Institute for
Interlaboratory Studies

Results of Proficiency Test
Hydraulic Oil (fresh)
November 2023

Organized by: Institute for Interlaboratory Studies
Spijkenisse, The Netherlands

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1 INTRODUCTION

Since 2014 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Hydraulic Oil (fresh) every year. During the annual proficiency testing program of 2023 it was decided to continue the round robin for the analysis of Hydraulic Oil (fresh).

In this interlaboratory study 38 laboratories in 27 countries registered for participation, see appendix 2 for the number of participants per country. In this report the results of the Hydraulic 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 a laboratory that has performed the tests in accordance with for ISO/IEC17043 relevant requirements of ISO/IEC17025.
It was decided to send one sample Hydraulic Oil (fresh) in a 1-liter amber glass bottle labelled #23215.
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 200 liters of fresh Hydraulic Oil was obtained from a local supplier. After homogenization 65 amber glass bottles of 1 L were filled and labelled #23215.
The homogeneity of the subsamples was checked by determination of Density at 15 °C in accordance with ISO12185 and Kinematic Viscosity at 40 °C in accordance with ASTM D445 on 8 stratified randomly selected subsamples.

	Density at 15 °C in kg/L	Kinematic Viscosity at 40 °C in mm^2/s
sample #23215-1	0.86063	65.98
sample #23215-2	0.86063	65.99
sample #23215-3	0.86063	66.02
sample #23215-4	0.86063	65.98
sample #23215-5	0.86061	66.00
sample #23215-6	0.86063	65.99
sample #23215-7	0.86063	66.03
sample #23215-8	0.86063	65.99

Table 1: homogeneity test results of subsamples #23215

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibility of the reference test methods in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Density at 15 °C in kg/L	Kinematic Viscosity at 40 °C in mm^2/s
r (observed)	0.00002	0.05
reference test method	ISO12185:96	D445:23
0.3 x R (reference test method)	0.00015	0.24

Table 2: evaluation of the repeatabilities of subsamples #23215

The calculated repeatabilities are in agreement with 0.3 times the corresponding reproducibility of the reference test methods. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one 1 L bottle of Hydraulic Oil (fresh) labelled #23215 was sent on October 11, 2023. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of fresh Hydraulic Oil packed in amber glass bottles was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYZES

The participants were requested to determine: Total Acid Number, Copper Corrosion 3 hrs at 50 °C, Density at 15 °C, Flash Point PMcc, Foaming Characteristics (Foaming Tendency, Foam Stability), Kinematic Viscosity at 40 °C and at 100 °C, Viscosity Index, Kinematic Viscosity Stabinger at 40 °C and at 100 °C, 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 determination of Total Acid Number (ASTM D664).

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 some problems were encountered with the dispatch of the samples. Four 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 36 participants reported 465 numerical test results. Observed were 23 outlying test results, which is 4.9%. 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 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: The group of participants met the target requirements. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of Inflection Point at titration volume 60 mL and Buffer End Point at titration volume 60 mL and 125 mL of ASTM D664-A:18e2, but not with Inflection Point at titration volume 125 mL.

Copper Corrosion: All reporting participants agreed on a test result of 1 (1A).

Density at 15 °C: The group of participants met the target requirements. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO12185:96.

Flash Point PMcc: The group of participants met the target requirements. No statistical outliers were observed but one test result was excluded. The calculated reproducibility after rejection of the suspect data is in agreement with the requirements of ASTM D93-A:20.

Foaming Characteristics (Tendency and Stability): The group of participants had difficulty to meet the target requirements. No statistical outliers were observed over the Foaming Tendency parameters. The calculated reproducibilities of sequence I, II and III are not in agreement with the requirements of ASTM D892:23. It was decided not to calculate z-scores for Foaming Tendency sequence III due to the large difference between the calculated and reference reproducibility.
All reporting participants reported 0 mL for Foam Stability.

Kinematic Viscosity at 40 °C: The group of participants met the target requirements. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D445:23.

Kinematic Viscosity at 100 °C: The group of participants had difficulty to meet the target requirements. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D445:23.

Viscosity Index: The group of participants had difficulty to meet the target requirements. One statistical outlier was observed and three other test results were excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of ASTM D2270:10R16.

Kinematic Viscosity Stabinger at 40 °C: The group of participants met the target requirements. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D7042:21a.

Kinematic Viscosity Stabinger at 100 °C: The group of participants met the target requirements. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D7042:21a.

Pour Point Manual: The group of participants had difficulty to meet the target requirements. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D97:17bR22.

Pour Point Automated 1 °C interval: The group of participants met the target requirements. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D5950:14R20.

Sulfur: The group of participants met the target requirements. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D4294:21.

Water: The group of participants met the target requirements. 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: The group of participants met the target requirements. One statistical outlier was observed over three parameters. The calculated reproducibilities for ≤3 mL emulsion and complete break after rejection of the statistical outlier are in agreement with the requirements of ASTM D1401:21, but not for 37 mL water.

Calcium as Ca: The group of participants may have had difficulty to meet the target requirements. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated reproducibility calculated with the Horwitz equation, and not at all with the strict requirements of ASTM D5185:18.

Phosphorus as P: The group of participants met the target requirements. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D5185:18.

Zinc as Zn: The group of participants had difficulty to meet the target requirements. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D5185:18.

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	25	0.43	0.14	0.20
Copper Corrosion 3 hrs at 50 °C		21	1 (1A)	n.a.	n.a.
Density at 15 °C	kg/L	30	0.8607	0.0005	0.0005
Flash Point PMcc	°C	26	229.5	14.0	16.3
Foaming Tendency Seq. I	mL	15	17.2	40.6	20.9
Foaming Tendency Seq. II	mL	14	9.3	27.4	13.9
Foaming Tendency Seq. III	mL	14	4.1	14.9	(1.8)
Foam Stability Seq. I	mL	15	0	n.e.	n.e.
Foam Stability Seq. II	mL	14	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	66.155	0.498	0.807
Kinematic Viscosity at 100 °C	mm^2/s	27	9.112	0.150	0.126
Viscosity Index		26	113.83	2.80	2
Kin.Viscosity Stabinger at 40 °C	mm^2/s	17	66.195	0.609	0.835
Kin.Viscosity Stabinger at 100 °C	mm^2/s	16	9.127	0.087	0.107
Pour Point Manual	°C	16	-36.3	11.0	9
Pour Point Automated 1 °C int.	°C	7	-40.4	4.2	4.5
Sulfur	mg/kg	17	643	104	124
Water	mg/kg	25	44.0	47.2	110.2
Water Separability at 54 °C, distilled water					
Time ≤ 3 mL emulsion	minutes	11	23.4	19.5	20
Time 37 mL water	minutes	13	25.7	23.6	20
Complete Break (40-40-0)	minutes	9	28.5	20.6	20
Calcium as Ca	mg/kg	18	12.5	4.8	3.8

Parameter	unit	n	average	2.8 * sd	R(lit)
Phosphorus as P	mg/kg	20	283	57	72
Zinc as Zn	mg/kg	22	340	86	51

Table 3: reproducibilities of tests on sample #23215

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 NOVEMBER 2023 WITH PREVIOUS PTS

	November 2023	November 2022	November 2021	November 2020	November 2019
Number of reporting laboratories	36	43	36	41	35
Number of test results	465	576	462	533	504
Number of statistical outliers	23	25	21	23	23
Percentage of statistical outliers	4.9%	4.3%	4.5%	4.3%	4.6%

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	November 2023	November 2022	November 2021	November 2020	November 2019
Total Acid Number	+	+	+	+	+/-
Density at 15 °C	+/-	-	+	-	+
Flash Point PMcc	+	+	++	+/-	++
Foaming Tendency Seq. I	-	+	(--)	--	+
Foaming Tendency Seq. II	-	-	-	+/-	-
Foaming Tendency Seq. III	(--)	(--)	(--)	(--)	(--)
Kinematic Viscosity at 40 °C	+	+/-	+/-	++	+/-
Kinematic Viscosity at 100 °C	-	+	+	+/-	+
Viscosity Index	-	+/-	-	+/-	-
Kin.Viscosity Stabinger at 40 °C	+	-	++	++	+
Kin.Viscosity Stabinger at 100 °C	+	+	+/-	+	-
Pour Point Manual	-	-	+/-	+/-	+
Pour Point Automated 1 °C int.	+/-	-	-	-	-
Sulfur	+	-	-	+/-	-
Water	++	+	+/-	++	++
Water Separability	+	++	++	--	++
Calcium as Ca	-	+	+/-	+	+/-

Parameter	November 2023	November 2022	November 2021	November 2020	November 2019
Phosphorus as P	+	+	+	++	+/-
Zinc as Zn	-	-	-	-	-

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:

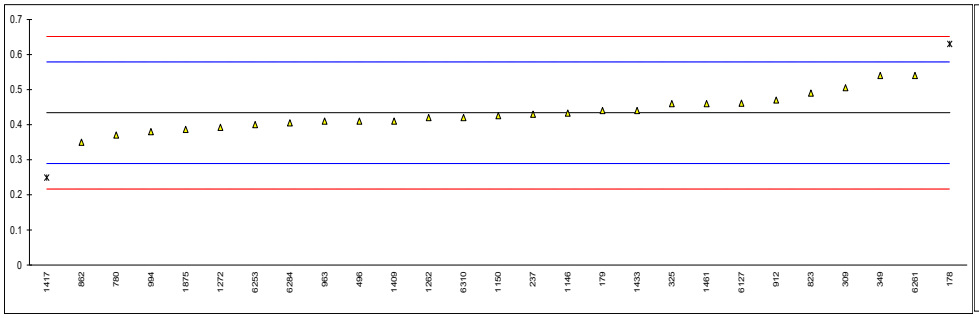
++ : group performed much better than the reference test method
+ : group performed better than the reference test method
+/- : group performance equals the reference test method
- : group performed worse than the reference test method
-- : group performed much worse than the reference test method
n.e.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

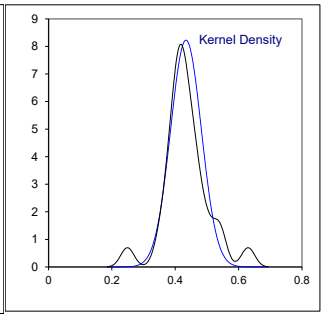
APPENDIX 1

Determination of Total Acid Number on sample #23215; results in mg KOH/g

lab	method	value	mark	z(targ)	end point	volume	remarks
178	D664-A	0.63	R(0.05)	2.71	---	---	
179	D664-A	0.44		0.08	Buffer End Point pH 11	60 mL	
237	D664-B	0.43		-0.05	Inflection Point	125 mL	
256		-----		-----	---	---	
257		-----		-----	---	---	
309	D664-A	0.505		0.98	Buffer End Point pH 10	125 mL	
325	D664-A	0.46		0.36	Buffer End Point pH 10	125 mL	
349	D664-A	0.54		1.46	Buffer End Point pH 10	125 mL	
432		-----		-----	---	---	
496	D664-A	0.410		-0.33	Buffer End Point pH 10	60 mL	
614		-----		-----	---	---	
780	D664-A	0.37		-0.88	Buffer End Point pH 10	60 mL	
823	D664-A	0.49		0.77	Inflection Point	125 mL	
862	D664-A	0.35		-1.16	Inflection Point	60 mL	
912	D664	0.47		0.50	---	---	
963	D974	0.41		-0.33	Inflection Point	60 mL	
994	D664-A	0.38		-0.74	Inflection Point	60 mL	
1146	D664-A	0.433		-0.01	Buffer End Point pH 10	125 mL	
1150	ISO6618	0.4256		-0.11	Inflection Point	125 mL	
1262	ISO6618	0.42		-0.19	Buffer End Point pH 10	60 mL	
1272	ISO6618	0.392		-0.58	---	60 mL	
1381		-----		-----	---	---	
1409	D664-A	0.41		-0.33	Buffer End Point pH 11	60 mL	
1417	D664-A	0.249	R(0.05)	-2.55	Inflection Point	60 mL	
1433	D664-A	0.44		0.08	Inflection Point	125 mL	
1461		0.46		0.36	---	---	
1682		-----		-----	---	---	
1748		-----		-----	---	---	
1875	ISO6618	0.3863		-0.66	---	---	
6117		-----		-----	---	---	
6118		-----		-----	---	---	
6127	D664-A	0.461		0.37	Inflection Point	125 mL	
6253	ISO6618	0.4		-0.47	---	125 mL	
6261	D664-A	0.54		1.46	Inflection Point	60 mL	
6284	D664-A	0.405		-0.40	Buffer End Point pH 11	60 mL	
6310	D664-A	0.42		-0.19	Buffer End Point pH 10	60 mL	
6442		-----		-----	---	---	
6554		-----		-----	---	---	

	normality	OK
	n	25
	outliers	2
	mean (n)	0.4339
	st.dev. (n)	0.04848
	R(calc.)	0.1357
	st.dev.(D664-A:18e2, IP 60 mL)	0.07244
	R(D664-A:18e2, IP 60 mL)	0.2028
Compare:		
	R(D664-A:18e2, IP 125 mL)	0.0916
	R(D664-A:18e2, BEP 60 mL)	0.2454
	R(D664-A:18e2, BEP 125 mL)	0.1314





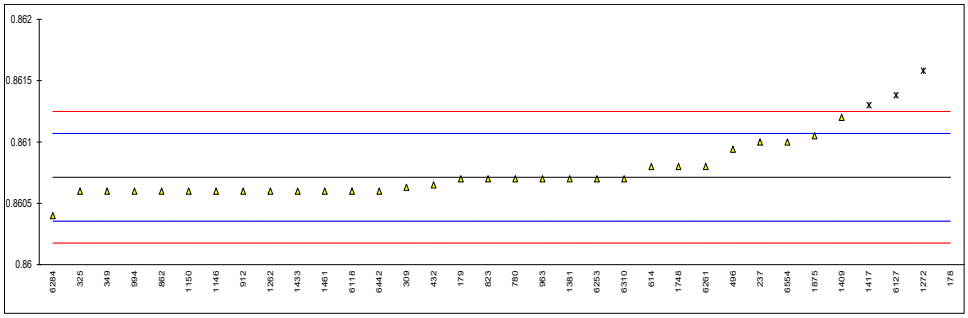
Determination of Copper Corrosion 3 hrs at 50 °C on sample #23215;

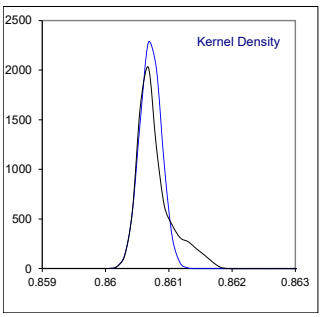
lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D130	1A		-----	
256		-----		-----	
257		-----		-----	
309	D130	1A		-----	
325	D130	1A		-----	
349		-----		-----	
432		-----		-----	
496	D130	1a		-----	
614	D130	1a		-----	
780	D130	1a		-----	
823	D130	1a		-----	
862	D130	1A		-----	
912		-----		-----	
963		-----		-----	
994	D130	1a		-----	
1146		-----		-----	
1150	ISO2160	1a		-----	
1262	ISO2160	1a		-----	
1272	ISO2160	1a		-----	
1381		-----		-----	
1409	ISO2160	1a		-----	
1417	IP154	1A		-----	
1433	ISO2160	1a		-----	
1461	ISO2160	1a		-----	
1682		-----		-----	
1748	D130	1a		-----	
1875		-----		-----	
6117		-----		-----	
6118		-----		-----	
6127		-----		-----	
6253	ISO2160	1a		-----	
6261	D130	1A		-----	
6284	D130	1a		-----	
6310		-----		-----	
6442	D130	1a		-----	
6554		-----		-----	
	n	21			
	mean (n)	1 (1A)			

Determination of Density at 15 °C on sample #23215; results in kg/L

lab	method	value	mark	z(targ)	remarks
178	D4052	0.8919	G(0.01)	174.65	
179	D4052	0.8607		-0.07	
237	D4052	0.8610		1.61	
256		-----		-----	
257		-----		-----	
309	D4052	0.86063		-0.46	
325	D4052	0.8606		-0.63	
349	D4052	0.8606		-0.63	
432	ISO12185	0.86065		-0.35	
496	ISO12185	0.86094		1.27	
614	D4052	0.8608		0.49	
780	ISO12185	0.8607		-0.07	
823	ISO12185	0.8607		-0.07	
862	D4052	0.8606		-0.63	
912	ISO12185	0.8606		-0.63	
963	D4052	0.8607		-0.07	
994	ISO12185	0.8606		-0.63	
1146	D4052	0.8606		-0.63	
1150	ISO12185	0.8606		-0.63	
1262	ISO3675	0.8606		-0.63	
1272	ISO12185	0.86158	G(0.05)	4.86	
1381	ISO12185	0.86070		-0.07	
1409	ISO12185	0.8612		2.73	
1417	IP365	0.8613	DG(0.05)	3.29	
1433	ISO12185	0.8606		-0.63	
1461	ISO3675	0.8606	C	-0.63	first reported 0.8592
1682		-----		-----	
1748	D4052	0.8608		0.49	
1875	DIN51757	0.86105		1.89	
6117		-----		-----	
6118	D4052	0.8606		-0.63	
6127	D4052	0.86138	DG(0.05)	3.74	
6253	ISO3675	0.8607		-0.07	
6261	D4052	0.8608	C	0.49	first reported 0.8615
6284	D4052	0.8604		-1.75	
6310	D4052	0.8607		-0.07	
6442	D4052	0.8606		-0.63	
6554	D4052	0.8610		1.61	

normality	not OK
n	30
outliers	4
mean (n)	0.86071
st.dev. (n)	0.000171
R(calc.)	0.00048
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

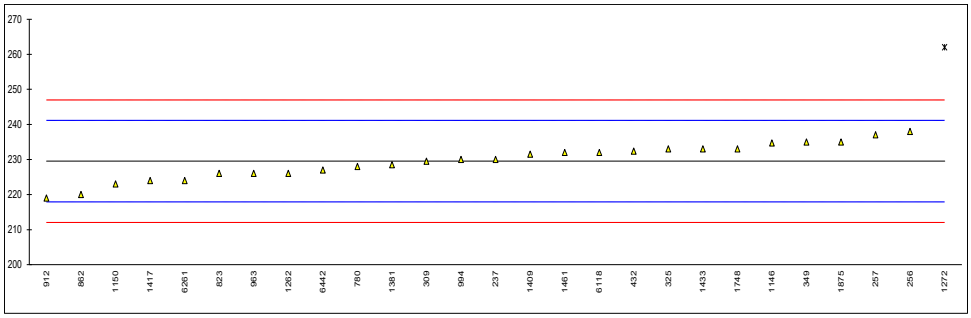


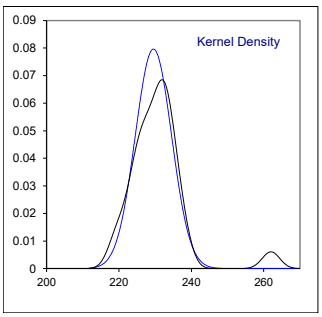


Determination of Flash Point PMcc on sample #23215; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D93-A	230.0		0.08	
256	D3828	238.0		1.46	
257	D3828	237.0		1.28	
309	D93-A	229.5		0.00	
325	D93	233.0		0.60	
349	D93-A	235		0.94	
432	D93-A	232.36		0.49	
496		-----		-----	
614		-----		-----	
780	D93-A	228.0		-0.26	
823	ISO2719-A	226		-0.61	
862	D93-A	220		-1.64	
912	D93	219		-1.81	
963	D93-A	226.0		-0.61	
994	D93-A	230.0		0.08	
1146	D93-A	234.7		0.89	
1150	ISO2719-A	223		-1.12	
1262	ISO2719-A	226.0		-0.61	
1272	ISO2592	262	ex	5.58	test result excluded as open cup test method was used
1381	ISO2719-A	228.5		-0.18	
1409	ISO2719-A	231.5		0.34	
1417	D93-A	224.0		-0.95	
1433	D93-A	233		0.60	
1461	ISO2719-A	232		0.43	
1682		-----		-----	
1748	D93-A	233		0.60	
1875	ISO2719-A	235		0.94	
6117		-----		-----	
6118	D93-A	232		0.43	
6127		-----		-----	
6253		-----		-----	
6261	D93-A	224.0		-0.95	
6284		-----		-----	
6310		-----		-----	
6442	D93-A	227.0	C	-0.43	first reported 252 with open cup test method D92
6554		-----		-----	

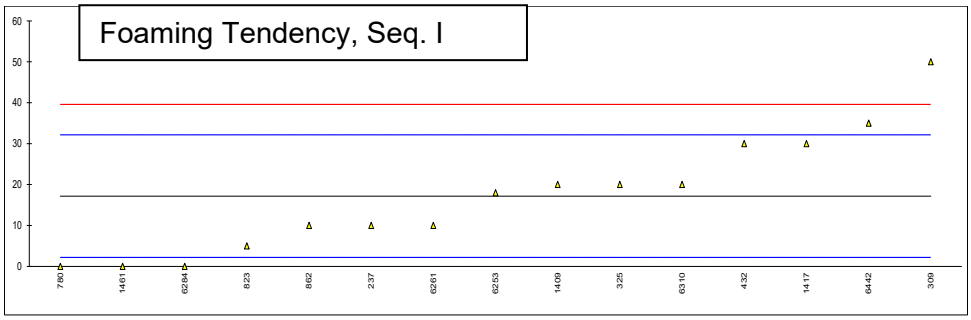
normality	OK
n	26
outliers	0 +1ex
mean (n)	229.52
st.dev. (n)	5.010
R(calc.)	14.03
st.dev.(D93-A:20)	5.820
R(D93-A:20)	16.30

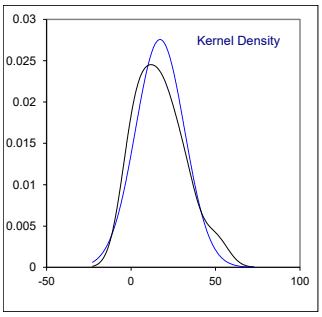


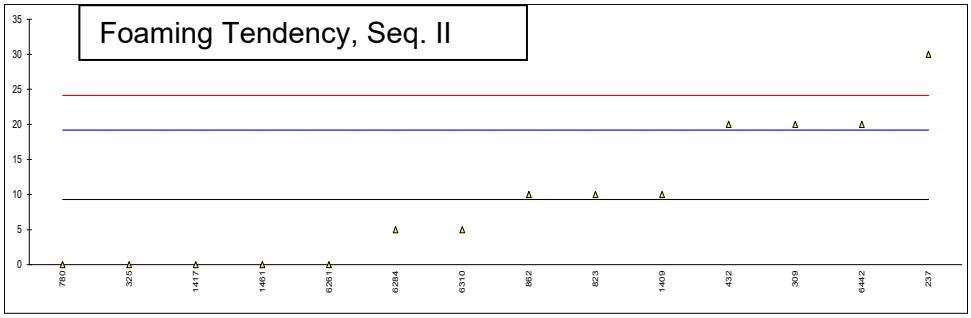


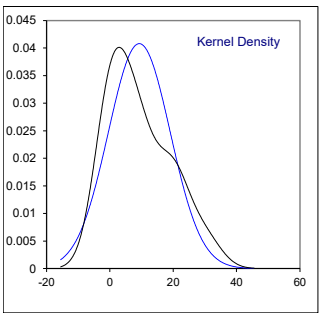
Determination of Foaming Characteristics, Foaming Tendency (5 minutes blowing period) on sample #23215; results in mL

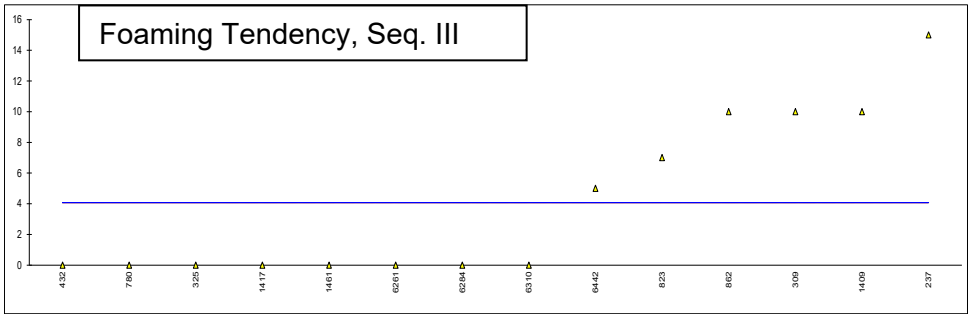
lab	method	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179		-----		-----	-----		-----	-----		-----
237	D892	10		-0.96	30		4.19	15		-----
256		-----		-----	-----		-----	-----		-----
257		-----		-----	-----		-----	-----		-----
309	D892	50		4.39	20		2.17	10		-----
325	D892	20		0.37	0		-1.88	0		-----
349		-----		-----	-----		-----	-----		-----
432	D892	30		1.71	20		2.17	0		-----
496		-----		-----	-----		-----	-----		-----
614		-----		-----	-----		-----	-----		-----
780	D892	0		-2.30	0		-1.88	0		-----
823	D892	5		-1.63	10		0.14	7		-----
862	D892	10		-0.96	10		0.14	10		-----
912		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
994		-----		-----	-----		-----	-----		-----
1146		-----		-----	-----		-----	-----		-----
1150		-----		-----	-----		-----	-----		-----
1262		-----		-----	-----		-----	-----		-----
1272		-----		-----	-----		-----	-----		-----
1381		-----		-----	-----		-----	-----		-----
1409	ISO6247	20		0.37	10		0.14	10		-----
1417	D892	30		1.71	0		-1.88	0		-----
1433		-----		-----	-----		-----	-----		-----
1461		0		-2.30	0		-1.88	0		-----
1682		-----		-----	-----		-----	-----		-----
1748		-----		-----	-----		-----	-----		-----
1875		-----		-----	-----		-----	-----		-----
6117		-----		-----	-----		-----	-----		-----
6118		-----		-----	-----		-----	-----		-----
6127		-----		-----	-----		-----	-----		-----
6253	ISO6247	18		0.11	-----		-----	-----		-----
6261	D892	10		-0.96	0		-1.88	0		-----
6284	D892	0		-2.30	5		-0.87	0		-----
6310	D892	20		0.37	5		-0.87	0		-----
6442	D892 (Alternative)	35		2.38	20		2.17	5		-----
6554		-----		-----	-----		-----	-----		-----
	normality	OK			OK			OK		
	n	15			14			14		
	outliers	0			0			0		
	mean (n)	17.20			9.29			4.07		
	st.dev. (n)	14.483			9.778			5.313		
	R(calc.)	40.55			27.38			14.88		
	st.dev.(D892:23)	7.478			4.948			(0.640)		
	R(D892:23)	20.94			13.85			(1.79)		

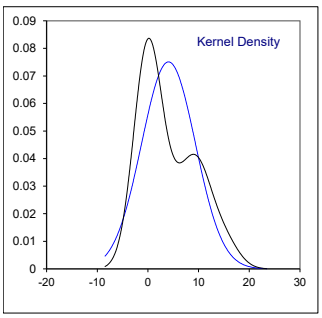

Foaming Tendency, Seq. I
60
50
40
30
20
10
0
7901
1461
6284
823
862
237
6261
6253
1409
325
6310
432
1417
6442
309


Kernel Density
0.03
0.025
0.02
0.015
0.01
0.005
0
-50
0
50
100


Foaming Tendency, Seq. II
35
30
25
20
15
10
5
0
7901
3251
1417
1461
6261
6284
6310
862
823
1409
432
309
6442
237


Kernel Density
0.045
0.04
0.035
0.03
0.025
0.02
0.015
0.01
0.005
0
-20
0
20
40
60


Foaming Tendency, Seq. III
16
14
12
10
8
6
4
2
0
432
7901
325
1417
1461
6261
6284
6310
6442
823
862
309
1409
237


Kernel Density
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
-20
-10
0
10
20
30

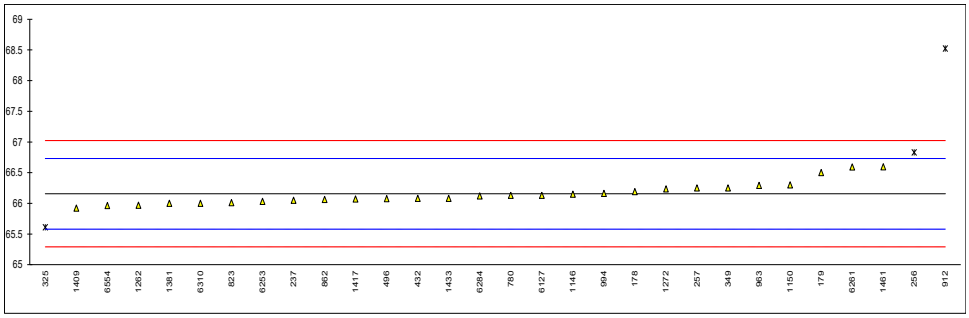
Determination of Foaming Characteristics, Foam Stability (10 minutes settling period) on sample #23215; results in mL

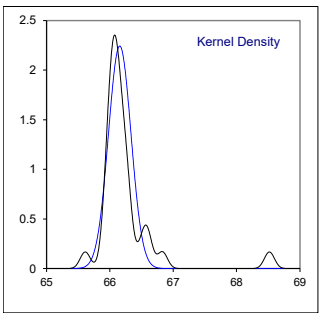
lab	method	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179		-----		-----	-----		-----	-----		-----
237	D892	0		-----	0		-----	0		-----
256		-----		-----	-----		-----	-----		-----
257		-----		-----	-----		-----	-----		-----
309	D892	0		-----	0		-----	0		-----
325	D892	0		-----	0		-----	0		-----
349		-----		-----	-----		-----	-----		-----
432	D892	0		-----	0		-----	0		-----
496		-----		-----	-----		-----	-----		-----
614		-----		-----	-----		-----	-----		-----
780	D892	0		-----	0		-----	0		-----
823	D892	0		-----	0		-----	0		-----
862	D892	0		-----	0		-----	0		-----
912		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
994		-----		-----	-----		-----	-----		-----
1146		-----		-----	-----		-----	-----		-----
1150		-----		-----	-----		-----	-----		-----
1262		-----		-----	-----		-----	-----		-----
1272		-----		-----	-----		-----	-----		-----
1381		-----		-----	-----		-----	-----		-----
1409	ISO6247	0		-----	0		-----	0		-----
1417	D892	0		-----	0		-----	0		-----
1433		-----		-----	-----		-----	-----		-----
1461		0		-----	0		-----	0		-----
1682		-----		-----	-----		-----	-----		-----
1748		-----		-----	-----		-----	-----		-----
1875		-----		-----	-----		-----	-----		-----
6117		-----		-----	-----		-----	-----		-----
6118		-----		-----	-----		-----	-----		-----
6127		-----		-----	-----		-----	-----		-----
6253	ISO6247	0		-----	-----		-----	-----		-----
6261	D892	0		-----	0		-----	0		-----
6284	D892	0		-----	0		-----	0		-----
6310	D892	0		-----	0		-----	0		-----
6442	D892 (Alternative)	0		-----	0		-----	0		-----
6554		-----		-----	-----		-----	-----		-----
	n	15			14			14		
	mean (n)	0			0			0		

Determination of Kinematic Viscosity at 40 °C on sample #23215; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178	D7279 cor. to D445	66.19		0.12	
179	D445	66.50		1.20	
237	D445	66.05		-0.37	
256	D7279 cor. to D445	66.83	C,R(0.05)	2.34	first reported 64.92
257	D7279 cor. to D445	66.25	C	0.33	first reported 65.32
309		-----		-----	
325	D445	65.61	R(0.05)	-1.89	
349	D445	66.25		0.33	
432	D445	66.08		-0.26	
496	D445	66.075		-0.28	
614		-----		-----	
780	D445	66.13		-0.09	
823	D445	66.01		-0.50	
862	D445	66.06		-0.33	
912	D445	68.52	R(0.01)	8.20	
963	D445	66.29		0.47	
994	D445	66.16		0.02	
1146	D445	66.149		-0.02	
1150	ISO3104	66.30		0.50	
1262	ISO3104	65.97		-0.64	
1272	ISO3104	66.234		0.27	
1381	ISO3104	66.00		-0.54	
1409	D445	65.92		-0.82	
1417	D445	66.07		-0.30	
1433	D445	66.08	C	-0.26	first reported 68.00
1461	ISO3104	66.5924		1.52	
1682		-----		-----	
1748		-----		-----	
1875		-----		-----	
6117		-----		-----	
6118		-----		-----	
6127	D445	66.13		-0.09	
6253	ISO3104	66.032		-0.43	
6261	D445	66.59	C	1.51	first reported 66.75
6284	D445	66.12		-0.12	
6310	D7279 cor. to D445	66.0		-0.54	
6442		-----		-----	
6554	D445	65.963	C	-0.67	first reported 70.113

normality	not OK
n	27
outliers	3
mean (n)	66.1554
st.dev. (n)	0.17784
R(calc.)	0.4980
st.dev.(D445:23)	0.28825
R(D445:23)	0.8071

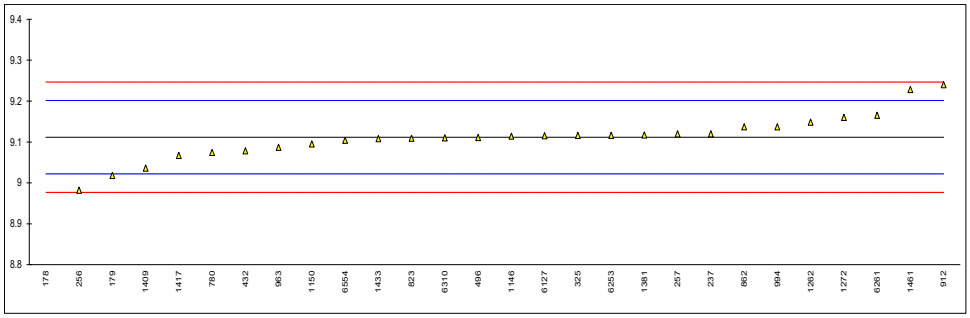


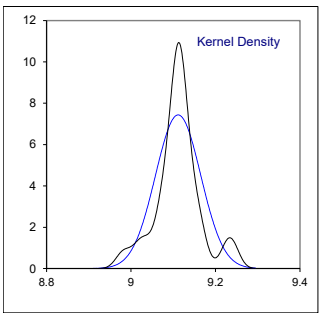


Determination of Kinematic Viscosity at 100 °C on sample #23215; results in mm²/s

lab	method	value	mark	z(targ)	remarks
178	D7279 cor. to D445	8.55	R(0.01)	-12.51	
179	D445	9.018		-2.08	
237	D445	9.120		0.19	
256	D7279 cor. to D445	8.982		-2.89	
257	D7279 cor. to D445	9.120		0.19	
309		-----		-----	
325	D445	9.116		0.10	
349		-----		-----	
432	D445	9.078		-0.75	
496	D445	9.1108		-0.02	
614		-----		-----	
780	D445	9.074		-0.84	
823	ISO3104	9.109		-0.06	
862	D445	9.137		0.57	
912	D445	9.240		2.86	
963	D445	9.087		-0.55	
994	D445	9.137		0.57	
1146	D445	9.1141		0.06	
1150	ISO3104	9.0952		-0.36	
1262	ISO3104	9.148		0.81	
1272	ISO3104	9.1602		1.08	
1381	ISO3104	9.117		0.12	
1409	D445	9.036		-1.68	
1417	D445	9.067		-0.99	
1433	D445	9.108		-0.08	
1461	ISO3104	9.2279	C	2.59	first reported 9.2544
1682		-----		-----	
1748		-----		-----	
1875		-----		-----	
6117		-----		-----	
6118		-----		-----	
6127	D445	9.115		0.08	
6253	ISO3104	9.1163		0.11	
6261	D445	9.165		1.19	
6284		-----		-----	
6310	D7279 cor. to D445	9.11		-0.04	
6442		-----		-----	
6554	D445	9.104	C	-0.17	first reported 9.257

normality	suspect
n	27
outliers	1
mean (n)	9.1116
st.dev. (n)	0.05365
R(calc.)	0.1502
st.dev.(D445:23)	0.04491
R(D445:23)	0.1257



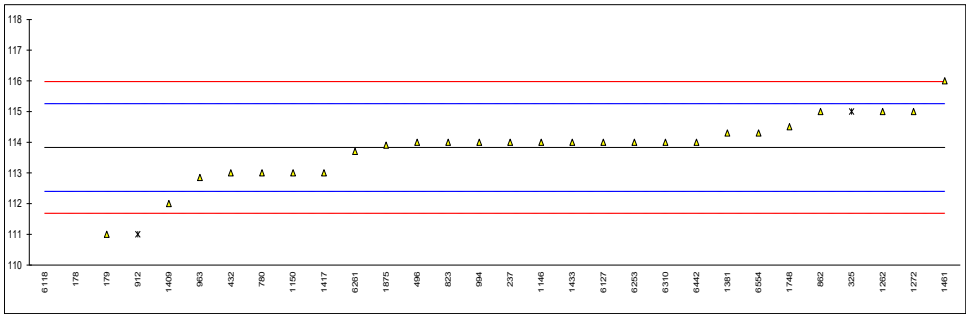


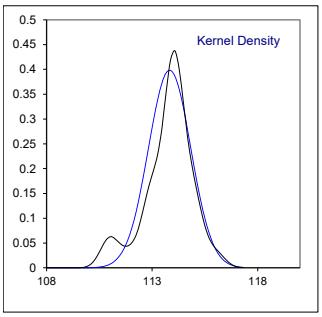
Determination of Viscosity Index on sample #23215;

lab	method	value	mark	z(targ)	remarks
178	D2270	100	ex,E	-19.36	test result excluded as statistical outlier at KV 100 °C
179	D2270	111		-3.96	
237	D2270	114		0.24	
256		-----		-----	
257		-----		-----	
309		-----		-----	
325	D2270	115	ex	1.64	test result excluded as statistical outlier at KV 40 °C
349		-----		-----	
432	D2270	113		-1.16	
496	D2270	114		0.24	
614		-----		-----	
780	D2270	113		-1.16	
823	D2270	114		0.24	
862	D2270	115		1.64	
912	D445	111	ex	-3.96	test result excluded as statistical outlier at KV 40 °C
963	D2270	112.853		-1.37	
994	D2270	114		0.24	
1146	D2270	114		0.24	
1150	ISO2909	113		-1.16	
1262	ISO2909	115		1.64	
1272	ISO2909	115.0		1.64	
1381	ISO2909	114.3		0.66	
1409	D2270	112		-2.56	
1417	D2270	113		-1.16	
1433	ISO2909	114	C	0.24	first reported 109
1461	ISO2909	116		3.04	
1682		-----		-----	
1748	D2270	114.5		0.94	
1875	ISO2909	113.9		0.10	
6117		-----		-----	
6118	Calculated	66.8	R(0.01)	-65.84	
6127	D2270	114		0.24	
6253	ISO2909	114		0.24	
6261	D2270	113.7		-0.18	
6284		-----		-----	
6310	D2270	114		0.24	
6442	D2270	114		0.24	
6554	D2270	114.3	C	0.66	first reported 108

normality	suspect
n	26
outliers	1 +3ex
mean (n)	113.83
st.dev. (n)	1.002
R(calc.)	2.80
st.dev.(D2270:10R16)	0.714
R(D2270:10R16)	2

Lab 178 calculation difference, iis calculated 99

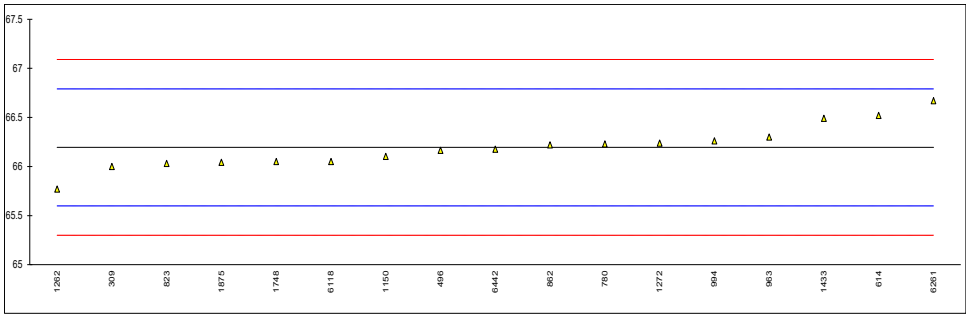


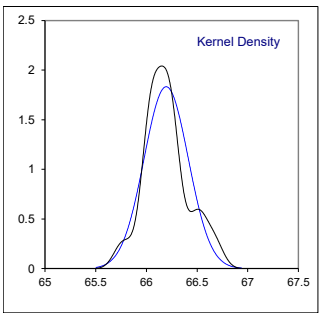


Determination of Kinematic Viscosity Stabinger at 40 °C on sample #23215; results in mm²/s

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
256		-----		-----	
257		-----		-----	
309	D7042	66.00		-0.65	
325		-----		-----	
349		-----		-----	
432		-----		-----	
496	D7042	66.165		-0.10	
614	D7042	66.52		1.09	
780	D7042	66.23		0.12	
823	D7042	66.03		-0.55	
862	D7042	66.22		0.08	
912		-----		-----	
963	D7042	66.30		0.35	
994	D7042	66.26		0.22	
1146		-----		-----	
1150	D7042	66.1031		-0.31	
1262	D7042	65.77		-1.43	
1272	D7042	66.237		0.14	
1381		-----		-----	
1409		-----		-----	
1417		-----		-----	
1433	D7042	66.49		0.99	
1461		-----		-----	
1682		-----		-----	
1748	D7042	66.05		-0.49	
1875	D7042	66.0428		-0.51	
6117		-----		-----	
6118	D7042	66.05		-0.49	
6127		-----		-----	
6253		-----		-----	
6261	D7042	66.67		1.59	
6284		-----		-----	
6310		-----		-----	
6442	D7042	66.175		-0.07	
6554		-----		-----	

normality	OK
n	17
outliers	0
mean (n)	66.1949
st.dev. (n)	0.21765
R(calc.)	0.6094
st.dev.(D7042:21a)	0.29813
R(D7042:21a)	0.8348

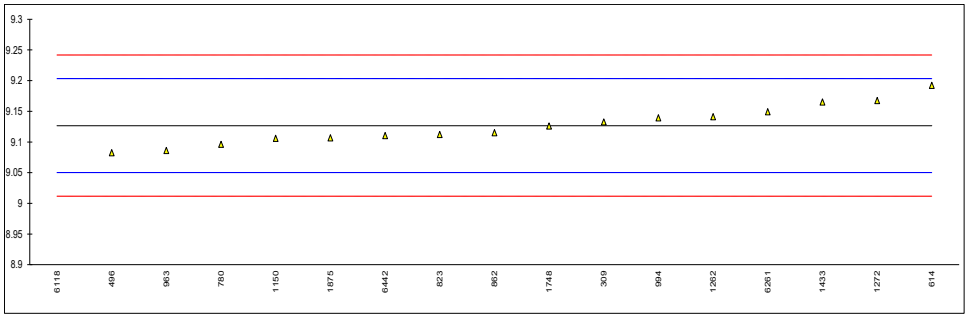


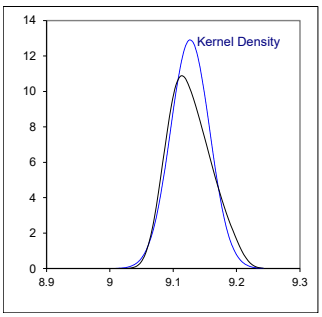


Determination of Kinematic Viscosity Stabinger at 100 °C on sample #23215; results in mm²/s

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
256		-----		-----	
257		-----		-----	
309	D7042	9.1326		0.16	
325		-----		-----	
349		-----		-----	
432		-----		-----	
496	D7042	9.0822		-1.16	
614	D7042	9.192		1.71	
780	D7042	9.096		-0.80	
823	D7042	9.112		-0.38	
862	D7042	9.115		-0.30	
912		-----		-----	
963	D7042	9.086		-1.06	
994	D7042	9.139		0.32	
1146		-----		-----	
1150	D7042	9.1058		-0.54	
1262	D7042	9.141		0.38	
1272	D7042	9.1675		1.07	
1381		-----		-----	
1409		-----		-----	
1417		-----		-----	
1433	D7042	9.165		1.00	
1461		-----		-----	
1682		-----		-----	
1748	D7042	9.126		-0.02	
1875	D7042	9.1067		-0.52	
6117		-----		-----	
6118	D7042	7.532	G(0.01)	-41.64	
6127		-----		-----	
6253		-----		-----	
6261	D7042	9.149		0.58	
6284		-----		-----	
6310		-----		-----	
6442	D7042	9.1103		-0.43	
6554		-----		-----	

normality	OK
n	16
outliers	1
mean (n)	9.1266
st.dev. (n)	0.03090
R(calc.)	0.0865
st.dev.(D7042:21a)	0.03830
R(D7042:21a)	0.1072

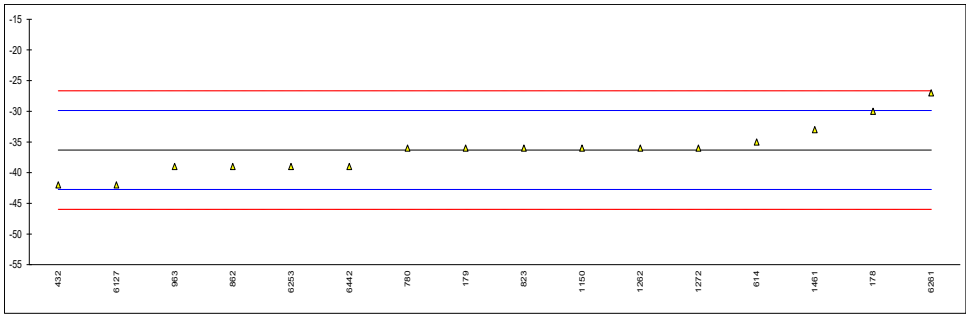


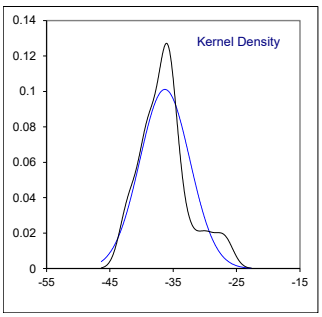


Determination of Pour Point Manual on sample #23215; results in °C

lab	method	value	mark	z(targ)	remarks
178	D97	-30		1.96	
179	D97	-36		0.10	
237	D97	<-21		-----	
256		-----		-----	
257		-----		-----	
309		-----		-----	
325		-----		-----	
349		-----		-----	
432	D97	-42		-1.77	
496		-----		-----	
614	D97	-35		0.41	
780	D97	-36		0.10	
823	ISO3016	-36		0.10	
862	D97	-39		-0.84	
912		-----		-----	
963	D97	-39		-0.84	
994		-----		-----	
1146		-----		-----	
1150	ISO3016	-36		0.10	
1262	ISO3016	-36		0.10	
1272	ISO3016	-36		0.10	
1381		-----		-----	
1409		-----		-----	
1417		-----		-----	
1433		-----		-----	
1461	ISO3016	-33		1.03	
1682		-----		-----	
1748		-----		-----	
1875		-----		-----	
6117		-----		-----	
6118		-----		-----	
6127	D97	-42	C	-1.77	first reported -51
6253	NF T60-105	-39		-0.84	
6261	D97	-27		2.90	
6284		-----		-----	
6310		-----		-----	
6442	D97	-39		-0.84	
6554		-----		-----	

normality	suspect
n	16
outliers	0
mean (n)	-36.31
st.dev. (n)	3.945
R(calc.)	11.05
st.dev.(D97:17bR22)	3.214
R(D97:17bR22)	9

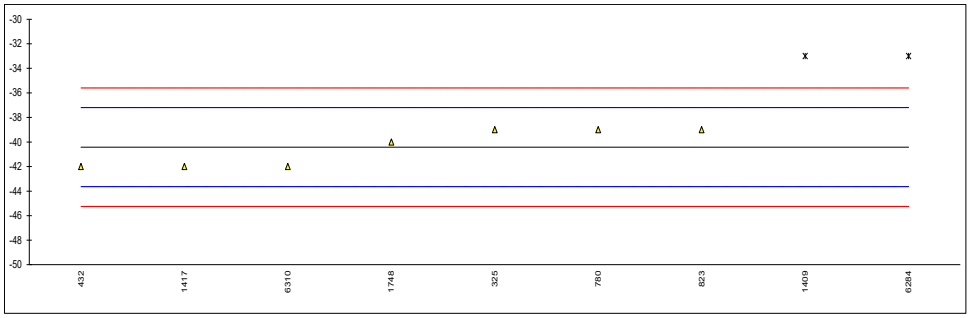


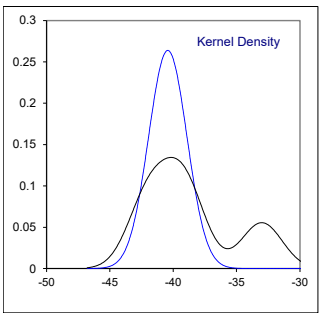


Determination of Pour Point Automated 1 °C interval on sample #23215; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
256		-----		-----	
257		-----		-----	
309		-----		-----	
325	D5950	-39		0.89	
349		-----		-----	
432	D5950	-42		-0.98	
496		-----		-----	
614		-----		-----	
780	D5950	-39		0.89	
823	D5950	-39		0.89	
862		-----		-----	
912		-----		-----	
963		-----		-----	
994		-----		-----	
1146		-----		-----	
1150		-----		-----	
1262		-----		-----	
1272		-----		-----	
1381		-----		-----	
1409	D5950	-33	DG(0.05)	4.62	
1417	D5950	-42.0		-0.98	
1433		-----		-----	
1461		-----		-----	
1682		-----		-----	
1748	D7346	-40		0.27	
1875		-----		-----	
6117		-----		-----	
6118		-----		-----	
6127		-----		-----	
6253		-----		-----	
6261		-----		-----	
6284	D5949	-33	DG(0.05)	4.62	
6310	D5950	-42		-0.98	
6442		-----		-----	
6554		-----		-----	

normality	OK
n	7
outliers	2
mean (n)	-40.43
st.dev. (n)	1.512
R(calc.)	4.23
st.dev.(D5950:14R20)	1.607
R(D5950:14R20)	4.5

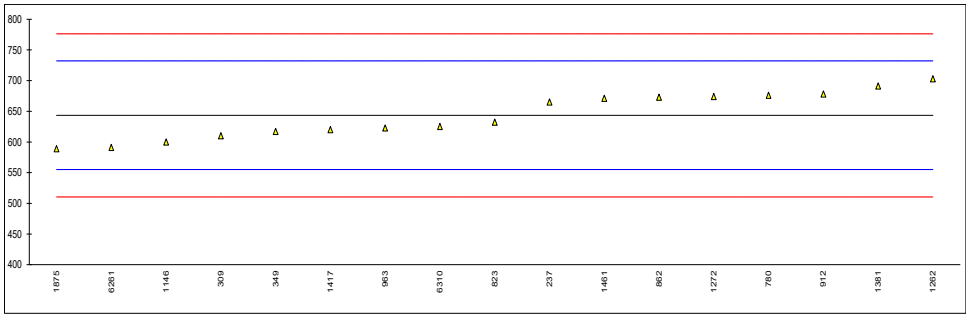


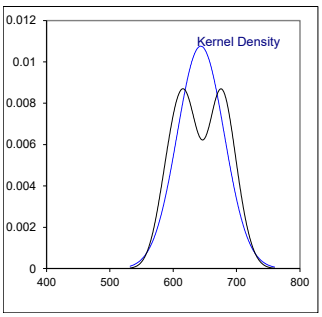


Determination of Sulfur on sample #23215; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D4294	665		0.49	
256		-----		-----	
257		-----		-----	
309	ISO8754	610		-0.75	
325		-----		-----	
349	D2622	617		-0.60	
432		-----		-----	
496		-----		-----	
614		-----		-----	
780	D4294	676		0.74	
823	D4294	632	C	-0.26	first reported 0.0632
862	D2622	673		0.67	
912	D4294	678		0.78	
963	D4294	622.54		-0.47	
994		-----		-----	
1146	D4294	600		-0.98	
1150		-----		-----	
1262	ISO8754	703		1.35	
1272	ISO8754	674		0.69	
1381	ISO8754	691		1.08	
1409		-----		-----	
1417	In house	620		-0.53	
1433		-----		-----	
1461	ISO8754	671		0.62	
1682		-----		-----	
1748		-----		-----	
1875	DIN51724	588.8		-1.23	
6117		-----		-----	
6118		-----		-----	
6127		-----		-----	
6253		-----		-----	
6261	D4294	591		-1.18	
6284		-----		-----	
6310	D7751	625		-0.42	
6442		-----		-----	
6554		-----		-----	

normality	OK
n	17
outliers	0
mean (n)	643.37
st.dev. (n)	37.075
R(calc.)	103.81
st.dev.(D4294:21)	44.266
R(D4294:21)	123.94





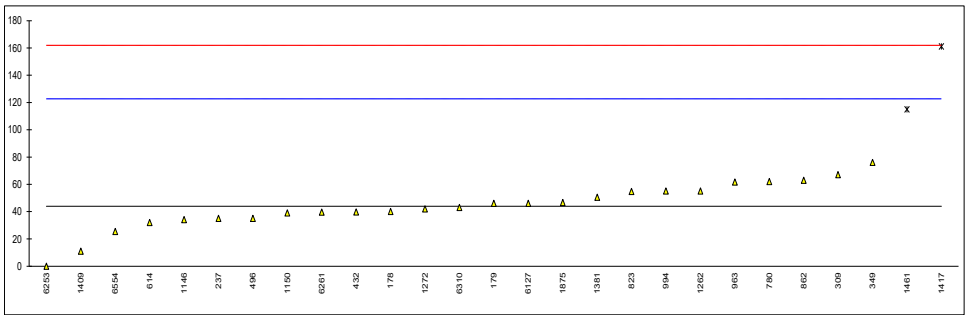
Determination of Water on sample #23215; results in mg/kg

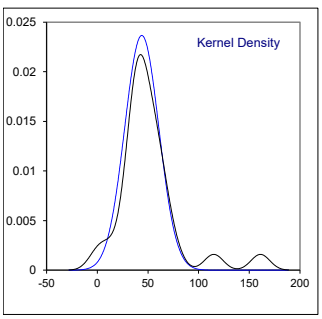
lab	method	value	mark	z(targ)	remarks
178	D6304-C:20	40		-0.10	
179	D6304-C:16e1	46		0.05	
237	D6304-B:16e1	35		-0.23	
256		-----		-----	
257		-----		-----	
309	D6304-C:20	67		0.59	
325	D6304-C	<100		-----	
349	D6304-A:20	76		0.81	
432	D6304-B:20	39.7		-0.11	
496	D6304-B:20	35		-0.23	
614	D6304-B:20	32		-0.30	
780	D6304-B:20	62.0		0.46	
823	D6304-B:20	54.7		0.27	
862	D6304-B	63		0.48	
912		-----		-----	
963	D6304-A:20	61.7		0.45	
994	D6304-C:20	55		0.28	
1146	D6304-B:20	34		-0.25	
1150	ISO12937	39		-0.13	
1262	EN60814	55		0.28	
1272	ISO12937	42		-0.05	
1381	EN60814	50.5		0.17	
1409	D6304-B:20	11	C	-0.84	first reported <30
1417	D6304-A:20	161.0	R(0.01)	2.97	
1433		-----		-----	
1461		115	C,R(0.01)	1.81	first reported absence
1682		-----		-----	
1748		-----		-----	
1875	ISO12937	46.67		0.07	
6117		-----		-----	
6118		-----		-----	
6127	D6304-A:20	46.0589		0.05	
6253	ISO3733	0		-1.12	
6261	D6304-A:20	39.6		-0.11	
6284		-----		-----	
6310	D6304-B:16e1	43		-0.02	
6442		-----		-----	
6554	D6304-A:20	25.4		-0.47	

normality	suspect
n	25
outliers	2
mean (n)	43.973
st.dev. (n)	16.8649
R(calc.)	47.222
st.dev.(D6304-B:20)	39.3461
R(D6304-B:20)	110.169

Compare:

R(D6304-A:20)	33.712
R(D6304-C:20)	23.042

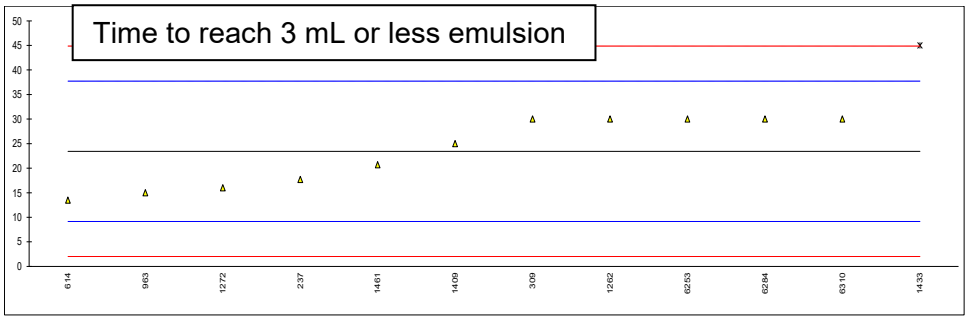


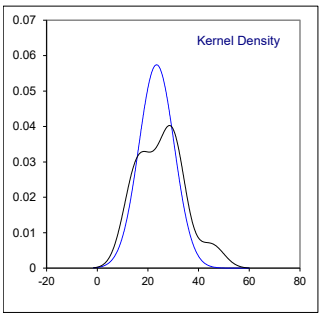


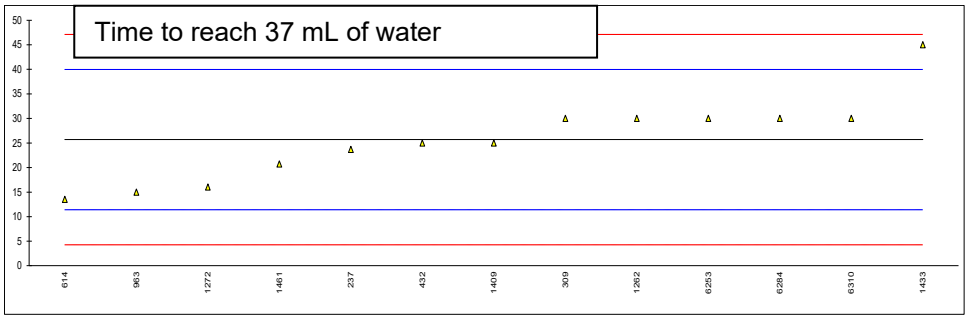
Determination of Water Separability at 54 °C, distilled water on sample #23215; results in minutes

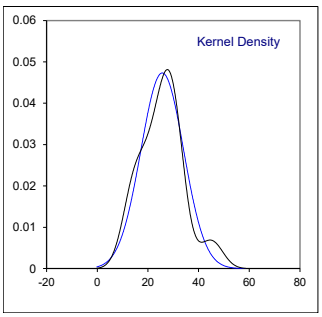
lab	method	≤3 mL emulsion	m.	z(targ)	37 mL water	m.	z(targ)	complete break (40-40-0)	m.	z(targ)	test aborted	time aborted
178		-----		-----	-----		-----	-----		-----	-----	-----
179		-----		-----	-----		-----	-----		-----	-----	-----
237	D1401	17.7		-0.80	23.7		-0.28	30.0		0.21	No	-----
256		-----		-----	-----		-----	-----		-----	-----	-----
257		-----		-----	-----		-----	-----		-----	-----	-----
309	D1401	30		0.92	30		0.60	30		0.21	Yes	30
325		-----		-----	-----		-----	-----		-----	-----	-----
349		-----		-----	-----		-----	-----		-----	-----	-----
432		-----		-----	25		-0.10	-----		-----	Yes	30
496		-----		-----	-----		-----	-----		-----	Yes	30
614	D1401	13.5		-1.39	13.5		-1.71	14.5		-1.96	No	-----
780		-----		-----	-----		-----	-----		-----	Yes	30
823		-----		-----	-----		-----	-----		-----	-----	-----
862		-----		-----	-----		-----	30		0.21	-----	-----
912		-----		-----	-----		-----	-----		-----	-----	-----
963	D1401	15		-1.18	15		-1.50	25		-0.49	Yes	25
994		-----		-----	-----		-----	-----		-----	-----	-----
1146		-----		-----	-----		-----	-----		-----	Yes	25
1150		-----		-----	-----		-----	-----		-----	-----	-----
1262	ISO6614	30		0.92	30		0.60	>60	f+?	>4.41	Yes	60
1272	ISO6614	16		-1.04	16		-1.36	22		-0.91	-----	-----
1381		-----		-----	-----		-----	-----		-----	-----	-----
1409	ISO6614	25		0.22	25		-0.10	-----		-----	Yes	60
1417		-----		-----	-----		-----	-----		-----	Yes	30
1433	D1401	45	D(5)	3.02	45		2.70	>60	f+?	>4.41	Yes	30
1461	ISO6614	20.7		-0.38	20.7		-0.70	35		0.91	-----	-----
1682		-----		-----	-----		-----	-----		-----	-----	-----
1748		-----		-----	-----		-----	-----		-----	-----	-----
1875		-----		-----	-----		-----	-----		-----	-----	-----
6117		-----		-----	-----		-----	-----		-----	-----	-----
6118		-----		-----	-----		-----	-----		-----	-----	-----
6127		-----		-----	-----		-----	-----		-----	-----	-----
6253	ISO6614	30		0.92	30		0.60	-----		-----	-----	-----
6261		-----		-----	-----		-----	-----		-----	Yes	30
6284		30		0.92	30		0.60	30		0.21	Yes	30
6310		30		0.92	30		0.60	40		1.61	No	-----
6442		-----		-----	-----		-----	-----		-----	-----	-----
6554		-----		-----	-----		-----	-----		-----	-----	-----
	normality	OK			suspect			OK				
	n	11			13			9				
	outliers	1			0			0				
	mean (n)	23.45			25.68			28.50				
	st.dev. (n)	6.953			8.428			7.365				
	R(calc.)	19.47			23.60			20.62				
	st.dev.(D1401:21)	7.143			7.143			7.143				
	R(D1401:21)	20			20			20				

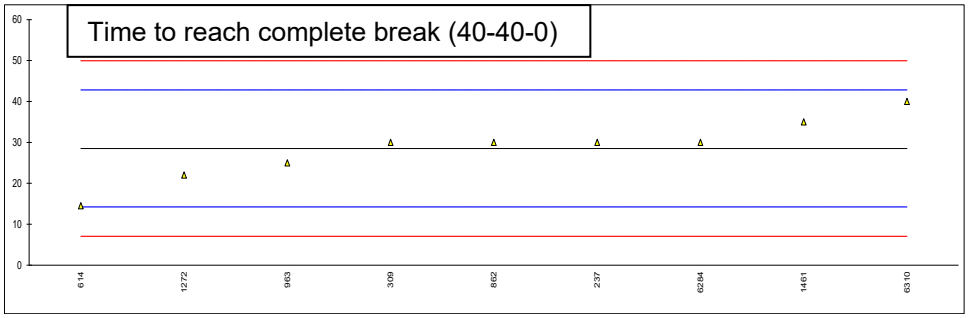
Lab 1262 possibly a false positive test result?
Lab 1433 possibly a false positive test result?

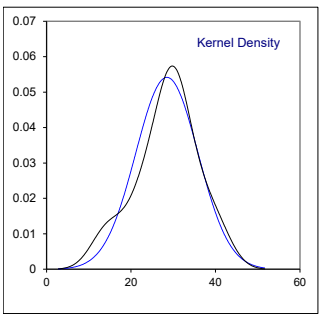

Time to reach 3 mL or less emulsion
614
963
1272
237
1461
1409
309
1262
6253
6284
6310
1433


Kernel Density


Time to reach 37 mL of water
614
963
1272
1461
237
432
1409
309
1262
6253
6284
6310
1433


Kernel Density


Time to reach complete break (40-40-0)
614
1272
963
309
862
237
6284
1461
6310


Kernel Density

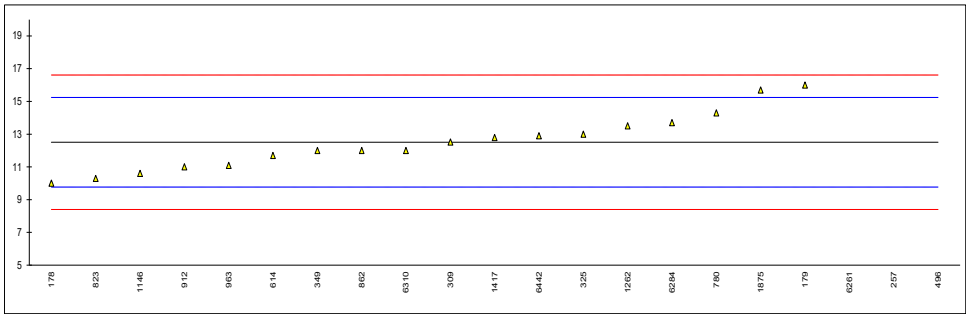
Determination of Water Separability at 54 °C, distilled water on sample #23215; results in mL
--- Continued ----

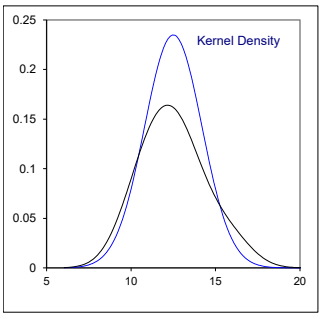
lab	method	oil	mark	z(targ)	water	mark	z(targ)	emulsion	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179		-----		-----	-----		-----	-----		-----
237	D1401	42		-----	38		-----	0		-----
256		-----		-----	-----		-----	-----		-----
257		-----		-----	-----		-----	-----		-----
309	D1401	0		-----	33		-----	47		-----
325		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
432		0		-----	35		-----	40		-----
496		34		-----	35		-----	11		-----
614	D1401	40		-----	40		-----	0		-----
780		43		-----	37		-----	0		-----
823		-----		-----	-----		-----	-----		-----
862		36		-----	35		-----	9		-----
912		-----		-----	-----		-----	-----		-----
963	D1401	40		-----	40		-----	0		-----
994		-----		-----	-----		-----	-----		-----
1146		43		-----	37		-----	0		-----
1150		-----		-----	-----		-----	-----		-----
1262	ISO6614	41		-----	38		-----	1		-----
1272	ISO6614	-----		-----	-----		-----	-----		-----
1381		-----		-----	-----		-----	-----		-----
1409	ISO6614	42		-----	38		-----	0		-----
1417		10		-----	10		-----	60		-----
1433	D1401	0		-----	12		-----	68		-----
1461	ISO6614	-----		-----	-----		-----	-----		-----
1682		-----		-----	-----		-----	-----		-----
1748		-----		-----	-----		-----	-----		-----
1875		-----		-----	-----		-----	-----		-----
6117		-----		-----	-----		-----	-----		-----
6118		-----		-----	-----		-----	-----		-----
6127		-----		-----	-----		-----	-----		-----
6253	ISO6614	-----		-----	-----		-----	-----		-----
6261		0		-----	30		-----	50		-----
6284		0		-----	21		-----	59		-----
6310		-----		-----	-----		-----	-----		-----
6442		-----		-----	-----		-----	-----		-----
6554		-----		-----	-----		-----	-----		-----

Determination of Calcium as Ca on sample #23215; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	10		-1.83	
179	D5185	16		2.55	
237		-----		-----	
256		-----		-----	
257	D6595	36.41	G(0.05)	17.47	
309	D5185	12.512		0.01	
325	D5185	13		0.36	
349	D5185	12		-0.37	
432		-----		-----	
496	D5185	69.50	G(0.01)	41.66	
614	D5185	11.7		-0.59	
780	D5185	14.30		1.31	
823	D5185	10.29		-1.62	
862	D5185	12		-0.37	
912	D5185	11		-1.10	
963	D5185	11.09		-1.03	
994	D5185	<40		-----	
1146	D5185	10.6		-1.39	
1150		-----		-----	
1262	D5185	13.5		0.73	
1272		-----		-----	
1381		-----		-----	
1409		-----		-----	
1417	D5185	12.8		0.22	
1433		-----		-----	
1461		-----		-----	
1682		-----		-----	
1748		-----		-----	
1875	EN11885	15.7		2.34	
6117		-----		-----	
6118		-----		-----	
6127		-----		-----	
6253		-----		-----	
6261	D5185	36.2	G(0.01)	17.32	
6284	D5185	13.7		0.87	
6310	D7751	12		-0.37	
6442		12.9		0.29	
6554		-----		-----	

	normality	OK
	n	18
	outliers	3
	mean (n)	12.505
	st.dev. (n)	1.6985
	R(calc.)	4.756
	st.dev.(Horwitz)	1.3680
	R(Horwitz)	3.830
Compare:		
	R(D5185:18)	0.400

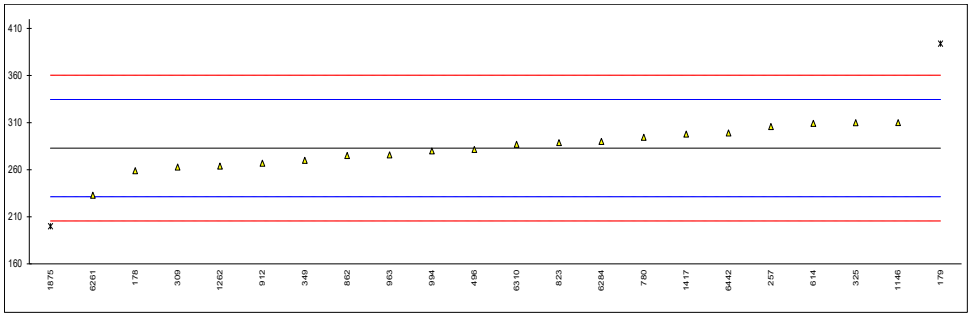


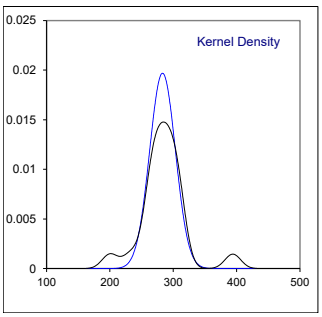


Determination of Phosphorus as P on sample #23215; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	259		-0.93	
179	D5185	394	R(0.01)	4.30	
237		-----		-----	
256		-----		-----	
257	D6595	306.01		0.89	
309	D5185	262.84		-0.78	
325	D5185	310		1.04	
349	D5185	270		-0.50	
432		-----		-----	
496	D5185	281.5		-0.06	
614	D5185	309.3		1.02	
780	D5185	294.50		0.44	
823	D5185	288.8		0.22	
862	D5185	275		-0.31	
912	D5185	267		-0.62	
963	D5185	275.85		-0.28	
994	D5185	280		-0.12	
1146	D5185	310.1		1.05	
1150		-----		-----	
1262	D5185	263.9		-0.74	
1272		-----		-----	
1381		-----		-----	
1409		-----		-----	
1417	D5185	297.8		0.57	
1433		-----		-----	
1461		-----		-----	
1682		-----		-----	
1748		-----		-----	
1875	EN11885	200.1	R(0.05)	-3.21	
6117		-----		-----	
6118		-----		-----	
6127		-----		-----	
6253		-----		-----	
6261	D5185	233		-1.94	
6284	D5185	290		0.27	
6310	D7751	287		0.15	
6442		299		0.62	
6554		-----		-----	

normality	OK
n	20
outliers	2
mean (n)	283.030
st.dev. (n)	20.2473
R(calc.)	56.692
st.dev.(D5185:18)	25.8361
R(D5185:18)	72.341

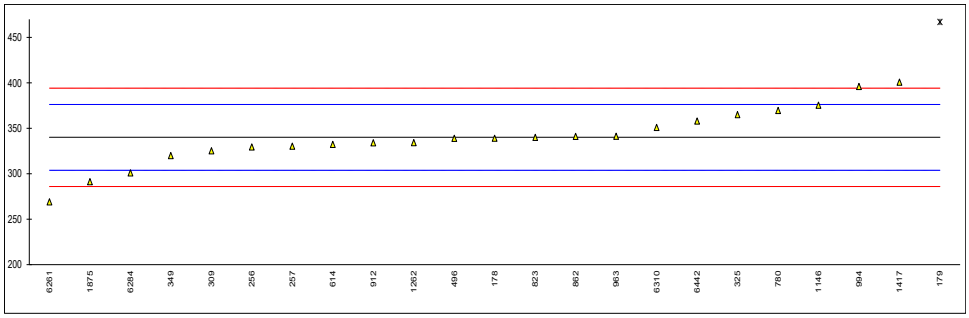


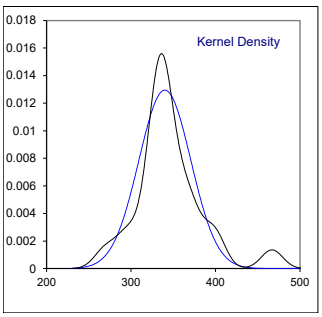


Determination of Zinc as Zn on sample #23215; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	339		-0.06	
179	D5185	467	R(0.05)	7.03	
237		-----		-----	
256	In house	329.38		-0.59	
257	D6595	330.38		-0.54	
309	D5185	325.128		-0.83	
325	D5185	365		1.38	
349	D5185	320		-1.11	
432		-----		-----	
496	D5185	338.8		-0.07	
614	D5185	332.2		-0.44	
780	D5185	369.50		1.63	
823	D5185	340.0		0.00	
862	D5185	341		0.05	
912	D5185	334		-0.34	
963	D5185	341.19		0.06	
994	D5185	396		3.10	
1146	D5185	375.4		1.96	
1150		-----		-----	
1262	D5185	334.1		-0.33	
1272		-----		-----	
1381		-----		-----	
1409		-----		-----	
1417	D5185	400.7		3.36	
1433		-----		-----	
1461		-----		-----	
1682		-----		-----	
1748		-----		-----	
1875	EN11885	291.2		-2.71	
6117		-----		-----	
6118		-----		-----	
6127		-----		-----	
6253		-----		-----	
6261	D5185	269		-3.94	
6284	D5185	301		-2.16	
6310	D7751	351		0.60	
6442		358		0.99	
6554		-----		-----	

normality	OK
n	22
outliers	1
mean (n)	340.090
st.dev. (n)	30.7990
R(calc.)	86.237
st.dev.(D5185:18)	18.0581
R(D5185:18)	50.563





APPENDIX 2

Number of participants per country

1 lab in	ALGERIA
1 lab in	AUSTRALIA
1 lab in	AUSTRIA
1 lab in	AZERBAIJAN
3 labs in	BELGIUM
3 labs in	BULGARIA
2 labs in	CHINA, People's Republic
1 lab in	ESTONIA
2 labs in	GERMANY
1 lab in	INDIA
1 lab in	JORDAN
1 lab in	KOREA, Republic of
2 labs in	MALAYSIA
1 lab in	MOROCCO
2 labs in	NETHERLANDS
1 lab in	NIGERIA
1 lab in	OMAN
1 lab in	POLAND
1 lab in	PORTUGAL
1 lab in	RUSSIAN FEDERATION
2 labs in	SAUDI ARABIA
1 lab in	SERBIA
1 lab in	SPAIN
1 lab in	TAIWAN
2 labs in	TANZANIA
1 lab in	UNITED KINGDOM
2 labs in	UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)/D5	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?
SDS	= Safety Data Sheet

Literature

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2 ISO5725:86
3 ISO5725 parts 1-6:94
4 ISO13528:05
5 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
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)
8 J.N. Miller, Analyst, 118, 455, (1993)
9 Analytical Methods Committee, Technical Brief, No 4, January 2001
10 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)
11 W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)

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