

**Results of Proficiency Test
Turbine Oil (fresh)
May 2021**

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2018 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of fresh Turbine Oil every year. During the annual proficiency testing program 2020/2021 it was decided to continue the round robin for the analysis of fresh Turbine Oil.

In this interlaboratory study 28 laboratories in 19 different countries registered for participation. See appendix 2 for the number of participants per country. In this report the results of the fresh Turbine 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 one sample of fresh Turbine Oil in an one-liter bottle labelled #21066. 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 110 liters of fresh Turbine Oil was obtained from a third party. After homogenization 58 amber glass bottles of 1L were filled and labelled #21066.

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 #21066-1	0.86796
sample #21066-2	0.86796
sample #21066-3	0.86796
sample #21066-4	0.86796
sample #21066-5	0.86796
sample #21066-6	0.86796
sample #21066-7	0.86796
sample #21066-8	0.86796

Table 1: homogeneity test results of subsamples #21066

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)	0.00000
reference test method	ISO12185:96
0.3 x R (reference test method)	0.00015

Table 2: evaluation of the repeatability of subsamples #21066

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 sample of fresh Turbine Oil labelled #21066 was sent on April 21, 2021. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of fresh Turbine 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, Air Release time at 50°C, Density at 15°C, Flash Point C.O.C., Foam Characteristics (Foaming Tendency, Foam Stability), Kinematic Viscosity at 40°C and at 100°C, Viscosity Index, Pour Point (Manual and Automated 1°C interval), Sulfur, Water, Water Separability at 54°C (Distilled water), Calcium as Ca, Phosphorus as P and Zinc as Zn.

Some extra information was asked about the determinations of Total Acid Number and Foam Characteristics.

It was explicitly requested to treat the sample as if it was a routine sample and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

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

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

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

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

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

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

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

3.2 GRAPHICS

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

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM 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.

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:

$$Z_{(\text{target})} = (\text{test result} - \text{average of PT}) / \text{target standard deviation}$$

The $Z_{(\text{target})}$ scores are listed in the test result tables in appendix 1.

Absolute values for $z < 2$ are very common and absolute values for $z > 3$ are very rare.

Therefore, the usual interpretation of z-scores is as follows:

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

4 EVALUATION

In this interlaboratory study some problems were encountered with the dispatch of the samples. Three participants reported test results after the final reporting date and one other participant did not report any test results. Not all participants were able to report test results for all requested tests. In total 27 participants reported 368 numerical test results. Observed were 8 outlying test results, which is 2.2%. In proficiency studies, 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 methods are referred to with a number (e.g. D2270) and an added designation for the year that the method was adopted or revised (e.g. D2270:10). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D2270:10(2016)). In the test results tables of appendix 1 only the method number and year of adoption or revision (e.g. D2270:10) will be used.

Total Acid Number: This determination was very problematic. No statistical outliers were observed. The calculated reproducibility is not at all in agreement with the requirements of ASTM D664-A:18e2 IP 60mL nor in agreement with BEP 60mL, IP 125mL or BEP 125mL.

When the test results for IP and BEP were evaluated separately, the calculated reproducibilities of the test results are also not in agreement. Remarkably, one participant still used pH 11 for BEP instead of pH 10. In test method ASTM D664-A:18e2 pH 10 is mentioned.

Air Release time at 50°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D3427:19.

Density at 15°C: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO12185:96.

Flash Point C.O.C.: This determination was problematic. No statistical outliers were observed but one test result was excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of ASTM D92:18.

Foam Characteristics (Tendency and Stability): This determination was problematic. In total one statistical outlier was observed. It was decided not to calculate z-scores for Foaming Tendency at sequence I and III due to the large difference between Calculated and Reference reproducibility. The Foaming Tendency determination for sequence II after rejection of the statistical outlier is in full agreement with the requirements of ASTM D892:18. Almost all reported test results for Foam Stability were zero. Therefore, it was decided to calculate no z-scores.

The determination of the Foam Characteristics is very sensitive in maintenance and execution. In ASTM D892:18 many tips and tricks are given in the test method part X1. Possible sources for the large variation are the cleaning and checking of the air diffuser, air tubes and test

cylinders, the air flow rate used during the blowing period. About 60% of the reporting participants used the sample as received. About 85% of the reporting participants used a metal diffuser.

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. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D445:19a.

Viscosity Index: This determination was problematic. No statistical outliers were observed but two test results were excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of ASTM D2270:10(2016).

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

Pour Point Automated: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5950:14(2020).

Sulfur: 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 D4294:16e1.

Water: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D6304:20 procedure C, nor with procedure A but is in agreement with procedure B.

Water Separability at 54°C, distilled water: This determination was not problematic. Two statistical outliers were observed over 3 parameters. The calculated reproducibilities after rejection of the statistical outliers are in good agreement with the requirements of ASTM D1401:19.

Calcium: This determination may not be problematic. All of the reporting participants agreed on a consensus value below the application range of ASTM D5185:18. Therefore, no z-scores were calculated.

Phosphorus: This determination may not be problematic. All of the reporting participants agreed on a consensus value below the application range of ASTM D5185:18. Therefore, no z-scores were calculated.

Zinc: This determination may not be problematic. All of the reporting participants agreed on a consensus value 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 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 ($2.8 \cdot$ standard deviation) and the target reproducibility derived from literature reference test methods (in casu ASTM 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	18	0.051	0.072	0.035
Air Release time at 50°C	minutes	13	2.8	1.4	1.9
Density at 15°C	kg/L	21	0.8680	0.0004	0.0005
Flash Point C.O.C.	°C	17	231.1	22.8	18
Foaming Tendency Seq. I	mL	15	224.0	349.2	(80.9)
Foaming Tendency Seq. II	mL	14	15.4	17.8	18.8
Foaming Tendency Seq. III	mL	15	190.0	128.3	(99.4)
Foam Stability Seq. I	mL	13	0	n.e.	n.e.
Foam Stability Seq. II	mL	15	0	n.e.	n.e.
Foam Stability Seq. III	mL	14	0	n.e.	n.e.
Kinematic Viscosity at 40°C	mm ² /s	27	46.012	0.563	0.561
Kinematic Viscosity at 100°C	mm ² /s	23	6.815	0.046	0.094
Viscosity Index		22	102.15	2.56	2
Pour Point Manual	°C	16	-13.2	10.1	9
Pour Point Automated 1°C interval	°C	7	-11.7	3.5	4.5
Sulfur	mg/kg	8	24	17	15
Water	mg/kg	21	27.5	29.0	15.8
Water Separability at 54°C, distilled water					
Time to reach ≤ 3 ml emulsion	minutes	12	7.5	7.2	20
Time to reach 37 ml water	minutes	12	8.6	13.3	20
Time to reach complete break	minutes	12	10.0	10.8	20
Calcium as Ca	mg/kg	21	<10	n.e	n.e.
Phosphorus as P	mg/kg	21	<10	n.e	n.e.
Zinc as Zn	mg/kg	21	<20	n.e	n.e.

Table 3: reproducibilities of tests on sample #21066

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 reference test methods. The tests that are problematic have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF MAY 2021 WITH PREVIOUS PTS

	May 2021	May 2020	May 2019	May 2018
Number of reporting laboratories	27	17	20	19
Number of test results	368	254	271	281
Number of statistical outliers	8	8	4	12
Percentage of statistical outliers	2.2%	3.1%	1.5%	4.3%

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	May 2021	May 2020	May 2019	May 2018
Total Acid Number	--	+/-	-	++
Air Release time at 50°C	+	+	-	++
Density at 15°C	+	+/-	+	-
Flash Point C.O.C.	-	+	+	++
Foaming Tendency Seq. I	(--)	(--)	(--)	(--)
Foaming Tendency Seq. II	+/-	-	+/-	+
Foaming Tendency Seq. III	(--)	(--)	(--)	(--)
Kinematic Viscosity at 40°C	+/-	-	+	+
Kinematic Viscosity at 100°C	++	+/-	+	+
Viscosity Index	-	-	-	-
Pour Point Manual	-	+	+	++
Pour Point Automated 1°C interval	+	+	+	+
Sulfur	-	-	+/-	+
Water	-	++	++	++
Water Separability	+	++	+	++
Calcium as Ca	n.e.	n.e.	n.e.	n.e.
Phosphorus as P	n.e.	+	n.e.	++
Zinc as Zn	n.e.	n.e.	n.e.	n.e.

Table 5: comparison determinations against the reference test methods

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

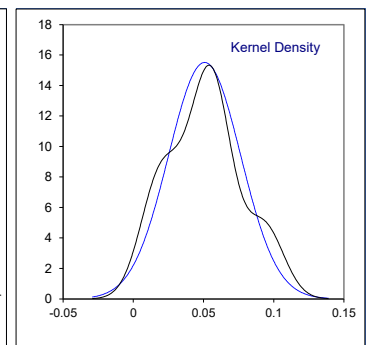
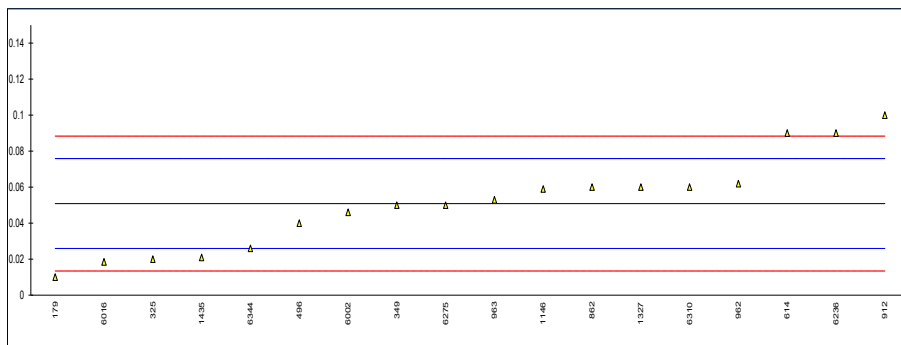
APPENDIX 1

Determination of Total Acid Number on sample #21066; results in mg KOH/g

lab	method	value	mark	z(targ)	Determination of end point	Volume of titration solvent
178		----		----	---	---
179	D664-A	0.01		-3.27	Inflection Point	60 mL
237	D664-A	<0.1		----	Inflection Point	125 mL
325	D664-A	0.02		-2.47	Buffer End Point (pH 10)	125 mL
349	D664-A	0.05		-0.07	Buffer End Point (pH 10)	125 mL
432		----		----	---	---
496	D664-A	0.040		-0.87	Buffer End Point (pH 10)	60 mL
614	D664-A	0.09		3.13	---	60 mL
862	D664-A	0.06		0.73	Inflection Point	60 mL
912	D664-A	0.1	C	3.93	---	---
962	D664-A	0.062		0.89	---	---
963	D974	0.053		0.17	---	---
1011	D664-A	< 0.07		----	Inflection Point	125 mL
1017		----		----	---	---
1026	D664-A	<0.05		----	---	---
1146	D664-A	0.059		0.65	Buffer End Point (pH 10)	125 mL
1184		----		----	---	---
1213	D664-A	<0.1		----	Buffer End Point (pH 10)	60 mL
1327	D664-A	0.06		0.73	Inflection Point	60 mL
1435	D664-A	0.021		-2.39	Buffer End Point (pH 10)	---
6002	D664-A	0.046		-0.39	Inflection Point	60 mL
6016	D664-A	0.0185		-2.59	Inflection Point	60 mL
6236	D8045	0.09		3.13	Inflection Point	---
6275	D974	0.05		-0.07	---	---
6310	D664-A	0.06		0.73	Buffer End Point (pH 10)	60 mL
6344	D664-A	0.026		-1.99	Buffer End Point (pH 11)	60 mL
6387		----		----	---	---
6401		----		----	---	---

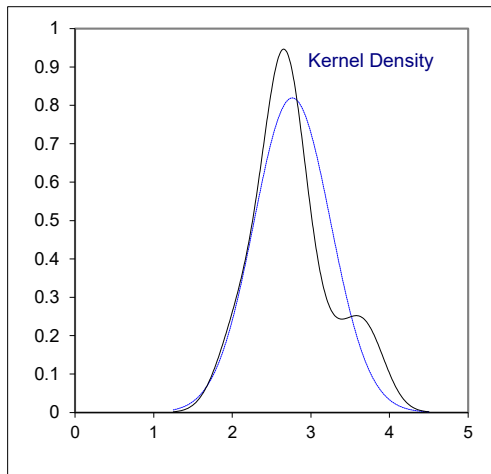
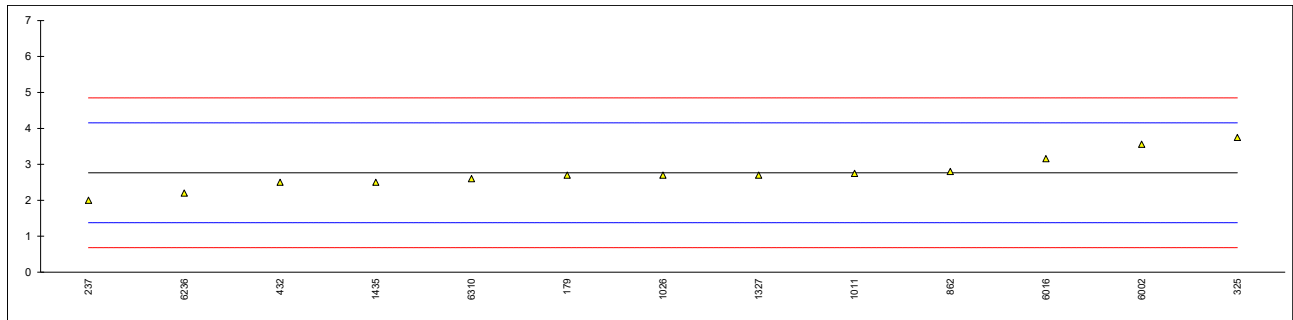
		<u>BEP only</u>	<u>Inflection point only</u>
normality	OK	OK	OK
n	18	8	6
outliers	0	0	0
mean (n)	0.0509	0.0408	0.0474
st.dev. (n)	0.02572	0.01653	0.02956
R(calc.)	0.0720	0.0463	0.0828
st.dev.(D664-A:18e2 IP 60ml)	0.01249	---	---
R(D664-A:18e2 IP 60ml)	0.0350	---	0.0330
Compare			
D664-A:18e2 BEP 60ml	0.0303	0.0244	---
D664-A:18e2 IP 125ml	0.0098	---	0.0091
D664-A:18e2 BEP 125ml	0.0138	0.0078	---

Lab 912 first reported 0.15



Determination of Air Release time at 50°C on sample #21066; results in minutes

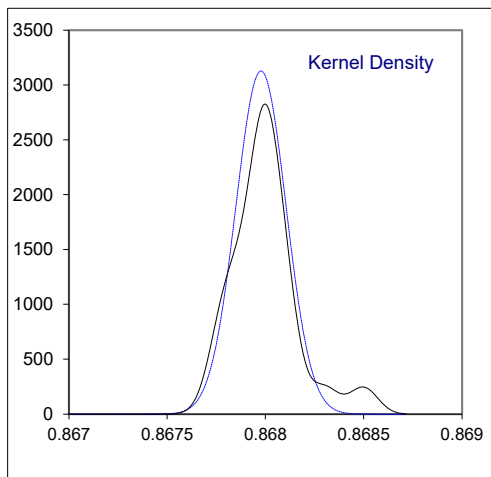
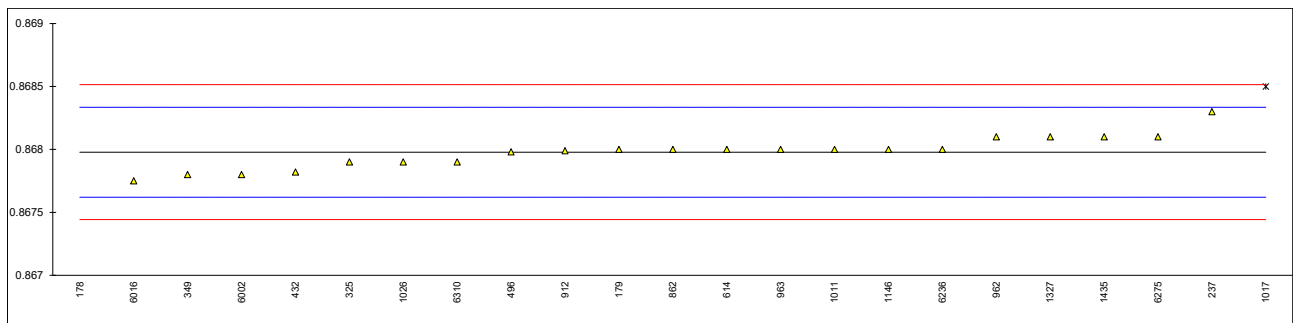
lab	method	value	mark	z(targ)	remarks
178		----		----	
179	D3427	2.7		-0.09	
237	D3427	2.0		-1.10	
325	D3427	3.75		1.42	
349		----		----	
432	ISO9120	2.5		-0.38	
496		----		----	
614		----		----	
862	D3427	2.8		0.05	
912		----		----	
962		----		----	
963		----		----	
1011	IP313/D3427	2.75		-0.02	
1017		----		----	
1026	D3427	2.7		-0.09	
1146		----		----	
1184		----		----	
1213		----		----	
1327	D3427	2.7		-0.09	
1435	D3427	2.5		-0.38	
6002	ISO9120	3.56		1.15	
6016	D3427	3.16		0.57	
6236	D3427	2.2		-0.81	
6275		----		----	
6310	D3427	2.6		-0.23	
6344		----		----	
6387		----		----	
6401		----		----	
normality		OK			
n		13			
outliers		0			
mean (n)		2.763			
st.dev. (n)		0.4869			
R(calc.)		1.363			
st.dev.(D3427:19)		0.6953			
R(D3427:19)		1.947			



Determination of Density at 15°C on sample #21066; results in kg/L

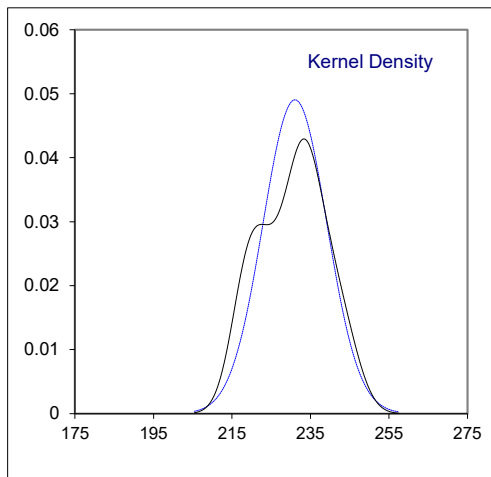
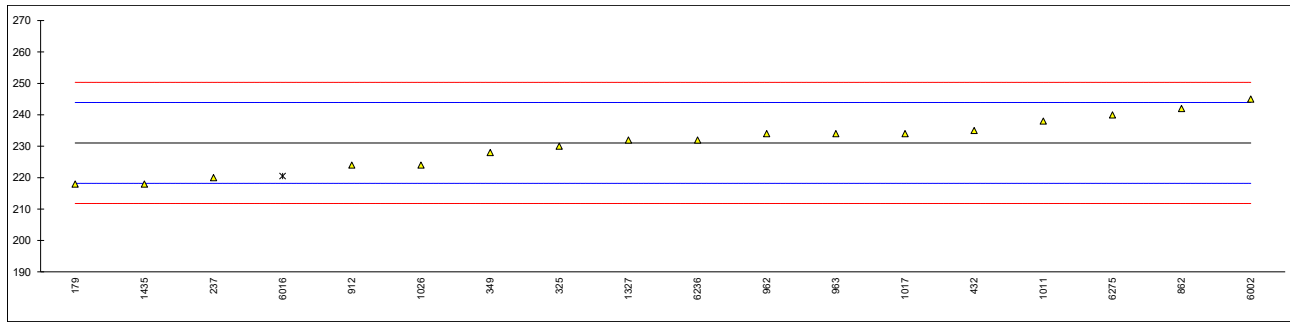
lab	method	value	mark	z(targ)	remarks
178	D4052	0.8660	R(0.01)	-11.08	
179	D4052	0.8680		0.12	
237	D4052	0.8683		1.80	
325	D4052	0.8679		-0.44	
349	D4052	0.8678		-1.00	
432	ISO12185	0.86782		-0.89	
496	ISO12185	0.86798		0.01	
614	D4052	0.8680		0.12	
862	D4052	0.8680		0.12	
912	ISO12185	0.86799		0.07	
962	ISO12185	0.8681		0.68	
963	D4052	0.8680		0.12	
1011	D4052	0.8680		0.12	
1017	ISO12185	0.8685	R(0.05)	2.92	
1026	D4052	0.8679		-0.44	
1146	D4052	0.8680		0.12	
1184		----		----	
1213		----		----	
1327	D4052	0.8681		0.68	
1435	D4052	0.8681		0.68	
6002	ISO3675	0.8678		-1.00	
6016	D4052	0.86775		-1.28	
6236	D4052	0.8680		0.12	
6275	D1298	0.8681		0.68	
6310	D4052	0.8679	C	-0.44	first reported 867.9 kg/L
6344		----		----	
6387		----		----	
6401		----		----	

normality OK
 n 21
 outliers 2
 mean (n) 0.86798
 st.dev. (n) 0.000128
 R(calc.) 0.00036
 st.dev.(ISO12185:96) 0.000179
 R(ISO12185:96) 0.0005



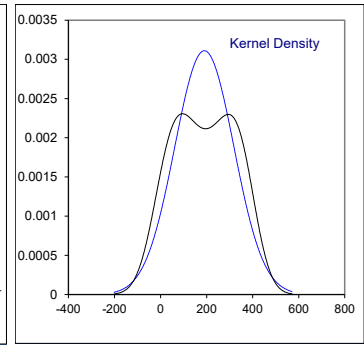
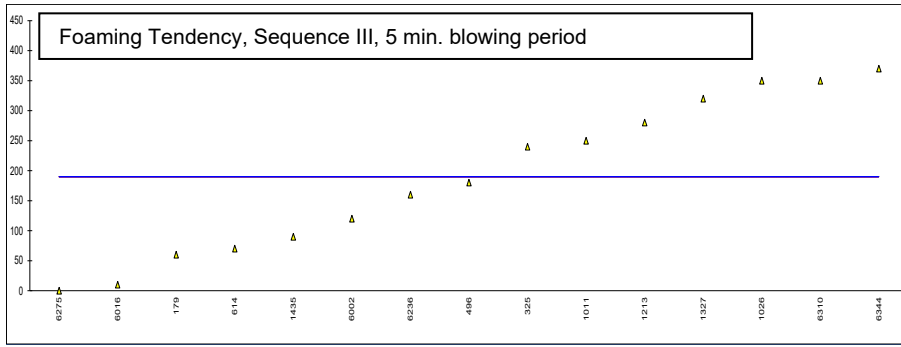
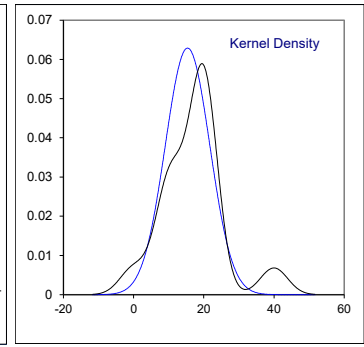
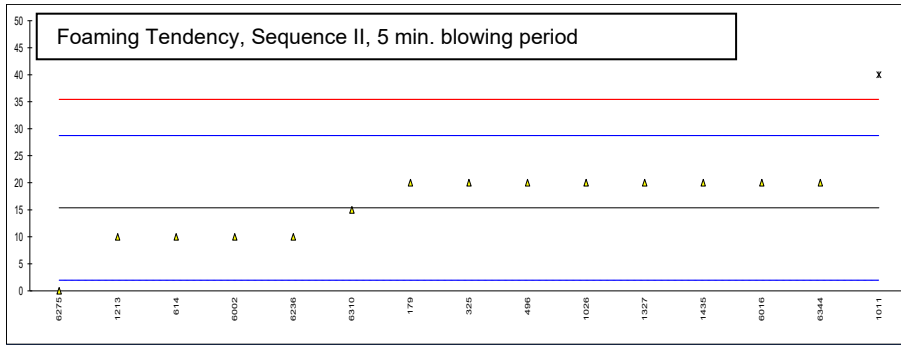
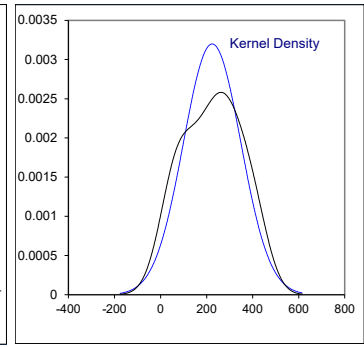
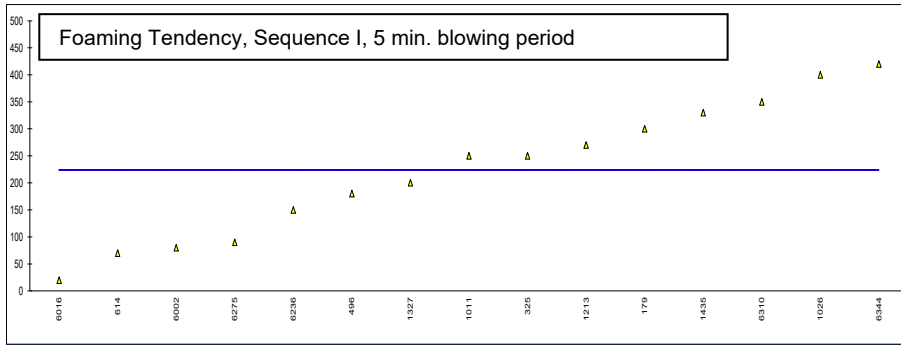
Determination of Flash Point C.O.C. on sample #21066; results in °C

lab	method	value	mark	z(targ)	remarks
178		----		----	
179	D92	218		-2.03	
237	D92	220		-1.72	
325	D92	230		-0.16	
349	D92	228		-0.48	
432	D92	235		0.61	
496		----		----	
614		----		----	
862	D92	242		1.70	
912	D92	224		-1.10	
962	D92	234.0		0.46	
963	D92	234.0		0.46	
1011	D92	238		1.08	
1017	D92	234		0.46	
1026	D92	224		-1.10	
1146		----		----	
1184		----		----	
1213		----		----	
1327	D92	232		0.15	
1435	D92	218.0		-2.03	
6002	ISO2592	245.0		2.17	
6016	D93	220.5	ex	-1.64	test result excluded as used test method is Flash Point PMcc
6236	D92	232		0.15	
6275	D92	240		1.39	
6310		----		----	
6344		----		----	
6387		----		----	
6401		----		----	
normality		OK			
n		17			
outliers		0 +1ex			
mean (n)		231.06			
st.dev. (n)		8.135			
R(calc.)		22.78			
st.dev.(D92:18)		6.429			
R(D92:18)		18			



Determination of Foaming Tendency, Sequence I, II and III (5 min. blowing period) on sample #21066; results in mL

lab	method	Sample used	Diffuser	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
178		---	---	----		----	----		----	----		----
179	D892	As received	Metal	300		----	20		0.69	60		----
237		---	---	----		----	----		----	----		----
325	D892	As received	Metal	250		----	20		0.69	240		----
349		---	---	----		----	----		----	----		----
432		---	---	----		----	----		----	----		----
496	D892	After agitation	Metal	180		----	20		0.69	180		----
614	D892	As received	Metal	70		----	10		-0.80	70		----
862		---	---	----		----	----		----	----		----
912		---	---	----		----	----		----	----		----
962		---	---	----		----	----		----	----		----
963		---	---	----		----	----		----	----		----
1011	D892 (Altern.)	---	Metal	250		----	40	G(0.05)	3.68	250		----
1017		---	---	----		----	----		----	----		----
1026	D892	---	---	400		----	20		0.69	350		----
1146		---	---	----		----	----		----	----		----
1184		---	---	----		----	----		----	----		----
1213	D892 (Altern.)	After agitation	---	270		----	10		-0.80	280		----
1327	D892	As received	Metal	200		----	20		0.69	320		----
1435	D892	As received	Metal	330		----	20		0.69	90		----
6002	ISO6247	After agitation	Metal	80		----	10		-0.80	120		----
6016	D892	After agitation	Stone	20		----	20		0.69	10		----
6236	D892	---	---	150		----	10		-0.80	160		----
6275	D892	As received	Stone	90		----	0		-2.29	0		----
6310	D892	After agitation	Metal	350		----	15		-0.05	350		----
6344	D892	As received	Metal	420		----	20		0.69	370		----
6387		---	---	----		----	----		----	----		----
6401		---	---	----		----	----		----	----		----
	normality			OK			OK			OK		
	n			15			14			15		
	outliers			0			1			0		
	mean (n)			224.00			15.36			190.00		
	st.dev. (n)			124.717			6.344			128.285		
	R(calc.)			349.21			17.76			359.20		
	st.dev.(D892:18)			(28.885)			6.699			(35.490)		
	R(D892:18)			(80.88)			18.76			(99.37)		



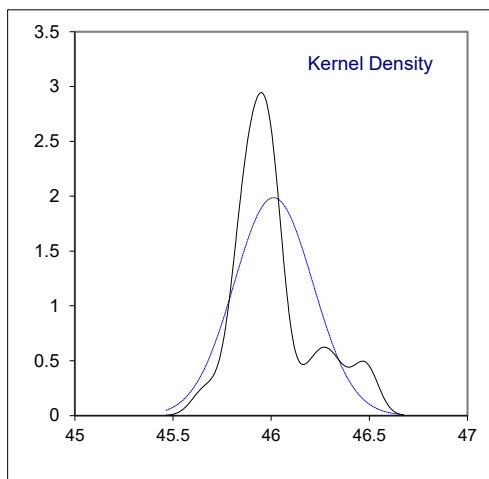
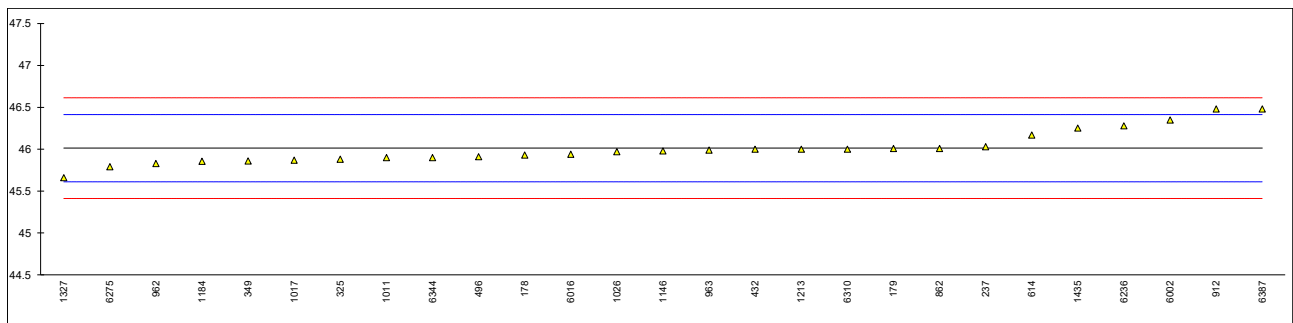
Determination of Foam Stability, Sequence I, II and III (10 min. settling period) on sample #21066; results in mL

lab	method	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
178		----		----	----		----	----		----
179		----		----	0		----	----		----
237		----		----	----		----	----		----
325	D892	0		----	0		----	0		----
349		----		----	----		----	----		----
432		----		----	----		----	----		----
496	D892	0		----	0		----	0		----
614	D892	0		----	0		----	0		----
862		----		----	----		----	----		----
912		----		----	----		----	----		----
962		----		----	----		----	----		----
963		----		----	----		----	----		----
1011	D892 (Alternative)	0		----	0		----	0		----
1017		----		----	----		----	----		----
1026	D892	50	f+?	----	0		----	0		----
1146		----		----	----		----	----		----
1184		----		----	----		----	----		----
1213	D892 (Alternative)	0		----	0		----	0		----
1327	D892	0		----	0		----	0		----
1435	D892	0		----	0		----	0		----
6002	ISO6247	0		----	0		----	0		----
6016	D892	5		----	0		----	0		----
6236	D892	0		----	0		----	0		----
6275	D892	0		----	0		----	0		----
6310	D892	0		----	0		----	0		----
6344	D892	0		----	0		----	0		----
6387		----		----	----		----	----		----
6401		----		----	----		----	----		----
	n	13			15			14		
	mean (n)	0			0			0		

Determination of Kinematic Viscosity at 40°C on sample #21066; results in mm²/s

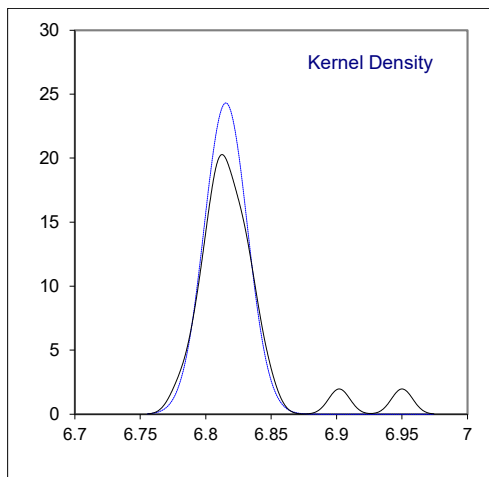
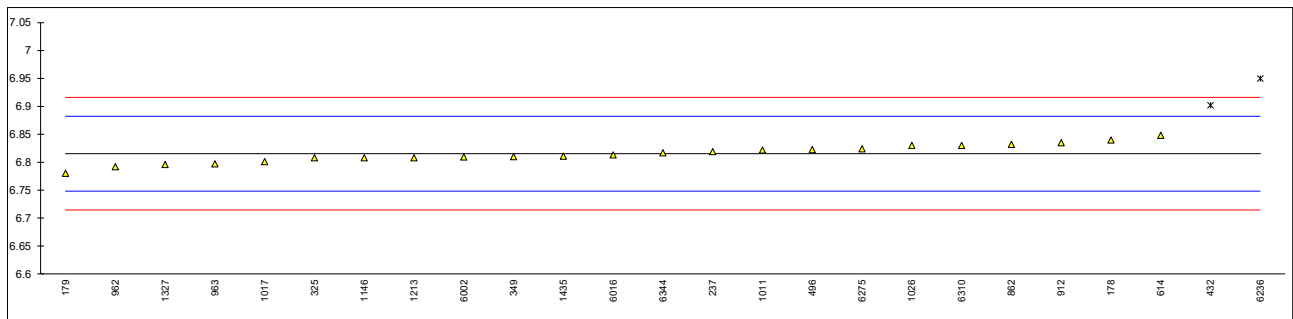
lab	method	value	mark	z(targ)	remarks
178	D445	45.93		-0.41	
179	D445	46.01		-0.01	
237	D445	46.03		0.09	
325	D445	45.88		-0.66	
349	D445	45.86		-0.76	
432	D445	46.00		-0.06	
496	D445	45.911		-0.50	
614	D7042	46.17		0.79	
862	D445	46.01		-0.01	
912	D445	46.48	C	2.33	first reported 46.82
962	D445	45.83	C	-0.91	first reported 45.28
963	D445	45.99		-0.11	
1011	D7042	45.90		-0.56	
1017	D445	45.87		-0.71	
1026	D445	45.97		-0.21	
1146	D445	45.98		-0.16	
1184	D445	45.856		-0.78	
1213	D445	46.00		-0.06	
1327	D445	45.66		-1.76	
1435	D7042	46.252		1.20	
6002	ISO3104	46.35		1.69	
6016	D7042	45.940		-0.36	
6236	D7279 corr. to D445	46.28		1.34	
6275	D445	45.79		-1.11	
6310	D7279 corr. to D445	46.0		-0.06	
6344	ISO3104	45.90		-0.56	
6387	D445	46.48		2.33	
6401		----		----	

normality suspect
 n 27
 outliers 0
 mean (n) 46.0122
 st.dev. (n) 0.20089
 R(calc.) 0.5625
 st.dev.(D445:19a) 0.20048
 R(D445:19a) 0.5613



Determination of Kinematic Viscosity at 100°C on sample #21066; results in mm²/s

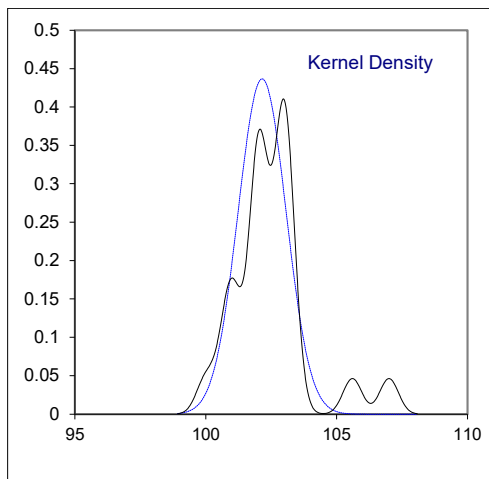
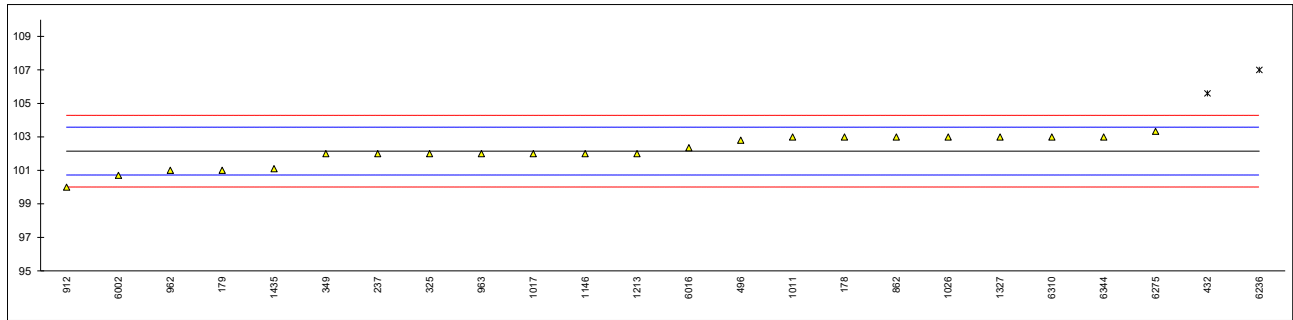
lab	method	value	mark	z(targ)	remarks
178	D445	6.84		0.73	
179	D445	6.78		-1.05	
237	D445	6.819		0.11	
325	D445	6.808		-0.22	
349	D445	6.810		-0.16	
432	D445	6.902	R(0.01)	2.58	
496	D445	6.8227		0.22	
614	D7042	6.848		0.97	
862	D445	6.832		0.50	
912	D445	6.835		0.58	
962	D445	6.792	C	-0.70	first reported 6.721
963	D445	6.797		-0.55	
1011	D7042	6.822		0.20	
1017	D445	6.801		-0.43	
1026	D445	6.83		0.44	
1146	D445	6.808		-0.22	
1184		----		----	
1213	D445	6.808		-0.22	
1327	D445	6.796		-0.58	
1435	D7042	6.8108		-0.14	
6002	ISO3104	6.8095		-0.17	
6016	D7042	6.8132		-0.06	
6236	D7279 corr. to D445	6.95	R(0.01)	4.01	
6275	D445	6.824		0.26	
6310	D7279 corr. to D445	6.83		0.44	
6344	ISO3104	6.817		0.05	
6387		----		----	
6401		----		----	
normality		OK			
n		23			
outliers		2			
mean (n)		6.8154			
st.dev. (n)		0.01640			
R(calc.)		0.0459			
st.dev.(D445:19a)		0.03359			
R(D445:19a)		0.0941			



Determination of Viscosity Index on sample #21066

lab	method	value	mark	z(targ)	remarks
178	D2270	103		1.19	
179	D2270	101		-1.61	
237	D2270	102		-0.21	
325	D2270	102		-0.21	
349	D2270	102		-0.21	
432	D2270	105.6	ex	4.83	test result excluded as statistical outlier in KV 100°C
496	D2270	102.8		0.91	
614		----		----	
862	D2270	103		1.19	
912	D2270	100		-3.01	iis calculated 101, iis calc. 100 with first reported KV 40°C
962	D2270	101		-1.61	iis calculated 102, iis calc. 101 with first reported KV 100°C
963	D2270	102		-0.21	
1011	D2270	103		1.19	
1017	D2270	102		-0.21	
1026	D2270	103		1.19	
1146	D2270	102		-0.21	
1184		----		----	
1213	D2270	102		-0.21	
1327	D2270	103		1.19	
1435	D2270	101.10		-1.47	
6002	ISO2909	100.7		-2.03	
6016	D2270	102.35		0.28	
6236	D2270	107	ex, E	6.79	test result excl. as statistical outlier in KV 100°C, iis calc. 106
6275	D2270	103.33		1.65	
6310	D2270	103		1.19	
6344	STN656218	103		1.19	
6387		----		----	
6401		----		----	

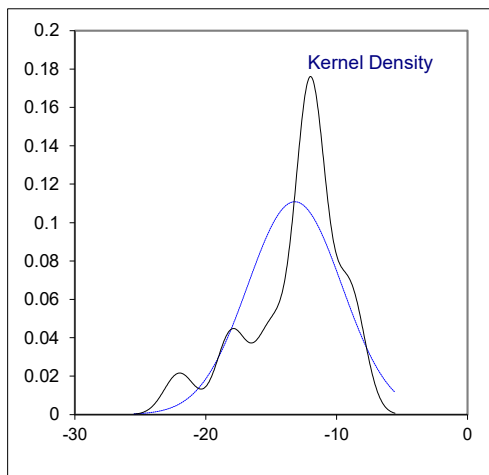
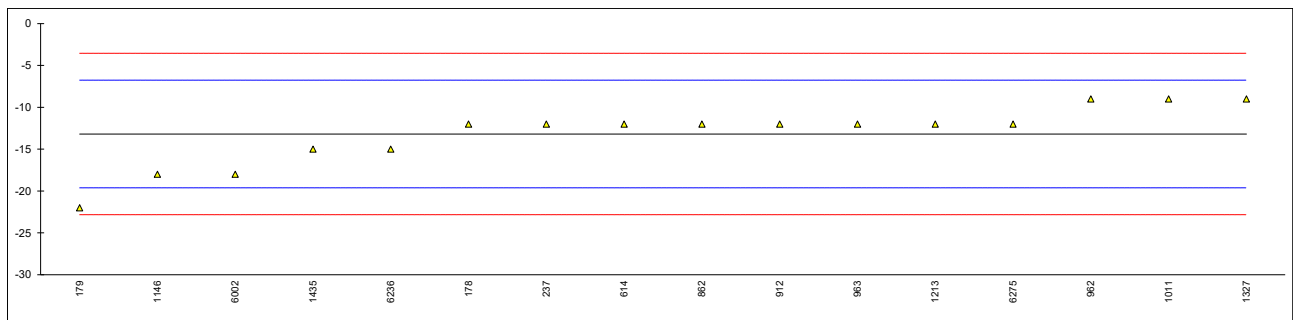
normality OK
 n 22
 outliers 0 +2ex
 mean (n) 102.15
 st.dev. (n) 0.914
 R(calc.) 2.56
 st.dev.(D2270:10) 0.714
 R(D2270:10) 2



Determination of Pour Point Manual on sample #21066; results in °C

lab	method	value	mark	z(targ)	remarks
178	D97	-12		0.37	
179	D97	-22		-2.74	
237	D97	-12		0.37	
325		----		----	
349		----		----	
432		----		----	
496		----		----	
614	D97	-12		0.37	
862	D97	-12		0.37	
912	D97	-12		0.37	
962	D97	-9		1.30	
963	D97	-12		0.37	
1011	D97	-9		1.30	
1017		----		----	
1026		----		----	
1146	D97	-18		-1.50	
1184		----		----	
1213	D97	-12		0.37	
1327	D97	-9		1.30	
1435	D97	-15		-0.56	
6002	ISO3016	-18		-1.50	
6016		----		----	
6236	D97	-15		-0.56	
6275	D97	-12		0.37	
6310		----		----	
6344		----		----	
6387		----		----	
6401		----		----	

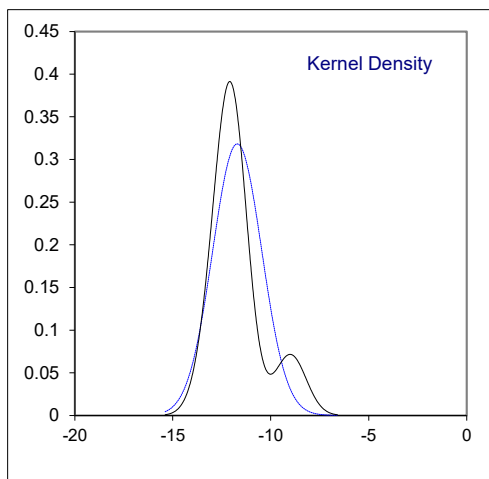
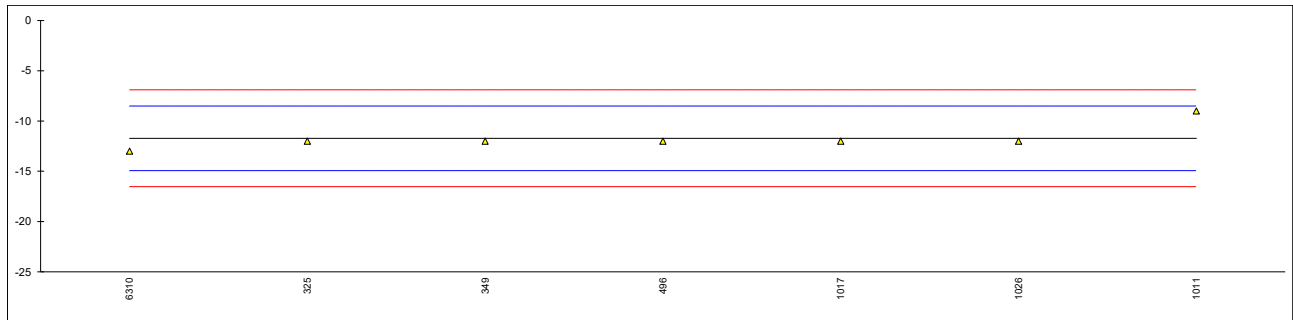
normality suspect
n 16
outliers 0
mean (n) -13.19
st.dev. (n) 3.600
R(calc.) 10.08
st.dev.(D97:17b) 3.214
R(D97:17b) 9



Determination of Pour Point Automated 1°C interval on sample #21066; results in °C

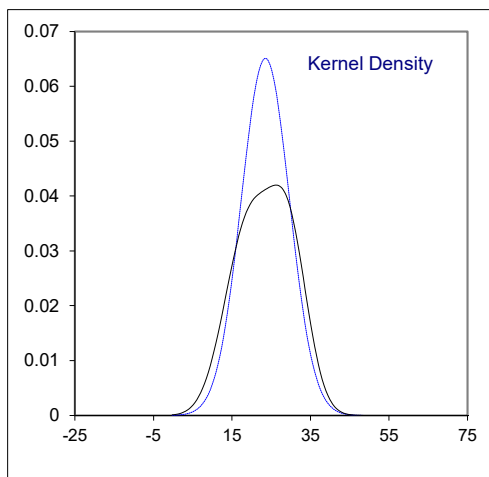
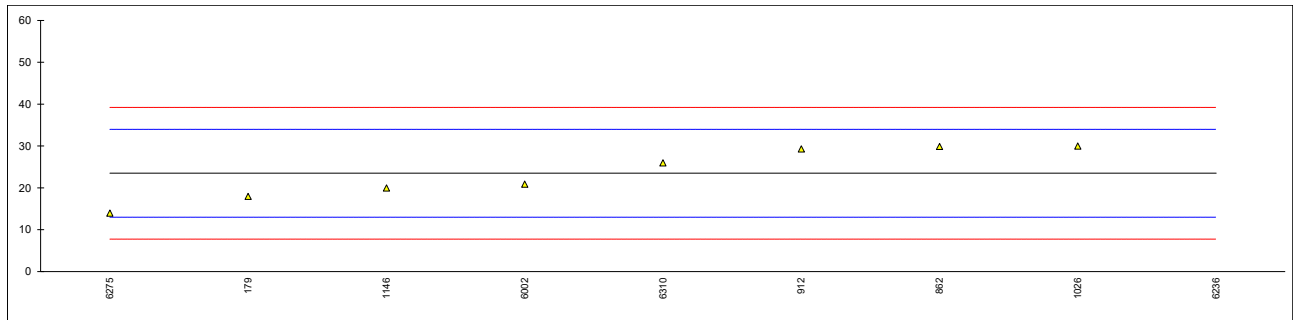
lab	method	value	mark	z(targ)	remarks
178		----		----	
179		----		----	
237		----		----	
325	D5950	-12		-0.18	
349	D5949	-12		-0.18	
432		----		----	
496	D5950	-12		-0.18	
614		----		----	
862		----		----	
912		----		----	
962		----		----	
963		----		----	
1011	D6892	-9		1.69	
1017	D5950	-12		-0.18	
1026	D5950	-12		-0.18	
1146		----		----	
1184		----		----	
1213		----		----	
1327		----		----	
1435		----		----	
6002		----		----	
6016		----		----	
6236		----		----	
6275		----		----	
6310	D5950	-13		-0.80	
6344		----		----	
6387		----		----	
6401		----		----	

normality unknown
n 7
outliers 0
mean (n) -11.71
st.dev. (n) 1.254
R(calc.) 3.51
st.dev.(D5950:14) 1.607
R(D5950:14) 4.5



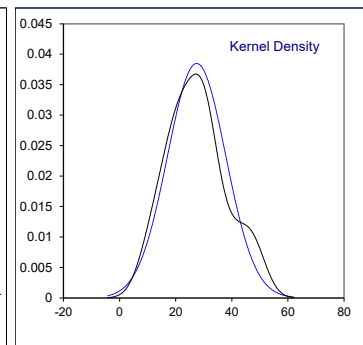
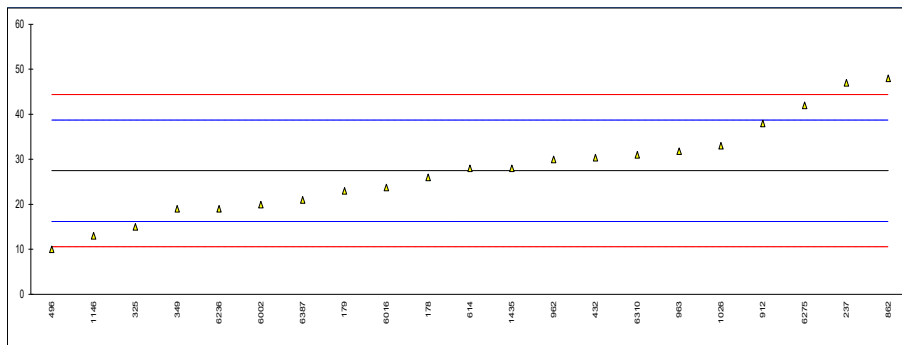
Determination of Sulfur on sample #21066; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		----		----	
179	D4294	18	C	-1.05	first reported <0.0020 mg/kg
237	D4294	<20		----	
325	D5185	<50		----	
349		----		----	
432		----		----	
496		----		----	
614		----		----	
862	D2622	29.9		1.22	
912	D4294	29.3		1.10	
962		----		----	
963		----		----	
1011		----		----	
1017		----		----	
1026	ISO20884	30		1.24	
1146	D4294	20		-0.67	
1184		----		----	
1213		----		----	
1327		----		----	
1435		----		----	
6002	D5185	20.9		-0.50	
6016		----		----	
6236	D5185	232.2	D(0.01)	39.79	
6275	D5185	14		-1.81	
6310	D7751	26		0.47	
6344		----		----	
6387		----		----	
6401		----		----	
normality		OK			
n		8			
outliers		1			
mean (n)		23.51			
st.dev. (n)		6.125			
R(calc.)		17.15			
st.dev.(D4294:16e1)		5.244			
R(D4294:16e1)		14.68			



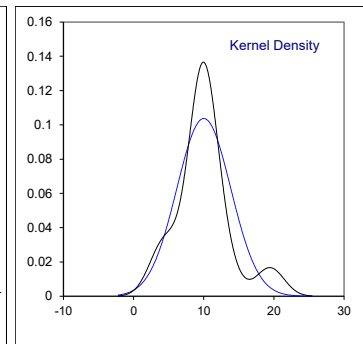
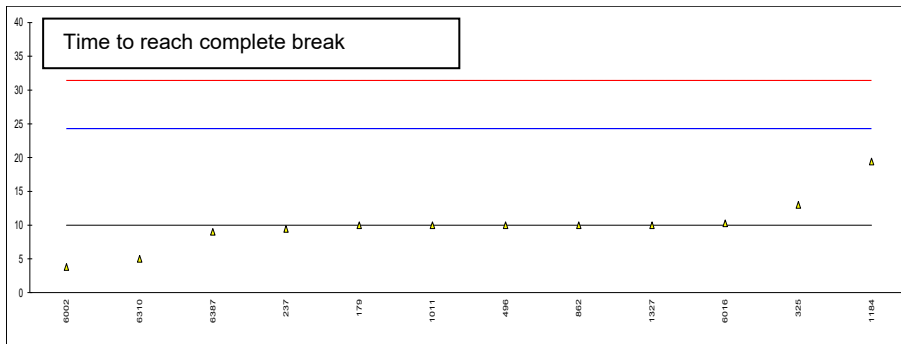
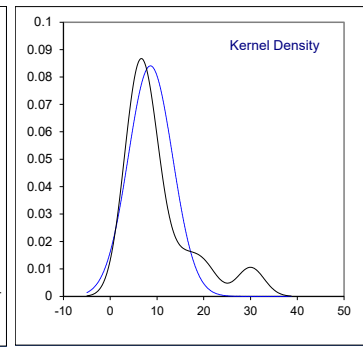
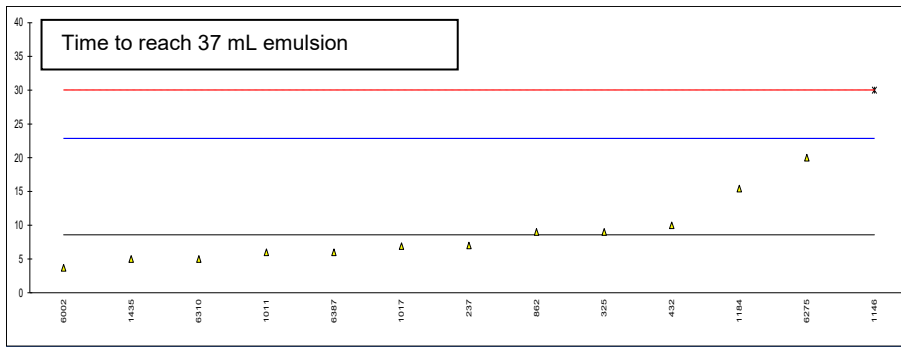
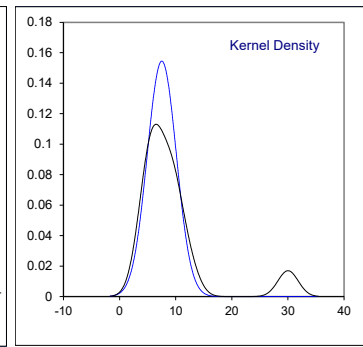
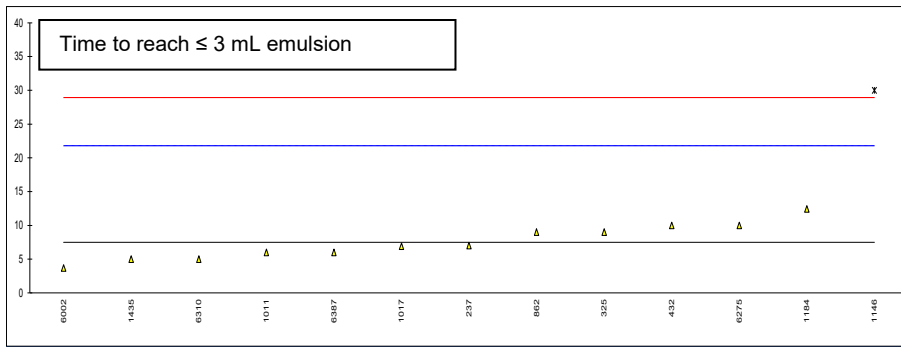
Determination of Water on sample #21066; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D6304-C	26		-0.26	
179	D6304-C	23		-0.79	
237	D6304-C	47		3.47	
325	D6304-C	15		-2.21	
349	D6304-A	19		-1.50	
432	D6304-C	30.35		0.51	
496	D6304-C	10		-3.10	
614	D6304-B	28		0.09	
862	D6304-A	48		3.65	
912	D6304-C	38		1.87	
962	D6304-C	29.96		0.44	
963	D6304-A	31.8		0.77	
1011		----		----	
1017		----		----	
1026	D6304-C	33		0.98	
1146	D6304-C	13		-2.57	
1184		----		----	
1213		----		----	
1327		----		----	
1435	D6304-A	28		0.09	
6002	In house	19.91		-1.34	
6016	D6304-A	23.75		-0.66	
6236	D6304-A	19		-1.50	
6275	D6304-A	42		2.58	
6310	D6304-C	31		0.63	
6344		----		----	
6387	D6304-A	21		-1.15	
6401		----		----	
normality		OK			
n		21			
outliers		0			
mean (n)		27.47			
st.dev. (n)		10.369			
R(calc.)		29.03			
st.dev.(D6304-C:20)		5.633			
R(D6304-C:20)		15.77			range 20 - 360 mg/kg
compare					
R(D6304-A:20)		24.19			range 20 - 25000 mg/kg
R(D6304-B:20)		91.51			range 30 - 2100 mg/kg



Determination of Water Separability at 54°C, distilled water on sample #21066; results in minutes

lab method	3mL or less emulsion			37mL of water			complete break (40-40-0)			test aborted	time test aborted
	mark	z(targ)		mark	z(targ)		mark	z(targ)			
178	----	----	----	----	----	----	----	----	----	----	----
179 D1401	----	----	----	----	----	----	10	0.00	----	----	----
237 D1401	7.0	-0.07	7.0	7.0	-0.22	9.46	9.46	-0.08	No	----	----
325 D1401	9	0.21	9	9	0.06	13	13	0.42	----	----	----
349	----	----	----	----	----	----	----	----	----	----	----
432 D1401	10	0.35	10	10	0.20	>30	>30	----	Yes	30	----
496	----	----	----	----	----	----	10	0.00	No	----	----
614	----	----	----	----	----	----	----	----	----	----	----
862 D1401	9	0.21	9	9	0.06	10	10	0.00	No	10	----
912	----	----	----	----	----	----	----	----	----	----	----
962	----	----	----	----	----	----	----	----	----	----	----
963	----	----	----	----	----	----	----	----	----	----	----
1011	6	-0.21	6	6	-0.36	10	10	0.00	No	----	----
1017 D1401	6.91	-0.08	6.91	6.91	-0.23	----	----	----	----	----	----
1026 D1401	----	----	----	----	----	----	----	----	Yes	10	----
1146 D1401	30	G1	3.15	30	G5	3.00	----	----	----	----	----
1184 D1401	12.43		0.69	15.43		0.96	19.43	1.32	----	----	----
1213	----	----	----	----	----	----	----	----	----	----	----
1327 D1401	----	----	----	----	----	----	10	0.00	No	----	----
1435 D1401	5	-0.35	5	5	-0.50	----	----	----	No	----	----
6002 ISO6614	3.7	-0.53	3.7	3.7	-0.68	3.8	3.8	-0.87	No	----	----
6016 D1401	----	----	----	----	----	----	10.28	0.04	No	----	----
6236 D1401	----	----	----	----	----	----	----	----	Yes	15	----
6275 D1401	10	0.35	20	20	1.60	----	----	----	Yes	30	----
6310	5	-0.35	5	5	-0.50	5	5	-0.70	No	40	----
6344	----	----	----	----	----	----	----	----	----	----	----
6387 D1401	6	-0.21	6	6	-0.36	9	9	-0.14	----	----	----
6401	----	----	----	----	----	----	----	----	----	----	----
normality	OK			not OK			not OK				
n	12			12			12				
outliers	1			1			0				
mean (n)	7.50			8.59			10.00				
st.dev. (n)	2.582			4.746			3.846				
R(calc.)	7.23			13.29			10.77				
st.dev.(D1401:19)	7.143			7.143			7.143				
R(D1401:19)	20			20			20				



Determination of Water Separability at 54°C, distilled water on sample #21066; results in mL

lab	method	oil phase	mark	z(targ)	water phase	mark	z(targ)	emulsion phase	mark	z(targ)
178		----		----	----		----	----		----
179	D1401	40		----	40		----	0		----
237	D1401	40		----	40		----	0		----
325		----		----	----		----	----		----
349		----		----	----		----	----		----
432	D1401	42		----	38		----	0		----
496		----		----	----		----	----		----
614		----		----	----		----	----		----
862	D1401	40		----	40		----	0		----
912		----		----	----		----	----		----
962		----		----	----		----	----		----
963		----		----	----		----	----		----
1011		----		----	----		----	----		----
1017		----		----	----		----	----		----
1026	D1401	40		----	40		----	0		----
1146	D1401	43		----	37		----	0		----
1184	D1401	42		----	37		----	1		----
1213		----		----	----		----	----		----
1327		----		----	----		----	----		----
1435		----		----	----		----	----		----
6002	ISO6614	40		----	40		----	0		----
6016		----		----	----		----	----		----
6236	D1401	43		----	37		----	0		----
6275	D1401	43		----	37		----	0		----
6310		40		----	40		----	0		----
6344		----		----	----		----	----		----
6387		----		----	----		----	----		----
6401		----		----	----		----	----		----

Determination of Calcium as Ca on sample #21066; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	2		----	
179	D5185	<1		----	
237	D5185	<1		----	
325	D5185	<1		----	
349	D5185	0		----	
432		----		----	
496	D5185	0.55		----	
614	D5185	<1		----	
862	D5185	<1		----	
912		----		----	
962	D5185	0.19		----	
963	D5185	0.140		----	
1011	D5185	0		----	
1017	D5185	0		----	
1026	D5185	3		----	
1146	In house	<5		----	
1184		----		----	
1213	D5185	<10		----	
1327	D5185	0		----	
1435		----		----	
6002	D5185	0		----	
6016	D5185	0.38		----	
6236	D5185	0.53		----	
6275	D5185	0.112		----	
6310	D7751	1		----	
6344		----		----	
6387		----		----	
6401		----		----	
	n	21			
	mean (n)	<10			Application range D5185:18: 40 – 9000 mg/kg

Determination of Phosphorus as P on sample #21066; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	3		----	
179	D5185	<1		----	
237	D5185	<1		----	
325	D5185	<1		----	
349	D5185	3		----	
432		----		----	
496	D5185	1.14		----	
614	D5185	<1		----	
862	D5185	<1		----	
912		----		----	
962	D5185	0.84		----	
963	D5185	0.790		----	
1011	D5185	0		----	
1017	D5185	0		----	
1026	D5185	0		----	
1146	In house	<10		----	
1184		----		----	
1213	D5185	<1		----	
1327	D5185	0		----	
1435		----		----	
6002	D5185	0		----	
6016	D5185	2.62	C	----	first reported 49
6236	D5185	0.78		----	
6275	D5185	0.107		----	
6310	D7751	1		----	
6344		----		----	
6387		----		----	
6401		----		----	
	n	21			
	mean (n)	<10			Application range D5185:18: 10 – 1000 mg/kg

Determination of Zinc as Zn on sample #21066; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	2		----	
179	D5185	1		----	
237	D5185	<1		----	
325	D5185	<1		----	
349	D5185	0		----	
432		----		----	
496	D5185	1.17		----	
614	D5185	<1		----	
862	D5185	<1		----	
912		----		----	
962	D5185	<0.1		----	
963	D5185	<0.10		----	
1011	D5185	1.7		----	
1017	D5185	0		----	
1026	D5185	0		----	
1146	In house	<5		----	
1184		----		----	
1213	D5185	<20		----	
1327	D5185	0		----	
1435		----		----	
6002	D5185	0		----	
6016	D5185	0.106		----	
6236	D5185	0.04		----	
6275	D5185	0.0250		----	
6310	D7751	1		----	
6344		----		----	
6387		----		----	
6401		----		----	
	n	21			
	mean (n)	<20			Application range D5185:18: 60 – 1600 mg/kg

APPENDIX 2

Number of participants per country

1 lab in ALGERIA
1 lab in AUSTRALIA
1 lab in AUSTRIA
5 labs in BELGIUM
2 labs in CHINA, People's Republic
1 lab in GERMANY
1 lab in INDIA
1 lab in INDONESIA
1 lab in KAZAKHSTAN
2 labs in NETHERLANDS
1 lab in NIGERIA
1 lab in POLAND
1 lab in PORTUGAL
2 labs in SAUDI ARABIA
1 lab in SLOVAKIA
2 labs in SOUTH KOREA
1 lab in SPAIN
2 labs in UNITED STATES OF AMERICA
1 lab in VIETNAM

APPENDIX 3

Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01), G1	= outlier in Grubbs' outlier test
G(0.05), G5	= 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
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- 4 ISO13528:05
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- 12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)