Institute for
Interlaboratory Studies

Results of Proficiency Test
Gear Oil (fresh)
March 2023

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Report: iis23L01

May 2023

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1 INTRODUCTION

Since 2015 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of fresh Gear Oil every year. During the annual proficiency testing program 2022/2023 it was decided to continue the round robin for the analysis of fresh Gear Oil.

In this interlaboratory study 21 laboratories in 15 countries registered for participation, see appendix 2 for the number of participants per country. In this report the results of the Gear Oil (fresh) proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.
It was decided to send two identical samples of fresh Gear Oil in a 1 L bottle and a 0.5 L bottle both labelled #23030.
The participants were requested to report rounded and unrounded test results. The unrounded test 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/IEC17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. 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 regular basis by sending out questionnaires.

2.2 PROTOCOL

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

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

A batch of approximately 200 liters of fresh Gear Oil was obtained from a local supplier. After homogenization 35 amber glass bottles of 1 L and 35 amber glass bottles of 0.5 L were filled and labelled #23030.
The homogeneity of the subsamples was checked by determination of Density at 15 °C in accordance with ISO12185 on 8 stratified randomly selected subsamples.

	Density at 15 °C in kg/L
sample #23030-1	0.88103
sample #23030-2	0.88103
sample #23030-3	0.88103
sample #23030-4	0.88103
sample #23030-5	0.88103
sample #23030-6	0.88102
sample #23030-7	0.88103
sample #23030-8	0.88103

Table 1: homogeneity test results of subsamples #23030

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Density at 15 °C in kg/L
r (observed)	0.00001
reference test method	ISO12185:96
0.3 x R (reference test method)	0.00015

Table 2: evaluation of the repeatability of subsamples #23030

The calculated repeatability is in agreement with 0.3 times the reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one 1 L bottle and one 0.5 L bottle both labelled #23030 were sent on February 15, 2023. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of fresh Gear Oil packed in amber glass bottles was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYZES

The participants were requested to determine: Total Acid Number, Copper Corrosion 3 hrs at 100 °C, Density at 15 °C, Flash Point (C.O.C. and PMcc), Foaming Tendency and Foam Stability, Kinematic Viscosity at 40 °C and 100 °C, Viscosity Index, Pour Point (Manual and Automated), Rust Prevention distilled water, Sulfur, Water, Water Separability at 82 °C, Calcium as Ca, Phosphorus as P and Zinc as Zn.
Some extra information was asked about the determinations of Total Acid Number and Foaming Characteristics.

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 evaluations.

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods (when applicable) 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 reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report ‘iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation’ of June 2018 (iis-protocol, version 3.5).

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

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

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

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

3.2 GRAPHICS

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

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms.

Also, a normal Gauss curve (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

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 (derived from e.g. ISO or ASTM test methods), 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, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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

The z-scores were calculated according to:

$z_{(target)}$ = (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. Therefore, the usual interpretation of z-scores is as follows:

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

4 EVALUATION

In this proficiency test no problems were encountered with the dispatch of the samples. One participant reported test results after the final reporting date and four other participants did not report any test results. Not all participants were able to report all tests requested. In total 17 participants reported 216 numerical test results. Observed were 10 outlying test results, which is 4.6%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

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

4.1 EVALUATION PER TEST

In this section the reported test results are discussed per test. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 3.

In the iis PT reports ASTM test methods are referred to with a number (e.g. D5950) and an added designation for the year that the test method was adopted or revised (e.g. D5950:14). When a method has been reapproved an “R” will be added and the year of approval (e.g. D5950:14R20).

Total Acid Number: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D664-A:18e2 for the end point modes IP 60 mL, IP 125 mL and BEP 125 mL, but is in agreement for end point mode BEP 60 mL.

Copper Corrosion: This determination was not problematic. All reporting participants agreed on a test result of 1 (1a/1b).

Density at 15 °C: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ISO12185:96 and ASTM D4052:22.

Flash Point C.O.C.: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D92:18.

Flash Point PMcc: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D93-A:20.

Foaming Characteristics (Tendency and Stability): This determination was problematic. In total three statistical outliers were observed over Foaming Tendency sequence I to III. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D892:18e1 for Foaming sequence III. For Foaming Tendency sequence I and II the variation in the test results was too large. Therefore, no z-scores are calculated.
For Foam Stability sequence I, II and III all reporting participants agreed on a test result of 0 (Nil). Therefore, no z-scores are calculated.

Kinematic Viscosity at 40 °C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D445:21e2.

Kinematic Viscosity at 100 °C: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D445:21e2.

Viscosity Index: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D2270:10R16.

Pour Point Manual: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D97:17bR22.

Pour Point Automated: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5950:14R20.

Rust Prevention: This determination was not problematic. All reporting participants agreed on a test result of “Pass”.

Sulfur: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D4294:21.

Water: This determination was problematic. No statistical outliers were observed. A new version of ASTM D6304 was published in 2020 with major changes. In the 2016 version one precision statement was mentioned for test results based on mass with a broad application range and one based on volume. In the 2020 version all precision statements are based on mass with three different procedures (A - direct injection, B - oven accessory and C - evaporation accessory) each with a different application range. In ASTM D6304:20 the reproducibility for all three procedures is much stricter compared to ASTM D6304:16e1. It was decided to use procedure B for the 2023 PTs of Gear Oil (fresh) and Gear Oil (used).
The calculated reproducibility is not in agreement with the requirements of ASTM D6304:20 for all three procedures A, B and C.

Water Separability at 82 °C: Only two participants reported test results for the Time to reach parameters. Therefore, no z-scores are calculated. For the volumes of the different separation phases eight participants reported test results. Unfortunately, the test results show two groups. Two participants reported a complete breakdown while six other participants observed a large emulsion phase.

Calcium as Ca: This determination was not problematic. All reporting participants agreed on a value near or below the limit of detection. Therefore, no z-scores are calculated.

Phosphorus as P: 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 ASTM D5185:18.

Zinc as Zn: This determination was not problematic. All reporting participants agreed on a value near or below the limit of detection. Therefore, no z-scores are calculated.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Parameter	unit	n	average	2.8 * sd	R(lit)
Total Acid Number	mg KOH/g	12	0.75	0.37	0.32
Copper Corrosion 3 hrs at 100 °C		10	1 (1a/1b)	n.a.	n.a.
Density at 15 °C	kg/L	15	0.8811	0.0007	0.0005
Flash Point C.O.C.	°C	10	234	20	18
Flash Point PMcc	°C	13	194	9	14
Foaming Tendency (Seq I) 5 min	mL	8	18	29	(11)
Foaming Tendency (Seq II) 5 min	mL	9	66	142	(41)
Foaming Tendency (Seq III) 5 min	mL	7	6	13	10
Foam Stability (Seq I) 10 min	mL	9	0	n.e.	n.e.
Foam Stability (Seq II) 10 min	mL	9	0	n.e.	n.e.
Foam Stability (Seq III) 10 min	mL	9	0	n.e.	n.e.
Kinematic Viscosity at 40 °C	mm^2/s	15	99.48	1.20	1.21
Kinematic Viscosity at 100 °C	mm^2/s	12	11.341	0.103	0.157
Viscosity Index		10	99.8	0.8	2
Pour Point Manual	°C	6	-23.0	4.3	9
Pour Point Automated 1 °C int.	°C	8	-24.0	4.7	4.5
Rust Prevention distilled water		7	Pass	n.a.	n.a.
Sulfur	mg/kg	10	9202	625	689
Water	mg/kg	11	88	171	146
Water Separability at 82 °C					
Time to ≤ 3 mL emulsion	minutes	2	n.e.	n.e.	n.e.
Time to 37 mL water	minutes	2	n.e.	n.e.	n.e.
Time to complete break	minutes	1	n.e.	n.e.	n.e.
Calcium as Ca	mg/kg	12	<40	n.e.	n.e.
Phosphorus as P	mg/kg	11	299	34	74
Zinc as Zn	mg/kg	13	<60	n.e.	n.e.

Table 3: reproducibilities of tests on sample #23030. For results between brackets no z-scores are calculated.

Without further statistical calculations it can be concluded that for many tests there is a good compliance of the group of participants with the reference test methods. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF MARCH 2023 WITH PREVIOUS PTS

	March 2023	March 2022	March 2021	March 2020	April 2019
Number of reporting laboratories	17	20	24	21	23
Number of test results	216	315	390	384	400
Number of statistical outliers	10	9	18	32	14
Percentage of statistical outliers	4.6%	2.9%	4.6%	8.3%	3.5%

Table 4: comparison with previous proficiency tests

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

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

Parameter	March 2023	March 2022	March 2021	March 2020	April 2019
Total Acid Number	-	+	-	+	+
Density at 15 °C	-	+	+	-	+
Flash Point C.O.C.	-	-	-	--	--
Flash Point PMcc	+	+	+	++	+
Foaming Tendency and Stability	-	-	-	--	--
Kinematic Viscosity at 40 °C	+/-	+	+	+	+/-
Kinematic Viscosity at 100 °C	+	+	+	+/-	+
Viscosity Index	++	+	+	+/-	+
Pour Point Manual	++	-	+	+/-	+/-
Pour Point Automated 1 °C int.	+/-	-	++	--	(--)
Sulfur	+/-	-	--	+/-	-
Water	-	+	--	++	++
Water Separability at 82 °C	n.e.	++	+	++	++
Calcium as Ca	n.e.	n.e.	n.e.	(--)	(--)
Phosphorus as P	++	++	+	++	+
Zinc as Zn	n.e.	n.e.	n.e.	(--)	(--)

Table 5: comparison of determinations to the reference test methods. For results between brackets no z-scores are calculated.

The following performance categories were used:

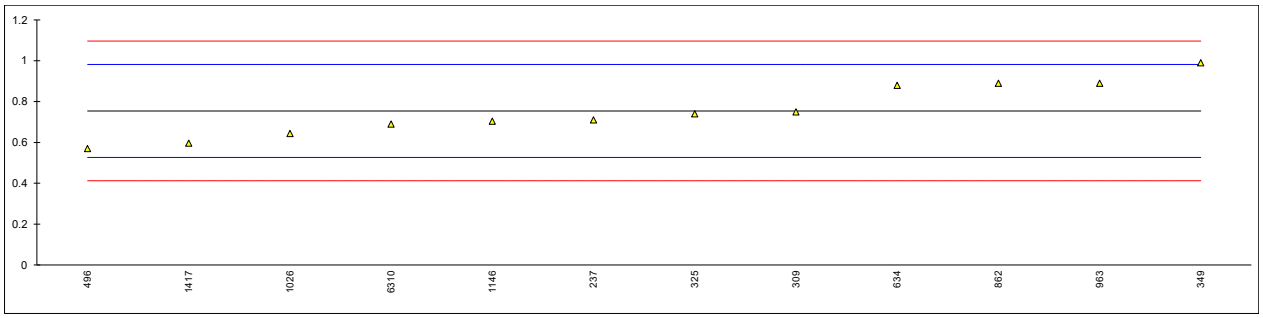
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test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

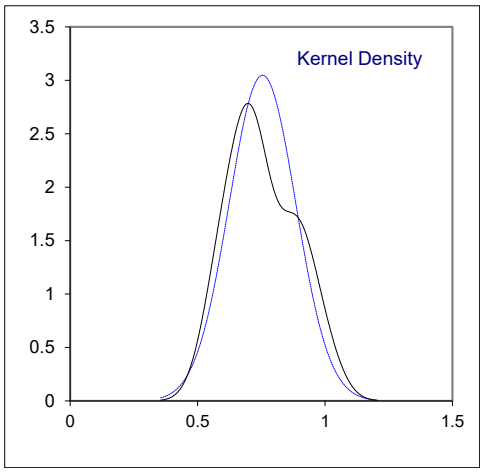
APPENDIX 1

Determination of Total Acid Number on sample #23030; results in mg KOH/g

lab	method	value	mark	z(targ)	end point type	volume (mL)	remarks
178		-----		-----	---	---	
179		-----		-----	---	---	
237	D664-B	0.710		-0.39	Inflection Point	125 mL	
256		-----		-----	---	---	
257		-----		-----	---	---	
309	D664-A	0.749		-0.05	Buffer End Point pH 10	125 mL	
325	D664-A	0.74		-0.13	Buffer End Point pH 10	125 mL	
329		-----		-----	---	---	
349	D664-A	0.99		2.07	Inflection Point	125 mL	
432		-----		-----	---	---	
496	D664-A	0.57		-1.62	Buffer End Point pH 10	60 mL	
634	D664-A	0.88		1.10	---	---	
862	D664-A	0.89		1.19	Inflection Point	60 mL	
963	D664-B	0.89		1.19	Inflection Point	60 mL	
1026	D664-A	0.6431		-0.98	Buffer End Point pH 10	125 mL	
1146	D664-A	0.704		-0.44	Buffer End Point pH 10	125 mL	
1417	D664-A	0.596		-1.39	Inflection Point	60 mL	
1748		-----		-----	---	---	
6310	D664-A	0.69		-0.56	Buffer End Point pH 10	60 mL	
6442		-----		-----	---	---	
6515		-----		-----	---	---	

normality	OK
n	12
outliers	0
mean (n)	0.7543
st.dev. (n)	0.13088
R(calc.)	0.3665
st.dev.(D664-A:18e2, IP 60 mL)	0.11400
R(D664-A:18e2, IP 60 mL)	0.3192
Compare:	
R(D664-A:18e2, IP 125 mL)	0.1631
R(D664-A:18e2, BEP 60 mL)	0.4209
R(D664-A:18e2, BEP 125 mL)	0.2349





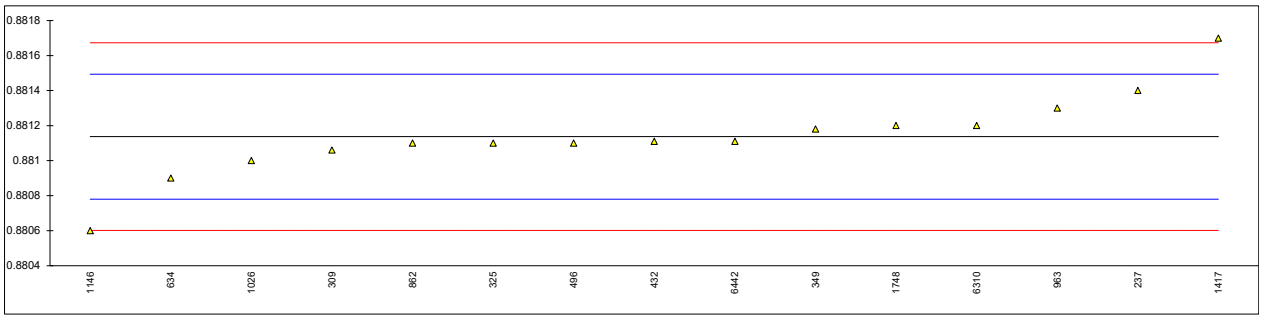
Determination of Copper Corrosion 3 hrs at 100 °C on sample #23030;

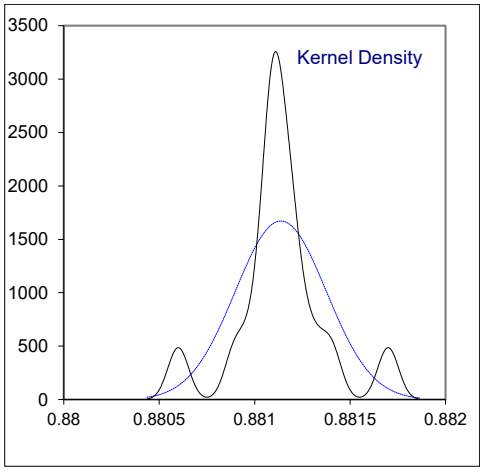
lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D130	1		-----	
256		-----		-----	
257		-----		-----	
309	D130	1A		-----	
325	D130	1B		-----	
329		-----		-----	
349		-----		-----	
432		-----		-----	
496	ISO2160	1A		-----	
634	D130	1a		-----	
862	D130	1a		-----	
963		-----		-----	
1026	D130	1A		-----	
1146		-----		-----	
1417	D130	1a		-----	
1748	D130	1a		-----	
6310		-----		-----	
6442	D130	1b		-----	
6515		-----		-----	
	n	10			
	mean (n)	1 (1a/1b)			

Determination of Density at 15 °C on sample #23030; results in kg/L

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D4052	0.8814		1.47	
256		-----		-----	
257		-----		-----	
309	D4052	0.88106		-0.43	
325	D4052	0.8811		-0.21	
329		-----		-----	
349	D4052	0.88118		0.24	
432	D4052	0.88111		-0.15	
496	ISO12185	0.8811		-0.21	
634	D4052	0.8809		-1.33	
862	D4052	0.8811		-0.21	
963	D4052	0.8813		0.91	
1026	D4052	0.8810		-0.77	
1146	D4052	0.8806		-3.01	
1417	IP365	0.8817	C	3.15	first reported 881.9 kg/m^3
1748	D4052	0.8812		0.35	
6310	D4052	0.8812		0.35	
6442	D4052	0.88111		-0.15	
6515		-----		-----	

	normality	not OK
	n	15
	outliers	0
	mean (n)	0.881137
	st.dev. (n)	0.0002386
	R(calc.)	0.000668
	st.dev.(ISO12185:96)	0.0001786
	R(ISO12185:96)	0.0005
Compare:		
	R(D4052:22)	0.00050

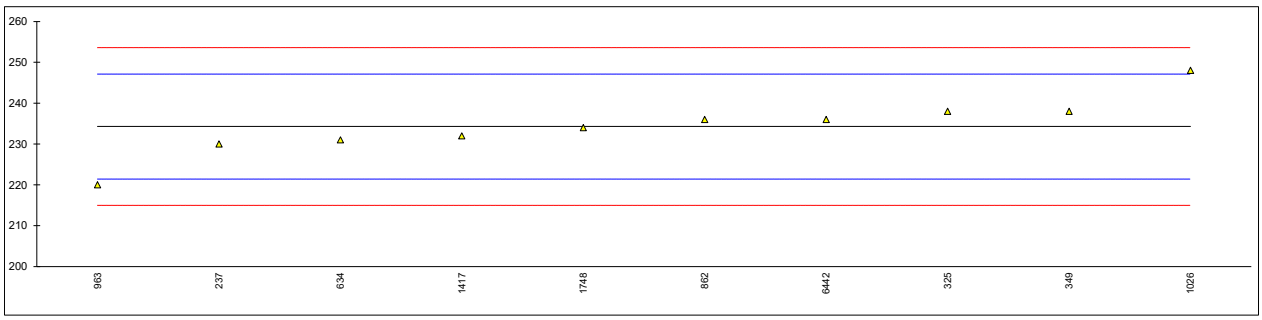


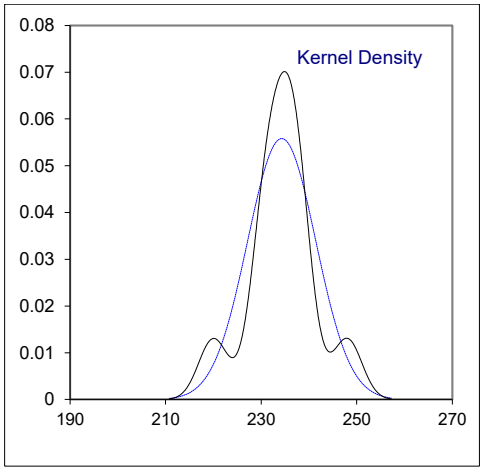


Determination of Flash Point C.O.C. on sample #23030; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D92	230		-0.67	
256		-----		-----	
257		-----		-----	
309		-----		-----	
325	D92	238		0.58	
329		-----		-----	
349	D92	238		0.58	
432		-----		-----	
496		-----		-----	
634	D92	231		-0.51	
862	D92	236		0.26	
963	D92	220.0		-2.22	
1026	D92	248		2.13	
1146		-----		-----	
1417	D92	232		-0.36	
1748	D92	234		-0.05	
6310		-----		-----	
6442	D92	236		0.26	
6515		-----		-----	

normality	not OK
n	10
outliers	0
mean (n)	234.30
st.dev. (n)	7.150
R(calc.)	20.02
st.dev.(D92:18)	6.429
R(D92:18)	18

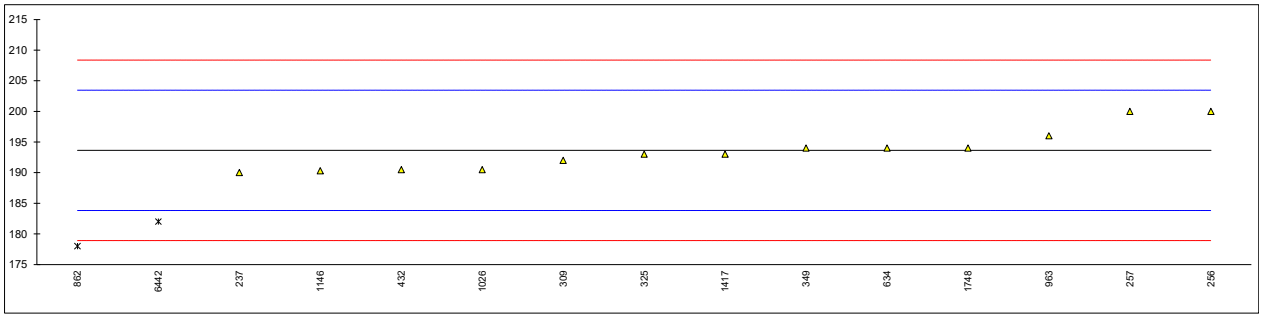


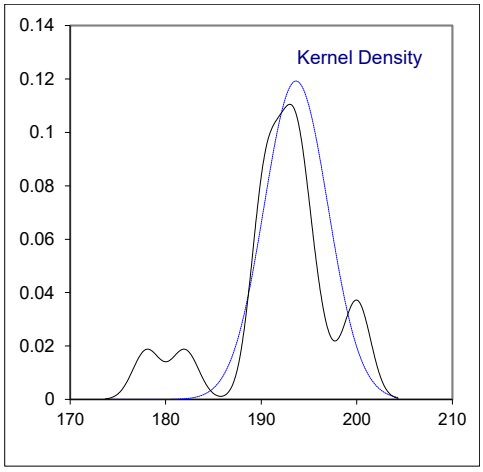


Determination of Flash Point PMcc on sample #23030; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D93-A	190.0		-0.74	
256	D3828	200.0		1.30	
257	D3828	200.0		1.30	
309	D93-A	192.0		-0.33	
325	D93-A	193.0		-0.13	
329		-----		-----	
349	D93-A	194		0.07	
432	D93-A	190.5		-0.64	
496		-----		-----	
634	D93-A	194.0		0.07	
862	D93-A	178	D(0.05)	-3.18	
963	D93-A	196.0		0.48	
1026	D93-A	190.5		-0.64	
1146	D93-A	190.3		-0.68	
1417	D93-A	193.0		-0.13	
1748	D93-A	194		0.07	
6310		-----		-----	
6442	D93-A	182.0	D(0.05)	-2.37	
6515		-----		-----	

normality	OK
n	13
outliers	2
mean (n)	193.64
st.dev. (n)	3.348
R(calc.)	9.37
st.dev.(D93-A:20)	4.910
R(D93-A:20)	13.75

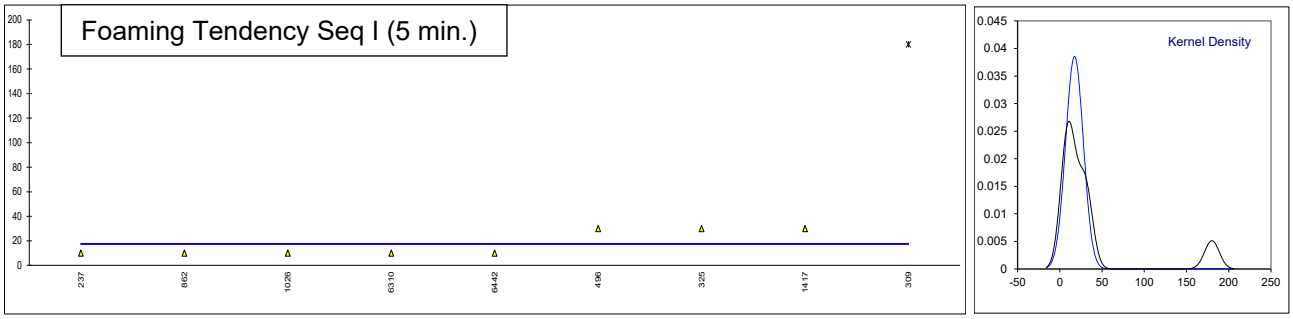


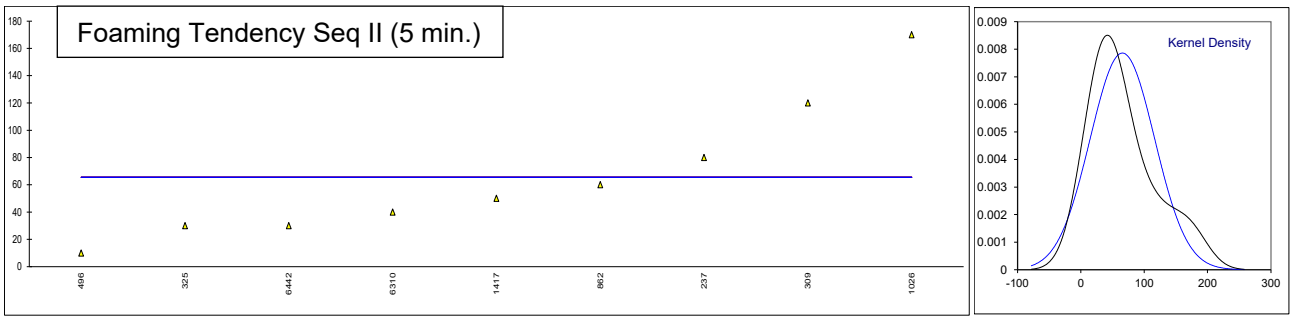


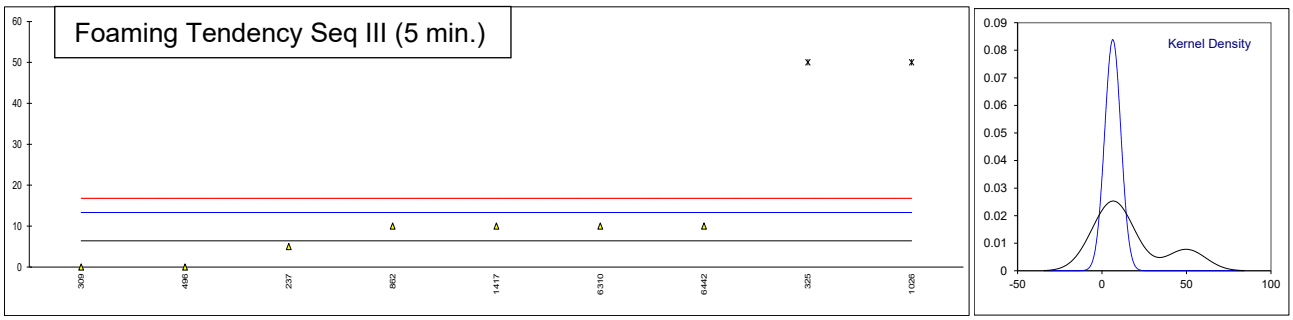
Determination of Foaming Tendency, Sequence I, II and III (5 min. blowing period) on sample #23030; results in mL

lab	method	Sample	Diffuser	Seq I	mark	z(targ)	Seq II	mark	z(targ)	Seq III	mark	z(targ)
178		---	---	-----		-----	-----		-----	-----		-----
179		---	---	-----		-----	-----		-----	-----		-----
237		As received	Metal	10		-----	80		-----	5		-0.41
256		---	---	-----		-----	-----		-----	-----		-----
257		---	---	-----		-----	-----		-----	-----		-----
309	D892	After agit/pre-heat	Metal	180	G(1)	-----	120		-----	0		-1.86
325	D892	As received	Stone	30	C	-----	30	C	-----	50	C,DG(1)	12.58
329		---	---	-----		-----	-----		-----	-----		-----
349		---	---	-----		-----	-----		-----	-----		-----
432		---	---	-----		-----	-----		-----	-----		-----
496	D892	---	Metal	30		-----	10		-----	0		-1.86
634		---	---	-----		-----	-----		-----	-----		-----
862	D892	As received	Metal	10		-----	60		-----	10		1.03
963		---	---	-----		-----	-----		-----	-----		-----
1026	D892	As received	Metal	10		-----	170		-----	50	DG(1)	12.58
1146		---	---	-----		-----	-----		-----	-----		-----
1417	D892	As received	Metal	30		-----	50		-----	10		1.03
1748		---	---	-----		-----	-----		-----	-----		-----
6310	D892	After agitation (A)	Stone	10		-----	40		-----	10		1.03
6442	D892 (Altern.)	After agitation (A)	Metal	10		-----	30		-----	10		1.03
6515		---	---	-----		-----	-----		-----	-----		-----
	normality			OK			not OK			OK		
	n			8			9			7		
	outliers			1			0			2		
	mean (n)			17.50			65.56			6.43		
	st.dev. (n)			10.351			50.772			4.756		
	R(calc.)			28.98			142.16			13.32		
	st.dev.(D892:18e1)			(3.846)			(14.601)			3.463		
	R(D892:18e1)			(10.77)			(40.88)			9.70		

Lab 325 first reported 130, 160 and 10 respectively







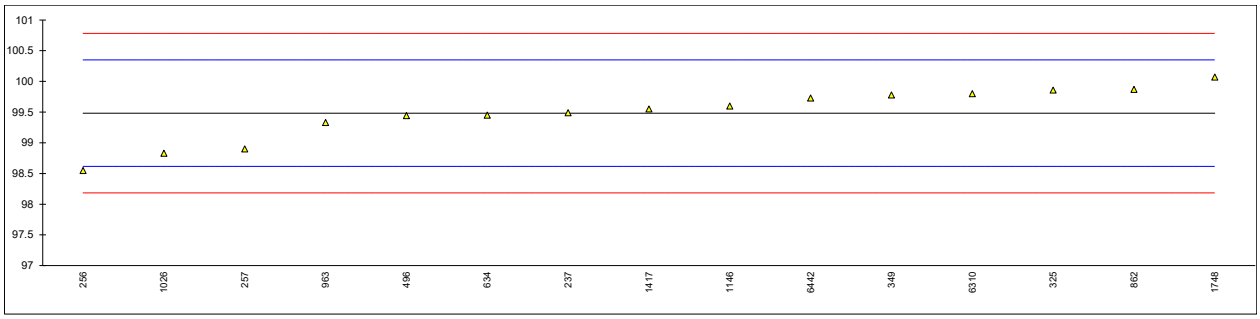
Determination of Foam Stability, Sequence I, II and III (10 min. settling period) on sample #23030; results in mL

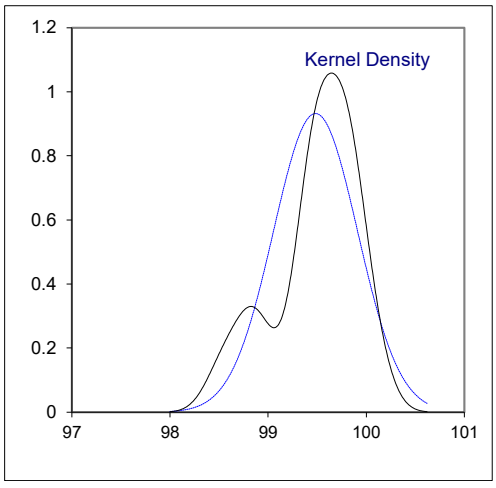
lab	method	Seq I	mark	z(targ)	Seq II	mark	z(targ)	Seq III	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179		-----		-----	-----		-----	-----		-----
237	D892	0		-----	0		-----	0		-----
256		-----		-----	-----		-----	-----		-----
257		-----		-----	-----		-----	-----		-----
309	D892	0		-----	0		-----	0		-----
325	D892	0		-----	0		-----	0		-----
329		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
432		-----		-----	-----		-----	-----		-----
496	D892	0		-----	0		-----	0		-----
634		-----		-----	-----		-----	-----		-----
862	D892	0		-----	0		-----	0		-----
963		-----		-----	-----		-----	-----		-----
1026	D892	0		-----	0		-----	0		-----
1146		-----		-----	-----		-----	-----		-----
1417	D892	0		-----	0		-----	0		-----
1748		-----		-----	-----		-----	-----		-----
6310	D892	0		-----	0		-----	0		-----
6442	D892 (Alternative)	0		-----	0		-----	0		-----
6515		-----		-----	-----		-----	-----		-----
	n	9			9			9		
	mean (n)	0			0			0		

Determination of Kinematic Viscosity at 40 °C on sample #23030; results in mm²/s

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D445	99.49		0.01	
256	D7279 corrected to D445	98.55		-2.15	
257	D7279 corrected to D445	98.90		-1.35	
309		-----		-----	
325	D445	99.86		0.87	
329		-----		-----	
349	D445	99.78		0.68	
432		-----		-----	
496	D445	99.444		-0.09	
634	D445	99.45		-0.08	
862	D445	99.87		0.89	
963	D445	99.331		-0.35	
1026	D445	98.83		-1.51	
1146	D445	99.60		0.27	
1417	D445	99.55		0.15	
1748	D7042	100.07		1.35	
6310	D7279 corrected to D445	99.8	C	0.73	first reported 11.31
6442	D7042	99.73		0.57	
6515		-----		-----	

normality	OK
n	15
outliers	0
mean (n)	99.484
st.dev. (n)	0.4282
R(calc.)	1.199
st.dev.(D445:21e2)	0.4335
R(D445:21e2)	1.214

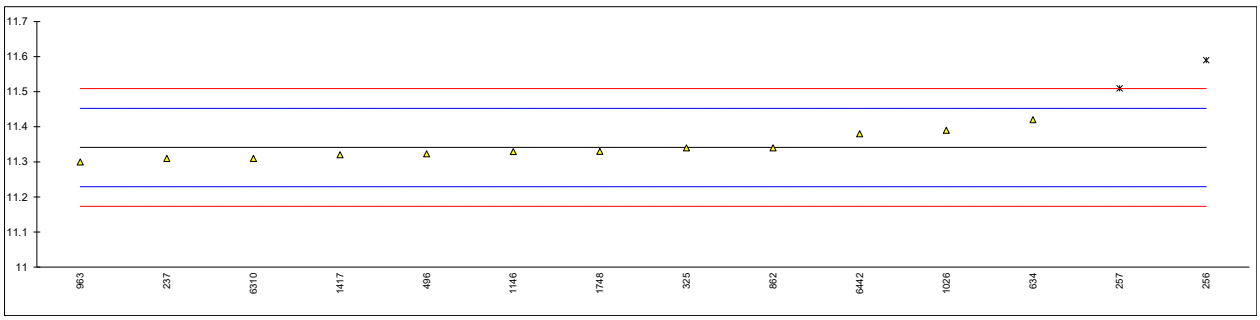


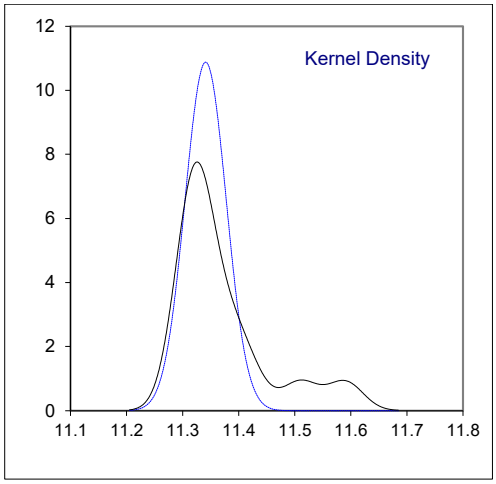


Determination of Kinematic Viscosity at 100 °C on sample #23030; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D445	11.31		-0.55	
256	D7279 corrected to D445	11.59	G(0.05)	4.45	
257	D7279 corrected to D445	11.51	G(0.05)	3.02	
309		-----		-----	
325	D445	11.34		-0.02	
329		-----		-----	
349		-----		-----	
432		-----		-----	
496	D445	11.323		-0.32	
634	D445	11.42		1.41	
862	D445	11.34		-0.02	
963	D445	11.30		-0.73	
1026	D445	11.39		0.88	
1146	D445	11.329		-0.21	
1417	D445	11.32		-0.38	
1748	D7042	11.33		-0.20	
6310	D7279 corrected to D445	11.31	C	-0.55	first reported 99.8
6442	D7042	11.38		0.70	
6515		-----		-----	

normality	suspect
n	12
outliers	2
mean (n)	11.341
st.dev. (n)	0.0367
R(calc.)	0.103
st.dev.(D445:21e2)	0.0559
R(D445:21e2)	0.157

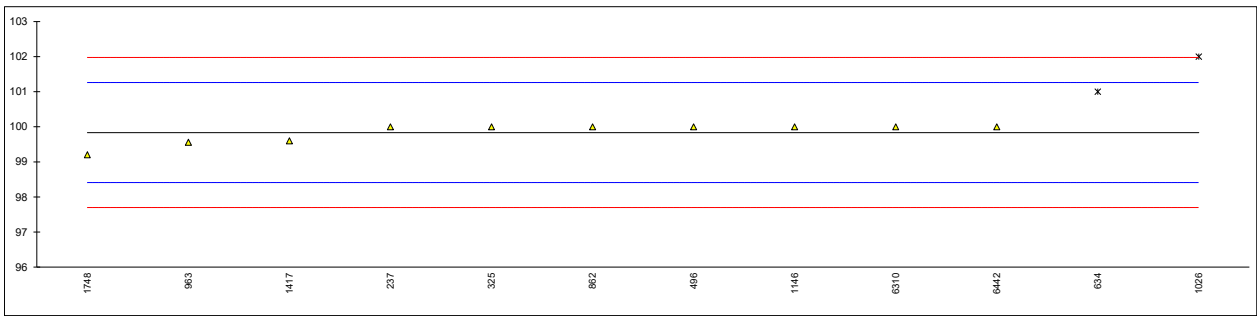


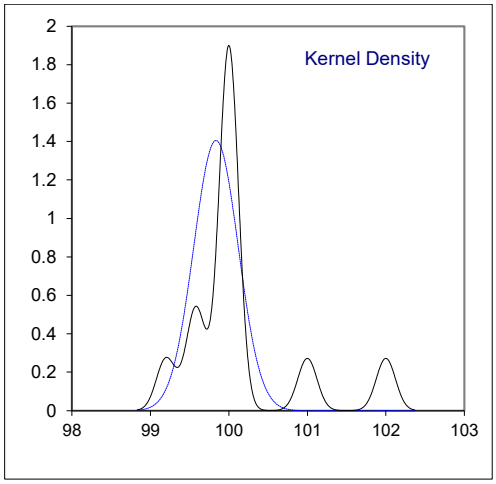


Determination of Viscosity Index on sample #23030

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D2270	100		0.23	
256		-----		-----	
257		-----		-----	
309		-----		-----	
325	D2270	100		0.23	
329		-----		-----	
349		-----		-----	
432		-----		-----	
496	D2270	100		0.23	
634	D2270	101	G(0.05)	1.63	
862	D2270	100		0.23	
963	D2270	99.56		-0.39	
1026	D2270	102	G(0.05)	3.03	
1146	D2270	100		0.23	
1417	D2270	99.6		-0.33	
1748	D2270	99.2		-0.89	
6310	D2270	100	E	0.23	calculation difference, iis calculated 99
6442	D2270	100		0.23	
6515		-----		-----	

normality	suspect
n	10
outliers	2
mean (n)	99.84
st.dev. (n)	0.284
R(calc.)	0.79
st.dev.(D2270:10R16)	0.714
R(D2270:10R16)	2

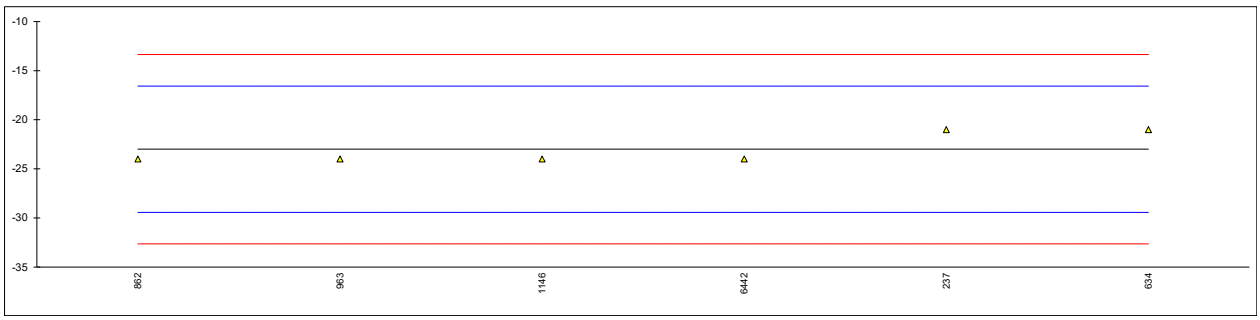




Determination of Pour Point Manual on sample #23030; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D97	-21		0.62	
256		-----		-----	
257		-----		-----	
309		-----		-----	
325		-----		-----	
329		-----		-----	
349		-----		-----	
432		-----		-----	
496		-----		-----	
634	D97	-21		0.62	
862	D97	-24		-0.31	
963	D97	-24		-0.31	
1026		-----		-----	
1146	D97	-24		-0.31	
1417		-----		-----	
1748		-----		-----	
6310		-----		-----	
6442	D97	-24		-0.31	
6515		-----		-----	

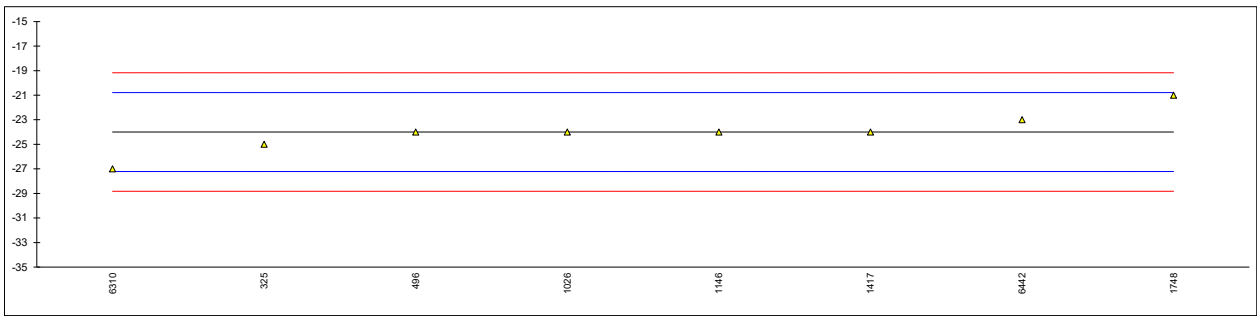
normality	unknown
n	6
outliers	0
mean (n)	-23.00
st.dev. (n)	1.549
R(calc.)	4.34
st.dev.(D97:17bR22)	3.214
R(D97:17bR22)	9

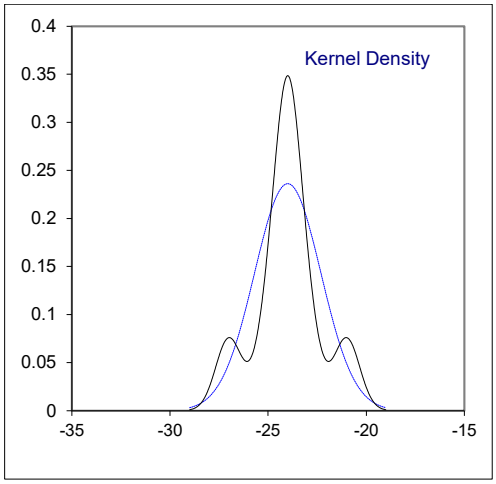


Determination of Pour Point Automated 1 °C interval on sample #23030; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
256		-----		-----	
257		-----		-----	
309		-----		-----	
325	D5950	-25		-0.62	
329		-----		-----	
349		-----		-----	
432		-----		-----	
496	D5950	-24		0.00	
634		-----		-----	
862		-----		-----	
963		-----		-----	
1026	D5950	-24		0.00	
1146	D6892	-24		0.00	
1417	D5950	-24		0.00	
1748	D7346	-21		1.87	
6310	D5950	-27		-1.87	
6442	D6892	-23		0.62	
6515		-----		-----	

normality	unknown
n	8
outliers	0
mean (n)	-24.00
st.dev. (n)	1.690
R(calc.)	4.73
st.dev.(D5950:14R20)	1.607
R(D5950:14R20)	4.5





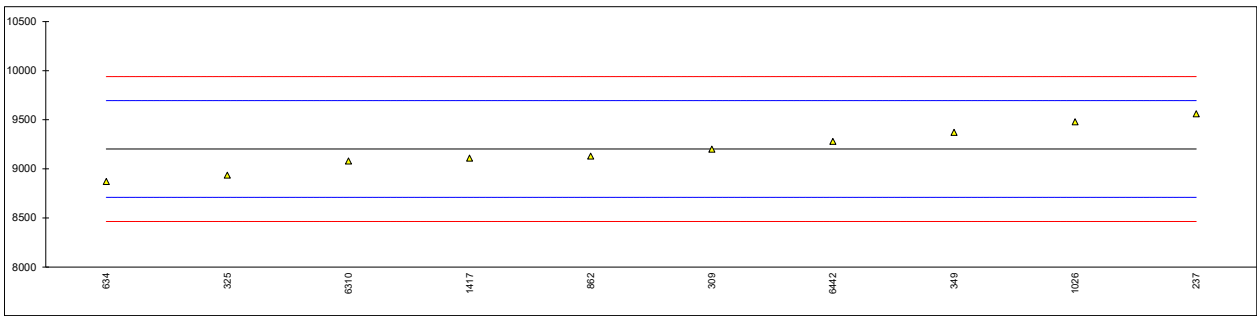
Determination of Rust Prevention distilled water on sample #23030

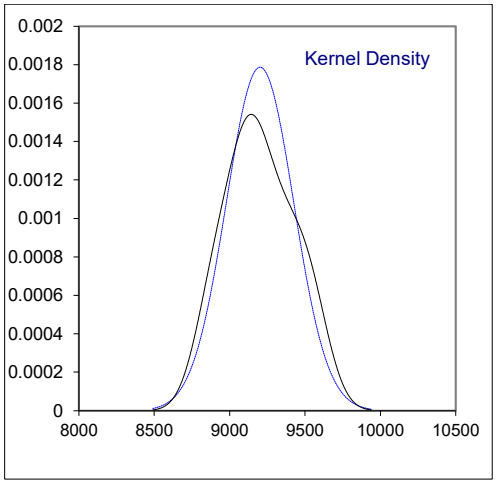
lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D665	PASS		-----	
256		-----		-----	
257		-----		-----	
309	D665	PASSES		-----	
325	D665	Pass		-----	
329		-----		-----	
349		-----		-----	
432		-----		-----	
496		-----		-----	
634		-----		-----	
862	D665	No Rusting		-----	
963		-----		-----	
1026	D665	Pass		-----	
1146		-----		-----	
1417	D665	PASS		-----	
1748		-----		-----	
6310		-----		-----	
6442	D665	Pass		-----	
6515		-----		-----	
	n	7			
	mean (n)	Pass			

Determination of Sulfur on sample #23030; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D4294	9560		1.46	
256		-----		-----	
257		-----		-----	
309	ISO8754	9200		-0.01	
325	D5185	8936		-1.08	
329		-----		-----	
349	D2622	9373	C	0.69	first reported 10692
432		-----		-----	
496		-----		-----	
634	D4294	8872		-1.34	
862	D2622	9130		-0.29	
963		-----		-----	
1026	D2622	9480		1.13	
1146		-----		-----	
1417	In house	9110		-0.37	
1748		-----		-----	
6310	D7751	9080	C	-0.50	first reported 908
6442	D6481	9280		0.32	
6515		-----		-----	

normality	OK
n	10
outliers	0
mean (n)	9202.1
st.dev. (n)	223.35
R(calc.)	625.4
st.dev.(D4294:21)	245.96
R(D4294:21)	688.7

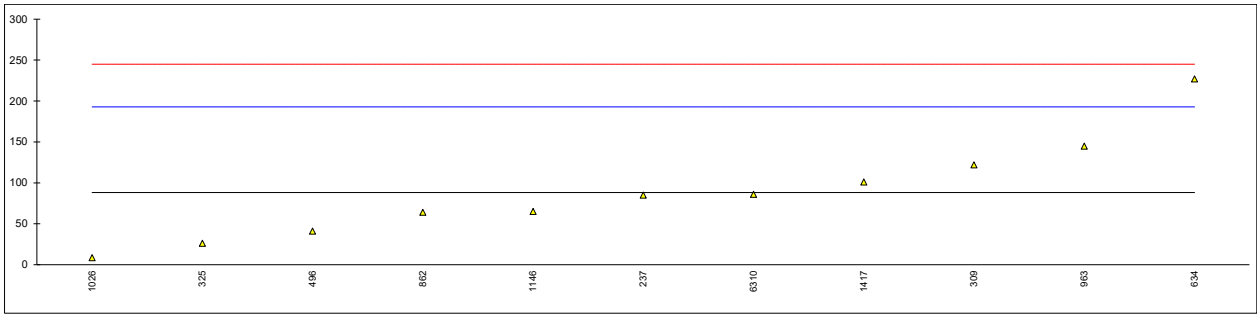


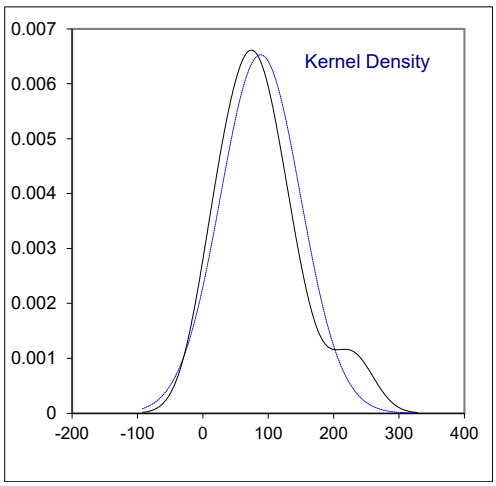


Determination of Water on sample #23030; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D6304-C:16e1	85		-0.06	
256		-----		-----	
257		-----		-----	
309	D6304-A:20	122		0.65	
325	D6304-C:16e1	26		-1.19	
329		-----		-----	
349		-----		-----	
432		-----		-----	
496	D6304-B:20	41		-0.90	
634	D6304-A:20	227		2.66	
862	D6304-B	64		-0.46	
963	D6304-A:20	145		1.09	
1026	D6304-C:20	8.64115		-1.52	
1146	D6304-B:20	65		-0.45	
1417	D6304-A:20	101		0.24	
1748		-----		-----	
6310	D6304-C:16e1	86		-0.04	
6442		-----		-----	
6515		-----		-----	

	normality	not OK
	n	11
	outliers	0
	mean (n)	88.24
	st.dev. (n)	61.125
	R(calc.)	171.15
	st.dev.(D6304-B:20)	52.215
	R(D6304-B:20)	146.20
Compare:		
	R(D6304-A:20)	55.10
	R(D6304-C:20)	40.38





Determination of Water Separability at 82 °C, distilled water on sample #23030; results in minutes

lab	method	3 mL or less emulsion	z(targ)	37 mL of water	z(targ)	complete break (40-40-0)	z(targ)	test aborted	time aborted
178		-----	-----	-----	-----	-----	-----	-----	-----
179		-----	-----	-----	-----	-----	-----	-----	-----
237		-----	-----	-----	-----	-----	-----	-----	-----
256		-----	-----	-----	-----	-----	-----	-----	-----
257		-----	-----	-----	-----	-----	-----	-----	-----
309	D1401	10	-----	10	-----	-----	-----	-----	10
325		-----	-----	-----	-----	-----	-----	Yes	60
329		-----	-----	-----	-----	-----	-----	-----	-----
349		-----	-----	-----	-----	-----	-----	-----	-----
432		-----	-----	-----	-----	-----	-----	-----	-----
496		-----	-----	-----	-----	-----	-----	Yes	60
634		-----	-----	-----	-----	-----	-----	-----	-----
862		-----	-----	-----	-----	-----	-----	-----	60
963		-----	-----	-----	-----	-----	-----	-----	-----
1026		-----	-----	-----	-----	-----	-----	No	60
1146		-----	-----	-----	-----	-----	-----	Yes	30
1417	D1401	35	-----	35	-----	35	-----	No	-----
1748		-----	-----	-----	-----	-----	-----	-----	-----
6310		-----	-----	-----	-----	-----	-----	Yes	60
6442		-----	-----	-----	-----	-----	-----	-----	-----
6515		-----	-----	-----	-----	-----	-----	-----	-----
	n	2		2		1			

Determination of Water Separability at 82 °C, distilled water on sample #23030; results in mL

lab	method	volume oil phase	mark	volume water phase	mark	volume emulsion phase	mark
178		-----		-----		-----	
179		-----		-----		-----	
237		-----		-----		-----	
256		-----		-----		-----	
257		-----		-----		-----	
309	D1401	41		39		0	
325	D1401	1		8		71	
329		-----		-----		-----	
349		-----		-----		-----	
432		-----		-----		-----	
496	D1401	2		5		73	
634		-----		-----		-----	
862	D1401	5		25		50	
963		-----		-----		-----	
1026	D1401	12		35		33	
1146		4		15		61	
1417	D1401	37	C	40		3	
1748		-----		-----		-----	
6310	D1401	2		12		66	
6442		-----		-----		-----	
6515		-----		-----		-----	

Lab 1417 first reported 40

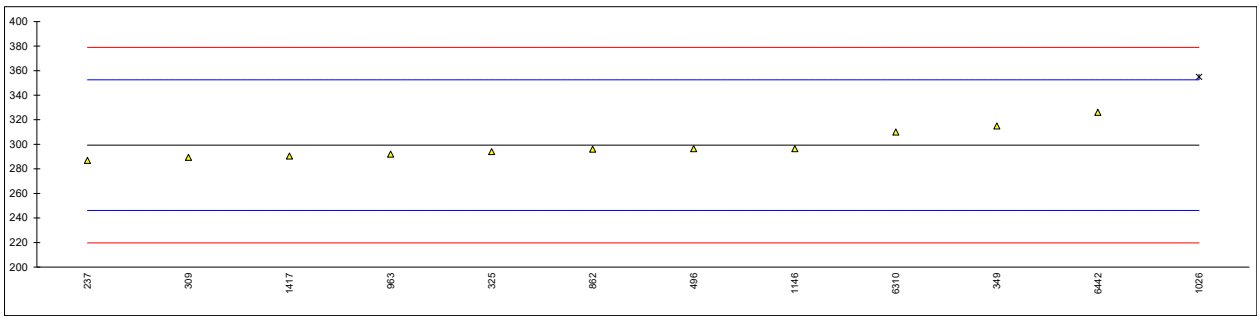
Determination of Calcium as Ca on sample #23030; results in mg/kg

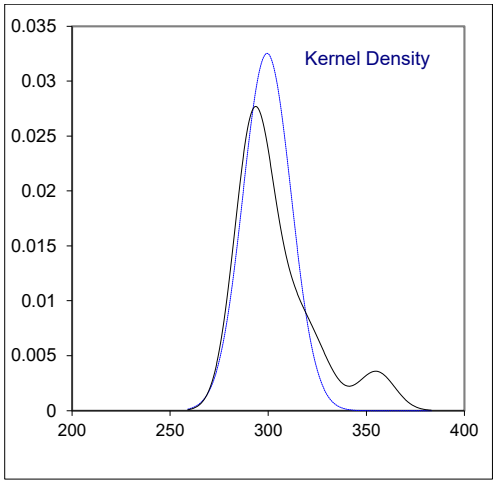
lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D5185	<40		-----	
256		-----		-----	
257	D6595	0.27		-----	
309	D5185	0.57		-----	
325	D5185	1		-----	
329		-----		-----	
349	D5185	2		-----	
432		-----		-----	
496	D5185	1.501		-----	
634		-----		-----	
862	D5185	<1		-----	
963	D5185	0.40		-----	
1026	D5185	0		-----	
1146	In house	<5		-----	
1417	D5185	0.689		-----	
1748		-----		-----	
6310	D7751	0		-----	
6442		-----		-----	
6515		-----		-----	
	n	12			
	mean (n)	<40			D5185:18 application range 40 – 9000 mg/kg

Determination of Phosphorus as P on sample #23030; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D5185	287		-0.47	
256		-----		-----	
257		-----		-----	
309	D5185	289.484		-0.37	
325	D5185	294		-0.20	
329		-----		-----	
349	D5185	315		0.59	
432		-----		-----	
496	D5185	296.5		-0.11	
634		-----		-----	
862	D5185	296		-0.13	
963	D5185	292.04		-0.28	
1026	D5185	355	G(0.05)	2.09	
1146	In house	296.55		-0.11	
1417	D5185	290.5		-0.33	
1748		-----		-----	
6310	D7751	310		0.40	
6442	D5185	326		1.00	
6515		-----		-----	

normality	suspect
n	11
outliers	1
mean (n)	299.37
st.dev. (n)	12.272
R(calc.)	34.36
st.dev.(D5185:18)	26.571
R(D5185:18)	74.40





Determination of Zinc as Zn on sample #23030; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D5185	<60		-----	
256	In house	5.0		-----	
257	D6595	0.14		-----	
309	D5185	0.08		-----	
325	D5185	<1		-----	
329		-----		-----	
349	D5185	2		-----	
432		-----		-----	
496	D5185	1.008		-----	
634		-----		-----	
862	D5185	<1		-----	
963	D5185	2.85		-----	
1026	D5185	0		-----	
1146	In house	<5		-----	
1417	D5185	0.981		-----	
1748		-----		-----	
6310	D7751	0		-----	
6442		-----		-----	
6515		-----		-----	
	n	13			
	mean (n)	<60			D5185:18 application range 60 – 1600 mg/kg

APPENDIX 2

Number of participants per country

1 lab in	AUSTRIA
3 labs in	BELGIUM
1 lab in	CHINA, People's Republic
1 lab in	GERMANY
1 lab in	JORDAN
1 lab in	MOROCCO
3 labs in	NETHERLANDS
1 lab in	NIGERIA
1 lab in	PHILIPPINES
1 lab in	PORTUGAL
1 lab in	SAUDI ARABIA
1 lab in	SPAIN
2 labs in	TANZANIA
1 lab 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)\G(1)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)\DG(1)	= 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	= calculation difference between reported test result and result calculated by iis
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
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?
SDS	= Safety Data Sheet

Literature

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