Results of Proficiency Test
Turbine Oil (fresh)
May 2019

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2013, the Institute for Interlaboratory Studies (iis) organizes a proficiency test (PT) for the analysis on used Turbine Oil every year. During the annual proficiency testing program 2018/2019 it was decided to continue the proficiency test (PT) for the analysis on fresh Turbine Oil.

In this interlaboratory study 22 laboratories in 18 different countries registered for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2019 Turbine Oil (fresh) proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, The Netherlands, was the organizer of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send one bottle of 1L labelled #19081 of fresh Turbine Oil.
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

The necessary bulk material of about 150 liters of fresh Turbine Oil was obtained from a third party. After homogenisation 40 amber glass bottles of 1L were filled and labelled #19081.
The homogeneity of the subsamples #19081 was checked by determination of Density at 15°C in accordance with ASTM D4052.

	Density at 15°C in kg/L
Sample #19081-1	0.86798
Sample #19081-2	0.86797
Sample #19081-3	0.86797
Sample #19081-4	0.86799
Sample #19081-5	0.86800
Sample #19081-6	0.86800
Sample #19081-7	0.86798
Sample #19081-8	0.86798

Table 1: homogeneity test results of Turbine Oil (fresh) subsamples #19081

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

	Density at 15°C in kg/L
r (observed)	0.00003
reference test method	ASTM D4052:18a
0.3 x R (ref. test method)	0.00015

Table 2: evaluation of the repeatability of the subsamples #19081

The calculated repeatability was less than 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneity of the subsamples #19081 was assumed.

To each of the participating laboratories, one sample of 1liter amber glass bottles labelled #19081 was sent on May 01, 2019.

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYSES

The participants were requested to determine on sample #19081; Acid Number (Total), Air Release Time at 50°C, Density at 15°C, Flash Point C.O.C., Foam Characteristics (Foaming Tendency, Foaming Stability), Kinematic Viscosity at 40°C and at 100°C, Viscosity Index, Pour Point (manual and automated, 1°C interval, Sulfur, Water, Water Separability at 54°C, distilled water and Calcium, Phosphorus and Zinc.
Also, some additional questions were asked about the Acid Number and 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 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 reanalyzes). Additional or corrected test results are used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.
Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report ‘iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation’ of June 2018 (iis-protocol, version 3.5).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as ‘<…’ or ‘>…’ were not used in the statistical evaluation.

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

According to ISO5725 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 them with a factor of 2.8.

3.2 GRAPHICS

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

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve 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, EN or ISO reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation of 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 test result tables in 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 interlaboratory study, no problems were encountered with the dispatch of the samples to laboratories. Two participants reported test results after the final reporting date and two other participants did not report any test results at all. Not all laboratories were able to report all analyses requested. In total 20 participants reported 271 test results. Observed were 4 outlying results, which is 1.5% of the numerical 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, see also paragraph 3.1.

4.1 EVALUATION PER TEST

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

In the iis PT reports, ASTM methods are referred to with a number (e.g. D2270) and an added designation for the year that the 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 results tables of Appendix 1 only the method number and year of adoption or revision e.g. D2270:10 will be used.

Acid Number (total): This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D664-A:18e2 for all modes of IP and BEP and volume of titration solvent used.
When the test results for IP and BEP were evaluated separately, the calculated reproducibility of the test results for BEP 60mL are in agreement with the precision data of ASTM D664-A:18e2. The calculated reproducibility of the test results for BEP 125mL and IP 60 and 125mL are not in agreement. ASTM D664 was updated in 2018. One of the major changes is the buffer used for in the end point detection (pH11 is changed into pH10).

Air-release time at 50°C: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D3427:15.

Density at 15°C: 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 D4052:18a.

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

Foaming Characteristics (Tendency and Stability): This determination was very problematic.
In total one statistical outlier was observed.
It was decided not to calculate z-scores at sequence I and III due to the large variation between the reported test results. The Foam Tendency determination for sequence II is in agreement with the requirements of ASTM D892:18.
All reported test results for Foam Stability were zero. Therefore, it was decided not to calculate z-scores.
The determination of the Foaming Characteristics is very sensitive in maintenance and execution. In ASTM D892:18 many tips and tricks are given in the test method part X1. Possible sources for the large variation are the cleaning and checking of the air diffuser, air tubes and test cylinders, the air flow rate used during the blowing period. All reporting participants mentioned to use the sample as received and a metal diffuser.

Kinematic Viscosity at 40°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D445:18.

Kinematic Viscosity at 100°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D445:18.

Viscosity Index: This determination was problematic. However, the viscosity test results were in agreement. No statistical outliers but one calculation error were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D2270:10(2016).

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

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.

Sulfur: 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 D4294:16e1.

Water: 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, distilled water: This determination was not problematic. No statistical outliers were observed over six parameters. The calculated are in good agreement with the requirements of ASTM D1401:18b.

Calcium: All reporting participants agreed on absence of Calcium (<40 mg/kg). Therefore, no z-scores were calculated.

Phosphorus: All reporting participants agreed on absence of Phosphorus (<10 mg/kg). Therefore, no z-scores were calculated.

Zinc: All reporting participants agreed on absence of Zinc (<60 mg/kg). 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 and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average result, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from literature reference test methods (e.g. ASTM, EN and ISO test methods) are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R (lit)
Acid Number (Total)	mg KOH/g	18	0.05	0.05	0.04
Air-release time at 50°C	minutes	9	2.7	2.7	2.1
Density at 15°C	kg/L	18	0.8679	0.0003	0.0005
Flash Point C.O.C.	°C	13	231.6	14.9	18
Foam Tendency Seq. I	mL	9	222.2	289.2	(80.4)
Foam Tendency Seq. II	mL	9	15.6	14.8	15.5
Foam Tendency Seq. III	mL	10	181.0	239.2	(79.6)
Foam Stability Seq. I	mL	10	0	n.a.	n.a.
Foam Stability Seq. II	mL	10	0	n.a.	n.a.
Foam Stability Seq. III	mL	10	0	n.a.	n.a.
Kinematic Viscosity at 40°C	mm^2/s	20	45.984	0.345	0.561
Kinematic Viscosity at 100°C	mm^2/s	19	6.818	0.075	0.094
Viscosity Index		18	102.4	3.4	2
Pour Point manual	°C	12	-11.3	5.2	9
Pour Point automated, 1°C interval	°C	6	-11.2	3.7	4.5
Sulfur	mg/kg	8	30.0	16.4	17.2
Water	mg/kg	15	29.7	22.7	129.1
Water Separability at 54°C, distilled water					
- Time ≤ 3 ml emulsion	minutes	9	11.9	14.5	20
- Time 37 ml water	minutes	6	11.0	14.9	20
- Time to complete break	minutes	8	12.2	12.6	20
- Volume Oil phase	mL	9	41.0	4.4	n.a.
- Volume Water phase	mL	9	38.7	4.6	n.a.
- Volume Emulsion phase	mL	9	0.3	2.8	n.a.
Calcium as Ca	mg/kg	16	<40	n.a.	n.a.
Phosphorus as P	mg/kg	16	<10	n.a.	n.a.
Zinc as Zn	mg/kg	16	<60	n.a.	n.a.

Table 3: reproducibilities of tests on sample #19081

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

4.3 OVERVIEW OF THE PROFICIENCY TEST OF MAY 2019

	May 2019	May 2018
Number of reporting labs	20	19
Number of results reported	271	281
Number of statistical outliers	4	12
Percentage outliers	1.5%	4.3%

Table 4: comparison with previous proficiency tests

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

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

Parameter	May 2019	May 2018
Acid Number (Total)	-	++
Air-release time at 50°C	-	++
Density at 15°C	+	-
Flash Point C.O.C.	+	++
Foam Tendency Seq. I	(--)	(--)
Foam Tendency Seq. II	+/-	+
Foam Tendency Seq. III	(--)	(--)
Kinematic Viscosity at 40°C	+	+
Kinematic Viscosity at 100°C	+	+
Viscosity Index	-	-
Pour Point manual	+	++
Pour Point automated, 1°C interval	+	+
Sulfur	+/-	+
Water	++	++
Water Separability 54°C, dist. water	+	++
Calcium as Ca	n.e.	n.e.
Phosphorus as P	n.e.	++
Zinc as Zn	n.e.	n.e.

Table 5: comparison determinations against the reference test methods

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

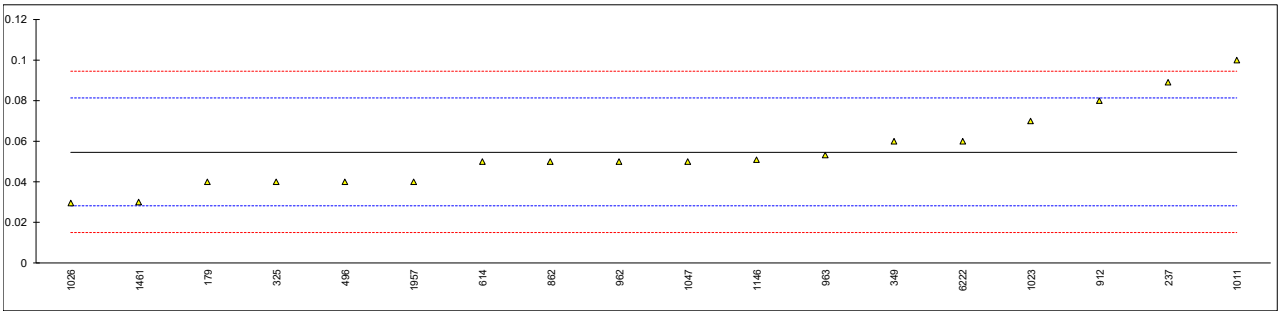
++:	group performed much better than the reference test method
+ :	group performed better than the reference test method
+/-:	group performance equals the reference test method
- :	group performed worse than the reference test method
-- :	group performed much worse than the reference test method
n.e.: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

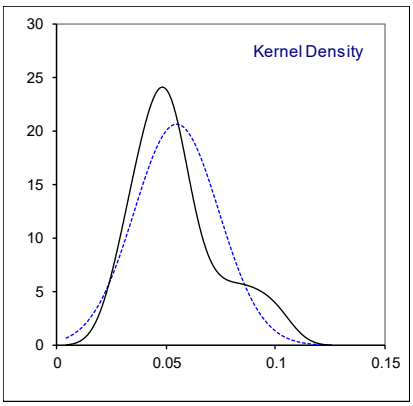
APPENDIX 1

Determination of Acid Number (Total) on sample #19081; results in mg KOH/g

lab	method	value	mark	z(targ)	Determination of end point	Volume of titration solvent
178	INH-1118	<0.10		-----	Inflection Point	---
179	D664-A	0.04		-1.10	Inflection Point	125 mL
237	D664-A	0.089		2.60	Inflection Point	125 mL
325	D664-A	0.04		-1.10	Buffer End Point (pH 10)	125 mL
349	D664-A	0.06		0.41	Inflection Point	125 mL
432		-----		-----	---	---
496	D664-A	0.04		-1.10	Buffer End Point (pH 11)	60 mL
614	D664-A	0.05		-0.35	---	60 mL
862	D664-A	0.05		-0.35	Inflection Point	60 mL
912	D974	0.08		1.92	---	---
962	D974	0.05		-0.35	---	---
963	D664-A	0.053		-0.12	Inflection Point	60 mL
1011	D664-A	0.10		3.43	---	---
1023	D8045Mod.	0.07		1.16	---	---
1026	D664-A	0.0295		-1.89	Buffer End Point (pH 11)	60 mL
1047	ISO6618	0.05		-0.35	Inflection Point	60 mL
1146	D664-A	0.051		-0.27	Buffer End Point (pH 11)	125 mL
1461	ISO6618	0.03		-1.86	---	---
1957	D664-A	0.04		-1.10	Buffer End Point (pH 10)	125 mL
6016		-----		-----	---	---
6222	D664-A	0.06		0.41	Inflection Point	60 mL
6253		-----		-----	---	---

		BEP (pH 10 and 11) only	Inflection point only
normality	suspect	not OK	not OK
n	18	5	7
outliers	0	0	0
mean (n)	0.0546	0.0401	0.0574
st.dev. (n)	0.01936	0.00760	0.01551
R(calc.)	0.0542	0.0213	0.0434
st.dev.(D664-A:18e2 IP 60ml)	0.01324	---	---
R(D664-A:18e2 IP 60ml)	0.0371	---	0.0386
Compare			
D664-A:18e2 BEP 60ml	0.0325	0.0240	---
D664-A:18e2 IP 125ml	0.0105	---	0.0157
D664-A:18e2 BEP 125ml	0.0149	0.0107	---

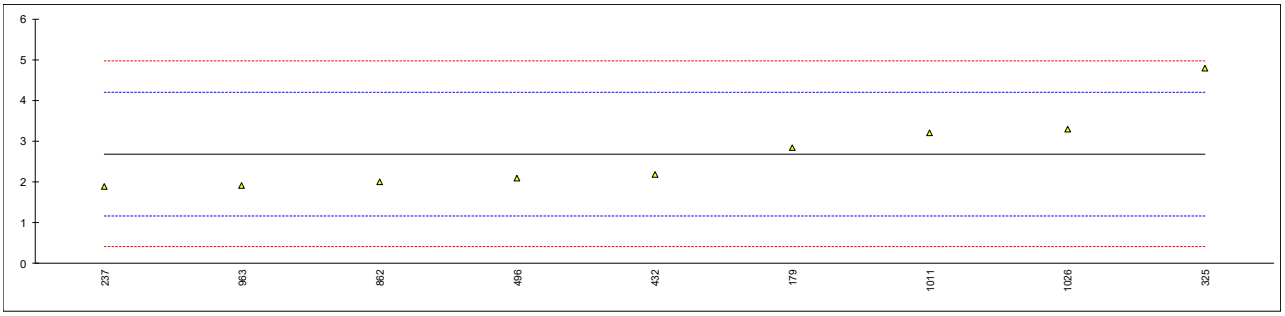


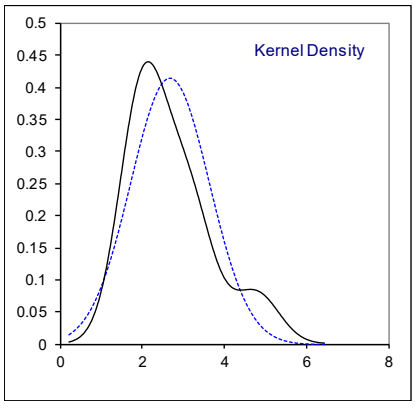


Determination of Air-release time at 50°C on sample #19081; results in minutes

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D3427	2.83	C	0.19	first reported: 7.2
237	D3427	1.89		-1.05	
325	D3427	4.7833		2.76	
349		-----		-----	
432	ISO9120	2.17		-0.68	
496	D3427	2.1		-0.77	
614		-----		-----	
862	D3427	2.0		-0.90	
912		-----		-----	
962		-----		-----	
963	D3427	1.9		-1.03	
1011	IP313	3.2		0.68	
1023		-----		-----	
1026	D3427	3.3		0.81	
1047		-----		-----	
1146		-----		-----	
1461		-----		-----	
1957		-----		-----	
6016		-----		-----	
6222		-----		-----	
6253		-----		-----	

normality	not OK
n	9
outliers	0
mean (n)	2.6859
st.dev. (n)	0.9613
R(calc.)	2.692
st.dev.(D3427:15)	0.7609
R(D3427:15)	2.131

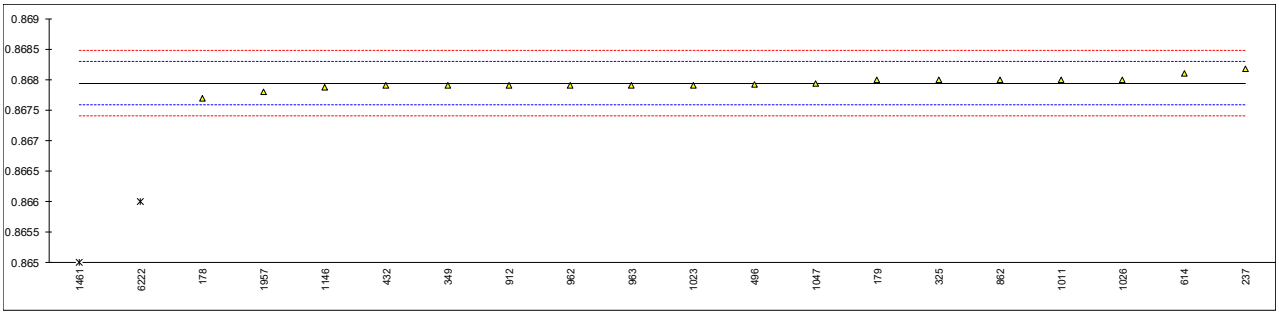


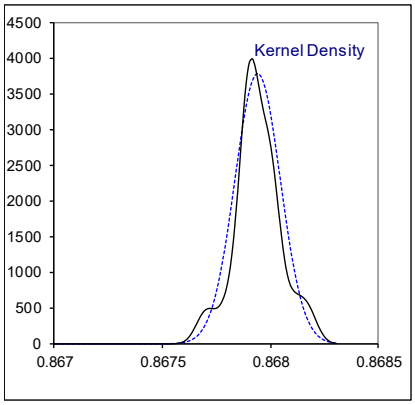


Determination of Density at 15°C on sample #19081; results in kg/L

lab	method	value	mark	z(targ)	remarks
178	D4052	0.8677		-1.34	
179	D4052	0.8680		0.34	
237	D4052	0.86817		1.29	
325	D4052	0.8680		0.34	
349	D4052	0.8679		-0.22	
432	D4052	0.86790		-0.22	
496	D4052	0.86792		-0.11	
614	D4052	0.8681		0.90	
862	D4052	0.8680		0.34	
912	D4052	0.8679		-0.22	
962	D4052	0.8679		-0.22	
963	D4052	0.8679		-0.22	
1011	D4052	0.8680		0.34	
1023	D4052	0.8679	C	-0.22	reported: 0.8679 kg/m^3
1026	D4052	0.8680		0.34	
1047	ISO12185	0.86794		0.01	
1146	D4052	0.86787		-0.39	
1461	ISO3675	0.8650	R(0.01)	-16.46	
1957	D4052	0.8678		-0.78	
6016		-----		-----	
6222	D7042	0.866	R(0.01)	-10.86	
6253		-----		-----	

normality	suspect
n	18
outliers	2
mean (n)	0.86794
st.dev. (n)	0.000105
R(calc.)	0.00029
st.dev.(D4052:18a)	0.000179
R(D4052:18a)	0.00050

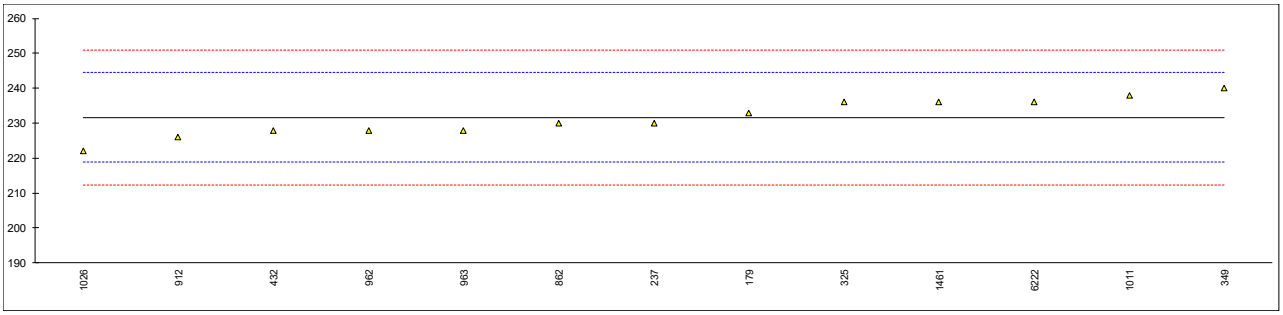


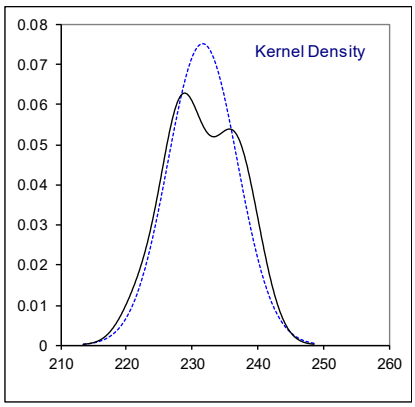


Determination of Flash Point C.O.C. on sample #19081; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D92	233		0.22	
237	D92	230		-0.25	
325	D92	236		0.68	
349	D92	240		1.30	
432	D92	228.0		-0.56	
496		-----		-----	
614		-----		-----	
862	D92	230		-0.25	
912	D92	226		-0.87	
962	D92	228		-0.56	
963	D92	228		-0.56	
1011	D92	238		0.99	
1023		-----		-----	
1026	D92	222		-1.50	
1047		-----		-----	
1146		-----		-----	
1461	ISO2592	236		0.68	
1957		-----	W	-----	test result withdrawn. first reported: 270
6016		-----		-----	
6222	ISO2592	236		0.68	
6253		-----		-----	

normality	OK
n	13
outliers	0
mean (n)	231.62
st.dev. (n)	5.316
R(calc.)	14.88
st.dev.(D92:18)	6.429
R(D92:18)	18

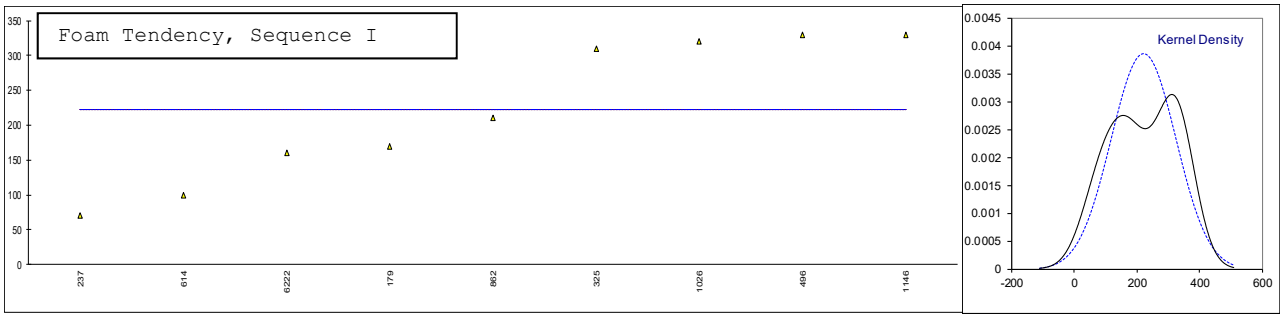


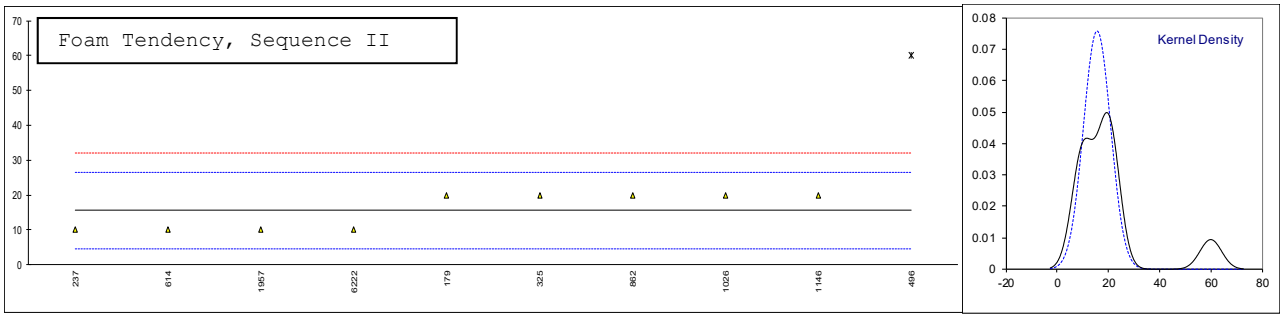


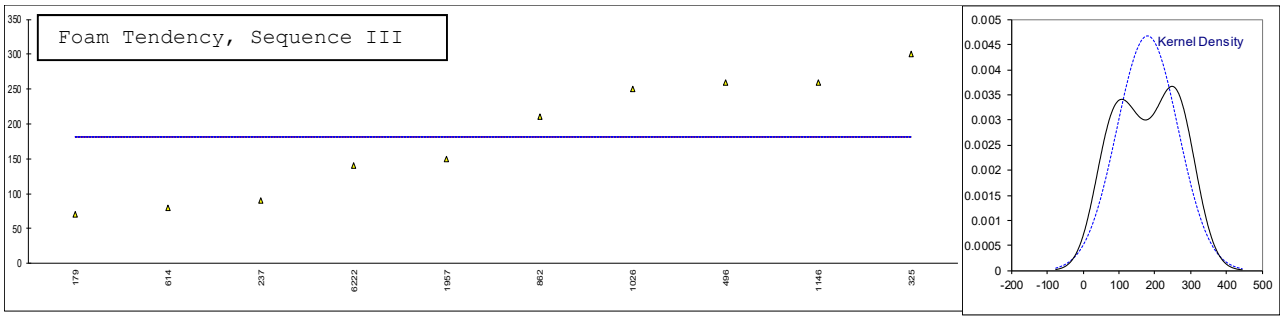
Determination of Foaming Tendency, Sequence I, II and III (5 min. blowing period) on sample #19081; results in mL

lab	method	Sample used	Diffuser	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
178		---	---	-----		-----	-----		-----	-----		-----
179	D892	As received	Metal	170		-----	20		0.80	70		-----
237	D892	As received	Metal	70	C	-----	10		-1.00	90		-----
325	D892	As received	Metal	310		-----	20		0.80	300		-----
349		---	---	-----		-----	-----		-----	-----		-----
432		---	---	-----		-----	-----		-----	-----		-----
496	D892	As received	Metal	330		-----	60	G(1)	8.04	260		-----
614	D892	As received	Metal	100		-----	10		-1.00	80		-----
862	D892	As received	Metal	210		-----	20		0.80	210		-----
912		---	---	-----		-----	-----		-----	-----		-----
962		---	---	-----		-----	-----		-----	-----		-----
963		---	---	-----		-----	-----		-----	-----		-----
1011		---	---	-----		-----	-----		-----	-----		-----
1023		---	---	-----		-----	-----		-----	-----		-----
1026	D892	As received	Metal	320		-----	20		0.80	250		-----
1047		---	---	-----		-----	-----		-----	-----		-----
1146	ISO6247	As received	Metal	330		-----	20		0.80	260		-----
1461		---	---	-----		-----	-----		-----	-----		-----
1957	D892	As received	Metal	-----	W	-----	10		-1.00	150		-----
6016		---	---	-----		-----	-----		-----	-----		-----
6222	ISO6247	As received	Metal	160		-----	10		-1.00	140		-----
6253		---	---	-----		-----	-----		-----	-----		-----
		normality		OK			OK			OK		
		n		9			9			10		
		outliers		0			1			0		
		mean (n)		222.22			15.56			181.00		
		st.dev. (n)		103.2930			5.2705			85.4335		
		R(calc.)		289.22			14.76			239.21		
		st.dev.(D892:18)		(28.7123)			5.5302			(28.4429)		
		R(D892:18)		(80.39)			15.48			(79.64)		

Lab 237 first reported: 50
Lab 1957 test result withdrawn. First reported: 20







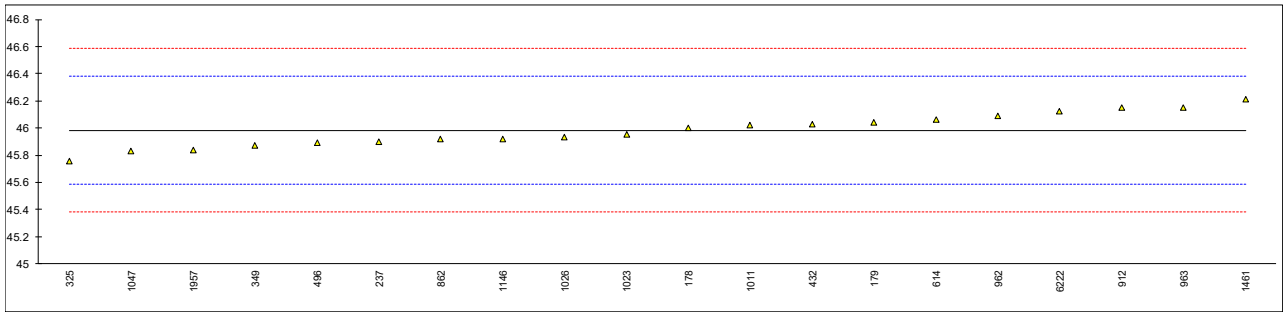
Determination of Foaming Stability, Sequence I, II and III (10 min. settling period) on sample #19081; results in mL

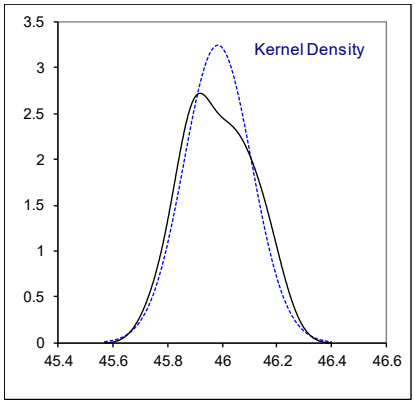
lab	method	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179	D892	0		-----	0		-----	0		-----
237	D892	0		-----	0		-----	0		-----
325	D892	0		-----	0		-----	0		-----
349		-----		-----	-----		-----	-----		-----
432		-----		-----	-----		-----	-----		-----
496	D892	0		-----	0		-----	0		-----
614	D892	0		-----	0		-----	0		-----
862	D892	0		-----	0		-----	0		-----
912		-----		-----	-----		-----	-----		-----
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
1011		-----		-----	-----		-----	-----		-----
1023		-----		-----	-----		-----	-----		-----
1026	D892	0		-----	0		-----	0		-----
1047		-----		-----	-----		-----	-----		-----
1146	ISO6247	0		-----	0		-----	0		-----
1461		-----		-----	-----		-----	-----		-----
1957	D892	0		-----	0		-----	0		-----
6016		-----		-----	-----		-----	-----		-----
6222	ISO6247	0		-----	0		-----	0		-----
6253		-----		-----	-----		-----	-----		-----
n		10			10			10		
mean (n)		0			0			0		

Determination of Kinematic Viscosity at 40°C on sample #19081; results in mm²/s

lab	method	value	mark	z(targ)	remarks
178	D445	46.0		0.08	
179	D445	46.04		0.28	
237	D445	45.8966		-0.44	
325	D445	45.755		-1.14	
349	D445	45.87		-0.57	
432	D445	46.03		0.23	
496	D445	45.892		-0.46	
614	D445	46.06		0.38	
862	D445	45.9183		-0.33	
912	D445	46.15		0.83	
962	D445	46.09		0.53	
963	D445	46.15		0.83	
1011	D7042	46.02		0.18	
1023	D445	45.95		-0.17	
1026	D445	45.93		-0.27	
1047	ISO3104	45.83		-0.77	
1146	D445	45.922		-0.31	
1461	ISO3104	46.2142		1.15	
1957	D7042	45.84		-0.72	
6016		-----		-----	
6222	D7042	46.12		0.68	
6253		-----		-----	

normality	OK
n	20
outliers	0
mean (n)	45.9839
st.dev. (n)	0.12325
R(calc.)	0.3451
st.dev.(D445:18)	0.20036
R(D445:18)	0.5610

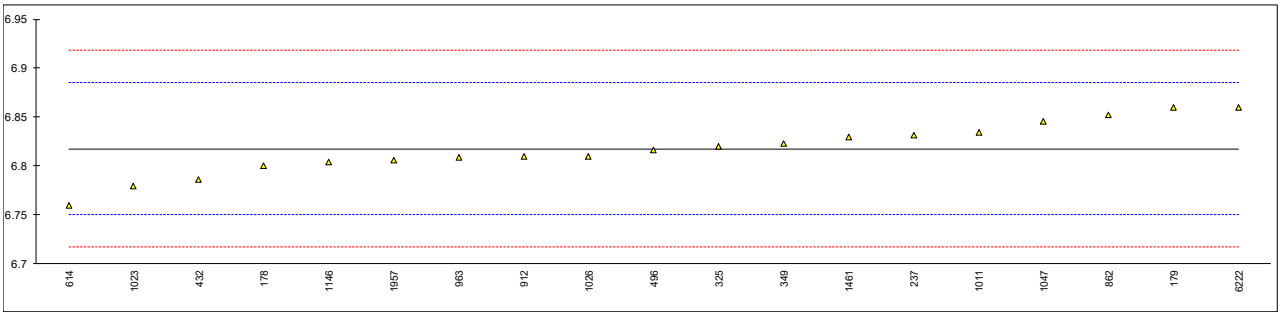


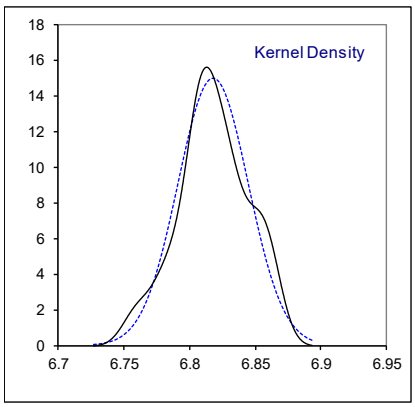


Determination of Kinematic Viscosity at 100°C on sample #19081; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178	D445	6.80	C	-0.53	first reported: 7.05
179	D445	6.86		1.26	
237	D445	6.831189		0.40	
325	D445	6.820		0.07	
349	D445	6.823		0.16	
432	D445	6.786		-0.95	
496	D445	6.8162		-0.05	
614	D445	6.76	C	-1.72	first reported: 6.7
862	D445	6.8526		1.04	
912	D445	6.810		-0.23	
962		-----		-----	
963	D445	6.809		-0.26	
1011	D7042	6.834		0.48	
1023	D445	6.7798		-1.13	
1026	D445	6.81		-0.23	
1047	ISO3104	6.846		0.84	
1146	D445	6.8045		-0.39	
1461	ISO3104	6.8293		0.34	
1957	D7042	6.806		-0.35	
6016		-----		-----	
6222	D7042	6.86		1.26	
6253		-----		-----	

normality	OK
n	19
outliers	0
mean (n)	6.8178
st.dev. (n)	0.02667
R(calc.)	0.0747
st.dev.(D445:18)	0.03360
R(D445:18)	0.0941

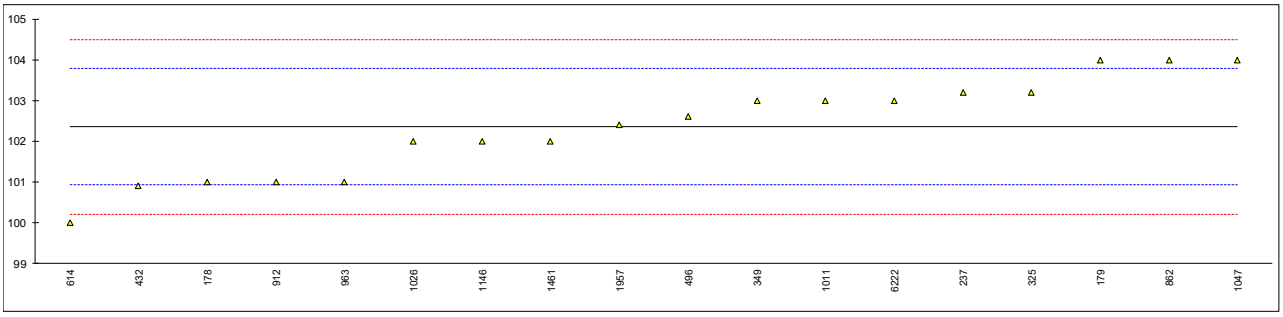


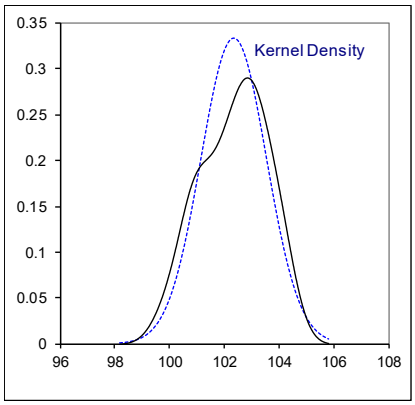


Determination of Viscosity Index on sample #19081

lab	method	value	mark	z(targ)	remarks
178	D2270	101	C	-1.89	first reported: 111
179	D2270	104		2.31	
237	D2270	103.189		1.18	
325	D2270	103.2		1.19	
349	D2270	103		0.91	
432	D2270	100.9		-2.03	
496	D2270	102.6		0.35	
614	D2270	100	C	-3.29	first reported: 97
862	D2270	104		2.31	
912	D2270	101		-1.89	
962		-----		-----	
963	D2270	101		-1.89	
1011	D2270	103		0.91	
1023		-----		-----	
1026	D2270	102		-0.49	
1047	ISO2909	104		2.31	
1146	D2270	102		-0.49	
1461	ISO2909	102		-0.49	
1957	D2270	102.4		0.07	
6016		-----		-----	
6222	D2270	103		0.91	
6253		-----		-----	

normality	OK
n	18
outliers	0
mean (n)	102.35
st.dev. (n)	1.198
R(calc.)	3.35
st.dev.(D2270:10)	0.714
R(D2270:10)	2

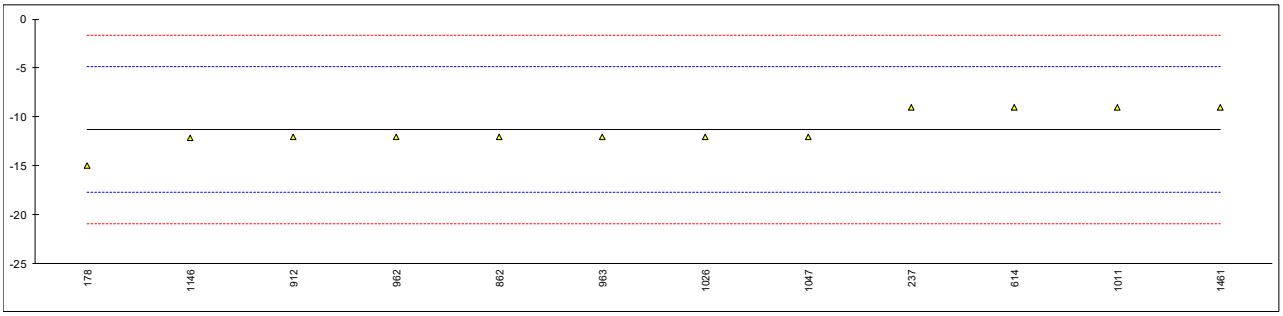


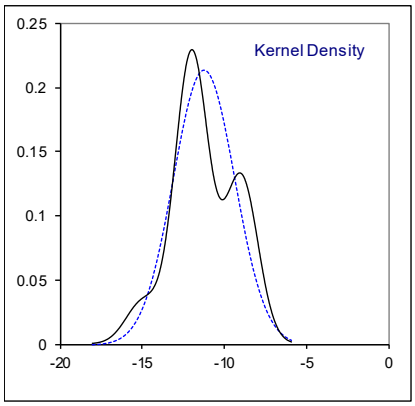


Determination of Pour Point manual on sample #19081; results in °C

lab	method	value	mark	z(targ)	remarks
178	D97	-15		-1.16	
179		-----		-----	
237	D97	-9		0.70	
325		-----		-----	
349		-----		-----	
432		-----		-----	
496		-----		-----	
614	D97	-9		0.70	
862	D97	-12		-0.23	
912	D97	-12		-0.23	
962	D97	-12		-0.23	
963	D97	-12		-0.23	
1011	D97	-9		0.70	
1023		-----		-----	
1026	D97	-12		-0.23	
1047	ISO3016	-12		-0.23	
1146	D97	-12.1		-0.26	
1461	ISO3016	-9		0.70	
1957		-----		-----	
6016		-----		-----	
6222		-----		-----	
6253		-----		-----	

normality	OK
n	12
outliers	0
mean (n)	-11.26
st.dev. (n)	1.869
R(calc.)	5.23
st.dev.(D97:17b)	3.214
R(D97:17b)	9

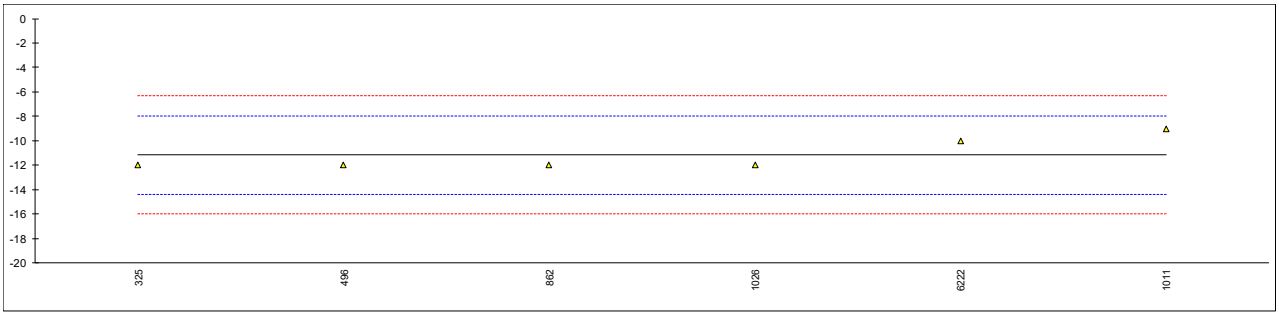




Determination of Pour Point automated 1°C interval on sample #19081; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
325	D5950	-12		-0.52	
349		-----		-----	
432		-----		-----	
496	D5950	-12		-0.52	
614		-----		-----	
862	D5950	-12		-0.52	
912		-----		-----	
962		-----		-----	
963		-----		-----	
1011	D6892	-9		1.35	
1023		-----		-----	
1026	D5950	-12		-0.52	
1047		-----		-----	
1146		-----		-----	
1461		-----		-----	
1957		-----		-----	
6016		-----		-----	
6222	ISO3016	-10		0.73	
6253		-----		-----	

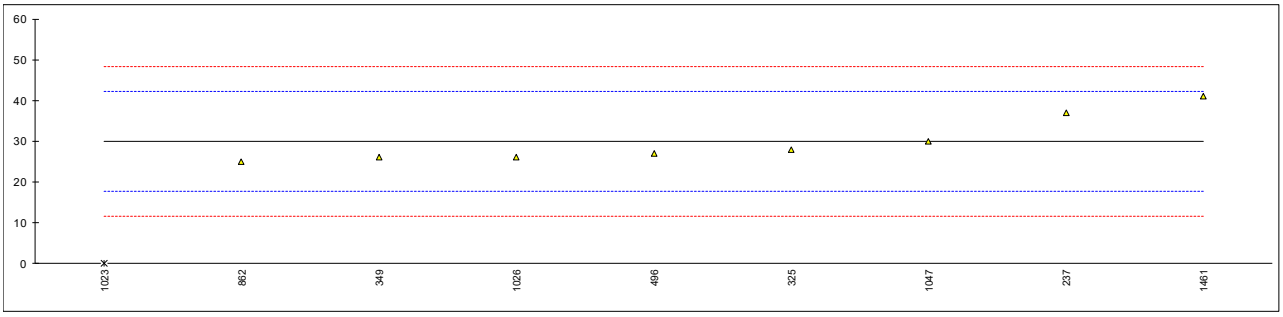
normality	unknown
n	6
outliers	0
mean (n)	-11.17
st.dev. (n)	1.329
R(calc.)	3.72
st.dev.(D5950:14)	1.607
R(D5950:14)	4.5

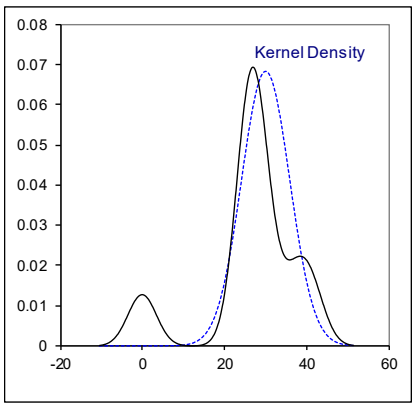


Determination of Sulfur on sample #19081; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D4294	37		1.14	
325	INH-4927	28		-0.33	
349	D2622	26		-0.65	
432		-----		-----	
496	D2622	27.1		-0.47	
614		-----		-----	
862	D2622	25		-0.82	
912	D4294	<17		-----	
962		-----		-----	
963		-----		-----	
1011	D6481	<200		-----	
1023	ISO14596	0.0	D(0.01)	-4.89	
1026	ISO20884	26.0	C	-0.65	first reported: 6.7
1047	ISO8754	30		0.00	
1146		-----		-----	
1461	ISO8754	41	C	1.79	Reported: 0.0041 mg/kg
1957		-----		-----	
6016		-----		-----	
6222	ISO11885	<10		<-3.26	
6253		-----		-----	

normality	suspect
n	8
outliers	1
mean (n)	30.01
st.dev. (n)	5.848
R(calc.)	16.37
st.dev.(D4294:16e1)	6.138
R(D4294:16e1)	17.19

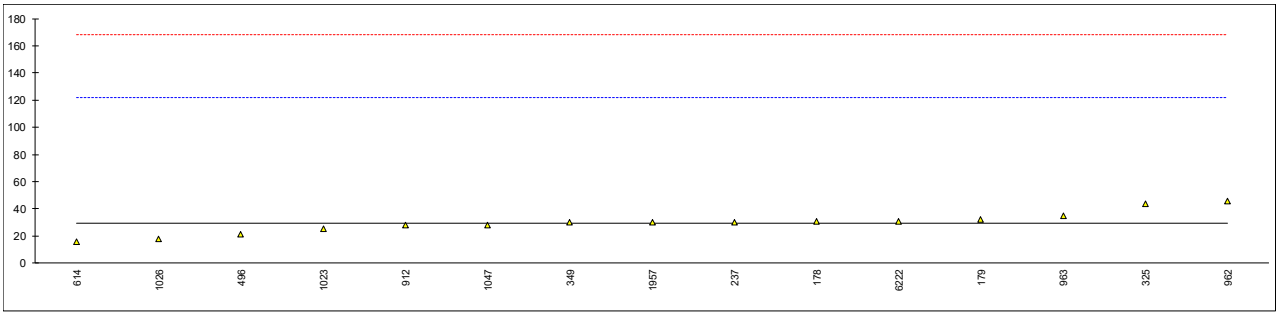


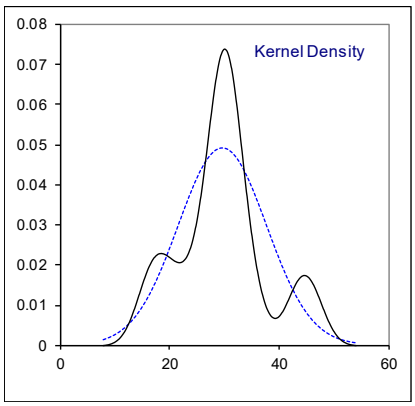


Determination of Water on sample #19081; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D6304-C	31		0.03	
179	D6304-C	32		0.05	
237	D6304-C	30.1		0.01	
325	D6304-C	43.5		0.30	
349	D6304-A	30		0.01	
432		-----		-----	
496	D6304-C	21		-0.19	
614	D6304-C	16		-0.30	
862		-----		-----	
912	D6304-C	28		-0.04	
962	D6304-A	46		0.35	
963	D6304-A	35		0.12	
1011		-----		-----	
1023	D6304-A	25		-0.10	
1026	D6304-C	18		-0.25	
1047	ISO12937	28.2		-0.03	
1146	D6304-C	<100		-----	
1461		-----		-----	
1957	D6304-A	30.0		0.01	
6016		-----		-----	
6222	ISO12937	31		0.03	
6253		-----		-----	

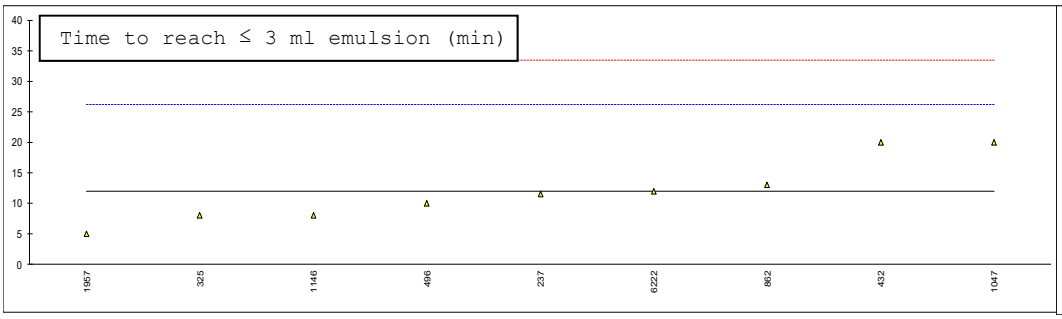
normality	OK
n	15
outliers	0
mean (n)	29.653
st.dev. (n)	8.1095
R(calc.)	22.707
st.dev.(D6304:16e1)	46.1062
R(D6304:16e1)	129.097

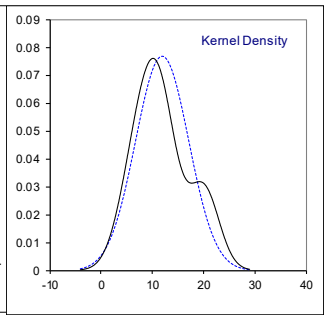


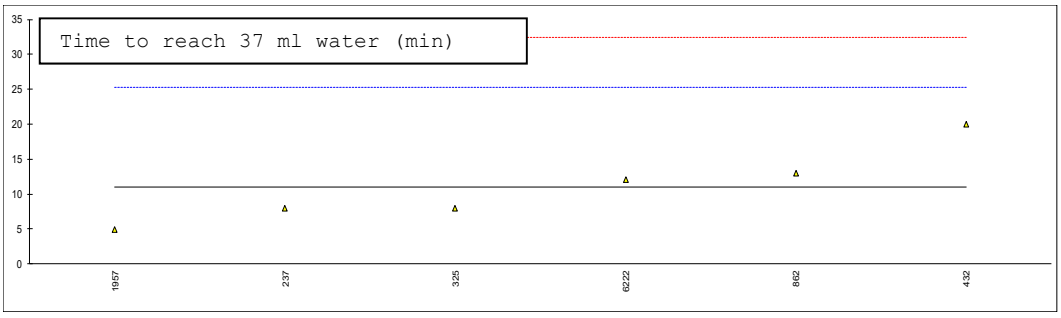


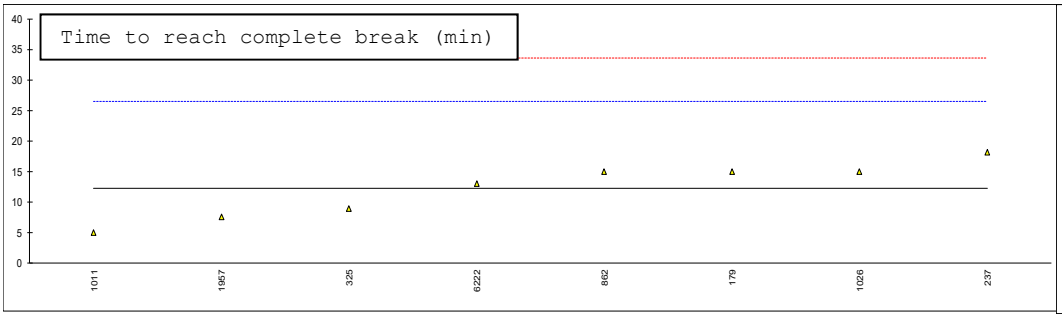
Determination of Water Separability at 54°C, distilled water on sample #19081; results in minutes

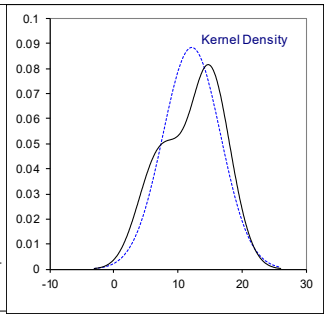
lab	method	time to reach 3 mL or less emulsion	mark	z(targ)	time to reach 37 mL of water	mark	z(targ)	time to reach complete break (40-40-0)	mark	z(targ)	test aborted	time test aborted
178		-----		-----	-----		-----	-----		-----		-----
179	D1401	-----		-----	-----		-----	15		0.39	NO	-----
237		11.5		-0.06	8.0		-0.42	18.1		0.82	NO	-----
325	D1401	8		-0.55	8		-0.42	9		-0.45	NO	-----
349		-----		-----	-----		-----	-----		-----		-----
432	D1401	20		1.13	20		1.26	>30		-----	YES	30
496	D1401	10		-0.27	-----		-----	-----		-----	NO	-----
614		-----		-----	-----		-----	-----		-----		-----
862	D1401	13		0.15	13		0.28	15		0.39	NO	15
912		-----		-----	-----		-----	-----		-----		-----
962		-----		-----	-----		-----	-----		-----		-----
963		-----		-----	-----		-----	-----		-----		-----
1011	D1401	-----		-----	-----		-----	5		-1.01		-----
1023		-----		-----	-----		-----	-----		-----		-----
1026		-----		-----	-----		-----	15		0.39	NO	-----
1047	ISO6614	20		1.13	-----		-----	-----		-----	YES	20
1146	D1401	8		-0.55	-----		-----	-----		-----		-----
1461		-----		-----	-----		-----	-----		-----		-----
1957	D1401	4.98		-0.97	4.88		-0.85	7.63		-0.64	NO	-----
6016		-----		-----	-----		-----	-----		-----		-----
6222	D1401	12		0.01	12		0.14	13		0.11	NO	-----
6253		-----		-----	-----		-----	-----		-----		-----
normality		OK			unknown			unknown				
n		9			6			8				
outliers		0			0			0				
mean (n)		11.94			10.98			12.22				
st.dev. (n)		5.178			5.319			4.503				
R(calc.)		14.50			14.89			12.61				
st.dev.(D1401:18b)		7.143			7.143			7.143				
R(D1401:18b)		20			20			20				











Determination of Water Separability at 54°C, distilled water on sample #19081; results in mL

lab	method	oil phase	mark	z(targ)	water phase	mark	z(targ)	emulsion phase	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179	D1401	40		-----	40		-----	0		-----
237		40		-----	40		-----	0		-----
325		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
432	D1401	42		-----	38		-----	0		-----
496	D1401	44		-----	36		-----	0		-----
614		-----		-----	-----		-----	-----		-----
862	D1401	40		-----	40		-----	0		-----
912		-----		-----	-----		-----	-----		-----
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
1011		-----		-----	-----		-----	-----		-----
1023		-----		-----	-----		-----	-----		-----
1026		40		-----	40		-----	0		-----
1047	ISO6614	43		-----	37		-----	0		-----
1146	D1401	40		-----	37		-----	3		-----
1461		-----		-----	-----		-----	-----		-----
1957	D1401	40		-----	40		-----	0		-----
6016		-----		-----	-----		-----	-----		-----
6222		-----		-----	-----		-----	-----		-----
6253		-----		-----	-----		-----	-----		-----

Determination of Calcium as Ca on sample #19081; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	1		-----	
179	D5185	<1		-----	
237		-----		-----	
325	D5185	0		-----	
349		1		-----	
432		-----		-----	
496	D5185	0.313		-----	
614	D5185	0.0		-----	
862	D5185	<1		-----	
912	D5185	1		-----	
962	D5185	<1		-----	
963	D5185	0.14		-----	
1011	D5185	<10		-----	
1023		-----		-----	
1026	D5185	2		-----	
1047	D5185	<5,0		-----	
1146	In house	0.1054		-----	
1461		-----		-----	
1957	D5185	2		-----	
6016		-----		-----	
6222	ISO11885	<5		-----	
6253		-----		-----	
	n	16			
	mean (n)	<40			Application range D5185:18: 40 – 9000 mg/kg

Determination of Phosphorus as P on sample #19081; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	1		-----	
179	D5185	<1		-----	
237		-----		-----	
325	D5185	0		-----	
349		0		-----	
432		-----		-----	
496	D5185	0		-----	
614	D5185	0.5		-----	
862	D5185	<1		-----	
912	D5185	<1		-----	
962	D5185	1		-----	
963	D5185	1.47		-----	
1011	D5185	<10		-----	
1023		-----		-----	
1026	D5185	0		-----	
1047	D5185	<10,0		-----	
1146	In house	0.4533		-----	
1461		-----		-----	
1957	D5185	0		-----	
6016		-----		-----	
6222	ISO11885	<10		-----	
6253		-----		-----	
	n	16			
	mean (n)	<10			Application range D5185:18: 10 – 1000 mg/kg

Determination of Zinc as Zn on sample #19081; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	1		-----	
179	D5185	<1		-----	
237		-----		-----	
325	D5185	0		-----	
349		1		-----	
432		-----		-----	
496	D5185	0.341		-----	
614	D5185	0.45		-----	
862	D5185	<1		-----	
912	D5185	<1		-----	
962	D5185	<1		-----	
963	D5185	0.34		-----	
1011	D5185	<10		-----	
1023		-----		-----	
1026	D5185	1		-----	
1047	D5185	<5,0		-----	
1146	In house	0.0644		-----	
1461		-----		-----	
1957	D5185	0		-----	
6016		-----		-----	
6222	ISO11885	5		-----	
6253		-----		-----	
	n	16			
	mean (n)	<60			Application range D5185:18: 60 – 1600 mg/kg

APPENDIX 2

Number of participants per country

1 lab in AUSTRALIA
2 labs in AUSTRIA
1 lab in BELGIUM
1 lab in BULGARIA
1 lab in CHINA, People's Republic
1 lab in GERMANY
1 lab in INDIA
1 lab in KAZAKHSTAN
1 lab in MALAYSIA
1 lab in MOROCCO
2 labs in NETHERLANDS
1 lab in NIGERIA
1 lab in NORWAY
1 lab in POLAND
1 lab in PORTUGAL
2 labs in SAUDI ARABIA
1 lab in SPAIN
2 labs in UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations:

C	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= 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
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
SDS	= Safety Data Sheet

Literature:

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