Results of Proficiency Test
Gear Oil (fresh)
April 2017

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. R.J. Starink
Correctors: dr. R.G. Visser & ing. G.A. Oosterlaken-Buijs
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CONTENTS

Appendices:

1 INTRODUCTION

In 2015 Institute for Interlaboratory Studies organised a new proficiency test for the analyses of fresh Gear Oil on request of several participants. During the annual proficiency testing program 2016/2017 it was decided to continue with the round robin for the analyses of fresh Gear Oil. In this interlaboratory study, 15 laboratories in 14 different countries registered for participation. See appendix 2 for the number of participants per country.
In this report, the results of the 2017 fresh Gear Oil 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 were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send one bottle of 1L (labelled #17035) with fresh Gear 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% confidentially 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 Organization, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4). This protocol can be downloaded from 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 purchased from a local supplier. The 20 litres bulk material was homogenized and transferred into 18 brown glass bottles of 1 litre and 8 brown glass bottles of 250 ml (labelled #17035). The homogeneity of the eight 250 ml subsamples was checked by determination of Density at 15°C in accordance with ASTM D4052 and Water content in accordance with ASTM D6304, proc A on 8 stratified randomly selected samples.

	Density at 15 °C in kg/m^3	Water in mg/kg
Sample #17035-1	891.40	180
Sample #17035-2	891.39	180
Sample #17035-3	891.40	185
Sample #17035-4	891.39	185
Sample #17035-5	891.36	185
Sample #17035-6	891.37	170
Sample #17035-7	891.37	180
Sample #17035-8	891.36	170

Table 1: homogeneity test results of subsamples #17035

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibilities 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/m^3	Water in mg/kg
r (observed)	0.05	17
reference test method	ASTM D4052:16	ASTM D6304:16e1
0.3 x R(ref. test method)	0.15	114

Table 2: evaluation of the repeatability of subsamples #17035

The calculated repeatabilities were less than 0.3 times the corresponding reproducibility of the reference test methods. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories, one sample of 1 L in a brown glass bottle (labelled #17035) was sent on March 15, 2017.

2.5 STABILITY OF THE SAMPLES

The stability of Gear Oil packed in amber glass bottle was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYSES

The participants were asked to determine Acid Number (Total), Copper Corrosion, Density at 15°C, Flash Point (COC and PMcc), Foaming Tendency and Stability, Kinematic Viscosity at 40°C and at 100°C, Viscosity Index, Pour Point Manual and Automated, Rust prevention (proc. A), Sulphur, Water, Water separability, Calcium, Phosphorus and Zinc.

It was explicitly requested to treat the samples as if they were routine samples. Therefore, each laboratory is advised to perform only those analyses that normally are done in daily routine (but the laboratories are allowed to do all analyses). Furthermore, it was requested to report the test results using the indicated units on the report form and not to round the test results more, but report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods 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 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. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

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. 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 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 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, except for one participant in Saudi Arabia. All, except one, participants reported test results in time.
Not all participants were able to report test results for all the requested tests.
In total 14 participants reported 177 test results. Observed were 8 outlying test results, which is 4.5% 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, see also paragraph 3.1.

4.1 EVALUATION PER TEST

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

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 D664:11ae1.

Copper Corrosion: Seven laboratories reported a test result. One laboratory reported a test result of 3A, which may be a false positive test result. All other participants agreed on classification 1.

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 full agreement with the requirements of ASTM D4052:16.

Flash Point COC: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with ASTM D92:16b.

Flash Point PMcc: This determination was not problematic. One statistical outlier was observed. One test result was excluded, test method ASTM D93B is not meant for fresh oils. The calculated reproducibility after rejection of the suspect data is in good agreement with ASTM D93A:16a.

Foaming Characteristics (Tendency and Stability): This determination was not problematic. No statistical outliers were observed in Foam Tendency. Both calculated reproducibilities are in agreement with the requirements of ASTM D892:13.

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

Kinematic Viscosity at 100°C: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D445:17.

Viscosity Index: This determination was not problematic. No statistical outliers were observed. Two test results were excluded for statistical calculations as the Kinematic Viscosity for these two laboratories was found to be an statistical outlier. However, the calculated reproducibility after rejection of the suspect data is in agreement with ASTM D2270:10.
Also iis calculated the Viscosity Index from the test results reported for the kinematic viscosities at 40°C and 100°C. The calculated reproducibility is somewhat smaller. Compared to the reported values none of the calculated (by iis) results deviate much.

Pour Point manual: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with requirements of ASTM D97:17. The low number of test results and the rounding of the test results to 3 degrees may (partly) explain the large variation.

Pour Point automated: This determination is not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with requirements of ASTM D5950:14.

Rust prevention: Five participants reported a result. All participants agreed on classification as “Pass”.

Sulphur: This determination may be problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D4294:16e1. The low number of test results may (partly) explain the large variation.

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 The determination may not be problematic. Only seven participants reported test results. No statistical outliers were observed in “time to reach 3ml or less emulsion” and “time to reach 37 ml of water”. The calculated reproducibilities are in agreement with the requirements of ASTM D1401:12e1. The calculated reproducibilities of the volume oil phase and the volume water phase are large compared to the calculated reproducibilities of the previous PT (3.9 vs 1.5).

Calcium as Ca: The consensus value for the Calcium determination was below the application range of ASTM D5185:13e1. Therefore, no significant conclusions could be drawn.

Phosphorus as P: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5185:13e1.

Zinc as Zn: The consensus value for the Zinc determination was below the application range of ASTM D5185:13e1. Therefore, no significant conclusions could be 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. The average test results, calculated reproducibilities (2.8*sd) and reproducibilities (R(lit)) derived from literature reference test methods (in casu ASTM test methods), are compared in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Acid Number (Total)	mg KOH/g	9	0.76	0.44	0.25
Copper Corrosion, 3hrs at 100°C	rating	6	1	n.a.	n.a.
Density at 15°C	kg/m^3	10	891.5	0.5	0.5
Flash Point COC	°C	8	237.9	20.9	18
Flash Point PMcc	°C	8	191.7	10.2	13.6
Foaming Tendency (Seq I)	mL	9	3.3	14.0	16.9
Foaming Stability (Seq I)	mL	7	0	0	16
Kinematic Viscosity at 40°C	mm^2/s	12	144.9	1.4	1.8
Kinematic Viscosity at 100°C	mm^2/s	12	14.43	0.14	0.20
Viscosity Index		11	97.2	1.7	2
Pour Point, Manual	°C	7	-22.2	15.7	9
Pour Point, Automated	°C	4	-27.5	2.8	4.5
Rust Prevention (proc. A)		5	pass	n.a.	n.a.
Sulphur	mg/kg	4	11586	1091	799
Water	mg/kg	8	120	159	298
Water Separability at 82°C, distilled water					
- Time ≤ 3 ml emulsion	min.	7	21.9	18.4	25
- Time 37 ml water	min.	7	23.3	18.5	25
- Time to complete break	min.	5	n.a.	n.a.	n.a.
- Volume Oil phase	mL	7	41.3	3.9	n.a.
- Volume Water phase	mL	7	38.3	3.9	n.a.
- Volume Emulsion phase	mL	7	0.4	3.2	n.a.
Calcium as C	mg/kg	9	<5	n.a.	n.a.
Phosphorus as P	mg/kg	10	332	31	78
Zinc as Zn	mg/kg	9	<5	n.a.	n.a.

Table 3: reproducibilities of the test results on sample #17035

Without further statistical calculations it can be concluded that for a number of tests there is not a good compliance of the group of participants with the relevant test methods. The tests that are problematic have been discussed in paragraph 4.1.

4.3 COMPARISON OF PROFICIENCY TEST OF APRIL 2017 WITH PREVIOUS PT

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

Determination	April 2017	April 2016	April 2015
Acid Number (Total)	-	+	+
Copper Corrosion, 3hrs at 100°C	n.e.	n.e.	n.e.
Density at 15 °C	+/-	+/-	-
Flash Point COC	-	n.e.	n.e.
Flash Point PMcc	+	-	-
Foaming Tendency/Stability	++	++	n.e.
Kinematic Viscosity at 40°C	+	+/-	+
Kinematic Viscosity at 100°C	++	++	+
Viscosity Index	+	+	+
Pour Point, Manual	-	+/-	--
Pour Point, Automated	++	+/-	+/-
Rust Prevention (proc. A)	n.e.	n.e.	n.e.
Sulphur	-	+/-	-
Water	++	n.e.	n.e.
Water Separability at 82°C	+	++	++
Calcium as C	n.e.	n.e.	n.e.
Phosphorus as P	++	+	--
Zinc as Zn	n.e.	n.e.	n.e.

Table 4: comparison determinations against the reference test method

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

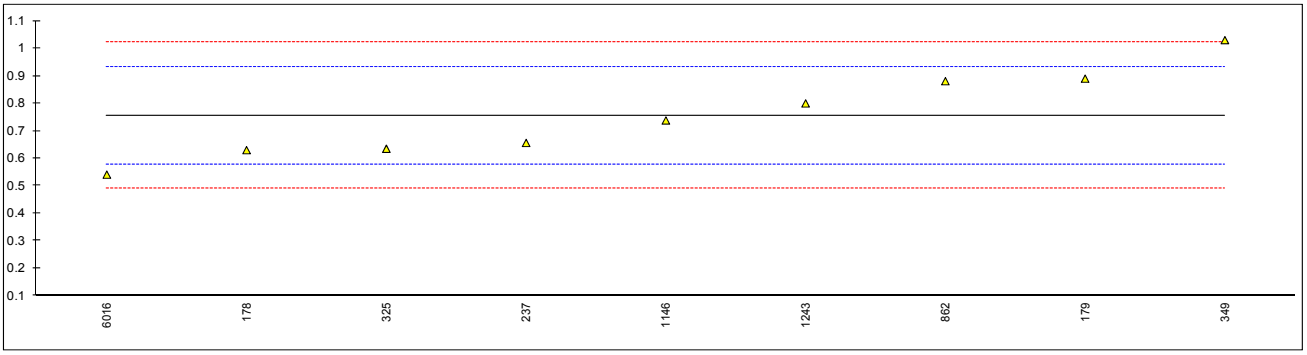
++: group performed much better than the reference test method
+ : group performed better than the reference test method
+/-: group performance equals the reference test method
- : group performed worse than the reference test method
-- : group performed much worse than the reference test method
n.e.: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

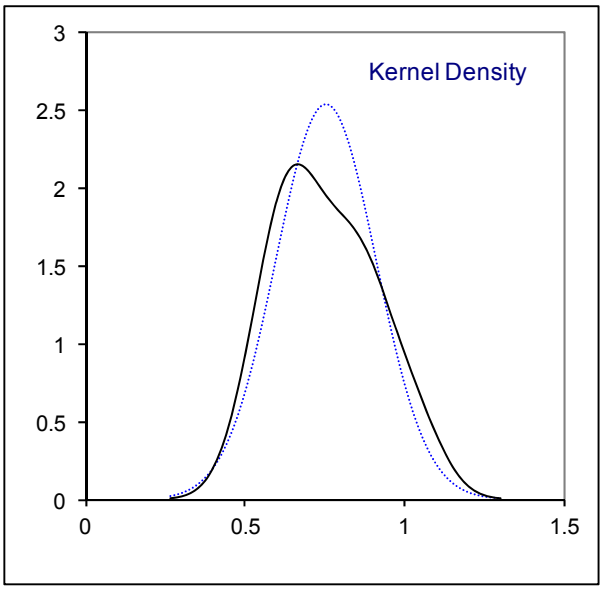
APPENDIX 1

Determination of Acid Number (Total) on sample #17035; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
178	INH-1118	0.63		-1.42	
179	D664-A	0.89		1.52	
237	D664-A	0.6562		-1.13	
325	D664-A	0.635		-1.36	
349	D664-A	1.03		3.10	
432		-----		-----	
862	D664-A	0.8808		1.42	
902		-----		-----	
963		-----		-----	
1146	D664-A	0.738		-0.20	
1174		-----		-----	
1243	D664-A	0.8		0.50	
1244		-----		-----	
1748		-----		-----	
6016	D664-A	0.541		-2.43	

normality	OK
n	9
outliers	0
mean (n)	0.756
st.dev. (n)	0.1571
R(calc.)	0.440
R(D664:11ae1)	0.248





Determination of Copper Corrosion 3 hours at 100°C on sample #17035;

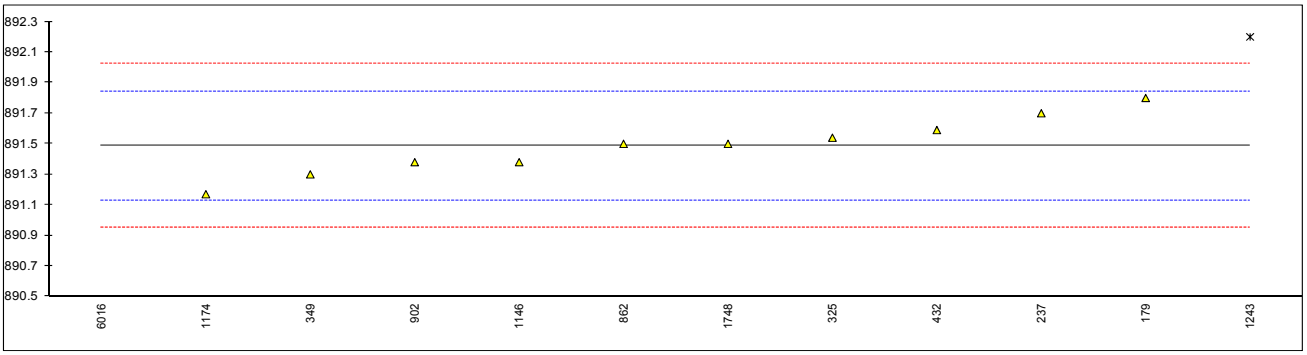
lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D130	1B		-----	
237	D130	1A		-----	
325	D130	1B		-----	
349		-----		-----	
432		-----		-----	
862	D130	1b		-----	
902		-----		-----	
963		-----		-----	
1146		-----		-----	
1174	ISO2160	3a		-----	False positive test result?
1243	ISO2160	1a		-----	
1244		-----		-----	
1748	D130	1a		-----	
6016		-----		-----	

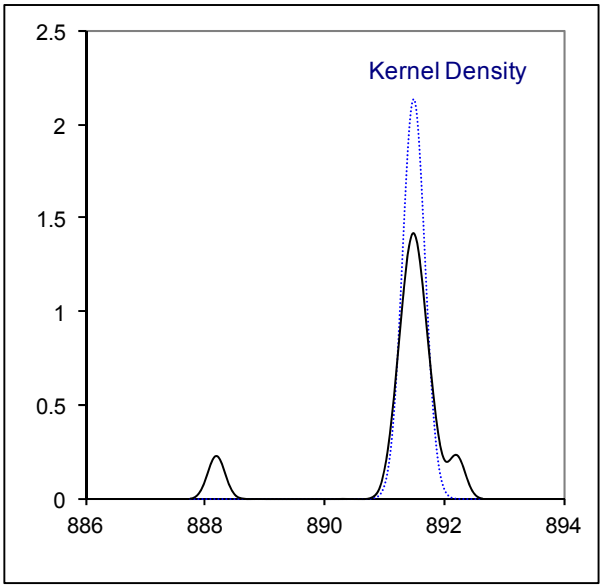
normality	n.a.
n	6
outliers	1
mean (n)	1 (1A+1B)
st.dev. (n)	n.a.
R(calc.)	n.a.
R(D130:12)	n.a.

Determination of Density at 15°C on sample #17035; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D4052	891.8		1.76	
237	D4052	891.7		1.20	
325	D4052	891.54		0.30	
349	D4052	891.3		-1.04	
432	D4052	891.59		0.58	
862	D4052	891.5		0.08	
902	D4052	891.38		-0.59	
963		-----		-----	
1146	D4052	891.38		-0.59	
1174	ISO3675	891.17	C	-1.77	First reported 890.5
1243	ISO12185	892.2	G(0.05)	4.00	
1244		-----		-----	
1748	D4052	891.5		0.08	
6016	D4052	888.2	C,G(0.01)	-18.40	Reported 0.8882 kg/m3

normality	OK
n	10
outliers	2
mean (n)	891.49
st.dev. (n)	0.187
R(calc.)	0.52
R(D4052:16)	0.5

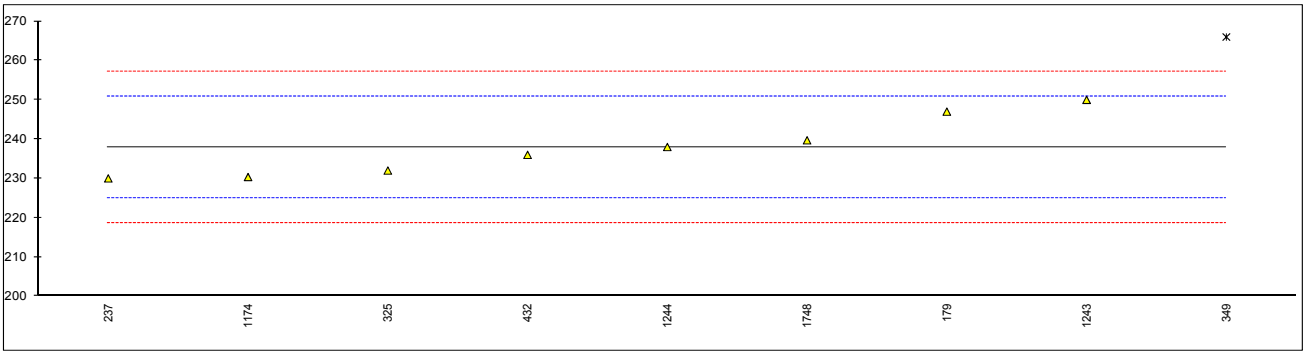


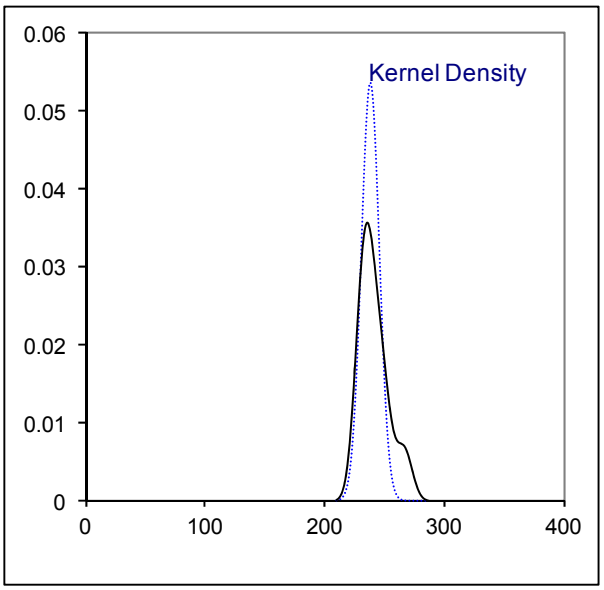


Determination of Flash Point C.O.C. on sample #17035; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D92	247		1.42	
237	D92	230.0	C	-1.23	First reported 192.0
325	D92	232		-0.91	
349	D92	266	G(0.05)	4.37	
432	D92	236		-0.29	
862		-----		-----	
902		-----		-----	
963		-----		-----	
1146		-----		-----	
1174	ISO2592	230.35		-1.17	
1243	ISO2592	250		1.89	
1244	ISO2592	238.0	C	0.02	First reported 208.0
1748	D92	239.7		0.28	
6016		-----		-----	

normality	OK
n	8
outliers	1
mean (n)	237.88
st.dev. (n)	7.467
R(calc.)	20.91
R(D92:16b)	18

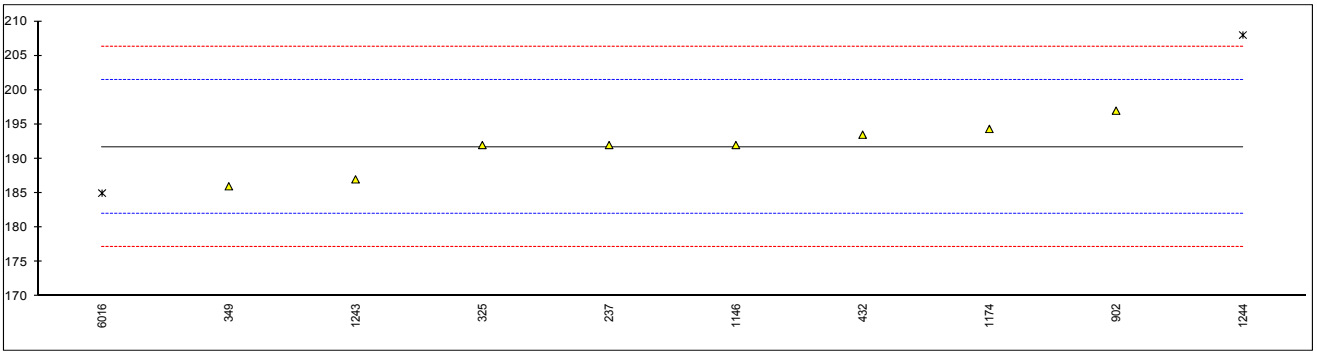


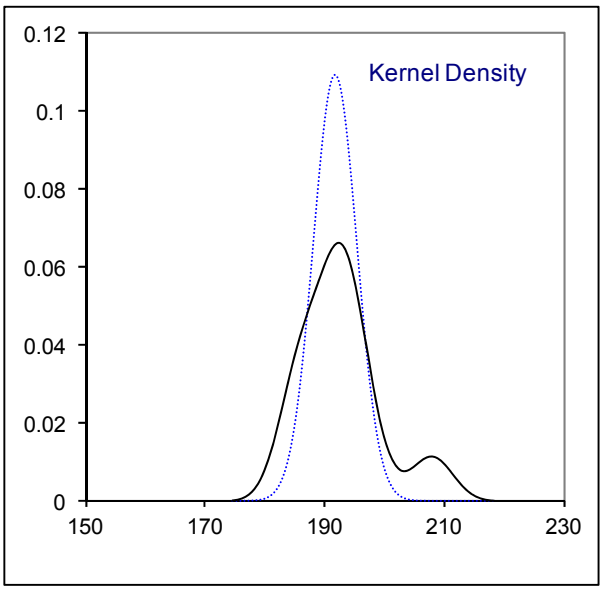


Determination of Flash Point PMcc on sample #17035; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237	D93-A	192.0	C	0.06	First reported 230.0
325	D93-A	192.0		0.06	
349	D93-A	186		-1.18	
432	D93-A	193.5		0.36	
862		-----		-----	
902	D93-A	197.0		1.08	
963		-----		-----	
1146	D93-A	192.0		0.06	
1174	ISO2719-A	194.34		0.54	
1243	ISO2719-A	187		-0.97	
1244	D93-A	208.0	C,G(0.05)	3.35	First reported 238.0
1748		-----		-----	
6016	D93-B	185	ex	-1.38	Test method B is not suitable for fresh oils

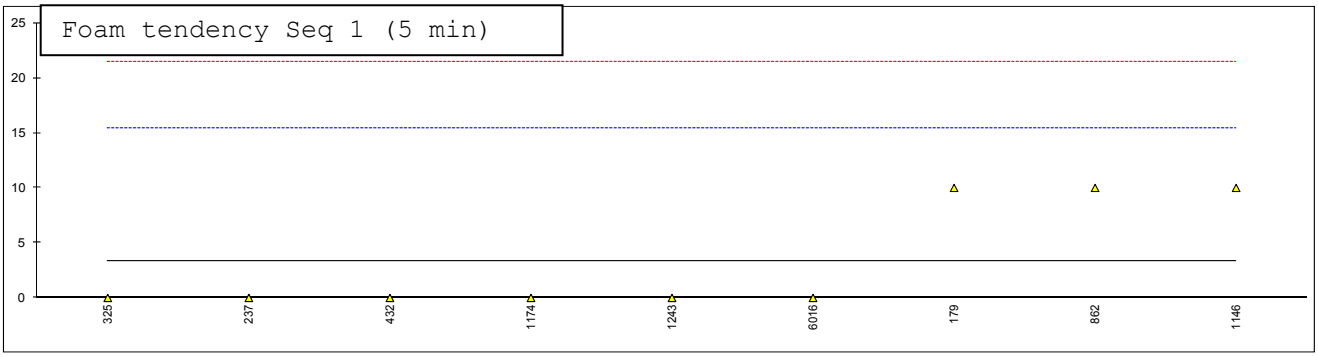
normality	OK
n	8
outliers	1 (+1excl)
mean (n)	191.73
st.dev. (n)	3.648
R(calc.)	10.21
R(D93-A:16a)	13.61





Determination of Foaming Tendency, Sequence I (5 min. blowing period) and Foaming Stability, Sequence I (10 min. settling period) on sample #17035; results in mL

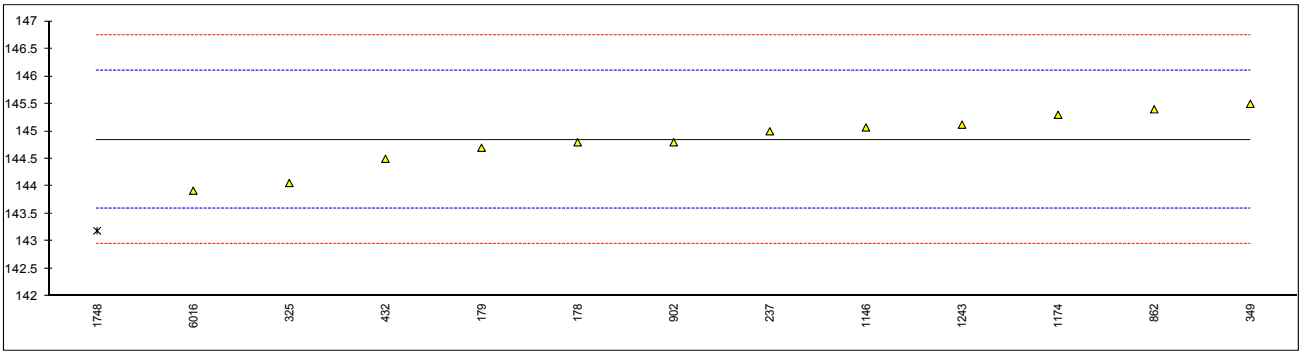
lab	method	Foam Tendency, Seq 1 (5 min)	mark	z(targ)	Foam Stability, Seq 1 (10 min)	mark	z(targ)	remarks
178		-----		-----	-----		-----	
179	D892	10		1.10	0		-----	
237	D892	0		-0.55	0		-----	Heated to 50°C
325	D892	0		-0.55	0		-----	
349		-----		-----	-----		-----	
432	D892	0		-0.55	0		-----	
862	D892	10		1.10	0		-----	
902		-----		-----	-----		-----	
963		-----		-----	-----		-----	
1146	D892	10		1.10	0		-----	
1174	ISO6247	0		-0.55	0		-----	
1243	D892	0		-0.55	0		-----	
1244		-----		-----	-----		-----	
1748		-----		-----	-----		-----	
6016	D892	0		-0.55	0		-----	
	normality	n.a.			n.a.			
	n	9			7			
	outliers	0			0			
	mean (n)	3.33			0			
	st.dev. (n)	5.0			0			
	R(calc.)	14.0			0			
	R(D892:13)	16.9			16			

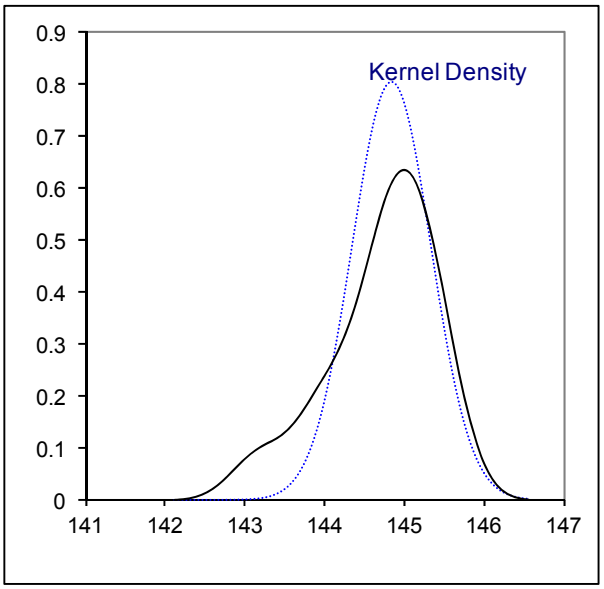


Determination of Kinematic Viscosity at 40°C on sample #17035; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178	D445	144.8		-0.08	
179	D445	144.7		-0.23	
237	D445	145.0		0.24	
325	D445	144.06		-1.25	
349	D445	145.5		1.03	
432	D445	144.5		-0.55	
862	D445	145.4		0.88	
902	D445	144.8		-0.08	
963		-----		-----	
1146	D445	145.07		0.35	
1174	ISO3104	145.3000		0.72	
1243	D7279 to D445	145.12		0.43	
1244		-----		-----	
1748	D7042	143.19	G(0.05)	-2.63	
6016	D7042	143.92		-1.47	

normality	OK
n	12
outliers	1
mean (n)	144.85
st.dev. (n)	0.496
R(calc.)	1.39
R(D445:17)	1.77

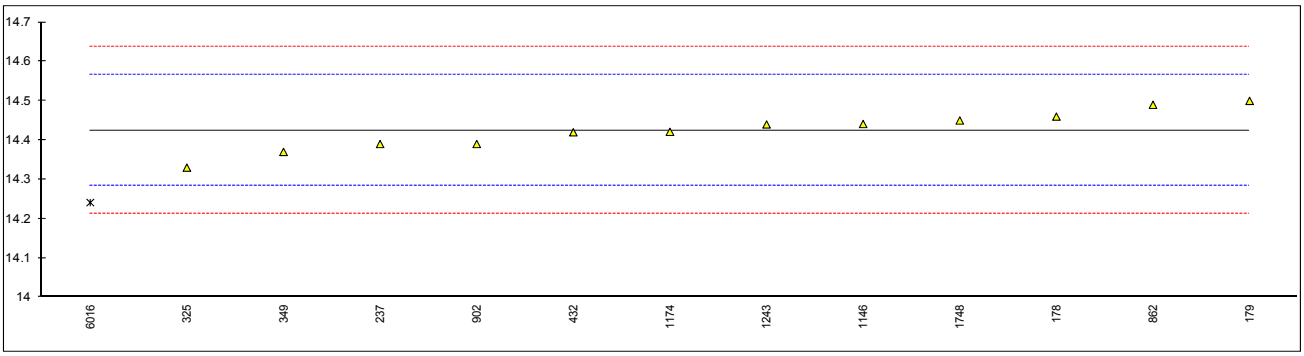


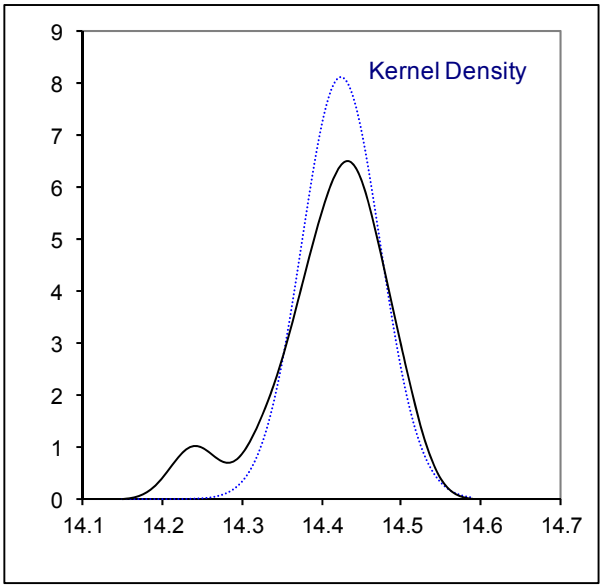


Determination of Kinematic Viscosity at 100°C on sample #17035; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178	D445	14.46		0.49	
179	D445	14.50		1.05	
237	D445	14.39		-0.49	
325	D445	14.33		-1.34	
349	D445	14.37		-0.78	
432	D445	14.42		-0.07	
862	D445	14.49		0.91	
902	D445	14.39		-0.49	
963		-----		-----	
1146	D445	14.441		0.22	
1174	ISO3104	14.4211		-0.06	
1243	D7279 to D445	14.44		0.21	
1244		-----		-----	
1748	D7042	14.45		0.35	
6016	D7042	14.241	G(0.05)	-2.59	

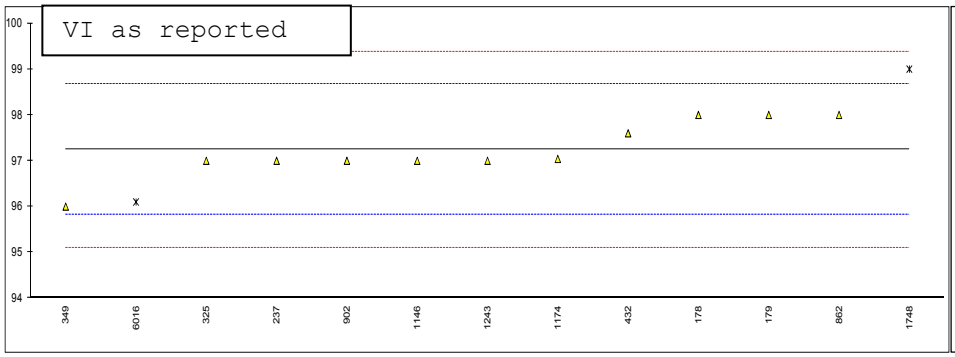
normality	OK
n	12
outliers	1
mean (n)	14.425
st.dev. (n)	0.0493
R(calc.)	0.138
R(D445:17)	0.199

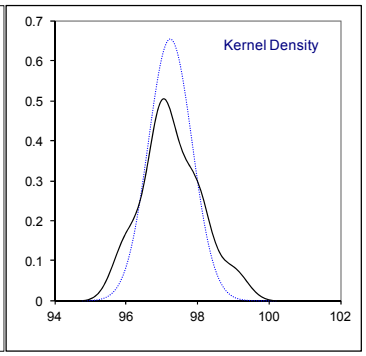


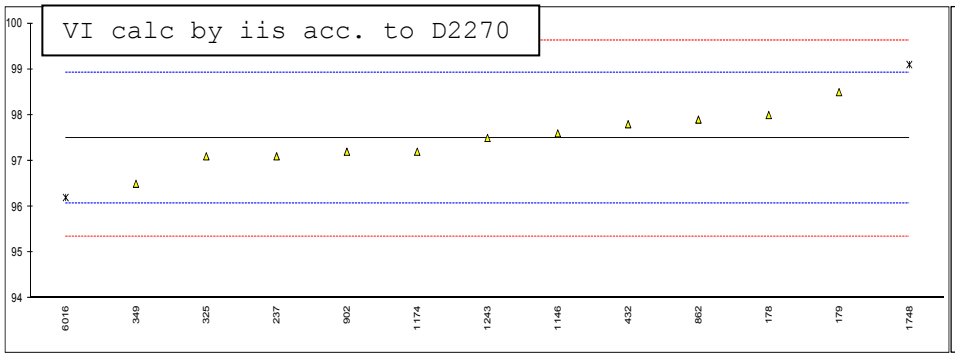


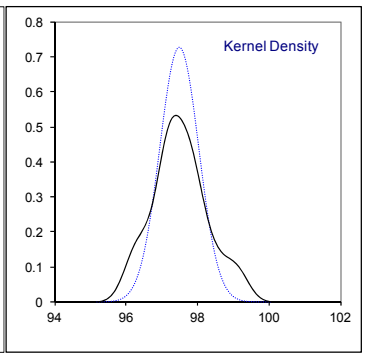
Determination of Viscosity Index (V.I.) on sample #17035

lab	method	value	mark	z(targ)	calc.iis	remarks
178	D2270	98		1.06	98.0	
179	D2270	98		1.06	98.5	
237	D2270	97		-0.34	97.1	
325	D2270	97		-0.34	97.1	
349	D2270	96		-1.74	96.5	
432	D2270	97.6		0.50	97.8	
862	D2270	98		1.06	97.9	
902	D2270	97		-0.34	97.2	
963		-----		-----	-----	
1146	D2270	97		-0.34	97.6	
1174	ISO2909	97.04		-0.28	97.2	
1243	ISO2909	97		-0.34	97.5	
1244		-----		-----	-----	
1748	D2270	99	ex	2.46	99.1	Results excluded, as Kin.Visco 40ºC is an outlier
6016	D2270	96.10	ex	-1.60	96.2	Results excluded, as Kin.Visco 100ºC is an outlier
	normality	OK			OK	
	n	11			11	
	outliers	0 (+2 excl)			0 (+2 excl)	
	mean (n)	97.24			97.49	
	st.dev. (n)	0.611			0.549	
	R(calc.)	1.71			1.54	
	R(D2270:10)	2			2	





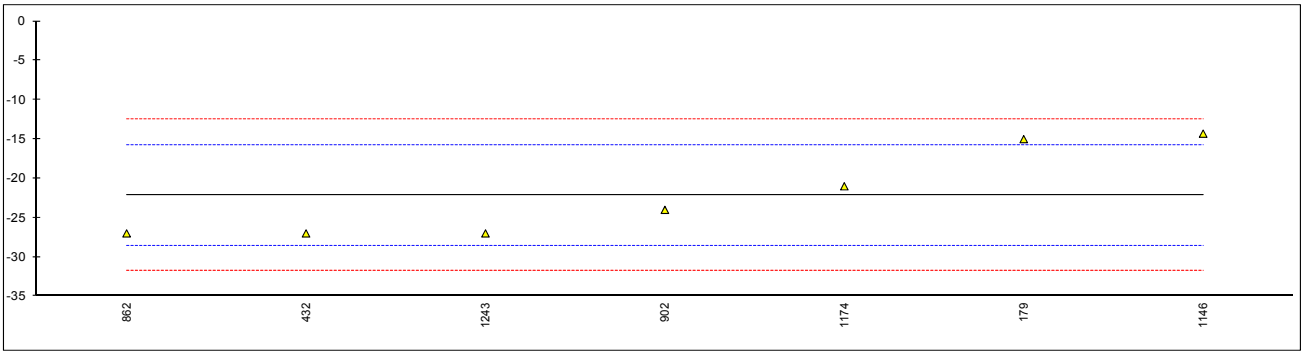




Determination of Pour Point, manual on sample #17035; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D97	-15		2.24	
237		-----		-----	
325		-----		-----	
349		-----		-----	
432	D97	-27		-1.50	
862	D97	-27		-1.50	
902	D97	-24		-0.56	
963		-----		-----	
1146	D97	-14.3		2.45	
1174	ISO3016	-21		0.37	
1243	D97	-27		-1.50	
1244		-----		-----	
1748		-----		-----	
6016		-----		-----	

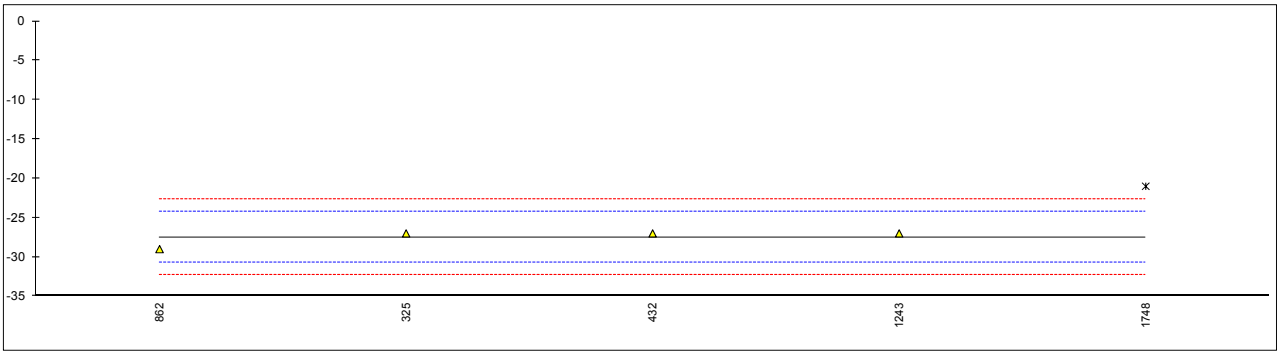
normality	unknown
n	7
outliers	0
mean (n)	-22.19
st.dev. (n)	5.598
R(calc.)	15.68
R(D97:17)	9



Determination of Pour Point, automated, 1°C interval on sample #17035; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
325	D5950	-27		0.31	
349		-----		-----	
432	D5950	-27		0.31	
862	D5950	-29		-0.93	
902		-----		-----	
963		-----		-----	
1146		-----		-----	
1174		-----		-----	
1243	D7346	-27		0.31	
1244		-----		-----	
1748	D5950	-21	D(0.05)	4.04	
6016		-----		-----	

normality	unknown
n	4
outliers	1
mean (n)	-27.5
st.dev. (n)	1.00
R(calc.)	2.8
R(D5950:14)	4.5



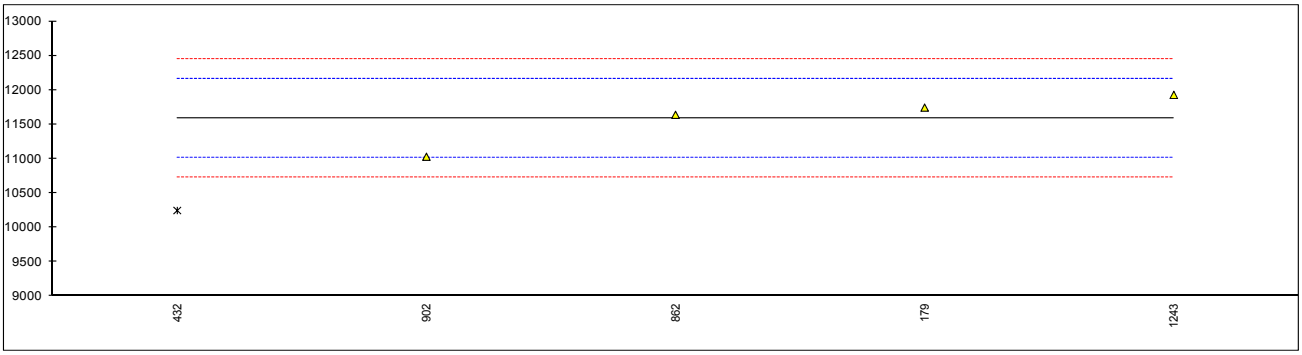
Determination of Rust prevention (procedure A), distilled water on sample #17035

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D665	Pass		-----	
237		-----		-----	
325	D665	PASS		-----	
349		-----		-----	
432		-----		-----	
862	D665	No Rusting		-----	
902		-----		-----	
963		-----		-----	
1146		-----		-----	
1174	ISO7120	pass		-----	
1243		-----		-----	
1244		-----		-----	
1748		-----		-----	
6016	D665	pass		-----	
	normality	unknown			
	n	5			
	outliers	n.a.			
	mean (n)	Pass			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D665:14e1)	n.a.			

Determination of Sulphur on sample #17035; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D4294	11745	C	0.56	First reported 1.1745
237		-----		-----	
325		-----		-----	
349		-----		-----	
432	D4951	10247	D(0.05)	-4.69	
862	D2622	11640		0.19	
902	D4294	11030		-1.95	
963		-----		-----	
1146		-----		-----	
1174		-----		-----	
1243	ISO8754	11930		1.20	
1244		-----		-----	
1748		-----		-----	
6016		-----		-----	

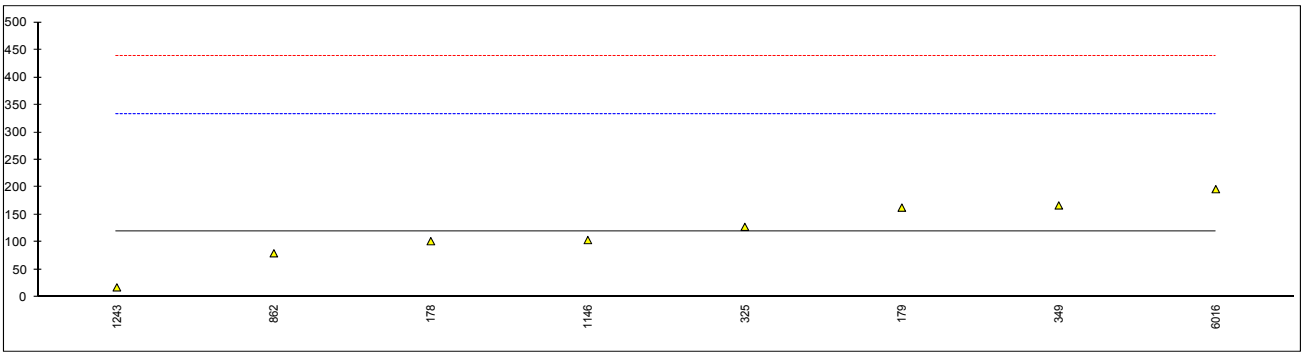
normality	unknown
n	4
outliers	1
mean (n)	11586.3
st.dev. (n)	389.73
R(calc.)	1091.2
R(D4294:16e1)	798.9

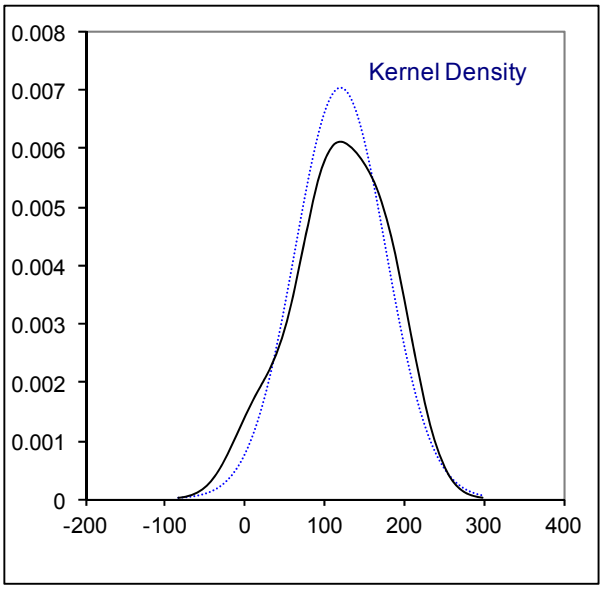


Determination of Water on sample #17035; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D6304-C	102		-0.17	
179	D6304-C	163		0.41	
237		-----		-----	
325	D6304-C	128		0.08	
349	D6304-A	167		0.44	
432		-----		-----	
862	D6304-C	80		-0.37	
902		-----		-----	
963		-----		-----	
1146	D6304-C	104		-0.15	
1174		-----		-----	
1243	DIN51777	18		-0.96	
1244		-----		-----	
1748		-----		-----	
6016	D6304-A	196.5		0.72	

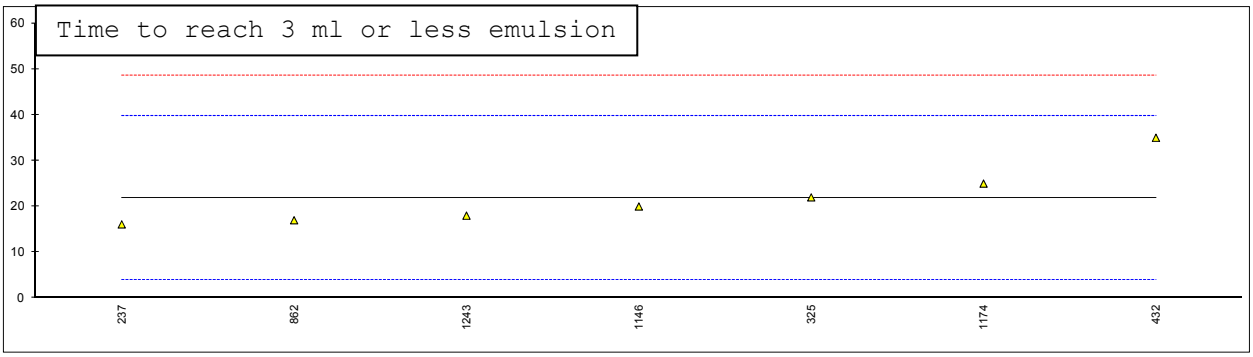
normality	unknown
n	8
outliers	0
mean (n)	119.8
st.dev. (n)	56.77
R(calc.)	158.9
R(D6304:16e1)	298.4

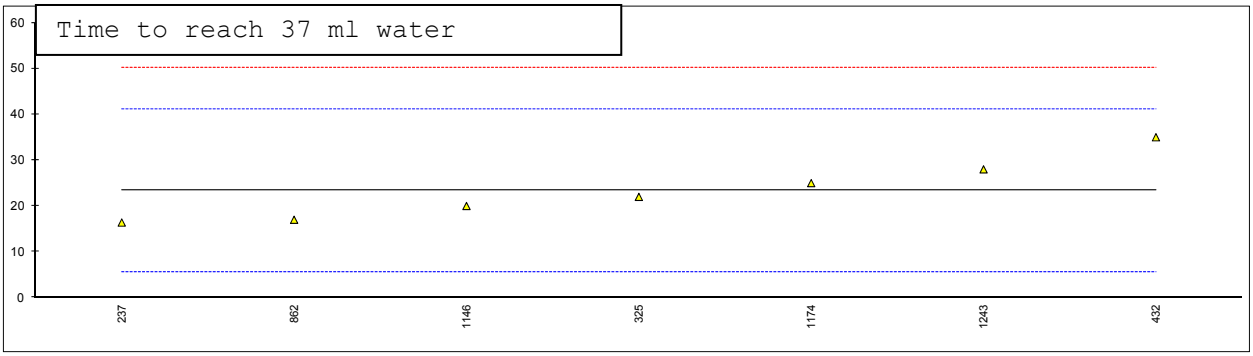




Determination of Water Separability at 82°C, distilled water on sample #17035; results in minutes

lab	method	time to reach 3 ml or less emulsion	z(targ)	time to reach 37 ml of water	z(targ)	time to reach complete break (40-40-0)	z(targ)	test aborted
178		-----	-----	-----	-----	-----		-----
179		-----	-----	-----	-----	-----		-----
237	D1401	16.1	-0.65	16.4	-0.78	24.3		NO
325	D1401	22	0.01	22	-0.15	>60		60
349		-----	-----	-----	-----	-----		-----
432	D1401	35	1.47	35	1.31	>60		-----
862	D1401	17	-0.55	17	-0.71	20		-----
902		-----	-----	-----	-----	-----		-----
963		-----	-----	-----	-----	-----		-----
1146	D1401	20	-0.21	20	-0.37	-----		-----
1174	ISO6614	25	0.35	25	0.19	> 60		-----
1243	DIN/ISO6614	18	-0.43	28	0.52	-----		-----
1244		-----	-----	-----	-----	-----		-----
1748		-----	-----	-----	-----	-----		-----
6016		-----	-----	-----	-----	-----		-----
	normality	unknown		unknown		unknown		
	n	7		7		5		
	outliers	0		0		n.a.		
	mean (n)	21.87		23.34		n.a.		
	st.dev. (n)	6.554		6.605		n.a.		
	R(calc.)	18.35		18.49		n.a.		
	R(D1401:12e1)	25		25		n.a.		

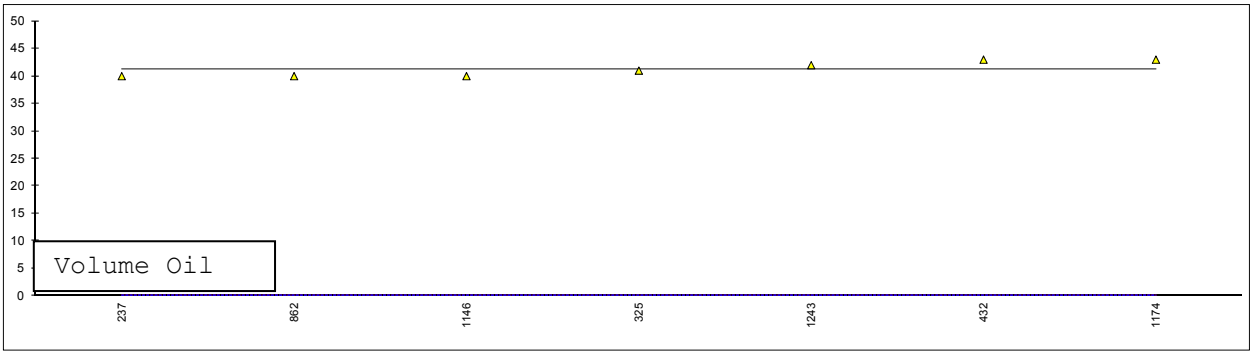


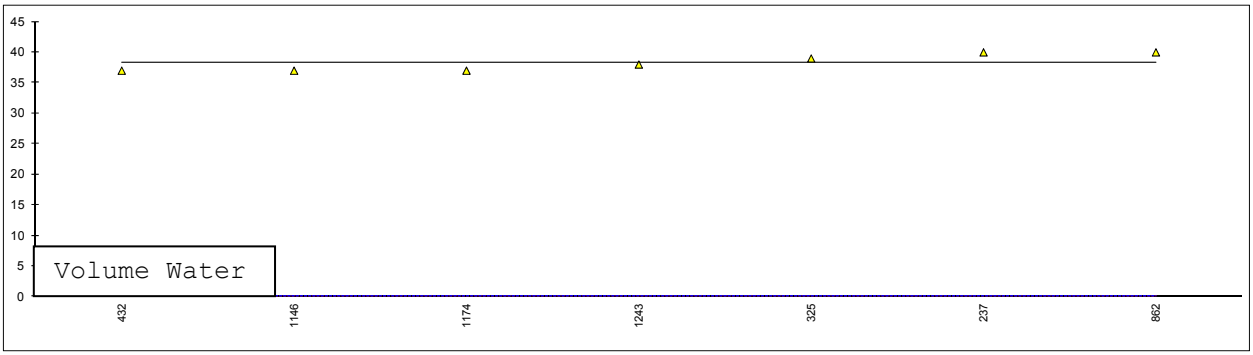


Determination of Water Separability at 82°C, distilled water on sample #17035; results in ml

--- Continued ---

lab	method	volume oil phase	mark	volume water phase	mark	volume emulsion phase	mark	remarks
178		-----		-----		-----		
179		-----		-----		-----		
237		40		40		0		
325		41		39		0		
349		-----		-----		-----		
432		43		37		0		
862		40		40		0		
902		-----		-----		-----		
963		-----		-----		-----		
1146		40		37		3		
1174		43		37		0		
1243		42		38		0		
1244		-----		-----		-----		
1748		-----		-----		-----		
6016		-----		-----		-----		
	normality	unknown		unknown		unknown		
	n	7		7		7		
	outliers	0		0		0		
	mean (n)	41.3		38.3		0.4		
	st.dev. (n)	1.38		1.38		1.13		
	R(calc.)	3.9		3.9		3.2		
	R()	n.a.		n.a.		n.a.		
Compare	R(iis16L01)	1.4		1.5		1.1		





Determination of Calcium (Ca) on sample #17035; results in mg/kg

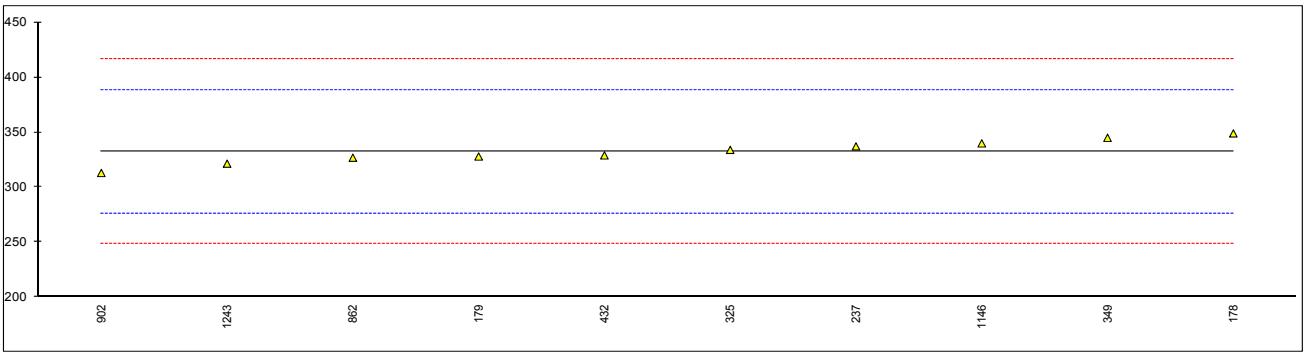
lab	method	value	mark	z(targ)	remarks
178		<1		-----	
179	D5185	1		-----	
237	D5185	<5		-----	
325	D5185	0		-----	
349	D5185	0		-----	
432	D4951	<1		-----	
862	D5185	<0.1		-----	
902	D5185	<40		-----	
963		-----		-----	
1146	INH-5185	0.7543		-----	
1174		-----		-----	
1243	DIN51399-1	<0,035		-----	
1244		-----		-----	
1748		-----		-----	
6016		-----		-----	
	normality	unknown			
	n	9			
	outliers	n.a.			
	mean (n)	<5			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D5185:13e1)	n.a.			Application range 40 – 9000 mg/kg

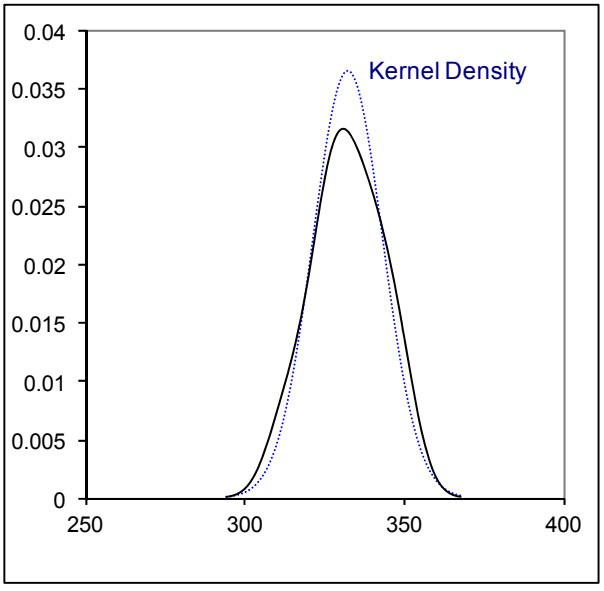
Determination of Phosphorus (P) on sample #17035; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		349		0.59	
179	D5185	328		-0.16	
237	D5185	337.2		0.17	
325	D5185	334		0.06	
349	D5185	345		0.45	
432	D4951	329		-0.12	
862	D5185	326.9		-0.19	
902	D5185	313		-0.69	
963		-----		-----	
1146	INH-5185	339.9		0.27	
1174		-----		-----	
1243	DIN51399-1	321.5		-0.39	
1244		-----		-----	
1748		-----		-----	
6016		-----		-----	

normality	OK
n	10
outliers	0
mean (n)	332.35
st.dev. (n)	10.916
R(calc.)	30.57
R(D5185:13e1)	78.39

Application range 10 -1000 mg/kg





Determination of Zinc (Zn) on sample #17035; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		<1		-----	
179	D5185	<1		-----	
237	D5185	<5		-----	
325	D5185	0		-----	
349	D5185	0		-----	
432	D4951	<1		-----	
862	D5185	0.4		-----	
902	D5185	<60		-----	
963		-----		-----	
1146	INH-5185	2.467		-----	
1174		-----		-----	
1243	DIN51399-1	<0,013		-----	
1244		-----		-----	
1748		-----		-----	
6016		-----		-----	
	normality	unknown			
	n	9			
	outliers	n.a.			
	mean (n)	<5			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D5185:13e1)	n.a.			Application range 60 – 1600 mg/kg

APPENDIX 2

Number of participants per country

1 lab in AUSTRIA
1 lab in BELGIUM
1 lab in BULGARIA
1 lab in CHINA, People's Republic
1 lab in GERMANY
1 lab in JORDAN
1 lab in KAZKHSTAN
1 lab in NETHERLANDS
1 lab in NIGERIA
1 lab in SAUDI ARABIA
1 lab in SOUTH KOREA
1 lab in SPAIN
1 lab in TURKEY
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 probably reported 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
SDS	= Safety Data Sheet

Literature:

1	iis Interlaboratory Studies, Protocol for the Organization, Statistics and Evaluation, March 2017
2	ASTM E178:08
3	ISO 5725:86
4	ISO 5725, parts 1-6, 1994
5	ISO13528:05
6	ISO17043:10
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