Results of Proficiency Test
Bitumen
December 2018

Organised by: Institute for Interlaboratory Studies
                Spijkenisse, the Netherlands

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## 1	INTRODUCTION

Since 2014, the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for Bitumen. During the annual proficiency testing program 2018/2019, it was decided to continue the round robin for the analysis of Bitumen based on the scope of the latest specification for EN12591 Paving Grade.

In this interlaboratory study 39 laboratories in 21 different countries registered for participation. See appendix 2 for the number of participants per country.

In this report, the results of the 2018 proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

## 2	SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send two different samples of Bitumen, 1x2.5L can of sample #18260 grade 70/100 and 1x1L can of sample #18261 grade 35/50. The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

### 2.1	QUALITY SYSTEM

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

### 2.2	PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of 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

In this proficiency test two samples with a different grade were supplied by a third party. Samples with grade 70/100 were supplied in a 2.5L can and labelled #18260, samples with grade 35/50 were supplied in a 1L can and labelled #18261.
The homogeneity of the subsamples #18260 was checked by determination of Penetration in accordance with EN1426 and Softening Point in accordance with EN1427 on 4 stratified randomly selected samples.

	Penetration at 25°C in 0.1 mm	Softening Point (Ring and Ball) in °C
Sample #18260-1	87	44.6
Sample #18260-2	87	44.8
Sample #18260-3	87	44.8
Sample #18260-4	87	44.2

Table 1: homogeneity test results of subsamples #18260

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

	Penetration at 25°C in 0.1 mm	Softening Point (Ring and Ball) in °C
r (observed)	0.0	0.79
reference test method	EN1426:15	EN1427:15
r (reference test method)	3.5	1.0

Table 2: evaluation of the repeatabilities of subsamples #18260

The calculated repeatabilities were in agreement with the repeatability of the respective reference test method. Therefore, homogeneity of the subsamples of #18260 was assumed.

The homogeneity of the subsamples #18261 was checked by determination of Penetration in accordance with EN1426 and Softening Point in accordance with EN1427 on 4 stratified randomly selected samples.

	Penetration at 25°C in 0.1 mm	Softening Point (Ring and Ball) in °C
Sample #18260-1	39	51.0
Sample #18260-2	39	51.0
Sample #18260-3	38	51.6
Sample #18260-4	38	51.6

Table 3: homogeneity test results of subsamples #18261

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

	Penetration at 25°C in 0.1 mm	Softening Point (Ring and Ball) in °C
r (observed)	1.6	0.97
reference test method	EN1426:15	EN1427:15
r (reference test method)	1.5	1.0

Table 4: evaluation of the repeatabilities of subsamples #18261

The calculated repeatabilities were in agreement with the repeatability of the respective reference test method. Therefore, homogeneity of the subsamples of #18261 was assumed.

To each of the participating laboratories 1*2.5L can of sample #18260 and 1*1L can of sample #18261 was sent on November 21, 2018. A SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYSES

The participants were asked to determine on sample #18260 and sample #18261: Density at 25°C, Dynamic Viscosity at 60°C, Flash Point COC, Fraass Breaking Point, Kinematic Viscosity at 135°C, Penetration at 25°C, Penetration Index, RTFOT at 163°C (Change of Mass, Retained Penetration, Viscosity Ratio and Increase in Softening Point), Softening Point (Ring & Ball) and Solubility in Xylene.

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

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

3   RESULTS

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

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

3.1   STATISTICS

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of 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 and/or Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. 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 these with a factor of 2.8.

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis, the reported 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. ASTM, EN or ISO reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency tests could be used.

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

   z(target) = (test result - average of PT) / target standard deviation

The z(target) scores are listed in the 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 major problems were encountered with the dispatch of the samples. Two participants did not report any test results at all. Not all participants were able to report all analyses requested.

Finally, 37 participants reported in total 511 numerical results. Observed were 15 outlying test results, which is 2.9%. 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 reported test results are discussed per sample and per test. The methods, which are 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 original data. The abbreviations, used in these tables, are listed in appendix 3.

Sample #18260 (grade 70/100)

Density at 25°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 EN15326:07.

Dynamic Viscosity at 60°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN12596:14.

Flash Point COC: 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 ISO2592:17 or ASTM D92:18.

Fraass Breaking Point: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of EN12593:15.

Kinematic Viscosity at 135°C: This determination was problematic depending on the test method used. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of EN12595:14, but is in agreement with the less strict requirements of ASTM D2170/2170M:10.

Penetration: This determination was problematic depending on the test method used. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of EN1426:15, but is in agreement with the less strict requirements of ASTM D5/D5M:13.

When the test results of test method EN1426 and ASTM D5/D5M are evaluated separately, the reproducibility for the EN1426 data is not in

agreement, but the ASTM D5 data is in agreement with the respective method requirements.

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

RTFOT: Four prarameters were determined: Change of Mass, Retained Penetration, Viscosity Ratio and Increase in Softening Point.

For the determination on Change of Mass no statistical outliers were observed. As the calculated reproducibility is not at all in agreement with the requirements of EN12607-1:14 and as the variation in the test results is large (test results between -0.406% and 0.4401%), no z-scores were calculated.

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

The determination on Viscosity Ratio was not problematic. No statistical outliers were observed. Based on only four test results, the calculated reproducibility is in agreement with the requirements of EN12607-1:14.

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

Softening Point (Ring & Ball): This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of EN1427:15 and ASTM D36/D36M:14e1.

Solubility in Xylene: This determination is not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of EN12592:14.

Sample #18261 (grade 35/50)

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

Dynamic Viscosity at 60°C: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of EN12596:14.

Flash Point COC: This determination was very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of ISO2592:17 or ASTM D92:18.

Fraass Breaking Point: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of EN12593:15.

Kinematic Viscosity at 135°C: This determination was problematic depending on the test method used. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of EN12595:14, but is in agreement with the less strict requirements of ASTM D2170/2170M:10.

Penetration: 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 EN1426:15 or ASTM D5/D5M:13. Also when the test results of test method EN1426 and ASTM D5/D5M are evaluated separately, the reproducibility for the EN1426 data and/or the reproducibility for the ASTM D5 data is not at all in agreement with the respective method requirements.

Penetration Index: This determination was not problematic. One statistical outlier was observed and one other test result was excluded. However, the calculated reproducibility after rejection of the suspect data is in agreement with the requirements of EN12591:09.

RTFOT: Four parameters were determined: Change of Mass, Retained Penetration, Viscosity Ratio and Increase in Softening Point.

The determination on Change of Mass may be problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is almost in agreement with the requirements of EN12607-1:14.

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

The determination on Viscosity Ratio was not problematic. No statistical outliers were observed. Based on only four test results, the calculated reproducibility is in agreement with the requirements of EN12607-1:14.

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

Softening Point (Ring & Ball): This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN1427:15 and ASTM D36/D36M:14e1.

Solubility in Xylene: This determination is not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of EN12592:14.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Parameter	unit	n	average	2.8 * sd	R (lit)
Density at 25°C	kg/m^3	26	1014.9	5.7	5
Dynamic Viscosity at 60°C	Pa.s	15	147.2	12.6	14.7
Flash Point COC	°C	23	318	23	18
Fraass Breaking Point	°C	10	-11.2	4.9	6
Kinematic Viscosity at 135°C	mm^2/s	16	359.6	28.8	21.6
Penetration at 25°C	0.1 mm	36	84.6	8.9	5.1
Penetration Index		15	-1.27	0.41	0.5
RTFOT - Change of Mass	%	23	-0.07	0.76	(0.20)
RTFOT - Retained Penetration	%	20	61.7	17.4	10
RTFOT - Viscosity Ratio		4	2.07	0.34	0.41
RTFOT - Increase in Soft. Point	°C	20	4.4	2.1	2.0
Softening Point (Ring & Ball)	°C	35	45.1	1.8	2.0
Solubility in Xylene	%M/M	15	99.88	0.14	0.15

Table 5: reproducibilities of test results on sample #18260 (grade 70/100)

Results between brackets should be used with care

Parameter	unit	n	average	2.8 * sd	R (lit)
Density at 25°C	kg/m^3	28	1022.0	11.0	5
Dynamic Viscosity at 60°C	Pa.s	13	402.6	47.3	40.3
Flash Point COC	°C	20	340	37	18
Fraass Breaking Point	°C	9	-6.9	6.2	6
Kinematic Viscosity at 135°C	mm^2/s	15	525.1	39.6	31.5
Penetration at 25°C	0.1 mm	34	38.8	7.2	2.3
Penetration Index		15	-1.38	0.39	0.5
RTFOT - Change of Mass	%	19	-0.10	0.22	0.20
RTFOT - Retained Penetration	%	16	64.8	14.4	10
RTFOT - Viscosity Ratio		4	2.05	0.45	0.41
RTFOT - Increase in Soft. Point	°C	17	4.6	2.0	2.0
Softening Point (Ring & Ball)	°C	34	51.6	1.7	2.0
Solubility in Xylene	%M/M	13	99.89	0.13	0.15

Table 6: reproducibilities of test results on sample #18261 (grade 35/50)

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

4.3   COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2018 WITH PREVIOUS PTS

	December 2018	December 2017	December 2016	December 2015	December 2014
Number of reporting labs	37	50	51	35	36
Number of results reported	511	289	318	388	464
Number of statistical outliers	15	7	8	30	18
Percentage statistical outliers	2.9%	2.4%	2.5%	7.7%	3.9%

Table 7: 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 test was compared against the requirements of the respective reference test methods. The conclusions are given in the following table:

Parameter	2018		2017	2016	2015		2014	
	#18260	#18261	#17260	#16275	#15255	#15256	#14260	#14261
Density at 25°C	-	--	+/-	-	+	+/-	+/-	-
Dynamic Viscosity at 60°C	+	-	--	+	--	+	+/-	+
Flash Point COC	-	--	--	--	-	+	-	--
Fraass Breaking Point	+	+/-	-	++	+	-	-	--
Kinematic Viscosity at 135°C	-	-	--	--	-	--	--	+/-
Penetration at 25°C	-	--	--	--	--	--	--	--
Penetration Index	+	+	-	--	++	+	--	+
RTFOT - Change of Mass	(--)	+/-	(--)	++	++	++	++	++
RTFOT - Retained Penetration	-	-	+	++	+	-	-	-
RTFOT - Viscosity Ratio	+	+/-	--	-	n.e.	n.e.	(++)	n.e.
RTFOT - Increase in Soft. Point	+/-	+/-	+	+	+	+/-	+/-	+
Softening Point (Ring and Ball)	+	+	-	-	+	+	--	--
Solubility in Xylene	+/-	+	n.e.	n.e.	+	+	+/-	+

Table 8: comparison determinations against the reference test method

Results between brackets should be used with care

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 #18260; results in kg/m^3

lab	method	Temp. (°C)		Density Temp. (kg/m^3)		Density at 25°C	mark	z(targ)	remarks
154	D70	25.0		1015.2	C	1015.2	C	0.16	fr. 1.0152, 1.0152 respectively
168	D70	25.0		1016	C	1016	C	0.61	fr. 1.016, 1.016 respectively
225	D70	15	C	1016	C	1017	C	1.17	reported as sample #18261
332		-----		-----		-----		-----	
333	EN15326	-----		-----		1015	C	0.05	fr. 1.015
335	EN15326	-----		-----		1017.0		1.17	
336		-----		-----		-----		-----	
353		-----		-----		-----		-----	
357	D70	25.0		-----		1012.9		-1.13	
360	EN15326	-----		-----		1014.96		0.02	
396		-----		-----		-----		-----	
398		-----		-----		-----		-----	
399	D70	-----		-----		1018.4		1.95	
444	D70	25		1012.0		1012.0		-1.63	
447		-----		-----		-----		-----	
604	D70	-----		-----		1012.6		-1.30	
657	D70	25.0		1014		1014		-0.51	
865	D70	25.0		1014.5		1014.5		-0.23	
962		-----		-----		-----		-----	
963	D70	25.0		1016		1016		0.61	
1011	D70	25		-----		1019	C	2.29	fr. 1.019
1016	EN15326	150		938.65		1016		0.61	
1026	EN15326	25.0		1013.9		1013.9		-0.57	
1108	EN15326	25.3		1012.4		1012.4		-1.41	
1135	EN15326	25		1014.4		1014.4		-0.29	
1378	D70	-----		-----		1013		-1.07	
1402	ISO3838	-----		-----		1017.2		1.28	
1468	EN15326	25		-----		1018.7		2.12	
1724		-----		-----		-----		-----	
1730		-----		-----		-----		-----	
1849		-----		-----		-----		-----	
1944	EN15326	-----		-----		1015.05		0.08	
6048	EN15326	-----		-----		1013.6		-0.74	
6054		-----		-----		-----		-----	
6076	EN15326	25.0		1013.8		1013.8		-0.62	
6182	D70	-----		-----		1012.2	C	-1.52	fr. 1.012
6219	EN15326	-----		-----		1026	R(0.01)	6.21	
6228		-----		-----		-----		-----	
6229	EN15326	25.0		1013	C	1013	C	-1.07	fr. 1027, 1027 respectively

normality	OK
n	26
outliers	1
mean (n)	1014.92
st.dev. (n)	2.049
R(calc.)	5.74
st.dev.(EN15326:07)	1.786
R(EN15326:07)	5
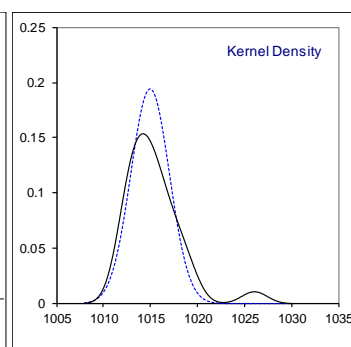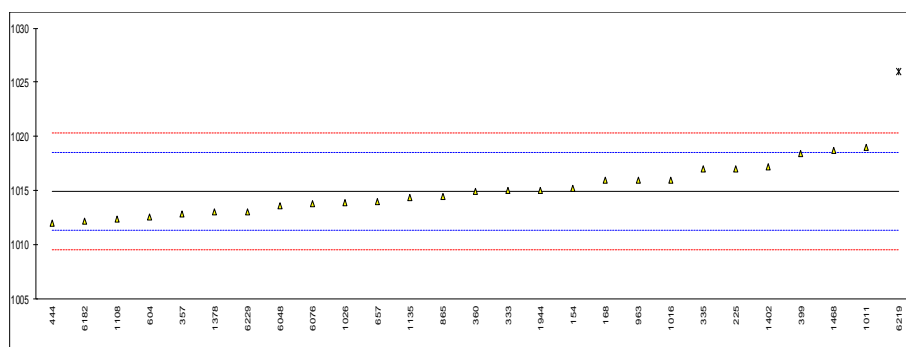
Determination of Dynamic Viscosity at 60°C on sample #18260; results in Pa.s

lab	method	value	mark	z(targ)	remarks
154	D2171	148.0		0.15	
168	D2171	151.0		0.72	
225	EN12596	147.9	C	0.13	reported as sample #18261
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357	EN12596	149.5		0.44	
360		-----		-----	
396	EN12596	143.4	C	-0.72	first reported 183.4
398		-----		-----	
399		-----		-----	
444		-----		-----	
447	EN13302	153		1.10	
604		-----		-----	
657	D2171	148		0.15	
865	D2171	145.5		-0.32	
962		-----		-----	
963		-----		-----	
1011		-----		-----	
1016	EN12596	148.6		0.27	
1026	EN12596	151.0		0.72	
1108		-----		-----	
1135	EN12596	142.9		-0.82	
1378		-----		-----	
1402	EN12596	137		-1.94	
1468	EN12596	153.60		1.22	
1724		-----		-----	
1730		-----		-----	
1849		-----		-----	
1944		-----		-----	
6048		-----		-----	
6054		-----		-----	
6076	EN13302	141.500		-1.08	
6182	D2171	147.1		-0.02	
6219		-----		-----	
6228		-----		-----	
6229		-----		-----	

normality	OK
n	15
outliers	0
mean (n)	147.20
st.dev. (n)	4.511
R(calc.)	12.63
st.dev.(EN12596:14)	5.257
R(EN12596:14)	14.72
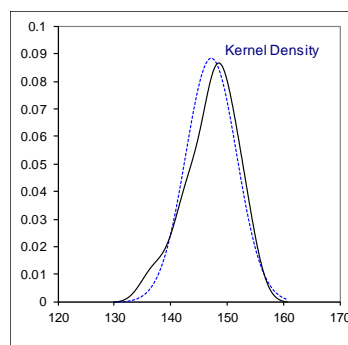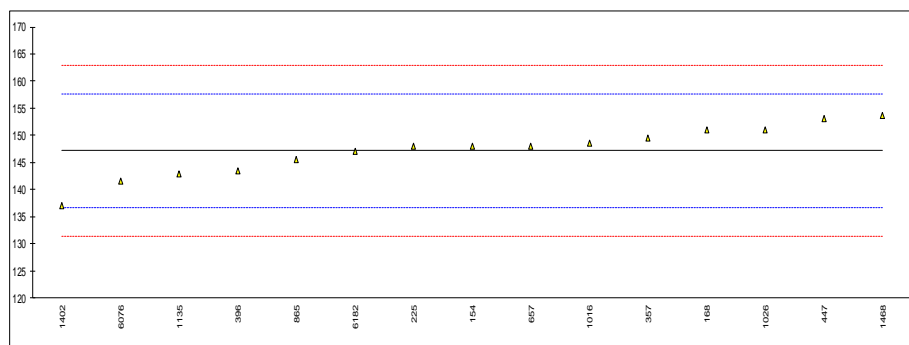
Determination of Flash Point COC on sample #18260; results in °C

lab	method	mode	value	mark	z(targ)	remarks
154	D92	Manual	320		0.27	
168	D92	Manual	316		-0.35	
225		-----	-----		-----	
332		-----	-----		-----	
333	ISO2592	Automated	326		1.21	
335		-----	-----		-----	
336		-----	-----		-----	
353		-----	-----		-----	
357	ISO2592	Automated	327.1		1.38	
360	ISO2592	-----	305		-2.06	
396	D92	Manual	330		1.83	
398		-----	-----		-----	
399		-----	-----		-----	
444		-----	-----		-----	
447		-----	-----		-----	
604	D92	Manual	328		1.52	
657	D92	Manual	324		0.89	
865	D92	Manual	311.6		-1.03	
962	D92	Manual	324		0.89	
963	D92	-----	326		1.21	
1011	ISO2592	Automated	318		-0.04	
1016	ISO2592	Automated	316	C	-0.35	first reported 116
1026	ISO2592	Manual	320		0.27	
1108		-----	-----		-----	
1135	ISO2592	Manual	324.0		0.89	
1378	D92	-----	310		-1.28	
1402	ISO2592	Manual	320		0.27	
1468	ISO2592	Automated	298		-3.15	
1724	D92	Manual	306		-1.91	
1730		-----	-----		-----	
1849	ISO2592	Automated	312		-0.97	
1944	ISO2592	Manual	324		0.89	
6048	ISO2592	Automated	318		-0.04	
6054		-----	-----		-----	
6076	ISO2592	Automated	283	D(0.05)	-5.48	
6182		-----	-----		-----	
6219		-----	-----		-----	
6228		-----	-----		-----	
6229	ISO2592	Automated	316		-0.35	

	normality		OK	
	n		23	
	outliers		1	
	mean (n)		318.25	
	st.dev. (n)		8.189	
	R(calc.)		22.93	
	st.dev.(ISO2592:17)		6.429	
	R(ISO2592:17)		18	
	compare			
	R(D92:18)		18	
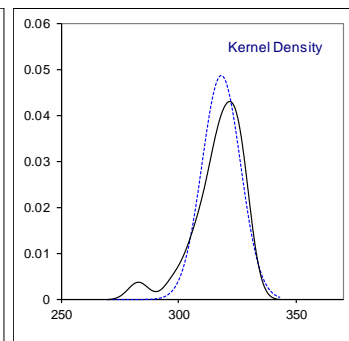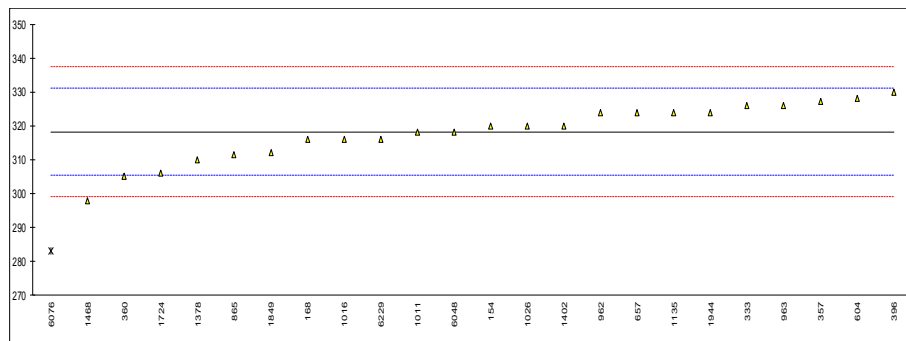
Determination of Fraass Breaking point on sample #18260; results in °C

lab	method	mode	value	mark	z(targ)	remarks
154		-----	-----		-----	
168		-----	-----		-----	
225		-----	-----		-----	
332		-----	-----		-----	
333	EN12593	Automated	-11		0.09	
335		-----	-----		-----	
336		-----	-----		-----	
353		-----	-----		-----	
357	EN12593	Automated	-12		-0.37	
360		-----	-----		-----	
396		-----	-----		-----	
398		-----	-----		-----	
399		-----	-----		-----	
444		-----	-----		-----	
447		-----	-----		-----	
604		-----	-----		-----	
657		-----	-----		-----	
865		-----	-----		-----	
962		-----	-----		-----	
963		-----	-----		-----	
1011	EN12593	Automated	-11		0.09	
1016	EN12593	Automated	-9		1.03	
1026	EN12593	Automated	-9		1.03	
1108		-----	-----		-----	
1135	EN12593	Automated	-11		0.09	
1378		-----	-----		-----	
1402	EN12593	Manual	-2	G(0.05)	4.29	
1468	EN12593	Automated	-20	G(0.05)	-4.11	
1724		-----	-----		-----	
1730		-----	-----		-----	
1849		-----	-----		-----	
1944	EN12593	Manual	-15		-1.77	
6048		-----	-----		-----	
6054		-----	-----		-----	
6076	EN12593	Automated	-12		-0.37	
6182		-----	-----		-----	
6219		-----	-----		-----	
6228	EN12593	Automated	-12		-0.37	
6229	EN12593	Manual	-10		0.56	

normality	suspect
n	10
outliers	2
mean (n)	-11.20
st.dev. (n)	1.751
R(calc.)	4.90
st.dev.(EN12593:15)	2.143
R(EN12593:15)	6
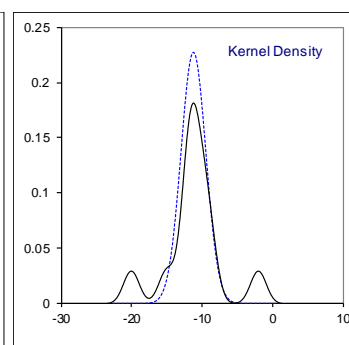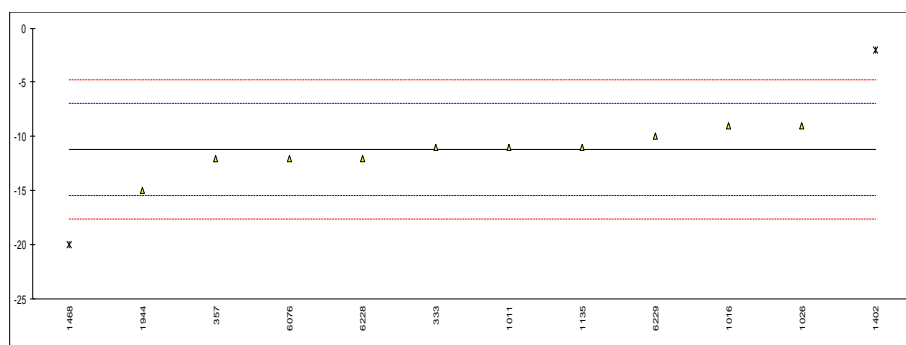
Determination of Kinematic Viscosity at 135°C on sample #18260; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
154	D2170	358		-0.21	
168	D2170	359		-0.08	
225	EN12595	375.3	C	2.04	reported as sample #18261
332		-----		-----	
333	EN12595	371	C	1.48	first reported 531
335		-----		-----	
336		-----		-----	
353		-----		-----	
357	EN12595	347.7		-1.54	
360		-----		-----	
396		-----		-----	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2170	358		-0.21	
865	D2170	374.0		1.87	
962		-----		-----	
963		-----		-----	
1011	EN12595	365		0.70	
1016	EN12595	354.3		-0.69	
1026	EN12595	335.7		-3.10	
1108	EN12595	356.5		-0.40	
1135	EN12595	366.1		0.84	
1378		-----		-----	
1402	EN12595	351		-1.12	
1468	EN12595	353.1		-0.84	
1724		-----		-----	
1730		-----		-----	
1849		-----		-----	
1944		-----		-----	
6048	EN12595	367.1		0.97	
6054		-----		-----	
6076		-----		-----	
6182		-----		-----	
6219		-----		-----	
6228		-----		-----	
6229	EN12595	361.7		0.27	

normality	OK	
n	16	
outliers	0	
mean (n)	359.59	
st.dev. (n)	10.289	
R(calc.)	28.81	
st.dev.(EN12595:14)	7.706	
R(EN12595:14)	21.58	
compare		
R(D2170/D217M:10)	31.64	
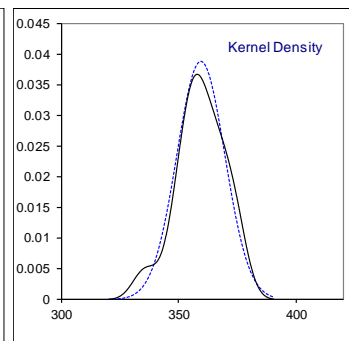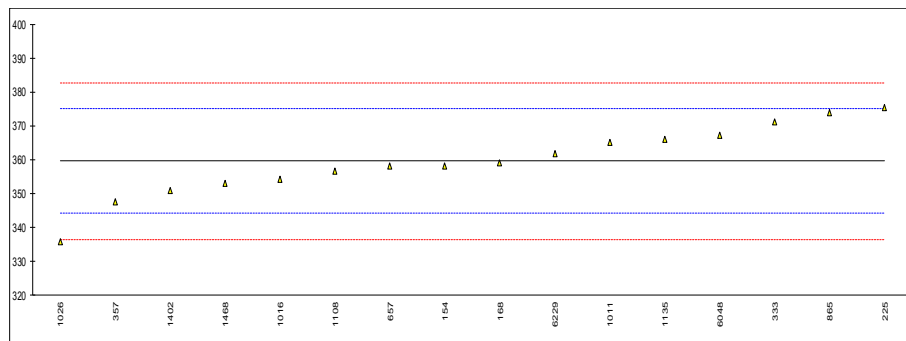
Determination of Penetration at 25°C on sample #18260; results in 0.1 mm

lab	method	value	mark	z(targ)	remarks
154	D5	80.2		-2.40	
168	D5	88		1.90	
225	D5	85	C	0.25	first reported as sample #18261
332		-----		-----	
333	EN1426	82		-1.41	
335	EN1426	86.9		1.30	
336	EN1426	85		0.25	
353	EN1426	86		0.80	
357	EN1426	84		-0.30	
360	EN1426	79.8		-2.62	
396	EN1426	83		-0.86	
398	EN1426	84.7		0.08	
399	EN1426	84		-0.30	
444	EN1426	85.1		0.30	
447	EN1426	84		-0.30	
604	D5	87		1.35	
657	D5	86		0.80	
865	D5	81.7		-1.57	
962		-----		-----	
963	D5	86		0.80	
1011	EN1426	80		-2.51	
1016	EN1426	84		-0.30	
1026	EN1426	84		-0.30	
1108	EN1426	84		-0.30	
1135	EN1426	86		0.80	
1378	D5	83		-0.86	
1402	EN1426	89		2.46	
1468	EN1426	82		-1.41	
1724	D5	83		-0.86	
1730	EN1426	81		-1.96	
1849	EN1426	87		1.35	
1944	EN1426	86		0.80	
6048	EN1426	87		1.35	
6054		-----		-----	
6076	EN1426	86		0.80	
6182	D5	89		2.46	
6219	EN1426	75.4		-5.05	
6228	EN1426	93		4.66	
6229	EN1426	86		0.80	

				EN1426 only	D5/D5M only
normality	suspect			not OK	OK
n	36			26	10
outliers	0			0	0
mean (n)	84.55			84.42	84.89
st.dev. (n)	3.165			3.323	2.844
R(calc.)	8.86			9.31	7.96
st.dev.(EN1426:15)	1.812			1.809	----
R(EN1426:15)	5.07			5.07	----
compare					
R(D5/D5M:13)	10.55			----	10.60
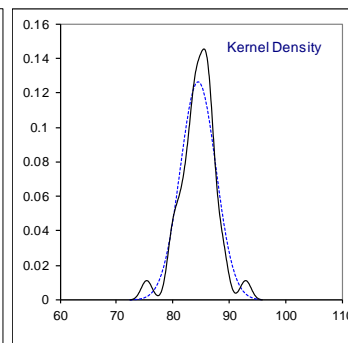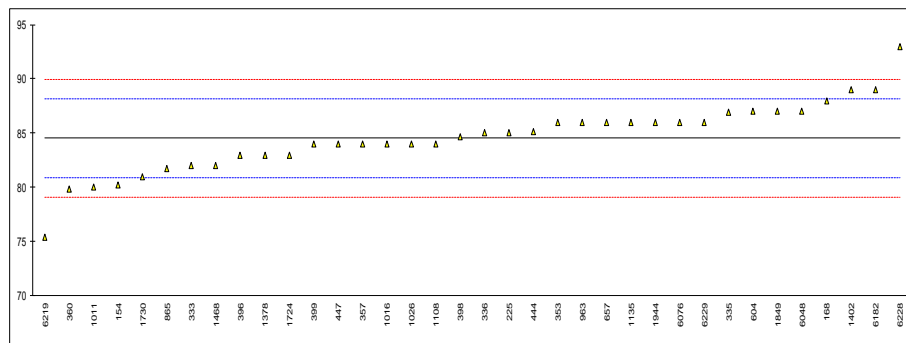
Determination of Penetration Index on sample #18260;

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225	EN12591	-0.23	C,E,D(0.01)	5.80	reported as sample #18261, iis calculated -1.27
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357	EN12591	-1.24		0.15	
360		-----		-----	
396	EN12591	-1.25		0.09	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	Calculation	-1.0		1.49	
865		-----		-----	
962		-----		-----	
963		-----		-----	
1011	EN12591	-1.5		-1.31	
1016	EN12591	-1.4		-0.75	
1026	EN12591	-1.19		0.43	
1108	EN12591	-1.37		-0.58	
1135	EN12591	-1.37		-0.58	
1378	EN12591	-1.2		0.37	
1402	EN12591	-1.3		-0.19	
1468		-----		-----	
1724		-----		-----	
1730		-----		-----	
1849		-----		-----	
1944	EN12591	-1.179		0.49	
6048	EN12591	-1.5		-1.31	
6054		-----		-----	
6076	EN12591	-1.1		0.93	
6182	EN12591	-1.1		0.93	
6219		-----		-----	
6228		-----		-----	
6229	EN12591	-1.3		-0.19	

normality	OK	
n	15	
outliers	1	
mean (n)	-1.267	
st.dev. (n)	0.1454	
R(calc.)	0.407	
st.dev.(EN12591:09)	0.1786	
R(EN12591:09)	0.5	
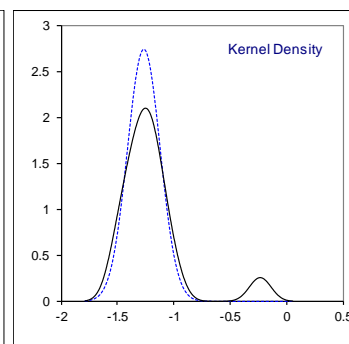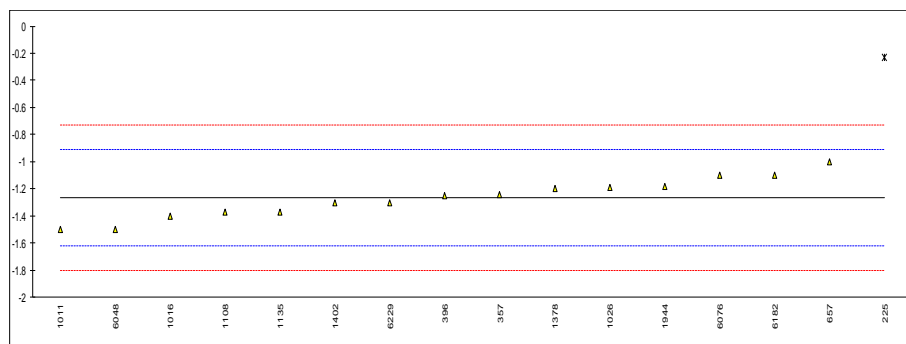
Determination of RTFOT at 163°C, Change of Mass on sample #18260; results in %

lab	method	value	mark	z(targ)	remarks
154	D2872	-0.321		-----	
168	D2872	-0.406		-----	
225	EN12607-1	0.32	C	-----	reported as sample #18261
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357	EN12607-1	-0.165		-----	
360	EN12607-1	-0.255		-----	
396	EN12607-1	-0.22		-----	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2872	-0.26		-----	
865	D2872	-0.291		-----	
962		-----		-----	
963		-----		-----	
1011	EN12607-1	-0.20		-----	
1016	EN12607-1	0.4401		-----	
1026	EN12607-1	-0.19		-----	
1108		-----		-----	
1135	EN12607-1	-0.32		-----	
1378	EN12607-1	-0.243		-----	
1402	EN12607-1	-0.25		-----	
1468	EN12607-1	0.14		-----	
1724	D2872	0.18		-----	
1730		0.32		-----	
1849	EN12607-1	0.43		-----	
1944	EN12607-1	0.068		-----	
6048	EN12607-1	-0.23		-----	
6054		-----		-----	
6076	EN12607-1	0.25		-----	
6182		-----		-----	
6219	EN12607-1	-0.07		-----	
6228		-----		-----	
6229		-0.33		-----	

normality	OK
n	23
outliers	0
mean (n)	-0.0697
st.dev. (n)	0.27122
R(calc.)	0.7594
st.dev.(EN12607-1:14)	(0.07143)
R(EN12607-1:14)	(0.20)
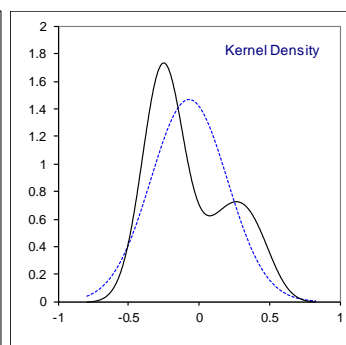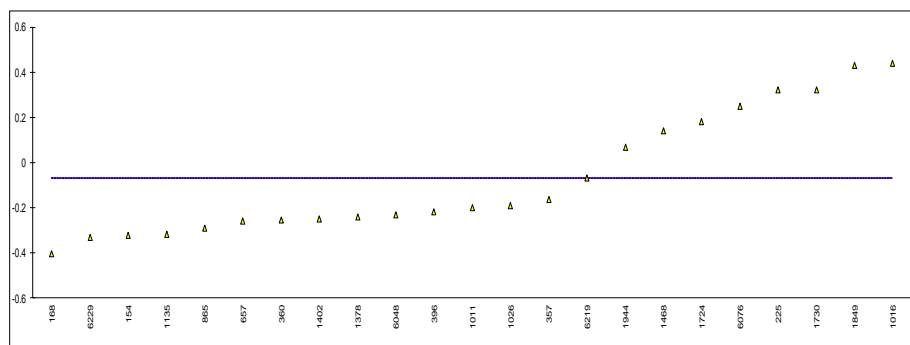
Determination of RTFOT at 163°C, Retained Penetration on sample #18260; results in %

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225	EN12607-1	72.94	C	3.16	reported as sample #18261
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357	EN12607-1	59.5		-0.60	
360	EN12607-1	64.4		0.77	
396	EN12607-1	59.04		-0.73	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2872	60		-0.46	
865		-----		-----	
962		-----		-----	
963		-----		-----	
1011	EN12607-1	67		1.50	
1016	EN12607-1	52	C	-2.70	first reported 44
1026	EN12607-1	65.5		1.08	
1108		-----		-----	
1135	EN12607-1	59		-0.74	
1378	EN12607-1	52.0		-2.70	
1402	EN12607-1	61		-0.18	
1468	EN12607-1	73.2		3.23	
1724	D2872	71		2.62	
1730		53		-2.42	
1849	EN12607-1	56.8		-1.36	
1944	EN12607-1	59.3		-0.66	
6048	EN12607-1	62.0		0.10	
6054		-----		-----	
6076	EN12607-1	65		0.94	
6182		-----		-----	
6219	EN12607-1	58.7		-0.83	
6228		-----		-----	
6229		61.6		-0.01	

normality	OK
n	20
outliers	0
mean (n)	61.65
st.dev. (n)	6.203
R(calc.)	17.37
st.dev.(EN12607-1:14)	3.571
R(EN12607-1:14)	10
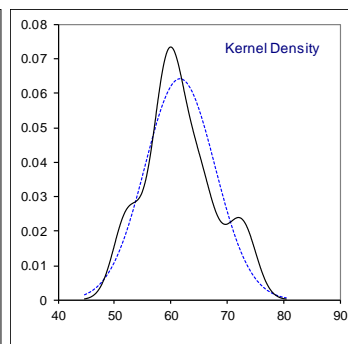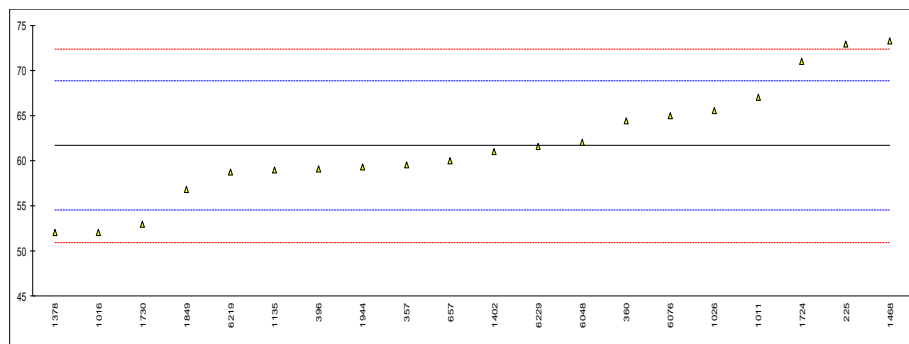
Determination of RTFOT at 163°C, Viscosity Ratio on sample #18260;

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225		-----		-----	
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357	EN12607-1	1.93		-0.93	
360		-----		-----	
396		-----		-----	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2872	2.13		0.42	
865		-----		-----	
962		-----		-----	
963		-----		-----	
1011		-----		-----	
1016		-----		-----	
1026		-----		-----	
1108		-----		-----	
1135	EN12607-1	2.2		0.90	
1378		-----		-----	
1402	EN12607-1	2.01		-0.39	
1468		-----		-----	
1724		-----		-----	
1730		-----		-----	
1849		-----		-----	
1944		-----		-----	
6048		-----		-----	
6054		-----		-----	
6076		-----		-----	
6182		-----		-----	
6219		-----		-----	
6228		-----		-----	
6229		-----		-----	

normality	unknown
n	4
outliers	0
mean (n)	2.068
st.dev. (n)	0.1207
R(calc.)	0.338
st.dev.(EN12607-1:14)	0.1477
R(EN12607-1:14)	0.414
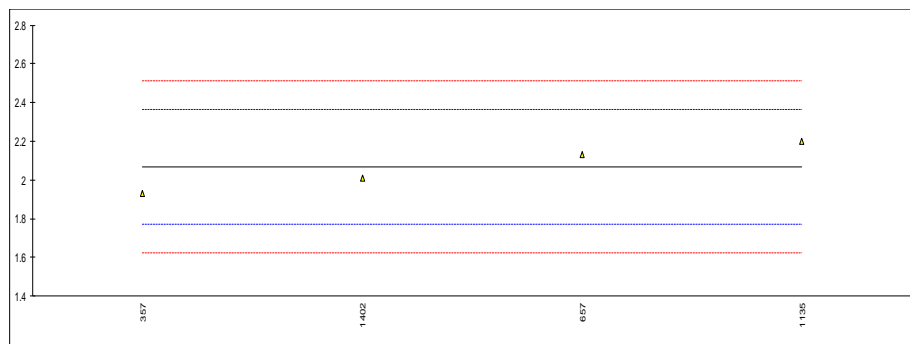
Determination of RTFOT at 163°C, Increase in Softening Point on sample #18260; results in °C

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225	EN12607-1	3.0	C	-2.01	reported as sample #18261
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357	EN12607-1	5.0		0.79	
360	EN12607-1	4.40		-0.05	
396	EN12607-1	4.8		0.51	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2872	4.2		-0.33	
865		-----		-----	
962		-----		-----	
963		-----		-----	
1011	EN12607-1	4		-0.61	
1016	EN12607-1	5.4		1.35	
1026	EN12607-1	4.8		0.51	
1108		-----		-----	
1135	EN12607-1	4.8		0.51	
1378	EN12607-1	4.5		0.09	
1402	EN12607-1	3.8		-0.89	
1468	EN12607-1	3.2		-1.73	
1724	D2872	3.4		-1.45	
1730		5.6		1.63	
1849	EN12607-1	5.2		1.07	
1944	EN12607-1	5.2		1.07	
6048	EN12607-1	4.6		0.23	
6054		-----		-----	
6076	EN12607-1	4.0		-0.61	
6182		-----		-----	
6219	EN12607-1	5.05		0.86	
6228		-----		-----	
6229		3.8		-0.89	

normality	OK	
n	20	
outliers	0	
mean (n)	4.44	
st.dev. (n)	0.743	
R(calc.)	2.08	
st.dev.(EN12607-1:14)	0.714	
R(EN12607-1:14)	2.0	
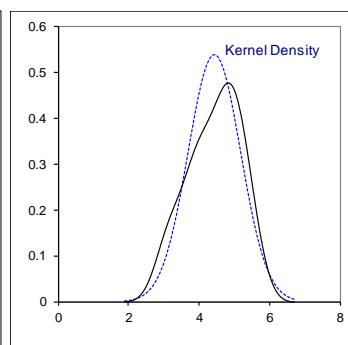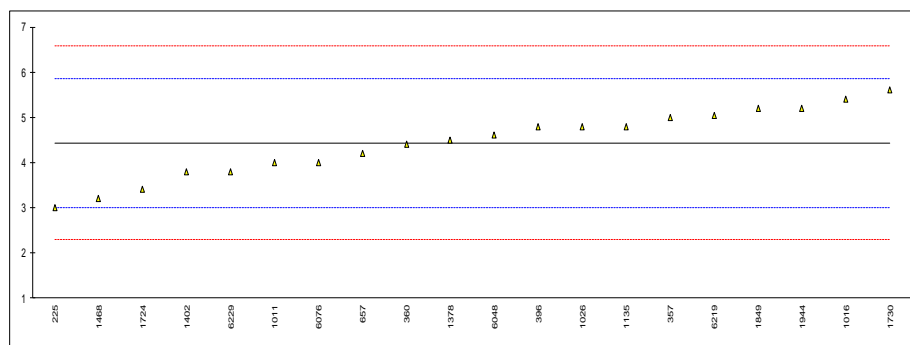
Determination of Softening Point (Ring & Ball) on sample #18260; results in °C

lab	method	value	mark	z(targ)	remarks
154	D36	43.6	C	-2.14	first reported 41.7
168	D36	40.0	C,R(0.01)	-7.18	first reported 38.3
225	D36	45.0	C	-0.18	reported as sample #18261
332		-----		-----	
333	EN1427	44.8		-0.46	
335	EN1427	45.1		-0.04	
336	EN1427	45.6		0.66	
353	EN1427	44.53		-0.84	
357	EN1427	45.2		0.10	
360	EN1427	45.17		0.06	
396	EN1427	45.2		0.10	
398	EN1427	44.8		-0.46	
399	EN1427	45.0		-0.18	
444	EN1427	44.5		-0.88	
447	EN1427	45.4		0.38	
604	D36	45.0	C	-0.18	first reported 48.5
657	D36	45.8		0.94	
865	D36	45.0		-0.18	
962		-----		-----	
963	D36	46.3	C	1.64	first reported 51.6
1011	EN1427	44.8		-0.46	
1016	EN1427	44.8		-0.46	
1026	EN1427	45.4		0.38	
1108	EN1427	44.8		-0.46	
1135	EN1427	44.6		-0.74	
1378	D36	45.5		0.52	
1402	EN1427	44.6		-0.74	
1468	EN1427	45.8		0.94	
1724	D36	45.6		0.66	
1730	EN1427	45.6		0.66	
1849	EN1427	44.2		-1.30	
1944	EN1427	45.2		0.10	
6048	EN1427	44.2		-1.30	
6054		-----		-----	
6076	EN1427	45.6		0.66	
6182	IP58	45.0		-0.18	
6219	EN1427	46.65		2.13	
6228	EN1427	46.4		1.78	
6229	EN1427	44.8		-0.46	

normality	OK	
n	35	
outliers	1	
mean (n)	45.130	
st.dev. (n)	0.6301	
R(calc.)	1.764	
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
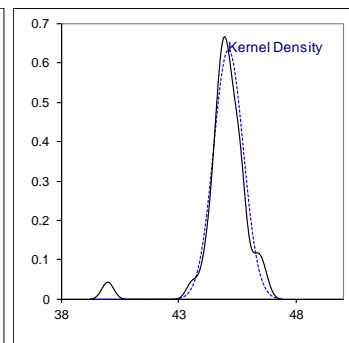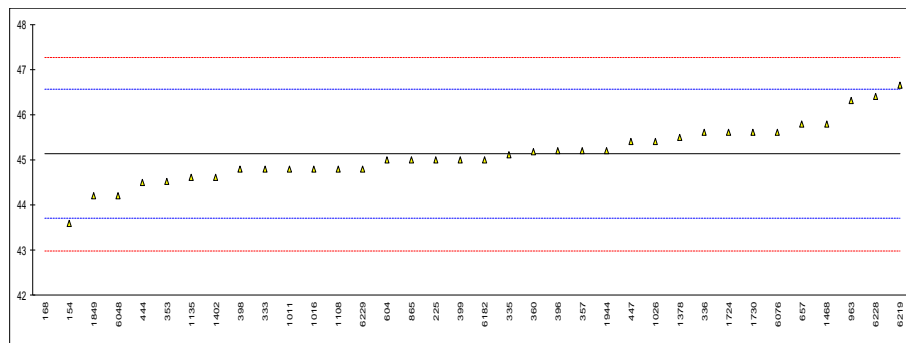
Determination of Solubility in Xylene on sample #18260; results in %M/M

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225		-----		-----	
332		-----		-----	
333	EN12592	99.80		-1.48	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357		-----		-----	
360	EN12592	99.943		1.18	
396		-----		-----	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657		-----		-----	
865	EN12592	99.90		0.38	
962		-----		-----	
963		-----		-----	
1011	EN12592	99.90		0.38	
1016		-----		-----	
1026	EN12592	99.9		0.38	
1108		-----		-----	
1135	EN12592	99.95		1.32	
1378	EN12592	99.9		0.38	
1402	EN12592	99.90		0.38	
1468	EN12592	99.88		0.01	
1724	EN12592	99.81		-1.30	
1730		-----		-----	
1849	EN12592	99.93		0.94	
1944	EN12592	99.90	C	0.38	first reported 99.48
6048	EN12592	99.85		-0.55	
6054		-----		-----	
6076	EN12592	99.8		-1.48	
6182		-----		-----	
6219		-----		-----	
6228		-----		-----	
6229	EN12592	99.83		-0.92	

normality	OK
n	15
outliers	0
mean (n)	99.880
st.dev. (n)	0.0499
R(calc.)	0.140
st.dev.(EN12592:14)	0.0536
R(EN12592:14)	0.15
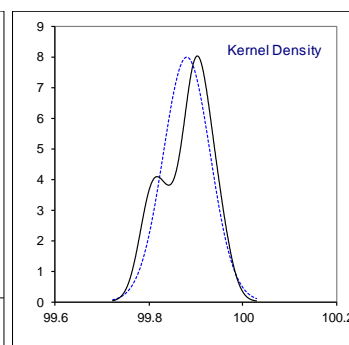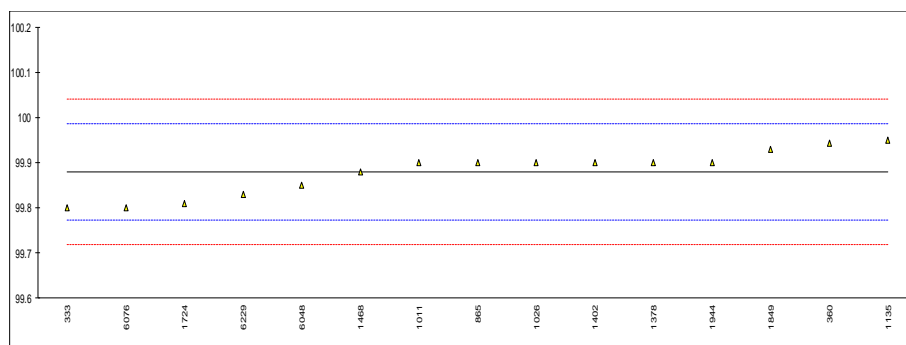
Determination of Density at 25°C on sample #18261; results in kg/m³

lab	method	Temp. (°C)		Density Temp. (kg/m³)		Density at 25°C	mark	z(targ)	remarks
154	D70	25.0		1022.2	C	1022.2	C	0.09	fr. 1.0222, 1.0222 respectively
168	D70	25.0		1019	C	1019	C	-1.70	fr. 1.019, 1.019 respectively
225	D70	15.0	C	1025.6	C	1026	C	2.22	reported as sample #18260
332		-----		-----		-----		-----	
333	EN15326	-----		-----		1024	C	1.10	fr. 1.024
335	EN15326	-----		-----		1025.0		1.66	
336	EN15326	-----		-----		1025	C	1.66	fr. 1.025
353		-----		-----		-----		-----	
357	D70	25.0		-----		1021.9		-0.08	
360	EN15326	-----		-----		1016.54		-3.08	
396	D70	25		1028.4		1028.4		3.56	
398		-----		-----		-----		-----	
399	D70	-----		-----		1013.0		-5.06	
444	D70	25		1021.3		1021.3		-0.41	
447		-----		-----		-----		-----	
604	D70	-----		-----		1021.0		-0.58	
657	D70	25		1021		1021		-0.58	
865	D70	25.0		1020.4		1020.4		-0.92	
962		-----		-----		-----		-----	
963	D70	25.0		1015		1015		-3.94	
1011	D70	25		-----		1020	C	-1.14	fr. 1.020
1016	EN15326	150		946.48		1025		1.66	
1026		-----		-----		-----		-----	
1108	EN15326	24.6		1020.0		1019.4		-1.48	
1135	EN15326	25		1024.7		1024.7		1.49	
1378	D70	-----		-----		1022		-0.02	
1402	ISO3838	-----		-----		1024.2		1.21	
1468	EN15326	25		-----		1026.3		2.39	
1724		-----		-----		-----		-----	
1730		-----		-----		-----		-----	
1849		-----		-----		-----		-----	
1944	EN15326	-----		-----		1022.40		0.20	
6048	EN15326	-----		-----		1021.1		-0.53	
6054		-----		-----		-----		-----	
6076	EN15326	25.0		1021.3		1021.3		-0.41	
6182	D70	-----		-----		1020.0	C	-1.14	fr. 1.1020
6219	EN15326	-----		-----		1032		5.58	
6228		-----		-----		-----		-----	
6229	EN15326	-----		-----		1019		-1.70	

normality		suspect
n		28
outliers		0
mean (n)		1022.04
st.dev. (n)		3.933
R(calc.)		11.01
st.dev.(EN15326:07)		1.786
R(EN15326:07)		5
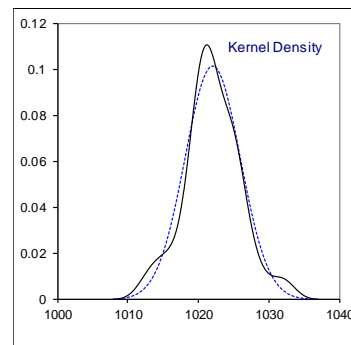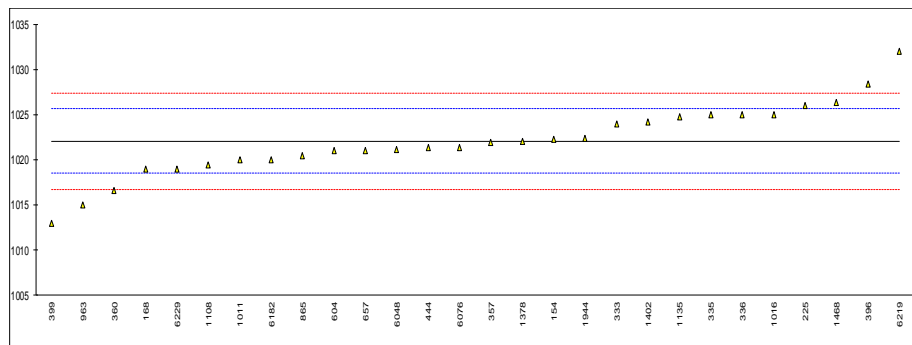
# Determination of Dynamic Viscosity at 60°C on sample #18261; results in Pa.s

lab	method	value	mark	z(targ)	remarks
154	D2171	400.5		-0.15	
168		-----		-----	
225	EN12596	386.9	C	-1.09	reported as sample #18260
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357	EN12596	398.9		-0.26	
360		-----		-----	
396	EN12596	408.8		0.43	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447	EN13302	420.5		1.24	
604		-----		-----	
657	D2171	393		-0.67	
865	D2171	416.9		0.99	
962		-----		-----	
963		-----		-----	
1011		-----		-----	
1016	EN12596	396.8		-0.41	
1026		-----		-----	
1108		-----		-----	
1135	EN12596	407.1		0.31	
1378		-----		-----	
1402	EN12596	407		0.30	
1468	EN12596	436.54		2.36	
1724		-----		-----	
1730		-----		-----	
1849		-----		-----	
1944		-----		-----	
6048		-----		-----	
6054		-----		-----	
6076	EN13302	368.125		-2.40	
6182	D2171	393.2		-0.66	
6219		-----		-----	
6228		-----		-----	
6229		-----		-----	

normality	suspect
n	13
outliers	0
mean (n)	402.64
st.dev. (n)	16.887
R(calc.)	47.28
st.dev.(EN12596:14)	14.380
R(EN12596:14)	40.26
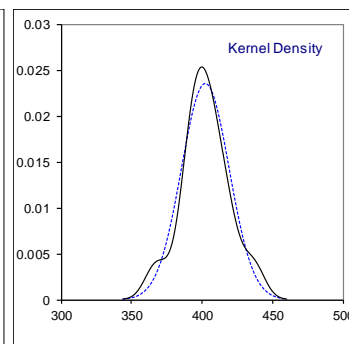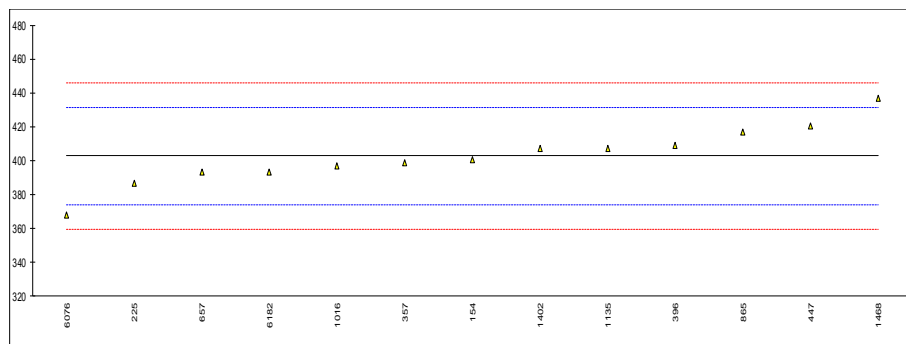
Determination of Flash Point COC on sample #18261; results in °C

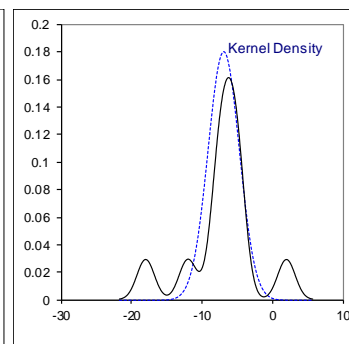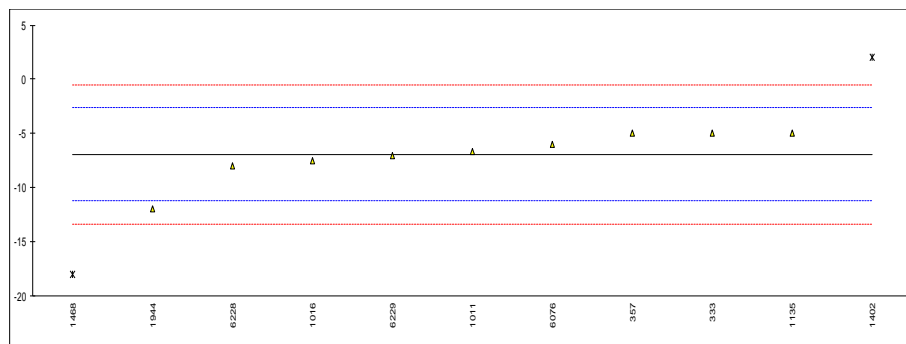| lab	method	mode	value	mark	z(targ)	remarks
154	D92	Manual	336		-0.65	
168	D92	Automated	334		-0.96	
225		-----	-----		-----	
332		-----	-----		-----	
333	ISO2592	Automated	348		1.22	
335		-----	-----		-----	
336		-----	-----		-----	
353		-----	-----		-----	
357		-----	-----		-----	
360	ISO2592	-----	330		-1.58	
396	D92	Manual	338		-0.34	
398		-----	-----		-----	
399		-----	-----		-----	
444		-----	-----		-----	
447		-----	-----		-----	
604	D92	Manual	330		-1.58	
657	D92	Manual	358		2.77	
865	D92	Manual	330.7		-1.48	
962	D92	-----	344		0.59	
963	D92	-----	344		0.59	
1011	ISO2592	Automated	358		2.77	
1016	ISO2592	Automated	366		4.02	
1026		-----	-----		-----	
1108		-----	-----		-----	
1135	ISO2592	Automated	326.0		-2.21	
1378	D92	-----	312		-4.38	
1402	ISO2592	Manual	352	C	1.84	first reported 252
1468	ISO2592	Automated	328		-1.90	
1724	D92	Automated	340		-0.03	
1730		-----	-----		-----	
1849		-----	-----		-----	
1944	ISO2592	Manual	358		2.77	
6048	ISO2592	Automated	335		-0.81	
6054		-----	-----		-----	
6076	ISO2592	Automated	277.3	R(0.01)	-9.78	
6182		-----	-----		-----	
6219		-----	-----		-----	
6228		-----	-----		-----	
6229	ISO2592	-----	336		-0.65	

	normality		OK
	n		20
	outliers		1
	mean (n)		340.18
	st.dev. (n)		13.367
	R(calc.)		37.43
	st.dev.(ISO2592:17)		6.429
	R(ISO2592:17)		18
	compare		
	R(D92:18)		18
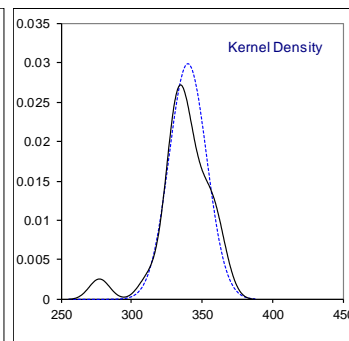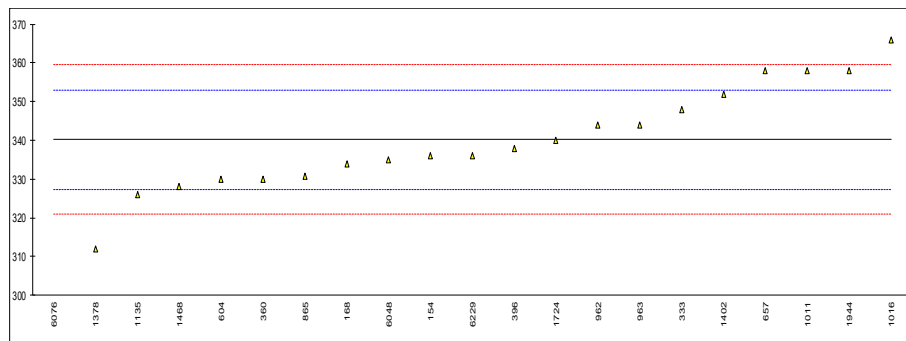
Determination of Fraass Breaking point on sample #18261; results in °C

lab	method	mode	value	mark	z(targ)	remarks
154		-----	-----		-----	
168		-----	-----		-----	
225		-----	-----		-----	
332		-----	-----		-----	
333	EN12593	Automated	-5		0.89	
335		-----	-----		-----	
336		-----	-----		-----	
353		-----	-----		-----	
357	EN12593	Automated	-5		0.89	
360		-----	-----		-----	
396		-----	-----		-----	
398		-----	-----		-----	
399		-----	-----		-----	
444		-----	-----		-----	
447		-----	-----		-----	
604		-----	-----		-----	
657		-----	-----		-----	
865		-----	-----		-----	
962		-----	-----		-----	
963		-----	-----		-----	
1011	EN12593	Automated	-6.7		0.10	
1016	EN12593	Automated	-7.5		-0.27	
1026		-----	-----		-----	
1108		-----	-----		-----	
1135	EN12593	Automated	-5		0.89	
1378		-----	-----		-----	
1402	EN12593	Manual	2	G(0.05)	4.16	
1468	EN12593	Automated	-18	G(0.05)	-5.17	
1724		-----	-----		-----	
1730		-----	-----		-----	
1849		-----	-----		-----	
1944	EN12593	Manual	-12		-2.37	
6048		-----	-----		-----	
6054		-----	-----		-----	
6076	EN12593	Automated	-6		0.43	
6182		-----	-----		-----	
6219		-----	-----		-----	
6228	EN12593	Automated	-8		-0.51	
6229	EN12593	Manual	-7		-0.04	
	normality		not OK			
	n		9			
	outliers		2			
	mean (n)		-6.91			
	st.dev. (n)		2.216			
	R(calc.)		6.20			
	st.dev.(EN12593:15)		2.143			
	R(EN12593:15)		6			

Determination of Kinematic Viscosity at 135°C on sample #18261; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
154	D2170	526.0		0.08	
168	D2170	523		-0.19	
225	EN12595	547.9	C	2.02	reported as sample #18260
332		-----		-----	
333	EN12595	531	C	0.52	first reported 371
335		-----		-----	
336		-----		-----	
353		-----		-----	
357	EN12595	501.2		-2.13	
360		-----		-----	
396		-----		-----	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2170	520		-0.46	
865	D2170	551.1		2.31	
962		-----		-----	
963		-----		-----	
1011	EN12595	531		0.52	
1016	EN12595	522.6		-0.22	
1026		-----		-----	
1108	EN12595	522.2		-0.26	
1135	EN12595	516.0		-0.81	
1378		-----		-----	
1402	EN12595	507		-1.61	
1468	EN12595	509.7		-1.37	
1724		-----		-----	
1730		-----		-----	
1849		-----		-----	
1944		-----		-----	
6048	EN12595	526.6		0.13	
6054		-----		-----	
6076		-----		-----	
6182		-----		-----	
6219		-----		-----	
6228		-----		-----	
6229	EN12595	541.5		1.46	

normality	OK
n	15
outliers	0
mean (n)	525.12
st.dev. (n)	14.146
R(calc.)	39.61
st.dev.(EN12595:14)	11.253
R(EN12595:14)	31.51
compare	
R(D2170/2170M:10)	46.21
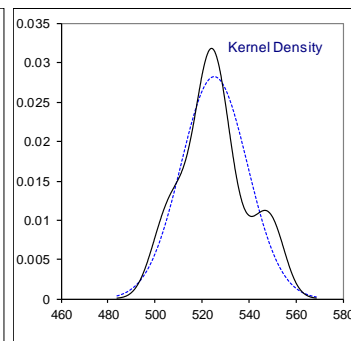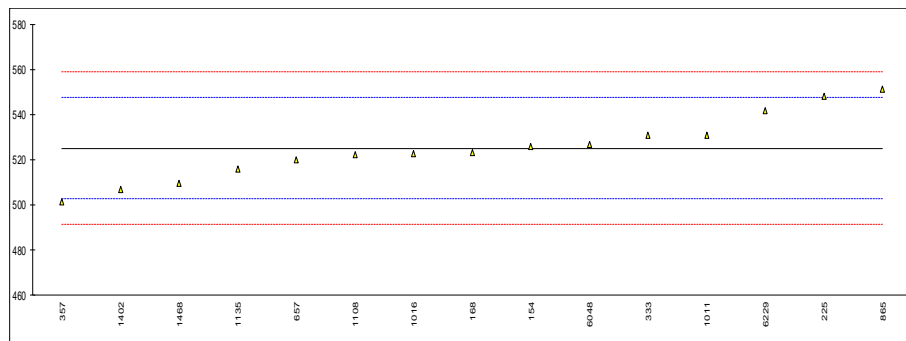
## Determination of Penetration at 25°C on sample #18261; results in 0.1 mm

lab	method	value	mark	z(targ)	remarks
154	D5	34.8		-4.82	
168	D5	40		1.43	
225	D5	36.0	C	-3.38	first reported 371
332		-----		-----	
333	EN1426	36		-3.38	
335	EN1426	37		-2.18	
336	EN1426	38		-0.97	
353	EN1426	42.33		4.23	
357	EN1426	38		-0.97	
360	EN1426	37.0		-2.18	
396	EN1426	39		0.23	
398	EN1426	36		-3.38	
399	EN1426	41		2.63	
444	EN1426	42.3		4.20	
447	EN1426	41		2.63	
604	D5	37		-2.18	
657	D5	39		0.23	
865	D5	39.4		0.71	
962		-----		-----	
963	D5	41		2.63	
1011	EN1426	38		-0.97	
1016	EN1426	38		-0.97	
1026	EN1426	35		-4.58	
1108	EN1426	38		-0.97	
1135	EN1426	26	R(0.01)	-15.40	
1378	D5	35		-4.58	
1402	EN1426	43		5.04	
1468	EN1426	37		-2.18	
1724	D5	38		-0.97	
1730	EN1426	36		-3.38	
1849		-----		-----	
1944	EN1426	42		3.84	
6048	EN1426	40		1.43	
6054		-----		-----	
6076	EN1426	42		3.84	
6182	D5	43		5.04	
6219	EN1426	37.7		-1.33	
6228	EN1426	44		6.24	
6229	EN1426	38		-0.97	

				EN1426 only	D5/D5M only
normality	OK			OK	OK
n	34			24	10
outliers	1			1	0
mean (n)	38.81			39.01	38.32
st.dev. (n)	2.583			2.576	2.670
R(calc.)	7.23			7.21	7.48
st.dev.(EN1426:15)	0.832			0.836	----
R(EN1426:15)	2.33			2.34	----
compare					
R(D5/D5M:13)	4.08			----	4.01
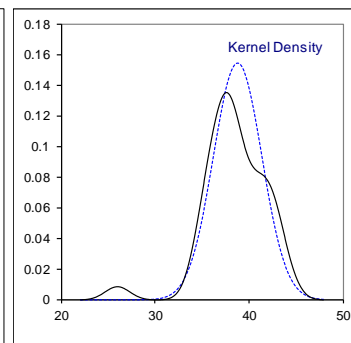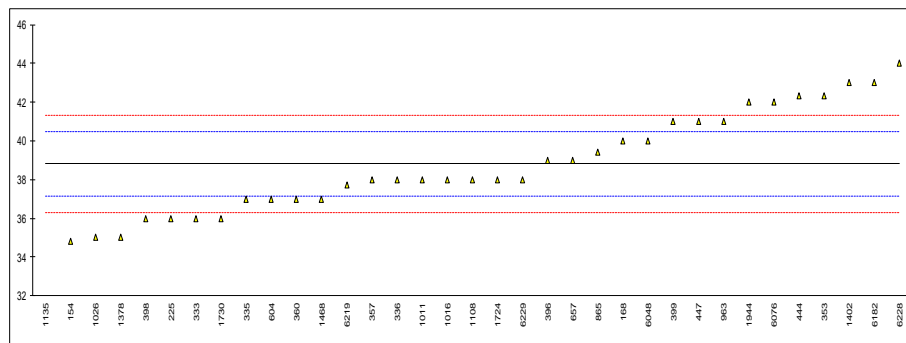
Determination of Penetration Index on sample #18261;

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225	EN12591	-0.06	C,E,D(0.01)	7.39	reported as sample #18260, iis calculated -1.65
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357	EN12591	-1.50		-0.67	
360		-----		-----	
396	EN12591	-1.28		0.56	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	Calculation	-1.3		0.45	
865		-----		-----	
962		-----		-----	
963		-----		-----	
1011	EN12591	-1.5		-0.67	
1016	EN12591	-1.5		-0.67	
1026	EN12591	-1.3		0.45	
1108	EN12591	-1.47		-0.51	
1135	EN12591	-2.33	ex	-5.32	excluded, outlier in Penetration at 25°C
1378	EN12591	-1.5		-0.67	
1402	EN12591	-1.2		1.01	
1468		-----		-----	
1724		-----		-----	
1730		-----		-----	
1849		-----		-----	
1944	EN12591	-1.245		0.75	
6048	EN12591	-1.5		-0.67	
6054		-----		-----	
6076	EN12591	-1.1		1.57	
6182	EN12591	-1.3		0.45	
6219	EN12591	-1.5		-0.67	
6228		-----		-----	
6229	EN12591	-1.5		-0.67	

	normality	OK
	n	15
	outliers	1 (+1 ex)
	mean (n)	-1.380
	st.dev. (n)	0.1381
	R(calc.)	0.387
	st.dev.(EN12591:09)	0.1786
	R(EN12591:09)	0.5
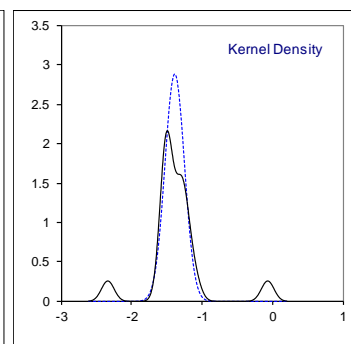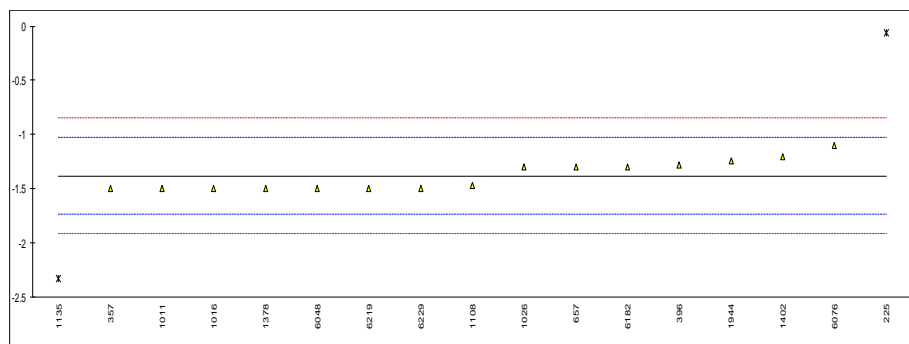
Determination of RTFOT at 163°C, Change of Mass on sample #18261; results in %

lab	method	value	mark	z(targ)	remarks
154	D2872	-0.158		-0.87	
168		-0.202		-1.49	
225	EN12607-1	-0.16	C	-0.90	reported as sample #18260
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357	EN12607-1	-0.041		0.76	
360	EN12607-1	-0.110		-0.20	
396	EN12607-1	-0.043		0.74	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2872	-0.11		-0.20	
865	D2872	-0.121		-0.36	
962		-----		-----	
963		-----		-----	
1011	EN12607-1	-0.11		-0.20	
1016	EN12607-1	0.2304	G(0.05)	4.56	
1026		-----		-----	
1108		-----		-----	
1135	EN12607-1	-0.15		-0.76	
1378	EN12607-1	-0.104		-0.12	
1402	EN12607-1	-0.11		-0.20	
1468	EN12607-1	0.00		1.34	
1724	D2872	0.09		2.60	
1730		-----		-----	
1849		-----		-----	
1944	EN12607-1	0.062		2.21	
6048	EN12607-1	-0.11		-0.20	
6054		-----		-----	
6076	EN12607-1	-0.08		0.22	
6182		-----		-----	
6219	EN12607-1	-0.22		-1.74	
6228		-----		-----	
6229	EN12607-1	-0.14		-0.62	

normality	OK	
n	19	
outliers	1	
mean (n)	-0.0956	
st.dev. (n)	0.08026	
R(calc.)	0.2247	
st.dev.(EN12607-1:14)	0.07143	
R(EN12607-1:14)	0.20	
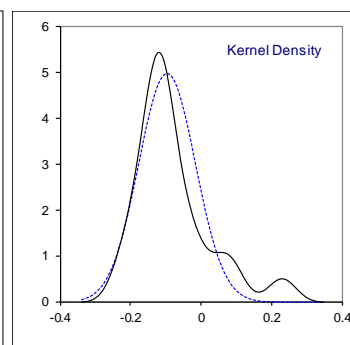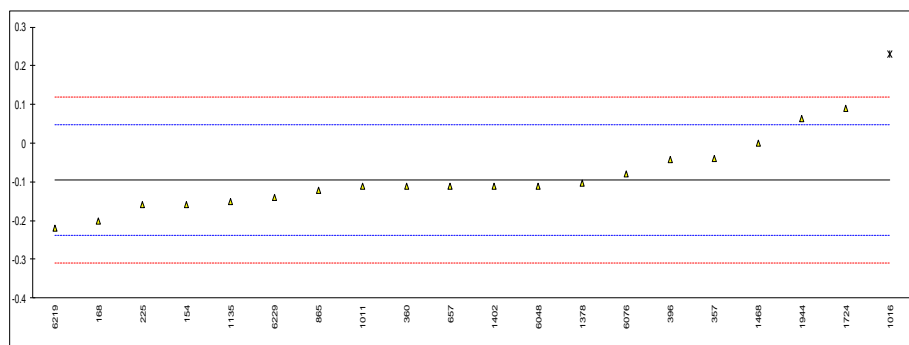
Determination of RTFOT at 163°C, Retained Penetration on sample #18261; results in %
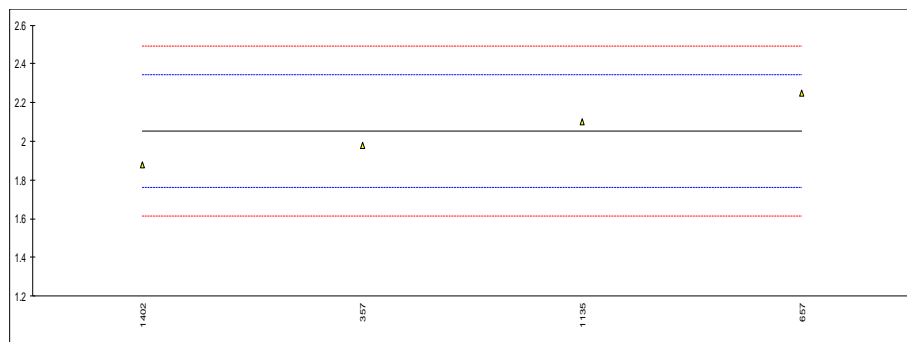
lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225	EN12607-1	83.3	C,DG(0.05)	5.19	reported as sample #18260
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357	EN12607-1	65.8		0.29	
360	EN12607-1	58.1		-1.87	
396	EN12607-1	58.97		-1.62	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2872	69		1.18	
865		-----		-----	
962		-----		-----	
963		-----		-----	
1011	EN12607-1	62		-0.78	
1016	EN12607-1	61	C	-1.06	first reported 23
1026		-----		-----	
1108		-----		-----	
1135	EN12607-1	65		0.06	
1378	EN12607-1	60.0		-1.34	
1402	EN12607-1	67		0.62	
1468	EN12607-1	86.5	DG(0.05)	6.08	
1724	D2872	78		3.70	
1730		67		0.62	
1849		-----		-----	
1944	EN12607-1	69.04		1.20	
6048	EN12607-1	68.0		0.90	
6054		-----		-----	
6076	EN12607-1	63		-0.50	
6182		-----		-----	
6219	EN12607-1	58.6		-1.73	
6228		-----		-----	
6229	EN12607-1	65.8		0.29	

normality	suspect
n	16
outliers	2
mean (n)	64.77
st.dev. (n)	5.149
R(calc.)	14.42
st.dev.(EN12607-1:14)	3.571
R(EN12607-1:14)	10
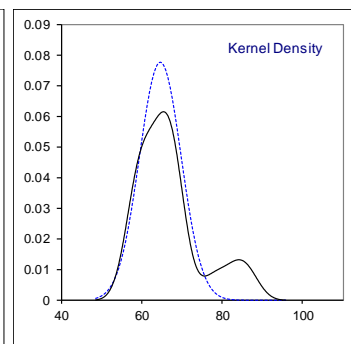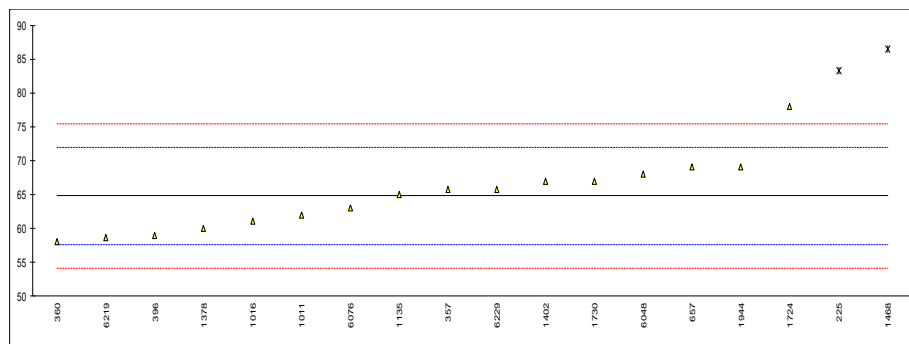
Determination of RTFOT at 163°C, Viscosity Ratio on sample #18261;

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225		-----		-----	
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357	EN12607-1	1.98		-0.49	
360		-----		-----	
396		-----		-----	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2872	2.25		1.35	
865		-----		-----	
962		-----		-----	
963		-----		-----	
1011		-----		-----	
1016		-----		-----	
1026		-----		-----	
1108		-----		-----	
1135	EN12607-1	2.1		0.32	
1378		-----		-----	
1402	EN12607-1	1.88		-1.18	
1468		-----		-----	
1724		-----		-----	
1730		-----		-----	
1849		-----		-----	
1944		-----		-----	
6048		-----		-----	
6054		-----		-----	
6076		-----		-----	
6182		-----		-----	
6219		-----		-----	
6228		-----		-----	
6229		-----		-----	

|	
normality	unknown
n	4
outliers	0
mean (n)	2.053
st.dev. (n)	0.1595
R(calc.)	0.446
st.dev.(EN12607-1:14)	0.1466
R(EN12607-1:14)	0.411

Determination of RTFOT at 163°C, Increase in Softening Point on sample #18261; results in °C

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225	EN12607-1	2.0	C,G(0.05)	-3.59	reported as sample #18260
332		-----		-----	
333		-----		-----	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357	EN12607-1	4.6		0.05	
360	EN12607-1	4.40		-0.23	
396	EN12607-1	5.4		1.17	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657	D2872	4.8		0.33	
865		-----		-----	
962		-----		-----	
963		-----		-----	
1011	EN12607-1	5.7		1.59	
1016	EN12607-1	4.8		0.33	
1026		-----		-----	
1108		-----		-----	
1135	EN12607-1	4.8		0.33	
1378	EN12607-1	5.4		1.17	
1402	EN12607-1	3.6		-1.35	
1468	EN12607-1	3.8		-1.07	
1724	D2872	3.2		-1.91	
1730		4.4		-0.23	
1849		-----		-----	
1944	EN12607-1	4.2		-0.51	
6048	EN12607-1	4.4		-0.23	
6054		-----		-----	
6076	EN12607-1	4.1		-0.65	
6182		-----		-----	
6219	EN12607-1	5.65		1.52	
6228		-----		-----	
6229	EN12607-1	4.4		-0.23	

normality	OK	
n	17	
outliers	1	
mean (n)	4.57	
st.dev. (n)	0.701	
R(calc.)	1.96	
st.dev.(EN12607-1:14)	0.714	
R(EN12607-1:14)	2.0	
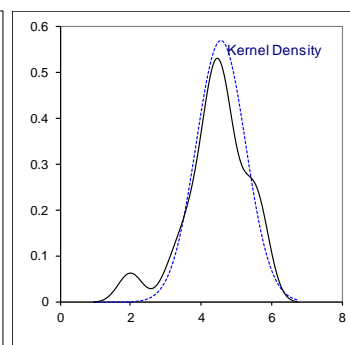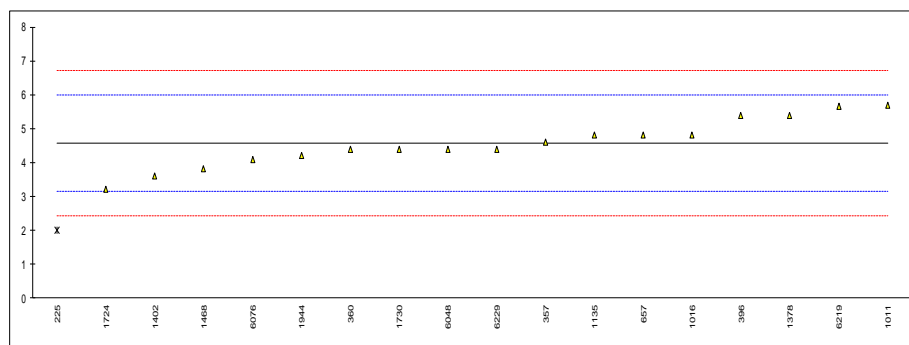
Determination of Softening Point (Ring & Ball) on sample #18261; results in °C

lab	method	value	mark	z(targ)	remarks
154	D36	50.8	C	-1.10	first reported 49.4
168		-----		-----	
225	D36	51.0	C	-0.82	reported as sample #18260
332		-----		-----	
333	EN1427	51.2		-0.54	
335	EN1427	51.45		-0.19	
336	EN1427	51.6		0.02	
353	EN1427	50.7		-1.24	
357	EN1427	51.2		-0.54	
360	EN1427	51.3		-0.40	
396	EN1427	51.8		0.30	
398	EN1427	51.0		-0.82	
399	EN1427	51.4		-0.26	
444	EN1427	52.0		0.58	
447	EN1427	52.4		1.14	
604	D36	52.4		1.14	
657	D36	52.0		0.58	
865	D36	51.2		-0.54	
962		-----		-----	
963	D36	52.2	C	0.86	first reported 68.0
1011	EN1427	51.2		-0.54	
1016	EN1427	51.4		-0.26	
1026	EN1427	52.6		1.42	
1108	EN1427	51.3		-0.40	
1135	EN1427	50.6		-1.38	
1378	D36	51.7		0.16	
1402	EN1427	51.2		-0.54	
1468	EN1427	52.4		1.14	
1724	D36	52.8		1.70	
1730	EN1427	52.2		0.86	
1849		-----		-----	
1944	EN1427	51.4		-0.26	
6048	EN1427	50.8		-1.10	
6054		-----		-----	
6076	EN1427	52.2		0.86	
6182	IP58	51.0		-0.82	
6219	EN1427	51.4		-0.26	
6228	EN1427	52.8		1.70	
6229	EN1427	51.2		-0.54	
	normality	OK			
	n	34			
	outliers	0			
	mean (n)	51.584			
	st.dev. (n)	0.6212			
	R(calc.)	1.739			
	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		
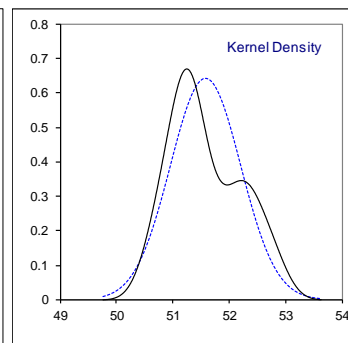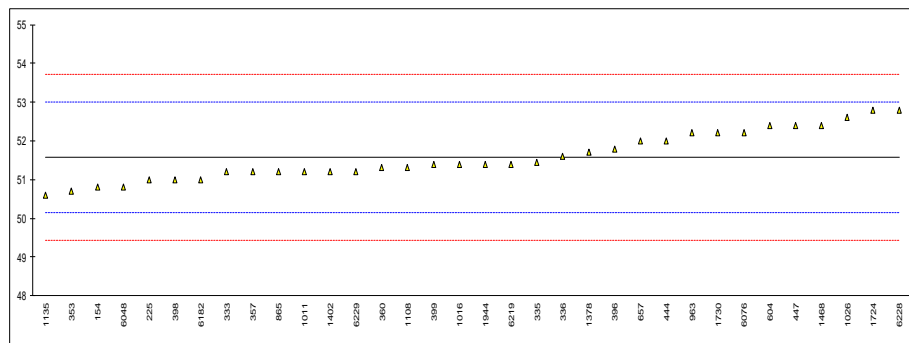
Determination of Solubility in Xylene on sample #18261; results in %M/M

lab	method	value	mark	z(targ)	remarks
154		-----		-----	
168		-----		-----	
225		-----		-----	
332		-----		-----	
333	EN12592	99.85		-0.67	
335		-----		-----	
336		-----		-----	
353		-----		-----	
357		-----		-----	
360	EN12592	99.958		1.34	
396		-----		-----	
398		-----		-----	
399		-----		-----	
444		-----		-----	
447		-----		-----	
604		-----		-----	
657		-----		-----	
865	EN12592	99.93		0.82	
962		-----		-----	
963		-----		-----	
1011	EN12592	99.80		-1.61	
1016		-----		-----	
1026		-----		-----	
1108		-----		-----	
1135	EN12592	99.89		0.07	
1378	EN12592	99.9		0.26	
1402	EN12592	99.90		0.26	
1468	EN12592	99.92		0.63	
1724	EN12592	99.9		0.26	
1730		-----		-----	
1849		-----		-----	
1944	EN12592	99.92	C	0.63	first reported 99.57
6048	EN12592	99.90		0.26	
6054		-----		-----	
6076	EN12592	99.8		-1.61	
6182		-----		-----	
6219		-----		-----	
6228		-----		-----	
6229	EN12592	99.85	C	-0.67	first reported 99.65

normality	OK	
n	13	
outliers	0	
mean (n)	99.886	
st.dev. (n)	0.0480	
R(calc.)	0.134	
st.dev.(EN12592:14)	0.0536	
R(EN12592:14)	0.15	
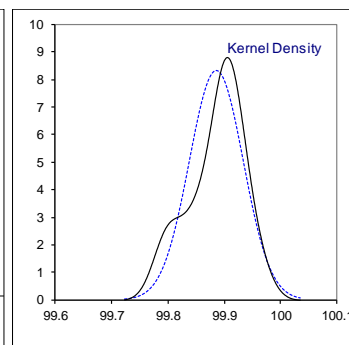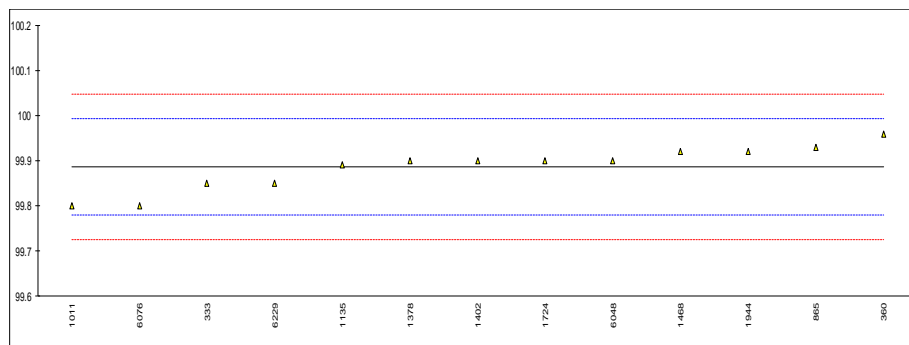
APPENDIX 2

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 FINLAND
4 labs in FRANCE
1 lab in GERMANY
3 labs in GREECE
1 lab in HONG KONG
1 lab in IRELAND
4 labs in ITALY
1 lab in MALAYSIA
5 labs in NETHERLANDS
1 lab in PORTUGAL
1 lab in ROMANIA
2 labs in SAUDI ARABIA
1 lab in SINGAPORE
2 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
E	= possibly an error in calculations
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
SDS	= safety data sheet

Literature:

1   iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018

2   W. Horwitz and R. Albert, J. AOAC Int., 79, 3, 589, (1996)

3   ASTM E178:02

4   ASTM E1301:03

5   ISO13528:05

6   ISO 5725:86

7   ISO 5725, parts 1-6, 1994

8   M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)

9   W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)

10   IP 367:84

11   DIN 38402 T41/42

12   P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)

13   J.N. Miller, Analyst, 118, 455, (1993)

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

15   P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)

16   Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), 165-172, (1983)