Results of Proficiency Test
Hydraulic Oil (fresh)
November 2021

Organized by: Institute for Interlaboratory Studies
Spijkenisse, The Netherlands

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CONTENTS

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 2021/2022 it was decided to continue the round robin for the analysis of Hydraulic Oil (fresh).

In this interlaboratory study 40 laboratories in 31 different 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 an ISO/IEC17025 accredited laboratory.
It was decided to send one sample Hydraulic Oil in a 1-liter amber glass bottle labelled #21210.
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 140 liters of fresh Hydraulic Oil was obtained from a local supplier. After homogenization 78 amber glass bottles of 1L were filled and labelled #21210.
The homogeneity of the subsamples was checked by determination of Density at 15°C in accordance with ASTM 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 #21210-1	0.86951	47.28
Sample #21210-2	0.86949	47.23
Sample #21210-3	0.86949	47.28
Sample #21210-4	0.86949	47.26
Sample #21210-5	0.86949	47.26
Sample #21210-6	0.86949	47.21
Sample #21210-7	0.86949	47.28
Sample #21210-8	0.86949	47.25

Table 1: homogeneity test results of subsamples #21210

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	0.072
reference test method	ISO12185:96	D445:21e1
0.3 x R (reference test method)	0.00015	0.173

Table 2: evaluation of the repeatabilities of subsamples #21210

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 sample of Hydraulic Oil (fresh) labelled #21210 was sent on October 6, 2021. 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), Sulfur, Water, Water Separability at 54°C (distilled water) and Calcium as Ca, Phosphorus as P and Zinc as Zn. Some extra information was asked about the determination of Total Acid Number.

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.

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 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 (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 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. Four participants reported test results after the extended reporting date and four other participants did not report any test results. Not all laboratories were able to report all tests requested.
In total 36 participants reported 462 numerical test results. Observed were 21 outlying test results, which is 4.5%. 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. The abbreviations, used in these tables, are explained in appendix 3.

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

Total Acid Number: 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 Inflection Point at titration volume 60 mL, with Buffer End Point at titration volume 60 mL and Buffer End Point at titration volume 125 mL from ASTM D664-A:18e2. The calculated reproducibility is not in agreement with the Inflection Point at titration volume 125 mL requirement.

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

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

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

Foaming Characteristics (Tendency and Stability): This determination was very problematic. In total one statistical outlier was observed over three foaming parameters. The calculated reproducibilities after rejection of the statistical outlier in the Foaming Tendency determination for sequence I, II and III are not at all in agreement with the requirements of ASTM D892:18. The variation in the test results for sequence I and III are very large. Therefore, it was decided not to calculate z-scores.
All reporting participants reported 0 mL for Foam Stability.

Kinematic Viscosity at 40°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D445:21e1.

Kinematic Viscosity at 100°C: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D445:21e1.

Viscosity Index: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D2270:10(2016).
Two calculation differences were found between the reported test results of the participants and the values calculated by iis. Remarkably, some laboratories used test results from ASTM D7279 to calculate Viscosity Index, while in the test method ASTM D2270:10 states in paragraph 1.3 that only D445, D7042, IP71 or ISO3104 can be used.

Kinematic Viscosity Stabinger at 40°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D7042:21a.

Kinematic Viscosity Stabinger at 100°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D7042:21a.

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. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D5950:14(2020).

Sulfur: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D4294:21.

Water: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ASTM D6304-A:20 and in agreement with ASTM D6304-B:20, but not in agreement with ASTM D6304-C:20.
A new version of ASTM D6304 was published in 2020 with major changes. In the 2016 version one precision statement was mentioned for test results based on mass with a broad application range and one based on volume. In the 2020 version all precision statements are based on mass with three different procedures (A - direct injection, B - oven accessory and C - evaporation accessory) each with a different application range. In ASTM D6304:20 the reproducibility for all three procedures A, B and C is much stricter compared to ASTM D6304:16e1. Although there is a new version of ASTM D6304 published in 2020 four participants mentioned to have used the A, B or C of the 2016 version.

Water Separability at 54°C: This determination was not problematic. Two statistical outliers were observed over three parameters. All calculated reproducibilities after rejection of the outliers are in agreement with the requirements of ASTM D1401:21.

Calcium as Ca: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full agreement with the estimated reproducibility calculated with the Horwitz equation, but not at all with the strict requirements of ASTM D5185:18.

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

Zinc as Zn: 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 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 literature reference test methods (in casu ASTM and ISO test methods) are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Total Acid Number	mg KOH/g	25	0.25	0.06	0.13
Copper Corrosion 3hrs at 50°C		22	1 (1A)	n.a.	n.a.
Density at 15°C	kg/L	30	0.8695	0.0003	0.0005
Flash Point PMcc	°C	27	208.9	7.3	14.8
Foaming Tendency Seq. I	mL	15	123	247	(52)
Foaming Tendency Seq. II	mL	13	26.9	35.0	18.4
Foaming Tendency Seq. III	mL	12	90.8	176.5	(40.0)
Foam Stability Seq. I	mL	15	0	n.e.	n.e.
Foam Stability Seq. II	mL	13	0	n.e.	n.e.
Foam Stability Seq. III	mL	13	0	n.e.	n.e.
Kinematic Viscosity at 40°C	mm^2/s	29	47.321	0.625	0.577
Kinematic Viscosity at 100°C	mm^2/s	28	7.237	0.066	0.100
Viscosity Index		28	112.92	2.68	2
Viscosity Stabinger at 40°C	mm^2/s	13	47.350	0.253	0.630
Viscosity Stabinger at 100°C	mm^2/s	13	7.232	0.086	0.095
Pour Point Manual	°C	15	-36.8	9.8	9
Pour Point Automated 1°C int.	°C	8	-41.3	7.3	4.5

Parameter	unit	n	average	2.8 * sd	R(lit)
Sulfur	mg/kg	17	1790	356	240
Water	mg/kg	20	47.6	32.3	35.7
Water Separability at 54°C, distilled water					
Time ≤ 3 mL emulsion	minutes	14	18.2	6.9	20
Time 37 mL water	minutes	16	18.2	6.2	20
Complete Break (40-40-0)	minutes	10	20.5	4.2	20
Calcium as Ca	mg/kg	19	13.1	3.9	4.0
Phosphorus as P	mg/kg	23	187	41	59
Zinc as Zn	mg/kg	25	97.9	19.8	12.9

Table 3: reproducibilities of tests on sample #21210

Results between brackets should be used with due care.

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 2021 WITH PREVIOUS PTS

	November 2021	November 2020	November 2019	November 2018	November 2017
Number of reporting laboratories	36	41	35	35	45
Number of test results	462	533	504	465	610
Number of statistical outliers	21	23	23	18	28
Percentage of statistical outliers	4.5%	4.3%	4.6%	3.9%	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 against the requirements of the reference test methods. The conclusions are given in the following table.

	November 2021	November 2020	November 2019	November 2018	November 2017
Total Acid Number	+	+	+/-	-	+
Density at 15°C	+	-	+	+	--
Flash Point PMcc	++	+/-	++	-	+/-
Foaming Tendency Seq. I	(--)	--	+	(--)	+/-
Foaming Tendency Seq. II	-	+/-	-	-	-
Foaming Tendency Seq. III	(--)	(--)	(--)	(--)	n.e.
Kinematic Viscosity at 40°C	+/-	++	+/-	++	+
Kinematic Viscosity at 100°C	+	+/-	+	+/-	+
Viscosity Index	-	+/-	-	+/-	+/-
Kin. Viscosity Stabinger at 40°C	++	++	+	++	-

	November 2021	November 2020	November 2019	November 2018	November 2017
Kin. Viscosity Stabinger at 100°C	+/-	+	-	-	-
Pour Point Manual	+/-	+/-	+	-	+/-
Pour Point Automated 1°C int.	-	-	-	+	-
Sulfur	-	+/-	-	+	+/-
Water	+/-	++	++	++	++
Water Separability	++	--	++	+	+
Calcium as Ca	+/-	+	+/-	-	+
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

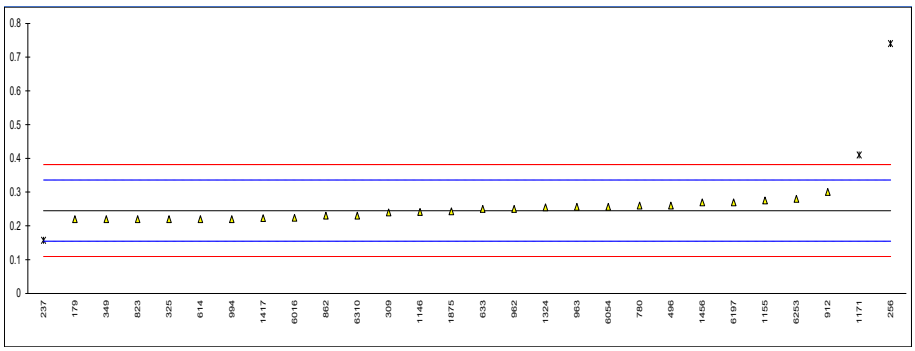
APPENDIX 1

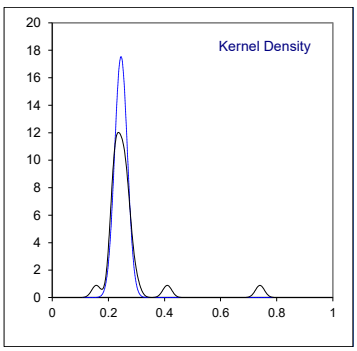
Determination of Total Acid Number on sample #21210; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks	End point	Volume
178		-----		-----		---	---
179	D664-A	0.22		-0.56		Inflection Point	60 mL
237	D664-A	0.157	R(0.05)	-1.95		Inflection Point	125 mL
256	D7889	0.74	C,R(0.01)	10.90	fr. 0.38	---	---
257		-----		-----		---	---
309	D664-A	0.24		-0.12		Buffer End Point pH 10	60 mL
325	D664-A	0.22		-0.56		Buffer End Point pH 10	125 mL
349	D664-A	0.22		-0.56		Inflection Point	125 mL
432		-----		-----		---	---
496	D664-A	0.26		0.32		Buffer End Point pH 10	60 mL
614	D664-A	0.22		-0.56		---	60 mL
633	D664-A	0.25		0.10		Inflection Point	125 mL
780	D664-A	0.26		0.32		Inflection Point	60 mL
823	D664-A	0.22		-0.56		Inflection Point	125 mL
862	D664-A	0.23		-0.34		Inflection Point	60 mL
912	D664-A	0.3		1.20		---	---
962	D664-A	0.25		0.10		---	---
963	D974	0.256		0.24		---	---
994	D664-A	0.22		-0.56		Inflection Point	125 mL
1011		-----		-----		---	---
1146	D664-A	0.241		-0.10		Buffer End Point pH 10	125 mL
1155	D664-A	0.2749		0.65		Inflection Point	125 mL
1171	ISO6618	0.41	R(0.01)	3.63		*)	100 mL
1213		-----		-----		---	---
1324	D664-A	0.255		0.21		Inflection Point	125 mL
1409		-----		-----		---	---
1417	D664-A	0.223		-0.49		Buffer End Point pH 10	60 mL
1448		-----		-----		---	---
1456	D974	0.27		0.54		---	---
1660		-----		-----		---	---
1748		-----		-----		---	---
1875	ISO6618	0.243		-0.05		---	---
6009		-----		-----		---	---
6016	D664-A	0.224		-0.47		Buffer End Point pH 11	60 mL
6034		-----		-----		---	---
6054	D974	0.256		0.24		---	---
6197	D664-A	0.27		0.54		Inflection Point	60 mL
6253	ISO6618	0.28		0.76		---	---
6310	D664-A	0.23		-0.34		---	---
6425		-----		-----		---	---

normality	OK
n	25
outliers	3
mean (n)	0.2453
st.dev. (n)	0.02273
R(calc.)	0.0638
st.dev.(D664-A:18e2, IP 60mL)	0.04538
R(D664-A:18e2, IP 60mL)	0.1271
Compare	
R(D664-A:18e2, IP 125mL)	0.0505
R(D664-A:18e2, BEP 60mL)	0.1407
R(D664-A:18e2, BEP 125mL)	0.0721

*) change of color mix of titration solvent and indicator





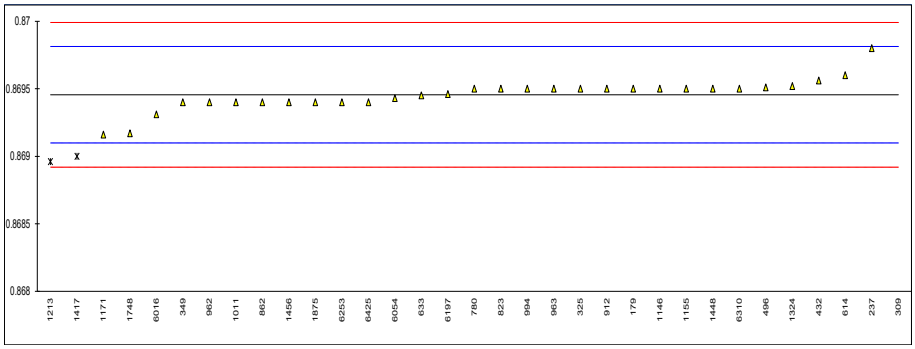
Determination of Copper Corrosion 3hrs at 50°C on sample #21210;

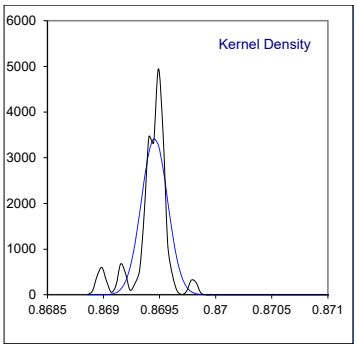
lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D130	1A		-----	
237	D130	1		-----	
256		-----		-----	
257		-----		-----	
309	D130	CUCOR1A		-----	
325	D130	1A		-----	
349		-----		-----	
432		-----		-----	
496		-----		-----	
614	D130	1a		-----	
633	D130	1a		-----	
780	D130	1a		-----	
823	D130	1a		-----	
862	D130	1a		-----	
912	D130	1a		-----	
962	D130	1A		-----	
963		-----		-----	
994	D130	1a		-----	
1011	D130	1a		-----	
1146		-----		-----	
1155	ISO2160	1a		-----	
1171	ISO2160	1A		-----	
1213	D130	1a		-----	
1324	D130	1a		-----	
1409		-----		-----	
1417	IP154	1A		-----	
1448		-----		-----	
1456	D130	1a		-----	
1660		-----		-----	
1748	D130	1a		-----	
1875		-----		-----	
6009		-----		-----	
6016		-----		-----	
6034		-----		-----	
6054		-----		-----	
6197	D130	1A		-----	
6253	ISO2160	1a		-----	
6310		-----		-----	
6425		-----		-----	
	n	22			
	mean (n)	1 (1A)			

Determination of Density at 15°C on sample #21210; results in kg/L

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D4052	0.8695		0.25	
237	D4052	0.8698		1.93	
256		-----		-----	
257		-----		-----	
309	D4052	0.87946	R(0.01)	56.02	
325	D4052	0.8695		0.25	
349	D4052	0.8694		-0.31	
432	D4052	0.86956		0.58	
496	ISO12185	0.86951		0.30	
614	D4052	0.8696		0.81	
633	D4052	0.86945		-0.03	
780	ISO12185	0.8695		0.25	
823	ISO12185	0.8695		0.25	
862	D4052	0.8694	C	-0.31	reported 869.4 kg/L
912	ISO12185	0.8695		0.25	
962	D4052	0.8694		-0.31	
963	D4052	0.8695		0.25	
994	ISO12185	0.8695		0.25	
1011	D4052	0.8694		-0.31	
1146	D4052	0.8695		0.25	
1155	ISO3675	0.8695		0.25	
1171	D4052	0.86916		-1.66	
1213	D4052	0.86896	R(0.05)	-2.78	
1324	D4052	0.86952		0.36	
1409		-----		-----	
1417	IP365	0.8690	C,R(0.05)	-2.55	first reported 870.2 kg/m³
1448	D4052	0.8695		0.25	
1456	D4052	0.8694		-0.31	
1660		-----		-----	
1748	D4052	0.86917		-1.60	
1875	DIN51757	0.8694		-0.31	
6009		-----		-----	
6016	D4052	0.86931	C	-0.82	first reported 869.3100 kg/L
6034		-----		-----	
6054	D4052	0.86943		-0.14	
6197	D4052	0.86946		0.02	
6253	ISO3675	0.8694	C	-0.31	reported 869.4 kg/L
6310	D4052	0.8695		0.25	
6425	D4052	0.8694		-0.31	

normality	not OK
n	30
outliers	3
mean (n)	0.86945
st.dev. (n)	0.000117
R(calc.)	0.00033
st.dev.(ISO12185:96)	0.000179
R(ISO12185:96)	0.0005

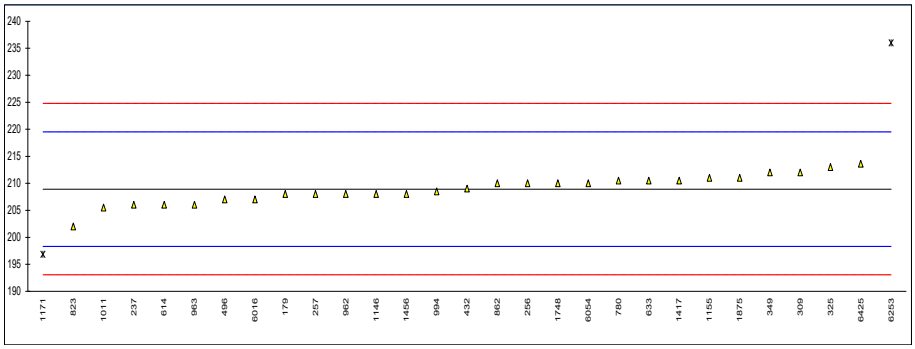


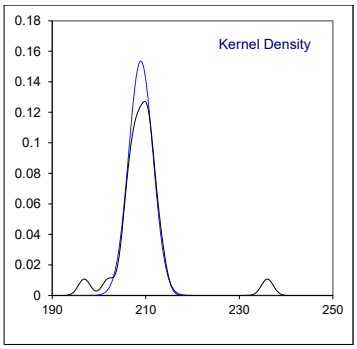


Determination of Flash Point PMcc on sample #21210; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D93-A	208.0		-0.18	
237	D93-A	206.0		-0.55	
256	D3828	210.0		0.20	
257	D93-A	208		-0.18	
309	D93-A	212.0		0.58	
325	D93-A	213		0.77	
349	D93-A	212		0.58	
432	D93-A	209.0		0.01	
496	D93-A	207.0		-0.36	
614	D93-A	206		-0.55	
633	D93-A	210.5		0.30	
780	D93-A	210.5		0.30	
823	ISO2719-A	202.0		-1.31	
862	D93-A	210		0.20	
912		-----		-----	
962	D93-A	208.0		-0.18	
963	D93-A	206.0		-0.55	
994	D93-A	208.5		-0.08	
1011	D93-A	205.5		-0.65	
1146	D93-A	208.0		-0.18	
1155	ISO2719-A	211		0.39	
1171	ISO2719-A	196.85	C,R(0.01)	-2.28	first reported 199.42
1213		-----		-----	
1324		-----		-----	
1409		-----		-----	
1417	D93-A	210.5		0.30	
1448		-----		-----	
1456	D93-A	208.0		-0.18	
1660		-----		-----	
1748	D93-A	210		0.20	
1875	ISO2719-A	211		0.39	
6009		-----		-----	
6016	D6450	207		-0.36	
6034		-----		-----	
6054	D93-A	210.0		0.20	
6197		-----		-----	
6253	ISO2592	236	R(0.01)	5.11	
6310		-----		-----	
6425	ISO2719-A	213.6		0.88	

normality	OK
n	27
outliers	2
mean (n)	208.93
st.dev. (n)	2.596
R(calc.)	7.27
st.dev.(D93-A:20)	5.298
R(D93-A:20)	14.83

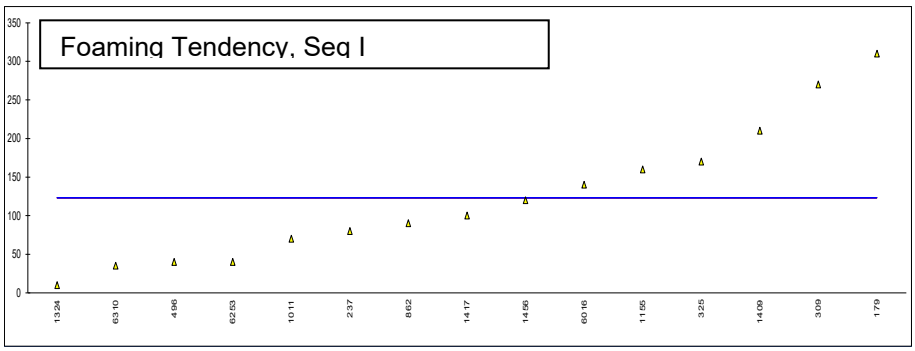


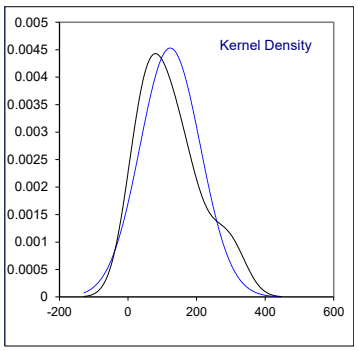


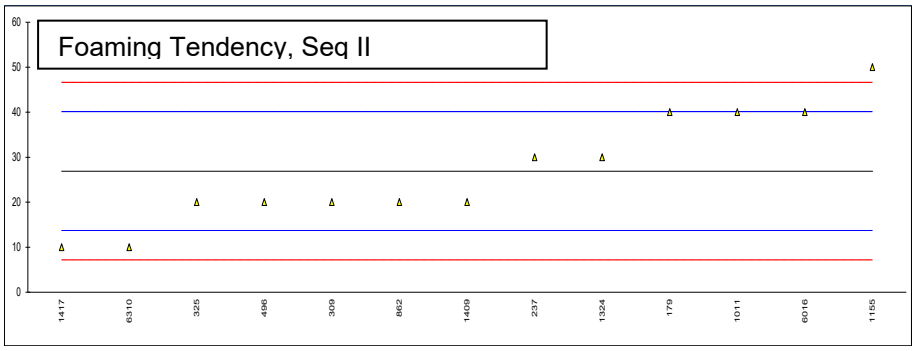
Determination of Foaming Characteristics, Foaming Tendency (5 minutes blowing period) on sample #21210; results in mL

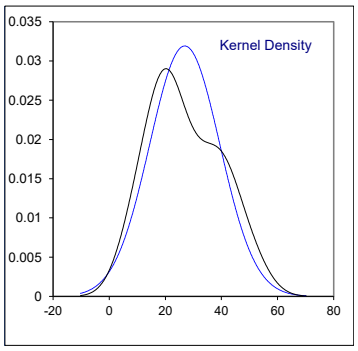
lab	method	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179	D892	310		-----	40		1.99	390	G(0.05)	-----
237	D892	80		-----	30		0.47	20		-----
256		-----		-----	-----		-----	-----		-----
257		-----		-----	-----		-----	-----		-----
309	D892	270		-----	20		-1.05	190		-----
325	D892	170		-----	20		-1.05	110		-----
349		-----		-----	-----		-----	-----		-----
432		-----		-----	-----		-----	-----		-----
496	D892	40		-----	20		-1.05	70		-----
614		-----		-----	-----		-----	-----		-----
633		-----		-----	-----		-----	-----		-----
780		-----		-----	-----		-----	-----		-----
823		-----		-----	-----		-----	-----		-----
862	D892	90		-----	20		-1.05	90		-----
912		-----		-----	-----		-----	-----		-----
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
994		-----		-----	-----		-----	-----		-----
1011	D892	70		-----	40		1.99	20		-----
1146		-----		-----	-----		-----	-----		-----
1155	D892	160		-----	50		3.50	170		-----
1171		-----		-----	-----		-----	-----		-----
1213		-----		-----	-----		-----	-----		-----
1324	D892	10		-----	30		0.47	10		-----
1409	ISO6247	210		-----	20		-1.05	150		-----
1417	D892	100		-----	10		-2.57	120		-----
1448		-----		-----	-----		-----	-----		-----
1456	D892	120		-----	-----	W	-----	-----	W	-----
1660		-----		-----	-----		-----	-----		-----
1748		-----		-----	-----		-----	-----		-----
1875		-----		-----	-----		-----	-----		-----
6009		-----		-----	-----		-----	-----		-----
6016		140		-----	40		1.99	120		-----
6034		-----		-----	-----		-----	-----		-----
6054		-----		-----	-----		-----	-----		-----
6197		-----		-----	-----		-----	-----		-----
6253	ISO6247	40		-----	-----		-----	-----		-----
6310	D892	35		-----	10		-2.57	20		-----
6425		-----		-----	-----		-----	-----		-----
	normality	OK			OK			OK		
	n	15			13			12		
	outliers	0			0			1		
	mean (n)	123			26.92			90.83		
	st.dev. (n)	88.071			12.506			63.024		
	R(calc.)	246.60			35.02			176.47		
	st.dev.(D892:18)	(18.436)			6.586			(14.274)		
	R(D892:18)	(51.62)			18.44			(39.97)		

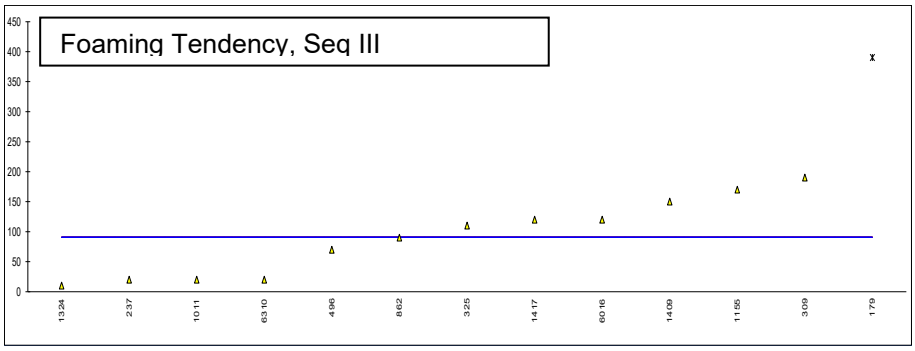
Lab 1456 test results withdrawn, first reported 120 and 120

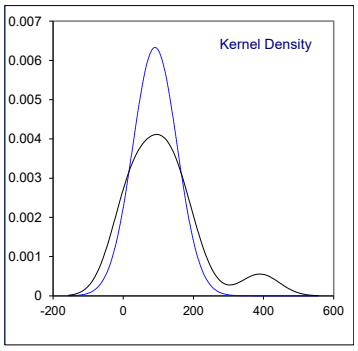

Foaming Tendency, Seq I
350
300
250
200
150
100
50
0
1324
6310
496
6253
1011
237
862
1417
1456
6016
1155
325
1409
309
179


Kernel Density
0.005
0.0045
0.004
0.0035
0.003
0.0025
0.002
0.0015
0.001
0.0005
0
-200
0
200
400
600


Foaming Tendency, Seq II
60
50
40
30
20
10
0
1417
6310
325
496
309
862
1409
237
1324
179
1011
6016
1155


Kernel Density
0.035
0.03
0.025
0.02
0.015
0.01
0.005
0
-20
0
20
40
60
80


Foaming Tendency, Seq III
450
400
350
300
250
200
150
100
50
0
1324
237
1011
6310
496
862
325
1417
6016
1409
1155
309
179


Kernel Density
0.007
0.006
0.005
0.004
0.003
0.002
0.001
0
-200
0
200
400
600

Determination of Foaming Characteristics, Foam Stability (10 minutes settling period) on sample #21210; results in mL

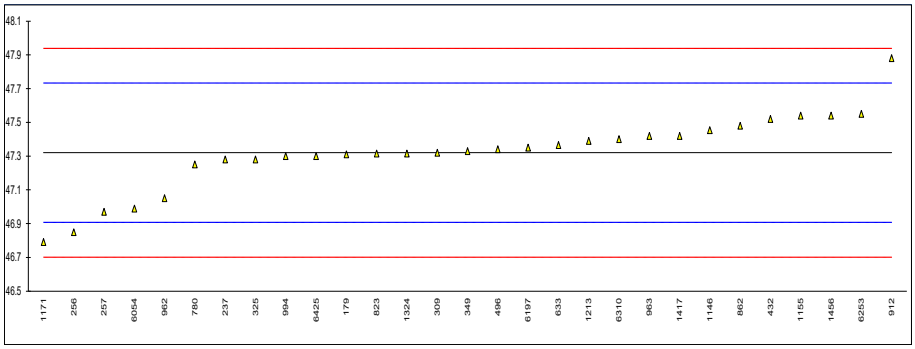
lab	method	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179	D892	0		-----	0		-----	0		-----
237	D892	0		-----	0		-----	0		-----
256		-----		-----	-----		-----	-----		-----
257		-----		-----	-----		-----	-----		-----
309	D892	0		-----	0		-----	0		-----
325	D892	0		-----	0		-----	0		-----
349		-----		-----	-----		-----	-----		-----
432		-----		-----	-----		-----	-----		-----
496	D892	0		-----	0		-----	0		-----
614		-----		-----	-----		-----	-----		-----
633		-----		-----	-----		-----	-----		-----
780		-----		-----	-----		-----	-----		-----
823		-----		-----	-----		-----	-----		-----
862	D892	0		-----	0		-----	0		-----
912		-----		-----	-----		-----	-----		-----
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
994		-----		-----	-----		-----	-----		-----
1011	D892	0		-----	0		-----	0		-----
1146		-----		-----	-----		-----	-----		-----
1155	D892	0		-----	0		-----	0		-----
1171		-----		-----	-----		-----	-----		-----
1213		-----		-----	-----		-----	-----		-----
1324	D892	0		-----	0		-----	0		-----
1409	ISO6247	0		-----	0		-----	0		-----
1417	D892	0		-----	0		-----	0		-----
1448		-----		-----	-----		-----	-----		-----
1456	D892	0		-----	-----	W	-----	-----	W	-----
1660		-----		-----	-----		-----	-----		-----
1748		-----		-----	-----		-----	-----		-----
1875		-----		-----	-----		-----	-----		-----
6009		-----		-----	-----		-----	-----		-----
6016		0		-----	0		-----	0		-----
6034		-----		-----	-----		-----	-----		-----
6054		-----		-----	-----		-----	-----		-----
6197		-----		-----	-----		-----	-----		-----
6253	ISO6247	0		-----	-----		-----	-----		-----
6310	D892	0		-----	0		-----	0		-----
6425		-----		-----	-----		-----	-----		-----
	n	15			13			13		
	mean (n)	0			0			0		

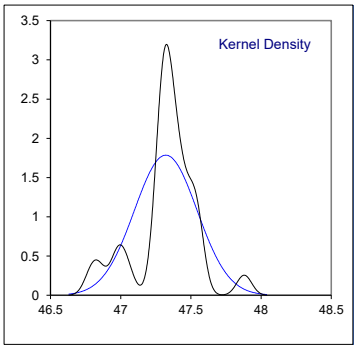
Lab 1456 test results withdrawn, first reported 0 and 0

Determination of Kinematic Viscosity at 40°C on sample #21210; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D445	47.31		-0.05	
237	D445	47.28		-0.20	
256	D7229	46.849		-2.29	
257	D7279 corr to D445	46.97		-1.70	
309	D445	47.32		0.00	
325	D445	47.28		-0.20	
349	D445	47.33		0.05	
432	D445	47.52		0.97	
496	D445	47.340		0.09	
614		-----		-----	
633	D445	47.365		0.22	
780	D445	47.25		-0.34	
823	D445	47.315		-0.03	
862	D445	47.48		0.77	
912	D445	47.88		2.71	
962	D445	47.05		-1.31	
963	D445	47.42		0.48	
994	D445	47.30		-0.10	
1011		-----		-----	
1146	D445	47.453		0.64	
1155	ISO3104	47.54		1.06	
1171	ISO3104	46.791		-2.57	
1213	D445	47.39		0.34	
1324	D445	47.315		-0.03	
1409		-----		-----	
1417	D445	47.42		0.48	
1448		-----		-----	
1456	D445	47.54	C	1.06	first reported 48.52
1660		-----		-----	
1748		-----		-----	
1875		-----		-----	
6009		-----		-----	
6016		-----		-----	
6034		-----		-----	
6054	D445	46.989		-1.61	
6197	D445	47.35		0.14	
6253	ISO3104	47.55		1.11	
6310	D7279 corr to D445	47.4		0.39	
6425	D7042	47.30		-0.10	

normality	suspect
n	29
outliers	0
mean (n)	47.3205
st.dev. (n)	0.22337
R(calc.)	0.6254
st.dev.(D445:21e1)	0.20618
R(D445:21e1)	0.5773

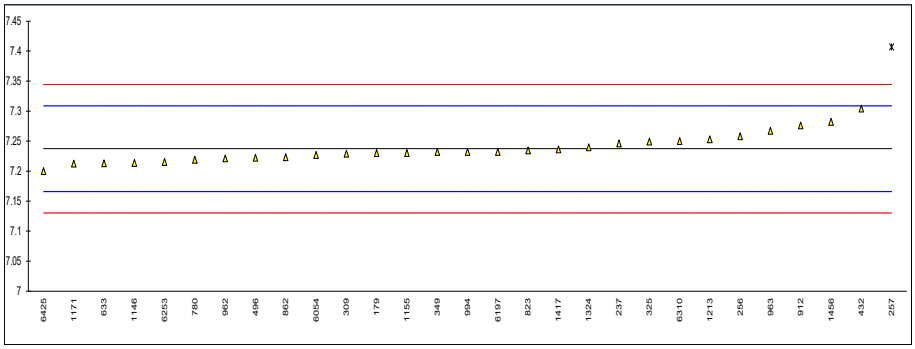


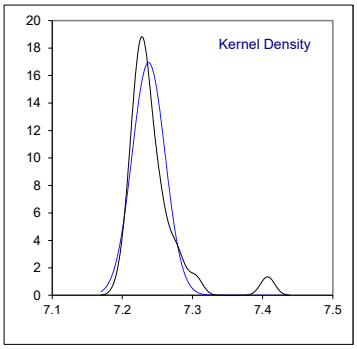


Determination of Kinematic Viscosity at 100°C on sample #21210; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D445	7.23		-0.21	
237	D445	7.246		0.24	
256	D7229	7.258	C	0.58	first reported 7.358
257	D7279 corr to D445	7.407	R(0.01)	4.76	
309	D445	7.229		-0.23	
325	D445	7.249		0.33	
349	D445	7.232		-0.15	
432	D445	7.304		1.87	
496	D445	7.2221		-0.43	
614		-----		-----	
633	D445	7.213	C	-0.68	first reported 8.727
780	D445	7.219		-0.51	
823	ISO3104	7.2344		-0.08	
862	D445	7.223		-0.40	
912	D445	7.276		1.08	
962	D445	7.221		-0.46	
963	D445	7.267		0.83	
994	D445	7.232		-0.15	
1011		-----		-----	
1146	D445	7.2135		-0.67	
1155	ISO3104	7.230		-0.21	
1171	ISO3104	7.2125		-0.70	
1213	D445	7.253		0.44	
1324	D445	7.2395		0.06	
1409		-----		-----	
1417	D445	7.236		-0.04	
1448		-----		-----	
1456	D445	7.282	C	1.25	first reported 7.410
1660		-----		-----	
1748		-----		-----	
1875		-----		-----	
6009		-----		-----	
6016		-----		-----	
6034		-----		-----	
6054	D445	7.2268		-0.30	
6197	D445	7.232		-0.15	
6253	ISO3104	7.215		-0.63	
6310	D7279 corr to D445	7.25		0.35	
6425	D7042	7.200		-1.05	

normality	not OK
n	28
outliers	1
mean (n)	7.2373
st.dev. (n)	0.02354
R(calc.)	0.0659
st.dev.(D445:21e1)	0.03567
R(D445:21e1)	0.0999

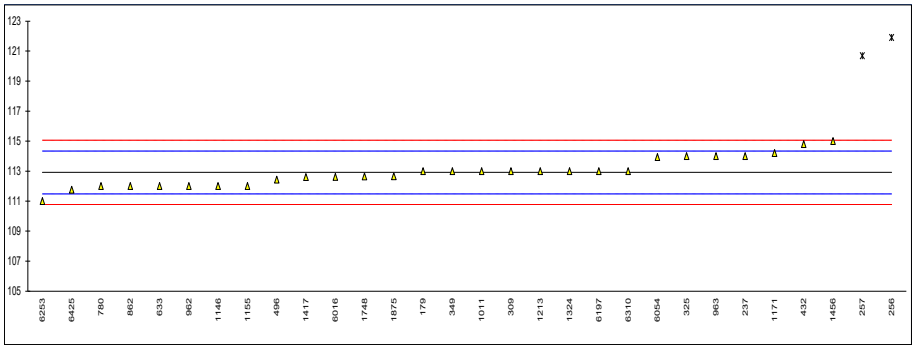


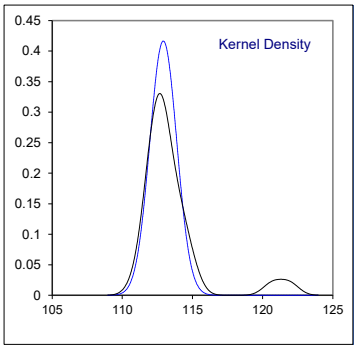


Determination of Viscosity Index on sample #21210;

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D2270	113		0.12	
237	D2270	114		1.52	
256	D2270	121.906	C,R(0.01),E	12.59	first reported 119.351, calculation difference, iis calc. 115.6
257	D2270	120.689	R(0.01)	10.88	
309	D2270	113		0.12	
325	D2270	114		1.52	
349	D2270	113		0.12	
432	D2270	114.8		2.64	
496	D2270	112.43		-0.68	
614		-----		-----	
633	D2270	112	C	-1.28	first reported 165.5
780	D2270	112		-1.28	
823		-----		-----	
862	D2270	112		-1.28	
912		-----		-----	
962	D2270	112		-1.28	
963	D2270	114		1.52	
994		-----		-----	
1011	D2270	113		0.12	
1146	D2270	112		-1.28	
1155	ISO2909	112		-1.28	
1171	D2270	114.2		1.80	
1213	D2270	113		0.12	
1324	D2270	113		0.12	
1409		-----		-----	
1417	D2270	112.6		-0.44	
1448		-----		-----	
1456	D2270	115	E	2.92	calculation difference, iis calculate 113.9
1660		-----		-----	
1748	D2270	112.65		-0.37	
1875	ISO2909	112.657		-0.36	
6009		-----		-----	
6016	D2270	112.610		-0.43	
6034		-----		-----	
6054	D2270	113.93		1.42	
6197	D2270	113		0.12	
6253	ISO2909	111		-2.68	
6310	D2270	113		0.12	
6425	D2270	111.76		-1.62	

normality	OK
n	28
outliers	2
mean (n)	112.92
st.dev. (n)	0.958
R(calc.)	2.68
st.dev.(D2270:10)	0.714
R(D2270:10)	2

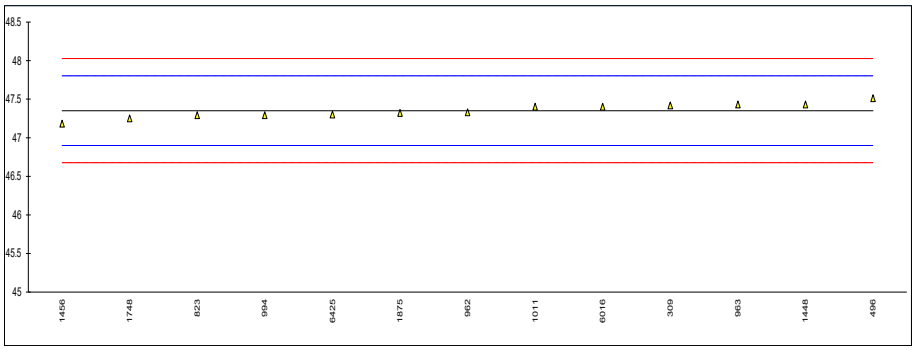


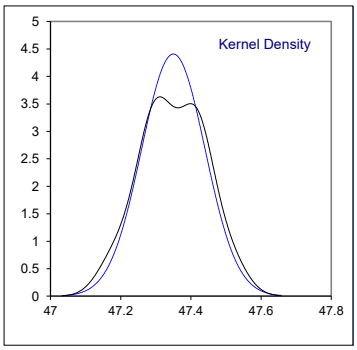


Determination of Viscosity Stabinger at 40°C on sample #21210; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
256		-----		-----	
257		-----		-----	
309	D7042	47.417		0.30	
325		-----		-----	
349		-----		-----	
432		-----		-----	
496	D7042	47.511		0.72	
614		-----		-----	
633		-----		-----	
780		-----		-----	
823	D7042	47.29		-0.27	
862		-----		-----	
912		-----		-----	
962	D7042	47.33		-0.09	
963	D7042	47.43		0.36	
994	D7042	47.29		-0.27	
1011	D7042	47.40		0.22	
1146		-----		-----	
1155		-----		-----	
1171		-----		-----	
1213		-----		-----	
1324		-----		-----	
1409		-----		-----	
1417		-----		-----	
1448	D7042	47.431		0.36	
1456	D7042	47.18		-0.76	
1660		-----		-----	
1748	D7042	47.25		-0.44	
1875	D7042	47.32		-0.13	
6009		-----		-----	
6016	D7042	47.400		0.22	
6034		-----		-----	
6054		-----		-----	
6197		-----		-----	
6253		-----		-----	
6310		-----		-----	
6425	D7042	47.30		-0.22	

normality	OK
n	13
outliers	0
mean (n)	47.3499
st.dev. (n)	0.09049
R(calc.)	0.2534
st.dev.(D7042:21a)	0.22497
R(D7042:21a)	0.6299

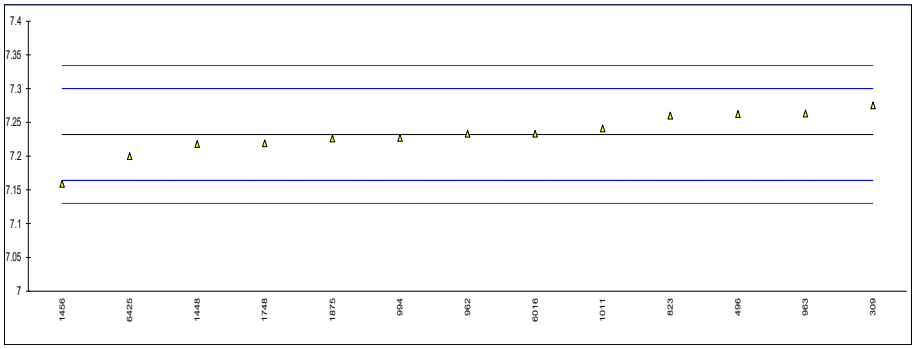


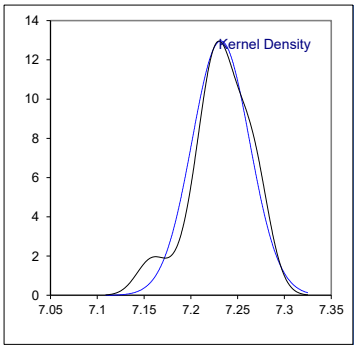


Determination of Viscosity Stabinger at 100°C on sample #21210; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
256		-----		-----	
257		-----		-----	
309	D7042	7.2750		1.26	
325		-----		-----	
349		-----		-----	
432		-----		-----	
496	D7042	7.2623		0.89	
614		-----		-----	
633		-----		-----	
780		-----		-----	
823	D7042	7.26		0.82	
862		-----		-----	
912		-----		-----	
962	D7042	7.233		0.03	
963	D7042	7.263		0.91	
994	D7042	7.227		-0.15	
1011	D7042	7.241		0.26	
1146		-----		-----	
1155		-----		-----	
1171		-----		-----	
1213		-----		-----	
1324		-----		-----	
1409		-----		-----	
1417		-----		-----	
1448	D7042	7.2181		-0.41	
1456	D7042	7.159		-2.15	
1660		-----		-----	
1748	D7042	7.219		-0.38	
1875	D7042	7.226		-0.18	
6009		-----		-----	
6016	D7042	7.233		0.03	
6034		-----		-----	
6054		-----		-----	
6197		-----		-----	
6253		-----		-----	
6310		-----		-----	
6425	D7042	7.200		-0.94	

normality	suspect
n	13
outliers	0
mean (n)	7.2320
st.dev. (n)	0.03078
R(calc.)	0.0862
st.dev.(D7042:21a)	0.03400
R(D7042:21a)	0.0952

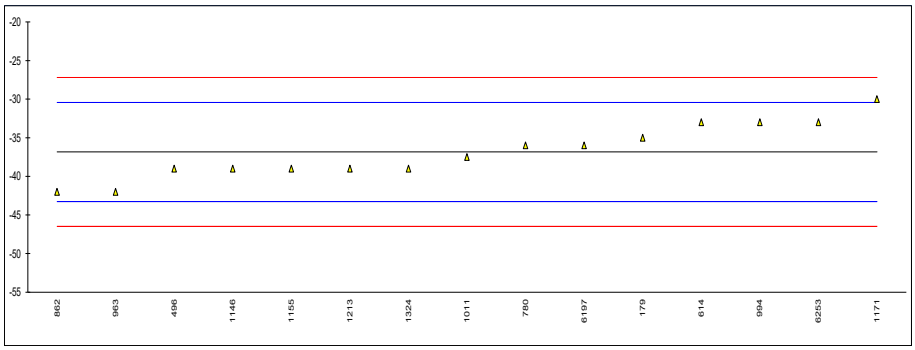


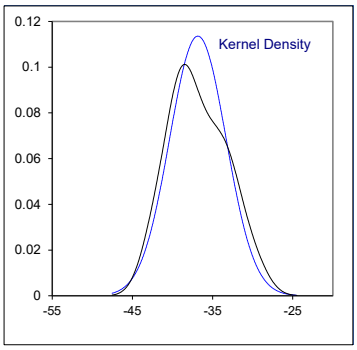


Determination of Pour Point Manual on sample #21210; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D97	-35		0.57	
237	D97	<-21		-----	
256		-----		-----	
257		-----		-----	
309		-----		-----	
325		-----		-----	
349		-----		-----	
432		-----		-----	
496	ISO3016	-39		-0.67	
614	D97	-33		1.19	
633		-----		-----	
780	D97	-36		0.26	
823		-----		-----	
862	D97	-42		-1.61	
912		-----		-----	
962		-----		-----	
963	D97	-42		-1.61	
994	D97	-33		1.19	
1011	D97	-37.5		-0.21	
1146	D97	-39		-0.67	
1155	ISO3016	-39		-0.67	
1171	ISO3016	-30.0		2.13	
1213	D97	-39		-0.67	
1324	D97	-39		-0.67	
1409		-----		-----	
1417		-----		-----	
1448		-----		-----	
1456		-----		-----	
1660		-----		-----	
1748		-----		-----	
1875		-----		-----	
6009		-----		-----	
6016		-----		-----	
6034		-----		-----	
6054		-----		-----	
6197	D97	-36		0.26	
6253	T60-105	-33		1.19	
6310		-----		-----	
6425		-----		-----	

normality	OK
n	15
outliers	0
mean (n)	-36.83
st.dev. (n)	3.514
R(calc.)	9.84
st.dev.(D97:17b)	3.214
R(D97:17b)	9

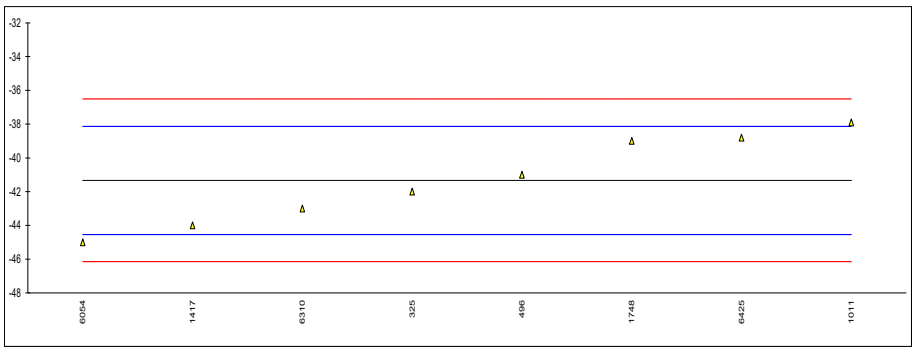


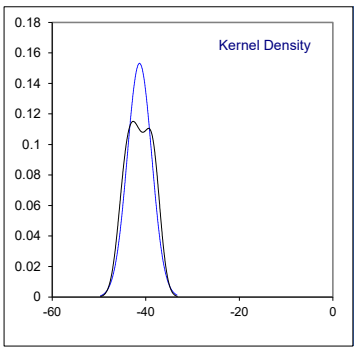


Determination of Pour Point Automated 1°C interval on sample #21210; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
256		-----		-----	
257		-----		-----	
309		-----		-----	
325	D5950	-42		-0.41	
349		-----		-----	
432		-----		-----	
496	D5950	-41		0.21	
614		-----		-----	
633		-----		-----	
780		-----		-----	
823		-----		-----	
862		-----		-----	
912		-----		-----	
962		-----		-----	
963		-----		-----	
994		-----		-----	
1011		-37.9		2.14	
1146		-----		-----	
1155		-----		-----	
1171		-----		-----	
1213		-----		-----	
1324		-----		-----	
1409		-----		-----	
1417	D5950	-44.0		-1.66	
1448		-----		-----	
1456		-----		-----	
1660		-----		-----	
1748	D7346	-39		1.45	
1875		-----		-----	
6009		-----		-----	
6016		-----		-----	
6034		-----		-----	
6054	D5950	-45.0		-2.28	
6197		-----		-----	
6253		-----		-----	
6310	D5950	-43		-1.03	
6425		-38.8		1.58	

normality	unknown
n	8
outliers	0
mean (n)	-41.34
st.dev. (n)	2.606
R(calc.)	7.30
st.dev.(D5950:14)	1.607
R(D5950:14)	4.5

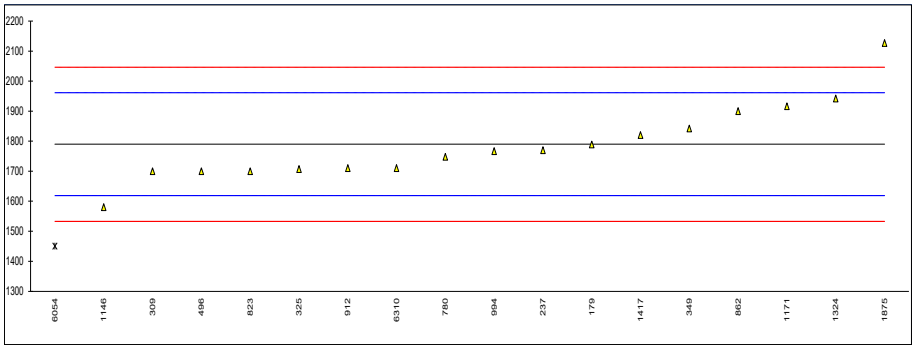


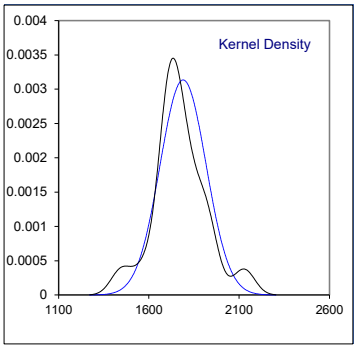


Determination of Sulfur on sample #21210; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D4294	1789		-0.01	
237	D4294	1770		-0.23	
256		-----		-----	
257		-----		-----	
309	D2622	1700		-1.05	
325	D5185	1707		-0.97	
349	D2622	1842		0.61	
432		-----		-----	
496	D4294	1700		-1.05	
614		-----		-----	
633		-----		-----	
780	D4294	1748		-0.49	
823	ISO8754	1700	C	-1.05	first reported 0.17 mg/kg
862	D2622	1900		1.29	
912	D4294	1710		-0.93	
962		-----		-----	
963		-----		-----	
994	D4294	1767		-0.27	
1011		-----		-----	
1146	D4294	1580		-2.45	
1155		-----		-----	
1171	D5453	1916.3		1.48	
1213		-----		-----	
1324	D4294	1942.0		1.78	
1409		-----		-----	
1417	In house	1820		0.35	
1448		-----		-----	
1456		-----		-----	
1660		-----		-----	
1748		-----		-----	
1875	DIN51724-1	2127		3.94	
6009		-----		-----	
6016		-----		-----	
6034		-----		-----	
6054	D4294	1450	D(0.05)	-3.97	
6197		-----		-----	
6253		-----		-----	
6310	D7751	1710		-0.93	
6425		-----		-----	

normality	not OK
n	17
outliers	1
mean (n)	1789.90
st.dev. (n)	127.258
R(calc.)	356.32
st.dev.(D4294:21)	85.607
R(D4294:21)	239.70

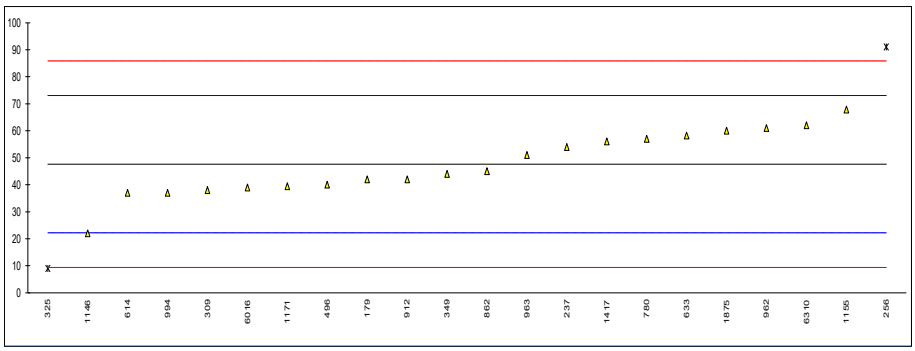


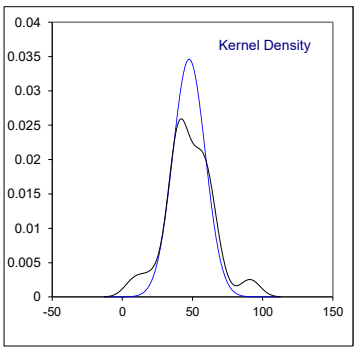


Determination of Water on sample #21210; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D6304	42		-0.44	
237	D6304-C:16e1	54		0.50	
256	D7889	91.0	D(0.05)	3.41	
257		-----		-----	
309	D6304-C:20	38		-0.76	
325	D6304-C:20	9	D(0.05)	-3.03	
349	D6304-A:20	44		-0.28	
432		-----		-----	
496	D6304-B:20	40		-0.60	
614	D6304-B:20	37		-0.83	
633	D6304-B:20	58.2		0.83	
780	D6304-B:20	57		0.74	
823	D6304-C:20	<20		-----	
862	D6304-B:20	45		-0.21	
912	D6304-C:16e1	42		-0.44	
962	D6304-C:20	61		1.05	
963	D6304-A:20	51		0.27	
994	D6304-C:20	37		-0.83	
1011		-----		-----	
1146		22		-2.01	
1155	D6304-B:20	67.8		1.58	
1171	ISO12937	39.49		-0.64	
1213		-----		-----	
1324		-----		-----	
1409		-----		-----	
1417	D6304-A:20	56		0.66	
1448		-----		-----	
1456		-----		-----	
1660		-----		-----	
1748		-----		-----	
1875	ISO12937	60		0.97	
6009		-----		-----	
6016	D6304-B:16e1	39.0		-0.68	
6034		-----		-----	
6054		-----		-----	
6197		-----		-----	
6253		-----		-----	
6310	D6304-C:16e1	62		1.13	
6425		-----		-----	

	normality	OK
	n	20
	outliers	2
	mean (n)	47.625
	st.dev. (n)	11.5273
	R(calc.)	32.276
	st.dev.(D6304-A:20)	12.7368
	R(D6304-A:20)	35.663
Compare		
	R(D6304-B:20)	113.75
	R(D6304-C:20)	24.571

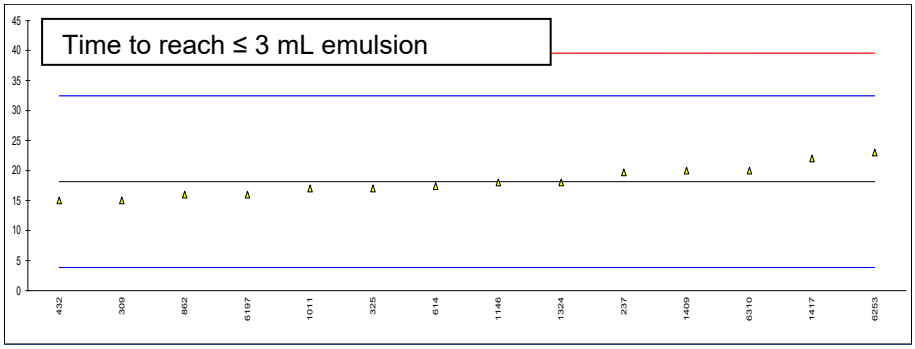


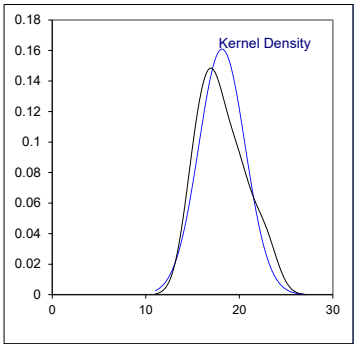


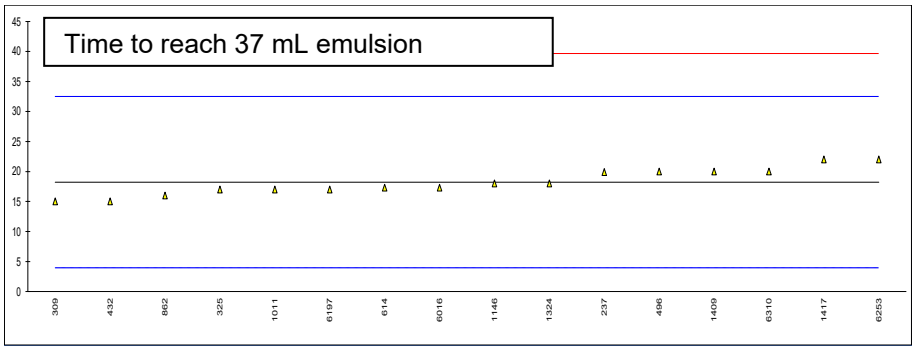
Determination of Water Separability at 54°C, distilled water on sample #21210; results in minutes

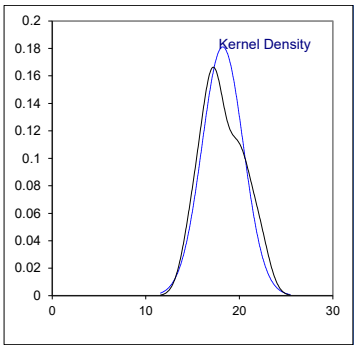
lab	method	≤3 mL emulsion	m.	z(targ)	37 mL water	m.	z(targ)	complete break (40-40-0)	m.	z(targ)	test aborted	time aborted
178		-----		-----	-----		-----	-----		-----	-----	-----
179		-----		-----	-----		-----	-----		-----	-----	25
237	D1401	19.7		0.22	19.9		0.24	23.9		0.47	No	-----
256		-----		-----	-----		-----	-----		-----	-----	-----
257		-----		-----	-----		-----	-----		-----	-----	-----
309	D1401	15		-0.44	15		-0.45	>30		-----	-----	-----
325	D1401	17		-0.16	17		-0.17	20		-0.07	Yes	17
349		-----		-----	-----		-----	-----		-----	-----	-----
432	D1401	15		-0.44	15		-0.45	>30		-----	Yes	>30
496		-----		-----	20		0.25	20		-0.07	No	-----
614	D1401	17.4		-0.10	17.3		-0.13	18.3		-0.31	No	-----
633		-----		-----	-----		-----	-----		-----	-----	-----
780		-----		-----	-----		-----	-----		-----	-----	-----
823		-----		-----	-----		-----	-----		-----	Yes	30
862	D1401	16		-0.30	16		-0.31	20		-0.07	No	-----
912		-----		-----	-----		-----	-----		-----	-----	-----
962		-----		-----	-----		-----	-----		-----	-----	-----
963		-----		-----	-----		-----	-----		-----	-----	-----
994		-----		-----	-----		-----	-----		-----	-----	-----
1011		17		-0.16	17		-0.17	20		-0.07	No	-----
1146	D1401	18		-0.02	18		-0.03	0	G1	-2.87	No	18
1155		-----		-----	-----		-----	-----		-----	-----	-----
1171		-----		-----	-----		-----	-----		-----	-----	-----
1213		-----		-----	-----		-----	-----		-----	-----	-----
1324	D1401	18		-0.02	18		-0.03	-----		-----	-----	-----
1409	ISO6614	20		0.26	20		0.25	20		-0.07	No	-----
1417	D1401	22		0.54	22		0.53	22		0.21	No	-----
1448		-----		-----	-----		-----	-----		-----	-----	-----
1456		-----		-----	-----		-----	-----		-----	-----	-----
1660		-----		-----	-----		-----	-----		-----	-----	-----
1748		-----		-----	-----		-----	-----		-----	-----	-----
1875		-----		-----	-----		-----	-----		-----	-----	-----
6009		-----		-----	-----		-----	-----		-----	-----	-----
6016		-----		-----	17.30		-0.13	-----		-----	No	-----
6034		-----		-----	-----		-----	-----		-----	-----	-----
6054		-----		-----	-----		-----	-----		-----	-----	-----
6197		16		-0.30	17		-0.17	21		0.07	No	-----
6253	ISO6614	23		0.68	22		0.53	30	G1	1.33	-----	-----
6310	D1401	20		0.26	20		0.25	20		-0.07	No	-----
6425		-----		-----	-----		-----	-----		-----	-----	-----

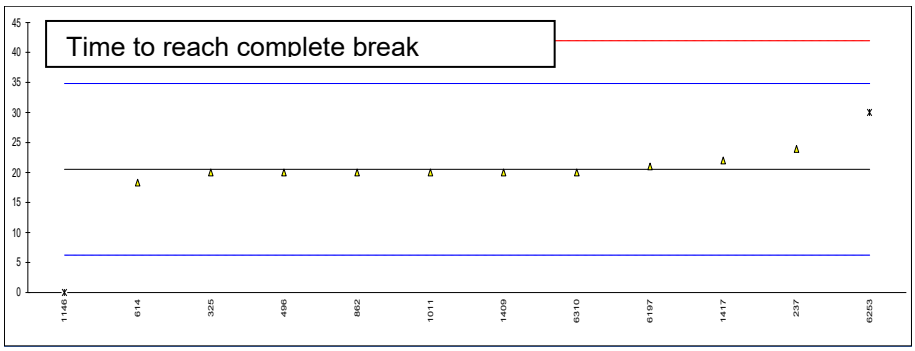
normality	OK	OK	not OK
n	14	16	10
outliers	0	0	2
mean (n)	18.15	18.22	20.52
st.dev. (n)	2.480	2.203	1.505
R(calc.)	6.94	6.17	4.22
st.dev.(D1401:21)	7.143	7.143	7.143
R(D1401:21)	20	20	20

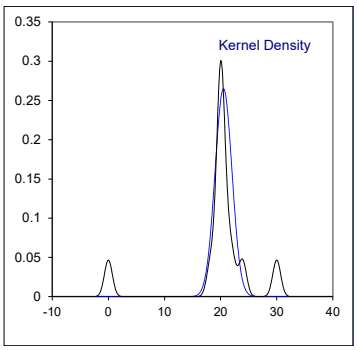

Time to reach ≤ 3 mL emulsion
45
40
35
30
25
20
15
10
5
0
432
309
862
6197
1011
325
614
1146
1324
237
1409
6310
1417
6253


Kernel Density
0.18
0.16
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
0
10
20
30


Time to reach 37 mL emulsion
45
40
35
30
25
20
15
10
5
0
309
432
862
325
1011
6197
614
6016
1146
1324
237
496
1409
6310
1417
6253


Kernel Density
0.2
0.18
0.16
0.14
0.12
0.1
0.08
0.06
0.04
0.02
0
0
10
20
30


Time to reach complete break
45
40
35
30
25
20
15
10
5
0
1146
614
325
496
862
1011
1409
6310
6197
1417
237
6253


Kernel Density
0.35
0.3
0.25
0.2
0.15
0.1
0.05
0
-10
0
10
20
30
40

Determination of Water Separability at 54°C, distilled water on sample #21210; results in mL
--- Continued ----

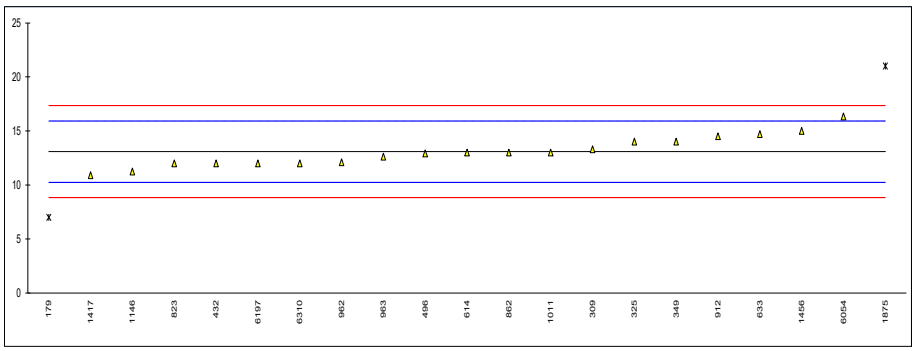
lab	method	oil	mark	z(targ)	water	mark	z(targ)	emulsion	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179	D1401	40		-----	40		-----	0		-----
237	D1401	40.0		-----	40.0		-----	0		-----
256		-----		-----	-----		-----	-----		-----
257		-----		-----	-----		-----	-----		-----
309		-----		-----	-----		-----	-----		-----
325	D1401	43		-----	37		-----	0		-----
349		-----		-----	-----		-----	-----		-----
432	D1401	43		-----	37		-----	0		-----
496	D1401	36		-----	38		-----	6		-----
614	D1401	40		-----	40		-----	0		-----
633		-----		-----	-----		-----	-----		-----
780		-----		-----	-----		-----	-----		-----
823	D1401	42	C	-----	38		-----	0	C	-----
862	D1401	40		-----	40		-----	0		-----
912		-----		-----	-----		-----	-----		-----
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
994		-----		-----	-----		-----	-----		-----
1011		40		-----	40		-----	0		-----
1146	D1401	43		-----	37		-----	0		-----
1155		-----		-----	-----		-----	-----		-----
1171		-----		-----	-----		-----	-----		-----
1213		-----		-----	-----		-----	-----		-----
1324		-----		-----	-----		-----	-----		-----
1409		-----		-----	-----		-----	-----		-----
1417	D1401	40		-----	40		-----	0		-----
1448		-----		-----	-----		-----	-----		-----
1456		-----		-----	-----		-----	-----		-----
1660		-----		-----	-----		-----	-----		-----
1748		-----		-----	-----		-----	-----		-----
1875		-----		-----	-----		-----	-----		-----
6009		-----		-----	-----		-----	-----		-----
6016		-----		-----	-----		-----	-----		-----
6034		-----		-----	-----		-----	-----		-----
6054		-----		-----	-----		-----	-----		-----
6197		-----		-----	-----		-----	-----		-----
6253		-----		-----	-----		-----	-----		-----
6310		-----		-----	-----		-----	-----		-----
6425		-----		-----	-----		-----	-----		-----

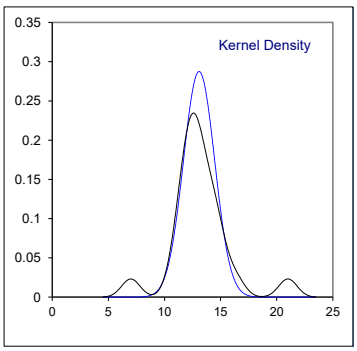
Lab 823 first reported 0, 42

Determination of Calcium as Ca on sample #21210; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D5185	7	R(0.01)	-4.28	
237		-----		-----	
256		-----		-----	
257		-----		-----	
309	D5185	13.3		0.15	
325	D5185	14		0.64	
349	D5185	14	C	0.64	first reported 0
432	D5185	12		-0.76	
496	D5185	12.92		-0.12	
614	D5185	13.0		-0.06	
633	D6595	14.7		1.14	
780	D5185	<40		-----	
823	D5185	12		-0.76	
862	D5185	13		-0.06	
912	D5185	14.5		1.00	
962	D5185	12.1		-0.69	
963	D5185	12.61		-0.33	
994	D5185	<40	C	-----	first reported 6.41
1011	D5185	13		-0.06	
1146	D5185	11.24		-1.30	
1155		-----		-----	
1171		-----		-----	
1213		-----		-----	
1324	D5185	-----		-----	
1409		-----		-----	
1417		10.9		-1.54	
1448		-----		-----	
1456	D5185	15		1.35	
1660		-----		-----	
1748		-----		-----	
1875	EN11885	21	R(0.01)	5.57	
6009		-----		-----	
6016	D6595	-----		-----	
6034		-----		-----	
6054	IP501	16.3339		2.29	
6197	D4951	12		-0.76	
6253		-----		-----	
6310	D7751	12		-0.76	
6425		-----		-----	

	normality	OK
	n	19
	outliers	2
	mean (n)	13.084
	st.dev. (n)	1.3882
	R(calc.)	3.887
	st.dev.(Horwitz)	1.4216
	R(Horwitz)	3.981
Compare		
	R(D5158:18)	0.424

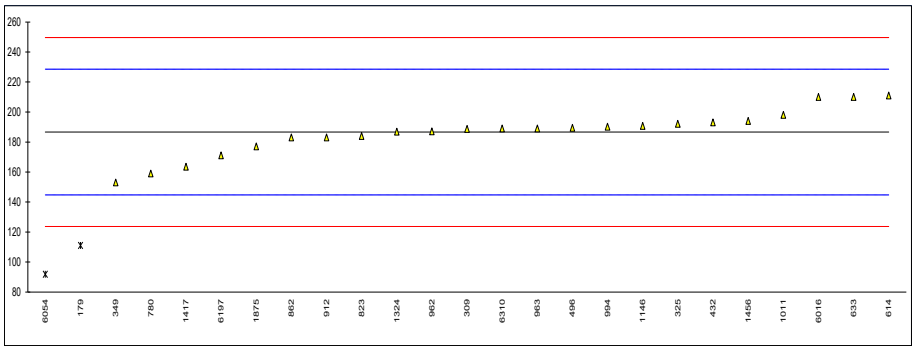


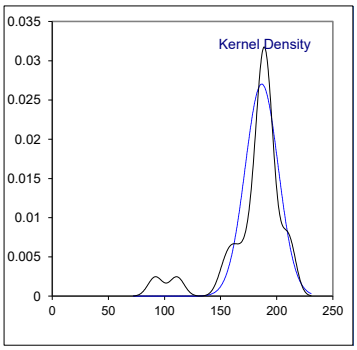


Determination of Phosphorus as P on sample #21210; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D5185	111	R(0.01)	-3.60	
237		-----		-----	
256		-----		-----	
257		-----		-----	
309	D5185	188.7		0.10	
325	D5185	192		0.26	
349	D5185	153		-1.60	
432	D5185	193		0.30	
496	D5185	189.4		0.13	
614	D5185	211.0		1.16	
633	D6595	210.05		1.12	
780	D5185	159		-1.32	
823	D5185	184	C	-0.13	first reported 94
862	D5185	183		-0.17	
912	D5185	183		-0.17	
962	D5185	187		0.02	
963	D5185	189.03		0.11	
994	D5185	190.2		0.17	
1011	D5185	198		0.54	
1146	D5185	190.79		0.20	
1155		-----		-----	
1171		-----		-----	
1213		-----		-----	
1324	D5185	186.8		0.01	
1409		-----		-----	
1417		163.5		-1.10	
1448		-----		-----	
1456	D5185	194		0.35	
1660		-----		-----	
1748		-----		-----	
1875	EN11885	177		-0.46	
6009		-----		-----	
6016	D6595	210	C	1.11	first reported 125
6034		-----		-----	
6054	IP501	91.8797	R(0.01)	-4.52	
6197	D4951	171		-0.74	
6253		-----		-----	
6310	D7751	189		0.11	
6425		-----		-----	

normality	OK
n	23
outliers	2
mean (n)	186.629
st.dev. (n)	14.7630
R(calc.)	41.336
st.dev.(D5185:18)	20.9797
R(D5185:18)	58.743

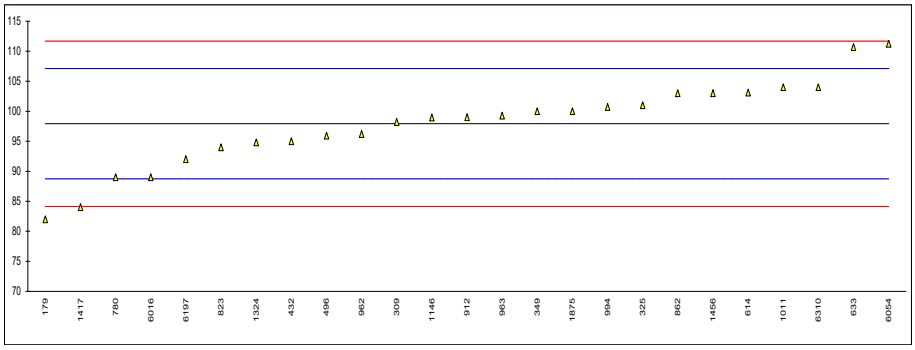


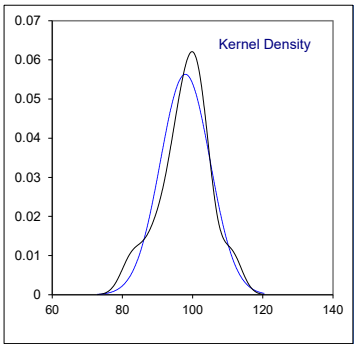


Determination of Zinc as Zn on sample #21210; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D5185	82		-3.47	
237		-----		-----	
256		-----		-----	
257		-----		-----	
309	D5185	98.2		0.06	
325	D5185	101		0.67	
349	D5185	100		0.45	
432	D5185	95		-0.64	
496	D5185	95.91		-0.44	
614	D5185	103.1		1.13	
633	D6595	110.7		2.78	
780	D5185	89		-1.94	
823	D5185	94	C	-0.85	first reported 184
862	D5185	103		1.11	
912	D5185	99		0.23	
962	D5185	96.2		-0.38	
963	D5185	99.26		0.29	
994	D5185	100.7		0.61	
1011	D5185	104		1.32	
1146	D5185	98.94		0.22	
1155		-----		-----	
1171		-----		-----	
1213		-----		-----	
1324	D5185	94.8		-0.68	
1409		-----		-----	
1417		84.0		-3.03	
1448		-----		-----	
1456	D5185	103		1.11	
1660		-----		-----	
1748		-----		-----	
1875	EN11885	100		0.45	
6009		-----		-----	
6016	D6595	89		-1.94	
6034		-----		-----	
6054	IP501	111.240		2.90	
6197	D4951	92		-1.29	
6253		-----		-----	
6310	D7751	104		1.32	
6425		-----		-----	

normality	OK
n	25
outliers	0
mean (n)	97.922
st.dev. (n)	7.0861
R(calc.)	19.841
st.dev.(D5185:18)	4.5908
R(D5185:18)	12.854





APPENDIX 2

Number of participants per country

1 lab in	ALGERIA
2 labs in	AUSTRALIA
1 lab in	AUSTRIA
1 lab in	AZERBAIJAN
3 labs in	BELGIUM
1 lab in	BOSNIA and HERZEGOVINA
2 labs in	CHINA, People's Republic
1 lab in	COTE D'IVOIRE
2 labs in	GERMANY
1 lab in	INDIA
1 lab in	ITALY
1 lab in	JORDAN
1 lab in	KAZAKHSTAN
1 lab in	KOREA, Republic of
1 lab in	MALAYSIA
1 lab in	MOROCCO
2 labs in	NETHERLANDS
1 lab in	NIGERIA
1 lab in	PHILIPPINES
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	SINGAPORE
1 lab in	SPAIN
2 labs in	TANZANIA
1 lab in	TURKEY
1 lab in	UNITED KINGDOM
2 labs in	UNITED STATES OF AMERICA
1 lab in	VIETNAM

APPENDIX 3

Abbreviations

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= 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)
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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)