Results of Proficiency Test
Base Oil
May 2013

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

On request of several participants, the Institute for Interlaboratory Studies decided to organise a new proficiency test for the analysis of Base Oil during the annual proficiency testing program 2012/2013. In this interlaboratory study 33 laboratories in 23 different countries have participated. See appendix 2 for the number of participants per country. In this report, the results of the 2013 Base 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 #13066) of Base Oil that was purchased from a local supplier. 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 and ILAC-G13:2007, (R007), 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 obtained from a local supplier. The 80 litre bulk material (HVI-650) was transferred after homogenizing into 77 brown glass bottles of 1 litre (labelled #13066). The homogeneity of the subsamples #13066 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/L
Sample #13066-1	0.9003
Sample #13066-2	0.9003
Sample #13066-3	0.9003
Sample #13066-4	0.9003
Sample #13066-5	0.9003
Sample #13066-6	0.9003
Sample #13066-7	0.9003
Sample #13066-8	0.9003

Table 1: homogeneity test results of subsamples #13066

From the above test results, the repeatabilities were 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 #13066)	0.00000
reference test	ASTM D4052:11
0.3 x R(reference test)	0.00015

Table 2: evaluation of the repeatabilities of the subsamples #13066

The calculated repeatability is less than 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneity of the subsamples #13066 was assumed.

To each of the participating laboratories, 1 sample of 1 L in a brown glass bottle (labelled #13066) was sent on May 1, 2013.

2.5 ANALYSES

The participants were requested to determine on sample #13066: Acid Number (Total), Air-release time @75ºC, Colour ASTM, Conradson/Micro Carbon Residue, Ramsbottom Carbon Residue, Density @ 15°C, Flash Point COC, Kinematic Viscosity @ 40°C and @ 100°C, Viscosity Stabinger @ 40°C and @100ºC, Viscosity Index, Evaporation loss by Noack test, Pour Point, Rust prevention (proc. B), Sulphur, Water, Water Separability @82ºC.
To get comparable results a detailed report form, on which the units were prescribed as well as some of the required standards, was sent together with each set of samples. Also, a letter of instructions and a SDS were added to the 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.13 and 14).

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:

$z_{(target)}$ = (result - average of PT) / 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 Brazil and Russia. Twelve participants reported the test results after the final reporting date and five participants did not report any test results at all. Not all laboratories were able to report all analyses requested. In total 28 participants reported 260 test results. Observed were 17 outlying results, which is 6.5% 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: Acid Number, Colour, Density @15°C, Kinematic Viscosity @40ºC, Viscosity Index and Pour Point. 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.

Acid Number (total): This determination was problematic for a number of laboratories. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D974:12.

Air-release time: Only three test results were reported, therefore no significant conclusions were drawn.

Colour: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with ASTM D1500:12.

Conradson CR: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D4530:11.

Ramsbottom CR: Only one result was reported, therefore no significant conclusions were drawn.

Density @ 15°C: This determination was very problematic. The group appears to be bimodally divided. Three statistical outliers were observed and the calculated reproducibility, after rejection of the statistical outliers, is not at all in agreement with the requirements of ASTM D4052:11. The large spread may be explained by possible not correcting the test result for viscosity or using an incorrect conversion table.

Flash Point COC: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in full agreement with ASTM D92:12b.

Kin.Visco.@ 40°C: This determination was very problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the (strict) requirements of ASTM D445:12. The large spread might be explained that a number of laboratories may have used a Canon Fenske Routine Viscometer instead of an Ubbelohde Viscometer.

Kin.Visco.@ 100°C: This determination was very problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the (strict) requirements of ASTM D445:12. The large spread might (party) be explained that a number of laboratories may have used a Canon Fenske Routine Viscometer instead of an Ubbelohde Viscometer.

Visco. Stabinger at 40ºC This determination was very problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with ASTM D7042:11.

Visco. Stabinger at 100ºC 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 ASTM D7042:11.

Viscosity Index: 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 ASTM D2270:10e1.

Evaporation loss: 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 ASTM D5800:10.

Pour Point: This determination was problematic. Only one statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with ASTM D6892:08. The large spread may (partly) be explained by rounding of the results up to 3 degrees.

Rust prevention: This determination may be problematic. Regretfully, only five participants reported a result. Two reported the presence of rust, while three other participants reported the test as “pass”.

Sulphur: This determination was problematic for a number of laboratories. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ASTM D2622:10.

Water: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D6304:07.

Water separability Regretfully, only for Time measurement statistical data is available. Only three participants reported a test result in minutes, therefore no significant conclusions were drawn. Also for the other reported test results no significant conclusion was drawn.

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 standards), are compared in the next table.

Parameter	unit	n	Average	2.8 * sd	R(lit)
Total Acid Number	mg KOH/g	15	0.018	0.038	0.040
Air-release time @25ºC	min	3	18.4	n.a.	n.a.
Colour ASTM		15	3.5	0.5	1.0
Conradson Carbon Residue	%M/M	16	0.50	0.24	0.16
Ramsbottom Carbon Residue	%M/M	1	0.46	n.a.	n.a.
Density @ 15 °C	kg/L	24	0.9002	0.0010	0.0005
Flash Point COC	^{o}C	21	325.9	14.2	18.0
Kinematic Viscosity @ 40 °C	mm^2/s	26	487.1	8.2	3.2
Kinematic Viscosity @ 100 °C	mm^2/s	25	31.82	0.32	0.21
Stabinger Viscosity @ 40 °C	mm^2/s	8	487.4	9.2	2.9
Stabinger Viscosity @ 100 °C	mm^2/s	7	31.99	0.34	0.11
Viscosity Index		23	96.2	1.5	2.0
Evaporation loss by Noack	%M/M	6	0.27	0.12	0.05
Pour Point	^{o}C	20	-10.6	4.1	3.2
Sulphur	%M/M	16	1.03	0.07	0.07
Water	mg/kg	13	58.4	135.7	193.8
Water Separability	min	3	21.7	8.1	25.0

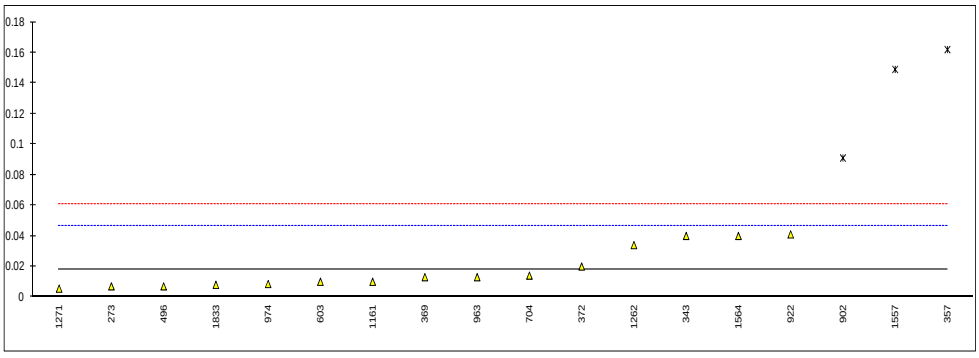
Table 3: reproducibilities of results of sample #13066

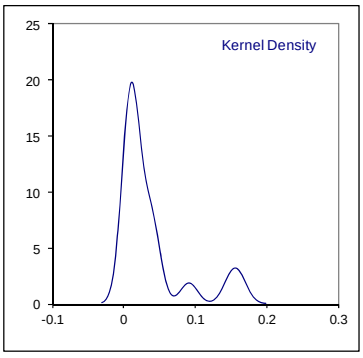
Without further statistical calculations it can be concluded that for a number of tests there is a not a good compliance of the group of participants with the relevant standards. The tests that are problematic have been discussed in paragraph 4.1.

APPENDIX 1

Determination of Acid Number (Total) on sample #13066; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
273	D974	0.007		-0.77	
315		-----		-----	
343	D974	0.04		1.54	
357	D664	0.162	DG(0.01)	10.08	
369	D974	0.013		-0.35	
372	D974	0.02		0.14	
432		-----		-----	
494		-----		-----	
496	D974	0.007		-0.77	
541	D974	<0.1		-----	
551		-----		-----	
603	D664	0.01		-0.56	
704	D974	0.014		-0.28	
781		-----		-----	
875		-----		-----	
902	D974	0.091	G(0.01)	5.11	
922	D664	0.041		1.61	
963	D974	0.013		-0.35	
974	D974	0.0085		-0.67	
1013	D974	<0.07		-----	
1082		-----		-----	
1161	D974	0.010		-0.56	
1191		-----		-----	
1231		-----		-----	
1243		-----		-----	
1262	D974	0.034		1.12	
1271	D974	0.0055		-0.88	
1340		-----		-----	
1557	EN14104	0.149	DG(0.01)	9.17	
1564	D664	0.04		1.54	
1652		-----		-----	
1706		-----		-----	
1833	D974	0.008		-0.70	
					Only ASTM D974 data:
	normality	not OK			not OK
	n	15			12
	outliers	3			1
	mean (n)	0.0181			0.0150
	st.dev. (n)	0.01347			0.01110
	R(calc.)	0.0377			0.0311
	R(D974:12)	0.0400			0.0400





Determination of Air-release time @ 25°C on sample #13066; results in min

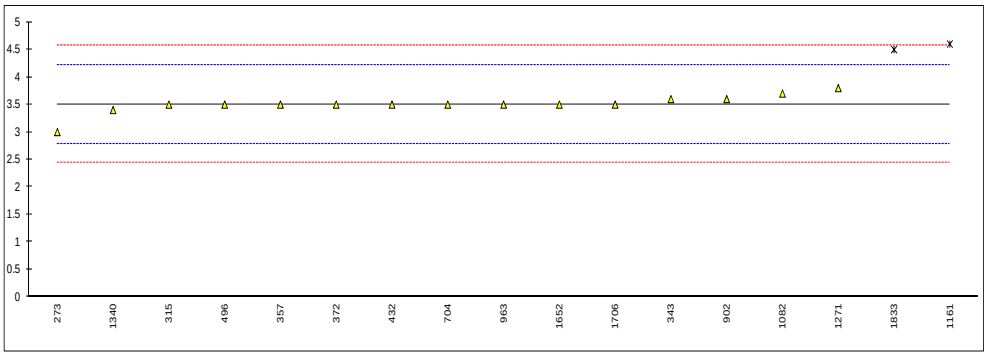
lab	method	value	mark	z(targ)	remarks
273		-----		-----	
315		-----		-----	
343		-----		-----	
357		-----		-----	
369		-----		-----	
372		-----		-----	
432	ISO9120	23.2		-----	
494		-----		-----	
496		-----		-----	
541		-----		-----	
551		-----		-----	
603		-----		-----	
704		-----		-----	
781		-----		-----	
875		-----		-----	
902		-----		-----	
922		-----		-----	
963		-----		-----	
974		-----		-----	
1013		-----		-----	
1082		-----		-----	
1161		-----		-----	
1191		-----		-----	
1231		-----		-----	
1243		-----		-----	
1262	D3427	21.1		-----	
1271	D3427	10.8		-----	
1340		-----		-----	
1557		-----		-----	
1564		-----		-----	
1652		-----		-----	
1706		-----		-----	
1833		-----		-----	

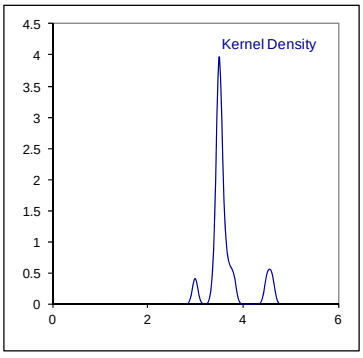
normality	n.a.
n	3
outliers	0
mean (n)	18.4
st.dev. (n)	n.a.
R(calc.)	n.a.
R(D3427:12)	n.a.

Determination of Colour on sample #13066

lab	method	value	mark	z(targ)	remarks
273	D1500	3.0		-1.42	
315	D1500	3.5		-0.02	
343	D1500	3.6		0.26	
357	D1500	3.5		-0.02	
369	D1500	L4.0		-----	
372	D1500	3.5		-0.02	
432	D1500	3.5		-0.02	
494	D1500	L3.5		-----	
496	D1500	3.5		-0.02	
541	D1500	L3.5		-----	
551		-----		-----	
603	D1500	L3.5		-----	
704	D1500	3.5		-0.02	
781		-----		-----	
875		-----		-----	
902	D1500	3.6		0.26	
922	D1500	L4.0		-----	
963	D1500	3.5		-0.02	
974	D1500	<3.5		-----	
1013	D1500	L4.0		-----	
1082	D1500	3.7		0.54	
1161	D1500	4.6	DG(0.01)	3.06	
1191		-----		-----	
1231		-----		-----	
1243		-----		-----	
1262	D1500	L3.5		-----	
1271	D6045	3.8		0.82	
1340	D6045	3.4		-0.30	
1557	D1500	L3.5		-----	
1564	D1500	L3		-----	
1652	D1500	3.5		-0.02	
1706	D1500	3.5		-0.02	
1833	D1500	4.5	DG(0.01)	2.78	

normality	not OK
n	15
outliers	2
mean (n)	3.51
st.dev. (n)	0.171
R(calc.)	0.48
R(D1500:12)	1.00

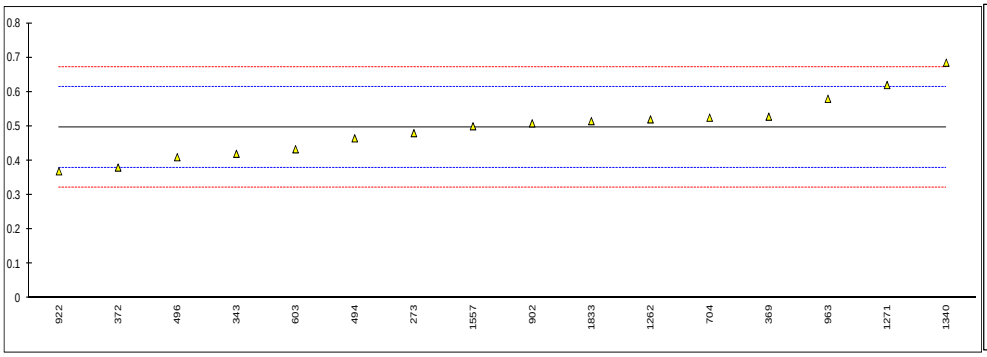


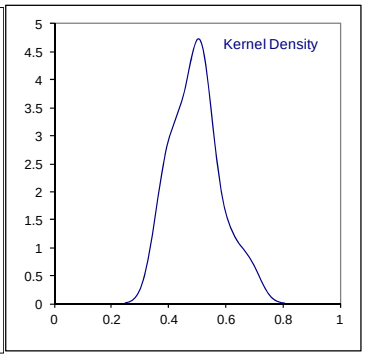


Determination of Conradson Carbon Residue on sample #13066; results in %M/M

lab	method	value	mark	z(targ)	remarks
273	D4530	0.48		-0.28	
315		-----		-----	
343	D4530	0.42		-1.30	
357		-----		-----	
369	D4530	0.528		0.54	
372	D4530	0.38		-1.99	
432		-----		-----	
494	D4530	0.465		-0.53	
496	D4530	0.41		-1.47	
541		-----		-----	
551		-----		-----	
603	D4530	0.4331		-1.08	
704	D4530	0.525		0.49	
781		-----		-----	
875		-----		-----	
902	D4530	0.5085		0.21	
922	D4530	0.369		-2.18	
963	D4530	0.58	C	1.43	First reported 0.19
974		-----		-----	
1013		-----		-----	
1082		-----		-----	
1161		-----		-----	
1191		-----		-----	
1231		-----		-----	
1243		-----		-----	
1262	D189	0.52		0.41	
1271	D4530	0.62		2.12	
1340	ISO10370	0.685		3.23	
1557	ISO10370	0.50		0.07	
1564		-----		-----	
1652		-----		-----	
1706		-----		-----	
1833	D4530	0.515		0.32	

normality	OK
n	16
outliers	0
mean (n)	0.4962
st.dev. (n)	0.08527
R(calc.)	0.2387
R(D4530:11)	0.1637





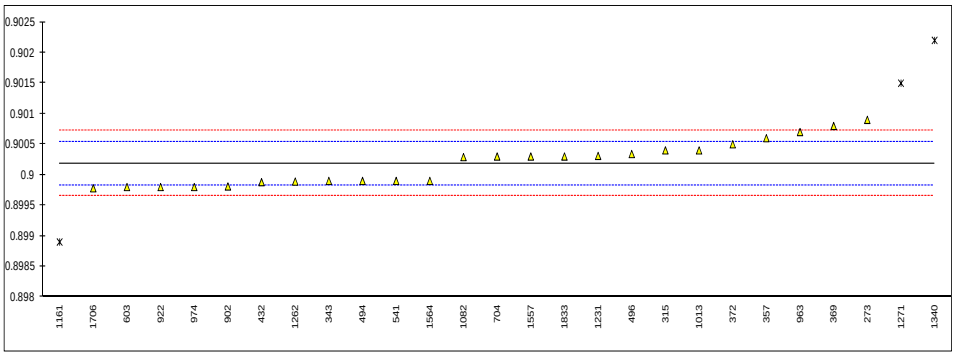
Determination of Ramsbottom Carbon Residue on sample #13066; results in %M/M

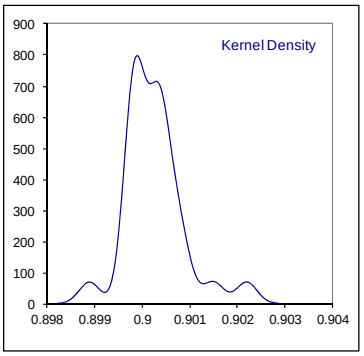
lab	method	value	mark	z(targ)	remarks
273		-----		-----	
315		-----		-----	
343		-----		-----	
357		-----		-----	
369		-----		-----	
372		-----		-----	
432		-----		-----	
494		-----		-----	
496		-----		-----	
541		-----		-----	
551		-----		-----	
603		-----		-----	
704		-----		-----	
781		-----		-----	
875		-----		-----	
902		-----		-----	
922		-----		-----	
963		-----		-----	
974		-----		-----	
1013		-----		-----	
1082		-----		-----	
1161		-----		-----	
1191		-----		-----	
1231		-----		-----	
1243		-----		-----	
1262		-----		-----	
1271		-----		-----	
1340		-----		-----	
1557		-----		-----	
1564		-----		-----	
1652		-----		-----	
1706		-----		-----	
1833	D524	0.459		-----	
	normality	n.a.			
	n	1			
	outliers	n.a.			
	mean (n)	n.a.			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D524:10)	n.a.			

Determination of Density @ 15°C on sample #13066; results in kg/L

lab	method	value	mark	z(targ)	remarks
273	D4052	0.9009		3.84	
315	D4052	0.9004		1.14	
343	D4052	0.8999		-1.55	
357	D4052	0.9006		2.22	
369	D4052	0.9008		3.30	
372	D4052	0.9005		1.68	
432	D4052	0.89988		-1.66	
494	D4052	0.8999		-1.55	
496	D4052	0.90034		0.82	
541	D4052	0.8999	C	-1.55	First reported 0.9030
551		-----		-----	
603	D4052	0.8998		-2.09	
704	D4052	0.9003		0.61	
781		-----		-----	
875		-----		-----	
902	D4052	0.89981		-2.03	
922	D4052	0.8998		-2.09	
963	D4052	0.9007		2.76	
974	D4052	0.8998		-2.09	
1013	D4052	0.9004		1.14	
1082	ISO12185	0.90029		0.55	
1161	ISO3675	0.8989	C,G(0.05)	-6.93	First reported 898.9
1191		-----		-----	
1231	D4052	0.90031		0.66	
1243		-----		-----	
1262	D4052	0.89989	C	-1.60	First reported 899.89
1271	D4052	0.9015	C,G(0.05)	7.07	First reported 901.5
1340	ISO3675	0.90220	G(0.05)	10.84	
1557	ISO3675	0.9003	C	0.61	First reported 0.8975
1564	D4052	0.8999	C	-1.55	First reported 899.9
1652		-----		-----	
1706	D4052	0.89978		-2.19	
1833	D4052	0.9003		0.61	

normality	not OK
n	24
outliers	3
mean (n)	0.90019
st.dev. (n)	0.000353
R(calc.)	0.00099
R(D4052:11)	0.00052



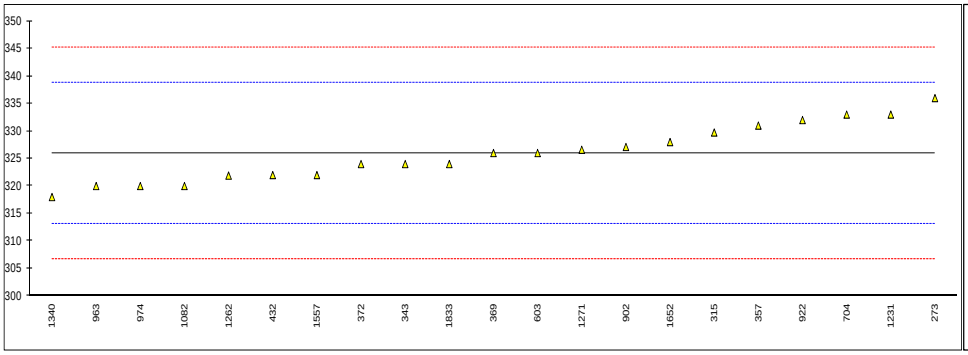


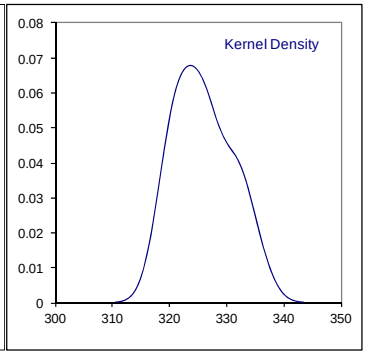
Determination of Flash Point C.O.C. on sample #13066; results in °C

lab	method	value	mark	z(targ)	remarks
273	D92	336		1.57	
315	D92	329.74		0.59	
343	D92	324		-0.30	
357	D92	331		0.79	
369	D92	326		0.01	
372	D92	324		-0.30	
432	D92	322		-0.61	
494	D92	>300		-----	
496		-----		-----	
541		-----		-----	
551		-----		-----	
603	D92	326.0		0.01	
704	D92	333		1.10	
781		-----		-----	
875		-----		-----	
902	D92	327.1		0.18	
922	D92	332		0.95	
963	D92	320		-0.92	
974	D92	320.0		-0.92	
1013		-----		-----	
1082	ISO2592	320		-0.92	
1161		-----		-----	
1191		-----		-----	
1231	D92	333		1.10	
1243		-----		-----	
1262	D92	321.9		-0.63	
1271	D92	326.6		0.11	
1340	ISO2592	318.0		-1.23	
1557	D92	322		-0.61	
1564		-----		-----	
1652	ISO2592	328.0		0.32	
1706		-----		-----	
1833	D92	324		-0.30	

normality	OK
n	21
outliers	0
mean (n)	325.92
st.dev. (n)	5.056
R(calc.)	14.16
R(D92:12b)	18.00

Compare R(ISO2592:00) = 17

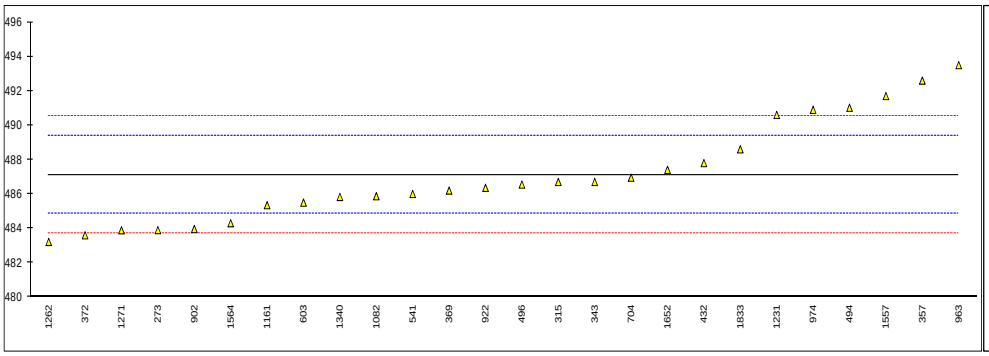


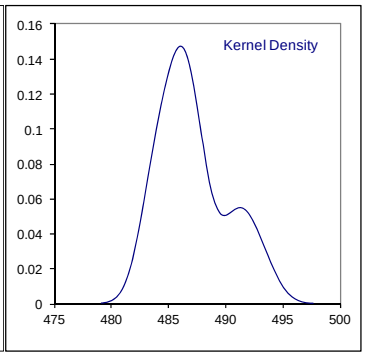


Determination of Kinematic Viscosity @ 40°C on sample #13066; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
273	D445	483.9		-2.84	
315	D445	486.7		-0.37	
343	D445	486.7		-0.37	
357	D445	492.6		4.85	
369	D445	486.2		-0.81	
372	D445	483.6		-3.11	
432	D445	487.8		0.61	
494	D445	491.02		3.45	
496	D445	486.55		-0.50	
541	D445	486.0		-0.99	
551		-----		-----	
603	D445	485.5		-1.43	
704	D445	486.95		-0.15	
781		-----		-----	
875		-----		-----	
902	D445	483.96		-2.79	
922	D445	486.36		-0.67	
963	D445	493.5		5.65	
974	D445	490.9		3.35	
1013		-----		-----	
1082	ISO3104	485.87		-1.10	
1161	ISO3104	485.35		-1.56	
1191		-----		-----	
1231	D445	490.6		3.08	
1243		-----		-----	
1262	D445	483.21		-3.45	
1271	ISO3104	483.89		-2.85	
1340	ISO3104	485.83		-1.14	
1557	ISO3104	491.7		4.05	
1564	D445	484.3		-2.49	
1652	ISO3104	487.41		0.26	
1706		-----		-----	
1833	D445	488.6		1.31	

normality	not OK
n	26
outliers	0
mean (n)	487.12
st.dev. (n)	2.932
R(calc.)	8.21
R(D445:12)	3.17

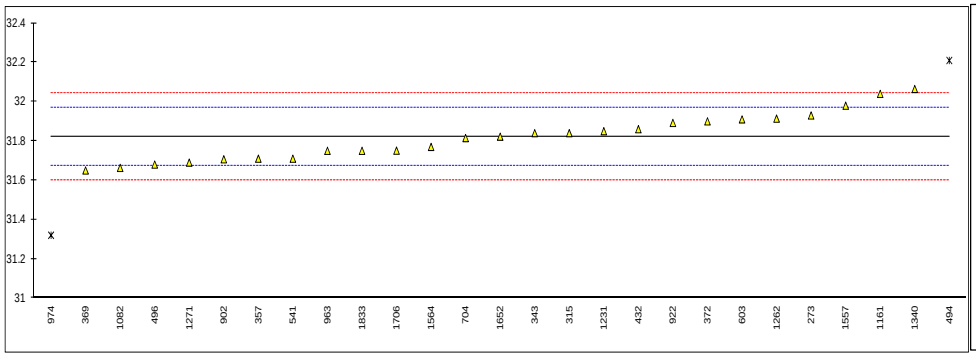


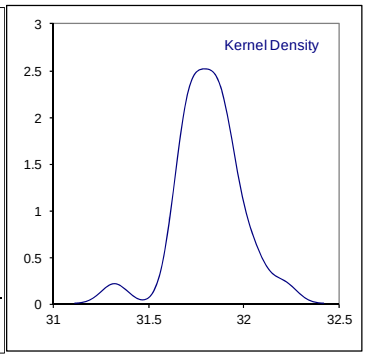


Determination of Kinematic Viscosity @ 100°C on sample #13066; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
273	D445	31.93		1.50	
315	D445	31.84		0.28	
343	D445	31.84		0.28	
357	D445	31.71		-1.48	
369	D445	31.65		-2.30	
372	D445	31.90		1.09	
432	D445	31.86		0.55	
494	D445	32.210	G(0.05)	5.29	
496	D445	31.680		-1.89	
541	D445	31.71		-1.48	
551		-----		-----	
603	D445	31.91		1.22	
704	D445	31.815		-0.06	
781		-----		-----	
875		-----		-----	
902	D445	31.707		-1.52	
922	D445	31.892		0.98	
963	D445	31.75		-0.94	
974	D445	31.32	G(0.05)	-6.76	
1013		-----		-----	
1082	ISO3104	31.663		-2.12	
1161	ISO3104	32.04		2.98	
1191		-----		-----	
1231	D445	31.85		0.41	
1243		-----		-----	
1262	D445	31.914		1.28	
1271	ISO3104	31.69		-1.75	
1340	ISO3104	32.065		3.32	
1557	ISO3104	31.98		2.17	
1564	D445	31.77		-0.67	
1652	ISO3104	31.822		0.03	
1706	ISO3104	31.751		-0.93	
1833	D445	31.75		-0.94	

normality	OK
n	25
outliers	2
mean (n)	31.820
st.dev. (n)	0.1158
R(calc.)	0.324
R(D445:12)	0.207

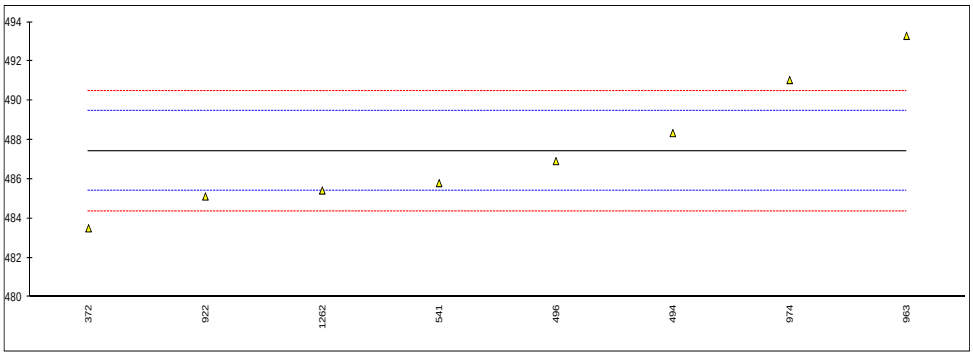


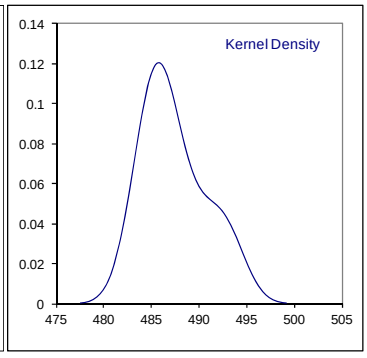


Determination of Viscosity Stabinger @ 40 °C on sample #13066; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
273		-----		-----	
315		-----		-----	
343		-----		-----	
357		-----		-----	
369		-----		-----	
372	D7042	483.5		-3.87	
432		-----		-----	
494	D7042	488.35		0.90	
496	D7042	486.92		-0.50	
541	D7042	485.8		-1.61	
551		-----		-----	
603		-----		-----	
704		-----		-----	
781		-----		-----	
875		-----		-----	
902		-----		-----	
922	D7042	485.12		-2.28	
963	D7042	493.3		5.77	
974	D7042	491.05		3.56	
1013		-----		-----	
1082		-----		-----	
1161		-----		-----	
1191		-----		-----	
1231		-----		-----	
1243		-----		-----	
1262	D7042	485.4250		-1.98	
1271		-----		-----	
1340		-----		-----	
1557		-----		-----	
1564		-----		-----	
1652		-----		-----	
1706		-----		-----	
1833		-----		-----	

normality	OK
n	8
outliers	0
mean (n)	487.43
st.dev. (n)	3.298
R(calc.)	9.23
R(D7042:11)	2.85

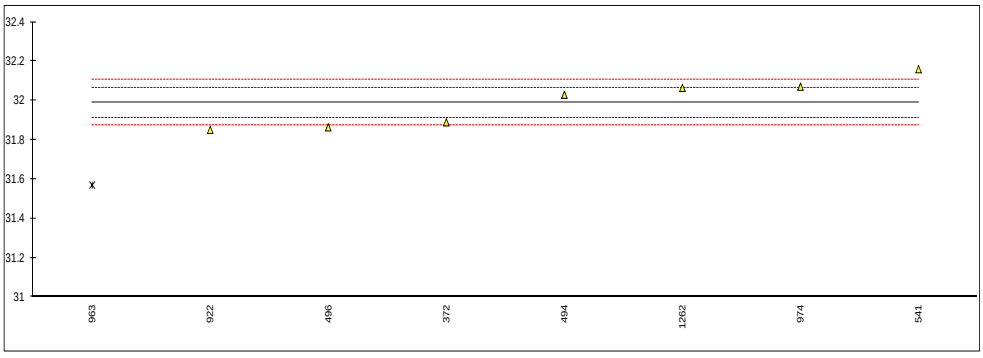


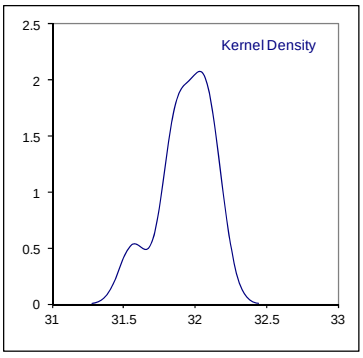


Determination of Viscosity Stabinger @ 100 °C on sample #13066; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
273		-----		-----	
315		-----		-----	
343		-----		-----	
357		-----		-----	
369		-----		-----	
372	D7042	31.89		-2.62	
432		-----		-----	
494	D7042	32.030		1.04	
496	D7042	31.864		-3.31	
541	D7042	32.16		4.45	
551		-----		-----	
603		-----		-----	
704		-----		-----	
781		-----		-----	
875		-----		-----	
902		-----		-----	
922	D7042	31.851		-3.65	
963	D7042	31.57	G(0.05)	-11.01	
974	D7042	32.071		2.12	
1013		-----		-----	
1082		-----		-----	
1161		-----		-----	
1191		-----		-----	
1231		-----		-----	
1243		-----		-----	
1262	D7042	32.065		1.96	
1271		-----		-----	
1340		-----		-----	
1557		-----		-----	
1564		-----		-----	
1652		-----		-----	
1706		-----		-----	
1833		-----		-----	

normality	OK
n	7
outliers	1
mean (n)	31.990
st.dev. (n)	0.1210
R(calc.)	0.339
R(D7042:11)	0.107

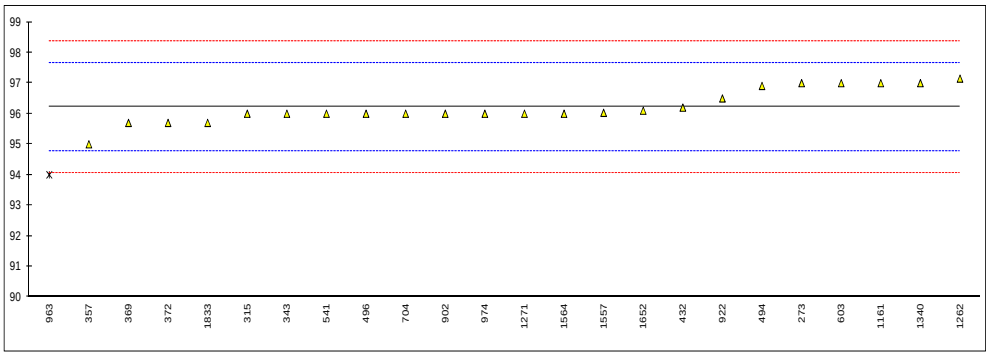


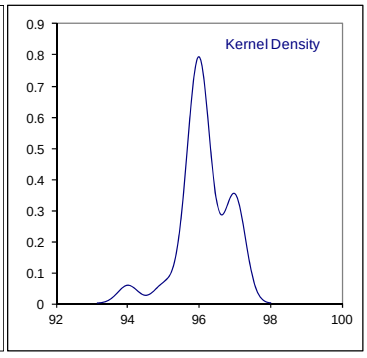


Determination of Viscosity index on sample #13066

lab	method	value	mark	z(targ)	remarks
273	D2270	97		1.10	
315	D2270	96		-0.30	
343	D2270	96		-0.30	
357	D2270	95		-1.70	
369	D2270	95.7		-0.72	
372	D2270	95.7		-0.72	
432	D2270	96.2		-0.02	
494	D2270	96.91		0.97	
496	D2270	96.0		-0.30	
541	D2270	96		-0.30	
551		-----		-----	
603	D2270	97		1.10	
704	D2270	96		-0.30	
781		-----		-----	
875		-----		-----	
902	D2270	96		-0.30	
922	D2270	96.5		0.40	
963	D2270	94	C,G(0.05)	-3.10	First reported 95
974	D2270	96		-0.30	
1013		-----		-----	
1082		-----		-----	
1161	D2270	97		1.10	
1191		-----		-----	
1231		-----		-----	
1243		-----		-----	
1262	D2270	97.15		1.31	
1271	ISO2909	96		-0.30	
1340	ISO2909	97		1.10	
1557	D2270	96.03		-0.26	
1564	D2270	96		-0.30	
1652	ISO2909	96.1		-0.16	
1706		-----		-----	
1833	D2270	95.7		-0.72	

normality	not OK
n	23
outliers	1
mean (n)	96.22
st.dev. (n)	0.549
R(calc.)	1.54
R(D2270:10e1)	2.00

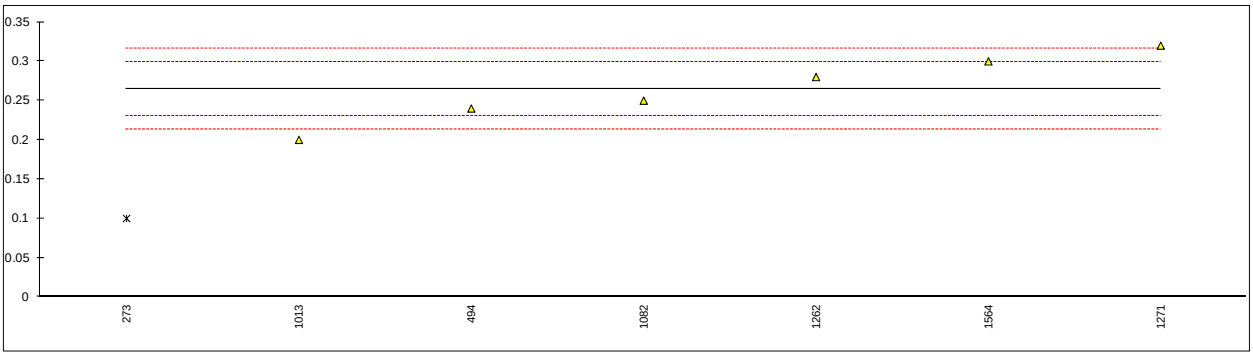




Determination of Evaporation loss by Noack test on sample #13066; results in %M/M

lab	method	value	mark	z(targ)	remarks
273	D5800	0.1	G(0.05)	-9.53	
315		-----		-----	
343		-----		-----	
357		-----		-----	
369		-----		-----	
372		-----		-----	
432		-----		-----	
494	D5800	0.24		-1.44	
496		-----		-----	
541		-----		-----	
551		-----		-----	
603		-----		-----	
704		-----		-----	
781		-----		-----	
875		-----		-----	
902		-----		-----	
922		-----		-----	
963		-----		-----	
974		-----		-----	
1013	CEC-L-40-93	0.2	C	-3.75	First reported 0.1
1082	CECL-40-93	0.25		-0.87	
1161		-----		-----	
1191		-----		-----	
1231		-----		-----	
1243		-----		-----	
1262	D5800	0.28		0.87	
1271	D5800	0.32		3.18	
1340		-----		-----	
1557		-----		-----	
1564	DIN51581	0.3		2.02	
1652		-----		-----	
1706		-----		-----	
1833		-----		-----	

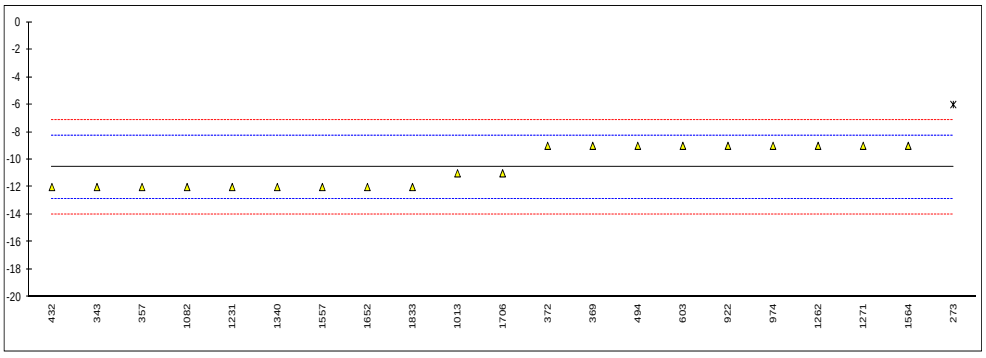
normality	OK
n	6
outliers	1
mean (n)	0.265
st.dev. (n)	0.0437
R(calc.)	0.122
R(D5800:10)	0.048

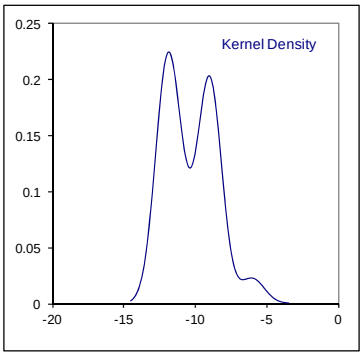


Determination of Pour Point on sample #13066; results in °C

lab	method	value	mark	z(targ)	remarks
273	D97	-6	G(0.05)	3.98	
315		-----		-----	
343	D97	-12		-1.27	
357	D5950	-12		-1.27	
369	D97	-9		1.36	
372	D5950	-9		1.36	
432	D5950	-12		-1.27	
494	D6892	-9		1.36	
496		-----		-----	
541		-----		-----	
551		-----		-----	
603	D97	-9		1.36	
704		-----		-----	
781		-----		-----	
875		-----		-----	
902		-----		-----	
922	D97	-9		1.36	
963		-----		-----	
974	D97	-9		1.36	
1013	D6892	-11		-0.39	
1082	D5950	-12		-1.27	
1161		-----		-----	
1191		-----		-----	
1231	D5950	-12		-1.27	
1243		-----		-----	
1262	D97	-9		1.36	
1271	ISO3016	-9		1.36	
1340	ISO3016	-12		-1.27	
1557	ISO3016	-12		-1.27	
1564	D97	-9		1.36	
1652	ISO3016	-12		-1.27	
1706	ISO3016	-11.0		-0.39	
1833	D5950	-12		-1.27	

	normality	not OK	
	n	20	
	outliers	1	
	mean (n)	-10.55	
	st.dev. (n)	1.468	
	R(calc.)	4.11	
	R(D6892:08)	3.20	Compare R(D97:12) = 8.0





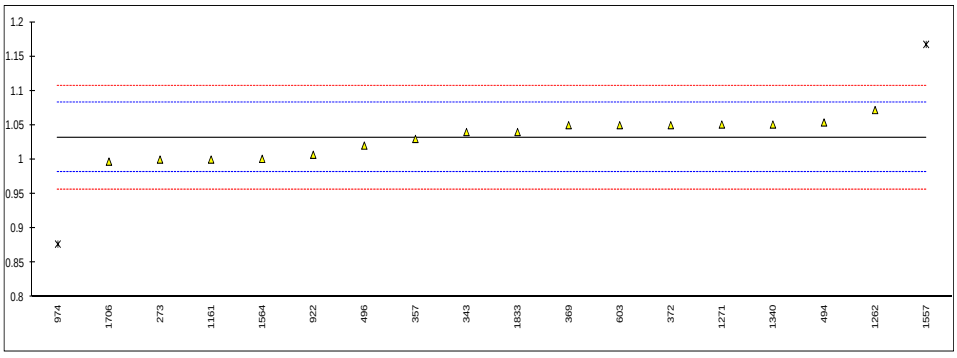
Determination of Rust prevention (proc.B) on sample #13066

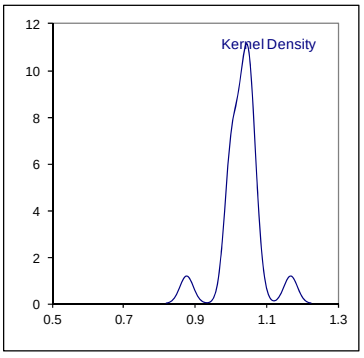
lab	method	value	mark	z(targ)	remarks
273		-----		-----	
315	D665	fails, moderate rust		-----	
343		-----		-----	
357		-----		-----	
369		-----		-----	
372		-----		-----	
432		-----		-----	
494		-----		-----	
496	D665	severe rusting		-----	
541		-----		-----	
551		-----		-----	
603		-----		-----	
704		-----		-----	
781		-----		-----	
875		-----		-----	
902		-----		-----	
922		-----		-----	
963	D665	Pass		-----	
974		-----		-----	
1013	D665	Pass		-----	
1082		-----		-----	
1161		-----		-----	
1191		-----		-----	
1231		-----		-----	
1243		-----		-----	
1262	D665	Pass		-----	
1271		-----		-----	
1340		-----		-----	
1557		-----		-----	
1564		-----		-----	
1652		-----		-----	
1706		-----		-----	
1833		-----		-----	

normality	n.a.
n	5
outliers	n.a.
mean (n)	n.a.
st.dev. (n)	n.a.
R(calc.)	n.a.
R(D665:12)	n.a.

Determination of Sulphur on sample #13066; results in %M/M

lab	method	value	mark	z(targ)	remarks
273	D4294	1.0		-1.28	
315		-----		-----	
343	ISO8754	1.04		0.31	
357	D4294	1.03		-0.08	
369	D2622	1.050		0.71	
372	D4294	1.05		0.71	
432		-----		-----	
494	D4294	1.054		0.87	
496	D2622	1.0203		-0.47	
541	D4294	>1	C	-----	First reported 0.387
551		-----		-----	
603	D4294	1.050		0.71	
704		-----		-----	
781		-----		-----	
875		-----		-----	
902		-----		-----	
922	D4294	1.007		-1.00	
963		-----		-----	
974	D4294	0.877	G(0.05)	-6.17	
1013		-----		-----	
1082		-----		-----	
1161	ISO8754	1.0		-1.28	
1191		-----		-----	
1231		-----		-----	
1243		-----		-----	
1262	D4927	1.072		1.59	
1271	D2622	1.051		0.75	
1340	ISO8754	1.051		0.75	
1557	ISO8754	1.1675	G(0.01)	5.38	
1564	D5453	1.001		-1.24	
1652		-----		-----	
1706	D2622	0.9970		-1.39	
1833	IP336	1.04		0.31	
	normality	OK			
	n	16			
	outliers	2			
	mean (n)	1.0321			
	st.dev. (n)	0.02437			
	R(calc.)	0.0682			
	R(D2622:10)	0.0704			Compare R (D4294:10) = 0.0742

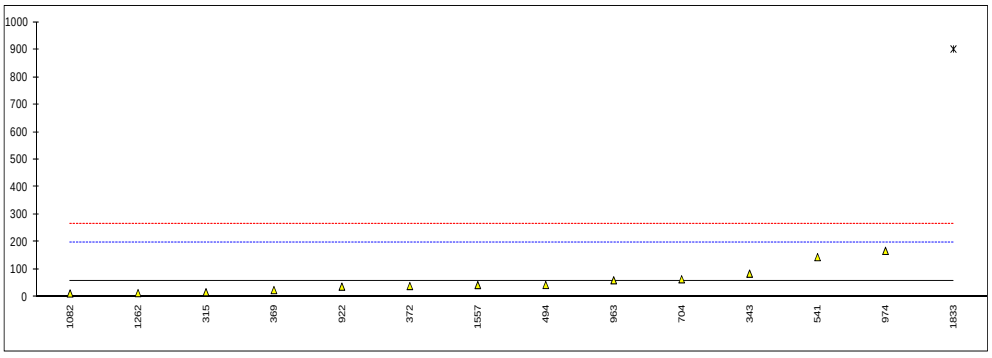


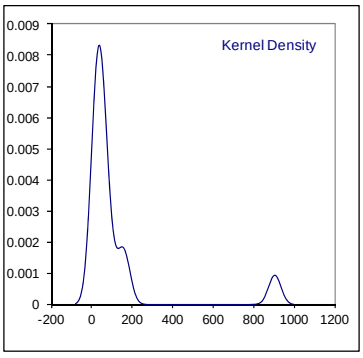


Determination of Water on sample #13066; results in mg/kg

lab	method	value	mark	z(targ)	remarks
273		-----		-----	
315	D6304	17.46		-0.59	
343	E203	85		0.38	
357		-----		-----	
369	D6304	25.3		-0.48	
372	D6304	40		-0.27	
432		-----		-----	
494	D6304	44		-0.21	
496	D6304	<10		<-0.70	False negative?
541	D6304	145		1.25	
551		-----		-----	
603		-----		-----	
704	D6304	64.6		0.09	
781		-----		-----	
875		-----		-----	
902		-----		-----	
922	D6304	37.8		-0.30	
963	D6304	61		0.04	
974	D6304	167.78		1.58	
1013		-----		-----	
1082	D6304	13		-0.66	
1161		-----		-----	
1191		-----		-----	
1231		-----		-----	
1243		-----		-----	
1262	D6304	14.3		-0.64	
1271		-----		-----	
1340		-----		-----	
1557	EN12937	43.53		-0.21	
1564		-----		-----	
1652		-----		-----	
1706		-----		-----	
1833	D6304	902	C,G(0.01)	12.19	First reported 215.9884

normality	OK
n	13
outliers	1
mean (n)	58.37
st.dev. (n)	48.464
R(calc.)	135.70
R(D6304:07)	193.81





Determination of Water Separability on sample #13066

lab	method	value	Time (min)	mark	z(targ)	remarks
273		-----		-----		
315		-----		-----		
343		-----		-----		
357		-----		-----		
369		-----		-----		
372		-----		-----		
432	ISO6614	0-38-42	25	-----		Wrong reporting? Oil – water - emulsion
494		-----		-----		
496		-----		-----		
541		-----		-----		
551		-----		-----		
603		-----		-----		
704		-----		-----		
781		-----		-----		
875		-----		-----		
902		-----		-----		
922		-----		-----		
963		-----		-----		
974		-----		-----		
1013	D1401	40-40-0	20	-----		
1082		-----		-----		
1161	D1401	<0.1		-----		Deviating reporting?
1191		-----		-----		
1231		-----		-----		
1243		-----		-----		
1262	D1401	40-39-1	20	-----		
1271		-----		-----		
1340		-----		-----		
1557		-----		-----		
1564		-----		-----		
1652		-----		-----		
1706		-----		-----		
1833		-----		-----		

normality	n.a.
n	3
outliers	0
mean (n)	21.7
st.dev. (n)	2.89
R(calc.)	8.1
R(D1401:12)	25

APPENDIX 2

Number of participants per country

1 laboratory in	ARGENTINA
2 laboratories in	AUSTRIA
2 laboratories in	BOSNIA and HERZEGOVINA
1 laboratory in	BRAZIL
1 laboratory in	BULGARIA
1 laboratory in	ESTONIA
3 laboratories in	FINLAND
3 laboratories in	GERMANY
1 laboratory in	LATVIA
1 laboratory in	MALAYSIA
1 laboratory in	PAKISTAN
1 laboratory in	POLAND
1 laboratory in	PORTUGAL
2 laboratories in	RUSSIA
1 laboratory in	SAUDI ARABIA
1 laboratory in	SERBIA
1 laboratory in	SOUTH AFRICA
2 laboratories in	SPAIN
1 laboratory in	THAILAND
1 laboratory in	THE NETHERLANDS
3 laboratories in	TURKEY
1 laboratory in	U.A.E.
1 laboratory in	UKRAINE

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 E178:08

3 ISO 5725-86

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6 ISO17043:2010

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)

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10 DIN 38402 T41/42

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

13 Analytical Methods Committee Technical brief, No4 January 2001.

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