Results of Proficiency Test
Hydraulic Fluid (fresh)
November 2018

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: A. Lewinska, MSc.
Corrector: ing. A.S. Noordman de – Neef & ing. R.J. Starink

Report no. iis18L09

February 2019

- EMPTY PAGE -

CONTENTS

1 INTRODUCTION

Since 2014, the Institute for Interlaboratory Studies (iis) organizes a proficiency test for the analysis of fresh Hydraulic Fluid every year. It was decided to continue this interlaboratory study during the annual program 2018/2019.
In this interlaboratory study, 36 laboratories from 28 different countries did register for participation. See appendix 3 for the number of participants per country. In this report, the test results of the 2018 interlaboratory study on fresh Hydraulic Fluid 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 organiser of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send one sample of 1L fresh Hydraulic Fluid, labelled #18225. 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 (iis) in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC 17043:2010 (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 organisation 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

The necessary bulk material was obtained from a local supplier. After homogenisation, 68 amber glass bottles of 1 litre were filled and labelled #18225.
The homogeneity of the subsamples #18225 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 8 stratified randomly selected samples.

	Density at 15°C in kg/L	*Kinematic Viscosity at 40°C in mm^2/s*
Sample #18225-1	0.87026	65.75
Sample #18225-2	0.87025	65.86
Sample #18225-3	0.87026	65.79
Sample #18225-4	0.87025	65.76
Sample #18225-5	0.87025	65.88
Sample #18225-6	0.87025	65.77
Sample #18225-7	0.87026	65.82
Sample #18225-8	0.87026	65.81

Table 1: homogeneity test results of subsamples #18225

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 ISO 13528, 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	0.13
reference test method	D4052:18	D445:18
0.3 x R (ref. test method)	0.00015	0.24

Table 2: evaluation of the repeatabilities of subsamples #18225

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibility of the reference test methods. Therefore, homogeneity of the subsamples #18225 was assumed.

To each of the participating laboratories, one 1 L amber glass bottle, labelled #18225, was sent on October 24, 2018. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYSES

The participants were asked to determine on sample #18225: Acid Number (Total), Copper Corrosion (3hrs at 50°C), Density at 15°C, Flash Point PMcc, Foam Characteristics (Foam 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), Sulphur, Water by KF, Water Separability at 54°C and Calcium, Phosphorus and Zinc.
Also, additional questions were asked about the Acid Number and the foam determination.

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’ 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 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 reanalysis). Additional or corrected test results are used for data analysis and 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 organisation 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 ISO 5725 the original test results per determination were submitted to Dixon’s, Grubbs' and/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 analysis 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. The Kernel Density Graph 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 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. In some cases, a reproducibility based on former iis proficiency tests could be used.

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 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

In this proficiency test no severe problems were encountered with the dispatch of the samples. One participant reported after the final reporting date and one other participant did not report any test results at all. Not all laboratories were able to report all analyses requested.
In total 35 participants reported 465 numerical test results. Observed were 18 outlying test results, which is 3.9% of the numerical test results. 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.

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 methods are also in the table together with the original data. The abbreviations, used in these tables, are listed 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.

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 reproducibility estimated from the Horwitz equation.

Acid Number (Total): 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 D664-A:17a; IP - 60 mL and BEP, pH 11, 60 and 125 mL. However, it is in agreement with the requirements of IP – 125 mL. When evaluated separately over BEP or IP the calculated reproducibility of IP is in full agreement with the precision data of the procedures of Inflection Point (60 mL).

Copper Corrosion: This determination was not problematic. Almost all participants agreed on a test result of 1 (1a, 1b). One participant reported “noncorrosive – 2E”.

Density at 15°C: This determination was problematic for a number of laboratories. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D4052:18.

Flash Point PMcc: This determination was problematic. No statistical outliers were observed and one test result was excluded. However, the calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of ASTM D93-A:18.

Foaming Characteristics (Tendency and Stability): This determination was problematic. No statistical outliers were observed. The calculated reproducibility in the Foam Tendency determination for sequence I, II and III is not in agreement with the requirements of ASTM D892:18. The variation over the test results for sequence I and III is very large. Therefore, it was decided not to calculate z-scores.
All reporting participants reported 0 mL for foam stability.
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).
Surprisingly all reporting participants reported the same answers.

Kinematic Viscosity at 40°C: This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D445:18.

Kinematic Viscosity at 100°C: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ASTM D445:18.

Viscosity Index This determination was not problematic. Two statistical outliers were observed and one test result was excluded. However, the calculated reproducibility after rejection of the suspect data is in full agreement with the requirements of ASTM D2270:10(2016).

Viscosity Stabinger at 40°C: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D7042:16e3.

Viscosity Stabinger at 100°C: 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 D7042:16e3.

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

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

Sulphur: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D4294:16e1.

Water content by KF: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D6304:16e1.

Water Separability at 54°C: This determination was not problematic. No statistical outliers were observed over six parameters. The calculated reproducibilities for “time to reach ≤ 3ml emulsion” and “time to reach 37ml water” are both in agreement with the requirements of ASTM D1401:18a.

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

Phosphorus: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5185:18.

Zinc: All reporting laboratories agreed on the absence of Zinc.
Therefore, no z-scores were calculated.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant 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 results, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from literature reference test methods (in casu ASTM methods) are compared in the next table.

Parameter	unit	n	average	2.8 * sd	R (lit)
Acid Number (Total)	mg KOH/g	24	0.11	0.08	0.07
Copper Corrosion, 3 hrs at 50°C	rating	17	1	n.a.	n.a.
Density at 15°C	kg/L	29	0.8702	0.0004	0.0005
Flash Point PMcc	°C	27	223.3	17.1	15.9
Foam Tendency Seq. I	ml	14	124.3	227.9	(52.0)
Foam Tendency Seq. II	ml	14	18.9	23.4	16.4
Foam Tendency Seq. III	ml	14	185.7	279.9	(81.7)
Foam Stability Seq. I	ml	13	0	0	n.a.
Foam Stability Seq. II	ml	13	0	0	n.a.
Foam Stability Seq. III	ml	13	0	0	n.a.
Kinematic viscosity at 40°C	mm^2/s	26	65.673	0.440	0.801
Kinematic viscosity at 100°C	mm^2/s	25	8.696	0.128	0.120
Viscosity Index		26	104.8	2.0	2
Viscosity Stabinger at 40°C	mm^2/s	13	65.716	0.389	0.830
Viscosity Stabinger at 100°C	mm^2/s	15	8.725	0.136	0.105
Pour Point (manual)	°C	18	-31.7	10.9	9
Pour Point (automated), 1°C int.	°C	11	-34.4	4.3	5
Sulphur	mg/kg	15	163	36	51
Water content by KF	mg/kg	27	37.4	63.8	148.4
Water Separability at 54°C, distilled water					
- Time ≤ 3 ml emulsion	min	8	24.4	14.2	20
- Time 37 ml water	min	8	25.3	15.1	20
- Complete Break	min	6	25.5	3.3	n.a.
- Volume Oil phase	ml	9	41.2	6.1	n.a.
- Volume Water phase	ml	9	37.3	6.9	n.a.
- Volume Emulsion phase	ml	9	1.4	5.3	n.a.
Calcium as Ca	mg/kg	24	22.7	9.2	6.4
Phosphorus as P	mg/kg	24	29.7	17.0	23.4
Zinc as Zn	mg/kg	24	<60	n.a.	n.a.

Table 3: reproducibilities of tests on sample #18225

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 2018 WITH THE PREVIOUS PTS.

	November 2018	*November 2017*	*November 2016*	*November 2015*	*November 2014*
Number of reporting labs	35	45	43	45	29
Number of test results reported	465	610	597	569	346
Statistical outliers	18	28	30	26	19
Percentage outliers	3.9%	4.6%	5.0%	4.6%	5.5%

Table 4: comparison with previous proficiency tests

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

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

Determination	*November 2018*	*November 2017*	*November 2016*	*November 2015*	*November 2014*
Acid Number (Total)	-	+	+	++	++
Density at 15°C	+	--	+	-	+/-
Flash Point PMcc	-	+/-	+	+	-
Foam Tendency Seq. I	(--)	+/-	n.e.	--	--
Foam Tendency Seq. II	-	-	+	+/-	--
Foam Tendency Seq. III	(--)	n.e.	n.e.	--	--
Foam Stability Seq. I	n.e.	n.e.	n.e.	n.e.	n.e.
Foam Stability Seq. II	n.e.	n.e.	n.e.	n.e.	n.e.
Foam Stability Seq. III	n.e.	n.e.	n.e.	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.	+	-	-	+	+
Sulphur	+	+/-	-	+	+
Water content (by KF)	++	++	++	++	++
Water Separability ≤ 3ml emul.	+	+	+	++	++
Water Separability 37ml water	+	+	++	++	++
Calcium as Ca	-	+	n.e.	n.e.	n.e.
Phosphorus as P	+	+	+	+	++
Zinc as Zn	n.e.	-	-	n.e.	n.e.

Table 5: comparison determinations against the reference test methods

The performance of the determinations against the requirements of the respective test methods is listed in the above table. The following performance categories were used:

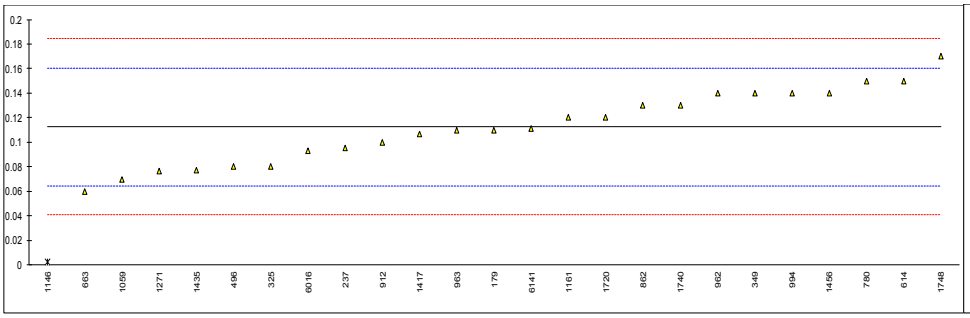
++:	group performed much better than the reference test method
+ :	group performed better than the reference test method
+/-:	group performance equals the reference test method
- :	group performed worse than the reference test method
-- :	group performed much worse than the reference test method
n.e.: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

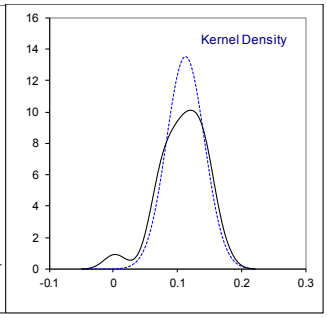
APPENDIX 1

Determination of Acid Number (Total) on sample #18225; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks	Determination of end point	Volume of titration solvent
173		-----		-----		---	---
178		-----		-----		---	---
179	D664-B	0.11		-0.10		Inflection Point	60 mL
237	D664-A	0.095		-0.73		Inflection Point	125 mL
255		-----		-----		---	---
325	D664-A	0.08		-1.36		Buffer End Point (pH 11)	125 mL
349	D664-A	0.14	C	1.15	fr. 0.39	Inflection Point	125 mL
432		-----		-----		---	---
496	D664-A	0.08		-1.36		Buffer End Point (pH 11)	60 mL
614	D664-A	0.15		1.57		---	60 mL
663	D664-A	0.06		-2.19		Inflection Point	125 mL
780	D664-A	0.15		1.57		Inflection Point	60 mL
862	D664-A	0.13		0.73		Inflection Point	60 mL
912	D664-A	0.1		-0.52		---	---
962	D974	0.14		1.15		---	---
963	D664-A	0.11		-0.10		Inflection Point	60 mL
994	D664-A	0.14		1.15		Inflection Point	125 mL
1011		-----		-----		---	---
1059	ISO6619	0.07		-1.77		Buffer End Point (pH 11)	60 mL
1146	D664-A	0.0027	R(0.05)	-4.58		Buffer End Point (pH 11)	125 mL
1161	D664-A	0.120		0.31		Inflection Point	125 mL
1174		-----		-----		---	---
1271	ISO6618	0.0765		-1.50		---	---
1331		-----		-----		---	---
1417	D664-A	0.107		-0.23		Inflection Point	125 mL
1435	D664-A	0.077		-1.48		Buffer End Point (pH 11)	---
1448		-----		-----		---	---
1456	D974	0.14		1.15		Buffer End Point (pH 11)	125 mL
1660		-----		-----		---	---
1720	D974	0.12		0.31		---	---
1740	D664-A	0.13		0.73		Inflection Point	60 mL
1748	D664-A	0.17		2.40		---	---
1797		-----		-----		---	---
1890		-----		-----		---	---
6016	D664-A	0.093		-0.81		Inflection Point	60 mL
6141	D974	0.11142		-0.04		---	125 mL

			BEP (pH 11) only (60+125mL)	Inflection point only (60+125mL)
normality	OK		not OK	OK
n	24		5	12
outliers	1		1	0
mean (n)	0.1125		0.0894	0.1154
st.dev. (n)	0.02948		0.02858	0.02517
R(calc.)	0.0825		0.0800	0.0705
st.dev.(D664-A:17a)	0.02395	IP – 60mL	-	-
R(D664-A:17a)	0.0671		-	0.0685
Compare				
R(D664-A:17a)	0.0536	BEP (pH 11) – 60mL	0.0428	-
R(D664-A:17a)	0.0495	BEP (pH 11) – 125mL		
R(D664-A:17a)	0.1569	IP – 125mL		





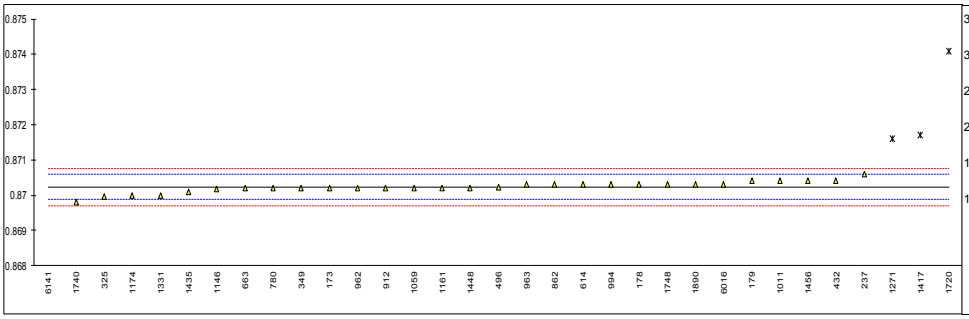
Determination of Copper Corrosion 3hrs at 50°C on sample #18225; results in rating

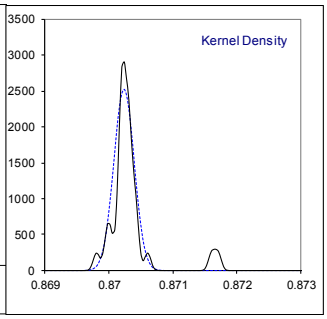
lab	method	value	mark	z(targ)	remarks
173		-----		-----	
178		-----		-----	
179	D130	1A		-----	
237	D130	1A		-----	
255		-----		-----	
325	D130	1A		-----	
349		-----		-----	
432		-----		-----	
496		-----		-----	
614	D130	1a		-----	
663	D130	1a		-----	
780	D130	1a		-----	
862	D130	1a		-----	
912	D130	1A		-----	
962		-----		-----	
963		-----		-----	
994	D130	1a		-----	
1011	D130	1b		-----	
1059	ISO2160	1a		-----	
1146		-----		-----	
1161	ISO2160	1a		-----	
1174	ISO2160	1a		-----	
1271	D130	1a		-----	
1331	GB/T5096	1a		-----	
1417		-----		-----	
1435		-----		-----	
1448		-----		-----	
1456	D130	1A		-----	
1660		-----		-----	
1720		-----		-----	
1740		-----		-----	
1748	D130	1a		-----	
1797		-----		-----	
1890		-----		-----	
6016		-----		-----	
6141	IEC62535	noncorrosive -2E		-----	
	n	17			
	mean (n)	1 (1A, 1B)			

Determination of Density at 15°C on sample #18225; results in kg/L

lab	method	value	mark	z(targ)	remarks
173	D4052	0.8702		-0.17	
178	D4052	0.8703		0.39	
179	D4052	0.8704		0.95	
237	D4052	0.8706		2.07	
255		-----		-----	
325	D4052	0.86995		-1.57	
349	D4052	0.8702		-0.17	
432	D4052	0.87042		1.06	
496	D4052	0.87023		0.00	
614	D1298	0.8703		0.39	
663	D4052	0.87020		-0.17	
780	D4052	0.8702		-0.17	
862	D4052	0.8703		0.39	
912	D4052	0.8702		-0.17	
962	D4052	0.8702		-0.17	
963	D4052	0.8703		0.39	
994	D4052	0.8703		0.39	
1011	D4052	0.8704		0.95	
1059	D4052	0.8702		-0.17	
1146	D4052	0.87017		-0.34	
1161	ISO3675	0.87020	C	-0.17	first reported 868.9 kg/m³
1174	ISO3675	0.8700		-1.29	
1271	D4052	0.8716	C,R(0.01)	7.67	first reported 873.3 kg/m³
1331	ISO12185	0.87000		-1.29	
1417	IP365	0.8717	R(0.01)	8.23	
1435	D4052	0.8701		-0.73	
1448	D4052	0.8702		-0.17	
1456	D4052	0.8704		0.95	
1660		-----		-----	
1720	D4052	0.8741	C,R(0.01)	21.67	first reported 869.6 kg/m³
1740	D4052	0.8698		-2.41	
1748	D4052	0.8703		0.39	
1797		-----		-----	
1890	ISO12185	0.8703		0.39	
6016	D4052	0.87030		0.39	
6141	D1298	0.865	R(0.01)	-29.29	

normality	suspect
n	29
outliers	4
mean (n)	0.87023
st.dev. (n)	0.000158
R(calc.)	0.00044
st.dev.(D4052:18)	0.000179
R(D4052:18)	0.00050

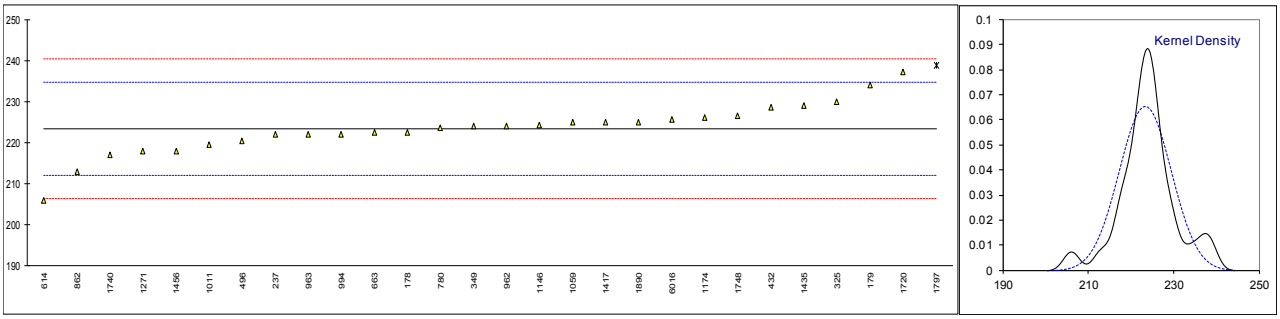




Determination of Flash Point PMcc on sample #18225; results in °C

lab	method	value	mark	z(targ)	remarks
173		-----		-----	
178	D93-A	222.5		-0.15	
179	D93-A	234.0		1.88	
237	D93-A	222		-0.24	
255		-----		-----	
325	D93-A	230		1.18	
349	D93-A	224		0.12	
432	D93-A	228.5		0.91	
496	D93-A	220.5		-0.50	
614	D93-A	206		-3.06	
663	D93-A	222.4		-0.17	
780	D93-A	223.5		0.03	
862	D93-A	213		-1.83	
912		-----		-----	
962	D93-A	224		0.12	
963	D93-A	222.0		-0.24	
994	D93-A	222.0		-0.24	
1011	D93-A	219.5		-0.68	
1059	ISO2719-A	225.0		0.29	
1146	D93-A	224.2		0.15	
1161		-----		-----	
1174	ISO2719-A	226		0.47	
1271	ISO2719-A	218		-0.94	
1331		-----		-----	
1417	IP34-A	225		0.29	
1435	D93-A	229.0	C	1.00	first reported 148.5
1448		-----		-----	
1456	D93-A	218.0		-0.94	
1660		-----		-----	
1720	D93-A	237.1		2.43	
1740	D93-B	217		-1.12	
1748	D93-A	226.5		0.56	
1797	ISO2592	238.7	ex	2.71	excluded: open cup method is not equivalent to closed cup method
1890	D93-A	225		0.29	
6016	D93-B	225.6		0.40	
6141		-----		-----	

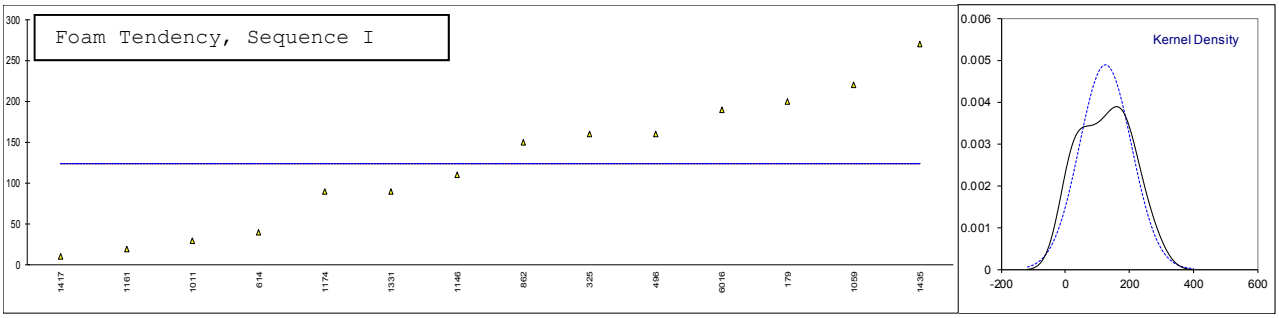
normality	not OK
n	27
outliers	0+1ex
mean (n)	223.34
st.dev. (n)	6.116
R(calc.)	17.13
st.dev.(D93-A:18)	5.663
R(D93-A:18)	15.86

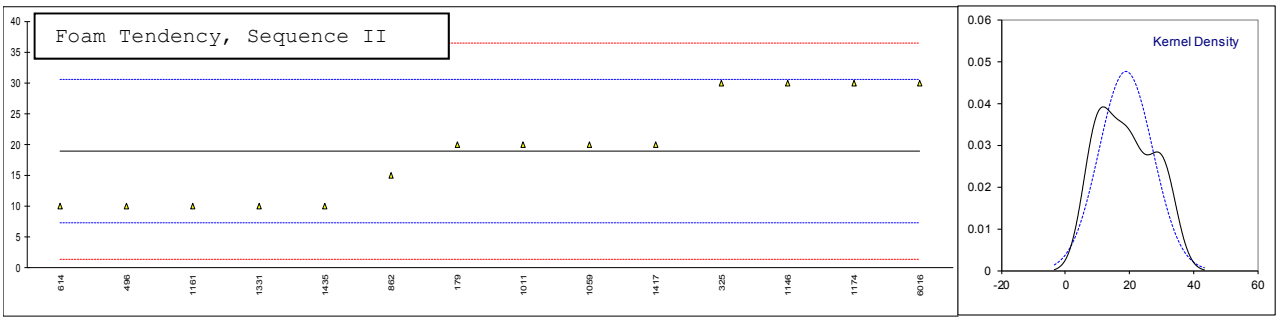


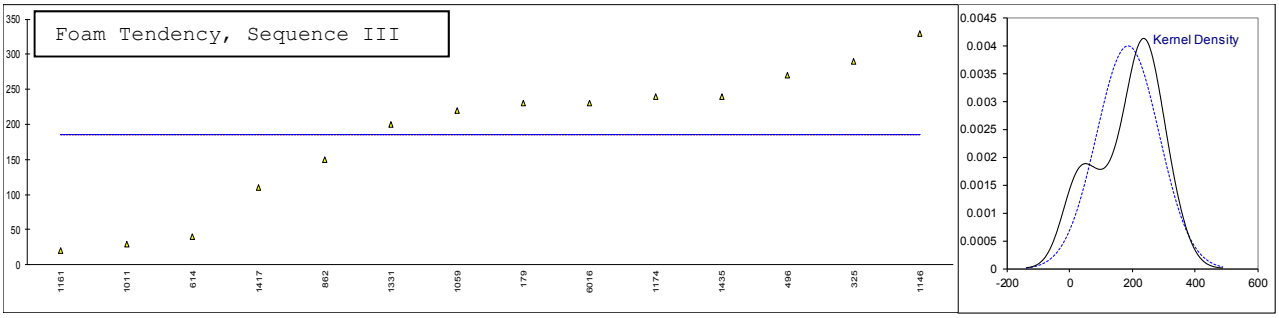
Determination of Foaming Characteristics, Foaming Tendency (at end of 5 min blowing period) on sample #18225; results in ml

lab	method	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
173		-----		-----	-----		-----	-----		-----
178		-----		-----	-----		-----	-----		-----
179	D892	200		-----	20		0.18	230		-----
237		-----		-----	-----		-----	-----		-----
255		-----		-----	-----		-----	-----		-----
325	D892	160		-----	30		1.89	290		-----
349		-----		-----	-----		-----	-----		-----
432		-----		-----	-----		-----	-----		-----
496	D892	160		-----	10		-1.53	270		-----
614	D892	40		-----	10		-1.53	40		-----
663		-----		-----	-----		-----	-----		-----
780		-----		-----	-----		-----	-----		-----
862	D892	150		-----	15		-0.67	150		-----
912		-----		-----	-----		-----	-----		-----
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
994		-----		-----	-----		-----	-----		-----
1011	D892	30		-----	20		0.18	30		-----
1059	D892	220		-----	20		0.18	220		-----
1146	ISO6247	110		-----	30		1.89	330		-----
1161	D892	20		-----	10		-1.53	20		-----
1174	ISO6247	90		-----	30		1.89	240		-----
1271		-----		-----	-----		-----	-----		-----
1331	GB/T12579	90		-----	10		-1.53	200		-----
1417	D892	10		-----	20		0.18	110		-----
1435	D892	270		-----	10		-1.53	240		-----
1448		-----		-----	-----		-----	-----		-----
1456		-----		-----	-----		-----	-----		-----
1660		-----		-----	-----		-----	-----		-----
1720		-----		-----	-----		-----	-----		-----
1740		-----		-----	-----		-----	-----		-----
1748		-----		-----	-----		-----	-----		-----
1797		-----		-----	-----		-----	-----		-----
1890		-----		-----	-----		-----	-----		-----
6016	D892	190		-----	30		1.89	230		-----
6141		-----		-----	-----		-----	-----		-----

normality	OK	OK	OK
n	14	14	14
outliers	0	0	0
mean (n)	124.29	18.93	185.71
st.dev. (n)	81.403	8.362	99.978
R(calc.)	227.93	23.41	279.94
st.dev.(D892:18)	(18.569)	5.843	(29.184)
R(D892:18)	(51.99)	16.36	(81.71)


Foam Tendency, Sequence I
Kernel Density


Foam Tendency, Sequence II
Kernel Density


Foam Tendency, Sequence III
Kernel Density

Determination of Foaming Characteristics, Foaming Stability (at end of 10 min settling period) on sample #18225; results in ml

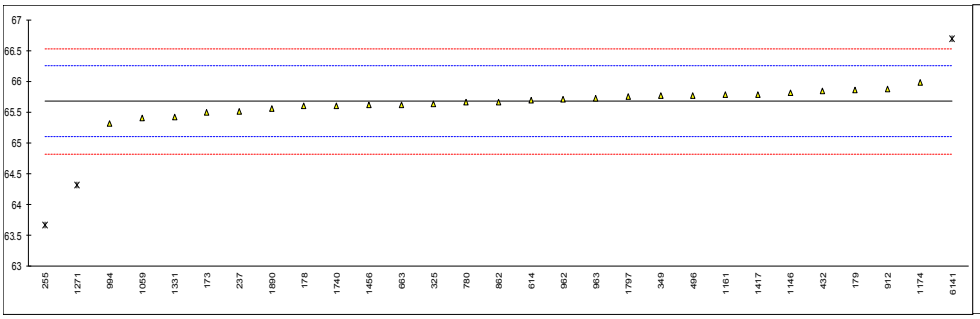
lab	method	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
173		-----		-----	-----		-----	-----		-----
178		-----		-----	-----		-----	-----		-----
179	D892	0		-----	0		-----	0		-----
237		-----		-----	-----		-----	-----		-----
255		-----		-----	-----		-----	-----		-----
325	D892	0		-----	0		-----	0		-----
349		-----		-----	-----		-----	-----		-----
432		-----		-----	-----		-----	-----		-----
496	D892	0		-----	0		-----	0		-----
614		0		-----	0		-----	0		-----
663		-----		-----	-----		-----	-----		-----
780		-----		-----	-----		-----	-----		-----
862	D892	0		-----	0		-----	0		-----
912		-----		-----	-----		-----	-----		-----
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
994		-----		-----	-----		-----	-----		-----
1011	D892	0		-----	0		-----	0		-----
1059	D892	0		-----	0		-----	0		-----
1146	ISO6247	0		-----	0		-----	0		-----
1161		-----		-----	-----		-----	-----		-----
1174	ISO6247	0		-----	0		-----	0		-----
1271		-----		-----	-----		-----	-----		-----
1331	GB/T12579	0		-----	0		-----	0		-----
1417	D892	0		-----	0		-----	0		-----
1435	D892	0	C	-----	0	C	-----	0	C	-----
1448		-----		-----	-----		-----	-----		-----
1456		-----		-----	-----		-----	-----		-----
1660		-----		-----	-----		-----	-----		-----
1720		-----		-----	-----		-----	-----		-----
1740		-----		-----	-----		-----	-----		-----
1748		-----		-----	-----		-----	-----		-----
1797		-----		-----	-----		-----	-----		-----
1890		-----		-----	-----		-----	-----		-----
6016	D892	0		-----	0		-----	0		-----
6141		-----		-----	-----		-----	-----		-----
n		13			13			13		
mean (n)		0			0			0		

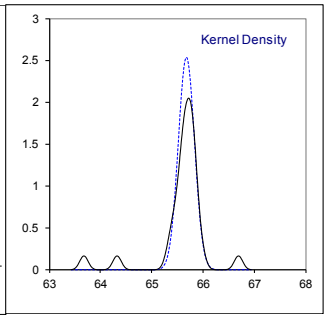
Lab 1435 first reported 340, 10, 276

Determination of Kinematic Viscosity at 40°C on sample #18225; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
173	D445	65.50		-0.60	
178	D445	65.60		-0.25	
179	D445	65.86		0.65	
237	D445	65.51		-0.57	
255	D7279 corrected to D445	63.67	C,R(0.01)	-7.00	first reported 64.62
325	D445	65.63		-0.15	
349	D445	65.77		0.34	
432	D445	65.85		0.62	
496	D445	65.773		0.35	
614	D445	65.70		0.09	
663	D445	65.623		-0.17	
780	D445	65.67		-0.01	
862	D445	65.67		-0.01	
912	D445	65.87		0.69	
962	D445	65.71		0.13	
963	D445	65.73		0.20	
994	D445	65.32		-1.23	
1011		-----		-----	
1059	ISO3104	65.41		-0.92	
1146	D445	65.817		0.50	
1161	ISO3104	65.776		0.36	
1174	ISO3104	65.9756		1.06	
1271	ISO3104	64.318	C,R(0.01)	-4.73	first reported 64.689
1331	D445	65.42		-0.88	
1417	D445	65.78		0.37	
1435		-----		-----	
1448		-----		-----	
1456	D445	65.61		-0.22	
1660		-----		-----	
1720		-----		-----	
1740	D445	65.60		-0.25	
1748		-----		-----	
1797	ISO3104	65.76		0.30	
1890	ISO3104	65.560		-0.39	
6016		-----		-----	
6141	D7279 corrected to D445	66.69	R(0.01)	3.55	

normality	OK
n	26
outliers	3
mean (n)	65.6729
st.dev. (n)	0.15721
R(calc.)	0.4402
st.dev.(D445:18)	0.28615
R(D445:18)	0.8012

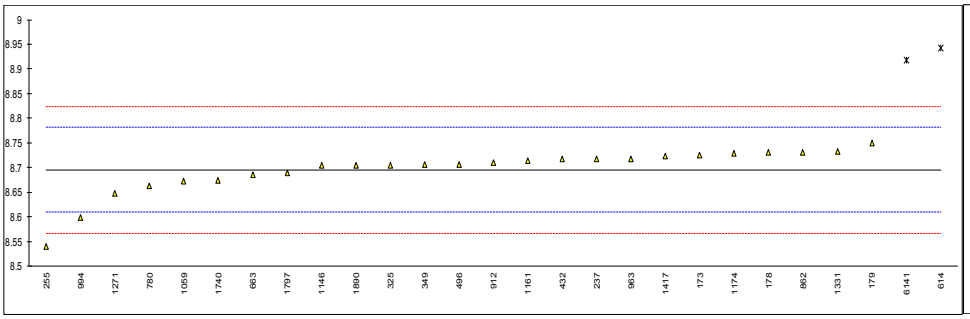


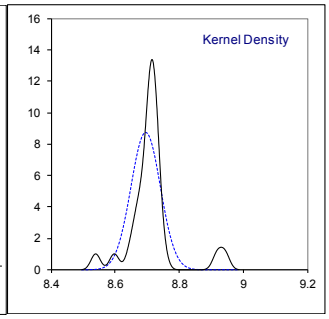


Determination of Kinematic Viscosity at 100°C on sample #18225; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
173	D445	8.725		0.68	
178	D445	8.730		0.80	
179	D445	8.750		1.27	
237	D445	8.718		0.52	
255	D7279 corrected to D445	8.54	C	-3.63	first reported 8.98
325	D445	8.705		0.22	
349	D445	8.706	C	0.24	first reported 10.44
432	D445	8.717		0.50	
496	D445	8.7064		0.25	
614	D445	8.943	R(0.01)	5.77	
663	D445	8.6864		-0.22	
780	D445	8.663		-0.76	
862	D445	8.73		0.80	
912	D445	8.710		0.33	
962		-----		-----	
963	D445	8.718		0.52	
994	D445	8.598		-2.28	
1011		-----		-----	
1059	ISO3104	8.673		-0.53	
1146	D445	8.7037		0.19	
1161	ISO3104	8.713		0.40	
1174	ISO3104	8.7285		0.76	
1271	ISO3104	8.647		-1.14	
1331	D445	8.733		0.87	
1417	D445	8.724		0.66	
1435		-----		-----	
1448		-----		-----	
1456		-----		-----	
1660		-----		-----	
1720		-----		-----	
1740	D445	8.674		-0.51	
1748		-----		-----	
1797	ISO3104	8.690		-0.13	
1890	ISO3104	8.704		0.19	
6016		-----		-----	
6141	D7279 corrected to D445	8.91875	R(0.01)	5.20	

normality	not OK
n	25
outliers	2
mean (n)	8.6957
st.dev. (n)	0.04571
R(calc.)	0.1280
st.dev.(D445:18)	0.04286
R(D445:18)	0.1200

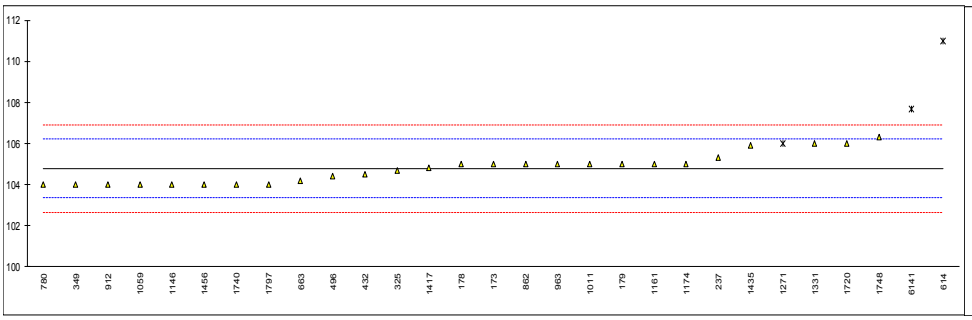


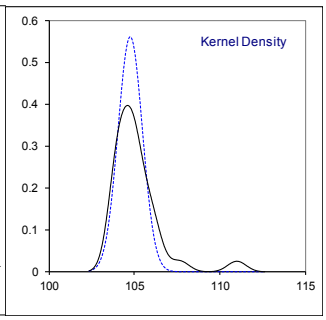


Determination of Viscosity Index on sample #18225; unit less results

lab	method	value	mark	z(targ)	iis calc.	mark	remarks
173	D2270	105		0.32	104.98		
178	D2270	105		0.32	104.89		
179	D2270	105		0.32	104.80		
237	D2270	105.3		0.74	104.80		
255		-----		-----	104.57		
325	D2270	104.7		-0.10	104.26		
349	D2270	104	C	-1.08	104.00		first reported 147
432	D2270	104.5		-0.38	104.08		
496	D2270	104.4		-0.52	104.00		
614	D2270	111	R(0.01)	8.72	109.24	R(0.01)	
663	D2270	104.2		-0.80	103.85		
780	D2270	104		-1.08	103.22		
862	D2270	105		0.32	104.74		
912	D2270	104		-1.08	103.88		
962		-----		-----	-----		
963	D2270	105		0.32	104.35		
994		-----		-----	102.43		
1011	D2270	105		0.32	105.00		
1059	ISO2909	104		-1.08	103.99		
1146	D2270	104		-1.08	103.85		
1161	D2270	105		0.32	104.14		
1174	ISO2909	105		0.32	104.08		
1271	ISO2909	106	ex	1.72	105.67		outlier in viscosity 40° C
1331	GB/T1995	106		1.72	105.32		
1417	D2270	104.8		0.04	104.38		
1435	D2270	105.88		1.55	106.00		
1448		-----		-----	-----		
1456	D2270	104		-1.08	104.00		
1660		-----		-----	-----		
1720	D2270	106		1.72	106.00		
1740	D2270	104		-1.08	103.61		
1748	D2270	106.3		2.14	106.00		
1797	ISO2909	104		-1.08	103.65		
1890		-----		-----	104.38		
6016		-----		-----	-----		
6141	D2270	107.669	R(0.05)	4.06	106.80		

			iis calculated
	normality	OK	OK
	n	26	31
	outliers	2+1ex	1
	mean (n)	104.77	104.51
	st.dev. (n)	0.710	0.917
	R(calc.)	1.99	2.57
	st.dev.(D2270:10)	0.714	0.714
	R(D2270:10)	2	2

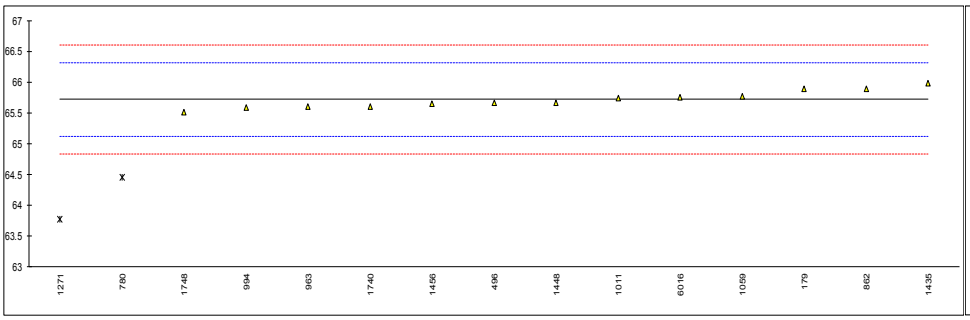


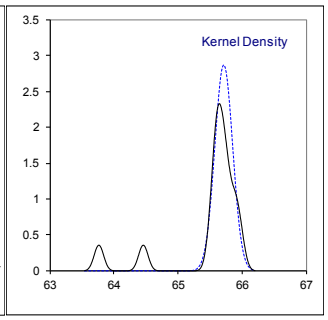


Determination of Viscosity Stabinger at 40°C on sample #18225; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
173		-----		-----	
178		-----		-----	
179	D7042	65.89		0.59	
237		-----		-----	
255		-----		-----	
325		-----		-----	
349		-----		-----	
432		-----		-----	
496	D7042	65.658		-0.19	
614		-----		-----	
663		-----		-----	
780	D7042	64.46	D(0.01)	-4.24	
862	D7042	65.89		0.59	
912		-----		-----	
962		-----		-----	
963	D7042	65.60		-0.39	
994	D7042	65.58		-0.46	
1011	D7042	65.74		0.08	
1059	D7042	65.77		0.18	
1146		-----		-----	
1161		-----		-----	
1174		-----		-----	
1271	D7042	63.767	C,D(0.01)	-6.58	first reported 64.653
1331		-----		-----	
1417		-----		-----	
1435	D7042	65.981		0.90	
1448	D7042	65.67		-0.15	
1456	D7042	65.65		-0.22	
1660		-----		-----	
1720		-----		-----	
1740	D7042	65.601		-0.39	
1748	D7042	65.514		-0.68	
1797		-----		-----	
1890		-----		-----	
6016	D7042	65.758		0.14	
6141		-----		-----	

normality	OK
n	13
outliers	2
mean (n)	65.7155
st.dev. (n)	0.13900
R(calc.)	0.3892
st.dev.(D7042:16e3)	0.29627
R(D7042:16e3)	0.8295

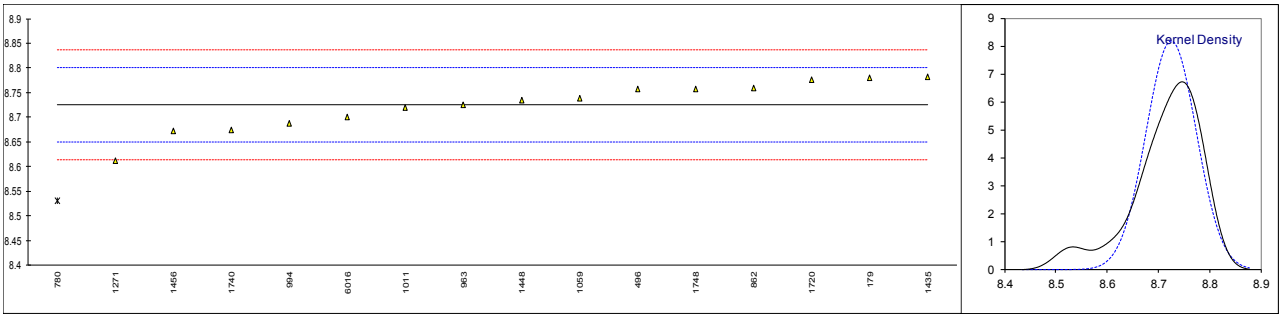




Determination of Viscosity Stabinger at 100°C on sample #18225; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
173		-----		-----	
178		-----		-----	
179	D7042	8.780		1.47	
237		-----		-----	
255		-----		-----	
325		-----		-----	
349		-----		-----	
432		-----		-----	
496	D7042	8.7579		0.87	
614		-----		-----	
663		-----		-----	
780	D7042	8.530	D(0.05)	-5.22	
862	D7042	8.76		0.93	
912		-----		-----	
962		-----		-----	
963	D7042	8.725		-0.01	
994	D7042	8.688		-1.00	
1011	D7042	8.720		-0.14	
1059	D7042	8.739		0.37	
1146		-----		-----	
1161		-----		-----	
1174		-----		-----	
1271	D7042	8.611		-3.06	
1331		-----		-----	
1417		-----		-----	
1435	D7042	8.7826		1.53	
1448	D7042	8.734		0.23	
1456	D7042	8.672		-1.42	
1660		-----		-----	
1720	D7042	8.777		1.38	
1740	D7042	8.6739		-1.37	
1748	D7042	8.758		0.88	
1797		-----		-----	
1890		-----		-----	
6016	D7042	8.700		-0.67	
6141		-----		-----	

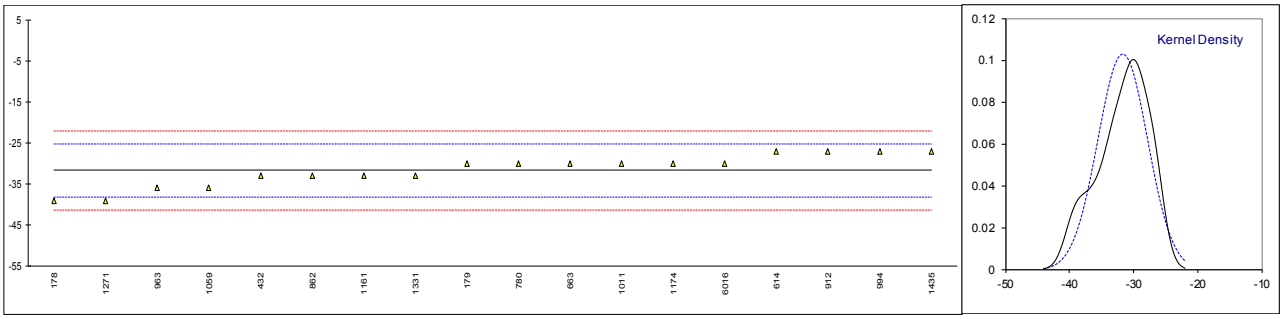
normality	OK
n	15
outliers	1
mean (n)	8.7252
st.dev. (n)	0.04866
R(calc.)	0.1363
st.dev.(D7042:16e3)	0.03739
R(D7042:16e3)	0.1047



Determination of Pour Point, manual on sample #18225; results in °C

lab	method	value	mark	z(targ)	remarks
173		-----		-----	
178	D97	-39		-2.28	
179	D97	-30		0.52	
237	D97	<-21		-----	
255		-----		-----	
325		-----		-----	
349		-----		-----	
432	D97	-33		-0.41	
496		-----		-----	
614	D97	-27		1.45	
663	D97	-30		0.52	
780	D97	-30		0.52	
862	D97	-33		-0.41	
912	D97	-27		1.45	
962		-----		-----	
963	D97	-36		-1.35	
994	D97	-27		1.45	
1011	D97	-30		0.52	
1059	ISO3016	-36		-1.35	
1146		-----		-----	
1161	D97	-33		-0.41	
1174	ISO3016	-30		0.52	
1271	ISO3016	-39		-2.28	
1331	GB/T3535	-33.0		-0.41	
1417		-----		-----	
1435	ISO3016	-27		1.45	
1448		-----		-----	
1456		-----		-----	
1660		-----		-----	
1720		-----		-----	
1740		-----		-----	
1748		-----		-----	
1797		-----		-----	
1890		-----		-----	
6016	D97	-30		0.52	
6141		-----		-----	

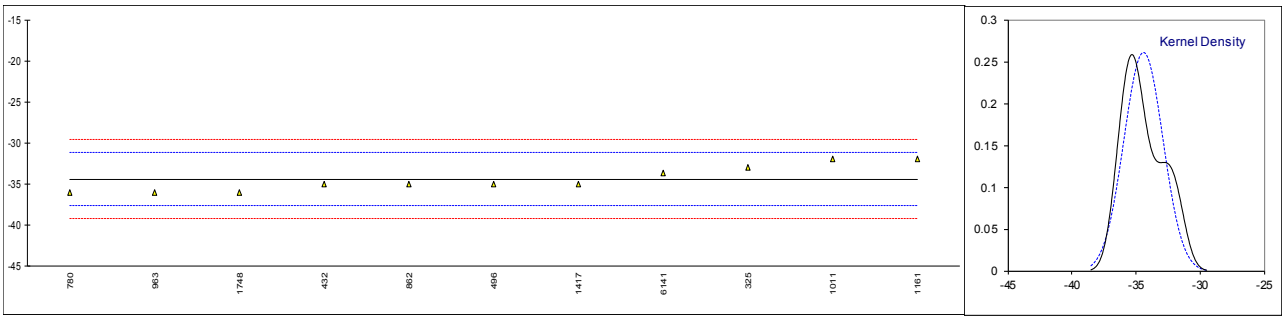
normality	OK
n	18
outliers	0
mean (n)	-31.67
st.dev. (n)	3.881
R(calc.)	10.87
st.dev.(D97:17b)	3.214
R(D97:17b)	9



Determination of Pour Point, automated, 1°C interval on sample #18225; results in °C

lab	method	value	mark	z(targ)	remarks
173		-----		-----	
178		-----		-----	
179		-----		-----	
237		-----		-----	
255		-----		-----	
325	D5950	-33		0.88	
349		-----		-----	
432	D5950	-35		-0.36	
496	D6892	-35		-0.36	
614		-----		-----	
663		-----		-----	
780	D5950	-36		-0.98	
862	D5950	-35		-0.36	
912		-----		-----	
962		-----		-----	
963	D5950	-36		-0.98	
994		-----		-----	
1011	D6892	-32		1.50	
1059		-----		-----	
1146		-----		-----	
1161	D6749	-32		1.50	
1174		-----		-----	
1271		-----		-----	
1331		-----		-----	
1417	D5950	-35		-0.36	
1435		-----		-----	
1448		-----		-----	
1456		-----		-----	
1660		-----		-----	
1720		-----		-----	
1740		-----		-----	
1748		-36		-0.98	
1797		-----		-----	
1890		-----		-----	
6016		-----		-----	
6141	D6892	-33.6		0.51	

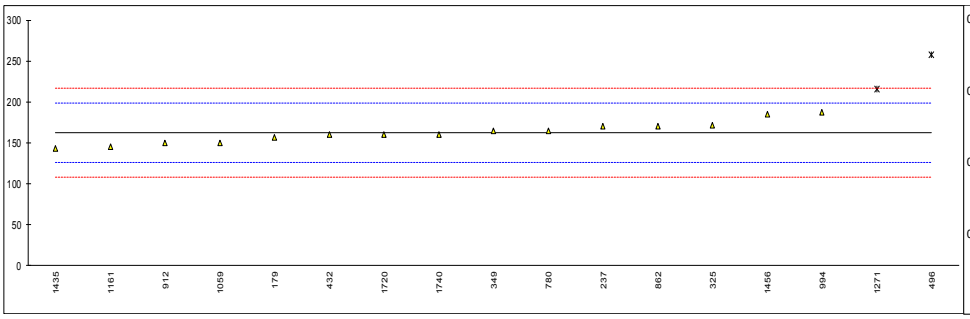
normality	OK
n	11
outliers	0
mean (n)	-34.42
st.dev. (n)	1.524
R(calc.)	4.27
st.dev.(D5950:14)	1.607
R(D5950:14)	5

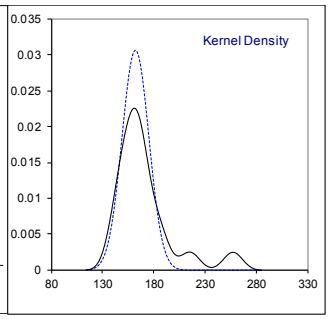


Determination of Sulphur on sample #18225; results in mg/kg

lab	method	value	mark	z(targ)	remarks
173		-----		-----	
178		-----		-----	
179	D4294	157		-0.30	
237	D4294	170		0.41	
255		-----		-----	
325	INH-6443/4929	171		0.47	
349	D2622	165	C	0.14	first reported 363
432	D5185	159.8		-0.15	
496	D2622	257.1	G(0.01)	5.19	
614		-----		-----	
663		-----		-----	
780	D4294	165		0.14	
862	D2622	170		0.41	
912	D4294	150		-0.69	
962		-----		-----	
963		-----		-----	
994	D4294	187.0		1.34	
1011		-----		-----	
1059	ISO14596	150		-0.69	
1146		-----		-----	
1161	ISO8754	145.14		-0.95	
1174		-----		-----	
1271		215.39	G(0.05)	2.90	
1331		-----		-----	
1417		-----		-----	
1435	D5185	142.5		-1.10	
1448		-----		-----	
1456	D5185	185		1.23	
1660		-----		-----	
1720	D4294	160		-0.14	
1740	D4294	160		-0.14	
1748		-----		-----	
1797		-----		-----	
1890		-----		-----	
6016		-----	W	-----	first reported 546.4
6141		-----		-----	

normality	OK
n	15
outliers	2
mean (n)	162.50
st.dev. (n)	13.005
R(calc.)	36.41
st.dev.(D4294:16e1)	18.232
R(D4294:16e1)	51.05

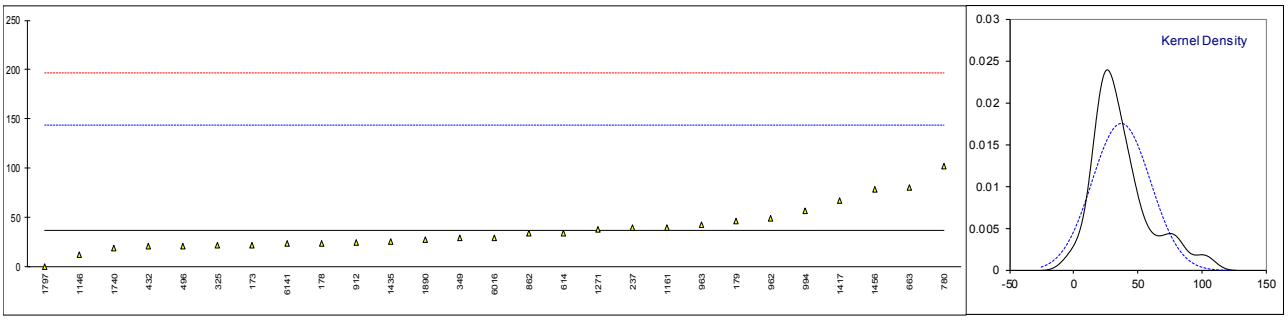




Determination of Water Content by KF on sample #18225; results in mg/kg

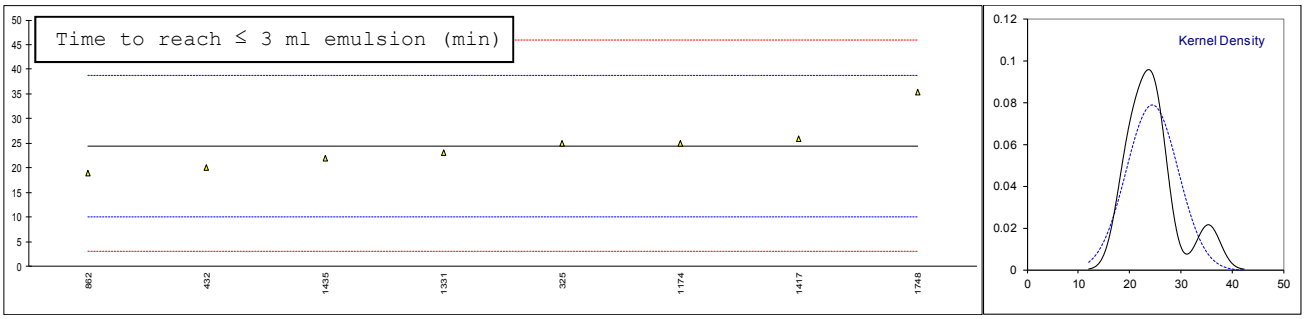
lab	method	value	mark	z(targ)	remarks
173	D6304-C	22		-0.29	
178	D6304-C	24		-0.25	
179	D6304-C	46		0.16	
237	D6304-C	39.42		0.04	
255		-----		-----	
325	D6304-C	22		-0.29	
349	D6304-A	29		-0.16	
432	D6304-C	20.8		-0.31	
496	D6304-C	21		-0.31	
614	D6304-C	34		-0.06	
663	D6304-C	80.15		0.81	
780	D6304-C	102		1.22	
862	D6304-C	33.9		-0.07	
912	D6304-C	25		-0.23	
962	D6304-A	49		0.22	
963	D6304-A	43		0.11	
994	IP438	57.0		0.37	
1011		-----		-----	
1059		-----		-----	
1146	D6304-C	12		-0.48	
1161	D6304-A	40.254		0.05	
1174		-----		-----	
1271	ISO12937	38		0.01	
1331		-----		-----	
1417	D6304-A	67		0.56	
1435	D6304-A	25.6		-0.22	
1448		-----		-----	
1456	D6304-A	78.4		0.77	
1660		-----		-----	
1720		-----		-----	
1740	D6304-C	19		-0.35	
1748		-----		-----	
1797	ISO13484/D95	0		-0.71	
1890	EN60814	27.4		-0.19	
6016	D6304-A	29.8		-0.14	
6141	D1533	23.8		-0.26	

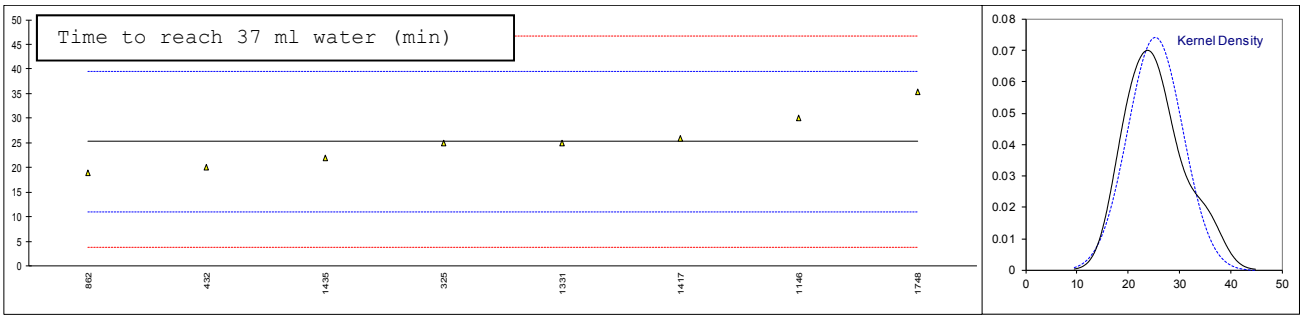
normality	not OK
n	27
outliers	0
mean (n)	37.390
st.dev. (n)	22.7913
R(calc.)	63.816
st.dev.(D6304:16e1)	52.9867
R(D6304:16e1)	148.363



Determination of Water Separability at 54 °C, distilled water on sample #18225; results in min.

lab	method	≤3 ml emul.	mk	z(targ)	37 ml water	mk	z(targ)	compl. break	mk	z(targ)	aborted	time aborted
173		-----		-----	-----		-----	-----		-----		-----
178		-----		-----	-----		-----	-----		-----		-----
179	D1401	-----		-----	-----		-----	25		-----	NO	-----
237	D1401	-----		-----	-----		-----	-----		-----	YES	30.07
255		-----		-----	-----		-----	-----		-----		-----
325	D1401	25		0.08	25		-0.04	-----		-----	YES	25
349		-----		-----	-----		-----	-----		-----		-----
432	D1401	20		-0.62	20		-0.74	30		-----	NO	-----
496		-----		-----	-----		-----	-----		-----		-----
614		-----		-----	-----		-----	-----		-----		-----
663		-----		-----	-----		-----	-----		-----		-----
780		-----		-----	-----		-----	-----		-----		-----
862	D1401	19		-0.76	19		-0.88	20		-----	YES	20
912		-----		-----	-----		-----	-----		-----		-----
962		-----		-----	-----		-----	-----		-----		-----
963		-----		-----	-----		-----	-----		-----		-----
994		-----		-----	-----		-----	-----		-----		-----
1011	D1401	-----		-----	-----		-----	25		-----	NO	-----
1059		-----		-----	-----		-----	-----		-----		-----
1146	D1401	-----		-----	30		0.66	-----		-----	YES	30
1161		-----		-----	-----		-----	-----		-----		-----
1174	ISO6614	25		0.08	-----		-----	-----		-----	YES	25
1271	ISO6614	-----		-----	-----		-----	27		-----		-----
1331	GB/T7305	23.0		-0.20	25.0		-0.04	-----		-----		30.0
1417		26		0.22	26		0.10	26		-----	NO	26
1435	D1401	22		-0.34	22		-0.46	-----		-----	YES	22
1448		-----		-----	-----		-----	-----		-----		-----
1456		-----		-----	-----		-----	-----		-----		-----
1660		-----		-----	-----		-----	-----		-----		-----
1720		-----		-----	-----		-----	-----		-----		-----
1740		-----		-----	-----		-----	-----		-----		-----
1748	D1401	35.35		1.53	35.35		1.41	-----		-----	YES	-----
1797		-----		-----	-----		-----	-----		-----		-----
1890		-----		-----	-----		-----	-----		-----		-----
6016	D1401	>30		-----	>30		-----	>30		-----	YES	30
6141		-----		-----	-----		-----	-----		-----		-----
	normality	unknown			unknown			unknown				
	n	8			8			6				
	outliers	0			0			0				
	mean (n)	24.42			25.29			25.50				
	st.dev. (n)	5.063			5.378			3.271				
	R(calc.)	14.18			15.06			9.16				
	st.dev.(D1401:18a)	7.143			7.143			unknown				
	R(D1401:18a)	20			20			unknown				

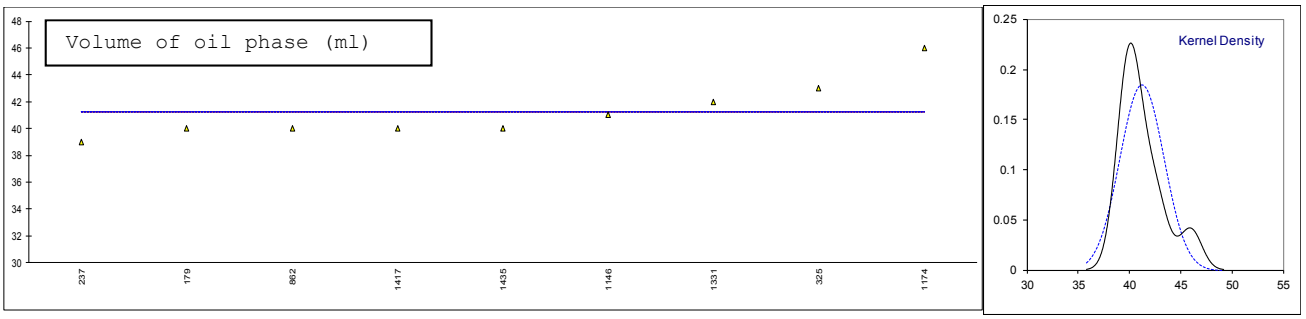


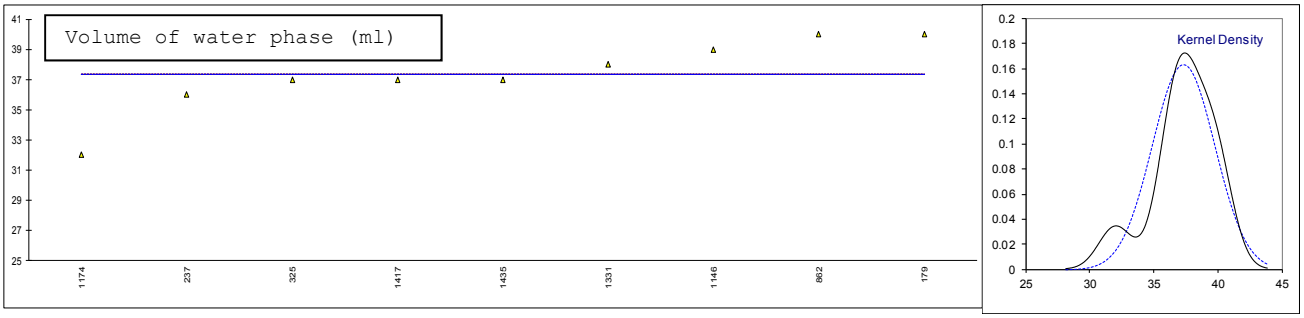


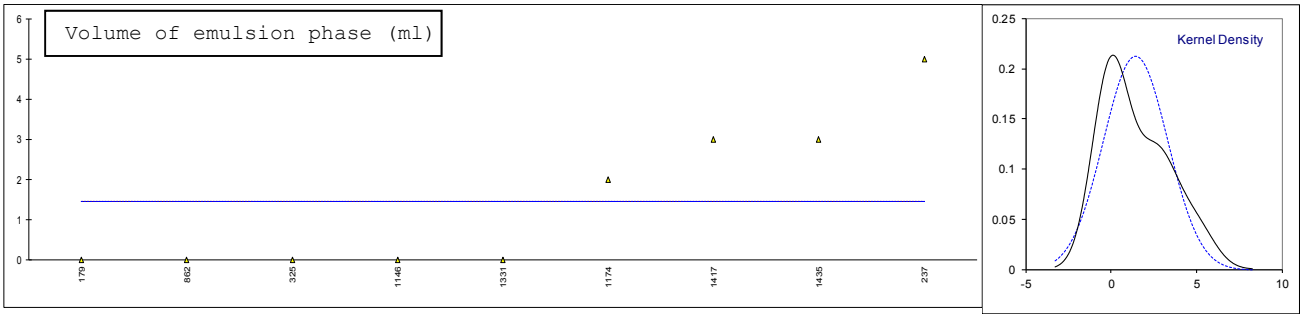
Determination of Water Separability at 54 °C, distilled water on sample #18225; results in ml.
--- Continued ----

lab	method	oil	mark	z(targ)	water	mark	z(targ)	emulsion	mark	z(targ)
173		-----		-----	-----		-----	-----		-----
178		-----		-----	-----		-----	-----		-----
179	D1401	40		-----	40		-----	0		-----
237	D1401	39		-----	36		-----	5		-----
255		-----		-----	-----		-----	-----		-----
325	D1401	43		-----	37		-----	0		-----
349		-----		-----	-----		-----	-----		-----
432	D1401	-----		-----	-----		-----	-----		-----
496		-----		-----	-----		-----	-----		-----
614		-----		-----	-----		-----	-----		-----
663		-----		-----	-----		-----	-----		-----
780		-----		-----	-----		-----	-----		-----
862	D1401	40		-----	40		-----	0		-----
912		-----		-----	-----		-----	-----		-----
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
994		-----		-----	-----		-----	-----		-----
1011	D1401	-----		-----	-----		-----	-----		-----
1059		-----		-----	-----		-----	-----		-----
1146	D1401	41	C	-----	39		-----	0	C	-----
1161		-----		-----	-----		-----	-----		-----
1174	ISO6614	46		-----	32		-----	2		-----
1271	ISO6614	-----		-----	-----		-----	-----		-----
1331	GB/T7305	42.0		-----	38.0		-----	0.0		-----
1417		40		-----	37		-----	3		-----
1435	D1401	40		-----	37	C	-----	3	C	-----
1448		-----		-----	-----		-----	-----		-----
1456		-----		-----	-----		-----	-----		-----
1660		-----		-----	-----		-----	-----		-----
1720		-----		-----	-----		-----	-----		-----
1740		-----		-----	-----		-----	-----		-----
1748	D1401	-----		-----	-----		-----	-----		-----
1797		-----		-----	-----		-----	-----		-----
1890		-----		-----	-----		-----	-----		-----
6016	D1401	-----		-----	-----		-----	-----		-----
6141		-----		-----	-----		-----	-----		-----
	normality	not OK			not OK			OK		
	n	9			9			9		
	outliers	0			0			0		
	mean (n)	41.22			37.33			1.44		
	st.dev. (n)	2.167			2.449			1.878		
	R(calc.)	6.07			6.86			5.26		
	st.dev.(target)	n.a.			n.a.			n.a.		
	R(target)	n.a.			n.a.			n.a.		

Lab 1146 first reported 0 and 41
Lab 1435 first reported 3 and 37


Volume of oil phase (ml)
Kernel Density


Volume of water phase (ml)
Kernel Density

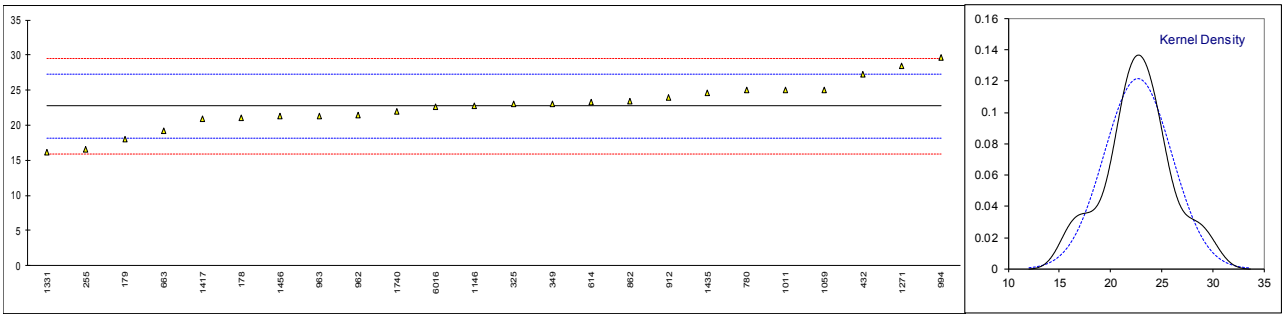

Volume of emulsion phase (ml)
Kernel Density

Determination of Calcium (Ca) on sample #18225; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
173		-----		-----	
178		21		-0.75	
179	D5185	18		-2.07	
237	D5185	<40		-----	
255	INH-OL1	16.57	C	-2.70	first reported 14.9
325	D5185	23		0.13	
349		23		0.13	
432	D4951	27.2		1.98	
496	D5185	-----		-----	
614	D4628	23.3		0.26	
663	D5185	19.232		-1.53	
780	D5185	25		1.01	
862	D5185	23.4		0.31	
912	D5185	24		0.57	
962	D5185	21.5		-0.53	
963	D5185	21.37		-0.59	
994	D5185	29.6		3.04	
1011	D5185	25		1.01	
1059	In house	25		1.01	
1146	In house	22.74		0.02	
1161		-----		-----	
1174		-----		-----	
1271	D5185	28.4		2.51	
1331	GB/T17476	16.15	C	-2.89	First reported 31.95
1417	INH-15	20.9		-0.79	
1435	D5185	24.58		0.83	
1448		-----		-----	
1456	D5185	21.3		-0.62	
1660		-----		-----	
1720		-----		-----	
1740	D5185	22		-0.31	
1748		-----		-----	
1797		-----		-----	
1890		-----		-----	
6016	D5185	22.65		-0.02	
6141		-----		-----	

	normality	OK
	n	24
	outliers	0
	mean (n)	22.704
	st.dev. (n)	3.2894
	R(calc.)	9.210
	st.dev.(Horwitz)	2.2704
	R(Horwitz)	6.357
Compare		
	R(D5185:18)	0.869

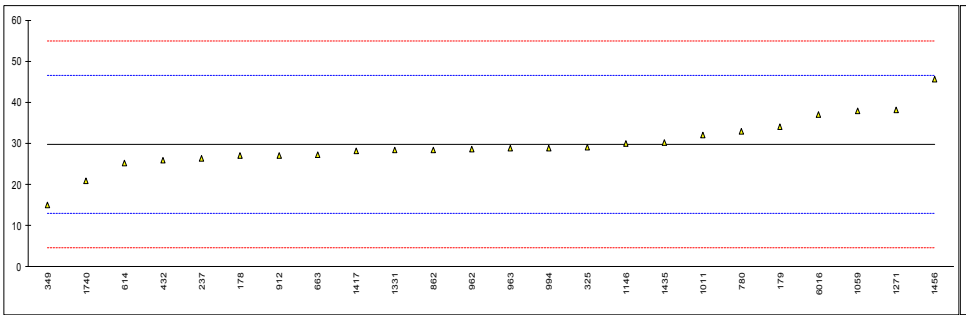
application range D5185:18 = 40 – 9000 mg/kg

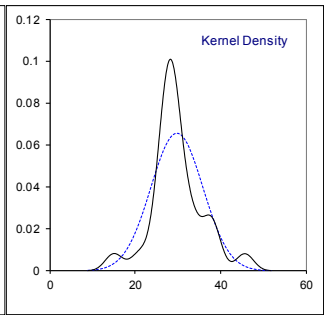


Determination of Phosphorus (P) on sample #18225; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
173		-----		-----	
178		27		-0.32	
179	D5185	34		0.51	
237	D5185	26.227		-0.42	
255	INH-OL1	-----		-----	
325	D5185	29		-0.09	
349		15		-1.76	
432	D4951	25.8		-0.47	
496	D5185	-----		-----	
614	D4628	25.3		-0.53	
663	D5185	27.357		-0.28	
780	D5185	33		0.39	
862	D5185	28.4		-0.16	
912	D5185	27		-0.32	
962	D5185	28.6		-0.13	
963	D5185	28.89		-0.10	
994	D5185	28.9		-0.10	
1011	D5185	32		0.27	
1059	In house	38		0.99	
1146	In house	30.00		0.03	
1161		-----		-----	
1174		-----		-----	
1271	D5185	38.2		1.01	
1331	GB/T17476	28.30		-0.17	
1417	INH-15	28.2		-0.18	
1435	D5185	30.28		0.07	
1448		-----		-----	
1456	D5185	45.6		1.90	
1660		-----		-----	
1720		-----		-----	
1740	D5185	21		-1.04	
1748		-----		-----	
1797		-----		-----	
1890		-----		-----	
6016	D5185	37.10		0.88	
6141		-----		-----	

normality	suspect
n	24
outliers	0
mean (n)	29.715
st.dev. (n)	6.0834
R(calc.)	17.034
st.dev.(D5185:18)	8.3714
R(D5185:18)	23.440





Determination of Zinc (Zn) on sample #18225; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
173		-----		-----	
178		<1		-----	
179	D5185	<1		-----	
237	D5185	0.4503		-----	
255	INH-OL1	-----		-----	
325	D5185	0		-----	
349		0		-----	
432	D4951	<1		-----	
496	D5185	-----		-----	
614	D4628	0.1		-----	
663	D5185	0.093		-----	
780	D5185	<1		-----	
862	D5185	<1		-----	
912	D5185	1		-----	
962	D5185	<1		-----	
963	D5185	0.13		-----	
994	D5185	<60		-----	
1011	D5185	<10		-----	
1059	In house	<3		-----	
1146	In house	1.299		-----	
1161		-----		-----	
1174		-----		-----	
1271	D5185	0.2		-----	
1331	GB/T17476	0.691	C	-----	First reported 29.86
1417	INH-15	<0.1		-----	
1435	D5185	0.1118		-----	
1448		-----		-----	
1456	D5185	0		-----	
1660		-----		-----	
1720		-----		-----	
1740	D5185	<1		-----	
1748		-----		-----	
1797		-----		-----	
1890		-----		-----	
6016	D5185	<1.0		-----	
6141		-----		-----	
	n	24			
	mean (n)	<60			Application range D5185:18 = 60 – 1600 mg/kg

APPENDIX 2

Reported details: Foam determination

lab	Sample used	Diffuser type	Cylinder cleansed	Gas diffuser cleansed	Air tube cleansed	Air flow rate constant
173	---	---	---	---	---	---
178	---	---	---	---	---	---
179	As received	Metal (Stainless Steel)	Yes	---	---	---
237	---	---	---	---	---	---
255	---	---	---	---	---	---
325	As received	Metal (Stainless Steel)	Yes	Yes	Yes	No: adjustment at start
349	---	---	---	---	---	---
432	---	---	---	---	---	---
496	As received	Metal (Stainless Steel)	Yes	Yes	---	Yes: without readjustment
614	As received	Metal (Stainless Steel)	Yes	Yes	No	No: adjustment at start
663	---	---	---	---	---	---
780	---	---	---	---	---	---
862	As received	Metal (Stainless Steel)	Yes	Yes	Yes	Yes: without readjustment
912	---	---	---	---	---	---
962	---	---	---	---	---	---
963	---	---	---	---	---	---
994	---	---	---	---	---	---
1011	---	---	---	---	---	---
1059	As received	Metal (Stainless Steel)	Yes	Yes	Yes	Yes: readjustment needed
1146	As received	Metal (Stainless Steel)	?	?	?	Yes: without readjustment
1161	As received	Metal (Stainless Steel)	Yes	Yes	Yes	?
1174	As received	Stone (Non-Metallic)	Yes	Yes	Yes	Yes: readjustment needed
1271	---	---	---	---	---	---
1331	After agitation, option A	Metal (Stainless Steel)	Yes	Yes	No	Yes: readjustment needed
1417	As received	Metal (Stainless Steel)	Yes	Yes	Yes	Yes: without readjustment
1435	As received	---	Yes	Yes	Yes	Yes: without readjustment
1448	---	---	---	---	---	---
1456	---	---	---	---	---	---
1660	---	---	---	---	---	---
1720	---	---	---	---	---	---
1740	---	---	---	---	---	---
1748	---	---	---	---	---	---
1797	---	---	---	---	---	---
1890	---	---	---	---	---	---
6016	---	---	---	---	---	---
6141	---	---	---	---	---	---

? = I do not know

APPENDIX 3

Number of participants per country

2 labs in	AUSTRALIA
1 lab in	AUSTRIA
1 lab in	AZERBAIJAN
3 labs in	BELGIUM
1 lab in	BOSNIA and HERZEGOVINA
1 lab in	BULGARIA
2 labs in	CHINA, People's Republic
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
1 lab in	NETHERLANDS
1 lab in	NIGERIA
1 lab in	PORTUGAL
1 lab in	ROMANIA
1 lab in	RUSSIAN FEDERATION
3 labs in	SAUDI ARABIA
1 lab in	SLOVENIA
1 lab in	SPAIN
1 lab in	SUDAN
1 lab in	TANZANIA
1 lab in	THAILAND
1 lab in	TURKEY
1 lab in	UNITED KINGDOM
3 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	= possibly an error in calculations
U	= test result possibly reported in a different unit
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:

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2 ASTM E178:16a
3 ASTM E1301:95(2003)
4 ISO 5725:86
5 ISO 5725, parts 1-6, 1994
6 ISO 13528:05
7 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
9 IP 367:84
10 DIN 38402 T41/42
11 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
12 J.N. Miller, Analyst, 118, 455, (1993)
13 Analytical Methods Committee Technical Brief, No 4 January 2001
14 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364 (2002)
15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)
16 Horwitz, R. Albert, J. AOAC Int, 79, 3, 589, (1996)