Results of Proficiency Test
Hydraulic Oil (fresh)
November 2020

Organized by: Institute for Interlaboratory Studies
Spijkenisse, The Netherlands

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Report: iis20L08

January 2021

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1 INTRODUCTION

Since 2014 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for Hydraulic Oil (fresh) every year. It was decided to continue the round robin for the analysis on Hydraulic Oil (fresh) during the annual proficiency test program of 2020/2021.
In this interlaboratory study 42 laboratories in 28 different countries registered for participation. See appendix 3 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 an ISO/IEC17025 accredited laboratory.
It was decided to send one sample of 1L Hydraulic Oil labelled #20210. 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 50 liters of fresh Hydraulic Oil was obtained from a local supplier. After homogenization 48 amber glass bottles of 1L were filled and labelled #20210.
The homogeneity of the subsamples was checked by determination of Density at 15°C in accordance with ASTM D4052 and Kinematic Viscosity at 40°C in accordance with ASTM D445 on 10 stratified randomly selected subsamples.

	Density at 15°C in kg/L	Kinematic Viscosity at 40°C in mm^2/s
Sample #20210-1	0.87460	97.91
Sample #20210-2	0.87460	97.92
Sample #20210-3	0.87460	97.91
Sample #20210-4	0.87461	97.89
Sample #20210-5	0.87460	97.92
Sample #20210-6	0.87460	97.93
Sample #20210-7	0.87460	97.90
Sample #20210-8	0.87461	97.93
Sample #20210-9	0.87461	97.88
Sample #20210-10	0.87461	97.92

Table 1: homogeneity test results of subsamples #20210

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)	0.00001	0.05
reference test method	ISO12185:96	D445:19a
0.3 x R (reference test method)	0.00015	0.36

Table 2: evaluation of the repeatabilities of subsamples #20210

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 1L sample labelled #20210 was sent on October 7, 2020. 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 asked to determine on sample #20210: 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, Viscosity Stabinger at 40°C and at 100°C, Pour Point (manual and automated), Sulfur, Water, Water Separability at 82°C (distilled water) and Calcium, Phosphorus and Zinc.
Also, some additional questions were asked about Total Acid Number and Foaming 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 test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.

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

According to ISO5725 the original test results per determination were submitted to Dixon’s, Grubbs' or Rosner’s outlier tests. Outliers are marked by D(0.01) for the Dixon’s test, by G(0.01) or DG(0.01) for the Grubbs’ test and by R(0.01) for the Rosner’s test. Stragglers are marked by D(0.05) for the Dixon’s test, by G(0.05) or DG(0.05) for the Grubbs’ test and by R(0.05) for the Rosner’s test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

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

3.2 GRAPHICS

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

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM, ISO reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

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

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

The z-scores were calculated according to:

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

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

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

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

4 EVALUATION

Some problems were encountered with the dispatch of the samples due to the COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with one week. Three participants reported test results after the final reporting date and one other participant did not report any test results. Not all laboratories were able to report all tests requested.
In total 41 participants reported 533 numerical test results. Observed were 23 outlying test results, which is 4.3%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

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

4.1 EVALUATION PER TEST

In this section the reported test results are discussed per test. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the reported test results in appendix 1. The abbreviations, used in these tables, are explained in appendix 4.

In iis PT reports 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). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D2270:10(2016)). In the tables of appendix 1 only the test method number and year of adoption or revision will be used (e.g. D2270:10).

Unfortunately, a suitable reference test method providing the precision data is not available for all determinations. For the tests that have no available precision data the calculated reproducibility was compared against the estimated reproducibility calculated from the Horwitz equation.

Total Acid Number: 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 inflection point at titration volume 60 mL and Buffer End Point at titration volume 60 mL from ASTM D664-A:18e2. However, the calculated reproducibility is not in agreement with the 125 mL requirements.
It is observed that seven participants reported to have used BEP (pH 11) as determination end point and six reported to have used BEP (pH 10). In method ASTM D664-A version 2018e2 the Buffer End Point has been changed to pH 10.

Copper Corrosion: This determination was not problematic. All reporting participants agreed on a test result of 1 (1a/1b).

Density at 15°C: This determination was problematic for a number of laboratories. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ISO12185:96.

Flash Point PMcc: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of ASTM D93-A:20.

Foaming Characteristics (Tendency and Stability): This determination was problematic. In total eight statistical outliers were observed over three foaming parameters. The calculated reproducibilities after rejection of the statistical outliers in the Foaming Tendency determination for sequence I and III are not in agreement with the requirements of ASTM D892:18. The calculated reproducibility for sequence II is in agreement with the requirements of ASTM D892:18. The variation in the test results for sequence III is very large. Therefore, it was decided not to calculate z-scores.
All reporting participants reported 0 mL for Foam Stability, except two laboratories.
This determination 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. Therefore, extra information was asked (see appendix 2).

Almost all participants have given the same answers or did not report this information. Therefore, no conclusions could be drawn.

Kinematic Viscosity at 40°C: This determination was not problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D445:19a.

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

Viscosity Index: This determination was not problematic. One statistical outlier was observed and one other test result was excluded. The calculated reproducibility after rejection of the suspect data is in full agreement with the requirements of ASTM D2270:10(2016). No calculation differences were found between the reported test results of the participants and the values calculated by iis.

Viscosity Stabinger at 40°C: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D7042:20.

Viscosity Stabinger at 100°C: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility is in good agreement with the requirements of ASTM D7042:20.

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

Pour Point Automated, 1°C interval: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D5950:14.

Sulfur: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D4294:16e1.

Water: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D6304:16e1.

Water Separability at 82°C: This determination was problematic. No statistical outliers were observed over three parameters. All calculated reproducibilities are not in agreement with the requirements of ASTM D1401:18a.

Calcium: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility calculated with the Horwitz equation, but not at all with the strict requirements of ASTM D5185:18.

Phosphorus: This determination was not problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D5185:18.

Zinc: This determination was problematic. No statistical outliers were observed. The calculated reproducibility 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 or as declared by the estimated target reproducibility using the Horwitz equation 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 reproducibilities derived from reference test methods (in casu ASTM and ISO test methods) are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Total Acid Number	mg KOH/g	30	0.35	0.12	0.17
Copper Corrosion 3hrs at 50°C		24	1 (1A/1B)	n.a.	n.a.
Density at 15°C	kg/L	37	0.8747	0.0007	0.0005
Flash Point PMcc	°C	30	236.6	15.9	16.8
Foaming Tendency Seq. I	mL	12	17.1	53.9	20.9
Foaming Tendency Seq. II	mL	15	17.0	16.6	15.9
Foaming Tendency Seq. III	mL	14	14.6	63.7	(6.4)
Foam Stability Seq. I	mL	14	0	n.a.	n.a.
Foam Stability Seq. II	mL	17	0	n.a.	n.a.
Foam Stability Seq. III	mL	16	0	n.a.	n.a.
Kinematic Viscosity at 40°C	mm^2/s	32	97.849	0.603	1.194
Kinematic Viscosity at 100°C	mm^2/s	34	11.166	0.149	0.154
Viscosity Index		34	99.25	2.17	2
Viscosity Stabinger at 40°C	mm^2/s	13	97.901	0.561	1.179
Viscosity Stabinger at 100°C	mm^2/s	13	11.177	0.087	0.120
Pour Point Manual	°C	18	-15.4	9.7	9
Pour Point Automated 1°C int.	°C	13	-16.0	7.5	4.5
Sulfur	mg/kg	21	534	108	110
Water	mg/kg	25	43.3	62.0	162.0
Water Separability at 82°C, distilled water					
Time ≤ 3 mL emulsion	minutes	10	23.8	40.9	25

Parameter	unit	n	average	2.8 * sd	R(lit)
Time 37 mL water	minutes	11	22.7	40.1	25
Complete Break (40-40-0)	minutes	10	18.9	36.3	25
Calcium as Ca	mg/kg	30	39.7	6.3	10.2
Phosphorus as P	mg/kg	28	257	32	69
Zinc as Zn	mg/kg	31	269	49	39

Table 3: reproducibilities of tests on sample #20210

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

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

	November 2020	November 2019	November 2018	November 2017	November 2016
Number of reporting laboratories	41	35	35	45	43
Number of test results	533	504	465	610	597
Number of statistical outliers	23	23	18	28	30
Percentage of statistical outliers	4.3%	4.6%	3.9%	4.6%	5.0%

Table 4: comparison with previous proficiency tests

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

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

	November 2020	November 2019	November 2018	November 2017	November 2016
Total Acid Number	+	+/-	-	+	+
Density at 15°C	-	+	+	--	+
Flash Point PMcc	+/-	++	-	+/-	+
Foaming Tendency Seq. I	--	+	(--)	+/-	n.e.
Foaming Tendency Seq. II	+/-	-	-	-	+
Foaming Tendency Seq. III	(--)	(--)	(--)	n.e.	n.e.
Kinematic Viscosity at 40°C	++	+/-	++	+	+
Kinematic Viscosity at 100°C	+/-	+	+/-	+	+/-
Viscosity Index	+/-	-	+/-	+/-	--
Viscosity Stabinger at 40°C	++	+	++	-	+
Viscosity Stabinger at 100°C	+	-	-	-	+/-
Pour Point Manual	+/-	+	-	+/-	+/-
Pour Point Automated 1°C int.	-	-	+	-	-
Sulfur	+/-	-	+	+/-	-

	November 2020	November 2019	November 2018	November 2017	November 2016
Water	++	++	++	++	++
Water Separability	--	++	+	+	+
Calcium as Ca	+	+/-	-	+	n.e.
Phosphorus as P	++	+/-	+	+	+
Zinc as Zn	-	-	n.e.	-	-

Table 5: comparison determinations against the reference test methods
Results between brackets should be used with due care

The following performance categories were used:

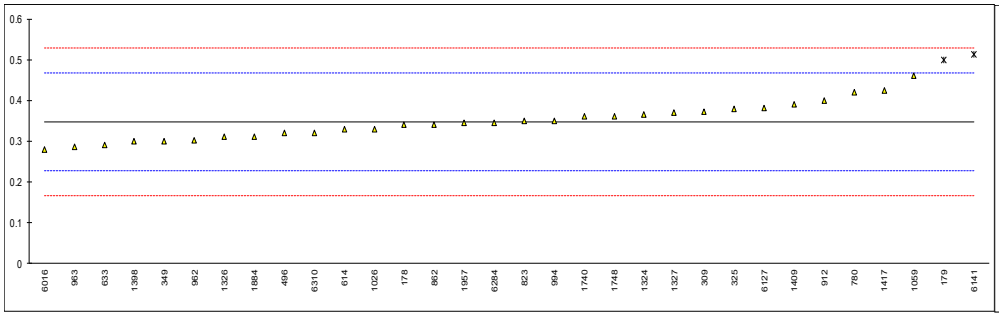
++: group performed much better than the reference test method
+ : group performed better than the reference test method
+/-: group performance equals the reference test method
- : group performed worse than the reference test method
-- : group performed much worse than the reference test method
n.e.: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

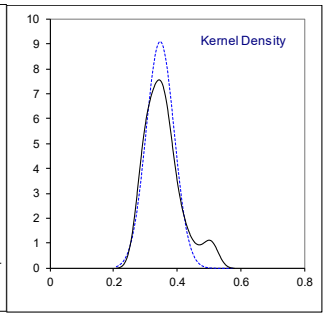
APPENDIX 1

Determination of Total Acid Number on sample #20210; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks	End point	Volume
178	D664-A	0.34		-0.13		Inflection Point	60 mL
179	D664-A	0.50	DG(0.05)	2.52		Inflection Point	60 mL
237		-----		-----		---	---
256		-----		-----		---	---
257		-----		-----		---	---
309	D664-A	0.3733		0.42		Buffer End Point (pH 10)	60 mL
325	D664-A	0.38		0.53		---	---
349	D664-A	0.30		-0.79		Inflection Point	125 mL
432		-----		-----		---	---
496	D664-A	0.32		-0.46		Buffer End Point (pH 10)	60 mL
614	D664-A	0.33		-0.29		---	---
633	D664-A	0.29		-0.95		Inflection Point	125 mL
780	D664-A	0.42		1.20		Buffer End Point (pH 10)	60 mL
823	D664-A	0.35		0.04		Inflection Point	125 mL
862	D664-A	0.34		-0.13		Inflection Point	60 mL
912	D664-A	0.4		0.87		---	---
962	D974	0.301		-0.77		Inflection Point	125 mL
963	D974	0.286		-1.02		---	---
994	D664-A	0.35		0.04		Inflection Point	125 mL
1011		-----		-----		---	---
1017		-----		-----		---	---
1026	D664-A	0.33		-0.29		Buffer End Point (pH 10)	125 mL
1059	ISO6619	0.46		1.86		Buffer End Point (pH 11)	60 mL
1146		-----		-----		Buffer End Point (pH 11)	125 mL
1150		-----		-----		---	---
1324	D664-A	0.366		0.30		Inflection Point	125 mL
1326	D974	0.31		-0.62		---	---
1327	D664-A	0.370		0.37		Inflection Point	60 mL
1398	D664-A	0.2998		-0.79		Inflection Point	60 mL
1409	D664-A	0.39		0.70		Buffer End Point (pH 11)	60 mL
1417	D664-A	0.424		1.26		Buffer End Point (pH 10)	60 mL
1660		-----		-----		---	---
1740	D664-A	0.36		0.20		Inflection Point	60 mL
1748	D664-A	0.36		0.20		Inflection Point	125 mL
1884	D664-A	0.31		-0.62		Buffer End Point (pH 11)	60 mL
1957	D664-A	0.345		-0.04		Buffer End Point (pH 11)	125 mL
6016	D664-A	0.279		-1.14		Inflection Point	60 mL
6127	D664-A	0.3806		0.54		Buffer End Point (pH 11)	125 mL
6141	D664-A	0.5125	C,DG(0.05)	2.73	fr.0.1125	Buffer End Point (pH 11)	60 mL
6284	D974	0.346		-0.03		Inflection Point	60 mL
6310	D664-A	0.32		-0.46		Buffer End Point (pH 10)	60 mL
6317		-----		-----		---	---

normality	OK
n	30
outliers	2
mean (n)	0.3477
st.dev. (n)	0.04388
R(calc.)	0.1229
st.dev.(D664-A:18e2, IP 60mL)	0.06041
R(D664-A:18e2, IP 60mL)	0.1691
Compare	
R(D664-A:18e2, BEP 60mL)	0.1977
R(D664-A:18e2, BEP 125mL)	0.1041
R(D664-A:18e2, IP 125mL)	0.0727





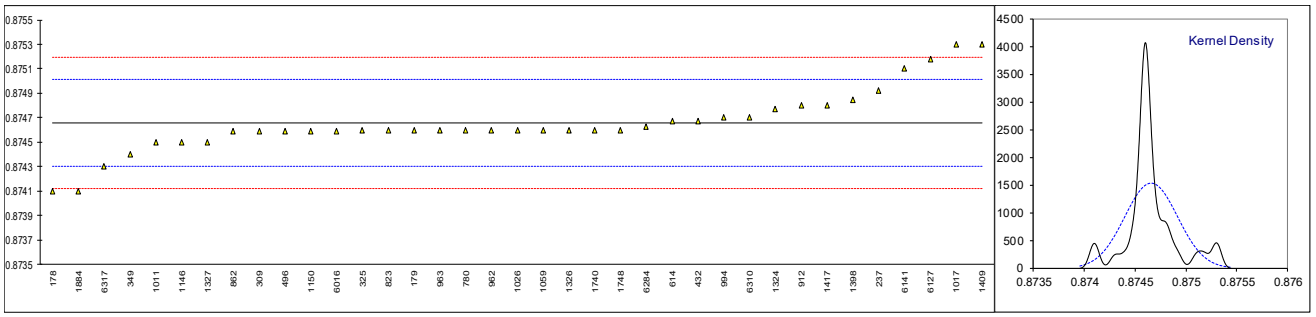
Determination of Copper Corrosion 3hrs at 50°C on sample #20210;

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D130	1A		-----	
237	D130	1A		-----	
256		-----		-----	
257		-----		-----	
309	D130	CUCOR1A		-----	
325	D130	1A		-----	
349	D130	1A		-----	
432		-----		-----	
496	ISO2160	1a		-----	
614		-----		-----	
633	D130	1a		-----	
780	D130	1a		-----	
823	D130	1a		-----	
862	D130	1a		-----	
912		-----		-----	
962		-----		-----	
963		-----		-----	
994	D130	1A		-----	
1011	D130	1a		-----	
1017		-----		-----	
1026	ISO2160	1A		-----	
1059	ISO2160	1a		-----	
1146		-----		-----	
1150		-----		-----	
1324	D130	1b		-----	
1326		-----		-----	
1327	D130	1b		-----	
1398		-----		-----	
1409	D130	1a		-----	
1417	IP154	1A		-----	
1660		-----		-----	
1740	D130	1A		-----	
1748	D130	1a		-----	
1884	D130	1a		-----	
1957		-----		-----	
6016		-----		-----	
6127	D130	1a		-----	
6141	D130	1a		-----	
6284		-----		-----	
6310		-----		-----	
6317	D130	1a		-----	
	n	24			
	mean (n)	1 (1A/ 1B)			

Determination of Density at 15°C on sample #20210; results in kg/L

lab	method	value	mark	z(targ)	remarks
178	D4052	0.8741		-3.12	
179	D4052	0.8746		-0.32	
237	D4052	0.87492		1.47	
256		-----		-----	
257		-----		-----	
309	D4052	0.87459	C	-0.38	First reported 0.87459 kg/m^3
325	D4052	0.8746		-0.32	
349	D4052	0.8744		-1.44	
432	ISO12185	0.87467		0.07	
496	ISO12185	0.87459		-0.38	
614	D4052	0.87467		0.07	
633		-----		-----	
780	ISO12185	0.8746		-0.32	
823	ISO12185	0.8746		-0.32	
862	D4052	0.87459		-0.38	
912	D1298	0.8748		0.80	
962	D4052	0.8746		-0.32	
963	D4052	0.8746		-0.32	
994	ISO12185	0.8747		0.24	
1011	D4052	0.8745		-0.88	
1017	D4052	0.8753		3.60	
1026	D4052	0.8746		-0.32	
1059	ISO12185	0.8746		-0.32	
1146	D4052	0.8745		-0.88	
1150	ISO12185	0.87459		-0.38	
1324	D4052	0.87477		0.63	
1326	D4052	0.8746		-0.32	
1327	D4052	0.8745		-0.88	
1398	D4052	0.87485		1.08	
1409	ISO12185	0.8753		3.60	
1417	IP365	0.8748		0.80	
1660		-----		-----	
1740	D7042	0.8746		-0.32	
1748	D4052	0.8746		-0.32	
1884	D4052	0.87410		-3.12	
1957		-----		-----	
6016	ISO12185	0.87459	C	-0.38	Reported 874.59 kg/L
6127	D4052	0.87518		2.92	
6141	D4052	0.8751		2.48	
6284	D4052	0.87463		-0.16	
6310	D4052	0.8747		0.24	
6317	D7042	0.8743	C	-2.00	First reported 874.3 kg/L

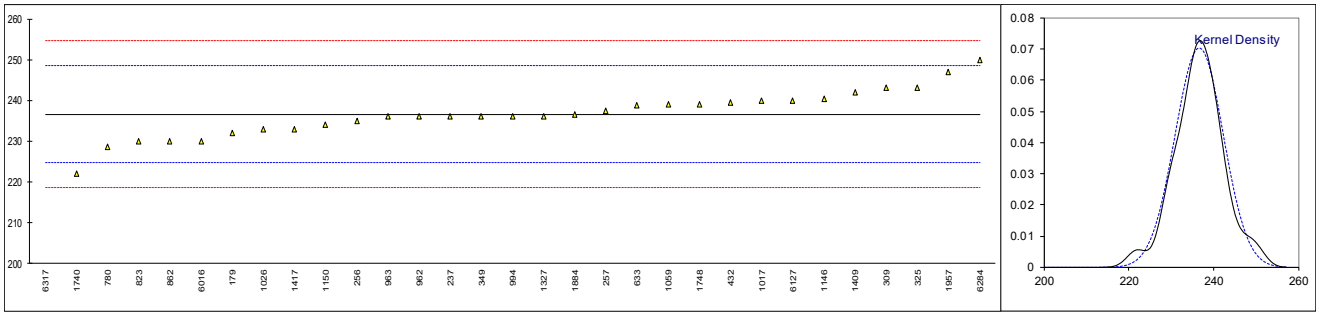
normality	suspect
n	37
outliers	0
mean (n)	0.87466
st.dev. (n)	0.000259
R(calc.)	0.00072
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005



Determination of Flash Point PMcc on sample #20210; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D93-A	232.0		-0.77	
237	D93-A	236.0		-0.11	
256	D3828	235.0		-0.27	
257	D3828	237.5		0.14	
309	D93-A	243.0		1.06	
325	D93-A	243.0		1.06	
349	D93-A	236		-0.11	
432	D93-A	239.5		0.48	
496		-----		-----	
614		-----		-----	
633	D93-A	238.72		0.35	
780	D93-A	228.5		-1.36	
823	ISO2719-A	230.0		-1.11	
862	D93-A	230		-1.11	
912		-----		-----	
962	D93-A	236.0		-0.11	
963	D93-A	236.0		-0.11	
994	D93-A	236.0		-0.11	
1011		-----		-----	
1017	D93-A	240		0.56	
1026	D93-A	233		-0.61	
1059	ISO2719-A	239.0		0.39	
1146	D93-A	240.5		0.64	
1150	ISO2719-A	234		-0.44	
1324		-----		-----	
1326		-----		-----	
1327	D93-A	236		-0.11	
1398		-----		-----	
1409	ISO2719-A	242.0		0.89	
1417	D93-A	233		-0.61	
1660		-----		-----	
1740	D93-A	222		-2.44	
1748	D93-A	239		0.39	
1884	D93-A	236.5		-0.02	
1957	D93-A	247.0		1.73	
6016	D93-B	230		-1.11	
6127	D93-A	240		0.56	
6141		-----		-----	
6284		250		2.23	
6310		-----		-----	
6317	D93-A	144.8	C,R(0.01)	-15.31	First reported 148.9

normality	OK
n	30
outliers	1
mean (n)	236.64
st.dev. (n)	5.664
R(calc.)	15.86
st.dev.(D93-A:20)	6.001
R(D93-A:20)	16.80

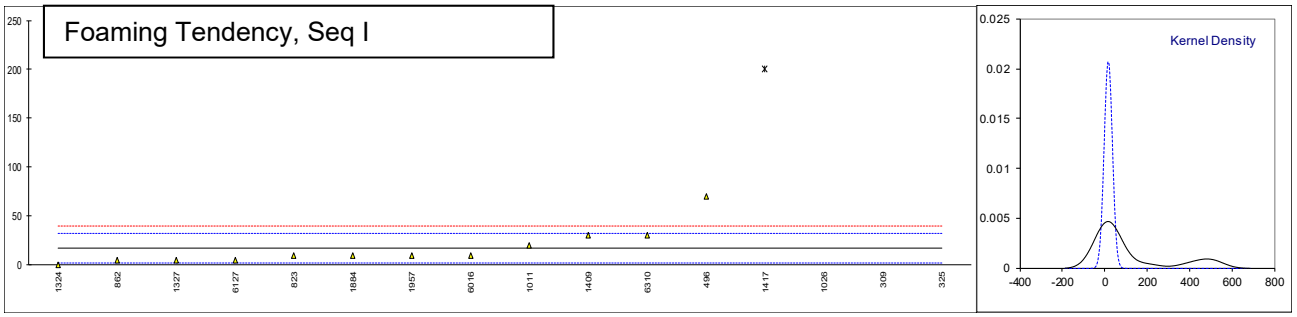


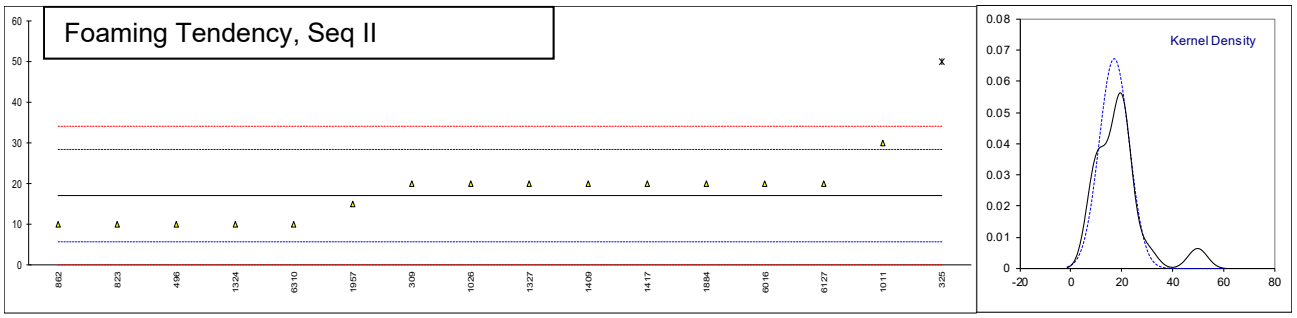
Determination of Foaming Characteristics, Foaming Tendency (at end of 5 min blowing period) on sample #20210; results in mL

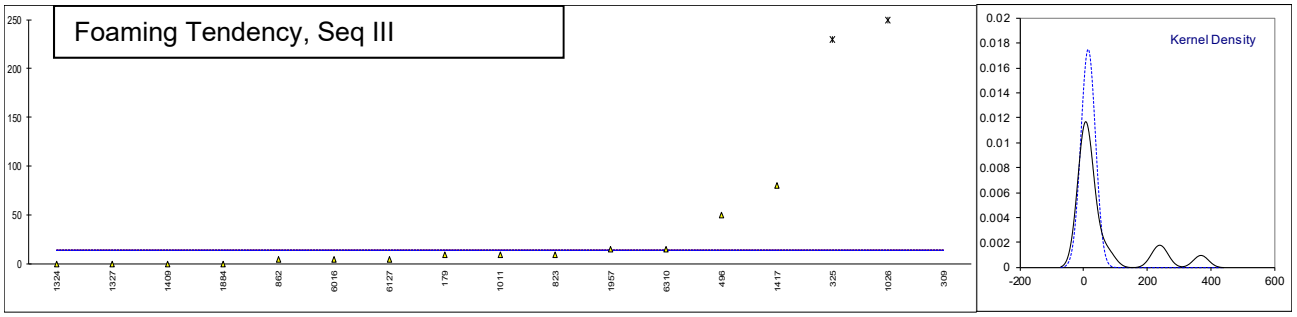
lab	method	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179	D892	-----		-----	-----		-----	10		-----
237	D892	-----		-----	-----		-----	-----		-----
256		-----		-----	-----		-----	-----		-----
257		-----		-----	-----		-----	-----		-----
309	D892	500	DG(0.05)	64.68	20		0.53	370	G(0.05)	-----
325	D892	500	DG(0.05)	64.68	50	G(0.01)	5.83	230	G(0.01)	-----
349		-----		-----	-----		-----	-----		-----
432	D892	-----		-----	-----		-----	-----		-----
496	D892	70		7.09	10		-1.24	50		-----
614		-----		-----	-----		-----	-----		-----
633		-----		-----	-----		-----	-----		-----
780		-----		-----	-----		-----	-----		-----
823	D892	10		-0.95	10		-1.24	10		-----
862	D892	5		-1.62	10		-1.24	5		-----
912		-----		-----	-----		-----	-----		-----
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
994		-----		-----	-----		-----	-----		-----
1011	D892	20		0.39	30		2.30	10		-----
1017		-----		-----	-----		-----	-----		-----
1026	D892	400	G(0.01)	51.29	20		0.53	250	G(0.05)	-----
1059		-----		-----	-----		-----	-----		-----
1146		-----		-----	-----		-----	-----		-----
1150		-----		-----	-----		-----	-----		-----
1324	D892	0		-2.29	10		-1.24	0		-----
1326		-----		-----	-----		-----	-----		-----
1327	D892	5		-1.62	20		0.53	0		-----
1398		-----		-----	-----		-----	-----		-----
1409	ISO6247	30		1.73	20		0.53	0		-----
1417	D892	200	G(0.05)	24.50	20		0.53	80		-----
1660		-----		-----	-----		-----	-----		-----
1740		-----		-----	-----		-----	-----		-----
1748		-----		-----	-----		-----	-----		-----
1884	D892	10		-0.95	20		0.53	0		-----
1957	D892	10		-0.95	15		-0.35	15		-----
6016	D892	10		-0.95	20		0.53	5		-----
6127	D892	5		-1.62	20		0.53	5		-----
6141		-----		-----	-----		-----	-----		-----
6284		-----		-----	-----		-----	-----		-----
6310	D892	30	C	1.73	10		-1.24	15		-----
6317		-----		-----	-----		-----	-----		-----

normality	not OK	OK	not OK
n	12	15	14
outliers	4	1	3
mean (n)	17.08	17.00	14.64
st.dev. (n)	19.242	5.916	22.741
R(calc.)	53.88	16.57	63.68
st.dev.(D892:18)	7.466	5.664	(2.301)
R(D892:18)	20.90	15.86	(6.44)

Lab 6310: first reported 80


Foaming Tendency, Seq I
Kernel Density


Foaming Tendency, Seq II
Kernel Density


Foaming Tendency, Seq III
Kernel Density

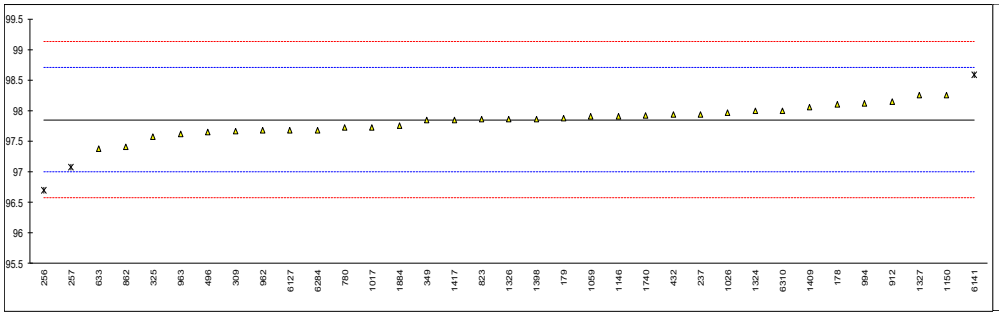
Determination of Foaming Characteristics, Foaming Stability (at end of 10 min settling period) on sample #20210; results in mL

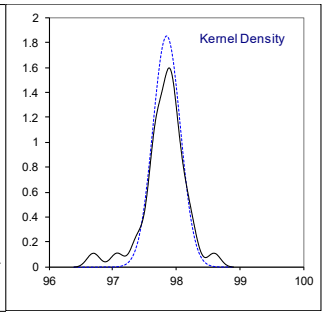
lab	method	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179	D892	-----		-----	0		-----	-----		-----
237	D892	-----		-----	-----		-----	-----		-----
256		-----		-----	-----		-----	-----		-----
257		-----		-----	-----		-----	-----		-----
309	D892	0		-----	0		-----	0		-----
325	D892	10	False +	-----	0		-----	0		-----
349		-----		-----	-----		-----	-----		-----
432		-----		-----	-----		-----	-----		-----
496	D892	0		-----	0		-----	0		-----
614		-----		-----	-----		-----	-----		-----
633		-----		-----	-----		-----	-----		-----
780		-----		-----	-----		-----	-----		-----
823	D892	0		-----	0		-----	0		-----
862	D892	0		-----	0		-----	0		-----
912		-----		-----	-----		-----	-----		-----
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
994		-----		-----	-----		-----	-----		-----
1011	D892	0		-----	0		-----	0		-----
1017		-----		-----	-----		-----	-----		-----
1026	D892	120	False +	-----	0		-----	0		-----
1059		-----		-----	-----		-----	-----		-----
1146		-----		-----	-----		-----	-----		-----
1150		-----		-----	-----		-----	-----		-----
1324	D892	0		-----	0		-----	0		-----
1326		-----		-----	-----		-----	-----		-----
1327	D892	0		-----	0		-----	0		-----
1398		-----		-----	-----		-----	-----		-----
1409	ISO6247	0		-----	0		-----	0		-----
1417	D892	0		-----	0		-----	0		-----
1660		-----		-----	-----		-----	-----		-----
1740		-----		-----	-----		-----	-----		-----
1748		-----		-----	-----		-----	-----		-----
1884	D892	0		-----	0		-----	0		-----
1957	D892	0		-----	0		-----	0		-----
6016	D892	0		-----	0		-----	0		-----
6127	D892	0		-----	0		-----	0		-----
6141		-----		-----	-----		-----	-----		-----
6284		-----		-----	-----		-----	-----		-----
6310	D892	0		-----	0		-----	0		-----
6317		-----		-----	-----		-----	-----		-----
	n	14			17			16		
	mean (n)	0			0			0		

Determination of Kinematic Viscosity at 40°C on sample #20210; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178	D445	98.1		0.59	
179	D445	97.88		0.07	
237	D445	97.941		0.22	
256	D7279 corrected to D445	96.7	R(0.05)	-2.69	
257	D7279 corrected to D445	97.07	R(0.05)	-1.83	
309	D445	97.66		-0.44	
325	D445	97.57		-0.65	
349	D445	97.84		-0.02	
432	D445	97.94		0.21	
496	D445	97.644		-0.48	
614		-----		-----	
633	D7279 corrected to D445	97.380		-1.10	
780	D445	97.72		-0.30	
823	ISO3104	97.86		0.03	
862	D445	97.40		-1.05	
912	D445	98.15		0.71	
962	D445	97.68		-0.40	
963	D445	97.62		-0.54	
994	D445	98.12		0.64	
1011		-----		-----	
1017	D445	97.72		-0.30	
1026	D445	97.96		0.26	
1059	ISO3104	97.90		0.12	
1146	D445	97.91		0.14	
1150	ISO3104	98.2508		0.94	
1324	D445	97.99		0.33	
1326	D445	97.86		0.03	
1327	D445	98.25		0.94	
1398	D445	97.861		0.03	
1409	D445	98.06	C	0.50	First reported 99.98
1417	D445	97.85		0.00	
1660		-----		-----	
1740	D445	97.92		0.17	
1748		-----		-----	
1884	D445	97.75		-0.23	
1957		-----		-----	
6016		-----		-----	
6127	D445	97.68		-0.40	
6141	D445	98.5922	R(0.05)	1.74	
6284	D445	97.6845		-0.38	
6310	D7279 corrected to D445	98.0		0.36	
6317		-----		-----	

normality	OK
n	32
outliers	3
mean (n)	97.8485
st.dev. (n)	0.21517
R(calc.)	0.6025
st.dev.(D445:19a)	0.42634
R(D445:19a)	1.1938

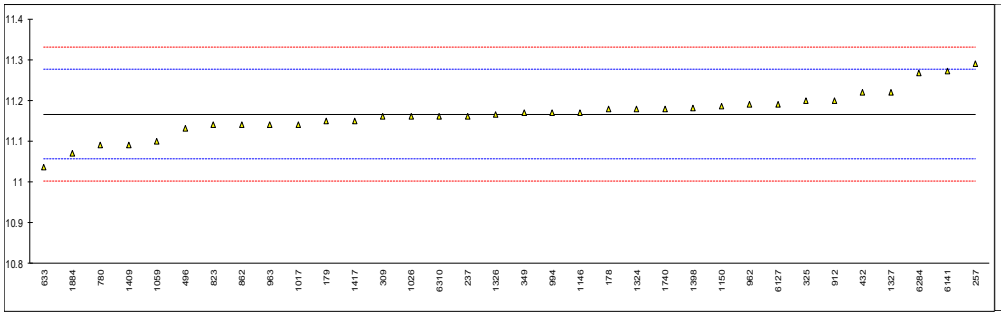


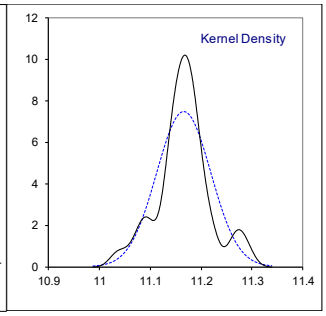


Determination of Kinematic Viscosity at 100°C on sample #20210; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178	D445	11.18		0.25	
179	D445	11.15		-0.29	
237	D445	11.161		-0.09	
256		-----		-----	
257	D7279 corrected to D445	11.29		2.25	
309	D445	11.16		-0.11	
325	D445	11.20		0.61	
349	D445	11.17		0.07	
432	D445	11.22		0.98	
496	D445	11.131		-0.64	
614		-----		-----	
633	D7279 corrected to D445	11.037		-2.35	
780	D445	11.09		-1.38	
823	D445	11.14		-0.48	
862	D445	11.14		-0.48	
912	D445	11.20		0.61	
962	D445	11.19		0.43	
963	D445	11.14		-0.48	
994	D445	11.17		0.07	
1011		-----		-----	
1017	D445	11.14		-0.48	
1026	D445	11.16		-0.11	
1059	ISO3104	11.10		-1.20	
1146	D445	11.17		0.07	
1150	ISO3104	11.1859	C	0.36	First reported 10.9888
1324	D445	11.18		0.25	
1326	D445	11.165		-0.02	
1327	D445	11.22		0.98	
1398	D445	11.182		0.29	
1409	D445	11.09		-1.38	
1417	D445	11.15		-0.29	
1660		-----		-----	
1740	D445	11.18		0.25	
1748		-----		-----	
1884	D445	11.07		-1.75	
1957		-----		-----	
6016		-----		-----	
6127	D445	11.19		0.43	
6141	D445	11.2719		1.92	
6284	D445	11.2674		1.84	
6310	D7279 corrected to D445	11.16		-0.11	
6317		-----		-----	

normality	OK
n	34
outliers	0
mean (n)	11.1662
st.dev. (n)	0.05331
R(calc.)	0.1493
st.dev.(D445:19a)	0.05503
R(D445:19a)	0.1541

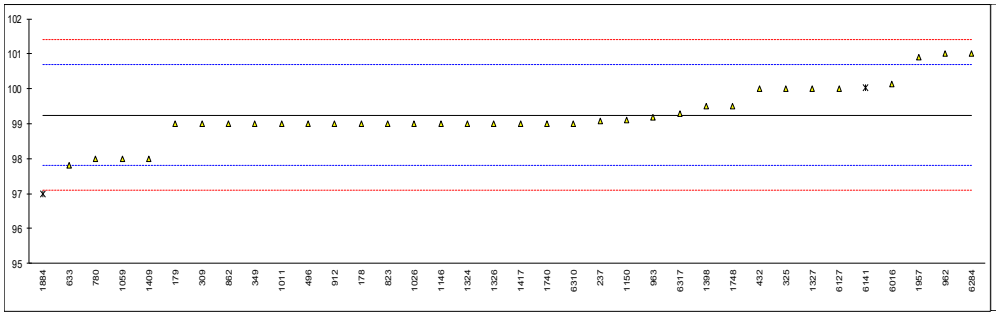


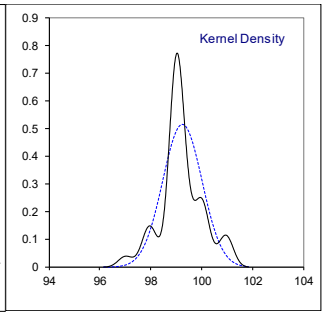


Determination of Viscosity Index on sample #20210;

lab	method	value	mark	z(targ)	iis calc		remarks
178	D2270	99		-0.35	99		
179	D2270	99		-0.35	99		
237	D2270	99.09		-0.22	99		
256		-----		-----	-----		
257		-----		-----	102	ex	Test result excluded, outlier in KV 40°C
309	D2270	99		-0.35	99		
325	D2270	100		1.05	100		
349	D2270	99		-0.35	99		
432	D2270	100.0		1.05	100		
496	D2270	99.0		-0.35	99		
614		-----		-----	-----		
633	D2270	97.8		-2.03	98		
780	D2270	98		-1.75	98		
823	D2270	99		-0.35	99		
862	D2270	99		-0.35	99		
912	D2270	99		-0.35	99		
962	D2270	101		2.45	100		
963	D2270	99.175		-0.10	99		
994		-----		-----	99		
1011	D2270	99		-0.35	-----		
1017		-----		-----	99		
1026	D2270	99		-0.35	99		
1059	ISO2909	98		-1.75	98		
1146	D2270	99		-0.35	99		
1150	ISO2909	99.1	C	-0.21	99		First reported 94.7
1324	D2270	99		-0.35	99		
1326	D2270	99		-0.35	99		
1327	D2270	100		1.05	100		
1398	D2270	99.50		0.35	99		
1409	D2270	98	C	-1.75	98		First reported 95
1417	D2270	99		-0.35	99		
1660		-----		-----	-----		
1740	D2270	99		-0.35	99		
1748	D2270	99.5		0.35	-----		
1884	D2270	97.0	G(0.01)	-3.15	98		
1957	D2270	100.9		2.31	-----		
6016	D2270	100.129		1.23	-----		
6127	D2270	100		1.05	100		
6141	D2270	100.032	ex	1.10	100	ex	Test result excluded, outlier in KV 40°C
6284	D2270	101		2.45	101		
6310	D2270	99		-0.35	99		
6317	D2270	99.291		0.06	-----		

normality	OK	suspect
n	34	32
outliers	1 (+1ex)	0 (+2ex)
mean (n)	99.25	99.06
st.dev. (n)	0.775	0.669
R(calc.)	2.17	1.87
st.dev.(D2270:10)	0.714	0.714
R(D2270:10)	2	2

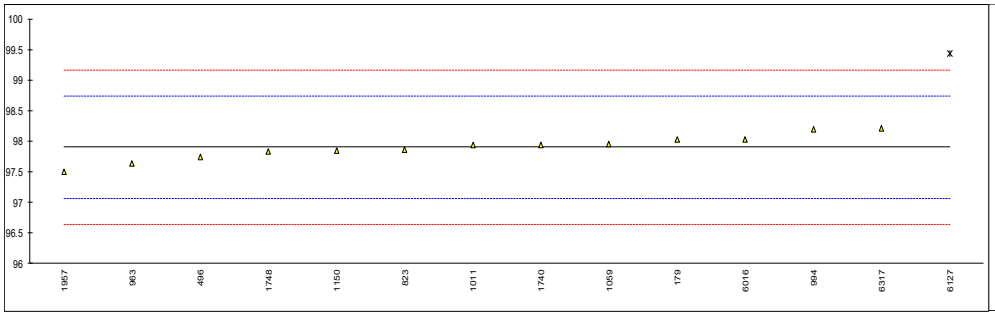


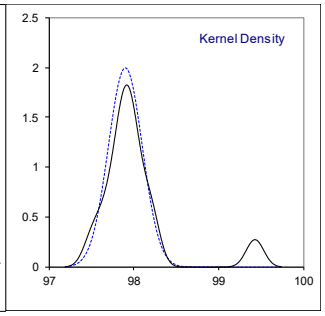


Determination of Viscosity Stabinger at 40°C on sample #20210; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D7042	98.03		0.31	
237		-----		-----	
256		-----		-----	
257		-----		-----	
309		-----		-----	
325		-----		-----	
349		-----		-----	
432		-----		-----	
496	D7042	97.736		-0.39	
614		-----		-----	
633		-----		-----	
780		-----		-----	
823	D7042	97.86		-0.10	
862		-----		-----	
912		-----		-----	
962		-----		-----	
963	D7042	97.64		-0.62	
994	D7042	98.19		0.69	
1011	D7042	97.94		0.09	
1017		-----		-----	
1026		-----		-----	
1059	D7042	97.95		0.12	
1146		-----		-----	
1150	D7042	97.841	C	-0.14	First reported 99.871
1324		-----		-----	
1326		-----		-----	
1327		-----		-----	
1398		-----		-----	
1409		-----		-----	
1417		-----		-----	
1660		-----		-----	
1740	D7042	97.94		0.09	
1748	D7042	97.832		-0.16	
1884		-----		-----	
1957	D7042	97.505		-0.94	
6016	D7042	98.031		0.31	
6127	D7042	99.43	G(0.01)	3.63	
6141		-----		-----	
6284		-----		-----	
6310		-----		-----	
6317	D7042	98.211		0.74	

normality	OK
n	13
outliers	1
mean (n)	97.9005
st.dev. (n)	0.20017
R(calc.)	0.5605
st.dev.(D7042:20)	0.42121
R(D7042:20)	1.1794

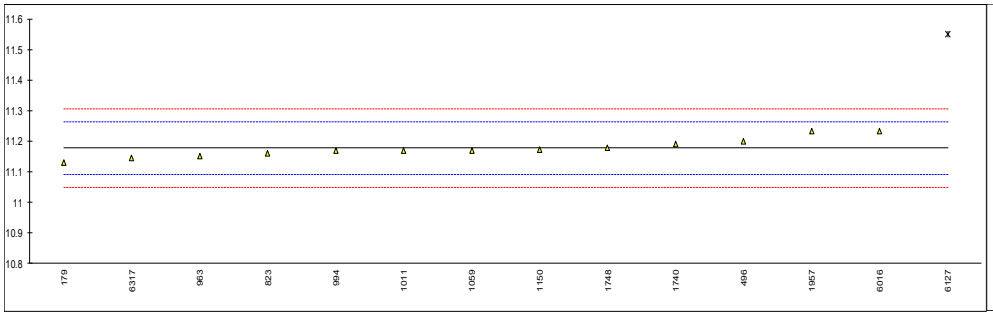


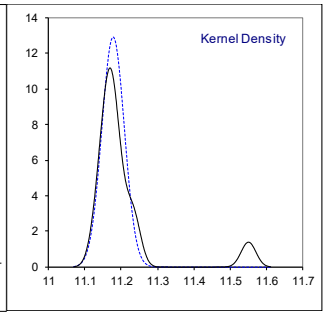


Determination of Viscosity Stabinger at 100°C on sample #20210; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D7042	11.13		-1.10	
237		-----		-----	
256		-----		-----	
257		-----		-----	
309		-----		-----	
325		-----		-----	
349		-----		-----	
432		-----		-----	
496	D7042	11.201		0.56	
614		-----		-----	
633		-----		-----	
780		-----		-----	
823	D7042	11.16		-0.40	
862		-----		-----	
912		-----		-----	
962		-----		-----	
963	D7042	11.15		-0.63	
994	D7042	11.17		-0.16	
1011	D7042	11.17		-0.16	
1017		-----		-----	
1026		-----		-----	
1059	D7042	11.17		-0.16	
1146		-----		-----	
1150	D7042	11.171	C	-0.14	First reported 11.504
1324		-----		-----	
1326		-----		-----	
1327		-----		-----	
1398		-----		-----	
1409		-----		-----	
1417		-----		-----	
1660		-----		-----	
1740	D7042	11.19		0.30	
1748	D7042	11.179		0.04	
1884		-----		-----	
1957	D7042	11.233		1.30	
6016	D7042	11.233		1.30	
6127	D7042	11.55	G(0.01)	8.68	
6141		-----		-----	
6284		-----		-----	
6310		-----		-----	
6317	D7042	11.145		-0.75	

normality	OK
n	13
outliers	1
mean (n)	11.1771
st.dev. (n)	0.03090
R(calc.)	0.0865
st.dev.(D7042:20)	0.04294
R(D7042:20)	0.1202

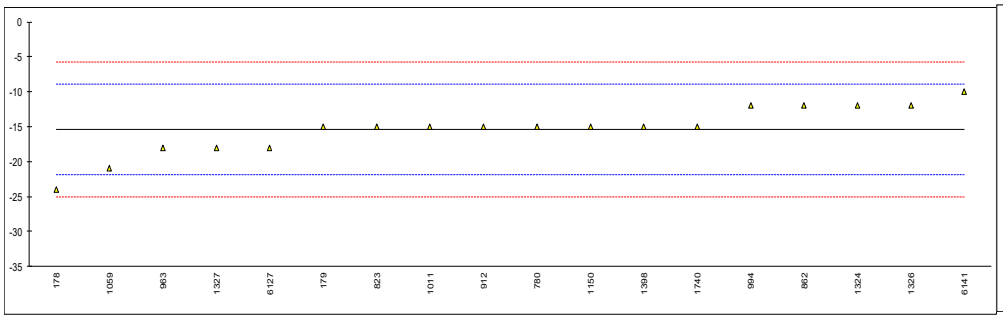


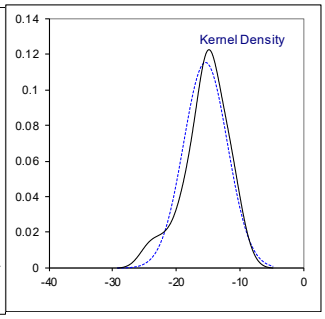


Determination of Pour Point, Manual on sample #20210; results in °C

lab	method	value	mark	z(targ)	remarks
178	D97	-24		-2.68	
179	D97	-15		0.12	
237		-----		-----	
256		-----		-----	
257		-----		-----	
309		-----		-----	
325		-----		-----	
349		-----		-----	
432		-----		-----	
496		-----		-----	
614		-----		-----	
633		-----		-----	
780	D97	-15		0.12	
823	ISO3016	-15		0.12	
862	D97	-12		1.05	
912	D97	-15		0.12	
962		-----		-----	
963	D97	-18		-0.81	
994	D97	-12		1.05	
1011	D97	-15		0.12	
1017		-----		-----	
1026		-----		-----	
1059	ISO3016	-21		-1.75	
1146		-----		-----	
1150	ISO3016	-15		0.12	
1324	D97	-12		1.05	
1326	D97	-12		1.05	
1327	D97	-18		-0.81	
1398	D97	-15		0.12	
1409		-----		-----	
1417		-----		-----	
1660		-----		-----	
1740	D97	-15		0.12	
1748		-----		-----	
1884		-----		-----	
1957		-----		-----	
6016		-----		-----	
6127	D97	-18		-0.81	
6141	D6892	-10		1.68	
6284		-----		-----	
6310		-----		-----	
6317		-----		-----	

normality	suspect
n	18
outliers	0
mean (n)	-15.39
st.dev. (n)	3.449
R(calc.)	9.66
st.dev.(D97:17b)	3.214
R(D97:17b)	9

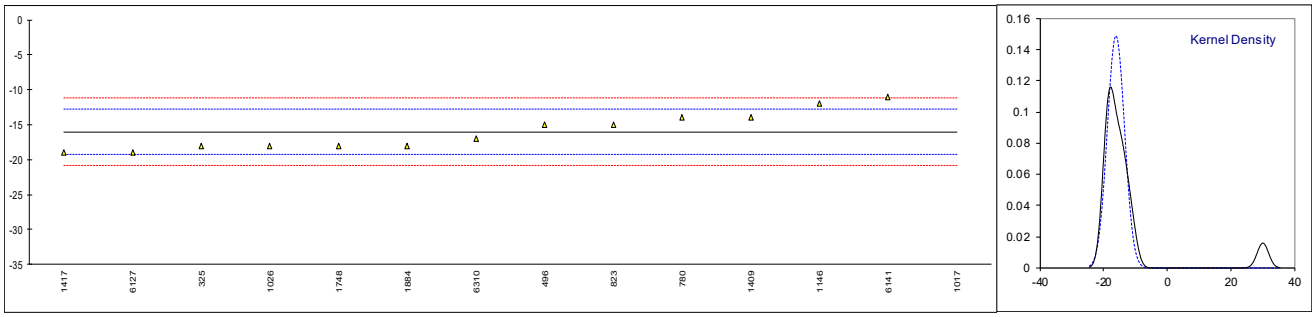




Determination of Pour Point, Automated, 1°C interval on sample #20210; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
256		-----		-----	
257		-----		-----	
309		-----		-----	
325	D5950	-18		-1.24	
349		-----		-----	
432		-----		-----	
496	D5950	-15		0.62	
614		-----		-----	
633		-----		-----	
780	D5950	-14		1.24	
823	D5950	-15		0.62	
862		-----		-----	
912		-----		-----	
962		-----		-----	
963		-----		-----	
994		-----		-----	
1011		-----		-----	
1017	D5950	30.1	G(0.01)	28.68	
1026	D5950	-18		-1.24	
1059		-----		-----	
1146	D5949	-12		2.49	
1150		-----		-----	
1324		-----		-----	
1326		-----		-----	
1327		-----		-----	
1398		-----		-----	
1409	D5950	-14		1.24	
1417	D5950	-19		-1.87	
1660		-----		-----	
1740		-----		-----	
1748	D7346	-18		-1.24	
1884	D5950	-18		-1.24	
1957		-----		-----	
6016		-----		-----	
6127	D5950	-19		-1.87	
6141	D6892	-11		3.11	
6284		-----		-----	
6310	D5950	-17		-0.62	
6317		-----		-----	

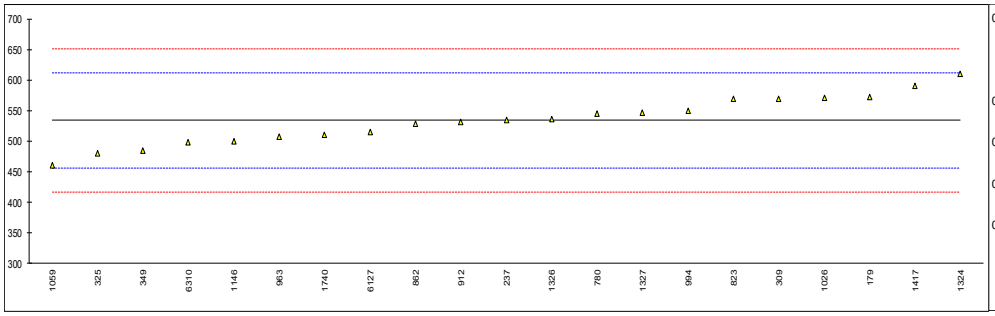
normality	OK
n	13
outliers	1
mean (n)	-16.00
st.dev. (n)	2.677
R(calc.)	7.50
st.dev.(D5950:14)	1.607
R(D5950:14)	4.5

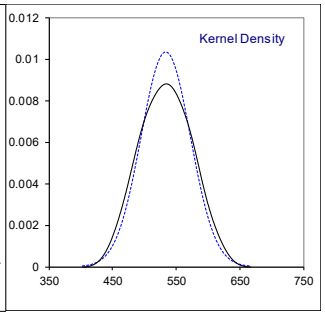


Determination of Sulfur on sample #20210; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D4294	573		1.00	
237	D4294	535		0.03	
256		-----		-----	
257		-----		-----	
309	D4294	570		0.92	
325	D5185	480		-1.37	
349	D2622	485		-1.24	
432		-----		-----	
496		-----		-----	
614		-----		-----	
633		-----		-----	
780	D4294	545		0.28	
823	D5453	569		0.90	
862	D2622	528		-0.15	
912	D4294	532		-0.05	
962		-----		-----	
963	D4294	508		-0.66	
994	D4294	549		0.39	
1011		-----		-----	
1017		-----		-----	
1026	D2622	571		0.95	
1059	ISO14596	460		-1.88	
1146	D4294	500		-0.86	
1150		-----		-----	
1324	D4294	610		1.94	
1326	D4294	536		0.05	
1327	D5185	547		0.34	
1398		-----		-----	
1409		-----		-----	
1417	In house	590		1.43	
1660		-----		-----	
1740	D4294	510		-0.61	
1748		-----		-----	
1884		-----		-----	
1957		-----		-----	
6016		-----		-----	
6127	D5185	514.7		-0.49	
6141		-----		-----	
6284		-----		-----	
6310	D7751	498		-0.91	
6317		-----		-----	

normality	OK
n	21
outliers	0
mean (n)	533.84
st.dev. (n)	38.569
R(calc.)	107.99
st.dev.(D4294:16e1)	39.249
R(D4294:16e1)	109.90

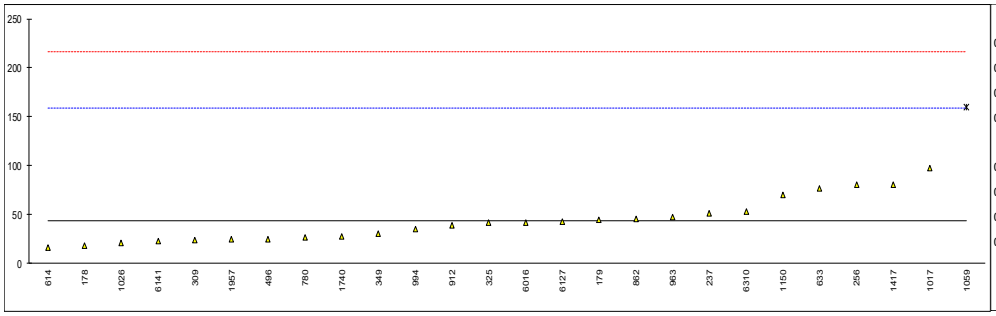


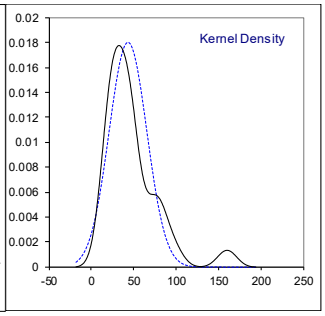


Determination of Water on sample #20210; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D6304-C	18		-0.44	
179	D6304-C	45		0.03	
237	D6304-C	51.55		0.14	
256	D7889	80		0.63	
257		-----		-----	
309	D6304-C	24		-0.33	
325	D6304-C	42		-0.02	
349	D6304-C	30		-0.23	
432		-----		-----	
496	D6304-A	25		-0.32	
614	D6304-C	16		-0.47	
633	D6304-C	76.7		0.58	
780	D6304-C	26.5		-0.29	
823	D6304-C	<10		-----	
862	D6304-C	45.2		0.03	
912	D6304-C	39		-0.07	
962		-----		-----	
963	D6304-A	47		0.06	
994	D6304-A	35		-0.14	
1011		-----		-----	
1017	D6304-A	97.4		0.93	
1026	D6304-C	21		-0.39	
1059	D6304-C	160	R(0.01)	2.02	
1146	D6304-C	<100		-----	
1150	ISO12937	70		0.46	
1324		-----		-----	
1326		-----		-----	
1327		-----		-----	
1398		-----		-----	
1409		-----		-----	
1417	D6304-A	80		0.63	
1660		-----		-----	
1740	D6304-C	28		-0.26	
1748		-----		-----	
1884		-----		-----	
1957	D6304-A	24.8		-0.32	
6016	D6304	42.2		-0.02	
6127	D6304-A	42.2342		-0.02	
6141	D1533	23.2		-0.35	
6284		-----		-----	
6310	D6304-C	53		0.17	
6317		-----		-----	

normality	OK
n	25
outliers	1
mean (n)	43.311
st.dev. (n)	22.1287
R(calc.)	61.960
st.dev.(D6304:16e1)	57.8730
R(D6304:16e1)	162.044



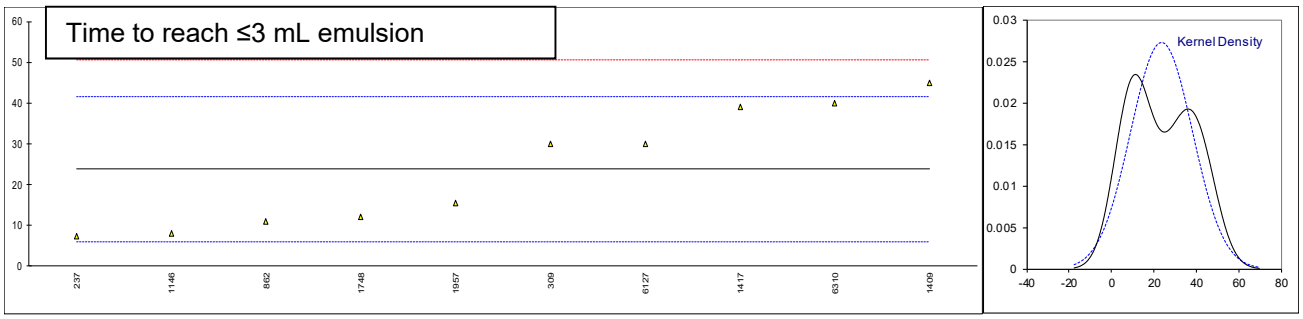


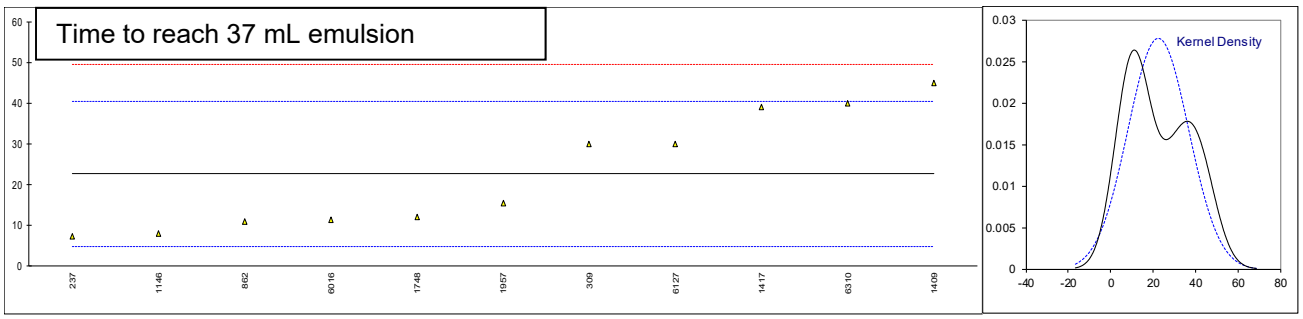
Determination of Water Separability at 82°C, distilled water on sample #20210; results in minutes

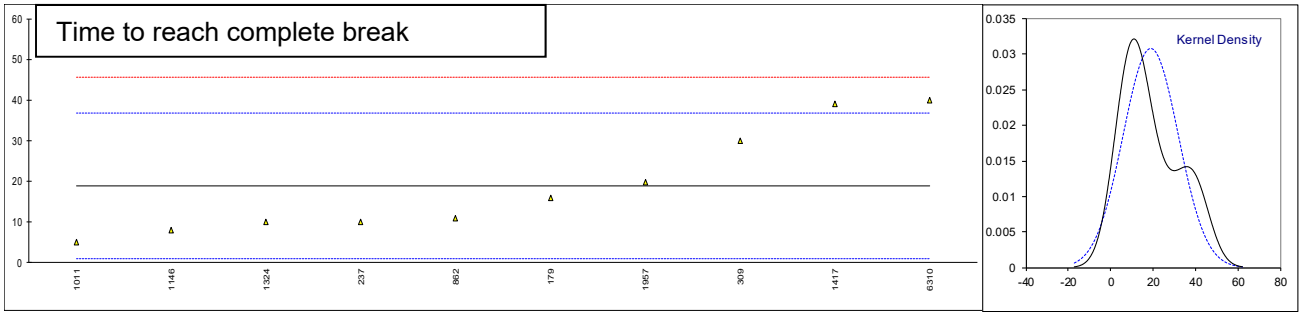
lab	method	≤3 mL emulsion	m.	z(targ)	37 mL water	m.	z(targ)	complete break (40-40-0)	m.	z(targ)	test aborted		time aborted	
178		-----		-----	-----		-----	-----		-----	-----		-----	
179	D1401	-----		-----	-----		-----	16		-0.32	-----		-----	
237	D1401	7.3		-1.84	7.4		-1.71	10.1		-0.98	No		-----	
256		-----		-----	-----		-----	-----		-----	-----		-----	
257		-----		-----	-----		-----	-----		-----	-----		-----	
309	D1401	30	C	0.70	30	C	0.82	30	C	1.24	Yes	C	30	C
325	D1401	>60	f+	-----	>60	f+	-----	>60	f+	-----	Yes		60	
349		-----		-----	-----		-----	-----		-----	-----		-----	
432		-----		-----	-----		-----	-----		-----	-----		-----	
496		-----		-----	-----		-----	-----		-----	Yes		30	
614		-----		-----	-----		-----	-----		-----	-----		-----	
633		-----		-----	-----		-----	-----		-----	-----		-----	
780		-----		-----	-----		-----	-----		-----	-----		-----	
823		-----		-----	-----		-----	-----		-----	-----		-----	
862	D1401	11		-1.43	11		-1.30	11		-0.88	No		-----	
912		-----		-----	-----		-----	-----		-----	-----		-----	
962		-----		-----	-----		-----	-----		-----	-----		-----	
963		-----		-----	-----		-----	-----		-----	-----		-----	
994		-----		-----	-----		-----	-----		-----	-----		-----	
1011		-----		-----	-----		-----	5		-1.56	-----		-----	
1017		-----		-----	-----		-----	-----		-----	-----		-----	
1026	D1401	-----		-----	-----		-----	-----		-----	Yes		60	
1059		-----		-----	-----		-----	-----		-----	-----		-----	
1146	D1401	8		-1.77	8		-1.64	8		-1.22	No		-----	
1150		-----		-----	-----		-----	-----		-----	-----		-----	
1324	D1401	-----		-----	-----		-----	10		-1.00	-----		-----	
1326		-----		-----	-----		-----	-----		-----	-----		-----	
1327		-----		-----	-----		-----	-----		-----	-----		-----	
1398		-----		-----	-----		-----	-----		-----	-----		-----	
1409	ISO6614	45		2.38	45		2.50	> 45		-----	Yes		45	
1417	D1401	39		1.71	39		1.83	39		2.25	No		-----	
1660		-----		-----	-----		-----	-----		-----	-----		-----	
1740		-----		-----	-----		-----	-----		-----	-----		-----	
1748	D1401	12		-1.32	12		-1.19	-----		-----	-----		-----	
1884		-----		-----	-----		-----	-----		-----	-----		-----	
1957	D1401	15.37		-0.94	15.43		-0.81	19.79		0.10	Yes		30	
6016	D1401	-----		-----	11.30		-1.27	-----		-----	-----		-----	
6127	D1401	30		0.70	30		0.82	-----		-----	Yes		60	
6141		-----		-----	-----		-----	-----		-----	-----		-----	
6284		-----		-----	-----		-----	-----		-----	-----		-----	
6310	D1401	40		1.82	40		1.94	40		2.36	No		-----	
6317		-----		-----	-----		-----	-----		-----	-----		-----	

normality	OK	OK	OK
n	10	11	10
outliers	0	0	0
mean (n)	23.77	22.65	18.89
st.dev. (n)	14.590	14.329	12.965
R(calc.)	40.85	40.12	36.30
st.dev.(D1401:18a)	8.929	8.929	8.929
R(D1401:18a)	25	25	25

Lab 309: First reported >60, >60, >60, No, >60
Lab 325: f+ = possibly a false positive test result?


Time to reach ≤3 mL emulsion
Kernel Density


Time to reach 37 mL emulsion
Kernel Density


Time to reach complete break
Kernel Density

Determination of Water Separability at 82°C, distilled water on sample #20210; results in mL
--- Continued ----

lab	method	oil	mark	z(targ)	water	mark	z(targ)	emulsion	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179	D1401	40		-----	37		-----	3		-----
237	D1401	40.0		-----	40.0		-----	0.0		-----
256		-----		-----	-----		-----	-----		-----
257		-----		-----	-----		-----	-----		-----
309	D1401	40	C	-----	40	C	-----	0	C	-----
325	D1401	0		-----	28		-----	52		-----
349		-----		-----	-----		-----	-----		-----
432		-----		-----	-----		-----	-----		-----
496	D1401	40		-----	30		-----	10		-----
614		-----		-----	-----		-----	-----		-----
633		-----		-----	-----		-----	-----		-----
780		-----		-----	-----		-----	-----		-----
823		-----		-----	-----		-----	-----		-----
862		-----		-----	-----		-----	-----		-----
912		-----		-----	-----		-----	-----		-----
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
994		-----		-----	-----		-----	-----		-----
1011		-----		-----	-----		-----	-----		-----
1017		-----		-----	-----		-----	-----		-----
1026	D1401	1		-----	0		-----	79		-----
1059		-----		-----	-----		-----	-----		-----
1146	D1401	40		-----	40		-----	0		-----
1150		-----		-----	-----		-----	-----		-----
1324		-----		-----	-----		-----	-----		-----
1326		-----		-----	-----		-----	-----		-----
1327		-----		-----	-----		-----	-----		-----
1398		-----		-----	-----		-----	-----		-----
1409	ISO6614	41		-----	37		-----	2		-----
1417	D1401	40		-----	40		-----	0		-----
1660		-----		-----	-----		-----	-----		-----
1740		-----		-----	-----		-----	-----		-----
1748		-----		-----	-----		-----	-----		-----
1884		-----		-----	-----		-----	-----		-----
1957	D1401	40		-----	40		-----	0		-----
6016		-----		-----	-----		-----	-----		-----
6127	D1401	40		-----	38		-----	2		-----
6141		-----		-----	-----		-----	-----		-----
6284		-----		-----	-----		-----	-----		-----
6310		-----		-----	-----		-----	-----		-----
6317		-----		-----	-----		-----	-----		-----

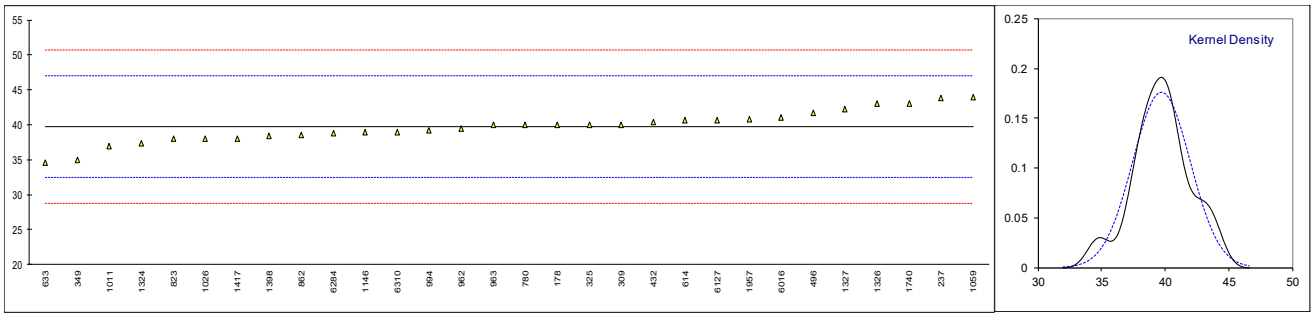
Lab 309: First reported 2, 11, 67

Determination of Calcium as Ca on sample #20210; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	40		0.07	
179		-----		-----	
237	D5185	43.80		1.11	
256		-----		-----	
257		-----		-----	
309	D5185	40.03		0.08	
325	D5185	40		0.07	
349		35		-1.30	
432	D5185	40.37		0.18	
496		41.7		0.54	
614	D5185	40.63		0.25	
633	D6595	34.6		-1.40	
780	D5185	40.0		0.07	
823	D5185	38		-0.47	
862	D5185	38.6		-0.31	
912		-----		-----	
962	D5185	39.5		-0.06	
963	D5185	39.93		0.05	
994	D5185	39.24		-0.13	
1011		37		-0.75	
1017		-----		-----	
1026	D5185	38		-0.47	
1059	In house	44		1.17	
1146	In house	39		-0.20	
1150		-----		-----	
1324	D5185	37.4		-0.64	
1326	D5185	43		0.90	
1327	D5185	42.3		0.70	
1398	D4951	38.39		-0.37	
1409		-----		-----	
1417	D5185	38		-0.47	
1660		-----		-----	
1740	D5185	43	C	0.90	First reported 29
1748		-----		-----	
1884		-----		-----	
1957	D5185	40.795		0.29	
6016	D5185	41.1		0.38	
6127	D5185	40.7		0.27	
6141		-----		-----	
6284		38.8120		-0.25	
6310	D7751	39		-0.20	
6317		-----		-----	

	normality	OK
	n	30
	outliers	0
	mean (n)	39.730
	st.dev. (n)	2.2592
	R(calc.)	6.326
	st.dev.(Horwitz)	3.6521
	R(Horwitz)	10.226
Compare		
	R(D5185:18)	1.799

Application range: 40 - 9000 mg/kg

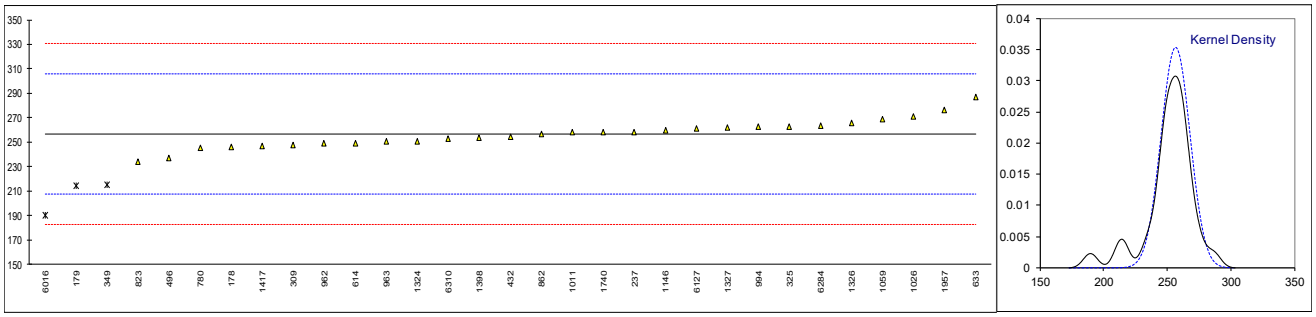


Determination of Phosphorus as P on sample #20210; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	246		-0.44	
179	D5185	214	R(0.05)	-1.74	
237	D5185	258.4		0.07	
256		-----		-----	
257		-----		-----	
309	D5185	247.82		-0.36	
325	D5185	263		0.25	
349		215	R(0.05)	-1.70	
432	D5185	254.51		-0.09	
496		237.4		-0.79	
614	D5185	249.2		-0.31	
633	D6595	286.8		1.22	
780	D5185	245		-0.48	
823	D5185	234		-0.93	
862	D5185	256.9		0.00	
912		-----		-----	
962	D5185	249		-0.32	
963	D5185	250.47		-0.26	
994	D5185	263		0.25	
1011		258		0.05	
1017		-----		-----	
1026	D5185	271		0.58	
1059	In house	269		0.50	
1146	In house	260		0.13	
1150		-----		-----	
1324	D5185	251		-0.24	
1326	D5185	266		0.37	
1327	D5185	262		0.21	
1398	D4951	253.73		-0.12	
1409		-----		-----	
1417	D5185	246.5		-0.42	
1660		-----		-----	
1740	D5185	258	C	0.05	First reported 177
1748		-----		-----	
1884		-----		-----	
1957	D5185	276.117		0.79	
6016	D5185	190	R(0.01)	-2.71	
6127	D5185	261.0		0.17	
6141		-----		-----	
6284		263.2000		0.26	
6310	D7751	253		-0.15	
6317		-----		-----	

normality	OK
n	28
outliers	3
mean (n)	256.787
st.dev. (n)	11.2985
R(calc.)	31.636
st.dev.(D5185:18)	24.6092
R(D5185:18)	68.906

Application range: 10 – 1000 mg/kg

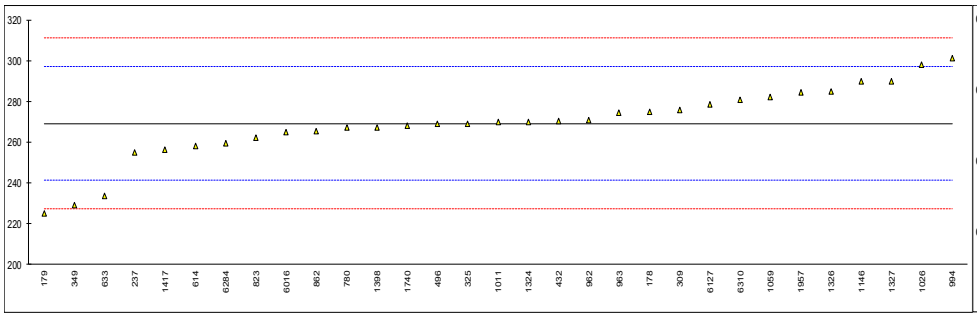


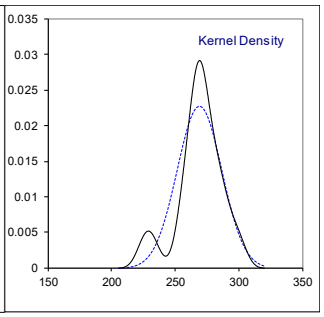
Determination of Zinc as Zn on sample #20210; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178	D5185	275		0.41	
179	D5185	225		-3.17	
237	D5185	255.0		-1.02	
256		-----		-----	
257		-----		-----	
309	D5185	275.88		0.48	
325	D5185	269		-0.01	
349		229		-2.88	
432	D5185	270.48		0.09	
496		268.8		-0.03	
614	D5185	258.2		-0.79	
633	D6595	233.5		-2.56	
780	D5185	267		-0.16	
823	D5185	262	C	-0.52	First reported 214
862	D5185	265.5		-0.27	
912		-----		-----	
962	D5185	271		0.13	
963	D5185	274.39		0.37	
994	D5185	301		2.28	
1011		270		0.06	
1017		-----		-----	
1026	D5185	298		2.06	
1059	In house	282		0.92	
1146	In house	290		1.49	
1150		-----		-----	
1324	D5185	270		0.06	
1326	D5185	285		1.13	
1327	D5185	290		1.49	
1398	D4951	267.37		-0.13	
1409		-----		-----	
1417	D5185	256.3		-0.92	
1660		-----		-----	
1740	D5185	268	C	-0.09	First reported 170
1748		-----		-----	
1884		-----		-----	
1957	D5185	284.349		1.08	
6016	D5185	265		-0.30	
6127	D5185	278.4		0.66	
6141		-----		-----	
6284		259.32		-0.71	
6310	D7751	281		0.84	
6317		-----		-----	

normality	suspect
n	31
outliers	0
mean (n)	269.209
st.dev. (n)	17.5195
R(calc.)	49.055
st.dev.(D5185:18)	13.9643
R(D5185:18)	39.100

Application range: 60 – 1600 mg/kg





APPENDIX 2

Analytical details: Foam determination

lab	Sample used	Diffuser type	Cylinder cleansed	Gas diffuser cleansed	Air tube cleansed	Air flow rate constant
178	---	---	---	---	---	---
179	As received	Metal (Stainless Steel)	?	?	Yes	re-adjustment NOT needed
237	---	---	---	---	---	---
256	---	---	---	---	Yes	re-adjustment NOT needed
257	---	---	---	---	---	---
309	After agitation (option A)	Metal (Stainless Steel)	Yes	Yes	---	---
325	---	---	---	---	?	re-adjustment NOT needed
349	---	---	---	---	Yes	re-adjustment NOT needed
432	---	---	---	---	---	---
496	As received	Metal (Stainless Steel)	Yes	Yes	---	---
614	---	---	---	---	---	---
633	---	---	---	---	---	---
780	---	---	---	---	---	---
823	As received	Stone (Non-Metallic)	Yes	Yes	---	---
862	As received	Metal (Stainless Steel)	Yes	Yes	Yes	re-adjustment NOT needed
912	---	---	---	---	---	---
962	---	---	---	---	---	---
963	---	---	---	---	---	---
994	---	---	---	---	---	---
1011	---	---	---	---	---	---
1017	---	---	---	---	---	---
1026	As received	Metal (Stainless Steel)	Yes	Yes	?	re-adjustment NOT needed
1059	---	---	---	---	Yes	re-adjustment NOT needed
1146	---	---	---	---	No	re-adjustment NOT needed
1150	---	---	---	---	Yes	re-adjustment NOT needed
1324	As received	Metal (Stainless Steel)	Yes	Yes	Yes	re-adjustment was needed
1326	---	---	---	---	---	---
1327	As received	Metal (Stainless Steel)	Yes	Yes	---	---
1398	---	---	---	---	---	---
1409	After agitation (option A)	Stone (Non-Metallic)	Yes	Yes	---	---
1417	As received	Metal (Stainless Steel)	Yes	Yes	---	---
1660	---	---	---	---	---	---
1740	---	---	---	---	---	---
1748	---	---	---	---	---	---
1884	As received	Metal (Stainless Steel)	Yes	Yes	---	---
1957	As received	Metal (Stainless Steel)	Yes	Yes	---	---
6016	After agitation (option A)	Stone (Non-Metallic)	---	Yes		
6127	As received	Stone (Non-Metallic)	No	No		
6141	---	---	---	---		
6284	After agitation (option A)	Stone (Non-Metallic)	?	?		
6310	After agitation (option A)	Metal (Stainless Steel)	Yes	Yes		
6317	---	---	---	---		

? = I do not know

APPENDIX 3

Number of participants per country

1 lab in	AUSTRALIA
1 lab in	AUSTRIA
1 lab in	AZERBAIJAN
4 labs in	BELGIUM
1 lab in	BULGARIA
4 labs in	CHINA, People's Republic
1 lab in	GEORGIA
1 lab in	GERMANY
1 lab in	GREECE
1 lab in	INDIA
1 lab in	ITALY
1 lab in	JORDAN
1 lab in	KAZAKHSTAN
1 lab in	MALAYSIA
3 labs in	NETHERLANDS
1 lab in	NIGERIA
1 lab in	OMAN
1 lab in	PHILIPPINES
1 lab in	PORTUGAL
2 labs in	RUSSIAN FEDERATION
3 labs in	SAUDI ARABIA
1 lab in	SLOVENIA
1 lab in	SOUTH KOREA
1 lab in	SPAIN
1 lab in	TAIWAN
3 labs in	TANZANIA
1 lab in	UNITED KINGDOM
2 labs in	UNITED STATES OF AMERICA

APPENDIX 4

Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= a 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
SDS	= Safety Data Sheet

Literature

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