Results of Proficiency Test
Gear Oil (fresh)
April 2018

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. R.J. Starink
Correctors: ing. A.S. Noordman-de Neef & ing. C.M. Nijssen-Wester
Report no.: iis18L01

June 2018

CONTENTS

Appendices:

1 INTRODUCTION

Since 2015, the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of fresh Gear Oil. During the annual proficiency testing program 2017/2018 it was decided to continue with the round robin for the analysis of fresh Gear Oil.
In this interlaboratory study, 21 laboratories in 18 different countries registered for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2018 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 1.5 L fresh Gear Oil labelled #18035.
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 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 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 fresh Gear Oil was purchased from a local supplier. The 40 litres bulk material was homogenized and transferred into 24 amber glass bottles of 1 litre and 30 amber glass bottles of 0.5 litre (all labelled #18035). The homogeneity of the subsamples #18035 was checked by the determination of Density at 15°C in accordance with ASTM D4052 and Particle Contamination in accordance with ASTM D7647 on 8 stratified randomly selected samples.

	Density at 15 °C in kg/m^3	Particle Size >4µm (c) in counts/ml	Particle Size >6µm (c) in counts/ml	Particle Size >14µm (c) in counts/ml
Sample #18035-1	887.98	8798	1063	31
Sample #18035-2	887.97	9254	1161	34
Sample #18035-3	887.97	9227	1156	33
Sample #18035-4	887.97	9536	1161	34
Sample #18035-5	887.97	9737	1169	35
Sample #18035-6	887.97	9403	1148	31
Sample #18035-7	887.98	9678	1198	35
Sample #18035-8	887.98	9536	1193	39

Table 1: homogeneity test results of subsamples #18035

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	Particle Size >4µm (c) in counts/ml	Particle Size >6µm (c) in counts/ml	Particle Size >14µm (c) in counts/ml
r (observed)	0.01	848	116	7
reference test method	ASTM D4052:18	ASTM D7647:10	ASTM D7647:10	ASTM D7647:10
0.3 x R(ref. test method)	0.15	3220	266	14

Table 2: evaluation of the repeatability of subsamples #18035

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

To each of the participating laboratories, one sample of 1L and one sample of 0.5L (both labelled #18035) were sent on March 14, 2018. A SDS was added to the sample package.

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: 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 (distilled water), Sulphur, Water, Water separability, Level of Contamination, Calcium, Phosphorus and Zinc.

It was explicitly requested to treat the samples as if they were routine samples 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' test results for 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 reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of 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 and/or 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 visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are on the X-axis.

The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. 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. ISO reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

This 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 in accordance with:

$z_{(target)}$ = (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.

Therefore, the usual interpretation of z-scores is as follows:

	$\lvert z\rvert < 1$	good
$1 <$	$\lvert z\rvert < 2$	satisfactory
$2 <$	$\lvert z\rvert < 3$	questionable
$3 <$	$\lvert z\rvert$	unsatisfactory

4 EVALUATION

In this interlaboratory study no problems were encountered with the dispatch of the samples. Two participants did not report any test results. One participant was not able to report the test results in time. Not all participants were able to report test results for all the requested tests. In total 19 participants reported 350 test results. Observed were 14 outlying test results, which is 4.0% 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 reported test results are discussed per test. The test methods, which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These 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. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D664:17a.

Copper Corrosion: All reporting participants agreed on classification 1.

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

Flash Point COC: 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 D92:16b.

Flash Point PMcc: This determination was not problematic. One statistical outlier was observed and one other test result was excluded. Test method ASTM D93-B is not applicable to fresh oils. However, the calculated reproducibility after rejection of the suspect data is in good agreement with ASTM D93-A:16a.

Foaming Characteristics (Tendency and Stability): This determination was only problematic for Sequence II of the Foaming Tendency. In total four statistical outliers were observed in Foam Tendency. The calculated reproducibility for Sequence I, after rejection of the statistical outlier is in agreement with the requirements of ASTM D892:18. The calculated reproducibility for Sequence II, after rejection of the statistical outlier is not in agreement with the requirements of ASTM D892:18.
For the Foaming stability, all reporting participants reported a zero for all three sequences. Therefore, no significant conclusions could be drawn.

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 D445:17a.

Kinematic Viscosity at 100°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D445:17a.

Viscosity Index: This determination was not problematic. No statistical outliers were observed. One test result was excluded for the statistical evaluation. 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. No calculation errors were observed.

Pour Point - Manual: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with requirements of ASTM D97:17b. The low number of test results and the rounding of a part 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: All reporting participants agreed on a classification as “Pass” / “no rusting”.

Sulphur: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility 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 This determination may 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 not in agreement with the requirements of ASTM D1401:12e1.
The calculated reproducibilities of the volume oil phase and the volume water phase are very large compared to the calculated reproducibilities of the previous PT (18.4 vs 3.9).

Level of Contamination: This determination was very problematic. Two statistical outliers were observed over six parameters. Only the calculated reproducibility for level of contamination acc. ISO4406 scale >14µm after rejection of the statistical outlier is in agreement with the requirements of ASTM D7647:10(2018). All other calculated reproducibilities are not in agreement with the requirements of ASTM D7646:10(2018).

Calcium as Ca: The consensus value for the Calcium determination was below the application range of ASTM D5185:18. 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 D5185:18.

Zinc as Zn: The consensus value for the Zinc determination was below the application range of ASTM D5185:18. 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	13	0.95	0.21	0.28
Copper Corrosion, 3hrs at 100°C	rating	12	1	n.a.	n.a.
Density at 15°C	kg/m^3	15	888.0	0.5	0.5
Flash Point COC	°C	13	222	11	18
Flash Point PMcc	°C	13	173	6	12
Foaming Tendency (Seq I)	mL	10	3	12	17
Foaming Tendency (Seq II)	mL	10	163	167	54
Kinematic Viscosity at 40°C	mm^2/s	17	77.90	0.55	0.95
Kinematic Viscosity at 100°C	mm^2/s	17	9.804	0.080	0.135
Viscosity Index		14	104.9	1.8	2
Pour Point, Manual	°C	10	-31	13	9
Pour Point, Automated	°C	6	-34	4	5
Rust Prevention (distilled water)		8	Pass	n.a.	n.a.
Sulphur	mg/kg	7	16095	2455	988
Water	mg/kg	15	84	53	241
Water Separability at 54°C, distilled water					
- Time ≤ 3 ml emulsion	min.	5	41	36	20
- Time 37 ml water	min.	5	40	35	20
- Time to complete break	min.	6	>30	n.a.	n.a.
- Volume Oil phase	mL	11	32	18	n.a.
- Volume Water phase	mL	11	21	39	n.a.
- Volume Emulsion phase	mL	11	27	57	n.a.

Parameter	unit	n	average	2.8 * sd	R(lit)
Level of contamination					
- ≥ 4µm (c)	counts/mL	7	2326	3846	2628
- ≥ 6µm (c)	counts/mL	7	400	766	304
- ≥14µm (c)	counts/mL	6	22	64	30
- ≥ 4µm (c)	Scale no.	9	18.4	4.2	1.7
- ≥ 6µm (c)	Scale no.	9	16.0	3.4	1.2
- ≥14µm (c)	Scale no.	8	11.9	1.8	2
Calcium as Ca	mg/kg	14	22	n.e.	n.e.
Phosphorus as P	mg/kg	15	673	69	112
Zinc as Zn	mg/kg	14	<2	n.a.	n.a.

Table 3: reproducibilities of the test results on sample #18035

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

4.3 COMPARISON OF PROFICIENCY TEST OF APRIL 2018 WITH PREVIOUS PT

	April 2018	April 2017	April 2016	April 2015
Number of reporting labs	18	14	18	13
Number of results reported	350	177	215	125
Statistical outliers	14	8	14	6
Percentage outliers	4.0%	4.5%	6.5%	4.8%

Table 4: comparison with previous proficiency tests

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 2018	April 2017	April 2016	April 2015
Acid Number (Total)	+	-	+	+
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	+/-	++	+/-	+/-
Sulphur	--	-	+/-	-
Water	++	++	n.e.	n.e.
Water Separability at 54°C	-	+	++	++
Level of Contamination	--	n.e.	n.e.	n.e.
Calcium as C	n.e.	n.e.	n.e.	n.e.
Phosphorus as P	++	++	+	--
Zinc as Zn	n.e.	n.e.	n.e.	n.e.

Table 5: 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:

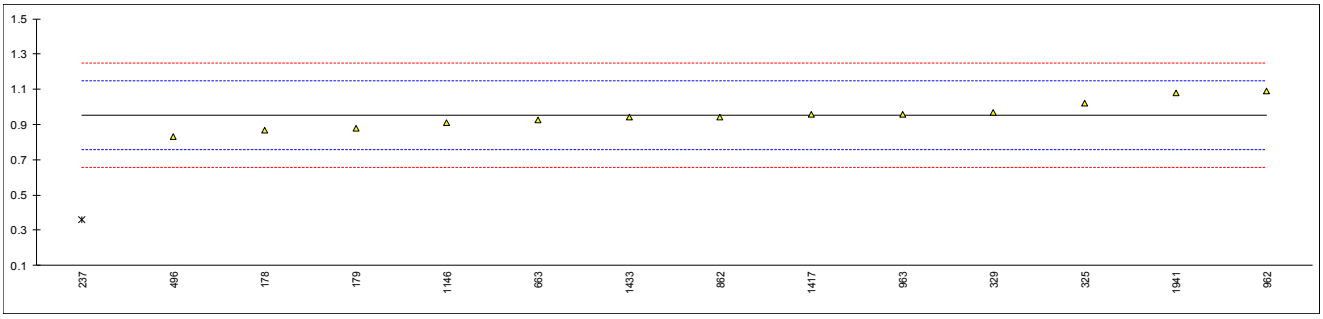
++:	group performed much better than the reference test method
+ :	group performed better than the reference test method
+/-:	group performance equals the reference test method
- :	group performed worse than the reference test method
-- :	group performed much worse than the reference test method
n.e.: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

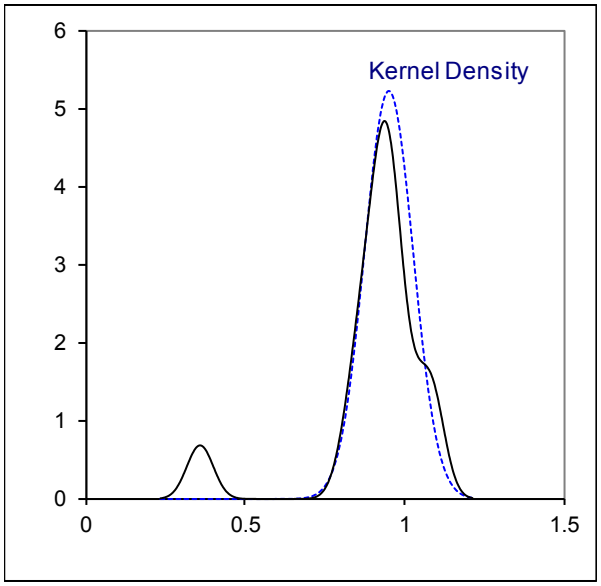
APPENDIX 1

Determination of Acid Number (Total) on sample #18035; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
178	INH-1118	0.87		-0.83	
179	D664-B	0.88		-0.73	
237	D664-A	0.3589	G(0.01)	-6.03	
325	D664-A	1.02		0.69	
329	D664-A	0.97		0.18	
349		-----		-----	
432		-----		-----	
496	D664-A	0.83		-1.24	
663	D664-A	0.926		-0.26	
862	D664-A	0.9420		-0.10	
962	D974	1.09		1.40	
963	D664-A	0.96		0.08	
1146	D664-A	0.909		-0.44	
1150		-----		-----	
1417	IP177	0.959		0.07	
1433	D664-B	0.94		-0.12	
1748		-----		-----	
1799		-----		-----	
1941	ISO6619	1.08		1.30	
6016		-----		-----	
6199		-----		-----	

normality	OK
n	13
outliers	1
mean (n)	0.9520
st.dev. (n)	0.07640
R(calc.)	0.2139
st.dev.(D664:17a)	0.09830
R(D664:17a)	0.2752





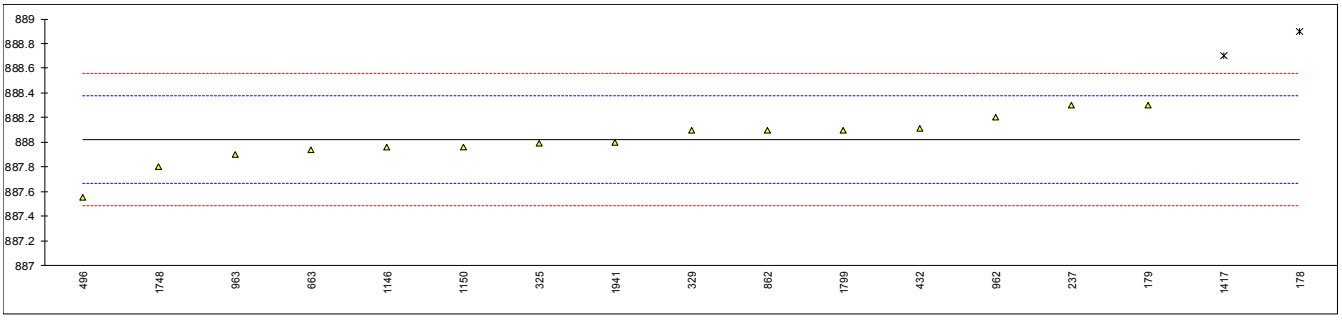
Determination of Copper Corrosion 3 hours at 100°C on sample #18035;

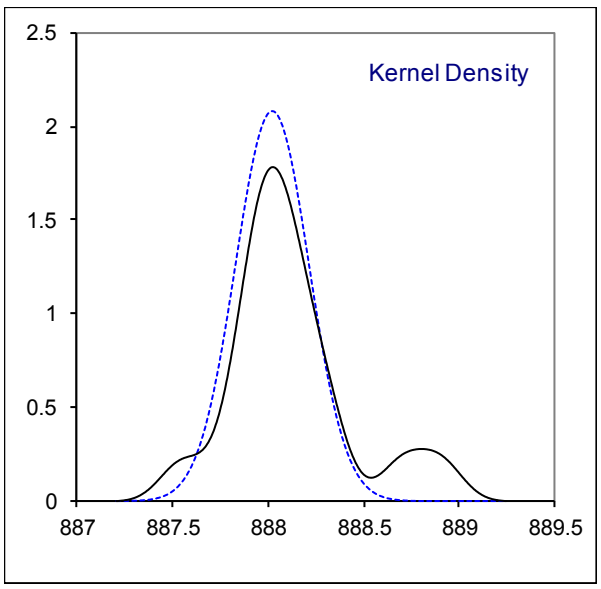
lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D130	1A		-----	
237	D130	1		-----	
325	D130	1B		-----	
329		-----		-----	
349		-----		-----	
432		-----		-----	
496	D130	1b		-----	
663		-----		-----	
862	D130	1a		-----	
962		-----		-----	
963	D130	1b		-----	
1146		-----		-----	
1150	ISO2160	1b		-----	
1417	IP154	1B		-----	
1433	D130	1a		-----	
1748	D130	1a		-----	
1799	D130	1a		-----	
1941	ISO2160	1 A		-----	
6016		-----		-----	
6199		-----		-----	
	n	12			
	mean (n)	1 (1a+1b)			

Determination of Density at 15°C on sample #18035; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
178	D4052	888.9	DG(0.05)	4.92	
179	D4052	888.3		1.56	
237	D4052	888.3		1.56	
325	D4052	887.99		-0.17	
329	D4052	888.1		0.44	
349		-----		-----	
432	D4052	888.11		0.50	
496	D4052	887.55	C	-2.64	First reported 891.01
663	D4052	887.94	C	-0.45	First reported 0.89148
862	D4052	888.1		0.44	
962	D4052	888.2		1.00	
963	D4052	887.9		-0.68	
1146	D4052	887.96		-0.34	
1150	ISO12185	887.96		-0.34	
1417	IP365	888.7	DG(0.05)	3.80	
1433		-----		-----	
1748	D4052	887.8		-1.24	
1799	D7042	888.1		0.44	
1941	D4052	888.0		-0.12	
6016		-----		-----	
6199		-----		-----	

normality	suspect
n	15
outliers	2
mean (n)	888.021
st.dev. (n)	0.1918
R(calc.)	0.537
st.dev.(D4052:18)	0.1786
R(D4052:18)	0.5

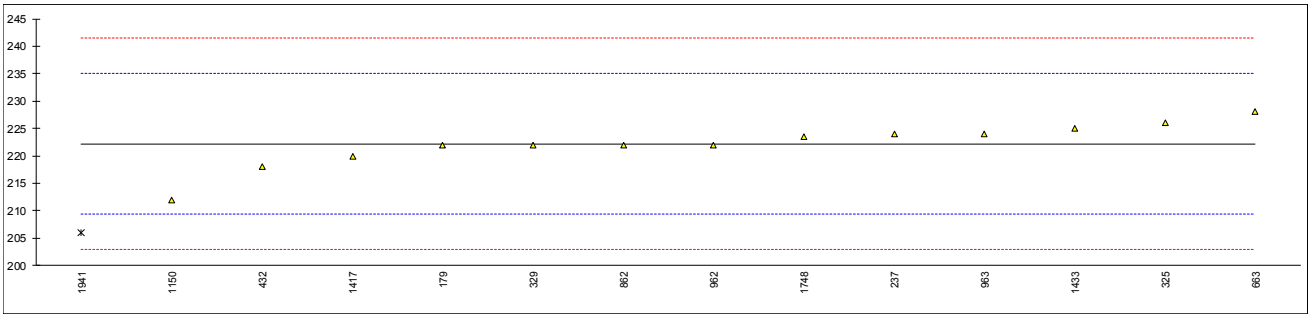


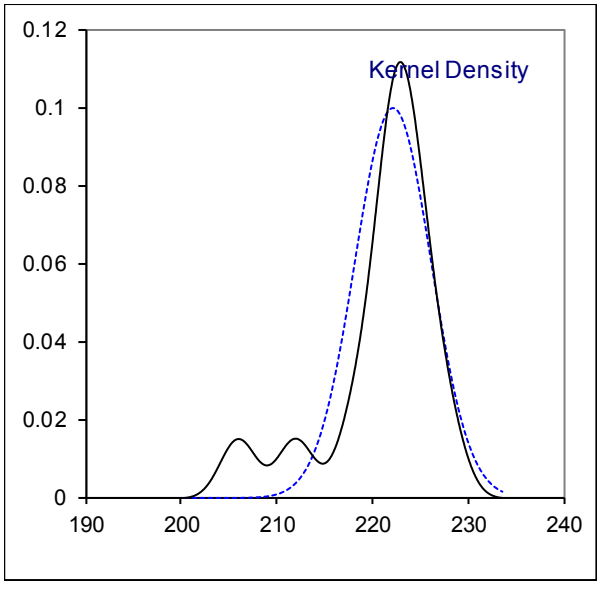


Determination of Flash Point COC on sample #18035; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D92	222		-0.03	
237	D92	224		0.28	
325	D92	226.0		0.59	
329	D92	222.0		-0.03	
349		-----		-----	
432	D92	218		-0.65	
496		-----		-----	
663	D92	228		0.90	
862	D92	222		-0.03	
962	D92	222.0		-0.03	
963	D92	224		0.28	
1146		-----		-----	
1150	ISO2592	212		-1.59	
1417	D92	220		-0.34	
1433	D92	225		0.44	
1748	D92	223.5		0.20	
1799		-----		-----	
1941	ISO2592	206	G(0.05)	-2.52	
6016		-----		-----	
6199		-----		-----	

normality	not OK
n	13
outliers	1
mean (n)	222.19
st.dev. (n)	3.987
R(calc.)	11.16
st.dev.(D92:16b)	6.429
R(D92:16b)	18

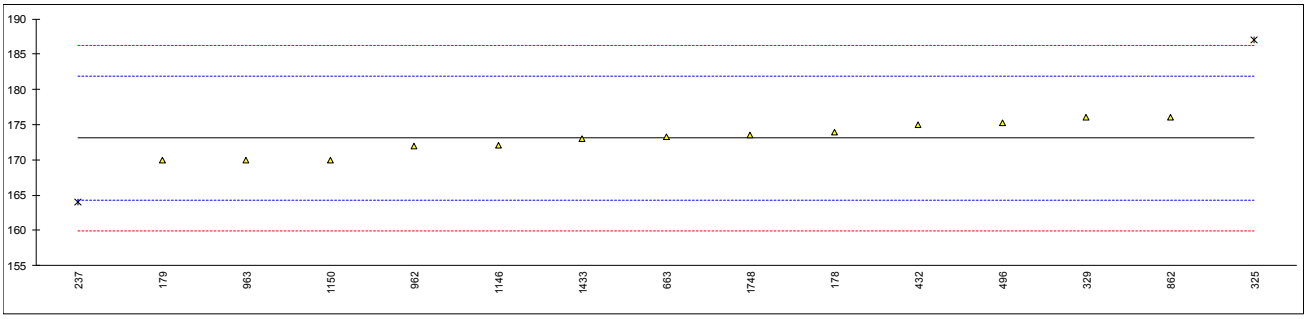


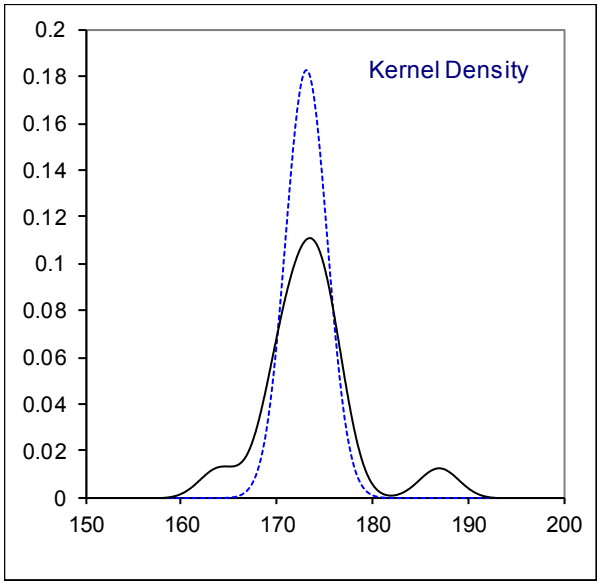


Determination of Flash Point PMcc on sample #18035; results in °C

lab	method	value	mark	z(targ)	remarks
178	D93-A	174.0		0.21	
179	D93-A	170		-0.70	
237	D93-B	164.0	ex	-2.07	Test method B is not suitable for fresh oils
325	D93-A	187.0	G(0.05)	3.17	
329	D93-A	176.0	C	0.66	First reported 184.0
349		-----		-----	
432	D93-A	175.0		0.43	
496	D93-A	175.3		0.50	
663	D93-A	173.3		0.05	
862	D93-A	176		0.66	
962	D93-A	172.0		-0.25	
963	D93-A	170.0		-0.70	
1146	In house	172.1		-0.23	
1150	ISO2719-A	170		-0.70	
1417		-----		-----	
1433	D93-A	173.0		-0.02	
1748	D93-A	173.5		0.09	
1799		-----		-----	
1941		-----		-----	
6016		-----		-----	
6199		-----		-----	

normality	OK
n	13
outliers	1 (+1excl)
mean (n)	173.09
st.dev. (n)	2.186
R(calc.)	6.12
st.dev.(D93-A:16a)	4.389
R(D93-A:16a)	12.29



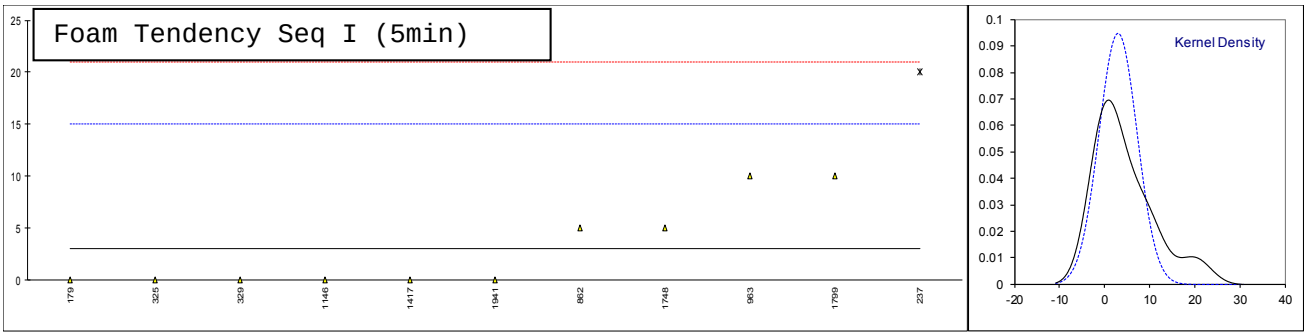


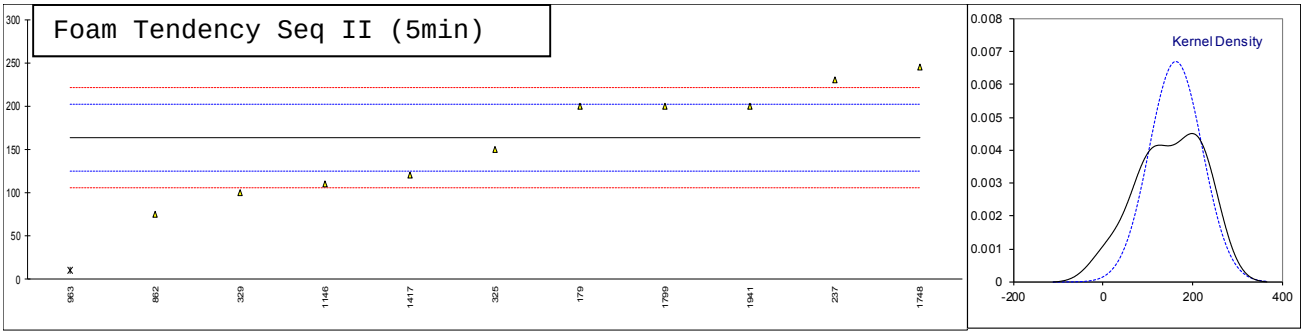
Determination of Foaming Tendency, Sequence I, II and III (5 min. blowing period) on sample #18035; results in mL

lab	method	Sample used	Diffuser	Seq I (5 min)	mark	z(targ)	Seq II (5 min)	mark	z(targ)	Seq III (5 min)	mark	z(targ)
178			---	-----		-----	-----		-----	-----		-----
179	D892	As rec.	Metal	0		-0.50	200		1.92	0		-----
237	D892	As rec.	Stone	20	G(5)	2.83	230		3.49	0		-----
325	D892	As rec.	Stone	0		-0.50	150		-0.68	0		-----
329	D892	As rec.	Stone	0		-0.50	100		-3.28	0		-----
349		---	---	-----		-----	-----		-----	-----		-----
432		---	---	-----		-----	-----		-----	-----		-----
496		---	---	-----		-----	-----		-----	-----		-----
663		---	---	-----		-----	-----		-----	-----		-----
862	D892	---	---	5		0.33	75		-4.58	5	G(1)	-----
962		---	---	-----		-----	-----		-----	-----		-----
963	D892	As rec.	Metal	10		1.17	10	G(5)	-7.96	10	G(1)	-----
1146	D892	As rec.	Metal	0		-0.50	110		-2.76	0		-----
1150		---	---	-----		-----	-----		-----	-----		-----
1417	D892	As rec.	Metal	0		-0.50	120		-2.24	0		-----
1433		---	---	-----		-----	-----		-----	-----		-----
1748	D892	As rec.	Stone	5		0.33	245		4.27	0		-----
1799	D892	---	---	10		1.17	200		1.92	0		-----
1941	ISO6247	As rec.	Metal	0		-0.50	200		1.92	0		-----
6016		---	---	-----		-----	-----		-----	-----		-----
6199				-----		-----	-----		-----	-----		-----

	Seq I (5 min)	Seq II (5 min)	Seq III (5 min)
normality	suspect	OK	n.a.
n	10	10	9
outliers	1	1	2
mean (n)	3	163	0
st.dev. (n)	4.22	59.50	0
R(calc.)	11.8	166.6	0
st.dev.(D892:18)	6.01	19.22	0
R(D892:18)	16.8	53.8	0

As rec. = As received





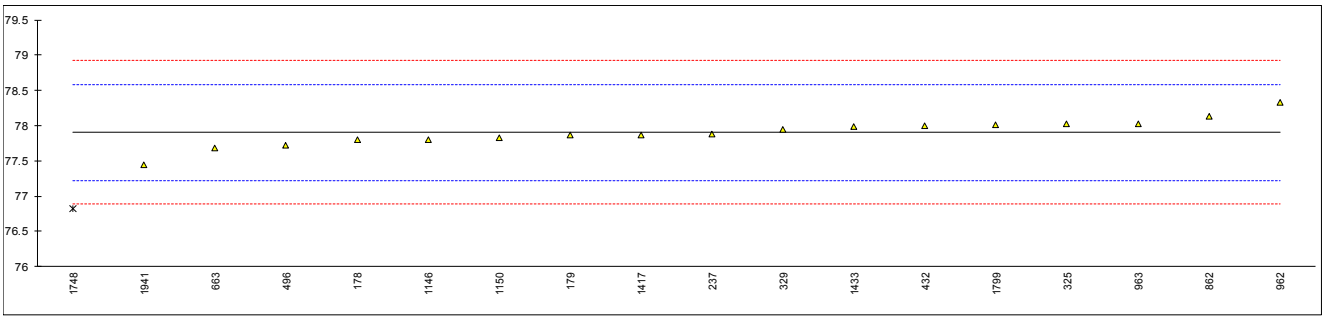
Determination of Foaming Stability, Sequence I, II and III (10 min. settling period) on sample #18035; results in mL

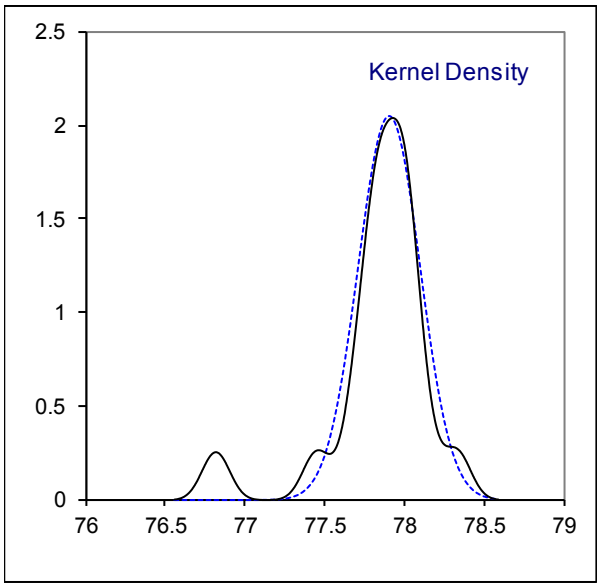
lab	method	Seq I (10 min)	mark	z(targ)	Seq II (10 min)	mark	z(targ)	Seq III (10 min)	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179	D892	0		-----	0		-----	0		-----
237	D892	0		-----	0		-----	0		-----
325	D892	0		-----	0		-----	0		-----
329	D892	0		-----	0		-----	0		-----
349		-----		-----	-----		-----	-----		-----
432		-----		-----	-----		-----	-----		-----
496		-----		-----	-----		-----	-----		-----
663		-----		-----	-----		-----	-----		-----
862	D892	0		-----	0		-----	0		-----
962		-----		-----	-----		-----	-----		-----
963	D892	0		-----	0		-----	0		-----
1146	D892	0		-----	0		-----	0		-----
1150		-----		-----	-----		-----	-----		-----
1417	D892	0		-----	0		-----	0		-----
1433		-----		-----	-----		-----	-----		-----
1748	D892	0		-----	0		-----	0		-----
1799	D892	0		-----	0		-----	0		-----
1941	ISO6247	0		-----	0		-----	0		-----
6016		-----		-----	-----		-----	-----		-----
6199		-----		-----	-----		-----	-----		-----

Determination of Kinematic Viscosity at 40°C on sample #18035; results in mm²/s

lab	method	value	mark	z(targ)	remarks
178	D445	77.8		-0.31	
179	D445	77.87	C	-0.10	First reported 44.06
237	D445	77.88		-0.07	
325	D445	78.02		0.34	
329	D445	77.95		0.14	
349		-----		-----	
432	D445	78.00		0.28	
496	D445	77.728		-0.52	
663	D445	77.682		-0.65	
862	D445	78.13		0.67	
962	D445	78.33		1.26	
963	D445	78.03		0.37	
1146	D445	77.80		-0.31	
1150	ISO3104	77.8281		-0.22	
1417	D445	77.87		-0.10	
1433	D445	77.99		0.25	
1748	D7042	76.816	G(0.01)	-3.21	
1799	D7042	78.01		0.31	
1941	ISO3104	77.45		-1.34	
6016		-----		-----	
6199		-----		-----	

normality	suspect
n	17
outliers	1
mean (n)	77.904
st.dev. (n)	0.1950
R(calc.)	0.546
st.dev.(D445:17a)	0.3394
R(D445:17a)	0.950

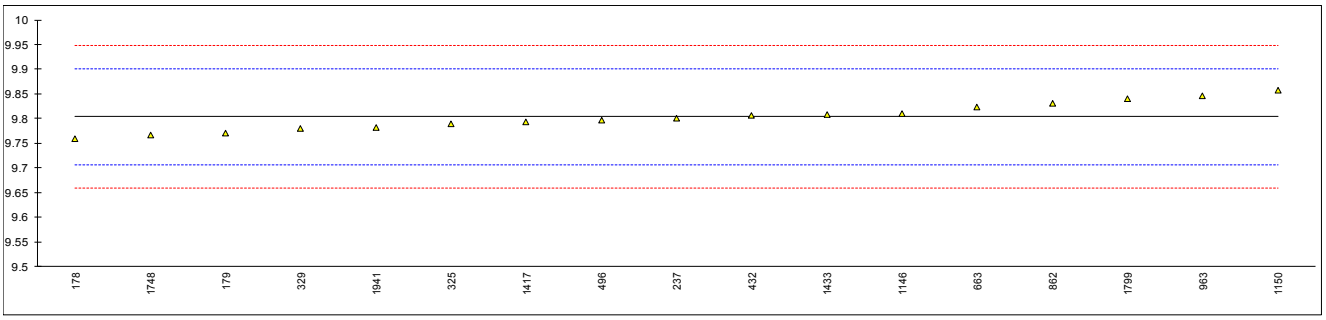


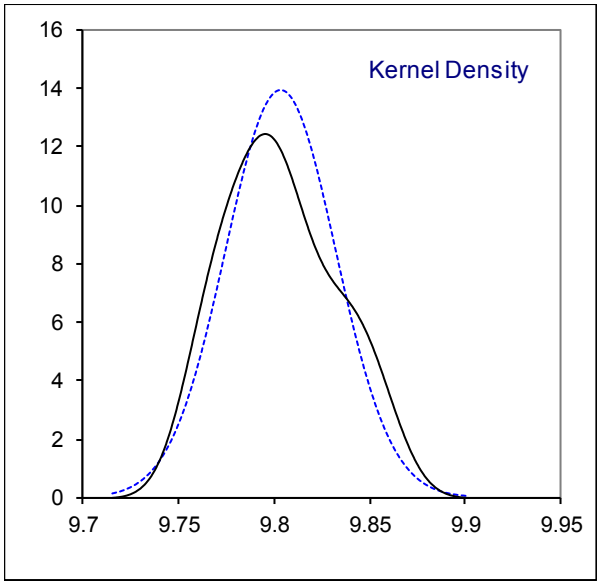


Determination of Kinematic Viscosity at 100°C on sample #18035; results in mm²/s

lab	method	value	mark	z(targ)	remarks
178	D445	9.760		-0.90	
179	D445	9.770		-0.70	
237	D445	9.80		-0.07	
325	D445	9.790		-0.28	
329	D445	9.779		-0.51	
349		-----		-----	
432	D445	9.806		0.05	
496	D445	9.7960		-0.16	
663	D445	9.8240		0.42	
862	D445	9.831		0.57	
962		-----		-----	
963	D445	9.846		0.88	
1146	D445	9.8108		0.15	
1150	ISO3104	9.8574		1.11	
1417	D445	9.793		-0.22	
1433	D445	9.808		0.09	
1748	D7042	9.767		-0.76	
1799	D7042	9.841		0.77	
1941	ISO3104	9.782		-0.45	
6016		-----		-----	
6199		-----		-----	

normality	OK
n	17
outliers	0
mean (n)	9.8036
st.dev. (n)	0.02866
R(calc.)	0.0803
st.dev.(D445:17a)	0.04832
R(D445:17a)	0.1353

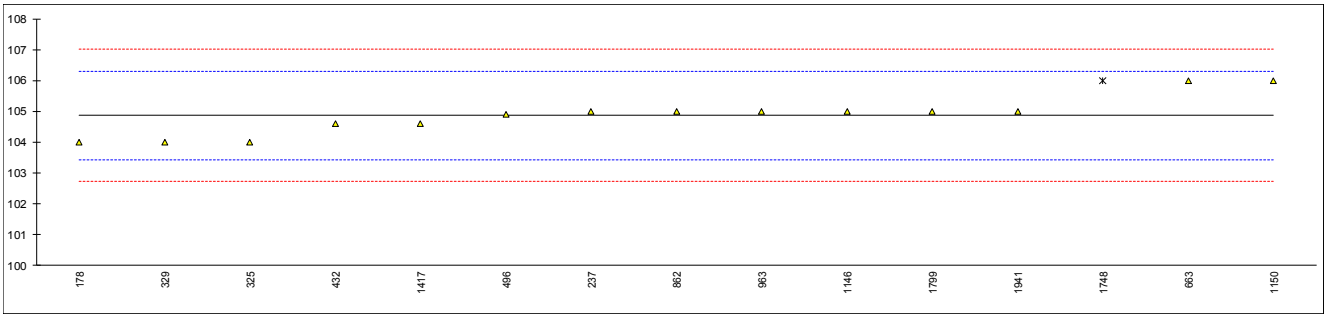


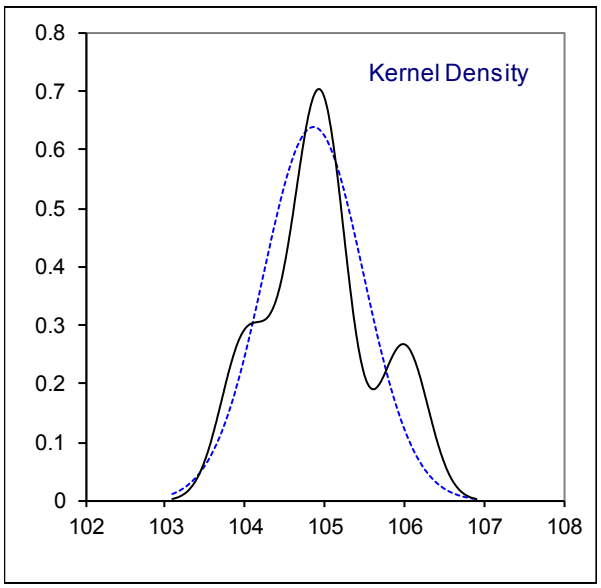


Determination of Viscosity Index (V.I.) on sample #18035

lab	method	value	mark	z(targ)	Calc.iis	remarks
178	D2270	104		-1.21	104.1	
179		-----		-----	104.2	
237	D2270	105		0.19	104.8	
325	D2270	104		-1.21	104.3	
329	D2270	104		-1.21	104.2	
349		-----		-----	-----	
432	D2270	104.6		-0.37	104.7	
496	D2270	104.9		0.05	105.0	
663	D2270	106		1.59	105.7	
862	D2270	105		0.19	105.0	
962		-----		-----	-----	
963	D2270	105		0.19	105.5	
1146	D2270	105		0.19	105.2	
1150	ISO2909	106		1.59	106.1	
1417	D2270	104.6		-0.37	104.6	
1433		-----		-----	104.7	
1748	D2270	105.98	ex	1.56	106.1	Result excluded, as Kin.Visco at 40°C is an outlier
1799	D2270	105		0.19	105.4	
1941	ISO2909	105		0.19	105.2	
6016		-----		-----	-----	
6199		-----		-----	-----	

normality	OK
n	14
outliers	0 (+1 excl)
mean (n)	104.86
st.dev. (n)	0.625
R(calc.)	1.75
st.dev.(D2270:10)	0.714
R(D2270:10)	2

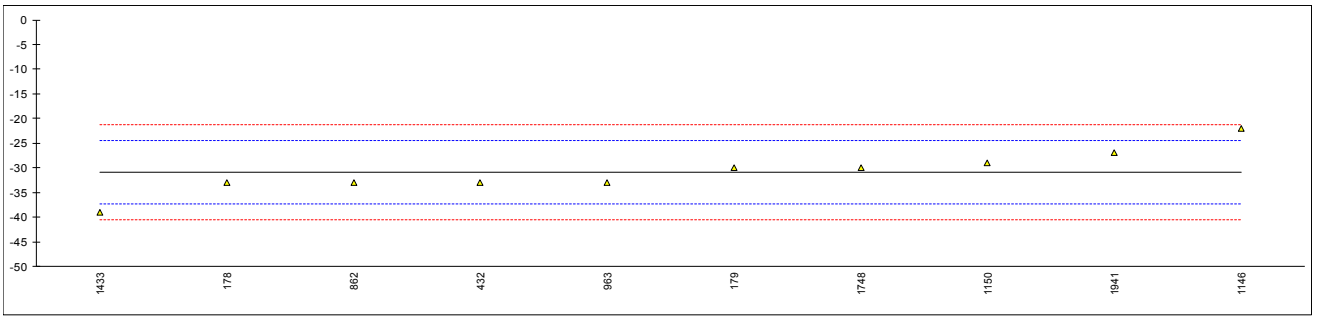


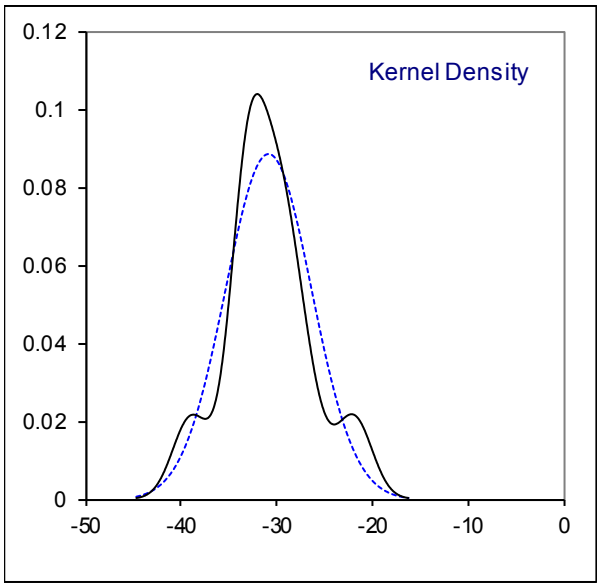


Determination of Pour Point, Manual on sample #18035; results in °C

lab	method	value	mark	z(targ)	remarks
178	D97	-33		-0.65	
179	D97	-30		0.28	
237		-----		-----	
325		-----		-----	
329		-----		-----	
349		-----		-----	
432	D97	-33		-0.65	
496		-----		-----	
663		-----		-----	
862	D97	-33		-0.65	
962		-----		-----	
963	D97	-33		-0.65	
1146	D97	-22		2.77	
1150	ISO3016	-29		0.59	
1417		-----		-----	
1433	D97	-39.0		-2.52	
1748	D97	-30		0.28	
1799		-----		-----	
1941	ISO3016	-27		1.21	
6016		-----		-----	
6199		-----		-----	

normality	suspect
n	10
outliers	0
mean (n)	-30.90
st.dev. (n)	4.508
R(calc.)	12.62
st.dev.(D97:17b)	3.21
R(D97:17b)	9

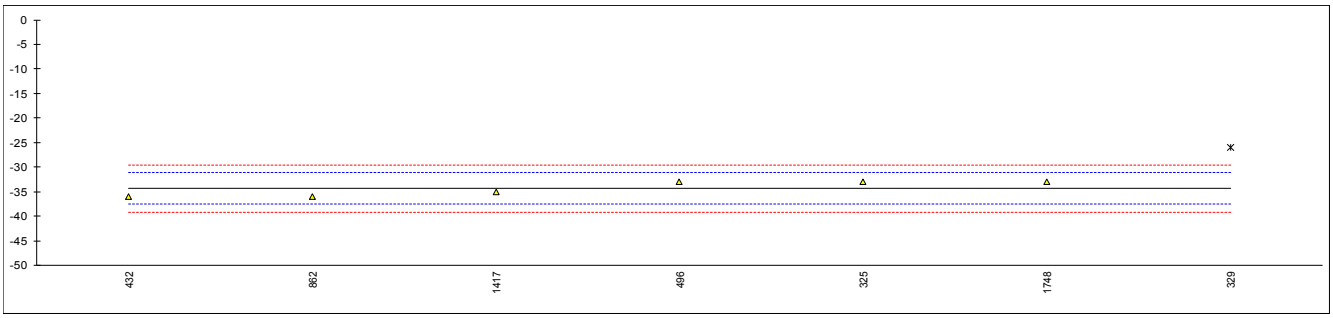


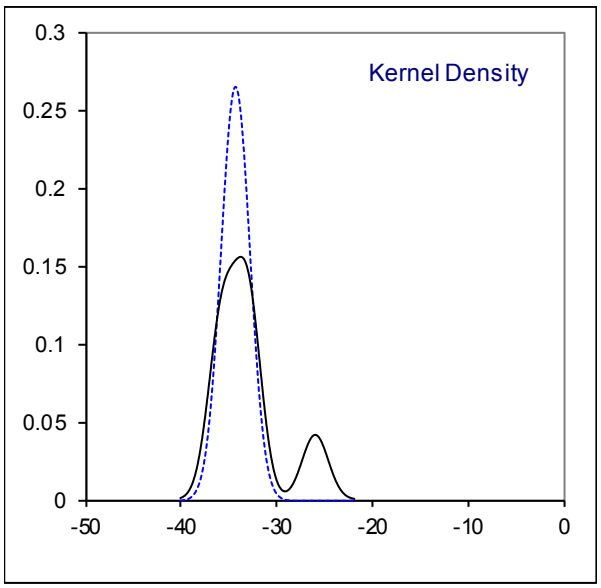


Determination of Pour Point, Automated, 1°C interval on sample #18035; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
325	D5950	-33		0.83	
329	D5950	-26	G(0.05)	5.19	
349		-----		-----	
432	D5950	-36		-1.04	
496	D5950	-33		0.83	
663		-----		-----	
862	D5950	-36		-1.04	
962		-----		-----	
963		-----		-----	
1146		-----		-----	
1150		-----		-----	
1417	D5950	-35		-0.41	
1433		-----		-----	
1748	D7346	-33		0.83	
1799		-----		-----	
1941		-----		-----	
6016		-----		-----	
6199		-----		-----	

normality	unknown
n	6
outliers	1
mean (n)	-34.33
st.dev. (n)	1.506
R(calc.)	4.22
st.dev.(D5950:14)	1.607
R(D5950:14)	4.5





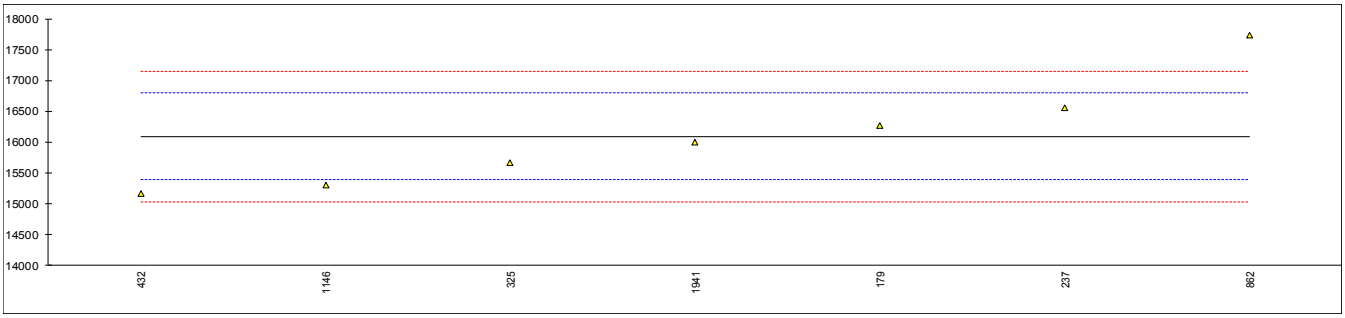
Determination of Rust prevention, distilled water on sample #18035

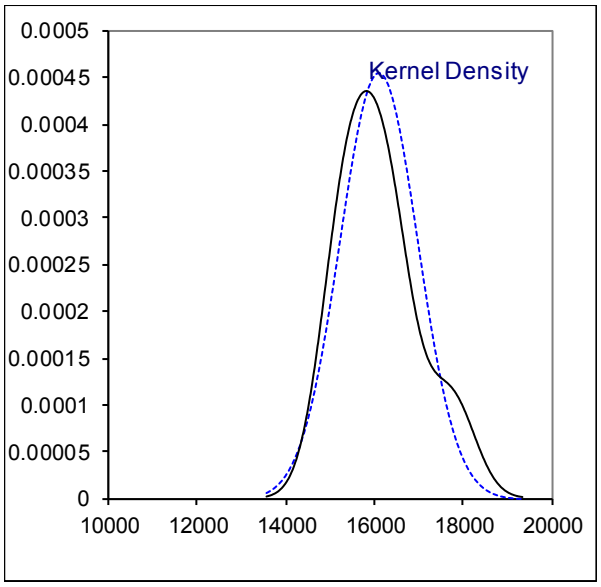
lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D665	Pass		-----	
237	D665	PASS		-----	
325	D665	PASS		-----	
329		-----		-----	
349		-----		-----	
432		-----		-----	
496	D665	pass		-----	
663		-----		-----	
862	D665	No Rusting		-----	
962		-----		-----	
963		-----		-----	
1146		-----		-----	
1150		-----		-----	
1417	D665	PASS		-----	
1433		-----		-----	
1748		-----		-----	
1799	D665	Pass		-----	
1941	D665	pass		-----	
6016		-----		-----	
6199		-----		-----	
	n	8			
	mean (n)	Pass			

Determination of Sulphur on sample #18035; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D4294	16264	C	0.48	First reported <20
237	D4294	16550	C	1.29	Reported 1.655, unit error
325	D5185	15660		-1.23	
329		-----		-----	
349		-----		-----	
432	D5185	15160		-2.65	
496		-----		-----	
663		-----		-----	
862	D2622	17730		4.64	
962		-----		-----	
963		-----		-----	
1146	D4294	15300		-2.25	
1150		-----		-----	
1417		-----	W	-----	Result withdrawn, reported 17620
1433		-----		-----	
1748		-----		-----	
1799		-----		-----	
1941	ISO8754	16001		-0.27	
6016		-----		-----	
6199		-----		-----	

normality	unknown
n	7
outliers	0
mean (n)	16095.0
st.dev. (n)	876.84
R(calc.)	2455.2
st.dev.(D4294:16e1)	352.67
R(D4294:16e1)	987.5

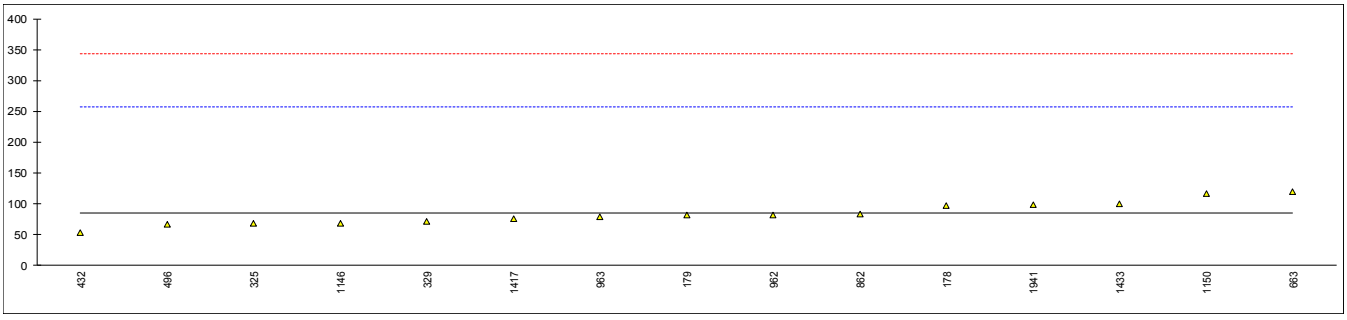


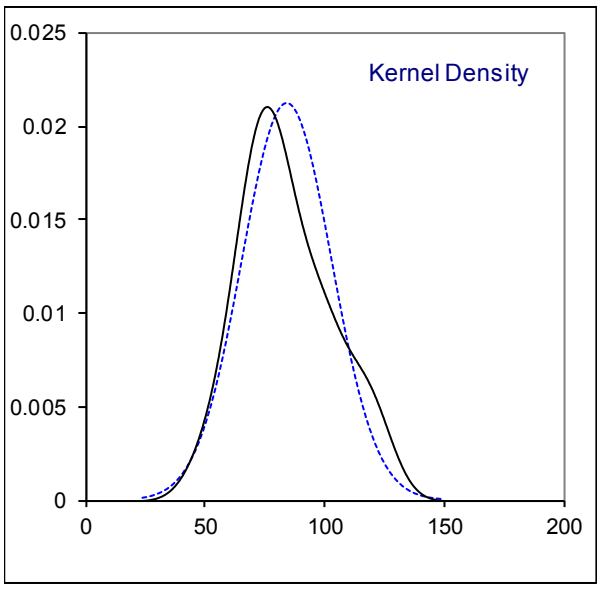


Determination of Water content by KF on sample #18035; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D6304-C	97		0.15	
179	D6304-C	82		-0.03	
237		-----		-----	
325	D6304-C	69		-0.18	
329	D6304-A	71		-0.15	
349		-----		-----	
432	D6304-C	53.2		-0.36	
496	D6304-C	67		-0.20	
663	D6304-C	120		0.41	
862	D6304-C	83		-0.01	
962	D6304-A	82.0		-0.03	
963	D6304-A	79		-0.06	
1146	D6304-C	69		-0.18	
1150	ISO12937	116		0.37	
1417	D6304-A	76		-0.10	
1433	ISO12937	100		0.18	
1748		-----		-----	
1799		-----		-----	
1941	D6304-C	99		0.17	
6016		-----		-----	
6199		-----		-----	

normality	OK
n	15
outliers	0
mean (n)	84.21
st.dev. (n)	18.754
R(calc.)	52.51
st.dev.(D6304:16e1)	86.247
R(D6304:16e1)	241.49

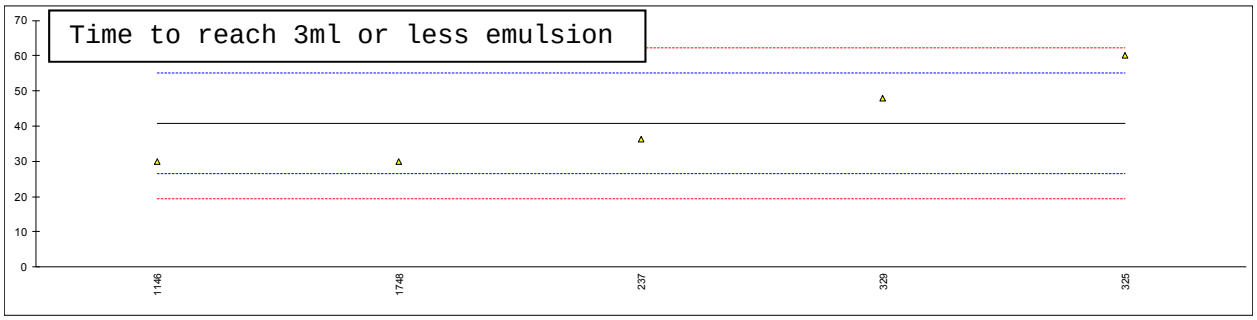


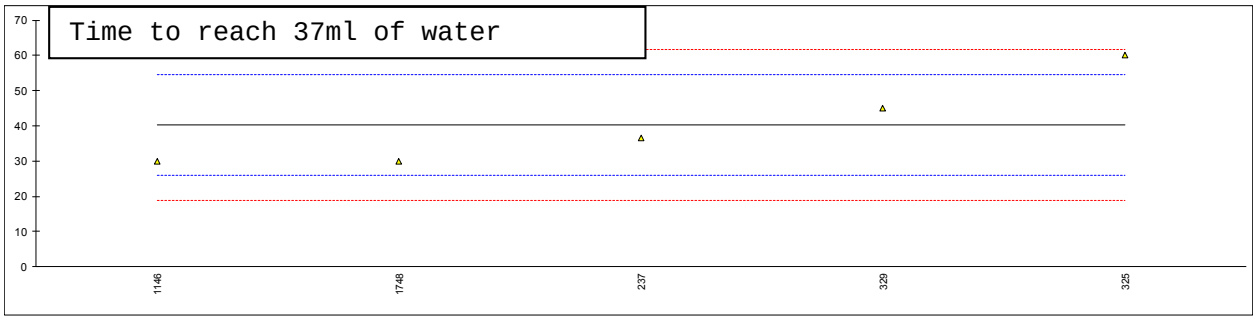


Determination of Water Separability at 54°C, distilled water on sample #18035; results in minutes

lab	method	Time to reach 3ml or less emulsion	z(targ)	Time to reach 37ml of water	z(targ)	Time to reach complete break (40-40-0)	z(targ)	Test aborted
178		-----	-----	-----	-----	-----	-----	-----
179	D1401	-----	-----	-----	-----	30	-----	-----
237	D1401	36.2	-0.65	36.6	-0.52	-----	-----	-----
325	D1401	60	2.68	60	2.76	>60	-----	60
329	D1401	48	1.00	45	0.66	60	-----	-----
349		-----	-----	-----	-----	-----	-----	-----
432	D1401	>30	-----	>30	-----	>30	-----	-----
496		-----	-----	-----	-----	-----	-----	-----
663		-----	-----	-----	-----	-----	-----	-----
862		-----	-----	-----	-----	-----	-----	30
962		-----	-----	-----	-----	-----	-----	-----
963		-----	-----	-----	-----	-----	-----	-----
1146	D1401	30	-1.52	30	-1.44	-----	-----	-----
1150		-----	-----	-----	-----	-----	-----	-----
1417		-----	-----	-----	-----	-----	-----	30
1433		-----	-----	-----	-----	-----	-----	-----
1748	D1401	30	-1.52	30	-1.44	35	-----	-----
1799		-----	-----	-----	-----	-----	-----	30
1941	D1401	>30	-----	>30	-----	>30	-----	-----
6016		-----	-----	-----	-----	-----	-----	-----
6199		-----	-----	-----	-----	-----	-----	-----

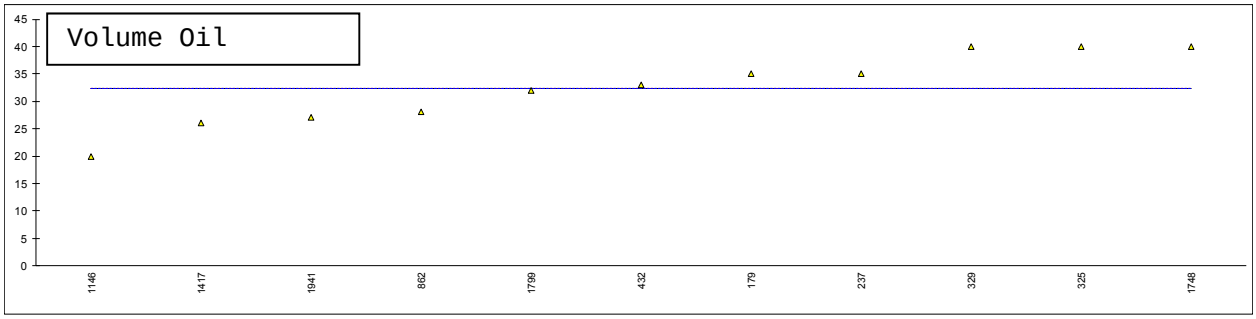
normality	unknown	unknown	n.a.
n	5	5	6
outliers	0	0	n.a.
mean (n)	40.8	40.3	>30
st.dev. (n)	12.99	12.61	n.a.
R(calc.)	36.4	35.3	n.a.
st.dev.(D1401:12e1)	7.14	7.14	n.a.
R(D1401:12e1)	20	20	n.a.

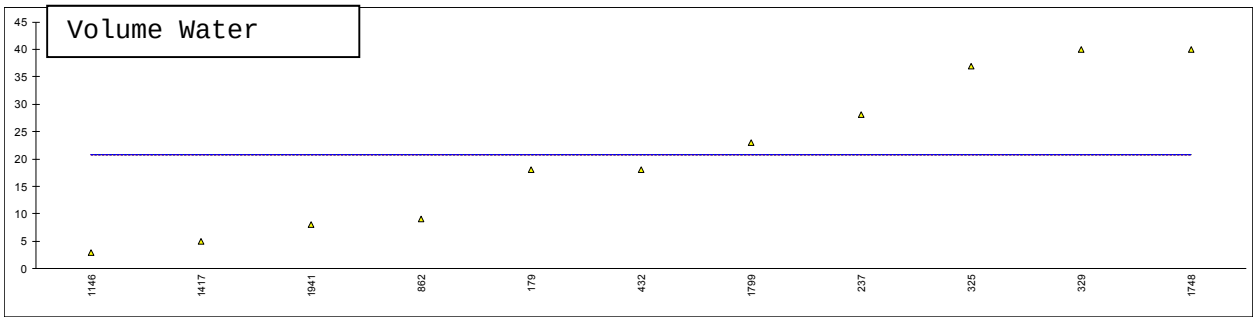


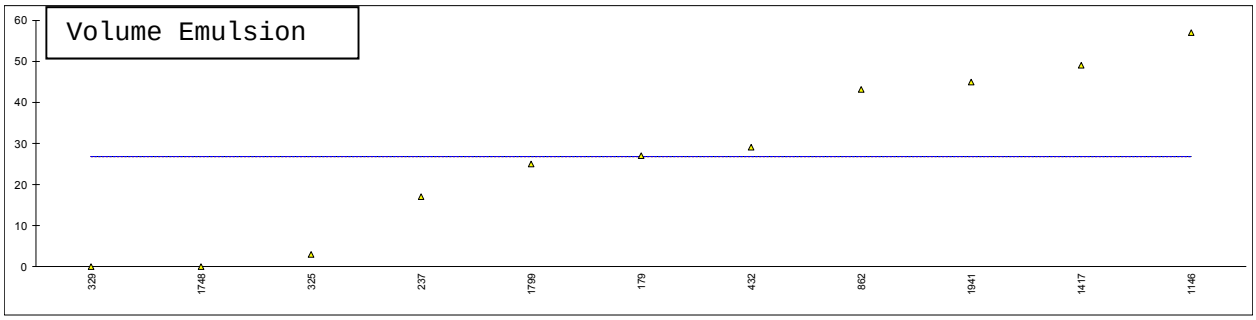


Determination of Water Separability at 54°C, distilled water on sample #18035; results in mL

lab	method	Volume oil phase	mark	Volume water phase	mark	Volume emulsion phase	mark	Remarks
178		-----		-----		-----		
179		35		18		27		
237		35		28		17		
325		40		37		3		
329		40		40		0		
349		-----		-----		-----		
432		33		18		29		
496		-----		-----		-----		
663		-----		-----		-----		
862		28		9		43		
962		-----		-----		-----		
963		-----		-----		-----		
1146		20		3		57		
1150		-----		-----		-----		
1417		26		5		49		
1433		-----		-----		-----		
1748		40		40		0		
1799		32		23		25		
1941		27		8		45		
6016		-----		-----		-----		
6199		-----		-----		-----		
	normality	OK		OK		OK		
	n	11		11		11		
	outliers	0		0		0		
	mean (n)	32.4		20.8		26.8		
	st.dev. (n)	6.56		13.93		20.26		
	R(calc.)	18.4		39.0		56.7		
compare								
	R(iis17L01)	3.9		3.9		3.2		





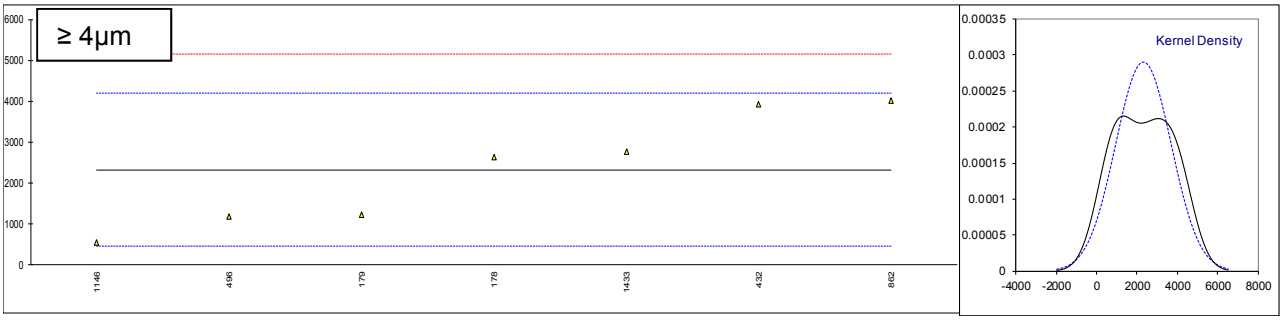


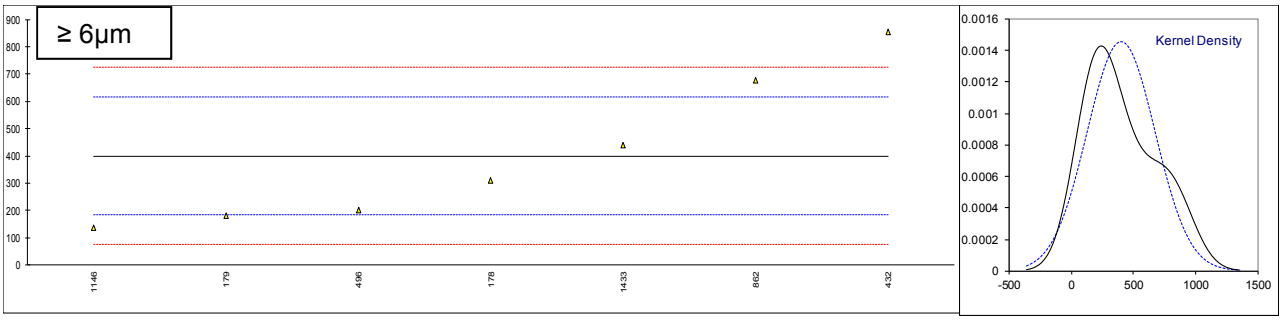
Determination of Level of Contamination on sample #18035; results in counts per ml.

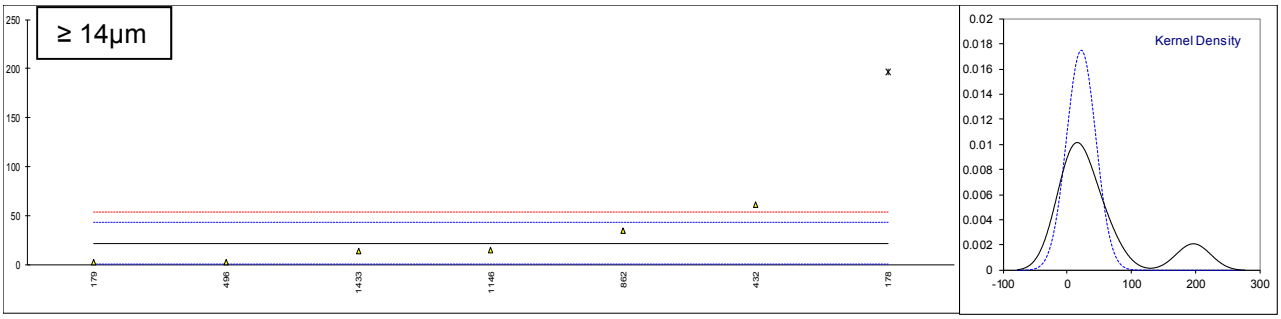
lab	method	≥ 4 µm (c)	mark	z(targ)	≥ 6 µm (c)	mark	z(targ)	≥ 14 µm (c)	mark	z(targ)
178	INH-1185	2630		0.32	311		-0.82	197	G(0.01)	16.50
179	D7647	1233	C	-1.16	181	C	-2.02	3	C	-1.79
237		-----		-----	-----		-----	-----		-----
325		-----		-----	-----		-----	-----		-----
329		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
432	ISO11500	3920		1.70	855		4.19	62		3.77
496	D7647	1178		-1.22	201		-1.83	3		-1.79
663		-----		-----	-----		-----	-----		-----
862	ISO11500	4007		1.79	676		2.54	35		1.23
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
1146	ISO11500	549		-1.89	138		-2.41	15		-0.66
1150		-----		-----	-----		-----	-----		-----
1417		-----		-----	-----		-----	-----		-----
1433	D7647	2764		0.47	439		0.36	14		-0.75
1748		-----		-----	-----		-----	-----		-----
1799		-----		-----	-----		-----	-----		-----
1941		-----		-----	-----		-----	-----		-----
6016		-----		-----	-----		-----	-----		-----
6199		-----		-----	-----		-----	-----		-----

normality	unknown	unknown	unknown
n	7	7	6
outliers	0	0	1
mean (n)	2325.9	400.14	22.000
st.dev. (n)	1373.63	273.511	22.8210
R(calc.)	3846.2	765.83	63.899
st.dev.(D7647:10)	938.65	108.610	10.6071
R(D7647:10)	2628.2	304.11	29.700

Lab 179: first reported 123303, 18138, 3213



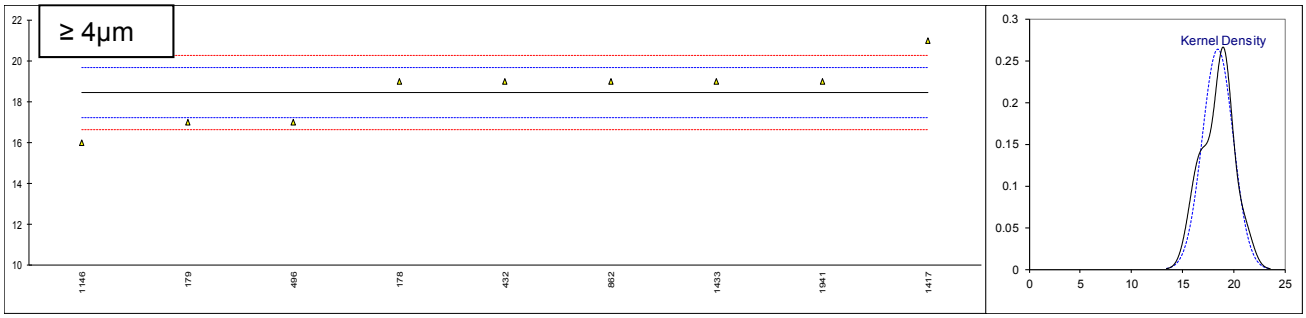


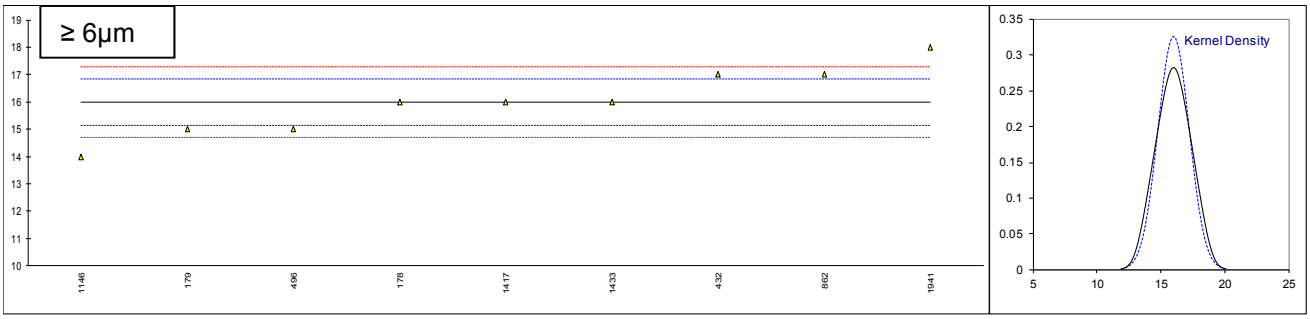


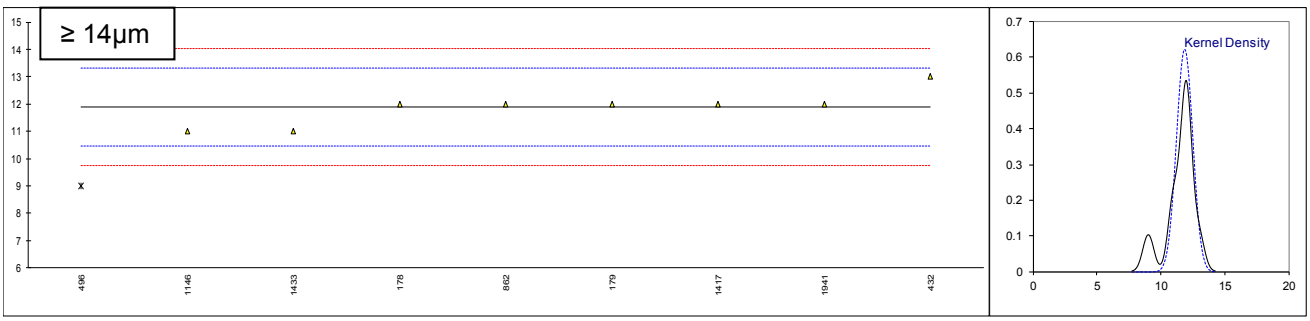
Determination of Level of Contamination acc. to ISO4406 scale on sample #18035; results in scale number

lab	method	≥ 4 µm (c)	mark	z(targ)	≥ 6 µm (c)	mark	z(targ)	≥ 14 µm (c)	mark	z(targ)
178	ISO4406	19		0.92	16		0.00	12		0.18
179	ISO4406	17		-2.38	15		-2.33	12		0.18
237		-----		-----	-----		-----	-----		-----
325		-----		-----	-----		-----	-----		-----
329		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
432	ISO4406	19		0.92	17		2.33	13		1.58
496	ISO4406	17		-2.38	15		-2.33	9	G(0.05)	-4.03
663		-----		-----	-----		-----	-----		-----
862	ISO11500	19		0.92	17		2.33	12		0.18
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
1146	ISO4406	16		-4.03	14		-4.67	11		-1.23
1150		-----		-----	-----		-----	-----		-----
1417	ISO4406	21		4.21	16		0.00	12		0.18
1433	D7647	19		0.92	16		0.00	11		-1.23
1748		-----		-----	-----		-----	-----		-----
1799		-----		-----	-----		-----	-----		-----
1941	ISO4406	19		0.92	18		4.67	12		0.18
6016		-----		-----	-----		-----	-----		-----
6199		-----		-----	-----		-----	-----		-----

normality	OK	OK	OK
n	9	9	8
outliers	0	0	1
mean (n)	18.4	16.0	11.9
st.dev. (n)	1.51	1.22	0.64
R(calc.)	4.2	3.4	1.8
st.dev.(D7647:10)	0.61	0.43	0.71
R(D7647:10)	1.7	1.2	2





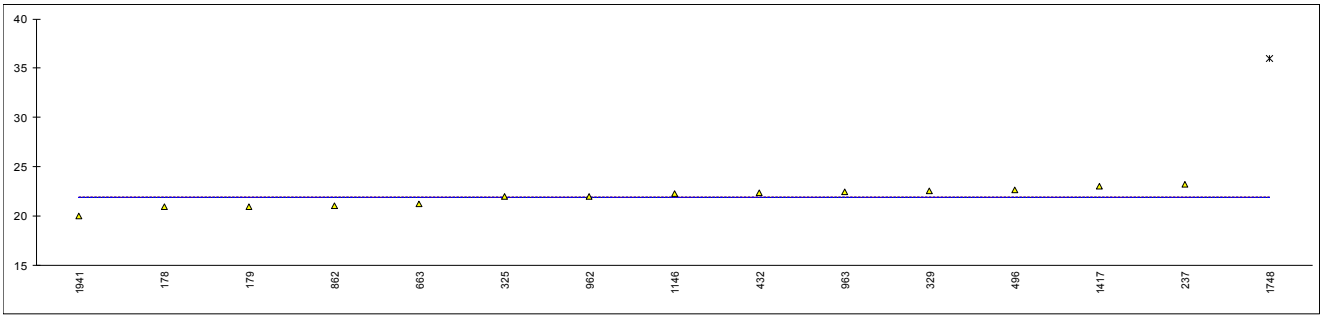


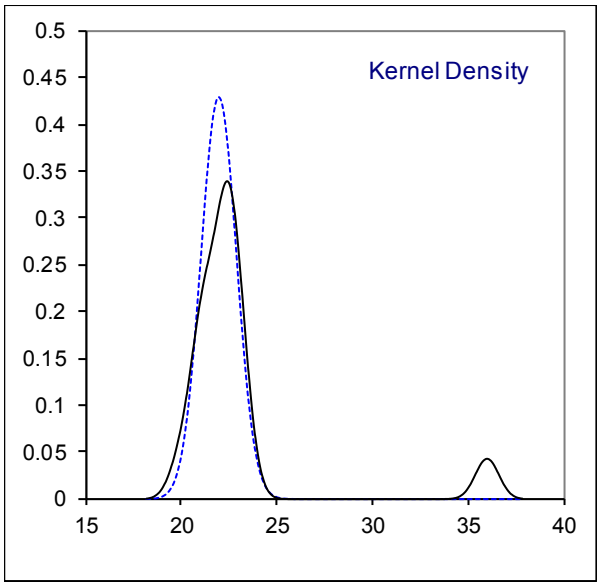
Determination of Calcium (Ca) on sample #18035; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	21		-----	
179	D5185	21		-----	
237	D5185	23.28		-----	
325	D5185	22		-----	
329	D4951	22.6		-----	
349		-----		-----	
432	D5185	22.4		-----	
496	D5185	22.68		-----	
663	D5185	21.28		-----	
862	D5185	21.1		-----	
962		22		-----	
963	D5185	22.5		-----	
1146	In house	22.31		-----	
1150		-----		-----	
1417	D5185	23		-----	
1433		-----		-----	
1748	D6481	36	G(0.01)	-----	
1799		-----		-----	
1941	D5185	20		-----	
6016		-----		-----	
6199		-----		-----	

	normality	OK
	n	14
	outliers	1
	mean (n)	21.939
	st.dev. (n)	0.9300
	R(calc.)	2.604
	st.dev.(D5185:18)	(0.2968)
	R(D5185:18)	(0.831)

Application range: 40 – 9000 mg/kg



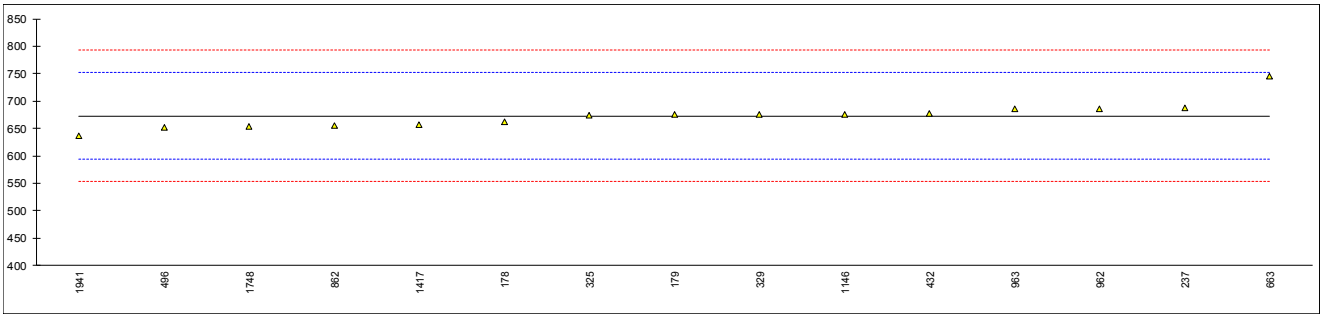


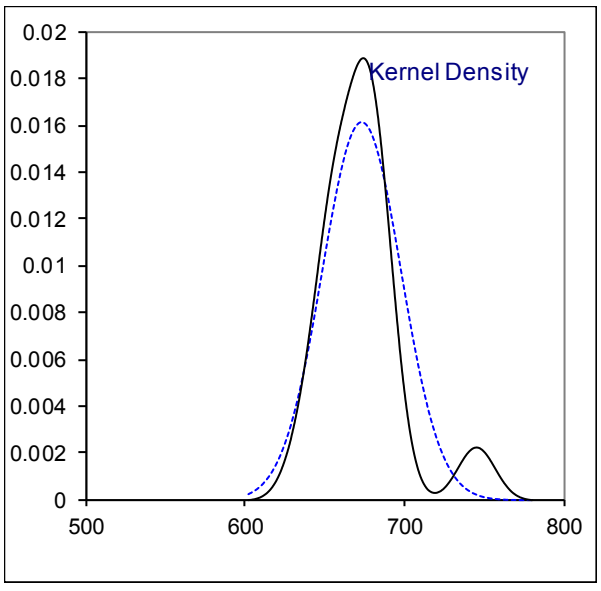
Determination of Phosphorus (P) on sample #18035; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	662		-0.28	
179	D5185	675		0.05	
237	D5185	687.2		0.35	
325	D5185	674		0.02	
329	D4951	675		0.05	
349		-----		-----	
432	D5185	676.6		0.09	
496	D5185	652.05		-0.53	
663	D5185	745.30		1.81	
862	D5185	655.5		-0.44	
962		686		0.32	
963	D5185	685.5		0.31	
1146	In house	675.1		0.05	
1150		-----		-----	
1417	D5185	657		-0.40	
1433		-----		-----	
1748	D6481	653		-0.50	
1799		-----		-----	
1941	D5185	637		-0.91	
6016		-----		-----	
6199		-----		-----	

normality	not OK
n	15
outliers	0
mean (n)	673.08
st.dev. (n)	24.783
R(calc.)	69.39
st.dev.(D5185:18)	39.842
R(D5185:18)	111.56

Application range: 10 – 1000 mg/kg





Determination of Zinc (Zn) on sample #18035; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	1		-----	
179	D5185	<1		-----	
237	D5185	2.102		-----	
325	D5185	2		-----	
329	D4951	2		-----	
349		-----		-----	
432	D5185	<1		-----	
496	D5185	1.868		-----	
663	D5185	1.433		-----	
862	D5185	<1		-----	
962		1		-----	
963	D5185	1.23		-----	
1146	In house	2.123		-----	
1150		-----		-----	
1417	D5185	0.183		-----	
1433		-----		-----	
1748	D6481	27		-----	False positive test result?
1799		-----		-----	
1941	D5185	<1		-----	
6016		-----		-----	
6199		-----		-----	
	normality	n.a.			
	n	14			
	outliers	n.a.			
	mean (n)	<2			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	st.dev.(D5185:18)	n.a.			
	R(D5185:18)	n.a.			Application range: 60 – 1600 mg/kg

APPENDIX 2

Number of participants per country

1 lab in	AUSTRIA
2 labs 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	POLAND
1 lab in	ROMANIA
2 labs in	SAUDI ARABIA
1 lab in	SERBIA
1 lab in	SPAIN
1 lab in	THAILAND
1 lab in	UNITED KINGDOM
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 E178:02
3 ISO 5725:86
4 ISO 5725, parts 1-6, 1994
5 ISO13528:05
6 ISO17043:10
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 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 W. Horwitz and R. Albert, J. AOAC Int., 79, 3, 589, (1996)