Results of Proficiency Test
Bitumen
November 2017

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.
Since 2014, the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for Bitumen. During the annual proficiency testing program 2017/2018, it was decided to continue the round robin for the analysis of Bitumen in accordance with the last applicable version of EN12591 Paving Grade. In this interlaboratory study 51 laboratories in 22 different countries registered for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2017 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 organiser of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send one container of 1 litre Bitumen, a 70/100 grade (labelled #17260). 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/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 March 2017 (iis-protocol, version 3.4). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

In this proficiency test one batch, a straight-run bitumen, 70/100 grade was supplied by a third party. After heating, 77 metal cans of 1 liter were filled and labelled #17260. The homogeneity of the subsamples #17260 was checked by determination of penetration (EN 1426) and softening point (EN 1427) on 4 stratified randomly selected samples.

	Penetration at 25°C in 0.1 mm	Softening Point in °C
Sample #17260-1	84	46.0
Sample #17260-2	83	45.8
Sample #17260-3	83	46.0
Sample #17260-4	85	46.2

Table 1: homogeneity test results of subsamples #17260

From the above test results, the repeatabilities were calculated and compared with the repeatability of the corresponding test method, 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)	2.7	0.5
Reference test method	EN1426:15	EN1427:15
r (Reference test method)	3.4	1.0

Table 2: repeatabilities of subsamples #17260

The calculated repeatability for Penetration and Softening Point were both in full agreement with the repeatability of the respective reference test method. Therefore, homogeneity of the subsamples of #17260 was assumed.

To each of the participating laboratories a 1 litre metal can, labelled #17260 was sent on November 22, 2017. A 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 ANALYSES

The participants were asked to determine on sample #17260: 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, Retained Penetration, Viscosity Ratio and Increase in Softening Point), Softening Point (Ring and Ball) and Solubility in Xylene.

It was explicitly requested to treat the samples as if they were routine samples 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 calculations.

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 that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

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

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment.

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

3.1 STATISTICS

The protocol followed in the 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 March 2017 (iis-protocol, version 3.4).
For the statistical evaluation, the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.

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

According to ISO 5725 the original test 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. 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 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 analysis 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 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. The Kernel Density Graph is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, 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. In some cases, a reproducibility based on former iis proficiency tests could be used.

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

The z-scores were calculated according to:

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

The $z_{(target)}$ scores are listed in the result tables of appendix 1.

Absolute values for z<2 are very common and absolute values for z>3 are very rare. Therefore, the usual interpretation of z-scores is as follows:

	$\lvert z \rvert$	< 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 three other laboratories reported the test results after the final reporting date. Not all laboratories were able to report all analyses requested.
Finally, 50 participants reported in total 289 numerical results. Observed were 7 outlying test results, which is 2.4%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

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

4.1 EVALUATION PER TEST

In this section, the reported test results are discussed per test. The 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.

Density at 25°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of EN15326:07.

Dynamic Viscosity at 60°C: This determination was very problematic. Only one statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of EN12596:14.

Flash Point COC: This determination was very problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ISO2592:00 and/or ASTM D92:12b.

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 very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of EN12595:14 and/or with the less strict requirements of ASTM D2170/2170M:10.

Penetration: This determination was problematic depending on the test method used. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the strict requirements of EN1426:15, but is in full agreement with the less strict requirements of ASTM D5/D5M:13.
When the test results of test method EN1426 (n=37) and ASTM D5/D5M (n=11) are evaluated separately, the reproducibility for the EN1426 data is not in agreement, but the ASTM D5 data is in good agreement with the respective method requirements.

Different factors could cause this large variation, such as preparation, temperature and needle. During the measurement, the temperature should be kept at 25°C, by immersing the sample in sufficient water of this temperature (for measurements outside of the waterbath, a transfer dish of 350 ml should be used). Deviations from this temperature will have influence on the penetration. Another factor is the tip of the needle used. This tip should keep the same

dimensions/surface through out testing in time. In practise, it will get abrasion and wear and should be replaced regularly.

Penetration Index: This determination was problematic. Five test results appeared to have an error in calculation and one other test result was excluded, because the test result of the Penetration of this laboratory was an outlier. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of EN12591:09.

The Penetration Index was calculated by iis for all laboratories, which reported both Penetration and Softening Point.
The values for Penetration Index, calculated by iis, were also statistically evaluated. One test result was excluded as this laboratory had an outlier in the Penetration test result. The calculated reproducibility was not in agreement with the requirements of EN12591:09.

RTFOT: The Rolling Thin Film Oven Test (RTFOT, EN12607-1 or ASTM D2872), 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 reported test results for Change of Mass appeared to be out of the application range ($0.3 < x < 0.8$ %absolute) of the test method EN12607-1:14. Therefore, no significant conclusions were drawn.

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

The determination on Viscosity Ratio was very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of EN12607-1:14. The low number of reported test results may (partly) explain the large variation found.

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

Softening Point: This determination was problematic depending on the test method used. One statistical outlier was observed. The calculated reproducibility is not in agreement with the requirements of EN1427:15. However, it is in good agreement with the less strict requirements of ASTM D36/D36M:13.

Solubility in Xylene: This determination may be very problematic. No statistical outliers were observed. The calculated reproducibility is larger than the calculated reproducibility of the previous PT: iis15F02 (1.84 vs 1.71). As it is not clear what the cause of the large variation may be, no z-scores were calculated.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

	Unit	n	average	2.8 * sd	R (lit)
Density at 25°C	kg/m^3	27	1015.5	5.0	5.0
Dynamic Viscosity at 60°C	Pa.s	13	197.9	40.2	19.8
Flash Point COC	°C	21	329	35	17
Fraass Breaking Point	°C	13	-15	8	6
Kinematic Viscosity at 135°C	mm^2/s	17	518.4	66.7	31.1
Penetration at 25°C	0.1 mm	48	82.7	10.5	5.0
Penetration Index		24	-0.98	0.89	0.50
RTFOT - Change of Mass	%	20	-0.034	0.45	(0.200)*
RTFOT - Retained Penetration	%	18	61.7	7.6	10.0
RTFOT - Viscosity Ratio		6	2.32	0.98	0.46
RTFOT - Increase in Soft. Point	°C	17	5.2	1.7	2.0
Softening Point (Ring and Ball)	°C	46	46.4	2.4	2.0
Solubility in Xylene	% M/M	11	99.10	1.84	(0.15)

Table 3: summary of test results on Bitumen 70/100 grade

()* Results between brackets are for information only

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2017 WITH PREVIOUS PTS

	December 2017	December 2016	December 2015	December 2014
Number of reporting labs	50	51	35	36
Number of results reported	289	318	388	464
Number of statistical outliers	7	8	30	18
Percentage statistical outliers	2.4%	2.5%	7.7%	3.9%

Table 4: comparison with previous proficiency tests

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

	2017	2016	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	n.e.	n.e.	+	+	+/-	+

Table 5: comparison determinations against the test method

()* Results between brackets are for information only

The following performance categories were used:

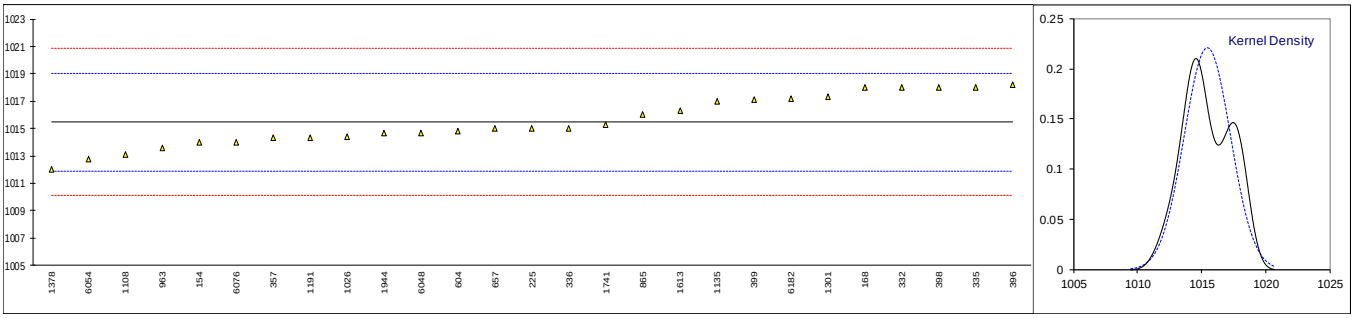
- ++: group performed much better than the reference test method
- + : group performed better than the reference test method
- +/-: group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test method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

APPENDIX 1

Determination of Density at 25°C on sample #17260; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
154	D70	1014		-0.83	
168	D70	1018		1.41	
225	D70	1015		-0.27	
332	EN15326	1018		1.41	
333		-----		-----	
335	EN15326	1018.0		1.41	
336	EN15326	1015	C	-0.27	Reported 1.015 kg/m3
353		-----		-----	
357	EN15326	1014.3		-0.66	
396	ISO3838	1018.2		1.52	
398	EN15326	1018		1.41	
399	D70	1017.1		0.91	
444		-----		-----	
447		-----		-----	
604	D70	1014.8		-0.38	
657	D70	1015		-0.27	
865	D70	1016.0		0.29	
962		-----		-----	
963	D70	1013.6		-1.05	
1011		-----		-----	
1026	EN15326	1014.4		-0.61	
1040		-----		-----	
1108	EN15326	1013.1		-1.33	
1135	EN15326	1017	C	0.85	Reported 1.017 kg/m3
1191	EN15326	1014.3		-0.66	
1301	EN15326	1017.3		1.02	
1378	D70	1012		-1.95	
1402		-----		-----	
1613	DIN51757	1016.3		0.46	
1724		-----		-----	
1730		-----		-----	
1741	EN15326	1015.3		-0.10	
1833		-----		-----	
1849		-----		-----	
1852		-----		-----	
1944	EN15326	1014.66		-0.46	
6048	EN15326	1014.7		-0.44	
6054	D70	1012.77200	C	-1.52	Reported 1.01277200 kg/m3
6076	EN15326	1014		-0.83	
6093		-----		-----	
6094		-----		-----	
6095		-----		-----	
6096		-----		-----	
6097		-----		-----	
6098		-----		-----	
6148		-----		-----	
6149		-----		-----	
6150		-----		-----	
6151		-----		-----	
6152		-----		-----	
6182	EN15326	1017.2	C	0.96	Reported 1.0172 kg/m3

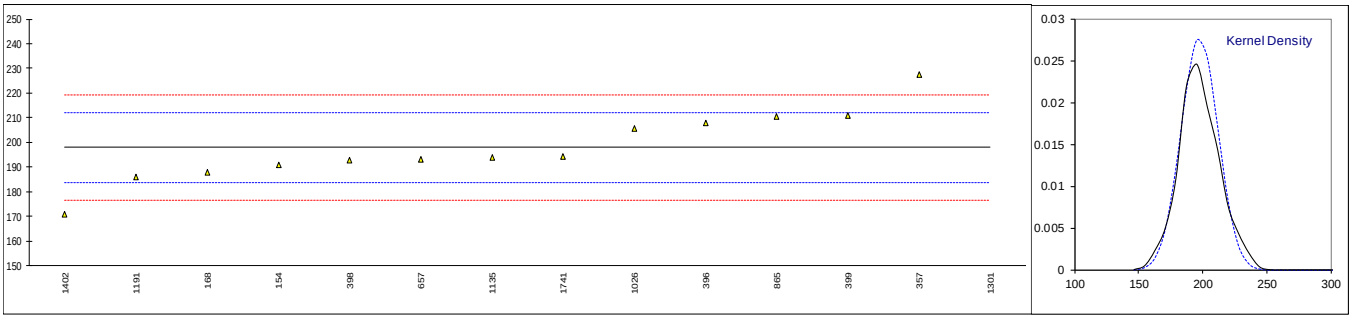
normality	OK
n	27
outliers	0
mean (n)	1015.48
st.dev. (n)	1.802
R(calc.)	5.04
st.dev.(EN15326:07)	1.786
R(EN15326:07)	5.00



Determination of Dynamic Viscosity at 60°C on sample #17260; results in Pa.s

lab	method	value	mark	z(targ)	remarks
154	D2171	191		-0.97	
168	D2171	188		-1.40	
225		-----		-----	
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357	EN12596	227.7		4.22	
396	EN12596	207.7		1.39	
398	EN12596	192.7		-0.73	
399	EN12596	211		1.86	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2171	193		-0.69	
865	D2171	210.4		1.77	
962		-----		-----	
963		-----		-----	
1011		-----		-----	
1026	EN12596	205.6		1.09	
1040		-----		-----	
1108		-----		-----	
1135	EN12956	194		-0.55	
1191	EN12596	186		-1.68	
1301	EN12596	2205	G(0.01)	284.03	
1378		-----		-----	
1402	EN12596	170.854803		-3.82	
1613		-----		-----	
1724		-----		-----	
1730		-----		-----	
1741	EN12596	194.3		-0.50	
1833		-----		-----	
1849		-----		-----	
1852		-----		-----	
1944		-----		-----	
6048		-----		-----	
6054		-----		-----	
6076		-----		-----	
6093		-----		-----	
6094		-----		-----	
6095		-----		-----	
6096		-----		-----	
6097		-----		-----	
6098		-----		-----	
6148		-----		-----	
6149		-----		-----	
6150		-----		-----	
6151		-----		-----	
6152		-----		-----	
6182		-----		-----	

normality	OK
n	13
outliers	1
mean (n)	197.87
st.dev. (n)	14.344
R(calc.)	40.16
st.dev.(EN12596:14)	7.067
R(EN12596:14)	19.79

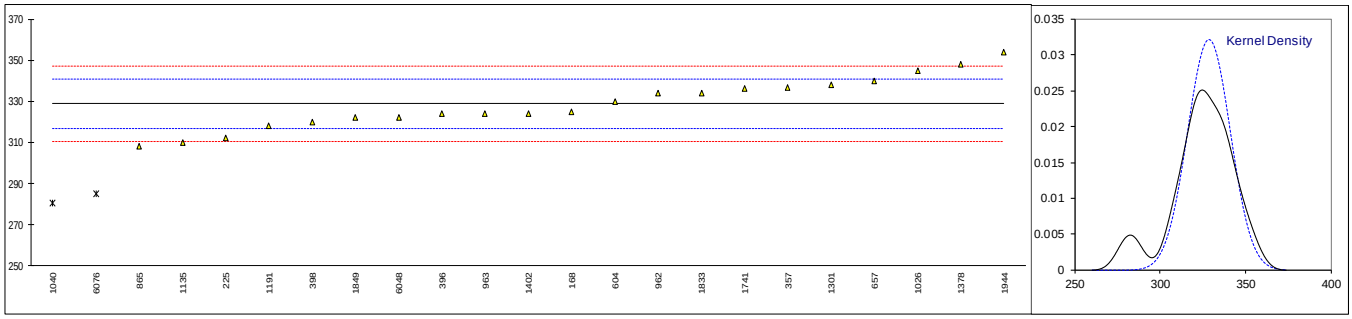


Determination of Flash Point COC on sample #17260; results in °C

lab	method	mode	value	mark	z(targ)	remarks
154		-----	-----		-----	
168	D92	-----	325		-0.63	
225	D92	-----	312		-2.77	
332		-----	-----		-----	
333		-----	-----		-----	
335		-----	-----		-----	
336		-----	-----		-----	
353		-----	-----		-----	
357	ISO2592	Automated	336.6		1.28	
396	ISO2592	Manual	324		-0.79	
398	ISO2592	Manual	320		-1.45	
399		-----	-----		-----	
444		-----	-----		-----	
447		-----	-----		-----	
604	D92	Manual	330		0.20	
657	D92	Manual	340		1.84	
865	D92	Manual	308.2		-3.40	
962	D92	-----	334		0.85	
963	D92	Manual	324		-0.79	
1011		-----	-----		-----	
1026	ISO2592	Manual	345		2.67	
1040	ISO2719	Automated	280.5	DG(0.05)	-7.96	
1108		-----	-----		-----	
1135	ISO2592	Automated	310.0		-3.10	
1191	ISO2592	Automated	318		-1.78	
1301	ISO2592	Automated	338.0		1.51	
1378	D92	Automated	348		3.16	
1402	ISO2592	Automated	324		-0.79	
1613		-----	-----		-----	
1724		-----	-----		-----	
1730		-----	-----		-----	
1741	ISO2592	-----	336.3		1.23	
1833	ISO2592	Automated	334		0.85	
1849	ISO2592	-----	322		-1.12	
1852		-----	-----		-----	
1944	ISO2592	Manual	354		4.15	
6048	ISO2592	Automated	322		-1.12	
6054		-----	-----		-----	
6076	ISO2592	Automated	285	DG(0.05)	-7.22	
6093		-----	-----		-----	
6094		-----	-----		-----	
6095		-----	-----		-----	
6096		-----	-----		-----	
6097		-----	-----		-----	
6098		-----	-----		-----	
6148		-----	-----		-----	
6149		-----	-----		-----	
6150		-----	-----		-----	
6151		-----	-----		-----	
6152		-----	-----		-----	
6182		-----	-----		-----	

normality	OK
n	21
outliers	2
mean (n)	328.81
st.dev. (n)	12.408
R(calc.)	34.74
st.dev.(ISO2592:00)	6.071
R(ISO2592:00)	17.00

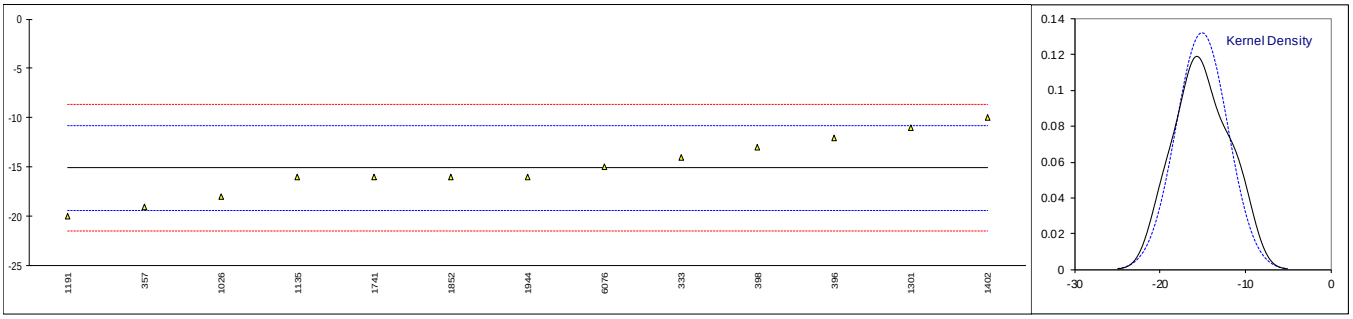
Comapre R(D92:12b) = 18.00



Determination of Fraass Breaking point on sample #17260; results in °C

lab	method	mode	value	mark	z(targ)	remarks
154		-----	-----		-----	
168		-----	-----		-----	
225		-----	-----		-----	
332		-----	-----		-----	
333	EN12593	Automated	-14		0.50	
335		-----	-----		-----	
336		-----	-----		-----	
353		-----	-----		-----	
357	EN12593	Automated	-19		-1.83	
396	EN12593	Manual	-12		1.44	
398	EN12593	Manual	-13		0.97	
399		-----	-----		-----	
444		-----	-----		-----	
447		-----	-----		-----	
604		-----	-----		-----	
657		-----	-----		-----	
865		-----	-----		-----	
962		-----	-----		-----	
963		-----	-----		-----	
1011		-----	-----		-----	
1026	EN12593	Automated	-18		-1.36	
1040		-----	-----		-----	
1108		-----	-----		-----	
1135	EN12593	Automated	-16		-0.43	
1191	EN12593	Automated	-20		-2.30	
1301	EN12593	-----	-11		1.90	
1378		-----	-----		-----	
1402	EN12593	Manual	-10		2.37	
1613		-----	-----		-----	
1724		-----	-----		-----	
1730		-----	-----		-----	
1741	EN12593	Automated	-16.0		-0.43	
1833		-----	-----		-----	
1849		-----	-----		-----	
1852	EN12593	Automated	-16.0		-0.43	
1944	EN12593	Manual	-16		-0.43	
6048		-----	-----		-----	
6054		-----	-----		-----	
6076	EN12593	Automated	-15		0.04	
6093		-----	-----		-----	
6094		-----	-----		-----	
6095		-----	-----		-----	
6096		-----	-----		-----	
6097		-----	-----		-----	
6098		-----	-----		-----	
6148		-----	-----		-----	
6149		-----	-----		-----	
6150		-----	-----		-----	
6151		-----	-----		-----	
6152		-----	-----		-----	
6182		-----	-----		-----	

normality	OK
n	13
outliers	0
mean (n)	-15.08
st.dev. (n)	3.013
R(calc.)	8.44
st.dev.(EN12593:15)	2.143
R(EN12593:15)	6.00

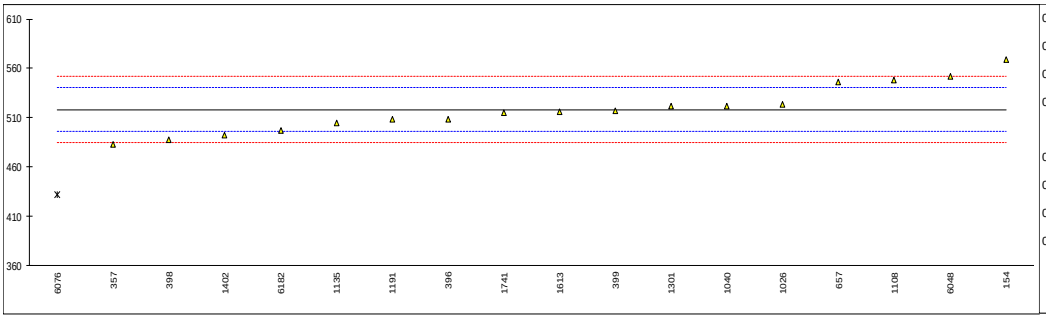


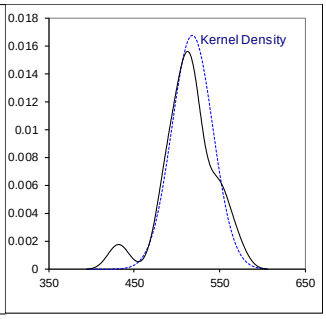
Determination of Kinematic Viscosity at 135°C on sample #17260; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
154	D2170	569		4.56	
168		-----		-----	
225		-----		-----	
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357	EN12595	483.4		-3.15	
396	EN12595	508.2		-0.91	
398	EN12595	487.4		-2.79	
399	EN12595	517.1		-0.11	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2170	546		2.49	
865		-----		-----	
962		-----		-----	
963		-----		-----	
1011		-----		-----	
1026	EN12595	523.6		0.47	
1040	D2170	522.1		0.34	
1108	EN12595	547.8		2.65	
1135	EN12595	505		-1.20	
1191	EN12595	508		-0.93	
1301	EN12595	522.0		0.33	
1378		-----		-----	
1402	EN12595	492.1218		-2.36	
1613	D2170	515.90		-0.22	
1724		-----		-----	
1730		-----		-----	
1741	EN12595	514.8		-0.32	
1833		-----		-----	
1849		-----		-----	
1852		-----		-----	
1944		-----		-----	
6048	EN12595	551.9		3.02	
6054		-----		-----	
6076	EN13302	432.4	G(0.01)	-7.74	
6093		-----		-----	
6094		-----		-----	
6095		-----		-----	
6096		-----		-----	
6097		-----		-----	
6098		-----		-----	
6148		-----		-----	
6149		-----		-----	
6150		-----		-----	
6151		-----		-----	
6152		-----		-----	
6182	EN12595	497.6		-1.87	

normality	OK
n	17
outliers	1
mean (n)	518.35
st.dev. (n)	23.828
R(calc.)	66.72
st.dev.(EN12595:14)	11.107
R(EN12595:14)	31.10

Compare R(D2170/D2170M:10) = 45.61



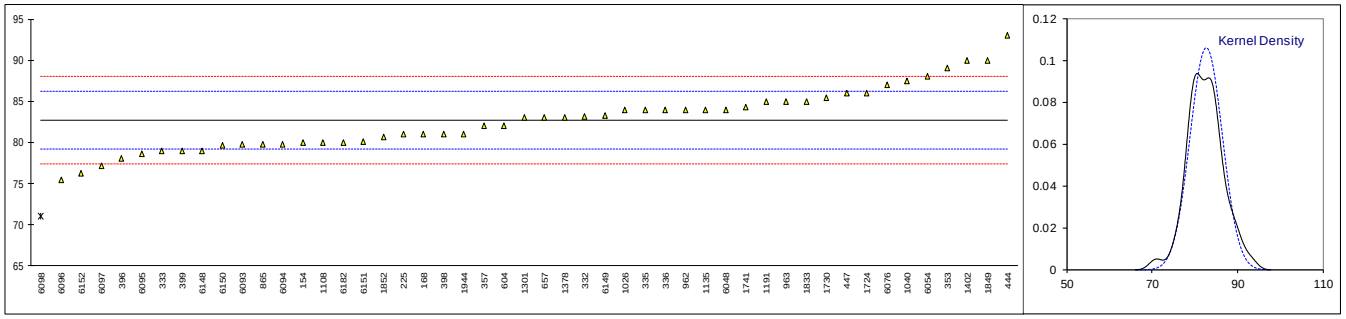


Determination of Penetration at 25°C on sample #17260; results in 0.1 mm

lab	method	value	mark	z(targ)	remarks
154	D5	80		-1.54	
168	D5	81		-0.97	
225	D5	81		-0.97	
332	EN1426	83.1		0.21	
333	EN1426	79		-2.10	
335	EN1426	84.0		0.72	
336	EN1426	84		0.72	
353	EN1426	89		3.54	
357	EN1426	82		-0.41	
396	EN1426	78		-2.66	
398	EN1426	81		-0.97	
399	EN1426	79		-2.10	
444	EN1426	93.0		5.80	
447	EN1426	86		1.85	
604	D5	82		-0.41	
657	D5	83		0.16	
865	D5	79.8		-1.65	
962	D5	84		0.72	
963	D5	85		1.28	
1011		-----		-----	
1026	EN1426	84		0.72	
1040	EN1426	87.5		2.69	
1108	EN1426	80.0		-1.54	
1135	EN1426	84		0.72	
1191	EN1426	85		1.28	
1301	EN1426	83		0.16	
1378	D5	83		0.16	
1402	EN1426	90		4.11	
1613		-----		-----	
1724	D5	86		1.85	
1730	EN1426	85.4		1.51	
1741	EN1426	84.3		0.89	
1833	EN1426	85		1.28	
1849	EN1426	90		4.11	
1852	EN1426	80.7		-1.14	
1944	EN1426	81		-0.97	
6048	EN1426	84		0.72	
6054	D5	88		2.98	
6076	EN1426	87		2.41	
6093	EN1426	79.7		-1.71	
6094	EN1426	79.8		-1.65	
6095	EN1426	78.6		-2.33	
6096	EN1426	75.4		-4.13	
6097	EN1426	77.2		-3.12	
6098	EN1426	71.0	G(0.05)	-6.61	
6148	EN1426	79.0		-2.10	
6149	EN1426	83.3		0.33	
6150	EN1426	79.6		-1.76	
6151	EN1426	80.1		-1.48	
6152	EN1426	76.2		-3.68	
6182	EN1426	80		-1.54	

			Only EN1426	Only D5
	normality	OK	OK	OK
	n	48	37	11
	outliers	1	1	0
	mean (n)	82.723	82.646	82.982
	st.dev. (n)	3.7664	4.0776	2.5930
	R(calc.)	10.546	11.417	7.260
	st.dev.(EN1426:15)	1.7726	1.7710	3.6882
	R(EN1426:15)	4.963	4.959	10.327

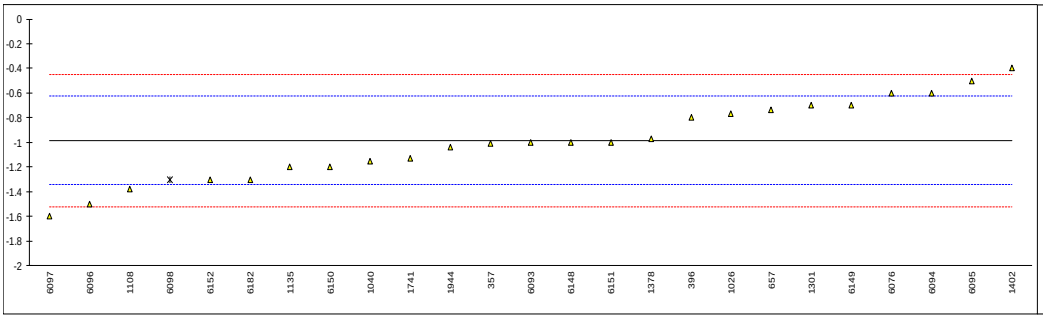
Compare R(D5/D5M) = 10.290

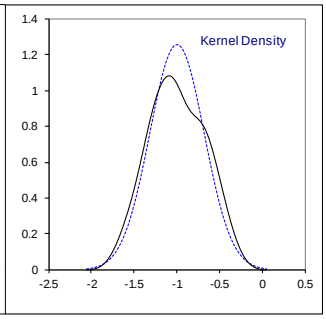


Determination of Penetration Index on sample #17260;

lab	method	value	mark	z(targ)	lis calc.	remarks
154		-----		-----	-----	
168		-----		-----	-----	
225		-----		-----	-1.909	
332		-----		-----	-1.152	
333		-----		-----	-1.229	
335		-----		-----	-0.911	
336		-----		-----	-0.763	
353		-----		-----	-0.715	
357	EN12591	-1.01		-0.15	-1.008	
396	EN12591	-0.8	E	1.03	-0.853	Error in calculation
398		-----		-----	-0.924	
399		-----		-----	-0.876	
444		-----		-----	-0.375	
447		-----		-----	-0.637	
604		-----		-----	-0.744	
657	EN12591	-0.74		1.36	-0.738	
865		-----		-----	-0.965	
962		-----		-----	-----	
963		-----		-----	-0.467	
1011		-----		-----	-----	
1026	EN12591	-0.77		1.19	-0.763	
1040	EN12591	-1.15		-0.93	-1.162	
1108	EN12591	-1.38		-2.22	-1.377	
1135	EN12591	-1.2		-1.21	-1.245	
1191		-----		-----	-0.788	
1301	EN12591	-0.7013		1.58	-0.709	
1378	EN12591	-0.97		0.07	-0.975	
1402	EN12591	-0.4		3.27	-0.386	
1613		-----		-----	-----	
1724		-----		-----	-0.637	
1730		-----		-----	-0.540	
1741	EN12591	-1.13		-0.82	-1.158	
1833		-----		-----	-0.848	
1849		-----		-----	-1.019	
1852		-----		-----	-1.232	
1944	EN12591	-1.042		-0.33	-1.042	
6048		-----		-----	-1.183	
6054		-----		-----	-0.808	
6076	EN12591	-0.6		2.15	-0.603	
6093	EN12591	-1.0		-0.09	-0.968	
6094	EN12591	-0.6		2.15	-0.562	
6095	EN12591	-0.5	E	2.71	-0.437	Error in calculation
6096	EN12591	-1.5	E	-2.89	-1.442	Error in calculation
6097	EN12591	-1.6		-3.45	-1.593	
6098	EN12591	-1.3	ex	-1.77	-1.303	Excluded, outlier in Penetration Point
6148	EN12591	-1.0		-0.09	-0.963	
6149	EN12591	-0.7		1.59	-0.728	
6150	EN12591	-1.2	E	-1.21	-1.149	Error in calculation
6151	EN12591	-1.0		-0.09	-0.984	
6152	EN12591	-1.3	E	-1.77	-1.236	Error in calculation
6182	EN12591	-1.3		-1.77	-1.256	

normality	OK	OK
n	24	45
outliers	0 (+ 1 excl)	0 (+ 1 excl)
mean (n)	-0.983	-0.935
st.dev. (n)	0.3176	0.3227
R(calc.)	0.889	0.904
st.dev.(EN12591:09)	0.1786	0.1786
R(EN12591:09)	0.500	0.500



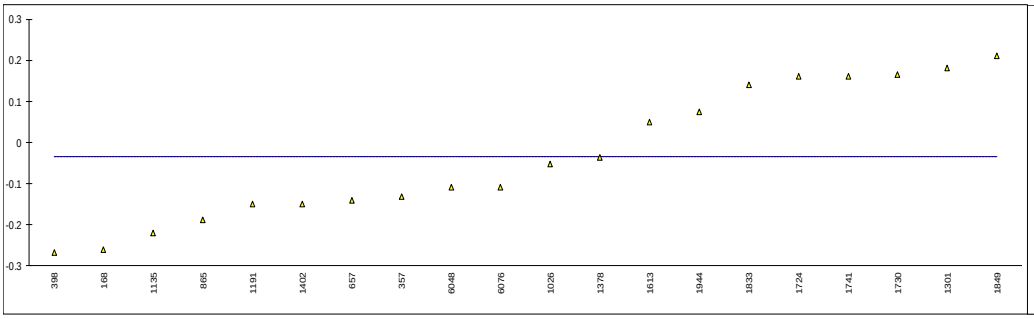


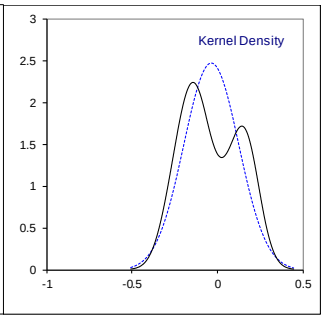
Determination of RTFOT at 163°C, Change of Mass on sample #17260; results in %

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168	D2872	-0.262		-----	
225		-----		-----	
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357	EN12607-1	-0.132		-----	
396		-----		-----	
398		-0.268		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657		-0.14		-----	
865	D2872	-0.1874		-----	
962		-----		-----	
963		-----		-----	
1011		-----		-----	
1026	EN12607-1	-0.053		-----	
1040		-----		-----	
1108		-----		-----	
1135	EN12607-1	-0.22		-----	
1191	EN12607-1	-0.15		-----	
1301		0.18		-----	
1378	EN12607-1	-0.037		-----	
1402	EN12607-1	-0.15		-----	
1613	D2872	0.05		-----	
1724		0.16		-----	
1730	EN12607-1	0.165		-----	
1741	EN12607-1	0.161		-----	
1833	EN12607-1	0.14		-----	
1849	EN12607-1	0.21		-----	
1852		-----		-----	
1944	EN12607-1	0.074		-----	
6048	EN12607-1	-0.11		-----	
6054		-----		-----	
6076	EN12607-1	-0.11		-----	
6093		-----		-----	
6094		-----		-----	
6095		-----		-----	
6096		-----		-----	
6097		-----		-----	
6098		-----		-----	
6148		-----		-----	
6149		-----		-----	
6150		-----		-----	
6151		-----		-----	
6152		-----		-----	
6182		-----		-----	

normality	OK
n	20
outliers	0
mean (n)	-0.0340
st.dev. (n)	0.16127
R(calc.)	0.4515
st.dev.(EN12607-1:14)	(0.07143)
R(EN12607-1:14)	(0.2000)

Application range >0.3% and <0.8%absolute

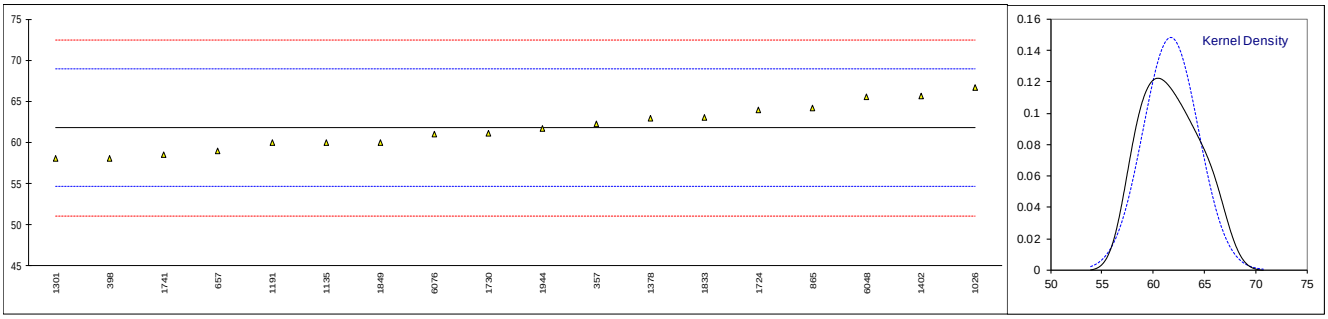




Determination of RTFOT at 163°C, Retained Penetration on sample #17260; results in %

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225		-----		-----	
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357	EN12607-1	62.2		0.13	
396		-----		-----	
398		58.02		-1.04	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657		59		-0.77	
865	D2872	64.2		0.69	
962		-----		-----	
963		-----		-----	
1011		-----		-----	
1026	EN12607-1	66.7		1.39	
1040		-----		-----	
1108		-----		-----	
1135	EN12607-1	60		-0.49	
1191	EN12607-1	60		-0.49	
1301		58		-1.05	
1378	EN12607-1	62.9		0.32	
1402	EN12607-1	65.6		1.08	
1613		-----		-----	
1724		63.95		0.62	
1730	EN12607-1	61.1		-0.18	
1741	EN12607-1	58.5		-0.91	
1833	EN12607-1	63		0.35	
1849	EN12607-1	60.0		-0.49	
1852		-----		-----	
1944	EN12607-1	61.7		-0.01	
6048	EN12607-1	65.5		1.05	
6054		-----		-----	
6076	EN12607-1	61		-0.21	
6093		-----		-----	
6094		-----		-----	
6095		-----		-----	
6096		-----		-----	
6097		-----		-----	
6098		-----		-----	
6148		-----		-----	
6149		-----		-----	
6150		-----		-----	
6151		-----		-----	
6152		-----		-----	
6182		-----		-----	

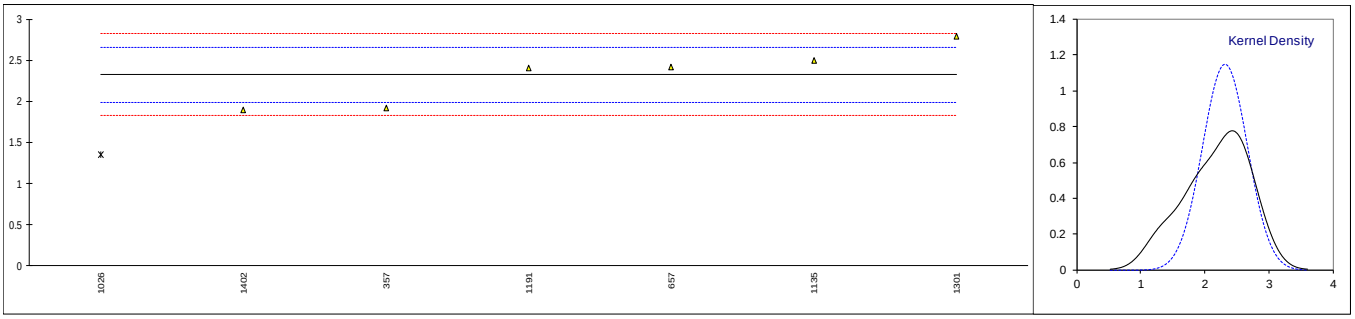
normality	OK
n	18
outliers	0
mean (n)	61.74
st.dev. (n)	2.696
R(calc.)	7.55
st.dev.(EN12607-1:14)	3.571
R(EN12607-1:14)	10.00



Determination of RTFOT at 163°C, Viscosity Ratio on sample #17260;

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225		-----		-----	
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357	EN12607-1	1.92		-2.42	
396		-----		-----	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657		2.42		0.59	
865		-----		-----	
962		-----		-----	
963		-----		-----	
1011		-----		-----	
1026	EN12607-1	1.35	G(0.05)	-5.86	
1040		-----		-----	
1108		-----		-----	
1135	EN12607-1	2.5		1.08	
1191	EN12607-1	2.4		0.47	
1301		2.79		2.82	
1378		-----		-----	
1402	EN12607-1	1.90		-2.54	
1613		-----		-----	
1724		-----		-----	
1730		-----		-----	
1741		-----		-----	
1833		-----		-----	
1849		-----		-----	
1852		-----		-----	
1944		-----		-----	
6048		-----		-----	
6054		-----		-----	
6076		-----		-----	
6093		-----		-----	
6094		-----		-----	
6095		-----		-----	
6096		-----		-----	
6097		-----		-----	
6098		-----		-----	
6148		-----		-----	
6149		-----		-----	
6150		-----		-----	
6151		-----		-----	
6152		-----		-----	
6182		-----		-----	

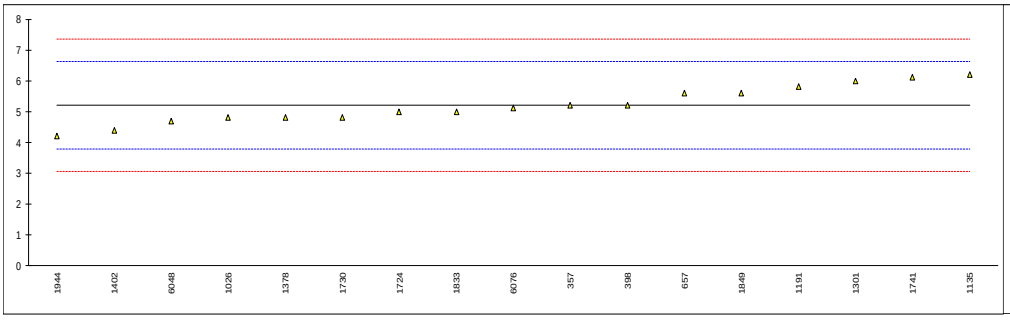
normality	unknown
n	6
outliers	1
mean (n)	2.322
st.dev. (n)	0.3482
R(calc.)	0.975
st.dev.(EN12607-1:14)	0.1658
R(EN12607-1:14)	0.464

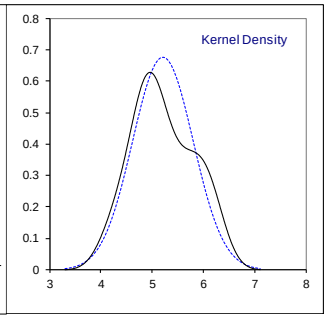


Determination of RTFOT at 163°C, Increase in Softening Point on sample #17260; results in °C

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225		-----		-----	
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357	EN12607-1	5.2		-0.01	
396		-----		-----	
398		5.2		-0.01	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657		5.6		0.55	
865		-----		-----	
962		-----		-----	
963		-----		-----	
1011		-----		-----	
1026	EN12607-1	4.8		-0.57	
1040		-----		-----	
1108		-----		-----	
1135	EN12607-1	6.2		1.39	
1191	EN12607-1	5.8		0.83	
1301		6.0		1.11	
1378	EN12607-1	4.8		-0.57	
1402	EN12607-1	4.4		-1.13	
1613		-----		-----	
1724		5		-0.29	
1730	EN12607-1	4.8		-0.57	
1741	EN12607-1	6.10		1.25	
1833	EN12607-1	5		-0.29	
1849	EN12607-1	5.6		0.55	
1852		-----		-----	
1944	EN12607-1	4.2		-1.41	
6048	EN12607-1	4.7		-0.71	
6054		-----		-----	
6076	EN12607-1	5.1		-0.15	
6093		-----		-----	
6094		-----		-----	
6095		-----		-----	
6096		-----		-----	
6097		-----		-----	
6098		-----		-----	
6148		-----		-----	
6149		-----		-----	
6150		-----		-----	
6151		-----		-----	
6152		-----		-----	
6182		-----		-----	

normality	OK
n	17
outliers	0
mean (n)	5.21
st.dev. (n)	0.591
R(calc.)	1.65
st.dev.(EN12607-1:14)	0.714
R(EN12607-1:14)	2.00



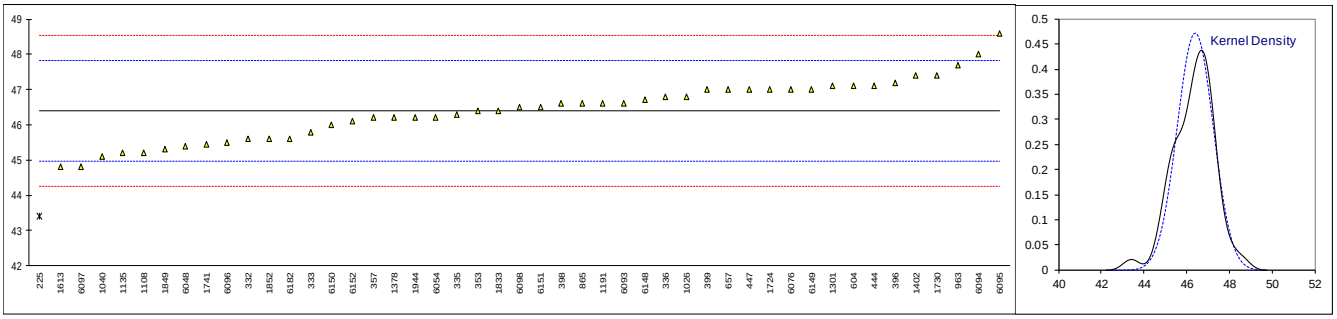


Determination of Softening Point (Ring & Ball) on sample #17260; results in °C

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225	D36	43.4	R(0.05)	-4.21	
332	EN1427	45.6		-1.13	
333	EN1427	45.8		-0.85	
335	EN1427	46.3		-0.15	
336	EN1427	46.8		0.55	
353	EN1427	46.4		-0.01	
357	EN1427	46.2		-0.29	
396	EN1427	47.2		1.11	
398	EN1427	46.6		0.27	
399	EN1427	47.0		0.83	
444	EN1427	47.1		0.97	
447	EN1427	47		0.83	
604	D36	47.1		0.97	
657	D36	47.0		0.83	
865	D36	46.6		0.27	
962		-----		-----	
963	D36	47.7		1.81	
1011		-----		-----	
1026	EN1427	46.8		0.55	
1040	EN1427	45.1		-1.83	
1108	EN1427	45.2		-1.69	
1135	EN1427	45.2		-1.69	
1191	EN1427	46.6		0.27	
1301	EN1427	47.1		0.97	
1378	D36	46.2		-0.29	
1402	EN1427	47.4		1.39	
1613	D36	44.8		-2.25	
1724	D36	47		0.83	
1730	EN1427	47.4		1.39	
1741	EN1427	45.45		-1.34	
1833	EN1427	46.4		-0.01	
1849	EN1427	45.3		-1.55	
1852	EN1427	45.6		-1.13	
1944	EN1427	46.2		-0.29	
6048	EN1427	45.4		-1.41	
6054	D36	46.2		-0.29	
6076	EN1427	47.0		0.83	
6093	EN1427	46.6		0.27	
6094	EN1427	48.0		2.23	
6095	EN1427	48.6		3.07	
6096	EN1427	45.5		-1.27	
6097	EN1427	44.8		-2.25	
6098	EN1427	46.5		0.13	
6148	EN1427	46.7		0.41	
6149	EN1427	47.0		0.83	
6150	EN1427	46.0		-0.57	
6151	EN1427	46.5		0.13	
6152	EN1427	46.1		-0.43	
6182	EN1427	45.6		-1.13	

normality	OK
n	46
outliers	1
mean (n)	46.405
st.dev. (n)	0.8467
R(calc.)	2.371
st.dev.(EN1427:15)	0.7143
R(EN1427:15)	2.000

Compare R(D36/36M:13) = 9.630

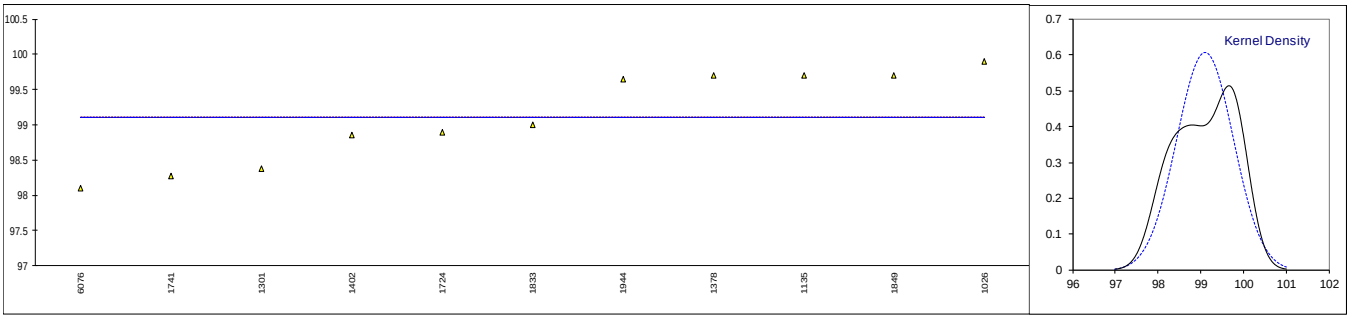


Determination of Solubility in Xylene on sample #17260; results in %M/M

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225		-----		-----	
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357		-----		-----	
396		-----		-----	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657		-----		-----	
865		-----		-----	
962		-----		-----	
963		-----		-----	
1011		-----		-----	
1026	EN12592	99.9		-----	
1040		-----		-----	
1108		-----		-----	
1135	EN12592	99.70		-----	
1191		-----		-----	
1301	EN12592	98.38		-----	
1378	EN12592	99.7		-----	
1402	EN12592	98.85		-----	
1613		-----		-----	
1724	EN12592	98.90		-----	
1730		-----		-----	
1741	EN12592	98.276		-----	
1833	EN12592	99.0		-----	
1849	EN12592	99.7		-----	
1852		-----		-----	
1944	EN12592	99.643		-----	
6048		-----		-----	
6054		-----		-----	
6076	EN12592	98.1		-----	
6093		-----		-----	
6094		-----		-----	
6095		-----		-----	
6096		-----		-----	
6097		-----		-----	
6098		-----		-----	
6148		-----		-----	
6149		-----		-----	
6150		-----		-----	
6151		-----		-----	
6152		-----		-----	
6182		-----		-----	

normality	OK
n	11
outliers	0
mean (n)	99.104
st.dev. (n)	0.6576
R(calc.)	1.841
st.dev.(EN12592:14)	(0.0536)
R(EN12592:14)	(0.150)

Compare R(iis16F02) = 0.714



APPENDIX 2

Number of participants per country

1 lab in	BELGIUM
1 lab in	CHINA, People’s Republic of
2 labs in	COTE D'IVOIRE
2 labs in	FINLAND
4 labs in	FRANCE
3 labs in	GERMANY
3 labs in	GREECE
1 lab in	HONG KONG
1 lab in	IRELAND
3 labs in	ITALY
1 lab in	JORDAN
1 lab in	MALAYSIA
13 labs in	NETHERLANDS
1 lab in	PORTUGAL
1 lab in	ROMANIA
2 labs in	SAUDI ARABIA
1 lab in	SERBIA
1 lab in	SINGAPORE
3 labs in	TURKEY
1 lab in	UNITED ARAB EMIRATES
3 labs in	UNITED KINGDOM
2 labs in	UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= probably an error in calculations
U	= test result probably reported in a different unit
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
SDS	= safety data sheet

Literature:

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11 DIN 38402 T41/42

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14 Analytical Methods Committee Technical brief, No 4.January 2001

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