

**Results of Proficiency Test
Bitumen
December 2016**

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 of Interlaboratory Studies (iis) organizes a proficiency scheme for Bitumen. During the annual proficiency testing program 2016/2017, it was decided to continue the round robin for the analysis of Bitumen.

In this interlaboratory study 52 laboratories in 25 different countries registered for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2016 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 third party laboratory. It was decided to send one container of 1 litre Bitumen, a 50/70 grade (labelled #16275). 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 April 2014 (iis-protocol, version 3.3). This protocol can be downloaded from 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, 50/70 grade was supplied by a third party. After heating, 56 metal cans of 1 liter were filled and labelled #16275. The homogeneity of the subsamples #16275 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 #16275-1	58	47.4
Sample #16275-2	59	47.2
Sample #16275-3	59	47.4
Sample #16275-4	58	47.2

Table 1: homogeneity test results of subsamples #16275

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)	1.6	0.3
Reference test method	EN1426:15	EN1427:15
r (Reference test method)	2.3	1.0

Table 2: repeatabilities of subsamples #16275

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 #16275 was assumed.

To each of the participating laboratories a 1 litre metal can, labelled #16275 was sent on November 23, 2016. 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 #16275: 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.

To get comparable test results, a detailed report form and a letter of instructions were 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 represented 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. Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the 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).

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 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. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. EN or ISO 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 of a former iis proficiency test 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, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated according to:

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

The $Z_{(\text{target})}$ 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. The usual interpretation of z-scores is as follows:

$ z < 1$	good
$1 < z < 2$	satisfactory
$2 < z < 3$	questionable
$3 < z $	unsatisfactory

4 EVALUATION

In this proficiency test no problems were encountered with the dispatch of the samples. One participant did not report any test results 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, 51 participants reported in total 318 numerical results. Observed were 8 outlying test results, which is 2.5%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

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

4.1 EVALUATION PER TEST

In this section, the 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 problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of EN15326:07.

Dynamic Viscosity at 60°C: 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 EN12596:14.

Flash Point COC: 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 ISO2592:00 and of ASTM D92:12b.

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

Kinematic Viscosity at 135°C: This determination was problematic. Only one statistical outlier was observed. However, the calculated reproducibility is not in agreement with the requirements of EN12595:14, but almost in agreement with the less strict requirements of ASTM D2170/2170M:10.

Penetration: This determination is very problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the strict requirements of EN1426:15, nor 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=10) are evaluated separately, the reproducibilities are still both not in agreement with the respective method requirements.

Different factors could cause this large variation, such as preparation, temperature and needle. During the measurement the temperature should be kept at 25°C, by immersing the sample in sufficient water of this temperature (for measurements outside of the waterbath, a transfer dish of 350 ml should be used). Deviations from this temperature will have influence on the penetration. Another factor is the tip of the needle used. This tip should keep the same dimensions/surface through out testing in time. In practise it will get abrasion and wear and should be replaced regularly.

Penetration Index: This determination was problematic for the laboratories that reported this parameter. One test result 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 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 EN12607-1:14.

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

The determination on Viscosity Ratio was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of EN12607-1:14. 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. No statistical outliers were 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. It is remarkable to see that the reported test results may be bimodally divided.

Solubility in Xylene: This determination may be very problematic. No statistical outliers were observed. The calculated reproducibility is much larger than the calculated reproducibility of the previous PT: iis15F02 (0.71 vs 0.10). As it is unclear what the cause of the large variation may be, no z-scores were calculated. The type of solvent (Toluene or Xylene) does not appear to be an important factor of influence. However, the oven temperature used to dry the sample, may be.

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	31	1015.0	5.8	5.0
Dynamic Viscosity at 60°C	Pa.s	14	196.4	16.3	19.6
Flash Point COC	°C	16	353	38	17
Fraass Breaking Point	°C	15	-10	5	6
Kinematic Viscosity at 135°C	mm ² /s	20	418.4	40.9	25.1
Penetration at 25°C	0.1 mm	48	57.8	8.2	3.5
Penetration Index		23	-1.39	0.69	0.50
RTFOT - Change of Mass	%	25	0.013	0.165	0.200
RTFOT - Retained Penetration	%	21	64.0	7.8	10.0
RTFOT - Viscosity Ratio		6	1.98	0.48	0.40
RTFOT - Increase in Soft. Point	°C	24	4.3	1.8	2.0
Softening Point (Ring and Ball)	°C	48	47.9	2.3	2.0
Solubility in Xylene	% M/M	17	99.70	0.71	(0.15)

Table 3: summary of test results on Bitumen 50/70 grade

Results between brackets are for information only

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 test methods. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2016 WITH PREVIOUS PTS

	December 2016	December 2015	December 2014
Number of reporting labs	51	35	36
Number of results reported	318	388	464
Number of statistical outliers	8	30	18
Percentage statistical outliers	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.

	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.	+	+	+/-	+

Table 5: comparison determinations against the test method

The following performance categories were used:

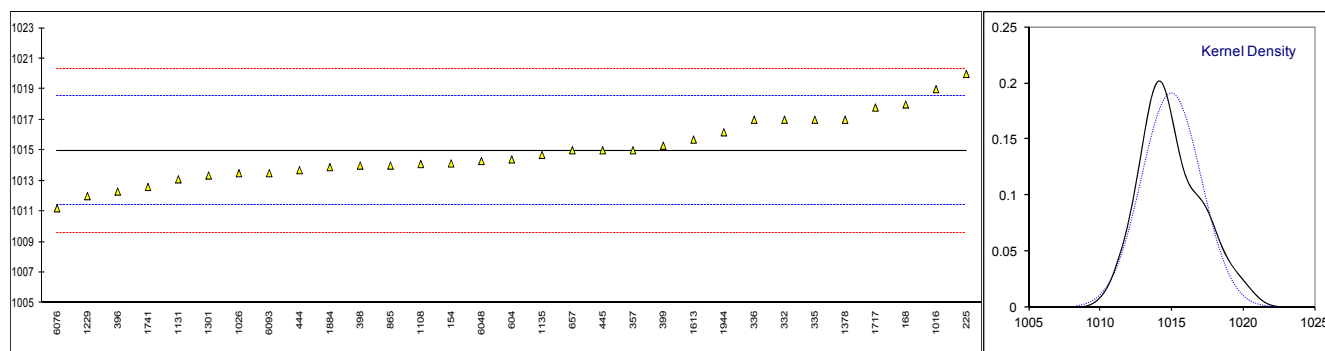
- ++: group performed much better than the reference test method
- + : group performed better than the reference test method
- +/-: group performance equals the reference test method
- : group performed worse than the reference test method
- : group performed much worse than the reference test method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

APPENDIX 1

Determination of Density at 25°C on sample #16275; results in kg/m³

lab	method	value	mark	z(targ)	remarks
154	D70	1014.14		-0.46	
168	D70	1018		1.70	
225	D70	1020		2.82	
332	EN15326	1017		1.14	
333		----		----	
335	EN15326	1017	C	1.14	First reported 1.017
336	EN15326	1017		1.14	
337		----		----	
353		----		----	
357	EN15326	1015.0		0.02	
396	ISO3838	1012.3		-1.49	
398	D70	1014		-0.54	
399	D70	1015.3		0.19	
444	D70	1013.7		-0.71	
445	D70	1015.0		0.02	
447		----		----	
604	D70	1014.4		-0.31	
657	D70	1015		0.02	
865	D70	1014.0		-0.54	
1011		----		----	
1016	EN15326	1019	C	2.26	First reported 1032.439
1026	EN15326	1013.5		-0.82	
1040		----		----	
1108	EN15326	1014.1	C	-0.48	First reported 1005.6
1131	EN15326	1013.1		-1.04	
1135	EN15326	1014.7	C	-0.15	First reported 1.0147 kg/m3
1229	EN15326	1012		-1.66	
1301	EN15326	1013.3508		-0.90	
1378	D70	1017	C	1.14	First reported 1.017 kg/m3
1394		----		----	
1402		----		----	
1613	DIN51757	1015.7		0.41	
1631		----		----	
1710		----		----	
1717	ISO3838	1017.8		1.59	
1724		----		----	
1730		----		----	
1741	EN15326	1012.6		-1.32	
1833		----		----	
1849		----		----	
1852		----		----	
1884	D70	1013.9		-0.59	
1944	EN15326	1016.18		0.68	
6037		----		----	
6048	EN15326	1014.3		-0.37	
6076	EN15326	1011.2	C	-2.11	First reported 1008.2
6093	EN15326	1013.5		-0.82	
6094		----		----	
6095		----		----	
6096		----		----	
6097		----		----	
6098		----		----	

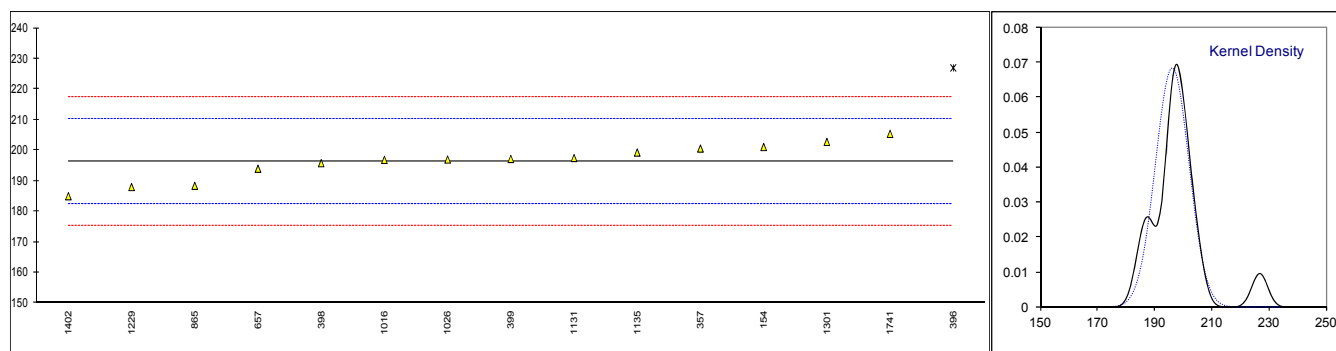
normality OK
n 31
outliers 0
mean (n) 1014.96
st.dev. (n) 2.085
R(calc.) 5.84
R(EN15326:07) 5.00



Determination of Dynamic Viscosity at 60°C on sample #16275; results in Pa.s

lab	method	value	mark	z(targ)	remarks
154	D2171	201.1		0.68	
168		----		----	
225		----		----	
332		----		----	
333		----		----	
335		----		----	
336		----		----	
337		----		----	
353		----		----	
357	EN12596	200.6		0.61	
396	EN12596	227	G(0.01)	4.37	
398	EN12596	195.8		-0.08	
399	EN12596	197.2		0.12	
444		----		----	
445		----		----	
447		----		----	
604		----		----	
657	D2171	194		-0.34	
865	D2171	188.4		-1.13	
1011		----		----	
1016	EN12596	196.9		0.08	
1026	EN12596	197		0.09	
1040		----		----	
1108		----		----	
1131	EN12596	197.5		0.16	
1135	EN12596	199.3		0.42	
1229	EN12596	188		-1.19	
1301	EN12596	202.76272		0.91	
1378		----		----	
1394		----		----	
1402	EN12596	185		-1.62	
1613		----		----	
1631		----		----	
1710		----		----	
1717		----		----	
1724		----		----	
1730		----		----	
1741	EN12596	205.4		1.29	
1833		----		----	
1849		----		----	
1852		----		----	
1884		----		----	
1944		----		----	
6037		----		----	
6048		----		----	
6076		----		----	
6093		----		----	
6094		----		----	
6095		----		----	
6096		----		----	
6097		----		----	
6098		----		----	

normality OK
n 14
outliers 1
mean (n) 196.35
st.dev. (n) 5.836
R(calc.) 16.34
R(EN12596:14) 19.64



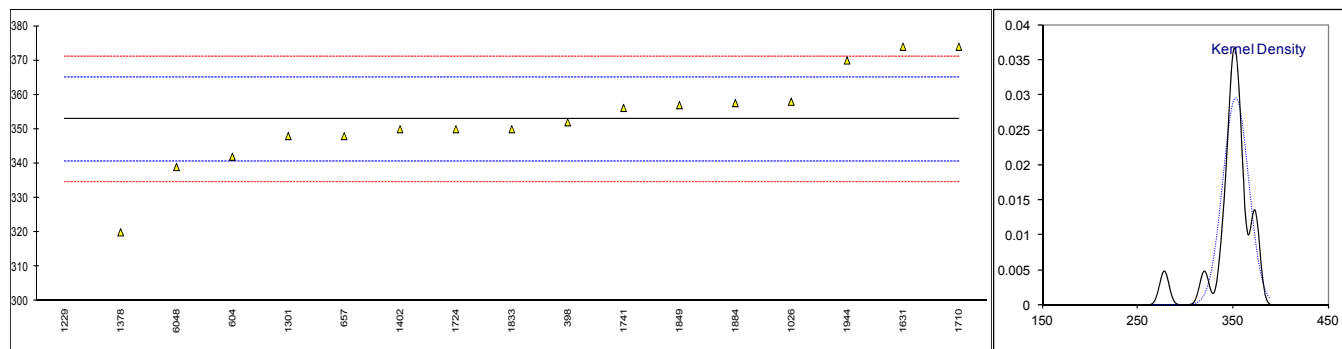
Determination of Flash Point COC on sample #16275; results in °C

lab	method	mode	value	mark	z(targ)	remarks
154		----	----		----	
168		----	----		----	
225		----	----		----	
332		----	----		----	
333		----	----		----	
335		----	----		----	
336		----	----		----	
337		----	----		----	
353		----	----		----	
357		----	----		----	
396	ISO2592	Manual	>320		----	
398	ISO2592	Manual	352		-0.14	
399		----	----		----	
444		----	----		----	
445	D92	Manual	----		----	Reported excess foam
447		----	----		----	
604	D92	Manual	342		-1.79	
657	D92	Manual	348		-0.80	
865	D92	Manual	>300		----	
1011		----	----		----	
1016		----	----		----	
1026	ISO2592	Manual	358		0.85	
1040		----	----		----	
1108		----	----		----	
1131		----	----		----	
1135		----	----		----	
1229	ISO2592	Automated	278	G(0.01)	-12.33	
1301	ISO2592	Manual	348		-0.80	
1378	D92	----	320		-5.41	
1394		----	----		----	
1402	ISO2592	Automated	350		-0.47	
1613		----	----		----	
1631	ISO2592	Automated	374		3.48	
1710	ISO2592	Automated	374		3.48	
1717	ISO2592	Automated	>350		----	
1724	D92	----	350		-0.47	
1730		----	----		----	
1741	ISO2592	Automated	356.2		0.55	
1833	ISO2592	----	350		-0.47	
1849	ISO2592	Automated	357.0		0.68	
1852		----	----		----	
1884	ISO2592	----	357.6		0.78	
1944	ISO2592	Manual	370		2.82	
6037		----	----		----	
6048	ISO2592	Automated	339		-2.28	
6076		----	----		----	
6093		----	----		----	
6094		----	----		----	
6095		----	----		----	
6096		----	----		----	
6097		----	----		----	
6098		----	----		----	

normality
n
outliers
mean (n)
st.dev. (n)
R(calc.)
R(ISO2592:00)

suspect
16
1
352.86
13.484
37.75
17.00

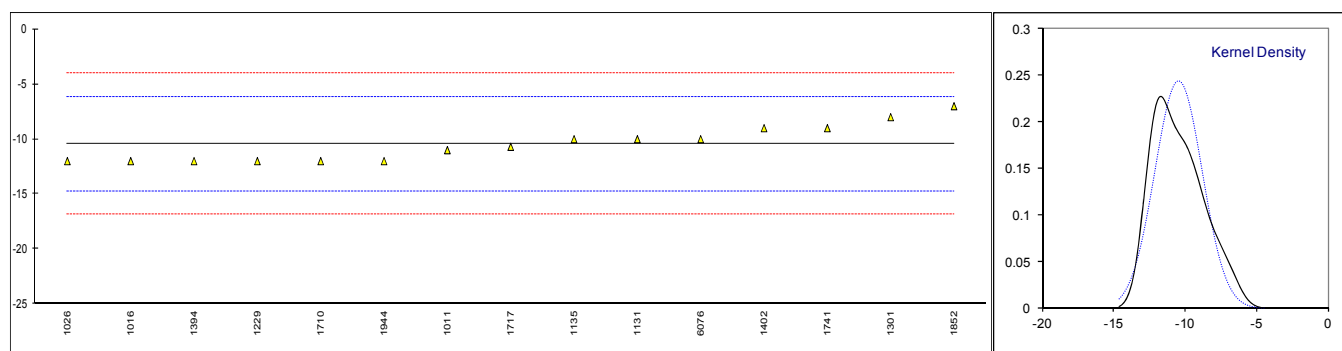
Compare R(D92:2012b) = 18.00



Determination of Fraass Breaking point on sample #16275; results in °C

lab	method	mode	value	mark	z(targ)	remarks
154		----	----		----	
168		----	----		----	
225		----	----		----	
332		----	----		----	
333		----	----		----	
335		----	----		----	
336		----	----		----	
337		----	----		----	
353		----	----		----	
357		----	----		----	
396		----	----		----	
398		----	----		----	
399		----	----		----	
444		----	----		----	
445		----	----		----	
447		----	----		----	
604		----	----		----	
657		----	----		----	
865		----	----		----	
1011	EN12593	Automated	-11		-0.26	
1016	EN12593	Automated	-12		-0.72	
1026	EN12593	Automated	-12		-0.72	
1040		----	----		----	
1108		----	----		----	
1131	EN12593	----	-10		0.21	
1135	EN12593	Automated	-10		0.21	
1229	EN12593	Automated	-12		-0.72	
1301	EN12593	Manual	-8	C	1.14	First reported -3
1378		----	----		----	
1394		Automated	-12		-0.72	
1402	EN12593	Manual	-9		0.68	
1613		----	----		----	
1631		----	----		----	
1710	EN12593	Automated	-12		-0.72	
1717	EN12593	Automated	-10.7		-0.12	
1724		----	----		----	
1730		----	----		----	
1741	EN12593	Automated	-9.0		0.68	
1833		----	----		----	
1849		----	----		----	
1852	EN12593	Automated	-7		1.61	
1884		----	----		----	
1944	EN12593	Manual	-12		-0.72	
6037		----	----		----	
6048		----	----		----	
6076	EN12593	Automated	-10		0.21	
6093		----	----		----	
6094		----	----		----	
6095		----	----		----	
6096		----	----		----	
6097		----	----		----	
6098		----	----		----	

normality OK
n 15
outliers 0
mean (n) -10.45
st.dev. (n) 1.637
R(calc.) 4.58
R(EN12593:15) 6.00

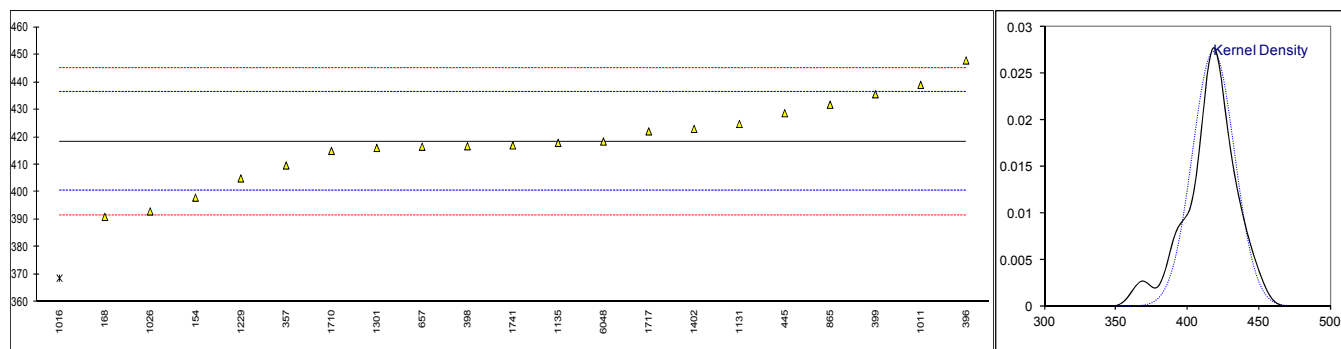


Determination of Kinematic Viscosity at 135°C on sample #16275; results in mm²/s

lab	method	value	mark	z(targ)	remarks
154	D2170	398	C	-2.27	First reported 378
168	D2170	391	C	-3.05	First reported 376
225		----		----	
332		----		----	
333		----		----	
335		----		----	
336		----		----	
337		----		----	
353		----		----	
357	EN12595	409.7		-0.97	
396	EN12595	447.9		3.30	
398	EN12595	416.7		-0.19	
399	EN12595	435.6		1.92	
444		----		----	
445	D2170	428.7		1.15	
447		----		----	
604		----		----	
657	D2170	416.5		-0.21	
865	D2170	431.8		1.50	
1011	EN12595	439		2.30	
1016	EN12595	368.7	G(0.05)	-5.54	
1026	EN12595	393		-2.83	
1040		----		----	
1108		----		----	
1131	EN12595	424.8		0.72	
1135	EN12595	417.9		-0.05	
1229	EN12595	405		-1.49	
1301	EN12595	416.084385		-0.25	
1378		----		----	
1394		----		----	
1402	EN12595	423		0.52	
1613		----		----	
1631		----		----	
1710	EN12595	415		-0.37	
1717	EN12595	422.1		0.42	
1724		----		----	
1730		----		----	
1741	EN12595	417.0		-0.15	
1833		----		----	
1849		----		----	
1852		----		----	
1884		----		----	
1944		----		----	
6037		----		----	
6048	EN12595	418.4		0.00	
6076		----		----	
6093		----		----	
6094		----		----	
6095		----		----	
6096		----		----	
6097		----		----	
6098		----		----	

normality OK
n 20
outliers 1
mean (n) 418.36
st.dev. (n) 14.591
R(calc.) 40.85
R(EN12595:14) 25.10

Compare R(D2170/D2170M:10) = 36.82



Determination of Penetration at 25°C on sample #16275; results in 0.1 mm

lab	method	value	mark	z(targ)	remarks
154	D5	63		4.21	
168	D5	62		3.40	
225	D5	55		-2.25	
332	EN1426	58.3		0.41	
333	EN1426	58		0.17	
335	EN1426	61.4		2.91	
336	EN1426	59		0.98	
337		----		----	
353	EN1426	57		-0.64	
357	EN1426	58		0.17	
396	EN1426	59		0.98	
398	EN1426	57.7		-0.07	
399	EN1426	57		-0.64	
444	IP49	67.4	R(0.05)	7.76	
445	D5	64		5.01	
447	EN1426	56		-1.45	
604	D5	57		-0.64	
657	D5	57		-0.64	
865	D5	54.4		-2.74	
1011	EN1426	60		1.78	
1016	EN1426	59		0.98	
1026	EN1426	60		1.78	
1040	EN1426	63.6		4.69	
1108	EN1426	55		-2.25	
1131	EN1426	57		-0.64	
1135	EN1426	59		0.98	
1229	EN1426	60		1.78	
1301	EN1426	57		-0.64	
1378	D5	53.5		-3.47	
1394	EN1426	59.5		1.38	
1402	EN1426	61		2.59	
1613		----		----	
1631	EN1426	52		-4.68	
1710	EN1426	58		0.17	
1717	EN1426	56.8		-0.80	
1724	D5	57		-0.64	
1730	EN1426	60.7		2.35	
1741	EN1426	59.7		1.54	
1833		52		-4.68	First reported as "Retained penetration"
1849	EN1426	61.5		2.99	
1852	EN1426	57		-0.64	
1884	D5	54.9		-2.34	
1944	EN1426	52		-4.68	
6037	EN1426	55.9	C	-1.53	First reported 45.9
6048	EN1426	59		0.98	
6076	EN1426	60		1.78	
6093	EN1426	54		-3.06	
6094	EN1426	57.0		-0.64	
6095	EN1426	58.7		0.73	
6096	EN1426	56.7		-0.88	
6097	EN1426	52.7		-4.11	
6098	EN1426	49	R(0.05)	-7.10	

normality OK
n 48
outliers 2
mean (n) 57.792
st.dev. (n) 2.9414
R(calc.) 8.236
R(EN1426:15) 3.467

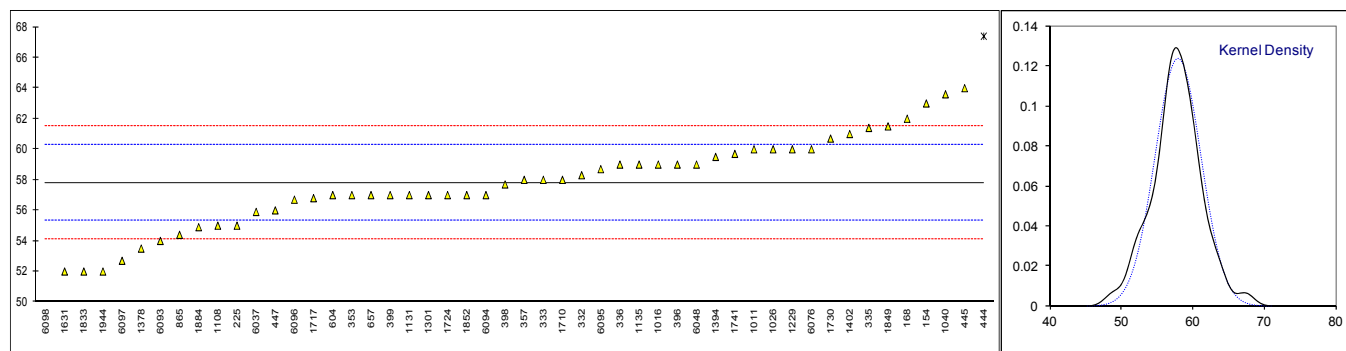
Compare R(D5/D5M) = 6.763

Only EN1426 data:

OK
37
1
57.951
2.5864
7.242
3.477

Only D5 data:

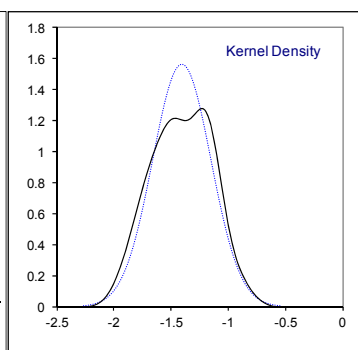
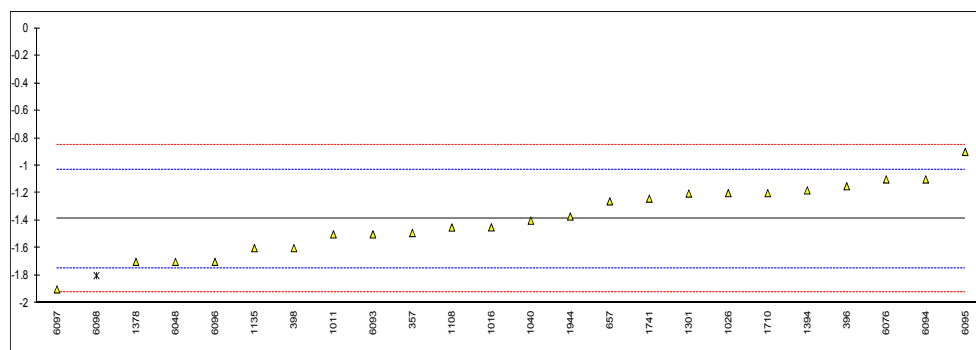
OK
10
1
57.780
3.8201
10.696
6.761



Determination of Penetration Index on sample #16275;

lab	method	value	mark	z(targ)	iis calc.	remarks
154		----		----	----	
168		----		----	----	
225		----		----	----	
332		----		----	-1.316	
333		----		----	-1.598	
335		----		----	-1.138	
336		----		----	-1.395	
337		----		----	----	
353		----		----	-1.638	
357	EN12591	-1.49		-0.58	-1.489	
396	EN12591	-1.15		1.32	-1.181	
398	EN12591	-1.6		-1.20	-1.610	
399		----		----	-1.638	
444		----		----	-0.743	
445		----		----	-0.823	
447		----		----	-1.570	
604		----		----	-1.289	
657	Calculation	-1.26		0.71	-1.263	
865		----		----	-1.529	
1011	EN12591	-1.5		-0.64	-1.519	
1016	EN12591	-1.449		-0.35	-1.449	
1026	EN12591	-1.2		1.04	-1.194	
1040	EN12591	-1.4		-0.08	-1.419	
1108	EN12591	-1.45		-0.36	-1.451	
1131		----		----	-1.693	
1135	EN12591	-1.6		-1.20	-1.558	
1229		----		----	-1.410	
1301	EN12591	-1.2026	E	1.03	-1.263	Error in calculation
1378	EN12591	-1.7		-1.76	-1.700	
1394	EN12591	-1.18		1.16	-1.188	
1402		----		----	-0.890	
1613		----		----	----	
1631		----		----	-1.843	
1710	EN12591	-1.2		1.04	-1.222	
1717		----		----	-1.605	
1724		----		----	-1.055	
1730		----		----	-1.233	
1741	EN12591	-1.24		0.82	-1.286	
1833		----		----	-2.060	
1849		----		----	-1.433	
1852		----		----	-1.803	
1884		----		----	-1.376	
1944	EN12591	-1.370		0.09	-1.370	
6037		----		----	-1.388	
6048	EN12591	-1.7		-1.76	-1.669	
6076	EN12591	-1.1		1.60	-1.141	
6093	EN12591	-1.5		-0.64	-1.519	
6094	EN12591	-1.1		1.60	-1.106	
6095	EN12591	-0.9		2.72	-0.932	
6096	EN12591	-1.7		-1.76	-1.732	
6097	EN12591	-1.9		-2.88	-1.895	
6098	EN12591	-1.8	ex	-2.32	-1.812	Excluded, outlier in Penetration point

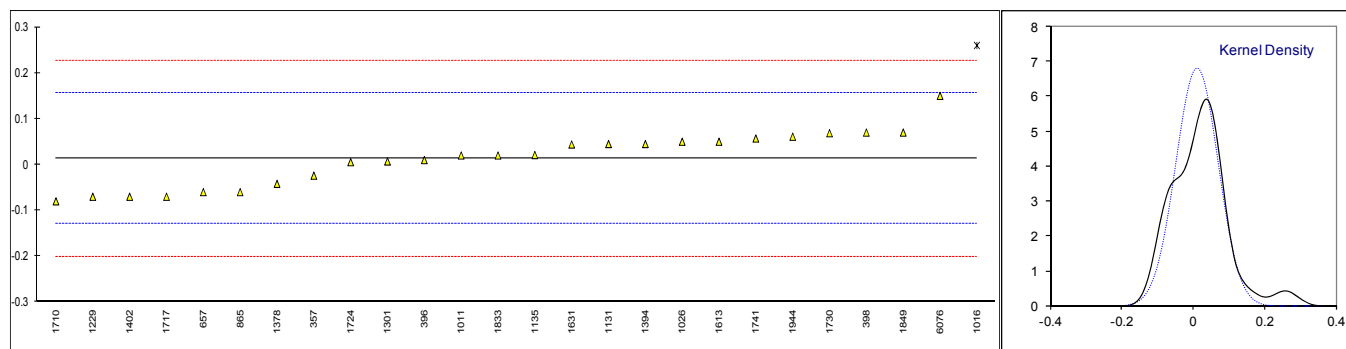
normality	OK	OK
n	23	46
outliers	0 (+1 excl)	0 (+1 excl)
mean (n)	-1.387	-1.404
st.dev. (n)	0.2463	0.2828
R(calc.)	0.690	0.792
R(EN12591:09)	0.500	0.500



Determination of RTFOT at 163°C, Change of Mass on sample #16275; results in %

lab	method	value	mark	z(targ)	remarks
154		----		----	
168		----		----	
225		----		----	
332		----		----	
333		----		----	
335		----		----	
336		----		----	
337		----		----	
353		----		----	
357	EN12607-1	-0.024		-0.51	
396	EN12607-1	0.01		-0.04	
398	EN12607-1	0.07		0.80	
399		----		----	
444		----		----	
445		----		----	
447		----		----	
604		----		----	
657	D2872	-0.06		-1.02	
865	D2872	-0.06		-1.02	
1011	EN12607-1	0.02		0.10	
1016	EN12607-1	0.26	C,R(0.05)	3.46	First reported -0.26
1026	EN12607-1	0.05		0.52	
1040		----		----	
1108		----		----	
1131		0.045		0.45	
1135	EN12607-1	0.021		0.12	
1229	EN12607-1	-0.07		-1.16	
1301	EN12607-1	0.007		-0.08	
1378	EN12607-1	-0.042		-0.77	
1394		0.045		0.45	
1402	EN12607-1	-0.07		-1.16	
1613	D2872	0.05		0.52	
1631	EN12607-1	0.044		0.44	
1710	EN12607-1	-0.08		-1.30	
1717	EN12607-1	-0.070		-1.16	
1724	D2872	0.0054		-0.10	
1730	EN12607-1	0.0686		0.78	
1741	EN12607-1	0.057		0.62	
1833	EN12607-1	0.02		0.10	
1849	EN12607-1	0.07		0.80	
1852		----		----	
1884		----		----	
1944	EN12607-1	0.061		0.68	
6037		----		----	
6048		----		----	
6076	EN12607-1	0.15		1.92	
6093		----		----	
6094		----		----	
6095		----		----	
6096		----		----	
6097		----		----	
6098		----		----	

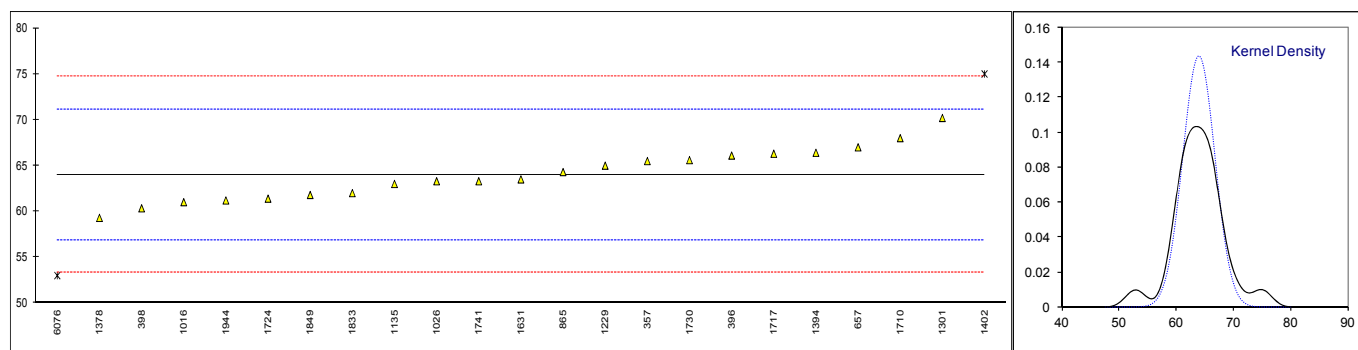
normality OK
n 25
outliers 1
mean (n) 0.0127
st.dev. (n) 0.05880
R(calc.) 0.1646
R(EN12607-1:14) 0.2000



Determination of RTFOT at 163°C, Retained Penetration on sample #16275; results in %

lab	method	value	mark	z(targ)	remarks
154		----		----	
168		----		----	
225		----		----	
332		----		----	
333		----		----	
335		----		----	
336		----		----	
337		----		----	
353		----		----	
357	EN12607-1	65.5		0.41	
396	EN12607-1	66.1		0.58	
398	EN12607-1	60.345		-1.03	
399		----		----	
444		----		----	
445		----		----	
447		----		----	
604		----		----	
657	D2872	67		0.83	
865	D2872	64.3		0.08	
1011	EN12607-1	----		----	
1016	EN12607-1	61.0169		-0.84	
1026	EN12607-1	63.3		-0.20	
1040		----		----	
1108		----		----	
1131		----		----	
1135	EN12607-1	63		-0.29	
1229	EN12607-1	65		0.27	
1301	EN12607-1	70.2		1.73	
1378	EN12607-1	59.3		-1.32	
1394		66.4		0.66	
1402	EN12607-1	75	R(0.05)	3.07	
1613	D2872	----		----	
1631	EN12607-1	63.5		-0.15	
1710	EN12607-1	68		1.11	
1717	EN12607-1	66.3		0.64	
1724	D2872	61.40		-0.74	
1730	EN12607-1	65.6		0.44	
1741	EN12607-1	63.3		-0.20	
1833	EN12607-1	62	C	-0.57	First reported 52
1849	EN12607-1	61.8		-0.62	
1852		----		----	
1884		----		----	
1944	EN12607-1	61.2		-0.79	
6037		----		----	
6048		----		----	
6076	EN12607-1	53	R(0.05)	-3.09	
6093		----		----	
6094		----		----	
6095		----		----	
6096		----		----	
6097		----		----	
6098		----		----	

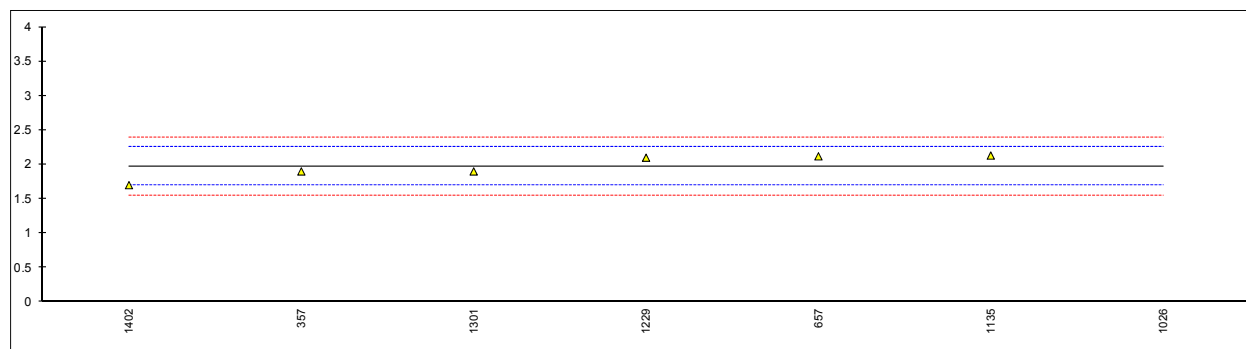
normality OK
n 21
outliers 2
mean (n) 64.03
st.dev. (n) 2.782
R(calc.) 7.79
R(EN12607-1:14) 10.00



Determination of RTFOT at 163°C, Viscosity Ratio on sample #16275;

lab	method	value	mark	z(targ)	remarks
154		----		----	
168		----		----	
225		----		----	
332		----		----	
333		----		----	
335		----		----	
336		----		----	
337		----		----	
353		----		----	
357	EN12607-1	1.90		-0.53	
396		----		----	
398		----		----	
399		----		----	
444		----		----	
445		----		----	
447		----		----	
604		----		----	
657	D2872	2.12		1.03	
865		----		----	
1011		----		----	
1016		----		----	
1026	EN12607-1	46	G(0.01)	312.05	
1040		----		----	
1108		----		----	
1131		----		----	
1135	EN12607-1	2.131		1.10	
1229	EN12607-1	2.1		0.88	
1301	EN12607-1	1.90		-0.53	
1378		----		----	
1394		----		----	
1402	EN12607-1	1.7		-1.95	
1613		----		----	
1631		----		----	
1710		----		----	
1717		----		----	
1724		----		----	
1730		----		----	
1741		----		----	
1833		----		----	
1849		----		----	
1852		----		----	
1884		----		----	
1944		----		----	
6037		----		----	
6048		----		----	
6076		----		----	
6093		----		----	
6094		----		----	
6095		----		----	
6096		----		----	
6097		----		----	
6098		----		----	

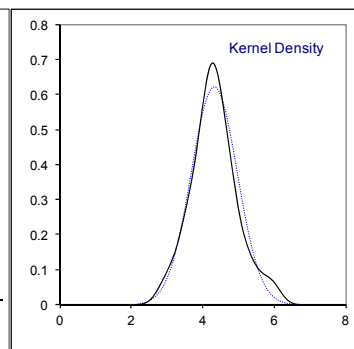
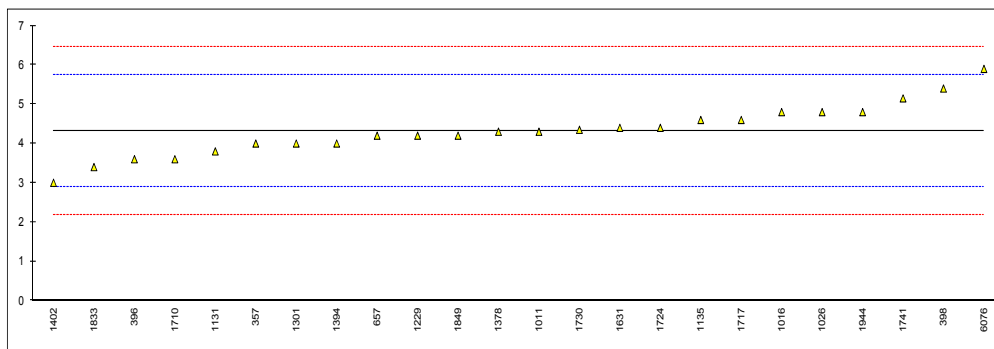
normality unknown
n 6
outliers 1
mean (n) 1.975
st.dev. (n) 0.1720
R(calc.) 0.482
R(EN12607-1:14) 0.395



Determination of RTFOT at 163°C, Increase in Softening Point on sample #16275; results in °C

lab	method	value	mark	z(targ)	remarks
154		----		----	
168		----		----	
225		----		----	
332		----		----	
333		----		----	
335		----		----	
336		----		----	
337		----		----	
353		----		----	
357	EN12607-1	4.0		-0.46	
396	EN12607-1	3.6		-1.02	
398	EN12607-1	5.4		1.50	
399		----		----	
444		----		----	
445		----		----	
447		----		----	
604		----		----	
657	D2872	4.2		-0.18	
865		----		----	
1011	EN12607-1	4.3		-0.04	
1016	EN12607-1	4.8		0.66	
1026	EN12607-1	4.8		0.66	
1040		----		----	
1108		----		----	
1131		3.8		-0.74	
1135	EN12607-1	4.6		0.38	
1229	EN12607-1	4.2		-0.18	
1301	EN12607-1	4.0		-0.46	
1378	EN12607-1	4.3		-0.04	
1394		4.0		-0.46	
1402	EN12607-1	3		-1.86	
1613		----		----	
1631	EN12607-1	4.4		0.10	
1710	EN12607-1	3.6		-1.02	
1717	EN12607-1	4.6		0.38	
1724	D2872	4.4		0.10	
1730	EN12607-1	4.35		0.03	
1741	EN12607-1	5.15		1.15	
1833	EN12607-1	3.4		-1.30	
1849	EN12607-1	4.2		-0.18	
1852		----		----	
1884		----		----	
1944	EN12607-1	4.8		0.66	
6037		----		----	
6048		----		----	
6076	EN12607-1	5.9		2.20	
6093		----		----	
6094		----		----	
6095		----		----	
6096		----		----	
6097		----		----	
6098		----		----	

normality OK
n 24
outliers 0
mean (n) 4.33
st.dev. (n) 0.641
R(calc.) 1.80
R(EN12607-1:14) 2.00

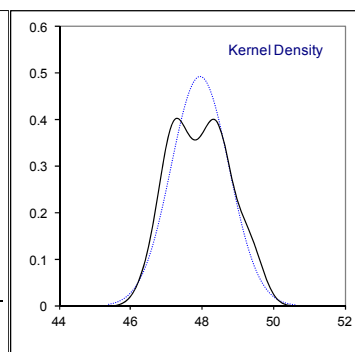
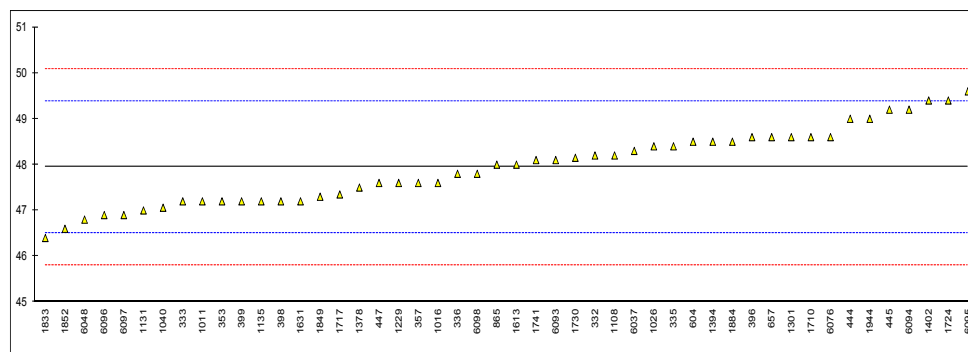


Determination of Softening Point (Ring & Ball) on sample #16275; results in °C

lab	method	value	mark	z(targ)	remarks
154		----		----	
168		----		----	
225		----		----	
332	EN1427	48.2		0.36	
333	EN1427	47.2		-1.04	
335	EN1427	48.4		0.64	
336	EN1427	47.8		-0.20	
337		----		----	
353	EN1427	47.2		-1.04	
357	EN1427	47.6		-0.48	
396	EN1427	48.6		0.92	
398	EN1427	47.2		-1.04	
399	EN1427	47.2		-1.04	
444	IP58	49.0		1.48	
445	D36	49.2		1.76	
447	EN1427	47.6		-0.48	
604	D36	48.5		0.78	
657	D36	48.6		0.92	
865	D36	48.0		0.08	
1011	EN1427	47.2		-1.04	
1016	EN1427	47.6		-0.48	
1026	EN1427	48.4		0.64	
1040	EN1427	47.06		-1.24	
1108	EN1427	48.2		0.36	
1131	EN1427	47.0		-1.32	
1135	EN1427	47.2		-1.04	
1229	EN1427	47.6		-0.48	
1301	EN1427	48.6		0.92	
1378	D36	47.5		-0.62	
1394		48.5		0.78	
1402	EN1427	49.4		2.04	
1613	D36	48.0		0.08	
1631	EN1427	47.2		-1.04	
1710	EN1427	48.6		0.92	
1717	EN1427	47.35		-0.83	
1724	D36	49.4		2.04	
1730	EN1427	48.15		0.29	
1741	EN1427	48.10		0.22	
1833	EN1427	46.4		-2.16	
1849	EN1427	47.3		-0.90	
1852	EN1427	46.6		-1.88	
1884	D36	48.5		0.78	
1944	EN1427	49		1.48	
6037	EN1427	48.3		0.50	
6048	EN1427	46.8		-1.60	
6076	EN1427	48.6		0.92	
6093	EN1427	48.1		0.22	
6094	EN1427	49.2		1.76	
6095	EN1427	49.6		2.32	
6096	EN1427	46.9		-1.46	
6097	EN1427	46.9		-1.46	
6098	EN1427	47.8		-0.20	

normality OK
n 48
outliers 0
mean (n) 47.945
st.dev. (n) 0.8111
R(calc.) 2.271
R(EN1427:15) 2.000

Compare R(D36/D36M:13) = 9.630

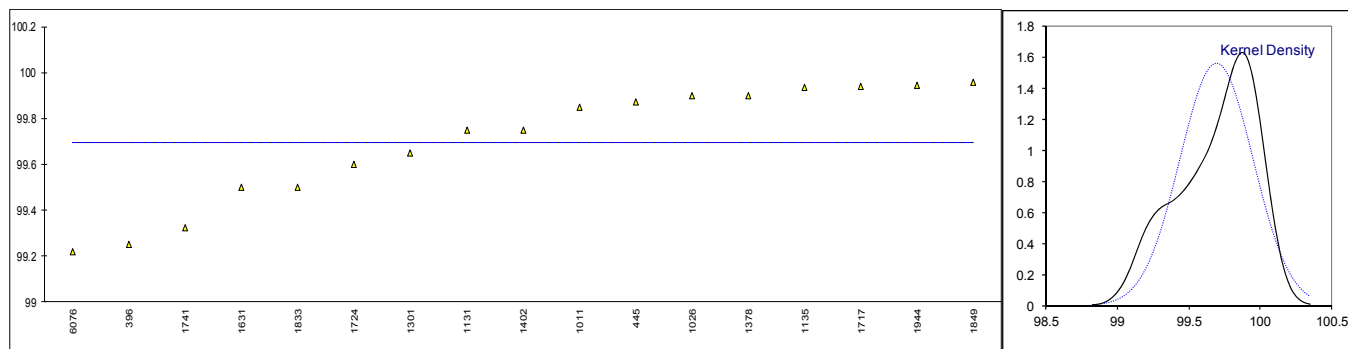


Determination of Solubility in Xylene on sample #16275; results in %M/M

lab	method	value	mark	z(targ)	remarks
154		----		----	
168		----		----	
225		----		----	
332		----		----	
333		----		----	
335		----		----	
336		----		----	
337		----		----	
353		----		----	
357		----		----	
396	EN12592	99.25		----	Solvent used: Xylene
398		----		----	
399		----		----	
444		----		----	
445	IP47	99.87		----	Solvent used: Xylene
447		----		----	
604		----		----	
657		----		----	
865		----		----	
1011	EN12592	99.85		----	
1016		----		----	
1026	EN12592	99.9		----	Solvent used: Xylene
1040		----		----	
1108		----		----	
1131	EN12592	99.750		----	Solvent used: Xylene
1135	EN12592	99.935		----	Solvent used: Xylene
1229		----		----	
1301	EN12592	99.64952		----	Solvent used: Xylene
1378	EN12592	99.9		----	Solvent used: Toluene
1394		----		----	
1402	EN12592	99.75		----	Solvent used: Xylene
1613		----		----	
1631	EN12592	99.50		----	Solvent used: Toluene
1710		----		----	
1717	EN12592	99.94		----	Solvent used: Toluene
1724	EN12592	99.6		----	Solvent used: Xylene
1730		----		----	
1741	EN12592	99.325		----	Solvent used: Xylene
1833	EN12592	99.50		----	Solvent used: Toluene
1849	EN12592	99.96		----	Solvent used: Toluene
1852		----		----	
1884		----		----	
1944	EN12592	99.943		----	Solvent used: Xylene
6037		----		----	
6048		----		----	
6076	EN12592	99.22		----	Solvent used: Xylene
6093		----		----	
6094		----		----	
6095		----		----	
6096		----		----	
6097		----		----	
6098		----		----	

normality OK
n 17
outliers 0
mean (n) 99.697
st.dev. (n) 0.2552
R(calc.) 0.714
R(EN12592:14) (0.150)

Compare R(iis15F02) = 0.101



APPENDIX 2

Number of participants per country

1 lab in ALBANIA
1 lab in BELGIUM
1 lab in CHINA, People's Republic of
1 lab in COTE D'IVOIRE
2 labs in FINLAND
5 labs in FRANCE
1 lab in GEORGIA
3 labs in GERMANY
3 labs in GREECE
2 labs in HUNGARY
1 lab in IRELAND
3 labs in ITALY
1 lab in JORDAN
1 lab in LITHUANIA
1 lab in MALAYSIA
9 labs in NETHERLANDS
1 lab in PORTUGAL
1 lab in ROMANIA
1 lab in RUSSIAN FEDERATION
1 lab in SERBIA
1 lab in SINGAPORE
4 labs in TURKEY
1 lab in UNITED ARAB EMIRATES
4 labs in UNITED KINGDOM
2 labs in UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations:

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= 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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