Results of Proficiency Test
Turbine Oil
March 2013

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. R.J. Starink
Correctors: dr. R.G. Visser & ing. L. Sweere

Report no.: iis13L10

May 2013

CONTENTS

1 INTRODUCTION 3

2 SET UP 3

2.1 ACCREDITATION 3

2.2 PROTOCOL 3

2.3 CONFIDENTIALITY STATEMENT 3

2.4 SAMPLES 4

2.5 ANALYSES 4

3 RESULTS 5

3.1 STATISTICS 5

3.2 GRAPHICS 6

3.3 Z-SCORES 6

4 EVALUATION 7

4.1 EVALUATION PER TEST 7

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES 9

Appendices:

1. Data and statistical results 10
2. Number of participants per country 24
3. Abbreviations and literature 25

1 INTRODUCTION

On request of several participants, the Institute for Interlaboratory Studies decided to organise a new proficiency test for the analysis of Turbine Oil during the annual proficiency testing program 2012/2013. In this interlaboratory study 29 laboratories in 15 different countries have participated. See appendix 2 for the number of participants per country. In this report, the results of the 2013 Turbine Oil proficiency test 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 organizer of this proficiency test. It was decided to send one bottle of 1L (labelled #13068) of used Turbine Oil that was provided by a third party. The analyses for fit-for-use and homogeneity were subcontracted. Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC 17043:2010, since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organization, Statistics and Evaluation' of January 2010 (iis-protocol, version 3.2), which can be downloaded from 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

The necessary bulk material was provided by a third party. The 60 litre bulk material was transferred after homogenizing into 60 brown glass bottles of 1 litre (labelled #13068). The homogeneity of the subsamples #13068 was checked by determination of Density @15°C in accordance with ASTM D4052:11 on 8 stratified randomly selected samples.

	Density @ 15 °C in kg/m^3
Sample #13068-1	868.38
Sample #13068-2	868.36
Sample #13068-3	868.36
Sample #13068-4	868.37
Sample #13068-5	868.37
Sample #13068-6	868.38
Sample #13068-7	868.37
Sample #13068-8	868.38

Table 1: homogeneity test results of subsamples #13068

From the above test results, the repeatability was calculated and compared with 0.3 times the corresponding reproducibilities in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Density @ 15 °C in kg/L
r (sample #13068)	0.00002
reference test	ASTM D1298:12b
0.3 x R(reference test)	0.00045

Table 2: evaluation of the repeatability of the subsamples #13068

The calculated repeatability is less than 0.3 times the corresponding reproducibility of the reference method. Therefore, homogeneity of the subsamples #13068 was assumed.

To each of the participating laboratories, 1 sample of 1 L in a brown glass bottle (labelled #13068) was sent on March 20, 2013.

2.5 ANALYSES

The participants were requested to determine on sample #13068: Total Acidity, Base Number, Acid Number, Color ASTM, Density @ 15°C, Flash Point PMcc, Kinematic Viscosity @ 40°C and @ 100°C, Viscosity Index, Level of contamination, Water, Water separability, Foaming Characteristics (at 5 min end point and at 10 min end point).
To get comparable results a detailed report form, on which the units were prescribed as well as some of the required standards and a letter of instructions were prepared and 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 gathered. The original data are tabulated per determination in the appendix of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder fax was sent to those laboratories that had not reported 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 data analysis and 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’ (iis-protocol, version 3.2) of January 2010. 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. 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, by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

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

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported 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 "x". Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. The Kernel Density 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.12 and 13).

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 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 the fit-for-useness of the reported test result.
The z-scores were calculated according to:

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

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

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

$\lvert z \rvert < 1$	good
$1 < \lvert z \rvert < 2$	satisfactory
$2 < \lvert z \rvert < 3$	questionable
$3 < \lvert z \rvert$	unsatisfactory

4 EVALUATION

In this interlaboratory study, some problems were encountered with the dispatch of the samples to laboratories in Norway, Peru and Spain. Six participants reported after the final reporting date and two participants did not report any test results at all. Not all laboratories were able to report all analyses requested. In total 27 participants reported 336 test results. Observed were 14 outlying results, which is 4.2% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

Not all original data sets proved to have a normal distribution. Non-Gaussian distributions were found for the following determinations: Base Number, Color ASTM, Density @ 15°C and Kinematic Viscosity @ 100ºC. In these cases the statistical evaluation should be used with due care.

4.1 EVALUATION PER TEST

In this section, the results are discussed per test. The methods, which are used by the various laboratories, are taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the original data. The abbreviations, used in these tables, are listed in appendix 3.

Total Acidity: This determination was not problematic. Only one statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of ASTM D974:12.

Acid Number: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D664:11a.

Base Number (total): This determination was very problematic. One statistical outlier was observed. The test results vary over a large range: 0.019 – 0.47 mg KOH/g and may be bimodally divided. Three laboratories reported to have used procedure A and four reported to have used procedure B (resp. 0.086 mg KOH/g for proc. A vs 0.364 mg KOH/g for proc. B), but the number of reported test results is too small to drawn any significant conclusions.

Color ASTM: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in full agreement with ASTM D1500:12.

Density @ 15°C: This determination was very problematic. Only one statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of ASTM D1298:12b. The large spread may be explained by the assumption that a number of laboratories possibly did not report density as requested, but specific gravity or apparent density instead.

Flash Point PMcc: This determination was problematic. Two statistical outliers were observed and the calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D93:12 procedure B.

Kin.Visco.@ 40°C: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D445:12.

Kin.Visco.@ 100°C: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D445:12.

Viscosity Index This determination was very problematic. No statistical outliers and/or calculation errors were observed. However, the calculated reproducibility is not at all in agreement with the strict requirements of ASTM D2270:10e1.

Level of Cont: No statistical conclusions were drawn as the reported test results are categorized. However, it is observed that the reported categories for contamination for each of the three levels vary over a large range. For ≥4µm, 17-23 (640/1300 – 40000/80000 particles), for ≥6µm, 14-20 (80/160 – 5000/10000 particles) and for ≥14µm, 9-14 (2.5/5 – 80/160 particles).

Water: This determination was not problematic. The preferred method to use for a product containing interfering components may be ASTM D6304:07 method C. This method is applicable for oils with difficult matrix interferences only. Almost all laboratories, except seven, reported results determined according ASTM D6304 method C. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D6304:07.

Water separability Regretfully, only for Time measurement statistical data is available. This determination was not problematic. One statistical outlier was observed and the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D1401:12. For the other reported test results no significant conclusion was drawn.

Foaming Characteristics: The Foaming characteristics can be divided into three groups, Sequence I, II and III. In total five statistical outliers were observed. The calculated reproducibilities after rejection of the statistical outliers are for all three Sequences not in agreement with the requirements of ASTM D892:11a.

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, calculated reproducibilities and reproducibilities derived from literature standards (in casu ASTM and IP standards), are compared in the next table.

Parameter	unit	n	Average	2.8 * sd	R(lit)
Total Acidity	mg KOH/g	11	0.072	0.040	0.040
Total Acid Number	mg KOH/g	16	0.091	0.049	0.040
Total Base Number	mg KOH/g	6	0.086/0.364	0.205/0.257	0.006/0.025
Color ASTM		12	3.9	0.8	1.0
Density @ 15 °C	kg/m^3	23	868.8	2.8	1.5
Flash Point PMcc	^oC	20	224.2	11.9	10.0
Kinematic Viscosity @ 40 °C	mm^2/s	27	46.720	1.188	2.189
Kinematic Viscosity @ 100 °C	mm^2/s	26	6.939	0.306	0.598
Viscosity Index		23	103.8	9.8	2.0
Level of Contamination		16/19/19	20/16/12	n.a.	n.a.
Water by KF	mg/kg	22	16.6	22.5	91.1
Water Separability, o-w-e (t)	min	5	11.2	11.6	20
Foaming Charac. Seq I, 5 min	ml	8	466.3	250.4	222.0
Foaming Charac. Seq II, 5 min	ml	9	28.9	34.8	25.2
Foaming Charac. Seq III, 5 min	ml	8	413.1	250.8	198.1
Foaming Charac. Seq I, 10 min	ml	8	0	n.a.	n.a.
Foaming Charac. Seq II, 10 min	ml	8	0	n.a.	n.a.
Foaming Charac. Seq III, 10min	ml	8	0	n.a.	n.a.

Table 3: reproducibilities of results of sample #13068

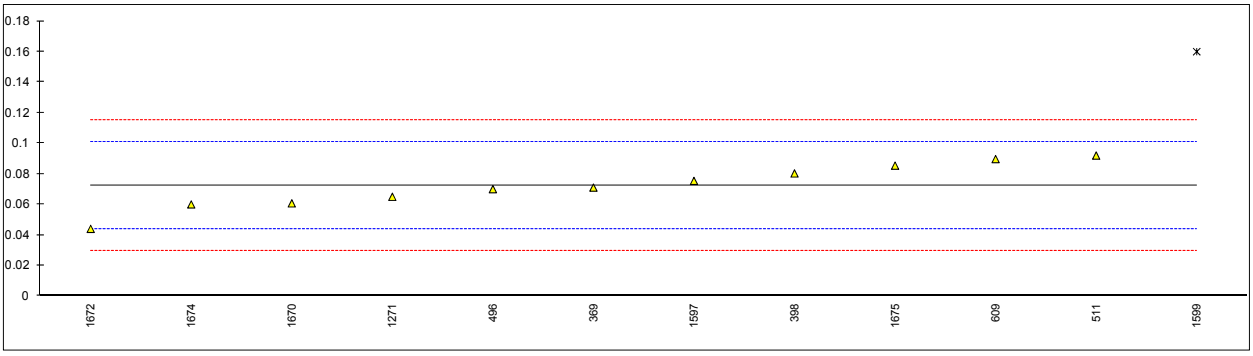
Without further statistical calculations it can be concluded that for a number of tests there is a good compliance of the group of participants with the relevant standards. The tests that are problematic have been discussed in paragraph 4.1.

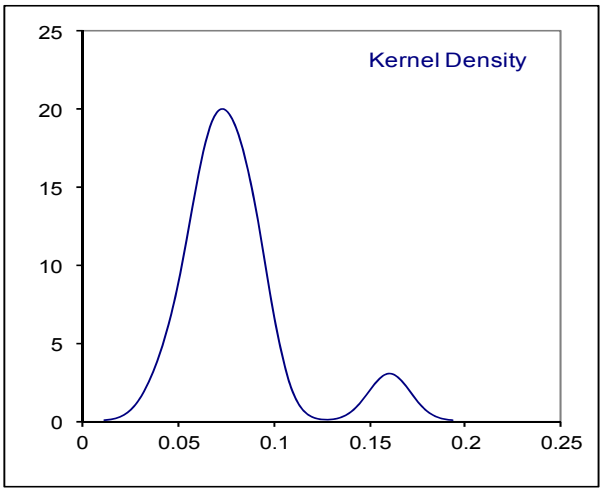
APPENDIX 1

Determination of Total Acidity on sample #13068; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
150		-----		-----	
228		-----		-----	
323		-----		-----	
331		-----		-----	
369	D974	0.071		-0.08	
398	D974	0.0803		0.57	
495		-----		-----	
496	D974	0.070		-0.15	
511	D974	0.092		1.39	
609	D974	0.0897		1.23	
657		-----		-----	
1023		-----		-----	
1146		-----		-----	
1271	D974	0.065		-0.50	
1435		-----		-----	
1596		-----		-----	
1597	INH-6070	0.0754		0.23	
1599	D664	0.160	G(0.01)	6.15	
1604		-----		-----	
1665		-----		-----	
1666		-----		-----	
1670	D974	0.0607		-0.80	
1671		-----		-----	
1672	D974	0.044		-1.97	
1673		-----		-----	
1674	D974	0.06		-0.85	
1675	D974	0.0854	C	0.93	First reported 0.854
1827		-----		-----	
1943		-----		-----	

normality	OK
n	11
outliers	1
mean (n)	0.0721
st.dev. (n)	0.01443
R(calc.)	0.0404
R(D974:12)	0.0400

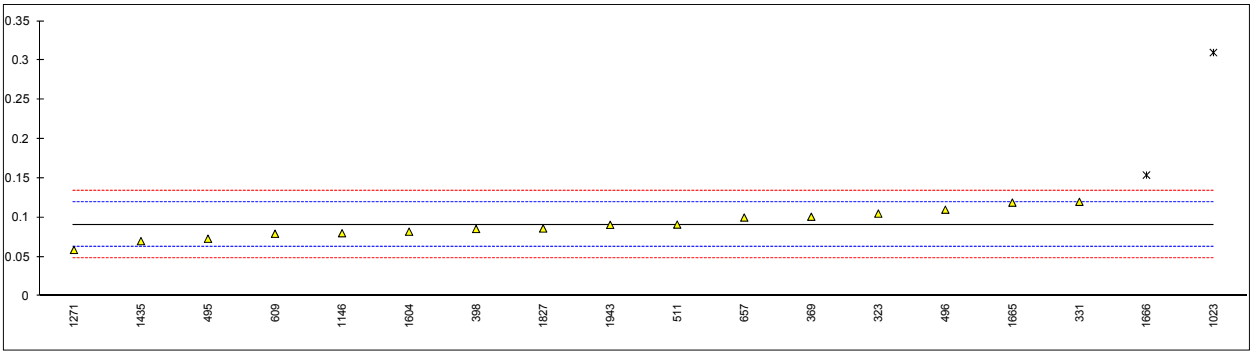


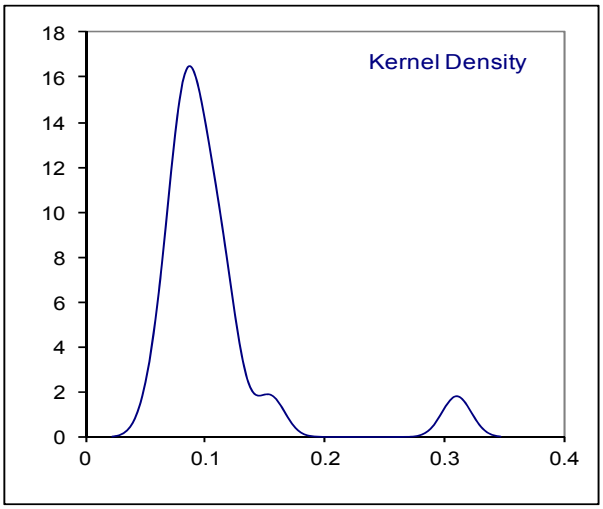


Determination of Acid Number on sample #13068; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
150		-----		-----	
228		-----		-----	
323	D664	0.105		1.00	
331	D664A	0.12		2.06	
369	D664A	0.101		0.72	
398	D664A	0.0854		-0.37	
495	D664A	0.073		-1.24	
496	D664A	0.11		1.35	
511	D664A	0.091		0.02	
609	D664A	0.0793		-0.80	
657	D664A	0.10		0.65	
1023	in house	0.31	G(0.01)	15.39	
1146	D664A	0.080		-0.75	
1271	D664A	0.0587		-2.24	
1435	D664A	0.07		-1.45	
1596		-----		-----	
1597		-----		-----	
1599		-----		-----	
1604	D664A	0.082		-0.61	
1665	D664A	0.119		1.99	
1666	D664A	0.154	G(0.05)	4.44	
1670		-----		-----	
1671		-----		-----	
1672		-----		-----	
1673		-----		-----	
1674		-----		-----	
1675		-----		-----	
1827	D664A	0.086		-0.33	
1943	ISO6618	0.0907		0.00	

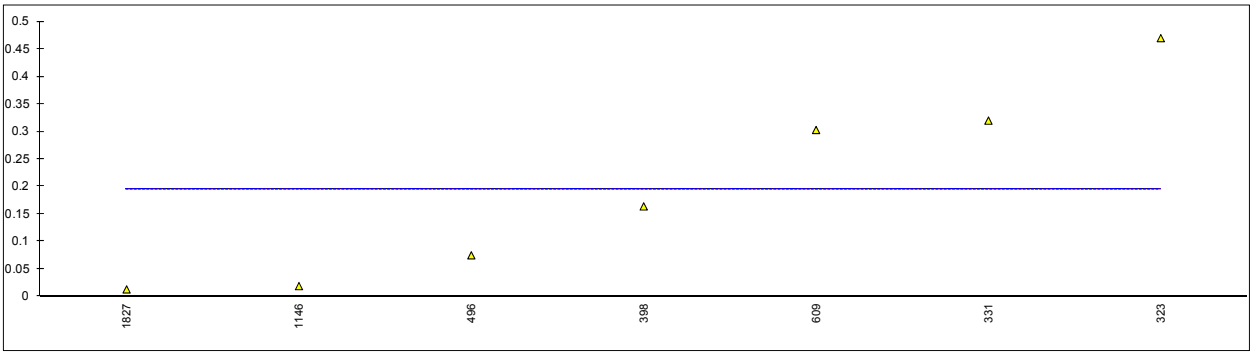
normality	OK
n	16
outliers	2
mean (n)	0.0907
st.dev. (n)	0.01745
R(calc.)	0.0488
R(D664:11a)	0.0399





Determination of Base Number on sample #13068; results in mg KOH/g

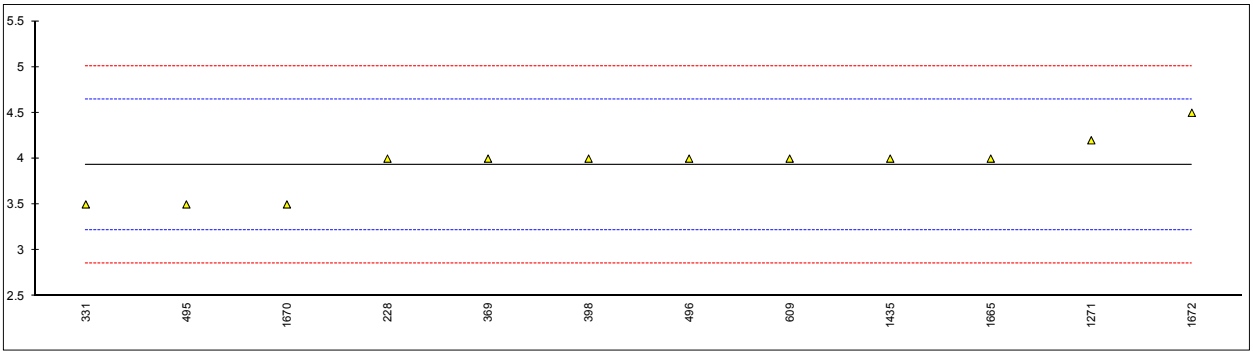
lab	method	value	mark	z(targ)	remarks
150		-----		-----	
228		-----		-----	
323	D2896B	0.47		-----	
331	D2896B	0.32		-----	
369		-----		-----	
398	D2896A	0.164		-----	
495		-----		-----	
496	D2896A	0.075		-----	
511		-----		-----	
609	D2896B	0.3029		-----	
657		-----		-----	
1023		-----		-----	
1146	D2896A	0.019		-----	
1271		-----		-----	
1435		-----		-----	
1596		-----		-----	
1597		-----		-----	
1599		-----		-----	
1604		-----		-----	
1665		-----		-----	
1666		-----		-----	
1670		-----		-----	
1671		-----		-----	
1672		-----		-----	
1673		-----		-----	
1674		-----		-----	
1675		-----		-----	
1827	D2896B	0.013	G(0.01)	-----	
1943		-----		-----	
		Only proc A	Only proc B		
	normality	not OK	not OK		
	n	3	3		
	outliers	0	1		
	mean (n)	0.086	0.364		
	st.dev. (n)	0.0731	0.0919		
	R(calc.)	0.205	0.257		
	R(D2896:11 (A))	0.006	n.a.		
	R(D2896:11 (B))	n.a.	0.025		

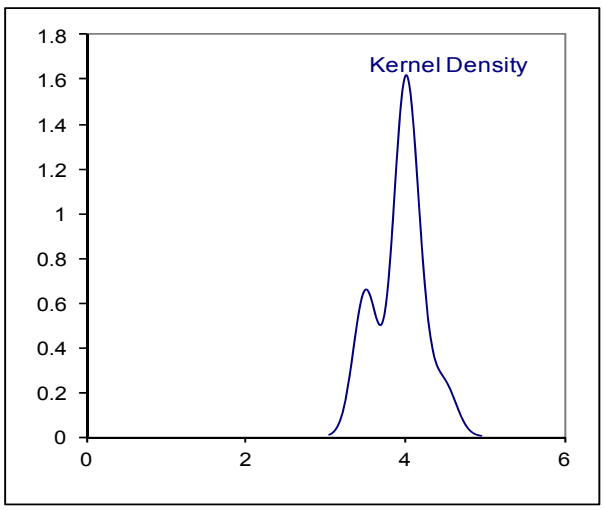


Determination of Color ASTM on sample #13068

lab	method	value	mark	z(targ)	remarks
150		-----		-----	
228	D1500	4.0		0.19	
323	D6045	L4.5		-----	
331	D1500	3.5		-1.21	
369	D1500	4.0		0.19	
398	D1500	4.0		0.19	
495	D1500	3.5		-1.21	
496	D1500	4.0		0.19	
511		-----		-----	
609	D1500	4.0		0.19	
657	D1500	L4.0		-----	
1023		-----		-----	
1146		-----		-----	
1271	D1500	4.2		0.75	
1435	D1500	4.0		0.19	
1596		-----		-----	
1597		-----		-----	
1599		-----		-----	
1604		-----		-----	
1665	D1500	4.0		0.19	
1666		-----		-----	
1670	D1500	3.5		-1.21	
1671		-----		-----	
1672	D1500	4.5		1.59	
1673		-----		-----	
1674		-----		-----	
1675	D1500	L4.5		-----	
1827		-----		-----	
1943		-----		-----	

normality	not OK
n	12
outliers	0
mean (n)	3.93
st.dev. (n)	0.299
R(calc.)	0.84
R(D1500:12)	1.00

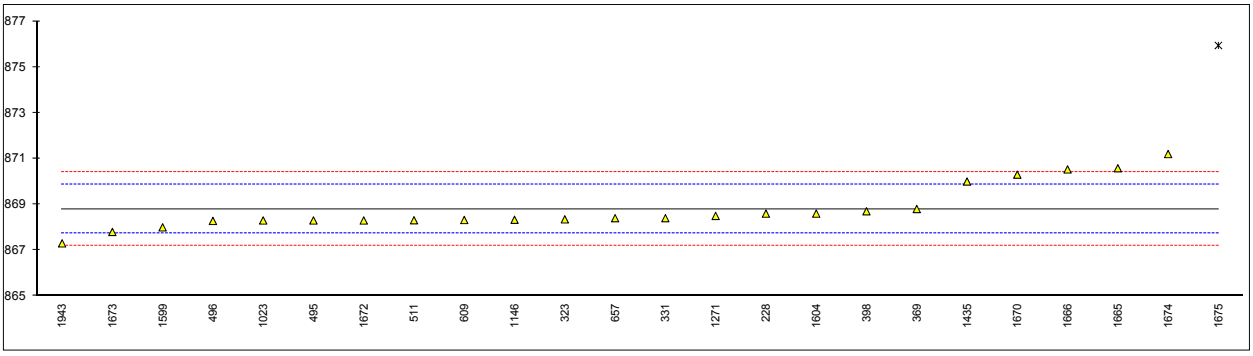


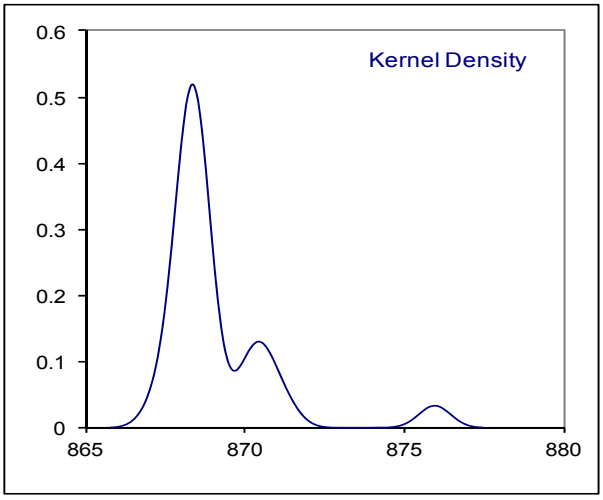


Determination of Density @ 15°C on sample #13068; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
150		-----		-----	
228	D1298	868.6		-0.36	
323	D4052	868.35	C	-0.82	First reported 0.86835
331	ISO12185	868.4		-0.73	
369	D4052	868.8		0.02	
398	D4052	868.7		-0.17	
495	D1298	868.3		-0.92	
496	D1298	868.28		-0.95	
511	D4052	868.31		-0.90	
609	D4052	868.32		-0.88	
657	D4052	868.4		-0.73	
1023	D1298	868.3	C	-0.92	Reported 0.8683 kg/l, probably an unit error
1146	D4052	868.33		-0.86	
1271	D4052	868.5		-0.54	
1435	D4052	870	C	2.26	Reported 0.870 kg/l, probably an unit error
1596		-----		-----	
1597		-----		-----	
1599	D5002	868	C	-1.48	First reported 0.868
1604	D1298	868.6		-0.36	
1665	D1298	870.58	C	3.34	First reported 872.98
1666	D1298	870.527		3.24	
1670	D1298	870.3	C	2.82	First reported 0.8703
1671		-----		-----	
1672	D1298	868.3	C	-0.92	Reported 0.8683 kg/l, probably an unit error
1673	D1298	867.8	C	-1.85	Reported 0.8678 kg/l, probably an unit error
1674	D4052	871.2	C	4.50	First reported 0.8712
1675	D1298	875.94	G(0.01)	13.34	
1827		-----		-----	
1943	ISO3675	867.3	C	-2.78	First reported 0.8673

normality	not OK
n	23
outliers	1
mean (n)	868.79
st.dev. (n)	0.998
R(calc.)	2.79
R(D1298:12b)	1.50

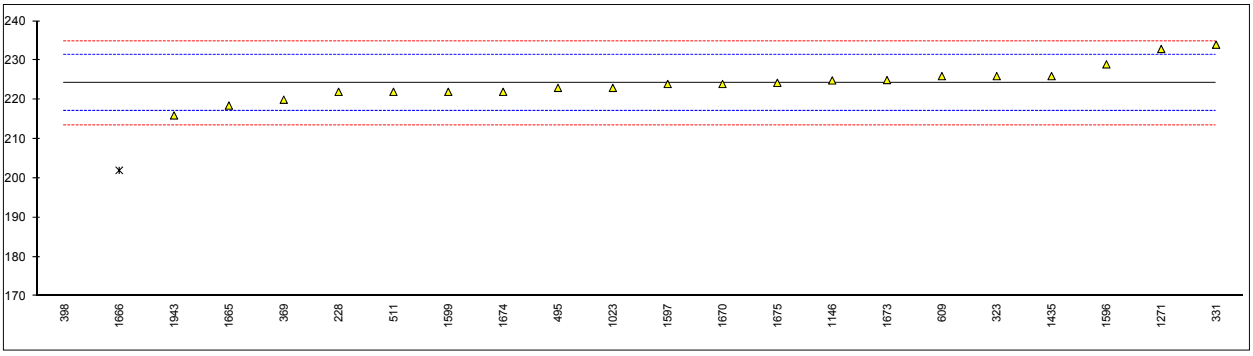


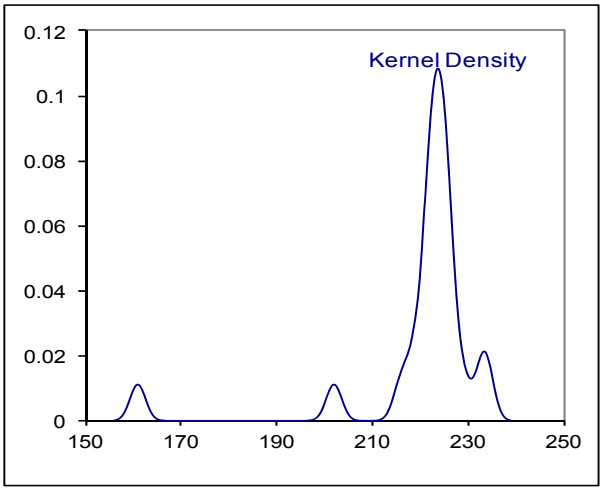


Determination of Flash Point PMcc on sample #13068; results in °C

lab	method	value	mark	z(targ)	remarks
150		-----		-----	
228	D93	222.0		-0.62	
323	D93	226.0		0.50	
331	D93	234.0		2.74	
369	D93	220.0		-1.18	
398	D93	161.0	G(0.01)	-17.70	
495	D93	223.0		-0.34	
496		-----		-----	
511	D93	222.0		-0.62	
609	D3828	226		0.50	
657		-----		-----	
1023	D93	223		-0.34	
1146	in house	224.9		0.19	
1271	ISO2719	232.9		2.43	
1435	D93	226		0.50	
1596	D92	229		1.34	
1597	ISO2592	224		-0.06	
1599	D93	222		-0.62	
1604		-----		-----	
1665	D93	218.5		-1.60	
1666	D93	202	G(0.01)	-6.22	
1670	D93	224		-0.06	
1671		-----		-----	
1672		-----		-----	
1673	D93	225		0.22	
1674	D93	222		-0.62	
1675	D93	224.3		0.02	
1827		-----		-----	
1943	ISO2719	216		-2.30	

normality	OK
n	20
outliers	2
mean (n)	224.23
st.dev. (n)	4.263
R(calc.)	11.94
R(D93:12)	10.00

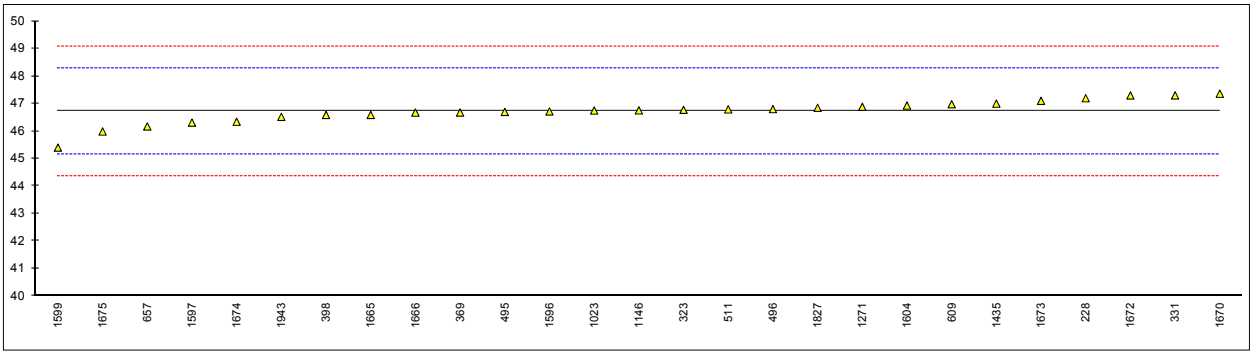


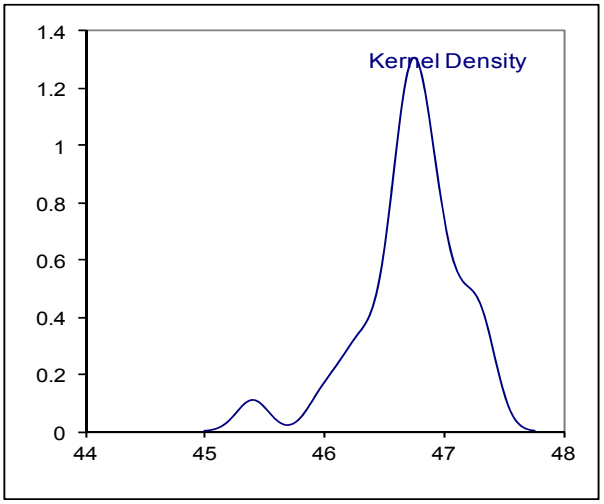


Determination of Kinematic Viscosity @ 40°C on sample #13068; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
150		-----		-----	
228	D445	47.197		0.61	
323	D445	46.775		0.07	
331	D7279	47.30		0.74	
369	D445	46.677		-0.05	
398	D445	46.59		-0.17	
495	D445	46.698		-0.03	
496	D445	46.800		0.10	
511	D445	46.7895		0.09	
609	D7042	46.979		0.33	
657	D7279	46.17		-0.70	
1023	D445	46.75		0.04	
1146	D445	46.754		0.04	
1271	ISO3104	46.885		0.21	
1435	D7042	47.0		0.36	
1596	D445	46.716		-0.01	
1597	D445	46.31		-0.52	
1599	D445	45.4		-1.69	
1604	D445	46.93		0.27	
1665	D445	46.59		-0.17	
1666	D445	46.6740		-0.06	
1670	D445	47.36		0.82	
1671		-----		-----	
1672	D445	47.298		0.74	
1673	D445	47.1		0.49	
1674	D445	46.34		-0.49	
1675	D445	45.986		-0.94	
1827	D445	46.85		0.17	
1943	ISO3104	46.52		-0.26	

normality	OK
n	27
outliers	0
mean (n)	46.7199
st.dev. (n)	0.42410
R(calc.)	1.1875
R(D445:12)	2.1888

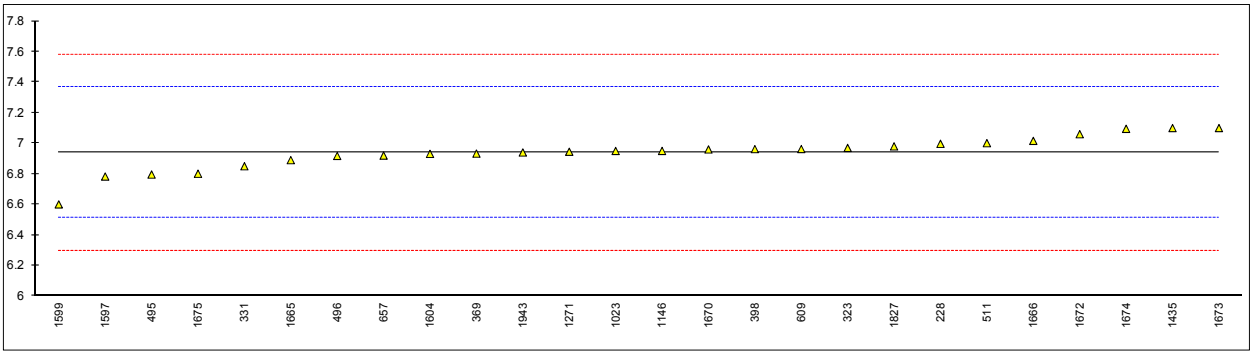


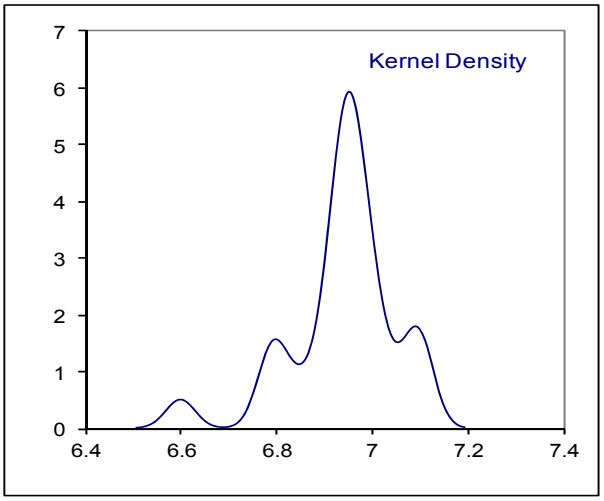


Determination of Kinematic Viscosity @ 100°C on sample #13068; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
150		-----		-----	
228	D445	6.9969		0.27	
323	D445	6.970		0.15	
331	D7279	6.85		-0.42	
369	D445	6.9327		-0.03	
398	D445	6.962		0.11	
495	D445	6.7958		-0.67	
496	D445	6.9175		-0.10	
511	D445	7.0011		0.29	
609	D7042	6.9627		0.11	
657	D7279	6.920		-0.09	
1023	D445	6.95		0.05	
1146	D445	6.9503		0.05	
1271	ISO3104	6.945		0.03	
1435	D7042	7.1		0.76	
1596		-----		-----	
1597	D445	6.7827		-0.73	
1599	D445	6.6		-1.59	
1604	D445	6.931		-0.04	
1665	D445	6.890		-0.23	
1666	D445	7.0161		0.36	
1670	D445	6.96		0.10	
1671		-----		-----	
1672	D445	7.0597		0.57	
1673	D445	7.1		0.76	
1674	D445	7.095		0.73	
1675	D445	6.801		-0.65	
1827	D445	6.98		0.19	
1943	ISO3104	6.94		0.01	

normality	not OK
n	26
outliers	0
mean (n)	6.9388
st.dev. (n)	0.10927
R(calc.)	0.3060
R(D445:12)	0.5976

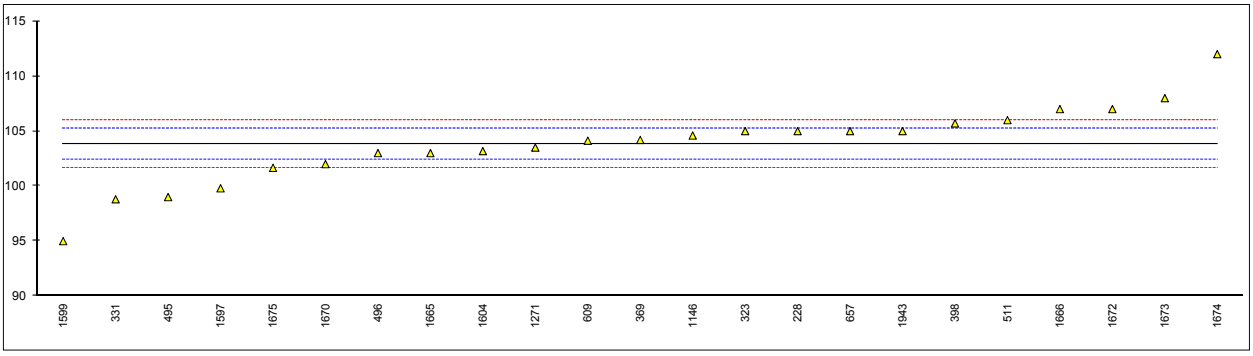


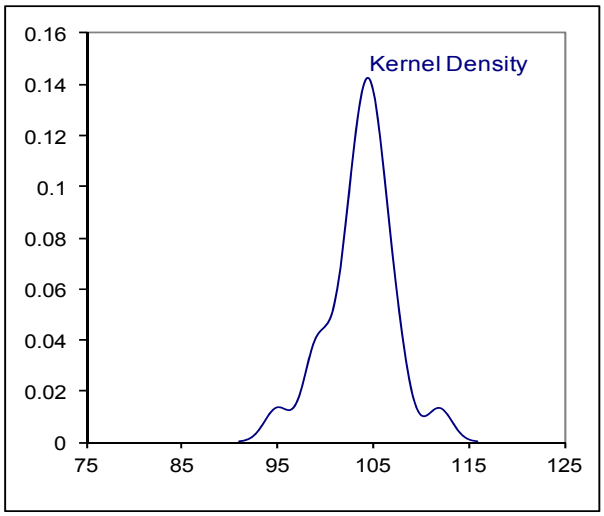


Determination of Viscosity Index from Kinematic Viscosity @ 40&100°C on sample #13068

lab	method	value	mark	z(targ)	Remarks
150		-----		-----	
228	D2270	105		1.67	
323	D2270	105		1.67	
331	D2270	98.8		-7.01	
369	D2270	104.2		0.55	
398	D2270	105.7		2.65	
495	D2270	99		-6.73	
496	D2270	103.0		-1.13	
511	D2270	106		3.07	
609	D2270	104.13		0.45	
657	D2270	105		1.67	
1023		-----		-----	
1146	D2270	104.6		1.11	
1271	ISO2909	103.5		-0.43	
1435		-----		-----	
1596		-----		-----	
1597	INH-6218	99.8		-5.61	
1599	D2270	95		-12.33	
1604	D2270	103.18		-0.88	
1665	D2270	103		-1.13	
1666	D2270	107		4.47	
1670	D2270	102		-2.53	
1671		-----		-----	
1672	D2270	107		4.47	
1673	D2270	108		5.87	
1674	ISO2909	112		11.47	
1675	D2270	101.66		-3.01	
1827		-----		-----	
1943	ISO2909	105		1.67	

normality	OK
n	23
outliers	0
mean (n)	103.81
st.dev. (n)	3.513
R(calc.)	9.84
R(D2270:10e1)	2.00





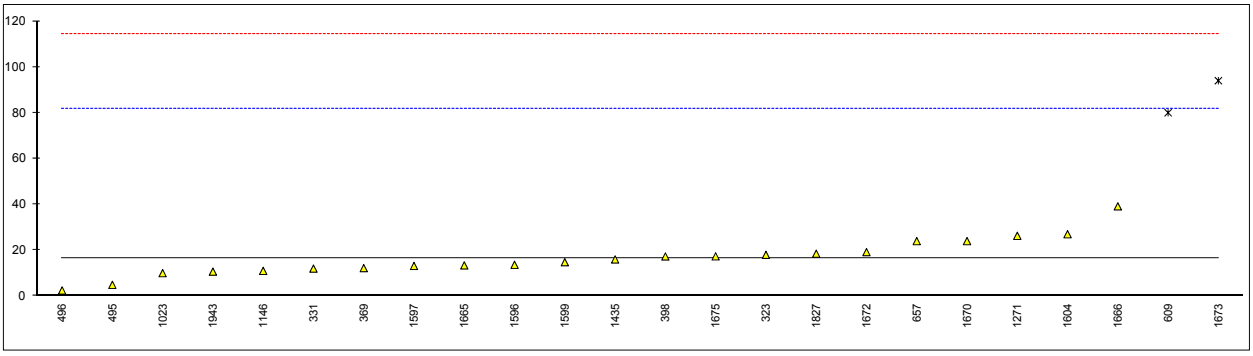
Determination of Level of contamination on sample #13068;

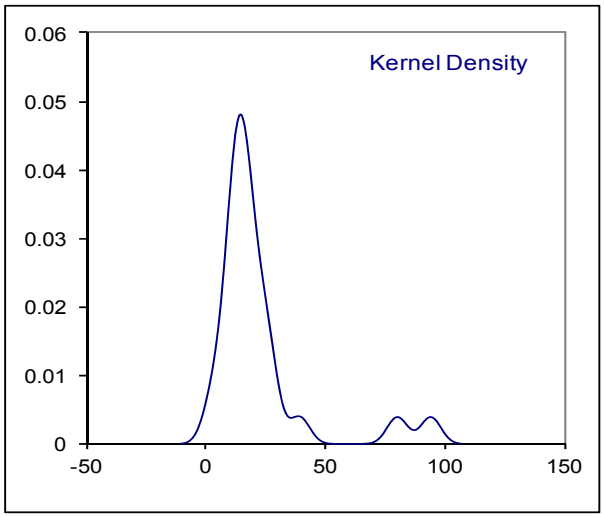
lab	method	>4µm (c)	>6µm (c)	>14µm (c)	mark	z(targ)	remarks
150		-----	-----	-----		-----	
228		-----	-----	-----		-----	
323	ISO4406	22	15	9		-----	
331	ISO4406/7	23	17	11		-----	
369		-----	-----	-----		-----	
398	ISO4406	23	16	10		-----	
495	ISO4406	19	16	13		-----	
496		-----	-----	-----		-----	
511	ISO4406	17	15	12		-----	
609	ISO4406	18	16	14		-----	
657	ISO4406	17	14	11		-----	
1023	ISO4406/7	23	20	11		-----	
1146	ISO4406/7	17	15	13		-----	
1271	ISO4406	19	16	14		-----	
1435	ISO4406	23	19	12		-----	
1596		-----	-----	-----		-----	
1597		-----	-----	-----		-----	
1599		-----	-----	-----		-----	
1604	ISO4406/7	19	14	9		-----	
1665	ISO4406	22	19	12		-----	
1666	ISO4406/7	22	15	12		-----	
1670	ISO4406	-----	14	11		-----	
1671		-----	-----	-----		-----	
1672	ISO4406/7	22	18	10		-----	
1673		-----	-----	-----		-----	
1674	ISO4406	-----	14	11		-----	
1675	INH-400	-----	17	13		-----	
1827		-----	-----	-----		-----	
1943	EN60970	20	14	12		-----	
	normality	n.a.	n.a.	n.a.			
	n	16	19	19			
	outliers	n.a.	n.a.	n.a.			
	mean (n)	20	16	12			
	st.dev. (n)	n.a.	n.a.	n.a.			
	R(calc.)	n.a.	n.a.	n.a.			
	R(ISO4406)	n.a.	n.a.	n.a.			

Determination of Water by KF on sample #13068; results in mg/kg

lab	method	value	mark	z(targ)	remarks
150		-----		-----	
228		-----		-----	
323	D6304C	18		0.04	
331	D6304C	12		-0.14	
369	D6304C	12.2		-0.13	
398	D6304C	17.3		0.02	
495	D6304C	4.9	C	-0.36	First reported 49
496	D6304C	2.45		-0.43	
511		-----		-----	
609	D6304C	80	G(0.01)	1.95	
657	D6304C	24	C	0.23	First reported 5.9
1023	D6304A	10		-0.20	
1146	D6304C	11		-0.17	
1271	ISO12937	26.3		0.30	
1435	D1744	16		-0.02	
1596	D6304C	13.66		-0.09	
1597	D6304C	13.15		-0.11	
1599	D6304C	14.8		-0.06	
1604	D6304C	27		0.32	
1665	D6304C	13.39		-0.10	
1666	D6304C	39.179		0.69	
1670	D1744	24		0.23	
1671		-----		-----	
1672	D6304C	19.2		0.08	
1673	D6304C	94	G(0.01)	2.38	
1674		-----		-----	
1675	D6304A	17.35		0.02	
1827	D6304A	18.50		0.06	
1943	EN60814	10.67		-0.18	

normality	OK
n	22
outliers	2
mean (n)	16.593
st.dev. (n)	8.0390
R(calc.)	22.509
R(D6304:07)	91.123

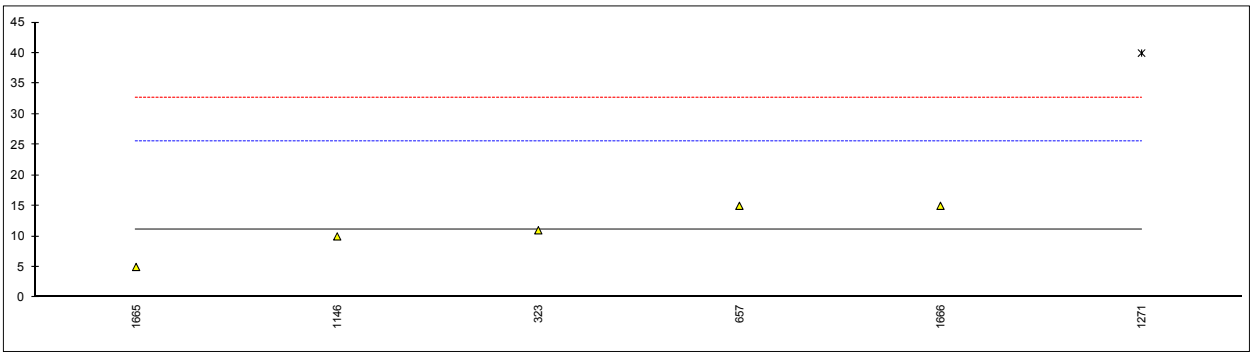




Determination of Water Separability on sample #13068; o-w-e / t

lab	method	value	Time (min)	mark	z(targ)	remarks
150		-----			-----	
228		-----			-----	
323	D1401	40-37-3	11		-----	
331		-----			-----	
369		-----			-----	
398		-----			-----	
495		-----			-----	
496		-----			-----	
511		-----			-----	
609		-----			-----	
657	D1401	40-40-0	15		-----	
1023		-----			-----	
1146	D1401	36-33-11	10		-----	
1271	D1401	40-37-3	40	G(0.05)	-----	
1435		-----			-----	
1596		-----			-----	
1597		-----			-----	
1599		-----			-----	
1604		-----			-----	
1665	D1401	40-40-0	5		-----	
1666	D1401	42-38-0	15		-----	
1670		-----			-----	
1671		-----			-----	
1672		-----			-----	
1673		-----			-----	
1674		-----			-----	
1675		-----			-----	
1827		-----			-----	
1943		-----			-----	

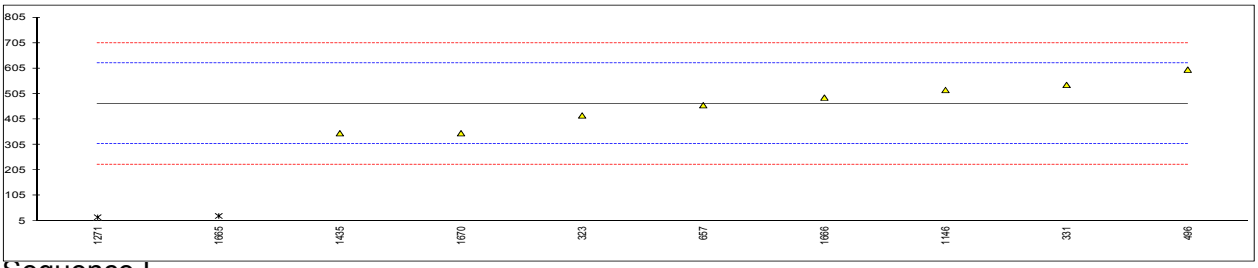
normality	OK
n	5
outliers	1
mean (n)	11.2
st.dev. (n)	4.15
R(calc.)	11.6
R(D1401:12)	20.0



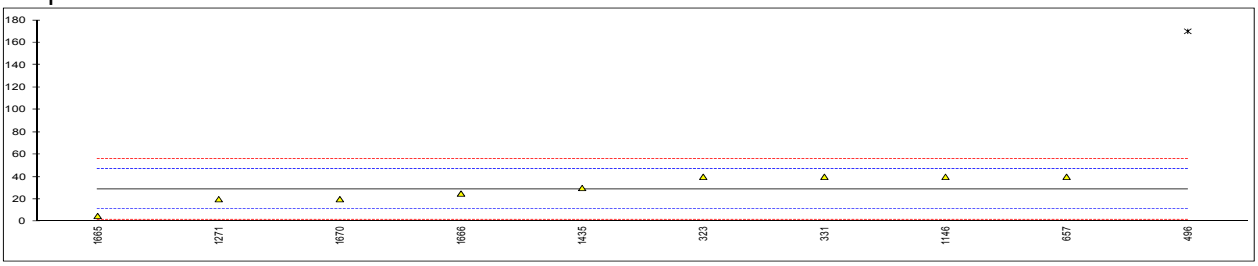
Time (minutes)

Determination of Foaming Characteristics, as received; Sequence I, II and III at the end of 5 min period on sample #13068; results in ml

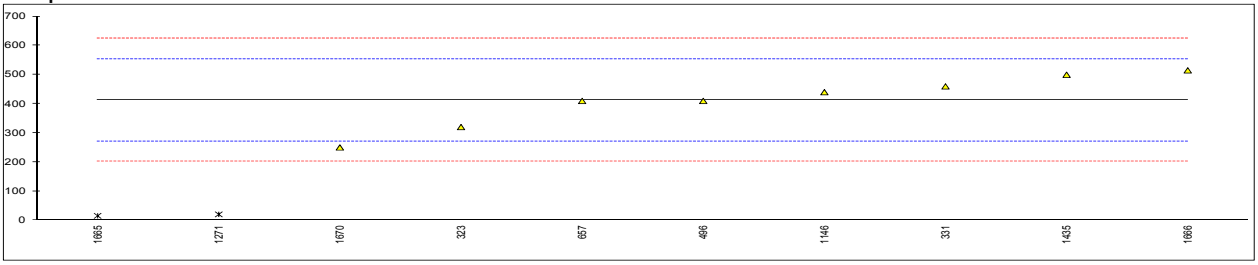
Lab	method	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
150		-----		-----	-----		-----	-----		-----
228		-----		-----	-----		-----	-----		-----
323	D892	420		-0.58	40		1.23	320	fr 260	-1.32
331	D892	540		0.93	40		1.23	460		0.66
369		-----		-----	-----		-----	-----		-----
398		-----		-----	-----		-----	-----		-----
495		-----		-----	-----		-----	-----		-----
496	D892	600		1.69	170	G(0.01)	15.66	410		-0.04
511		-----		-----	-----		-----	-----		-----
609		-----		-----	-----		-----	-----		-----
657	D892	460		-0.08	40		1.23	410		-0.04
1023		-----		-----	-----		-----	-----		-----
1146	D892	520		0.68	40		1.23	440		0.38
1271	D892	20	DG(0.05)	-5.63	20		-0.99	20	DG(0.05)	-5.56
1435	D892	350		-1.47	30		0.12	500		1.23
1596		-----		-----	-----		-----	-----		-----
1597		-----		-----	-----		-----	-----		-----
1599		-----		-----	-----		-----	-----		-----
1604		-----		-----	-----		-----	-----		-----
1665	D892	25	DG(0.05)	-5.56	5		-2.65	15	DG(0.05)	-5.63
1666	D892	490		0.30	25		-0.43	515		1.44
1670	D892	350		-1.47	20		-0.99	250		-2.31
1671		-----		-----	-----		-----	-----		-----
1672		-----		-----	-----		-----	-----		-----
1673		-----		-----	-----		-----	-----		-----
1674		-----		-----	-----		-----	-----		-----
1675		-----		-----	-----		-----	-----		-----
1827		-----		-----	-----		-----	-----		-----
1943		-----		-----	-----		-----	-----		-----
	normality	OK			OK			OK		
	n	8			9			8		
	outliers	2			1			2		
	mean (n)	466.3			28.9			413.1		
	st.dev. (n)	89.43			12.44			89.56		
	R(calc.)	250.4			34.8			250.8		
	R(D892:11a)	222.0			25.2			198.1		



Sequence I



Sequence II



Sequence III

Determination of Foaming Characteristics, as received; Sequence I, II and III at the end of 10 min period on sample #13068; results in ml

Lab	method	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
150		-----		-----	-----		-----	-----		-----
228		-----		-----	-----		-----	-----		-----
323	D892	0		-----	0		-----	0		-----
331	D892	0		-----	0		-----	0		-----
369		-----		-----	-----		-----	-----		-----
398		-----		-----	-----		-----	-----		-----
495		-----		-----	-----		-----	-----		-----
496	D892	0		-----	0		-----	0		-----
511		-----		-----	-----		-----	-----		-----
609		-----		-----	-----		-----	-----		-----
657	D892	NIL		-----	NIL		-----	NIL		-----
1023		-----		-----	-----		-----	-----		-----
1146	D892	0		-----	0		-----	0		-----
1271	D892	0		-----	0		-----	0		-----
1435		-----		-----	-----		-----	-----		-----
1596		-----		-----	-----		-----	-----		-----
1597		-----		-----	-----		-----	-----		-----
1599		-----		-----	-----		-----	-----		-----
1604		-----		-----	-----		-----	-----		-----
1665	D892	0		-----	0		-----	0		-----
1666	D892	0		-----	0		-----	0		-----
1670	D892	0		-----	0		-----	0		-----
1671		-----		-----	-----		-----	-----		-----
1672		-----		-----	-----		-----	-----		-----
1673		-----		-----	-----		-----	-----		-----
1674		-----		-----	-----		-----	-----		-----
1675		-----		-----	-----		-----	-----		-----
1827		-----		-----	-----		-----	-----		-----
1943		-----		-----	-----		-----	-----		-----
	normality	n.a.			n.a.			n.a.		
	n	8			8			8		
	outliers	n.a.			n.a.			n.a.		
	mean (n)	0			0			0		
	st.dev. (n)	n.a.			n.a.			n.a.		
	R(calc.)	n.a.			n.a.			n.a.		
	R(D892:11a)	n.a.			n.a.			n.a.		

APPENDIX 2

Number of participants per country

2 laboratories in	BELGIUM
1 laboratory in	BOSNIA and HERZEGOVINA
1 laboratory in	FRANCE
2 laboratories in	GERMANY
7 laboratories in	ITALY
2 laboratories in	LATVIA
2 laboratories in	MALAYSIA
1 laboratory in	NORWAY
1 laboratory in	PERU
1 laboratory in	SINGAPORE
2 laboratories in	SLOVAKIA
4 laboratories in	SPAIN
1 laboratory in	THE NETHERLANDS
1 laboratory in	TOGO
1 laboratory in	U.S.A.

APPENDIX 3

Abbreviations:

C	= 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
ex	= excluded from calculations
U	= reported in different unit
W	= result withdrawn on request of the participants
fr.	= first reported
S	= scope of the reported method is not applicable
n.a.	= not applicable
n.e.	= not evaluated
SDS	= Material Safety Data Sheet

Literature:

1 iis Interlaboratory Studies, Protocol for the Organization, Statistics and Evaluation, January 2010

2 ASTM E178-89

3 ASTM E1301-89

4 ISO 5725-86

5 ISO 5725, parts 1-6, 1994

6 ISO13528-05

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

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

9 IP 367/84

10 DIN 38402 T41/42

11 P.L. Davies, First reported Z. Anal. Chem, 331, 513, (1988)

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

13 Analytical Methods Committee Technical brief, No4 January 2001.

14 The Royal Society of Chemistry 2002, Analyst 2002, 127 pages 1359-1364, P.J. Lowthian and M. Thompson (see http://www.rsc.org/suppdata/an/b2/b205600n/).