Results of Proficiency Test
Gear Oil (fresh)
April 2019

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: A. Lewinska, MSc.
Correctors: ing. A.S. Noordman-de Neef & ing. M. Meijer
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1 INTRODUCTION

Since 2015, the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of fresh Gear Oil every year. During the annual proficiency testing program 2018/2019, it was decided to continue the proficiency tests for the analysis of fresh Gear Oil. In this interlaboratory study, 26 laboratories in 20 different countries did register for participation. See appendix 2 for the number of participants per country.
In this report, the results of the 2019 proficiency test for fresh Gear Oil are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send one sample fresh Gear Oil of 1.5L in two bottles and labelled #19055.
Participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the 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 June 2018 (iis-protocol, version 3.5).
This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

The necessary bulk material of fresh Gear Oil was purchased from a local supplier. The 100 liters bulk material was homogenized and transferred into 35 amber glass bottles of 1 liter and 35 amber glass bottles of 0.5 liter. All bottles were labelled #19055. The homogeneity of the subsamples #19055 was checked by the determination of Density at 15°C in accordance with ASTM D4052 on 8 stratified randomly selected samples.

	Density at 15°C in kg/L
Sample #19055-1	0.88757
Sample #19055-2	0.88757
Sample #19055-3	0.88757
Sample #19055-4	0.88757
Sample #19055-5	0.88757
Sample #19055-6	0.88758
Sample #19055-7	0.88757
Sample #19055-8	0.88758

Table 1: homogeneity test results of subsamples #19055

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 ISO13528, Annex B2 in the next table.

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

Table 2: evaluation of the repeatability of subsamples #19055

The calculated repeatability was in agreement with 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneity of the subsamples #19055 was assumed.

To each of the participating laboratories, one 1L bottle and one 0.5L bottle both labelled #19055 were sent on April 03, 2019. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYSES

The participants were requested to determine on sample #19055: Acid Number (Total), Copper Corrosion 3hrs at 100°C, 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), Sulfur, Water, Water Separability at 82°C, Level of Contamination, Calcium, Phosphorus and Zinc.
Also, some extra information were asked about the determination of Acid Number and Foaming Characteristics.

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 test results, but report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the appropriate reference test methods that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyses). Additional or corrected test results are used for data analysis and 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 June 2018 (iis-protocol, version 3.5).
For the statistical evaluation the unrounded (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.
First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'.
After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

According to ISO5725 the original test results per determination were submitted to Dixon's 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. In this PT, the criterion of ISO13528, paragraph 9.2.1, was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis.

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

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. 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.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency tests could be used.
When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated according to:

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

The $z_{(target)}$ scores are listed in the result tables of appendix 1.

Absolute values for z<2 are very common and absolute values for z>3 are very rare.
The usual interpretation of z-scores is as follows:

	$\lvert z\rvert < 1$	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. The reporting participants were on time with submitting the test results. Three participants did not report any test results. Not all participants were able to report test results for all the requested tests. In total 23 participants reported 400 test results. Observed were 14 outlying test results, which is 3.5% 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 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 original data. The abbreviations, used in these tables, are listed in appendix 3.

In the iis PT reports, ASTM methods are referred to with a number (e.g. D7647) and an added designation for the year that the method was adopted or revised (e.g. D7647:10).
If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D7647:10(2018)). In the results tables of appendix 1 only the method number and year of adoption or revision (e.g. D7647:10) will be used.

Sample #19055

Acid Number (Total): This determination was problematic dependent on mode used (end point and volume of titration solvent). No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D664-A:18e2; IP and BEP 60mL but not in agreement with D664-A:18e2; IP and BEP 125mL. When evaluated separately over BEP or IP the calculated reproducibilities of BEP and IP are in agreement with the precision data of the procedures of BEP and IP (60mL).
Remarkably, only three participants used pH 10 for BEP instead of pH 11 as mentioned in method ASTM D664:18e2.

Copper Corrosion: This determination was not problematic. All reporting participants agreed on classification 1 (1a,1b).

Density at 15°C: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D4052:18a.

Flash Point COC: This determination was very problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements of ASTM D92:18.

Flash Point PMcc: This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D93-A:18.

Foaming Characteristics (Tendency and Stability): This determination was very problematic. No statistical outliers were observed. However, the calculated reproducibilities in the Foam Tendency determination for sequence I, II and III are not at all in agreement with the requirements of ASTM D892:18.
The test results for sequence I is bimodal distributed. Therefore, it was decided not to calculate z-scores.
The calculated reproducibility in the Foam Stability for sequence I is not at all in agreement with the requirements of ASTM D892:18.
All reporting participants reported 0 mL for Foam Stability for sequence II and III. Therefore, no z-scores were calculated.

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 D445:18.

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

Viscosity Index: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D2270:10.
Also, iis calculated the Viscosity Index from the test results reported for the kinematic viscosities at 40°C and 100°C. Two calculation errors were observed.

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

Pour Point - Automated: This determination was very problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements of ASTM D5950:14.
The test results are bimodal distributed. Therefore, it was decided not to calculate z-scores.

Rust Prevention: All reporting participants agreed on a classification as “Pass” / “no rusting”.

Sulfur: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D4294:16e1.

Water: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D6304:16e1.

Water Separability: This determination is not problematic. No statistical outliers were observed in “time to reach 3mL or less emulsion” and “time to reach 37mL of water”. The calculated reproducibilities are in agreement with the requirements of ASTM D1401:18b.
Remarkably, the calculated reproducibilities of the volume oil phase, the volume water phase and the volume emulsion phase are very low compared to the calculated reproducibilities of iis18L01 PT of 2018 (e.g. 18.4 vs 3.0).

Level of Contamination: This determination was very problematic. In total six statistical outliers were observed over six parameters. Six other test results were excluded because the related test values were statistical outliers. Only the calculated reproducibility for Level of Contamination acc. ISO4406 scale >14µm after rejection of the suspect data is in full agreement with the requirements of ASTM D7647:10(2018). All other calculated reproducibilities are not at all in agreement with the requirements of ASTM D7647:10(2018).

Calcium as Ca: The consensus value for the Calcium determination was below the application range of ASTM D5185:18. Therefore, no z-scores were calculated.

Phosphorus as P: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D5185:18.

Zinc as Zn: The consensus value for the Zinc determination was below the application range of ASTM D5185:18. Therefore, no z-scores were calculated.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average results, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from literature reference test methods (in casu ASTM, EN test methods) or previous proficiency tests are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Acid Number (Total)	mg KOH/g	19	0.36	0.13	0.18
Copper Corrosion, 3hrs at 100°C	rating	17	1	n.a.	n.a.
Density at 15°C	kg/m^3	20	887.6	0.3	0.5
Flash Point COC	°C	17	271	37	18
Flash Point PMcc	°C	17	201	8	14
Foaming Tendency (Seq I)	mL	10	174	437	(66)
Foaming Tendency (Seq II)	mL	10	236	220	116
Foaming Tendency (Seq III)	mL	10	9	21	12
Kinematic Viscosity at 40°C	mm^2/s	23	217.24	2.57	2.65
Kinematic Viscosity at 100°C	mm^2/s	22	19.395	0.209	0.268
Viscosity Index		22	101.0	1.2	2
Pour Point, Manual	°C	11	-17	10	9
Pour Point, Automated	°C	10	-18	17	(5)
Rust Prevention (distilled water)		4	Pass	n.a.	n.a.
Sulfur	mg/kg	9	8326	1099	646
Water	mg/kg	17	77	55	229
Water Separability at 82°C					
- Time ≤ 3 mL emulsion	minutes	7	21	12	25
- Time 37 mL water	minutes	7	22	12	25
- Time to complete break	minutes	6	24	10	25
- Volume Oil phase	mL	10	41	3	18
- Volume Water phase	mL	10	39	5	39
- Volume Emulsion phase	mL	10	1	3	57
Level of Contamination					
- ≥ 4µm (c)	counts/mL	4	3354	5033	3790
- ≥ 6µm (c)	counts/mL	4	710	978	540
- ≥14µm (c)	counts/mL	4	46	64	62
- ≥ 4µm (c)	scale no.	5	19.4	6.1	1.7
- ≥ 6µm (c)	scale no.	5	16.8	4.2	1.2
- ≥14µm (c)	scale no.	7	13.6	4.8	2.0
Calcium as Ca	mg/kg	19	12.5	3.0	(0.4)
Phosphorus as P	mg/kg	18	286	47	73
Zinc as Zn	mg/kg	18	6.0	3.0	(0.6)

Table 3: reproducibilities of the test results on sample #19055
NB. Results between brackets no z-scores are calculated

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 problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF APRIL 2019 WITH PREVIOUS PTS

	April 2019	April 2018	April 2017	April 2016	April 2015
Number of reporting labs	23	18	14	18	13
Number of results reported	400	350	177	215	125
Number of statistical outliers	14	14	8	14	6
Percentage outliers	3.5%	4.0%	4.5%	6.5%	4.8%

Table 4: comparison with previous proficiency tests

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

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

Determination	April 2019	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	(--)	+/-	++	+/-	+/-
Sulfur	-	--	-	+/-	-
Water	++	++	++	n.e.	n.e.
Water Separability at 82°C	++	-	+	++	++
Level of Contamination	--	--	n.e.	n.e.	n.e.
Calcium as Ca	(--)	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
nb. Results between brackets no z-scores are calculated

The following performance categories were used:

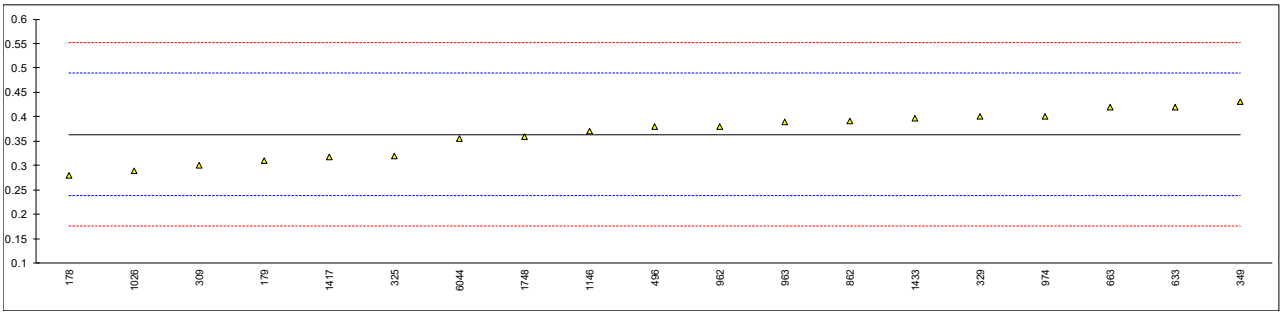
++: group performed much better than the reference test method
+ : group performed better than the reference test method
+/-: group performance similar to the reference test method
- : group performed worse than the reference test method
-- : group performed much worse than the reference test method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

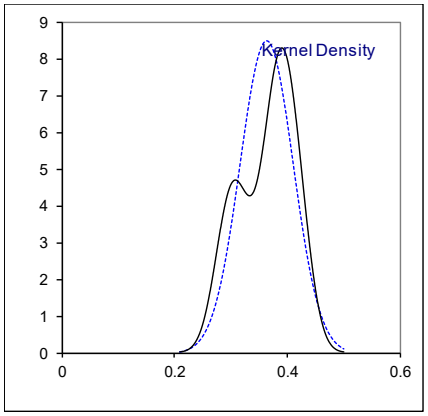
APPENDIX 1

Determination of Acid Number (Total) on sample #19055; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks	end point determination	titration solvent volume
178	INH-1118	0.28		-1.33		Inflection Point	---
179	D664-A	0.31		-0.86		Inflection Point	125 mL
237		-----		-----		---	---
257		-----		-----		---	---
309	D664	0.30		-1.02		Buffer End Point (pH 10)	60 mL
325	D664-A	0.32		-0.70		Buffer End Point (pH 10)	125 mL
329	D664-A	0.40		0.58		Inflection Point	60 mL
349	D664-A	0.43		1.06		Buffer End Point (pH 10)	125 mL
432		-----		-----		---	---
496	D664-A	0.38		0.26		Buffer End Point (pH 11)	60 mL
633	D664-A	0.42		0.90		Inflection Point	125 mL
663	D664-A	0.419		0.88		Inflection Point	60 mL
862	D664-A	0.391		0.44		Inflection Point	60 mL
962	D664-A	0.38		0.26		Inflection Point	125 mL
963	D664-A	0.39		0.42		Inflection Point	60 mL
974	D664-A	0.4		0.58		Inflection Point	125 mL
1011		-----		-----		---	---
1026	D664-A	0.29		-1.18		Buffer End Point (pH 11)	60 mL
1146	D664-A	0.370		0.10		Buffer End Point (pH 11)	60 mL
1417	D664-A	0.317		-0.74		Buffer End Point (pH 11)	125 mL
1433	D664-A	0.397		0.53		Inflection Point	125 mL
1748	D664-A	0.36		-0.06		Inflection Point	125 mL
6016		-----		-----		---	---
6035		-----		-----		---	---
6044	D664-A	0.356		-0.12		---	---
6253		-----		-----		---	---

			BEP only	Inflection point only
normality	OK		OK	suspect
n	19		7	11
outliers	0		0	0
mean (n)	0.3637		0.3439	0.3770
st.dev. (n)	0.04708		0.05087	0.04429
R(calc.)	0.1318		0.1424	0.1240
st.dev.(D664-A:18e2)	0.06268		---	---
R(D664-A:18e2)	0.1755	IP-60mL	---	0.1808
Compare:				
R(D664-A:18e2)	0.2065	BEP (pH-10)-60mL	0.1956	---
R(D664-A:18e2)	0.1091	BEP (pH-10)-125mL		
R(D664-A:18e2)	0.0762	IP-125mL		





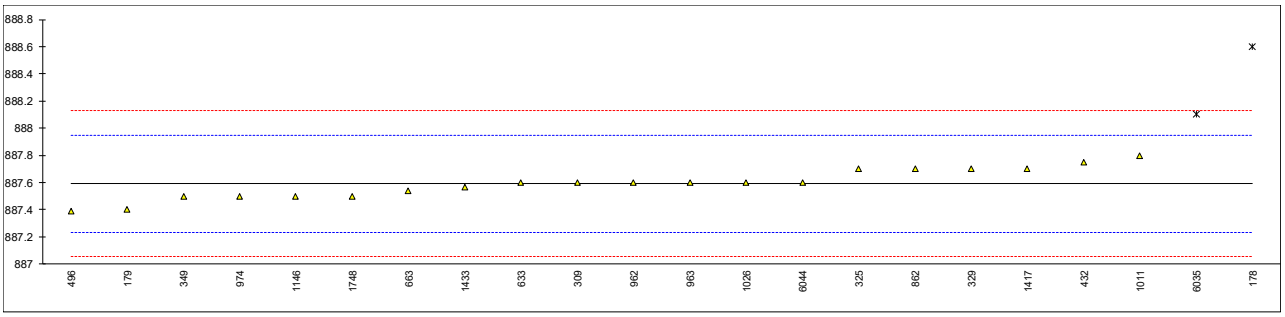
Determination of Copper Corrosion 3 hours at 100°C on sample #19055;

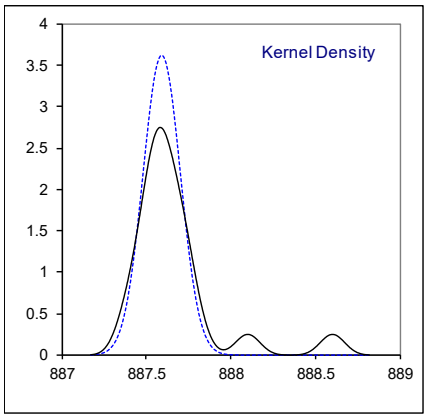
lab	method	value	mark	z(targ)	remarks
178	D130	1A		-----	
179	D130	1A		-----	
237		-----		-----	
257		-----		-----	
309	D130	1A		-----	
325	D130	1A		-----	
329		-----		-----	
349		-----		-----	
432		-----		-----	
496	D130	1a		-----	
633	D130	1a		-----	
663	D130	1a		-----	
862	D130	1a		-----	
962	D130	1A		-----	
963	D130	1a		-----	
974	D130	1a		-----	
1011	D130	1b		-----	
1026	D130	1A		-----	
1146		-----		-----	
1417	IP154	1A		-----	
1433	D130	1a		-----	
1748	D130	1a		-----	
6016		-----		-----	
6035	ISO2160	1		-----	
6044		-----		-----	
6253		-----		-----	
	n	17			
	mean (n)	1 (1a, 1b)			

Determination of Density at 15°C on sample #19055; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
178	D4052	888.6	R(0.01)	5.64	
179	D4052	887.4		-1.08	
237		-----		-----	
257		-----		-----	
309	D4052	887.60	C	0.04	Reported 0.88760 kg/m^3
325	D4052	887.7		0.60	
329	D4052	887.7		0.60	
349	D4052	887.5		-0.52	
432	D4052	887.75		0.88	
496	D4052	887.39		-1.13	
633	D4052	887.60		0.04	
663	D4052	887.54		-0.29	
862	D4052	887.7		0.60	
962	D4052	887.6		0.04	
963	D4052	887.6		0.04	
974	D4052	887.5		-0.52	
1011	D4052	887.8		1.16	
1026	D4052	887.6		0.04	
1146	D4052	887.5		-0.52	
1417	IP365	887.7		0.60	
1433	D4052	887.57		-0.14	
1748	D4052	887.5		-0.52	
6016		-----		-----	
6035	ISO12185	888.1	R(0.01)	2.84	
6044	D4052	887.6	C	0.04	first reported 0.8876 kg/m^3
6253		-----		-----	

normality	OK
n	20
outliers	2
mean (n)	887.592
st.dev. (n)	0.1101
R(calc.)	0.308
st.dev.(D4052:18a)	0.1786
R(D4052:18a)	0.5

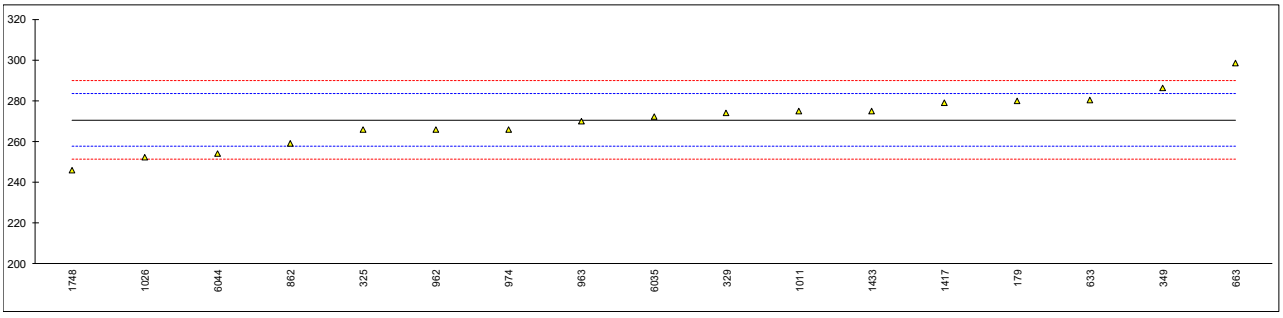


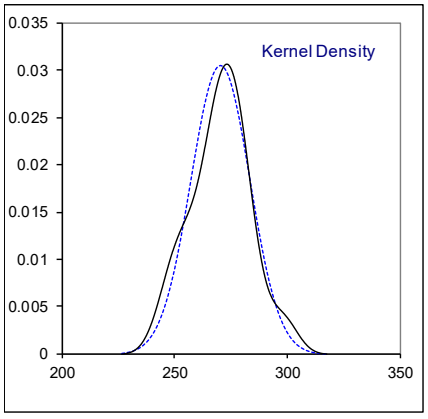


Determination of Flash Point COC on sample #19055; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D92	280		1.47	
237		-----		-----	
257		-----		-----	
309		-----		-----	
325	D92	266		-0.70	
329	D92	274.0		0.54	
349	D92	286		2.41	
432		-----		-----	
496		-----		-----	
633	D92	280.4		1.54	
663	D92	298.45		4.34	
862	D92	259		-1.79	
962	D92	266.0		-0.70	
963	D92	270		-0.08	
974	D92	266.0		-0.70	
1011	D92	275		0.70	
1026	D92	252		-2.88	
1146		-----		-----	
1417	D92	279		1.32	
1433	D92	275		0.70	
1748	D92	246		-3.81	
6016		-----		-----	
6035	ISO2592	272.0		0.23	
6044	D92	254		-2.57	
6253		-----		-----	

normality	OK
n	17
outliers	0
mean (n)	270.52
st.dev. (n)	13.098
R(calc.)	36.67
st.dev.(D92:18)	6.429
R(D92:18)	18

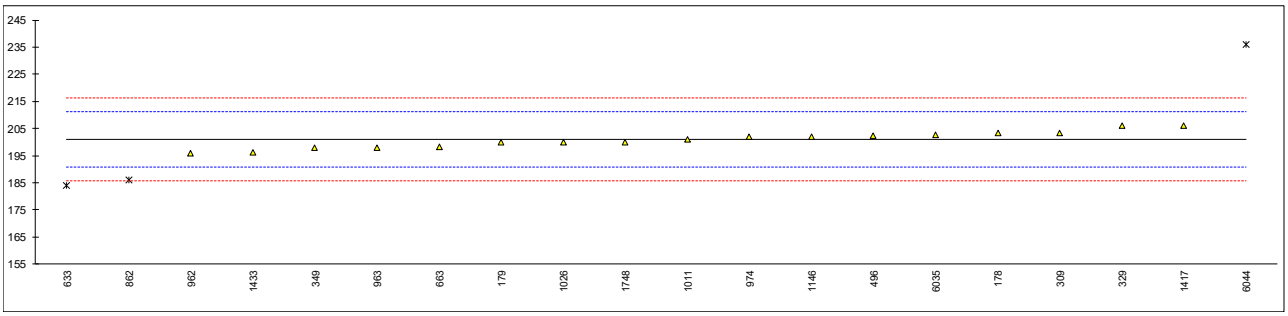


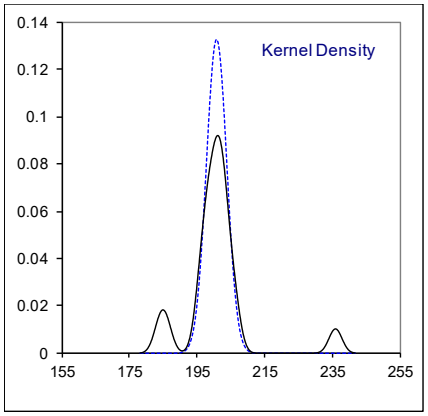


Determination of Flash Point PMcc on sample #19055; results in °C

lab	method	value	mark	z(targ)	remarks
178	D93-A	203.5		0.51	
179	D93-A	200.0		-0.18	
237		-----		-----	
257		-----		-----	
309	D93	203.5		0.51	
325		-----		-----	
329	D93-A	206.0		1.00	
349	D93-A	198		-0.57	
432		-----		-----	
496	D93-A	202.2		0.25	
633	D93-A	184.13	DG(0.01)	-3.29	
663	D93-A	198.27		-0.52	
862	D93-A	186	DG(0.01)	-2.93	
962	D93-A	196.0		-0.96	
963	D93-A	198		-0.57	
974	D93-A	202.0		0.22	
1011	D93	201.0		0.02	
1026	ISO2719-A	200.0		-0.18	
1146	D93-A	202.1		0.24	
1417	D93-A	206		1.00	
1433	D93-A	196.25		-0.91	
1748	D93-A	200		-0.18	
6016		-----		-----	
6035	ISO2719-A	202.5		0.31	
6044	D93-A	236	C,G(0.01)	6.89	first reported 216
6253		-----		-----	

normality	OK
n	17
outliers	3
mean (n)	200.90
st.dev. (n)	3.003
R(calc.)	8.41
st.dev.(D93-A:18)	5.094
R(D93-A:18)	14.26



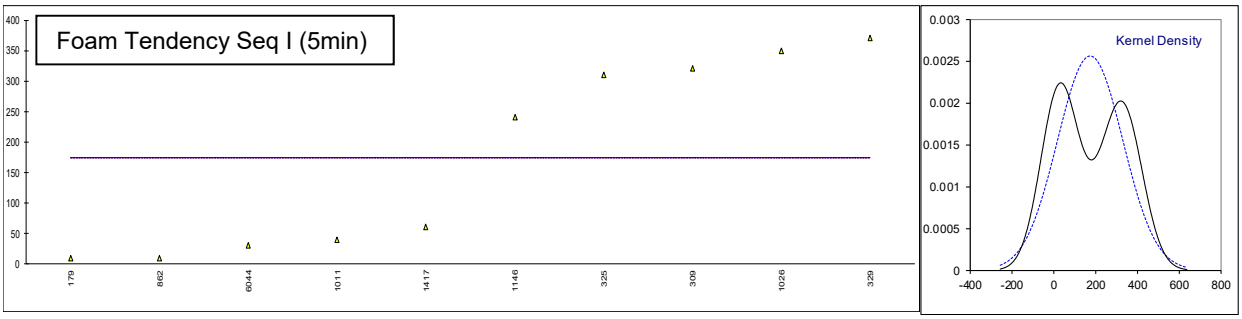


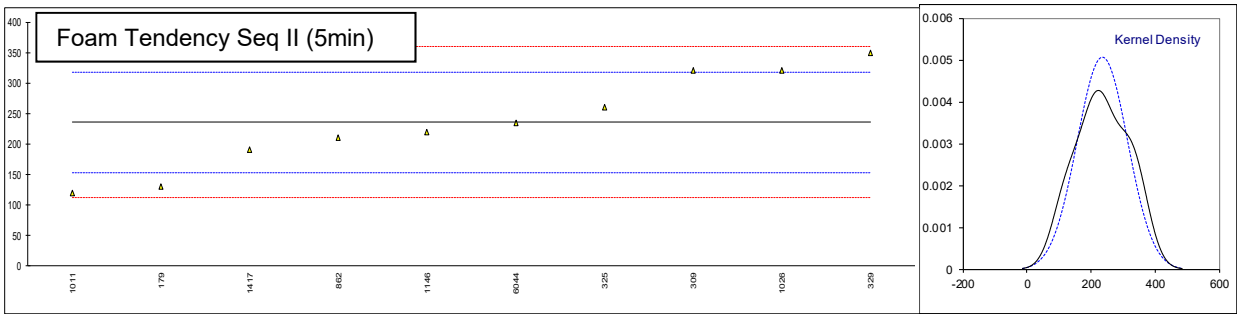
Determination of Foaming Tendency, Sequence I, II and III (5 min. blowing period) on sample #19055; results in mL

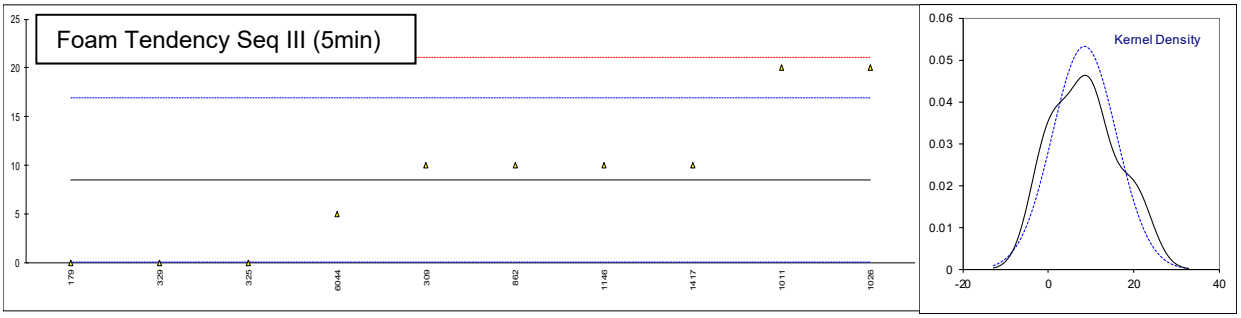
lab	method	Sample used	Diffuser	Seq I	mark	z(targ)	Seq II	mark	z(targ)	Seq III	mark	z(targ)
178		---	---	-----		-----	-----		-----	-----		-----
179	D892	As rec.	Metal	10		-----	130		-2.55	0		-2.03
237		---	---	-----		-----	-----		-----	-----		-----
257		---	---	-----		-----	-----		-----	-----		-----
309	D892	As rec.	Metal	320		-----	320		2.04	10		0.36
325	D892	As rec.	Metal	310		-----	260		0.59	0		-2.03
329	D892	After agit. (A)	Stone	370		-----	350		2.77	0		-2.03
349		---	---	-----		-----	-----		-----	-----		-----
432		---	---	-----		-----	-----		-----	-----		-----
496		---	---	-----		-----	-----		-----	-----		-----
633		---	---	-----		-----	-----		-----	-----		-----
663		---	---	-----		-----	-----		-----	-----		-----
862	D892	As rec.	Metal	10		-----	210		-0.62	10		0.36
962		---	---	-----		-----	-----		-----	-----		-----
963		---	---	-----		-----	-----		-----	-----		-----
974		---	---	-----		-----	-----		-----	-----		-----
1011	D892	---	---	40		-----	120		-2.79	20		2.74
1026	D892	As rec.	Stone	350		-----	320		2.04	20		2.74
1146	ISO6247	As rec.	Metal	240		-----	220		-0.37	10		0.36
1417	D892	As rec.	Metal	60		-----	190		-1.10	10		0.36
1433		---	---	-----		-----	-----		-----	-----		-----
1748		---	---	-----		-----	-----		-----	-----		-----
6016		---	---	-----		-----	-----		-----	-----		-----
6035		---	---	-----		-----	-----		-----	-----		-----
6044	D892	As rec.	Metal	30		-----	235	C	-0.01	5	C	-0.83
6253				-----		-----	-----		-----	-----		-----

	Seq I	Seq II	Seq III
normality	OK	OK	OK
n	10	10	10
outliers	0	0	0
mean (n)	174	235.5	8.5
st.dev. (n)	156.01	78.47	7.47
R(calc.)	436.8	219.7	20.9
st.dev.(D892:18)	(23.65)	41.36	4.20
R(D892:18)	(66.2)	115.8	11.7

As rec. = As received
Lab 6044 first reported 5 and 235 respectively


Foam Tendency Seq I (5min)
Kernel Density


Foam Tendency Seq II (5min)
Kernel Density


Foam Tendency Seq III (5min)
Kernel Density

Determination of Foaming Stability, Sequence I, II and III (10 min. settling period) on sample #19055; results in mL

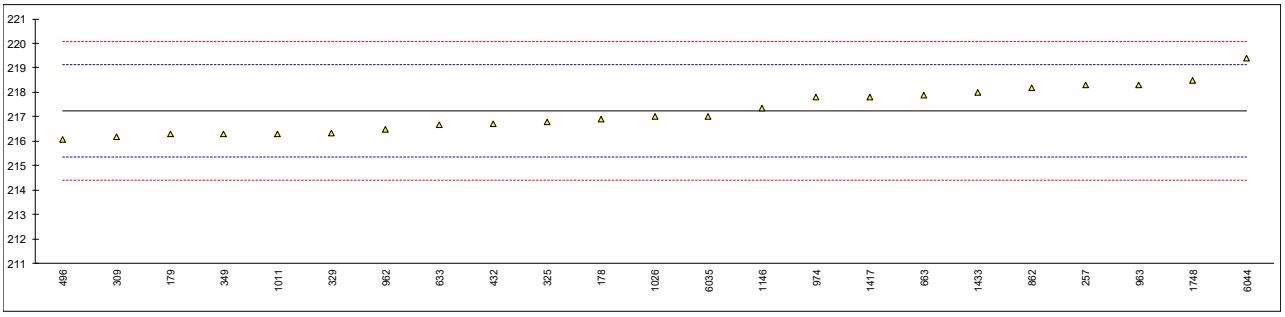
lab	method	Seq I	mark	z(targ)	Seq II	mark	z(targ)	Seq III	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179	D892	0		-----	0		-----	0		-----
237		-----		-----	-----		-----	-----		-----
257		-----		-----	-----		-----	-----		-----
309	D892	80		-----	0		-----	0		-----
325	D892	60		-----	0		-----	0		-----
329	D892	100		-----	0		-----	0		-----
349		-----		-----	-----		-----	-----		-----
432		-----		-----	-----		-----	-----		-----
496		-----		-----	-----		-----	-----		-----
633		-----		-----	-----		-----	-----		-----
663		-----		-----	-----		-----	-----		-----
862	D892	0		-----	0		-----	0		-----
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
974		-----		-----	-----		-----	-----		-----
1011	D892	0		-----	0		-----	0		-----
1026	D892	140		-----	0		-----	0		-----
1146	ISO6247	30		-----	0		-----	0		-----
1417	D892	0		-----	0		-----	0		-----
1433		-----		-----	-----		-----	-----		-----
1748		-----		-----	-----		-----	-----		-----
6016		-----		-----	-----		-----	-----		-----
6035		-----		-----	-----		-----	-----		-----
6044	D892	0		-----	0		-----	0	C	-----
6253		-----		-----	-----		-----	-----		-----
	normality	OK			n.a.			n.a.		
	n	10			10			10		
	outliers	0			0			0		
	mean (n)	41			0			0		
	st.dev. (n)	51.31			0			0		
	R(calc.)	143.7			0			0		
	st.dev.(D892:18)	(7.54)			(4.28)			0		
	R(D892:18)	(21.1)			(12.0)			0		

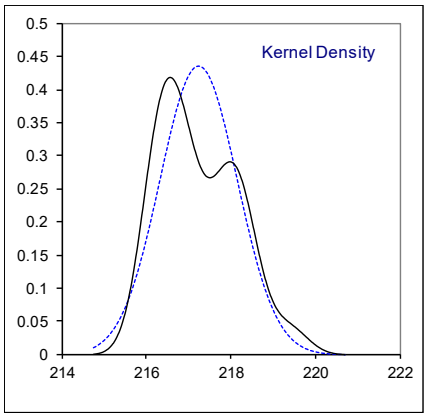
Lab 6044 first reported 20

Determination of Kinematic Viscosity at 40°C on sample #19055; results in mm²/s

lab	method	value	mark	z(targ)	remarks
178	D445	216.9		-0.36	
179	D445	216.3		-1.00	
237		-----		-----	
257	D7279	218.3	C	1.12	first reported 218.1
309	D445	216.2		-1.10	
325	D445	216.8		-0.47	
329	D445	216.35		-0.94	
349	D445	216.3		-1.00	
432	D445	216.7		-0.57	
496	D445	216.07		-1.24	
633	D7279 corrected to D445	216.68		-0.60	
663	D445	217.895		0.69	
862	D445	218.2		1.01	
962	D445	216.5		-0.79	
963	D445	218.3		1.12	
974	D445	217.8		0.59	
1011	D7042	216.3		-1.00	
1026	ISO3104	217.0		-0.26	
1146	D445	217.35		0.11	
1417	D445	217.8		0.59	
1433	D445	217.978		0.78	
1748	D7042	218.48		1.31	
6016		-----		-----	
6035	ISO3104	217.0		-0.26	
6044	D445	219.4		2.28	
6253		-----		-----	

normality	OK
n	23
outliers	0
mean (n)	217.244
st.dev. (n)	0.9159
R(calc.)	2.565
st.dev.(D445:18)	0.9466
R(D445:18)	2.650

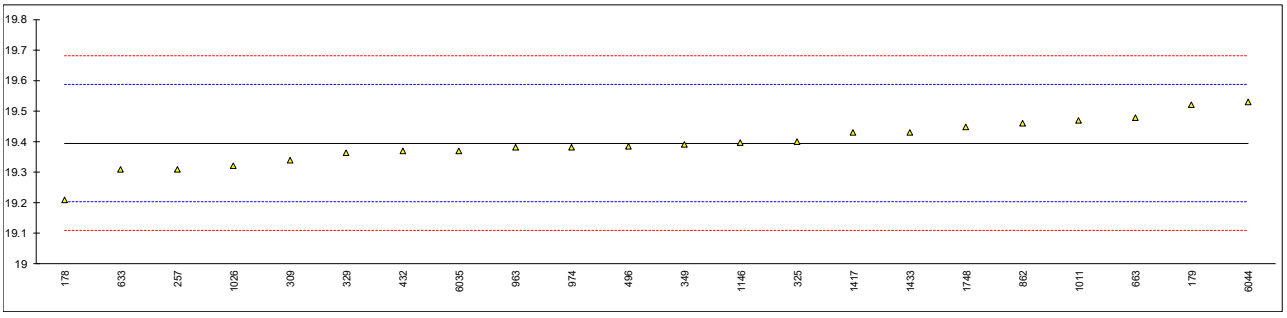


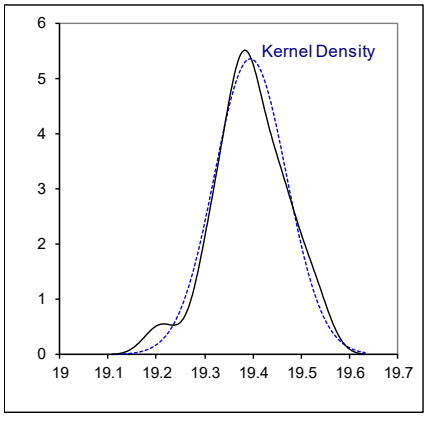


Determination of Kinematic Viscosity at 100°C on sample #19055; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178	D445	19.21		-1.93	
179	D445	19.52		1.31	
237		-----		-----	
257	D7279	19.31	C	-0.89	first reported 19.16
309	D445	19.34		-0.57	
325	D445	19.40		0.05	
329	D445	19.362		-0.34	
349	D445	19.39		-0.05	
432	D445	19.37		-0.26	
496	D445	19.383		-0.12	
633	D7279 corrected to D445	19.308		-0.91	
663	D445	19.479		0.88	
862	D445	19.46		0.68	
962		-----		-----	
963	D445	19.38		-0.16	
974	D445	19.38		-0.16	
1011	D7042	19.47		0.79	
1026	ISO3104	19.32		-0.78	
1146	D445	19.398		0.03	
1417	D445	19.43		0.37	
1433	D445	19.43		0.37	
1748	D7042	19.447		0.55	
6016		-----		-----	
6035	ISO3104	19.37		-0.26	
6044	D445	19.53		1.41	
6253		-----		-----	

normality	OK
n	22
outliers	0
mean (n)	19.395
st.dev. (n)	0.0745
R(calc.)	0.209
st.dev.(D445:18)	0.0956
R(D445:18)	0.268

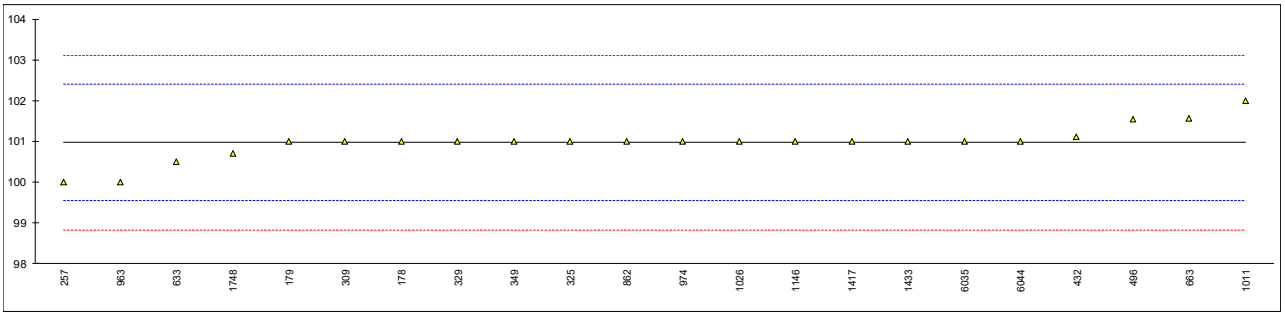


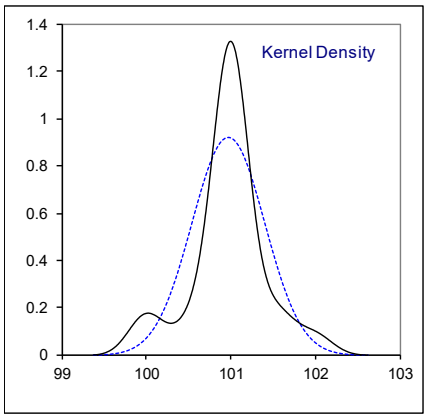


Determination of Viscosity Index (V.I.) on sample #19055

lab	method	value	mark	z(targ)	remarks
178	D2270	101	E	0.04	calc. error, iis calculated 99.7
179	D2270	101	E	0.04	calc. error, iis calculated 102.6
237		-----		-----	
257	D2270	100	C	-1.36	first reported 99
309	D2270	101		0.04	
325	D2270	101		0.04	
329	D2270	101		0.04	
349	D2270	101		0.04	
432	D2270	101.1		0.18	
496	D2270	101.53		0.78	
633	D2270	100.5		-0.66	
663	D2270	101.57		0.84	
862	D2270	101		0.04	
962		-----		-----	
963	D2270	100		-1.36	
974	D2270	101		0.04	
1011	D2270	102		1.44	
1026	D2270	101		0.04	
1146	D2270	101		0.04	
1417	D2270	101		0.04	
1433	ISO2909	101		0.04	
1748	D2270	100.7		-0.38	
6016		-----		-----	
6035	ISO2909	101		0.04	
6044	D2270	101		0.04	
6253		-----		-----	

normality	not OK
n	22
outliers	0
mean (n)	100.97
st.dev. (n)	0.433
R(calc.)	1.21
st.dev.(D2270:10)	0.714
R(D2270:10)	2

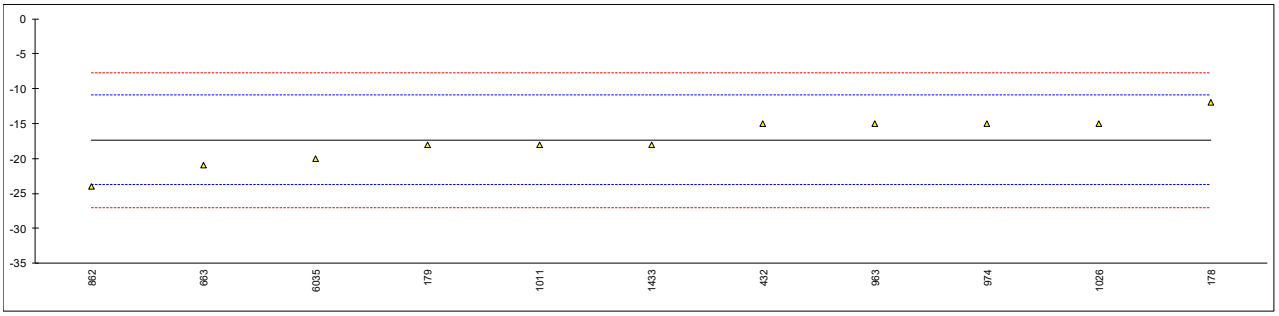


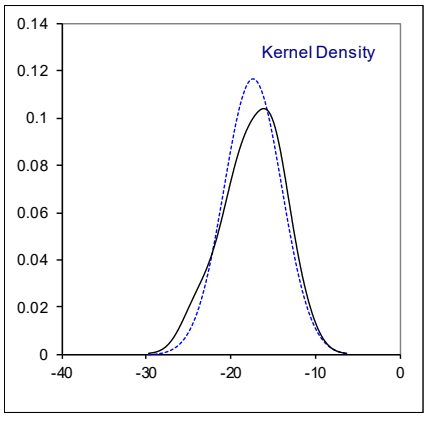


Determination of Pour Point, Manual on sample #19055; results in °C

lab	method	value	mark	z(targ)	remarks
178	D97	-12		1.67	
179	D97	-18		-0.20	
237		-----		-----	
257		-----		-----	
309		-----		-----	
325		-----		-----	
329		-----		-----	
349		-----		-----	
432	D97	-15		0.74	
496		-----		-----	
633		-----		-----	
663	D97	-21		-1.13	
862	D97	-24		-2.06	
962		-----		-----	
963	D97	-15		0.74	
974	D97	-15		0.74	
1011	D97	-18		-0.20	
1026	D97	-15		0.74	
1146		-----		-----	
1417		-----		-----	
1433	D97	-18		-0.20	
1748		-----		-----	
6016		-----		-----	
6035	ISO3016	-20.0		-0.82	
6044		-----		-----	
6253		-----		-----	

normality	OK
n	11
outliers	0
mean (n)	-17.36
st.dev. (n)	3.414
R(calc.)	9.56
st.dev.(D97:17b)	3.214
R(D97:17b)	9

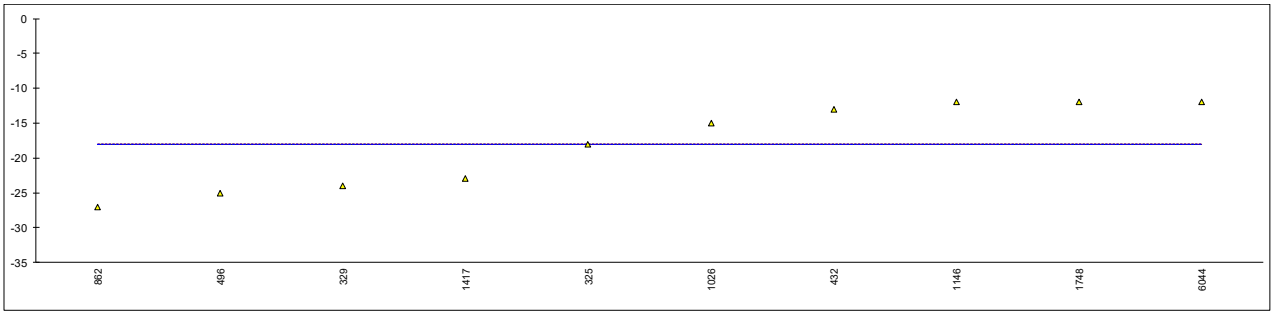


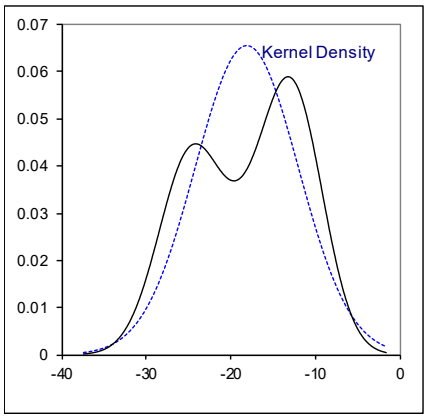


Determination of Pour Point, Automated, 1°C interval on sample #19055; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
257		-----		-----	
309		-----		-----	
325	D5950	-18		-----	
329		-24		-----	
349		-----		-----	
432	D5950	-13		-----	
496	D5950	-25		-----	
633		-----		-----	
663		-----		-----	
862	D5950	-27		-----	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1011		-----		-----	
1026	D5950	-15		-----	
1146	D6892	-12		-----	
1417	D5950	-23		-----	
1433		-----		-----	
1748	D7346	-12		-----	
6016		-----		-----	
6035		-----		-----	
6044	D6749	-12		-----	
6253		-----		-----	

		Only D5950
normality	OK	OK
n	10	6
outliers	0	0
mean (n)	-18.10	-20.17
st.dev. (n)	6.082	5.672
R(calc.)	17.03	15.88
st.dev.(D5950:14)	(1.607)	1.607
R(D5950:14)	(4.5)	4.5





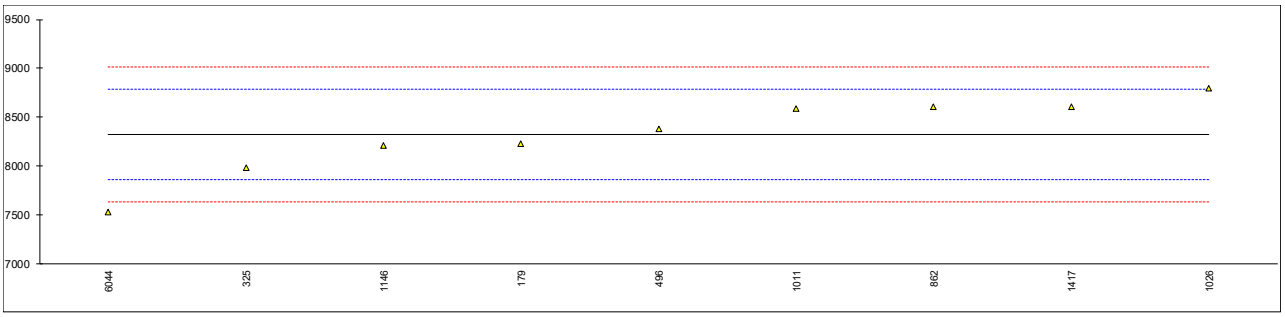
Determination of Rust Prevention, distilled water on sample #19055

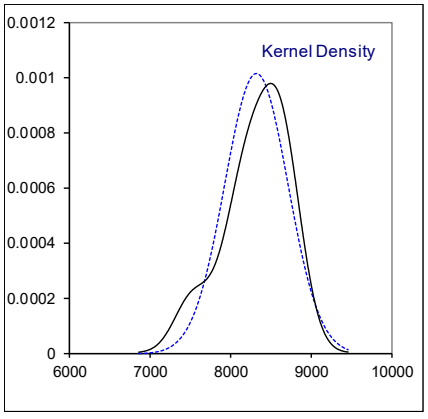
lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
237		-----		-----	
257		-----		-----	
309	D665	Passes		-----	
325		-----		-----	
329		-----		-----	
349		-----		-----	
432		-----		-----	
496	D665	pass - no rusting		-----	
633		-----		-----	
663		-----		-----	
862		-----		-----	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1011		-----		-----	
1026	D665	PASS		-----	
1146		-----		-----	
1417	D665	PASS		-----	
1433		-----		-----	
1748		-----		-----	
6016		-----		-----	
6035		-----		-----	
6044		-----		-----	
6253		-----		-----	
	n	4			
	mean (n)	Pass			

Determination of Sulfur on sample #19055; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D4294	8225		-0.44	
237		-----		-----	
257		-----		-----	
309		-----		-----	
325	D5185	7984		-1.48	
329		-----		-----	
349		-----		-----	
432		-----		-----	
496	D2622	8378.2		0.22	
633		-----		-----	
663		-----		-----	
862	D2622	8610		1.23	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1011	D6481	8590		1.14	
1026	D4294	8800	C	2.05	first reported 820
1146	D4294	8210		-0.50	
1417	In house	8610		1.23	
1433		-----		-----	
1748		-----		-----	
6016		-----		-----	
6035		-----		-----	
6044	D4294	7530	C	-3.45	first reported 0.753 mg/kg
6253		-----		-----	

normality	OK
n	9
outliers	0
mean (n)	8326.4
st.dev. (n)	392.53
R(calc.)	1099.1
st.dev.(D4294:16e1)	230.60
R(D4294:16e1)	645.7

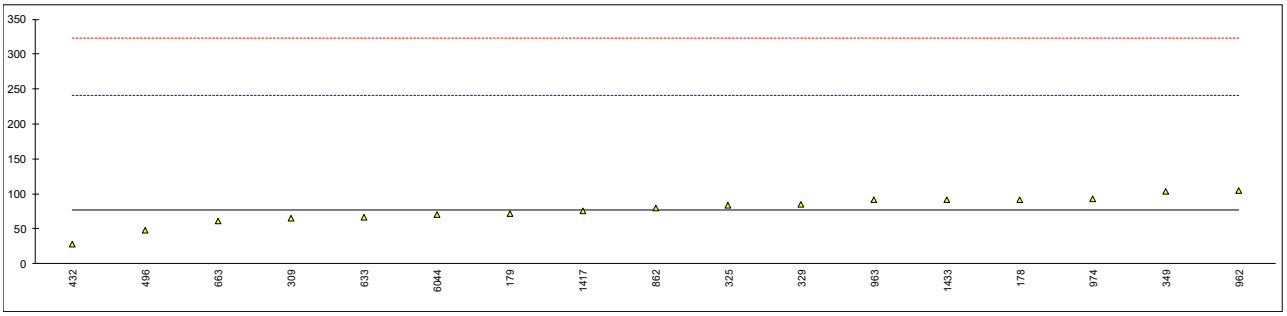


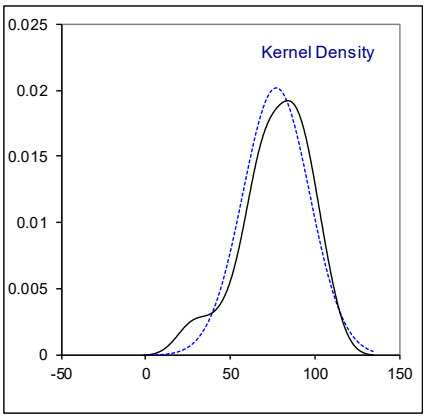


Determination of Water on sample #19055; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D6304-C	92		0.18	
179	D6304-C	72		-0.06	
237		-----		-----	
257		-----		-----	
309	D6304-C	65.5		-0.14	
325	D6304-C	84		0.08	
329	D6304-A	85		0.10	
349	D6304-A	103		0.32	
432	D6304-C	28.3		-0.60	
496	D6304-C	48	C	-0.36	first reported 48 % M/M
633	D6304-C	66.7		-0.13	
663	D6304-C	61.45		-0.19	
862	D6304-C	79.8		0.03	
962	D6304-A	105		0.34	
963	D6304-A	91		0.17	
974	D6304-A	93		0.19	
1011		-----		-----	
1026	D6304-C	<10		-----	
1146	D6304-C	<100		-----	
1417	D6304-A	76		-0.01	
1433	ISO12937	91.787736		0.18	
1748		-----		-----	
6016		-----		-----	
6035		-----		-----	
6044	D6304-A	70		-0.09	
6253		-----		-----	

normality	OK
n	17
outliers	0
mean (n)	77.21
st.dev. (n)	19.754
R(calc.)	55.31
st.dev.(D6304:16e1)	81.868
R(D6304:16e1)	229.23

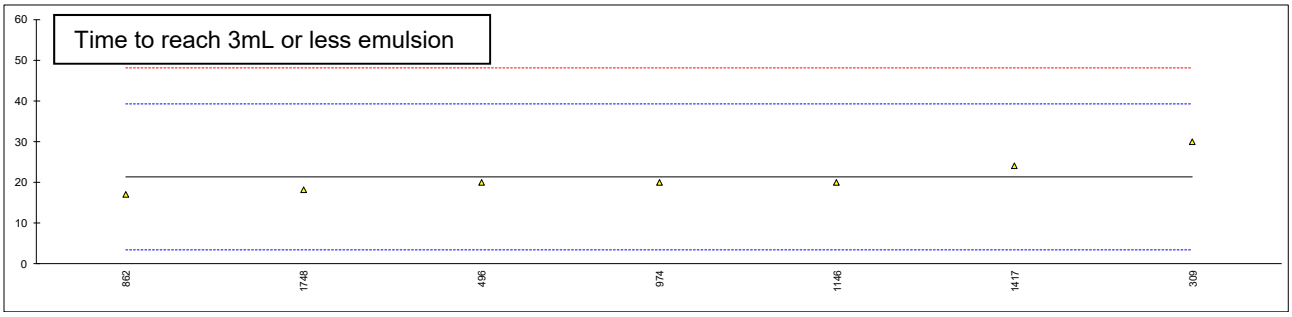


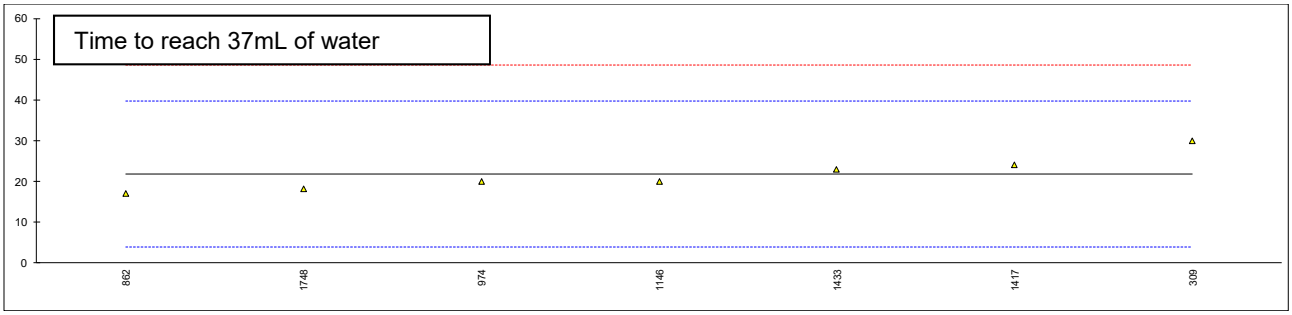


Determination of Water Separability at 82°C, distilled water on sample #19055; results in minutes

lab	method	3mL or less emulsion	z(targ)	37mL of water	z(targ)	complete break (40-40-0)	z(targ)	Test aborted
178		-----	-----	-----	-----	-----	-----	
179		-----	-----	-----	-----	30	0.71	NO
237		-----	-----	-----	-----	-----	-----	
257		-----	-----	-----	-----	-----	-----	
309	D1401	30	0.97	30	0.92	>30	-----	YES / 30
325		-----	-----	-----	-----	-----	-----	
329		-----	-----	-----	-----	-----	-----	YES
349		-----	-----	-----	-----	-----	-----	
432		-----	-----	-----	-----	-----	-----	
496	D1401	20	-0.15	-----	-----	-----	-----	NO
633		-----	-----	-----	-----	-----	-----	
663		-----	-----	-----	-----	-----	-----	
862	D1401	17	-0.48	17	-0.53	20	-0.41	NO
962		-----	-----	-----	-----	-----	-----	
963		-----	-----	-----	-----	-----	-----	
974	D1401	20	-0.15	20	-0.20	-----	-----	NO
1011		-----	-----	-----	-----	20	-0.41	
1026		-----	-----	-----	-----	25	0.15	NO
1146	D1401	20	-0.15	20	-0.20	-----	-----	YES
1417	D1401	24	0.30	24	0.25	24	0.04	NO
1433		>60	-----	23	0.14	>60	-----	YES
1748	D1401	18.2	-0.35	18.2	-0.40	-----	-----	YES
6016		-----	-----	-----	-----	-----	-----	
6035		-----	-----	-----	-----	-----	-----	
6044		-----	-----	-----	-----	23	-0.07	YES
6253		-----	-----	-----	-----	-----	-----	

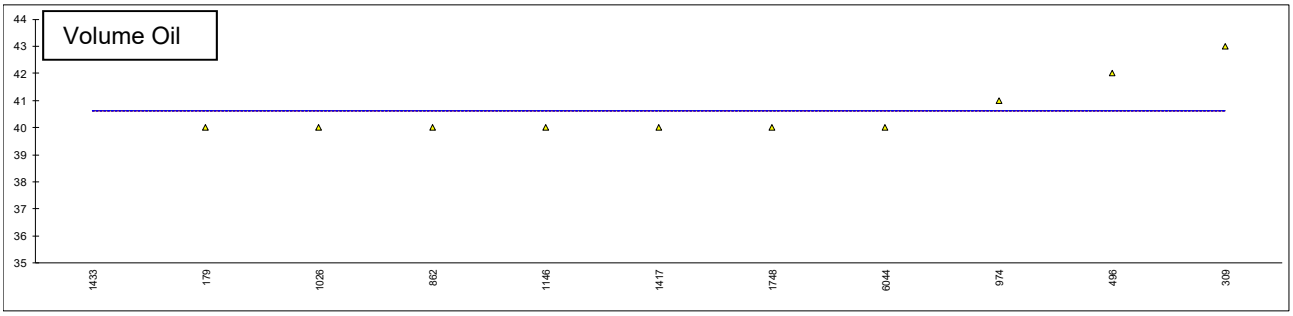
normality	unknown	unknown	unknown
n	7	7	6
outliers	0	0	0
mean (n)	21.3	21.7	23.7
st.dev. (n)	4.40	4.40	3.72
R(calc.)	12.3	12.3	10.4
st.dev.(D1401:18b)	8.93	8.93	8.93
R(D1401:18b)	25	25	25

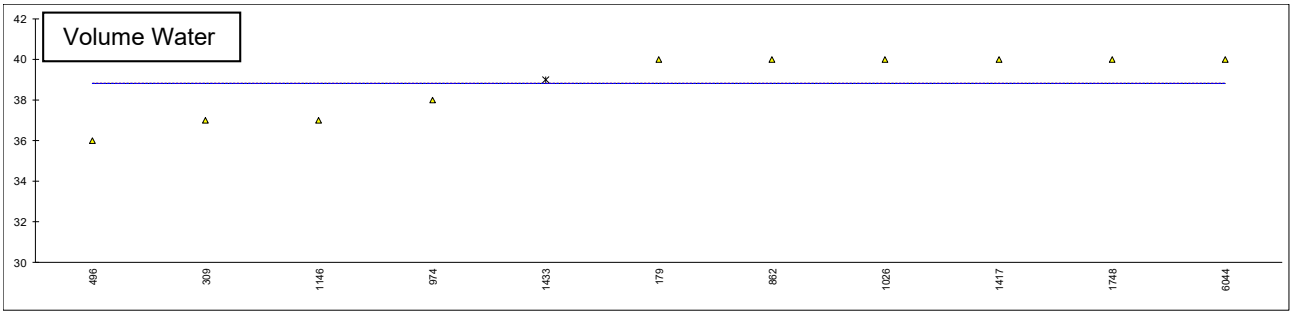


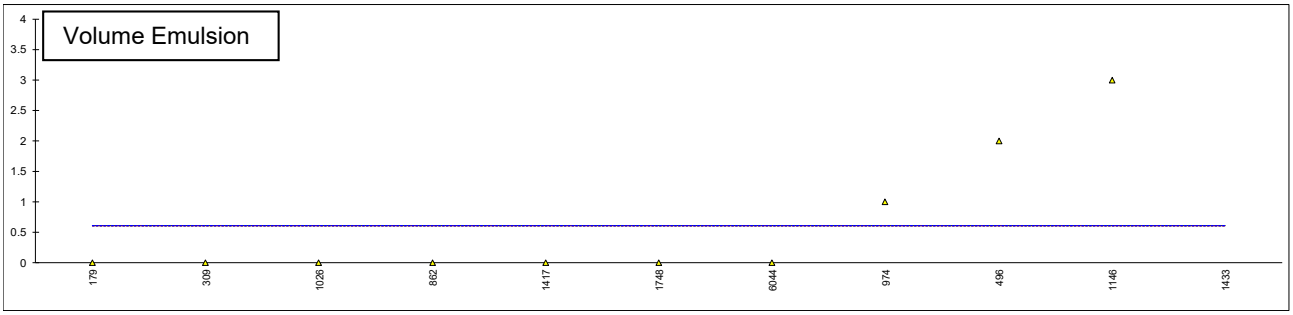


Determination of Water Separability at 82°C, distilled water on sample #19055; results in mL

lab	method	oil phase	mark	water phase	mark	emulsion phase	mark	Remarks
178		-----		-----		-----		
179		40		40		0		
237		-----		-----		-----		
257		-----		-----		-----		
309	D1401	43		37		0		
325		-----		-----		-----		
329		-----		-----		-----		
349		-----		-----		-----		
432		-----		-----		-----		
496	D1401	42		36		2		
633		-----		-----		-----		
663		-----		-----		-----		
862	D1401	40		40		0		
962		-----		-----		-----		
963		-----		-----		-----		
974	D1401	41		38		1		
1011		-----		-----		-----		
1026		40		40		0		
1146	D1401	40		37		3		
1417	D1401	40		40		0		
1433		3	D(0.01)	39	ex	38	D(0.01)	
1748	D1401	40		40		0		
6016		-----		-----		-----		
6035		-----		-----		-----		
6044		40		40		0		
6253		-----		-----		-----		
	normality	not OK		OK		not OK		
	n	10		10		10		
	outliers	1		0+1ex		1		
	mean (n)	40.6		38.8		0.6		
	st.dev. (n)	1.07		1.62		1.07		
	R(calc.)	3.0		4.5		3.0		
	Compare R(iis18L01)	18.4		39.0		56.7		



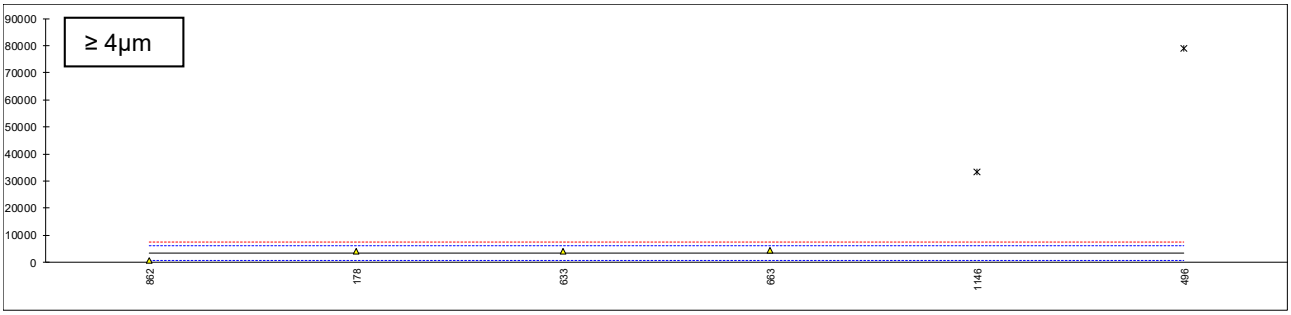


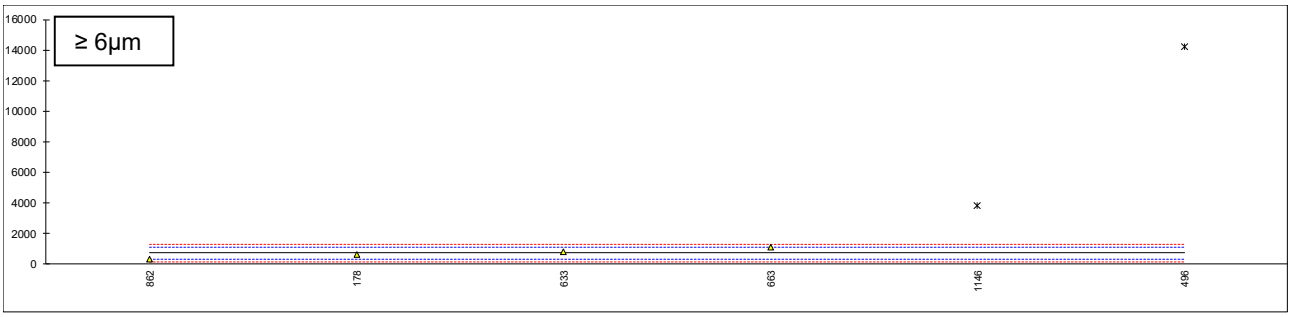


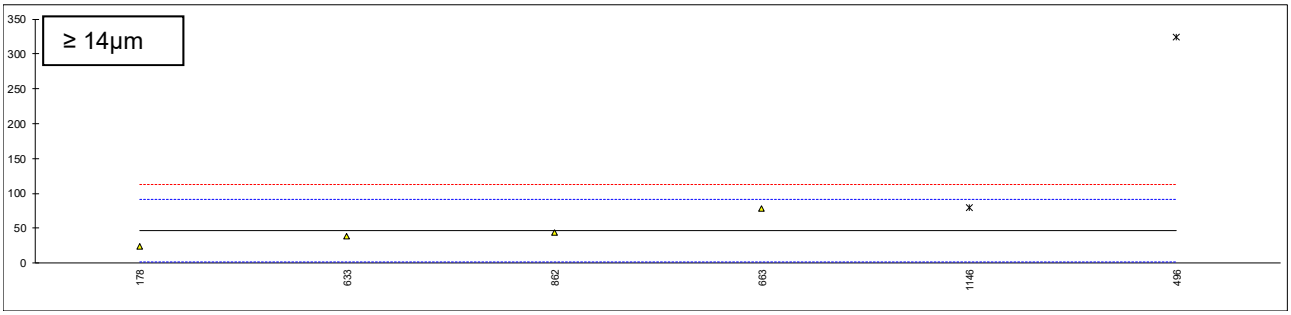
Determination of Level of Contamination on sample #19055; results in counts per mLz.

lab	method	≥ 4 µm (c)	mark	z(targ)	≥ 6 µm (c)	mark	z(targ)	≥ 14 µm (c)	mark	z(targ)
178	INH-1185	4083		0.54	608		-0.53	24		-1.00
179		-----		-----	-----		-----	-----		-----
237		-----		-----	-----		-----	-----		-----
257		-----		-----	-----		-----	-----		-----
309		-----		-----	-----		-----	-----		-----
325		-----		-----	-----		-----	-----		-----
329		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
432		-----		-----	-----		-----	-----		-----
496	D7647	79109	DG(0.01)	55.96	14190	D(0.01)	69.95	324	D(0.01)	12.46
633	D7647	4213		0.63	814		0.54	39		-0.33
663	D7647	4453		0.81	1124		2.15	78		1.42
862	ISO4407	668		-1.98	294		-2.16	44		-0.10
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
974		-----		-----	-----		-----	-----		-----
1011		-----		-----	-----		-----	-----		-----
1026		-----		-----	-----		-----	-----		-----
1146	ISO11500	33248	DG(0.01)	22.08	3835	D(0.05)	16.22	79	ex	1.47
1417		-----		-----	-----		-----	-----		-----
1433		-----		-----	-----		-----	-----		-----
1748		-----		-----	-----		-----	-----		-----
6016		-----		-----	-----		-----	-----		-----
6035		-----		-----	-----		-----	-----		-----
6044		-----		-----	-----		-----	-----		-----
6253		-----		-----	-----		-----	-----		-----

normality	unknown	unknown	unknown
n	4	4	4
outliers	2	2	1+1ex
mean (n)	3354.3	710.0	46.3
st.dev. (n)	1797.38	349.13	22.81
R(calc.)	5032.7	977.6	63.9
st.dev.(D7647:10)	1353.68	192.71	22.30
R(D7647:10)	3790.3	539.6	62.4



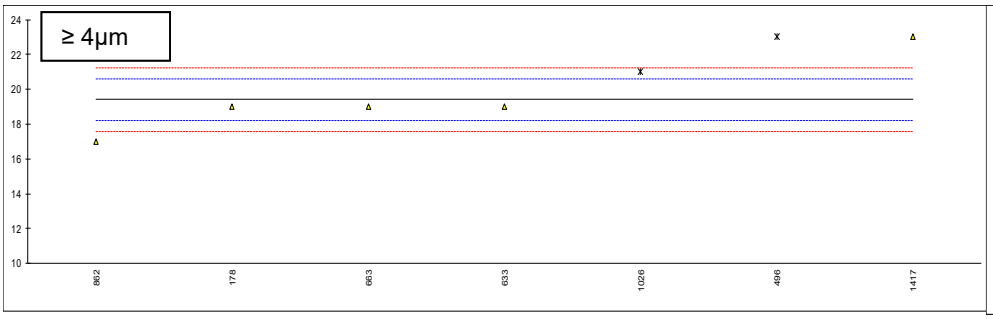


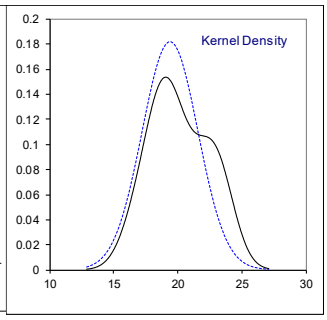


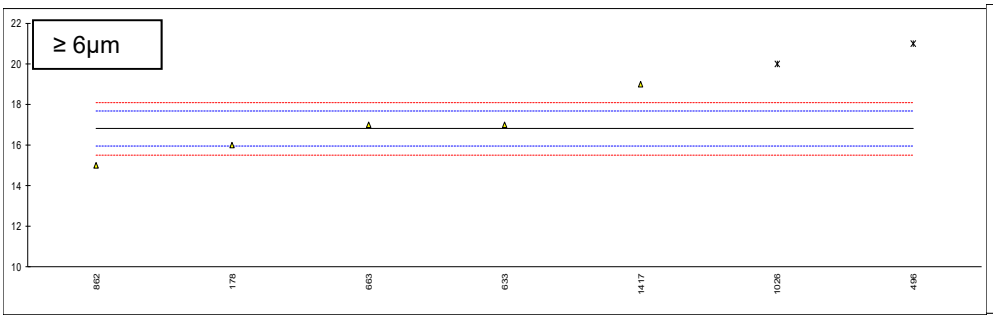
Determination of Level of Contamination acc. to ISO4406 scale on sample #19055; results in scale number

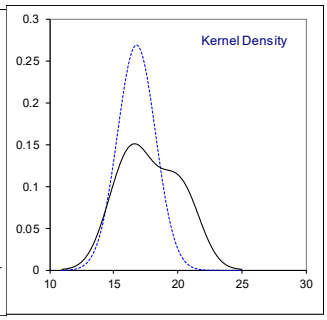
lab	method	≥ 4 µm (c)	mark	z(targ)	≥ 6 µm (c)	mark	z(targ)	≥ 14 µm (c)	mark	z(targ)
178	ISO4406	19		-0.66	16		-1.87	12		-0.84
179		-----		-----	-----		-----	-----		-----
237		-----		-----	-----		-----	-----		-----
257		-----		-----	-----		-----	-----		-----
309		-----		-----	-----		-----	-----		-----
325		-----		-----	-----		-----	-----		-----
329		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
432		-----		-----	-----		-----	-----		-----
496	ISO4406	23	ex	5.93	21	ex	9.80	16	ex	4.76
633	ISO4406	19		-0.66	17		0.47	12		-0.84
663	ISO4406	19		-0.66	17		0.47	13		0.56
862	ISO4406	17		-3.95	15		-4.20	13		0.56
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
974		-----		-----	-----		-----	-----		-----
1011		-----		-----	-----		-----	-----		-----
1026	ISO4406	21	ex	2.64	20	ex	7.47	16	D(0.01)	4.76
1146		-----		-----	-----		-----	-----		-----
1417	ISO4406	23		5.93	19		5.13	13		0.56
1433		-----		-----	-----		-----	-----		-----
1748		-----		-----	-----		-----	-----		-----
6016		-----		-----	-----		-----	-----		-----
6035		-----		-----	-----		-----	-----		-----
6044		-----		-----	-----		-----	-----		-----
6253		-----		-----	-----		-----	-----		-----

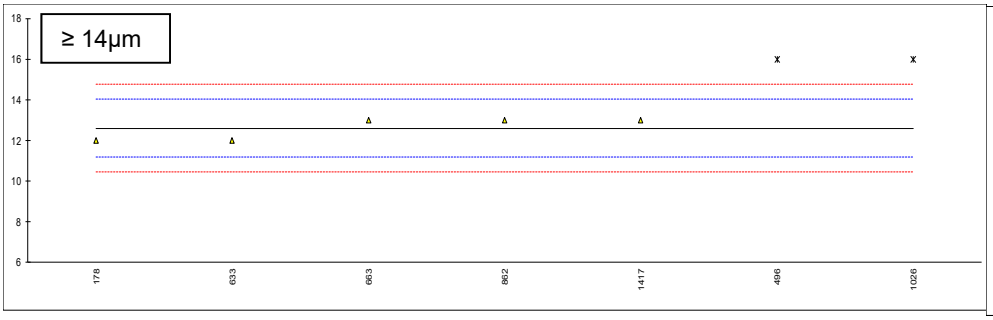
normality	unknown	unknown	unknown
n	5	5	7
outliers	0 +2ex	0+2ex	1+1ex
mean (n)	19.4	16.8	13.6
st.dev. (n)	2.19	1.48	1.72
R(calc.)	6.1	4.2	4.8
st.dev.(D7647:10)	0.61	0.43	0.71
R(D7647:10)	1.7	1.2	2.0

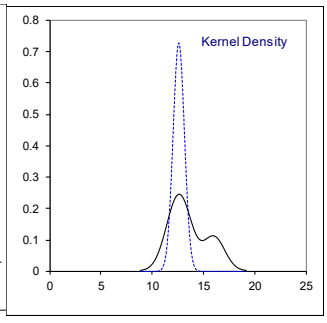










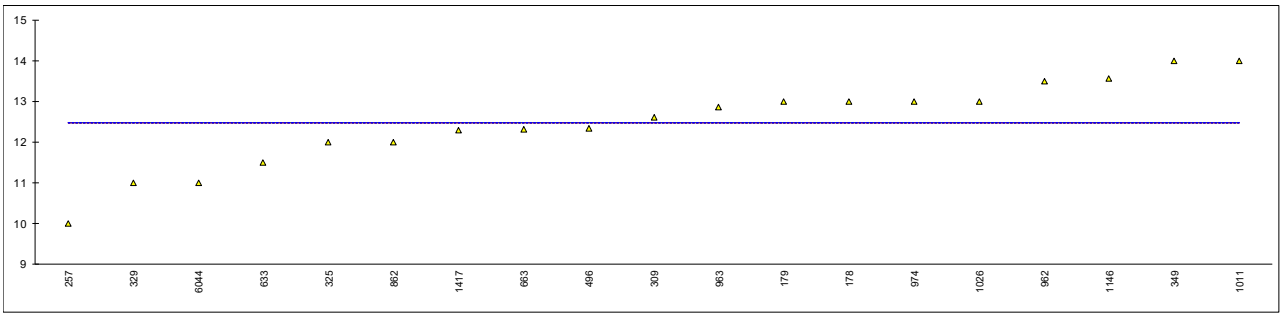


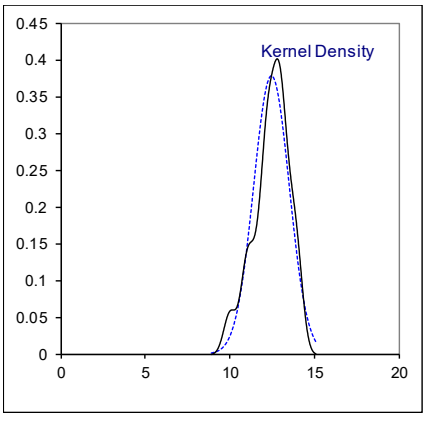
Determination of Calcium (Ca) on sample #19055; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	13		-----	
179	D5185	13		-----	
237		-----		-----	
257	D6595	10.01	C	-----	first reported 10.48
309	D5185	12.6		-----	
325	D5185	12		-----	
329	D4951	11		-----	
349		14		-----	
432		-----		-----	
496	D5185	12.326		-----	
633	D6595	11.5		-----	
663	D5185	12.32		-----	
862	D5185	12		-----	
962		13.5		-----	
963	D5185	12.85		-----	
974	D5185	13		-----	
1011	D5185	14		-----	
1026	D5185	13		-----	
1146	In house	13.55		-----	
1417		12.3		-----	
1433		-----		-----	
1748		-----		-----	
6016		-----		-----	
6035		-----		-----	
6044	D5185	11		-----	
6253		-----		-----	

	normality	OK
	n	19
	outliers	0
	mean (n)	12.471
	st.dev. (n)	1.0547
	R(calc.)	2.953
	st.dev.(D5185:18)	(0.1424)
	R(D5185:18)	(0.399)

Application range: 40- 9000 mg/kg



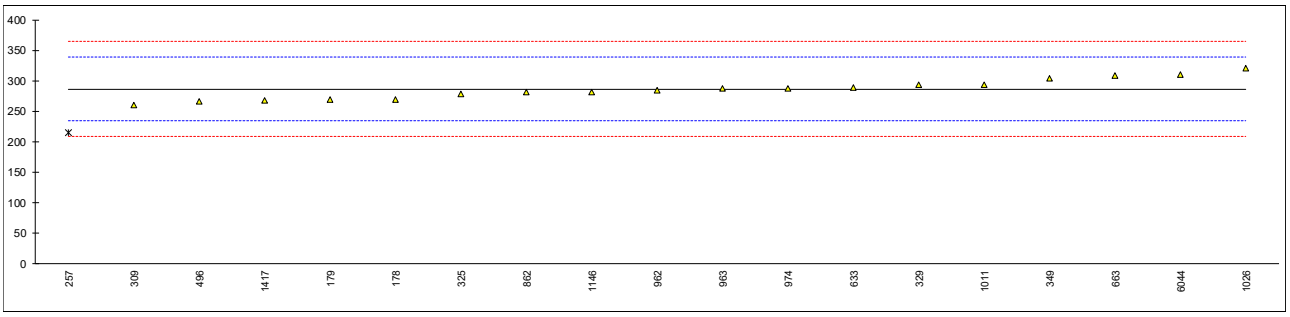


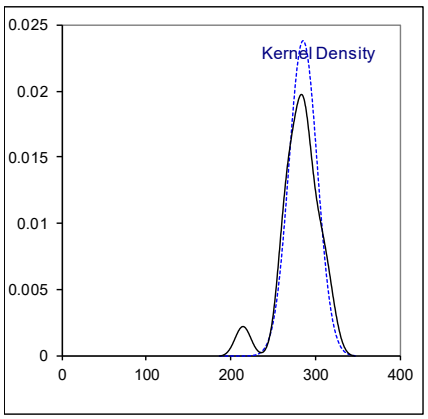
Determination of Phosphorus (P) on sample #19055; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	270		-0.63	
179	D5185	269		-0.67	
237		-----		-----	
257	D6595	215.26	C,G(0.05)	-2.74	first reported 214.0
309	D5185	260.5		-1.00	
325	D5185	279		-0.28	
329	D4951	294		0.29	
349		304		0.68	
432		-----		-----	
496	D5185	265.52		-0.80	
633	D6595	288.6		0.09	
663	D5185	309.15	C	0.88	first reported 5.87
862	D5185	281		-0.21	
962		285		-0.05	
963	D5185	287.63		0.05	
974	D5185	288		0.06	
1011	D5185	294		0.29	
1026	D5185	320	C	1.29	first reported 32
1146	In house	281.5		-0.19	
1417		267.7		-0.72	
1433		-----		-----	
1748		-----		-----	
6016		-----		-----	
6035		-----		-----	
6044	D5185	310		0.91	
6253		-----		-----	

	normality	OK
	n	18
	outliers	1
	mean (n)	286.367
	st.dev. (n)	16.7608
	R(calc.)	46.930
	st.dev.(D5185:18)	25.9879
	R(D5185:18)	72.766

Application range: 10-1000 mg/kg



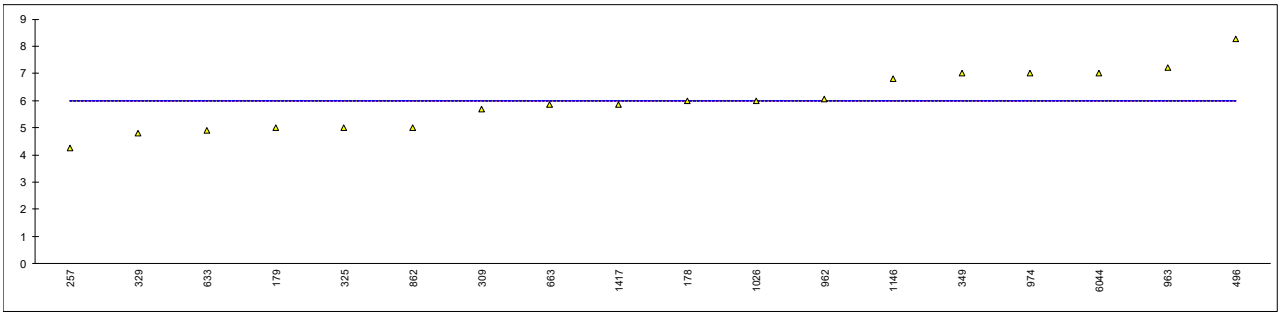


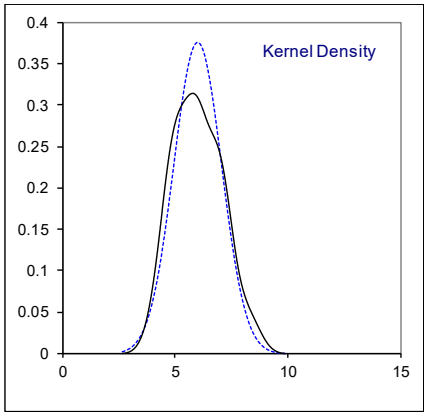
Determination of Zinc (Zn) on sample #19055; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	6		-----	
179	D5185	5		-----	
237		-----		-----	
257	D6595	4.25	C	-----	first reported 4.22
309	D5185	5.7		-----	
325	D5185	5		-----	
329	D4951	4.8		-----	
349		7		-----	
432		-----		-----	
496	D5185	8.256		-----	
633	D6595	4.9		-----	
663	D5185	5.87	C	-----	first reported 309.15
862	D5185	5		-----	
962		6.07		-----	
963	D5185	7.23		-----	
974	D5185	7		-----	
1011	D5185	<10		-----	
1026	D5185	6		-----	
1146	In house	6.807		-----	
1417		5.87		-----	
1433		-----		-----	
1748		-----		-----	
6016		-----		-----	
6035		-----		-----	
6044	D5185	7		-----	
6253		-----		-----	

	normality	OK
	n	18
	outliers	0
	mean (n)	5.986
	st.dev. (n)	1.0597
	R(calc.)	2.967
	st.dev.(D5185:18)	(0.2122)
	R(D5185:18)	(0.594)

Application range: 60-1600 mg/kg





APPENDIX 2

Number of participants per country

1 lab in	AUSTRIA
2 labs in	BELGIUM
1 lab in	CHINA, People's Republic
1 lab in	GERMANY
1 lab in	GREECE
1 lab in	JORDAN
1 lab in	KAZAKHSTAN
1 lab in	MOROCCO
3 labs in	NETHERLANDS
1 lab in	NIGERIA
1 lab in	PHILIPPINES
2 labs in	POLAND
1 lab in	PORTUGAL
2 labs in	SAUDI ARABIA
1 lab in	SPAIN
1 lab in	TANZANIA
1 lab in	THAILAND
1 lab in	UNITED ARAB EMIRATES
1 lab in	UNITED KINGDOM
2 labs in	UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations:

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

Literature:

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