Institute for
Interlaboratory Studies

Results of Proficiency Test
Gear Oil (fresh)
March 2022

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 2021/2022 it was decided to continue the round robin for the analysis of fresh Gear Oil.

In this interlaboratory study 23 laboratories in 17 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 #22030.
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 150 liters of fresh Gear Oil was obtained from a local supplier. After homogenization 36 amber glass bottles of 1 L and 36 amber glass bottles of 0.5 L were filled and labelled #22030.
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 #22030-1	0.88702
sample #22030-2	0.88701
sample #22030-3	0.88702
sample #22030-4	0.88703
sample #22030-5	0.88701
sample #22030-6	0.88702
sample #22030-7	0.88702
sample #22030-8	0.88702

Table 1: homogeneity test results of subsamples #22030

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)	0.00002
reference test method	ISO12185:96
0.3 x R (reference test method)	0.00015

Table 2: evaluation of the repeatability of subsamples #22030

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 #22030 were sent on February 16, 2022. 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 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

Some problems were encountered with the dispatch of the samples due to COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with another week. One participant reported test results after the extended reporting date and three other participants did not report any test results. Not all participants were able to report all tests requested.
In total 20 participants reported 315 numerical test results. Observed were 9 outlying test results, which is 2.9%. 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). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D5950:14(2020)). In the results tables of appendix 1 only the method number and year of adoption or revision (e.g. D5950:14) will be used.

Total Acid Number: 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 D664-A:18e2 for the end point modes IP 60 mL, BEP 60 mL and BEP 125 mL, but is not in agreement for end point mode IP 125 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 not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ISO12185:96 and ASTM D4052:18a.

Flash Point C.O.C.: This determination was problematic. No statistical outliers were observed. However, 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 may be problematic.
For Foaming Tendency sequence I and III and for Foam Stability sequence I, II and III (almost) all participants agreed on a test result of 0 (Nil). Therefore, no z-scores are calculated.
For Foaming Tendency sequence II the variation in the test results was too large. 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. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D445:21e2.

Viscosity Index: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D2270:10(2016).

Pour Point Manual: 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 D97:17b.

Pour Point Automated: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D5950:14(2020).

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

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

Water: This determination may be problematic depending on the procedure used. 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 2022 PTs of Gear Oil (fresh) and Gear Oil (used).
The calculated reproducibility is in agreement with the requirements of ASTM D6304:20 procedure B but not in agreement with procedure A and C.

Water Separability: This determination was not problematic. No statistical outliers were observed. The calculated reproducibilities of “time to reach 3mL or less emulsion”, “time to reach 37mL of water” and “time to reach complete break” are in agreement with the requirements of ASTM D1401:21.

Level of Contamination: This determination may be problematic. In total three statistical outliers were observed over three parameters. The calculated reproducibilities of counts/mL (≥4µm and ≥6µm) after rejection of the statistical outliers are in agreement with the requirements of ASTM D7647:10(2018). For counts/mL ≥14µm was the variation in the test results too large. Therefore, no z-scores are calculated.
Only four participants reported test results for Level of Contamination acc. to ISO4406 scale. Therefore, no z-scores are calculated.

Calcium as Ca: This determination was not problematic. Almost all reporting participants agreed on a value near or below the application range of ASTM D5185:18. 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. Almost all reporting participants agreed on a value near or below the application range of ASTM D5185:18. 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 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	15	0.38	0.10	0.18
Copper Corrosion 3 hrs at 100 °C		12	1 (1a/1b)	n.a.	n.a.
Density at 15 °C	kg/L	18	0.8870	0.0004	0.0005
Flash Point C.O.C.	°C	16	259	22	18
Flash Point PMcc	°C	17	205	12	15
Foaming Tendency (Seq I) 5min	mL	9	0	n.e.	n.e
Foaming Tendency (Seq II) 5min	mL	9	7.8	30.6	(15.4)
Foaming Tendency (Seq III) 5min	mL	9	0	n.e.	n.e
Foam Stability (Seq I) 10min	mL	10	0	n.e.	n.e.
Foam Stability (Seq II) 10min	mL	10	0	n.e.	n.e.
Foam Stability (Seq III) 10min	mL	10	0	n.e.	n.e.
Kinematic Viscosity at 40 °C	mm^2/s	19	218.73	2.34	2.67
Kinematic Viscosity at 100 °C	mm^2/s	18	19.312	0.145	0.267
Viscosity Index		18	99.7	1.5	2
Pour Point Manual	°C	7	-14.0	10.5	9
Pour Point Automated	°C	7	-14.3	5.3	4.5

Parameter	unit	n	average	2.8 * sd	R(lit)
Rust Prevention distilled water		5	Pass	n.a.	n.a.
Sulfur	mg/kg	10	6581	912	555
Water	mg/kg	14	68	94	131
Water Separability at 82 °C					
Time to ≤ 3 mL emulsion	minutes	12	12.3	5.7	25
Time to 37 mL water	minutes	12	12.4	5.7	25
Time to complete break	minutes	8	15.6	4.7	25
Level of Contamination					
≥ 4µm (c)	counts/mL	4	57984	39195	65522
≥ 6µm (c)	counts/mL	4	27096	13370	20593
≥14µm (c)	counts/mL	4	596	1895	(804)
≥ 4µm (c)	scale no	4	23	n.e.	n.e
≥ 6µm (c)	scale no	4	22	n.e.	n.e
≥14µm (c)	scale no	4	16	n.e.	n.e
Calcium as Ca	mg/kg	14	<40	n.e.	n.e.
Phosphorus as P	mg/kg	14	276	32	71
Zinc as Zn	mg/kg	15	<60	n.e.	n.e.

Table 3: reproducibilities of tests on sample #22030

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 2022 WITH PREVIOUS PTS

	March 2022	March 2021	March 2020	April 2019	April 2018
Number of reporting laboratories	20	24	21	23	18
Number of test results	315	390	384	400	350
Number of statistical outliers	9	18	32	14	14
Percentage of statistical outliers	2.9%	4.6%	8.3%	3.5%	4.0%

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 2022	March 2021	March 2020	April 2019	April 2018
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	+/-	--	+/-	--	--
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

Results between brackets should be used with due care

The following performance categories were used:

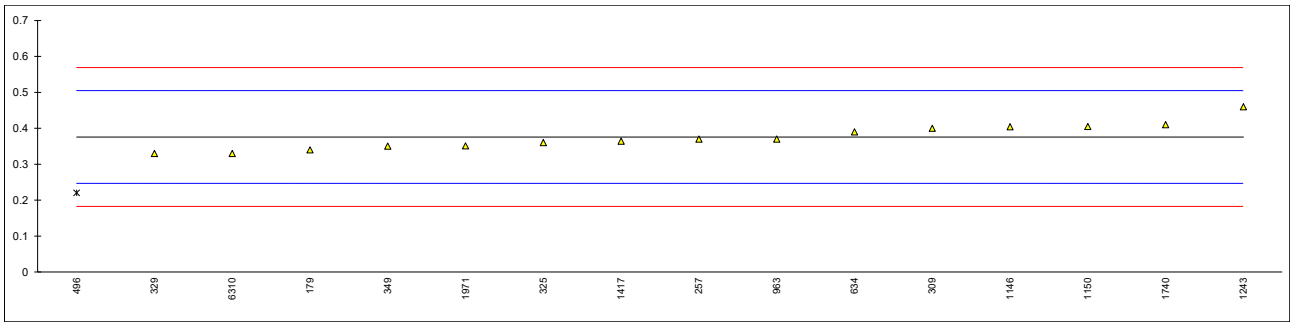
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test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

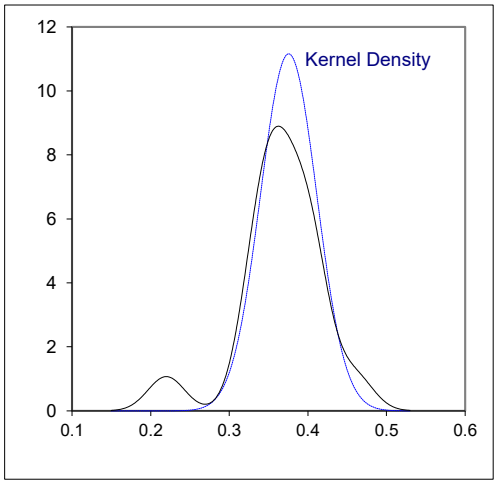
APPENDIX 1

Determination of Total Acid Number on sample #22030; results in mg KOH/g

lab	method	value	mark	z(targ)	end point type	volume (mL)	remarks
178		-----		-----	---	---	
179	D664-A	0.34		-0.55	Inflection Point	60 mL	
237		-----		-----	---	---	
257	D7889	0.37		-0.09	---	---	
309	D664-A	0.40		0.38	Buffer End Point pH 10	125 mL	
325	D664-A	0.36		-0.24	Buffer End Point pH 10	125 mL	
329	D664-A	0.33		-0.71	Buffer End Point pH 10	125 mL	
349	D664-A	0.35		-0.40	Buffer End Point pH 10	125 mL	
432		-----		-----	---	---	
496	D664-A	0.22	D(0.05)	-2.42	Buffer End Point pH 10	60 mL	
634	D664-A	0.39		0.22	---	---	
862		-----		-----	---	---	
962		-----		-----	---	---	
963	D664-B	0.37		-0.09	Inflection Point	60 mL	
1146	D664-A	0.404		0.44	Buffer End Point pH 10	125 mL	
1150	BDS9776	0.405		0.46	---	---	
1243	ISO6618	0.46		1.31	Inflection Point	60 mL	
1417	D664-A	0.364		-0.18	Inflection Point	60 mL	
1740	D664-A	0.41		0.53	Inflection Point	60 mL	
1748		-----		-----	---	---	
1971	ISO6618	0.351		-0.38	---	---	
6310	D664-A	0.33		-0.71	Buffer End Point pH 10	60 mL	
6442		-----		-----	---	---	

normality	OK
n	15
outliers	1
mean (n)	0.3756
st.dev. (n)	0.03576
R(calc.)	0.1001
st.dev.(D664-A:18e2, IP 60 mL)	0.06436
R(D664-A:18e2, IP 60 mL)	0.1802
compare	
R(D664-A:18e2, IP 125 mL)	0.0788
R(D664-A:18e2, BEP 60 mL)	0.2131
R(D664-A:18e2, BEP 125 mL)	0.1129





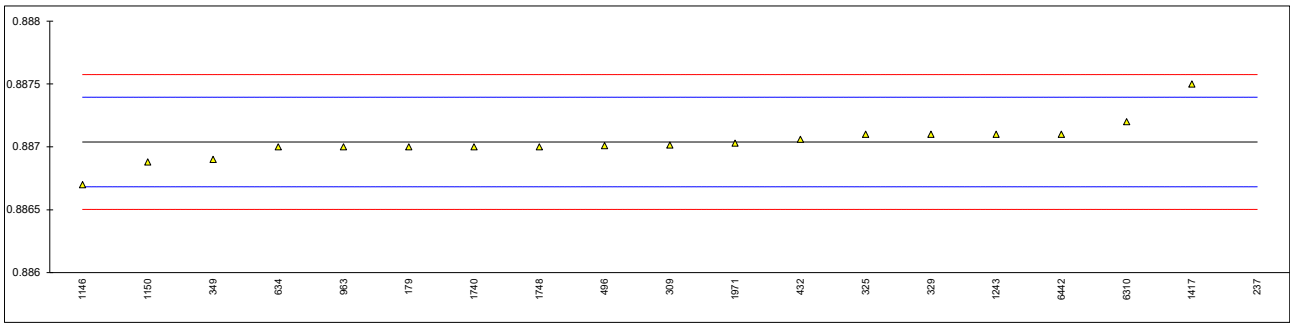
Determination of Copper Corrosion 3 hours at 100 °C on sample #22030;

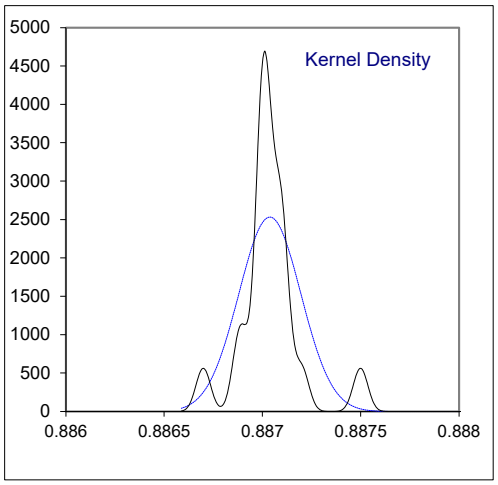
lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D130	1A		-----	
237	D130	1A		-----	
257		-----		-----	
309	D130	1A		-----	
325	D130	1A		-----	
329		-----		-----	
349		-----		-----	
432		-----		-----	
496		-----		-----	
634	D130	1a		-----	
862		-----		-----	
962		-----		-----	
963		-----		-----	
1146		-----		-----	
1150	ISO2160	1a		-----	
1243	ISO2160	1a		-----	
1417	IP154	1B		-----	
1740	D130	1A		-----	
1748	D130	1a		-----	
1971	D130	1a		-----	
6310		-----		-----	
6442	D130	1b		-----	
	n	12			
	mean (n)	1 (1a/1b)			

Determination of Density at 15 °C on sample #22030; results in kg/L

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D4052	0.8870		-0.22	
237	D4052	0.8894	D(0.01)	13.22	
257		-----		-----	
309	D4052	0.887015		-0.13	
325	D4052	0.8871		0.34	
329	D4052	0.8871		0.34	
349	D4052	0.8869		-0.78	
432	D4052	0.88706		0.12	
496	ISO12185	0.88701		-0.16	
634	D4052	0.8870		-0.22	
862		-----		-----	
962		-----		-----	
963	D4052	0.8870		-0.22	
1146	D4052	0.8867		-1.90	
1150	ISO12185	0.88688		-0.89	
1243	ISO12185	0.8871		0.34	
1417	IP365	0.8875	C	2.58	first reported 0.8881
1740	D7042	0.8870		-0.22	
1748	D4052	0.8870		-0.22	
1971	ISO12185	0.88703		-0.05	
6310	D4052	0.8872		0.90	
6442	D4052	0.8871		0.34	

normality	not OK
n	18
outliers	1
mean (n)	0.887039
st.dev. (n)	0.0001576
R(calc.)	0.000441
st.dev.(ISO12185:96)	0.0001786
R(ISO12185:96)	0.0005
compare	
R(D4052:18a)	0.00050

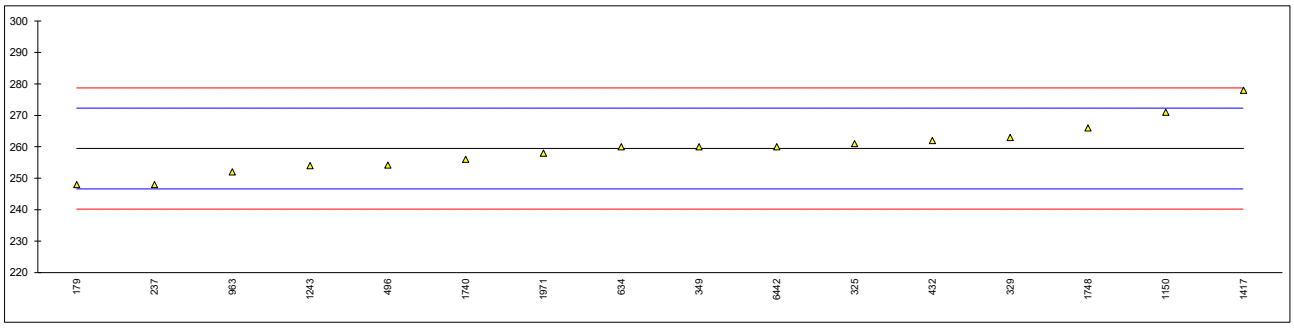


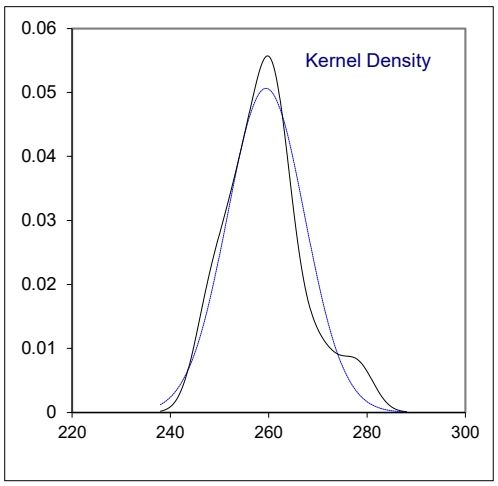


Determination of Flash Point C.O.C. on sample #22030; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D92	248		-1.78	
237	D92	248		-1.78	
257		-----		-----	
309		-----		-----	
325	D92	261		0.24	
329	D92	263		0.55	
349	D92	260		0.09	
432	D92	262		0.40	
496	D92	254.2		-0.82	
634	D92	260	C	0.09	first reported 284
862		-----		-----	
962		-----		-----	
963	D92	252.0		-1.16	
1146		-----		-----	
1150	ISO2592	271		1.80	
1243	ISO2592	254		-0.85	
1417	D92	278		2.89	
1740	D92	256		-0.54	
1748	D92	266		1.02	
1971	ISO2592	258		-0.23	
6310		-----		-----	
6442	D92	260		0.09	

normality	OK
n	16
outliers	0
mean (n)	259.45
st.dev. (n)	7.882
R(calc.)	22.07
st.dev.(D92:18)	6.429
R(D92:18)	18

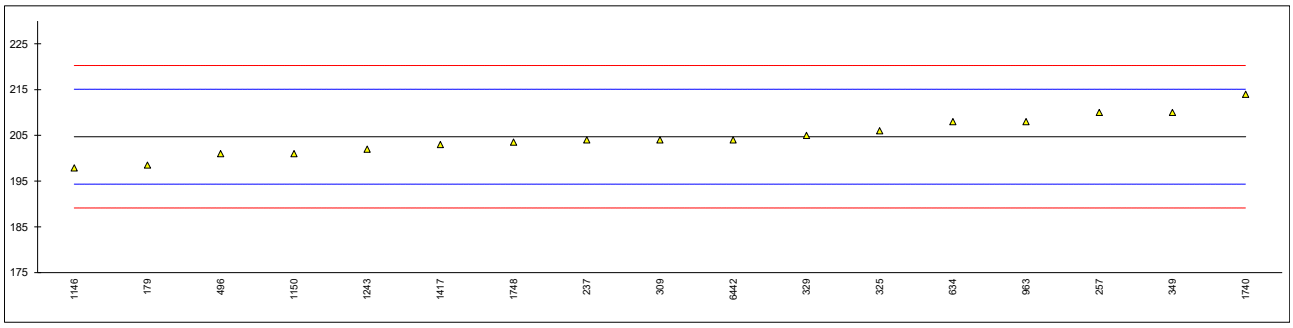


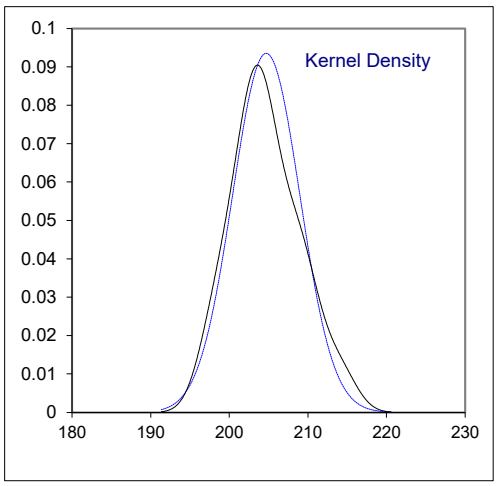


Determination of Flash Point PMcc on sample #22030; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D93-A	198.5		-1.19	
237	D93-A	204		-0.13	
257	D3828	210		1.02	
309	D93-A	204.0		-0.13	
325	D93-A	206.0		0.25	
329	D93-A	205		0.06	
349	D93-A	210		1.02	
432		-----		-----	
496	D93-A	201.0		-0.71	
634	D93-A	208.0		0.64	
862		-----		-----	
962		-----		-----	
963	D93-A	208.0		0.64	
1146	D93-A	197.9		-1.31	
1150	ISO2719-A	201		-0.71	
1243	ISO2719-A	202		-0.52	
1417	D93-A	203		-0.33	
1740	D93-A	214		1.79	
1748	D93-A	203.5		-0.23	
1971		-----		-----	
6310		-----		-----	
6442	D93-A	204		-0.13	

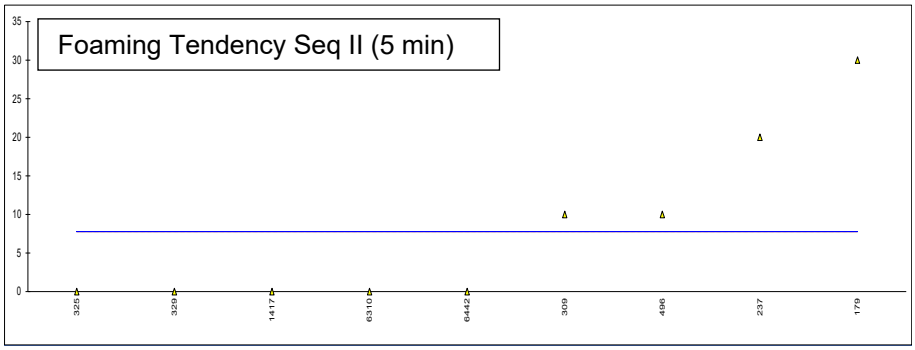
normality	OK
n	17
outliers	0
mean (n)	204.70
st.dev. (n)	4.267
R(calc.)	11.95
st.dev.(D93-A:20)	5.191
R(D93-A:20)	14.53

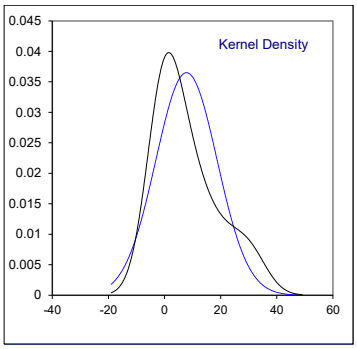




Determination of Foaming Tendency, Sequence I, II and III (5 min. blowing period) on sample #22030; results in mL

lab	method	Sample	Diffuser	Seq I	mark	z(targ)	Seq II	mark	z(targ)	Seq III	mark	z(targ)
178		---	---	-----		-----	-----		-----	-----		-----
179	D892	As received	Metal	0		-----	30		-----	0		-----
237	D892	---	---	10		-----	20		-----	10		-----
257		---	---	-----		-----	-----		-----	-----		-----
309	D892	As received	Metal	0		-----	10		-----	0		-----
325	D892	As received	Metal	0		-----	0		-----	0		-----
329	D892	As received	Metal	0		-----	0		-----	0		-----
349		---	Metal	-----		-----	-----		-----	-----		-----
432		---	---	-----		-----	-----		-----	-----		-----
496	D892	After agit (A)	Metal	0		-----	10		-----	0		-----
634		---	---	-----		-----	-----		-----	-----		-----
862		---	---	-----		-----	-----		-----	-----		-----
962		---	---	-----		-----	-----		-----	-----		-----
963		---	---	-----		-----	-----		-----	-----		-----
1146		---	---	-----		-----	-----		-----	-----		-----
1150		---	---	-----		-----	-----		-----	-----		-----
1243	D892	As received	Stone	0		-----	<10		-----	0		-----
1417	D892	As received	Metal	0		-----	0		-----	0		-----
1740		---	---	-----		-----	-----		-----	-----		-----
1748		---	---	-----		-----	-----		-----	-----		-----
1971		---	---	-----		-----	-----		-----	-----		-----
6310	D892	After agit (A)	Metal	0		-----	0		-----	0		-----
6442	D892	After agit (A)	Metal	0		-----	0		-----	0		-----
	normality						suspect					
	n			9			9			9		
	outliers						0					
	mean (n)			0			7.78			0		
	st.dev. (n)						10.929					
	R(calc.)						30.60					
	st.dev.(D892:18)						(5.505)					
	R(D892:18)						(15.42)					





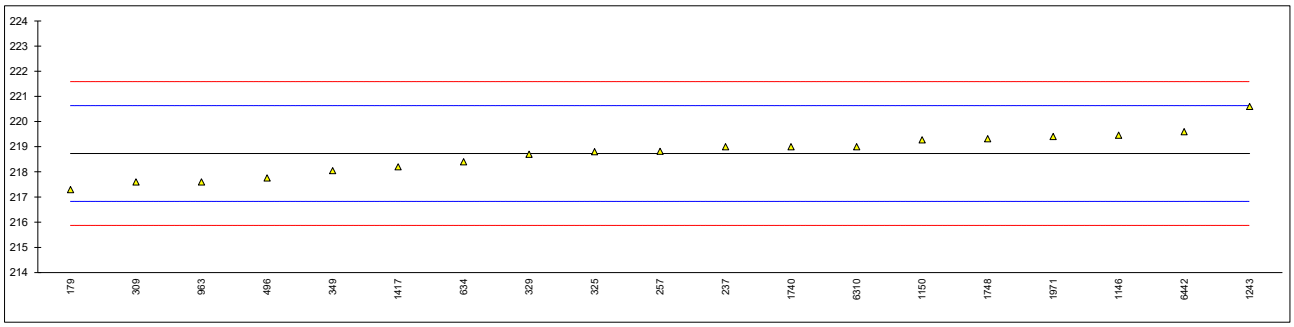
Determination of Foam Stability, Sequence I, II and III (10 min. settling period) on sample #22030; results in mL

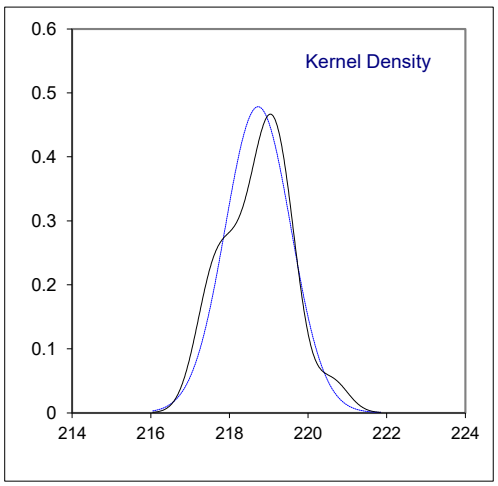
lab	method	Seq I	mark	z(targ)	Seq II	mark	z(targ)	Seq III	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179	D892	0		-----	0		-----	0		-----
237	D892	0		-----	0		-----	0		-----
257		-----		-----	-----		-----	-----		-----
309	D892	0		-----	0		-----	0		-----
325	D892	0		-----	0		-----	0		-----
329	D892	0		-----	0		-----	0		-----
349		-----		-----	-----		-----	-----		-----
432		-----		-----	-----		-----	-----		-----
496	D892	0		-----	0		-----	0		-----
634		-----		-----	-----		-----	-----		-----
862		-----		-----	-----		-----	-----		-----
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
1146		-----		-----	-----		-----	-----		-----
1150		-----		-----	-----		-----	-----		-----
1243	D892	0		-----	0		-----	0		-----
1417	D892	0		-----	0		-----	0		-----
1740		-----		-----	-----		-----	-----		-----
1748		-----		-----	-----		-----	-----		-----
1971		-----		-----	-----		-----	-----		-----
6310	D892	0		-----	0		-----	0		-----
6442	D892	0		-----	0		-----	0		-----
	n	10			10			10		
	mean (n)	0			0			0		

Determination of Kinematic Viscosity at 40 °C on sample #22030; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D445	217.30		-1.50	
237	D445	219.0		0.28	
257	D7279 corrected to D445	218.82		0.09	
309	D445	217.6		-1.19	
325	D445	218.8		0.07	
329	D445	218.7		-0.03	
349	D445	218.05		-0.71	
432		-----		-----	
496	D445	217.76		-1.02	
634	D445	218.4		-0.35	
862		-----		-----	
962		-----		-----	
963	D445	217.6		-1.19	
1146	D445	219.46		0.76	
1150	ISO3104	219.275		0.57	
1243	D7279 corrected to D445	220.60		1.96	
1417	D445	218.2		-0.56	
1740	D445	219.0		0.28	
1748	D7042	219.32		0.62	
1971	D445	219.41		0.71	
6310	D7279 corrected to D445	219		0.28	
6442	D7042	219.6		0.91	

normality	OK
n	19
outliers	0
mean (n)	218.731
st.dev. (n)	0.8340
R(calc.)	2.335
st.dev.(D445:21e2)	0.9530
R(D445:21e2)	2.669

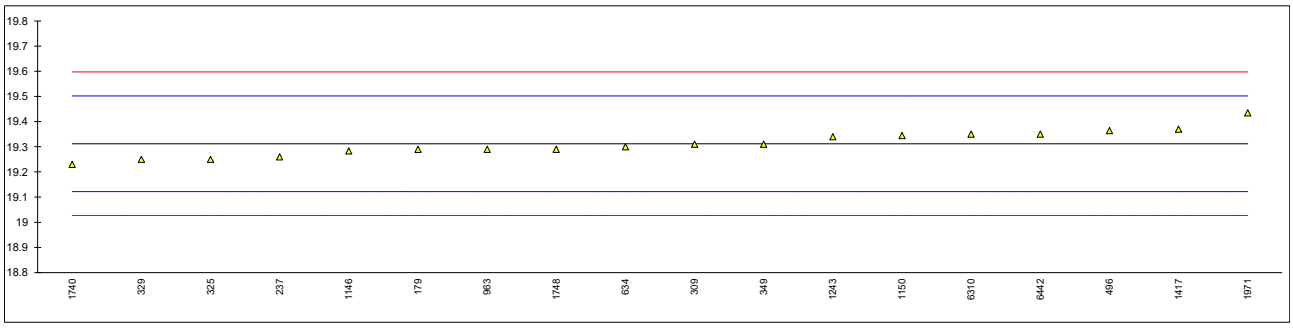


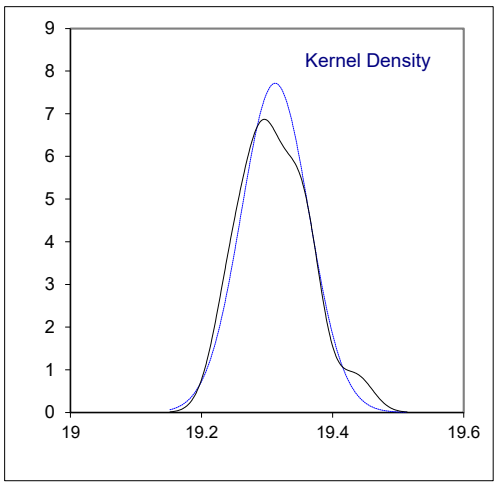


Determination of Kinematic Viscosity at 100 °C on sample #22030; results in mm²/s

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D445	19.29		-0.23	
237	D445	19.26		-0.55	
257		-----		-----	
309	D445	19.31		-0.02	
325	D445	19.25		-0.65	
329	D445	19.25		-0.65	
349	D445	19.31		-0.02	
432		-----		-----	
496	D445	19.364		0.55	
634	D445	19.30		-0.13	
862		-----		-----	
962		-----		-----	
963	D445	19.29		-0.23	
1146	D445	19.284		-0.30	
1150	ISO3104	19.345		0.35	
1243	D7279 corrected to D445	19.34		0.29	
1417	D445	19.37	C	0.61	first reported 25.58
1740	D445	19.23		-0.86	
1748	D7042	19.29		-0.23	
1971	D445	19.435		1.29	
6310	D7279 corrected to D445	19.35		0.40	
6442	D7042	19.35		0.40	

normality	OK
n	18
outliers	0
mean (n)	19.312
st.dev. (n)	0.0517
R(calc.)	0.145
st.dev.(D445:21e2)	0.0952
R(D445:21e2)	0.267

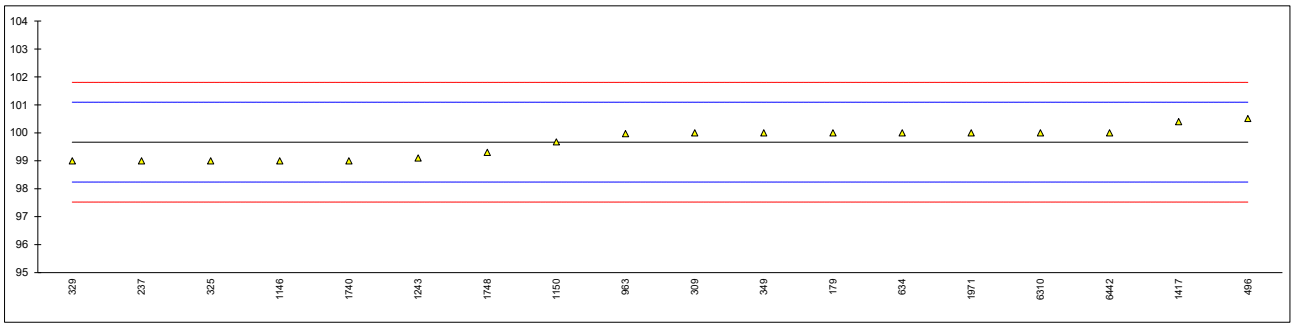


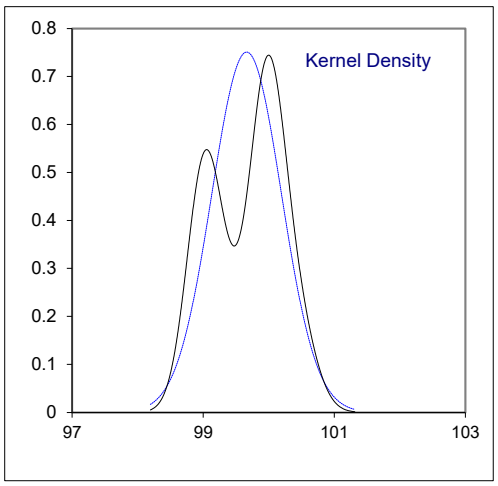


Determination of Viscosity Index on sample #22030

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D2270	100		0.47	
237	D2270	99		-0.93	
257		-----		-----	
309	D2270	100		0.47	
325	D2270	99		-0.93	
329	D2270	99		-0.93	
349	D2270	100		0.47	
432		-----		-----	
496	D2270	100.51		1.18	
634	D2270	100		0.47	
862		-----		-----	
962		-----		-----	
963	D2270	99.975		0.43	
1146	D2270	99		-0.93	
1150	ISO2909	99.675		0.01	
1243	ISO2909	99.1		-0.79	
1417	D2270	100.4	C	1.03	first reported 148.7
1740	D2270	99.0		-0.93	
1748	D2270	99.3		-0.51	
1971	D2270	100		0.47	
6310	D2270	100		0.47	
6442	D2270	100		0.47	

normality	OK
n	18
outliers	0
mean (n)	99.66
st.dev. (n)	0.531
R(calc.)	1.49
st.dev.(D2270:10)	0.714
R(D2270:10)	2

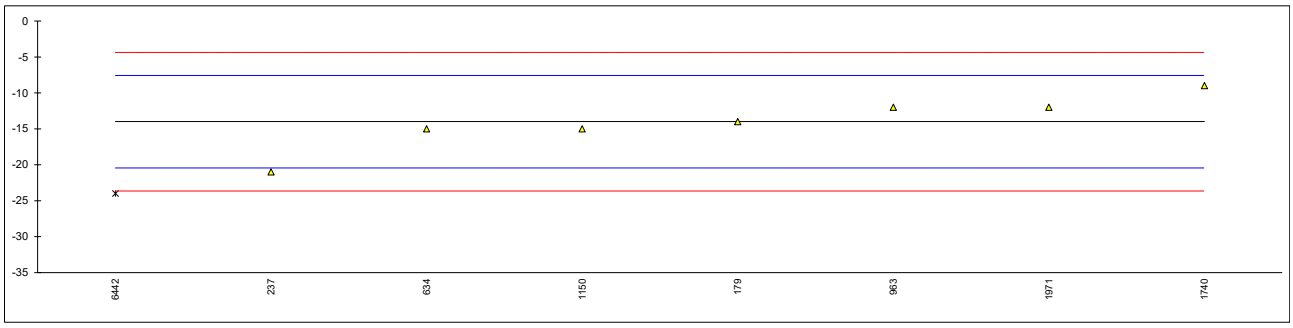


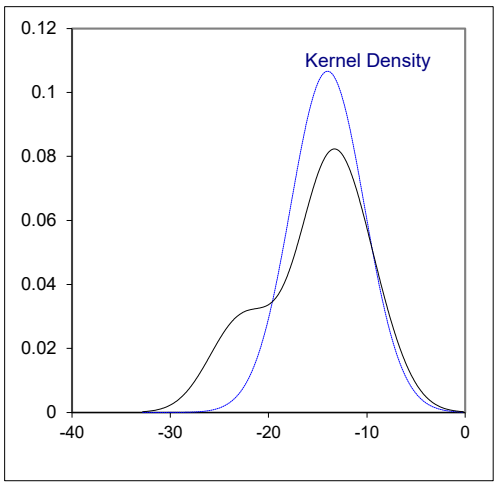


Determination of Pour Point Manual on sample #22030; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D97	-14		0.00	
237	D97	-21		-2.18	
257		-----		-----	
309		-----		-----	
325		-----		-----	
329		-----		-----	
349		-----		-----	
432		-----		-----	
496		-----		-----	
634	D97	-15		-0.31	
862		-----		-----	
962		-----		-----	
963	D97	-12		0.62	
1146		-----		-----	
1150	ISO3016	-15		-0.31	
1243		-----		-----	
1417		-----		-----	
1740	ISO3016	-9		1.56	
1748		-----		-----	
1971	D97	-12		0.62	
6310		-----		-----	
6442	D97	-24	G(0.05)	-3.11	

normality	unknown
n	7
outliers	1
mean (n)	-14.00
st.dev. (n)	3.742
R(calc.)	10.48
st.dev.(D97:17b)	3.214
R(D97:17b)	9

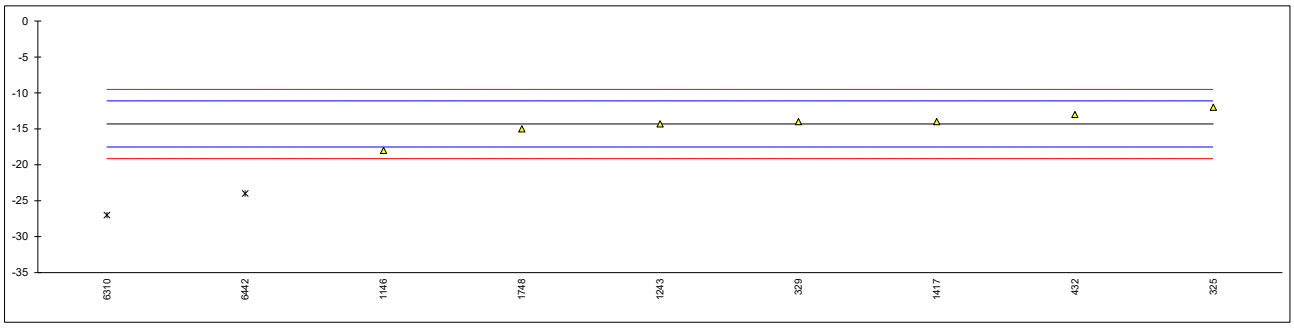


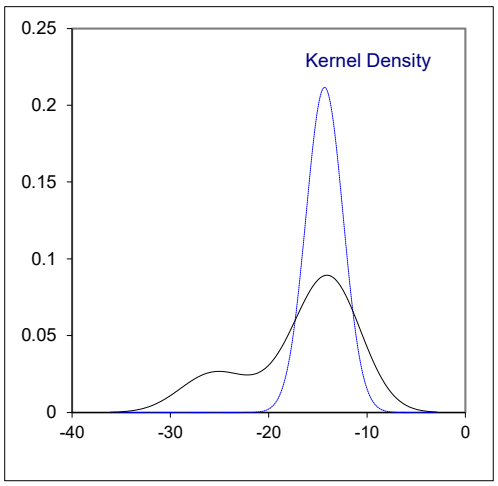


Determination of Pour Point Automated 1 °C interval on sample #22030; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
257		-----		-----	
309		-----		-----	
325	D5950	-12		1.45	
329	D5950	-14		0.20	
349		-----		-----	
432	D5950	-13		0.83	
496		-----		-----	
634		-----		-----	
862		-----		-----	
962		-----		-----	
963		-----		-----	
1146	D6892	-18	C	-2.28	reported as PP Manual
1150		-----		-----	
1243	D7346	-14.3		0.02	
1417	D5950	-14		0.20	
1740		-----		-----	
1748	D7346	-15		-0.42	
1971		-----		-----	
6310	D5950	-27	DG(0.05)	-7.88	
6442	D6892	-24	DG(0.05)	-6.02	

normality	not OK
n	7
outliers	2
mean (n)	-14.33
st.dev. (n)	1.886
R(calc.)	5.28
st.dev.(D5950:14)	1.607
R(D5950:14)	4.5





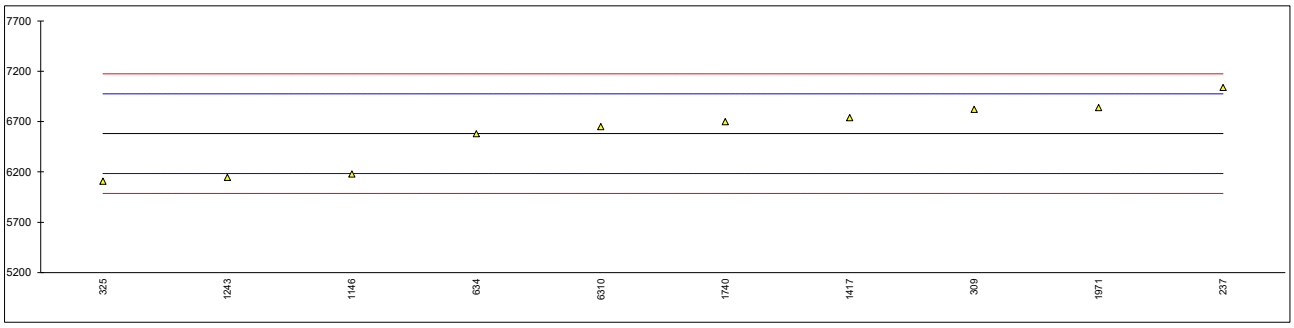
Determination of Rust Prevention distilled water on sample #22030

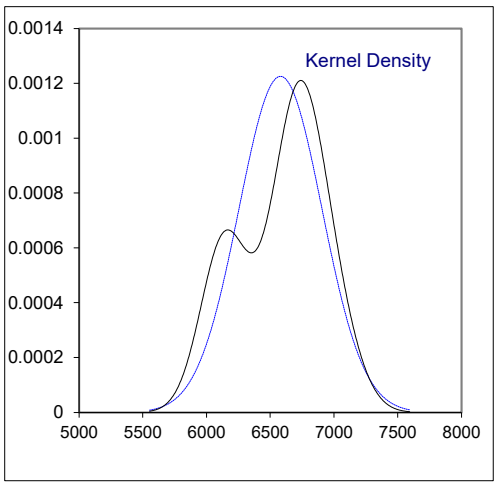
lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D665	PASS		-----	
237	D665	PASS		-----	
257		-----		-----	
309		-----		-----	
325	D665	pass		-----	
329		-----		-----	
349		-----		-----	
432		-----		-----	
496		-----		-----	
634		-----		-----	
862		-----		-----	
962		-----		-----	
963		-----		-----	
1146		-----		-----	
1150		-----		-----	
1243		-----		-----	
1417	D665	PASS		-----	
1740		-----		-----	
1748		-----		-----	
1971		-----		-----	
6310		-----		-----	
6442	D665	Pass		-----	4 hrs
	n	5			
	mean (n)	Pass			

Determination of Sulfur on sample #22030; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D4294	7040		2.32	
257		-----		-----	
309	D2622	6822		1.22	
325	D5185	6108		-2.39	
329		-----		-----	
349		-----		-----	
432		-----		-----	
496		-----		-----	
634	D4294	6580		0.00	
862		-----		-----	
962		-----		-----	
963		-----		-----	
1146	D4294	6180		-2.02	
1150		-----		-----	
1243	ISO8754	6146		-2.19	
1417	In house	6740		0.80	
1740	D4294	6700		0.60	
1748		-----		-----	
1971	ISO8754	6840		1.31	
6310	D7751	6650		0.35	
6442		-----		-----	

normality	OK
n	10
outliers	0
mean (n)	6580.6
st.dev. (n)	325.54
R(calc.)	911.5
st.dev.(D4294:21)	198.15
R(D4294:21)	554.8

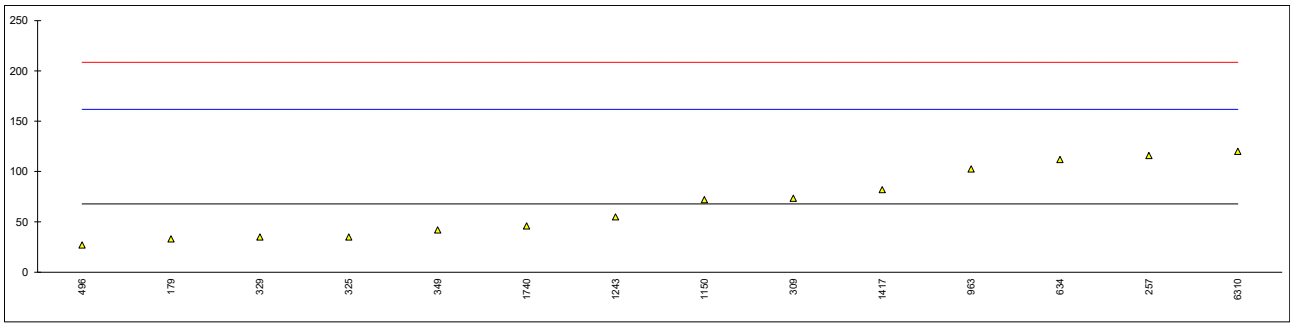


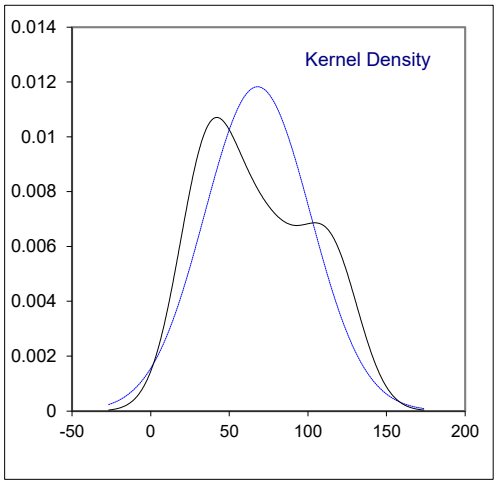


Determination of Water on sample #22030; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D6304-C:20	33		-0.74	
237		-----		-----	
257	D7889	116		1.02	
309	D6304-A:20	73.5		0.12	
325	D6304-C:20	35		-0.70	
329	D6304-C:20	35		-0.70	
349	D6304-C:20	42	C	-0.55	first reported 12
432		-----		-----	
496	D6304-B:20	27		-0.87	
634	D6304-B:20	112		0.94	
862		-----		-----	
962		-----		-----	
963	D6304-A:16e1	102.5		0.74	
1146	D6304-B:20	<100		-----	
1150	ISO12937	72		0.09	
1243	ISO12937	55		-0.28	
1417	D6304-A:20	82		0.30	
1740	D6304-B:20	46		-0.47	
1748		-----		-----	
1971		-----		-----	
6310	D6304-C:20	120		1.11	
6442		-----		-----	

normality	OK
n	14
outliers	0
mean (n)	67.93
st.dev. (n)	33.729
R(calc.)	94.44
st.dev.(D6304-B:20)	46.909
R(D6304-B:20)	131.34
compare	
R(D6304-A:20)	45.82
R(D6304-C:20)	32.71

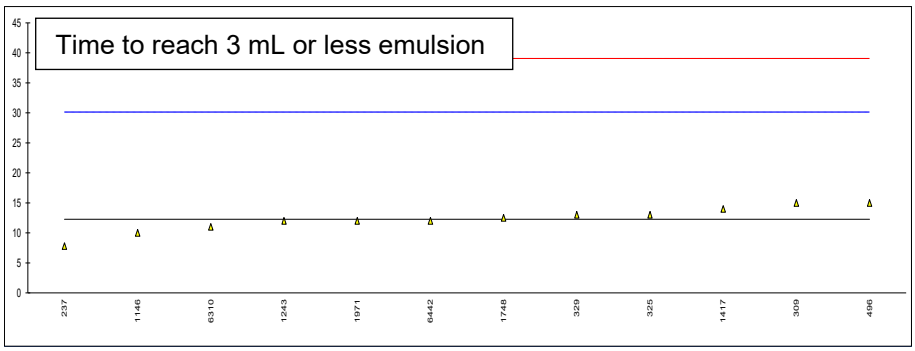


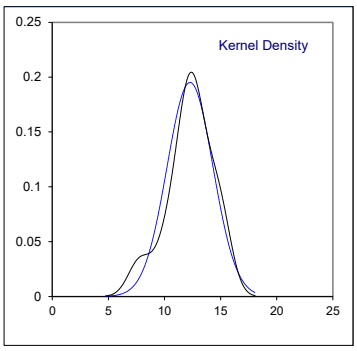


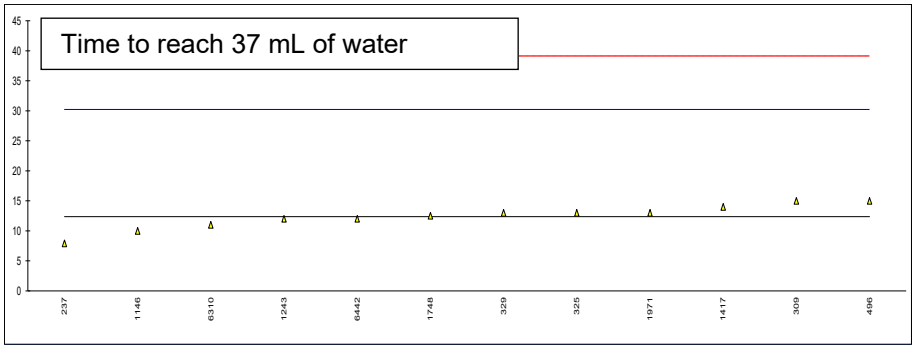
Determination of Water Separability at 82 °C, distilled water on sample #22030; results in minutes

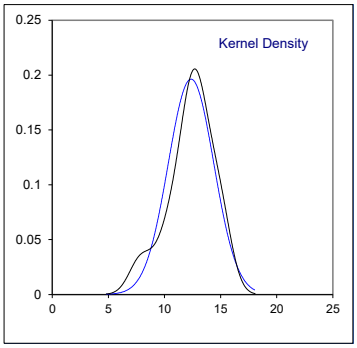
lab	method	3 mL or less emulsion	z(targ)	37 mL of water	z(targ)	complete break (40-40-0)		z(targ)	test aborted	time aborted
178		-----	-----	-----	-----	-----		-----	-----	-----
179		-----	-----	-----	-----	-----		-----	-----	-----
237	D1401	7.8	-0.50	7.9	-0.50	13.4		-0.24	No	-----
257		-----	-----	-----	-----	-----		-----	-----	-----
309	D1401	15	0.31	15	0.29	15		-0.06	Yes	15
325	D1401	13	0.08	13	0.07	18		0.27	-----	-----
329	D1401	13	0.08	13	0.07	18		0.27	No	-----
349		-----	-----	-----	-----	-----		-----	-----	-----
432		-----	-----	-----	-----	-----		-----	-----	-----
496	D1401	15	0.31	15	0.29	-----		-----	No	-----
634		-----	-----	-----	-----	-----		-----	-----	-----
862		-----	-----	-----	-----	-----		-----	-----	-----
962		-----	-----	-----	-----	-----		-----	-----	-----
963		-----	-----	-----	-----	-----		-----	-----	-----
1146	D1401	10	-0.25	10	-0.27	-----		-----	Yes	10
1150		-----	-----	-----	-----	-----		-----	-----	-----
1243	ISO6614	12	-0.03	12	-0.04	16	C	0.05	Yes	30
1417	D1401	14	0.19	14	0.18	14		-0.17	No	-----
1740		-----	-----	-----	-----	-----		-----	-----	-----
1748	D1401	12.5	0.03	12.5	0.01	-----		-----	Yes	-----
1971		12	-0.03	13	0.07	-----		-----	Yes	60
6310		11	-0.14	11	-0.15	15		-0.06	No	-----
6442	D1401	12	-0.03	12	-0.04	15		-0.06	No	-----
	normality	OK		OK		unknown				
	n	12		12		8				
	outliers	0		0		0				
	mean (n)	12.3		12.4		15.6				
	st.dev. (n)	2.04		2.03		1.70				
	R(calc.)	5.7		5.7		4.7				
	st.dev.(D1401:21)	8.93		8.93		8.93				
	R(D1401:21)	25		25		25				

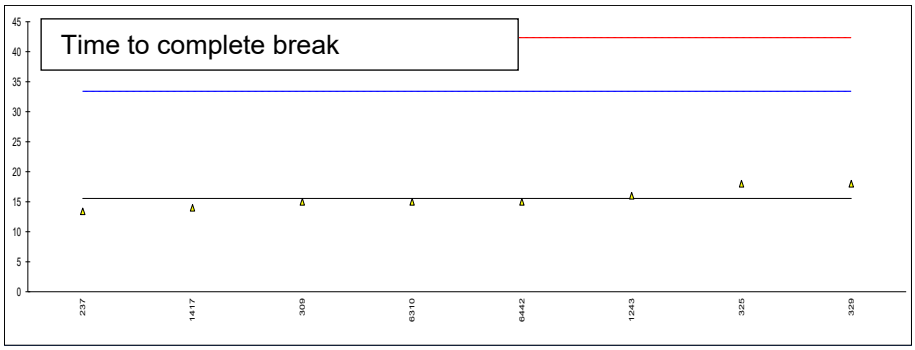
Lab 1243: first reported >30

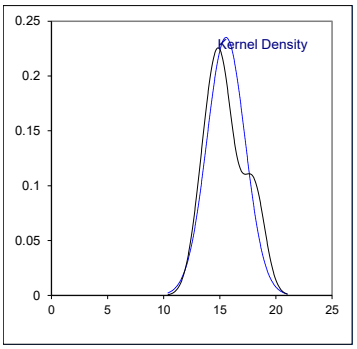

Time to reach 3 mL or less emulsion
45
40
35
30
25
20
15
10
5
0
237
1146
6310
1243
1971
6442
1748
329
325
1417
309
496


Kernel Density
0.25
0.2
0.15
0.1
0.05
0
0
5
10
15
20
25


Time to reach 37 mL of water
45
40
35
30
25
20
15
10
5
0
237
1146
6310
1243
6442
1748
329
325
1971
1417
309
496


Kernel Density
0.25
0.2
0.15
0.1
0.05
0
0
5
10
15
20
25


Time to complete break
45
40
35
30
25
20
15
10
5
0
237
1417
309
6310
6442
1243
325
329


Kernel Density
0.25
0.2
0.15
0.1
0.05
0
0
5
10
15
20
25

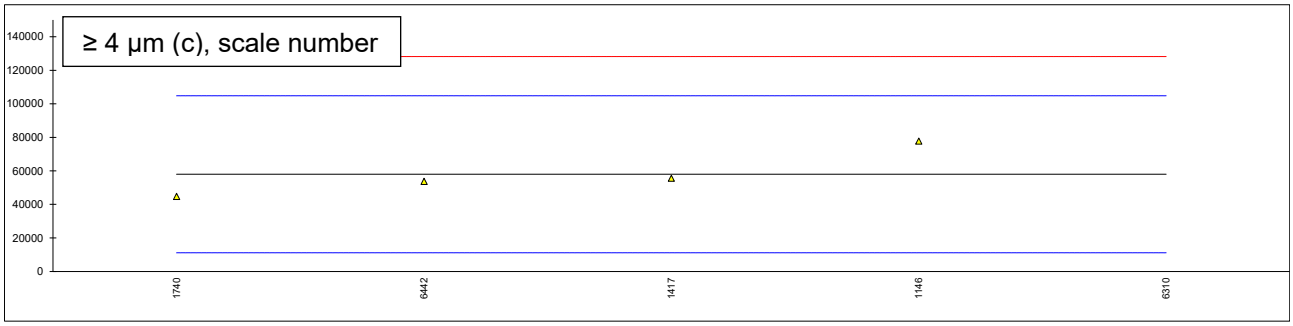
Determination of Water Separability at 82 °C, distilled water on sample #22030; results in mL

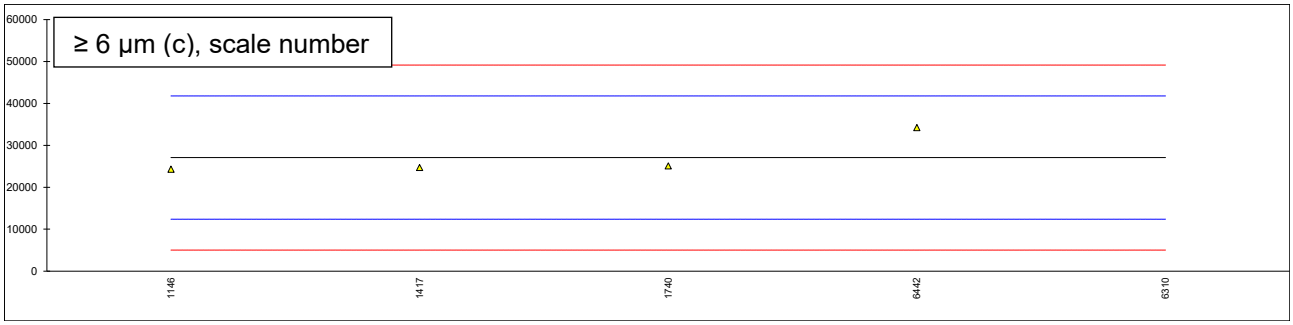
lab	method	volume oil phase	mark	volume water phase	mark	volume emulsion phase	mark
178		-----		-----		-----	
179		-----		-----		-----	
237	D1401	40.0		40.0		0	
257		-----		-----		-----	
309	D1401	40		40		0	
325		-----		-----		-----	
329		-----		-----		-----	
349		-----		-----		-----	
432		-----		-----		-----	
496	D1401	39		38		3	
634		-----		-----		-----	
862		-----		-----		-----	
962		-----		-----		-----	
963		-----		-----		-----	
1146	D1401	43		37		0	
1150		-----		-----		-----	
1243	ISO6614	41		39		0	
1417	D1401	40		40		0	
1740		-----		-----		-----	
1748	D1401	40		40		0	
1971		42		38		0	
6310		-----		-----		-----	
6442		-----		-----		-----	

Determination of Level of Contamination on sample #22030; 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		-----		-----	-----		-----	-----		-----
634		-----		-----	-----		-----	-----		-----
862		-----		-----	-----		-----	-----		-----
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
1146	D7647	77754		0.84	24320		-0.38	209		-----
1150		-----		-----	-----		-----	-----		-----
1243		-----		-----	-----		-----	-----		-----
1417	ISO4406	55593	C	-0.10	24725	C	-0.32	171	C	-----
1740	ISO4407	44807		-0.56	25097		-0.27	403		-----
1748		-----		-----	-----		-----	-----		-----
1971		-----		-----	-----		-----	-----		-----
6310	ISO4407	7096017	G(0.01)	300.76	1014282	E,G(0.01)	134.23	52672	E,G(0.01)	-----
6442	D7647	53782		-0.18	34243		0.97	1599		-----
	normality	unknown			unknown			unknown		
	n	4			4			4		
	outliers	1			1			1		
	mean (n)	57984.0			27096.3			595.5		
	st.dev. (n)	13998.33			4775.05			676.67		
	R(calc.)	39195.3			13370.2			1894.7		
	st.dev.(D7647:10)	23400.69			7354.70			(287.12)		
	R(D7647:10)	65521.9			20593.2			(803.9)		

Lab 1417: reported test results as ISO4406 scale number
Lab 6310: test result in counts/ml and ISO4406 scale number does not match for ≥ 6 µm and ≥ 14 µm





Determination of Level of Contamination acc. to ISO4406 scale on sample #22030; 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		-----		-----	-----		-----	-----		-----
634		-----		-----	-----		-----	-----		-----
862		-----		-----	-----		-----	-----		-----
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
1146	ISO4406	23		-----	22		-----	15		-----
1150		-----		-----	-----		-----	-----		-----
1243		-----		-----	-----		-----	-----		-----
1417		-----		-----	-----		-----	-----		-----
1740	ISO4406	23		-----	22		-----	16		-----
1748		-----		-----	-----		-----	-----		-----
1971		-----		-----	-----		-----	-----		-----
6310	ISO4406	23		-----	21	E	-----	16	E	-----
6442	D7647	23		-----	22		-----	18		-----
	n	4			4			4		
	mean (n)	23			22			16		

Lab 6310: test result in counts/ml and ISO4406 scale number does not match for ≥ 6 µm and ≥ 14 µm

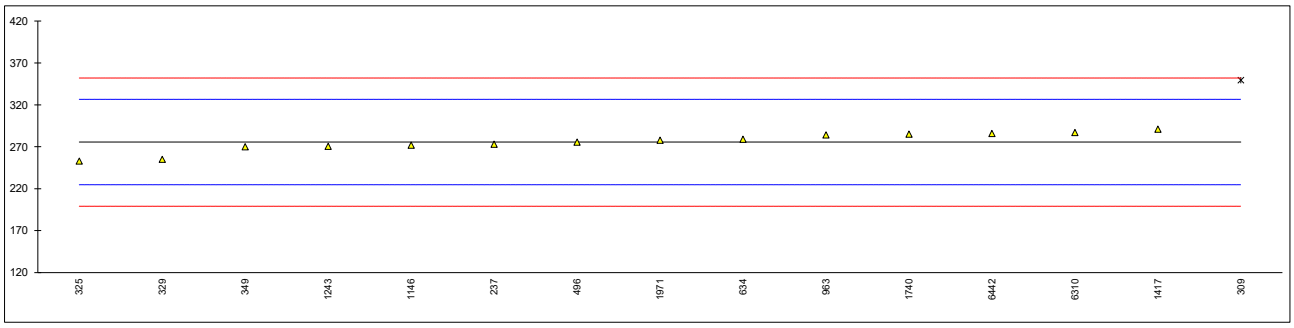
Determination of Calcium as Ca on sample #22030; results in mg/kg

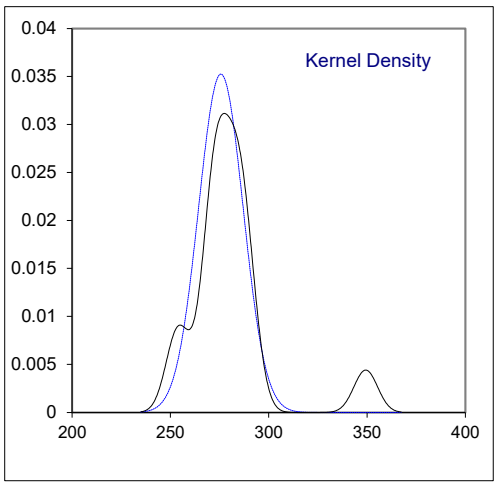
lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D5185	<40		-----	
257		-----		-----	
309		283.4		-----	possibly a false positive test result?
325	D5185	1		-----	
329	D5185	1		-----	
349	D5185	0		-----	
432		-----		-----	
496	D5185	<0.1		-----	
634	D6595	1.0		-----	
862		-----		-----	
962		-----		-----	
963	D5185	0.69		-----	
1146	D4951	<5		-----	
1150		-----		-----	
1243	DIN51399	0.4		-----	
1417	D5185	1.51		-----	
1740	D5185	0.2		-----	
1748		-----		-----	
1971	D4951	0.88		-----	
6310	D7751	<1		-----	
6442	D5185	<10		-----	
	n	14			
	mean (n)	<40			D5185:18 application range: 40 – 9000 mg/kg

Determination of Phosphorus as P on sample #22030; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		273		-0.10	
257		-----		-----	
309		349.4	D(0.01)	2.89	
325		253		-0.89	
329		255		-0.81	
349		270		-0.22	
432		-----		-----	
496		275.6		0.00	
634		279	C	0.13	first reported 169
862		-----		-----	
962		-----		-----	
963		284.19		0.33	
1146		272		-0.14	
1150		-----		-----	
1243		270.6		-0.20	
1417		291		0.60	
1740		285		0.37	
1748		-----		-----	
1971		277.8		0.08	
6310		287		0.44	
6442		286		0.41	

normality	OK
n	14
outliers	1
mean (n)	275.66
st.dev. (n)	11.317
R(calc.)	31.69
st.dev.(D5185:18)	25.497
R(D5185:18)	71.39





Determination of Zinc as Zn on sample #22030; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		<60		-----	
257		0.3		-----	
309		121.8		-----	possibly a false positive test result?
325		<1		-----	
329		<1		-----	
349		1		-----	
432		-----		-----	
496		0.049		-----	
634		0.6		-----	
862		-----		-----	
962		-----		-----	
963		0.45		-----	
1146		<5		-----	
1150		-----		-----	
1243		n.n.		-----	
1417		0.71		-----	
1740		<0.1		-----	
1748		-----		-----	
1971		1.32		-----	
6310		<1		-----	
6442		<10		-----	
	n	15			
	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	BULGARIA
1 lab in	CHINA, People's Republic
2 labs in	GERMANY
1 lab in	GREECE
1 lab in	JORDAN
2 labs in	NETHERLANDS
1 lab in	NIGERIA
1 lab in	PHILIPPINES
1 lab in	POLAND
1 lab in	PORTUGAL
2 labs in	SAUDI ARABIA
1 lab in	SPAIN
1 lab 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)	= 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

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2 ISO5725:86
3 ISO5725 parts 1-6:94
4 ISO13528:05
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6 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
7 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
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10 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)
11 W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)