Results of Proficiency Test
Turbine Oil (fresh)
May 2018

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 2017/2018 it was decided to organize a new proficiency test (PT) for the analyse on fresh Turbine Oil also. In this interlaboratory study 19 laboratories in 13 different countries registered for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2018 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/IEC 17025 accredited laboratory. It was decided to send one bottle of 1L (labelled #18076) 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/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 organization of these proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organization, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4). 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 a fresh Turbine Oil was obtained from a third party.
The approximately 150 litre of the bulk material was homogenized in a precleaned drum. After homogenisation, 38 amber glass bottles were filled and labelled #18076. The homogeneity of the subsamples #18076 was checked by determination of Density at 15°C in accordance with ASTM D4052.

	Density at 15°C in kg/L
Sample #18076-1	0.86281
Sample #18076-2	0.86281
Sample #18076-3	0.86281
Sample #18076-4	0.86281
Sample #18076-5	0.86282
Sample #18076-6	0.86281
Sample #18076-7	0.86282
Sample #18076-8	0.86281

Table 1: homogeneity test results of Turbine Oil (fresh) subsamples #18076

From the above test results, the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibilities in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Density at 15°C in kg/L
r (observed)	0.00001
reference test method	ASTM D4052:18
0.3 x R(reference test method)	0.00015

Table 2: evaluation of the repeatabilities of the subsamples #18076

The calculated repeatability was less than 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneity of the subsamples #18076 was assumed.

To each of the participating laboratories, one sample of 1 L amber glass bottle (labelled #18076) was sent on April 25, 2018.

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 #18076; 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 int.), Sulphur, Water content (by KF), Water Separability at 54°C 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 participants 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 reanalyses). 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 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 March 2017 (iis-protocol, version 3.4).
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 them with a factor of 2.8.

3.2 GRAPHICS

In order to visualise 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. 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 targets 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 interlaboratory study, no problems were encountered with the dispatch of the samples to laboratories. All participating laboratories reported test results, but not all laboratories were able to report all analyses requested. In total 19 participants reported 281 test results. Observed were 12 outlying results, which is 4.3% 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 not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D664-A:17a (Inflection Point and BEP).
When the test results determined with Inflection Point and Buffer End Point were evaluated separately no clear effect was observed.

Air-release time at 50°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 D3427:15.

Density at 15°C: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D4052:18.

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 problematic. In total two statistical outliers were observed. The Foam Tendency determination for sequence II is in agreement with the requirements of ASTM D892:18. It was decided not to calculate z-scores at sequence I and III due to the large variation between the reported test results.
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. From the extra information reported (see appendix 1) no clear conclusions could be drawn.

Kinematic Viscosity at 40°C: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D445:17a.

Kinematic Viscosity at 100°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 D445:17a.

Viscosity Index This determination was problematic. One statistical outlier was observed and two other test results were excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of ASTM D2270:10(2016).

Pour Point (manual): 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 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. 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 content: 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. In total two statistical outliers were observed over six parameters. However, the calculated reproducibilities after rejection of the statistical outliers are in good agreement with the requirements of ASTM D1401:18a.

Calcium: All reporting participants, except one agreed on absence of Calcium (<1 mg/kg). Therefore, no significant conclusions were drawn.

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 participants, except one agreed on absence of Zinc (<1 mg/kg). Therefore, no significant conclusions were drawn.

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 that participated. The average results, calculated reproducibilities and reproducibilities derived from reference test methods (in casu ASTM and IP standards) are compared in the next table.

Parameter	unit	n	average	2.8 * sd	R (lit)
Acid Number (total)	mg KOH/g	16	0.16	0.04	0.09
Air-release time at 50°C	min	7	3.3	1.0	2.4
Density at 15°C	kg/L	18	0.8629	0.0006	0.0005
Flash Point C.O.C.	°C	12	220.9	5.7	18
Foam Tendency Seq. I	ml	8	50.6	98.8	(30.6)
Foam Tendency Seq. II	ml	9	17.2	12.4	15.9
Foam Tendency Seq. III	ml	8	69.4	128.8	(30.5)
Foam Stability Seq. I	ml	9	0	n.a.	n.a.
Foam Stability Seq. II	ml	9	0	n.a.	n.a.
Foam Stability Seq. III	ml	9	0	n.a.	n.a.
Kinematic viscosity at 40°C	mm^2/s	18	31.352	0.206	0.383
Kinematic viscosity at 100°C	mm^2/s	15	5.394	0.064	0.074
Viscosity Index		13	105.7	2.6	2
Pour Point (manual)	°C	11	-12.3	4.0	9
Pour Point (automated), 1°C int.	°C	5	-13.0	3.4	4.5
Sulphur	mg/kg	8	287	51	74
Water content (by KF)	mg/kg	14	37.9	45.5	149.5
Water Separability at 54°C, distilled water					
- Time ≤ 3 ml emulsion	min	9	8.3	3.0	20
- Time 37 ml water	min	9	8.4	2.3	20
- Time to complete break	min	7	9.7	1.6	20
- Volume Oil phase	ml	8	40	2.1	n.a.
- Volume Water phase	ml	8	39	3.3	n.a.
- Volume Emulsion phase	ml	8	0	3.0	n.a.
Calcium as Ca	mg/kg	12	<1	n.a.	n.a.
Phosphorus as P	mg/kg	13	49.7	8.2	30.3
Zinc as Zn	mg/kg	12	<1	n.a.	n.a.

Table 3: reproducibilities of tests on sample #18076

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 relevant reference test methods. The tests that are problematic have been discussed in paragraph 4.1.

4.3 OVERVIEW OF THE PROFICIENCY TEST OF MAY 2018

	May 2018
Number of reporting labs	19
Number of results reported	281
Statistical outliers	12
Percentage outliers	4.3%

Table 4: overview of the proficiency test

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 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 int.	+
Sulphur	+
Water content (by KF)	++
- Time ≤ 3 ml emulsion	++
- Time 37 ml water	++
- Time to complete break	++
Calcium as Ca	n.e.
Phosphorus as P	++
Zinc as Zn	n.e.

Table 5: comparison determinations against the respective 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:

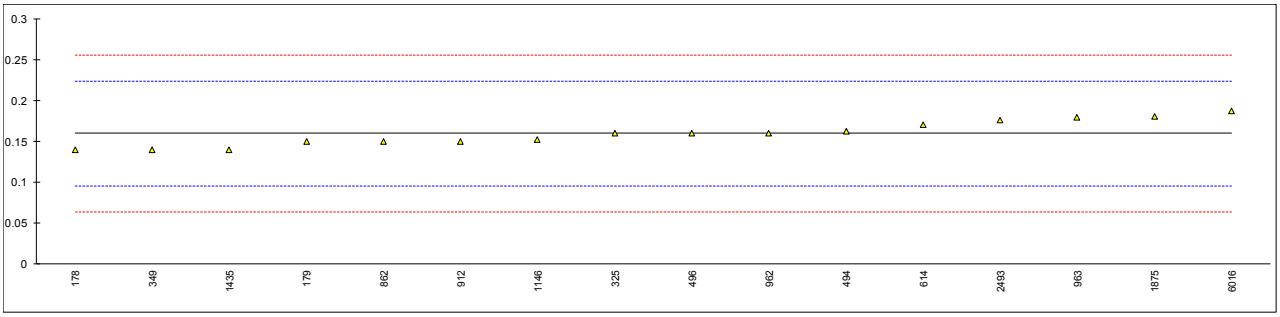
++:	group performed much better than the reference test method
+ :	group performed better than the reference test method
+/-:	group performance equals the reference test method
- :	group performed worse than the reference test method
-- :	group performed much worse than the reference test method
n.e.: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

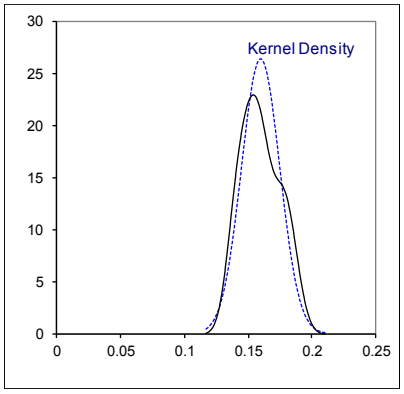
APPENDIX 1

Determination of Acid Number (total) on sample #18076; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks	Determination of end point	Volume of titration solvent
178	D664Mod.	0.14		-0.62		Buffer End Point (pH 11)	---
179	D664-A	0.15		-0.31		Buffer End Point (pH 11)	60 mL
237	D664-A	<0.1		-----		---	---
325	D664-A	0.16		0.01		Buffer End Point (pH 11)	125 mL
349	D664-A	0.14		-0.62		Inflection Point	125 mL
494	D664-A	0.162		0.07		Inflection Point	125 mL
496	D664-A	0.16		0.01		Buffer End Point (pH 11)	60 mL
614	D974	0.17		0.32		---	60 mL
862	D664-A	0.15		-0.31		---	---
912	D664-A	0.15		-0.31		---	---
962	D974	0.16		0.01		---	---
963	D664-A	0.179		0.60		Inflection Point	60 mL
1146	D664-A	0.152		-0.24		Buffer End Point (pH 11)	125 mL
1184		-----		-----		---	---
1243		-----		-----		---	---
1435	D664-A	0.140		-0.62		Buffer End Point (pH 11)	---
1875	ISO6618	0.18		0.63		Inflection Point	60 mL
2493	ISO6618	0.176		0.51		---	---
6016	D664-A	0.187		0.85		Inflection Point	60 mL

			BEP (pH 11) only	Inflection point only
normality	OK		OK	OK
n	16		6	5
outliers	0		0	0
mean (n)	0.1597		0.1503	0.1696
st.dev. (n)	0.01508		0.00898	0.01893
R(calc.)	0.0422		0.0251	0.0530
st.dev.(D664-A:17a)	0.03193		0.02537	0.03354
R(D664-A:17a)	0.0894	Inflection – 60 ml	--	0.0939
Compare				
R(D664-A:17a)	0.0754	BEP (pH 11)	0.0710	--

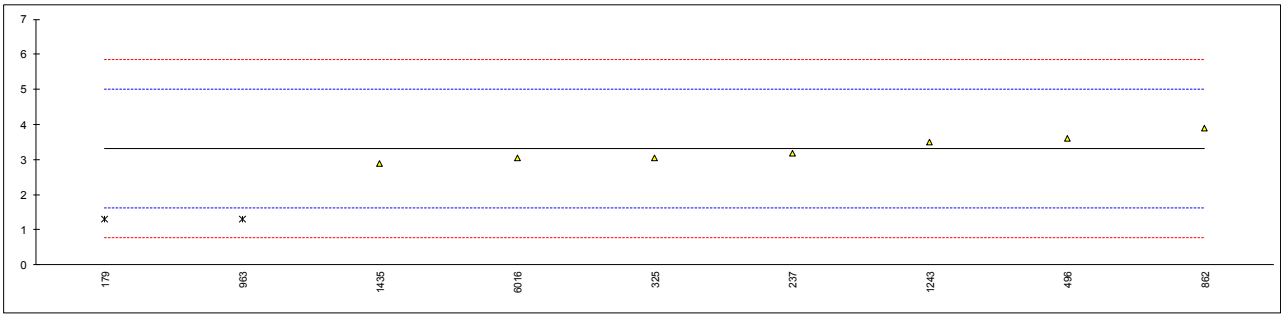


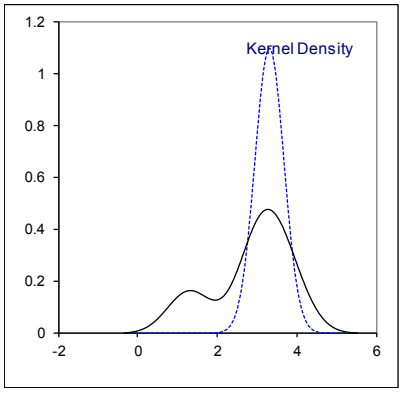


Determination of Air-release time at 50°C on sample #18076; results in minutes

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D3427	1.3	DG(0.05)	-2.38	
237	D3427	3.18		-0.15	
325	D3427	3.05		-0.31	
349		-----		-----	
494		-----		-----	
496	D3427	3.6		0.34	
614		-----		-----	
862	D3427	3.9		0.70	
912		-----		-----	
962		-----		-----	
963	D3427	1.3	DG(0.05)	-2.38	
1146		-----		-----	
1184		-----		-----	
1243	ISO9120	3.5		0.22	
1435	ISO9120	2.9		-0.49	
1875		-----		-----	
2493		-----		-----	
6016	D3427	3.04		-0.32	

normality	OK
n	7
outliers	2
mean (n)	3.3100
st.dev. (n)	0.3637
R(calc.)	1.018
st.dev.(D3427:15)	0.8447
R(D3427:15)	2.365

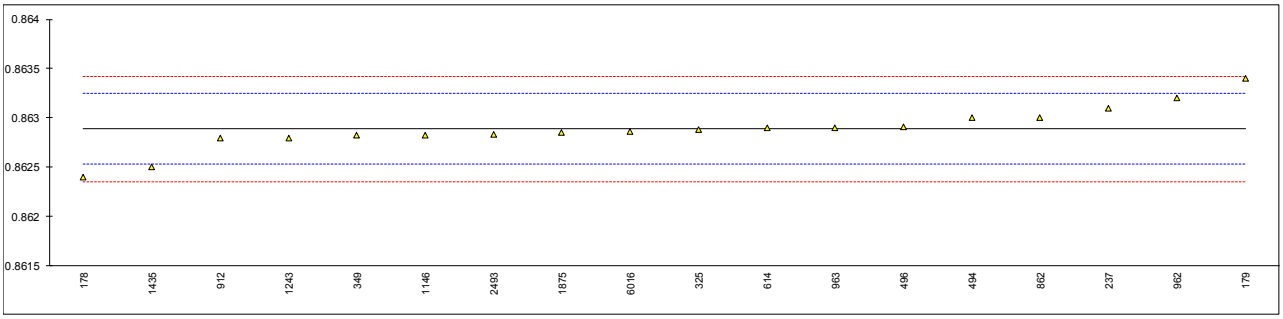


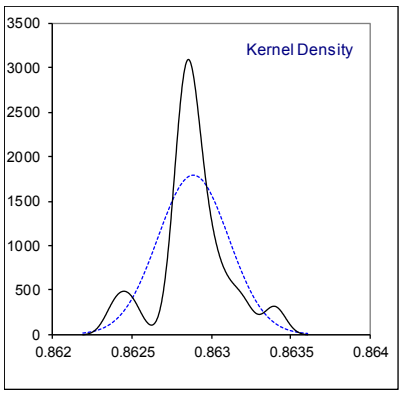


Determination of Density at 15°C on sample #18076; results in kg/L

lab	method	value	mark	z(targ)	remarks
178	D4052	0.8624		-2.73	
179	D4052	0.8634		2.87	
237	D4052	0.8631		1.19	
325	D4052	0.86288		-0.04	
349	D4052	0.86282		-0.38	
494	D4052	0.8630		0.63	
496	D4052	0.86291		0.13	
614	D4052	0.8629		0.07	
862	D4052	0.8630		0.63	
912	D4052	0.8628		-0.49	
962	D4052	0.8632		1.75	
963	D4052	0.8629		0.07	
1146	D4052	0.86282		-0.38	
1184		-----		-----	
1243	ISO12185	0.8628		-0.49	
1435	D4052	0.8625		-2.17	
1875	ISO12185	0.86285		-0.21	
2493	ISO12185	0.862837		-0.28	
6016	D4052	0.86286		-0.15	

normality	suspect
n	18
outliers	0
mean (n)	0.86289
st.dev. (n)	0.000223
R(calc.)	0.00063
st.dev.(D4052:18)	0.000175
R(D4052:18)	0.00050

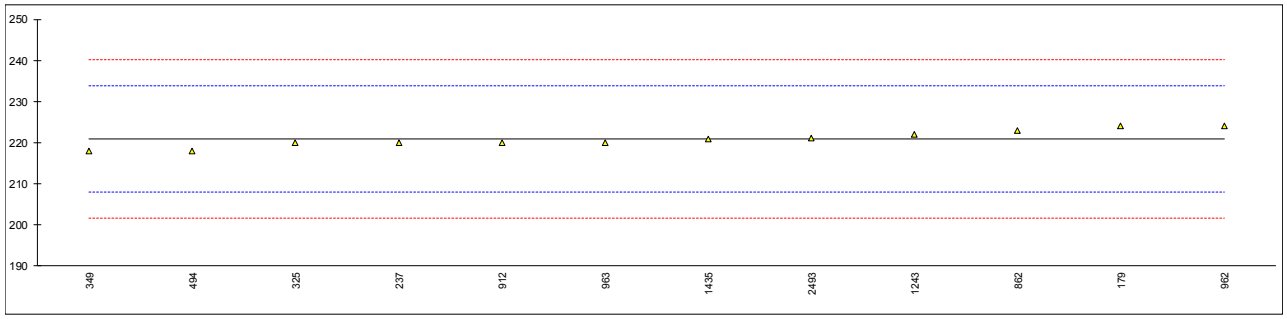


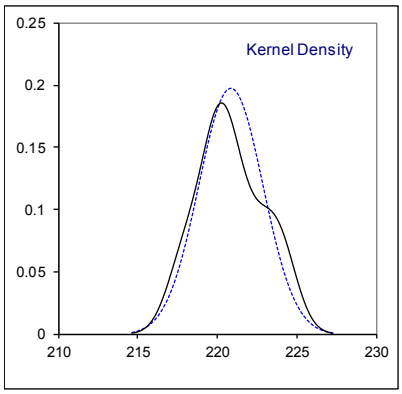


Determination of Flash Point C.O.C. on sample #18076; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D92	224		0.48	
237	D92	220		-0.14	
325	D92	220		-0.14	
349	D92	218		-0.45	
494	D92	218		-0.45	
496		-----		-----	
614		-----		-----	
862	D92	223		0.33	
912	D92	220		-0.14	
962	D92	224		0.48	
963	D92	220		-0.14	
1146		-----		-----	
1184		-----		-----	
1243	ISO2592	222		0.17	
1435	D92	220.8		-0.02	
1875		-----		-----	
2493	ISO2592	221		0.02	
6016		-----		-----	

normality	OK
n	12
outliers	0
mean (n)	220.90
st.dev. (n)	2.021
R(calc.)	5.66
st.dev.(D92:18)	6.429
R(D92:18)	18



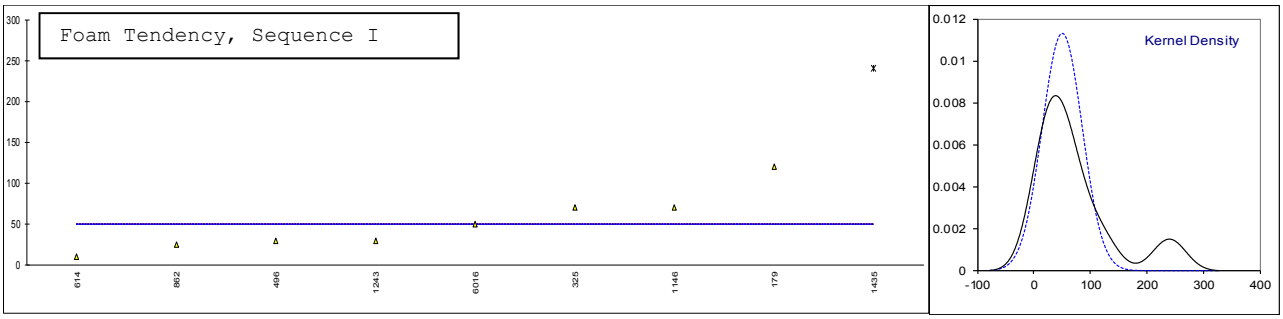


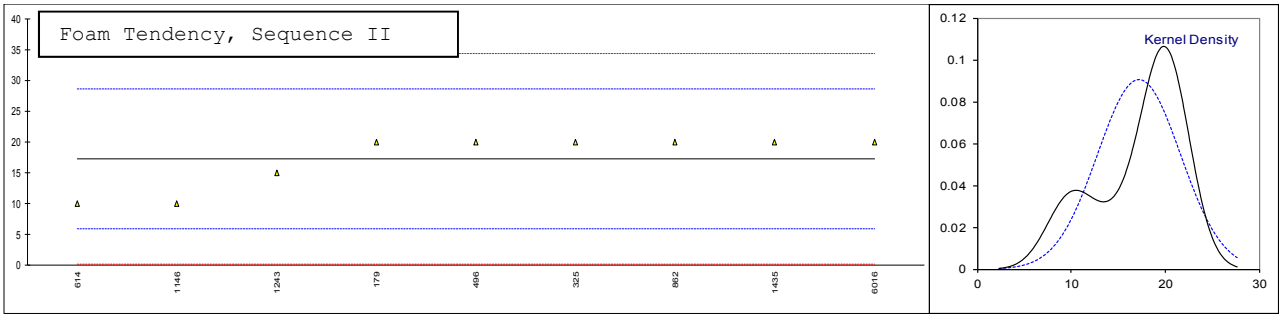
Determination of Foaming Tendency, Sequence I, II and III (5 min. blowing period) on sample #18076; results in mL

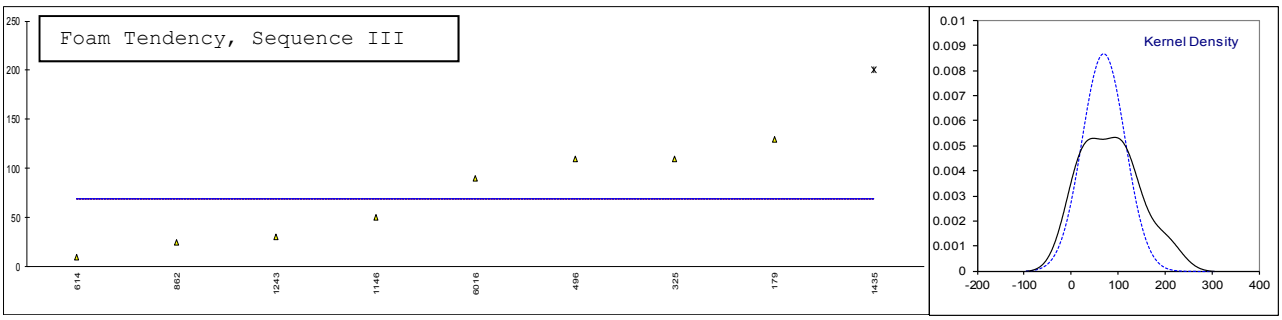
lab	method	Sample used	Diffuser	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
178		---	---	-----		-----	-----		-----	-----		-----
179	D892	As received	Metal	120		-----	20		0.49	130		-----
237		---	---	-----		-----	-----		-----	-----		-----
325	D892	As received	Metal	70		-----	20		0.49	110		-----
349		---	---	-----		-----	-----		-----	-----		-----
494		As received	---	-----		-----	-----		-----	-----		-----
496	D892	As received	Metal	30		-----	20		0.49	110		-----
614	D892	As received	Metal	10		-----	10		-1.27	10		-----
862	D892	---	---	25		-----	20		0.49	25		-----
912		---	---	-----		-----	-----		-----	-----		-----
962		---	---	-----		-----	-----		-----	-----		-----
963		---	---	-----		-----	-----		-----	-----		-----
1146	ISO6247	As received	Metal	70		-----	10		-1.27	50		-----
1184				-----		-----	-----		-----	-----		-----
1243	D892	As received	Stone	30		-----	15		-0.39	30		-----
1435	D892	As received	Metal	240	G(5)	-----	20		0.49	200	G(5)	-----
1875		---	---	-----		-----	-----		-----	-----		-----
2493		---	---	-----		-----	-----		-----	-----		-----
6016	D892	Aft ag. (A)	Stone	50		-----	20		0.49	90		-----

	Seq. I	Seq. II	Seq. III
normality	not OK	OK	OK
n	8	9	8
outliers	1	0	1
mean (n)	50.63	17.22	69.38
st.dev. (n)	35.2985	4.4096	46.0153
R(calc.)	98.84	12.35	128.84
st.dev.(D892:18)	(10.9397)	5.6849	(10.9018)
R(D892:18)	(30.63)	15.92	(30.53)

As rec. = As received
Aft. Ag. = after agitation (option A)







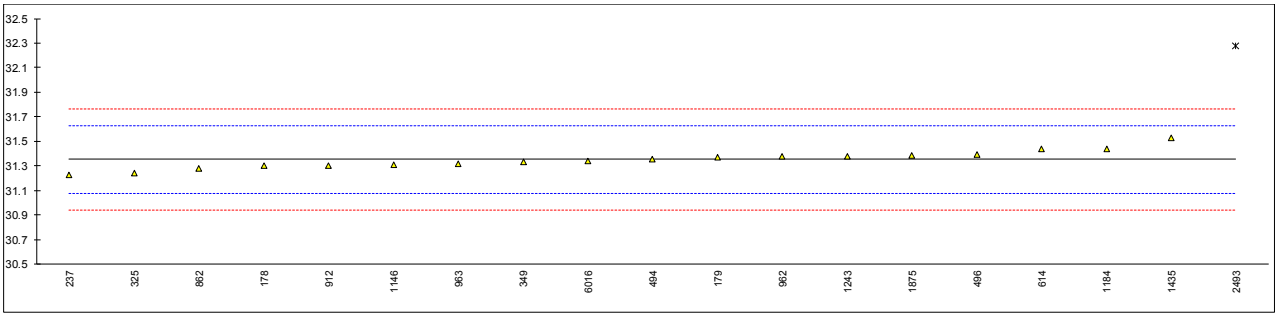
Determination of Foaming Stability, Sequence I, II and III (10 min. settling period) on sample #18076; results in mL

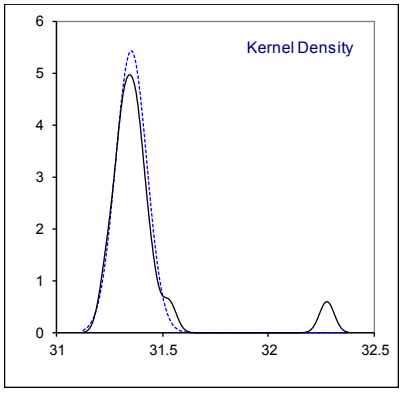
lab	method	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179	D892	0		-----	0		-----	0		-----
237		-----		-----	-----		-----	-----		-----
325	D892	0		-----	0		-----	0		-----
349		-----		-----	-----		-----	-----		-----
494		-----		-----	-----		-----	-----		-----
496	D892	0		-----	0		-----	0		-----
614	D892	0		-----	0		-----	0		-----
862	D892	0		-----	0		-----	0		-----
912		-----		-----	-----		-----	-----		-----
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
1146	ISO6247	0		-----	0		-----	0		-----
1184		-----		-----	-----		-----	-----		-----
1243	D892	0		-----	0		-----	0		-----
1435	D892	0		-----	0		-----	0		-----
1875		-----		-----	-----		-----	-----		-----
2493		-----		-----	-----		-----	-----		-----
6016	D892	0		-----	0		-----	0		-----
n		9			9			9		
mean (n)		0			0			0		

Determination of Kinematic Viscosity at 40°C on sample #18076; results in mm²/s

lab	method	value	mark	z(targ)	remarks
178	D445	31.3		-0.38	
179	D445	31.37		0.14	
237	D445	31.23		-0.89	
325	D445	31.245		-0.78	
349	D445	31.33		-0.16	
494	D445	31.353		0.01	
496	D445	31.394		0.31	
614	D445	31.44		0.65	
862	D445	31.28		-0.52	
912	D445	31.30		-0.38	
962	D445	31.38		0.21	
963	D445	31.32		-0.23	
1146	D445	31.313		-0.28	
1184	D445	31.44		0.65	
1243	D7279 corrected to D445	31.38		0.21	
1435	D7042	31.529		1.30	
1875	D7042	31.386		0.25	
2493	ISO3104	32.2735	D(0.01)	6.75	
6016	D7042	31.338		-0.10	

normality	OK
n	18
outliers	1
mean (n)	31.3516
st.dev. (n)	0.07348
R(calc.)	0.2057
st.dev.(D445:17a)	0.13660
R(D445:17a)	0.3825

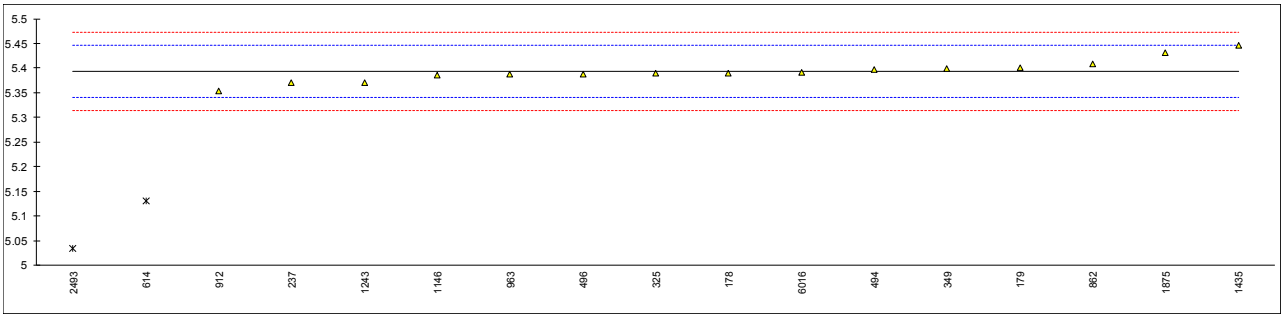


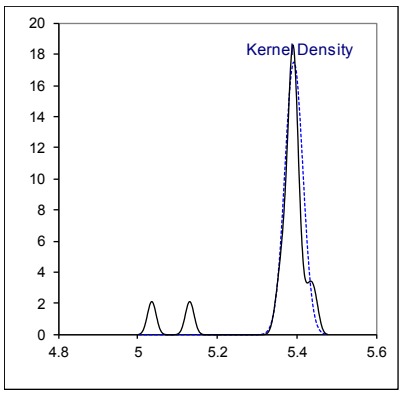


Determination of Kinematic Viscosity at 100°C on sample #18076; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178	D445	5.39		-0.14	
179	D445	5.40		0.23	
237	D445	5.370		-0.89	
325	D445	5.3889		-0.18	
349	D445	5.398		0.16	
494	D445	5.3975		0.14	
496	D445	5.3878		-0.22	
614	D445	5.13	D(0.01)	-9.92	
862	D445	5.409		0.57	
912	D445	5.354		-1.50	
962		-----		-----	
963	D445	5.387		-0.25	
1146	D445	5.3859		-0.30	
1184		-----		-----	
1243	D7279 corrected to D445	5.37		-0.89	
1435	D7042	5.446		1.96	
1875	D7042	5.43075		1.39	
2493	ISO3104	5.034	D(0.01)	-13.53	
6016	D7042	5.3917		-0.08	

normality	suspect
n	15
outliers	2
mean (n)	5.3938
st.dev. (n)	0.02283
R(calc.)	0.0639
st.dev.(D445:17a)	0.02658
R(D445:17a)	0.0744

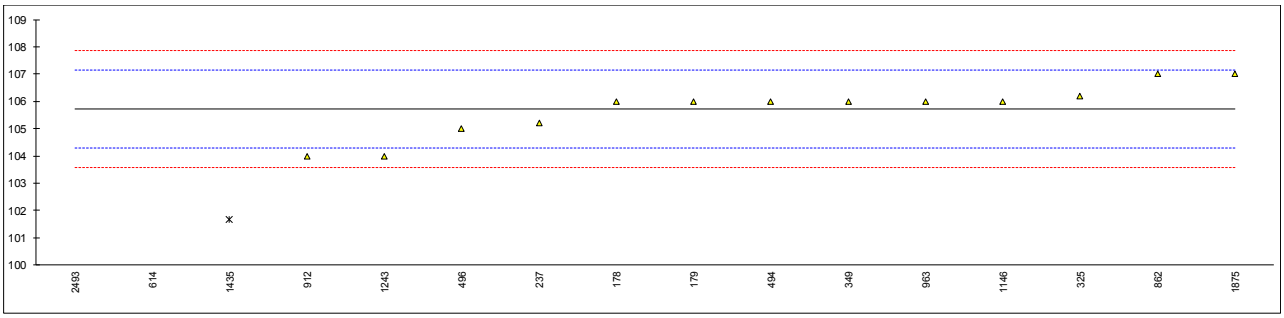


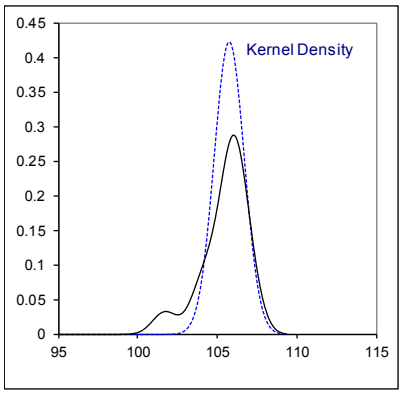


Determination of Viscosity Index on sample #18076

lab	method	value	mark	z(targ)	remarks
178	D2270	106		0.39	
179	D2270	106		0.39	
237	D2270	105.2		-0.73	
325	D2270	106.2		0.67	
349	D2270	106		0.39	
494	D2270	106		0.39	
496	D2270	105		-1.01	
614	D2270	87	ex	-26.21	Outlier in viscosity 100°C
862	D2270	107		1.79	
912	D2270	104		-2.41	
962		-----		-----	
963	D2270	106		0.39	
1146	D2270	106		0.39	
1184		-----		-----	
1243	ISO2909	104		-2.41	
1435	D2270	101.68	E,G(0.05)	-5.66	Probably calculation error? (iis calc. 107.81)
1875	ISO2909	107		1.79	
2493	ISO2909	71	ex	-48.61	Outlier in viscosity 40°C and 100°C
6016		-----		-----	

normality	OK
n	13
outliers	1 (+2ex)
mean (n)	105.72
st.dev. (n)	0.944
R(calc.)	2.64
st.dev.(D2270:10)	0.714
R(D2270:10)	2

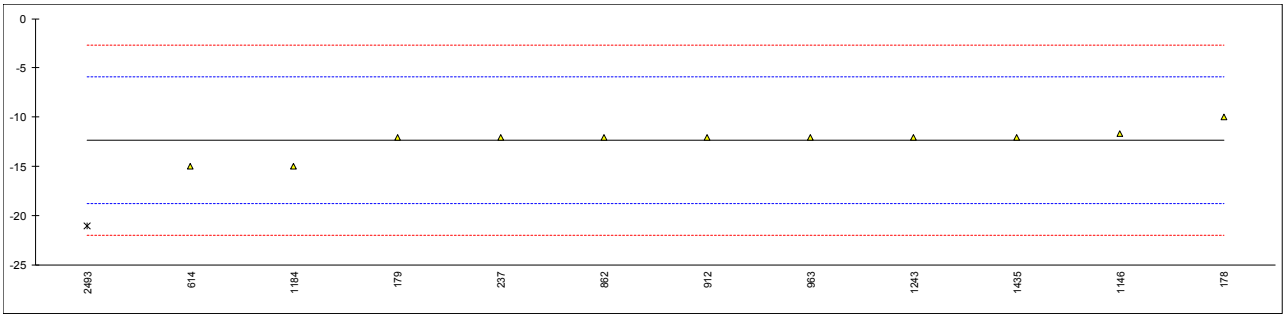


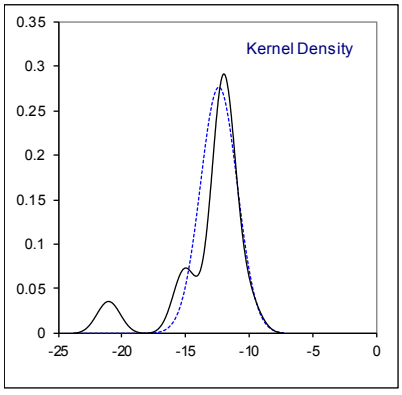


Determination of Pour Point manual on sample #18076; results in °C

lab	method	value	mark	z(targ)	remarks
178	D97	-10		0.73	
179	D97	-12		0.10	
237	D97	-12		0.10	
325		-----		-----	
349		-----		-----	
494		-----		-----	
496		-----		-----	
614	D97	-15		-0.83	
862	D97	-12		0.10	
912	D97	-12		0.10	
962		-----		-----	
963	D97	-12		0.10	
1146	D97	-11.7		0.20	
1184	D97	-15		-0.83	
1243	ISO3016	-12		0.10	
1435	D97	-12		0.10	
1875		-----		-----	
2493	ISO3016	-21	G(0.01)	-2.70	
6016		-----		-----	

normality	suspect
n	11
outliers	1
mean (n)	-12.34
st.dev. (n)	1.444
R(calc.)	4.04
st.dev.(D97:17b)	3.214
R(D97:17b)	9

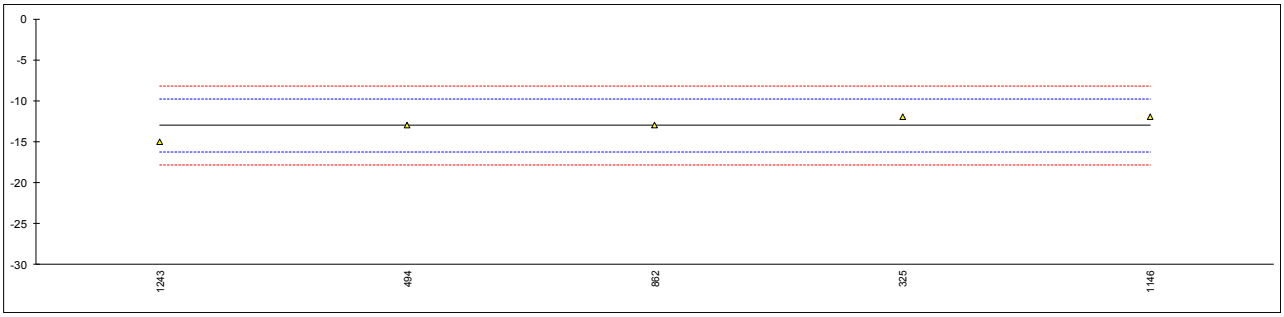




Determination of Pour Point automated 1°C int. on sample #18076; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
325	D5950	-12		0.62	
349		-----		-----	
494	D5950	-13		0.00	
496		-----		-----	
614		-----		-----	
862	D5950	-13		0.00	
912		-----		-----	
962		-----		-----	
963		-----		-----	
1146	D6892	-12		0.62	reported 3°C interval
1184		-----		-----	
1243	D7346	-15		-1.24	
1435		-----		-----	
1875		-----		-----	
2493		-----		-----	
6016		-----		-----	

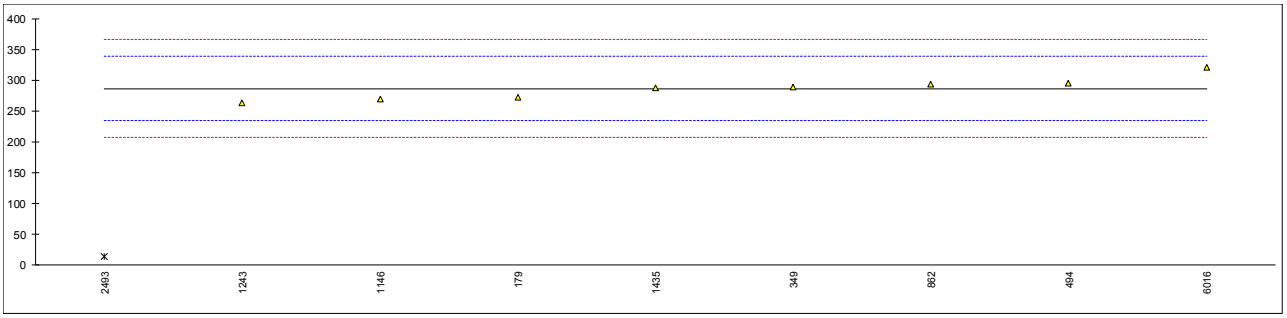
normality	unknown
n	5
outliers	0
mean (n)	-13.00
st.dev. (n)	1.225
R(calc.)	3.43
st.dev.(D5950:14)	1.607
R(D5950:14)	4.5

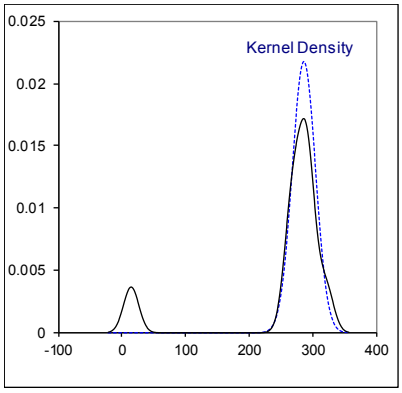


Determination of Sulphur on sample #18076; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D4294	273		-0.52	
237		-----		-----	
325		-----		-----	
349	D2622	289		0.09	
494	D4294	295		0.32	
496		-----		-----	
614		-----		-----	
862	D2622	294		0.28	
912		-----		-----	
962		-----		-----	
963		-----		-----	
1146	D4294	270		-0.63	
1184		-----		-----	
1243	ISO8754	263		-0.90	
1435	D5185	287.4		0.03	
1875		-----		-----	
2493	EN15492	14	C,D(0.01)	-10.37	First reported 88.2
6016	D4294	321.2		1.32	

normality	OK
n	8
outliers	1
mean (n)	286.58
st.dev. (n)	18.308
R(calc.)	51.26
st.dev.(D4294:16e1)	26.283
R(D4294:16e1)	73.59

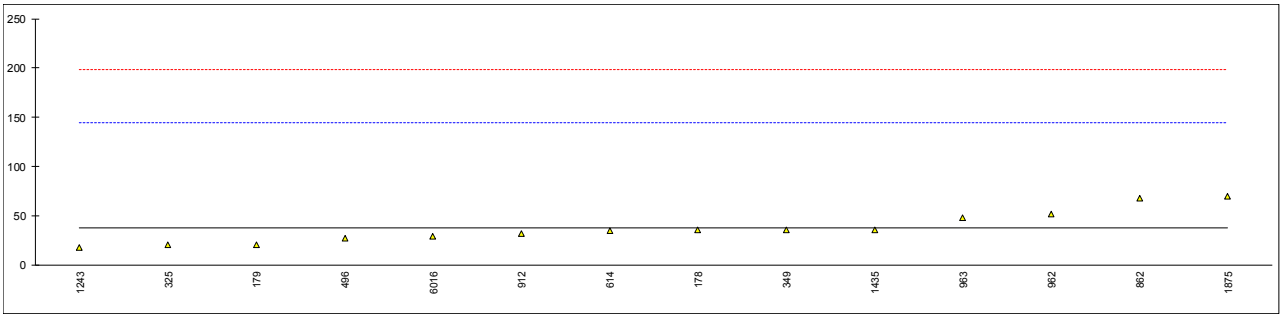


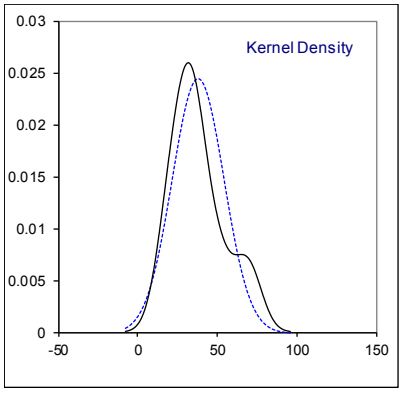


Determination of Water by KF on sample #18076; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D6304-C	36		-0.04	
179	D6304-C	21		-0.32	
237		-----		-----	
325	D6304-C	20.5		-0.33	
349	D6304-A	36		-0.04	
494		-----		-----	
496	D6304-C	28		-0.18	
614	D6304-C	35		-0.05	
862	D6304-C	68		0.56	
912	D6304-C	32		-0.11	
962	D6304-A	52		0.26	
963	D6304-A	48		0.19	
1146		-----		-----	
1184		-----		-----	
1243	ISO12937	18		-0.37	
1435	D6304-A	36		-0.04	
1875	ISO12937	70		0.60	
2493		-----		-----	
6016	D6304-A	29.75		-0.15	

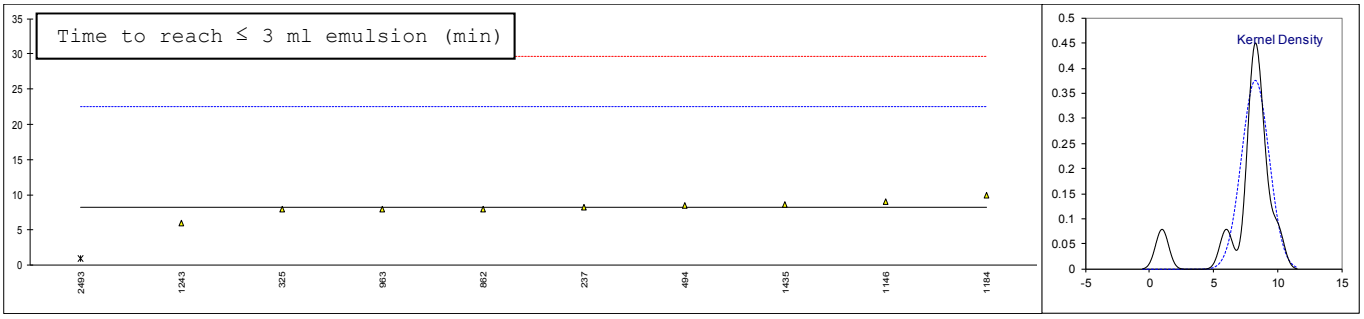
normality	OK
n	14
outliers	0
mean (n)	37.875
st.dev. (n)	16.2672
R(calc.)	45.548
st.dev.(D6304:16e1)	53.3982
R(D6304:16e1)	149.515

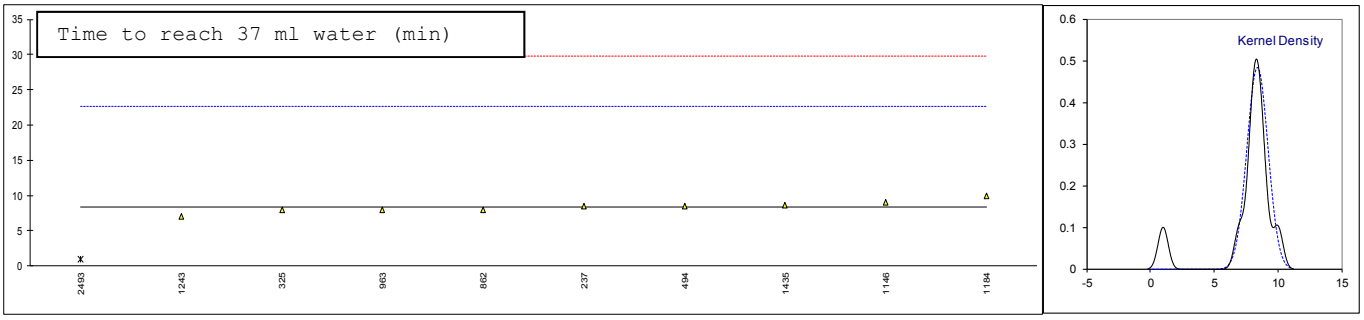


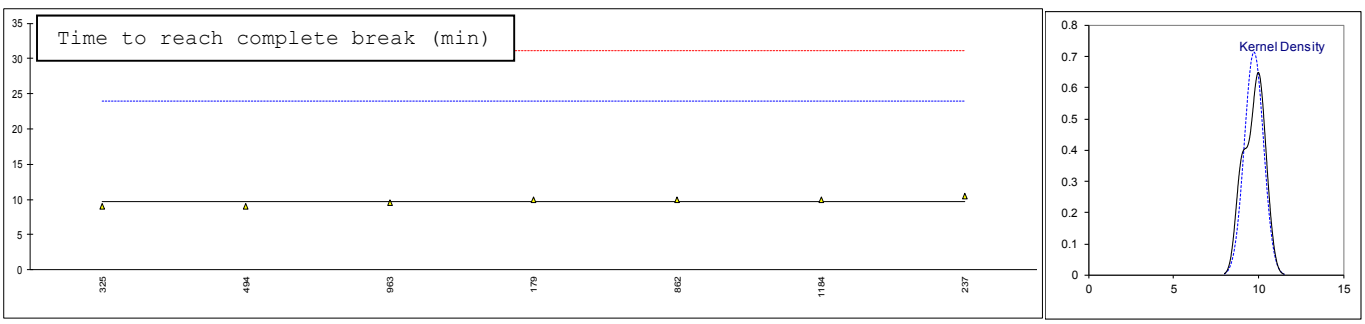


Determination of Water Separability at 54°C, distilled water on sample #18076; results in minutes

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	-----		-----	-----		-----	10		0.04	-----	-----
237		8.26		0.00	8.5		0.01	10.48		0.11	-----	-----
325	D1401	8		-0.04	8		-0.06	9		-0.10	-----	-----
349		-----		-----	-----		-----	-----		-----	-----	-----
494	D1401	8.5		0.03	8.5		0.01	9.0		-0.10	-----	-----
496		-----		-----	-----		-----	-----		-----	-----	-----
614		-----		-----	-----		-----	-----		-----	-----	-----
862	D1401	8		-0.04	8		-0.06	10		0.04	-----	10
912		-----		-----	-----		-----	-----		-----	-----	-----
962		-----		-----	-----		-----	-----		-----	-----	-----
963	D1401	8		-0.04	8		-0.06	9.58		-0.02	-----	-----
1146	D1401	9		0.10	9		0.08	-----		-----	-----	-----
1184	D1401	10		0.24	10		0.22	10		0.04	-----	-----
1243	ISO6614	6		-0.32	7		-0.20	-----		-----	-----	30
1435	ISO6614	8.58		0.04	8.58		0.03	-----		-----	-----	-----
1875		-----		-----	-----		-----	-----		-----	-----	-----
2493	ISO6614	1	D(5)	-1.02	1	D(1)	-1.04	-----	W, fr. 2	-----	-----	-----
6016		-----		-----	-----		-----	-----		-----	-----	-----
normality		not OK			suspect			unknown				
n		9			9			7				
outliers		1			1			0				
mean (n)		8.26			8.40			9.72				
st.dev. (n)		1.063			0.823			0.558				
R(calc.)		2.98			2.30			1.56				
st.dev.(D1401:18a)		7.143			7.143			7.143				
R(D1401:18a)		20			20			20				







Determination of Water Separability at 54°C, distilled water on sample #18076; results in ml

lab	method	oil	mark	z(targ)	water	mark	z(targ)	emulsion	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179		40		-----	40		-----	0		-----
237		40		-----	40		-----	0		-----
325		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
494		40		-----	40		-----	0		-----
496		-----		-----	-----		-----	-----		-----
614		-----		-----	-----		-----	-----		-----
862		40		-----	40		-----	0		-----
912		-----		-----	-----		-----	-----		-----
962		-----		-----	-----		-----	-----		-----
963		40		-----	40		-----	0		-----
1146		40		-----	37		-----	3		-----
1184		42		-----	38		-----	0		-----
1243		41		-----	39		-----	0		-----
1435		-----		-----	-----		-----	-----		-----
1875		-----		-----	-----		-----	-----		-----
2493		-----		-----	-----		-----	-----		-----
6016		-----		-----	-----		-----	-----		-----
normality		unknown			unknown			unknown		
n		8			8			8		
outliers		0			0			0		
mean (n)		40.38			39.25			0.38		
st.dev. (n)		0.744			1.165			1.061		
R(calc.)		2.08			3.26			2.97		
st.dev.(target)		n.a.			n.a.			n.a.		
R(target)		n.a.			n.a.			n.a.		

Determination of Calcium as Ca on sample #18076; results in mg/kg

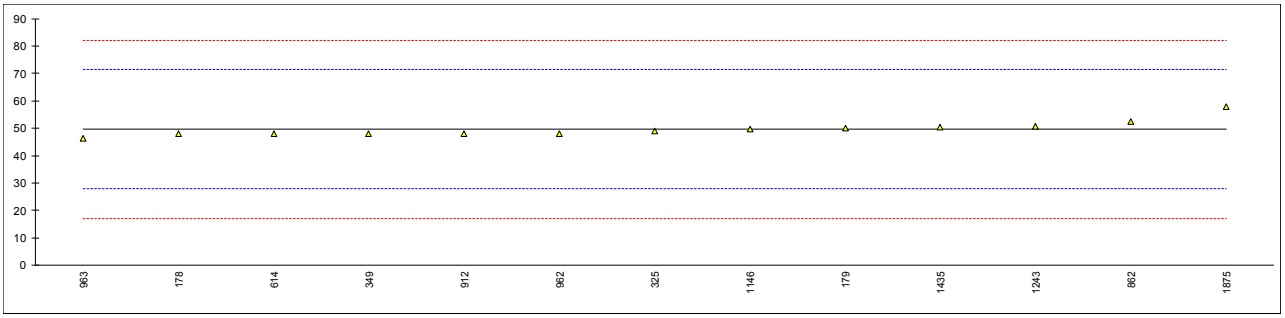
lab	method	value	mark	z(targ)	remarks
178	D5185	1		-----	
179	D5185	<1		-----	
237		-----		-----	
325	D5185	0		-----	
349	D5185	<1	C	-----	First reported 48
494		-----		-----	
496		-----		-----	
614	D5185	<1		-----	
862	D5185	<1		-----	
912		<1		-----	
962	D5185	<1		-----	
963	D5185	0.15		-----	
1146	D5185/D4951	0.8073		-----	
1184		-----		-----	
1243	DIN51399	0		-----	
1435	D5185	0.0545		-----	
1875		25		-----	Possibly a false positive test result?
2493		-----		-----	
6016		-----		-----	
	n	12			
	mean (n)	<1			Application range D5185:18: 40 – 9000 mg/kg

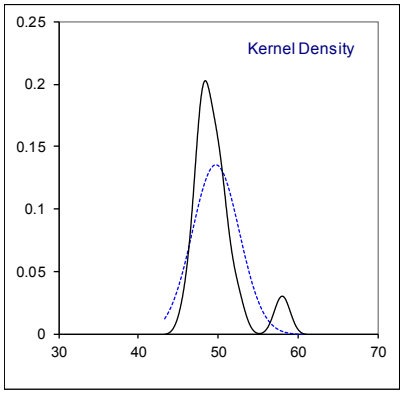
Determination of Phosphorus as P on sample #18076; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	48		-0.16	
179	D5185	50		0.03	
237		-----		-----	
325	D5185	49		-0.06	
349	D5185	48	C	-0.16	First reported <1
494		-----		-----	
496		-----		-----	
614	D5185	48	C	-0.16	First reported <1
862	D5185	52.3	C	0.24	First reported <1
912		48		-0.16	
962	D5185	48		-0.16	
963	D5185	46.28		-0.32	
1146	D5185/D4951	49.55		-0.01	
1184		-----		-----	
1243	DIN51399	50.6		0.08	
1435	D5185	50.36		0.06	
1875		58		0.77	
2493		-----		-----	
6016		-----		-----	

	normality	not OK
	n	13
	outliers	0
	mean (n)	49.699
	st.dev. (n)	2.9389
	R(calc.)	8.229
	st.dev.(D5185:18)	10.8264
	R(D5185:18)	30.314

Application range D5185:18: 10 – 1000 mg/kg





Determination of Zinc as Zn on sample #18076; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	1		-----	
179	D5185	<1		-----	
237		-----		-----	
325	D5185	0		-----	
349	D5185	<1		-----	
494		-----		-----	
496		-----		-----	
614	D5185	<1	C	-----	First reported 48
862	D5185	<1		-----	
912		<1		-----	
962	D5185	<1		-----	
963	D5185	0.19		-----	
1146	D5185/D4951	0.7061		-----	
1184		-----		-----	
1243	DIN51399	0		-----	
1435	D5185	0.1957		-----	
1875		16		-----	Possibly a false positive test result?
2493		-----		-----	
6016		-----		-----	
	n	12			
	mean (n)	<1			Application range D5185:18: 60 – 1600 mg/kg

APPENDIX 2

Number of participants per country

1 lab in AUSTRALIA
2 labs in BELGIUM
1 lab in CHINA, People's Republic
4 labs in GERMANY
1 lab in HUNGARY
1 lab in INDIA
1 lab in KAZAKHSTAN
1 lab in NETHERLANDS
1 lab in NIGERIA
2 labs in SAUDI ARABIA
1 lab in SOUTH KOREA
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	= probably an error in calculations
U	= test result reported probably in a different unit
W	= test result withdrawn on request of participant
ex	= test result excluded from the statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
SDS	= Material Safety Data Sheet

Literature:

1 iis Interlaboratory Studies, Protocol for the Organization, Statistics and Evaluation, March 2017
2 ASTM E178:89
3 ASTM E1301:89
4 ISO 5725:86
5 ISO 5725, parts 1-6, 1994
6 ISO13528:05
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 R.G. Visser and C. Nijssen-Wester, Estimation of reproducibility and measurement uncertainty of a viscosity test method from proficiency test data, Accred Qual Assur (2015) 20:125-129, DOI 10.1007/s00769-015-1110-y