Results of Proficiency Test Bitumen November 2014

Organised by: Institute for Interlaboratory Studies Spijkenisse, the Netherlands

Authors:ing. C.M. Nijssen-WesterCorrector:dr. R.G. VisserReport:iis14F02

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1 INTRODUCTION

Bitumen is a highly viscous, black and sticky form of petroleum. In the United States, bitumen is often referred to as asphalt. In this report we will use the word 'bitumen' for the substance that is the bottom product of the vacuum distillation step in oil refining. This bitumen can be used in road pavement as a binder for the sand and stones that build this pavement. But it is also used, among other applications for waterproofing products, like sealing of roofs and it can be a part of printing inks.

At the request of several participants, the Institute of Interlaboratory Studies decided to organise an interlaboratory study for bitumen in the 2014-2015 PT program.

In this interlaboratory study 37 laboratories in 23 different countries have participated. See appendix 2 for the number of participants per country. In this report, the results of the 2014 interlaboratory study on Bitumen are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

2 SET-UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. Sample analyses for fit-for-use and homogeneity testing were subcontracted to a third party laboratory. It was decided to send two containers of 1 litre bitumen (labelled #14260 and #14261), one is a soft bitumen, the other a hard bitumen. Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC 17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on a regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol can be downloaded from the iis website http://www.iisnl.com.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

In this proficiency test two different samples were prepared, one bitumen with a softening point around 40°C and a bitumen with a softening point around 55°C. Both bitumen samples were supplied by a third party laboratory.

The first batch is a straight-run bitumen with a softening point around 40°C. This batch was heated, filled into 44 one litre metal cans and labelled (#14260). The homogeneity of the subsamples #14260 was checked by determination of penetration (EN 1426) and softening point (EN 1427) at the beginning and the end of the straight run.

	Penetration in 0.1 mm	Softening Point in °C	
Sample #14260-1	201	39.0	
Sample #14260-2	204	39.2	

Table 1 : homogeneity test results of subsamples #14260

The differences between the two test results were calculated and compared with the repeatability of the method in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Penetration in 0.1 mm	Softening Point in °C	
Difference	3	0.2	
Reference method	EN1426:07	EN1427:07	
r (Reference method)	8	1.0	

Table 2: repeatabilities of subsamples #14260

The observed differences for Penetration and Softening Point were in full agreement with the repeatabilities of the respective reference methods. Therefore, homogeneity of the subsamples of #14260 was assumed.

The second batch is another straight-run bitumen with a softening point around 55°C. This batch was heated, filled into 44 one litre metal cans and labelled (#14261). The homogeneity of the subsamples #14261 was checked by determination of penetration (EN 1426) and softening point (EN 1427) at the beginning and the end of the straight run.

	Penetration in 0.1 mm	Softening Point in °C	
Sample #14260-1	21	59.2	
Sample #14260-2	22	59.2	

Table 3: homogeneity test results of subsamples #14261

The difference between the two test results were calculated and compared with the repeatability of the method in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Penetration in 0.1 mm	Softening Point in °C	
r (Observed)	1	0.0	
Reference method	EN1426:07	EN1427:07	
r (Reference method)	2	1.0	

Table 4: repeatabilities of subsamples #14261

The observed differences for Penetration and Softening Point were in full agreement with the repeatabilities of the respective reference methods. Therefore, homogeneity of the subsamples of #14261 was assumed.

To each of the participating laboratories a 1 litre metal can, labelled #14260 and a 1 litre metal can, labelled #14261 was sent on November 19, 2014.

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYSES

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

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

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were received. The original data are tabulated per sample in the appendix 1 of this report. The laboratories are represented by their code numbers.

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

3.1 STATISTICS

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

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

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

In accordance to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests. Outliers are marked by D(0.01) for the Dixon test and by G(0.01) or DG(0.01) for the Grubbs test and by R(0.01) for the Rosner General ESD test (see appendix 3, ref. 15). Stragglers are marked by D(0.05) for the Dixon test and by G(0.05) or DG(0.05) for the Grubbs test and by R(0.05) for the Rosner General ESD test. Both outliers and stragglers were not included in the calculations of the averages and the standard deviations.

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

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

3.2 GRAPHICS

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

3.3 Z-SCORES

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

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

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

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

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

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

4 EVALUATION

In this proficiency test some problems were encountered with the despatch of the samples. Laboratories in Jordan and South Africa received the samples late. One participant did not report any test results and six laboratories reported the test results after the final reporting date. Not all laboratories were able to report all analyses requested. Finally, 36 participants reported in total 464 numerical results. Observed were 18 statistically outlying results, which is 3.9%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

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

4.1 EVALUATION PER SAMPLE AND PER TEST

In this section, the results are discussed per sample and per test. The methods, which are used by the various laboratories, are taken into account for explaining the observed differences where possible and applicable. These methods are also in the tables together with the reported data. The abbreviations, used in these tables, are listed in appendix 3. In the iis PT reports, ASTM methods are referred to with a number (e.g. D2086) and an added designation for the year that the method was adopted or revised (e.g. D2086-08). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D2086-08 (2013)). In the results tables of Appendix 1 only the method number and year of adoption or revision will be used.

Sample #14260:

<u>Density:</u> This determination was somewhat problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is almost in agreement with the requirements of EN15326:2007.

Two laboratories reported the density at a higher temperature of 100 or 120°C. Unfortunately these laboratories did not convert this density to 25°C using the table from ASTM D4311/4311M.

Dynamic Shear Rheometer: Two properties were tested: Shear Modulus G* and Phase Angle. Only 3 participants reported results for these tests.

The test results for the determination of Modulus G* were very different and did not meet the requirements of EN11770:2012.

The test results for the determination of the Phase Angle were close together and did meet the requirements of EN11770:2012.

<u>Dynamic Viscosity:</u>This determination may be problematic. Two statistical outliers were observed and two test results were excluded. However, the calculated reproducibility after rejection of suspect data is just in agreement with the requirements of EN12596:2014.

Two participants used test method ASTM D4402, which is a rotating viscometer, not a capillary. Therefore these two test results were excluded.

<u>Flash Point COC:</u> This determination was problematic. No statistical outlier was observed. However, the calculated reproducibility is not in agreement with the requirements of ISO2592:2000. A possible cause for the large spread could be that both manual

determinations and automated equipment was used.

 <u>Fraass Breaking Point:</u> This determination was problematic. No statistical outlier was observed. However, the calculated reproducibility is not in agreement with the requirements of EN12593:2007. A possible cause for the large spread could be that both manual determinations and automated equipment was used.

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

<u>Penetration:</u> This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the strict requirements of EN1426:2007. However, the calculated reproducibility is in good agreement with the requirements of ASTM D5/5M:2013.

When the test results of test method EN1426 (n=23) are evaluated separately, the spread remains large. When the same is done for the test results of ASTM D5/5M (n=6), the spread is smaller by a factor of three.

Different factors could cause this large spread, such as preparation, temperature and needle.

During 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.

<u>Penetration Index</u>: This determination was very problematic. No statistical outliers were observed. However the calculated reproducibility is not at all in agreement with the strict requirements of EN12591:2009.

The Penetration Index was calculated by iis for all the laboratories, which reported Penetration and Softening Point. Six out of the twelve reported test results for Penetration Index appeared to have an error in calculation. The values for Penetration Index, which were calculated by iis, were also statistically evaluated. The calculated reproducibility was still very large compared to the requirements of EN12591:2009. Since the calculated reproducibilities for both Penetration and Softening Point are not meeting the requirements for the respective test methods, it can be expected that the spread in the (calculated) Penetration Index is large too.

RTFOT:This is a Rolling Thin Film Oven Test (RTFOT, EN12607-1 or ASTM D2872).It is a bitumen ageing test, in which rotating glass vessels with a coating of
bitumen is used. Unfortunately four participants performed a Thin Film Oven
Test (TFOT, EN12607-2 or ASTM D1754), which is a test in which the
bitumen is poured on a dish and is aged like this. This last test is not
equivalent to RTFOT. Therefore these four TFOT results were excluded from
the statistical evaluation.

Four properties were determined after the RTFOT ageing test: Change of Mass, Retained Penetration, Viscosity Ratio and Increase in Softening Point.

This determination on Change of Mass may not be problematic. Two statistical outliers were observed and four test results were excluded. However the calculated reproducibility after rejection of the suspect data is in good agreement with the requirements of EN12607-1:2014.

This determination on Retained Penetration is problematic. One statistical outlier was observed and three test results were excluded. The calculated reproducibility after rejection of the suspect data is in not agreement with the requirements of EN12607-1:2014.

This determination on Viscosity Ratio may not to be problematic. However, only four participants reported results. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of EN12607-1:2014.

This determination on Increase in Softening Point was not problematic. No outliers were observed. Three test results were excluded. The calculated

reproducibility after rejection of the suspect data is in good agreement with the requirements of EN12607-1:2014.

Softening Point: This determination was very problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements of EN1427:2007.
 Looking at the Kernel Density graph, a trimodal distribution appears to be present. When these three different groups (n=26 and softening point of 39.2, n=6 and softening point of 42.9 and n=2 and softening point of 45.6), the calculated reproducibilities are still not in agreement with the requirements of EN1427:2007, but have significantly improved.
 The large spread may be caused by preparation of the sample surface, the start temperature and the exact heating rate. Higher test results can be

start temperature and the exact heating rate. Higher test results can be caused by not removing the excess bitumen after filling the ring. The surface might harden during cooling, removing the excess bitumen will create a 'fresh' surface. Furthermore, in the test method start temperature and heating rate are very well defined, not following will create more spread as well. Also stirring of the waterbath can be an issue. ASTM D36 does not use stirring, while EN 1427 uses a stirring of the waterbath during measurement.

<u>Solubility in Xylene:</u> This determination was not problematic. Two statistical outliers were observed and one test result was excluded. The calculated reproducibility after rejection of the suspect data is in agreement with the requirements of EN12592:2014.

One participant reported to have used ASTM D4042, which is solubility in trichloroethylene. This test result was excluded.

Sample #14261:

Density: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 EN15326:2007.
One laboratory reported the density at a higher temperature of 120°C.
Unfortunately this laboratory did not convert this density to 25°C using the
table from ASTM D4311/4311M.

Dynamic Shear Rheometer: Two properties were tested: Shear Modulus G* and Phase Angle. Only 2 participants reported results for these tests. The two test results for the determination of Modulus G* were different in value, but the difference was within the requirements of EN11770:2012. The two test results for the determination of the Phase Angle were close together and this difference was well within the requirements of EN11770:2012.

<u>Dynamic Viscosity</u>: This determination was not problematic. One statistical outlier was observed and one test result was excluded. The calculated reproducibility after rejection of suspect data is in good agreement with the requirements of EN12596:2014. One participant used test method ASTM D4402, which is a rotating viscometer, not a capillary. Therefore this test result was excluded.

<u>Flash Point COC:</u> This determination was very problematic. No statistical outlier was observed. However, the calculated reproducibility is not at all in agreement with the requirements of ISO2592:2000. A possible cause for the large spread could be that both manual determinations and automated equipment was used.

<u>Fraass Breaking Point:</u> This determination was very problematic. No statistical outlier was observed. However, the calculated reproducibility is not at all in agreement with the requirements of EN12593:2007. A possible cause for the large spread could be that both manual determinations and automated equipment was used

<u>Kinematic Viscosity:</u> This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN12595:2014 or the requirements of ASTM D2170/2170M:2010.

<u>Penetration:</u> This determination was very problematic. No statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the strict requirements of EN1426:2007 and even not in agreement with the less strict requirements of ASTM D5/5M:2013.

When the test results of test method EN1426 (n=23) are evaluated separately, the spread remains large. When the same is done for the test results of ASTM D5/5M (n=8), the spread also remains large.

Possible causes for the large spread have already been explained in the discussion of the penetration of sample #14260. Additionnally for this harder bitumen: when the bitumen is poured in a cup, air bubbles will remain trapped just below the surface. When the penetration needle hits such a bubble, it will not measure penetration of the bitumen, but penetration of the bubble. With heat (like a flame) the bubbles can be removed.

<u>Penetration Index</u>: This determination may not be problematic. One statistical outlier was observed. The calculated reproducibility is in agreement with the strict requirements of EN12591:2009. The test result of one laboratory was excluded, because the Softening Point test result of this laboratory was an outlier.

The Penetration Index was calculated by iis for all the laboratories, which reported Penetration and Softening Point. Three out of the eleven reported test results for Penetration Index appeared to have an error in calculation. The values for Penetration Index, which were calculated by iis, were also statistically evaluated. The calculated reproducibility was larger and not at all in agreement with the strict requirements of EN12591:2009. Since the calculated reproducibilities for both Penetration and Softening Point are not

meeting the requirements for the respective test methods, it can be expected that the spread in the (calculated) Penetration Index is large too.

RTFOT:This is a Rolling Thin Film Oven Test (RTFOT, EN12607-1 or ASTM D2872).It is a bitumen ageing test, in which rotating glass vessels with a coating of
bitumen is used. Unfortunately four participants performed a Thin Film Oven
Test (TFOT, EN12607-2 or ASTM D1754), which is a test in which the
bitumen is poured on a dish and is aged like this. This last test is not
equivalent to RTFOT. Therefore these four TFOT results were excluded from
the statistical evaluation.

Four properties were determined after the RTFOT ageing test: Change of Mass, Retained Penetration, Viscosity Ratio and Increase in Softening Point.

This determination on Change of Mass may not be problematic. No statistical outliers were observed and four test results were excluded. The calculated reproducibility after rejection of the suspect data is in good agreement with the requirements of EN12607-1:2014.

This determination on Retained Penetration is problematic. One statistical outlier was observed and three test results were excluded. The calculated reproducibility after rejection of the suspect data is in not agreement with the requirements of EN12607-1:2014.

This determination on Viscosity Ratio could not be evaluated, for only two participants reported a test result. The difference of the two results was not within the requirements of EN12607-1:2014.

This determination on Increase in Softening Point was not problematic. No outliers were observed. Two test results were excluded. The calculated reproducibility after rejection of the suspect data is in good agreement with the requirements of EN12607-1:2014.

<u>Softening Point:</u> 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 EN1427:2007. Possible causes for the large spread have already been explained in the discussion of the softening point of sample #14260.

<u>Solubility in Xylene:</u> This determination was not problematic. One statistical outlier was observed and one test result was excluded. The calculated reproducibility after rejection of the suspect data is in agreement with the requirements of EN12592:2014. One participant reported to have used ASTM D4042, which is solubility in trichloroethylene. This test result was excluded.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Parameters #14260	Unit	n	average	2.8 * sd	R (lit)
Density at 25°C	kg/m ³	27	1022.2	5.6	5.0
Dynamic Shear Rheometer, at 60°	C and 1.59 H	Z			
- Shear Mod. G*	Pa	3	618.1	(408.3)	278.1
- Phase Angle	0	3	88.1	(1.4)	9.0
Dynamic Viscosity at 60°C	Pa.s	10	49.1	5.4	4.9
Flash Point COC	°C	19	331	27	17
Fraass Breaking Point	°C	14	-20	10	6
Kinematic Viscosity at 135°C	mm²/s	14	232.3	31.6	13.9
Penetration at 25°C	0.1 mm	33	187.8	22.3	11.3
Penetration Index		12	-0.63	1.74	0.50
RTFOT at 163°C					
- Change of Mass	%	14	0.078	0.070	0.200
- Retained Penetration	%	14	63.3	11.7	10.0
- Viscosity Ratio		3	1.91	(0.09)	0.38
- Increase in Softening Point	°C	13	4.0	1.9	2.0
Softening Point (Ring and Ball)	°C	34	40.2	6.1	2.0
Solubility in Xylene	% M/M	12	99.942	0.160	0.150

Table 5: summary of test results on Fuel Oil sample #14260

Parameters #14261	Unit	n	average	2.8 * sd	R (lit)
Density at 25°C	kg/m ³	25	1048.4	6.5	5.0
Dynamic Shear Rheometer, at 60°	C and 1.59 H	Z			
- Shear Mod. G*	Ра	2	12520	n.a.	5634
- Phase Angle	o	2	85.1	n.a.	9.0
Dynamic Viscosity at 60°C	Pa.s	9	1407.4	130.5	168.9
Flash Point COC	°C	17	358	36	17
Fraass Breaking Point	°C	11	-3	18	6
Kinematic Viscosity at 135°C	mm²/s	13	1024.2	99.7	93.8
Penetration at 25°C	0.1 mm	34	22.0	10.3	3.0
Penetration Index		9	-1.03	0.40	0.50
RTFOT at 163°C					
- Change of Mass	%	12	0.066	0.079	0.200
- Retained Penetration	%	9	74.3	13.9	10.0
- Viscosity Ratio		2	1.4	n.a.	0.3
- Increase in Softening Point	°C	10	4.7	1.2	2.0
Softening Point (Ring and Ball)	°C	30	58.7	5.2	2.0
Solubility in Xylene	% M/M	11	99.960	0.126	0.150

Table 6: summary of test results on Fuel Oil sample #14261

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

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

Parameters	#14260	#14261
Density at 25°C	+/-	-
Dyn. Shear Rheometer Shear Mod. G*	()	n.e
Dyn. Shear Rheometer Phase Angle	(++)	n.e.
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.
RTFOT at 163°C Increase in Soft. Point	+/-	+
Softening Point (Ring and Ball)		
Solubility in Xylene	+/-	+

 Table 7: comparison determinations against the standard

results between brackets should used with care, because the amount of reported values was 3 or less.

The following performance categories were used:

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

4.3 DISCUSSION

The spread of both penetration and softening point after the RTFOT test (for example for #14260 R_{softening point}: 1.9°C) is much smaller than the spread in the penetration and softening point before RTFOT (for example for #14260 R_{softening point}: 6.1°C). The reason for this could be that the penetration and softening point after RTFOT are expressed as a result in ratio for the penetration and a result in difference for the softening point. If a systematic error is present, this may be hidden when a difference value or a ratio of two measurements is calculated. When a significant systematic error is present, a laboratory will find low values on both samples or high values on both samples. When this happens, a scatter diagram of all test results will show a linear relation with an angle of 45°. When the values are randomly scattered, the distribution is more random and a systematic error is less likely to be the issue.

To investigate whether the test results of both penetration and softening point show systematic errors, the results of the tests concerned of both samples were plotted in a scatter diagram (see figure 1 and 2).





Figure 1: Penetration #14260 versus Penetration #14261

Figure 2: Softening Point #14260 versus Softening Point #14261

As can be seen from figure 1, the penetration results are indeed scattered. No linear relation with an angle of 45° is observed between the test results of both samples. It should be noted that the observed spread is of the penetration test (see section 4.2) is relatively large and therefore may hide a possible systematic error. So if the spread should get smaller in future PTs, systematic errors may get visible than.

However, the graph of the softening point clearly shows a linear relation. So when the test result for #14260 is lower or higher, the test result of #14261 is also lower or higher for that laboratory. This means that laboratories within this group may have a systematic error in the softening point test. As explained in section 4.1, the reasons for this could be preparation of the sample (surface), start temperature and heating rate and stirring or not of the waterbath.

APPENDIX 1

	Determination of Densit	v at 25°C on same	ple #14260: results in k	a/m ³
--	-------------------------	-------------------	--------------------------	------------------

lab method value mark z(targ)	remarks
154 EN15326 1021.4 C -0.42	First reported: 6.7
168 D70 1023.2 C 0.59	Reported 1.0232 (probably a unit error)
225 1026 C 2.15	First reported: 1033
332 EN15326 1024 C 1.03	First reported: 1.024
333	
335 EN15326 1023.0 C 0.47	First reported: 1.023
336 EN15326 1025 1.59	
337 EN15326 1025.0 1.59	
353	
357 EN15326 1020.1 -1.15	
360 ISO3838 1018.64 -1.97	
398 EN15326 1022 -0.09	
399 D70 1021 -0.65	
440	
444 D70 1023.5 0.75	
445 D70 1019.6 -1.43	
604 D70 1020.7 -0.81	
657 D70 1021 -0.65	
1016 EN15326 1021.25 -0.51	
1026 EN15326 1020.7 -0.81	
1040	Density measured at 100°C acc. to EN12185, reported: 973.2
1082 EN15326 1019.9 -1.26	
1229 EN15326 1022 -0.09	
1340 EN15326 1022 -0.09	
1300 ID70 1021 0 D(0.01) 4.05	
1469 EN15226 1022.0 K(0.01) 4.95	
1612 INTUS20 1023.0 0.47	Density at 15°C reported: 1026 5, measured at 120°C: 062.60
1631	Density at 15 C reported. 1020.5, measured at 120 C. 902.09
1710 ISO3838 1021 C -0.65	First reported: 1030
1717 ISO3838 1026 2.15	
1810 EN15326 1020 -0.93	
1842 IP365 1025.1 1.65	
1849	
1884 D70 1021 C -0.65	First reported: 1.021
1970 EN15326 1021.6 -0.31	· · · · · · · · · · · · · · · · · · ·
normality OK	
n 27	
outliers 1	
mean (n) 1022.16	
st.dev. (n) 2.004	
R(calc.) 5.61	





Determination of Shear Mod G* at 60°C, 1.59 Hz on sample #14260; results in Pa

lab	method	value	mark	z(targ)	remarks	
154						
168						
225						
332						
333						
336						
337						
353						
357						
360						
398						
399						
440						
444 445						
604						
657						
1011						
1016						
1026	EN14770	484.55		-1.34		
1040						
1082	EN14770	596		-0.22		
1229						
1385						
1399						
1468	EN14770	773.7		1.57		
1613						
1631						
1710						
1810						
1842						
1849						
1884						
1970						
	normality	unknown				
	n	3				
	outliers	0				
	st dev (n)	(145 834)				
	R(calc.)	(408.34)				
	R(EN14770:12)	278.14				
	. ,					
¹⁰⁰⁰ T						
900 -						-
800 -						Δ
700 -						_
600 -					Δ	
500		Δ				
400						-
300 -	Ę	1026			1082	1468

Determination of Phase Angle at 60°C, 1.59 Hz on sample #14260; results in °

lab	method	value	mark	z(targ)	remarks	
154						
168						
225						
332						
333						
336						
337						
353						
357						
360						
398						
399						
440						
445						
604						
657						
1011						
1016	EN4 4770					
1026	EN14770	87.88		-0.08		
1040	EN14770	88.7		0.17		
1229						
1340						
1385						
1399						
1468	EN14770	87.85		-0.09		
1613						
1710						
1717						
1810						
1842						
1849						
1884						
1970						
	normality	unknown				
	n	3				
	outliers	0				
	mean (n)	88.14				
	St.dev. (n)	(0.482)				
	R(EN14770.12)	9.00				
	(LITI 1170.12)	0.00				
¹⁰⁰ T						
	-					
95 -						
90 -						
	Δ	•			Δ	_▲
85 -						
	-					
80 -						
	-					
75	~				ø	N
	1465	-			05	801

Determination of Dynamic Viscosity at 60°C on sample #14260; results in Pa.s

154 EN12596 469.1 C,G(0.01) 293.69 First reported: 451 225	lab	method	value	mark	z(targ)	remarks
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$	154	EN12596	469.1	C,G(0.01)	239.69	First reported: 451
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	168	D2171	47.84	-,-()	-0.70	
332 333 336 337 338 337 338 339 EN12596 50.28 0.09 360 399 EN12596 52.85 2.16 399 604 604 604 604 604 604 604 604 604 604 1016 EN12596 48.75 0.18 10220 EN12596 47.7 -0.72 1380 D-402 50.3 ex 0.70 1399 D4402 50.4 ex 3.61 1613 D402 50.4 ex 3.61 1849	225					
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	332					
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	333					
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	335					
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	336					
$ \begin{array}{c ccccccccccccccccccccccccccccccccccc$	337					
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	353					
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	357	EN12596	50.28		0.69	
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	360					
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	398	EN12596	52.85		2.16	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	399					
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	440					
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	444					
	445					
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	604					
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	657	D2171	47.8		-0.72	
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	1011					
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	1016	EN12596	48.75		-0.18	
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	1026	EN12596	58.05	G(0.05)	5.13	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	1040	EN12596	48.56	0(0100)	-0.29	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	1082	EN12596	47.14		-1.10	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	1229	EN12596	47.7		-0.78	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	1340	EN12596	48.03		-0.59	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	1385	2.1.2000				
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	1399	D4402	50.3	ex	0.70	Result excluded, see §4.1
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	1468	EN12596	51.72	0,1	1.51	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	1613	D4402	55.4	ex	3.61	Result excluded, see §4.1
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	1631	2 2		en		
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	1710					
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	1717					
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	1810					
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	1842					
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	1849					
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	1884					
normality suspect n 10 outliers 2 (+2ex) mean (n) 49.07 st.dev. (n) 1.913 R(calc.) 5.36 R(EN12596:14) 4.91 $rac{60}{58}$	1970					
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$						
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$		normality	suspect			
outliers 2 (+2ex) mean (n) 49.07 st.dev. (n) 1.913 R(calc.) 5.36 R(EN12596:14) 4.91 60		n	10			
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		outliers	2 (+2ex)			
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		mean (n)	49.07			
R(calc.) 5.36 R(EN12596:14) 4.91		st.dev. (n)	1.913			
R(EN12596:14) 4.91		R(calc.)	5.36			
60 58 - ×		R(EN12596:14)	4.91			
60 58 - ×						
58 - ×	⁶⁰ T					
	58 -					ж





Determination of Flash Point COC on sample #14260; results in °C

lab	method	value	mark	z(targ)	remarks
154	ISO2592	326	С	-0.85	First reported 605
168	D92	329.4		-0.29	•
225	D92	340		1.45	
332	-				
333	ISO2592	354		3.76	
335	·				
336					
337					
353					
357	ISO2592	326		-0.85	
360	ISO2592	336		0.80	
398	ISO2592	326		-0.85	
399					
440					
444					
445	IP36	331.7		0.09	
604	D92	324		-1.18	
657	D92	330		-0.19	
1011					
1016	ISO2592	330.1		-0.18	
1026	ISO2592	325		-1.02	
1040					
1082	ISO2592	330		-0.19	
1229	ISO2592	324		-1.18	
1340	ISO2592	338		1.13	
1385	EN22592	310		-3.49	
1399					
1468	ISO2592	326		-0.85	
1613					
1631					
1710	ISO2592	344		2.11	
1717	1802592	342		1.78	
1810					
1842					
1849	D02				
1084	D92	>250			
1970					
	normality	cuencet			
	normality	suspeci 10			
	outlions	19			
	mean (n)	0			
	st day (n)	0.50			
	B(calc)	9.09 26.8			
	R(ISO2502.00)	20.0 17 0			Compare $R(D92.12h) = 18.0$
	1(1302382.00)	17.0			000000000000000000000000000000000000
260 -					
300					
350					Δ
					A
340 -					. <u>A</u>
					Δ Δ
330					Δ Δ Δ Δ
	Δ Δ	Δ Δ	Δ Δ	Δ	
320					
-					
310 - 🔺					
200					
1300					



Determination of Fraass Breaking point on sample #14260; results in °C

lab	method	value	mark	z(targ)	remarks
154					
168					
225					
332					
333	EN12593	-19		0.25	
335					
336					
337					
353	EN12502	21		0.69	
360	EN12593	-21		2.82	
398	EN12593	-17		1.18	
399					
440					
444					
445					
604					
657					
1011					
1016	EN12593	-22		-1.15	
1026	EN12593	-24		-2.08	
1040	EN12503			-1 15	
1229	EN12593	-22		-1.13	
1340	EN12593	-15		2.12	
1385					
1399					
1468	EN12593	-24.5		-2.32	
1613					
1631					
1710	EN12593	-21		-0.68	
1/1/	EN12593	-17.0		1.18	
18/2	EIN12093	-19		0.25	
1849					
1884					
1970	EN12593	-15.5		1.88	
	normality	OK			
	n	14			
	outliers	0			
	mean (n)	-19.5			
	st.dev. (n)	3.51			
	R(Calc.) R(EN12503:07)	9.0 6.0			
	R(EN12393.07)	0.0			
-10 T					
-12 -					
-14 -					۵
-16 -					Δ
-18 -					Δ Δ
-10					Δ Δ
-20				۵	۵
-22		Δ	Δ Δ		





Determination of Kinematic Viscosity at 135°C on sample #14260; results in mm²/s

lab	method	value		mark	z(targ)	remarl	ks						
154	EN12595	224.4			-1.58								
168													
225													
332													
333													
335													
336													
337													
353													
357	EN12595	225.8			-1.30								
360													
398	EN12595	232.1			-0.03								
399													
440													
444													
445	D2170	234.6			0.47								
604													
657	D2170	206.55		С	-5.16	First re	ported: 2	205.84					
1011													
1016	EN12595	230.5			-0.35								
1026	EN12595	223.11			-1.84								
1040													
1082	EN12595	244.8			2.52								
1229	EN12595	233			0.15								
1340	EN12595	227.6			-0.94								
1385													
1399													
1468	EN12595	251.7			3.91								
1613													
1631													
1710	EN12595	235.7			0.69								
1717	EN12595	234.6			0.47								
1810	EN12595	247.1			2.98								
1842													
1849													
1884													
1970													
	normality	suspect											
	n	14											
	outliers	0											
	mean (n)	232.25											
	st.dev. (n)	11.272											
	R(Calc.)	31.00				Compo		470/0470	(1.10) 0(
	R(EN12595.14)	13.94				Compa	are R(D2	170/2170	v(1, 10) = 20	J.44			
260 -													
200													
250 -													Δ
											Λ	Δ	
240 -													
								Δ	Δ	Δ			
230 -					۵	Δ	Δ						
	•	Δ	Δ	Δ									
220 -	A												
210 -	•												
	<u>A</u>												
200	657)26	154	357	340	016	398	229	445	717	710	382	310	468
	÷			.	-		-	-	-	.	÷	÷	÷



Determination of Penetration at 25°C on sample #14260; results in 0.1 mm

154 EN1426 195 1.78 168 D5 189 0.29 225 D5 194 1.53 332 EN1426 188.8 0.24 333 EN1426 195 1.78 335 336 EN1426 191 0.79 337 EN1426 189 0.29 360 EN1426 189 0.29 360 EN1426 189 0.29 380 EN1426 189 0.29 380 EN1426 189 0.29 380 EN1426 189.66 0.45 399 D5 185 -0.70 444 IP49 189.2 0.34 444 IP49 189.2 0.34 444 IP49 189.2 0.34 657 D5 192 1.03 1010 EN1426 193 -2.44 1026 EN1426 192 1.03 1040 EN1426 191 0	lab	method	value	mark	z(targ)	remarks			
168D51890.29225D51941.53332EN1426188.80.24333EN14261951.78335336EN14261910.79337EN14261992.77353EN14261890.29360EN1426173-3.69389EN1426189.660.45399D5185-0.70440IP491900.54444IP491900.54604D51931.28657D51921.0310111016EN1426178-2.441026EN14261951.781040EN14261921.03105151.781040EN14261921.0310511921.031052EN1426187-0.211046EN14261910.791345EN1426183.3-1.131613D5158R(0.05)-7.411631EN1426189C-4.681770EN14261982.241345EN14261880.041345EN14261880.041346178.8-2.241310EN14261880.041345EN14261880.041342IP49173-3.69 <td>154</td> <td>EN1426</td> <td>195</td> <td></td> <td>1.78</td> <td></td> <td></td> <td></td> <td></td>	154	EN1426	195		1.78				
225D51941.53332EN1426188.80.24333EN14261951.78335336EN14261910.79337EN14261951.78353EN14261951.78354EN14261890.29360EN1426189.660.45398EN1426189.660.45399D5185-0.70444IP49189.20.34445EN14261931.28667D51921.0310111016EN14261951.781040EN14261921.0310511.28-2.441026EN14261951.781039D51921.0310411052EN14261951163D5152129EN14261871390D51911390D519113910.791385EN14261531EN14261521631EN142615421531710EN142615511581613D5158R(0.05)1710EN14261631EN14261642178.1717EN14261731-3.69	168	D5	189		0.29				
332EN1426188.8 0.24 333EN1426195 1.78 336EN1426191 0.79 337EN1426199 2.77 333EN1426195 1.78 357EN1426189 0.29 360EN1426173 -3.69 388EN1426189.66 0.45 399D5185 -0.70 440IP49189.2 0.34 444IP49189.2 0.34 445EN1426193 1.28 657D5192 1.03 10111026EN1426195 1.78 1026EN1426192 1.03 10111022EN1426195 1.78 1031D5193 1.28 657D5192 1.03 1014115EN1426196 2.03 1229EN1426187 -0.21 1340EN1426191 0.79 1385EN1426183.3 -1.13 1613D5158R(0.05) -7.41 1631EN1426169C -4.68 1717EN1426178.8 -2.24 1810D5158R(0.05) -7.41 1831EN1426178.8 -2.24 1810EN1426188 0.04 1842IP49173 -3.69 </td <td>225</td> <td>D5</td> <td>194</td> <td></td> <td>1.53</td> <td></td> <td></td> <td></td> <td></td>	225	D5	194		1.53				
333EN1426195 1.78 335336EN1426199 2.77 333EN1426199 2.77 333EN1426195 1.78 357EN1426189 0.29 360EN1426189.66 0.45 399D5185 -0.70 440IP49189.2 0.34 444IP49189.2 0.34 445EN1426193 1.28 607D5193 1.28 677D5192 1.03 1016EN1426178 -2.44 1026EN1426195 1.78 10141016EN1426195 1.78 1026EN1426195 1.78 1030EN1426195 1.78 1040EN1426195 1.78 1040EN1426196 2.03 1229EN1426187 -0.21 1385EN1426152R(0.05)1399D5191 0.79 1385EN1426158R(0.05)1399D5191 0.79 1385EN1426188.3 -1.13 1613D5158R(0.05)1710EN1426188 -2.24 1810EN1426188 0.04 1842IP49173 -3.69	332	EN1426	188.8		0.24				
335 336 EN1426 191 0.79 337 EN1426 199 2.77 333 EN1426 195 1.78 357 EN1426 189 0.29 360 EN1426 173 -3.69 388 EN1426 189.66 0.45 399 D5 185 -0.70 440 IP49 190 0.54 444 IP49 189.2 0.34 445 EN1426 190 0.54 604 D5 193 1.28 657 D5 192 1.03 1011 1026 EN1426 195 1.78 1040 EN1426 192 1.03 1042 EN1426 191 0.79 1385 EN1426 187 -0.21 1340 EN1426 181 0.79 1385 EN1426 152 R(0.05) -7.41 1613 D5	333	EN1426	195		1.78				
336EN1426191 0.79 337EN1426199 2.77 353EN1426195 1.78 357EN1426189 0.29 360EN1426173 -3.69 398EN1426189.66 0.45 399D5185 -0.70 440IP49190 0.54 444IP49189.2 0.34 445EN1426190 0.54 604D5193 1.28 657D5192 1.03 10111016EN1426178 -2.44 1026EN1426195 1.78 1040EN1426196 2.03 1229EN1426196 2.03 1230EN1426196 2.03 1340EN1426152R(0.05)1399D5191 0.79 1385EN1426152R(0.05)1399D5191 0.79 1385EN1426183.3 -1.13 1613D5158R(0.05)1710EN1426198 2.53 1717EN1426178.8 -2.24 1810EN1426188 0.04 1842IP49173 -3.69	335	2111120							
337EN1426195 1.78 353 EN1426195 1.78 357 EN1426189 0.29 360 EN1426173 -3.69 398 EN1426189.66 0.45 399 D5185 -0.70 440 IP49190 0.54 444 IP49189.2 0.34 445 EN1426190 0.54 604 D5193 1.28 657 D5192 1.03 1011 1016 EN1426178 -2.44 1026 EN1426195 1.78 1040 EN1426195 1.78 1040 EN1426195 1.78 1040 EN1426196 2.03 1229 EN1426197 -0.21 1340 EN1426191 0.79 1385 EN1426152R(0.05) 1399 D5191 0.79 1385 EN1426183.3 -1.13 1613 D5158R(0.05) 1710 EN1426198 2.53 1717 EN1426178.8 -2.24 1810 EN1426188 0.04 1842 IP49173 -3.69	336	EN1426	191		0 79				
353 $E11426$ 195 1.78 357 $EN1426$ 189 0.29 360 $EN1426$ 173 -3.69 398 $EN1426$ 189.66 0.45 399 $D5$ 185 -0.70 440 $IP49$ 190 0.54 444 $IP49$ 189.2 0.34 445 $EN1426$ 190 0.54 604 $D5$ 193 1.28 657 $D5$ 192 1.03 1011 $$ $$ 1016 $EN1426$ 195 1.78 1026 $EN1426$ 195 1.78 1040 $EN1426$ 195 1.78 1042 $EN1426$ 195 1.78 1042 $EN1426$ 195 1.78 1042 $EN1426$ 196 2.03 1229 $EN1426$ 197 -0.21 1340 $EN1426$ 191 0.79 1385 $EN1426$ 152 $R(0.05)$ 7.11 0.79 1.13 1613 $D5$ 158 $R(0.05)$ 7.14 1631 $EN1426$ 183.3 113 1613 $D5$ 158 $R(0.05)$ 7.17 $EN1426$ 178.8 -2.24 110 $EN1426$ 188 0.04 1242 198 2.53 1717 $EN1426$ 178.8 -2.24 1842 173 -3.69	337	EN1426	100		2 77				
357EN1426189 0.29 360EN1426173 -3.69 398EN1426189.66 0.45 399D5185 -0.70 440IP49190 0.54 444IP49189.2 0.34 445EN1426190 0.54 604D5193 1.28 657D5192 1.03 10111016EN1426195 1.78 1040EN1426195 1.78 1042EN1426196 2.03 1229EN1426187 -0.21 1340EN1426191 0.79 1385EN1426152 $R(0.05)$ -7.41 1631D5158 $R(0.05)$ -7.41 1631EN1426198 2.53 1717EN1426178.8 -2.24 1810EN1426198 2.53 1717EN1426178.8 -2.24 1810EN1426198 2.53	353	EN1426	195		1 78				
360EN1426173 -3.69 398EN1426189.66 0.45 399D5185 -0.70 440IP49190 0.54 444IP49189.2 0.34 445EN1426190 0.54 604D5193 1.28 657D5192 1.03 10111016EN1426178 -2.44 1026EN1426195 1.78 1040EN1426192 1.03 1022EN1426196 2.03 1229EN1426196 2.03 1229EN1426196 2.03 1385EN1426152 $R(0.05)$ 7.411631D51581631D5158 $R(0.05)$ 1710EN1426169 C 468EN1426178.1717EN1426178.172EN1426178.173 -3.69	357	EN1426	180		0.29				
300 1132 103 103 398 $EN1426$ 189.66 0.45 399 $D5$ 185 -0.70 440 $IP49$ 190 0.54 444 $IP49$ 189.2 0.34 445 $EN1426$ 190 0.54 604 $D5$ 193 1.28 657 $D5$ 192 1.03 1011 1016 $EN1426$ 178 -2.44 1026 $EN1426$ 195 1.78 1040 $EN1426$ 192 1.03 1082 $EN1426$ 196 2.03 1229 $EN1426$ 196 2.03 1239 $EN1426$ 197 -0.21 1340 $EN1426$ 152 $R(0.05)$ 8.90 1.33 -1.13 1613 $D5$ 158 $R(0.05)$ 7.41 169 C 1631 $EN1426$ 198 2.53 171 $EN1426$ 171 $EN1426$ 178.8 2.53 171 173 1842 173 -3.69	360	EN1426	173		-3.60				
399D5185 0.43 440IP49190 0.54 444IP49189.2 0.34 445EN1426190 0.54 604D5193 1.28 657D5192 1.03 10111016EN1426178 -2.44 1026EN1426195 1.78 1040EN1426196 2.03 1229EN1426196 2.03 1229EN1426197 0.79 1385EN1426152R(0.05)1399D5191 0.79 1386EN1426183.3 -1.13 1613D5158R(0.05)1710EN1426198 2.53 1717EN1426178.8 -2.24 1810EN1426188 0.04	200	EN1420	170 66		-3.09				
359 440D3 -0.70 440IP49190 0.54 444IP49189.2 0.34 445EN1426190 0.54 604D5193 1.28 657D5192 1.03 10111016EN1426195 1.78 1026EN1426195 1.78 1040EN1426196 2.03 1229EN1426187 0.21 1340EN1426191 0.79 1385EN1426152R(0.05)1399D5191 0.79 1361D5158R(0.05)1631EN1426198 2.53 1710EN1426198 2.53 1717EN1426178.8 -2.24 1842IP49173 -3.69	200	D5	109.00		0.45				
440 1749 190 0.54 444 $1P49$ 189.2 0.34 445 $EN1426$ 190 0.54 604 $D5$ 193 1.28 657 $D5$ 192 1.03 10111016 $EN1426$ 178 -2.44 1026 $EN1426$ 195 1.78 1040 $EN1426$ 192 1.03 1082 $EN1426$ 196 2.03 1229 $EN1426$ 196 2.03 1229 $EN1426$ 191 0.79 1385 $EN1426$ 152 $R(0.05)$ -8.90 1399 $D5$ 191 0.79 1368 $EN1426$ 169 C -4.68 First reported 159 1710 $EN1426$ 198 2.53 1710 $EN1426$ 178.8 -2.24 1810 $EN1426$ 188 0.04	399		100		-0.70				
444 1749 189.2 0.34 445EN1426190 0.54 604D51931.28657D51921.0310111016EN1426178-2.441026EN14261951.781040EN14261921.031082EN14261962.031229EN14261910.791385EN14261910.791385EN1426152R(0.05)-8.901399D51910.791468EN1426183.3-1.131613D5158R(0.05)-7.411631EN14261982.531710EN1426178.8-2.241810EN14261880.041842IP49173-3.69	440	IP49	190		0.54				
445EN1426190 0.54 604D51931.28657D51921.0310111016EN1426178-2.441026EN14261951.781040EN14261921.031082EN14261962.031229EN1426187-0.211340EN14261910.791385EN1426152R(0.05)1399D51910.791468EN1426183.3-1.131613D5158R(0.05)7.411631EN14261691710EN1426178.8-2.241810EN1426178.8-2.241810EN1426178-3.69	444	IP49	189.2		0.34				
604D51931.28 657 D51921.03 1011 1016 EN1426178-2.44 1026 EN14261951.78 1040 EN14261921.03 1082 EN14261962.03 1229 EN1426187-0.21 1340 EN14261910.79 1385 EN1426152R(0.05)-8.90 1399 D51910.79 1468 EN1426183.3-1.13 1613 D5158R(0.05)-7.41 1631 EN1426169C-4.68 1710 EN14261982.53 1717 EN1426178.8-2.24 1810 EN14261880.04 1842 IP49173-3.69	445	EIN1426	190		0.54				
657 $D5$ 192 1.03 1011 1016 $EN1426$ 178 -2.44 1026 $EN1426$ 195 1.78 1040 $EN1426$ 192 1.03 1082 $EN1426$ 196 2.03 1229 $EN1426$ 187 -0.21 1340 $EN1426$ 191 0.79 1385 $EN1426$ 152 $R(0.05)$ -8.90 1399 $D5$ 191 0.79 1468 $EN1426$ 183.3 -1.13 1613 $D5$ 158 $R(0.05)$ -7.41 1631 $EN1426$ 169 C -4.68 First reported 159 1710 $EN1426$ 198 2.53 7.741 1710 $EN1426$ 178.8 -2.24 1810 $EN1426$ 178.8 -2.24 1810 $EN1426$ 188 0.04	604	D5	193		1.28				
1011 1016 EN1426 178 -2.44 1026 EN1426 195 1.78 1040 EN1426 192 1.03 1082 EN1426 196 2.03 1229 EN1426 187 -0.21 1340 EN1426 191 0.79 1385 EN1426 152 $R(0.05)$ -8.90 1399 $D5$ 191 0.79 1468 EN1426 183.3 -1.13 1613 $D5$ 158 $R(0.05)$ -7.41 1631 EN1426 169 C -4.68 First reported 159 1710 EN1426 178.8 -2.24 1810 EN1426 178.8 -2.24 1812 173 -3.69	657	D5	192		1.03				
1016 $EN1426$ 178 -2.44 1026 $EN1426$ 1951.781040 $EN1426$ 1921.031082 $EN1426$ 1962.031229 $EN1426$ 187-0.211340 $EN1426$ 1910.791385 $EN1426$ 152 $R(0.05)$ -8.901399 $D5$ 1910.791468 $EN1426$ 183.3-1.131613 $D5$ 158 $R(0.05)$ -7.411631 $EN1426$ 169C-4.681710 $EN1426$ 1982.531717 $EN1426$ 178.8-2.241810 $EN1426$ 1880.0418421P49173-3.69	1011								
1026EN1426195 1.78 1040EN1426192 1.03 1082EN1426196 2.03 1229EN1426187 -0.21 1340EN1426191 0.79 1385EN1426152R(0.05) -8.90 1399D5191 0.79 1468EN1426183.3 -1.13 1613D5158R(0.05) -7.41 1631EN1426169C -4.68 1710EN1426198 2.53 1717EN1426178.8 -2.24 1810EN1426188 0.04	1016	EN1426	178		-2.44				
1040EN14261921.031082EN14261962.031229EN1426187-0.211340EN14261910.791385EN1426152 $R(0.05)$ -8.901399D51910.791468EN1426183.3-1.131613D5158 $R(0.05)$ -7.411631EN1426169C-4.681710EN14261982.531717EN1426178.8-2.241810EN14261880.041842IP49173-3.69	1026	EN1426	195		1.78				
1082EN14261962.03 1229 EN1426187-0.21 1340 EN14261910.79 1385 EN1426152R(0.05)-8.90 1399 D51910.79 1468 EN1426183.3-1.13 1613 D5158R(0.05)-7.41 1631 EN1426169C-4.68First reported 1591710EN1426198 2.53 1717EN1426178.8-2.24 1810 EN14261880.04 1842 IP49173-3.69	1040	EN1426	192		1.03				
1229EN1426187 -0.21 1340EN1426191 0.79 1385EN1426152 $R(0.05)$ -8.90 1399D5191 0.79 1468EN1426183.3 -1.13 1613D5158 $R(0.05)$ -7.41 1631EN1426169C -4.68 1710EN1426198 2.53 1717EN1426178.8 -2.24 1810EN1426188 0.04	1082	EN1426	196		2.03				
1340EN1426191 0.79 1385EN1426152R(0.05) -8.90 1399D5191 0.79 1468EN1426183.3 -1.13 1613D5158R(0.05) -7.41 1631EN1426169C -4.68 First reported 1591710EN14261982.531717EN1426178.8 -2.24 1810EN1426188 0.04	1229	EN1426	187		-0.21				
1385 EN1426 152 R(0.05) -8.90 1399 D5 191 0.79 1468 EN1426 183.3 -1.13 1613 D5 158 R(0.05) -7.41 1631 EN1426 169 C -4.68 First reported 159 1710 EN1426 198 2.53 1717 EN1426 178.8 -2.24 1810 EN1426 188 0.04 1842 IP49 173 -3.69	1340	EN1426	191		0.79				
1399 D5 191 0.79 1468 EN1426 183.3 -1.13 1613 D5 158 R(0.05) -7.41 1631 EN1426 169 C -4.68 First reported 159 1710 EN1426 198 2.53 1717 EN1426 178.8 -2.24 1810 EN1426 188 0.04	1385	EN1426	152	R(0.05)	-8.90				
1468 EN1426 183.3 -1.13 1613 D5 158 R(0.05) -7.41 1631 EN1426 169 C -4.68 First reported 159 1710 EN1426 198 2.53 1717 EN1426 178.8 -2.24 1810 EN1426 188 0.04 1842 IP49 173 -3.69	1399	D5	191		0.79				
1613 D5 158 R(0.05) -7.41 1631 EN1426 169 C -4.68 First reported 159 1710 EN1426 198 2.53 1717 EN1426 178.8 -2.24 1810 EN1426 188 0.04 1842 IP49 173 -3.69	1468	EN1426	183.3		-1.13				
1631 EN1426 169 C -4.68 First reported 159 1710 EN1426 198 2.53 1717 EN1426 178.8 -2.24 1810 EN1426 188 0.04 1842 IP49 173 -3.69	1613	D5	158	R(0.05)	-7.41				
1710 EN1426 198 2.53 1717 EN1426 178.8 -2.24 1810 EN1426 188 0.04 1842 IP49 173 -3.69	1631	EN1426	169	С	-4.68	First reported 159			
1717 EN1426 178.8 -2.24 1810 EN1426 188 0.04 1842 IP49 173 -3.69	1710	EN1426	198		2.53				
1810 EN1426 188 0.04 1842 IP49 173 -3.69	1717	EN1426	178.8		-2.24				
1842 IP49 173 -3.69	1810	EN1426	188		0.04				
· · · · · · · · · · · · · · · · · · ·	1842	IP49	173		-3.69				
1849 EN1426 170 -4.43	1849	EN1426	170		-4.43				
1884 D5 190 0.54	1884	D5	190		0.54				
1970 EN1426 180.83 -1.74	1970	EN1426	180.83		-1.74				
EN1426 only D5 only							EN1426 only	D5 only	
normality OK OK suspect		normality	OK				OK	suspect	
n 33 23 6		n	33				23	6	
outliers 2 1 1		outliers	2				1	1	
mean (n) 187.84 187.50 189.83		mean (n)	187.84				187.50	189.83	
st.dev. (n) 7.949 8.756 2.927		st.dev. (n)	7.949				8.756	2.927	
R(calc.) 22.26 24.52 8.20		R(calc.)	22.26				24.52	8.20	
R(EN1426:07) 11.27 Compare $R(D5/5M:13) = 25.16$ 11.25 25.45		R(EN1426:07)	11.27	Compare	R(D5/5M	13) = 25.16	11.25	25.45	
					-(_ 3, 0.11	-,			





Determination of Penetration Index on sample #14260;

lab	method	value	mark	z(targ)	iis calc.	remarks
154						
168						
225	EN12591	0.13	E	4.24	-1.497	Error in calculation
332					-0.570	
333					-0.242	
335						
336					0.181	
337					-0.237	
353					-0.984	
357	EN12591	-0.66		-0.18	-0.656	
360					-1.147	
398	EN12591	0.7	E	7.44	0.642	Error in calculation
399					0.033	
440					-0.186	
444					0.052	
445					0.488	
604					-1.030	
657	Calc.	-1.0	E	-2.08	-0.494	Error in calculation
1011						
1016	EN12591	-1.189		-3.14	-1.189	
1026	EN12591	-0.34		1.61	-0.332	
1040	EN12591	-1.4	E	-4.32	-1.197	Error in calculation
1082					1.119	
1229					1.118	
1340	EN12591	-1.0		-2.08	-0.980	
1385					1.035	(excluded in calc. Pen. Index, outlier in Pen. 25°C test)
1399					1.773	
1468	EN12591	-0.5		0.72	-0.480	
1613					-1.264	(excluded in calc. Pen. Index, outlier in Pen. 25°C test)
1631					-0.869	
1710	EN12591	-0.17	E	2.56	-0.261	Error in calculation
1717	EN12591	-1.17		-3.04	-1.170	
1810					0.719	
1842					-2.062	
1849					-1.286	
1884					-0.274	
1970	EN12591	-0.936	E	-1.73	1.108	Error in calculation
	normality	ОК			ОК	
	n	12			31	
	outliers	0			0 (+2ex)	
	mean (n)	-0.628			-0.320 ´	
	st.dev. (n)	0.6197			0.8937	
	R(calc.)	1.735			2.502	
	R(EN12591:09)	0.500			0.500	





Determination of RTFOT at 163°C, Change of Mass on sample #14260; results in %

lab	method	value	mark	z(targ)	remarks
154					
168	D2872	0.067		-0.15	
225	D1754	0.03	ex	-0.67	TFOT test, excluded, see §4.1
332					
333					
335					
336					
353					
357	EN12607-1	0.099		0.30	
360					
398	EN12607-1	0.09		0.17	
399	D1754	0.06	ex	-0.25	TFOT test, excluded, see §4.1
440	-				, ,
444					
445					
604					
657	D2872	-0.04	DG(0.05)	-1.65	
1011					
1016	EN12607-1	0.086		0.11	
1026	EN12607-1	0.07		-0.11	
1040	EN12607 1	0.050		-0.26	
1229	EN12607-1	0.039		-0.20	
1340	EN12607-1	0.09		0.17	
1385	EN12607-2	0.053	ex	-0.35	TFOT test, excluded, see §4.1
1399	D2872	0	DG(0.05)	-1.09	- ····, - · ····, - · · · · · · · · · ·
1468	EN12607-1	0.021	- ()	-0.80	
1613					
1631	EN12607-1	0.07		-0.11	
1710	EN12607-1	0.06		-0.25	
1717	EN12607-1	0.087		0.13	
1810	EN12607-1	0.134		0.79	
1842					
1849	EN12607-1	0.079		0.02	
1004		0.0601	ox	0.25	TEOT test evoluded see 84.1
1970	SKES D. 10.019	0.0001	ex	-0.25	
	normality	not OK			
	n	14			
	outliers	2 (+4ex)			
	mean (n)	0.0778			
	st.dev. (n)	0.02511			
	R(calc.)	0.0703			
	R(EN12607-1:14)	0.2000			





Determination of RTFOT at 163°C, Retained Penetration on sample #14260; results in %

lab	method	value	mark	z(targ)	remarks
154					
168					
225	D1754	93.30	ex	8.40	TFOT test, excluded, see §4.1
332					
333					
335					
336					
331 252					
357	EN12607-1	50.8		-0.08	
360				-0.30	
398	EN12607-1	58.4		-1.37	
399					
440					
444					
445					
604					
657	D2872	64		0.20	
1011					
1016	EN12607-1	65.7		0.67	
1026	EN12607-1	67.2		1.09	
1040					
1082	EN12607-1	62		-0.36	
1229	EN12607-1	66 50.16		0.76	
1340	EN12007-1	09.10 124	OY	-1.10	TEOT tost evoluded see 84.1
1300	EN12007-2 EN12607-1	120		15.80	TFOT lesi, excluded, see §4.1
1468	EN12607-1	60.7	0(0.01)	-0.73	
1613					
1631	EN12607-1	74.2		3.05	
1710	EN12607-1	61		-0.64	
1717	EN12607-1	61.2		-0.59	
1810	EN12607-1	61.2		-0.59	
1842					
1849	EN12607-1	65.5		0.62	
1884					
1970	EN12607-2	62.8	ex	-0.14	TFOT test, excluded, see §4.1
	n ormolity (not OK			
	normality				
	outliere	14 1 (±30x)			
	mean (n)	63 29			
	st dev (n)	4 191			
	R(calc.)	11.74			
	R(EN12607-1:14)	10.00			
	. ,				
140 T					
11					*





Determination of RTFOT at 163°C, Viscosity Ratio on sample #14260;

154	lab	method	value	mark	z(targ)	remarks
$1 \\ 1 \\ 1 \\ 2 \\ 3 \\ 3 \\ 3 \\ 3 \\ 3 \\ 3 \\ 3 \\ 3 \\ 3$	154					
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	168					
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	225					
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	332 333					
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	335					
337 353 EN12607-1 1.89 -0.17 360 399 399 440 441 442 604 6057 1016 1028 EN12607-1 0.589 G(0.01) -9.69 1040 1380 1381 1389 EN12607-1 1.95 0.27 1613 1381	336					
$ \frac{353}{24} = \frac{100}{24} $ $ \frac{357}{24} = \frac{100}{24} $ $ \frac{357}{4} = \frac{100}{4} $ $ \frac{357}{4} = \frac{100}{$	337					
$ \frac{357}{920} = \frac{11, 189}{11, 189} = \frac{-0.17}{11, 180} = \frac{-0.17}{11, 190} = \frac{-0.17}{11, 100} = -0.17$	353					
$ \frac{360}{998}$	357	EN12607-1	1.89		-0.17	
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	360					
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	390					
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	440					
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	444					
$ \begin{bmatrix} 604 & & & & & & & & & & & & & & & & & & &$	445					
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	604					
$\begin{bmatrix} 1011 & & & & & & & & & & & & & & & & & $	657					
$\begin{bmatrix} 1026 \\ 1026 \\ EN12607-1 \\ 1.9 \\ $	1011					
$\begin{bmatrix} 1 \\ 1060 \\ 1082 \\ 1229 \\ 1340 \\ 1385 \\ 1399 \\ 1468 \\ EN12607-1 \\ 1.95 \\ 1027 \\ 1631 \\ 1631 \\ 1710 \\ 1717 \\ 1710 \\ 1842 \\ 1842 \\ 1849 \\ 1849 \\ 1849 \\ 1884 \\ 1970 \\ 1884 \\ 1970 \\ 1884 \\ 1970 \\ 1970 \\ 1000 \\ 1.913 \\ st.dev. (n) (0.0321) \\ remain (n) 1.913 \\ remain (n) $	1010	EN12607-1	0.589	G(0.01)	-9.69	
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	1040			0(0.01)		
$1229 \qquad \qquad 1340 \qquad \qquad 1385 \qquad \qquad 1385 \qquad \qquad 1385 \qquad \qquad 1386 EN12607-1 1.95 \qquad 0.27 1613 \qquad \qquad 1613 \qquad \qquad 1613 \qquad \qquad 1710 \qquad \qquad 1810 \qquad \qquad 1842 \qquad \qquad 1844 \qquad \qquad 1884 \qquad \qquad 1884 \qquad \qquad 1970 \qquad \qquad normality unknown n mean (n) 1.913 st.dev. (n) (0.0321) R(EN12607-1:14) 0.383 3$	1082	EN12607-1	1.9		-0.10	
$1340 \qquad \qquad 1385 \qquad \qquad 1399 \qquad \qquad 1463 EN12607-1 \qquad 1.95 \qquad 0.27 1613 \qquad \qquad 1631 \qquad \qquad 1710 \qquad \qquad 1810 \qquad \qquad 1810 \qquad \qquad 1842 \qquad \qquad 1844 \qquad \qquad 1884 \qquad \qquad 1884 \qquad \qquad 1970 \qquad \qquad 1970 \qquad \qquad 1970 \qquad \qquad 1970 \qquad \qquad 107 \qquad \qquad$	1229					
1399 1399 1468 EN12607-1 1.95 0.27 1613 1631 1710 1717 1842 1844 1884 1884 1970 1970 1970 10 1884 1970 10 10 1884 1970 10 1884 1970 10 10 1884 1970 10 10	1340					
$\begin{bmatrix} 1399 \\ 1468 \\ EN12607-1 \\ 1.95 \\ 0.27 \\ 1613 \\ \\ 1631 \\ \\ 1710 \\ \\ 1717 \\ \\ 1717 \\ \\ 1717 \\ \\ 1840 \\ \\ 1842 \\ \\ 1844 \\ \\ 1884 \\ \\ 1884 \\ \\ 1884 \\ \\ 1884 \\ \\ 1884 \\ \\ 1884 \\ \\ \\ 1884 \\ \\ 1884 \\ \\ \\ 1884 \\ \\ 1884 \\ \\ \\ 1884 \\ \\ 1884 \\ \\ \\ 1884 \\ \\ \\ 1884 \\ \\ \\ \\ 1884 \\ \\ \\ \\ 1884 \\ \\ \\ 1884 \\ \\ \\ 1884 \\ \\ \\ \\ 1884 \\ \\ \\ 1884 \\ \\ \\ 1884 \\ \\ \\ \\ 1884 \\ \\ \\ \\ 1884 \\ \\ \\ \\ 1884 \\ \\ \\ \\ 1884 \\ \\ \\ \\ \\ 1884 \\ \\ \\ \\ \\ 1884 \\ \\ \\ \\ \\ \\ \\ 1884 \\$	1385					
$\begin{bmatrix} 1613 & & & & & & & & \\ 1631 & & & & & & & & \\ 1710 & & & & & & & & \\ 1717 & & & & & & & & \\ 1717 & & & & & & & & \\ 1810 & & & & & & & & \\ 1842 & & & & & & & & \\ 1849 & & & & & & & & \\ 1884 & & & & & & & & \\ 1970 & & & & & & & & \\ 1970 & & & & & & & & \\ normality & unknown & n & & & & \\ n & & & & & & & & \\ 1970 & & & & & & & & \\ normality & unknown & n & & & \\ n & & & & & & & & \\ normality & unknown & n & & \\ n & & & & & & & \\ 1884 & & & & & & & \\ 1970 & & & & & & & \\ 1884 & & & & & & & \\ 1970 & & & & & & & \\ normality & unknown & n & & \\ n & & & & & & & \\ n & & & & $	1468	EN12607-1	1.95		0.27	
$1631 \qquad \qquad 1710 \qquad \qquad 1717 \qquad \qquad 1810 \qquad \qquad 1842 \qquad \qquad 1849 \qquad \qquad 1884 \qquad \qquad 1970 \qquad \qquad normality unknown n 3 outliers 1 mean (n) 1.913 st.dev. (n) (0.0321) R(calc.) (0.090) R(EN12607-1:14) 0.383 3 \frac{1}{25} \frac{1}{2} $	1613					
$1710 \qquad \qquad \\ 1717 \qquad \qquad \\ 1842 \qquad \qquad \\ 1849 \qquad \qquad \\ 1849 \qquad \qquad \\ 1884 \qquad \qquad \\ 1970 \qquad \qquad \\ 1970 \qquad \qquad \\ normality \qquad unknown \\ n \qquad 3 \\ outliers \qquad 1 \\ mean (n) \qquad 1.913 \\ st.dev. (n) \qquad (0.0321) \\ R(calc.) \qquad (0.090) \\ R(EN12607-1:14) \qquad 0.383 \\ \\ \hline \\ 3 \\ 2 \\ - \\ - \\ - \\ - \\ - \\ - \\ - \\ - \\ -$	1631					
1717	1710					
$1810 \qquad \qquad \\ 1842 \qquad \qquad \\ 1849 \qquad \qquad \\ 1970 \qquad \qquad \\ normality \qquad unknown \\ n \qquad 3 \\ outliers \qquad 1 \\ mean (n) \qquad 1.913 \\ st.dev. (n) \qquad (0.0321) \\ R(calc.) \qquad (0.090) \\ R(EN12607-1:14) \qquad 0.383 \\ \hline 3 \\ 2 \\ 2 \\ 2 \\ 2 \\ 2 \\ 2 \\ 2 \\ 2 \\ 2 \\ 2$	1717					
1349	1810					
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	1849					
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	1884					
normality unknown n 3 outliers 1 mean (n) 1.913 st.dev. (n) (0.0321) R(calc.) (0.090) R(EN12607-1:14) 0.383	1970					
normality unknown n 3 outliers 1 mean (n) 1.913 st.dev. (n) (0.0321) R(calc.) (0.090) R(EN12607-1:14) 0.383						
$\begin{bmatrix} n & & 3 \\ outliers & 1 \\ mean (n) & 1.913 \\ st.dev. (n) & (0.0321) \\ R(calc.) & (0.090) \\ R(EN12607-1:14) & 0.383 \end{bmatrix}$		normality	unknown			
Outlet's 1 mean (n) 1.913 st.dev. (n) (0.0321) R(calc.) (0.090) R(EN12607-1:14) 0.383		n outliere	3 1			
st.dev. (n) (0.0321) R(calc.) (0.090) R(EN12607-1:14) 0.383		mean (n)	1.913			
R(calc.) (0.090) R(EN12607-1:14) 0.383		st.dev. (n)	(0.0321)			
R(EN12607-1:14) 0.383		R(calc.)	(0.090)			
		R(EN12607-1:14)	0.383			
а 25 2 2 						
2.5 - 2 - A	3					
	2.5 -					
	2 -				4	ΔΔ
1.5 -	1.5 -					
	1 -					
x		×				
	0.5	226		36.7	ž	468

Determination of RTFOT at 163°C, Increase in Softening point on sample #14260; results in °C

lab	method	value	mark	z(targ)	remarks
154					
168					
225	D1754	4.6	ex	0.85	TFOT test, excluded, see §4.1
332	-		-		, · · · · · · · · · · · · · · · ·
333					
335					
336					
337					
353					
357	EN12607-1	4.2		0.29	
360					
398	EN12607-1	3.6		-0.55	
399					
440					
444					
445					
604					
657					
1011					
1016	EN12607-1	4.0		0.01	
1026	EN12607-1	3.4		-0.83	
1040					
1082	EN12607-1	4.4		0.57	
1229	EN12607-1	4.2		0.29	
1340	EN12607-1	5.0		1.41	
1385	EN12607-2	49	ex	63.01	TFOT test, excluded, see §4.1
1399	EN12607-1	4.15		0.22	,, <u>u</u>
1468	EN12607-1	3.65		-0.48	
1613					
1631	EN12607-1	2.6		-1.95	
1710	EN12607-1	4.5		0.71	
1717	EN12607-1	3.2		-1.11	
1810	EN12607-1	5.0		1.41	
1842					
1849					
1884					
1970	EN12607-2	5.0	ex	1.41	TFOT test, excluded, see §4.1
		-		-	,, <u>u</u>
	normality	OK			
	n	13			
	outliers	0 (+3ex)			
	mean (n)	3.99			
	st.dev. (n)	0.692			
	R(calc.)	1.94			
	R(EN12607-1:14)	2.00			
	(





Determination of Softening Point (Ring & Ball) on sample #14260; results in °C

lab	method	value	mark	z(targ)	remarks			
154								
168								
225	D36	37.2		-4.24				
332	EN1427	39.4		-1.16				
333	EN1427	39.8		-0.60				
335	EN1427	39.0	С	-1 72	First report	ed 39 0 as res	sult for RTF	OT Increase in softening point
336	EN1427	41.0	U	1.02	i not report	00.000		or morease in solitening point
337	EN1427	39.6		-0.88				
252	EN1427	29.0		-0.00				
252	EN1427	20.2		-2.04				
201		39.2		-1.44				
300		36.95		-1.79				
398	EN1427	42.2		2.76				
399	D36	41		1.08				
440	IP58	40.2		-0.04				
444	IP58	40.8		0.80				
445	EN1427	41.8		2.20				
604	D36	38.2		-2.84				
657	D36	39.4		-1.16				
1011								
1016	EN1427	38.6		-2.28				
1026	EN1427	39.6		-0.88				
1040	EN1427	37.9		-3.26				
1082	EN1427	43.0		3.88				
1229	EN1427	43.6		4.72				
1340	EN1427	38.4		-2.56				
1385	EN1427	46		8.08				
1399	D36	45 1		6.82				
1468	EN1427	39.9		-0.46				
1613	D36	39.5		-1 02				
1631	EN1427	30.8		-0.60				
1710	EN1427	20.6		-0.00				
1710		39.0		-0.00				
1/1/		30.0		-2.20				
1010		42.0 27		3.10				
1842	1200 EN14 407	31		-4.52				
1849	EN1427	38.8		-2.00				
1884	D36	40		-0.32				
1970	EN1427	44.0		5.28	a (•	
					Group 1	Group 2	Group 3	
	normality	suspect			OK	OK	Unknowr	1
	n	34			26	6	2	
	outliers	0			0	0	0	
	mean (n)	40.23			39.22	42.85	45.6	
	st.dev. (n)	2.168			1.033	0.843	n.a.	
	R(calc.)	6.07			2.89	2.36	n.a.	
	R(EN1427:07)	2.00			2.00	2.00	2.00	Compare R(D36/36M:12) = 2.00
	,							
40								





Determination of Solubility in Xylene on sample #14260; results in %M/M

lab	method	value	mark	z(targ)	remarks
154					
168					
225					
332					
333	EN12592	100		1.07	
335					
336					
337					
353					
357	EN12592	99.69	G(0.05)	-4.71	
360	EN12592	99.989		0.87	
398	EN12592	99.99		0.89	
399	EN12592	99.99		0.89	
440					
444	ENICOSOO				
445	EN12592	99.98		0.70	
604	D2042	99.7789	ex	-3.05	Excluded, method not equivalent, see §4.1
657					
1011	ENICOSOO		0(0.04)		
1016	EN12592	99.45	G(0.01)	-9.19	
1026	EN12592	99.9		-0.79	
1040					
1082					
1229				0.14	
1340	EIN12592	99.95		0.14	
1300					
1399	EN12502	00.96		1 5 4	
1400	EINT2092	99.00		-1.04	
1631	EN12502	00.86		-1 5/	
1710	EN12502	99.00		-0.05	In toluone instead of vulene
1717	EN12592	99.94		0.05	In toluene instead of xylene
1810	LIN12002			0.00	
1842					
1849	EN12592	99 86		-1.54	
1884	LITILOOL				
1970					
	normality	ОК			
	n	12			
	outliers	2 (+1ex)			
	mean (n)	99.9424́			
	st.dev. (n)	0.05715			
	R(calc.)	0.1600			
	R(EN12592:14)	0.1500			
	. ,				
100.2 T					
100.1 -					





Determination of Density at 25°C on sample #14261; results in kg/m³

lab	method	value	mark	z(targ)	remarks
154	EN15326	1048.0	С	-0.20	First reported: 3.2
168	D70	1049.9	С	0.87	Reported: 1.0499 (probably a unit error)
225		1048		-0.20	
332	EN15326	1048	С	-0.20	First reported: 1.048
333					•
335	EN15326	1050.0	С	0.92	First reported: 1.050
336	EN15326	1054		3.16	•
337	EN15326	1051.0		1.48	
353					
357	EN15326	1049.7		0.76	
360	ISO3838	1045.06		-1.84	
398	EN15326	1049		0.36	
399	D70	1048		-0.20	
440					
444	D70	1051.8		1.93	
445	D70	1046.3		-1.15	
604	D70	1046.3		-1.15	
657	D70	1044		-2.44	
1011					
1016	EN15326	1046.45		-1.06	
1026	EN15326	1049.5		0.64	
1040					
1082	EN15326	1046.0		-1.32	
1229	EN15326	1046		-1.32	
1340	EN15326	1048		-0.20	
1385					
1399	IP70	1040.1	R(0.05)	-4.62	
1468			, ,		
1613					Reported 1053.0 at 15°C, measured at 120°C: 990.8
1631					
1710	ISO3838	1050	С	0.92	First reported: 1060
1717	ISO3838	1051		1.48	
1810	EN15326	1046.5		-1.04	
1842					
1849					
1884	D70	1049	С	0.36	First reported: 1.049
1970	EN15326	1047.2		-0.64	
					
	normality	OK			
	n	25			
	outliers	1			
	mean (n)	1048.35			
	st.dev. (n)	2.308			
	R(calc.)	6.46			
	R(EN15326:07)	5.00			





Determination of Shear Mod G* at 60°C, 1.59 Hz on sample #14261; results in Pa

lab	method	value	mark	z(targ)	remarks
154					
168					
225					
332					
333					
335					
336					
337					
353					
357					
360					
390					
399 440					
440					
445					
604					
657					
1011					
1016					
1026	EN14770	11140			
1040					
1082	EN14770	13900			
1229					
1340					
1385					
1399					
1400					
1631					
1710					
1717					
1810					
1842					
1849					
1884					
1970					
	normality	unknown			
	n	2			
	outliers	– n.a.			
	mean (n)	12520			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(EN14770:12)	5634			

Determination of Phase Angle at 60°C, 1.59 Hz on sample #14261; results in °

lab	method	value	mark z(targ)	remarks
154				
168				
225				
332				
333				
335				
336				
337				
353				
357				
360				
398				
399				
440				
444				
445				
604				
657				
1011				
1016				
1026	EN14770	85.33		
1040				
1082	EN14770	84.8		
1229				
1340				
1200				
1469				
1613				
1631				
1710				
1717				
1810				
1842				
1849				
1884				
1970				
	normality	unknown		
	n	2		
	outliers	n.a.		
	mean (n)	85.06		
	st.dev. (n)	n.a.		
	R(calc.)	n.a.		
	R(EN14770:12)	9.00		

Determination of Dynamic Viscosity at 60°C on sample #14261; results in Pa.s

lab	method	value	mark	z(targ)	remarks
154	EN12596	12509	C,G(0.01)	184.05	First reported: 11063
168	D2171	1444.6		0.62	·
225					
332					
333					
335					
336					
337					
353					
357	EN12596	1396.8		-0.18	
360					
398	EN12596	1345.05		-1.03	
399					
440					
444					
445					
604					
657	D2171	1375		-0.54	
1011					
1016	EN12596	1418		0.18	
1026	EN12596	1392.4		-0.25	
1040	EN12596	1470.1		1.04	
1082	ENICOSOO				
1229	EN12596	1355		-0.87	
1340	EN12596	1470		1.04	
1385					
1399					
1400	D4402	1600	<u></u>	4.60	
1013	D4402	1690	ex	4.00	Excluded, see §4.1
1710					
1710					
1010					
18/2					
18/0					
1884					
1004					
1370					
	normality	OK			
	n	9			
	outliers	0 1 (+1ex)			
	mean (n)	1407.44			
	st dev (n)	46 592			
	R(calc.)	130.46			
	R(EN12596:14)	168.89			
1800 -					
1700 -					¥
					~
1600					





Determination of Flash Point COC on sample #14261; results in °C

lab	method	value	mark	z(targ)	remarks
154	ISO2592	356	С	-0.38	First reported: 650
168	D92	357.2	-	-0.19	
225	D92	376		2.91	
332	-				
333	ISO2592	374		2.58	
335					
336					
337					
353					
357	ISO2592	354		-0.71	
360	ISO2592	352		-1.04	
398	ISO2592	340		-3.02	
399					
440					
444					
445	IP36	357.9		-0.07	
604	D92	354		-0.71	
657	D92	362		0.60	
1011					
1016	ISO2592	356.6		-0.29	
1026	ISO2592	362		0.60	
1040					
1082					
1229					
1340	ISO2592	378		3.24	
1385	EN22592	330		-4.67	
1399					
1468					
1613					
1631					
1710	ISO2592	372		2.25	
1717	ISO2592	364		0.93	
1810					
1842					
1849	ISO2592	346		-2.03	
1884	D92	>250			
1970					
	normality	OK			
	n	17			
	outliers	0			
	mean (n)	358.3			
	st.dev. (n)	12.67			
	R(calc.)	35.5			
	R(ISO2592:00)	17.0			Compare R(D92:12b) = 18.0
³⁹⁰ T					
380 -					
-					ΔΔ
1270 -					A





Determination of Fraass Breaking point on sample #14261; results in °C

lab	method	value	mark	z(targ)	remarks
154					
168					
225					
332					
333	EN12593	0		1.37	
335					
336					
337					
353	EN12502	5		0.06	
307	EN12090	-0 5 5		-0.90	
308	EN12090	0.0 8		5.94	
300	LIN12393	0		5.10	
440					
444					
445					
604					
657					
1011					
1016	EN12593	-3		-0.03	
1026	EN12593	-5		-0.96	
1040					
1082					
1229					
1340	EN12593	0		1.37	Reported: ±0
1385					
1399					
1468					
1613					
1631					
1710	EN12593	-8		-2.30	
1010	EN12090	-9.0		-3.20	
18/2	LIN12393	-14		-5.10	
1840					
1884					
1970	EN12593	-1.0		0.90	
	22000			0.00	
	normality	OK			
	n	11			
	outliers	0			
	mean (n)	-2.9			
	st.dev. (n)	6.45			
	R(calc.)	18.0			
	R(EN12593:07)	6.0			
					Δ
5 -					Δ
0					





Determination of Kinematic Viscosity at 135°C on sample #14261; results in mm²/s

lab	method	value	mark	z(targ)	remarks
154	EN12595	1009		-0.99	
168	D2170	1047.0		0.14	
225					
332					
333					
335					
336					
337					
353					
357	EN12595	964.3		-2.33	
360					
398	EN12595	1058.65		0.49	
399					
440					
444					
445	D2170	1029		-0.40	
604					
657	D2170	879.17	C,G(0.05)	-4.87	First reported: 883.18
1011					
1016	EN12595	1016		-0.78	
1026	EN12595	1038.7		-0.11	
1040					
1082	EN12595	1084		1.25	
1229	EN12595	1021		-0.63	
1340	EN12595	1040.5		-0.05	
1385					
1399					
1468					
1613					
1631					
1710	EN12595	1080		1.13	
1/1/	EN12595	1068		0.77	
1810	EN12595	1093		1.52	
1842					
1849					
1884					
1970					
	Pt				
	normality				
	n tl'	13			
		1042.24			
	mean (n)	1042.24			
	St.dev. (n)	33.604			
	R(Calc.)	99.69			$C_{\text{ampore }} D/D2170/2170M(10) = 01.72$
	R(EN12395.14)	33.00			Compare R(D21/0/21/0W1.10) = 91.72
¹²⁰⁰ T					
1150					
1150 -					
1100 -					





Determination of Penetration at 25°C on sample #14261; results in 0.1 mm

lab	method	value	mark	z(targ)	remarks			
154	EN1426	18		-3.69				
168	D5	20		-1.82				
225	D5	17		-4.62				
332	EN1426	20.5		-1.35				
333	EN1426	22		0.05				
335	EN1426	25.7	С	3.50	First reported 25.	.7 as kinematic viscos	sity at 135°C	
336	EN1426	28		5.65	1		,	
337	EN1426	29		6.58				
353	EN1426	22		0.05				
357	EN1426	21		-0.89				
360	EN1426	20		-1.82				
398	EN1426	20		-1.82				
399	D5	21		-0.89				
440	IP49	21.0		-0.89				
444	IP49	21.7		-0.23				
445	EN1426	25		2.85				
604	D5	21		-0.89				
657	D5	24		1.91				
1011	20							
1016	EN1426	20		-1.82				
1026	EN1426	21		-0.89				
1040	EN1426	20.4		-1.45				
1082	EN1426	25		2 85				
1229	EN1426	21		-0.89				
1340	EN1426	23		0.98				
1385	EN1426	31		8 45				
1399	D5	22		0.05				
1468	20							
1613	D5	12		-9.29				
1631	20							
1710	EN1426	21		-0.89				
1717	EN1426	21.6		-0.33				
1810	EN1426	18.5		-3.22				
1842	IP49	23		0.98				
1849	EN1426	17		-4 62				
1884	D5	26		3.78				
1970	EN1426	26 875		4 60				
1010	2111120	20.010				EN1426 only	D5 only	
	normality	suspect				OK	OK	
	n	34				23	8	
	outliers	0				0	0	
	mean (n)	21.95				22.50	21.09	
	st dev (n)	3 691				3 636	4 811	
	R(calc.)	10.34				10.18	13.47	
	R(FN1426:07)	3.00	Compare	R(D5/5M)	13) = 7.08	3.00	7.08	
	(LIT 120.01)	0.00	oompare		10) = 1.00	0.00	1.00	
35 -								
								۵
30 -								_ ▲
								<u>م</u>
25 -							Δ Δ Δ	
_					· · · · · · · · · · · · · · · · · · ·			
20	•							





Determination of Penetration Index on sample #14261;

lab	method	value	mark	z(targ)	iis calc.	remarks
154						
168						
225	Calc.	0.01	C,G(0.01),E	5.81	-1.444	First reported: 0, Error in calculation
332					-1.294	
333					-0.938	
335					-0.926	
336					-0.454	
337					-0.424	
353					-1.556	
357	EN12591	-1.09		-0.35	-1.094	
360					-1.168	
398	EN12591	-0.8		1.27	-0.847	
399					-0.908	
440					-0.982	
444					-0.869	
445					-0.371	
604					-1.132	
657	Calc.	-1.0	E	0.15	-0.782	Error in calculation
1011						
1016	EN12591	-1.1		-0.41	-1.103	
1026	EN12591	-0.86		0.93	-0.835	
1040	EN12591	-1.3		-1.53	-1.314	
1082					-0.784	
1229						
1340	EN12591	-1.0		0.15	-1.011	
1385					-0.060	
1399					-0.007	
1468						
1613					-2.675	(outlier in evaluation calculated Penetration Index)
1631						(,
1710	EN12591	-1.04		-0.07	-1.038	
1717	EN12591	-1.05		-0.13	-1.083	
1810					-0.574	
1842					-1.560	
1849					-1.732	
1884					-0.596	
1970	EN12591	-0.815	ex, E	1.19	0.856	Excluded as outlier in Softening Point test, Error in calc.
	normality	auanaat			OK	
	normality	suspect				
	u outlioro	9 1 (110x)			29 1 (1103)	
	outliers	1 (+1ex)			1 (+1ex)	
	niedn (n)	-1.027			-0.927	
	SLUEV. (II)	0.1438			0.4176	
		0.403			1.169	
	R(EN12591:09)	0.500			0.500	





Determination of RTFOT at 163°C, Change of Mass on sample #14261; results in %

lab	method	value	mark	z(targ)	remarks
154 168 225	D2872 D1754	 0.094 0.04	ex	0.39 -0.36	TEOT test, excluded, see §4.1
332 333	51101		U.		
335					
336					
353					
357	EN12607-1	0.06		-0.08	
360	EN12607 1			0.24	
398 399	D1754	0.09	ex	-0.22	TEOT test, excluded, see §4.1
440	2		0,1		
444					
445					
604 657	D2872	0.04		-0.36	
1011	22012				
1016	EN12607-1	0.065		-0.01	
1026	EN12607-1	0.07		0.06	
1040					
1229					
1340	EN12607-1	0.07		0.06	
1385	EN12607-2	0.055	ex	-0.15	TFOT test, excluded, see §4.1
1399	EN12007-1	0.03		-0.50	
1613					
1631					
1710	EN12607-1	0.03		-0.50	
1810	EN12607-1	0.128		-0.14	
1842					
1849 1884	EN12607-1	0.0565		-0.13	
1970	SRPS B.H8.619	0.020	ex	-0.64	TFOT test, excluded, see §4.1
	normality	OK			
	n outliers	12 0 (+4ex)			
	mean (n)	0.0658			
	st.dev. (n)	0.02817			
	R(calc.)	0.0789			
	R(EN12007-1:14)	0.2000			





Determination of RTFOT at 163°C, Retained Penetration on sample #14261; results in %

lab	method	value	mark	z(targ)	remarks
154					
168					
225	D1754	94.44	ex,C	5.65	First reported: 100, TFOT test, excluded, see §4.1
332					
333					
335					
336					
337					
353					
357	EN12607-1	71.4		-0.81	
360					
398	EN12607-1	65.0		-2.60	
399					
440					
444					
445					
604					
657	D2872	75		0.20	
1011					
1016	EN12607-1	80		1.60	
1026	EN12607-1	71.4		-0.81	
1040					
1082					
1229	EN40007 4				
1340	EN12607-1	73.91		-0.10	TEOT toot avaluated and \$4.4
1300	EN12007-2	12		-17.44	TFOT test, excluded, see §4.1
1399	EIN12607-1	20	G(0.01)	-15.20	
1400					
1621					
1710	EN12607 1	92		2 16	
1710	EN12007-1	7/1		-0.05	
1810	EN12607-1	74.1		-0.05	
1842				0.40	
1849					
1884					
1970	EN12607-2	70.37	ex	-1 09	TEOT test excluded see §4.1
1010		10.01	<u>o</u> x	1.00	
	normality	ОК			
	n	9			
	outliers	1 (+3ex)			
	mean (n)	74.29			
	st.dev. (n)	4.972			
	R(calc.)	13.92			
	R(EN12607-1:14)	10.00			





Determination of RTFOT at 163°C, Viscosity Ratio on sample #14261;

lab	method	value	mark	z(targ)	remarks
154					
168					
225					
332 222					
335					
336					
337					
353					
357	EN12607-1	2.34			
360					
398					
399					
440					
444					
445					
604					
1011					
1011					
1010	EN12607-1	0.5			
1020					
1082					
1229					
1340					
1385					
1399					
1468					
1613					
1631					
1710					
1/1/					
1010					
1042					
1884					
1970					
1310					
	normality	n.a.			
	n	2			
	outliers	n.a.			
	mean (n)	1.42			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(EN12607-1:14)	0.28			

Determination of RTFOT at 163°C, Increase in Softening point on sample #14261; results in °C

lab	method	value	mark	z(targ)	remarks
154					
168					
225	D1754	0.02	ex	-6.48	TFOT test, excluded, see §4.1
332					
333					
335					
336					
337					
353					
357	EN12607-1	4.8		0.21	
360					
398	EN12607-1	5.0		0.49	
399					
440					
444					
445					
604					
1011					
1011	EN12607 1	 A A		0.25	
1010	EN12007-1	4.4		-0.35	
1020		5.0		-1.19	
1040					
1220					
1340	EN12607-1	44		-0 35	
1385					
1399	EN12607-1	50		0 49	
1468	21112007				
1613					
1631					
1710	EN12607-1	4.7		0.07	
1717	EN12607-1	5.0		0.49	
1810	EN12607-1	5.1		0.63	
1842					
1849	EN12607-1	4.3		-0.49	
1884					
1970	EN12607-2	9.3	ex	6.51	TFOT test, excluded, see §4.1
	normality	OK			
	n	10			
	outliers	0 (+2ex)			
	mean (n)	4.65			
	st.dev. (n)	0.417			
	R(calc.)	1.17			
	R(EN126071:14)	2.00			





Determination of Softening Point (Ring & Ball) on sample #14261; results in °C

lab	method	value	mark	z(targ)	remarks
154					
168					
225	D36	58.4		-0.34	
332	EN1427	57 56		-1 52	
333	EN1427	58.8		0.22	
335	EN1427	57.4		-1 74	
336	EN1427	50.0		0.50	
227	EN1427	59.0		0.30	
331		50.0		0.22	
353	EN1427	55.6		-4.20	
357	EN1427	58.4		-0.34	
360	EN1427	58.45		-0.27	
398	EN1427	60.2		2.18	
399	D36	59.4		1.06	
440	IP58	59.0		0.50	
444	EN1427	59.3		0.92	
445	EN1427	60.60		2.74	
604	D36	58.2		-0.62	
657	D36	58.8		0.22	
1011					
1016	EN1427	58.8		0.22	
1026	EN1427	59.8		1.62	
1040	EN1427	57.5		-1.60	
1082	EN1427	58.4		-0.34	
1229					
1340	FN1427	58.0		-0.90	
1385	EN1427	60		1.90	
1399	D36	64 0		7.50	
1468	200				
1613	D36	54 5		-5.80	
1631	000	04.0		-5.00	
1710	EN1427	59.7		0.09	
1710	EN1427	50.7		0.00	
1/1/		56.Z		-0.62	
1810	EN1427	62.5		5.40	
1842	IP58	55.2		-4.82	
1849	EN1427	56.85		-2.51	
1884	D36	59		0.50	
1970	EN1427	66.8	R(0.01)	11.42	
	normality	not OK			
	n	30			
	outliers	1			
	mean (n)	58.65			
	st.dev. (n)	1.867			
	R(calc.)	5.23			
	R(EN1427:07)	2.00			Compare R(D36/36M:13) = 2.00
⁶⁸ T					
66					×
00 -					
64 -					Δ
62 -					۵
					Δ
				<u> </u>	
58	Δ	ΔΔ.Δ	<u> </u>		
56 -	Δ Δ				
54 - ^Δ	_				



Determination of Solubility in Xylene on sample #14261; results in %M/M

lab	method	value	mark	z(targ)	remarks
154					
168					
225					
332					
333	EN12592	100		0.74	
335					
336					
337					
353					
357	EN12592	99.99		0.55	
360	EN12592	99.955		-0.10	
398	EN12592	99.99		0.55	
399	EN12592	99.99		0.55	
440					
444	EN12502	00.00		0.55	
604	D2042	99.99	07	0.55	Evaluated method not equivalent, see \$4.1
657	D2042	99.9079	ex	0.51	Excluded, method not equivalent, see 34.1
1011					
1016	EN12592	99.65	G(0.01)	-5.80	
1026	EN12592	99.90	0(0.01)	-1 13	
1040	21112002				
1082					
1229					
1340	EN12592	99.90		-1.13	
1385					
1399					
1468					
1613					
1631					
1710	EN12592	99.98		0.36	In toluene instead of xylene
1717	EN12592	99.99		0.55	In toluene instead of xylene
1810					
1842					
1849	EN12592	99.88	С	-1.50	First reported: 99.77
1884					
1970					
	normality	ОК			
	n	11			
	outliers	1 (+1ex)			
	mean (n)	99.9604			
	st.dev. (n)	0.04486			
	R(calc.)	0.1256			
	R(EN12592:14)	0.1500			
	. ,				
100.2 -					





APPENDIX 2

Number of participants per country

1 lab in AUSTRIA 1 lab in BOSNIA and HERZEGOVINA 1 lab in BULGARIA 1 lab in COTE D'IVOIRE 1 lab in CZECH REPUBLIC 3 labs in FINLAND 5 labs in FRANCE 1 lab in GEORGIA 1 lab in GERMANY 2 labs in HUNGARY 1 lab in IRELAND 2 labs in ITALY 1 lab in JORDAN 1 lab in LEBANON 1 lab in MALAYSIA 2 labs in NETHERLANDS 1 lab in PORTUGAL 1 lab in SERBIA 1 lab in SINGAPORE

1 lab in SOUTH AFRICA

2 labs in TURKEY

4 labs in UNITED KINGDOM

2 labs in UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations:

С	= final result after checking of first reported suspect result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.05)	= straggler in Rosner's outlier test
R(0.01)	= outlier in Rosner's outlier test
ex	= excluded from calculations
E	= error in calculations
n.a.	= not applicable

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