Results of Proficiency Test
Gear Oil (fresh)
March 2020

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 2019/2020 it was decided to continue the proficiency test for the analysis of fresh Gear Oil.

In this interlaboratory study 27 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 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 fresh Gear Oil, a 1L bottle and a 0.5L bottle, both labelled #20030.
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 125 liters of fresh Gear Oil was obtained from a local refinery. After homogenization 44 amber glass bottles of 1L and 44 amber glass bottles of 0.5L were filled and labelled #20030. The homogeneity of subsamples #20030 was checked by the determination of Density in accordance with ISO12185 on 8 stratified randomly selected subsamples.

	Density at 15°C in kg/L
Sample #20030-1	0.88759
Sample #20030-2	0.88760
Sample #20030-3	0.88759
Sample #20030-4	0.88760
Sample #20030-5	0.88760
Sample #20030-6	0.88760
Sample #20030-7	0.88760
Sample #20030-8	0.88760

Table 1: homogeneity test results of subsamples #20030

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

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

Table 2: evaluation of the repeatability of subsamples #20030

The calculated repeatability was 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 #20030 were sent on February 26, 2020. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Gear Oil fresh 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 on sample #20030: Total Acid Number, Copper Corrosion 3 hrs at 100°C, Density at 15°C, Flash Point (C.O.C. and PMcc), Foaming Tendency and Stability, Kinematic Viscosity at 40°C and at 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 appropriate reference test methods 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 test 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.

According to ISO5725 the original test results per determination were submitted to Dixon's and/or Grubbs' and/or Rosner's outlier tests. 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 was projected over the Kernel Density Graph for reference.

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. EN or 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.

This 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. In some cases, a reproducibility based on former iis proficiency tests could be used.

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 result tables of appendix 1.

Absolute values for z<2 are very common and absolute values for z>3 are very rare.
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 interlaboratory study some problems were encountered with the dispatch of the samples due to several reasons with transportation (e.g. closed borders due to COVID-19 pandemic). Therefore, the data entry portal was kept open for a few weeks. Six participants did not report any test results. Not all participants were able to report test results for all the requested tests.
In total 21 participants reported 384 numerical test results. Observed were 32 outlying test results, which is 8.3% of the numerical test results. In proficiency studies, outlier percentages of 3%-7.5% are quite normal.

Not all original 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 methods are referred to with a number (e.g. D7647) and an added designation for the year that the 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) is used.

Total Acid Number: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D664-A:18e2; IP 60mL and BEP 60mL but is not in agreement with BEP 125mL and IP 125mL. 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 problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ISO12185:96.

Flash Point C.O.C.: This determination was very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of ASTM D92:18.

Flash Point PMcc: This determination was problematic for a number of laboratories. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D93-A:19.

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

The calculated reproducibilities in the Foam Stability for sequence I is not at all in agreement with the requirements of ASTM D892:18. All reporting participants reported 0mL for Foam Stability for sequence II. Therefore, no z-scores were calculated. For Foam Stability for sequence III the variation in the test results was very high. Therefore, no z-scores were calculated.

Kinematic Viscosity at 40°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D445:19a.

Kinematic Viscosity at 100°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D445:19a.

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).
The Viscosity Index was also calculated by iis from the test results reported for the kinematic viscosities at 40°C and 100°C. No calculation errors were observed.

Pour Point - Manual: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of ASTM D97:17b.

Pour Point - Automated: This determination was very problematic. One statistical outlier was observed. The calculated reproducibility is not at all 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 not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of ASTM D4294:16e1.

Water: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D6304:16e1.

Water Separability: This determination is not problematic. In total seven statistical outliers were observed in 6 parameters and two other test results were excluded. The calculated reproducibilities of “time to reach 3mL or less emulsion”, “time to reach 37mL of water” and “complete break” after rejection of the suspect data are all in agreement with the requirements of ASTM D1401:19.
Remarkably, the calculated reproducibilities of the volume oil phase, the volume water phase and the volume emulsion phase are smaller compared to the calculated reproducibilities of iis19L01 PT of 2019 (e.g. 3.0 vs 2.5).

Level of Contamination: This determination may not be problematic. In total eleven statistical outliers were observed over six parameters. Eleven other test results were excluded because the related test values were statistical outliers.
The calculated reproducibilities for counts/mL (>4µm, >6µm and >14µm) after rejection of the suspect data are all in agreement with the requirements of ASTM D7647:10(2018).
The calculated reproducibilities for Level of Contamination acc. ISO4406 scale (>4µm, >6µm and >14µm) after rejection of the suspect data are not in agreement with the requirements of ASTM D7647:10(2018).

Calcium as Ca: This determination may not be problematic. No statistical outliers were observed. However, the consensus value for the Calcium determination was below the application range of ASTM D5185:18. Therefore, no z-scores were calculated.

Phosphorus as P: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D5185:18.

Zinc as Zn: This determination may not be problematic. One statistical outlier was observed. However, the consensus value for the Zinc determination was below the application range of ASTM D5185:18. 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 relevant 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 test methods) or previous proficiency tests are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Total Acid Number	mg KOH/g	15	0.36	0.14	0.17
Copper Corrosion, 3hrs at 100°C		13	1 (1a/1b)	n.a.	n.a.
Density at 15°C	kg/L	17	0.8876	0.0006	0.0005
Flash Point C.O.C.	°C	10	269	37	18
Flash Point PMcc	°C	11	203	7	14
Foaming Tendency (Seq I) (5min)	mL	12	473	216	146
Foaming Tendency (Seq II) (5min)	mL	13	467	291	218
Foaming Tendency (Seq III) (5min)	mL	13	194	457	(101)
Foaming Stability (Seq I) (10min)	mL	12	323	316	108
Foaming Stability (Seq II) (10min)	mL	13	0	0	(0)
Foaming Stability (Seq III) (10min)	mL	11	21	62	(22)

Parameter	unit	n	average	2.8 * sd	R(lit)
Kinematic Viscosity at 40°C	mm^2/s	20	217.14	2.40	2.65
Kinematic Viscosity at 100°C	mm^2/s	19	19.395	0.270	0.268
Viscosity Index		19	101.1	2.2	2
Pour Point, Manual	°C	5	-18.8	9.2	9
Pour Point, Automated	°C	5	-16.3	13.9	4.5
Rust Prevention (distilled water)		6	Pass	n.a.	n.a.
Sulfur	mg/kg	6	8486	655	654
Water	mg/kg	15	83	88	239
Water Separability at 82°C					
- Time ≤ 3 mL emulsion	minutes	10	27	15	25
- Time 37 mL water	minutes	11	26	15	25
- Time to complete break	minutes	8	30	16	25
- Volume Oil phase	mL	9	41	3	(3)
- Volume Water phase	mL	9	39	2	(5)
- Volume Emulsion phase	mL	9	0	1	(3)
Level of Contamination					
- ≥ 4µm (c)	counts/mL	3	81035	75381	91570
- ≥ 6µm (c)	counts/mL	3	17277	10041	13131
- ≥14µm (c)	counts/mL	3	347	338	469
- ≥ 4µm (c)	scale no.	4	23.0	2.3	1.7
- ≥ 6µm (c)	scale no.	5	20.2	3.7	1.2
- ≥14µm (c)	scale no.	5	15.8	2.3	2
Calcium as Ca	mg/kg	16	12.0	4.7	(0.4)
Phosphorus as P	mg/kg	13	284	35	72
Zinc as Zn	mg/kg	15	5.9	2.7	(0.6)

Table 3: reproducibilities of tests on sample #20030

NB. Results between brackets no z-scores are calculated

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF MARCH 2020 WITH PREVIOUS PTS

	March 2020	April 2019	April 2018	April 2017	April 2016
Number of reporting laboratories	21	23	18	14	18
Number of test results	384	400	350	177	215
Number of statistical outliers	32	14	14	8	14
Percentage of statistical outliers	8.3%	3.5%	4.0%	4.5%	6.5%

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 against the requirements of the respective reference test methods. The conclusions are given in the following table.

Parameter	March 2020	April 2019	April 2018	April 2017	April 2016
Total Acid Number	+	+	+	-	+
Density at 15°C	-	+	+/-	+/-	+/-
Flash Point C.O.C.	--	--	+	-	n.e.
Flash Point PMcc	++	+	++	+	-
Foaming Tendency/Stability	--	--	-	++	++
Kinematic Viscosity at 40°C	+	+/-	++	+	+/-
Kinematic Viscosity at 100°C	+/-	+	++	++	++
Viscosity Index	+/-	+	+	+	+
Pour Point, Manual	+/-	+/-	-	-	+/-
Pour Point, Automated	--	(--)	+/-	++	+/-
Sulfur	+/-	-	--	-	+/-
Water	++	++	++	++	n.e.
Water Separability at 82°C	++	++	-	+	++
Level of Contamination	+/-	--	--	n.e.	n.e.
Calcium as Ca	(--)	(--)	n.e.	n.e.	n.e.
Phosphorus as P	++	+	++	++	+
Zinc as Zn	(--)	(--)	n.e.	n.e.	n.e.

Table 5: comparison determinations against the reference test method

NB. Results between brackets no z-scores are calculated

The following performance categories were used:

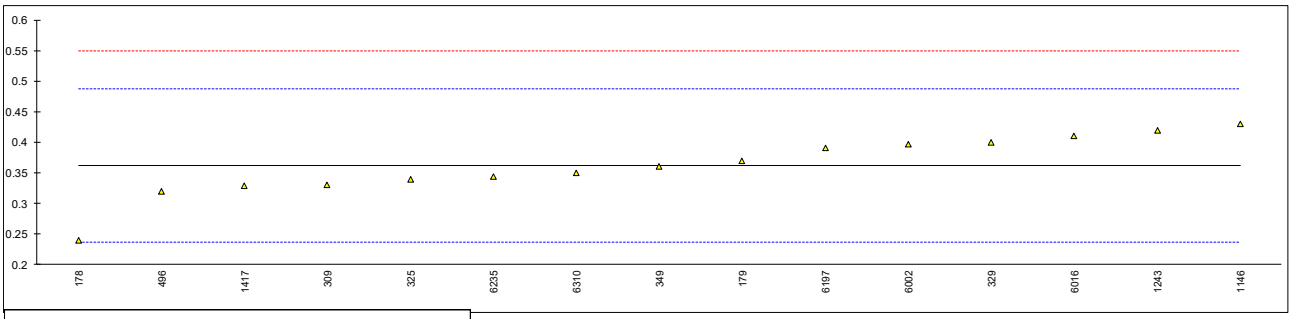
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance similar to the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference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

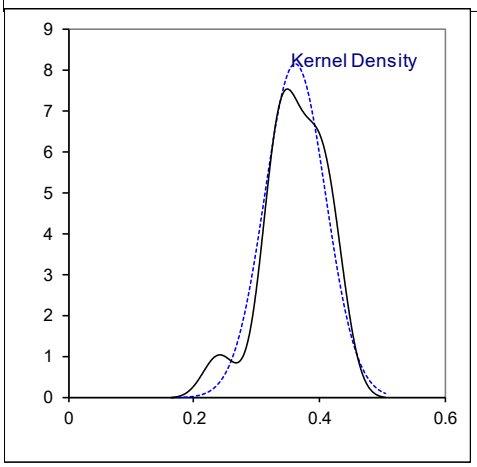
APPENDIX 1

Determination of Total Acid Number on sample #20030; results in mg KOH/g

lab	method	value	mark	z(targ)	End Point Det.	Tit.Solv.Vol.	remarks
178	D664-A	0.24		-1.95	Buffer End Point pH 11	60 mL	
179	D664	0.37		0.13	Inflection Point	125 mL	
237		-----		-----	---	---	
257		-----		-----	---	---	
309	D664-A	0.33		-0.51	Buffer End Point pH 10	60 mL	
325	D664-A	0.34		-0.35	Buffer End Point pH 10	125 mL	
329	D664-A	0.40		0.61	Inflection Point	60 mL	
349	D664-A	0.36		-0.03	Buffer End Point pH 10	125 mL	
432		-----		-----	---	---	
496	D664-A	0.32		-0.67	Buffer End Point pH 10	60 mL	
633		-----		-----	---	---	
862		-----		-----	---	---	
962		-----		-----	---	---	
963		-----		-----	---	---	
974		-----		-----	---	---	
1011		-----		-----	---	---	
1146	D664-A	0.430		1.09	Buffer End Point pH 11	125 mL	
1243	ISO6618	0.42		0.93	Inflection Point	60 mL	
1417	IP177	0.329		-0.53	Inflection Point	125 mL	
1748		-----		-----	---	---	
1799		-----		-----	---	---	
6002	D664-A	0.396		0.55	Buffer End Point pH 10	60 mL	
6016	D664-A	0.410		0.77	---	---	
6197	D664-A	0.39		0.45	Inflection Point	60 mL	
6235	D664-A	0.344		-0.29	Inflection Point	60 mL	
6253		-----		-----	---	---	
6310	D664-A	0.35		-0.19	Buffer End Point pH 10	60 mL	

	normality	suspect
	n	15
	outliers	0
	mean (n)	0.3619
	st.dev. (n)	0.04889
	R(calc.)	0.1369
	st.dev.(D664-A:18e2, IP 60mL)	0.06243
	R(D664-A:18e2, IP 60mL)	0.1748
Compare		
	R(D664-A:18e2, IP 125mL	0.0758
	R(D664-A:18e2, BEP 60mL	0.2056
	R(D664-A:18e2, BEP 125mL	0.1086





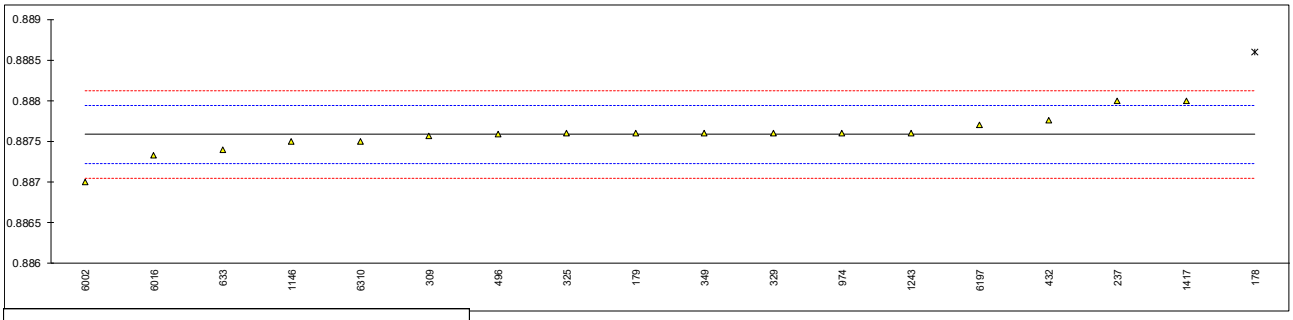
Determination of Copper Corrosion 3 hours at 100°C on sample #20030;

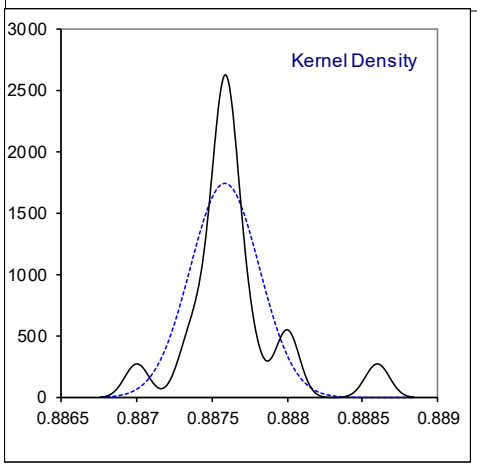
lab	method	value	mark	z(targ)	remarks
178	D130	1a		-----	
179		-----		-----	
237	D130	1A		-----	
257		-----		-----	
309	D130	CUCOR1A		-----	
325	D130	1A		-----	
329		-----		-----	
349	D130	1A		-----	
432		-----		-----	
496	D130	1a		-----	
633	D130	1a		-----	
862		-----		-----	
962		-----		-----	
963		-----		-----	
974	D130	1a		-----	
1011		-----		-----	
1146		-----		-----	
1243	ISO2160	1a		-----	
1417	IP154	1A		-----	
1748		-----		-----	
1799	D130	class 1		-----	
6002	ISO2160	1a		-----	
6016		-----		-----	
6197	D130	1b		-----	
6235		-----		-----	
6253		-----		-----	
6310		-----		-----	
	n	13			
	mean (n)	1 (1a/1b)			

Determination of Density at 15°C on sample #20030; results in kg/L

lab	method	value	mark	z(targ)	remarks
178	D4052	0.8886	G(0.05)	5.68	
179	D4052	0.8876		0.08	
237	D4052	0.8880		2.32	
257		-----		-----	
309	D4052	0.88757		-0.09	
325	D4052	0.8876		0.08	
329	D4052	0.8876		0.08	
349	D4052	0.8876		0.08	
432	D4052	0.88776		0.98	
496	ISO12185	0.88759		0.03	
633	D4052	0.8874		-1.04	
862		-----		-----	
962		-----		-----	
963		-----		-----	
974	D4052	0.8876		0.08	
1011		-----		-----	
1146	D4052	0.8875		-0.48	
1243	ISO12185	0.8876		0.08	
1417	IP365	0.8880	C	2.32	First reported 0.8870
1748		-----		-----	
1799		-----		-----	
6002	ISO12185	0.8870	C	-3.28	First reported 0.8866
6016	D4052	0.88733		-1.43	
6197	D4052	0.8877		0.64	
6235		-----		-----	
6253		-----		-----	
6310	D4052	0.8875		-0.48	

normality	not OK
n	17
outliers	1
mean (n)	0.887585
st.dev. (n)	0.0002299
R(calc.)	0.000644
st.dev.(ISO12185:96)	0.0001786
R(ISO12185:96)	0.0005

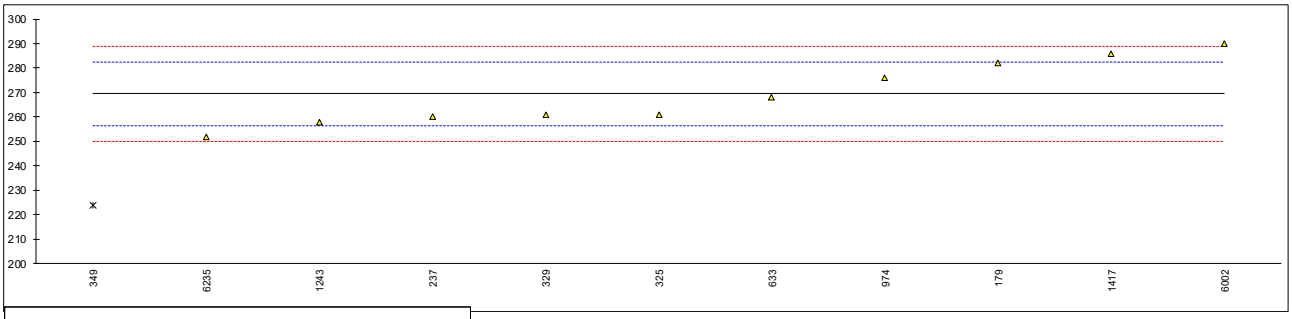


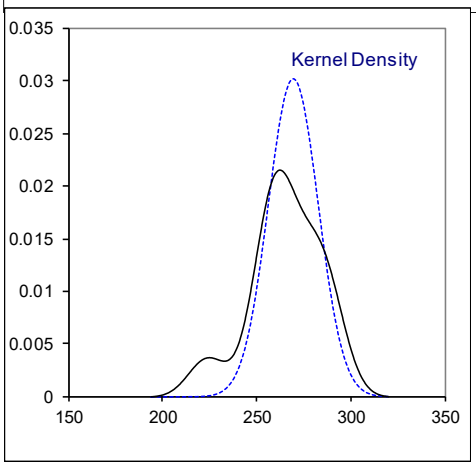


Determination of Flash Point C.O.C. on sample #20030; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D92	282		1.96	
237	D92	260		-1.46	
257		-----		-----	
309		-----		-----	
325	D92	261		-1.31	
329	D92	261		-1.31	
349	D92	224	G(0.05)	-7.06	
432		-----		-----	
496		-----		-----	
633	D92	268.0		-0.22	
862		-----		-----	
962		-----		-----	
963		-----		-----	
974	D92	276		1.03	
1011		-----		-----	
1146		-----		-----	
1243	ISO2592	258		-1.77	
1417	D92	286		2.58	
1748		-----		-----	
1799		-----		-----	
6002	ISO2592	290.0		3.20	
6016		-----		-----	
6197		-----		-----	
6235	ISO2592	252		-2.71	
6253		-----		-----	
6310		-----		-----	

normality	OK
n	10
outliers	1
mean (n)	269.40
st.dev. (n)	13.193
R(calc.)	36.94
st.dev.(D92:18)	6.429
R(D92:18)	18

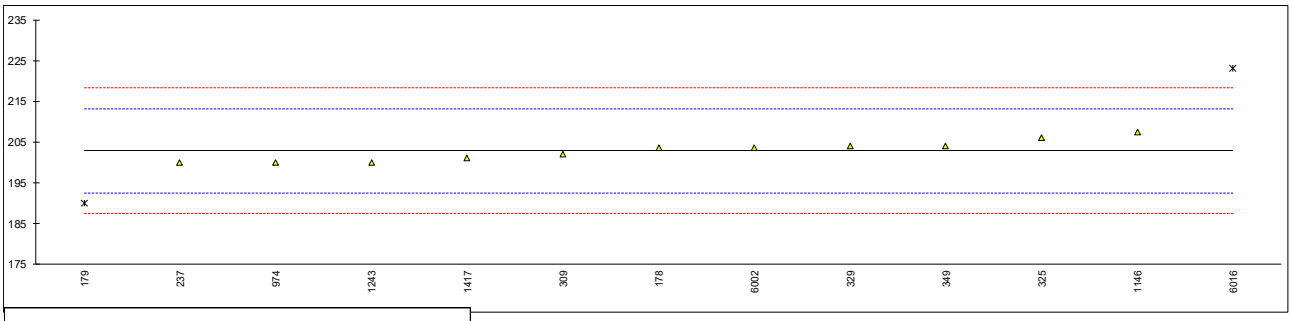


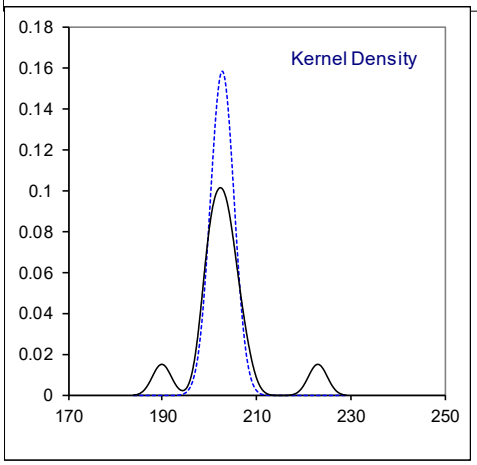


Determination of Flash Point PMcc on sample #20030; results in °C

lab	method	value	mark	z(targ)	remarks
178	D93-A	203.5		0.12	
179	D93-A	190.0	C,G(0.01)	-2.50	First reported 187.0
237	D93-A	200		-0.56	
257		-----		-----	
309	D93-A	202.0		-0.17	
325	D93-A	206.0		0.61	
329	D93-A	204.0		0.22	
349	D93-A	204		0.22	
432		-----		-----	
496		-----		-----	
633		-----		-----	
862		-----		-----	
962		-----		-----	
963		-----		-----	
974	D93-A	200		-0.56	
1011		-----		-----	
1146	D93-A	207.5		0.90	
1243	ISO2719-A	200		-0.56	
1417	D93-A	201		-0.36	
1748		-----		-----	
1799		-----		-----	
6002	ISO2719-A	203.5		0.12	
6016	D6450	223.2	G(0.01)	3.95	
6197		-----		-----	
6235		-----		-----	
6253		-----		-----	
6310		-----		-----	

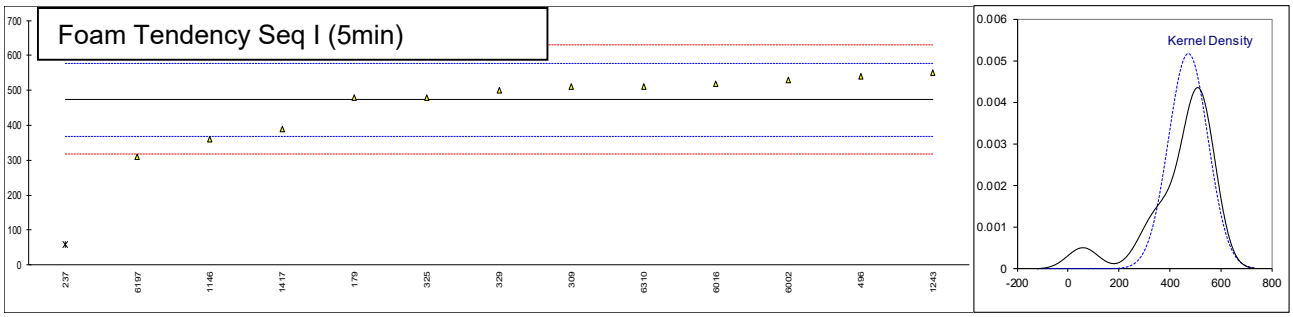
normality	OK
n	11
outliers	2
mean (n)	202.86
st.dev. (n)	2.521
R(calc.)	7.06
st.dev.(D93-A:19)	5.144
R(D93-A:19)	14.40

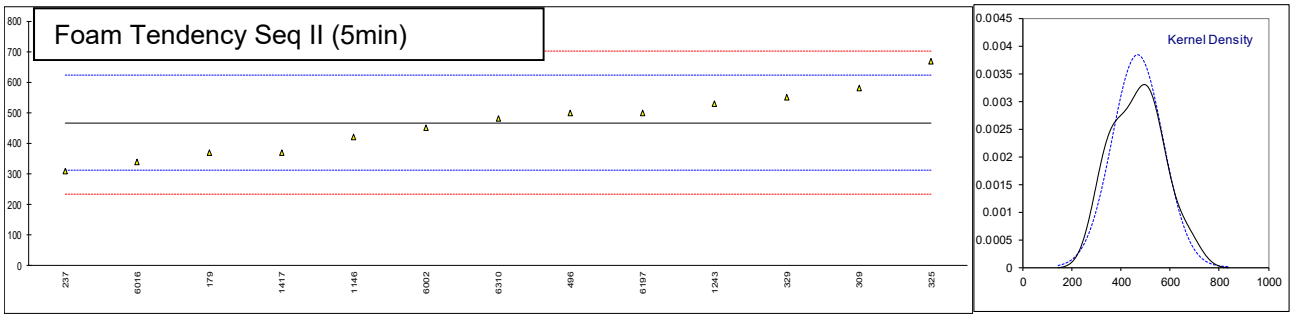


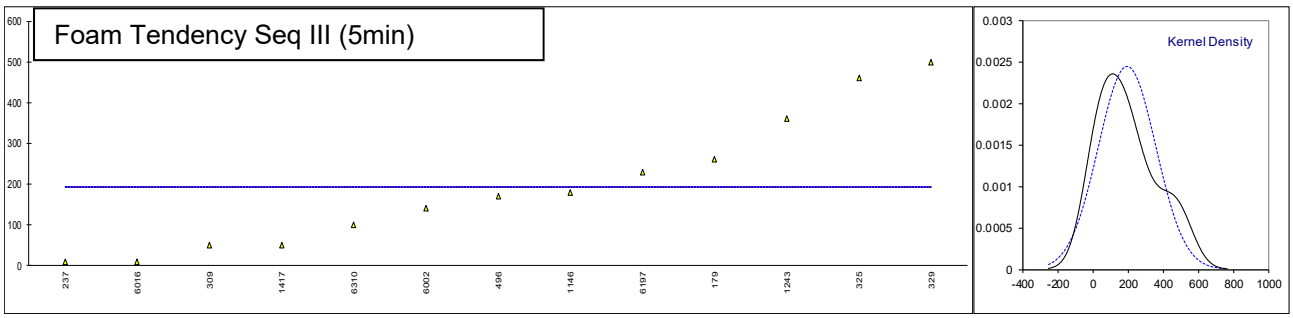


Determination of Foaming Tendency, Sequence I, II and III (5 min. blowing period) on sample #20030; results in mL

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	480		0.13	370		-1.25	260		-----
237	D892	As received	Metal	60	G(0.01)	-7.92	310		-2.02	10		-----
257		---	---	-----		-----	-----		-----	-----		-----
309	D892	After agit (A)	Metal	510		0.70	580		1.45	50		-----
325	D892	As received	Metal	480		0.13	670		2.61	460		-----
329	D892	After agit (A)	Stone	500		0.51	550		1.07	500		-----
349		---	---	-----		-----	-----		-----	-----		-----
432		---	---	-----		-----	-----		-----	-----		-----
496	D892	After agit (A)	Metal	540		1.28	500		0.43	170		-----
633		---	---	-----		-----	-----		-----	-----		-----
862		---	---	-----		-----	-----		-----	-----		-----
962		---	---	-----		-----	-----		-----	-----		-----
963		---	---	-----		-----	-----		-----	-----		-----
974		---	---	-----		-----	-----		-----	-----		-----
1011		---	---	-----		-----	-----		-----	-----		-----
1146	ISO6247	As received	Metal	360		-2.17	420		-0.60	180		-----
1243	D892	As received	Stone	550		1.47	530		0.81	360		-----
1417	D892	As received	Metal	390		-1.60	370		-1.25	50		-----
1748		---	---	-----		-----	-----		-----	-----		-----
1799		---	---	-----		-----	-----		-----	-----		-----
6002	ISO6247	As received	Metal	530		1.09	450		-0.22	140		-----
6016	D892	---	---	520		0.89	340		-1.63	10		-----
6197	D892	After agit (A)	Metal	310		-3.13	500		0.43	230		-----
6235		---	---	-----		-----	-----		-----	-----		-----
6253		---	---	-----		-----	-----		-----	-----		-----
6310	D892	After agit (A)	Metal	510		0.70	480		0.17	100		-----
	normality			OK			OK			OK		
	n			12			13			13		
	outliers			1			0			0		
	mean (n)			473.3			466.9			193.8		
	st.dev. (n)			77.26			103.79			163.12		
	R(calc.)			216.3			290.6			456.7		
	st.dev.(D892:18)			52.20			77.79			(35.98)		
	R(D892:18)			146.2			217.8			(100.7)		


Foam Tendency Seq I (5min)
Kernel Density

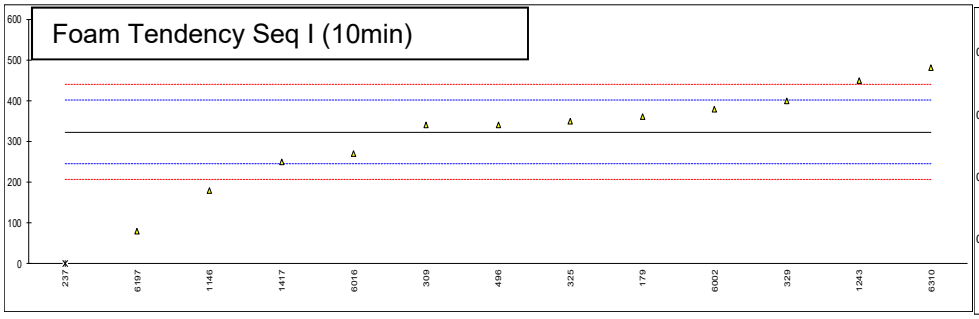

Foam Tendency Seq II (5min)
Kernel Density

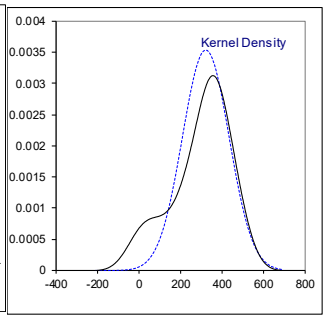

Foam Tendency Seq III (5min)
Kernel Density

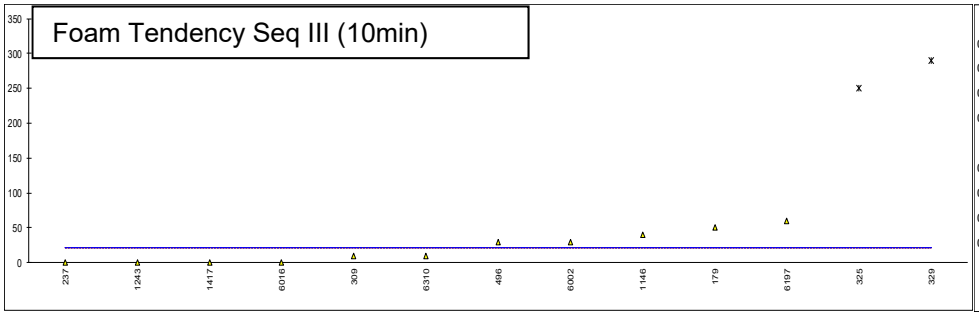
Determination of Foaming Stability, Sequence I, II and III (10 min. settling period) on sample #20030; results in mL

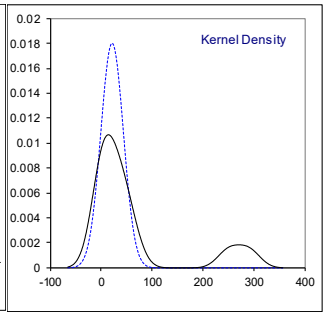
lab	method	Seq I	mark	z(targ)	Seq II	mark	z(targ)	Seq III	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179	D892	360		0.95	0		-----	50		-----
237	D892	0	G(0.05)	-8.37	0		-----	0		-----
257		-----		-----	-----		-----	-----		-----
309	D892	340		0.43	0		-----	10		-----
325	D892	350		0.69	0		-----	250	DG(0.01)	-----
329	D892	400		1.99	0		-----	290	C,DG(0.01)	-----
349		-----		-----	-----		-----	-----		-----
432		-----		-----	-----		-----	-----		-----
496	D892	340		0.43	0		-----	30		-----
633		-----		-----	-----		-----	-----		-----
862		-----		-----	-----		-----	-----		-----
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
974		-----		-----	-----		-----	-----		-----
1011		-----		-----	-----		-----	-----		-----
1146	ISO6247	180		-3.71	0		-----	40		-----
1243	D892	450		3.28	0		-----	0	C	-----
1417	D892	250		-1.90	0		-----	0		-----
1748		-----		-----	-----		-----	-----		-----
1799		-----		-----	-----		-----	-----		-----
6002	ISO6247	380		1.47	0		-----	30		-----
6016	D892	270		-1.38	0		-----	0		-----
6197	D892	80		-6.30	0		-----	60		-----
6235		-----		-----	-----		-----	-----		-----
6253		-----		-----	-----		-----	-----		-----
6310	D892	480		4.06	0		-----	10		-----
	normality	OK			unknown			OK		
	n	12			13			11		
	outliers	1			0			2		
	mean (n)	323.3			0			20.9		
	st.dev. (n)	112.92			0			22.12		
	R(calc.)	316.2			0			61.9		
	st.dev.(D892:18)	38.61			(0)			(7.79)		
	R(D892:18)	108.1			(0)			(21.8)		

Lab 329: first reported 260
Lab 1243: first reported 140





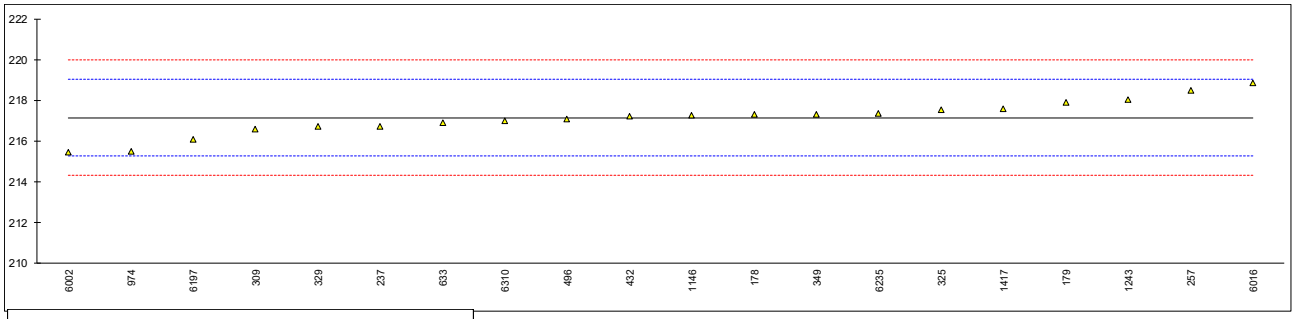


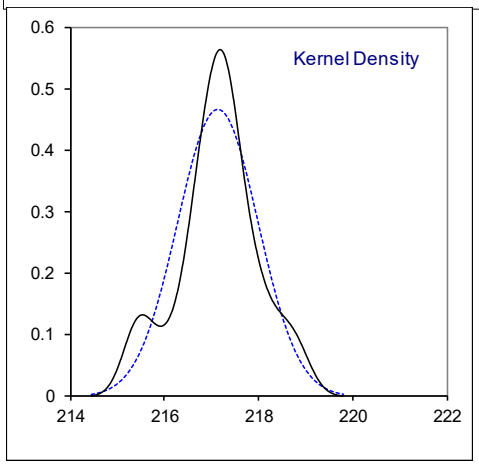


Determination of Kinematic Viscosity at 40°C on sample #20030; results in mm²/s

lab	method	value	mark	z(targ)	remarks
178	D445	217.3		0.17	
179	D445	217.9		0.80	
237	D445	216.7		-0.47	
257	D7279 corrected to D445	218.47		1.41	
309	D445	216.6		-0.57	
325	D445	217.55		0.43	
329	D445	216.7		-0.47	
349	D445	217.3		0.17	
432	D445	217.2		0.06	
496	D445	217.09		-0.05	
633	D445	216.9138		-0.24	
862		-----		-----	
962		-----		-----	
963		-----		-----	
974	D445	215.5		-1.73	
1011		-----		-----	
1146	D445	217.24		0.11	
1243	D7279 corrected to D445	218.04		0.95	
1417	D445	217.6		0.49	
1748		-----		-----	
1799		-----		-----	
6002	ISO3104	215.43		-1.81	
6016	D7042	218.83		1.79	
6197	D445	216.1		-1.10	
6235	ISO3104	217.34		0.21	
6253		-----		-----	
6310	D7279 corrected to D445	217		-0.15	

normality	OK
n	20
outliers	0
mean (n)	217.140
st.dev. (n)	0.8567
R(calc.)	2.399
st.dev.(D445:19a)	0.9461
R(D445:19a)	2.649

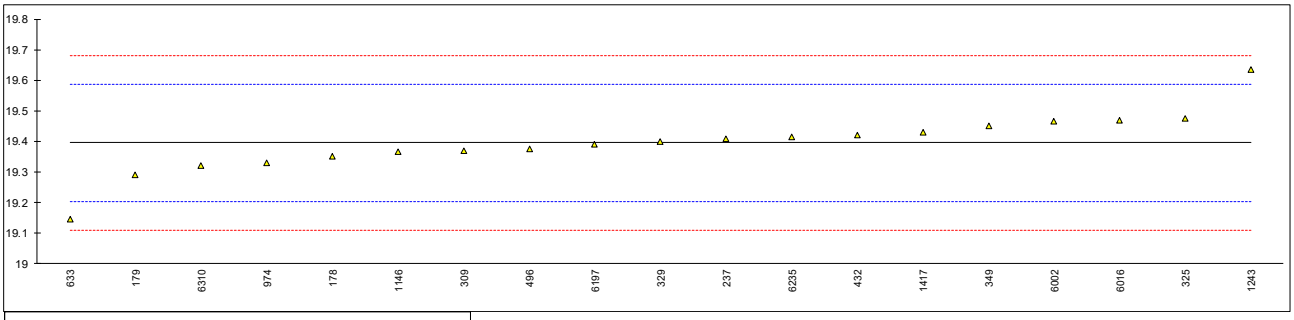


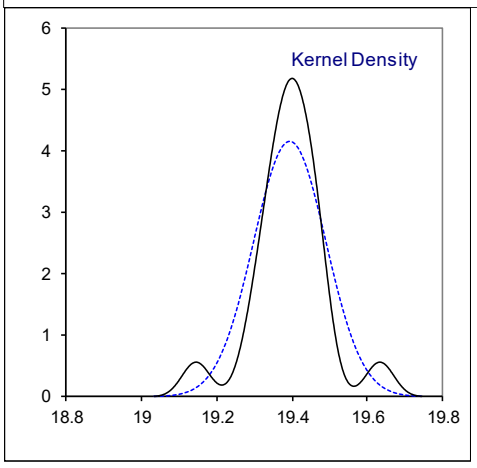


Determination of Kinematic Viscosity at 100°C on sample #20030; results in mm²/s

lab	method	value	mark	z(targ)	remarks
178	D445	19.35		-0.47	
179	D445	19.29		-1.10	
237	D445	19.41		0.16	
257		-----		-----	
309	D445	19.37		-0.26	
325	D445	19.475		0.84	
329	D445	19.40		0.05	
349	D445	19.45		0.57	
432	D445	19.42		0.26	
496	D445	19.374		-0.22	
633	D445	19.1457		-2.61	
862		-----		-----	
962		-----		-----	
963		-----		-----	
974	D445	19.33		-0.68	
1011		-----		-----	
1146	D445	19.365		-0.31	
1243	D7279 corrected to D445	19.635		2.51	
1417	D445	19.43		0.36	
1748		-----		-----	
1799		-----		-----	
6002	ISO3104	19.466		0.74	
6016	D7042	19.47		0.78	
6197	D445	19.39		-0.05	
6235	ISO3104	19.416		0.22	
6253		-----		-----	
6310	D7279 corrected to D445	19.32		-0.79	

normality	not OK
n	19
outliers	0
mean (n)	19.395
st.dev. (n)	0.0964
R(calc.)	0.270
st.dev.(D445:19a)	0.0956
R(D445:19a)	0.268

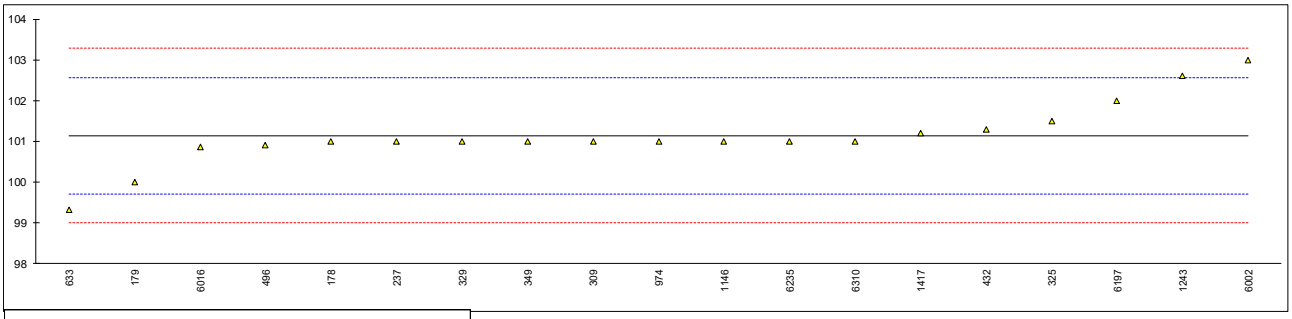


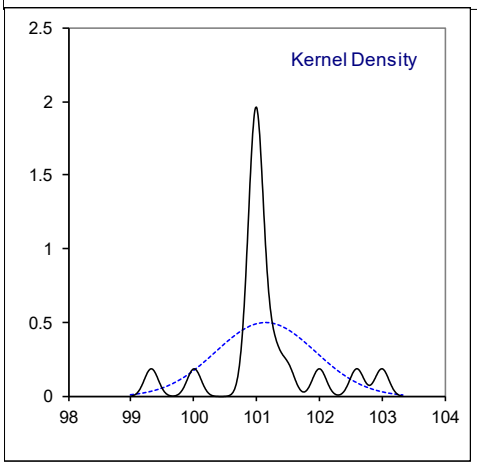


Determination of Viscosity Index on sample #20030

lab	method	value	mark	z(targ)	remarks
178	D2270	101		-0.20	
179	D2270	100		-1.60	
237	D2270	101		-0.20	
257		-----		-----	
309	D2270	101		-0.20	
325	D2270	101.5		0.50	
329	D2270	101		-0.20	
349	D2270	101		-0.20	
432	D2270	101.3		0.22	
496	D2270	100.9		-0.34	
633	D2270	99.315		-2.56	
862		-----		-----	
962		-----		-----	
963		-----		-----	
974	D2270	101		-0.20	
1011		-----		-----	
1146	D2270	101		-0.20	
1243	ISO2909	102.6		2.04	
1417	D2270	101.2		0.08	
1748		-----		-----	
1799		-----		-----	
6002	ISO2909	103		2.60	
6016	D2270	100.87		-0.38	
6197	D2270	102		1.20	
6235	ISO2909	101		-0.20	
6253		-----		-----	
6310	D2270	101		-0.20	

normality	not OK
n	19
outliers	0
mean (n)	101.14
st.dev. (n)	0.797
R(calc.)	2.23
st.dev.(D2270:10)	0.714
R(D2270:10)	2

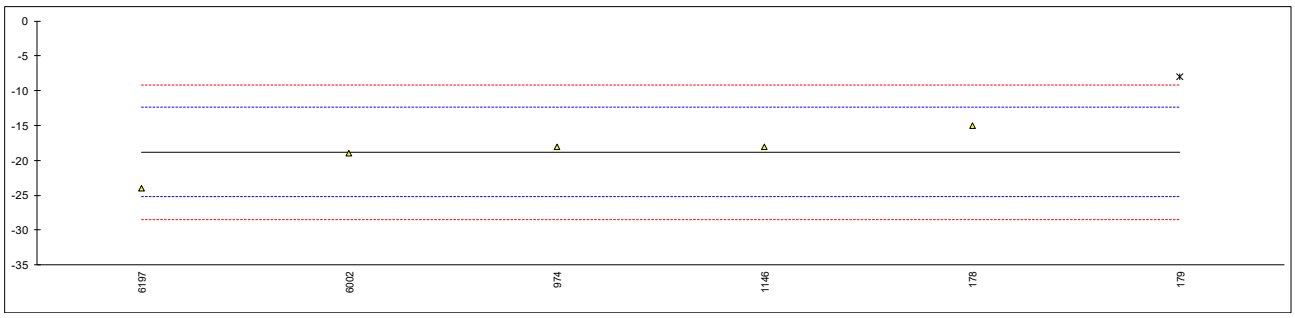




Determination of Pour Point, Manual on sample #20030; results in °C

lab	method	value	mark	z(targ)	remarks
178	D97	-15		1.18	
179	D97	-8	D(0.05)	3.36	
237		-----		-----	
257		-----		-----	
309		-----		-----	
325		-----		-----	
329		-----		-----	
349		-----		-----	
432		-----		-----	
496		-----		-----	
633		-----		-----	
862		-----		-----	
962		-----		-----	
963		-----		-----	
974	D97	-18		0.25	
1011		-----		-----	
1146	D97	-18		0.25	
1243		-----		-----	
1417		-----		-----	
1748		-----		-----	
1799		-----		-----	
6002	ISO3016	-19		-0.06	
6016		-----		-----	
6197	D97	-24		-1.62	
6235		-----		-----	
6253		-----		-----	
6310		-----		-----	

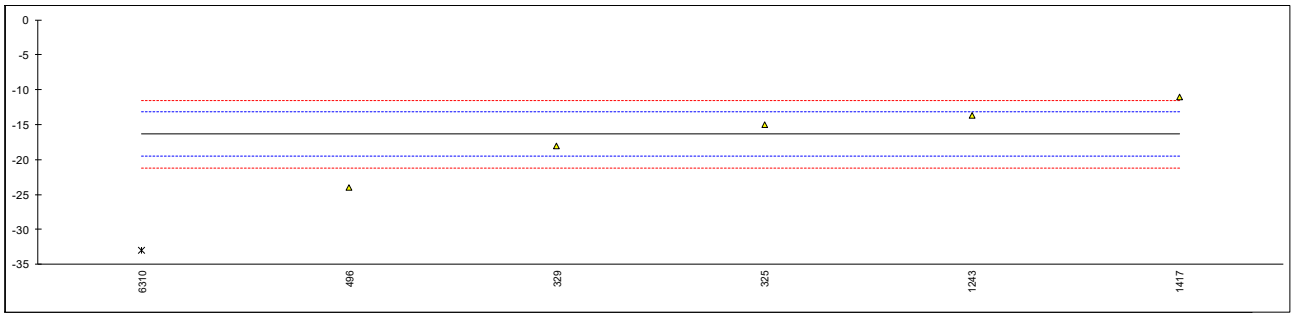
normality	unknown
n	5
outliers	1
mean (n)	-18.80
st.dev. (n)	3.271
R(calc.)	9.16
st.dev.(D97:17b)	3.214
R(D97:17b)	9



Determination of Pour Point, Automated, 1°C interval on sample #20030; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
257		-----		-----	
309		-----		-----	
325	D5950	-15		0.83	
329	D5950	-18		-1.03	
349		-----		-----	
432		-----		-----	
496	D5950	-24		-4.77	
633		-----		-----	
862		-----		-----	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1011		-----		-----	
1146		-----		-----	
1243	D7346	-13.7		1.64	
1417	D5950	-11		3.32	
1748		-----		-----	
1799		-----		-----	
6002		-----		-----	
6016		-----		-----	
6197		-----		-----	
6235		-----		-----	
6253		-----		-----	
6310	D5950	-33	G(0.05)	-10.37	

normality	unknown
n	5
outliers	1
mean (n)	-16.34
st.dev. (n)	4.968
R(calc.)	13.91
st.dev.(D5950:14)	1.607
R(D5950:14)	4.5



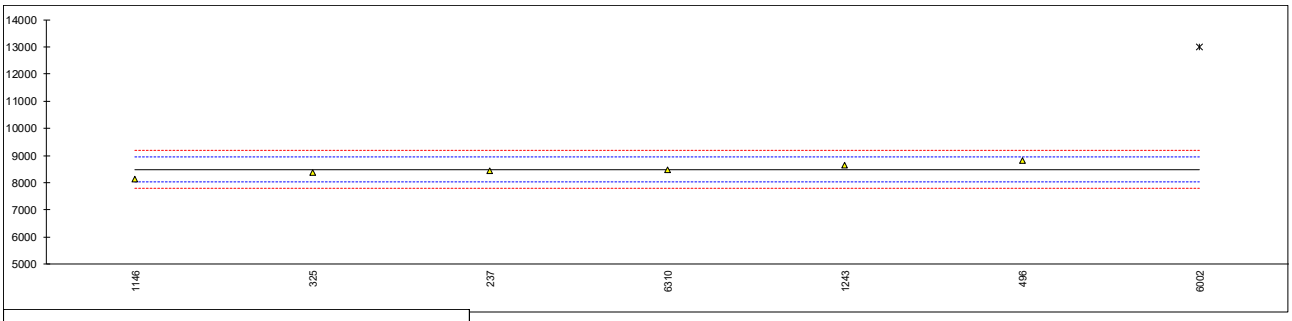
Determination of Rust Prevention, distilled water on sample #20030

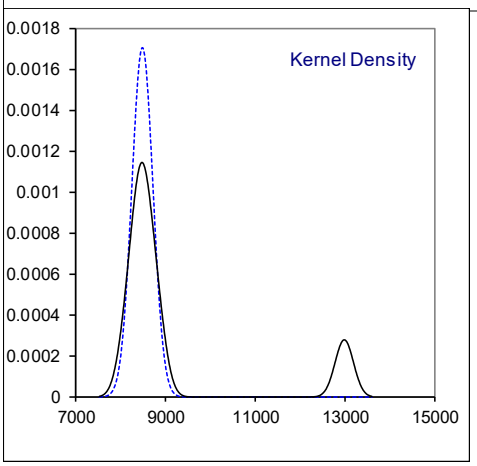
lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D665	PASS		-----	
257		-----		-----	
309		-----		-----	
325	D665	PASS		-----	
329		-----		-----	
349		-----		-----	
432		-----		-----	
496		-----		-----	
633		-----		-----	
862		-----		-----	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1011		-----		-----	
1146		-----		-----	
1243		-----		-----	
1417	D665	PASS		-----	
1748		-----		-----	
1799	D665	pass		-----	
6002	ISO7120	pass		-----	
6016	D665	Pass		-----	
6197		-----		-----	
6235		-----		-----	
6253		-----		-----	
6310		-----		-----	
	n	6			
	mean (n)	Pass			

Determination of Sulfur on sample #20030; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D4294	8440		-0.20	
257		-----		-----	
309		-----		-----	
325	INH-6443	8380		-0.45	
329		-----		-----	
349		-----		-----	
432		-----		-----	
496	D4294	8830		1.47	
633		-----		-----	
862		-----		-----	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1011		-----		-----	
1146	D4294	8140		-1.48	
1243	ISO8754	8635		0.64	
1417		-----		-----	
1748		-----		-----	
1799		-----		-----	
6002	D5185	12985	C,G(0.01)	19.27	First reported 10106
6016		-----		-----	
6197		-----		-----	
6235		-----		-----	
6253		-----		-----	
6310	D7751	8490	C	0.02	First reported 849

normality	unknown
n	6
outliers	1
mean (n)	8485.8
st.dev. (n)	233.93
R(calc.)	655.0
st.dev.(D4294:16e1)	233.44
R(D4294:16e1)	653.6

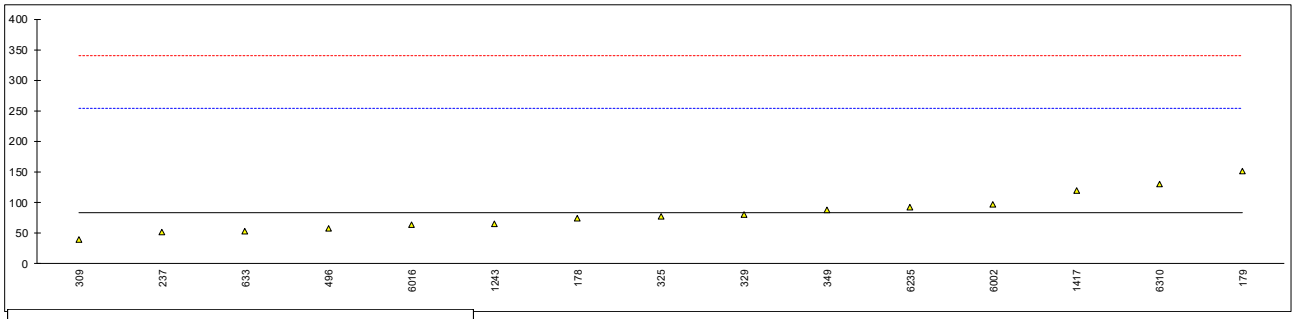


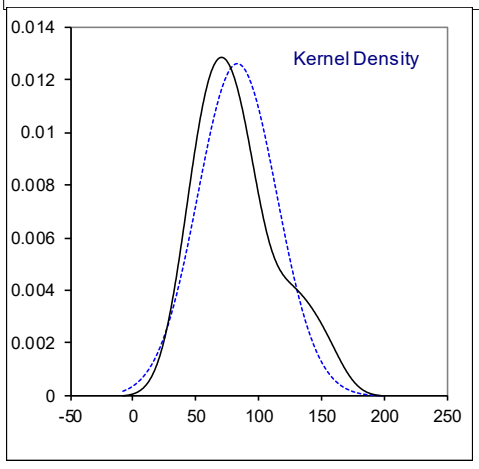


Determination of Water on sample #20030; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D6304-C	74		-0.11	
179	D6304	152		0.81	
237	D6304-C	52.4		-0.36	
257		-----		-----	
309	D6304-C	39		-0.51	
325	D6304-C	78		-0.06	
329	D6304-A	80		-0.04	
349	D6304-C	88		0.06	
432		-----		-----	
496	D6304-C	57		-0.30	
633	D6304-C	53.3		-0.35	
862		-----		-----	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1011		-----		-----	
1146	D6304-C	<100		-----	
1243	ISO12937	66		-0.20	
1417	D6304-A	120		0.43	
1748		-----		-----	
1799		-----		-----	
6002	In house	97.41		0.17	
6016	D6304-A	64.4		-0.22	
6197		-----		-----	
6235	In house	93		0.12	
6253		-----		-----	
6310	D6304-C	131	C	0.56	First reported 0.0131 mg/kg

normality	OK
n	15
outliers	0
mean (n)	83.03
st.dev. (n)	31.563
R(calc.)	88.38
st.dev.(D6304:16e1)	85.520
R(D6304:16e1)	239.46

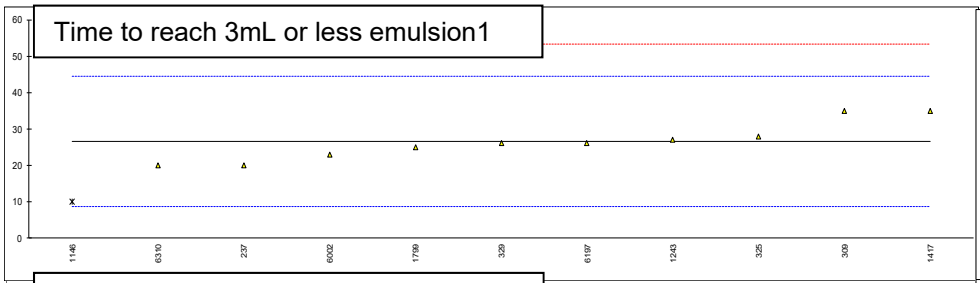


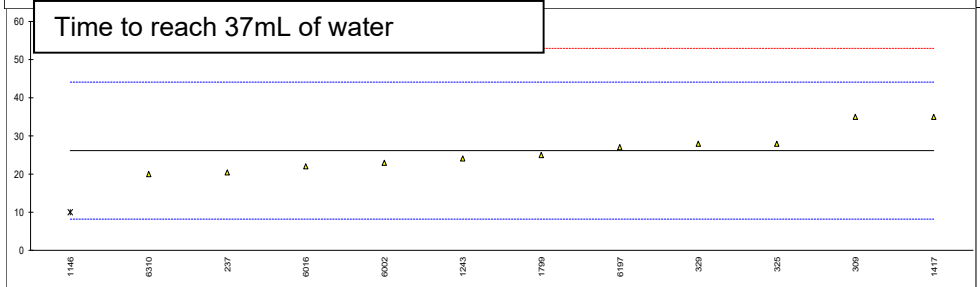


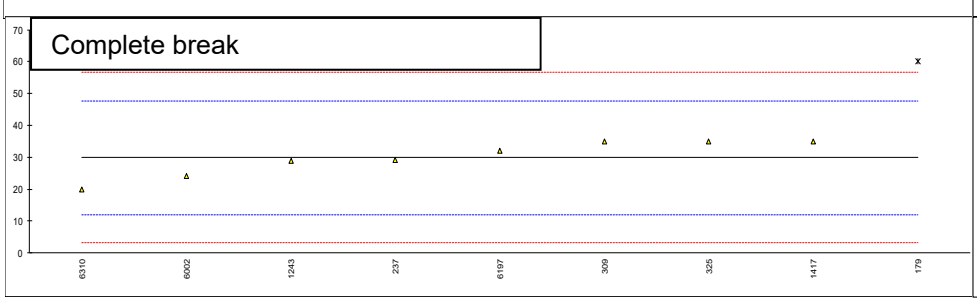
Determination of Water Separability at 82°C, distilled water on sample #20030; results in minutes

lab	method	3mL or less emulsion		z(targ)	37mL of water		z(targ)	complete break (40-40-0)		z(targ)	test aborted
178		-----		-----	-----		-----	-----		-----	-----
179		-----		-----	-----		-----	60	R(0.05)	3.37	NO
237	D1401	20.1		-0.72	20.5		-0.63	29.1		-0.09	NO
257		-----		-----	-----		-----	-----		-----	-----
309	D1401	35		0.95	35		0.99	35		0.57	YES
325	D1401	28		0.17	28		0.21	35		0.57	-----
329	D1401	26		-0.06	28		0.21	>30		-----	YES
349		-----		-----	-----		-----	-----		-----	-----
432		-----		-----	-----		-----	-----		-----	-----
496		-----		-----	-----		-----	-----		-----	YES
633		-----		-----	-----		-----	-----		-----	-----
862		-----		-----	-----		-----	-----		-----	-----
962		-----		-----	-----		-----	-----		-----	-----
963		-----		-----	-----		-----	-----		-----	-----
974		-----		-----	-----		-----	-----		-----	-----
1011		-----		-----	-----		-----	-----		-----	-----
1146	D1401	10	ex	-1.85	10	ex	-1.81	-----		-----	-----
1243	ISO6614	27		0.05	24		-0.24	29		-0.10	NO
1417	D1401	35		0.95	35		0.99	35		0.57	NO
1748		-----		-----	-----		-----	-----		-----	-----
1799		25		-0.17	25		-0.13	no break		-----	NO
6002	ISO6614	23		-0.39	23		-0.35	24		-0.66	NO
6016	D1401	>60	f+?	>3.75	22		-0.46	>60	f+?	>3.37	YES
6197	D1401	26		-0.06	27		0.10	32		0.24	NO
6235		-----		-----	-----		-----	-----		-----	-----
6253		-----		-----	-----		-----	-----		-----	-----
6310	D1401	20		-0.73	20		-0.69	20		-1.11	NO
	normality	OK			OK			OK			
	n	10			11			8			
	outliers	0 (+1ex)			0 (+1ex)			1			
	mean (n)	26.5			26.1			29.9			
	st.dev. (n)	5.22			5.17			5.56			
	R(calc.)	14.6			14.5			15.6			
	st.dev.(D1401:19)	8.93			8.93			8.93			
	R(D1401:19)	25			25			25			

lab 1146 test results excluded as volume of Oil, Water and Emulsion phase are outliers
f+? = possibly a false positive test result?

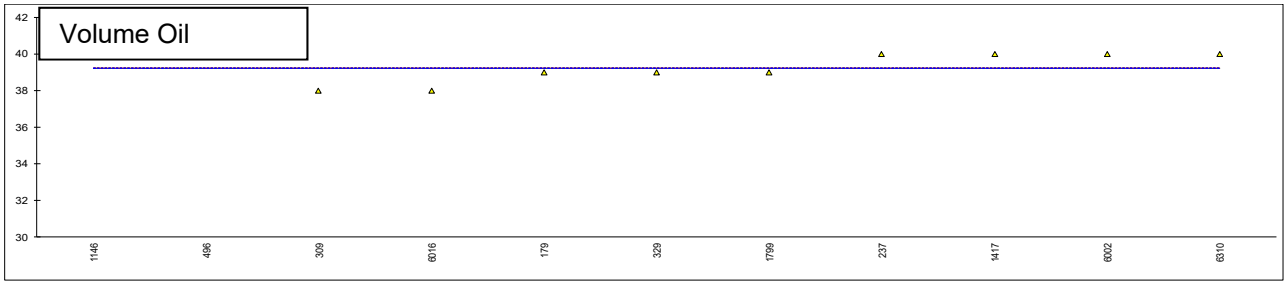


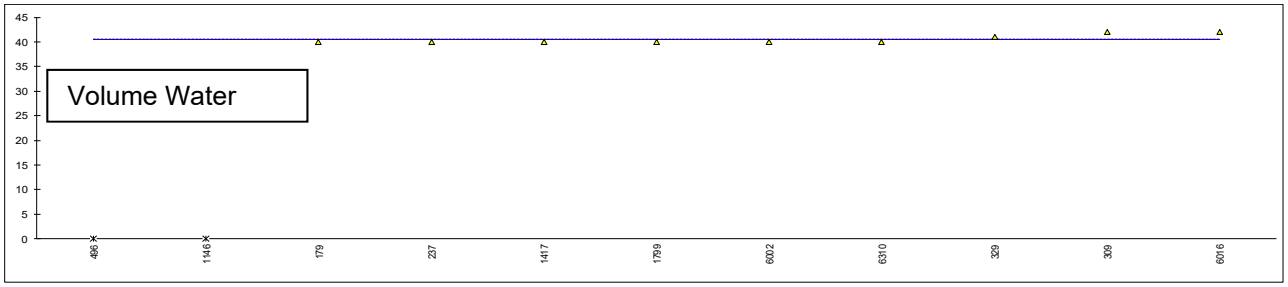


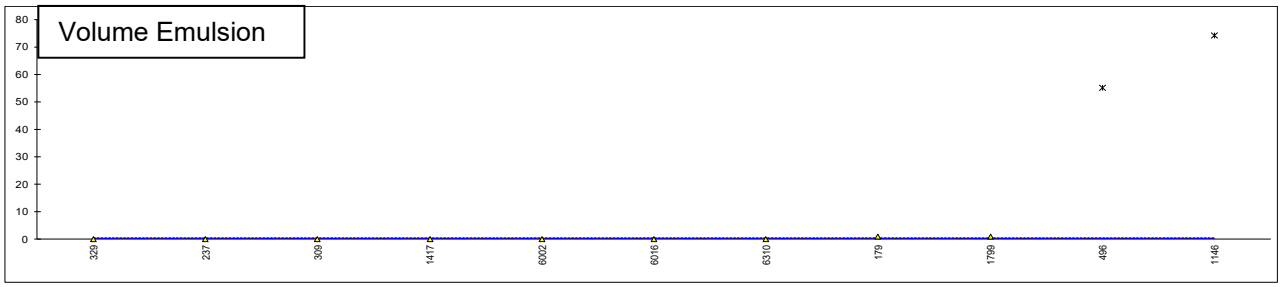


Determination of Water Separability at 82°C, distilled water on sample #20030; results in mL

lab	method	oil phase	mark	water phase	mark	emulsion phase	mark	remarks
178		-----		-----		-----		
179	D1401	40		39		1		
237	D1401	40		40		0		
257		-----		-----		-----		
309	D1401	42		38		0		
325		-----		-----		-----		
329	D1401	41		39		0		
349		-----		-----		-----		
432		-----		-----		-----		
496		0	DG(0.01)	25	G(0.01)	55	G(0.01)	
633		-----		-----		-----		
862		-----		-----		-----		
962		-----		-----		-----		
963		-----		-----		-----		
974		-----		-----		-----		
1011		-----		-----		-----		
1146	D1401	0	DG(0.01)	6	G(0.01)	74	G(0.01)	
1243		-----		-----		-----		
1417	D1401	40		40		0		
1748		-----		-----		-----		
1799		40		39		1		
6002	ISO6614	40		40		0		
6016	D1401	42	C	38		0	C	fr.. 1, 41
6197		-----		-----		-----		
6235		-----		-----		-----		
6253		-----		-----		-----		
6310	D1401	40		40		0		
	normality	suspect		OK		suspect		
	n	9		9		9		
	outliers	2		2		2		
	mean (n)	40.6		39.2		0.2		
	st.dev. (n)	0.88		0.83		0.44		
	R(calc.)	2.5		2.3		1.2		
	Compare R(iis19L01)	3.0		4.5		3.0		



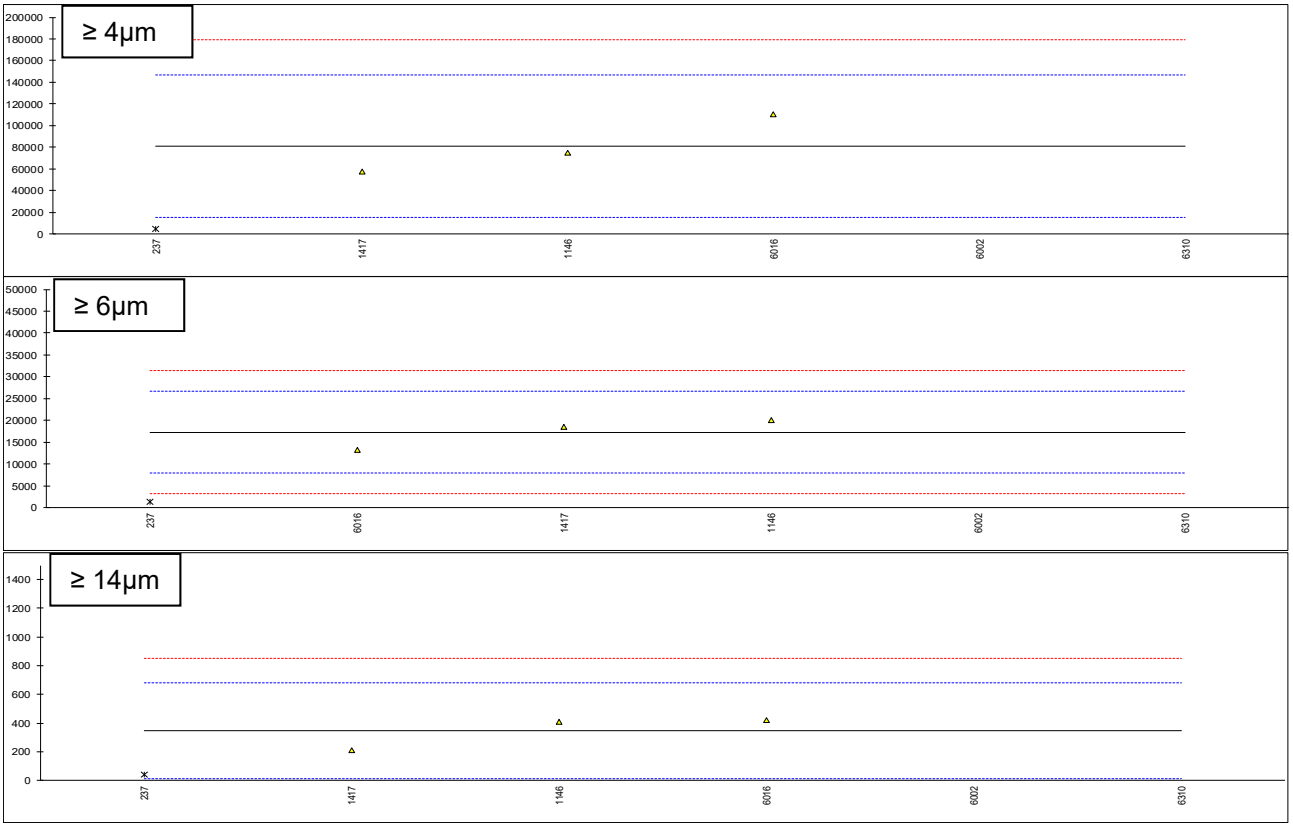




Determination of Level of Contamination on sample #20030; results in counts/mL

lab	method	≥ 4 µm (c)	mark	z(targ)	≥ 6 µm (c)	mark	z(targ)	≥ 14 µm (c)	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179		-----		-----	-----		-----	-----		-----
237	D7647	4343.0	ex	-2.35	1419.3	ex	-3.38	40.4	ex	-1.83
257		-----		-----	-----		-----	-----		-----
309		-----		-----	-----		-----	-----		-----
325		-----		-----	-----		-----	-----		-----
329		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
432		-----		-----	-----		-----	-----		-----
496		-----		-----	-----		-----	-----		-----
633		-----		-----	-----		-----	-----		-----
862		-----		-----	-----		-----	-----		-----
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
974		-----		-----	-----		-----	-----		-----
1011		-----		-----	-----		-----	-----		-----
1146	ISO11500	75000		-0.18	20000		0.58	410		0.38
1243		-----		-----	-----		-----	-----		-----
1417	In house	57644		-0.72	18617		0.29	208		-0.83
1748		-----		-----	-----		-----	-----		-----
1799		-----		-----	-----		-----	-----		-----
6002	D7647	3438062	C,DG(0.01)	102.65	402284	C,G(0.01)	82.10	5009	C, ex	27.87
6016	ISO4406	110463		0.90	13214		-0.87	423		0.45
6197		-----		-----	-----		-----	-----		-----
6235		-----		-----	-----		-----	-----		-----
6253		-----		-----	-----		-----	-----		-----
6310	ISO4407	5288855	DG(0.01)	159.24	1872580	G(0.01)	395.63	129872	C,G(0.01)	774.19
	normality	unknown			unknown			unknown		
	n	3			3			3		
	outliers	2 (+1ex)			2 (+1ex)			1 (+2ex)		
	mean (n)	81035.7			17277.0			347.0		
	st.dev. (n)	26921.80			3585.97			120.55		
	R(calc.)	75381.1			10040.7			337.5		
	st.dev.(D7647:10)	32703.68			4689.47			167.30		
	R(D7647:10)	91570.3			13130.5			468.5		

Lab 237: test results excluded, as test results in ISO4406 scale were statistical outliers
Lab 6002: first reported 4338062, 532673, 6527. Test result excluded as other two test results were statistical outliers
Lab 6310: first reported 1872580



Determination of Level of Contamination acc. to ISO4406 scale on sample #20030; results in scale number

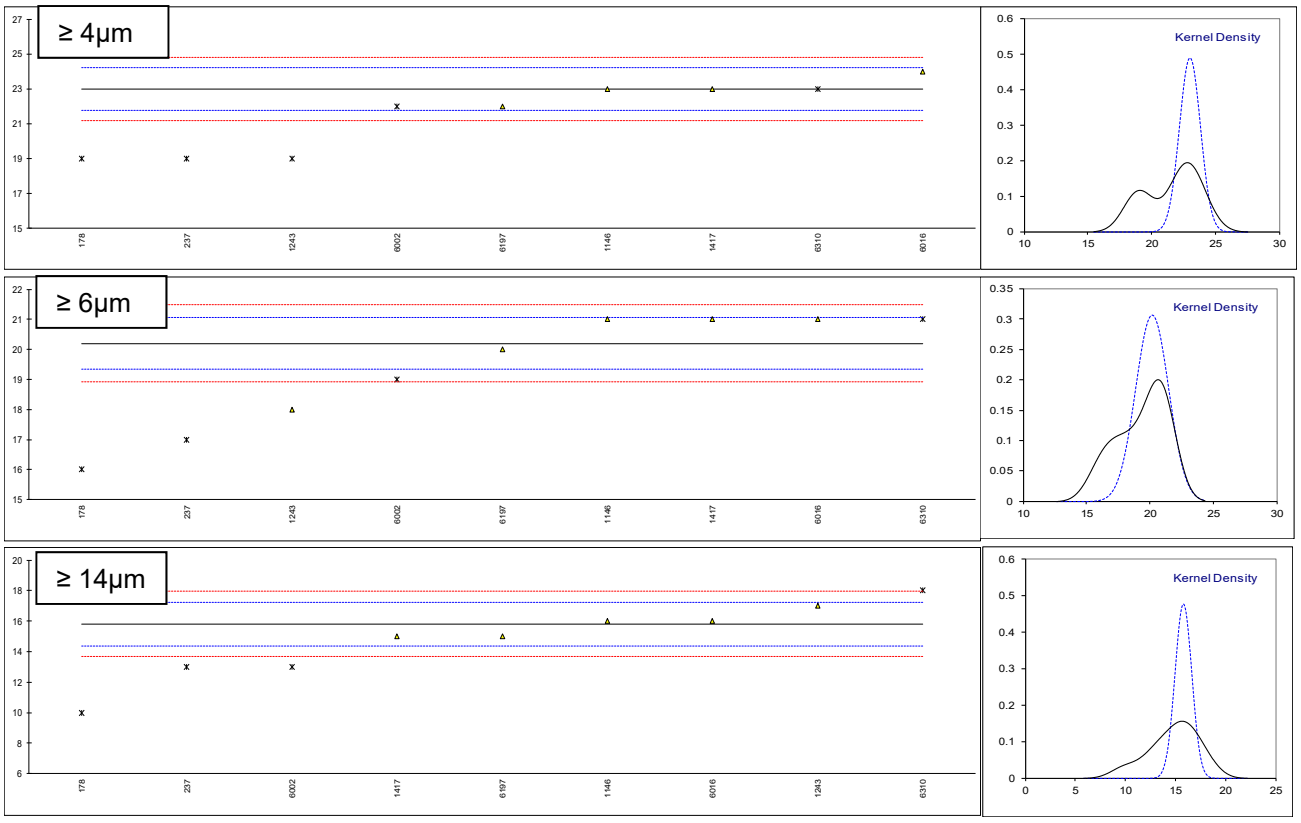
lab	method	≥ 4 µm (c)	mark	z(targ)	≥ 6 µm (c)	mark	z(targ)	≥ 14 µm (c)	mark	z(targ)
178	ISO4406	19	G(0.05)	-6.59	16	G(0.05)	-9.80	10	G(0.05)	-8.12
179		-----		-----	-----		-----	-----		-----
237	ISO4406	19	G(0.05)	-6.59	17	E,G(0.05)	-7.47	13	ex	-3.92
257		-----		-----	-----		-----	-----		-----
309		-----		-----	-----		-----	-----		-----
325		-----		-----	-----		-----	-----		-----
329		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
432		-----		-----	-----		-----	-----		-----
496		-----		-----	-----		-----	-----		-----
633		-----		-----	-----		-----	-----		-----
862		-----		-----	-----		-----	-----		-----
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
974		-----		-----	-----		-----	-----		-----
1011		-----		-----	-----		-----	-----		-----
1146	ISO4406	23		0.00	21		1.87	16		0.28
1243	ISO4406	19	G(0.05)	-6.59	18		-5.13	17		1.68
1417	ISO4406	23		0.00	21		1.87	15		-1.12
1748		-----		-----	-----		-----	-----		-----
1799		-----		-----	-----		-----	-----		-----
6002	D7647	22	C,E,ex	-1.65	19	C,E,ex	-2.80	13	E,ex	-3.92
6016	ISO4406	24		1.65	21		1.87	16		0.28
6197	ISO4406	22		-1.65	20		-0.47	15		-1.12
6235		-----		-----	-----		-----	-----		-----
6253		-----		-----	-----		-----	-----		-----
6310	ISO4406	23	E,ex	0.00	21	E,ex	1.87	18	E,ex	3.08

normality	not OK	not OK	OK
n	4	5	5
outliers	3 (+2ex)	2 (+2ex)	1 (+3ex)
mean (n)	23.0	20.2	15.8
st.dev. (n)	0.82	1.30	0.84
R(calc.)	2.3	3.7	2.3
st.dev.(D7647:10)	0.61	0.43	0.71
R(D7647:10)	1.7	1.2	2

Lab 237: test result was excluded as other two test results were statistical outliers.

Lab 6002: first reported 23, 20. Test results excluded as reported counts/mL do not match the correct ISO scale

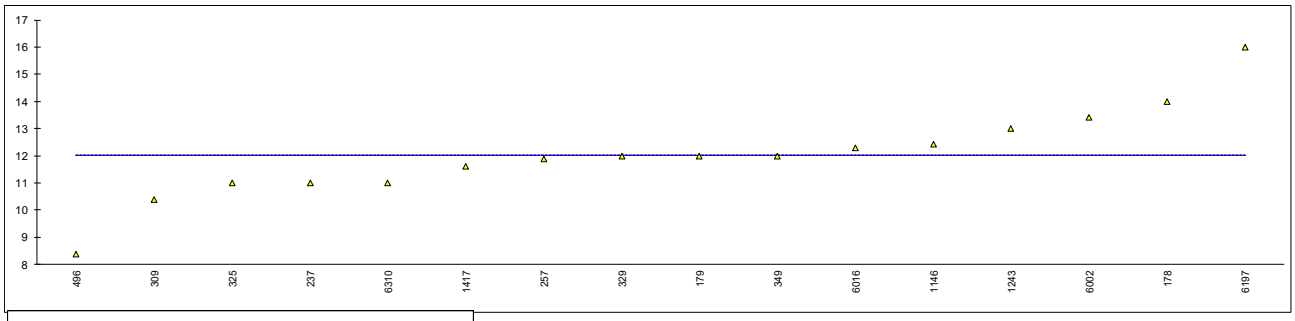
Lab 6310: Test results excluded as reported counts/mL do not match the correct ISO scale

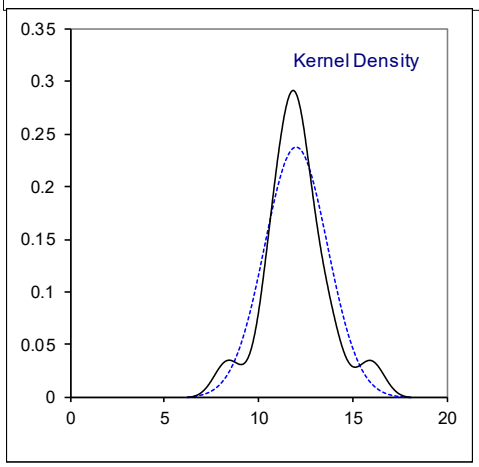


Determination of Calcium (Ca) on sample #20030; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	14		-----	
179	D5185	12		-----	
237	D5185	11		-----	
257	D6595	11.87		-----	
309	D5185	10.38		-----	
325	D5185	11		-----	
329	D4951	12		-----	
349	D5185	12		-----	
432		-----		-----	
496	D5185	8.39		-----	
633		-----		-----	
862		-----		-----	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1011		-----		-----	
1146	D5185	12.44		-----	
1243	DIN51399	13		-----	
1417	D5185	11.6		-----	
1748		-----		-----	
1799		-----		-----	
6002	D5185	13.41		-----	
6016	D5185	12.3		-----	
6197	D4951	16		-----	
6235		-----		-----	
6253		-----		-----	
6310	D7751	11		-----	

	normality	not OK	
	n	16	
	outliers	0	
	mean (n)	12.024	
	st.dev. (n)	1.6752	
	R(calc.)	4.691	
	st.dev.(D5185:18)	(0.1358)	
	R(D5185:18)	(0.380)	Application range : 40 – 9000 mg/kg
Compare			
	R(Horwitz)	3.705	



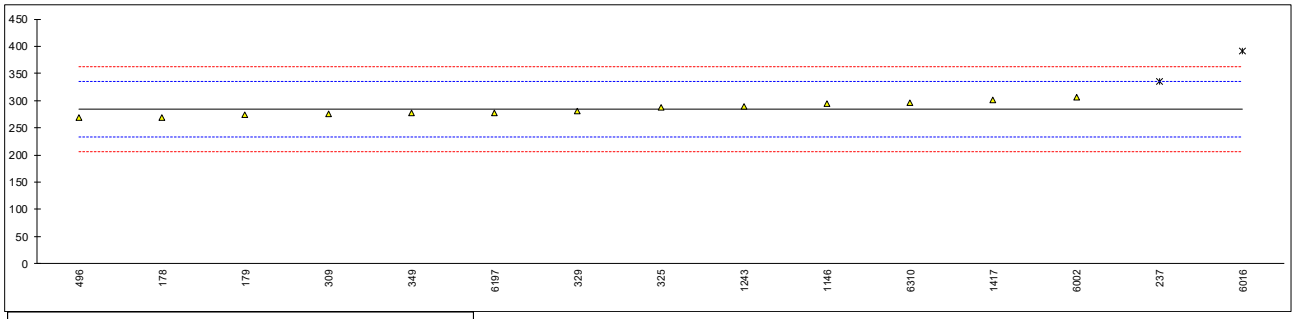


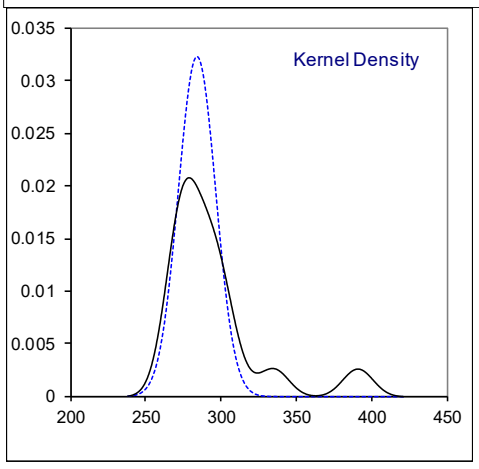
Determination of Phosphorus (P) on sample #20030; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	269		-0.59	
179	D5185	274		-0.40	
237	D5185	335	G(0.05)	1.96	
257		-----		-----	
309	D5185	275.0		-0.36	
325	D5185	287		0.11	
329	D4951	280		-0.16	
349	D5185	277		-0.28	
432		-----		-----	
496	D5185	268.4		-0.61	
633		-----		-----	
862		-----		-----	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1011		-----		-----	
1146	D5185	295.0		0.42	
1243	DIN51399	290		0.22	
1417	D5185	300.7		0.64	
1748		-----		-----	
1799		-----		-----	
6002	D5185	306		0.84	
6016	D5185	391.0	G(0.01)	4.12	
6197	D4951	277		-0.28	
6235		-----		-----	
6253		-----		-----	
6310	D7751	296		0.45	

normality	OK
n	13
outliers	2
mean (n)	284.238
st.dev. (n)	12.3677
R(calc.)	34.629
st.dev.(D5185:18)	25.8912
R(D5185:18)	72.495

Application range: 10 – 1000 mg/kg

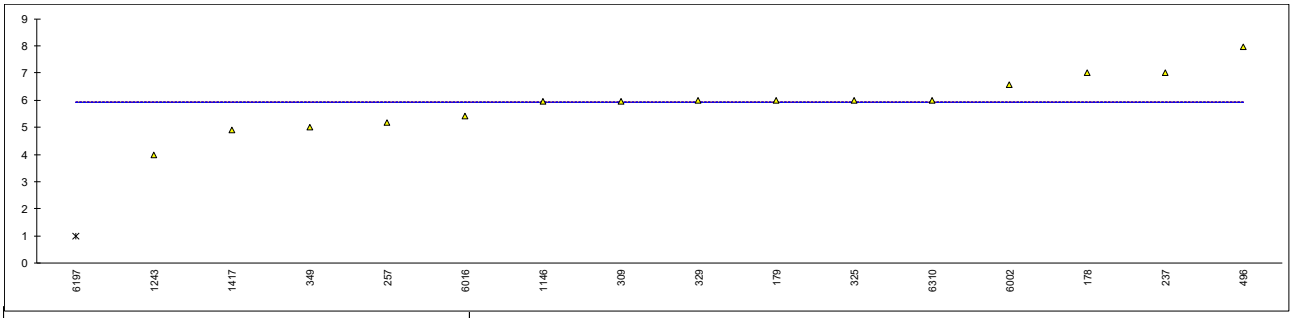


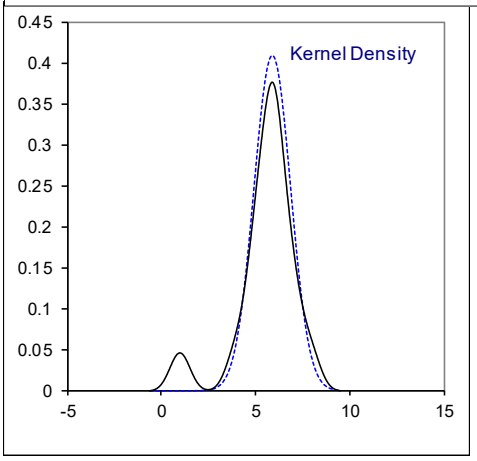


Determination of Zinc (Zn) on sample #20030; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	7		-----	
179	D5185	6		-----	
237	D5185	7		-----	
257	D6595	5.16		-----	
309	D5185	5.96		-----	
325	D5185	6		-----	
329	D4951	6		-----	
349	D5185	5		-----	
432		-----		-----	
496	D5185	7.95		-----	
633		-----		-----	
862		-----		-----	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1011		-----		-----	
1146	D5185	5.940		-----	
1243	DIN51399	4		-----	
1417	D5185	4.9		-----	
1748		-----		-----	
1799		-----		-----	
6002	D5185	6.57		-----	
6016	D5185	5.4		-----	
6197	D4951	1	G(0.01)	-----	
6235		-----		-----	
6253		-----		-----	
6310	D7751	6		-----	

	normality	OK	
	n	15	
	outliers	1	
	mean (n)	5.925	
	st.dev. (n)	0.9756	
	R(calc.)	2.732	
	st.dev.(D5185:18)	(0.2098)	
	R(D5185:18)	(0.588)	Application range: 60 – 1600 mg/kg
Compare			
	R(Horwitz)	2.031	





APPENDIX 2

Number of participants per country

1 lab in	AUSTRIA
3 labs in	BELGIUM
1 lab in	CHINA, People's Republic
2 labs in	GERMANY
1 lab in	JORDAN
1 lab in	KAZAKHSTAN
1 lab in	MOROCCO
2 labs in	NETHERLANDS
1 lab in	NIGERIA
1 lab in	PHILIPPINES
2 labs in	POLAND
1 lab in	PORTUGAL
1 lab in	ROMANIA
2 labs in	SAUDI ARABIA
1 lab in	SINGAPORE
1 lab in	SPAIN
1 lab in	TANZANIA
1 lab in	UNITED ARAB EMIRATES
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	= possibly an error in calculations
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
SDS	= Safety Data Sheet

Literature

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