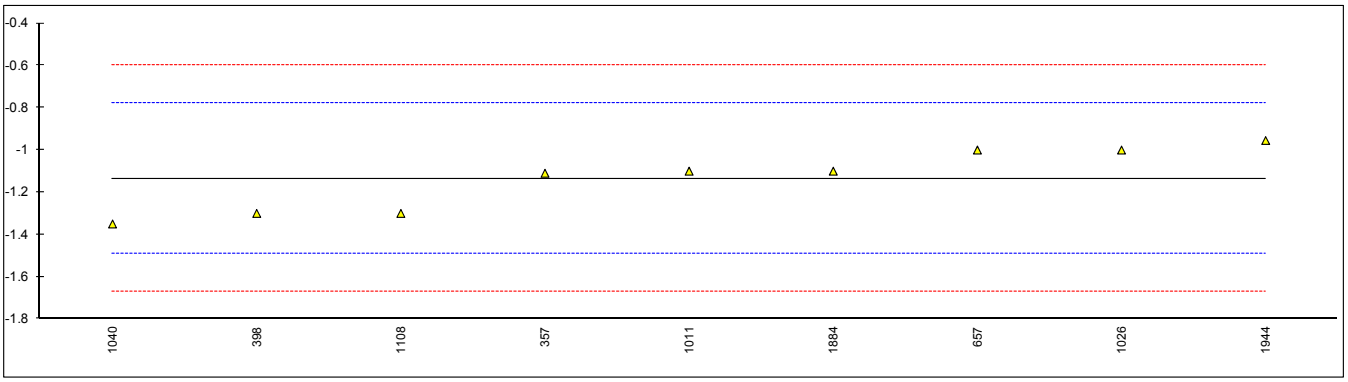
			EN1426 only	D5 only
normality	OK		OK	unknown
n	28		20	6
outliers	4		1	2
mean (n)	56.33		56.03	55.67
st.dev. (n)	2.762		2.613	1.862
R(calc.)	7.73		7.32	5.21
R(EN1426:15)	3.35	Compare R(D5/D5M:13) = 7.08	3.36	7.08

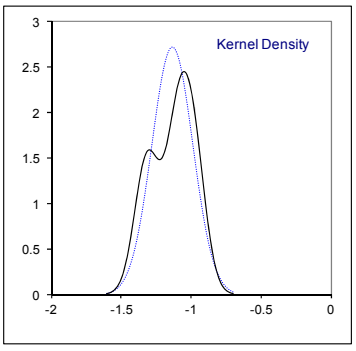




Determination of Penetration Index on sample #15256;

lab	method	value	mark	z(targ)	iis calc.	remarks
154		-----		-----	-----	
168		-----		-----	-----	
225		-----		-----	-1.463	
332		-----		-----	-1.223	
333		-----		-----	-----	
335		-----		-----	-1.024	
336		-----		-----	-----	
337		-----		-----	-1.097	
353		-----		-----	-1.294	
357	EN12591	-1.11		0.14	-1.106	
360		-----		-----	-1.281	
398	EN12591	-1.3		-0.92	-1.332	
399		-----		-----	-1.578	(excluded for outlier in Pen. and Soft. Point)
440		-----		-----	-0.399	(outlier in calc. penetration index)
444		-----		-----	-0.848	
445		-----		-----	-----	
604		-----		-----	-0.994	
657	Calc.	-1.0	E	0.76	-1.097	error in calculation
962		-----		-----	-----	
1011	EN12591	-1.1		0.20	-1.141	
1026	EN12591	-1.0		0.76	-0.994	
1040	EN12591	-1.35		-1.20	-1.322	
1108	EN12591	-1.3		-0.92	-1.276	
1340		-----		-----	-1.117	
1543		-----		-----	-----	
1613		-----		-----	-1.441	(excluded for outlier in penetration)
1724		-----		-----	-1.017	
1730		-----		-----	-1.200	
1833		-----		-----	-1.431	
1842		-----		-----	-0.488	(excluded for outlier in Pen. and Soft. Point)
1849		-----		-----	-1.058	
1852		-----		-----	-1.252	
1884	EN12591	-1.1		0.20	-1.089	
1944	EN12591	-0.955		1.01	-0.955	
6022		-----		-----	-1.422	
6023		-----		-----	-1.491	
	normality	OK			OK	
	n	9			25	
	outliers	0			4	
	mean (n)	-1.135			-1.181	
	st.dev. (n)	0.1469			0.1720	
	R(calc.)	0.411			0.482	
	R(EN12591:09)	0.500			0.500	

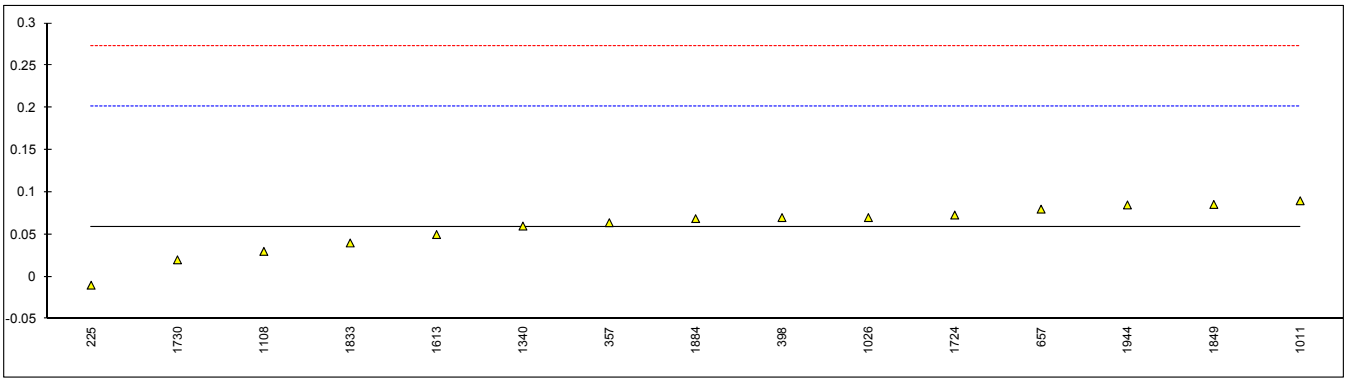


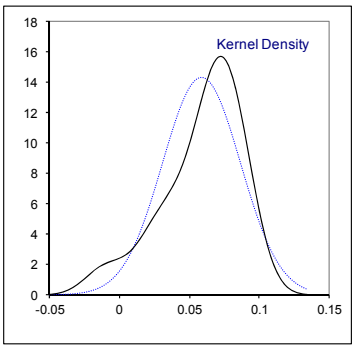


Determination of RTFOT at 163°C, Change of Mass on sample #15256; results in %

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225	EN12607-1	-0.01		-0.96	
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
337		-----		-----	
353		-----		-----	
357	EN12607-1	0.064		0.08	
360		-----		-----	
398	EN12607-1	0.07		0.16	
399		-----		-----	
440		-----		-----	
444		-----		-----	
445		-----		-----	
604		-----		-----	
657	D2872	0.08		0.30	
962		-----		-----	
1011	EN12607-1	0.09		0.44	
1026	EN12607-1	0.07		0.16	
1040		-----		-----	
1108	EN12607-1	0.03		-0.40	
1340	EN12607-1	0.06		0.02	
1543		-----		-----	
1613	D2872	0.05		-0.12	
1724	D2872	0.073		0.20	
1730	EN12607-1	0.02		-0.54	
1833	EN12607-1	0.04		-0.26	
1842		-----		-----	
1849	EN12607-1	0.0856		0.38	
1852		-----		-----	
1884	EN12607-1	0.0688		0.15	
1944	EN12607-1	0.085	C	0.37	first reported: 0.15
6022		-----		-----	
6023		-----		-----	

normality	suspect
n	15
outliers	0
mean (n)	0.0584
st.dev. (n)	0.02791
R(calc.)	0.0781
R(EN12607-1:14)	0.2000

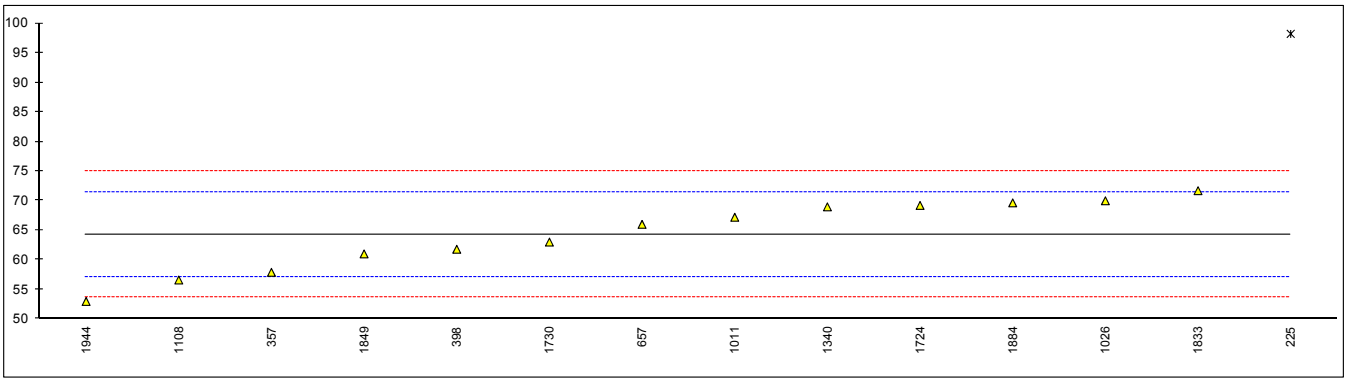


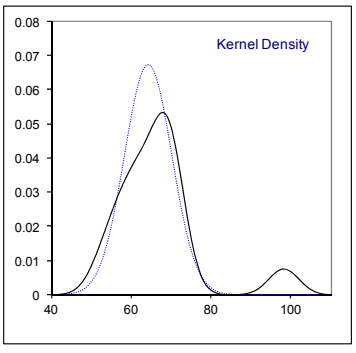


Determination of RTFOT at 163°C, Retained Penetration on sample #15256; results in %

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225	EN12607-1	98.21	G(0.01)	9.49	
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
337		-----		-----	
353		-----		-----	
357	EN12607-1	57.9		-1.79	
360		-----		-----	
398	EN12607-1	61.8		-0.70	
399		-----		-----	
440		-----		-----	
444		-----		-----	
445		-----		-----	
604		-----		-----	
657	D2872	66		0.47	
962		-----		-----	
1011	EN12607-1	67.2		0.81	
1026	EN12607-1	70		1.59	
1040		-----		-----	
1108	EN12607-1	56.6		-2.16	
1340	EN12607-1	68.97		1.31	
1543		-----		-----	
1613		-----		-----	
1724	D2872	69.2		1.37	
1730	EN12607-1	63		-0.37	
1833	EN12607-1	71.7		2.07	
1842		-----		-----	
1849	EN12607-1	61		-0.93	
1852		-----		-----	
1884	EN12607-1	69.64		1.49	
1944	EN12607-1	52.96		-3.18	
6022		-----		-----	
6023		-----		-----	

normality	OK
n	13
outliers	1
mean (n)	64.31
st.dev. (n)	5.920
R(calc.)	16.58
R(EN12607-1:14)	10.00





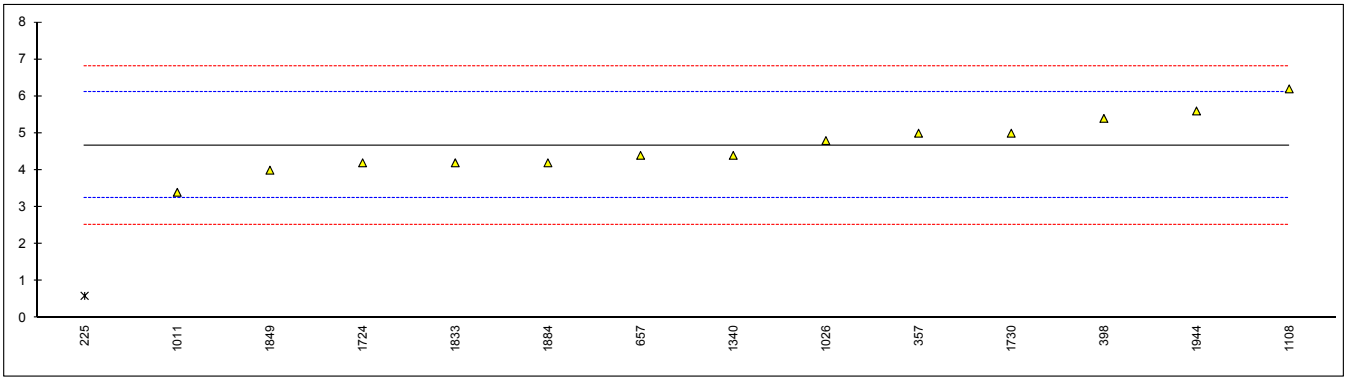
Determination of RTFOT at 163°C, Viscosity Ratio on sample #15256;

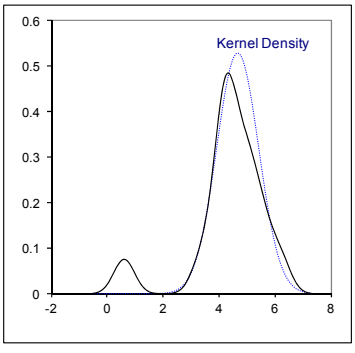
lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225		-----		-----	
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
337		-----		-----	
353		-----		-----	
357	EN12607-1	2.28		-----	
360		-----		-----	
398		-----		-----	
399		-----		-----	
440		-----		-----	
444		-----		-----	
445		-----		-----	
604		-----		-----	
657	D2872	211		-----	Possibly not a ratio, but the viscosity result?
962		-----		-----	
1011		-----		-----	
1026		-----		-----	
1040		-----		-----	
1108		-----		-----	
1340		-----		-----	
1543		-----		-----	
1613		-----		-----	
1724		-----		-----	
1730		-----		-----	
1833		-----		-----	
1842		-----		-----	
1849		-----		-----	
1852		-----		-----	
1884		-----		-----	
1944		-----		-----	
6022		-----		-----	
6023		-----		-----	
	n	2			

Determination of RTFOT at 163°C, Increase in Softening Point on sample #15256; results in °C

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225	EN12607-1	0.6	G(0.01)	-5.71	
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
337		-----		-----	
353		-----		-----	
357	EN12607-1	5.0		0.45	
360		-----		-----	
398	EN12607-1	5.4		1.01	
399		-----		-----	
440		-----		-----	
444		-----		-----	
445		-----		-----	
604		-----		-----	
657	D2872	4.4		-0.39	
962		-----		-----	
1011	EN12607-1	3.4		-1.79	
1026	EN12607-1	4.8		0.17	
1040		-----		-----	
1108	EN12607-1	6.2		2.13	
1340	EN12607-1	4.4		-0.39	
1543		-----		-----	
1613		-----		-----	
1724	D2872	4.2		-0.67	
1730	EN12607-1	5.0		0.45	
1833	EN12607-1	4.2		-0.67	
1842		-----		-----	
1849	EN12607-1	4		-0.95	
1852		-----		-----	
1884	EN12607-1	4.2	C	-0.67	first reported: 2.1
1944	EN12607-1	5.6		1.29	
6022		-----		-----	
6023		-----		-----	

normality	OK
n	13
outliers	1
mean (n)	4.68
st.dev. (n)	0.755
R(calc.)	2.11
R(EN12607-1:14)	2.00

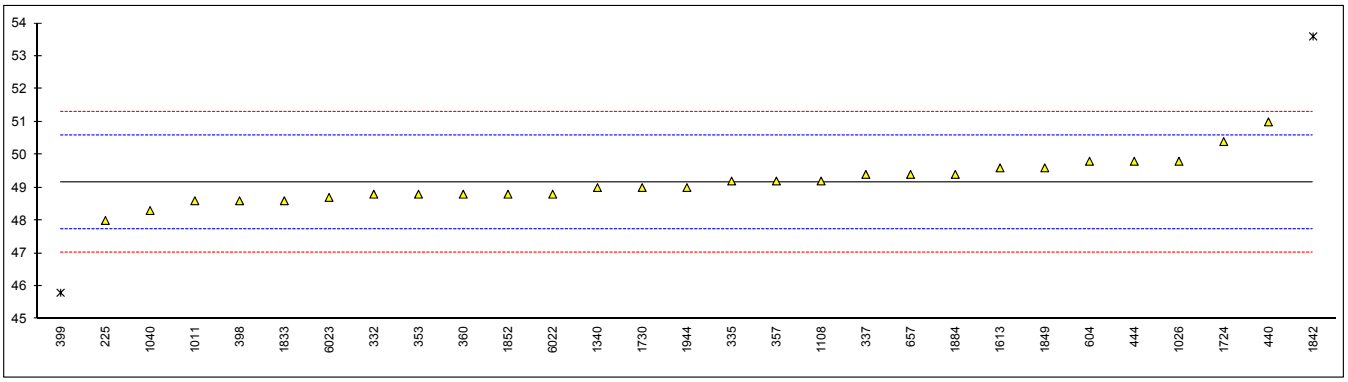


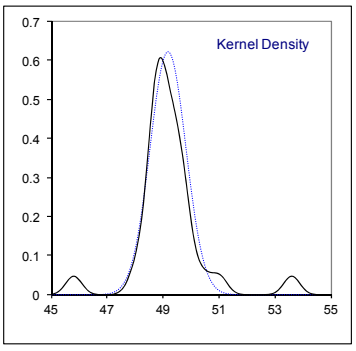


Determination of Softening Point (Ring & Ball) on sample #15256; results in °C

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225	EN1427	48.0		-1.64	
332	EN1427	48.8		-0.52	
333		-----		-----	
335	EN1427	49.2		0.04	
336		-----		-----	
337	EN1427	49.4	C	0.32	first reported: 46.0
353	EN1427	48.8		-0.52	
357	EN1427	49.2		0.04	
360	EN1427	48.80		-0.52	
398	EN1427	48.6		-0.80	
399	EN1427	45.8	R(0.01)	-4.72	
440	IP58	51.0		2.56	
444	IP58	49.8		0.88	
445		-----		-----	
604	D36	49.8		0.88	
657	D36	49.4	C	0.32	first reported: 53.8
962		-----		-----	
1011	EN1427	48.6		-0.80	
1026	EN1427	49.8		0.88	
1040	EN1427	48.3		-1.22	
1108	EN1427	49.2		0.04	
1340	EN1427	49.0		-0.24	
1543		-----		-----	
1613	D36	49.6		0.60	
1724	D36	50.4		1.72	
1730	EN1427	49.0		-0.24	
1833	EN1427	48.6		-0.80	
1842	IP58	53.6	R(0.01)	6.20	
1849	EN1427	49.6		0.60	
1852	EN1427	48.8		-0.52	
1884	EN1427	49.4		0.32	
1944	EN1427	49		-0.24	
6022	EN1427	48.8	C	-0.52	first reported: 48.4 / 48.6 / 49.0 / 49.2
6023	EN1427	48.7	C	-0.66	first reported: 48.0 / 48.4 / 49.0 / 49.2

normality	suspect
n	27
outliers	2
mean (n)	49.17
st.dev. (n)	0.641
R(calc.)	1.79
R(EN1427:15)	2.00

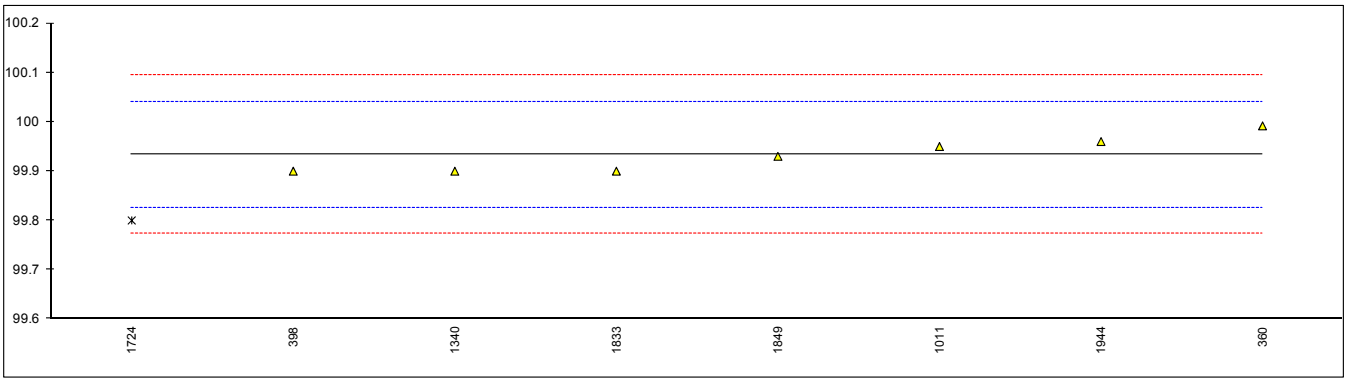


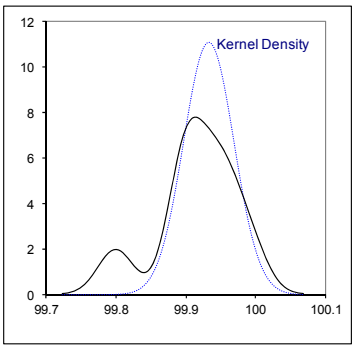


Determination of Solubility in Xylene on sample #15256; results in %M/M

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225		-----		-----	
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
337		-----		-----	
353		-----		-----	
357		-----		-----	
360	EN12592	99.992		1.10	
398	EN12592	99.90		-0.62	
399		-----		-----	
440		-----		-----	
444		-----		-----	
445		-----		-----	
604		-----		-----	
657		-----		-----	
962		-----		-----	
1011	EN12592	99.95		0.31	
1026	EN12592	> 99.0		-----	
1040		-----		-----	
1108		-----		-----	
1340	EN12592	99.90		-0.62	
1543		-----		-----	
1613		-----		-----	
1724	EN12592	99.80	D(0.05)	-2.49	
1730		-----		-----	
1833	EN12592	99.90		-0.62	
1842		-----		-----	
1849	EN12592	99.93		-0.06	
1852		-----		-----	
1884		-----		-----	
1944	EN12592	99.96	C	0.50	first reported: 99.65
6022		-----		-----	
6023		-----		-----	

normality	unknown
n	7
outliers	1
mean (n)	99.933
st.dev. (n)	0.0360
R(calc.)	0.101
R(EN12592:14)	0.150





APPENDIX 2

Number of participants per country

2 labs in ALBANIA
1 lab in BOSNIA and HERZEGOVINA
1 lab in BULGARIA
1 lab in COTE D'IVOIRE
1 lab in FINLAND
5 labs in FRANCE
1 lab in GEORGIA
2 labs in GERMANY
2 labs in GREECE
1 lab in IRELAND
2 labs in ITALY
1 lab in JORDAN
1 lab in MALAYSIA
1 lab in NETHERLANDS
1 lab in PORTUGAL
1 lab in ROMANIA
1 lab in SAUDI ARABIA
1 lab in SINGAPORE
1 lab in SUDAN
3 labs in TURKEY
4 labs in UNITED KINGDOM
2 labs in UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations:

C = final result after checking of first reported suspect result
D(0.01) = outlier in Dixon's outlier test
D(0.05) = straggler in Dixon's outlier test
G(0.01) = outlier in Grubbs' outlier test
G(0.05) = straggler in Grubbs' outlier test
DG(0.01) = outlier in Double Grubbs' outlier test
DG(0.05) = straggler in Double Grubbs' outlier test
R(0.01) = outlier in Rosner outlier test
R(0.05) = straggler in Rosner outlier test
E = probably an error in calculations
W = result withdrawn on request of participant
ex = excluded from calculations
n.a. = not applicable
n.d. = not detected
fr. = first reported
SDS = Safety Data Sheet

Literature:

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3 ASTM E1301-03
4 ISO 5725-86
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6 ISO 13528:05
7 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
9 IP 367/84
10 DIN 38402 T41/42
11 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
12 J.N. Miller, Analyst, 118, 455, (1993)
13 Analytical Methods Committee Technical brief, No 4.January 2001
14 The Royal Society of Chemistry 2002, Analyst, 2002, 127, page 1359-1364, P.J. Lowthian and M. Thompson. (see http://www.rsc.org/suppdata/an/b2/b205600n/)
15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), pp. 165-172, (1983)