Institute for
Interlaboratory Studies

Results of Proficiency Test
Turbine Oil (fresh)
May 2023

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 2022/2023 it was decided to continue the round robin for the analysis of fresh Turbine Oil.

In this interlaboratory study 26 laboratories in 19 countries registered for participation, see appendix 2 for the number of participants per country. In this report the results of the Turbine Oil (fresh) proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.
It was decided to send one sample of fresh Turbine Oil in a 1 L bottle labelled #23071.
The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

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

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

A batch of approximately 60 liters of fresh Turbine Oil was obtained from a third party. After homogenization 45 amber glass bottles of 1 L were filled and labelled #23071.
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 #23071-1	0.86794
sample #23071-2	0.86794
sample #23071-3	0.86793
sample #23071-4	0.86794
sample #23071-5	0.86794
sample #23071-6	0.86794
sample #23071-7	0.86794
sample #23071-8	0.86794

Table 1: homogeneity test results of subsamples #23071

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

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

Table 2: evaluation of the repeatability of subsamples #23071

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

To each of the participating laboratories one 1 L bottle of fresh Turbine Oil labelled #23071 was sent on April 19, 2023. 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 (derived from e.g. ISO or ASTM test methods), the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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

The z-scores were calculated according to:

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

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

Absolute values for z<2 are very common and absolute values for z>3 are very rare. Therefore, the usual interpretation of z-scores is as follows:

	$\lvert z \rvert$	< 1	good
1 <	$\lvert z \rvert$	< 2	satisfactory
2 <	$\lvert z \rvert$	< 3	questionable
3 <	$\lvert z \rvert$		unsatisfactory

4 EVALUATION

In this proficiency test no problems were encountered with the dispatch of the samples. Three participants reported test results after the final reporting date and two other participants did not report any test results. Not all participants were able to report all tests requested.
In total 24 participants reported 303 numerical test results. Observed were 11 outlying test results, which is 3.6%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

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

4.1 EVALUATION PER TEST

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

In the iis PT reports ASTM test methods are referred to with a number (e.g. D2270) and an added designation for the year that the test method was adopted or revised (e.g. D2270:10). When a method has been reapproved an “R” will be added and the year of approval (e.g. D2270:10R16).

Total Acid Number: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D664-A:18e2 IP and BEP at 60 mL, nor with the requirements of IP and BEP at 125 mL. When the test results for IP and BEP were evaluated separately, the calculated reproducibilities are only in agreement with IP at 60 mL, not with the requirements of BEP at 60 mL and 125 mL and IP at 125 mL.

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

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

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

Foam Characteristics (Tendency and Stability): This determination was problematic. No statistical outliers were observed over six parameters. 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 is in agreement with the requirements of ASTM D892:18.
Almost all reported test results for Foam Stability were zero. Therefore, it was decided not to calculate 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.
Of the reporting participants seven used the sample as received and five after agitation. All reporting participants used a metal diffuser, except one.

Kinematic Viscosity at 40 °C: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D445:21e2.

Kinematic Viscosity at 100 °C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D445:21e2.

Viscosity Index: This determination was not problematic. No statistical outliers were observed but two test results were excluded. The calculated reproducibility after rejection of the suspect data is in agreement with the requirements of ASTM D2270:10R16.

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

Pour Point Automated: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D5950:14R20.

Sulfur: 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 D4294:21.

Water: 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 D6304:20 procedure B, but not with procedure A and C.

Water Separability at 54 °C, distilled water: This determination was not problematic. One statistical outlier was observed over 3 parameters. The calculated reproducibilities after rejection of the statistical outlier are in agreement with the requirements of ASTM D1401:21.

Calcium: This determination was not problematic. All reporting participants agreed on a value below the application range. Therefore, no z-scores are calculated.

Phosphorus: This determination was not problematic. All reporting participants agreed on a value below the application range. Therefore, no z-scores are calculated.

Zinc: This determination was not problematic. All reporting participants agreed on a value below the application range. Therefore, no z-scores are calculated.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Parameter	unit	n	average	2.8 * sd	R(lit)
Total Acid Number	mg KOH/g	14	0.051	0.046	0.035
Air Release time at 50 °C	minutes	12	3.9	4.3	2.6
Density at 15 °C	kg/L	20	0.8680	0.0003	0.0005
Flash Point C.O.C.	°C	14	235.1	15.1	18
Foaming Tendency Seq. I	mL	12	110.4	309.0	(46.2)
Foaming Tendency Seq. II	mL	11	16.7	18.5	19.3
Foaming Tendency Seq. III	mL	11	107.5	262.4	(67.2)
Foam Stability Seq. I	mL	11	0	n.e.	n.e.
Foam Stability Seq. II	mL	11	0	n.e.	n.e.
Foam Stability Seq. III	mL	10	0	n.e.	n.e.
Kinematic Viscosity at 40 °C	mm^2/s	21	45.965	0.261	0.561
Kinematic Viscosity at 100 °C	mm^2/s	22	6.815	0.076	0.094
Viscosity Index		19	102.26	1.93	2
Pour Point Manual	°C	13	-13.2	7.3	9
Pour Point Automated 1 °C int.	°C	7	-12.1	1.9	4.5
Sulfur	mg/kg	8	28.5	5.0	16.6
Water	mg/kg	14	24.7	26.2	87.8
Water Separability at 54 °C, distilled water					
Time to reach ≤ 3 mL emulsion	minutes	10	8.3	5.6	20
Time to reach 37 mL water	minutes	10	8.4	5.5	20
Time to reach complete break	minutes	12	12.2	13.5	20
Calcium as Ca	mg/kg	18	<40	n.e	n.e.
Phosphorus as P	mg/kg	19	<10	n.e	n.e.
Zinc as Zn	mg/kg	19	<60	n.e	n.e.

Table 3: reproducibilities of tests on sample #23071

For results between brackets no z-scores are calculated.

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

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

	May 2023	May 2022	May 2021	May 2020	May 2019
Number of reporting laboratories	24	26	27	17	20
Number of test results	303	377	368	254	271
Number of statistical outliers	11	16	8	8	4
Percentage of statistical outliers	3.6%	4.2%	2.2%	3.1%	1.5%

Table 4: comparison with previous proficiency tests

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

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

Parameter	May 2023	May 2022	May 2021	May 2020	May 2019
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 int.	++	+	+	+	+
Sulfur	++	+	-	-	+/-
Water	++	++	-	++	++
Water Separability	++	++	+	++	+
Calcium as Ca	n.e.	n.e.	n.e.	n.e.	n.e.
Phosphorus as P	n.e.	++	n.e.	+	n.e.
Zinc as Zn	n.e.	n.e.	n.e.	n.e.	n.e.

Table 5: comparison of determinations to the reference test methods

For results between brackets no z-scores are calculated.

The following performance categories were used:

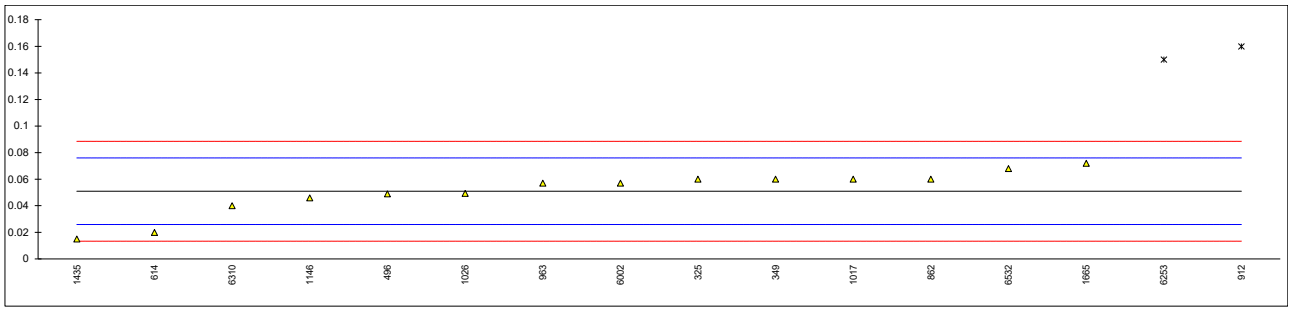
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test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

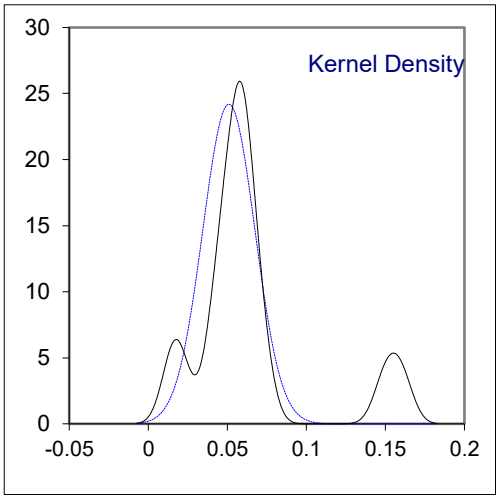
APPENDIX 1

Determination of Total Acid Number on sample #23071; results in mg KOH/g

lab	method	value	mark	z(targ)	End point determination	Volume titration solvent
178		-----		-----	---	---
179		-----		-----	---	---
237	D664-B	<0.1		-----	Inflection Point	125 mL
325	D664-A	0.06		0.72	Buffer End Point pH 10	125 mL
349	D664-A	0.06		0.72	Buffer End Point pH 10	60 mL
432		-----		-----	---	---
496	D664-A	0.049		-0.16	Buffer End Point pH 10	60 mL
614	D664-A	0.02		-2.47	---	---
862	D664-A	0.06		0.72	Inflection Point	60 mL
912	D664-A	0.16	DG(0.01)	8.72	---	---
963	D974	0.057		0.48	---	---
1017	D664-A	0.06		0.72	Inflection Point	125 mL
1026	D664-A	0.0493		-0.13	Buffer End Point pH 10	125 mL
1146	D664-A	0.046		-0.40	Buffer End Point pH 10	125 mL
1184		-----		-----	---	---
1244		-----		-----	---	---
1435	D664-A	0.015		-2.87	Buffer End Point pH 10	---
1665	D664-A	0.072		1.68	Inflection Point	60 mL
1740	D664-A	<0.05		-----	Inflection Point	60 mL
6002	D664-A	0.057		0.48	Inflection Point	60 mL
6016		-----		-----	---	---
6197		-----		-----	---	---
6253	ISO6618	0.15	DG(0.01)	7.92	---	---
6310	D664-A	0.04		-0.88	Buffer End Point pH 10	60 mL
6442		-----		-----	---	---
6532	D664-A	0.068		1.36	Buffer End Point pH 11	60 mL

			BEP only	Inflection Point only
	normality	OK	not OK	not OK
	n	14	8	4
	outliers	2	0	0
	mean (n)	0.0509	0.0484	0.0622
	st.dev. (n)	0.01649	0.01624	0.00665
	R(calc.)	0.0462	0.0455	0.0186
	st.dev.(D664-A:18e2 IP 60ml)	0.01251	-----	0.01474
	R(D664-A:18e2 IP 60ml)	0.0350	-----	0.0413
compare				
	R(D664-A:18e2 BEP 60ml)	0.0303	0.0289	-----
	R(D664-A:18e2 IP 125 ml)	0.0098	-----	0.0121
	R(D664-A:18e2 BEP 125 ml)	0.0138	0.0131	-----

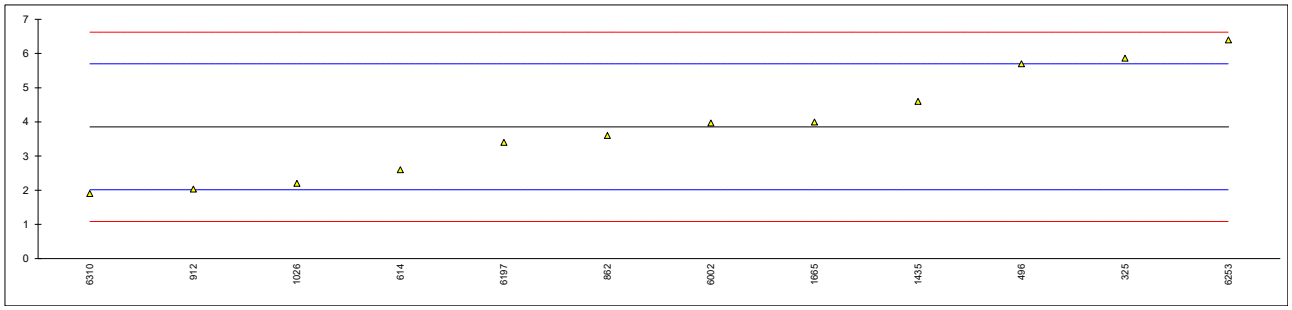


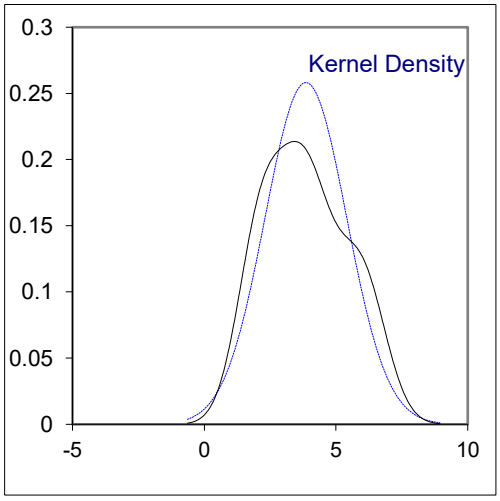


Determination of Air Release time at 50 °C on sample #23071; results in minutes

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
325	D3427	5.8667		2.18	
349		-----		-----	
432		-----		-----	
496	ISO9120	5.7		2.00	
614	D3427	2.6		-1.36	
862	D3427	3.6		-0.28	
912	D3427	2.03		-1.98	
963		-----		-----	
1017		-----		-----	
1026	D3427	2.2		-1.79	
1146		-----		-----	
1184		-----		-----	
1244		-----		-----	
1435	D3427	4.6		0.81	
1665	D3427	4		0.16	
1740		-----		-----	
6002	ISO9120	3.97		0.12	
6016		-----		-----	
6197	D3427	3.4		-0.49	
6253	ISO9120	6.4		2.76	
6310	D3427	1.9		-2.12	
6442		-----		-----	
6532		-----		-----	

normality	OK
n	12
outliers	0
mean (n)	3.856
st.dev. (n)	1.5450
R(calc.)	4.326
st.dev.(D3427:19)	0.9229
R(D3427:19)	2.584

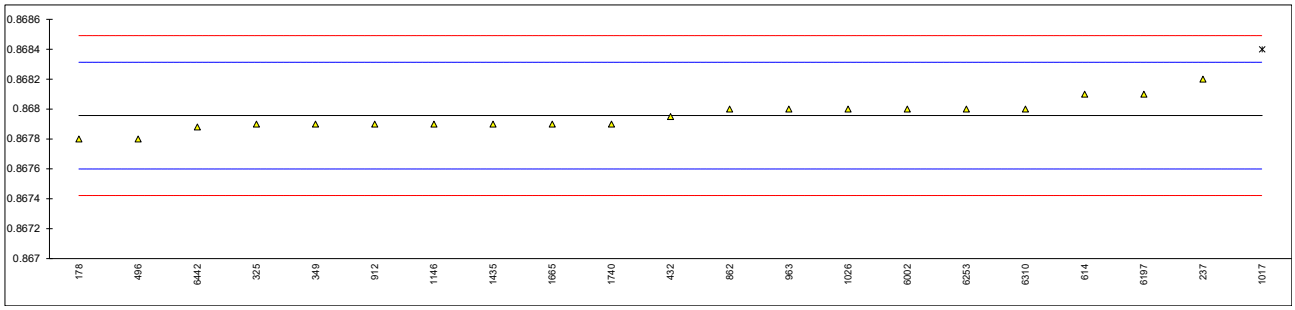


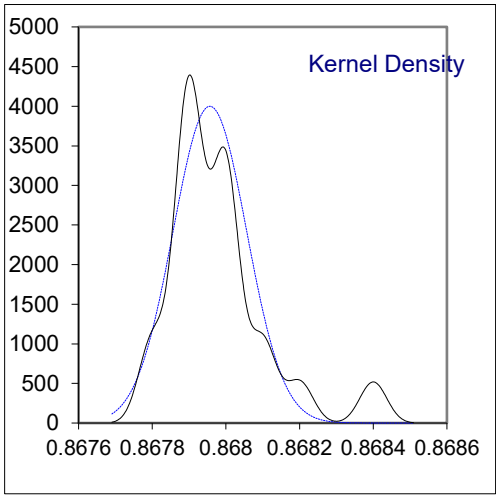


Determination of Density at 15 °C on sample #23071; results in kg/L

lab	method	value	mark	z(targ)	remarks
178	D4052	0.8678		-0.88	
179		-----		-----	
237	D4052	0.8682		1.36	
325	D4052	0.8679		-0.32	
349	D4052	0.8679		-0.32	
432	D4052	0.86795		-0.04	
496	ISO12185	0.8678		-0.88	
614	D4052	0.8681		0.80	
862	D4052	0.8680		0.24	
912	ISO12185	0.8679		-0.32	
963	D4052	0.8680		0.24	
1017	ISO12185	0.8684	R(0.01)	2.48	
1026	D4052	0.8680		0.24	
1146	D4052	0.8679		-0.32	
1184		-----		-----	
1244		-----		-----	
1435	D4052	0.8679		-0.32	
1665	D4052	0.8679		-0.32	
1740	D4052	0.8679		-0.32	
6002	ISO12185	0.8680		0.24	
6016		-----		-----	
6197	D4052	0.8681		0.80	
6253	ISO3675	0.8680	C	0.24	reported 868.0 kg/L
6310	D4052	0.868		0.24	
6442	D4052	0.86788		-0.43	
6532		-----		-----	

normality	OK
n	20
outliers	1
mean (n)	0.86796
st.dev. (n)	0.000100
R(calc.)	0.00028
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

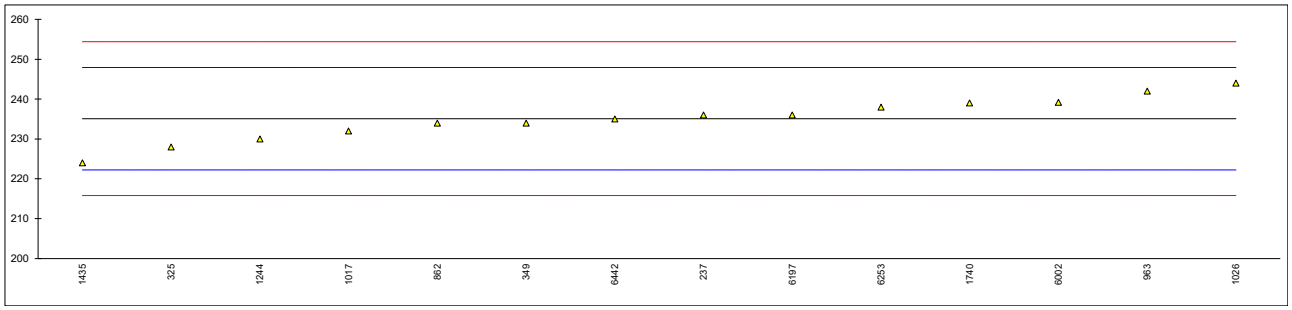


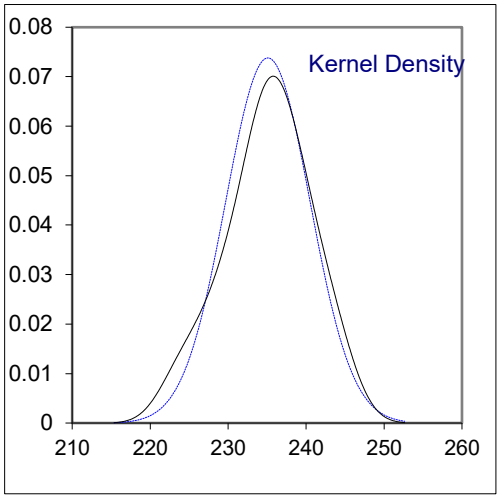


Determination of Flash Point C.O.C. on sample #23071; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D92	236		0.14	
325	D92	228		-1.10	
349	D92	234		-0.17	
432		-----		-----	
496		-----		-----	
614		-----		-----	
862	D92	234		-0.17	
912		-----		-----	
963	D92	242.0		1.08	
1017	D92	232		-0.48	
1026	D92	244		1.39	
1146		-----		-----	
1184		-----		-----	
1244	ISO2592	230.0		-0.79	
1435	D92	224.0		-1.72	
1665		-----	W	-----	test result withdrawn, reported 212 as Flash Point PMcc
1740	D92	239		0.61	
6002	ISO2592	239.2		0.64	
6016		-----		-----	
6197	D92	236		0.14	
6253	ISO2592	238		0.45	
6310		-----		-----	
6442	D92	235		-0.01	
6532		-----		-----	

normality	OK
n	14
outliers	0
mean (n)	235.09
st.dev. (n)	5.410
R(calc.)	15.15
st.dev.(D92:18)	6.429
R(D92:18)	18

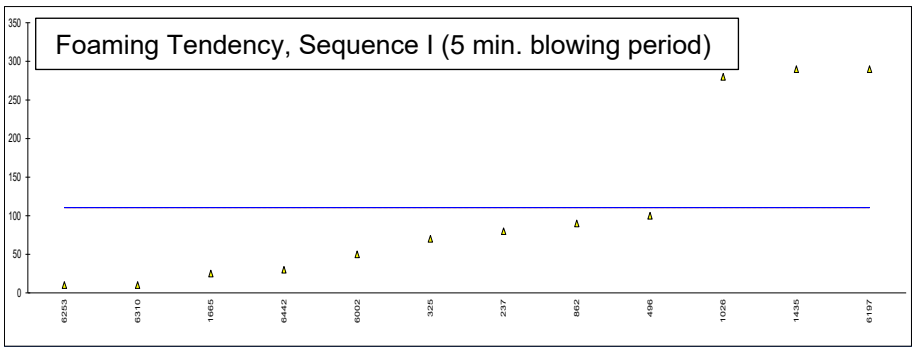


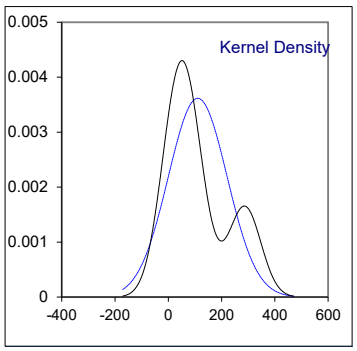


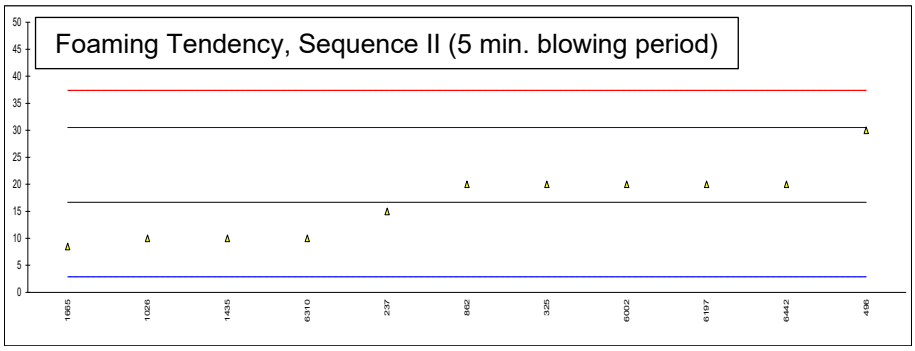
Determination of Foaming Tendency, Sequence I, II and III (5 min. blowing period) on sample #23071; results in mL

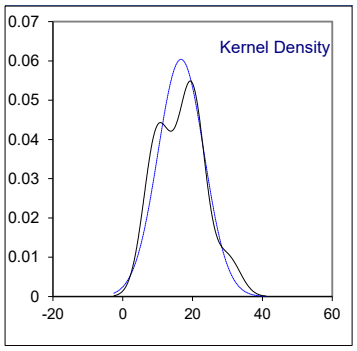
lab	method	Sample used	Diffuser	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
178		---	---	-----		-----	-----		-----	-----		-----
179		---	---	-----		-----	-----		-----	-----		-----
237	D892	As received	Metal	80		-----	15		-0.24	60		-----
325	D892	As received	Metal	70		-----	20		0.48	30		-----
349		---	---	-----		-----	-----		-----	-----		-----
432		---	---	-----		-----	-----		-----	-----		-----
496	D892	As received	Metal	100		-----	30		1.93	200		-----
614		---	---	-----		-----	-----		-----	-----		-----
862	D892	As received	Metal	90		-----	20		0.48	90		-----
912		---	---	-----		-----	-----		-----	-----		-----
963		---	---	-----		-----	-----		-----	-----		-----
1017		---	---	-----		-----	-----		-----	-----		-----
1026	D892	As received	Metal	280		-----	10		-0.97	240		-----
1146		---	---	-----		-----	-----		-----	-----		-----
1184		---	---	-----		-----	-----		-----	-----		-----
1244		---	---	-----		-----	-----		-----	-----		-----
1435	D892	After agitation	Metal	290		-----	10		-0.97	230		-----
1665	D892	After agitation	Stone	25		-----	8.5		-1.18	43		-----
1740		---	---	-----		-----	-----		-----	-----		-----
6002	ISO6247	After agitation	Metal	50		-----	20		0.48	30		-----
6016		---	---	-----		-----	-----		-----	-----		-----
6197	D892	As received	Metal	290		-----	20		0.48	220		-----
6253	ISO6247	As received	---	10		-----	-----		-----	-----		-----
6310	D892	After agitation	Metal	10		-----	10		-0.97	20		-----
6442	D892	After agitation	---	30		-----	20		0.48	20		-----
6532		---	---	-----		-----	-----		-----	-----		-----

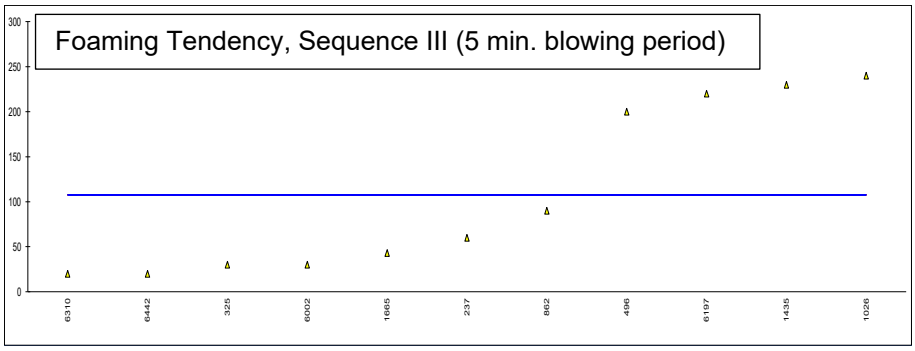
normality	suspect	OK	OK
n	12	11	11
outliers	0	0	0
mean (n)	110.42	16.68	107.55
st.dev. (n)	110.340	6.604	93.714
R(calc.)	308.95	18.49	262.40
st.dev.(D892:18)	(16.508)	6.907	(24.002)
R(D892:18)	(46.22)	19.34	(67.21)

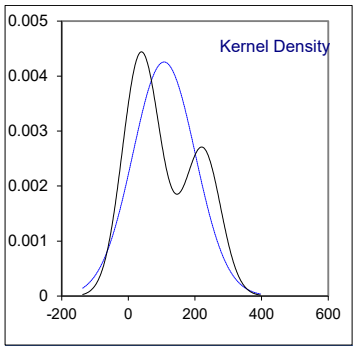

Foaming Tendency, Sequence I (5 min. blowing period)
350
300
250
200
150
100
50
0
6253
6310
1665
6442
6002
325
237
862
496
1026
1435
6197


Kernel Density
0.005
0.004
0.003
0.002
0.001
0
-400
-200
0
200
400
600


Foaming Tendency, Sequence II (5 min. blowing period)
50
45
40
35
30
25
20
15
10
5
0
1665
1026
1435
6310
237
862
325
6002
6197
6442
496


Kernel Density
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
-20
0
20
40
60


Foaming Tendency, Sequence III (5 min. blowing period)
300
250
200
150
100
50
0
6310
6442
325
6002
1665
237
862
496
6197
1435
1026


Kernel Density
0.005
0.004
0.003
0.002
0.001
0
-200
0
200
400
600

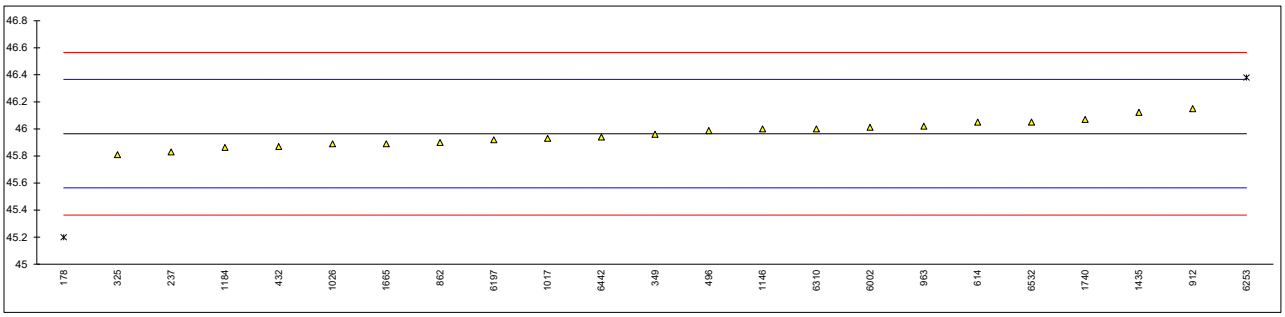
Determination of Foam Stability, Sequence I, II and III (10 min. settling period) on sample #23071; results in mL

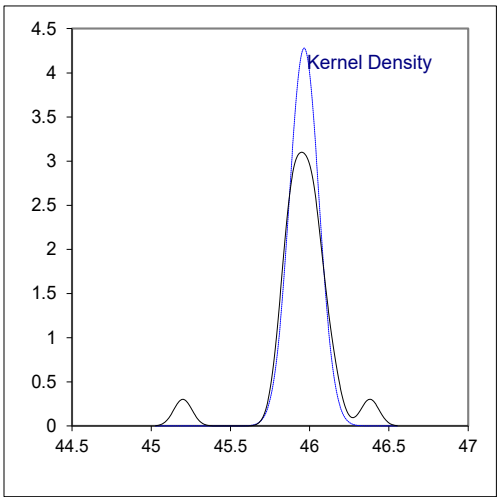
lab	method	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179		-----		-----	-----		-----	-----		-----
237	D892	0		-----	0		-----	0		-----
325	D892	0		-----	0		-----	0		-----
349		-----		-----	-----		-----	-----		-----
432		-----		-----	-----		-----	-----		-----
496	D892	0		-----	0		-----	0		-----
614		-----		-----	-----		-----	-----		-----
862		0		-----	0		-----	0		-----
912		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
1017		-----		-----	-----		-----	-----		-----
1026	D892	10	f+?	-----	0		-----	10	f+?	-----
1146		-----		-----	-----		-----	-----		-----
1184		-----		-----	-----		-----	-----		-----
1244		-----		-----	-----		-----	-----		-----
1435	D892	0		-----	0		-----	0		-----
1665	D892	0		-----	0		-----	0		-----
1740		-----		-----	-----		-----	-----		-----
6002	ISO6247	0		-----	0		-----	0		-----
6016		-----		-----	-----		-----	-----		-----
6197	D892	0		-----	0		-----	0		-----
6253	ISO6247	0		-----	-----		-----	-----		-----
6310	D892	0		-----	0		-----	0		-----
6442	D892 (Alternative)	0		-----	0		-----	0		-----
6532		-----		-----	-----		-----	-----		-----
	n	11			11			10		
	mean (n)	0			0			0		

Determination of Kinematic Viscosity at 40 °C on sample #23071; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178	D7279 corr. to D445	45.2	C,R(0.01)	-3.82	first reported 41.5
179		-----		-----	
237	D445	45.83		-0.67	
325	D445	45.81		-0.77	
349	D445	45.96		-0.02	
432	D445	45.87		-0.47	
496	D445	45.988		0.11	
614	D7042	46.05		0.42	
862	D445	45.90		-0.32	
912	D445	46.15		0.92	
963	D445	46.02		0.27	
1017	D445	45.93		-0.17	
1026	D445	45.89	C	-0.37	first reported 45.59
1146	D445	46		0.17	
1184	D445	45.863		-0.51	
1244		-----		-----	
1435	D7042	46.122		0.78	
1665	D445	45.89		-0.37	
1740	D445	46.07		0.52	
6002	ISO3104	46.0121		0.24	
6016		-----		-----	
6197	D445	45.92		-0.22	
6253	ISO3104	46.38	R(0.01)	2.07	
6310	D7279 corr. to D445	46.0		0.17	
6442	D7042	45.94		-0.12	
6532	D445	46.05		0.42	

normality	OK
n	21
outliers	2
mean (n)	45.9650
st.dev. (n)	0.09317
R(calc.)	0.2609
st.dev.(D445:21e2)	0.20028
R(D445:21e2)	0.5608

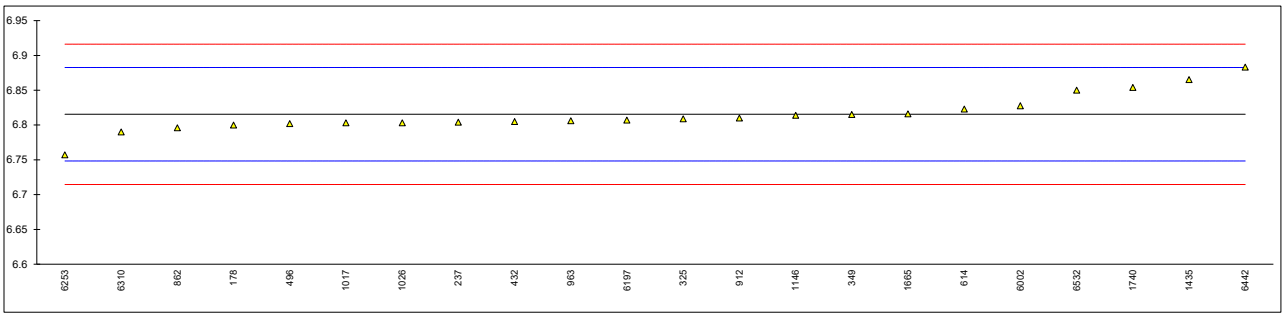


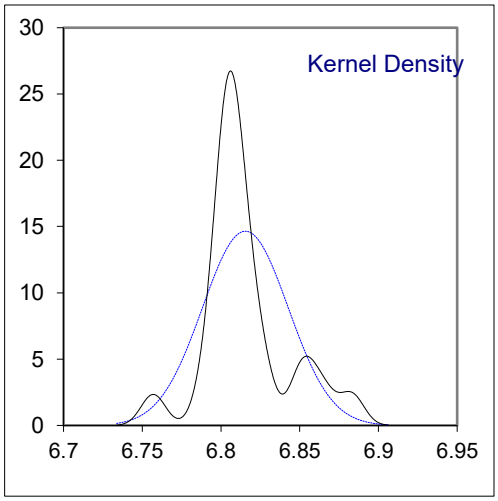


Determination of Kinematic Viscosity at 100 °C on sample #23071; results in mm²/s

lab	method	value	mark	z(targ)	remarks
178	D7279 corr. to D445	6.80	C	-0.46	first reported 6.51
179		-----		-----	
237	D445	6.804		-0.34	
325	D445	6.809		-0.19	
349	D445	6.815		-0.01	
432	D445	6.805		-0.31	
496	D445	6.8021		-0.40	
614	D7042	6.823		0.22	
862	D445	6.796		-0.58	
912	D445	6.810		-0.16	
963	D445	6.806	C	-0.28	first reported 6.725
1017	D445	6.803		-0.37	
1026	D445	6.803		-0.37	
1146	D445	6.814		-0.04	
1184		-----		-----	
1244		-----		-----	
1435	D7042	6.8652		1.48	
1665	D445	6.816		0.02	
1740	D445	6.854		1.15	
6002	ISO3104	6.8277		0.36	
6016		-----		-----	
6197	D445	6.807		-0.25	
6253	ISO3104	6.757		-1.74	
6310	D7279 corr. to D445	6.79		-0.76	
6442	D7042	6.883		2.01	
6532	D445	6.85		1.03	

normality	suspect
n	22
outliers	0
mean (n)	6.8155
st.dev. (n)	0.02723
R(calc.)	0.0762
st.dev.(D445:21e2)	0.03359
R(D445:21e2)	0.0941

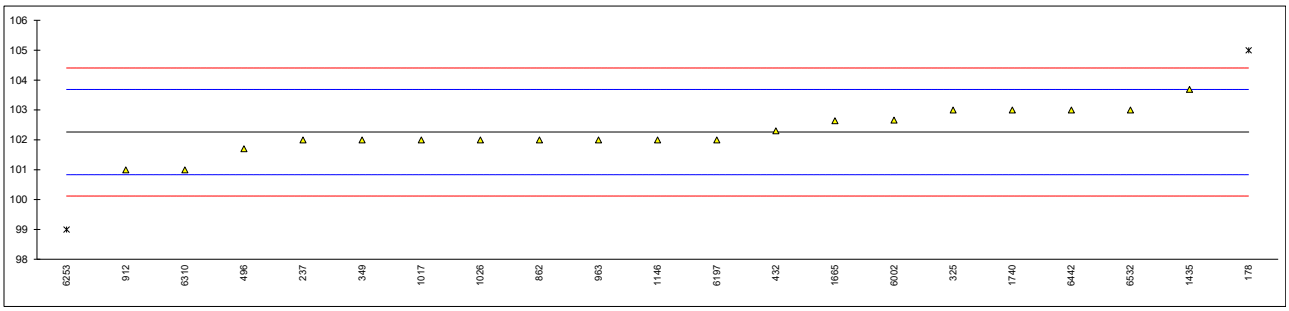


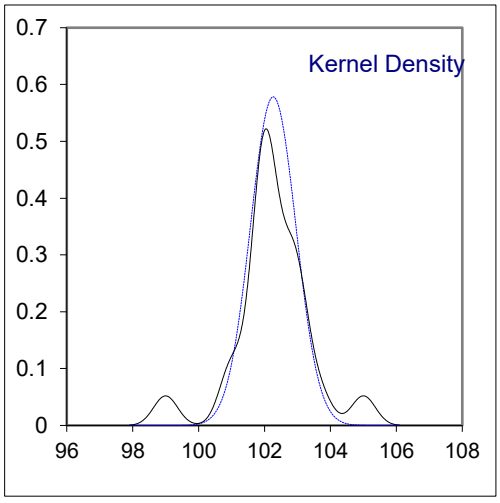


Determination of Viscosity Index on sample #23071

lab	method	value	mark	z(targ)	remarks
178	D2270	105	ex,C	3.83	ex = statistical outlier in KV 40 °C, first reported 108
179		-----		-----	
237	D2270	102		-0.37	
325	D2270	103		1.03	
349	D2270	102		-0.37	
432	D2270	102.3		0.05	
496	D2270	101.7		-0.79	
614		-----		-----	
862	D2270	102		-0.37	
912	D2270	101		-1.77	
963	D2270	102	C	-0.37	first reported 98
1017	D2270	102		-0.37	
1026	D2270	102		-0.37	
1146	D2270	102		-0.37	
1184		-----		-----	
1244		-----		-----	
1435	D2270	103.69		2.00	
1665	D2270	102.641		0.53	
1740	D2270	103		1.03	
6002	ISO2909	102.66		0.56	
6016		-----		-----	
6197	D2270	102		-0.37	
6253	ISO2909	99	ex,E	-4.57	ex= statistical outlier in KV 40 °C, calc. diff. iis calculated 98
6310	D2270	101		-1.77	
6442	D2270	103	E	1.03	calc. difference, iis calc. 105
6532	D2270	103		1.03	

normality	OK
n	19
outliers	0+2ex
mean (n)	102.26
st.dev. (n)	0.690
R(calc.)	1.93
st.dev.(D2270:10R16)	0.714
R(D2270:10R16)	2

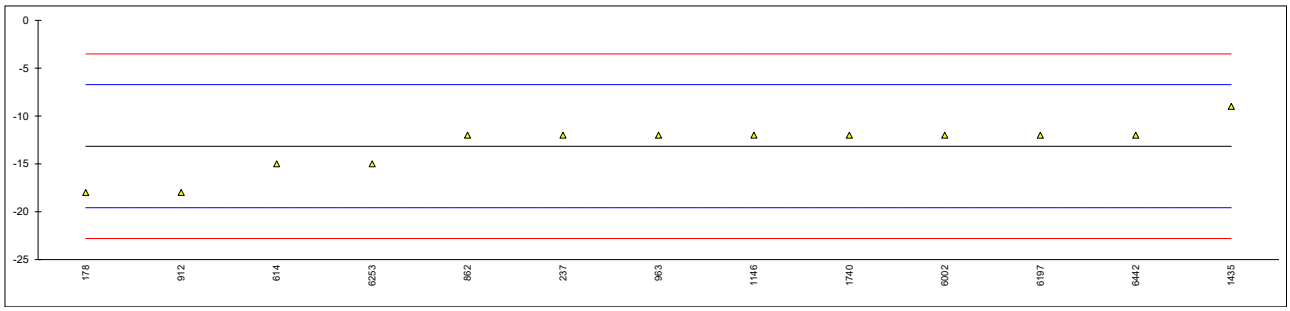


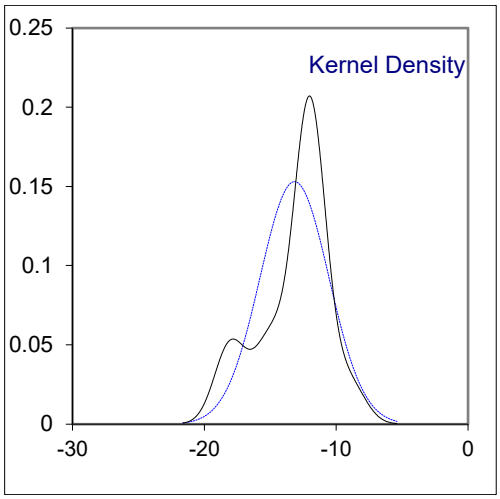


Determination of Pour Point Manual on sample #23071; results in °C

lab	method	value	mark	z(targ)	remarks
178	D97	-18		-1.51	
179		-----		-----	
237	D97	-12		0.36	
325		-----		-----	
349		-----		-----	
432		-----		-----	
496		-----		-----	
614	D97	-15		-0.57	
862	D97	-12		0.36	
912	D97	-18		-1.51	
963	D97	-12		0.36	
1017		-----		-----	
1026		-----		-----	
1146	D97	-12		0.36	
1184		-----		-----	
1244		-----		-----	
1435	ISO3016	-9		1.29	
1665		-----		-----	
1740	D97	-12		0.36	
6002	ISO3016	-12		0.36	
6016		-----		-----	
6197	D97	-12		0.36	
6253	NF T60-105	-15		-0.57	
6310		-----		-----	
6442	D97	-12		0.36	
6532		-----		-----	

normality	OK
n	13
outliers	0
mean (n)	-13.15
st.dev. (n)	2.609
R(calc.)	7.31
st.dev.(D97:17bR22)	3.214
R(D97:17bR22)	9

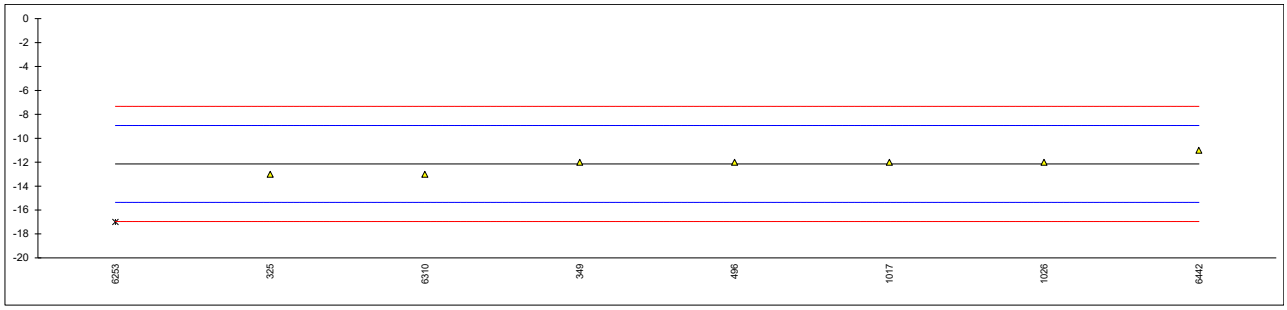


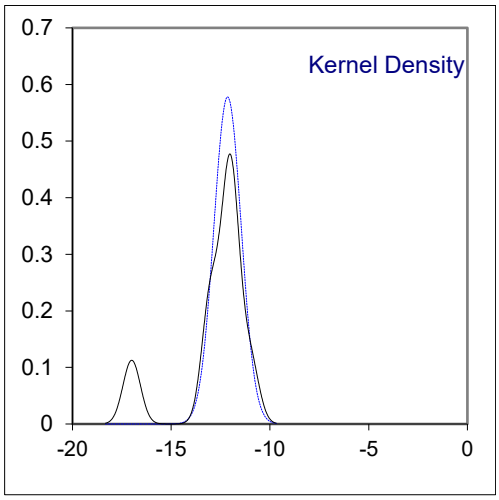


Determination of Pour Point Automated 1 °C interval on sample #23071; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
325	D5950	-13		-0.53	
349	D5950	-12		0.09	
432		-----		-----	
496	D5950	-12		0.09	
614		-----		-----	
862		-----		-----	
912		-----		-----	
963		-----		-----	
1017	D5950	-12		0.09	
1026	D5950	-12		0.09	
1146		-----		-----	
1184		-----		-----	
1244		-----		-----	
1435		-----		-----	
1665		-----		-----	
1740		-----		-----	
6002		-----		-----	
6016		-----		-----	
6197		-----		-----	
6253	NF T60-105	-17	G(0.01)	-3.02	
6310	D5950	-13		-0.53	
6442	D6892	-11		0.71	
6532		-----		-----	

normality	unknown
n	7
outliers	1
mean (n)	-12.14
st.dev. (n)	0.690
R(calc.)	1.93
st.dev.(D5950:14R20)	1.607
R(D5950:14R20)	4.5

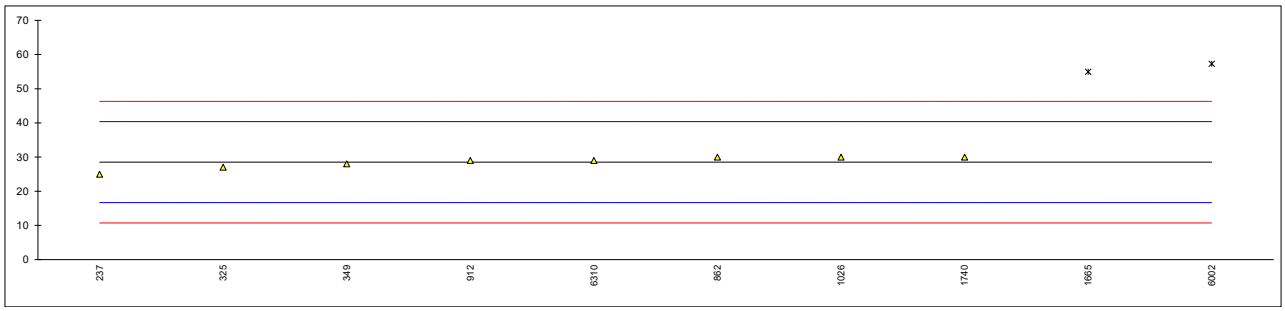


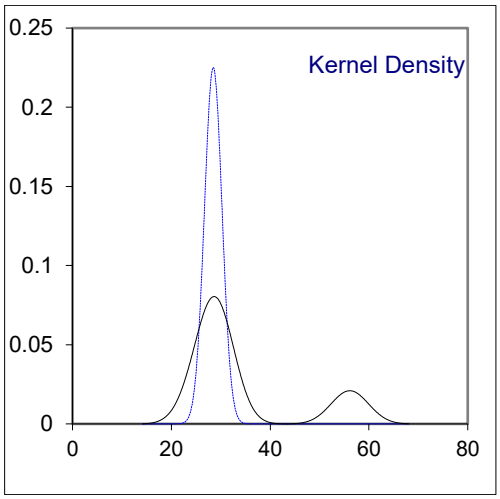


Determination of Sulfur on sample #23071; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D4294	25		-0.59	
325	INH-D6443/D4927	27		-0.25	
349	D2622	28		-0.08	
432		-----		-----	
496		-----		-----	
614		-----		-----	
862	D2622	30		0.25	
912	D4294	29		0.08	
963		-----		-----	
1017		-----		-----	
1026	D5185	30		0.25	
1146		-----		-----	
1184		-----		-----	
1244		-----		-----	
1435		-----		-----	
1665	ISO19579	55	DG(0.01)	4.46	
1740	D4294	30		0.25	
6002	D5185	57.3	DG(0.01)	4.85	
6016		-----		-----	
6197		-----		-----	
6253		-----		-----	
6310	D7751	29		0.08	
6442		-----		-----	
6532		-----		-----	

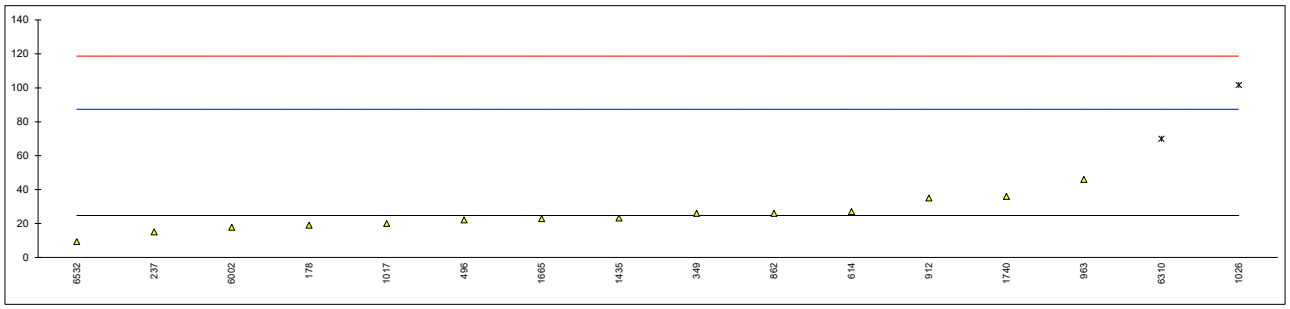
normality	suspect
n	8
outliers	2
mean (n)	28.50
st.dev. (n)	1.773
R(calc.)	4.96
st.dev.(D4294:21)	5.936
R(D4294:21)	16.62

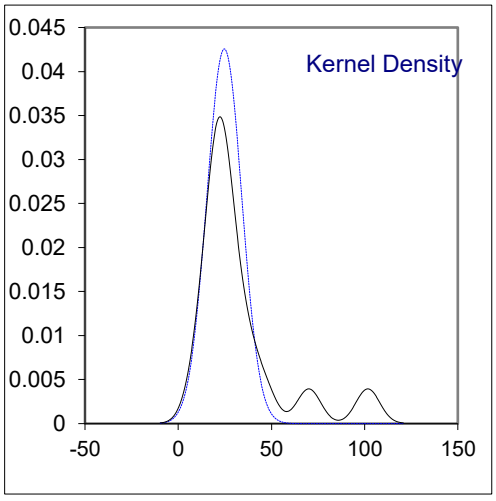




Determination of Water on sample #23071; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D6304-B:20	19		-0.18	
179		-----		-----	
237	D6304-C:16e1	15		-0.31	
325	D6304-C:20	<100		-----	
349	D6304-A:20	26		0.04	
432		-----		-----	
496	D6304-B:20	22.1		-0.08	
614	D6304-B:20	27		0.07	
862	D6304-A	26		0.04	
912	D6304	35		0.33	
963	D6304-A:16e1	46		0.68	
1017	ISO12937	20		-0.15	
1026	D6304-C:20	101.69	G(0.01)	2.46	
1146	D6304-B:20	<100		-----	
1184		-----		-----	
1244		-----		-----	
1435	D6304-A:20	23.2		-0.05	
1665	D6304-A:20	22.9		-0.06	
1740	D6304-B:20	36	C	0.36	first reported 106
6002	In house	17.74		-0.22	
6016		-----		-----	
6197		-----		-----	
6253		-----		-----	
6310	D6304-B:16e1	70	C,G(0.01)	1.45	first reported 76.4
6442		-----		-----	
6532	ISO12937	9.35		-0.49	
	normality	OK			
	n	14			
	outliers	2			
	mean (n)	24.66			
	st.dev. (n)	9.369			
	R(calc.)	26.23			
	st.dev.(D6304-B:20)	31.353			range 30-2100 mg/kg
	R(D6304-B:20)	87.79			
Compare					
	R(D6304-A:20)	22.42			range 20-25000 mg/kg
	R(D6304-C:20)	14.46			range 20-360 mg/kg



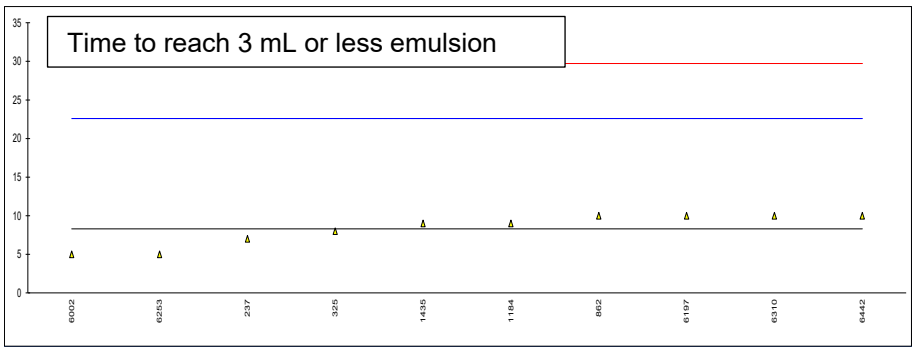


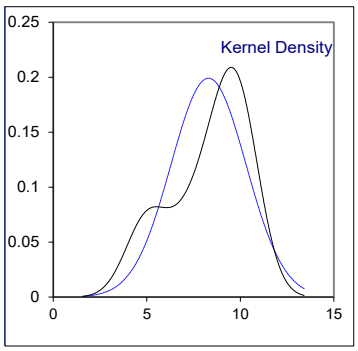
Determination of Water Separability at 54 °C, distilled water on sample #23071; results in minutes

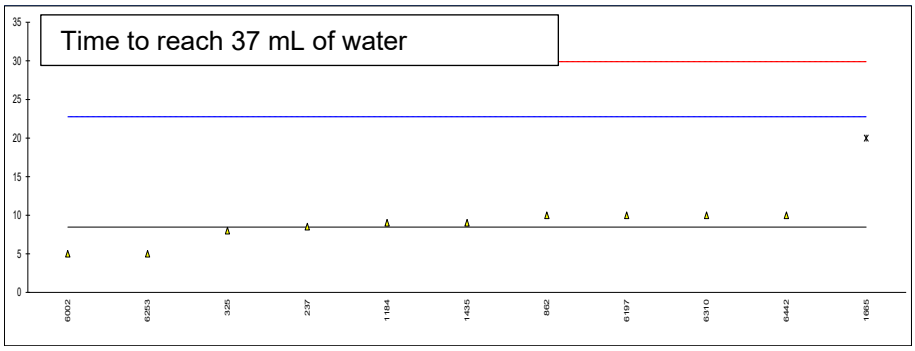
lab	method	3 mL or less emulsion	mark	z(targ)	37 mL of water	mark	z(targ)	complete break (40-40-0)	mark	z(targ)	test aborted	time test aborted
178		-----		-----	-----		-----	-----		-----	-----	-----
179		-----		-----	-----		-----	-----		-----	-----	-----
237	D1401	7.0		-0.18	8.5		0.01	13.6		0.19	No	-----
325	D1401	8		-0.04	8		-0.06	9		-0.45	No	9
349		-----		-----	-----		-----	-----		-----	-----	-----
432		-----		-----	-----		-----	-----		-----	-----	-----
496		-----		-----	-----		-----	10		-0.31	No	-----
614		-----		-----	-----		-----	-----		-----	-----	-----
862	D1401	10		0.24	10		0.22	-----		-----	-----	10
912		-----		-----	-----		-----	15		0.39	-----	-----
963		-----		-----	-----		-----	-----		-----	-----	-----
1017		-----		-----	-----		-----	-----		-----	-----	-----
1026		-----		-----	-----		-----	10		-0.31	Yes	10
1146		-----		-----	-----		-----	-----		-----	Yes	30
1184	D1401	9		0.10	9		0.08	13		0.11	-----	-----
1244		-----		-----	-----		-----	-----		-----	-----	-----
1435	D1401	9		0.10	9		0.08	-----		-----	No	-----
1665		-----		-----	20	G(1)	1.62	20		1.09	No	-----
1740		-----		-----	-----		-----	-----		-----	-----	-----
6002	ISO6614	5		-0.46	5		-0.48	5		-1.01	No	5
6016		-----		-----	-----		-----	-----		-----	-----	-----
6197	D1401	10		0.24	10		0.22	15		0.39	No	-----
6253	ISO6614	5		-0.46	5		-0.48	6		-0.87	-----	-----
6310	D1401	10	C	0.24	10	C	0.22	20		1.09	No	-----
6442		10		0.24	10		0.22	10		-0.31	No	-----
6532		-----		-----	-----		-----	-----		-----	-----	-----

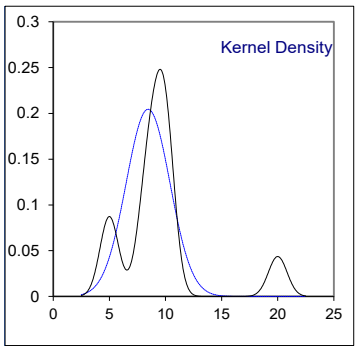
normality	OK	OK	OK
n	10	10	12
outliers	0	1	0
mean (n)	8.30	8.45	12.22
st.dev. (n)	2.003	1.950	4.815
R(calc.)	5.61	5.46	13.48
st.dev.(D1401:21)	7.143	7.143	7.143
R(D1401:21)	20	20	20

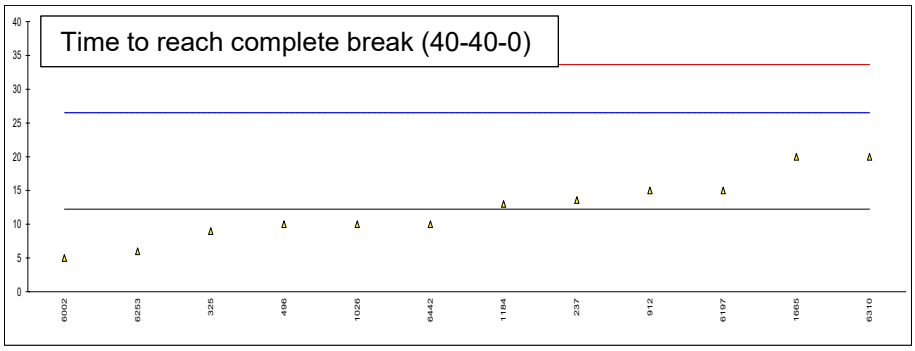
Lab 6310 first reported 20 min for time to reach 3 mL or less emulsion and 20 min for time to reach 37 mL of water

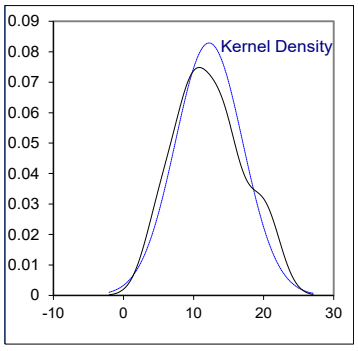

Time to reach 3 mL or less emulsion
6002
6253
237
325
1435
1184
862
6197
6310
6442


Kernel Density


Time to reach 37 mL of water
6002
6253
325
237
1184
1435
862
6197
6310
6442
1665


Kernel Density


Time to reach complete break (40-40-0)
6002
6253
325
496
1026
6442
1184
237
912
6197
1665
6310


Kernel Density

Determination of Water Separability at 54 °C, distilled water on sample #23071; results in mL

lab	method	oil phase	mark	z(targ)	water phase	mark	z(targ)	emulsion phase	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179		-----		-----	-----		-----	-----		-----
237	D1401	40.0		-----	40.0		-----	0		-----
325	D1401	40		-----	40		-----	0		-----
349		-----		-----	-----		-----	-----		-----
432		-----		-----	-----		-----	-----		-----
496	D1401	40		-----	40		-----	0		-----
614		-----		-----	-----		-----	-----		-----
862	D1401	40		-----	37		-----	3		-----
912	D1401	40		-----	40		-----	0		-----
963		-----		-----	-----		-----	-----		-----
1017		-----		-----	-----		-----	-----		-----
1026	D1401	40		-----	40		-----	0		-----
1146	D1401	0		-----	36		-----	44		-----
1184	D1401	43		-----	36		-----	1		-----
1244		-----		-----	-----		-----	-----		-----
1435		-----		-----	-----		-----	-----		-----
1665	D1401	43		-----	37		-----	0		-----
1740		-----		-----	-----		-----	-----		-----
6002	ISO6614	40		-----	40		-----	0		-----
6016		-----		-----	-----		-----	-----		-----
6197		-----		-----	-----		-----	-----		-----
6253		-----		-----	-----		-----	-----		-----
6310		-----		-----	-----		-----	-----		-----
6442		-----		-----	-----		-----	-----		-----
6532		-----		-----	-----		-----	-----		-----

Determination of Calcium as Ca on sample #23071; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	5		-----	
179		-----		-----	
237	D5185	<40		-----	
325	D5185	<1		-----	
349		0		-----	
432	D5185	<40		-----	
496	D5185	1.784		-----	
614	D5185	<1		-----	
862	D5185	<1		-----	
912	D5185	<1		-----	
963		<0.10		-----	
1017		-----		-----	
1026	D5185	0.1451		-----	
1146		<5		-----	
1184		-----		-----	
1244		-----		-----	
1435	D5185	0.081		-----	
1665		-----		-----	
1740	D5185	<1		-----	
6002	D5185	0.07		-----	
6016		-----		-----	
6197	D4951	5.99		-----	
6253		-----		-----	
6310	D7751	<1		-----	
6442	D5185	<10		-----	
6532		-----		-----	
	n	18			
	mean (n)	<40			

Determination of Phosphorus as P on sample #23071; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	3		-----	
179		-----		-----	
237	D5185	<10		-----	
325	D5185	<2		-----	
349		0		-----	
432	D5185	<10		-----	
496	D5185	1.112		-----	
614	D5185	<2		-----	
862	D5185	<1		-----	
912	D5185	<1		-----	
963		<0.10		-----	
1017		-----		-----	
1026	D5185	0.2986		-----	
1146		<10		-----	
1184		-----		-----	
1244		-----		-----	
1435	D5185	0		-----	
1665	D5185	5.11		-----	
1740	D5185	2		-----	
6002	D5185	0.597		-----	
6016		-----		-----	
6197	D4951	4.64		-----	
6253		-----		-----	
6310	D7751	<1		-----	
6442	D5185	<10		-----	
6532		-----		-----	
	n	19			
	mean (n)	<10			

Determination of Zinc as Zn on sample #23071; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	1		-----	
179		-----		-----	
237	D5185	<60		-----	
325	D5185	<1		-----	
349		0		-----	
432	D5185	<60		-----	
496	D5185	0.941		-----	
614	D5185	<1		-----	
862	D5185	<1		-----	
912	D5185	<1		-----	
963		0.14		-----	
1017		-----		-----	
1026	D5185	0.1119		-----	
1146		<5		-----	
1184		-----		-----	
1244		-----		-----	
1435	D5185	0.075		-----	
1665	D5185	0.38		-----	
1740	D5185	<1		-----	
6002	D5185	0.318		-----	
6016		-----		-----	
6197	D4951	2.14		-----	
6253		-----		-----	
6310	D7751	1		-----	
6442	D5185	<10		-----	
6532		-----		-----	
	n	19			
	mean (n)	<60			

APPENDIX 2

Number of participants per country

1 lab in	AUSTRALIA
1 lab in	AUSTRIA
4 labs in	BELGIUM
1 lab in	CHINA, People's Republic
1 lab in	FRANCE
1 lab in	GERMANY
1 lab in	GREECE
1 lab in	INDIA
1 lab in	KAZAKHSTAN
2 labs in	KOREA, Republic of
1 lab in	MOROCCO
2 labs in	NETHERLANDS
1 lab in	NIGERIA
1 lab in	POLAND
1 lab in	PORTUGAL
1 lab in	SAUDI ARABIA
1 lab in	SINGAPORE
2 labs in	SPAIN
2 labs in	UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations

C = final test result after checking of first reported suspect test result
D(0.01) = outlier in Dixon's outlier test
D(0.05) = straggler in Dixon's outlier test
G(0.01)/G(1)= outlier in Grubbs' outlier test
G(0.05) = straggler in Grubbs' outlier test
DG(0.01) = outlier in Double Grubbs' outlier test
DG(0.05) = straggler in Double Grubbs' outlier test
R(0.01) = outlier in Rosner's outlier test
R(0.05) = straggler in Rosner's outlier test
E = calculation difference between reported test result and result calculated by iis
W = test result withdrawn on request of participant
ex = test result excluded from statistical evaluation
n.a. = not applicable
n.e. = not evaluated
n.d. = not detected
fr. = first reported
f+? = possibly a false positive test result?
f-? = possibly a false negative test result?
SDS = Safety Data Sheet

Literature

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