Results of Proficiency Test
Gear Oil (used)
April 2019

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2017, the Institute for Laboratory Studies (iis) organizes a proficiency test for used Gear Oil. During the annual proficiency testing program 2018/2019, it was decided to continue the proficiency test for the analysis of used Gear Oil.
In this interlaboratory study, 27 laboratories in 21 different countries registered for participation. See appendix 4 for the number of participants per country.
In this report, the results of the 2019 proficiency test for used 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 analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send one sample used Gear Oil of 0.5L for the main round, labelled #19056 and one sample of 50mL for metals determination only, labelled #19057.
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/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 organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of 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 for the main round, approximately 20 liters, of used Gear Oil was obtained from a local supplier. After homogenization, 40 amber glass bottles of 0.5 liter were filled and labelled #19056. The homogeneity of the subsamples #19056 was checked by determination of Density at 15°C in accordance with ASTM D4052 and Water according to ASTM D6304-A on 8 stratified randomly selected samples.

	Density at 15°C in kg/m^3	Water in mg/kg
Sample #19056-1	891.48	1589
Sample #19056-2	891.52	1890
Sample #19056-3	891.52	1500
Sample #19056-4	891.53	1736
Sample #19056-5	891.53	1756
Sample #19056-6	891.52	1739
Sample #19056-7	891.53	1613
Sample #19056-8	891.53	1586

Table 1: homogeneity test results of subsamples #19056

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

	Density at 15°C in kg/m^3	Water in mg/kg
r (observed)	0.00	352
reference test method	ASTM D4052:18a	ASTM D6304:16e1
0.3 * R (reference test method)	0.15	436

Table 2: evaluation of the repeatabilities of subsamples #19056

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibilities of the reference test methods. Therefore, homogeneity of the subsamples #19056 was assumed.

For the preparation of the metals sample #19057, approximately 3 liters was taken from a previous PT batch of used Gear Oil (iis17L02) and spiked with various metals Conostan standards: Aluminum, Copper, Iron, Silicon and Tin.

After homogenization, 52 amber glass bottles of 50mL were filled and labelled #19057. The homogeneity of the subsamples #19057 was checked by determination of Iron as Fe in accordance with ASTM D5185 on 8 stratified randomly selected samples.

	Iron as Fe in mg/kg
Sample #19057-1	27
Sample #19057-2	27
Sample #19057-3	27
Sample #19057-4	27
Sample #19057-5	27
Sample #19057-6	27
Sample #19057-7	27
Sample #19057-8	27

Table 3: homogeneity test results of subsamples #19057

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.

	Iron as Fe in mg/kg
r (observed)	0
Reference test method	ASTM D5185:18
0.3 * R (reference test method)	2

Table 4: evaluation of the repeatability of subsamples #19057

The calculated repeatability was in agreement with 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneity of the subsamples #19057 was assumed.

To each of the participating laboratories, one 0.5L bottle, labelled #19056 and one 50mL bottle, labelled #19057 was sent on April 03, 2019. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of used Gear Oil 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 #19056: Acid Number (Total), Density at 15°C, Flash Point PMcc, Kinematic Viscosity at 40°C and at 100°C, Viscosity Index, Membrane Filtration 5µm, Water and Level of Contamination (counts/mL and Scale Number) and to determine on sample #19057: 24 elements (wear metals and additives).
Also, some extra information was asked about the determination of Acid Number and level of Contamination.

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 (when applicable) 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 and 2 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 organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of 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,

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. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used. In general, when no literature reproducibility is available, another target may be used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated according to:

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

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

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

	|z| < 1	good
1 <	|z| < 2	satisfactory
2 <	|z| < 3	questionable
3 <	|z|	unsatisfactory

4 EVALUATION

In this interlaboratory study no problems were encountered with the dispatch of the samples. The reporting participants were on time with submitting the test results. Three participants did not report any test results. Not all laboratories were able to report all analyses requested. In total 24 participants reported 421 numerical test results. Observed were 22 outlying test results, which is 5.2% of the statistically evaluated 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 SAMPLE AND PER TEST

In this section, the reported test results are discussed per sample and 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 5.

In the iis PT reports, ASTM test methods are referred to with a number (e.g. D2270) and an added designation for the year that the test method was adopted or revised (e.g. D2270:10). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D2270:10(2016)). In the test results tables of appendix 1 only the method number and year of adoption or revision e.g. D2270:10 will be used.

Sample #19056

Acid Number (Total): This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D664-A:18e2 for IP and BEP 60mL and 125mL. When the test results for IP and BEP were evaluated separately, the calculated reproducibility of the test results for BEP are in agreement with the precision data of ASTM D 664-A:18e2. The calculated reproducibility of the test results for IP are not in agreement with the precision data of ASTM D 664-A:18e2.
Remarkably, only three participants used pH 10 for BEP instead of pH 11 as mentioned in method ASTM D664:18e2.

Density at 15°C: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D4052:18a.

Flash Point PMcc: Both methods (A and B) of ASTM D93 may be applicable for this determination (in-use vs used lubricating oil). The majority of the participants used method A, only one participant used method B.
This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D93-A:18.

Kinematic Viscosity at 40°C: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D445:18.

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

Viscosity Index: This determination was not problematic. No statistical outliers were observed but one test result was excluded. The calculated reproducibility after rejection of the suspect data is in full agreement with the requirements of ASTM D2270:10(2016). Also, iis calculated the Viscosity Index from the test results reported for the Kinematic Viscosity at 40°C and 100°C. No calculation errors were observed.

Membrane Filtration 5µm: 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 D4055:04(2013).

Water: This determination was problematic for a number of laboratories. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D6304:16e1.

Level of Contamination: This determination was problematic. In total seven statistical outliers were observed over six parameters (4 in counts per ml and 3 in scale number) and twenty-three! test results were excluded. The calculated reproducibilities after rejection of the suspect data is not in agreement with the requirements of ASTM D7647:10(2018). No clear conclusion could be drawn from the reported analytical details (see appendix 3).

Sample #19057

Aluminum: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5185:18.

Boron: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5185:18.

Copper: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ASTM D5185:18.

Iron: 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.

Silicon: 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 D5185:18.

Tin: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5185:18.

Calcium: This determination may be problematic at a level of 3.84 mg/kg. One statistical outlier was observed. The calculated reproducibility is not in agreement with the estimated reproducibility using the Horwitz equation. And is also not in agreement with the requirements of ASTM D5185:18 (application range 40 - 9000 mg/kg), but is in full agreement with the requirements of ASTM D6595:17 (application range 3.7 - 11460 mg/kg).

Phosphorus: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D5185:18.

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

Other elements: The majority of the participants agreed on a concentration near or below the limit of detection for Antimony, Barium, Cadmium, Chromium, Lead, Lithium, Magnesium, Manganese, Molybdenum, Nickel, Potassium, Silver, Sodium, Titanium and Vanadium.

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 or as declared by the estimated target reproducibility using the Horwitz equation 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	20	2.46	1.52	1.33
Density at 15°C	kg/m^3	20	891.6	0.8	0.5
Flash Point PMcc	°C	15	200.7	15.7	14.3
Kinematic Viscosity at 40°C	mm^2/s	22	102.7	1.10	1.73
Kinematic Viscosity at 100°C	mm^2/s	23	12.123	0.165	0.130
Viscosity Index		21	108.63	2.18	2
Membrane Filtration 5µm	%M/M	4	0.001	0.004	0.008
Water	mg/kg	16	431	403	643
Level of contamination					
- ≥ 4µm (c)	counts/mL	6	23996	30577	27116
- ≥ 6µm (c)	counts/mL	6	343	423	261
- ≥14µm (c)	counts/mL	6	23	42	31
- ≥ 4µm (c)	scale number	8	21.6	1.5	1.7
- ≥ 6µm (c)	scale number	8	15.8	2.0	1.2
- ≥14µm (c)	scale number	8	11.3	4.2	2.0

Table 5: reproducibilities of test results of sample #19056.

Parameter	unit	n	average	2.8 * sd	R(lit)
Aluminum as Al	mg/kg	23	9.1	4.8	6.7
Boron as B	mg/kg	19	7.0	7.7	13.3
Copper as Cu	mg/kg	21	11.0	2.5	2.7
Iron as Fe	mg/kg	21	25.0	4.9	6.7
Silicon as Si	mg/kg	21	11.3	1.9	7.5
Tin as Sn	mg/kg	21	10.4	6.4	9.0
Calcium as Ca	mg/kg	21	3.8	1.9	1.4
Phosphorus as P	mg/kg	23	316	90	76
Zinc as Zn	mg/kg	22	3.6	4.2	(1.3)

Table 6: reproducibilities of test results on sample #19057.

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
Number of reporting labs	24	22	17
Number of test results	421	391	362
Statistical outliers	22	18	23
Percentage outliers	5.2%	4.6%	6.4%

Table 7: comparison with previous proficiency test

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

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

	April 2019	April 2018	April 2017
Acid Number (Total)	-	-	-
Density at 15°C	-	-	-
Flash Point PMcc	-	-	+
Kinematic Viscosity at 40°C	+	+	++
Kinematic Viscosity at 100°C	-	--	+
Viscosity Index	+/-	--	+
Membrane Filtration 5µm	++	n.e.	n.e.
Water	+	++	++
Level of contamination			
counts/ml (≥4 / ≥6 / ≥14µm)	-	-	--
scale number (≥4 / ≥6 / ≥14µm)	-	-	--

	April 2019	April 2018	April 2017
Aluminum as Al	+	+	n.e.
Antimony as Sb	n.e.	n.e.	n.e.
Barium as Ba	n.e.	n.e.	n.e.
Boron as B	++	+	+
Cadmium as Cd	n.e.	n.e.	n.e.
Chromium as Cr	n.e.	++	n.e.
Copper as Cu	+	+	(-)
Iron as Fe	+	+	+
Lead as Pb	n.e.	n.e.	n.e.
Lithium as Li	n.e.	n.e.	n.e.
Magnesium as Mg	n.e.	n.e.	n.e.
Manganese as Mn	n.e.	n.e.	n.e.
Molybdenum as Mo	n.e.	n.e.	n.e.
Nickel as Ni	n.e.	n.e.	n.e.
Potassium as K	n.e.	n.e.	n.e.

	April 2019	April 2018	April 2017
Silicon as Si	++	++	n.e.
Silver as Ag	n.e.	n.e.	n.e.
Sodium as Na	n.e.	n.e.	n.e.
Tin as Sn	+	+	n.e.
Titanium as Ti	n.e.	n.e.	n.e.
Vanadium as V	n.e.	n.e.	n.e.
Calcium as Ca	-	-	(-)
Phosphorus as P	-	++	+
Zinc as Zn	(--)	+/-	(-)

Table 8: comparison against the requirements of the reference test methods

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

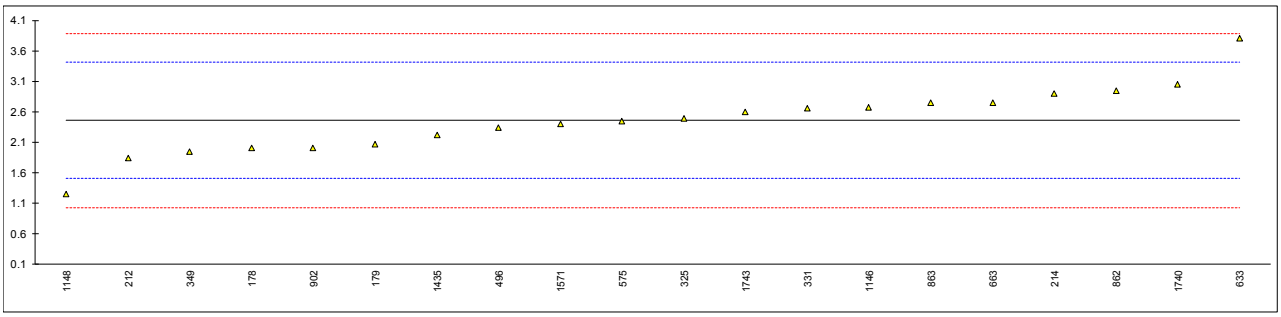
APPENDIX 1

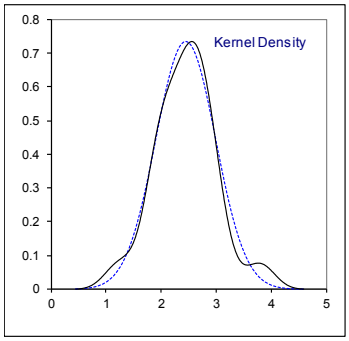
Determination of Acid Number (Total) on sample #19056; results in mg KOH/g

lab	method	value	mark	z(targ)	Determination of end point	Volume of titration solvent
178	INH-1118	2.00		-0.96	---	---
179	D664-A	2.06		-0.83	---	---
212	D664-A	1.84		-1.29	Inflection Point	125 mL
214	D664-A	2.893		0.92	Inflection Point	125 mL
237		-----		-----	---	---
257		-----		-----	---	---
325	D664-A	2.49		0.07	Buffer End Point (pH 10)	125 mL
331	D664Mod.	2.66		0.43	---	---
349	D664-A	1.94	C	-1.08	Buffer End Point (pH 10)	125 mL
451		-----		-----	---	---
496	D664-A	2.34		-0.24	Buffer End Point (pH 11)	60 mL
575	D664-A	2.44		-0.03	Inflection Point	60 mL
633	D664-A	3.80		2.83	Inflection Point	125 mL
663	D664-A	2.7405		0.60	Buffer End Point (pH 11)	60 mL
862	D664-A	2.95		1.04	Inflection Point	60 mL
863	D664-A	2.74		0.60	---	---
902	D664-A	2.01		-0.94	Inflection Point	60 mL
962		-----		-----	---	---
963		-----		-----	Inflection Point	60 mL
974		-----		-----	---	---
1146	D664-A	2.674		0.46	Buffer End Point (pH 11)	125 mL
1148	D8045	1.25431		-2.53	---	---
1435	D664-A	2.225		-0.48	Buffer End Point (pH 10)	100 mL
1571	D664-A	2.40		-0.12	Buffer End Point (pH 11)	60 mL
1740	D664-A	3.05		1.25	Inflection Point	60 mL
1743	D664-A	2.6		0.30	Buffer End Point (pH 11)	60 mL
6016		-----		-----	---	---

			BEP only	Inflection point only
normality	suspect		OK	OK
n	20		8	8
outliers	0		0	0
mean (n)	2.4553		2.4262	2.7054
st.dev. (n)	0.54243		0.2617	0.6230
R(calc.)	1.5188		0.7328	1.7444
st.dev.(D664-A:18e2)	0.47553		---	---
R(D664-A:18e2)	1.3315	BEP (pH-10)- 60mL	1.3161	---
Compare:				
R(D664-A:18e2)	0.8400	IP-60mL		0.9096
R(D664-A:18e2)	0.8125	BEP (pH-10)- 125mL		
R(D664-A:18e2)	0.5584	IP-125mL		

Lab 349 first reported 4.12

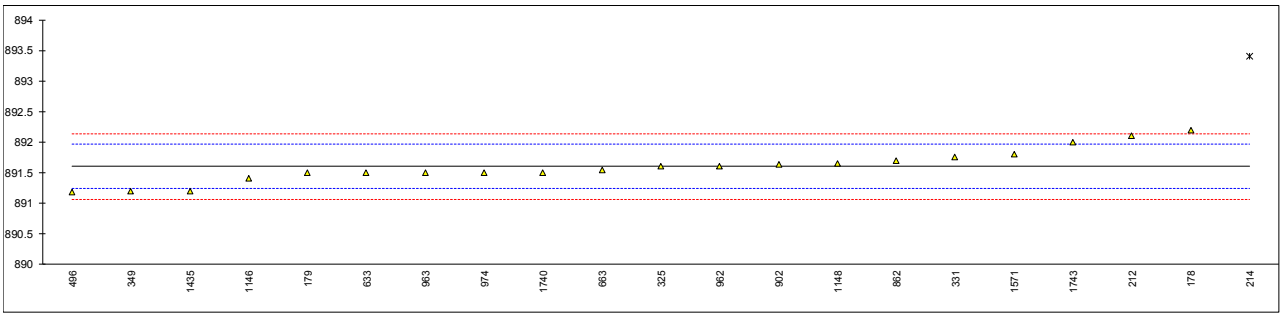


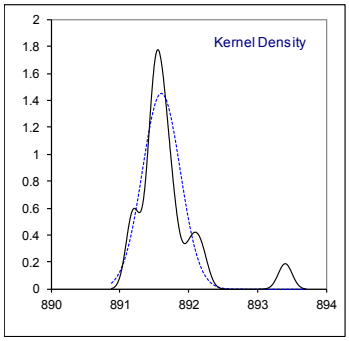


Determination of Density at 15°C on sample #19056; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
178	D4052	892.2		3.34	
179	D4052	891.5		-0.58	
212	ISO12185	892.10		2.78	
214	D1298	893.4	C,R(0.01)	10.06	first reported 0.887 kg/L
237		-----		-----	
257		-----		-----	
325	D4052	891.6		-0.02	
331	ISO12185	891.75		0.82	
349	D4052	891.2		-2.26	
451		-----		-----	
496	D4052	891.18		-2.37	
575		-----		-----	
633	D4052	891.50		-0.58	
663	D4052	891.54		-0.35	
862	D4052	891.7		0.54	
863		-----		-----	
902	D4052	891.64		0.21	
962	D4052	891.6		-0.02	
963	D4052	891.5		-0.58	
974	D4052	891.5		-0.58	
1146	D4052	891.41		-1.08	
1148	ISO12185	891.645		0.23	
1435	D4052	891.2		-2.26	
1571	D7042	891.8		1.10	
1740	D4052	891.5		-0.58	
1743	ISO12185	892.0		2.22	
6016		-----		-----	

normality	OK
n	20
outliers	1
mean (n)	891.603
st.dev. (n)	0.2745
R(calc.)	0.769
st.dev.(D4052:18a)	0.1786
R(D4052:18a)	0.5

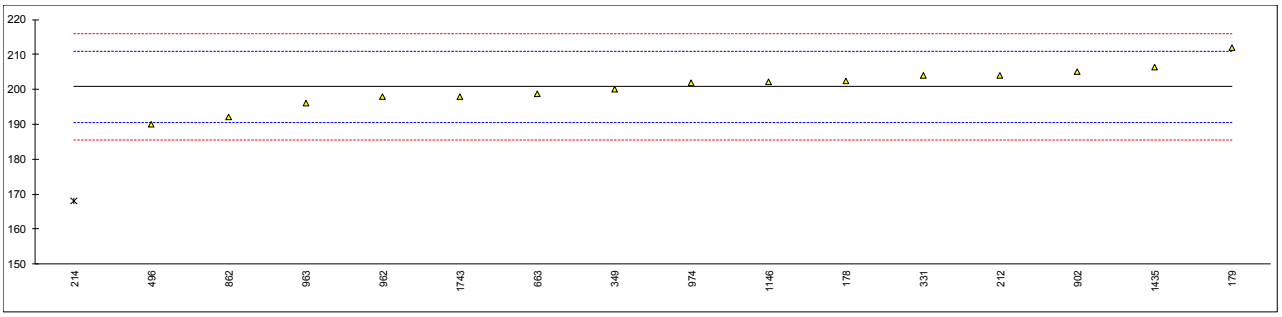


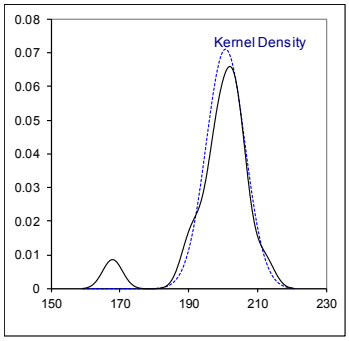


Determination of Flash Point PMcc on sample #19056; results in °C

lab	method	value	mark	z(targ)	remarks
178	D93-A	202.5		0.35	
179	D93-A	212.0		2.22	
212	ISO2719-A	204.0		0.65	
214	D93-A	168	G(0.01)	-6.43	
237		-----		-----	
257		-----		-----	
325		-----		-----	
331	D93-A	203.9		0.63	
349	D93-A	200		-0.14	
451		-----		-----	
496	D93-B	190.0		-2.10	
575		-----		-----	
633		-----		-----	
663	D93-A	198.58		-0.42	
862	D93-A	192		-1.71	
863		-----		-----	
902	D93-A	205		0.84	
962	D93-A	198.0		-0.53	
963	D93-A	196		-0.93	
974	D93-A	202		0.25	
1146	D93-A	202.2		0.29	
1148	D6450	<200		-----	
1435	D93-A	206.5		1.14	
1571		-----		-----	
1740		-----		-----	
1743	D93-A	198		-0.53	
6016		-----		-----	

normality	OK
n	15
outliers	1
mean (n)	200.71
st.dev. (n)	5.606
R(calc.)	15.70
st.dev.(D93-A:18)	5.089
R(D93-A:18)	14.25

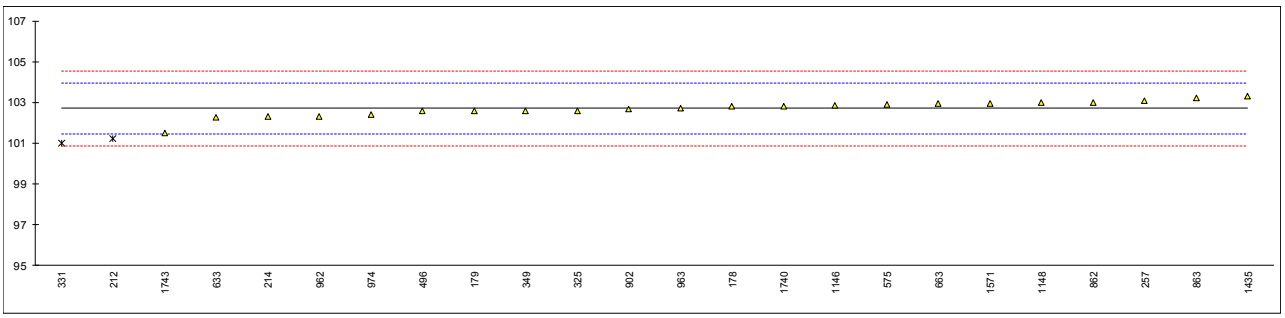


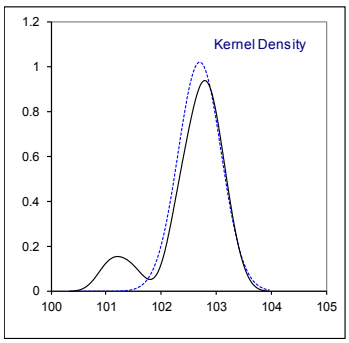


Determination of Kinematic Viscosity at 40°C on sample #19056; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178	D445	102.8		0.17	
179	D445	102.6		-0.16	
212	ISO3104	101.22	R(0.05)	-2.39	
214	D445	102.30		-0.64	
237		-----		-----	
257	D7279	103.1	C	0.65	first reported 103.0
325	D445	102.6		-0.16	
331	D7279Mod.	101.0	R(0.05)	-2.75	
349	D445	102.6		-0.16	
451		-----		-----	
496	D445	102.59	C	-0.17	first reported 12.089
575	D445	102.9		0.33	
633	D7279 corrected to D445	102.27		-0.69	
663	D445	102.92		0.36	
862	D445	103.0		0.49	
863	D445	103.2		0.81	
902	D445	102.69		-0.01	
962	D445	102.3		-0.64	
963	D445	102.7		0.00	
974	D445	102.4		-0.48	
1146	D445	102.85		0.25	
1148	DIN51659-2	102.98		0.46	
1435	D7042	103.3		0.98	
1571	D7042	102.95		0.41	
1740	D445	102.8		0.17	
1743	D7279 corrected to D445	101.5	C	-1.94	first reported 100.7
6016		-----		-----	

normality	not OK
n	22
outliers	2
mean (n)	102.6977
st.dev. (n)	0.39090
R(calc.)	1.0945
st.dev.(D445:18)	0.61736
R(D445:18)	1.7286

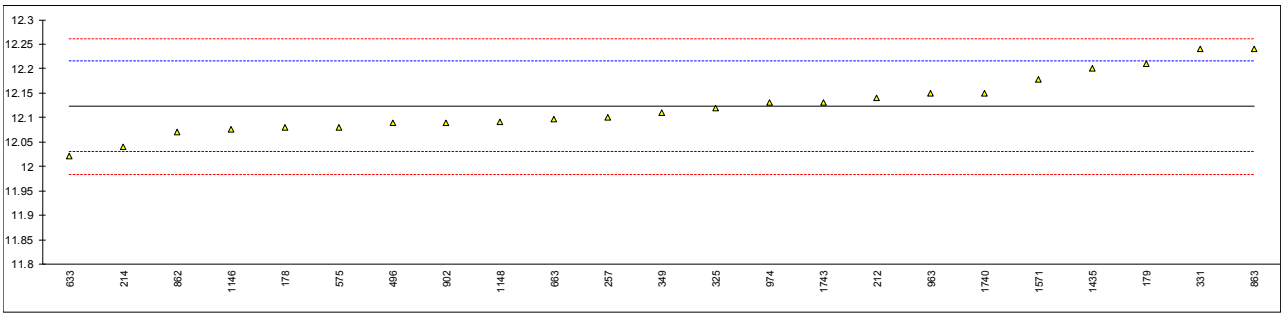


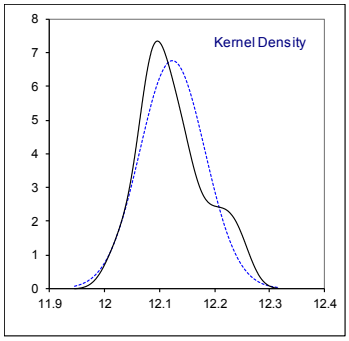


Determination of Kinematic Viscosity at 100°C on sample #19056; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178	D445	12.08		-0.93	
179	D445	12.21		1.88	
212	ISO3104	12.141		0.39	
214	D445	12.04		-1.80	
237		-----		-----	
257	D7279	12.10	C	-0.50	first reported 12.06
325	D445	12.12		-0.07	
331	D7279Mod.	12.24		2.53	
349	D445	12.11		-0.28	
451		-----		-----	
496	D445	12.09	C	-0.74	first reported 102.59
575	D445	12.08		-0.93	
633	D7279 corrected to D445	12.021		-2.21	
663	D445	12.0965		-0.58	
862	D445	12.07		-1.15	
863	D445	12.24		2.53	
902	D445	12.09		-0.72	
962		-----		-----	
963	D445	12.15		0.58	
974	D445	12.13		0.15	
1146	D445	12.076		-1.02	
1148	DIN51659-2	12.091		-0.70	
1435	D7042	12.20		1.66	
1571	D7042	12.178		1.19	
1740	D445	12.15		0.58	
1743	D7279 corrected to D445	12.13	C	0.15	first reported 11.90
6016		-----		-----	

normality	OK
n	23
outliers	0
mean (n)	12.1232
st.dev. (n)	0.05896
R(calc.)	0.1651
st.dev.(D445:18)	0.04623
R(D445:18)	0.1295

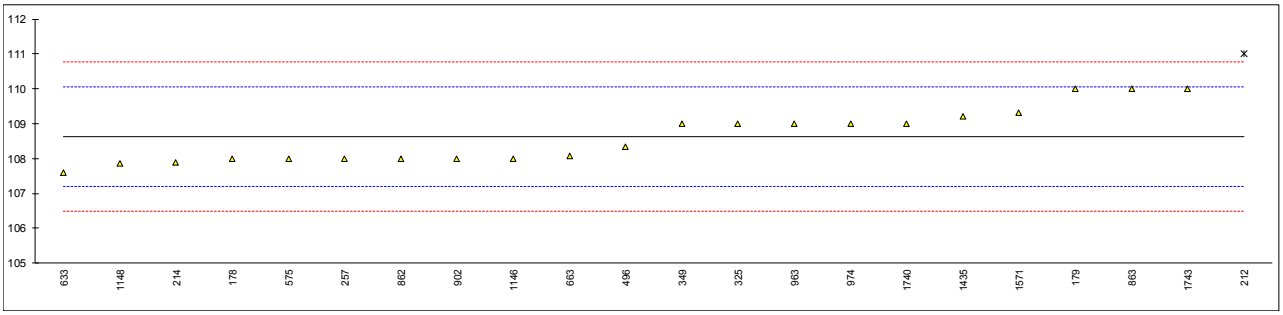


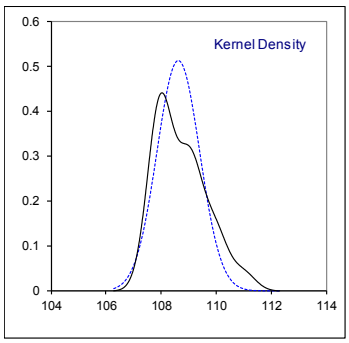


Determination of Viscosity Index (V.I.) on sample #19056

lab	method	value	mark	z(targ)	remarks
178	D2270	108		-0.89	
179	D2270	110		1.91	
212	D2270	111	ex	3.31	Outlier in Kinematic Viscosity at 40°C
214	D2270	107.90		-1.03	
237		-----		-----	
257	D2270	108	C	-0.89	first reported 107
325	D2270	109		0.51	
331		-----		-----	
349	D2270	109		0.51	
451		-----		-----	
496	D2270	108.35		-0.40	
575	D2270	108		-0.89	
633	D2270	107.6		-1.45	
663	D2270	108.06		-0.80	
862	D2270	108		-0.89	
863	D2270	110		1.91	
902	D2270	108		-0.89	
962		-----		-----	
963	D2270	109		0.51	
974	D2270	109		0.51	
1146	D2270	108		-0.89	
1148	ISO2909	107.86		-1.08	
1435	D2270	109.197		0.79	
1571	D2270	109.315		0.96	
1740	D2270	109		0.51	
1743	ISO2909	110		1.91	
6016		-----		-----	

normality	OK
n	21
outliers	0+1ex
mean (n)	108.632
st.dev. (n)	0.7771
R(calc.)	2.176
st.dev.(D2270:10)	0.7143
R(D2270:10)	2

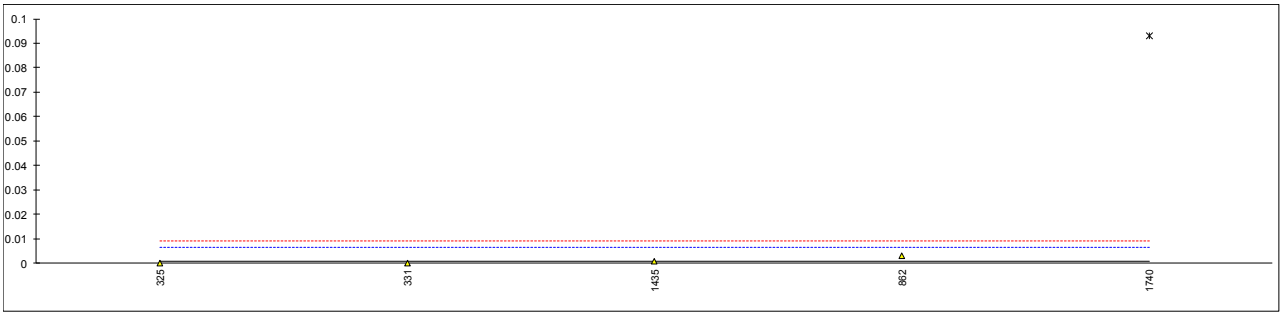




Determination of Membrane Filtration 5µm on sample #19056; results in %M/M

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
212		-----		-----	
214		-----		-----	
237		-----		-----	
257		-----		-----	
325	D4055	0.0000		-0.37	
331	NF E48652Mod.	0.000		-0.37	
349		-----		-----	
451		-----		-----	
496		-----		-----	
575		-----		-----	
633		-----		-----	
663		-----		-----	
862	D4055	0.003		0.74	
863		-----		-----	
902		-----		-----	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1146		-----		-----	
1148		-----		-----	
1435	D4055	0.0010	C	0.00	first reported 10% M/M
1571		-----		-----	
1740	D4055	0.093	D(0.01)	33.94	Possibly a false positive test result?
1743		-----		-----	
6016		-----		-----	

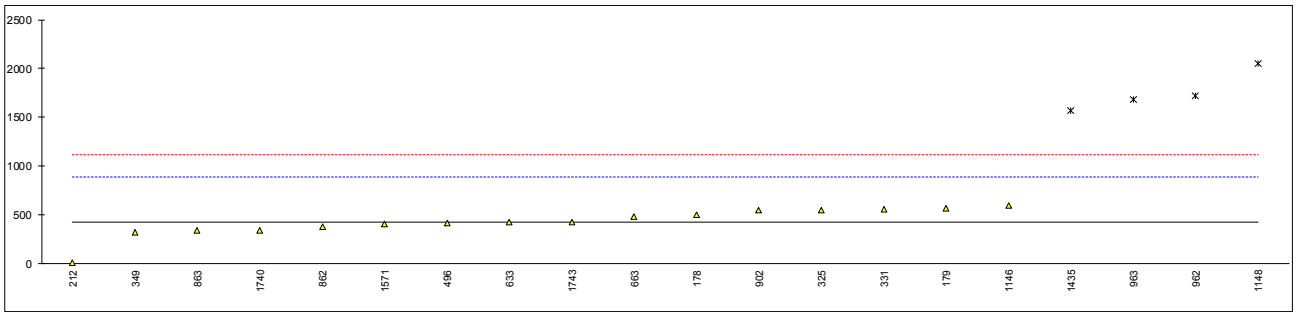
normality	unknown
n	4
outliers	1
mean (n)	0.001
st.dev. (n)	0.0014
R(calc.)	0.004
st.dev.(D4055:04)	0.0027
R(D4055:04)	0.008

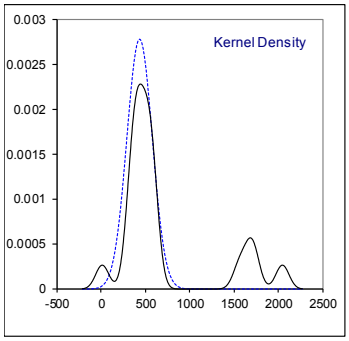


Determination of Water on sample #19056; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D6304-C	506		0.33	
179	D6304-C	567		0.59	
212	D6304-A	6.92		-1.85	
214		-----		-----	
237		-----		-----	
257		-----		-----	
325	D6304-C	553		0.53	
331	In house	556.3		0.55	
349	D6304-C	321		-0.48	
451		-----		-----	
496	D6304-C	414		-0.07	
575		-----		-----	
633	D6304-C	428.8		-0.01	
663	D6304-C	479.25		0.21	
862	D6304-C	381		-0.22	
863	D6304-C	343		-0.38	
902	D6304-C	549.4		0.52	
962	D6304-A	1725	R(0.01)	5.64	
963	D6304-A	1685	R(0.01)	5.46	
974		-----		-----	
1146	D6304-C	600		0.74	
1148	DIN51777-1	2050	C,R(0.01)	7.05	first reported 0.2243% M/M
1435	D6304-A	1565	R(0.01)	4.94	
1571	D6304-C	410		-0.09	
1740	D6304-C	343		-0.38	
1743	ISO12937	430		0.00	
6016		-----		-----	

normality	not OK
n	16
outliers	4
mean (n)	430.542
st.dev. (n)	143.8233
R(calc.)	402.705
st.dev.(D6304:16e1)	229.5748
R(D6304:16e1)	642.809

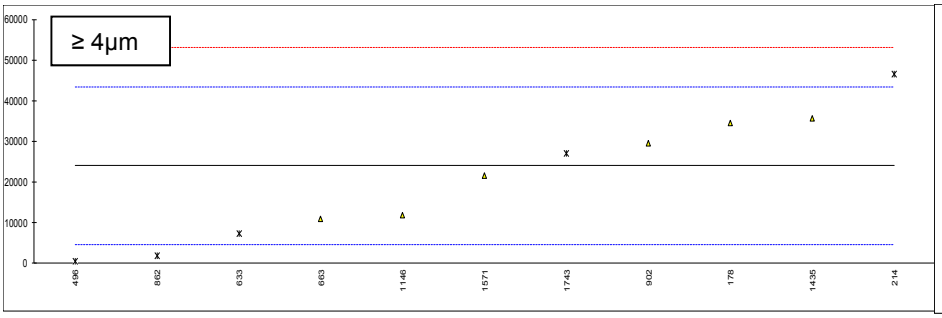


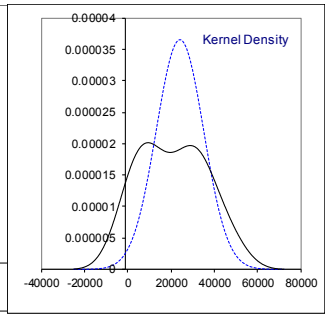


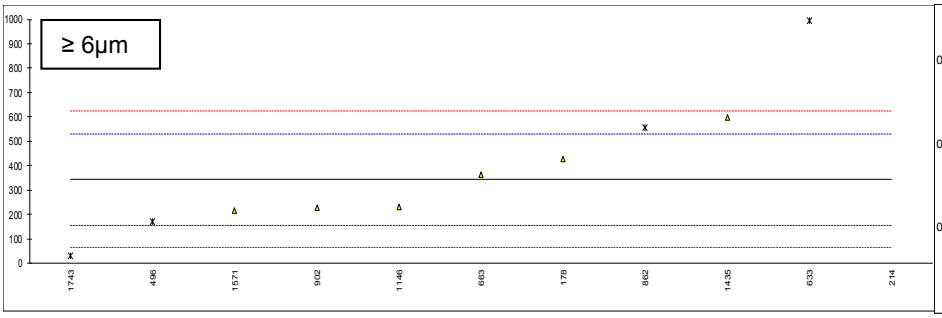
Determination of Level of Contamination on sample #19056; results in counts per mL

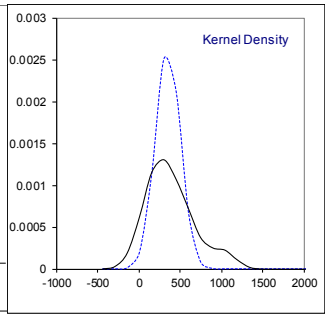
lab	method	≥ 4 µm	mark	z(targ)	≥ 6 µm	mark	z(targ)	≥ 14 µm	mark	z(targ)
178	INH-1185	34502		1.08	427		0.90	31		0.75
179		-----		-----	-----		-----	-----		-----
212		-----		-----	-----		-----	-----		-----
214		46590.4	ex	2.33	36910.4	G(0.01)	392.27	4576.6	G(0.01)	414.46
237		-----		-----	-----		-----	-----		-----
257		-----		-----	-----		-----	-----		-----
325		-----		-----	-----		-----	-----		-----
331		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
451		-----		-----	-----		-----	-----		-----
496	D7647	473	ex	-2.43	171	ex	-1.85	7	ex	-1.44
575		-----		-----	-----		-----	-----		-----
633	D7647	7316	ex	-1.72	994	G(0.01)	6.98	104	G(0.05)	7.39
663	D7647	11023		-1.34	364		0.22	42		1.75
862	ISO4407	1830	ex	-2.29	555	ex	2.27	80	ex	5.21
863		-----		-----	-----		-----	-----		-----
902	D7647	29404		0.56	226		-1.26	12.4		-0.95
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
974		-----		-----	-----		-----	-----		-----
1146	ISO11500	11833		-1.26	232		-1.20	9		-1.25
1148		-----	W	-----	-----	W	-----	-----	W	-----
1435		35603.13		1.20	596.6		2.72	7.3		-1.41
1571	ISO4407	21613.75		-0.25	215.02		-1.38	35.03		1.11
1740		-----		-----	-----		-----	-----		-----
1743	ISO4407	27058	ex	0.32	30	ex	-3.36	7	ex	-1.44
6016		-----		-----	-----		-----	-----		-----
	normality	OK			suspect			OK		
	n	6			6			6		
	outliers	0+5ex			2+3ex			2+3ex		
	mean (n)	23996.48			343.44			22.79		
	st.dev. (n)	10920.260			151.128			14.995		
	R(calc.)	30576.73			423.16			41.99		
	st.dev.(D7647:10)	9684.293			93.219			10.987		
	R(D7647:10)	27116.02			261.01			30.76		

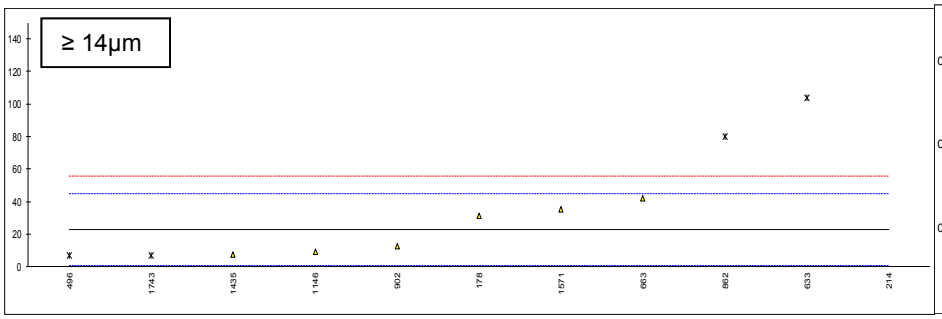
Lab 1148 first reported 1823304, 39151 and 297
Ex = excluded as related test results are statistical outliers

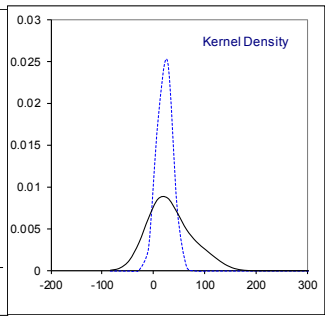

≥ 4µm
60000
50000
40000
30000
20000
10000
0
496
862
633
663
1146
1571
1743
902
178
1435
214


Kernel Density
0.00004
0.000035
0.00003
0.000025
0.00002
0.000015
0.00001
0.000005
0
-40000
-20000
0
20000
40000
60000
80000


≥ 6µm
1000
900
800
700
600
500
400
300
200
100
0
1743
496
1571
902
1146
663
178
862
1435
633
214


Kernel Density
0.003
0.0025
0.002
0.0015
0.001
0.0005
0
-1000
-500
0
500
1000
1500
2000

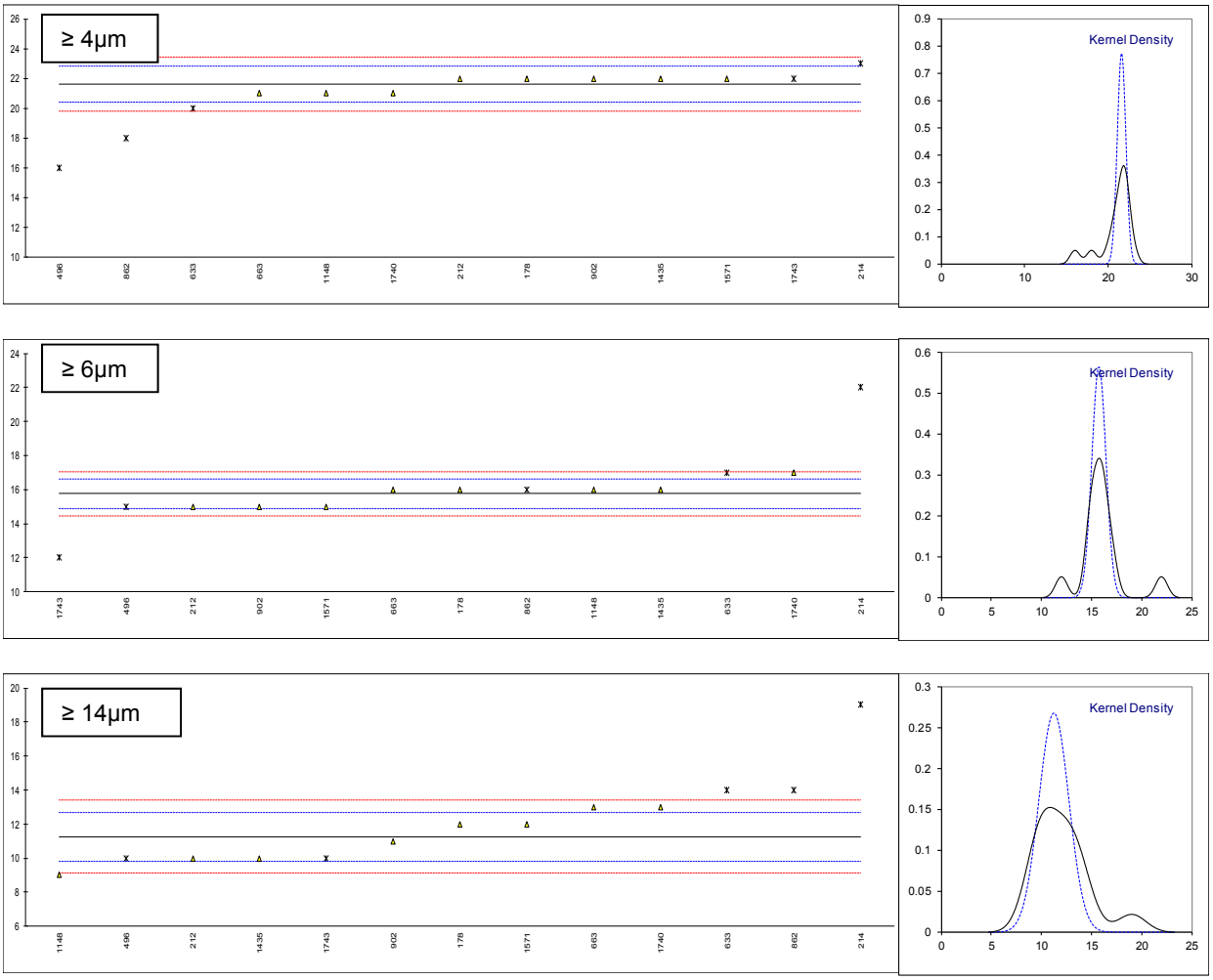

≥ 14µm
140
120
100
80
60
40
20
0
496
1743
1435
1146
902
178
1571
663
862
633
214


Kernel Density
0.03
0.025
0.02
0.015
0.01
0.005
0
-200
-100
0
100
200
300

Determination of Level of Contamination acc. to ISO4406 scale on sample #19056; results in scale number

lab	method	≥ 4 µm	mark	z(targ)	≥ 6 µm	mark	z(targ)	≥ 14 µm	mark	z(targ)
178	ISO4406	22		0.62	16		0.58	12		1.05
179		-----		-----	-----		-----	-----		-----
212	ISO4406	22		0.62	15		-1.75	10		-1.75
214	ISO4406	23	ex	2.26	22	ex	14.58	19	ex	10.85
237		-----		-----	-----		-----	-----		-----
257		-----		-----	-----		-----	-----		-----
325		-----		-----	-----		-----	-----		-----
331		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
451		-----		-----	-----		-----	-----		-----
496	ISO4406	16	G(0.05)	-9.26	15	ex	-1.75	10	ex	-1.75
575		-----		-----	-----		-----	-----		-----
633	ISO4406	20	ex	-2.68	17	ex	2.92	14	ex	3.85
663	ISO4406	21		-1.03	16		0.58	13		2.45
862	ISO4406	18	G(0.05)	-5.97	16	ex	0.58	14	ex	3.85
863		-----		-----	-----		-----	-----		-----
902	D7647	22		0.62	15		-1.75	11		-0.35
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
974		-----		-----	-----		-----	-----		-----
1146		-----		-----	-----		-----	-----		-----
1148	ISO4406	21		-1.03	16		0.58	9		-3.15
1435	ISO4406	22		0.62	16		0.58	10		-1.75
1571	ISO4406	22		0.62	15		-1.75	12		1.05
1740	ISO4406	21		-1.03	17		2.92	13		2.45
1743	ISO4406	22	ex	0.62	12	G(0.01)	-8.75	10	ex	-1.75
6016		-----		-----	-----		-----	-----		-----
	normality	OK			OK			OK		
	n	8			8			8		
	outliers	2+3ex			1+4ex			0+5ex		
	mean (n)	21.63			15.75			11.25		
	st.dev. (n)	0.518			0.707			1.488		
	R(calc.)	1.45			1.98			4.17		
	st.dev.(D7647:10)	0.607			0.429			0.714		
	R(D7647:10)	1.7			1.2			2.0		

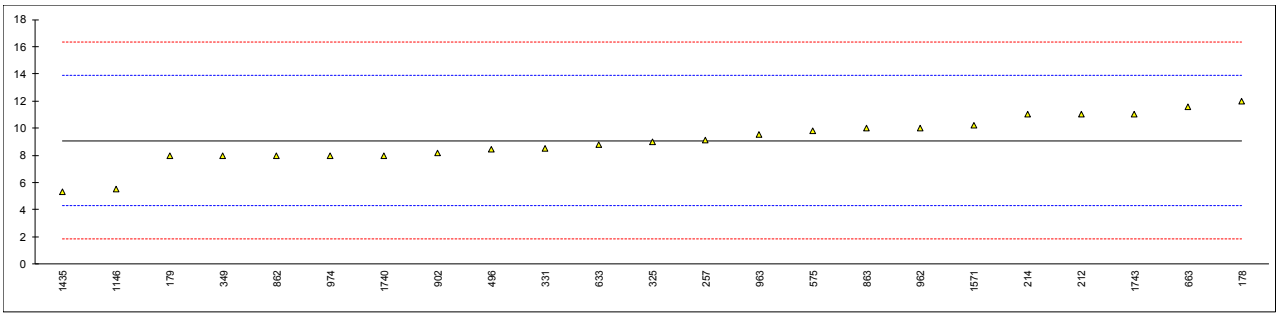
Ex = excluded as related test results are statistical outliers

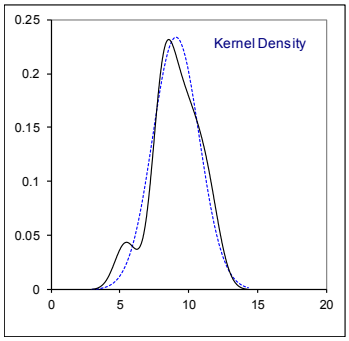

≥ 4µm
Kernel Density
≥ 6µm
Kernel Density
≥ 14µm
Kernel Density

Determination of Aluminum as Al on sample #19057; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	12		1.21	
179	D5185	8		-0.45	
212	D5185	11		0.79	
214	D6595	11	C	0.79	first reported 0
237		-----		-----	
257	D6595	9.13	C	0.02	first reported 9.45
325	D5185	9		-0.04	
331	D5185Mod.	8.5		-0.24	
349	D5185	8		-0.45	
451		-----		-----	
496	D5185	8.448		-0.27	
575	D6595	9.80		0.30	
633	D6595	8.81		-0.12	
663	D5185	11.58		1.03	
862	D5185	8		-0.45	
863	D5185	10		0.38	
902	D5185	8.14		-0.39	
962	D5185	10.0		0.38	
963	D5185	9.54		0.19	
974	D5185	8		-0.45	
1146	In house	5.525		-1.48	
1148		-----		-----	
1435	D5185	5.32	C	-1.56	first reported < 1
1571	D5185	10.2193		0.47	
1740	D6595	8		-0.45	
1743	D5185	11		0.79	
6016		-----		-----	

normality	OK
n	23
outliers	0
mean (n)	9.087
st.dev. (n)	1.7038
R(calc.)	4.771
st.dev.(D5185:18)	2.4089
R(D5185:18)	6.745

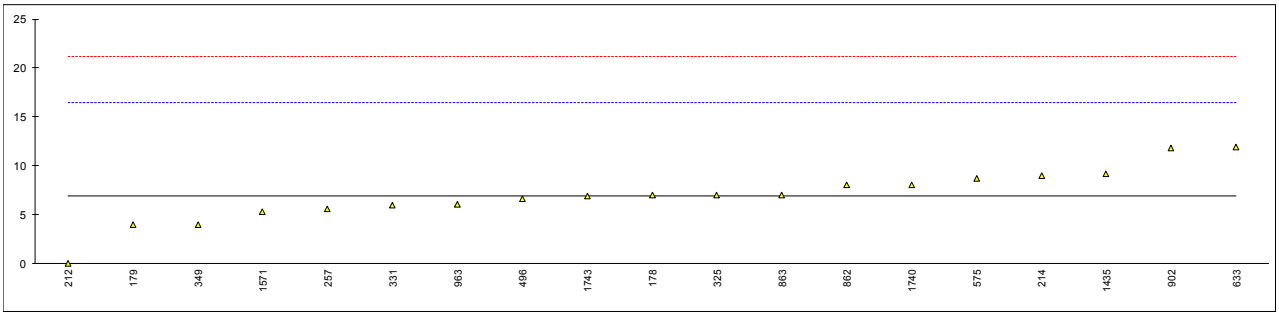


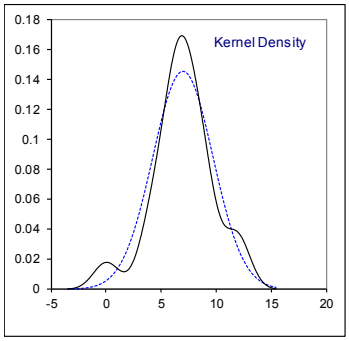


Determination of Boron as B on sample #19057; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	7		0.01	
179	D5185	4		-0.62	
212	D5185	0		-1.47	
214	D6595	9		0.43	
237		-----		-----	
257	D6595	5.57	C	-0.29	first reported 5.54
325	D5185	7		0.01	
331	D5185Mod.	6.0		-0.20	
349	D5185	4		-0.62	
451		-----		-----	
496	D5185	6.647		-0.07	
575	D6595	8.72		0.37	
633	D6595	11.9		1.04	
663		-----		-----	
862	D5185	8		0.22	
863	D5185	7		0.01	
902	D5185	11.85		1.03	
962		-----		-----	
963	D5185	6.07		-0.19	
974		-----		-----	
1146		-----		-----	
1148		-----		-----	
1435	D5185	9.19		0.47	
1571	D5185	5.30200		-0.35	
1740	D6595	8		0.22	
1743	D5185	6.9		-0.01	
6016		-----		-----	

normality	suspect
n	19
outliers	0
mean (n)	6.955
st.dev. (n)	2.7404
R(calc.)	7.673
st.dev.(D5185:18)	4.7338
R(D5185:18)	13.255

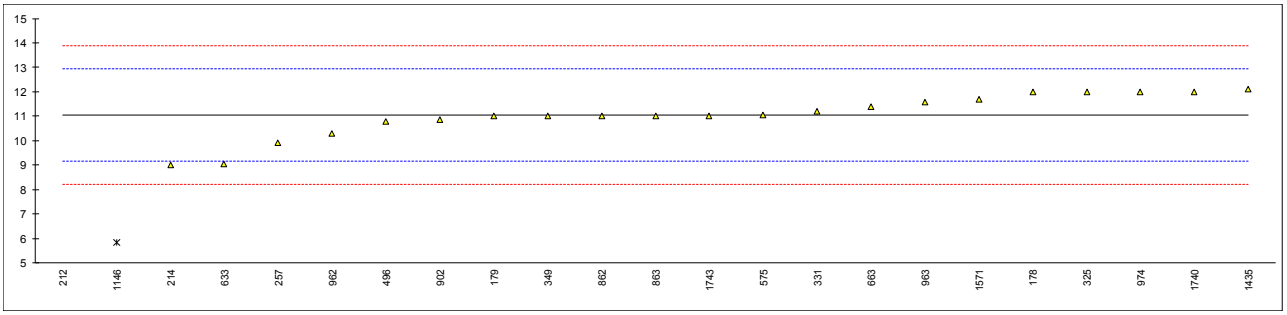


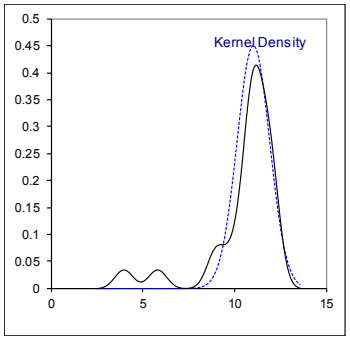


Determination of Copper as Cu on sample #19057; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	12		1.01	
179	D5185	11		-0.05	
212	D5185	4	R(0.01)	-7.44	
214	D6595	9		-2.16	
237		-----		-----	
257	D6595	9.91	C	-1.20	first reported 10.16
325	D5185	12		1.01	
331	D5185Mod.	11.2		0.17	
349	D5185	11		-0.05	
451		-----		-----	
496	D5185	10.796		-0.26	
575	D6595	11.06		0.02	
633	D6595	9.04		-2.12	
663	D5185	11.38		0.36	
862	D5185	11		-0.05	
863	D5185	11		-0.05	
902	D5185	10.86		-0.19	
962	D5185	10.3		-0.79	
963	D5185	11.58		0.57	
974	D5185	12		1.01	
1146	In house	5.837	R(0.01)	-5.50	
1148		-----		-----	
1435	D5185	12.10		1.12	
1571	D5185	11.6881		0.68	
1740	D6595	12		1.01	
1743	D5185	11		-0.05	
6016		-----		-----	

normality	OK
n	21
outliers	2
mean (n)	11.044
st.dev. (n)	0.8874
R(calc.)	2.485
st.dev.(D5185:18)	0.9466
R(D5185:18)	2.650

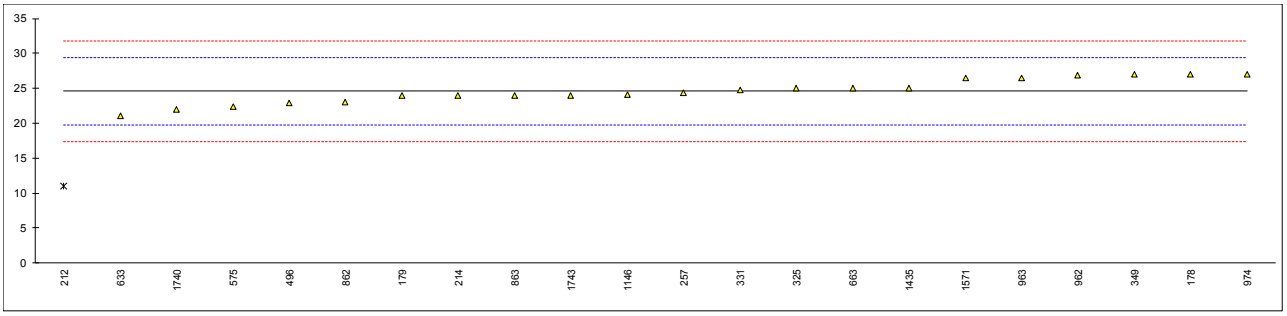


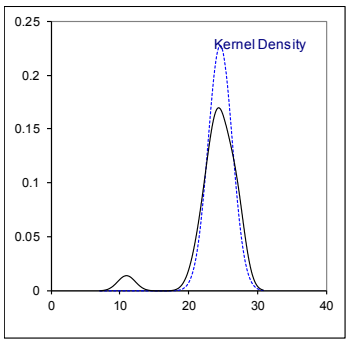


Determination of Iron as Fe on sample #19057; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	27		1.00	
179	D5185	24		-0.25	
212	D5185	11	R(0.01)	-5.65	
214	D6595	24		-0.25	
237		-----		-----	
257	D6595	24.38	C	-0.09	first reported 25.37
325	D5185	25		0.17	
331	D5185Mod.	24.7		0.04	
349	D5185	27		1.00	
451		-----		-----	
496	D5185	22.860		-0.72	
575	D6595	22.4		-0.91	
633	D6595	21.1		-1.45	
663	D5185	25.02		0.18	
862	D5185	23		-0.66	
863	D5185	24		-0.25	
902		-----		-----	
962	D5185	26.9		0.96	
963	D5185	26.53		0.80	
974	D5185	27		1.00	
1146	In house	24.03		-0.23	
1148		-----		-----	
1435	D5185	25.06		0.19	
1571	D5185	26.5119		0.80	
1740	D6595	22		-1.08	
1743	D5185	24		-0.25	
6016		-----		-----	

normality	OK
n	21
outliers	1
mean (n)	24.595
st.dev. (n)	1.7573
R(calc.)	4.920
st.dev.(D5185:18)	2.4072
R(D5185:18)	6.740

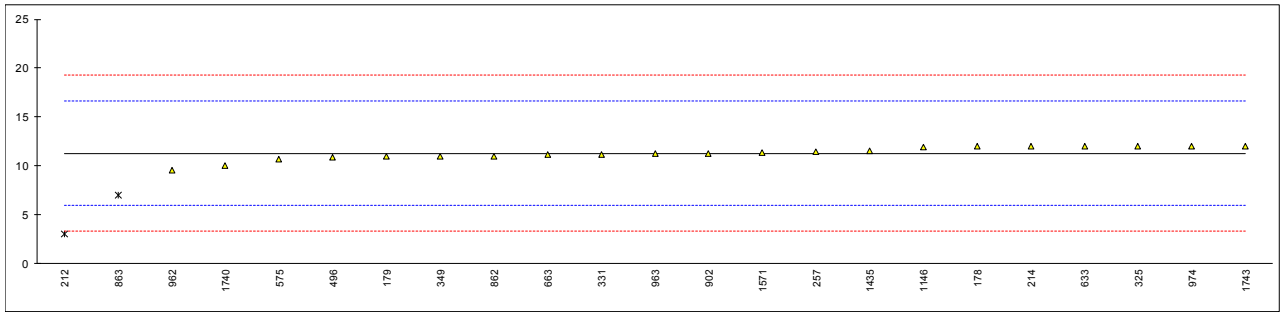


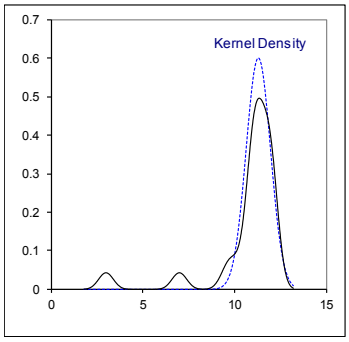


Determination of Silicon as Si on sample #19057; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	12		0.26	
179	D5185	11		-0.11	
212	D5185	3	R(0.01)	-3.11	
214	D6595	12		0.26	
237		-----		-----	
257	D6595	11.49	C	0.07	first reported 11.89
325	D5185	12		0.26	
331	D5185Mod.	11.2		-0.04	
349	D5185	11		-0.11	
451		-----		-----	
496	D5185	10.875		-0.16	
575	D6595	10.7		-0.23	
633	D6595	12.0		0.26	
663	D5185	11.15		-0.06	
862	D5185	11		-0.11	
863	D5185	7	R(0.01)	-1.61	
902	D5185	11.29		0.00	
962	D5185	9.6		-0.64	
963	D5185	11.26		-0.02	
974	D5185	12		0.26	
1146	In house	11.89		0.22	
1148		-----		-----	
1435	D5185	11.51		0.08	
1571	D5185	11.3987		0.04	
1740	D6595	10		-0.49	
1743	D5185	12		0.26	
6016		-----		-----	

normality	suspect
n	21
outliers	2
mean (n)	11.303
st.dev. (n)	0.6634
R(calc.)	1.858
st.dev.(D5185:18)	2.6668
R(D5185:18)	7.467

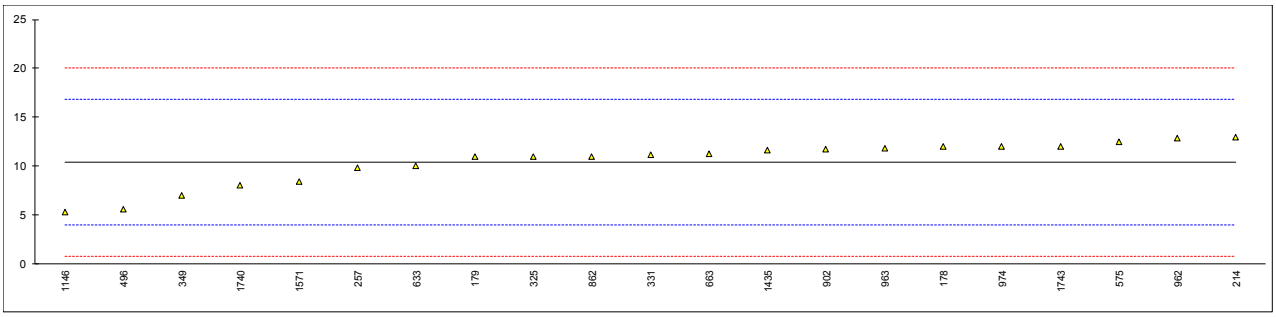


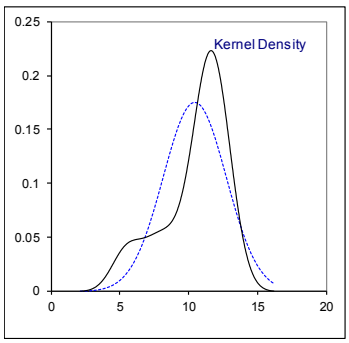


Determination of Tin as Sn on sample #19057; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	12		0.48	
179	D5185	11		0.17	
212		-----		-----	
214	D6595	13		0.80	
237		-----		-----	
257	D6595	9.86	C	-0.18	first reported 10.05
325	D5185	11		0.17	
331	D5185Mod.	11.2		0.24	
349	D5185	7		-1.07	
451		-----		-----	
496	D5185	5.611		-1.50	
575	D6595	12.5		0.64	
633	D6595	10.0		-0.14	
663	D5185	11.29		0.26	
862	D5185	11		0.17	
863	D5185	<1		<-2.94	Possibly a false positive test result?
902	D5185	11.76		0.41	
962	D5185	12.9		0.76	
963	D5185	11.87		0.44	
974	D5185	12		0.48	
1146	In house	5.329		-1.59	
1148		-----		-----	
1435	D5185	11.62		0.37	
1571	D5185	8.3802		-0.64	
1740	D6595	8		-0.76	
1743	D5185	12		0.48	
6016		-----		-----	

normality	OK
n	21
outliers	0
mean (n)	10.444
st.dev. (n)	2.2756
R(calc.)	6.372
st.dev.(D5185:18)	3.2118
R(D5185:18)	8.993

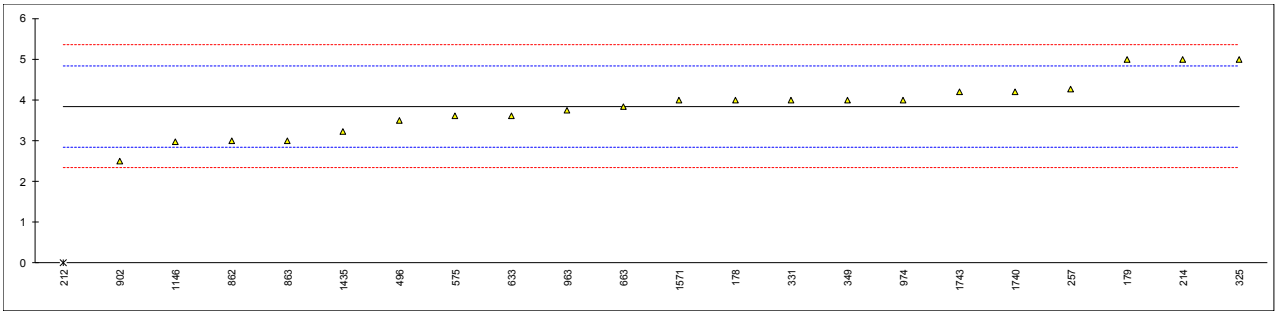


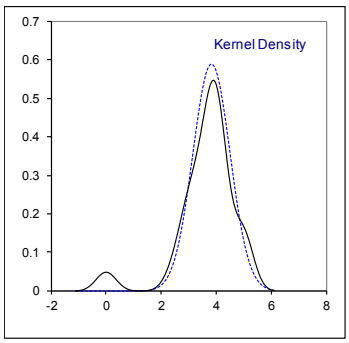


Determination of Calcium as Ca on sample #19057; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	4		0.32	
179	D5185	5		2.31	
212	D5185	0	R(0.01)	-7.65	
214	D6595	5		2.31	
237		-----		-----	
257	D6595	4.27	C	0.86	first reported 4.87
325	D5185	5		2.31	
331	D5185Mod.	4.0		0.32	
349	D5185	4		0.32	
451		-----		-----	
496	D5185	3.502		-0.67	
575	D6595	3.6		-0.48	
633	D6595	3.6		-0.48	
663	D5185	3.83		-0.02	
862	D5185	3		-1.67	
863	D5185	3		-1.67	
902	D5185	2.50		-2.67	
962		-----		-----	
963	D5185	3.75		-0.18	
974	D5185	4		0.32	
1146	In house	2.964		-1.74	
1148		-----		-----	
1435	D5185	3.23		-1.21	
1571	D5185	3.9849		0.29	
1740	D6595	4.2	C	0.72	first reported 1
1743	D5185	4.19		0.70	
6016		-----		-----	

normality	OK	
n	21	
outliers	1	
mean (n)	3.839	
st.dev. (n)	0.6775	
R(calc.)	1.897	
st.dev.(Horwitz)	0.5017	
R(Horwitz)	1.405	
compare		
R(D5185:18)	0.086	application range: 40 – 9000 mg/kg
R(D6595:17)	1.777	application range: 3.7 – 11460 mg/kg

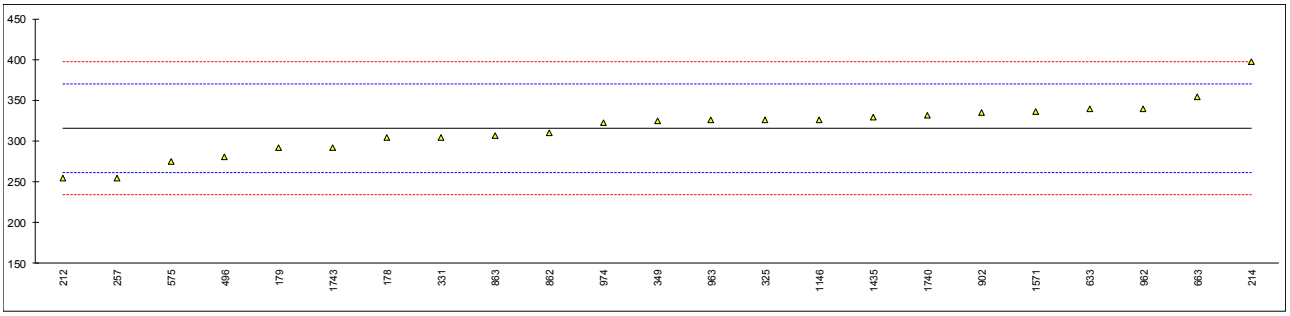


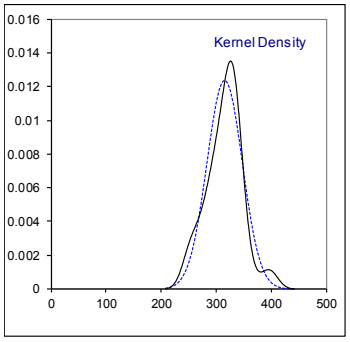


Determination of Phosphorus as P on sample #19057; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	304		-0.43	
179	D5185	292		-0.87	
212	D5185	254		-2.26	
214	D6595	397		2.98	
237		-----		-----	
257	D6595	254.02	C	-2.26	first reported 257.06
325	D5185	326		0.38	
331	D5185Mod.	304.2		-0.42	
349	D5185	325		0.34	
451		-----		-----	
496	D5185	280.46		-1.29	
575	D6595	275.3		-1.48	
633	D6595	339.9		0.88	
663	D5185	354.30		1.41	
862	D5185	310		-0.21	
863	D5185	307		-0.32	
902	D5185	335		0.71	
962	D5185	340		0.89	
963	D5185	325.74		0.37	
974	D5185	323		0.27	
1146	In house	326.1		0.38	
1148		-----		-----	
1435	D5185	329.2		0.49	
1571	D5185	336.038		0.74	
1740	D6595	332		0.60	
1743	D5185	292		-0.87	
6016		-----		-----	

normality	OK
n	23
outliers	0
mean (n)	315.750
st.dev. (n)	32.2988
R(calc.)	90.437
st.dev.(D5185:18)	27.2887
R(D5185:18)	76.408

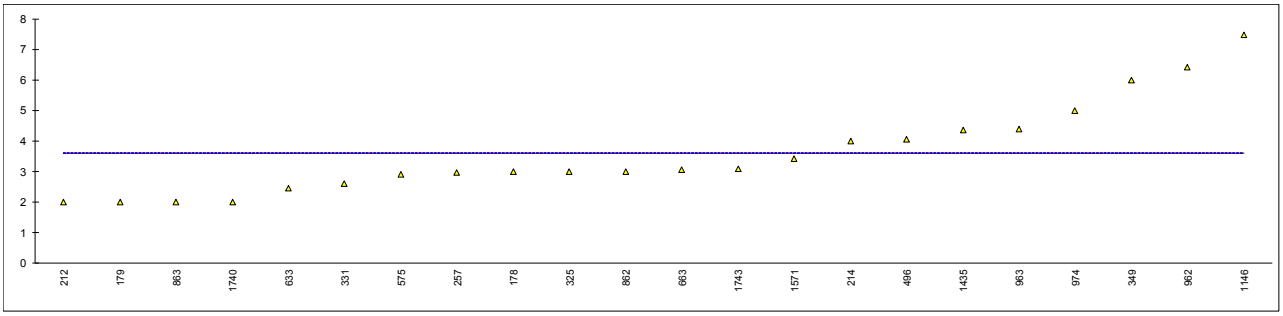


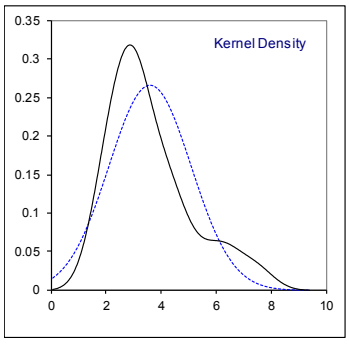


Determination of Zinc as Zn on sample #19057; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	3		-----	
179	D5185	2		-----	
212	D5185	2		-----	
214	D6595	4		-----	
237		-----		-----	
257	D6595	2.97	C	-----	first reported 3.83
325	D5185	3		-----	
331	D5185Mod.	2.6		-----	
349	D5185	6		-----	
451		-----		-----	
496	D5185	4.055		-----	
575	D6595	2.9		-----	
633	D6595	2.45		-----	
663	D5185	3.07		-----	
862	D5185	3		-----	
863	D5185	2		-----	
902		-----		-----	
962	D5185	6.4		-----	
963	D5185	4.39		-----	
974	D5185	5		-----	
1146	In house	7.462		-----	
1148		-----		-----	
1435	D5185	4.35		-----	
1571	D5185	3.4307		-----	
1740	D6595	2		-----	
1743	D5185	3.10		-----	
6016		-----		-----	

normality	suspect	
n	22	
outliers	0	
mean (n)	3.599	
st.dev. (n)	1.4979	
R(calc.)	4.194	
st.dev.(Horwitz)	(0.4749)	
R(Horwitz)	(1.330)	
compare		
R(D5185:18)	(0.340)	application range: 60 – 1600 mg/kg
R(D6595:17)	(2.488)	application range: 5.3 – 1345 mg/kg





APPENDIX 2 Other reported test results

Reported test results of other elements on sample #19057; results in mg/kg.

lab	method	Antimony as Sb	Barium as Ba	Cadmium as Cd	Chromium as Cr	Lead as Pb
178	D5185	0	<1	-----	<1	<1
179	D5185	<1	<1	<1	<1	<1
212	D5185	0	0	0	0	0
214	D6595	0	0	0	0	0
237		-----	-----	-----	-----	-----
257	D6595	-----	0.67 C	-----	0.30 C	0.10 C
325	D5185	0	0	-----	0	1
331	D5185 Mod.	<0.5	<0.5	-----	<0.5	<0.5
349	D5185	0	0	0	0	0
451		-----	-----	-----	-----	-----
496	D5185	-----	0.041	-----	0.264	0.228
575	D6595	-----	0.0	-----	0.27	0.0
633	D6595	-----	0.01	0	0.17	0
663	D5185	0.18	0.09	-----	0.30	0.12
862	D5185	<1	<1	<1	<1	<1
863	D5185	-----	<1	<1	<1	<1
902	D5185	-----	-----	-----	-----	-----
962	D5185	-----	-----	-----	<1	-----
963	D5185	-----	0.05	0.04	0.03	0.05
974	D5185	-----	<1	<1	1	<1
1146	In house	-----	0.020	-----	0.2034	0
1148		-----	-----	-----	-----	-----
1435	D5185	<1	<1	<1	<1	<1
1571	D5185	-----	0.06354	0.06594	0.18276	0.17166
1740	D6595	-----	0.1	-----	0.2	0.5
1743	D5185	0.15	0.05	0.02	0.28	0.20
6016		-----	-----	-----	-----	-----

Lab 257 first reported: 1.31, 0.28 and 0.04 respectively

lab	method	Lithium as Li	Magnesium as Mg	Manganese as Mn	Molybdenum as Mo	Nickel as Ni
178	D5185	-----	1	-----	<1	<1
179	D5185	1	<1	<1	<1	<1
212	D5185	-----	0	0	54	0
214	D6595	0	1	0	1	1
237		-----	-----	-----	-----	-----
257	D6595	-----	1.07 C	0.06 C	0.32 C	0.08 C
325	D5185	-----	0	0	0	0
331	D5185 Mod.	<0.5	<0.5	<0.5	<0.5	<0.5
349	D5185	0	0	0	0	0
451		-----	-----	-----	-----	-----
496	D5185	-----	0.316	0.256	0.046	0.111
575	D6595	-----	0.0	0.0	0.0	0.2
633	D6595	0	0.1	0.37	0.17	0.20
663	D5185	-----	0.36	-----	0.02	0.18
862	D5185	<1	<1	<1	<1	<1
863	D5185	-----	<1	<1	<1	<1
902	D5185	-----	-----	-----	-----	-----
962	D5185	-----	<1	<1	<1	<1
963	D5185	-----	0.41	0.48	0.02	0.09
974	D5185	<1	<1	<1	<1	<1
1146	In house	0.0092	0.3134	0.1546	-----	0.0346
1148		-----	-----	-----	-----	-----
1435	D5185	<1	<1	<1	<1	<1
1571	D5185	-----	0.31251	0.2869	0.002	0.0393
1740	D6595	0.1	0.1	0.1	0.1	0.5
1743	D5185	0.02	0.24	0.21	0.00	0.10
6016		-----	-----	-----	-----	-----

Lab 257 first reported: 1.93, 0.01, 0.29 and 0.1 respectively

lab	method	Potassium as K	Silver as Ag	Sodium as Na	Titanium as Ti	Vanadium as V
178	D5185	-----	<0.1	<3	-----	<1
179	D5185	<1	<0.1	<3	<1	<1
212	D5185	0	0	0	0	0
214	D6595	0	0	6	0	0
237		-----	-----	-----	-----	-----
257	D6595	0.16	-----	0.63 C	0.04	0.28 C
325	D5185	0	0	0	0	0
331	D5185 Mod.	1.0	<0.5	0.8	<0.5	<0.5
349	D5185	0	0	0	0	0
451		-----	-----	-----	-----	-----
496	D5185	0.037	<0.5	0.367	0.026	<1
575	D6595	-----	0.0	0.0	0.0	0.0
633	D6595	0.13	0	1.87	0.08	0.05
663	D5185	0.54	0.00	-----	0.05	0.13
862	D5185	<1	<1	1	<1	<1
863	D5185	<1	<1	1	<1	<1
902	D5185	-----	-----	-----	-----	-----
962	D5185	-----	-----	-----	-----	-----
963	D5185	0.07	0.00	0.99	0.03	0.26
974	D5185	<1	<1	1	<1	<1
1146	In house	-----	0	2.011	0.0397	0.5495
1148		-----	-----	-----	-----	-----
1435	D5185	<1	<1	<1	<1	<1
1571	D5185	-----	0.11172	0.52	<0,01	<0,01
1740	D6595	-----	0.1	1.5	0.1	0.2
1743	D5185	0.02	0.00	0.38	0.03	0.01
6016		-----	-----	-----	-----	-----

Lab 257 first reported: 0.65 and 0.29 respectively

APPENDIX 3 Reported analytical details

Level of Contamination

lab	Sample rolled	Sample diluted	Dilution ratio	Manufacturer / model of equipment
178	---	---		
179	---	---		
212	---	---		
214	Yes	No		PAMAS S40
237	---	---		
257	---	---		
325	---	---		
331	---	---		
349	---	---		
451	---	---		
496	Yes	No		
575	---	---		
633	---	Yes	5:95 sample: diluent dilution ratio. D(r,s) = 2 (dil. ratio with respect to sample)	Pamas / S40
663	Yes	Yes	D(r,d) = 2 (dil. ratio with respect to diluent)	
862	Yes	Yes	2	Manufacturer :Olympus Model:CIX100
863	---	---		
902	Yes	Yes	The device is diluting automaticly.	CINRG
962	---	---		
963	---	---		
974	---	---		
1146	No	Yes	1:2.0	Pamas SBSS-C HCB-LD-50/50
1148	No	No		
1435	Yes	No		SBSS - Pamas
1571	Yes	No	-	Pamas SBSS
1740	Yes	No		PAMAS SBSS
1743	No	No		Microscope Olympus BH-2
6016	---	---		

APPENDIX 4

Number of participants per country

1 lab in ALGERIA
2 labs in BELGIUM
2 labs in CHINA, People's Republic
1 lab in COLOMBIA
2 labs in FRANCE
2 labs in GERMANY
1 lab in GREECE
1 lab in KAZAKHSTAN
1 lab in MOROCCO
1 lab in NETHERLANDS
1 lab in NIGERIA
1 lab in PHILIPPINES
2 labs in SAUDI ARABIA
1 lab in SPAIN
1 lab in SWEDEN
1 lab in TANZANIA
1 lab in THAILAND
1 lab in TURKEY
1 lab in UNITED ARAB EMIRATES
1 lab in UNITED KINGDOM
2 labs in UNITED STATES OF AMERICA

APPENDIX 5

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

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