Results of Proficiency Test
Gear Oil (fresh)
March 2021

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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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 2020/2021 it was decided to continue the round robin for the analysis of fresh Gear Oil.

In this interlaboratory study 26 laboratories in 20 different countries registered for participation. See appendix 2 for the number of participants per country. In this report the results of the fresh Gear Oil 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 1L bottle and a 0.5L bottle both labelled #21030.
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 60 liters of fresh Gear Oil was obtained from a local refinery. After homogenization 40 amber glass bottles of 1L and 34 amber glass bottles of 0.5L were filled and labelled #21030.
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 #21030-1	0.88515
Sample #21030-2	0.88514
Sample #21030-3	0.88514
Sample #21030-4	0.88514
Sample #21030-5	0.88515
Sample #21030-6	0.88512
Sample #21030-7	0.88515
Sample #21030-8	0.88515

Table 1: homogeneity test results of subsamples #21030

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)	0.00003
reference test method	ISO12185:96
0.3 x R (reference test method)	0.00015

Table 2: evaluation of the repeatability of subsamples #21030

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 1L bottle and one 0.5L bottle both labelled #21030 were sent on February 24, 2021. 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, Level of Contamination (counts/mL and scale number), Calcium, Phosphorus and Zinc.
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, e.g. ISO reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, 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 some problems were encountered with the dispatch of the samples. Two participants reported test results after the final reporting date and two other participants did not report any test results. Not all participants were able to report all tests requested.
In total 24 participants reported 390 numerical test results. Observed were 18 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. 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. D7647) and an added designation for the year that the test method was adopted or revised (e.g. D7647:10). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D7647:10(2018)). In the results tables of appendix 1 only the method number and year of adoption or revision (e.g. D7647:10) will be used.

Total Acid Number: 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 D664-A:18e2 IP 60mL. It is also not in agreement with IP 125mL or BEP 125mL, but it is in agreement with BEP 60mL. Remarkably, two participants still used pH 11 for BEP instead of pH 10. In test method ASTM D664:18e2 pH 10 is mentioned.

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

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

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. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D93-A:20.

Foaming Characteristics (Tendency and Stability): This determination was problematic. Two statistical outliers were observed over six parameters. The calculated reproducibilities after rejection of the statistical outliers in the Foaming Tendency determination for sequence I, II and III are not in agreement with the requirements of ASTM D892:18.
For the test results of Foaming Tendency sequence I the variation in the test results was very high. Therefore, no z-scores were calculated.
Almost all reporting participants reported 0mL for Foam Stability for sequence I, II and III. Therefore, no z-scores were calculated.

Kinematic Viscosity at 40°C: 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 D445:19a.

Kinematic Viscosity at 100°C: 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 D445:19a.

Viscosity Index: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D2270:10(2016). The Viscosity Index was also calculated by iis from the test results reported for the Kinematic Viscosities at 40°C and 100°C. One difference in the calculation was observed.

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

Pour Point Automated: 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 D5950:14.

Rust Prevention: This determination was not problematic. All reporting participants agreed on a classification as “Pass”.

Sulfur: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D4294:16e1.

Water: This determination was very problematic. No statistical outliers were observed. The calculated reproducibility is not at all in agreement with the requirements of ASTM D6304-C:20.

Water Separability: This determination was not problematic. In total three statistical outliers were observed in 6 parameters. The calculated reproducibilities of “time to reach 3mL or less emulsion” and “time to reach 37mL of water” after rejection of the statistical outliers are in agreement with the requirements of ASTM D1401:19, but it is not in agreement for “time to reach complete break”.

Level of Contamination: This determination was very problematic. In total eight statistical outliers were observed and fifteen other test results were excluded over six parameters. Only the calculated reproducibility for counts/mL (≥14µm) after rejection of the suspect data is in agreement with the requirements of ASTM D7647:10(2018).
All other calculated reproducibilities for counts/mL (≥4µm and ≥6µm) and acc. ISO4406 scale number (≥4µm, ≥6µm and ≥14µm) after rejection of the suspect data are not at all in agreement with the requirements of ASTM D7647:10(2018).

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

Phosphorus as P: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5185:18.

Zinc as Zn: This determination was not problematic. All reporting participants agreed on a level near or below the detection limit. Therefore, no z-scores were 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 literature reference test methods (in casu ASTM, EN and ISO test methods) are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Total Acid Number	mg KOH/g	16	0.71	0.36	0.30
Copper Corrosion 3 hrs at 100°C		12	1 (1a/1b)	n.a.	n.a.
Density at 15°C	kg/L	23	0.8852	0.0004	0.0005
Flash Point C.O.C.	°C	14	233	23	18
Flash Point PMcc	°C	17	196	11	14
Foaming Tendency (Seq I) 5min	mL	14	87	254	(38)
Foaming Tendency (Seq II) 5min	mL	13	43	58	31
Foaming Tendency (Seq III) 5min	mL	13	13	22	16
Foam Stability (Seq I) 10min	mL	13	0	n.e.	n.e.
Foam Stability (Seq II) 10min	mL	13	0	n.e.	n.e.
Foam Stability (Seq III) 10min	mL	13	0	n.e.	n.e.
Kinematic Viscosity at 40°C	mm^2/s	21	96.48	0.84	1.18
Kinematic Viscosity at 100°C	mm^2/s	21	11.070	0.082	0.153
Viscosity Index		19	99.4	1.3	2
Pour Point Manual	°C	10	-23.4	5.3	9
Pour Point Automated	°C	7	-24.1	1.1	4.5
Rust Prevention distilled water		6	Pass	n.a.	n.a.
Sulfur	mg/kg	9	10925	1613	769
Water	mg/kg	18	165	145	67
Water Separability at 82°C					
Time to ≤ 3 mL emulsion	minutes	8	33.9	16.6	25
Time to 37 mL water	minutes	11	33.6	16.4	25
Time to complete break	minutes	9	42.5	28.9	25
Level of Contamination					
≥ 4µm (c)	counts/mL	6	758	1640	856
≥ 6µm (c)	counts/mL	6	112	220	85
≥14µm (c)	counts/mL	4	4.4	4.9	5.9

Parameter	unit	n	average	2.8 * sd	R(lit)
≥ 4µm (c)	scale no	7	16.9	3.0	1.7
≥ 6µm (c)	scale no	8	14.9	6.6	1.2
≥14µm (c)	scale no	6	9.7	4.9	2
Calcium as Ca	mg/kg	19	<40	n.e.	n.e.
Phosphorus as P	mg/kg	18	304	60	75
Zinc as Zn	mg/kg	19	<60	n.e.	n.e.

Table 3: reproducibilities of tests on sample #21030

NB. For results between brackets no z-scores are calculated

Without further statistical calculations, it can be concluded that for most 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 2021 WITH PREVIOUS PTS

	March 2021	March 2020	April 2019	April 2018	April 2017
Number of reporting laboratories	24	21	23	18	14
Number of test results	390	384	400	350	177
Number of statistical outliers	18	32	14	14	8
Percentage of statistical outliers	4.6%	8.3%	3.5%	4.0%	4.5%

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 2021	March 2020	April 2019	April 2018	April 2017
Total Acid Number	-	+	+	+	-
Density at 15°C	+	-	+	+/-	+/-
Flash Point C.O.C.	-	--	--	+	-
Flash Point PMcc	+	++	+	++	+
FoamingTendency and Stability	-	--	--	-	++
Kinematic Viscosity at 40°C	+	+	+/-	++	+
Kinematic Viscosity at 100°C	+	+/-	+	++	++
Viscosity Index	+	+/-	+	+	+
Pour Point Manual	+	+/-	+/-	-	-
Pour Point Automated	++	--	(--)	+/-	++
Sulfur	--	+/-	-	--	-
Water	--	++	++	++	++
Water Separability at 82°C	+	++	++	-	+
Level of Contamination	--	+/-	--	--	n.e.

Parameter	March 2021	March 2020	April 2019	April 2018	April 2017
Calcium as Ca	n.e.	(--)	(--)	n.e.	n.e.
Phosphorus as P	+	++	+	++	++
Zinc as Zn	n.e.	(--)	(--)	n.e.	n.e.

Table 5: comparison determinations against the reference test methods

NB. For results between brackets no z-scores are calculated

The following performance categories were used:

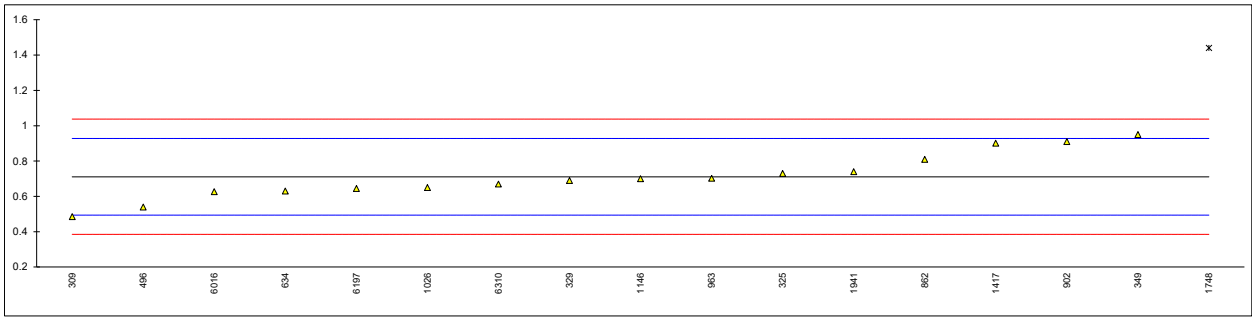
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test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

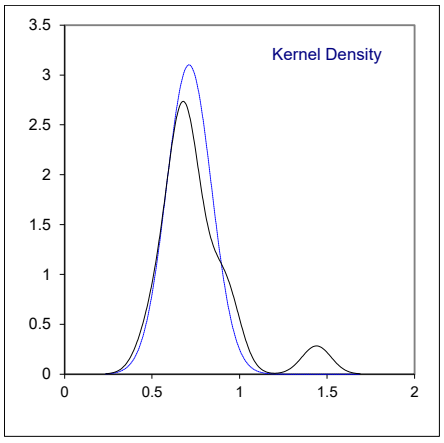
APPENDIX 1

Determination of Total Acid Number on sample #21030; results in mg KOH/g

lab	method	value	mark	z(targ)	end point type	volume (mL)	remarks
178		-----		-----	---	---	
179		-----		-----	---	---	
237		-----		-----	---	---	
257		-----		-----	---	---	
309	D664-A	0.485		-2.08	Buffer End Point pH 10	60	
325	D664-A	0.73		0.17	Buffer End Point pH 11	125	
329	D664-A	0.69		-0.20	Inflection Point	125	
349	D664-A	0.95		2.20	Buffer End Point pH 10	125	
432		-----		-----	---	---	
496	D664-A	0.54		-1.58	Buffer End Point pH 10	60	
634	D664-A	0.63	C	-0.75	---	---	fr. 1.34
862	D664-A	0.81		0.91	Inflection Point	60	
902	D664-A	0.91		1.83	Inflection Point	60	
962		-----		-----	---	---	
963	D664-A	0.702		-0.09	Inflection Point	60	
1011		-----		-----	---	---	
1026	D664-A	0.65		-0.56	Buffer End Point pH 10	125	
1146	D664-A	0.700		-0.10	Buffer End Point pH 11	125	
1213		-----		-----	---	---	
1417	D664-A	0.901		1.75	Inflection Point	60	
1448		-----		-----	---	---	
1748	D664-A	1.44	G(0.01)	6.71	Inflection Point	125	
1941	ISO6619	0.740		0.26	Inflection Point	60	
6016	D664-A	0.627		-0.78	Inflection Point	60	
6197	D664-A	0.645		-0.61	Inflection Point	60	
6310	D664-A	0.67		-0.38	Buffer End Point pH 10	60	

	normality	OK
	n	16
	outliers	1
	mean (n)	0.7113
	st.dev. (n)	0.12862
	R(calc.)	0.3601
	st.dev.(D664-A:18e2, IP 60mL)	0.10863
	R(D664-A:18e2, IP 60mL)	0.3042
Compare		
	R(D664-A:18e2, IP 125mL)	0.1534
	R(D664-A:18e2, BEP 60mL)	0.3974
	R(D664-A:18e2, BEP 125mL)	0.2209





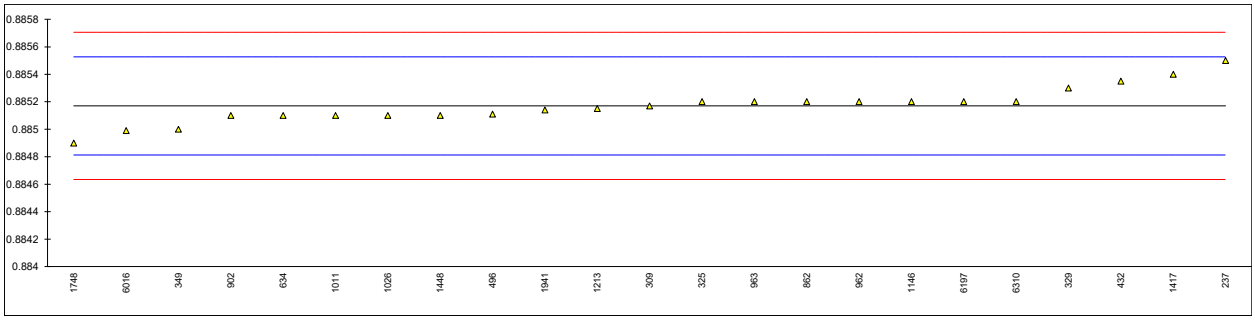
Determination of Copper Corrosion 3 hours at 100°C on sample #21030;

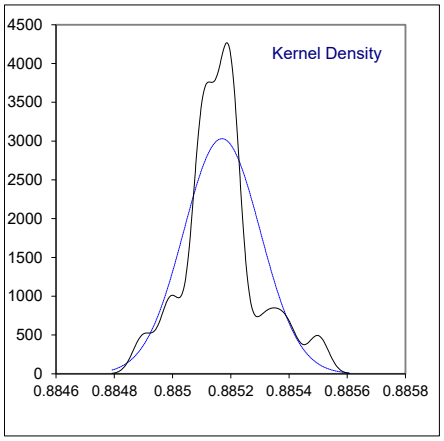
lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D130	1		-----	
257		-----		-----	
309	D130	1A		-----	
325	D130	1A		-----	
329		-----		-----	
349		-----		-----	
432		-----		-----	
496		-----		-----	
634	D130	1a		-----	
862	D130	1A		-----	
902	D130	1a		-----	
962		-----		-----	
963		-----		-----	
1011	D130	1b		-----	
1026	D130	1A		-----	
1146		-----		-----	
1213		-----		-----	
1417	IP154	1A		-----	
1448		-----		-----	
1748	D130	1b		-----	
1941	ISO2160	1b		-----	
6016		-----		-----	
6197	D130	1A		-----	
6310		-----		-----	
	n	12			
	mean (n)	1 (1a/1b)			

Determination of Density at 15°C on sample #21030; results in kg/L

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D4052	0.8855		1.85	
257		-----		-----	
309	D4052	0.88517		0.00	
325	D4052	0.8852		0.17	
329	D4052	0.8853		0.73	
349	D4052	0.8850		-0.95	
432	D4052	0.88535		1.01	
496	ISO12185	0.88511		-0.34	
634	D4052	0.8851		-0.39	
862	D4052	0.8852		0.17	
902	D4052	0.8851		-0.39	
962	ISO12185	0.8852		0.17	
963	D4052	0.8852		0.17	
1011	D4052	0.8851		-0.39	
1026	D4052	0.8851		-0.39	
1146	D4052	0.8852		0.17	
1213	D4052	0.88515		-0.11	
1417	IP365	0.8854		1.29	
1448	D4052	0.8851	C	-0.39	first reported 856.7 kg/m^3
1748	D4052	0.8849		-1.51	
1941	D4052	0.88514		-0.17	
6016	D4052	0.88499	C	-1.01	first reported 884.99 kg/L
6197	D4052	0.8852		0.17	
6310	D4052	0.8852		0.17	

	normality	suspect
	n	23
	outliers	0
	mean (n)	0.885170
	st.dev. (n)	0.0001316
	R(calc.)	0.000368
	st.dev.(ISO12185:96)	0.0001786
	R(ISO12185:96)	0.0005
Compare		
	R(D4052:18a)	0.0005

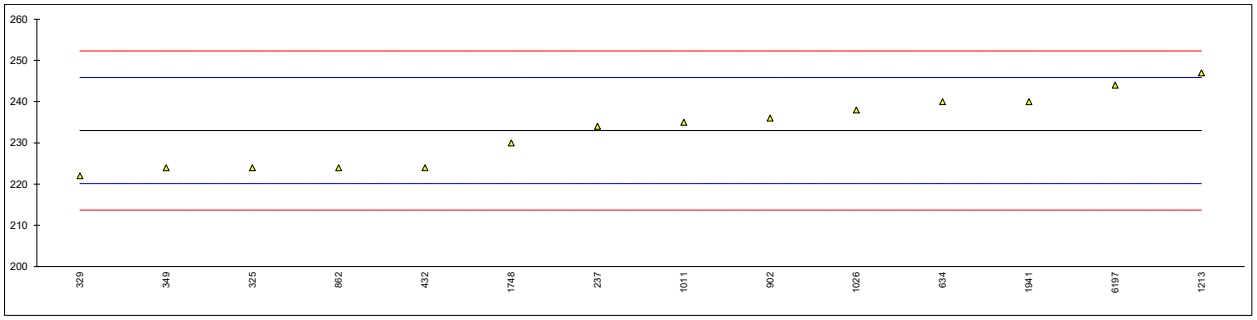


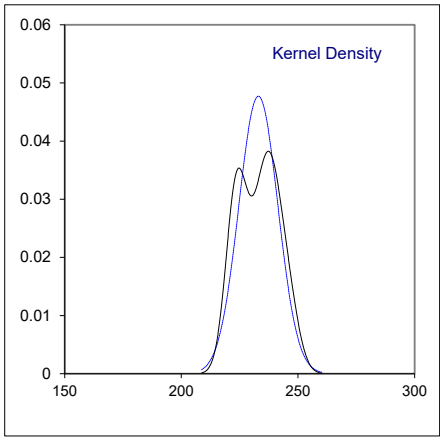


Determination of Flash Point C.O.C. on sample #21030; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D92	234		0.16	
257		-----		-----	
309		-----		-----	
325	D92	224		-1.40	
329	D92	222		-1.71	
349	D92	224		-1.40	
432	D92	224		-1.40	
496		-----		-----	
634	D92	240		1.09	
862	D92	224		-1.40	
902	D92	236		0.47	
962		-----		-----	
963		-----		-----	
1011	D92	235		0.31	
1026	D92	238		0.78	
1146		-----		-----	
1213	D92	247	C	2.18	first reported 261
1417		-----		-----	
1448		-----		-----	
1748	D92	230		-0.47	
1941	ISO2592	240		1.09	
6016		-----		-----	
6197	D92	244		1.71	
6310		-----		-----	

normality	OK
n	14
outliers	0
mean (n)	233.00
st.dev. (n)	8.357
R(calc.)	23.40
st.dev.(D92:18)	6.429
R(D92:18)	18

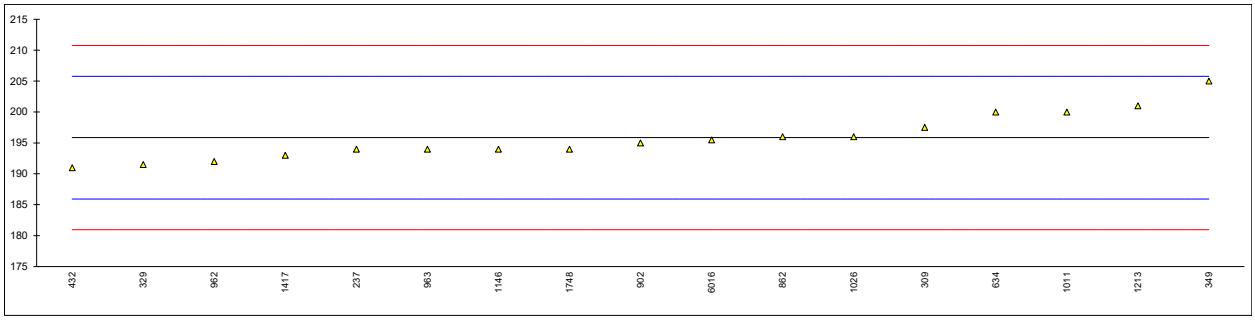


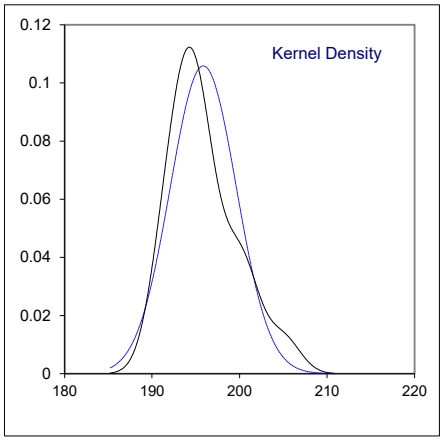


Determination of Flash Point PMcc on sample #21030; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D93-A	194.0		-0.37	
257		-----		-----	
309	D93-A	197.5		0.33	
325		-----		-----	
329	D93-A	191.5		-0.88	
349	D93-A	205		1.84	
432	D93-A	191.0		-0.98	
496		-----		-----	
634	D93-A	200.0		0.84	
862	D93-A	196		0.03	
902	D93-A	195		-0.17	
962	D93-A	192.0		-0.78	
963	D93-A	194.0		-0.37	
1011	D93	200.0		0.84	
1026	D93-A	196		0.03	
1146	D93-A	194.0		-0.37	
1213	D93-A	201		1.04	
1417	D93-A	193.0		-0.57	
1448		-----		-----	
1748	D93-A	194		-0.37	
1941		-----		-----	
6016	D93-A	195.5		-0.07	
6197		-----		-----	
6310		-----		-----	

normality	OK
n	17
outliers	0
mean (n)	195.85
st.dev. (n)	3.770
R(calc.)	10.56
st.dev.(D93-A:20)	4.966
R(D93-A:20)	13.91

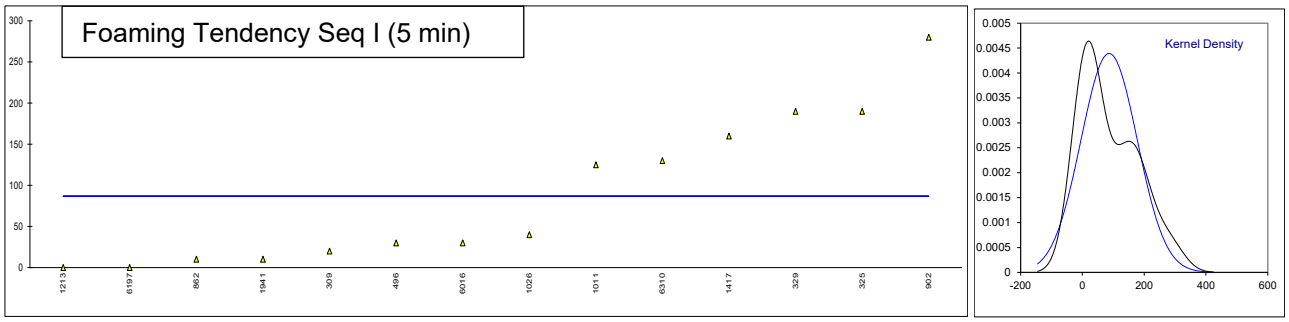


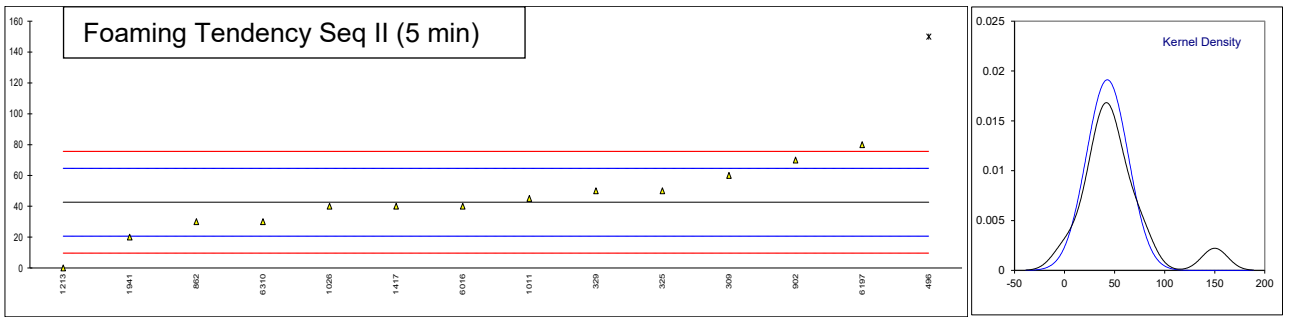


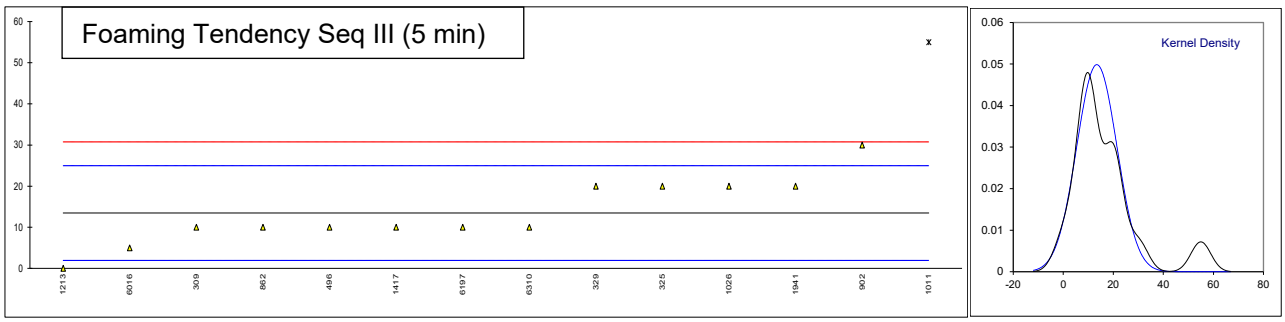
Determination of Foaming Tendency, Sequence I, II and III (5 min. blowing period) on sample #21030; results in mL

lab	method	Sample	Diffuser	Seq I	mark	z(targ)	Seq II	mark	z(targ)	Seq III	mark	z(targ)
178		---	---	-----		-----	-----		-----	-----		-----
179		---	---	-----		-----	-----		-----	-----		-----
237		---	---	-----		-----	-----		-----	-----		-----
257		---	---	-----		-----	-----		-----	-----		-----
309	D892	---	---	20		-----	60		1.57	10		-0.60
325	D892	As received	Metal	190		-----	50		0.66	20		1.14
329	D892	After agit (A)	Stone	190		-----	50		0.66	20		1.14
349		---	---	-----		-----	-----		-----	-----		-----
432		---	---	-----		-----	-----		-----	-----		-----
496	D892	As received	Metal	30		-----	150	G(0.01)	9.75	10		-0.60
634		---	---	-----		-----	-----		-----	-----		-----
862	D892	As received	Metal	10		-----	30		-1.15	10		-0.60
902	D892	After agit (A)	Metal	280	C	-----	70		2.48	30		2.87
962		---	---	-----		-----	-----		-----	-----		-----
963		---	---	-----		-----	-----		-----	-----		-----
1011	D892	---	---	125		-----	45		0.21	55	G(0.01)	7.22
1026	D892	As received	Metal	40		-----	40	C	-0.24	20		1.14
1146		---	---	-----		-----	-----		-----	-----		-----
1213	D892	---	---	0		-----	0		-3.88	0		-2.34
1417	D892	As received	Metal	160		-----	40		-0.24	10		-0.60
1448		---	---	-----		-----	-----		-----	-----		-----
1748		---	---	-----		-----	-----		-----	-----		-----
1941	ISO6247	As received	Metal	10		-----	20		-2.06	20		1.14
6016	D892	After agit (A)	Stone	30		-----	40		-0.24	5		-1.47
6197	D892	After agit (A)	Metal	0		-----	80		3.39	10		-0.60
6310	D892	After agit (A)	Metal	130		-----	30		-1.15	10		-0.60
	normality			OK			OK			OK		
	n			14			13			13		
	outliers			0			1			1		
	mean (n)			86.79			42.69			13.46		
	st.dev. (n)			90.84			20.88			8.01		
	R(calc.)			254.36			58.46			22.42		
	st.dev.(D892:18)			(13.65)			11.00			5.76		
	R(D892:18)			(38.21)			30.81			16.11		

Lab 902 first reported 320
Lab 1026 first reported 140


Foaming Tendency Seq I (5 min)
Kernel Density


Foaming Tendency Seq II (5 min)
Kernel Density


Foaming Tendency Seq III (5 min)
Kernel Density

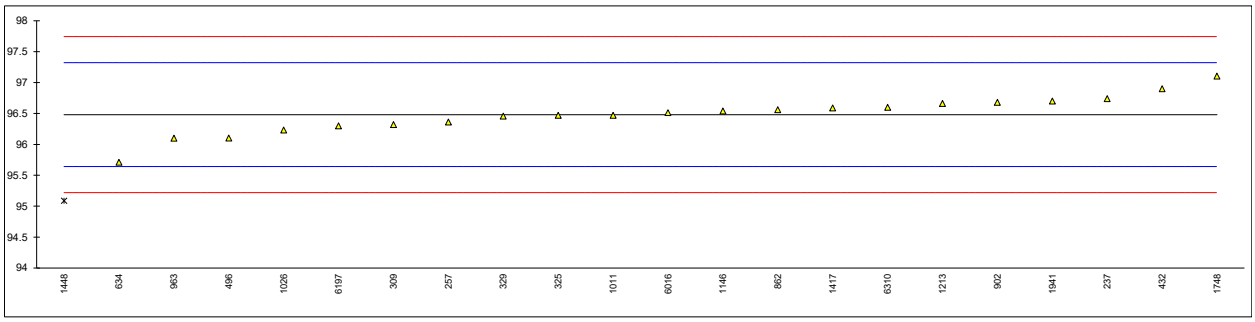
Determination of Foam Stability, Sequence I, II and III (10 min. settling period) on sample #21030; results in mL

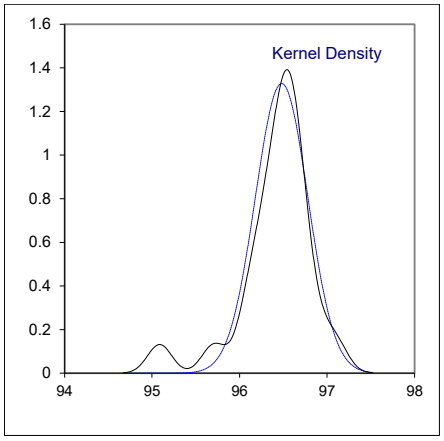
lab	method	Seq I	mark	z(targ)	Seq II	mark	z(targ)	Seq III	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179		-----		-----	-----		-----	-----		-----
237		-----		-----	-----		-----	-----		-----
257		-----		-----	-----		-----	-----		-----
309	D892	0		-----	0		-----	0		-----
325	D892	0		-----	0		-----	0		-----
329	D892	0		-----	0		-----	0		-----
349		-----		-----	-----		-----	-----		-----
432		-----		-----	-----		-----	-----		-----
496	D892	0		-----	0		-----	0		-----
634		-----		-----	-----		-----	-----		-----
862	D892	0		-----	0		-----	0		-----
902	D892	0		-----	0		-----	0		-----
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
1011	D892	0		-----	0		-----	0		-----
1026	D892	0		-----	0		-----	0		-----
1146		-----		-----	-----		-----	-----		-----
1213	D892	0		-----	0		-----	0		-----
1417	D892	0		-----	0		-----	0		-----
1448		-----		-----	-----		-----	-----		-----
1748		-----		-----	-----		-----	-----		-----
1941	ISO6247	0		-----	0		-----	0		-----
6016	D892	30		-----	40		-----	5		-----
6197	D892	0		-----	0		-----	0		-----
6310	D892	0		-----	0		-----	0		-----
	n	13			13			13		
	mean (n)	0			0			0		

Determination of Kinematic Viscosity at 40°C on sample #21030; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D445	96.74		0.62	
257	D7279 corrected to D445	96.36		-0.29	
309	D445	96.32		-0.38	
325	D445	96.47		-0.03	
329	D445	96.455		-0.06	
349		-----		-----	
432	D445	96.90		1.00	
496	D445	96.104		-0.90	
634	D445	95.71		-1.83	
862	D445	96.56		0.19	
902	D445	96.68		0.47	
962		-----		-----	
963	D445	96.10		-0.91	
1011	D7042	96.47		-0.03	
1026	D445	96.23		-0.60	
1146	D445	96.54		0.14	
1213	D445	96.66		0.43	
1417	D445	96.59		0.26	
1448	D7042	95.087	C,R(0.01)	-3.32	first reported 95.197
1748	D7042	97.104		1.48	
1941	ISO3104	96.70		0.52	
6016	D7042	96.514		0.08	
6197	D445	96.300		-0.43	
6310	D7279 corrected to D445	96.6		0.28	

normality	suspect
n	21
outliers	1
mean (n)	96.481
st.dev. (n)	0.3005
R(calc.)	0.841
st.dev.(D445:19a)	0.4204
R(D445:19a)	1.177

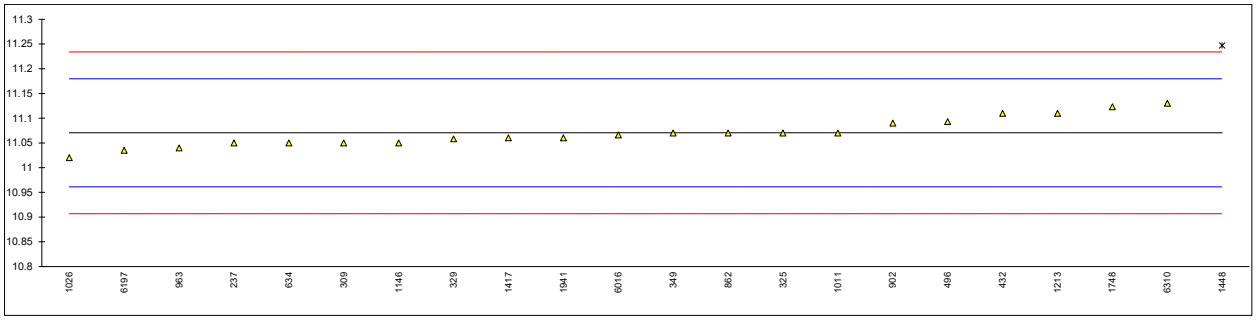


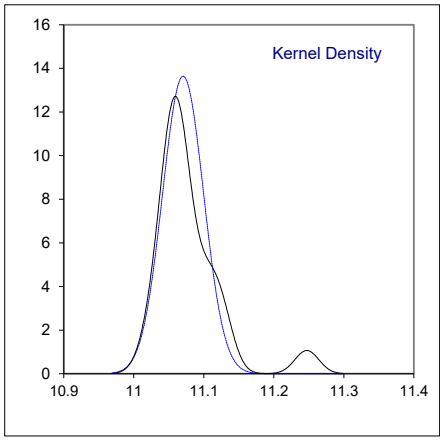


Determination of Kinematic Viscosity at 100°C on sample #21030; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D445	11.05		-0.37	
257		-----		-----	
309	D445	11.05		-0.37	
325	D445	11.07		0.00	
329	D445	11.058		-0.22	
349	D445	11.07		0.00	
432	D445	11.11		0.73	
496	D445	11.093		0.42	
634	D445	11.05		-0.37	
862	D445	11.07		0.00	
902	D445	11.09		0.36	
962		-----		-----	
963	D445	11.04		-0.55	
1011	D7042	11.07		0.00	
1026	D445	11.02		-0.92	
1146	D445	11.05		-0.37	
1213	D445	11.11		0.73	
1417	D445	11.06		-0.19	
1448	D7042	11.247	C,R(0.01)	3.24	first reported 11.2358
1748	D7042	11.123		0.97	
1941	ISO3104	11.06		-0.19	
6016	D7042	11.066		-0.08	
6197	D445	11.035		-0.65	
6310	D7279 corrected to D445	11.13		1.10	

normality	OK
n	21
outliers	1
mean (n)	11.070
st.dev. (n)	0.0293
R(calc.)	0.082
st.dev.(D445:19a)	0.0546
R(D445:19a)	0.153

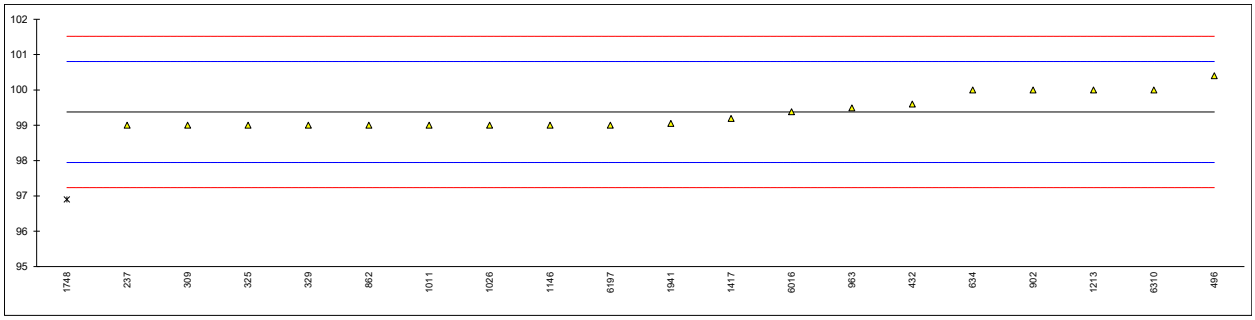


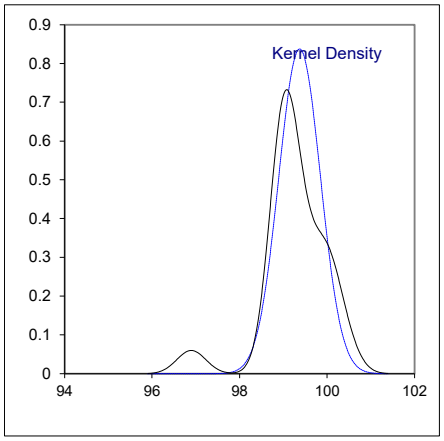


Determination of Viscosity Index on sample #21030

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D2270	99		-0.52	
257		-----		-----	
309	D2270	99		-0.52	
325	D2270	99		-0.52	
329	D2270	99		-0.52	
349		-----		-----	
432	D2270	99.6		0.32	
496	D2270	100.4		1.44	
634	D2270	100		0.88	
862	D2270	99		-0.52	
902	D2270	100		0.88	
962		-----		-----	
963	D2270	99.49		0.16	
1011	D2270	99		-0.52	
1026	D2270	99		-0.52	
1146	D2270	99		-0.52	
1213	D2270	100		0.88	
1417	D2270	99.19		-0.26	
1448		-----		-----	
1748	D2270	96.9	E,R(0.01)	-3.46	iis calculated 99.5
1941	ISO2909	99.05		-0.45	
6016	D2270	99.38		0.01	
6197	D2270	99		-0.52	
6310	D2270	100		0.88	

normality	OK
n	19
outliers	1
mean (n)	99.37
st.dev. (n)	0.476
R(calc.)	1.33
st.dev.(D2270:10)	0.714
R(D2270:10)	2

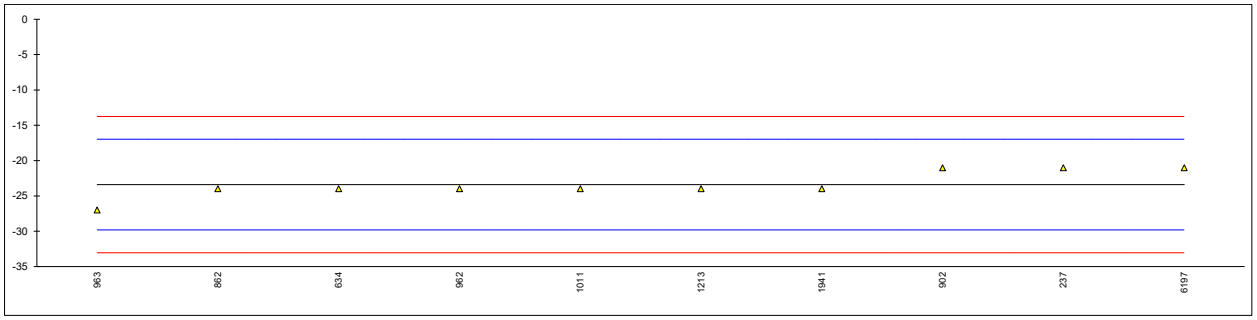


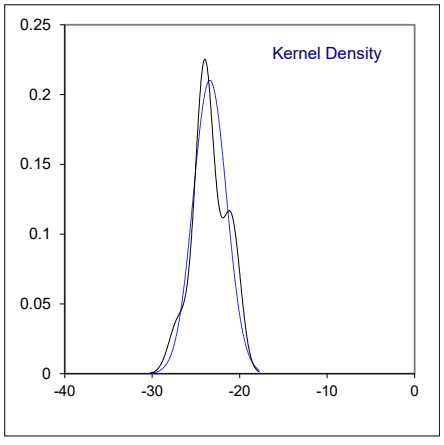


Determination of Pour Point Manual on sample #21030; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D97	-21		0.75	
257		-----		-----	
309		-----		-----	
325		-----		-----	
329		-----		-----	
349		-----		-----	
432		-----		-----	
496		-----		-----	
634	D97	-24		-0.19	
862	D97	-24		-0.19	
902	D97	-21		0.75	
962	D97	-24		-0.19	
963	D97	-27		-1.12	
1011	D97	-24		-0.19	
1026		-----		-----	
1146		-----		-----	
1213	D97	-24		-0.19	
1417		-----		-----	
1448		-----		-----	
1748		-----		-----	
1941	ISO3016	-24		-0.19	
6016		-----		-----	
6197	D97	-21		0.75	
6310		-----		-----	

normality	OK
n	10
outliers	0
mean (n)	-23.40
st.dev. (n)	1.897
R(calc.)	5.31
st.dev.(D97:17b)	3.214
R(D97:17b)	9

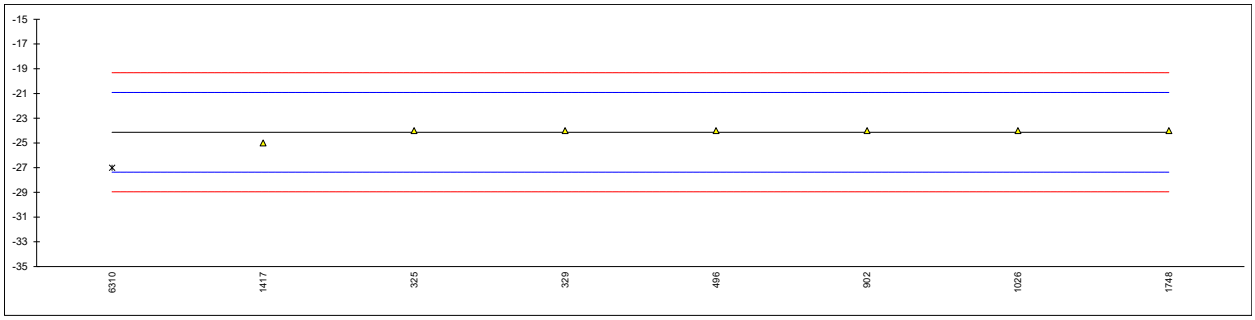


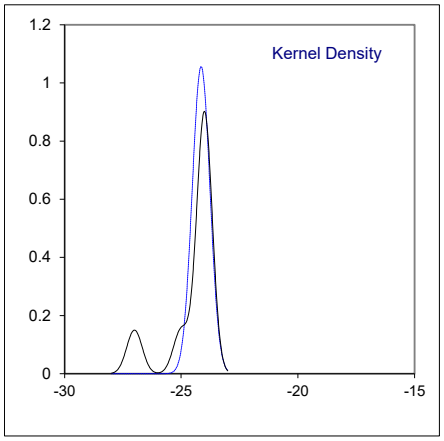


Determination of Pour Point Automated 1°C interval on sample #21030; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
257		-----		-----	
309		-----		-----	
325	D5950	-24		0.09	
329	D5950	-24		0.09	
349		-----		-----	
432		-----		-----	
496	D5950	-24		0.09	
634		-----		-----	
862		-----		-----	
902	D5950	-24		0.09	
962		-----		-----	
963		-----		-----	
1011		-----		-----	
1026	D5950	-24		0.09	
1146		-----		-----	
1213		-----		-----	
1417	D5950	-25		-0.53	
1448		-----		-----	
1748	D7346	-24		0.09	
1941		-----		-----	
6016		-----		-----	
6197		-----		-----	
6310	D5950	-27	D(0.05)	-1.78	

normality	unknown
n	7
outliers	1
mean (n)	-24.14
st.dev. (n)	0.378
R(calc.)	1.06
st.dev.(D5950:14)	1.607
R(D5950:14)	4.5





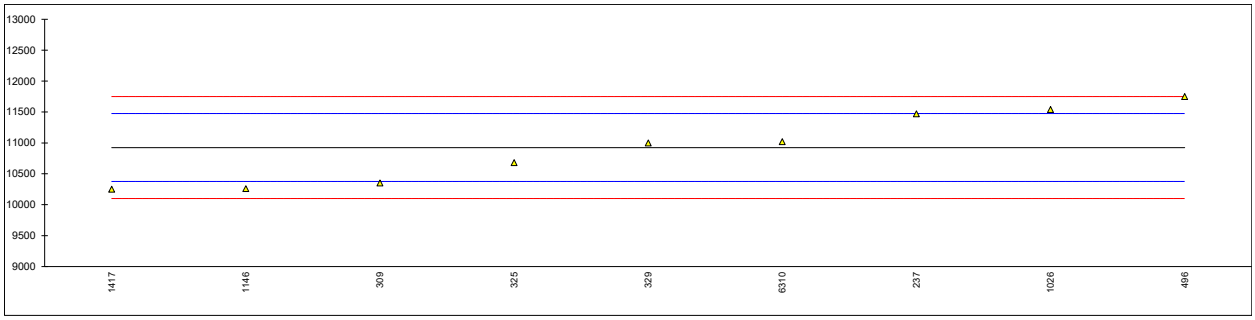
Determination of Rust Prevention distilled water on sample #21030

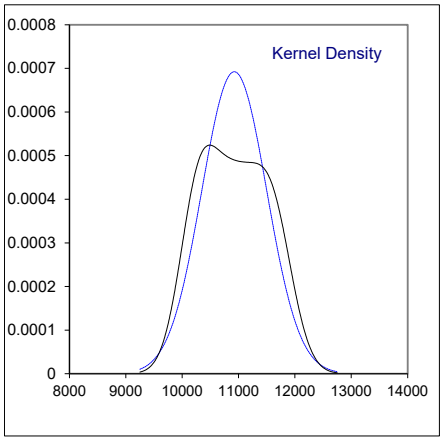
lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
257		-----		-----	
309		-----		-----	
325	D665	PASS		-----	
329		-----		-----	
349		-----		-----	
432		-----		-----	
496		-----		-----	
634		-----		-----	
862		-----		-----	
902		-----		-----	
962		-----		-----	
963		-----		-----	
1011		-----		-----	
1026	D665	Pass		-----	
1146		-----		-----	
1213		-----		-----	
1417	D665	PASS		-----	
1448		-----		-----	
1748		-----		-----	
1941	D665	Pass		-----	
6016	D665	pass		-----	
6197	D665	Rust Free		-----	
6310		-----		-----	
	n	6			
	mean (n)	Pass			

Determination of Sulfur on sample #21030; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D4294	11470		1.99	
257		-----		-----	
309	D4294	10350		-2.09	
325	D5185	10680		-0.89	
329	D4294	11000		0.27	
349		-----		-----	
432		-----		-----	
496	D4294	11750		3.00	
634		-----		-----	
862		-----		-----	
902		-----		-----	
962		-----		-----	
963		-----		-----	
1011		-----		-----	
1026	D2622	11540	C	2.24	first reported 1.15 mg/kg
1146	D4294	10260		-2.42	
1213		-----		-----	
1417	In house	10252		-2.45	
1448		-----		-----	
1748		-----		-----	
1941		-----		-----	
6016		-----		-----	
6197		-----		-----	
6310	D7751	11020	C	0.35	first reported 1102

normality	OK
n	9
outliers	0
mean (n)	10924.7
st.dev. (n)	576.23
R(calc.)	1613.4
st.dev.(D4294:16e1)	274.72
R(D4294:16e1)	769.2

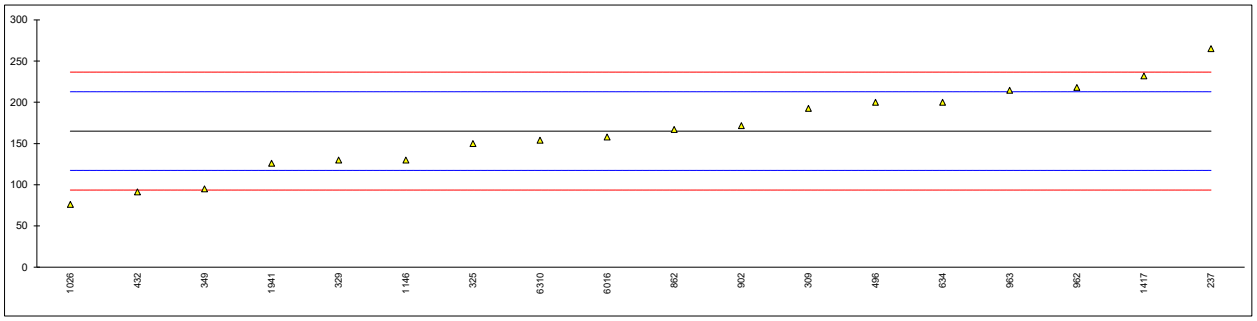


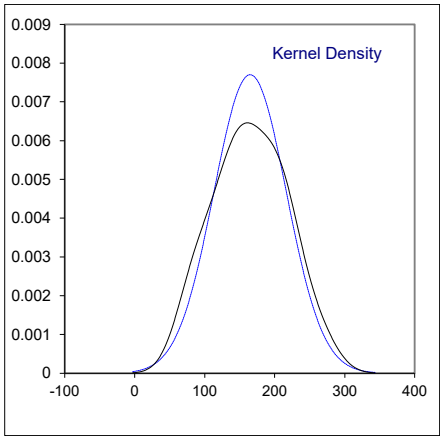


Determination of Water on sample #21030; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D6304-C	265		4.19	
257		-----		-----	
309	D6304-A	192.5		1.15	
325	D6304-C	150		-0.63	
329	D6304-C	130		-1.47	
349	D6304-C	95		-2.93	
432	D6304-C	91.20		-3.09	
496	D6304-C	200		1.46	
634	D6304-A	200		1.46	
862	D6304-B	167		0.08	
902	D6304-C	171.6		0.27	
962	D6304-A	218		2.22	
963	D6304-A	214.54		2.07	
1011		-----		-----	
1026	D6304-C	76		-3.73	
1146	D6304-C	130		-1.47	
1213		-----		-----	
1417	D6304-A	232		2.80	
1448		-----		-----	
1748		-----		-----	
1941	D6304-C	126		-1.64	
6016	D6304	158		-0.30	
6197		-----		-----	
6310	D6304-C	154		-0.46	

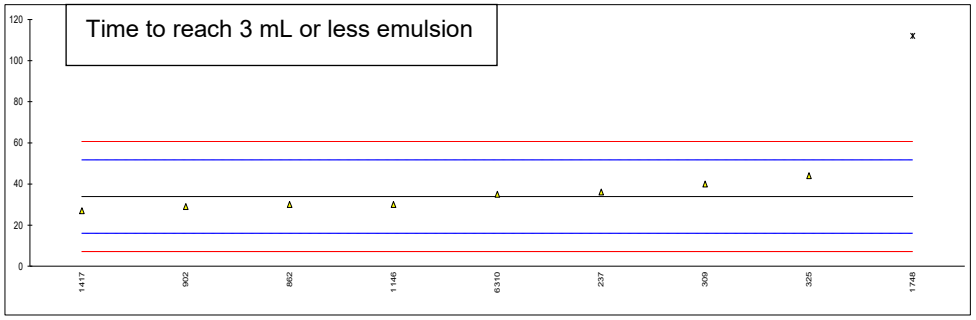
	normality	OK
	n	18
	outliers	0
	mean (n)	165.05
	st.dev. (n)	51.814
	R(calc.)	145.08
	st.dev.(D6304-C:20)	23.882
	R(D6304-C:20)	66.87
Compare		
	R(D6304-A:20)	85.71
	R(D6304-B:20)	189.49

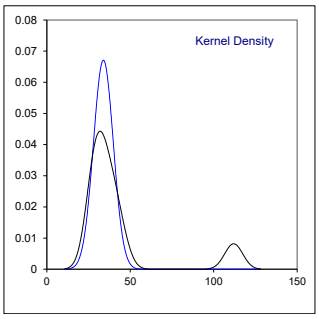


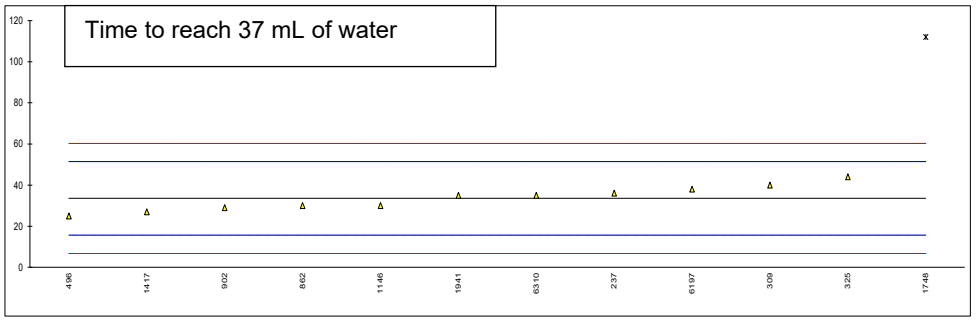


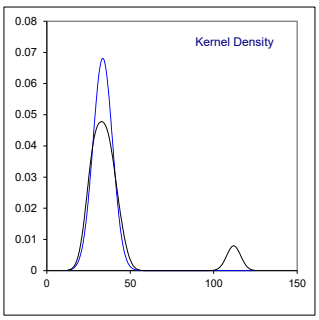
Determination of Water Separability at 82°C, distilled water on sample #21030; results in minutes

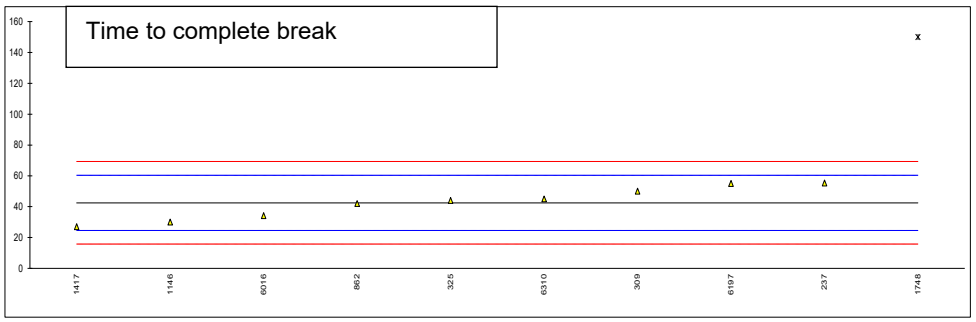
lab	method	3 mL or less emulsion		z(targ)	37 mL of water		z(targ)	complete break (40-40-0)		z(targ)	test aborted	time aborted
178		-----		-----	-----		-----	-----		-----	-----	-----
179		-----		-----	-----		-----	-----		-----	-----	-----
237	D1401	36.1		0.25	36.1		0.29	55.4		1.44	No	-----
257		-----		-----	-----		-----	-----		-----	-----	-----
309	D1401	40		0.68	40		0.72	50		0.84	Yes	50
325	D1401	44		1.13	44		1.17	44		0.17	No	-----
329	D1401	-----		-----	-----		-----	-----		-----	Yes	-----
349		-----		-----	-----		-----	-----		-----	-----	-----
432		-----		-----	-----		-----	-----		-----	-----	-----
496	D1401	-----		-----	25		-0.96	40-37-3		-----	No	-----
634		-----		-----	-----		-----	-----		-----	-----	-----
862	D1401	30		-0.44	30		-0.40	42		-0.06	No	-----
902	D1401	29		-0.55	29		-0.51	-----		-----	No	60
962		-----		-----	-----		-----	-----		-----	-----	-----
963		-----		-----	-----		-----	-----		-----	-----	-----
1011		-----		-----	-----		-----	-----		-----	-----	-----
1026	D1401	-----		-----	-----		-----	-----		-----	Yes	30
1146	D1401	30		-0.44	30		-0.40	30		-1.40	No	-----
1213		-----		-----	-----		-----	-----		-----	-----	-----
1417		27		-0.77	27		-0.73	27		-1.74	No	-----
1448		-----		-----	-----		-----	-----		-----	-----	-----
1748	D1401	112	D(0.01)	8.75	112	D(0.01)	8.79	150	D(0.01)	12.04	Yes	150
1941	D1401	-----		-----	35		0.16	-----		-----	Yes	60
6016	D1401	-----		-----	-----		-----	34.2		-0.93	No	-----
6197		-----		-----	38		0.50	55		1.40	No	-----
6310		35		0.12	35		0.16	45		0.28	No	-----
	normality	OK			OK			OK				
	n	8			11			9				
	outliers	1			1			1				
	mean (n)	33.9			33.6			42.5				
	st.dev. (n)	5.94			5.86			10.33				
	R(calc.)	16.6			16.4			28.9				
	st.dev.(D1401:19)	8.93			8.93			8.93				
	R(D1401:19)	25			25			25				

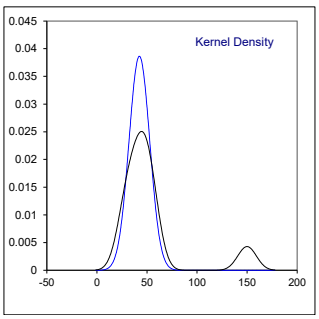












Determination of Water Separability at 82°C, distilled water on sample #21030; results in mL

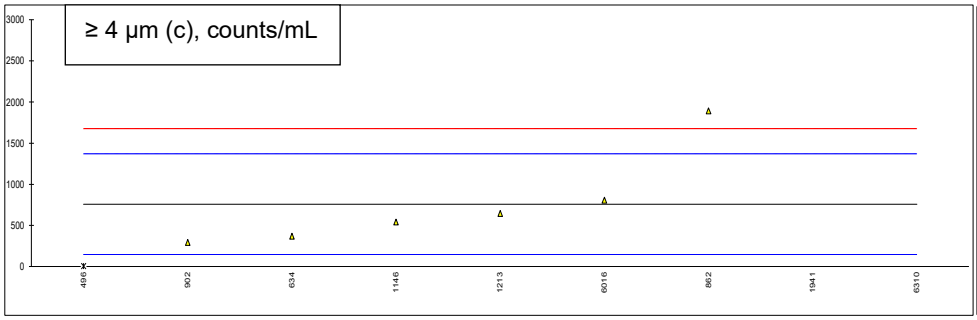
lab	method	volume oil phase	mark	volume water phase	mark	volume emulsion phase	mark
178		-----		-----		-----	
179		-----		-----		-----	
237	D1401	40.0		40.0		0.0	
257		-----		-----		-----	
309	D1401	40		40		0	
325		-----		-----		-----	
329		-----		-----		-----	
349		-----		-----		-----	
432		-----		-----		-----	
496		-----		-----		-----	
634		-----		-----		-----	
862		-----		-----		-----	
902	D1401	41		39		0	
962		-----		-----		-----	
963		-----		-----		-----	
1011		-----		-----		-----	
1026	D1401	43		37		0	
1146	D1401	43		37		0	
1213		-----		-----		-----	
1417		40		40		0	
1448		-----		-----		-----	
1748	D1401	40		40		0	
1941	D1401	42		38		0	
6016		-----		-----		-----	
6197		-----		-----		-----	
6310		40		40		0	

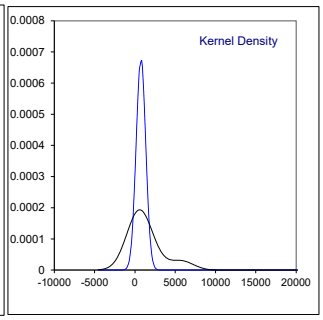
Determination of Level of Contamination on sample #21030; results in counts/mL

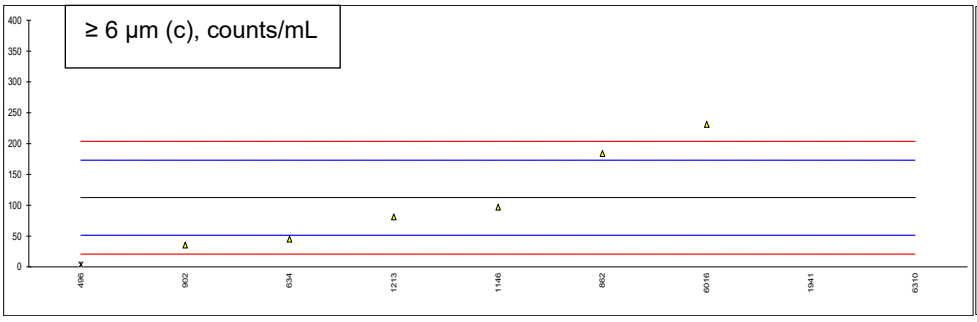
lab	method	≥ 4 µm (c)	mark	z(targ)	≥ 6 µm (c)	mark	z(targ)	≥ 14 µm (c)	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179		-----		-----	-----		-----	-----		-----
237		-----		-----	-----		-----	-----		-----
257		-----		-----	-----		-----	-----		-----
309		-----		-----	-----		-----	-----		-----
325		-----		-----	-----		-----	-----		-----
329		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
432		-----		-----	-----		-----	-----		-----
496	D7596	6	ex	-2.46	4	ex	-3.55	4	ex	-0.19
634	ISO11500	369		-1.27	45		-2.21	2		-1.13
862	ISO11500	1892		3.71	184		2.35	5		0.28
902	D7647	294.4		-1.52	35.6		-2.52	1.0	ex	-1.60
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
1011		-----		-----	-----		-----	-----		-----
1026		-----		-----	-----		-----	-----		-----
1146	ISO11500	540		-0.71	97		-0.50	6.1		0.80
1213	D7647	644.1		-0.37	81.1		-1.02	4.5		0.05
1417		-----		-----	-----		-----	-----		-----
1448		-----		-----	-----		-----	-----		-----
1748		-----		-----	-----		-----	-----		-----
1941	ISO4406	5739.8	G(0.01)	16.29	870.5	G(0.01)	24.88	56.6	G(0.05)	24.61
6016	D7596	807.3		0.16	231.0		3.90	29.2	G(0.01)	11.69
6197		-----		-----	-----		-----	-----		-----
6310	ISO4407	71637	G(0.01)	231.76	18182	G(0.01)	592.90	1127	G(0.01)	529.17

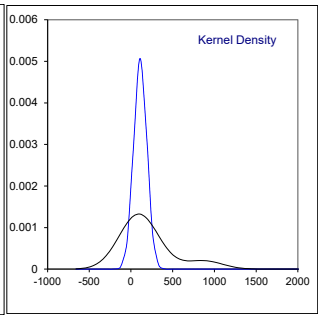
normality	not OK	OK	not OK
n	6	6	4
outliers	2 +1ex	2 +1ex	3 +2ex
mean (n)	757.8	112.3	4.4
st.dev. (n)	585.68	78.55	1.73
R(calc.)	1639.9	219.9	4.9
st.dev.(D7647:10)	305.83	30.48	2.12
R(D7647:10)	856.3	85.3	5.9

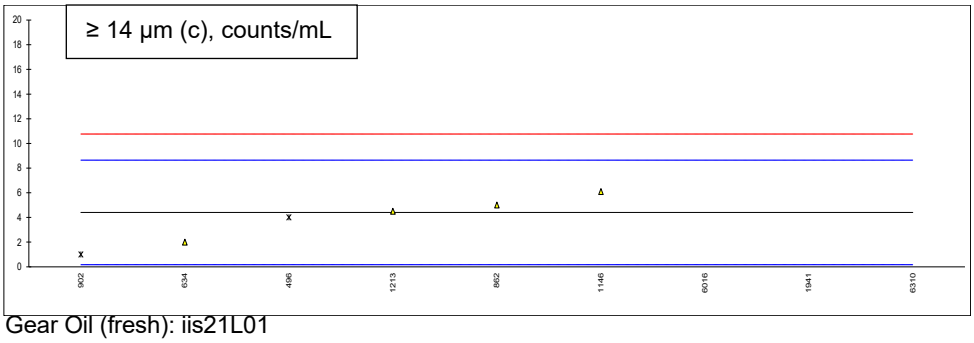
Lab 496 test results excluded as test results in counts/mL and ISO4406 scale number did not match
Lab 902 test result at ≥ 14 µm excluded as test result in counts/mL and ISO4406 scale number did not match

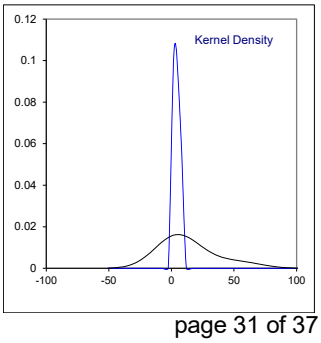










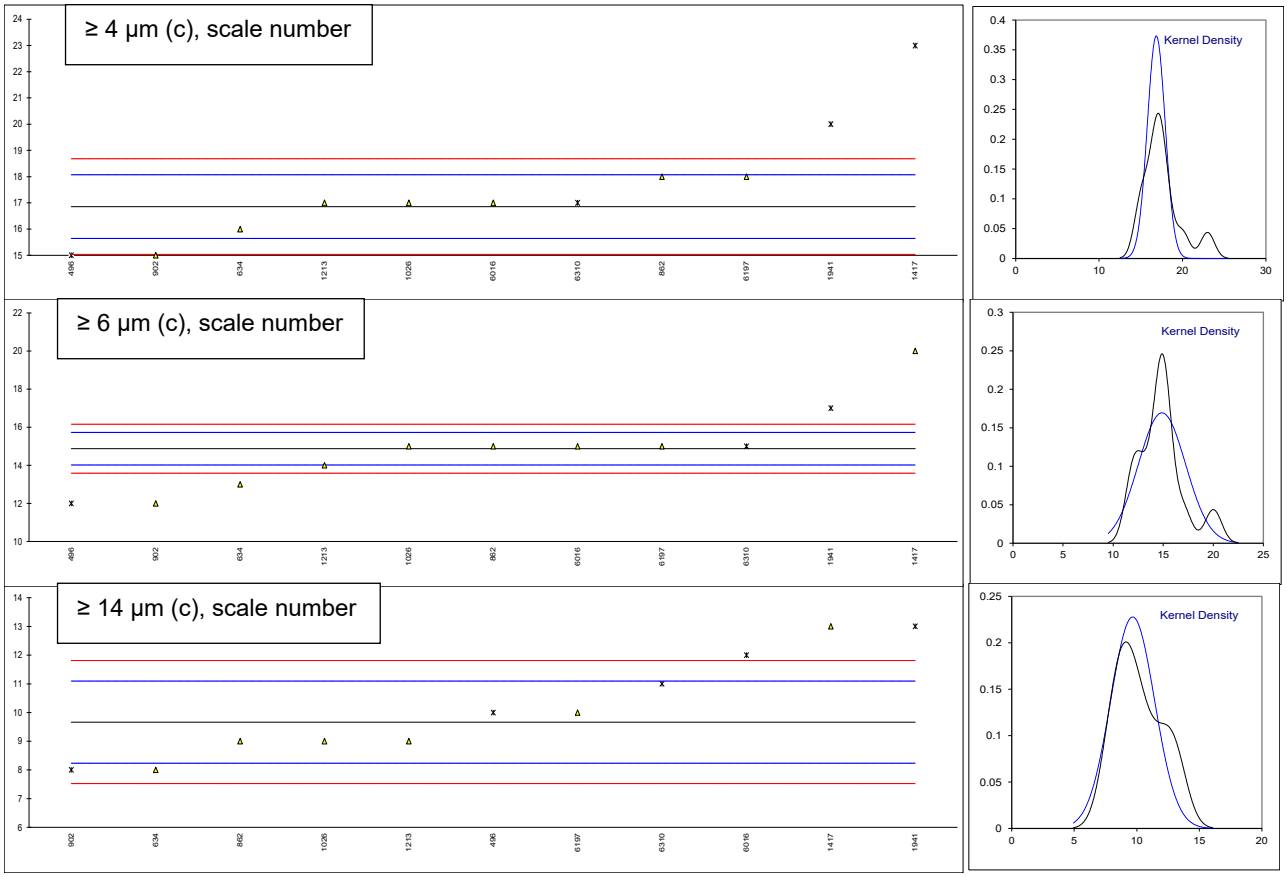


Determination of Level of Contamination acc. to ISO4406 scale on sample #21030; results in scale number

lab	method	≥ 4 µm (c)	mark	z(targ)	≥ 6 µm (c)	mark	z(targ)	≥ 14 µm (c)	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179		-----		-----	-----		-----	-----		-----
237		-----		-----	-----		-----	-----		-----
257		-----		-----	-----		-----	-----		-----
309		-----		-----	-----		-----	-----		-----
325		-----		-----	-----		-----	-----		-----
329		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
432		-----		-----	-----		-----	-----		-----
496	ISO4406	15	ex	-3.06	12	ex	-6.71	10	ex	0.47
634	ISO4406	16		-1.41	13		-4.38	8		-2.33
862	ISO4406	18		1.88	15		0.29	9		-0.93
902	ISO4406	15		-3.06	12		-6.71	8	ex	-2.33
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
1011		-----		-----	-----		-----	-----		-----
1026	ISO4406	17		0.24	15		0.29	9		-0.93
1146		-----		-----	-----		-----	-----		-----
1213	D7647	17		0.24	14		-2.04	9		-0.93
1417		23	G(0.05)	10.12	20		11.96	13		4.67
1448		-----		-----	-----		-----	-----		-----
1748		-----		-----	-----		-----	-----		-----
1941	ISO4406	20	ex	5.18	17	ex	4.96	13	ex	4.67
6016	ISO4406	17		0.24	15		0.29	12	ex	3.27
6197	ISO4406	18		1.88	15		0.29	10		0.47
6310	ISO4406	17	ex	0.24	15	ex	0.29	11	ex	1.87

normality	OK	not OK	not OK
n	7	8	6
outliers	1 +3ex	0 +3ex	0 +5ex
mean (n)	16.9	14.9	9.7
st.dev. (n)	1.07	2.36	1.75
R(calc.)	3.0	6.6	4.9
st.dev.(D7647:10)	0.61	0.43	0.71
R(D7647:10)	1.7	1.2	2

Lab 496 test results excluded as test results in counts/mL and ISO4406 scale number did not match
Lab 902 test result at ≥ 14 µm excluded as test result in counts/mL and ISO4406 scale number did not match
Lab 1941 test results excluded because of statistical outliers at related measurements for counts/mL
Lab 6016 test result at ≥ 14 µm excluded because of statistical outlier at related measurement for counts/mL
Lab 6310 test results excluded because of statistical outliers at related measurements for counts/mL



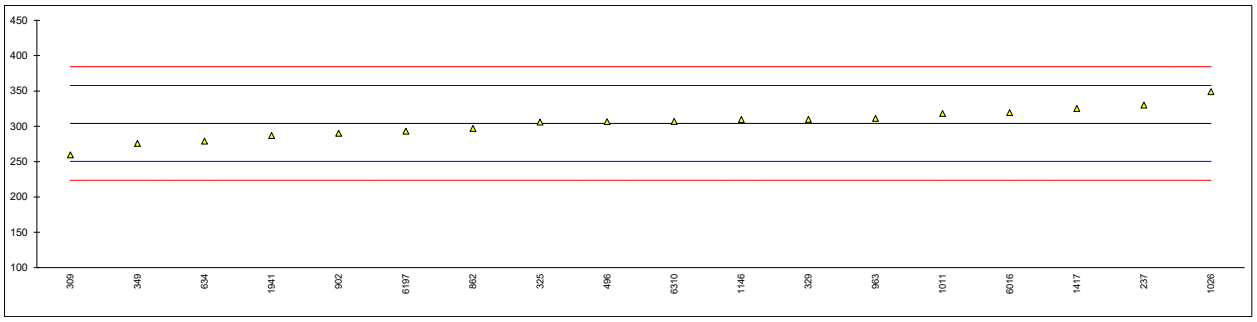
Determination of Calcium as Ca on sample #21030; results in mg/kg

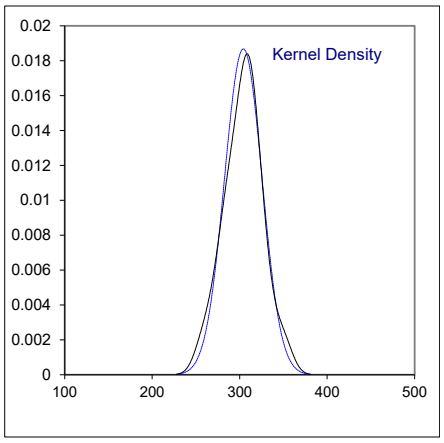
lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D5185	4.684		-----	
257	D6595	1		-----	
309	D5185	0.043		-----	
325	D5185	3		-----	
329	D5185	<3		-----	
349	D5185	1		-----	
432		-----		-----	
496	D5185	0.765		-----	
634	D6595	0.256		-----	
862	D5185	<1		-----	
902	D5185	<40		-----	
962		-----		-----	
963		0.22		-----	
1011	D5185	<10		-----	
1026	D5185	1	C	-----	first reported 0.0001 mg/kg
1146	D5185	<5		-----	
1213		-----		-----	
1417	D5185	3.987		-----	
1448		-----		-----	
1748		-----		-----	
1941	D5185	<1		-----	
6016	D5185	0.670		-----	
6197	D4951	<1		-----	
6310	D7751	<1		-----	
	n	19			
	mean (n)	<40			D5185:18 application range: 40 – 9000 mg/kg

Determination of Phosphorus as P on sample #21030; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D5185	330		0.97	
257		-----		-----	
309	D5185	259.6		-1.66	
325	D5185	306		0.07	
329	D5185	310		0.22	
349	D5185	276		-1.05	
432		-----		-----	
496	D5185	306.5		0.09	
634	D6595	279	C	-0.94	first reported 179
862	D5185	297		-0.26	
902	D5185	290		-0.53	
962		-----		-----	
963		311.11		0.26	
1011	D5185	318		0.52	
1026	D5185	349	C	1.68	first reported 0.0349 mg/kg
1146	D5185	309.49		0.20	
1213		-----		-----	
1417	D5185	325.3		0.79	
1448		-----		-----	
1748		-----		-----	
1941	D5185	287		-0.64	
6016	D5185	319.3		0.57	
6197	D4951	293		-0.41	
6310	D7751	307		0.11	

normality	OK	
n	18	
outliers	0	
mean (n)	304.072	
st.dev. (n)	21.3596	
R(calc.)	59.807	
st.dev.(D5185:18)	26.7793	
R(D5185:18)	74.982	D5185:18 application range: 10 – 1000 mg/kg





Determination of Zinc as Zn on sample #21030; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D5185	1.028		-----	
257	D6595	0.23		-----	
309	D5185	0		-----	
325	D5185	1		-----	
329	D5185	<3		-----	
349	D5185	0		-----	
432		-----		-----	
496	D5185	2.65	C	-----	first reported 8.38
634	D6595	0.2		-----	
862	D5185	<1		-----	
902	D5185	<60		-----	
962		-----		-----	
963		0.49		-----	
1011	D5185	<10		-----	
1026	D5185	1	C	-----	first reported 0.0001 mg/kg
1146	D5185	<5		-----	
1213		-----		-----	
1417	D5185	1.94		-----	
1448		-----		-----	
1748		-----		-----	
1941	D5185	<1		-----	
6016	D5185	0.115		-----	
6197	D4951	<1		-----	
6310	D7751	<1		-----	
	n	19			
	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	KAZAKHSTAN
1 lab in	MALAYSIA
3 labs in	NETHERLANDS
1 lab in	NIGERIA
1 lab in	PHILIPPINES
1 lab in	PORTUGAL
2 labs in	SAUDI ARABIA
1 lab in	SERBIA
1 lab in	SINGAPORE
1 lab in	SPAIN
1 lab in	TANZANIA
1 lab in	TURKEY
1 lab in	UNITED KINGDOM
2 labs in	UNITED STATES OF AMERICA
1 lab in	VIETNAM

APPENDIX 3

Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= 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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