

**Results of Proficiency Test
Gear Oil (fresh)
April 2016**

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. A.S. Noordman – de Neef
Correctors: dr. R.G. Visser & ing. R.J. Starink
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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 2015/2016 it was decided to continue with the round robin for the analyses of fresh Gear Oil. In this interlaboratory study 18 laboratories in 14 different countries have registered for participation. See appendix 2 for the number of participants per country.

In this report, the results of the 2016 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 accredited laboratory. It was decided to send one bottle of 1L (labelled #16035) of 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 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC 17043:2010. 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 a 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 Organization, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol is available from 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 150 litres bulk material was homogenized and part of this bulk was transferred into 34 brown glass bottles of 1 litre (labelled #16035). The homogeneity of the subsamples #16035 was checked by determination of Density at 15°C in accordance with ASTM D4052 on 4 stratified randomly selected samples.

	Density at 15 °C in kg/m ³
Sample #16035-1	887.00
Sample #16035-2	886.99
Sample #16035-3	887.00
Sample #16035-4	887.00

Table 1: homogeneity test results of subsamples #16035

From the above test results the repeatability was calculated and compared with 0.3 times the corresponding reproducibility of the reference test method in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Density at 15 °C in kg/m ³
r (observed)	0.01
reference test method	ASTM D4052:15
0.3 x R(ref. test method)	0.16

Table 2: evaluation of the repeatability of the subsamples #16035

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

To each of the participating laboratories, one sample of 1 L in a brown glass bottle (labelled #16035) was sent on March 16, 2016.

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 requested to determine on sample #16035: Acid Number (Total), Copper Corrosion, Density at 15°C, Flash Point 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 separability, Calcium, Phosphorus and Zinc.

To get comparable test results a detailed report form, on which the units were prescribed as well as the reference test methods and a letter of instructions were prepared and made

available on the data entry portal www.kpmd.co.uk/sgs-iis/. A SDS and a form to confirm receipt of the samples were added to the sample package.

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 April 2014 (iis-protocol, version 3.3).

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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the test results 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.

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:

$$z_{(\text{target})} = (\text{test result} - \text{average of PT}) / \text{target standard deviation}$$

The $z_{(\text{target})}$ 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, except for one participant in Brazil. Most participants reported test results in time, one participant reported test results after the final reporting date.

In total 18 participants reported 215 test results. Observed were 14 outlying test results, which is 6.5% 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 not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D664:11a.

Copper Corrosion: Eleven laboratories reported a test result. No problems have been observed. The participants agreed on classification 1.

Density at 15°C: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of ASTM D4052:15.

Flash Point PMcc: This determination was problematic. No statistical outliers were observed. Three test results were excluded, test method ASTM D93B is not meant for fresh oils. The calculated reproducibility after rejection of the suspect data is not in agreement with ASTM D93A:15a.

Foaming Characteristics (Tendency and Stability): This determination was not problematic. One statistical outlier was observed in Foam Tendency. However, the calculated reproducibilities after rejection of the statistical outlier are in agreement with the requirements of ASTM D892:13.

Kinematic Viscosity at 40°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D445:15a.

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 full agreement with the requirements of ASTM D445:15a.

Viscosity Index: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with ASTM D2270:10e1. Also iis calculated the Viscosity Index from the test results reported for the kinematic viscosities at 40°C and 100°C. These calculated test results were compared to the reported test results. The test results of two participants were slightly different.

Pour Point manual: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in full agreement with requirements of ASTM D97:16.

Pour Point automated: This determination may be problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is just not in agreement with requirements of ASTM D5950:14. The low number of test results may (partly) explain the slightly higher reproducibility.

Rust prevention: Regretfully, only two participants reported a test result. Both participants reported the test as "Pass".

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

Water separability The determination may not be problematic. Only seven participants reported test results. One statistical outlier was observed in volume oil, volume water and volume emulsion, all from the same participant. However, the calculated reproducibilities are in agreement with the requirements of ASTM D1401:12e1. For the test result 'time aborted' different interpretations of the test results were reported, e.g. "No" or "Yes" while complete break (40-40-0) was reported. Test method ASTM D1401 describes complete break as '40-40-0' when no emulsion layer was present. However, a complete break was also reported when a small emulsion layer was present, for example lab 432; o-w-e(t) '41-39-1(15)'.

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. However, two outliers were observed.

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. No outliers were observed.

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 ($2.8 \cdot sd$) 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	14	0.39	0.17	0.20
Copper Corrosion, 3hrs at 100°C	rating	11	1	n.a.	n.a.
Density at 15°C	kg/m ³	15	887.05	0.50	0.52
Flash Point PMcc	°C	13	201	16	14
Foaming Tendency (Seq I)	mL	7	0	0	16
Foaming Stability (Seq I)	mL	7	0	0	16
Kinematic Viscosity at 40°C	mm ² /s	18	218.6	2.5	2.7
Kinematic Viscosity at 100°C	mm ² /s	16	19.28	0.15	0.27
Viscosity Index		16	99.6	1.7	2.0
Pour Point, Manual	°C	9	-14	9	9
Pour Point, Automated	°C	8	-14	6	5
Rust Prevention (proc. A)		2	pass	n.a.	n.a.
Sulphur	mg/kg	10	6757	553	564
Water Separability at 82°C, distilled water					
- Time ≤ 3 ml emulsion	min.	6	13	6	25
- Time 37 ml water	min.	6	13	6	25
- Time to complete break	min.	5	16	4	n.a.
- Volume Oil phase	mL	6	40.3	1.4	n.a.
- Volume Water phase	mL	6	39.5	1.5	n.a.
- Volume Emulsion phase	mL	6	0.2	1.1	n.a.
Calcium as C	mg/kg	9	0.9	0.6	n.a.
Phosphorus as P	mg/kg	13	275	46	71
Zinc as Zn	mg/kg	9	0.6	1.3	n.a.

Table 3: reproducibilities of the test results of sample #16035

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 2016 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 2016	April 2015
Acid Number (Total)	+	+
Copper Corrosion, 3hrs at 100°C	n.e.	n.e.
Density at 15 °C	+/-	-
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.
Sulphur	+/-	-
Water Separability at 82°C	++	++
Calcium as C	n.e.	n.e.
Phosphorus as P	+	--
Zinc as Zn	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:

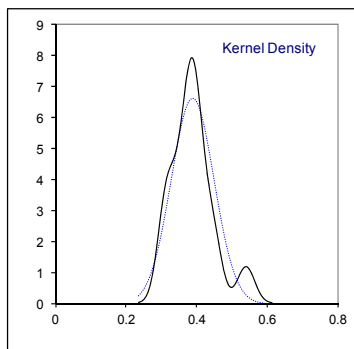
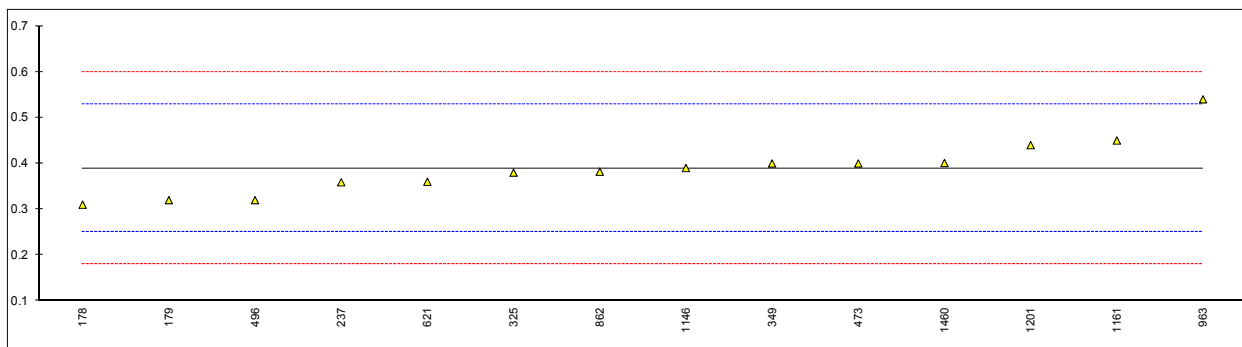
- ++: group performed much better than the reference test method
- + : group performed better than the reference test method
- +/-: group performance equals the reference test method
- : group performed worse than the reference test method
- : group performed much worse than the reference test method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

APPENDIX 1

Determination of Acid Number (Total) on sample #16035; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
178	D664Mod.	0.31		-1.14	
179	D664	0.32		-0.99	
237	D974	0.359		-0.43	
315		----		----	
325	D664	0.38		-0.13	
349	D664	0.40		0.15	
432		----		----	
473	D664	0.4		0.15	
496	D664	0.32		-0.99	
551		----		----	
621	D664	0.36		-0.42	
862	D664	0.382		-0.11	
963	D664	0.54		2.15	
1146	D664	0.390		0.01	
1161	D664	0.45		0.87	
1201	D664	0.44		0.72	
1460	D664	0.401		0.17	
1748		----		----	

normality not OK
n 14
outliers 0
mean (n) 0.389
st.dev. (n) 0.0602
R(calc.) 0.169
R(D664:11a) 0.196

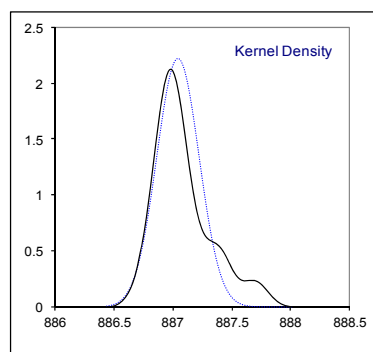
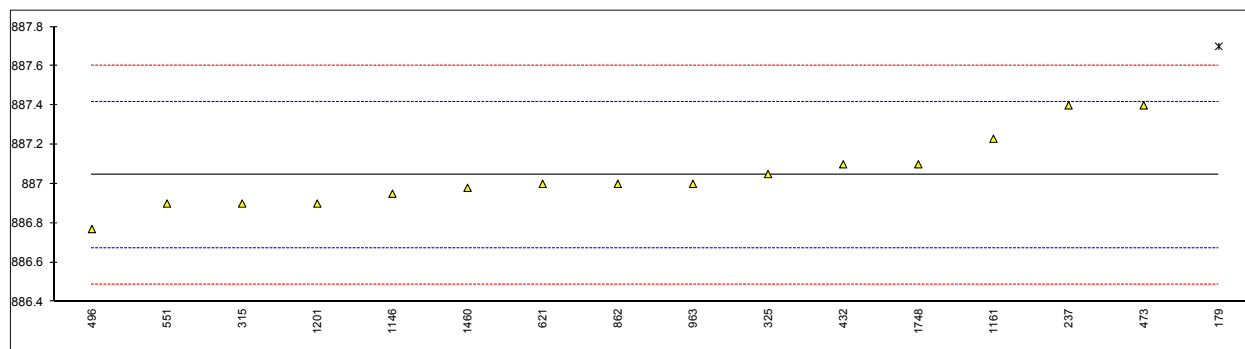


Determination of Copper Corrosion 3 hours at 100°C on sample #16035;

lab	method	value	mark	z(targ)	remarks
178		----		----	
179	D130	1A		----	
237	D130	1A		----	
315	D130	1A		----	
325	D130	1A		----	
349		----		----	
432		----		----	
473		----		----	
496	D130	1b		----	
551	D130	1A		----	
621	D130	1A		----	
862	D130	1a		----	
963		----		----	
1146		----		----	
1161	ISO2160	1A		----	
1201	D130	1a		----	
1460	D130	1a		----	
1748		----		----	
	normality	n.a.			
	n	11			
	outliers	0			
	mean (n)	1			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D130:12)	n.a.			

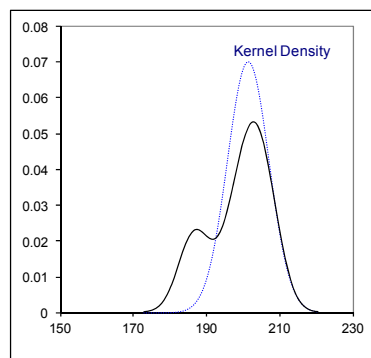
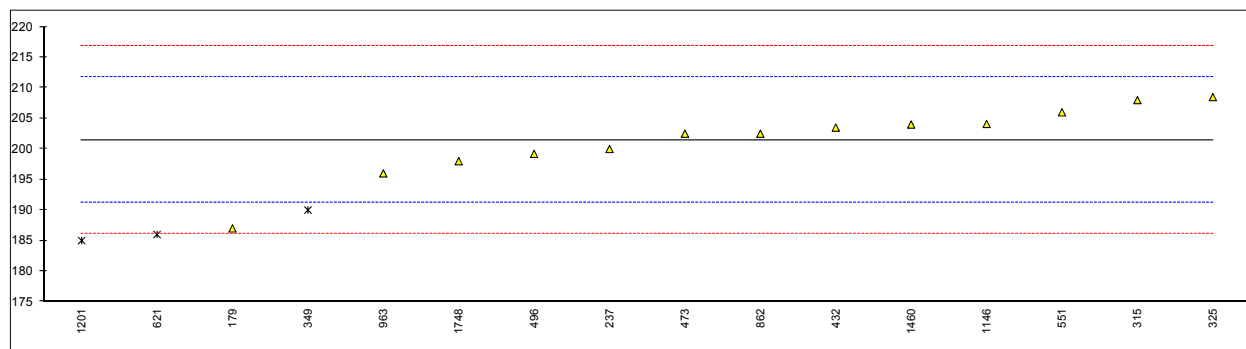
Determination of Density at 15°C on sample #16035; results in kg/m³

lab	method	value	mark	z(targ)	remarks
178		----		----	
179	D4052	887.7	D(0.05)	3.53	
237	D4052	887.4		1.91	
315	D4052	886.9		-0.78	
325	D4052	887.05		0.03	
349		----		----	
432	D4052	887.10		0.29	
473	D4052	887.4		1.91	
496	D4052	886.77		-1.48	
551	D4052	886.9		-0.78	
621	D4052	887.0		-0.24	
862	D4052	887.0		-0.24	
963	D4052	887.0	C	-0.24	first reported 0.8870 kg/m ³
1146	ISO12185	886.95		-0.51	
1161	D4052	887.23		0.99	
1201	D4052	886.9		-0.78	
1460	D4052	886.98		-0.35	
1748	D4052	887.1	C	0.29	first reported 0.8871 kg/m ³
normality		OK			
n		15			
outliers		1			
mean (n)		887.0453			
st.dev. (n)		0.17936			
R(calc.)		0.5022			
R(D4052:15)		0.5200			



Determination of Flash Point PMcc on sample #16035; results in °C

lab	method	value	mark	z(targ)	remarks
178		----		----	
179	D93-A	187.0		-2.84	
237	D93-A	200.0		-0.29	
315	D93-A	208.0		1.28	
325	D93-A	208.5		1.37	
349	D93-B	190	ex	-2.25	test method B not suitable for fresh oils
432	D93-A	203.5		0.39	
473	D93-A	202.5		0.20	
496	D93-A	199.2		-0.45	
551	D93	206		0.88	
621	D93-B	186.0	ex	-3.03	test method B not suitable for fresh oils
862	D93-A	202.5		0.20	
963	D93-A	196		-1.07	
1146	In house	204.1		0.51	
1161		----		----	
1201	D93-B	185.0	ex	-3.23	test method B not suitable for fresh oils
1460	D93-A	204		0.49	
1748	D93-A	198		-0.68	
normality		not OK			
n		13			
outliers		0+3ex			
mean (n)		201.48			
st.dev. (n)		5.707			
R(calc.)		15.98			
R(D93-A:15a)		14.31			



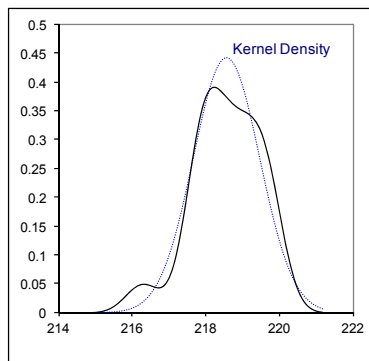
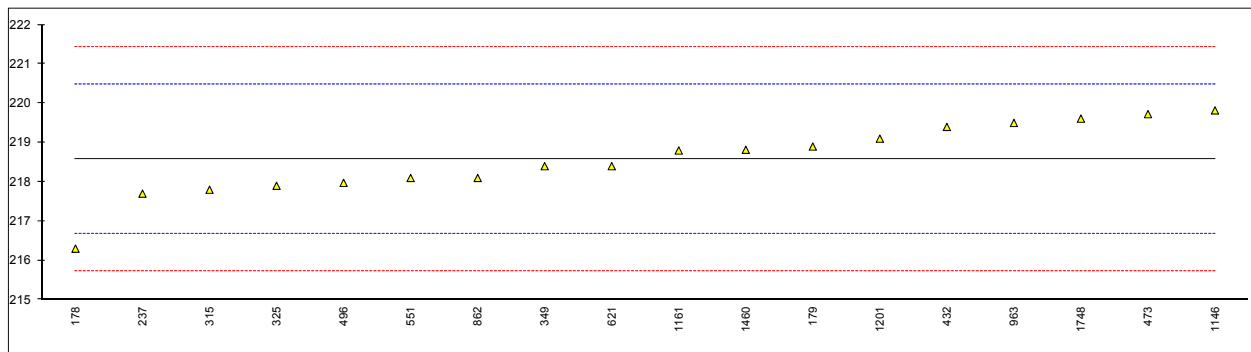
Determination of Foaming Tendency, Sequence I (5 min. blowing period) and Foam Stability, Sequence I (10 min. settling period) on sample #16035; results in mL

lab	method	Foam Tendency, Seq 1 (5 min)	mark	z(targ)	Foam Stability, Seq 1 (10 min)	mark	z(targ)	remarks
178		----		----	----		----	
179	D892	0		0.00	0		0.00	
237		----		----	----		----	
315	D892	0		0.00	0		0.00	
325	D892	0		0.00	0		0.00	
349		----		----	----		----	
432	D892	0		0.00	0		0.00	
473		----		----	----		----	
496	D892	0		0.00	0		0.00	
551		----		----	----		----	
621		----		----	----		----	
862		----		----	----		----	
963		----		----	----		----	
1146	D892	0		0.00	0		0.00	
1161	D892	10	D(0.01)	1.76	----		----	
1201		----		----	----		----	
1460	D892	0		0.00	0		0.00	
1748		----		----	----		----	
	normality	n.a.			n.a.			
	n	7			7			
	outliers	1			0			
	mean (n)	0			0			
	st.dev. (n)	0			0			
	R(calc.)	0			0			
	R(D892:13)	15.95			15.95			

Determination of Kinematic Viscosity at 40°C on sample #16035; results in mm²/s

lab	method	value	mark	z(targ)	remarks
178	D445	216.3		-2.39	
179	D445	218.9		0.34	
237	D445	217.7		-0.92	
315	D445	217.8		-0.81	
325	D445	217.9		-0.71	
349	D445	218.4		-0.18	
432	D445	219.4		0.87	
473	D7042	219.725		1.21	
496	D445	217.975		-0.63	
551	D445	218.1		-0.50	
621	D445	218.4		-0.18	
862	D445	218.1		-0.50	
963	D445	219.5		0.97	
1146	D445	219.82		1.31	
1161	ISO3104	218.8		0.24	
1201	D445	219.1		0.55	
1460	D445	218.817275		0.25	
1748	D7042	219.61		1.09	

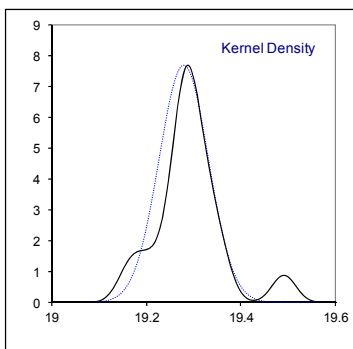
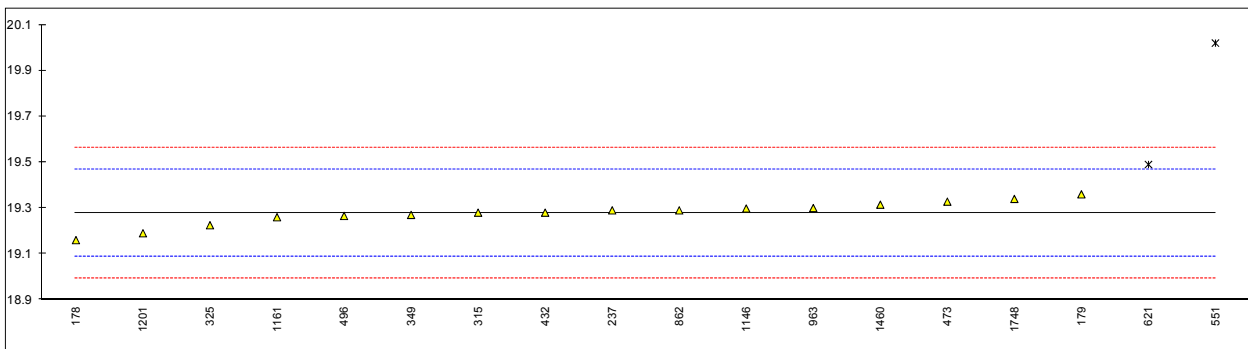
normality OK
n 18
outliers 0
mean (n) 218.57
st.dev. (n) 0.902
R(calc.) 2.53
R(D445:15a) 2.67



Determination of Kinematic Viscosity at 100°C on sample #16035; results in mm²/s

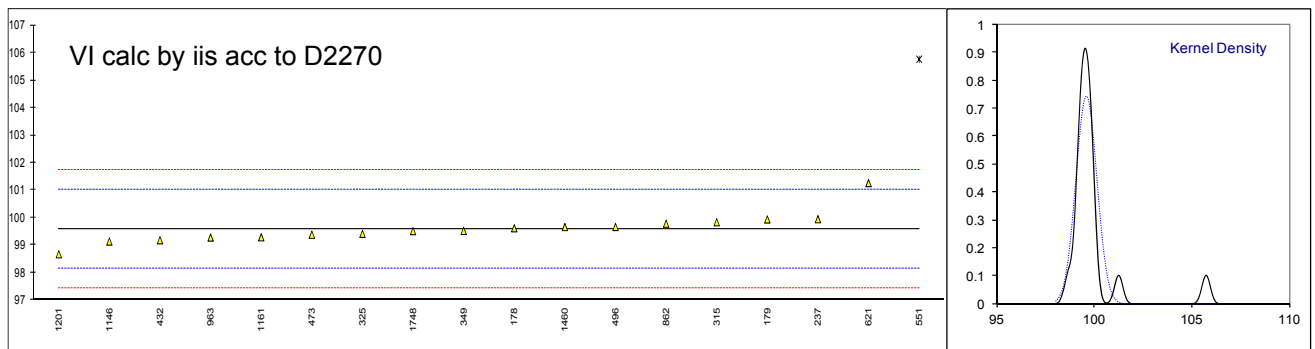
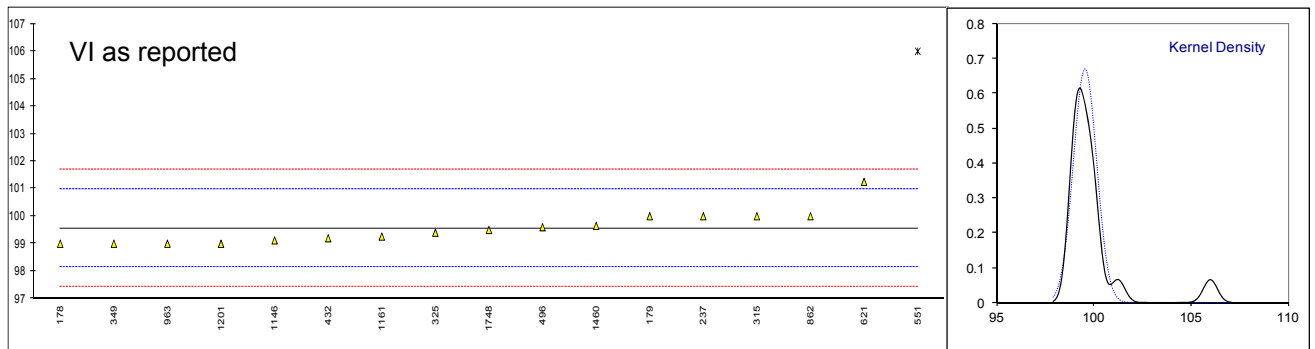
lab	method	value	mark	z(targ)	remarks
178	D445	19.16		-1.24	
179	D445	19.36		0.86	
237	D445	19.29		0.12	
315	D445	19.28		0.02	
325	D445	19.225		-0.56	
349	D445	19.27		-0.09	
432	D445	19.28		0.02	
473	D7042	19.328		0.52	
496	D445	19.2655		-0.13	
551	D445	20.02	D(0.01)	7.81	
621	D445	19.49	D(0.05)	2.23	
862	D445	19.29		0.12	
963	D445	19.30		0.23	
1146	D445	19.298		0.21	
1161	ISO3104	19.26	C	-0.19	first reported 19.87
1201	D445	19.19		-0.93	
1460	D445	19.314682		0.38	
1748	D7042	19.34		0.65	

normality OK
n 16
outliers 2
mean (n) 19.2782
st.dev. (n) 0.05203
R(calc.) 0.1457
R(D445:15a) 0.2660



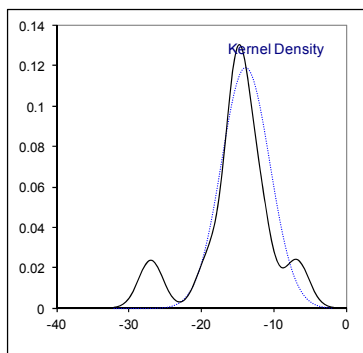
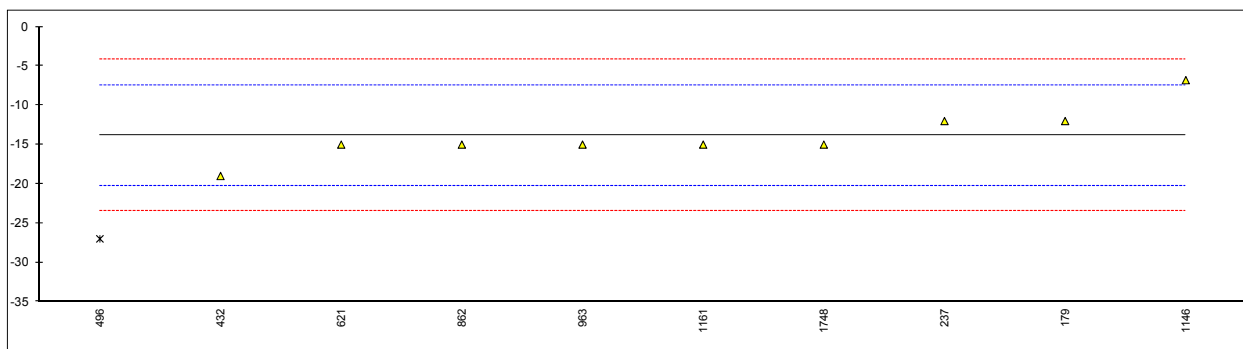
Determination of Viscosity Index (V.I.) on sample #16035

lab	method	value	mark	z(targ)	calc. iis	mark	remarks
178	D2270	99		-0.79	99.62		iis calculated slightly different V.I.
179	D2270	100		0.61	99.94		
237	D2270	100		0.61	99.95		
315	D2270	100		0.61	99.84		
325	D2270	99.4		-0.23	99.41		
349	D2270	99		-0.79	99.52		iis calculated slightly different V.I.
432	D2270	99.2		-0.51	99.18		
473		----		----	99.38		
496	D2270	99.6		0.05	99.66		
551	D2270	106	D(0.01)	9.01	105.76	D(0.01)	
621	D2270	101.25		2.36	101.26		
862	D2270	100		0.61	99.78		
963	D2270	99		-0.79	99.28		
1146	D2270	99.12		-0.62	99.14		
1161	D2270	99.26	C	-0.42	99.29		first reported 104
1201	D2270	99		-0.79	98.67		
1460	D2270	99.65		0.12	99.66		
1748	D2270	99.5		-0.09	99.51		
normality		not OK			not OK		
n		16			17		
outliers		1			1		
mean (n)		99.561			99.594		
st.dev. (n)		0.5965			0.5378		
R(calc.)		1.670			1.506		
R(D2270:10)		2.000			2.000		



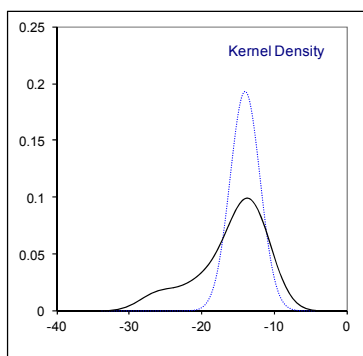
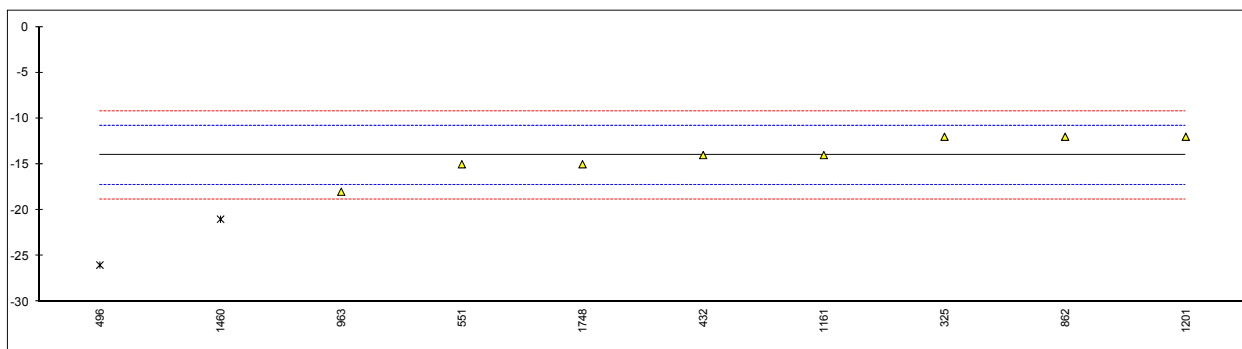
Determination of Pour Point, manual on sample #16035; results in °C

lab	method	value	mark	z(targ)	remarks
178		----		----	
179	D97	-12		0.58	
237	D97	-12		0.58	
315		----		----	
325		----		----	
349		----		----	
432	D97	-19		-1.60	
473		----		----	
496	D97	-27	D(0.05)	-4.09	
551		----		----	
621	D97	-15.0		-0.35	
862	D97	-15		-0.35	
963	D97	-15		-0.35	
1146	D97	-6.8		2.20	
1161	D97	-15		-0.35	
1201		----		----	
1460		----		----	
1748	D97	-15		-0.35	
normality		not OK			
n		9			
outliers		1			
mean (n)		-13.87			
st.dev. (n)		3.348			
R(calc.)		9.37			
R(D97:16)		9.00			



Determination of Pour Point, automated, 1°C interval on sample #16035; results in °C

lab	method	value	mark	z(targ)	remarks
178		----		----	
179		----		----	
237		----		----	
315		----		----	
325	D5950	-12		1.24	
349		----		----	
432	D5950	-14		0.00	
473		----		----	
496	D6892	-26	DG(0.05)	-7.47	
551	D5950	-15		-0.62	
621		----		----	
862	D5950	-12		1.24	
963	D5950	-18		-2.49	
1146		----		----	
1161	D6749	-14		0.00	
1201	D5950	-12		1.24	
1460	D5949	-21	DG(0.05)	-4.36	
1748	D7346	-15		-0.62	
normality		OK			
n		8			
outliers		2			
mean (n)		-14.0			
st.dev. (n)		2.07			
R(calc.)		5.8			
R(D5950:14)		4.5			



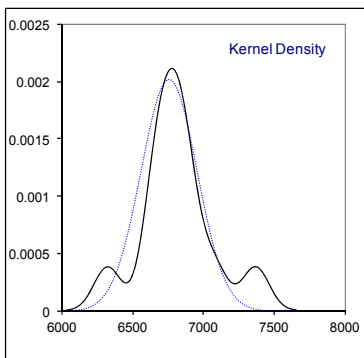
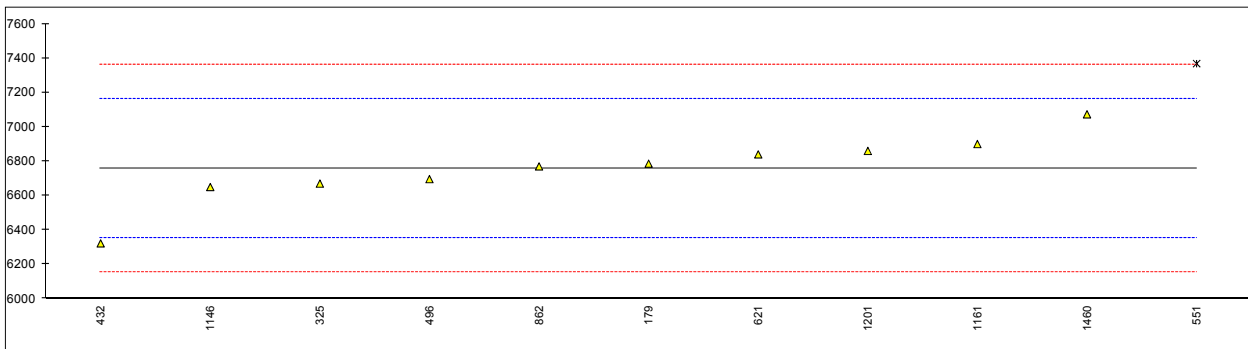
Determination of Rust prevention (procedure A), distilled water on sample #16035

lab	method	value	mark	z(targ)	remarks
178		----		----	
179		----		----	
237		----		----	
315	D665	pass		----	
325		----		----	
349		----		----	
432		----		----	
473		----		----	
496		----		----	
551		----		----	
621		----		----	
862		----		----	
963		----		----	
1146		----		----	
1161		----		----	
1201		----		----	
1460	D665	Pass		----	
1748		----		----	
	normality	unknown			
	n	2			
	outliers	n.a.			
	mean (n)	n.a.			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D665:14e1)	n.a.			

Determination of Sulphur on sample #16035; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		----		----	
179	D4294	6786		0.15	
237		----		----	
315		----		----	
325	D6443Mod.	6670		-0.43	
349		----		----	
432	D4951	6322		-2.16	
473		----		----	
496	D2622	6696		-0.30	
551	D4294	7368	D(0.05)	3.03	
621	D4294	6840		0.41	
862	D2622	6770		0.07	
963		----		----	
1146	ISO8754	6650		-0.53	
1161	ISO8754	6900		0.71	
1201	D4294	6860	C	0.51	first reported 0.686 mg/kg
1460	D4294	7073	C	1.57	first reported 0.7073 mg/kg
1748		----		----	

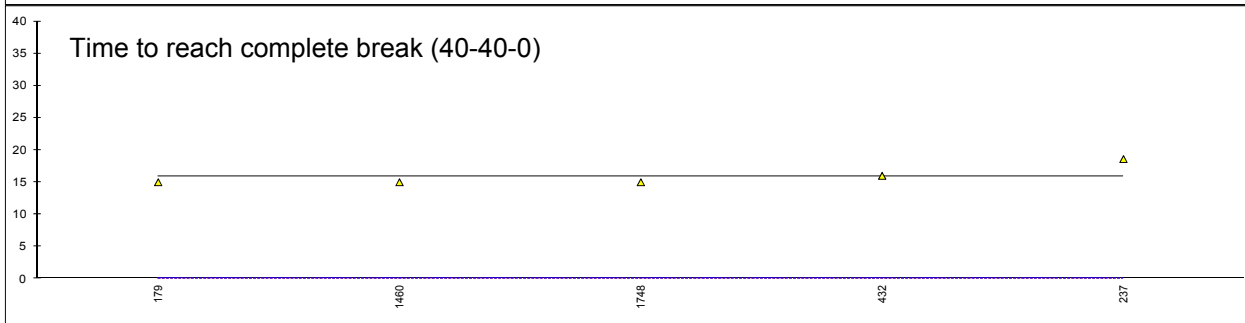
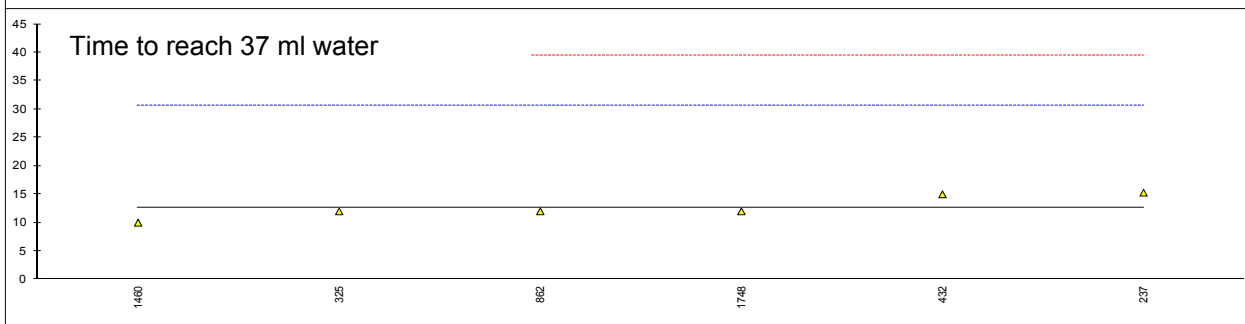
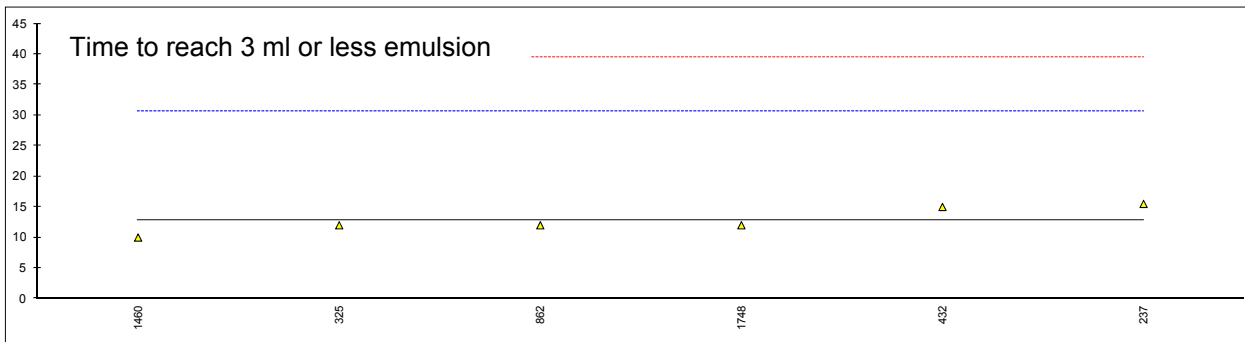
normality not OK
 n 10
 outliers 1
 mean (n) 6756.7
 st.dev. (n) 197.42
 R(calc.) 552.8
 R(D4294:16e1) 564.3



Determination of Water Separability at 82°C, distilled water on sample #16035; 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	o-w-e(t)
178		----	----	----	----	----	----	----	----
179	D1401	----	----	----	----	15	----	----	40-40-0
237	D1401	15.5	0.31	15.3	0.29	18.6	----	NO	----
315		----	----	----	----	----	----	----	----
325	D1401	12	-0.08	12	-0.08	>60	----	After 60 min	40-37-3(12)
349		----	----	----	----	----	----	----	----
432	D1401	15	0.25	15	0.26	16	----	NO	40-39-1(15)
473		----	----	----	----	----	----	----	----
496		----	----	----	----	----	----	----	----
551		----	----	----	----	----	----	----	----
621		----	----	----	----	----	----	----	----
862	D1401	12	-0.08	12	-0.08	----	----	YES	41-39-0(15)
963		----	----	----	----	----	----	----	----
1146	D1401	n.a.	----	n.a.	----	n.a.	----	After 30 min	0-32-48(30)
1161		----	----	----	----	----	----	----	----
1201		----	----	----	----	----	----	----	----
1460	D1401	10	-0.31	10	-0.30	15	----	NO	40-40-0
1748	D1401	12	-0.08	12	-0.08	15	----	YES	40-40-0

normality	unknown	unknown	unknown
n	6	6	5
outliers	0	0	0
mean (n)	12.75	12.72	15.92
st.dev. (n)	2.092	2.040	1.559
R(calc.)	5.86	5.71	4.37
R(D1401:12e1)	25.00	25.00	n.a.



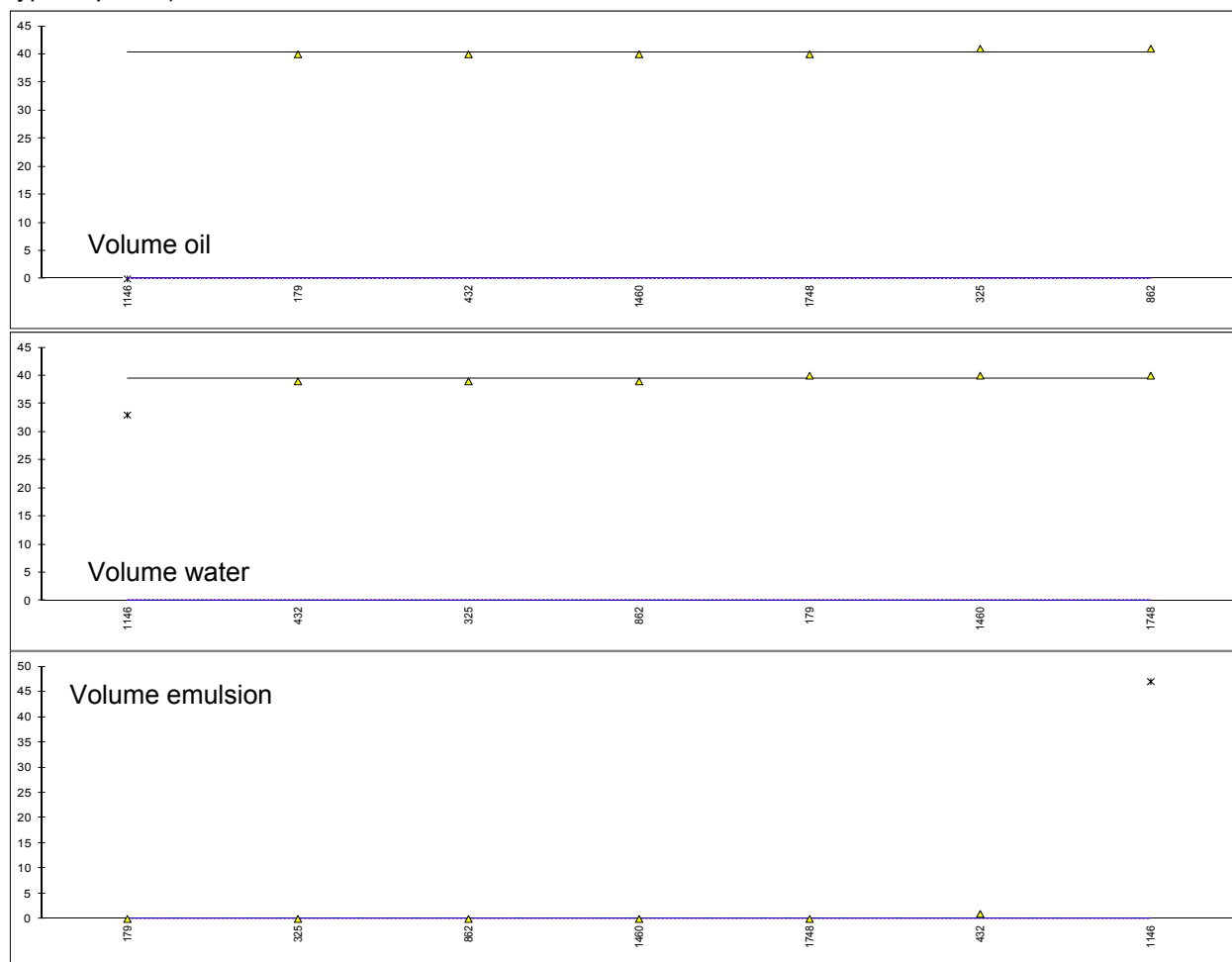
Determination of Water Separability at 82°C, distilled water on sample #16035; results in ml

--- Continued ---

lab	method	volume oil phase	mark	volume water phase	mark	volume emulsion phase	mark	o-w-e(t) reported
178		----		----		----		----
179	D1401	40		40		0		40-40-0
237	D1401	----		----		----		----
315		----		----		----		----
325	D1401	41		39		0		40-37-3(12)
349		----		----		----		----
432	D1401	40		39		1		40-39-1(15)
473		----		----		----		----
496		----		----		----		----
551		----		----		----		----
621		----		----		----		----
862	D1401	41		39		0		41-39-0(15)
963		----		----		----		----
1146	D1401	0	D(0.01)	32	C,D(0.01)	48	C,D(0.01)	0-32-48(30) C
1161		----		----		----		----
1201		----		----		----		----
1460	D1401	40		40		0		40-40-0
1748	D1401	40		40		0		40-40-0
normality		unknown		unknown		unknown		
n		6		6		6		
outliers		1		1		1		
mean (n)		40.3		39.5		0.2		
st.dev. (n)		0.52		0.55		0.41		
R(calc.)		1.4		1.5		1.1		
R(D1401:12e1)		n.a.		n.a.		n.a.		

Lab 325 reported inconsistent values in o-w-e(t) and volume oil, water and emulsion phase

Lab 1146 first reported 0/33/47 respectively for o/w/e and commented at remarks: Because of upper layer (oil type of phase) > 43 ml it should be seen as emulsion instead of an oil

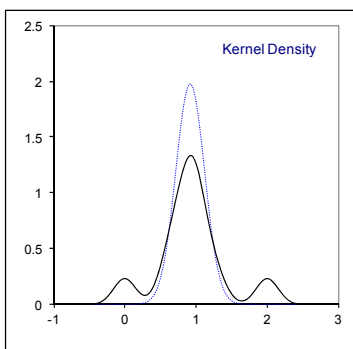
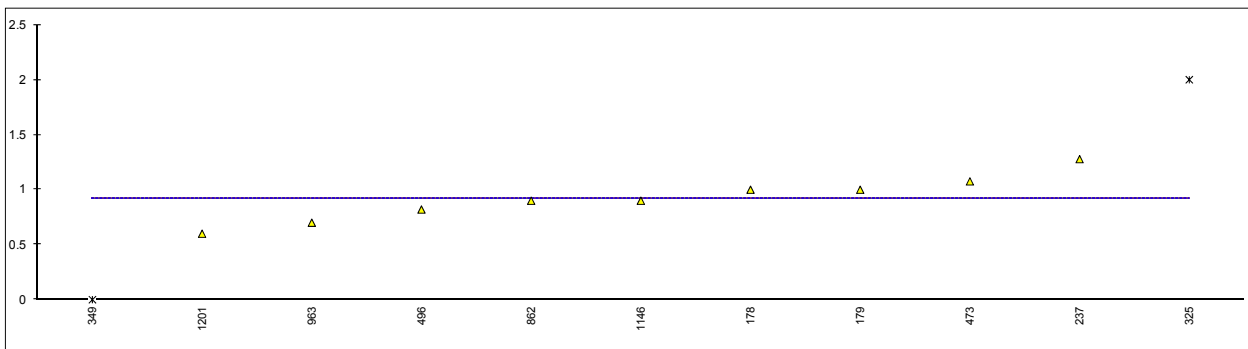


Determination of Calcium (Ca) on sample #16035; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	1		----	
179	D5185	1		----	
237	D5185	1.278	C	----	first reported 2.453
315		----		----	
325	D5185	2	D(0.05)	----	
349	D5185	0	D(0.05)	----	
432	D4951	<5		----	
473	D5185	1.076		----	
496	D5185	0.82	C	----	first reported 287.12
551		----		----	
621		----		----	
862	D5185	0.9		----	
963	D5185	0.7		----	
1146	In house	0.9		----	
1161		----		----	
1201	D5185	0.6		----	
1460	D5185	< 1.0		----	
1748		----		----	

normality OK
n 9
outliers 2
mean (n) 0.919
st.dev. (n) 0.2021
R(calc.) 0.566
R(D5185:13e1) n.a.

Application range 40-9000 mg/kg

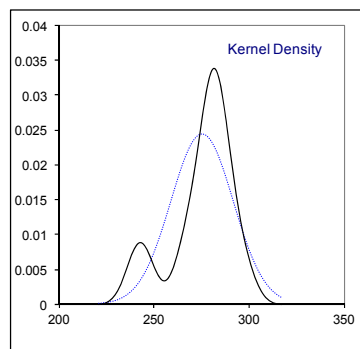
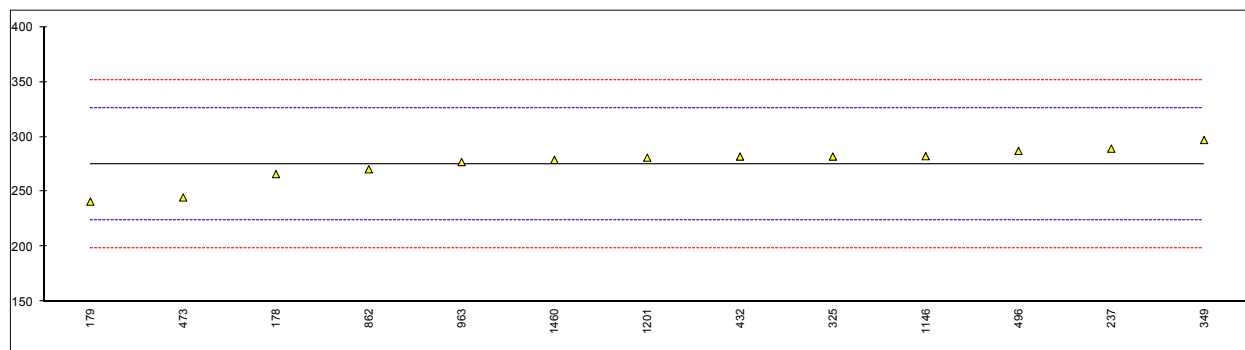


Determination of Phosphorus (P) on sample #16035; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	266		-0.36	
179	D5185	241		-1.35	
237	D5185	289.2		0.55	
315		----		----	
325	D5185	282		0.26	
349	D5185	297		0.85	
432	D4951	282		0.26	
473	D5185	244.8		-1.20	
496	D5185	287.12	C	0.46	first reported 0.82
551		----		----	
621		----		----	
862	D5185	270.4		-0.19	
963	D5185	277		0.07	
1146	In house	282.3		0.27	
1161		----		----	
1201	D5185	281		0.22	
1460	D5185	279		0.15	
1748		----		----	

normality OK
n 13
outliers 0
mean (n) 275.29
st.dev. (n) 16.353
R(calc.) 45.79
R(D5185:13e1) 71.35

Application range 10-1000 mg/kg

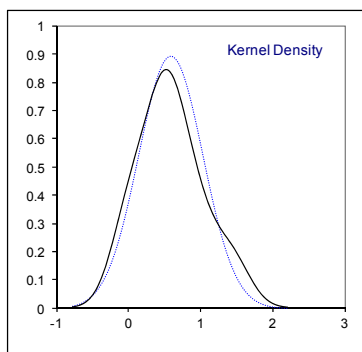
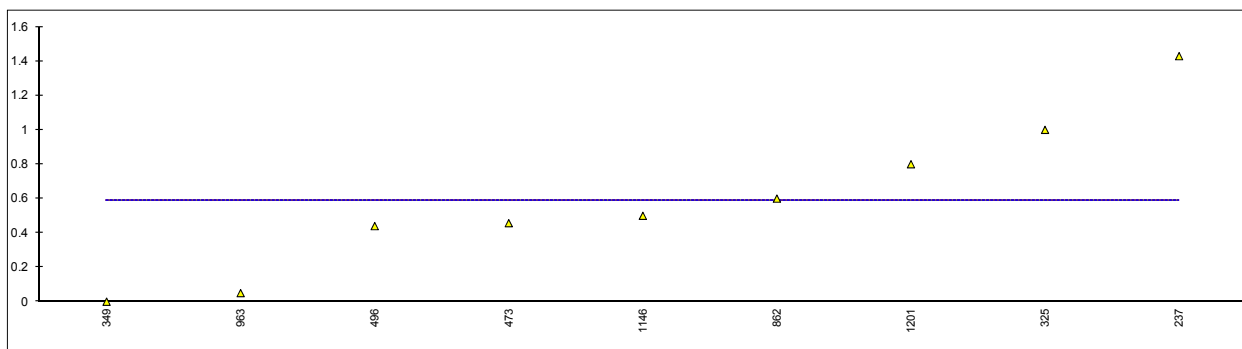


Determination of Zinc (Zn) on sample #16035; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	<1		----	
179	D5185	<1		----	
237	D5185	1.429	C	----	first reported 2.727
315		----		----	
325	D5185	1		----	
349	D5185	0		----	
432	D4951	<1		----	
473	D5185	0.457		----	
496	D5185	0.44		----	
551		----		----	
621		----		----	
862	D5185	0.6		----	
963	D5185	0.05		----	
1146	In house	0.5		----	
1161		----		----	
1201	D5185	0.8		----	
1460	D5185	< 1.0		----	
1748		----		----	

normality OK
 n 9
 outliers 0
 mean (n) 0.586
 st.dev. (n) 0.4478
 R(calc.) 1.254
 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 BRAZIL
1 lab in CHINA, People's Republic
1 lab in GERMANY
1 lab in INDONESIA
1 lab in JORDAN
3 labs in NETHERLANDS
1 lab in NIGERIA
1 lab in NORWAY
2 labs in SAUDI ARABIA
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 outlier test
R(0.05)	= straggler in Rosner 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 calculations
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
SDS	= Safety Data Sheet

Literature:

- 1 iis Interlaboratory Studies, Protocol for the Organization, Statistics and Evaluation, April 2014
- 2 ASTM E178:08
- 3 ISO 5725:86
- 4 ISO 5725, parts 1-6, 1994
- 5 ISO13528:05
- 6 ISO17043:2010
- 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, First reported 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 The Royal Society of Chemistry 2002, Analyst 2002, 127, pages 1359-1364, P.J. Lowthian and M. Thompson (see <http://www.rsc.org/suppdata/an/b2/b205600n/>).
- 15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), pp. 165-172, (1983)