Results of Proficiency Test
Bitumen
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Appendices:

1   INTRODUCTION

Since 2014 the Institute for Interlaboratory Studies (iis) organizes a proficiency test for Bitumen every year. During the annual proficiency testing program 2020/2021 it was decided to continue the round robin for the analysis on Bitumen based on the scope of the latest specification of EN12591 Paving Grade. In this interlaboratory study 50 laboratories in 27 different countries registered for participation. See appendix 2 for the number of participants per country.

In this report the results of the Bitumen proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2   SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send one 2.5L can of Bitumen grade 35/50 labelled #20240. The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1   QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on a regular basis by sending out questionnaires.

2.2   PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3   CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

A batch of 60 subsamples Bitumen grade 35/50 in 2.5 L cans was obtained from a local supplier. Each can has been labelled #20240.
The homogeneity of the subsamples was checked by determination of Softening Point in accordance with EN1427 and Penetration at 25°C in accordance with EN1426 on 4 stratified randomly selected subsamples.

	Softening Point (Ring and Ball) in °C	Penetration at 25°C in 0.1mm
Sample #20240-1	61.2	43
Sample #20240-2	61.6	43
Sample #20240-3	61.2	43
Sample #20240-4	61.0	42

Table 1: homogeneity test results of subsamples #20240

From the above test results the repeatabilities were calculated and compared with the corresponding repeatability of the reference test methods in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Softening Point (Ring and Ball) in °C	Penetration at 25°C in 0.1mm
r (observed)	0.7	1.4
reference test method	EN1427:15	EN1426:15
r (reference test method)	1.0	2.0

Table 2: evaluation of the repeatabilities of subsamples #20240

The calculated repeatabilities were in agreement with the repeatabilities of the corresponding reference test method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one 2.5 L can of sample #20240 was sent on November 4, 2020. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Bitumen stored in the metal cans was checked. The material has been found sufficiently stable for the period of the proficiency test.

2.6 ANALYZES

The participants were asked to determine on sample #20240: Density at 25°C, Dynamic Viscosity at 60°C, Flash Point C.O.C., Fraass Breaking Point, Kinematic Viscosity at 135°C, Penetration at 25°C, Penetration Index, RTFOT at 163°C (Change of Mass, Retained Penetration, Viscosity Ratio and Increase in Softening Point), Softening Point (Ring and Ball) and Solubility in Xylene.

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

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the appropriate reference test methods that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3   RESULTS

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

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

3.1   STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5).
For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.

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

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

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

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

3.2   GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3   Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ISO, EN reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation of this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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

The z-scores were calculated according to:

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

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

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

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

4   EVALUATION

In this proficiency test no problems were encountered with the dispatch of the samples.
All participants reported test results. Three participants reported the test results after the final reporting date. Not all participants were able to report all analyzes requested.
Finally, 50 participants reported in total 315 numerical test results. Observed were 14 outlying test results, which is 4.4%. 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 test methods, which were used by the various laboratories, were taken into account for explaining the observed differences where possible and applicable. These test methods are also in the tables together with the reported test results in appendix 1. The abbreviations, used in these tables, are explained in appendix 3.

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

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

Flash Point C.O.C.: This determination was very problematic. One statistical outlier was observed. Six of the fifteen participants reported a "larger than" value. These six obervations will have an influence on the average, whereby the actual value will be higher than calculated. Because of this significant influence on the mean value it was decided not to calculate z-scores.

Fraass Breaking Point: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of EN12593:15.

Kinematic Viscosity at 135°C: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of EN12595:14 or ASTM D2170/2170M:18.

Penetration at 25°C: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of EN1426:15 or with the requirements of ASTM D5/5M:19a. When the test results of test method EN1426 and ASTM D5/5M are evaluated separately, both reproducibilities are not in agreement with the requirements of the respective test methods.

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

Penetration Index: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN12591:09.

RTFOT at 163°C: The determination on Change of Mass was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN12607-1:14.

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 EN12607-1:14.

For the determination on Viscosity Ratio only a few participants (three) reported test results. Due to the large variation in the test results it was decided not to calculate z-scores.

The determination on Increase in Softening Point was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of EN12607-1:14.

Softening Point (Ring and Ball): This determination was problematic depending on the test method used. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the strict requirements of EN1427:15, but is in agreement with the requirements of ASTM D36/36M:14e1.

Solubility in Xylene: Eighteen participants reported test results. No statistical outliers were observed. However, due to the large variation in the test results, it was decided not to calculate z-scores. The groups seem to be divided bimodally.

4.2   PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Parameter	unit	n	average	2.8 * sd	R(lit)
Density at 25°C	kg/m^3	38	1090.0	8.1	5
Dynamic Viscosity at 60°C	Pa.s	15	2591	581	259
Flash Point C.O.C.	°C	14	n.e.	n.e.	n.e.
Fraass Breaking Point	°C	16	-17.6	8.7	6
Kinematic Viscosity at 135°C	mm^2/s	17	1268.9	247.1	114.2
Penetration at 25°C	0.1 mm	48	41.5	7.7	2.5
Penetration Index		19	0.94	0.51	0.5
RTFOT - Change of Mass	%	28	-0.08	0.09	0.20
RTFOT - Retained Penetration	%	23	67.0	17.3	10
RTFOT - Viscosity Ratio		3	n.e.	n.e.	n.e.
RTFOT - Increase in Soft. Point	°C	24	13.2	4.4	4.0
Softening Point (Ring & Ball)	°C	44	61.8	2.4	2.0
Solubility in Xylene	%M/M	18	91.65	15.33	(0.15)

Table 3: reproducibilities of tests on sample #20240

Results between brackets should be used with due care

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

4.3   COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2020 WITH PREVIOUS PTS

	December 2020	December 2019	December 2018	December 2017	December 2016
Number of reporting laboratories	50	45	37	50	51
Number of test results	315	310	511	289	318
Number of statistical outliers	14	11	15	7	8
Percentage of statistical outliers	4.4%	3.5%	2.9%	2.4%	2.5%

Table 4: comparison with previous proficiency tests

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

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

Parameter	December 2020	December 2019	December 2018		December 2017	December 2016
Paving Grade	35/50	70/100	70/100	35/50	70/100	50/70
Density at 25°C	-	-	-	--	+/-	-
Dynamic Viscosity at 60°C	--	-	+	-	--	+
Flash Point C.O.C.	(--)	-	-	--	--	--
Fraass Breaking Point	-	+/-	+	+/-	-	++
Kinematic Viscosity at 135°C	--	-	-	-	--	--
Penetration at 25°C	--	-	-	--	--	--
Penetration Index	+/-	-	+	+	-	--
RTFOT - Change of Mass	++	--	(--)	+/-	(--)	++
RTFOT - Retained Penetration	-	+/-	-	-	+	++
RTFOT - Viscosity Ratio	n.e.	(--)	+	+/-	--	-
RTFOT - Increase in Soft. Point	+/-	-	+/-	+/-	+	+
Softening Point (Ring and Ball)	-	+/-	+	+	-	-
Solubility in Xylene	(--)	(--)	+/-	+	n.e.	n.e.

Table 5: comparison determinations against the reference test methods

Results between brackets should be used with care

In the table above the following performance categories were used:

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

APPENDIX 1

Determination of Density at 25°C on sample #20240; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
154	D70	1089		-0.58	
168	D70	1087		-1.70	
225	D70	1090		-0.02	
333		-----		-----	
335	EN15326	1093	C	1.66	First reported 1.093 kg/m^3
336	EN15326	1091	C	0.54	First reported 1.091 kg/m^3
342	D70	1089		-0.58	
353	D70	1090.2		0.10	
357	D70	1089.9		-0.07	
360	EN15326	1090.38		0.20	
396	ISO3838	1090.6		0.32	
398		-----		-----	
399	D70	1093.0		1.66	
444	D70	1089.4		-0.35	
447		-----		-----	
604	D70	1086.7		-1.86	
657	D70	1091	C	0.54	First reported 1.103 kg/m^3
781	EN15326	1086.5		-1.98	
865	D70	1091.3		0.71	
963	D70	1083.5		-3.66	
1011	D70	1087	C	-1.70	First reported 1.087 kg/m^3
1016	EN15326	1096.3	C	3.51	First reported 1102.8
1026	EN15326	1091		0.54	
1040	ISO12185	1089.2		-0.46	
1047		-----		-----	
1082	EN15326	1088.19		-1.03	
1108	EN15326	1085.7	C	-2.42	First reported 1081.4
1135	EN15326	1088.0		-1.14	
1229	EN15326	1094		2.22	
1259	EN15326	1085		-2.82	
1378	D70	1090.1		0.04	
1402	IP189/190	1091.6	C	0.88	First reported 1.0916 kg/m^3
1468	EN15326	1089.5		-0.30	
1539	EN15326	1094	C	2.22	First reported 1096.2
1613	DIN51757	1092.3		1.27	
1631		-----		-----	
1724		-----		-----	
1730		-----		-----	
1741	EN15326	1094.84		2.69	
1833		-----		-----	
1849		-----		-----	
1852		-----		-----	
1881	D70	1094.3		2.39	
1944	EN15326	1085.37		-2.61	
1990	D70	1092.4		1.33	
6054	D70	1089.82788		-0.11	
6076	EN15326	1091		0.54	
6225		-----		-----	
6228		-----		-----	
6229	EN15326	1090		-0.02	

normality	OK	
n	38	
outliers	0	
mean (n)	1090.029	
st.dev. (n)	2.8960	
R(calc.)	8.109	
st.dev.(EN15326:07)	1.7857	
R(EN15326:07)	5	
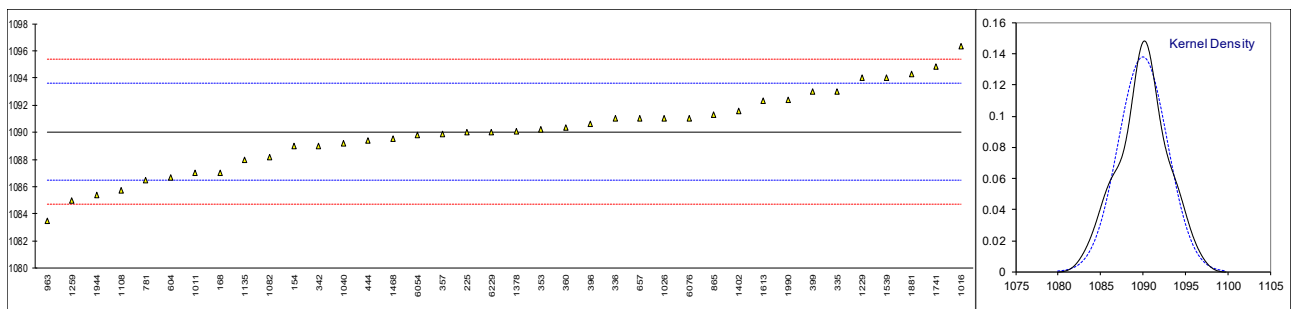
Determination of Dynamic Viscosity at 60°C on sample #20240; results in Pa.s

lab	method	value	mark	z(targ)	remarks
154	D2171	2410		-1.96	
168	D2171	2434		-1.70	
225		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
342		-----		-----	
353		-----		-----	
357	EN12596	2855		2.85	
360		-----		-----	
396	EN12596	2752		1.74	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447	EN13302	>500		-----	
604		-----		-----	
657	D2171	2274		-3.43	
781	EN12596	>800		-----	
865	D2171	2970		4.09	
963		-----		-----	
1011		-----		-----	
1016	EN12596	2514		-0.84	
1026	EN12596	2730		1.50	
1040		-----		-----	
1047		-----		-----	
1082	EN12596	2388		-2.20	
1108		-----		-----	
1135	EN12596	2391		-2.16	
1229		-----		-----	
1259		-----		-----	
1378		-----		-----	
1402	EN12596	2478		-1.22	
1468	EN12596	3550.1	C,G(0.01)	10.36	First reported 3.5501
1539	EN12596	2868		2.99	
1613	D2171	--		-----	
1631		-----		-----	
1724		-----		-----	
1730		-----		-----	
1741	EN12596	2539.6		-0.56	
1833		-----		-----	
1849		-----		-----	
1852		-----		-----	
1881	EN12596	2594		0.03	
1944		-----		-----	
1990	D2171	2672.91		0.88	
6054		-----		-----	
6076		-----		-----	
6225		-----		-----	
6228		-----		-----	
6229		-----		-----	

normality	OK
n	15
outliers	1
mean (n)	2591.37
st.dev. (n)	207.446
R(calc.)	580.85
st.dev.(EN12596:14)	92.549
R(EN12596:14)	259.14
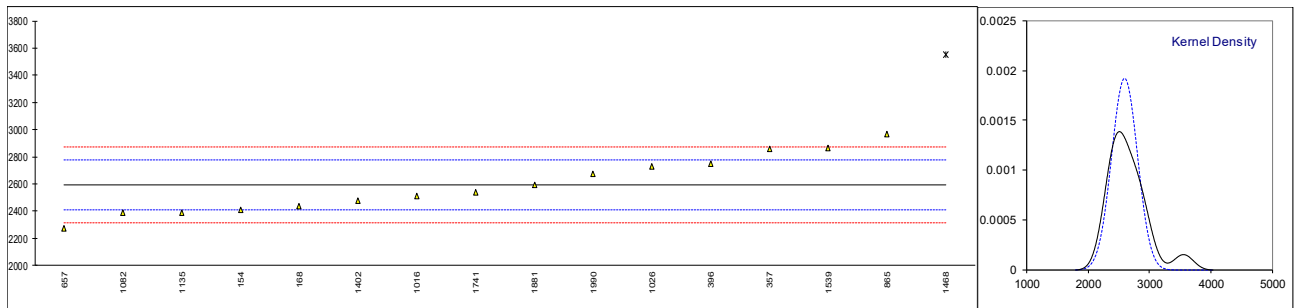
Determination of Flash Point C.O.C. on sample #20240; results in °C

lab	method	mode	value	mark	z(targ)	remarks
154			-----		-----	
168			-----		-----	
225			-----		-----	
333			-----		-----	
335			-----		-----	
336			-----		-----	
342			-----		-----	
353			-----		-----	
357			-----		-----	
360	ISO2592		296.0		-----	
396	ISO2592	Manual	>340		-----	
398			-----		-----	
399			-----		-----	
444			-----		-----	
447			-----		-----	
604			-----		-----	*)
657			-----		-----	
781	ISO2592	Automated	>330		-----	
865			-----		-----	
963			-----		-----	*)
1011	ISO2592	Automated	>280		-----	
1016			-----		-----	
1026			-----		-----	*)
1040	ISO2719	Automated	291.0		-----	
1047			-----		-----	
1082	ISO2592		272.0		-----	
1108			-----		-----	
1135	ISO2592	Automated	279.0		-----	
1229	ISO2592	Automated	>290		-----	
1259			-----		-----	*)
1378	D92		254		-----	
1402	ISO2592	Manual	>290		-----	
1468			-----		-----	*)
1539			-----		-----	
1613	D92	Manual	270		-----	
1631			-----		-----	
1724			-----		-----	*)
1730			-----		-----	
1741			-----		-----	*)
1833	ISO2592		300		-----	
1849	ISO2592	Automated	302		-----	
1852			-----		-----	
1881			-----		-----	
1944	ISO2592	Manual	364	C,G(0.05)	-----	First reported 344
1990			-----		-----	
6054			-----		-----	
6076	ISO2592	Automated	> 302		-----	*)
6225			-----		-----	
6228			-----		-----	
6229			-----		-----	

			Without the > values
normality	OK		OK
n	14		8
outliers	1		1
mean (n)	292.57		283.00
st.dev. (n)	22.657		17.0294
R(calc.)	63.44		47.68
st.dev.(ISO2592:17)	(6.429)		(6.429)
R(ISO2592:17)	(18)		(18)
Compare			
R(D92:18)	(18)		(18)

*) Several laboratories reported that it was not possible to determine the Flash Point as the Bitumen sample showed a deviating behaviour, e.g. foam was produced.
In the calculation of the mean for the graphics results a reported value of '>x'is changed into x (for example >302 into 302).
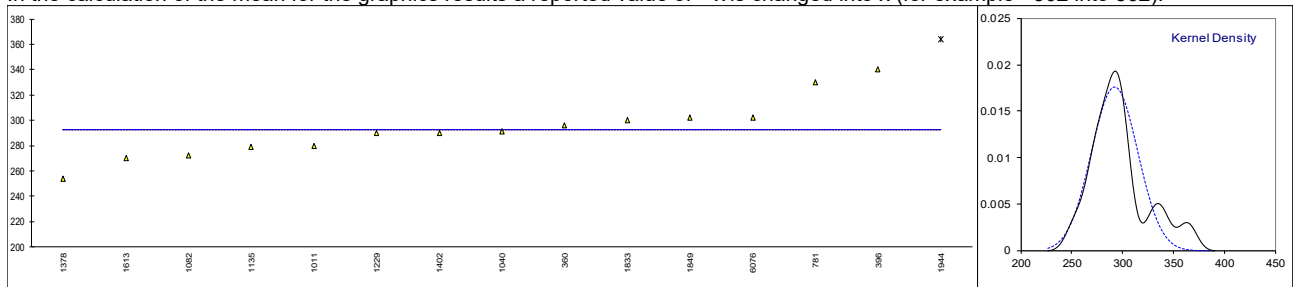
Determination of Fraass Breaking Point on sample #20240; results in °C

lab	method	mode	value	mark	z(targ)	remarks
154			-----		-----	
168			-----		-----	
225			-----		-----	
333			-----		-----	
335			-----		-----	
336			-----		-----	
342			-----		-----	
353			-----		-----	
357	EN12593	Automated	-18		-0.21	
360	EN12593		-18.0		-0.21	
396	EN12593	Manual	-10		3.53	
398			-----		-----	
399			-----		-----	
444			-----		-----	
447			-----		-----	
604			-----		-----	
657			-----		-----	
781	EN12593	Automated	-18		-0.21	
865			-----		-----	
963			-----		-----	
1011	EN12593	Automated	-19		-0.67	
1016	EN12593	Automated	-18		-0.21	
1026			-----		-----	
1040			-----		-----	
1047			-----		-----	
1082			-----		-----	
1108			-----		-----	
1135	EN12593	Automated	-20		-1.14	
1229	EN12593		-22		-2.07	
1259			-----		-----	
1378			-----		-----	
1402	EN12593	Manual	-4	G(0.05)	6.33	
1468	EN12593	Manual	-15		1.19	
1539	EN12593		-16		0.73	
1613			--		-----	
1631			-----		-----	
1724			-----		-----	
1730			-----		-----	
1741	EN12593		-22.0		-2.07	
1833			-----		-----	
1849			-----		-----	
1852	EN12593	Automated	-15.9		0.77	
1881			-----		-----	
1944	EN12593	Manual	-13		2.13	
1990			-----		-----	
6054			-----		-----	
6076	EN12593	Automated	-20		-1.14	
6225			-----		-----	
6228	EN12593	Automated	-18		-0.21	
6229	EN12593	Manual	-18		-0.21	

normality	suspect	
n	16	
outliers	1	
mean (n)	-17.56	
st.dev. (n)	3.101	
R(calc.)	8.68	
st.dev.(EN12593:15)	2.143	
R(EN12593:15)	6	
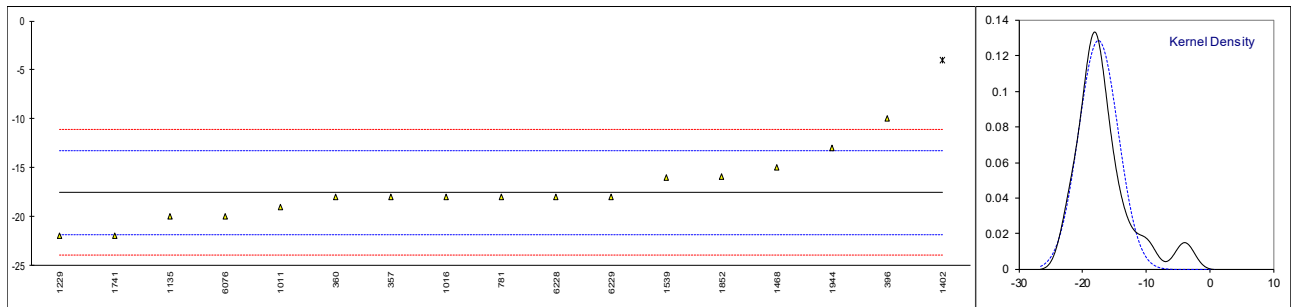
Determination of Kinematic Viscosity at 135°C on sample #20240; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
154	D2170	1310		1.01	
168		-----		-----	
225	EN12595	1402		3.26	
333		-----		-----	
335		-----		-----	
336		-----		-----	
342		-----		-----	
353		-----		-----	
357	EN12595	1136		-3.26	
360		-----		-----	
396	EN12595	1188		-1.98	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2170	1122.5		-3.59	
781	EN12595	>1200		-----	
865	D2170	1292		0.57	
963		-----		-----	
1011		-----		-----	
1016	EN12595	1151		-2.89	
1026	EN12595	1270		0.03	
1040		-----		-----	
1047		-----		-----	
1082	EN12595	1291.8		0.56	
1108	EN12595	1569	C,G(0.05)	7.36	First reported 1065
1135	EN12595	1343		1.82	
1229	EN12595	1165		-2.55	
1259	EN12595	1365		2.36	
1378		-----		-----	
1402	EN12595	1309		0.98	
1468		-----		-----	
1539	EN12595	1362	C	2.28	First reported 1441.6
1613	D2170	1290		0.52	
1631		-----		-----	
1724		-----		-----	
1730		-----		-----	
1741	EN12595	1224.8		-1.08	
1833		-----		-----	
1849		-----		-----	
1852		-----		-----	
1881		-----		-----	
1944		-----		-----	
1990		-----		-----	
6054		-----		-----	
6076		-----		-----	
6225		-----		-----	
6228		-----		-----	
6229	EN12595	1348.8		1.96	

normality	OK
n	17
outliers	1
mean (n)	1268.88
st.dev. (n)	88.238
R(calc.)	247.07
st.dev.(EN12595:14)	40.785
R(EN12595:14)	114.20
Compare	
R(D2170/D2170M:18)	111.66
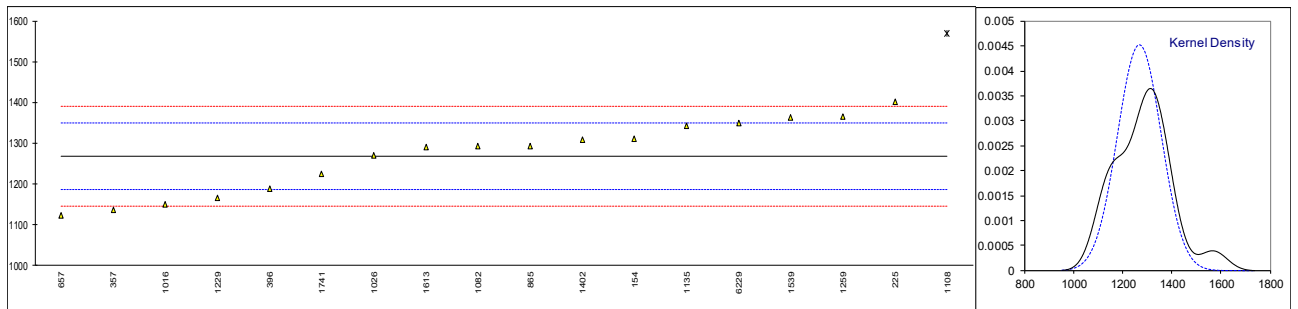
Determination of Penetration at 25°C on sample #20240; results in 0.1 mm

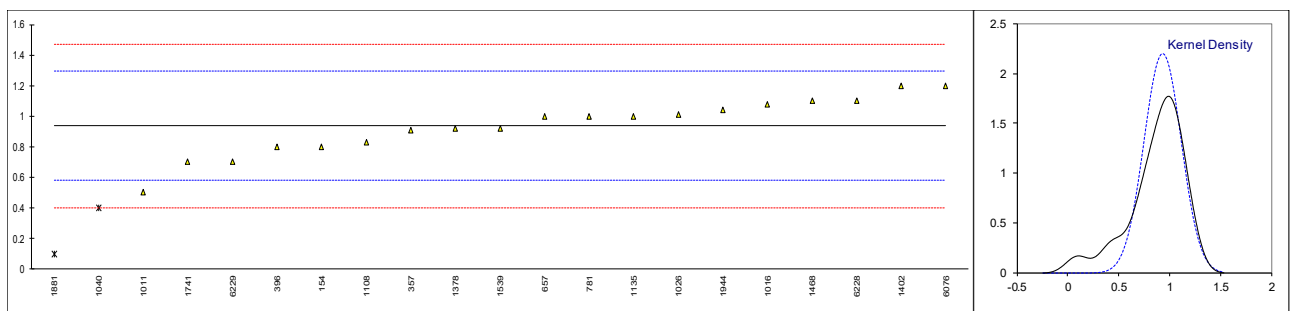| lab	method	mode	value	mark	z(targ)	remarks
154	D5	Manual	39.4		-2.38	
168	D5	Manual	43.3		2.00	
225	D5	Manual	40		-1.71	
333	EN1426		43.0		1.66	
335	EN1426		40		-1.71	
336	EN1426		43		1.66	
342	EN1426		39.0		-2.83	
353	EN1426		45		3.91	
357	EN1426	Automated	44		2.79	
360	EN1426		48.0		7.28	
396	EN1426	Manual	38		-3.96	
398	EN1426	Manual	40		-1.71	
399	EN1426	Manual	40		-1.71	
444	EN1426	Automated	38.9		-2.94	
447	EN1426	Automated	38.67		-3.20	
604	D5	Manual	44		2.79	
657	D5	Manual	45		3.91	
781	EN1426	Automated	41		-0.58	
865	D5		41.7		0.20	
963	D5		45.0		3.91	
1011	EN1426	Automated	39		-2.83	
1016	EN1426	Automated	45		3.91	
1026	EN1426		43		1.66	
1040	EN1426	Automated	37.0		-5.08	
1047	EN1426	Manual	42		0.54	
1082	EN1426		46.6		5.71	
1108	EN1426		41.2		-0.36	
1135	EN1426	Automated	43		1.66	
1229	EN1426	Automated	43		1.66	
1259	EN1426	Automated	43		1.66	
1378	D5		37		-5.08	
1402	EN1426	Manual	46		5.04	
1468	EN1426	Automated	42		0.54	
1539	EN1426		42		0.54	
1613			-----	W	-----	Test result withdrawn, reported 34
1631	EN1426	Automated	40		-1.71	
1724	EN1426		38		-3.96	
1730	EN1426		37.5		-4.52	
1741	EN1426		40.0		-1.71	
1833	EN1426		37		-5.08	
1849	EN1426	Automated	38		-3.96	
1852	EN1426	Automated	40		-1.71	
1881	EN1426	Automated	39.4		-2.38	
1944	EN1426	Automated	45		3.91	
1990	D5		40.7		-0.92	
6054	D5		44		2.79	
6076	EN1426	Automated	44		2.79	
6225			-----		-----	
6228	EN1426	Automated	41.6		0.09	
6229	EN1426	Automated	41		-0.58	

					Only EN1426:	Only ASTM D5/5M:
	normality		OK		OK	OK
	n		48		38	10
	outliers		0		0	0
	mean (n)		41.520		41.391	42.010
	st.dev. (n)		2.7632		2.8025	2.6921
	R(calc.)		7.737		7.847	7.538
	st.dev.(EN1426:15)		0.8898		0.8870	-----
	R(EN1426:15)		2.491		2.483	-----
Comapre						
	ASTM D5/D15M:19a		4.460		-----	4.529
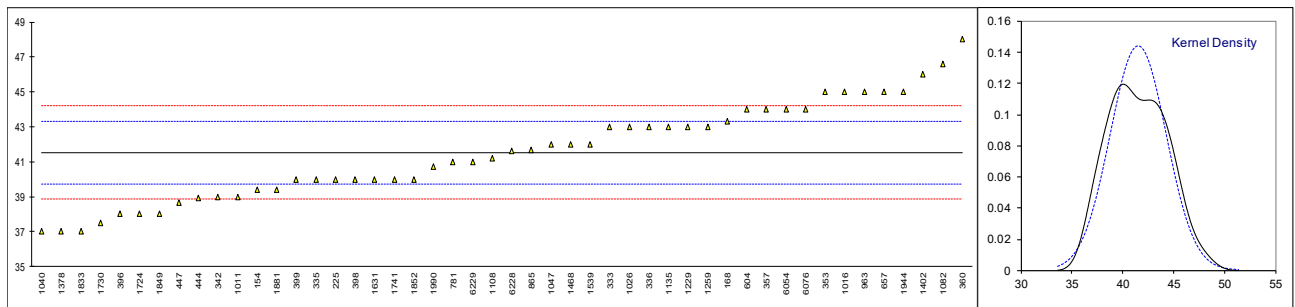
Determination of Penetration Index on sample #20240;

lab	method	value	mark	z(targ)	remarks
154	EN12591	0.8		-0.77	
168		-----		-----	
225		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
342		-----		-----	
353		-----		-----	
357	EN12591	0.91		-0.15	
360		-----		-----	
396	EN12591	0.8		-0.77	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	Calculation	1.0		0.35	
781	EN12591	1.0		0.35	
865		-----		-----	
963		-----		-----	
1011	EN12591	0.5		-2.45	
1016	EN12591	1.08		0.80	
1026	EN12591	1.01		0.41	
1040	EN12591	0.4	G(0.05)	-3.01	
1047		-----		-----	
1082		-----		-----	
1108	EN12591	0.829		-0.61	
1135	EN12591	1.0		0.35	
1229		-----		-----	
1259		-----		-----	
1378	EN12591	0.92		-0.10	
1402	EN12591	1.2		1.47	
1468	EN12591	1.1		0.91	
1539	EN12591	0.92		-0.10	
1613	EN12591	--		-----	
1631		-----		-----	
1724		-----		-----	
1730		-----		-----	
1741	EN12591	0.70		-1.33	
1833		-----		-----	
1849		-----		-----	
1852		-----		-----	
1881	EN12591	0.1	C,G(0.05)	-4.69	First reported -0.1
1944	EN12591	1.039		0.57	
1990		-----		-----	
6054		-----		-----	
6076	EN12591	1.2		1.47	
6225		-----		-----	
6228	EN12591	1.1		0.91	
6229	EN12591	0.7		-1.33	

normality	OK	
n	19	
outliers	2	
mean (n)	0.937	
st.dev. (n)	0.1816	
R(calc.)	0.508	
st.dev.(EN12591:09)	0.1786	
R(EN12591:09)	0.5	

Determination of RTFOT at 163°C, Change of Mass on sample #20240; results in %
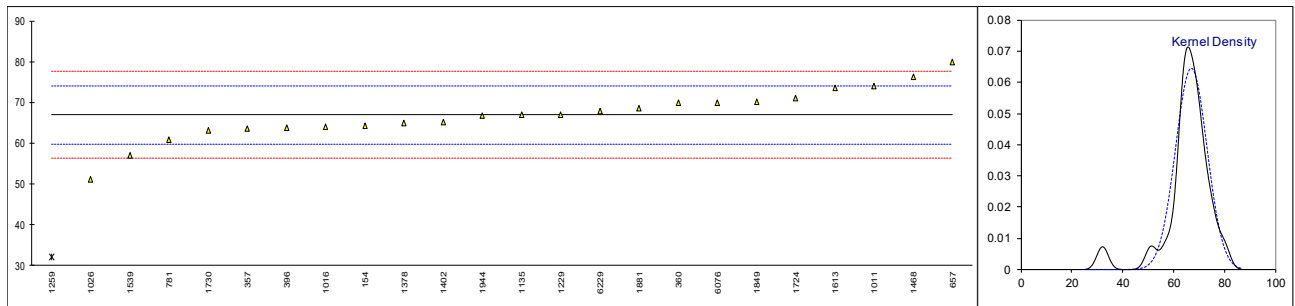
lab	method	value	mark	z(targ)	remarks
154	D2872	-0.117		-0.51	
168	D2872	-0.121		-0.57	
225		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
342		-----		-----	
353		-----		-----	
357	EN12607-1	-0.056		0.34	
360	EN12607-1	-0.053		0.39	
396	EN12607-1	-0.062		0.26	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2872	-0.11		-0.41	
781	EN12607-1	-0.10		-0.27	
865	D2872	-0.108		-0.38	
963		-----		-----	
1011	EN12607-1	-0.08		0.01	
1016	EN12607-1	-0.14		-0.83	
1026	EN12607-1	-0.02		0.85	
1040		-----		-----	
1047		-----		-----	
1082		-0.1227959		-0.59	
1108		-----		-----	
1135	EN12607-1	-0.09		-0.13	
1229	EN12607-1	-0.08		0.01	
1259	EN12607-1	-0.09		-0.13	
1378	EN12607-1	-0.059		0.30	
1402	EN12607-1	-0.07		0.15	
1468		-0.075		0.08	
1539		-0.09		-0.13	
1613	D2872	-0.04		0.57	
1631	EN12607-1	-0.11		-0.41	
1724	EN12607-1	-0.0880		-0.10	
1730		0.139	C,R(0.01)	3.07	First reported 0.364
1741		-0.0451		0.50	
1833	EN12607-1	-0.06		0.29	
1849	EN12607-1	-0.1	C	-0.27	First reported 0.1
1852		-----		-----	
1881	EN12607-1	-0.07		0.15	
1944	EN12607-1	0.121	C,R(0.01)	2.82	First reported 0.081
1990		-----		-----	
6054		-----		-----	
6076	EN12607-1	-0.08		0.01	
6225		-----		-----	
6228		-----		-----	
6229	EN12607-1	-0.02		0.85	

normality	OK
n	28
outliers	2
mean (n)	-0.0806
st.dev. (n)	0.03039
R(calc.)	0.0851
st.dev.(EN12607-1:14)	0.07143
R(EN12607-1:14)	0.20
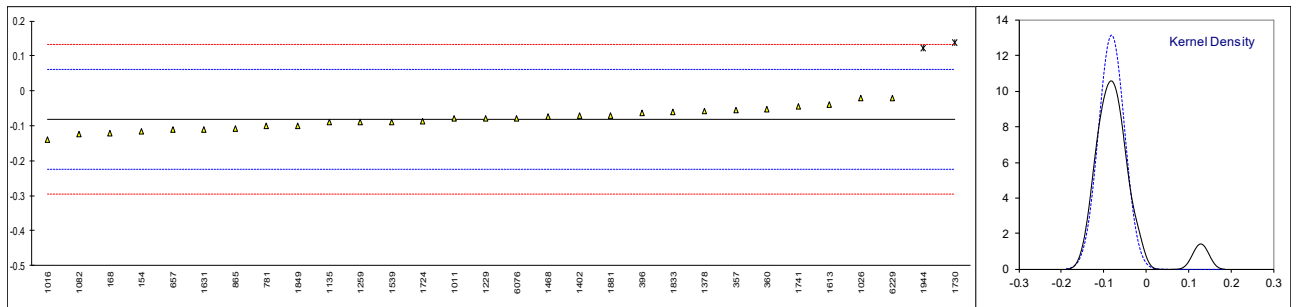
Determination of RTFOT at 163°C, Retained Penetration on sample #20240; results in %

lab	method	value	mark	z(targ)	remarks
154	D2872	64.21		-0.77	
168		-----		-----	
225		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
342		-----		-----	
353		-----		-----	
357	EN12607-1	63.6		-0.94	
360	EN12607-1	70.0		0.85	
396	EN12607-1	63.89		-0.86	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2872	80		3.65	
781	EN12607-1	60.98		-1.68	
865		-----		-----	
963		-----		-----	
1011	EN12607-1	74		1.97	
1016	EN12607-1	64	C	-0.83	First reported 55.56
1026	EN12607-1	51.1	C	-4.44	First reported 52
1040		-----		-----	
1047		-----		-----	
1082		-----		-----	
1108		-----		-----	
1135	EN12607-1	67		0.01	
1229	EN12607-1	67		0.01	
1259	EN12607-1	32	R(0.01)	-9.79	
1378	EN12607-1	64.9		-0.58	
1402	EN12607-1	65.2		-0.49	
1468		76.19		2.58	
1539		57.1		-2.76	
1613	D2872	73.5		1.83	
1631		-----		-----	
1724	EN12607-1	71.052		1.14	
1730		63.2		-1.05	
1741		-----		-----	
1833		-----		-----	
1849	EN12607-1	70.1		0.88	
1852		-----		-----	
1881	EN12607-1	68.5		0.43	
1944	EN12607-1	66.66		-0.09	
1990		-----	W	-----	Test result withdrawn, reported 95.33
6054		-----		-----	
6076	EN12607-1	70		0.85	
6225		-----		-----	
6228		-----		-----	
6229	EN12607-1	68		0.29	

|	
normality	suspect
n	23
outliers	1
mean (n)	66.96
st.dev. (n)	6.176
R(calc.)	17.29
st.dev.(EN12607-1:14)	3.571
R(EN12607-1:14)	10

Determination of RTFOT at 163°C, Viscosity Ratio on sample #20240;

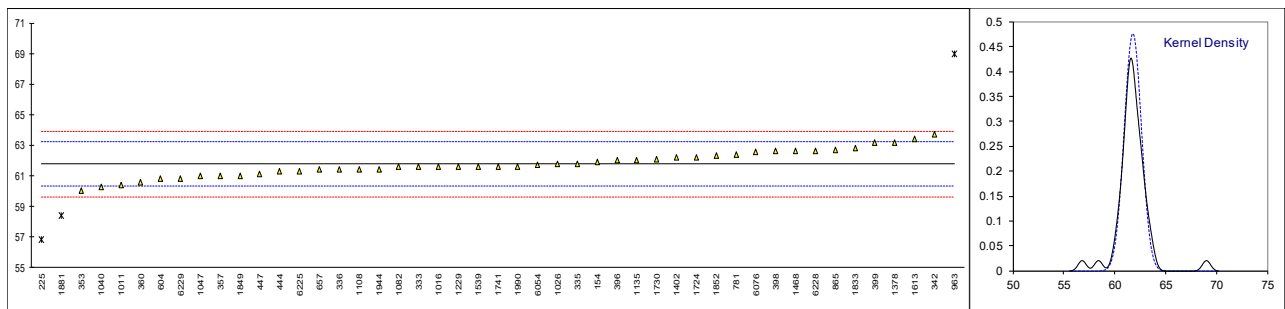| lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
342		-----		-----	
353		-----		-----	
357		-----		-----	
360		-----		-----	
396		-----		-----	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2872	6.10		-----	
781		-----		-----	
865		-----		-----	
963		-----		-----	
1011	EN12607-1	60.4		-----	
1016		-----		-----	
1026		-----		-----	
1040		-----		-----	
1047		-----		-----	
1082		-----		-----	
1108		-----		-----	
1135	EN12607-1	18.3		-----	
1229		-----		-----	
1259		-----		-----	
1378		-----		-----	
1402		-----		-----	
1468		-----		-----	
1539		-----		-----	
1613		-----		-----	
1631		-----		-----	
1724		-----		-----	
1730		-----		-----	
1741		-----		-----	
1833		-----		-----	
1849		-----		-----	
1852		-----		-----	
1881		-----		-----	
1944		-----		-----	
1990		-----		-----	
6054		-----		-----	
6076		-----		-----	
6225		-----		-----	
6228		-----		-----	
6229		-----		-----	

| | n | 3 | | | |

Determination of RTFOT at 163°C, Increase in Softening Point on sample #20240; results in °C

lab	method	value	mark	z(targ)	remarks
154	D2872	11.7		-1.06	
168		-----		-----	
225		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
342		-----		-----	
353		-----		-----	
357	EN12607-1	12.8		-0.29	
360	EN12607-1	14.60		0.97	
396	EN12607-1	13.0		-0.15	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2872	67.6	C,R(0.01)	38.07	First reported 73.2
781	EN12607-1	13.6		0.27	
865		-----		-----	
963		-----		-----	
1011	EN12607-1	13.4		0.13	
1016	EN12607-1	15.8		1.81	
1026	EN12607-1	15.0		1.25	
1040		-----		-----	
1047		-----		-----	
1082		13.2		-0.01	
1108		-----		-----	
1135	EN12607-1	13.4		0.13	
1229	EN12607-1	13.6		0.27	
1259		-----		-----	
1378	EN12607-1	11.9		-0.92	
1402	EN12607-1	13.0		-0.15	
1468		10.4		-1.97	
1539		14.6		0.97	
1613	D2872	11.2		-1.41	
1631	EN12607-1	13.3		0.06	
1724	EN12607-1	11.6		-1.13	
1730		6.2	C,R(0.01)	-4.91	First reported 6.6
1741		-----		-----	
1833	EN12607-1	12.8		-0.29	
1849	EN12607-1	14.0		0.55	
1852		-----		-----	
1881	EN12607-1	13.0		-0.15	
1944	EN12607-1	17.2		2.79	
1990		-----		-----	
6054		-----		-----	
6076	EN12607-1	10.8		-1.69	
6225		-----		-----	
6228		-----		-----	
6229	EN12607-1	13.2		-0.01	

|		
normality	OK	
n	24	
outliers	2	
mean (n)	13.213	
st.dev. (n)	1.5535	
R(calc.)	4.350	
st.dev.(EN12607-1:14)	1.4286	
R(EN12607-1:14)	4.0	
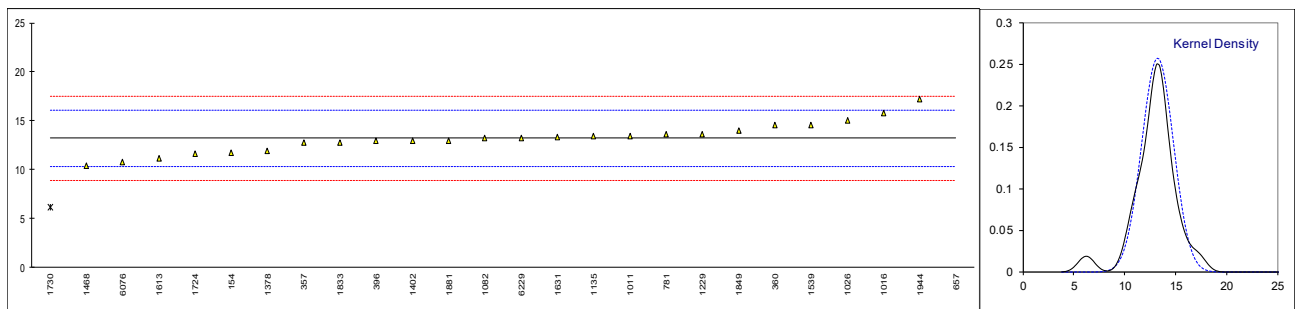
Determination of Softening Point (Ring and Ball) on sample #20240; results in °C

lab	method	value	mark	z(targ)	remarks
154	D36	61.9		0.17	
168		-----		-----	
225	D36	56.8	R(0.01)	-6.97	
333	EN1427	61.6		-0.25	
335	EN1427	61.8		0.03	
336	EN1427	61.4		-0.53	
342	EN1427	63.7		2.69	
353	EN1427	60.0		-2.49	
357	EN1427	61.0	C	-1.09	First reported 73.8
360	EN1427	60.6		-1.65	
396	EN1427	62.0		0.31	
398	EN1427	62.6		1.15	
399	EN1427	63.2		1.99	
444	EN1427	61.3		-0.67	
447	EN1427	61.1		-0.95	
604	D36	60.8		-1.37	
657	D36	61.4		-0.53	
781	EN1427	62.4		0.87	
865	D36	62.7		1.29	
963	D36	69.0	R(0.01)	10.11	
1011	EN1427	60.4	C	-1.93	First reported 73.8
1016	EN1427	61.6		-0.25	
1026	EN1427	61.8		0.03	
1040	EN1427	60.30		-2.07	
1047	EN1427	61.0		-1.09	
1082	EN1427	61.58		-0.28	
1108	EN1427	61.4		-0.53	
1135	EN1427	62.0		0.31	
1229	EN1427	61.6		-0.25	
1259		-----		-----	
1378	D36	63.2		1.99	
1402	EN1427	62.2		0.59	
1468	EN1427	62.6		1.15	
1539	EN1427	61.6		-0.25	
1613	D36	63.4		2.27	
1631		-----		-----	
1724	EN1427	62.2		0.59	
1730	EN1427	62.1		0.45	
1741	EN1427	61.60		-0.25	
1833	EN1427	62.8		1.43	
1849	EN1427	61.0		-1.09	
1852	EN1427	62.3		0.73	
1881	EN1427	58.4	C,R(0.05)	-4.73	First reported 57.2
1944	EN1427	61.4		-0.53	
1990	D36	61.6		-0.25	
6054	D36	61.7		-0.11	
6076	EN1427	62.55		1.08	
6225	D36	61.3		-0.67	
6228	EN1427	62.65		1.22	
6229	EN1427	60.8		-1.37	

	normality	OK
	n	44
	outliers	3
	mean (n)	61.777
	st.dev. (n)	0.8393
	R(calc.)	2.350
	st.dev.(EN1427:15)	0.7143
	R(EN1427:15)	2.0
Compare		
	R(D36/D36M:14e1)	9.32	Automated electronic thermometer
	R(D36/D36M:14e1)	9.63	Mercury thermometer

Determination of Solubility in Xylene on sample #20240; results in %M/M

lab	method	value	mark	z(targ)	remarks
154	D2042	87.10		-----	
168		-----		-----	
225		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
342		-----		-----	
353		-----		-----	
357		-----		-----	
360		-----		-----	
396	EN12592	99.24		-----	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657		-----		-----	
781	EN12592	86.96		-----	
865	EN12592	90.63		-----	
963		-----		-----	
1011	EN12592	87		-----	
1016		-----		-----	
1026	EN12592	99.0		-----	
1040		-----		-----	
1047		-----		-----	
1082		-----		-----	
1108		-----		-----	
1135	EN12592	91.45		-----	
1229		-----		-----	
1259	EN12592	99.97		-----	
1378	EN12592	99.8		-----	
1402	EN12592	98.40		-----	
1468	EN12592	97.11		-----	
1539	EN12592	87.00		-----	
1613	IP47	--		-----	
1631	EN12592	87.55		-----	
1724	EN12592	86.85		-----	
1730		-----		-----	
1741	EN12592	86.600		-----	
1833		-----		-----	
1849	EN12592	87.66		-----	
1852		-----		-----	
1881		-----		-----	
1944	EN12592	88.108		-----	
1990		-----		-----	
6054		-----		-----	
6076		-----		-----	
6225		-----		-----	
6228		-----		-----	
6229	EN12592	89.20		-----	

normality	OK	
n	18	
outliers	0	
mean (n)	91.646	
st.dev. (n)	5.4745	
R(calc.)	15.329	
st.dev.(EN12592:14)	(0.0536)	
R(EN12592:14)	(0.15)	
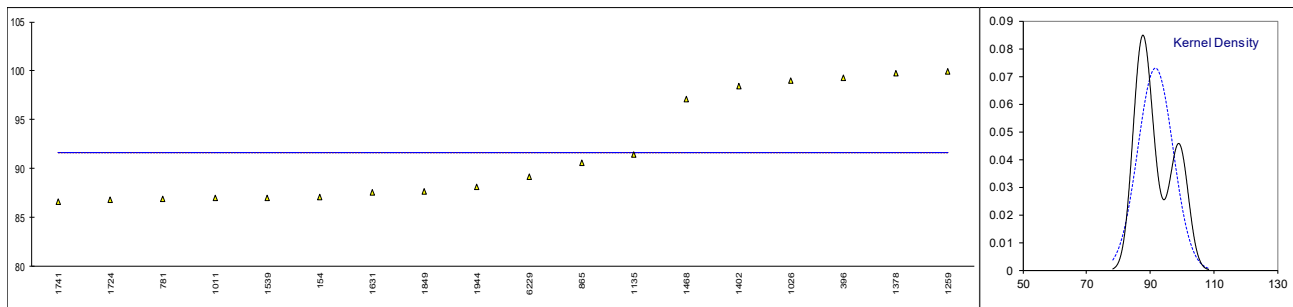
APPENDIX 2

Number of participants per country

    1 lab in AUSTRIA
    1 lab in BELGIUM
    1 lab in BULGARIA
    1 lab in CHINA, People's Republic
  2 labs in COTE D'IVOIRE
    1 lab in CROATIA
  3 labs in FINLAND
  3 labs in FRANCE
  2 labs in GERMANY
  3 labs in GREECE
    1 lab in IRELAND
  4 labs in ITALY
  2 labs in JORDAN
    1 lab in LITHUANIA
  2 labs in MALAYSIA
  4 labs in NETHERLANDS
  2 labs in POLAND
    1 lab in PORTUGAL
    1 lab in ROMANIA
    1 lab in RUSSIAN FEDERATION
    1 lab in SAUDI ARABIA
    1 lab in SERBIA
    1 lab in SINGAPORE
    1 lab in SPAIN
  4 labs in TURKEY
  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
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

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