Results of Proficiency Test
Gear Oil (used)
March 2021

Organized 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 scheme for the analysis of used Gear Oil every year. During the annual proficiency testing program 2020/2021 it was decided to continue the round robin for the analysis of used Gear Oil.

In this interlaboratory study 29 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 used Gear Oil proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.
It was decided to send two different samples of used Gear Oil, one 0.5L bottle labelled #21031 for the regular analyzes and one 50mL HDPE bottle labelled #21032 for metals determination only.
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

For the preparation of the sample for the regular PT a batch of approximately 60 liters of used Gear Oil was obtained from a third party. After homogenization 50 amber glass bottles of 0.5L were filled and labelled #21031.
The homogeneity of the subsamples was checked by the determination of Density at 15 °C in accordance with ISO12185 on 8 stratified randomly selected subsamples.

	Density at 15°C in kg/L
Sample #21031-1	0.85769
Sample #21031-2	0.85769
Sample #21031-3	0.85769
Sample #21031-4	0.85769
Sample #21031-5	0.85768
Sample #21031-6	0.85769
Sample #21031-7	0.85768
Sample #21031-8	0.85768

Table 1: homogeneity test results of subsamples #21031

From the above test results the repeatability was calculated and compared with 0.3 times the 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	ISO12185:96
0.3 x R (reference test	0.00015

Table 2: evaluation of the repeatability of subsamples #21031

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

For the preparation of the sample for the Metals PT a batch of approximately 35 liters of used Gear Oil was obtained from a third party. After homogenization 50 HDPE bottles of 50mL were filled and labelled #21032.
The homogeneity of the subsamples was checked by the determination of Iron in accordance with ASTM D5185 on 8 stratified randomly selected subsamples.

	Iron as Fe in mg/kg
Sample #21032-1	193
Sample #21032-2	193
Sample #21032-3	193
Sample #21032-4	193
Sample #21032-5	192
Sample #21032-6	193
Sample #21032-7	195
Sample #21032-8	195

Table 3: homogeneity test results of subsamples #21032

From the above test results the repeatability was calculated and compared with 0.3 times the 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)	3.0
reference test method	ASTM D5185:18
0.3 x R (reference test method)	10.5

Table 4: evaluation of the repeatability of subsamples #21032

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

To each of the participating laboratories one sample used Gear Oil labelled #21031 and one sample used Gear Oil labelled #21032 were sent on February 24, 2021. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYZES

The participants were requested to determine on sample #21031: Total Acid Number, Density at 15°C, Flash Point PMcc, Kinematic Viscosity at 40°C and 100°C, Viscosity Index, Membrane Filtration 5.0µm, Water and Level of Contamination (counts/mL and scale number).
Also, some extra information was asked about the determination of Total Acid Number and Level of Contamination.

On sample #21032 it was requested to determine the elements Aluminum as Al, Barium as Ba, Boron as B, Cadmium as Cd, Chromium as Cr, Copper as Cu, Iron as Fe, Lead as Pb, Lithium as Li, Magnesium as Mg, Manganese as Mn, Molybdenum as Mo, Nickel as Ni, Potassium as K, Silicon as Si, Silver as Ag, Sodium as Na, Tin as Sn, Titanium as Ti, Vanadium as V, Calcium as Ca, Phosphorus as P, Zinc as Zn.

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 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 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.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner’s outlier test can be used. 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 (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

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 or 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, 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 test result tables in appendix 1.

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

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

4 EVALUATION

In this proficiency test no severe problems were encountered with the dispatch of the samples. Considering both the regular and metals samples one participant reported test results after the final reporting date and one other participant did not report any test results. Not all participants were able to report all tests requested.
In total 28 participants reported 616 numerical test results. Observed were 31 outlying test results, which is 5.0%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

Not all 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 original data. The abbreviations, used in these tables, are explained in appendix 5.

Unfortunately, a suitable reference test method, providing the precision data, is not available for all determinations. For these tests the calculated reproducibility was compared against the estimated reproducibility calculated with the Horwitz equation.

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 #21031

Total Acid Number: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D664-A:18e2 for all end point modes (BEP 60mL and 125mL and IP 60mL and 125mL).
Remarkably, three participants still have used pH 11 for BEP instead of pH 10. In test method ASTM D664:18e2 pH 10 is mentioned.

Density at 15°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ISO12185:96.

Flash Point PMcc: 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 D93:20 procedures A and B.
Both procedures (A and B) of ASTM D93 may be applicable for this determination (in-use vs used lubricating oil). The majority of the participants used procedure A. Remarkably, one participant used procedure C which is applicable to Biodiesel (B100).

Kinematic Viscosity at 40°C: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ASTM D445:19a.

Kinematic Viscosity at 100°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 D445:19a.

Viscosity Index: This determination was not problematic. One statistical outlier was observed and two other test results were excluded. The calculated reproducibility after rejection of the suspect data is in full agreement with the requirements of ASTM D2270:10(2016).

Membrane Filtration 5.0µm: 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(2019).

Water: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D6304:20 procedure C, but is in agreement with procedure A and B.

Level of Contamination: This determination was problematic. In total nine statistical outliers were observed over six parameters (4 in counts/mL and 5 in scale number) and seven other test results were excluded. The calculated reproducibilities after rejection of the suspect data are not in agreement with the requirements of ASTM D7647:10(2018). No clear conclusion could be drawn from the reported analytical details given in appendix 3.

Sample #21032

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

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

Boron as B: 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.

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

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

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

Lithium as Li: This determination may be problematic. No statistical outliers were observed but one test result was excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the estimated reproducibility calculated with the Horwitz equation.

Magnesium as Mg: 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.

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

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

Potassium as K: 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 as Si: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D5185:18.

Sodium as Na: 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.

Calcium as Ca: This determination may be problematic. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the estimated reproducibility calculated with the Horwitz equation but also not in agreement with the strict requirements of ASTM D5185:18.

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

Zinc as Zn: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated reproducibility calculated with the Horwitz equation but not in agreement with the strict requirements of ASTM D5185:18.

The majority of the participants agreed on a concentration near or below the limit of detection for all other elements mentioned in paragraph 2.6. Therefore, no z-scores were calculated for these elements. The test results of these components are given in appendix 2.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Parameter	unit	n	average	2.8 * sd	R(lit)
Total Acid Number	mg KOH/g	22	0.62	0.41	0.35
Density at 15°C	kg/L	23	0.8576	0.0004	0.0005
Flash Point PMcc	°C	18	198.4	8.0	14.1
Kinematic Viscosity at 40°C	mm^2/s	21	51.63	0.50	0.53
Kinematic Viscosity at 100°C	mm^2/s	22	8.576	0.119	0.078
Viscosity Index		15	142.1	2.2	2
Membrane Filtration 5.0µm	%M/M	5	0.0138	0.0342	0.0436
Water	mg/kg	19	249	109	93
Level of Contamination					
≥ 4µm (c)	counts/mL	10	70693	121191	79883
≥ 6µm (c)	counts/mL	10	2384	3285	1812
≥14µm (c)	counts/mL	11	32.9	62.3	44.4
≥ 4µm (c)	scale number	12	23.0	2.4	1.7
≥ 6µm (c)	scale number	12	18.0	2.4	1.2
≥14µm (c)	scale number	13	11.8	3.1	2

Table 5: reproducibilities of tests on sample #21031

Parameter	unit	n	average	2.8 * sd	R(lit)
Aluminum as Al	mg/kg	26	12.6	3.6	7.4
Barium as Ba	mg/kg	19	0.69	0.62	0.42
Boron as B	mg/kg	24	28.7	11.7	13.4
Chromium as Cr	mg/kg	25	2.3	1.1	1.3
Copper as Cu	mg/kg	24	6.8	1.2	1.6
Iron as Fe	mg/kg	25	197	42	36
Lithium as Li	mg/kg	12	0.61	0.62	0.29
Magnesium as Mg	mg/kg	25	7.7	2.4	3.5
Manganese as Mn	mg/kg	23	3.2	0.7	0.5
Molybdenum as Mo	mg/kg	24	4.7	1.7	1.9
Potassium as K	mg/kg	18	1.0	1.2	6.6
Silicon as Si	mg/kg	24	7.9	5.4	6.5
Sodium as Na	mg/kg	24	45.8	8.5	16.6
Calcium as Ca	mg/kg	22	635	123	108
Phosphorus as P	mg/kg	23	519	122	98
Zinc as Zn	mg/kg	24	65.5	10.3	15.6

Table 6: reproducibilities of tests on sample #21032

Without further statistical calculations, it can be concluded that for many tests there is a good compliance of the group of participants with the reference test methods. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF MARCH 2021 WITH PREVIOUS PTS

	March 2021	March 2020	April 2019	April 2018	April 2017
Number of reporting laboratories	28	24	24	22	17
Number of test results	616	414	421	391	362
Number of statistical outliers	31	25	22	18	23
Percentage of statistical outliers	5.0%	6.0%	5.2%	4.6%	6.4%

Table 7: 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 to the requirements of the reference test methods. The conclusions are given in the following table.

Parameter	March 2021	March 2020	April 2019	April 2018	April 2017
Total Acid Number	-	+/-	-	-	-
Density at 15°C	+	-	-	-	-
Flash Point PMcc	+	+/-	-	-	+
Kinematic Viscosity at 40°C	+/-	++	+	+	++
Kinematic Viscosity at 100°C	-	-	-	--	+
Viscosity Index	+/-	-	+/-	--	+
Membrane Filtration 5.0µm	+	++	++	n.e.	n.e.
Water	-	+	+	++	++
Level of Contamination	-	-	-	-	--
Aluminum as Al	++	+	+	+	n.e.
Barium as Ba	-	n.a.	n.a.	n.a.	n.a.
Boron as B	+	++	++	+	+
Chromium as Cr	+	n.e.	n.e.	++	n.e.
Copper as Cu	+	+	+	+	(-)
Iron as Fe	-	+	+	+	+
Lithium as Li	--	n.a.	n.a.	n.a.	n.a.
Magnesium as Mg	+	n.a.	n.a.	n.a.	n.a.
Manganese as Mn	-	n.a.	n.a.	n.a.	n.a.
Molybdenum as Mo	+	n.a.	n.a.	n.a.	n.a.
Potassium as K	++	n.a.	n.a.	n.a.	n.a.
Silicon as Si	+	++	++	++	n.e.
Tin as Sn	n.e.	+	+	+	n.e.
Sodium as Na	+	+	+	+	n.e.
Calcium as Ca	-	(--)	-	-	(-)
Phosphorus as P	-	+	-	++	+
Zinc as Zn	+	(--)	(--)	+/-	(-)

Table 8: comparison determinations against the reference test methods

The following performance categories were used:

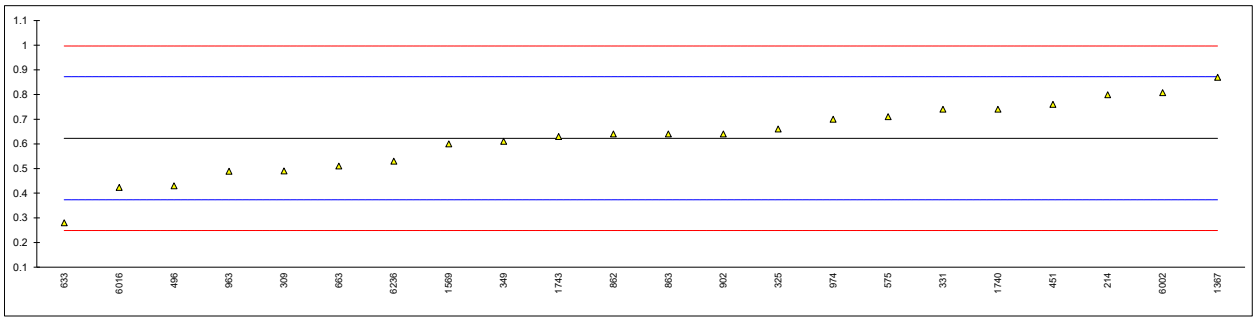
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test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

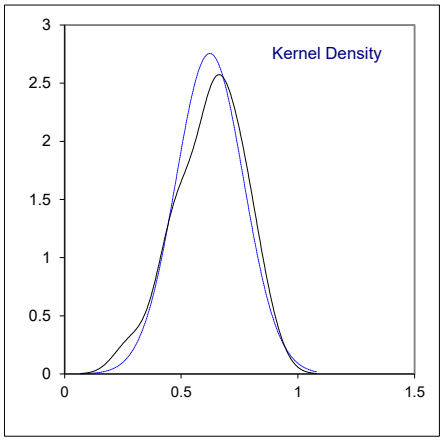
APPENDIX 1

Determination of Total Acid Number on sample #21031; results in mg KOH/g

lab	method	value	mark	z(targ)	end point type	volume (mL)	remarks
178		-----		-----	---	---	
179		-----		-----	---	---	
214	D664-A	0.799		1.41	Inflection Point	125	
237		-----		-----	---	---	
257		-----		-----	---	---	
309	D664-A	0.49		-1.06	Buffer End Point pH 10	60	
325	D664-A	0.66		0.30	Buffer End Point pH 11	125	
331	D664Mod.	0.74		0.94	Inflection Point	60	
349	D664-A	0.61		-0.10	Buffer End Point pH 10	125	
451	D664-A	0.76		1.10	Buffer End Point pH 10	60	
496	D664-A	0.43		-1.55	Buffer End Point pH 10	60	
575	D664-A	0.71		0.70	Buffer End Point pH 10	60	
633	D664-A	0.28		-2.75	Inflection Point	125	
663	D664-A	0.51		-0.90	Buffer End Point pH 10	60	
862	D664-A	0.64		0.14	Inflection Point	60	
863	D664-A	0.64		0.14	Inflection Point	60	
902	D664-A	0.64		0.14	Inflection Point	60	
962		-----		-----	Inflection Point	125	
963	D664-A	0.489		-1.07	Inflection Point	60	
974	D664-A	0.70		0.62	Buffer End Point pH 10	125	
1146		-----		-----	---	---	
1367	IP177	0.87		1.98	Buffer End Point pH 11	60	
1448		-----		-----	---	---	
1569	D664-B	0.60		-0.18	Inflection Point	125	
1740	D664-A	0.74		0.94	Inflection Point	60	
1743	D664-A	0.63		0.06	Buffer End Point pH 11	60	
6002	D664-A	0.807		1.48	Buffer End Point pH 10	60	
6016	D664-A	0.4235		-1.60	Inflection Point	60	
6236	D8045	0.530		-0.74	Inflection Point	60	

	normality	OK
	n	22
	outliers	0
	mean (n)	0.6227
	st.dev. (n)	0.14479
	R(calc.)	0.4054
	st.dev.(D664-A:18e2, BEP 60mL)	0.12466
	R(D664-A:18e2, BEP 60mL)	0.3491
Compare		
	R(D664-A:18e2, BEP 125mL)	0.1920
	R(D664-A:18e2, IP 60mL)	0.2727
	R(D664-A:18e2, IP 125mL)	0.1335

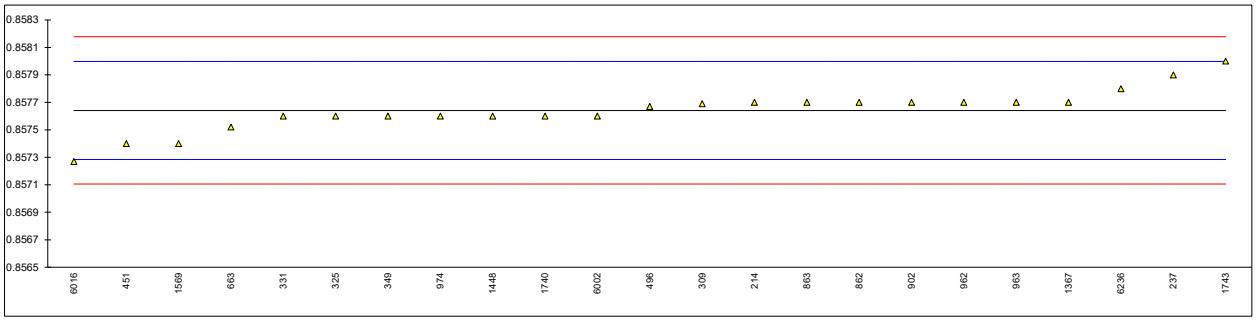


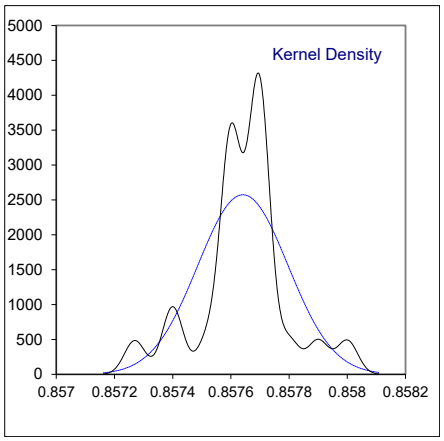


Determination of Density at 15°C on sample #21031; results in kg/L

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214	D7042	0.8577		0.33	
237	D4052	0.8579		1.45	
257		-----		-----	
309	D4052	0.85769		0.27	
325	D4052	0.8576		-0.23	
331	ISO12185	0.8576	C	-0.23	first reported 857.6 without unit
349	D4052	0.8576		-0.23	
451	D4052	0.8574		-1.35	
496	ISO12185	0.85767		0.16	
575		-----		-----	
633		-----		-----	
663	D4052	0.85752		-0.68	
862	D4052	0.8577		0.33	
863	D4052	0.8577		0.33	
902	D4052	0.8577		0.33	
962	ISO12185	0.8577		0.33	
963	D4052	0.8577		0.33	
974	D4052	0.8576		-0.23	
1146		-----		-----	
1367	IP365	0.8577	C	0.33	first reported 854.4 kg/m^3
1448	D4052	0.8576	C	-0.23	first reported 885.1 kg/m^3
1569	D4052	0.8574		-1.35	
1740	D4052	0.8576		-0.23	
1743	ISO12185	0.8580		2.01	
6002	ISO12185	0.8576		-0.23	
6016	D4052	0.85727	C	-2.08	first reported 857.27 kg/L
6236	D4052	0.8578		0.89	

normality	suspect
n	23
outliers	0
mean (n)	0.857641
st.dev. (n)	0.0001551
R(calc.)	0.000434
st.dev.(ISO12185:96)	0.0001786
R(ISO12185:96)	0.0005

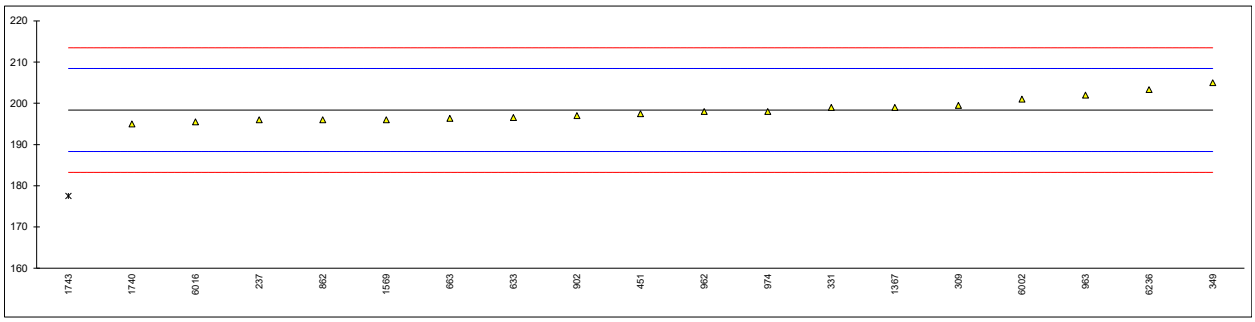


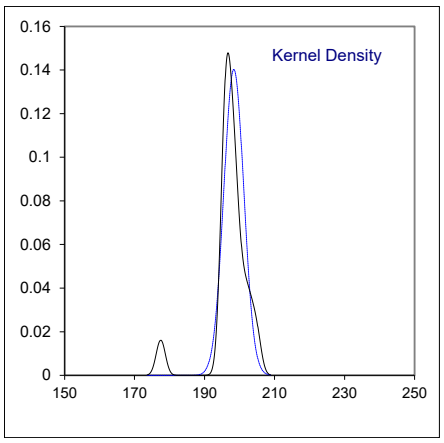


Determination of Flash Point PMcc on sample #21031; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214		-----		-----	
237	D93-B	196.0		-0.47	
257		-----		-----	
309	D93-A	199.5		0.22	
325		-----		-----	
331	D93-A	199		0.12	
349	D93-A	205		1.32	
451	D93-A	197.5		-0.17	
496		-----		-----	
575		-----		-----	
633	D93-A	196.55		-0.36	
663	D93-A	196.35		-0.40	
862	D93-A	196		-0.47	
863		-----		-----	
902	D93-A	197		-0.27	
962	D93-A	198.0		-0.07	
963	D93-A	202.0		0.72	
974	D93-A	198		-0.07	
1146		-----		-----	
1367	D93-A	199.0		0.12	
1448		-----		-----	
1569	D93-B	196.0		-0.47	
1740	D93-A	195	C	-0.67	first reported 213
1743	ISO2719-A	177.5	G(0.01)	-4.15	
6002	ISO2719-A	201.0		0.52	
6016	D93-A	195.5		-0.57	
6236	D93-C	203.33		0.99	

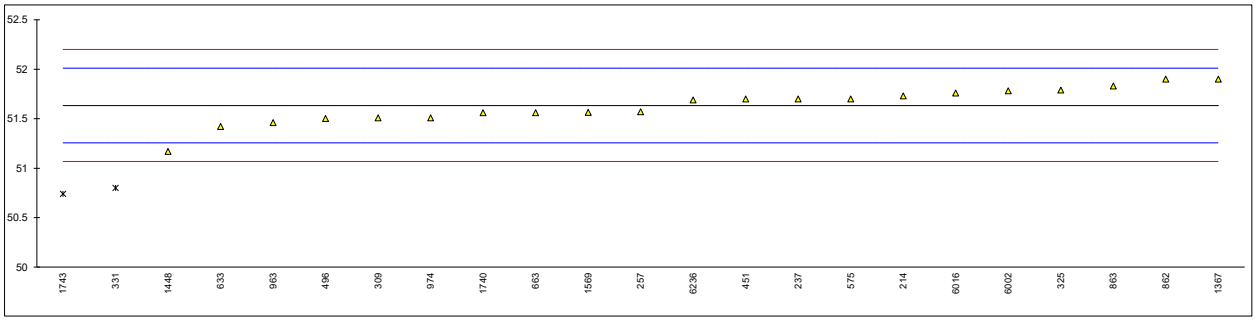
	normality	suspect
	n	18
	outliers	1
	mean (n)	198.37
	st.dev. (n)	2.844
	R(calc.)	7.96
	st.dev.(D93-A:20)	5.030
	R(D93-A:20)	14.08
Compare:		
	R(D93-B:20)	10

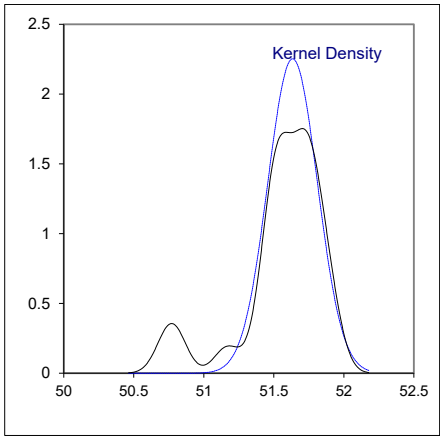




Determination of Kinematic Viscosity at 40°C on sample #21031; results in mm^2/s

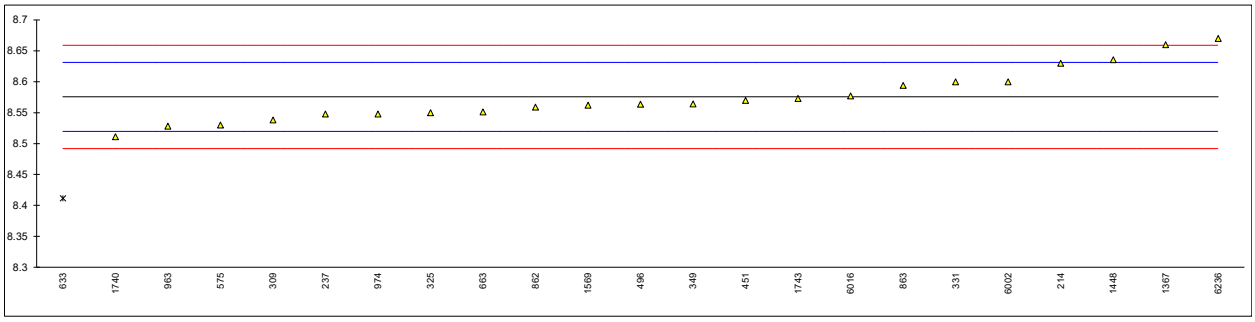
lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214	D445	51.73		0.51	
237	D445	51.70		0.35	
257	D7279 corrected to D445	51.57		-0.34	
309	D445	51.51		-0.66	
325	D445	51.79		0.83	
331	D7279 corrected to D445	50.8	R(0.01)	-4.41	
349		-----		-----	
451	D7279 corrected to D445	51.70		0.35	
496	D445	51.502		-0.70	
575	D7279 corrected to D445	51.7		0.35	
633	D7279 corrected to D445	51.422		-1.12	
663	D445	51.561		-0.39	
862	D445	51.90		1.41	
863	D445	51.83		1.04	
902		-----		-----	
962		-----		-----	
963	D445	51.46		-0.92	
974	D445	51.51		-0.66	
1146		-----		-----	
1367	D7279 corrected to D445	51.9		1.41	
1448	D7042	51.1687		-2.46	
1569	D445	51.563		-0.37	
1740	D445	51.56		-0.39	
1743	D7279 corrected to D445	50.74	R(0.01)	-4.73	
6002	ISO3104	51.783		0.79	
6016	D7042	51.760		0.67	
6236	D7279 corrected to D445	51.69		0.30	
	normality	OK			
	n	21			
	outliers	2			
	mean (n)	51.6338			
	st.dev. (n)	0.17714			
	R(calc.)	0.4960			
	st.dev.(D445:19a)	0.18893			
	R(D445:19a)	0.5290			R(D445:19a - used formulated oils)

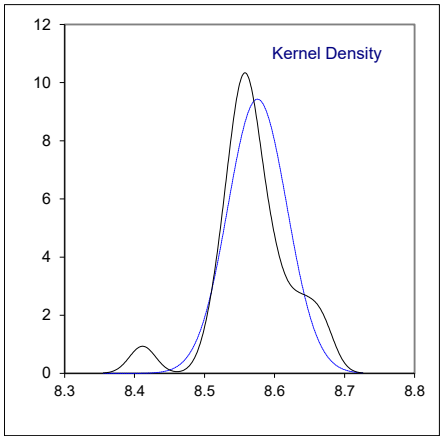




Determination of Kinematic Viscosity at 100°C on sample #21031; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214	D445	8.63		1.95	
237	D445	8.548		-0.99	
257		-----		-----	
309	D445	8.538		-1.35	
325	D445	8.550		-0.92	
331	D7279 corrected to D445	8.6		0.88	
349	D445	8.564		-0.42	
451	D7279 corrected to D445	8.57		-0.20	
496	D445	8.5635		-0.43	
575	D7279 corrected to D445	8.53		-1.64	
633	D7279 corrected to D445	8.4115	R(0.05)	-5.89	
663	D445	8.5513		-0.87	
862	D445	8.559		-0.59	
863	D445	8.594		0.66	
902		-----		-----	
962		-----		-----	
963	D445	8.528		-1.71	
974	D445	8.548		-0.99	
1146		-----		-----	
1367	D7279 corrected to D445	8.66		3.03	
1448	D7042	8.6357		2.16	
1569	D445	8.562		-0.49	
1740	D445	8.511		-2.32	
1743	D7279 corrected to D445	8.573		-0.09	
6002	ISO3104	8.600		0.88	
6016	D7042	8.577		0.05	
6236	D7279 corrected to D445	8.67	C	3.39	first reported 8.72
	normality	OK			
	n	22			
	outliers	1			
	mean (n)	8.5756			
	st.dev. (n)	0.04232			
	R(calc.)	0.1185			
	st.dev.(D445:19a)	0.02786			
	R(D445:19a)	0.0780			R(D445:19a - used formulated oils)

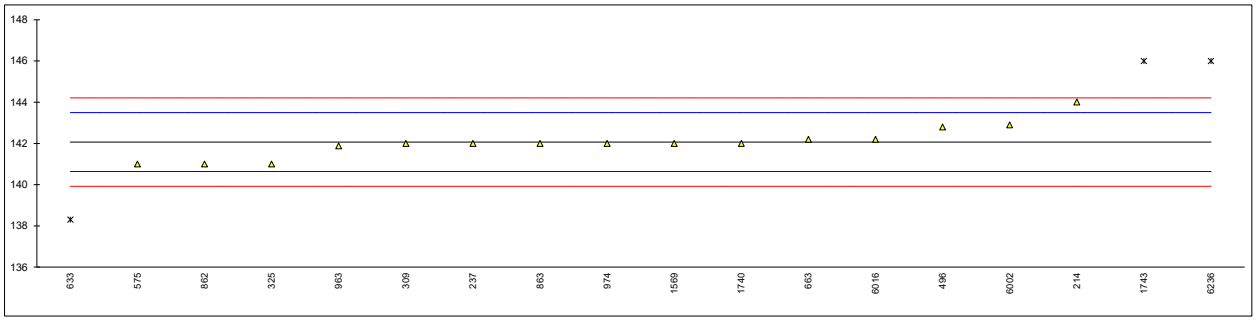


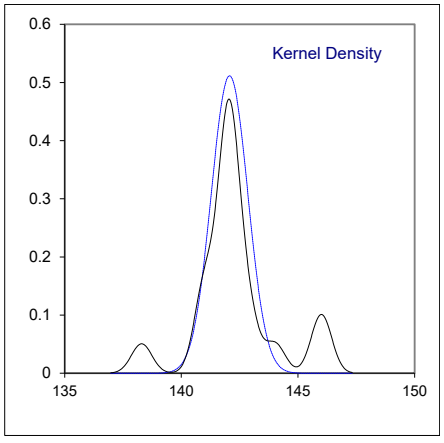


Determination of Viscosity Index on sample #21031

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214	D2270	144.011		2.72	
237	D2270	142		-0.09	
257		-----		-----	
309	D2270	142		-0.09	
325	D2270	141		-1.49	
331		-----		-----	
349		-----		-----	
451		-----		-----	
496	D2270	142.8		1.03	
575	D2270	141		-1.49	
633	D2270	138.3	ex	-5.27	test result excluded as statistical outlier in KV 100°C
663	D2270	142.2		0.19	
862	D2270	141		-1.49	
863	D2270	142		-0.09	
902		-----		-----	
962		-----		-----	
963	D2270	141.880		-0.26	
974	D2270	142		-0.09	
1146		-----		-----	
1367		-----		-----	
1448		-----		-----	
1569	D2270	142		-0.09	
1740	D2270	142	E	-0.09	iis calculated 141
1743	ISO2909	146	ex	5.51	test result excluded as statistical outlier in KV 40°C
6002	ISO2909	142.9		1.17	
6016	D2270	142.2		0.19	
6236	D2270	146	C,G(0.01),E	5.51	first reported 147 / iis calculated 145

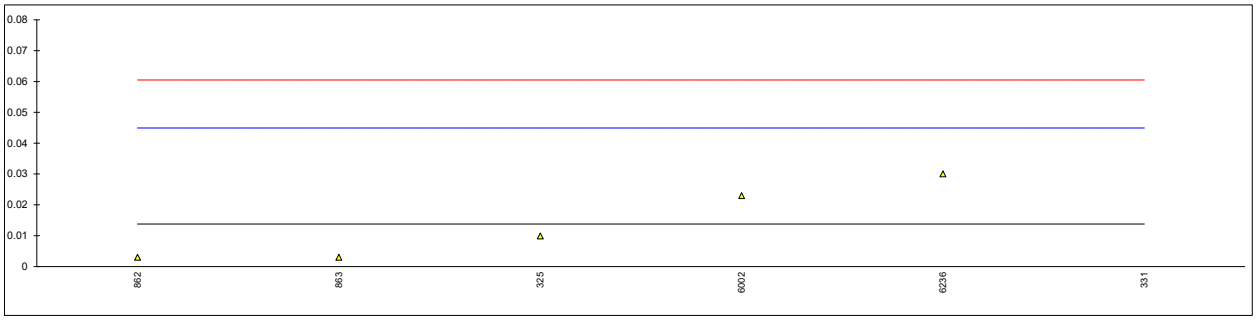
normality	suspect
n	15
outliers	1 +2ex
mean (n)	142.07
st.dev. (n)	0.780
R(calc.)	2.18
st.dev.(D2270:10)	0.714
R(D2270:10)	2





Determination of Membrane Filtration 5.0 µm on sample #21031; results in %M/M

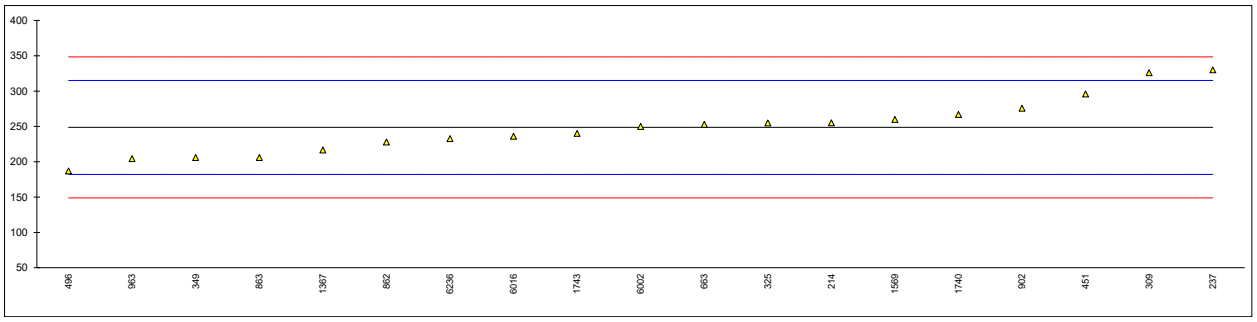
lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214		-----		-----	
237		-----		-----	
257		-----		-----	
309		-----		-----	
325	D4055	0.0099		-0.25	
331	NF E18652	8	D(0.01)	512.72	Possibly reported in different unit (mg/kg)?
349		-----		-----	
451		-----		-----	
496		-----		-----	
575		-----		-----	
633		-----		-----	
663		-----		-----	
862	D4055	0.003		-0.69	
863	D4055	0.003		-0.69	
902		-----		-----	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1146		-----		-----	
1367		-----		-----	
1448		-----		-----	
1569		-----		-----	
1740		-----		-----	
1743		-----		-----	
6002	D4055	0.02297	C	0.59	first reported 0.2297
6016		-----		-----	
6236	D4055	0.030		1.04	
	normality	unknown			
	n	5			
	outliers	1			
	mean (n)	0.0138			
	st.dev. (n)	0.01220			
	R(calc.)	0.0342			
	st.dev.(D4055:04)	0.01558			
	R(D4055:04)	0.0436			

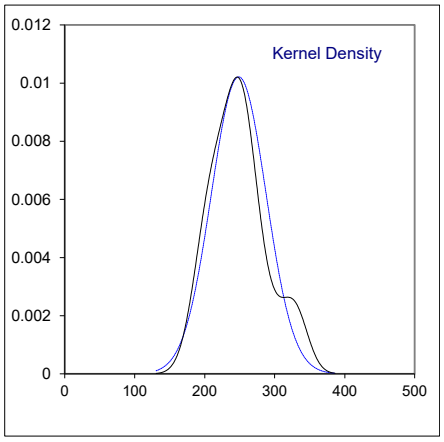


Determination of Water on sample #21031; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214	D6304-A	255.12		0.19	
237	D6304-C	330		2.45	
257		-----		-----	
309	D6304-A	326		2.33	
325	D6304-C	255		0.19	
331		-----		-----	
349	D6304-C	206		-1.28	
451	D6304-C	296		1.43	
496	D6304-C	187		-1.86	
575		-----		-----	
633		-----		-----	
663	D6304-C	252.8		0.12	
862	D6304-B	228	C	-0.62	first reported 104
863	D6304-B	206	C	-1.28	first reported 102
902	D6304-C	275.7		0.81	
962		-----		-----	
963	D6304-C	204.5		-1.33	
974		-----		-----	
1146		-----		-----	
1367	D6304-C	216.6		-0.96	
1448		-----		-----	
1569	D6304-B	260		0.34	
1740	D6304-B	267		0.55	
1743	ISO12937	240		-0.26	
6002	In house	249.95		0.04	
6016	D6304	236		-0.38	
6236	D6304-A	232.67		-0.48	

	normality	OK
	n	19
	outliers	0
	mean (n)	248.65
	st.dev. (n)	39.068
	R(calc.)	109.39
	st.dev.(D6304-C:20)	33.222
	R(D6304-C:20)	93.02
Compare:		
	R(D6304-A:20)	114.44
	R(D6304-B:20)	224.87



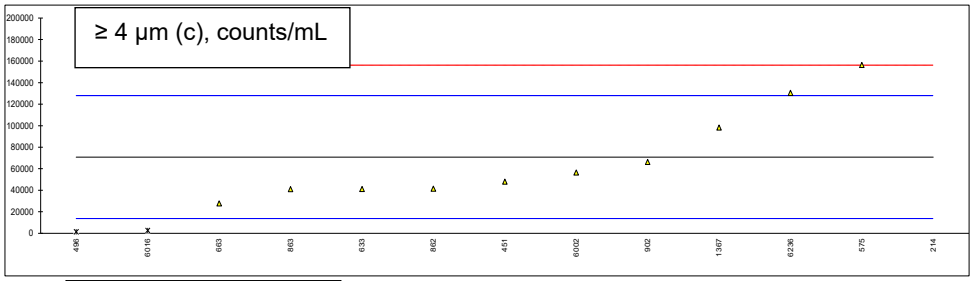


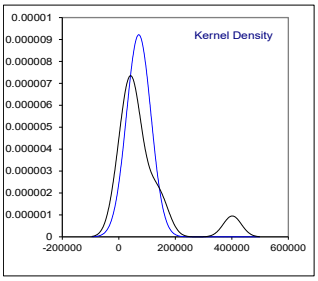
Determination of Level of Contamination on sample #21031; results in counts/mL

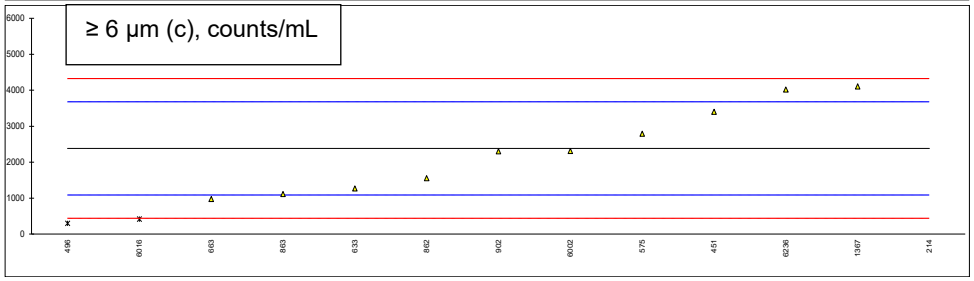
lab	method	≥ 4 µm (c)	mark	z(targ)	≥ 6 µm (c)	mark	z(targ)	≥ 14 µm (c)	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179		-----		-----	-----		-----	-----		-----
214	ISO4406	401745	G(0.01)	11.60	17436	G(0.01)	23.27	327	G(0.01)	18.54
237		-----		-----	-----		-----	-----		-----
257		-----		-----	-----		-----	-----		-----
309		-----		-----	-----		-----	-----		-----
325		-----		-----	-----		-----	-----		-----
331		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
451	ISO11500	47972		-0.80	3401		1.57	10		-1.44
496	D7596	1377	ex	-2.43	297	ex	-3.23	24		-0.56
575	D7647	156309		3.00	2792	C	0.63	551	C,G(0.01)	32.66
633	D7647	41149		-1.04	1269		-1.72	55		1.39
663	D7647	27714		-1.51	976		-2.18	60		1.71
862	ISO11500	41372		-1.03	1551		-1.29	12		-1.32
863	ISO11500	40911		-1.04	1112		-1.97	27		-0.37
902	D7647	66348		-0.15	2302		-0.13	17		-1.00
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
974		-----		-----	-----		-----	-----		-----
1146		-----		-----	-----		-----	-----		-----
1367	D7647	98274.5		0.97	4106		2.66	28		-0.31
1448		-----		-----	-----		-----	-----		-----
1569		-----		-----	-----		-----	-----		-----
1740		-----		-----	-----		-----	-----		-----
1743		-----		-----	-----		-----	-----		-----
6002	D7647	56476.8		-0.50	2309.1		-0.12	64.2		1.97
6016	D7596	2650	ex	-2.38	417.9	ex	-3.04	6		-1.70
6236	D7647	130405.4		2.09	4018.4		2.53	58.7		1.63

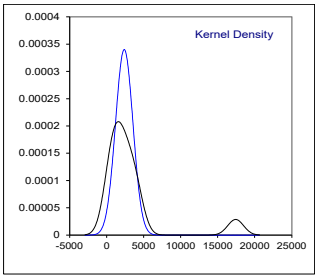
normality	suspect	OK	OK
n	10	10	11
outliers	1 +2ex	1 +2ex	2
mean (n)	70693	2384	32.9
st.dev. (n)	43282.5	1173.2	22.23
R(calc.)	121191	3285	62.3
st.dev.(D7647:10)	28529.7	647.0	15.86
R(D7647:10)	79883	1812	44.4

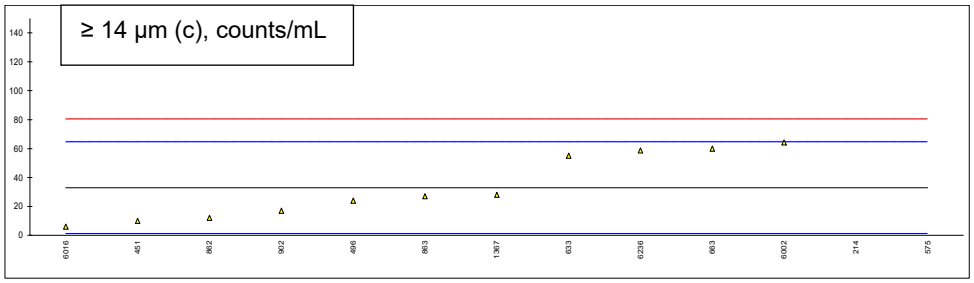
Lab 496 test results excluded at ≥ 4 µm and ≥ 6 µm as corresponding test results in scale number are statistical outliers
Lab 575 first reported 30437 at ≥ 6 µm and 278 at ≥ 14 µm
Lab 6016 test results excluded at ≥ 4 µm and ≥ 6 µm as corresponding test results in scale number are statistical outliers

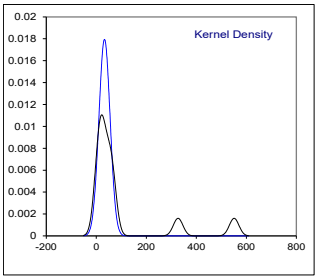










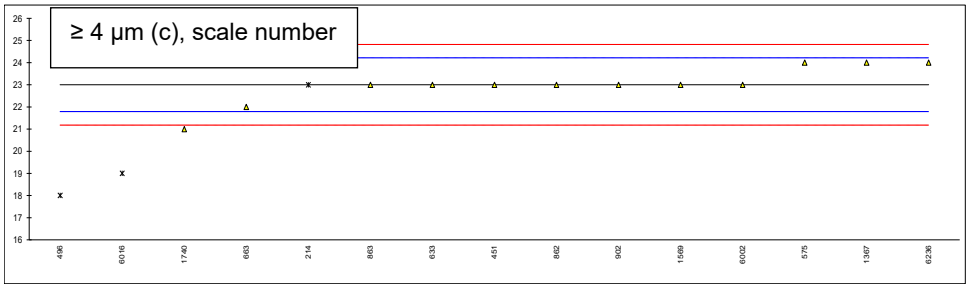


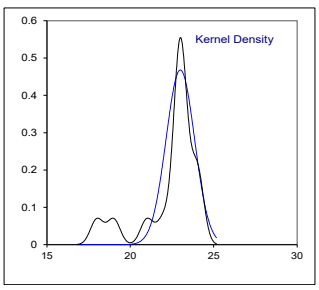
Determination of Level of Contamination acc. to ISO4406 scale on sample #21031; results in scale number

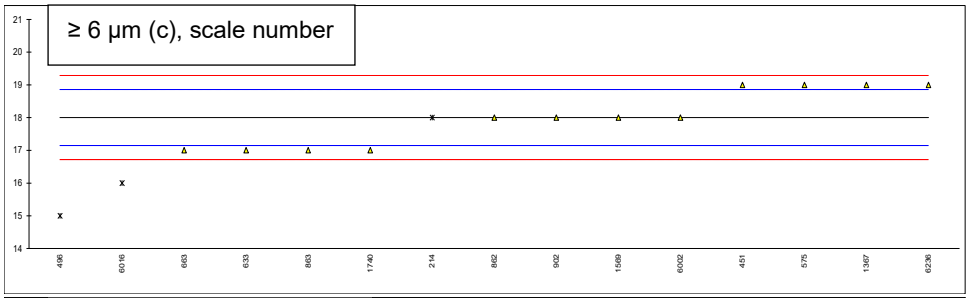
lab	method	≥ 4 µm (c)	mark	z(targ)	≥ 6 µm (c)	mark	z(targ)	≥ 14 µm (c)	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179		-----		-----	-----		-----	-----		-----
214	ISO4406	23	E,ex	0.00	18	E,ex	0.00	12	E,ex	0.32
237		-----		-----	-----		-----	-----		-----
257		-----		-----	-----		-----	-----		-----
309		-----		-----	-----		-----	-----		-----
325		-----		-----	-----		-----	-----		-----
331		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
451	ISO4406	23		0.00	19		2.33	11		-1.08
496	ISO4406	18	DG(0.01)	-8.24	15	G(0.05)	-7.00	12		0.32
575	ISO4406	24		1.65	19	C	2.33	16	C,G(0.05)	5.92
633	ISO4406	23		0.00	17		-2.33	13		1.72
663	D7647	22		-1.65	17		-2.33	13		1.72
862	ISO4406	23		0.00	18		0.00	11		-1.08
863	ISO4406	23		0.00	17		-2.33	12		0.32
902	ISO4406	23		0.00	18		0.00	11		-1.08
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
974		-----		-----	-----		-----	-----		-----
1146		-----		-----	-----		-----	-----		-----
1367	D7647	24		1.65	19		2.33	12		0.32
1448		-----		-----	-----		-----	-----		-----
1569	ISO4406	23		0.00	18		0.00	10		-2.48
1740	ISO4406	21		-3.29	17		-2.33	12		0.32
1743		-----		-----	-----		-----	-----		-----
6002	D7647	23		0.00	18		0.00	13		1.72
6016	ISO4406	19	DG(0.01)	-6.59	16	G(0.05)	-4.67	10		-2.48
6236	ISO4406	24		1.65	19		2.33	13		1.72

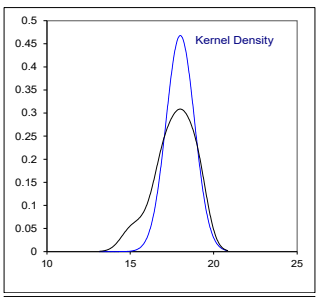
normality	suspect	OK	OK
n	12	12	13
outliers	2 +1ex	2 +1ex	1 +1ex
mean (n)	23.0	18.0	11.8
st.dev. (n)	0.85	0.85	1.09
R(calc.)	2.4	2.4	3.1
st.dev.(D7647:10)	0.61	0.43	0.71
R(D7647:10)	1.7	1.2	2

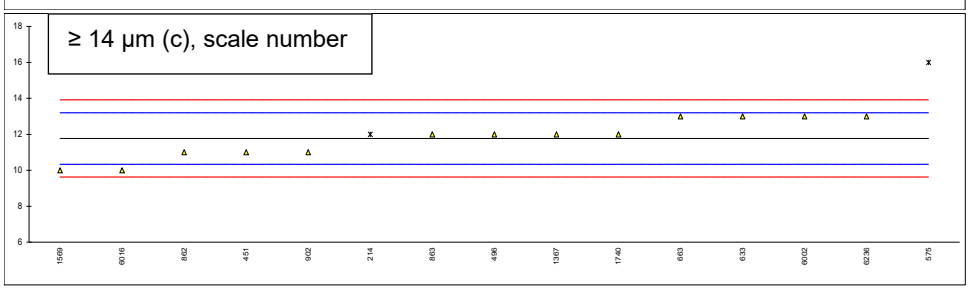
Lab 214 test results excluded as corresponding test results in counts/mL are statistical outliers. Count/mL and ISO class do not match.
Lab 575 first reported 22 at ≥ 6 µm and 15 at ≥ 14 µm

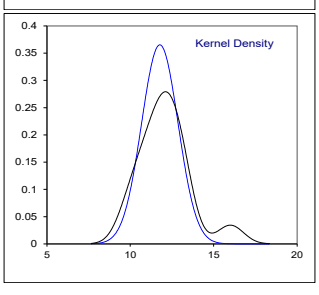








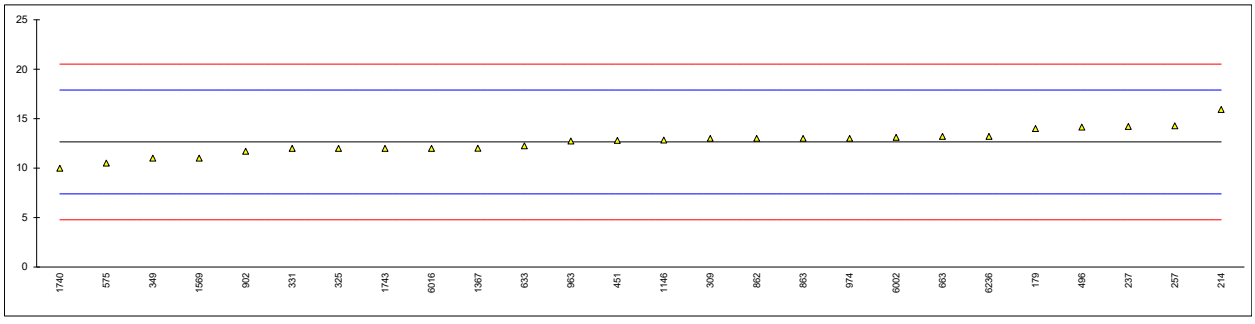


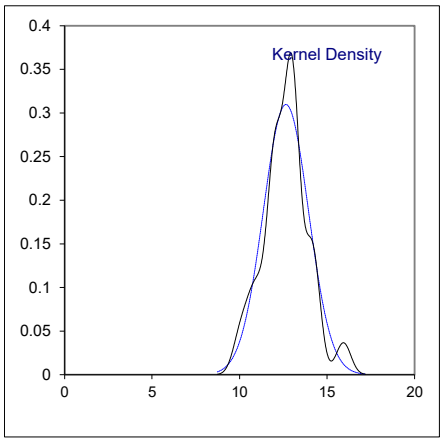


Determination of Aluminum as Al on sample #21032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D5185	14		0.51	
214	D6595	15.93		1.25	
237	D5185	14.22		0.60	
257	D6595	14.27		0.62	
309	D5185	13.00		0.13	
325	D5185	12		-0.25	
331	D5185	12		-0.25	
349	D5185	11		-0.63	
451	D5185	12.8		0.06	
496	D5185	14.14		0.57	
575	D6595	10.5		-0.82	
633	D6595	12.25		-0.15	
663	D5185	13.2		0.21	
862	D5185	13		0.13	
863	D5185	13		0.13	
902	D5185	11.7		-0.36	
962		-----		-----	
963	D5185	12.73		0.03	
974		13		0.13	
1146	D5185	12.83		0.07	
1367	D5185	12.01		-0.24	
1448		-----		-----	
1569	D5185	11		-0.63	
1740	D6595	10		-1.01	
1743		12		-0.25	
6002	D5185	13.1		0.17	
6016	D5185	12		-0.25	
6236	D5185	13.2		0.21	

normality	OK
n	26
outliers	0
mean (n)	12.649
st.dev. (n)	1.2884
R(calc.)	3.608
st.dev.(D5185:18)	2.6252
R(D5185:18)	7.351

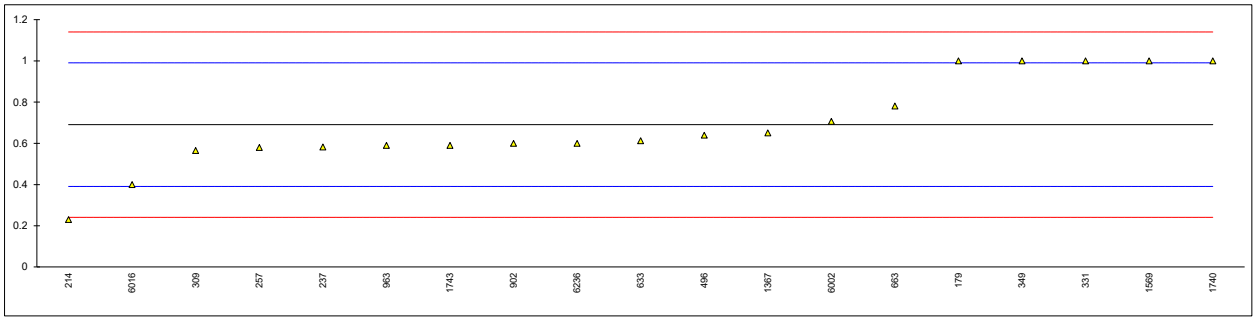


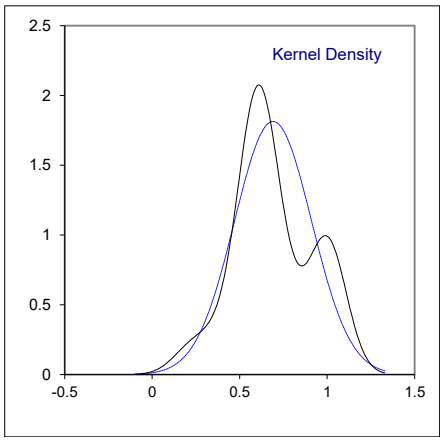


Determination of Barium as Ba on sample #21032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D5185	1		2.06	
214	D6595	0.23		-3.07	
237	D5185	0.5822		-0.73	
257	D6595	0.58		-0.74	
309	D5185	0.565		-0.84	
325	D5185	<1		-----	
331	D5185	1		2.06	
349	D5185	1		2.06	
451	D5185	<1		-----	
496	D5185	0.639		-0.35	
575		-----		-----	
633	D6595	0.613		-0.52	
663	D5185	0.7815		0.60	
862	D5185	<1		-----	
863	D5185	<1		-----	
902	D5185	0.6		-0.61	
962		-----		-----	
963	D5185	0.59		-0.67	
974		<1		-----	
1146	D5185	<5		-----	
1367	D5185	0.651		-0.27	
1448		-----		-----	
1569	D5185	1		2.06	
1740	D6595	1		2.06	
1743		0.59		-0.67	
6002	D5185	0.707		0.11	
6016	D5185	0.4		-1.94	
6236	D5185	0.6		-0.61	

normality	OK
n	19
outliers	0
mean (n)	0.691
st.dev. (n)	0.2199
R(calc.)	0.616
st.dev.(D5185:18)	0.1500
R(D5185:18)	0.420

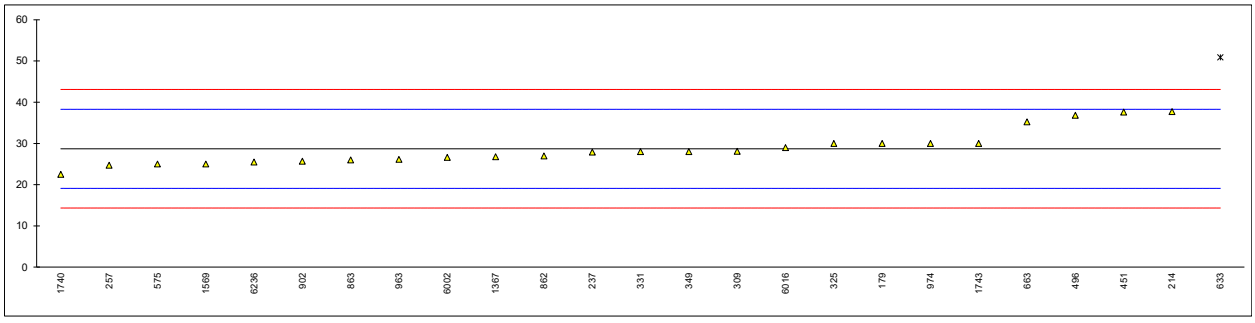


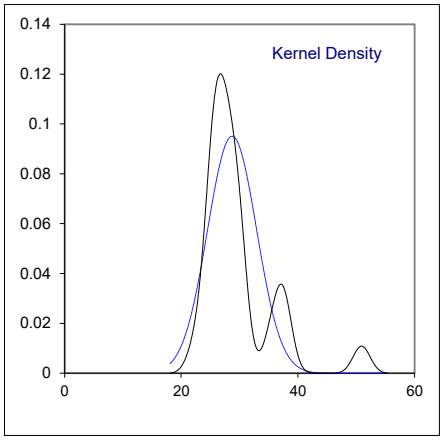


Determination of Boron as B on sample #21032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D5185	30		0.27	
214	D6595	37.74		1.88	
237	D5185	27.91		-0.17	
257	D6595	24.70		-0.84	
309	D5185	28.11		-0.13	
325	D5185	30		0.27	
331	D5185	28		-0.15	
349	D5185	28		-0.15	
451	D5185	37.6		1.85	
496	D5185	36.85		1.69	
575	D6595	25		-0.78	
633	D6595	50.9	R(0.01)	4.62	
663	D5185	35.23		1.36	
862	D5185	27		-0.36	
863	D5185	26		-0.57	
902	D5185	25.7		-0.63	
962		-----		-----	
963	D5185	26.13		-0.54	
974		30		0.27	
1146		-----		-----	
1367	D5185	26.78		-0.40	
1448		-----		-----	
1569	D5185	25		-0.78	
1740	D6595	22.5		-1.30	
1743		30		0.27	
6002	D5185	26.6		-0.44	
6016	D5185	29		0.06	
6236	D5185	25.5		-0.67	

normality	suspect
n	24
outliers	1
mean (n)	28.723
st.dev. (n)	4.1949
R(calc.)	11.746
st.dev.(D5185:18)	4.8014
R(D5185:18)	13.444

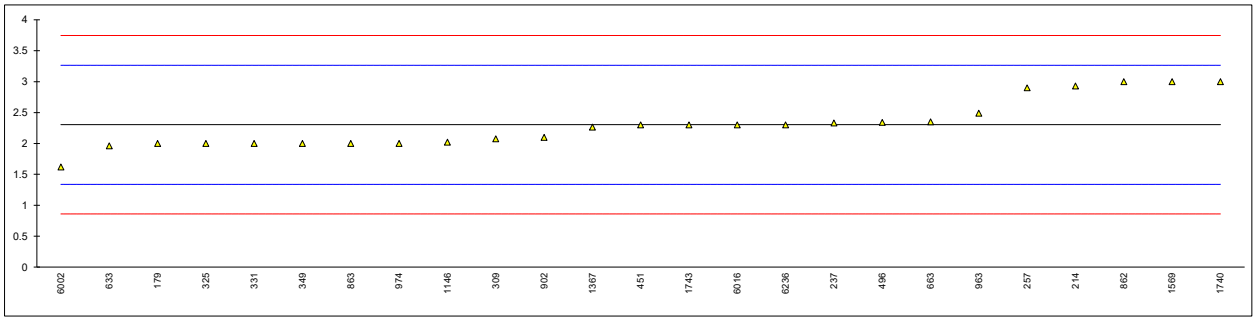


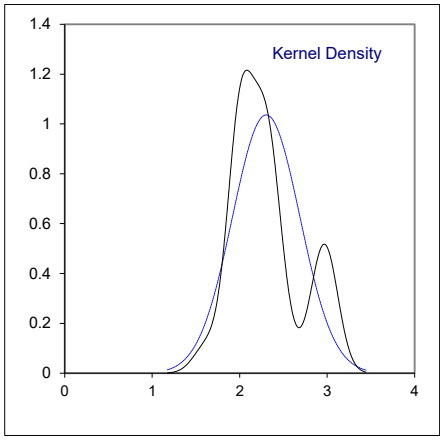


Determination of Chromium as Cr on sample #21032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D5185	2		-0.63	
214	D6595	2.93		1.30	
237	D5185	2.332		0.06	
257	D6595	2.90		1.24	
309	D5185	2.073		-0.48	
325	D5185	2		-0.63	
331	D5185	2		-0.63	
349	D5185	2		-0.63	
451	D5185	2.3		-0.01	
496	D5185	2.34		0.08	
575		-----		-----	
633	D6595	1.96		-0.71	
663	D5185	2.35		0.10	
862	D5185	3		1.45	
863	D5185	2		-0.63	
902	D5185	2.1		-0.42	
962		-----		-----	
963	D5185	2.49		0.39	
974		2		-0.63	
1146	D5185	2.02		-0.59	
1367	D5185	2.262		-0.09	
1448		-----		-----	
1569	D5185	3		1.45	
1740	D6595	3		1.45	
1743		2.3		-0.01	
6002	D5185	1.62		-1.42	
6016	D5185	2.3		-0.01	
6236	D5185	2.3		-0.01	

normality	OK
n	25
outliers	0
mean (n)	2.303
st.dev. (n)	0.3850
R(calc.)	1.078
st.dev.(D5185:18)	0.4812
R(D5185:18)	1.347

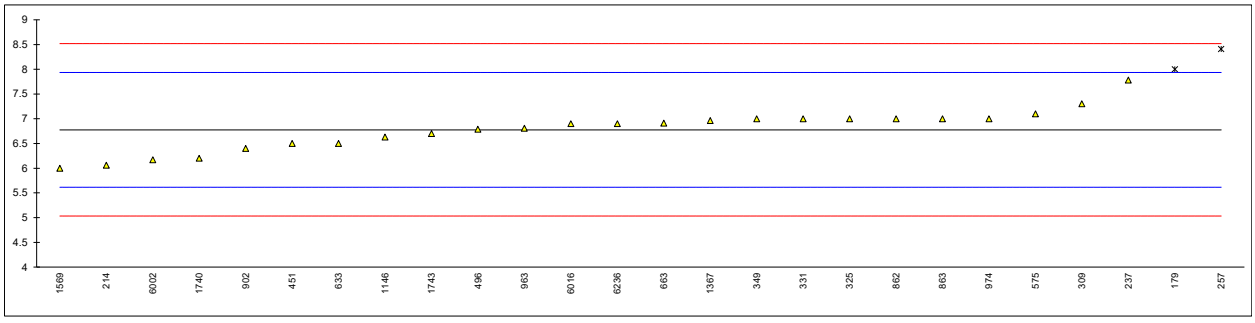


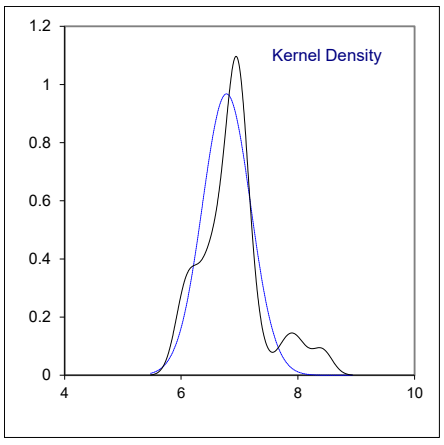


Determination of Copper as Cu on sample #21032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D5185	8	DG(0.05)	2.11	
214	D6595	6.06		-1.23	
237	D5185	7.781		1.73	
257	D6595	8.41	DG(0.05)	2.81	
309	D5185	7.302		0.91	
325	D5185	7		0.39	
331	D5185	7		0.39	
349	D5185	7		0.39	
451	D5185	6.5		-0.47	
496	D5185	6.79		0.02	
575	D6595	7.1		0.56	
633	D6595	6.50		-0.47	
663	D5185	6.91		0.23	
862	D5185	7		0.39	
863	D5185	7		0.39	
902	D5185	6.4		-0.65	
962		-----		-----	
963	D5185	6.81		0.06	
974		7		0.39	
1146	D5185	6.631		-0.25	
1367	D5185	6.961		0.32	
1448		-----		-----	
1569	D5185	6		-1.34	
1740	D6595	6.2		-0.99	
1743		6.7		-0.13	
6002	D5185	6.17		-1.04	
6016	D5185	6.9		0.21	
6236	D5185	6.9		0.21	

normality	OK
n	24
outliers	2
mean (n)	6.776
st.dev. (n)	0.4124
R(calc.)	1.155
st.dev.(D5185:18)	0.5808
R(D5185:18)	1.626

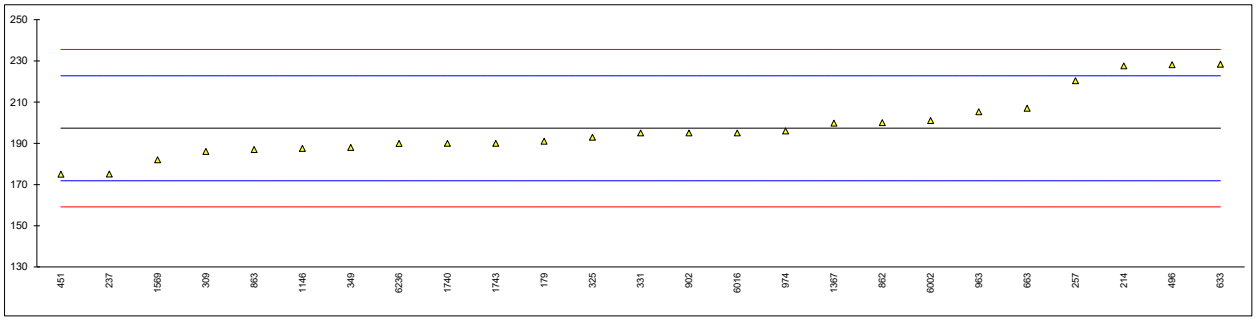


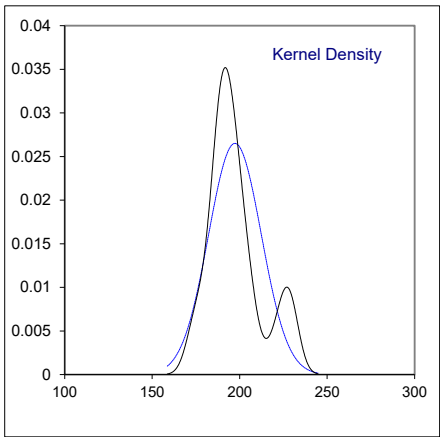


Determination of Iron as Fe on sample #21032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D5185	191		-0.50	
214	D6595	227.57		2.37	
237	D5185	175.1		-1.75	
257	D6595	220.4		1.81	
309	D5185	186.0		-0.89	
325	D5185	193		-0.34	
331	D5185	195		-0.18	
349	D5185	188		-0.73	
451	D5185	175		-1.75	
496	D5185	228.2		2.42	
575		-----		-----	
633	D6595	228.4		2.44	
663	D5185	207		0.76	
862	D5185	200		0.21	
863	D5185	187		-0.81	
902	D5185	195		-0.18	
962		-----		-----	
963	D5185	205.35		0.63	
974		196		-0.10	
1146	D5185	187.5		-0.77	
1367	D5185	199.8		0.19	
1448		-----		-----	
1569	D5185	182		-1.20	
1740	D6595	190		-0.58	
1743		190		-0.58	
6002	D5185	201.0		0.29	
6016	D5185	195		-0.18	
6236	D5185	189.9		-0.58	

normality	OK
n	25
outliers	0
mean (n)	197.329
st.dev. (n)	15.0494
R(calc.)	42.138
st.dev.(D5185:18)	12.7350
R(D5185:18)	35.658

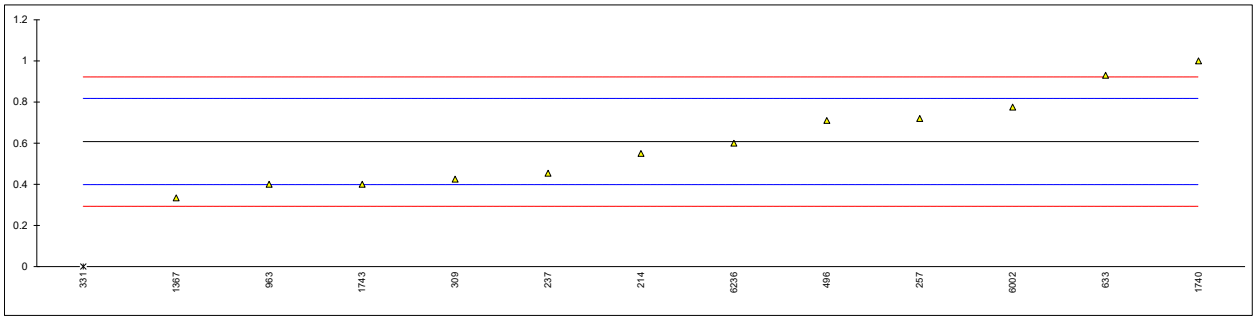


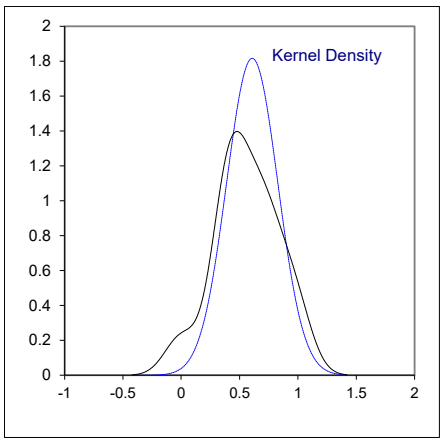


Determination of Lithium as Li on sample #21032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214	D6595	0.55		-0.55	
237	D5185	0.4534		-1.48	
257	D6595	0.72		1.07	
309	D5185	0.425		-1.75	
325		-----		-----	
331	D5185	0	ex	-5.80	test result excluded as zero is not a real test value
349		-----		-----	
451	D5185	<1		-----	
496	D5185	0.71		0.97	
575		-----		-----	
633	D6595	0.93		3.07	
663		-----		-----	
862	D5185	<1		-----	
863		-----		-----	
902		-----		-----	
962		-----		-----	
963	D5185	0.40		-1.98	
974		<1		-----	
1146	D5185	<1		-----	
1367	D5185	0.334		-2.61	
1448		-----		-----	
1569		-----		-----	
1740	D6595	1		3.74	
1743		0.4		-1.98	
6002	D5185	0.775		1.59	
6016		-----		-----	
6236	D5185	0.6		-0.08	

normality	OK
n	12
outliers	0 +1ex
mean (n)	0.608
st.dev. (n)	0.2196
R(calc.)	0.615
st.dev.(Horwitz)	0.1049
R(Horwitz)	0.294

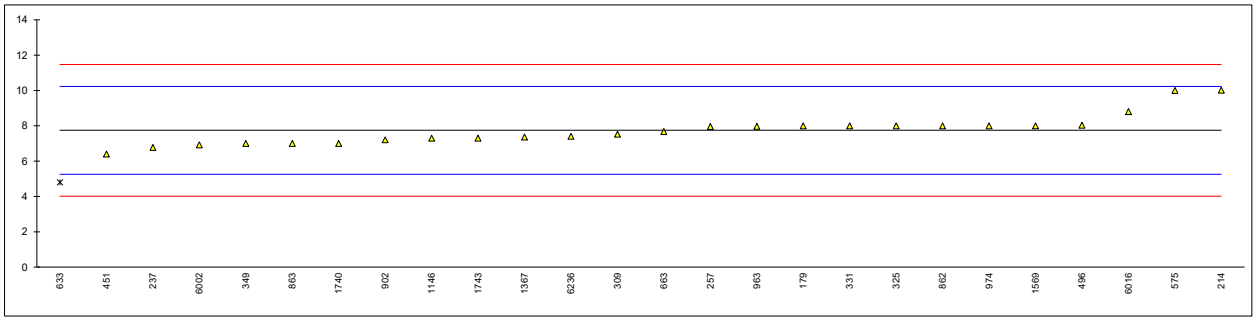


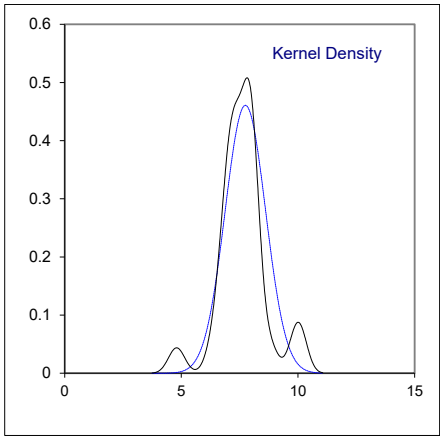


Determination of Magnesium as Mg on sample #21032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D5185	8		0.21	
214	D6595	10.01		1.82	
237	D5185	6.774		-0.78	
257	D6595	7.96		0.17	
309	D5185	7.522		-0.18	
325	D5185	8		0.21	
331	D5185	8		0.21	
349	D5185	7		-0.60	
451	D5185	6.4		-1.08	
496	D5185	8.030		0.23	
575	D6595	10		1.81	
633	D6595	4.80	D(0.05)	-2.37	
663	D5185	7.67		-0.06	
862	D5185	8		0.21	
863	D5185	7		-0.60	
902	D5185	7.2		-0.44	
962		-----		-----	
963	D5185	7.97		0.18	
974		8		0.21	
1146	D5185	7.30		-0.36	
1367	D5185	7.366		-0.30	
1448		-----		-----	
1569	D5185	8		0.21	
1740	D6595	7		-0.60	
1743		7.30		-0.36	
6002	D5185	6.92		-0.66	
6016	D5185	8.8		0.85	
6236	D5185	7.4		-0.28	

normality	not OK
n	25
outliers	1
mean (n)	7.745
st.dev. (n)	0.8661
R(calc.)	2.425
st.dev.(D5185:18)	1.2437
R(D5185:18)	3.482

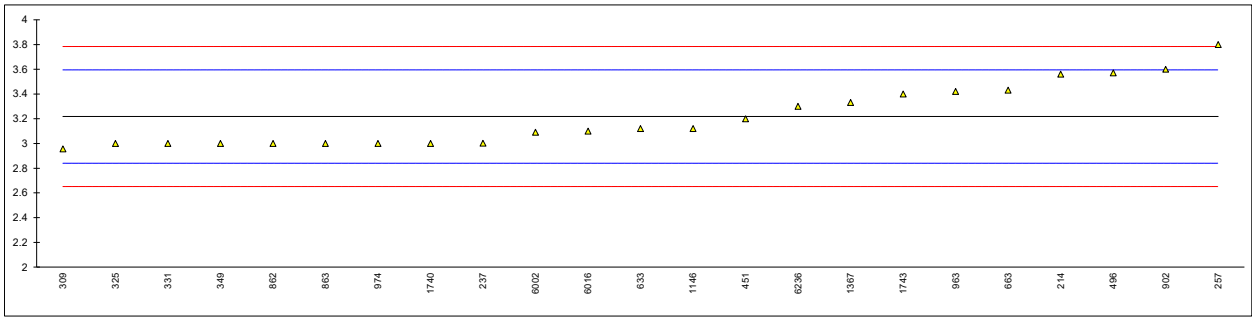


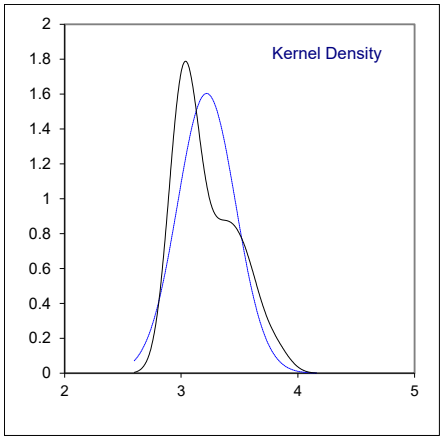


Determination of Manganese as Mn on sample #21032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214	D6595	3.56		1.82	
237	D5185	3.002		-1.14	
257	D6595	3.80		3.09	
309	D5185	2.956		-1.39	
325	D5185	3		-1.15	
331	D5185	3		-1.15	
349	D5185	3		-1.15	
451	D5185	3.2		-0.09	
496	D5185	3.57		1.87	
575		-----		-----	
633	D6595	3.12		-0.52	
663	D5185	3.431		1.13	
862	D5185	3		-1.15	
863	D5185	3		-1.15	
902	D5185	3.6		2.03	
962		-----		-----	
963	D5185	3.42		1.07	
974		3		-1.15	
1146	D5185	3.12		-0.52	
1367	D5185	3.332		0.61	
1448		-----		-----	
1569		-----		-----	
1740	D6595	3		-1.15	
1743		3.4		0.97	
6002	D5185	3.09		-0.68	
6016	D5185	3.1		-0.62	
6236	D5185	3.3		0.44	

normality	OK
n	23
outliers	0
mean (n)	3.217
st.dev. (n)	0.2488
R(calc.)	0.697
st.dev.(D5185:18)	0.1887
R(D5185:18)	0.528

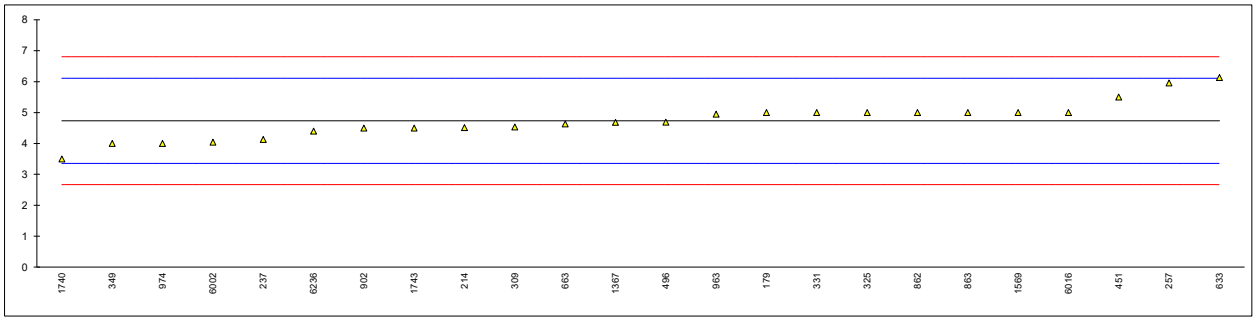


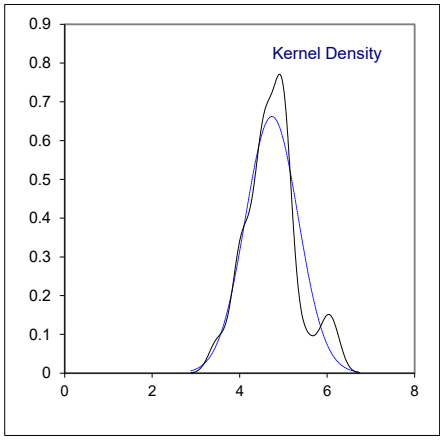


Determination of Molybdenum as Mo on sample #21032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D5185	5		0.38	
214	D6595	4.51		-0.33	
237	D5185	4.132		-0.88	
257	D6595	5.96		1.78	
309	D5185	4.535		-0.29	
325	D5185	5		0.38	
331	D5185	5		0.38	
349	D5185	4		-1.07	
451	D5185	5.5		1.11	
496	D5185	4.69		-0.07	
575		-----		-----	
633	D6595	6.13		2.02	
663	D5185	4.63		-0.15	
862	D5185	5		0.38	
863	D5185	5		0.38	
902	D5185	4.5		-0.34	
962		-----		-----	
963	D5185	4.95		0.31	
974		4		-1.07	
1146		-----		-----	
1367	D5185	4.682		-0.08	
1448		-----		-----	
1569	D5185	5		0.38	
1740	D6595	3.5		-1.79	
1743		4.5		-0.34	
6002	D5185	4.04		-1.01	
6016	D5185	5		0.38	
6236	D5185	4.4		-0.49	

normality	OK
n	24
outliers	0
mean (n)	4.736
st.dev. (n)	0.6026
R(calc.)	1.687
st.dev.(D5185:18)	0.6895
R(D5185:18)	1.931

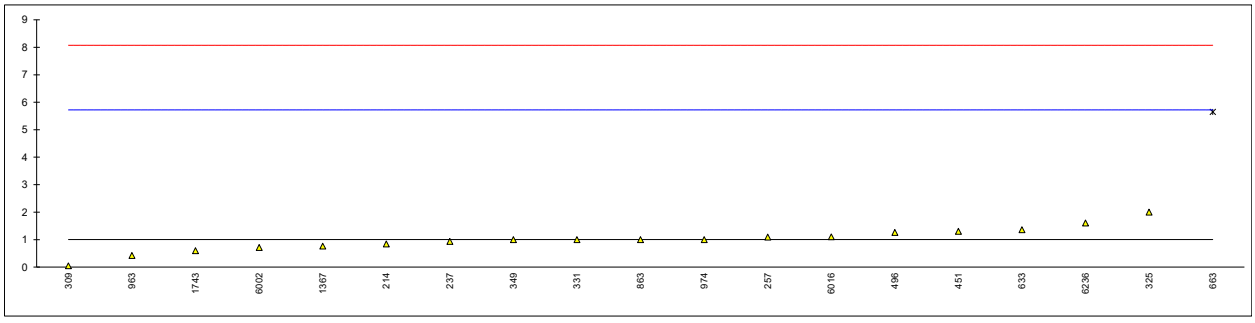


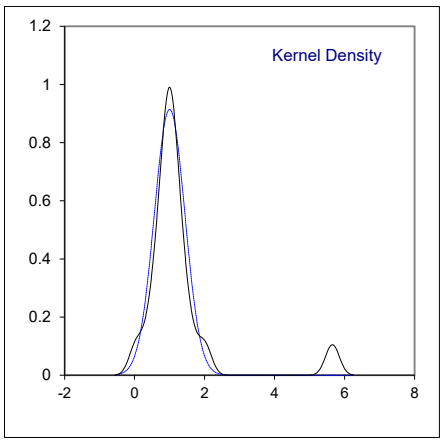


Determination of Potassium as K on sample #21032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214	D6595	0.84		-0.07	
237	D5185	0.9357		-0.03	
257	D6595	1.09		0.04	
309	D5185	0.048		-0.40	
325	D5185	2		0.42	
331	D5185	1		0.00	
349	D5185	1		0.00	
451	D5185	1.3		0.13	
496	D5185	1.26		0.11	
575		-----		-----	
633	D6595	1.36		0.15	
663	D5185	5.65	G(0.01)	1.97	
862	D5185	<1		-----	
863	D5185	1		0.00	
902	D5185	<40		-----	
962		-----		-----	
963	D5185	0.42		-0.25	
974		1		0.00	
1146		-----		-----	
1367	D5185	0.764		-0.10	
1448		-----		-----	
1569		-----		-----	
1740	D6595	<1		-----	
1743		0.6		-0.17	
6002	D5185	0.712		-0.12	
6016	D5185	1.1		0.04	
6236	D5185	1.6		0.25	

normality	suspect
n	18
outliers	1
mean (n)	1.002
st.dev. (n)	0.4363
R(calc.)	1.222
st.dev.(D5185:18)	2.3583
R(D5185:18)	6.603

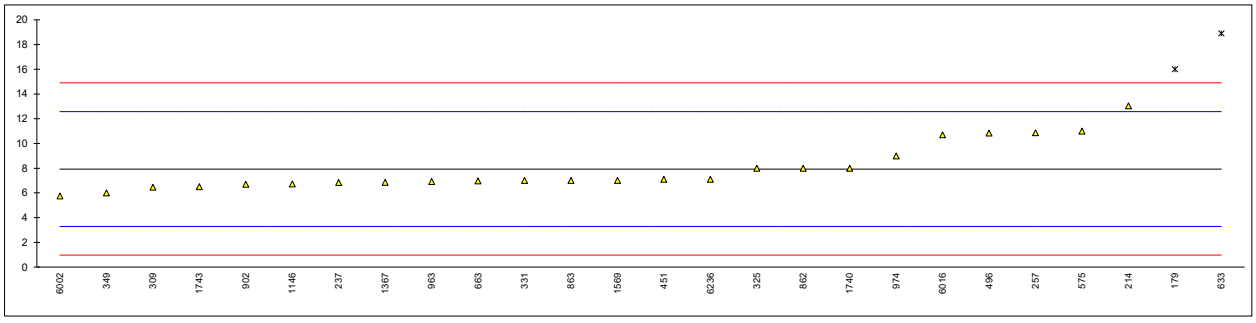


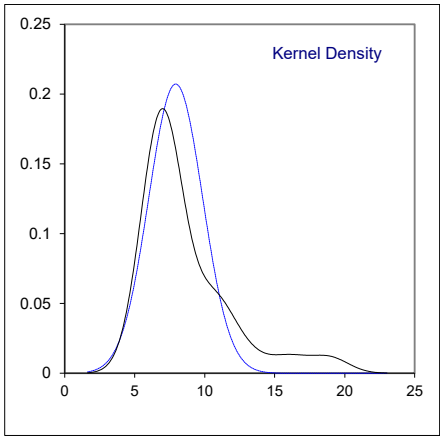


Determination of Silicon as Si on sample #21032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D5185	16	R(0.05)	3.47	
214	D6595	13.04	C	2.20	first reported 14.75
237	D5185	6.850		-0.47	
257	D6595	10.855		1.26	
309	D5185	6.459		-0.63	
325	D5185	8		0.03	
331	D5185	7		-0.40	
349	D5185	6		-0.83	
451	D5185	7.1		-0.36	
496	D5185	10.84		1.25	
575	D6595	11		1.32	
633	D6595	18.9	R(0.01)	4.72	
663	D5185	6.97		-0.41	
862	D5185	8		0.03	
863	D5185	7		-0.40	
902	D5185	6.7		-0.53	
962		-----		-----	
963	D5185	6.91		-0.44	
974		9		0.46	
1146	D5185	6.718		-0.52	
1367	D5185	6.861		-0.46	
1448		-----		-----	
1569	D5185	7		-0.40	
1740	D6595	8		0.03	
1743		6.5		-0.62	
6002	D5185	5.75		-0.94	
6016	D5185	10.7		1.19	
6236	D5185	7.1		-0.36	

normality	suspect
n	24
outliers	2
mean (n)	7.931
st.dev. (n)	1.9259
R(calc.)	5.392
st.dev.(D5185:18)	2.3227
R(D5185:18)	6.503

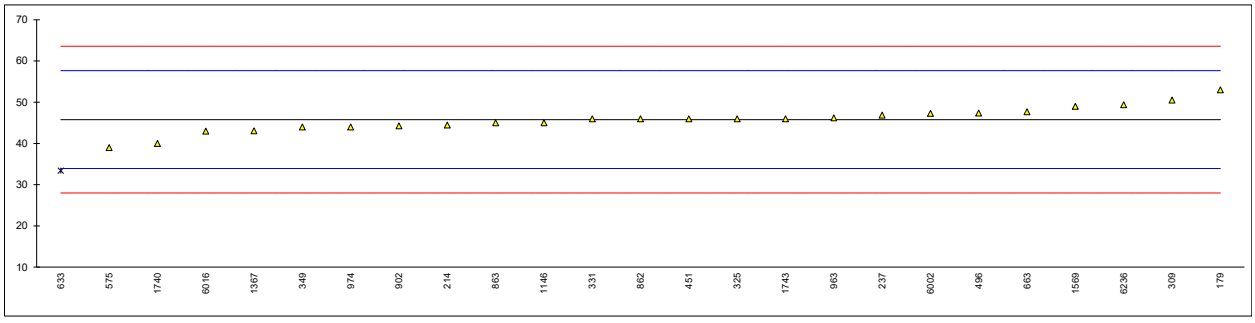


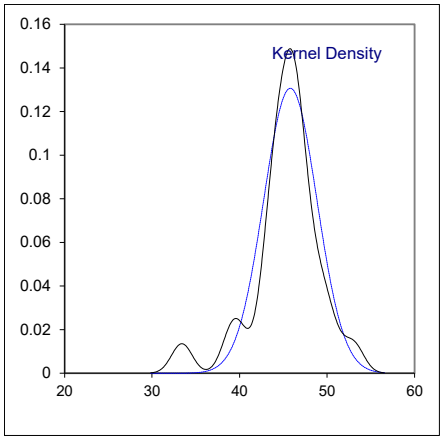


Determination of Sodium as Na on sample #21032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D5185	53		1.21	
214	D6595	44.45		-0.23	
237	D5185	46.86		0.18	
257		-----		-----	
309	D5185	50.52		0.80	
325	D5185	46		0.03	
331	D5185	46		0.03	
349	D5185	44		-0.30	
451	D5185	46		0.03	
496	D5185	47.4		0.27	
575	D6595	39		-1.15	
633	D6595	33.4	R(0.05)	-2.09	
663	D5185	47.7		0.32	
862	D5185	46		0.03	
863	D5185	45		-0.13	
902	D5185	44.3		-0.25	
962		-----		-----	
963	D5185	46.18		0.06	
974		44		-0.30	
1146	D5185	45.00		-0.13	
1367	D5185	43.04		-0.46	
1448		-----		-----	
1569	D5185	49		0.54	
1740	D6595	40		-0.98	
1743		46		0.03	
6002	D5185	47.3		0.25	
6016	D5185	43		-0.47	
6236	D5185	49.4		0.61	

normality	suspect
n	24
outliers	1
mean (n)	45.798
st.dev. (n)	3.0530
R(calc.)	8.548
st.dev.(D5185:18)	5.9352
R(D5185:18)	16.619

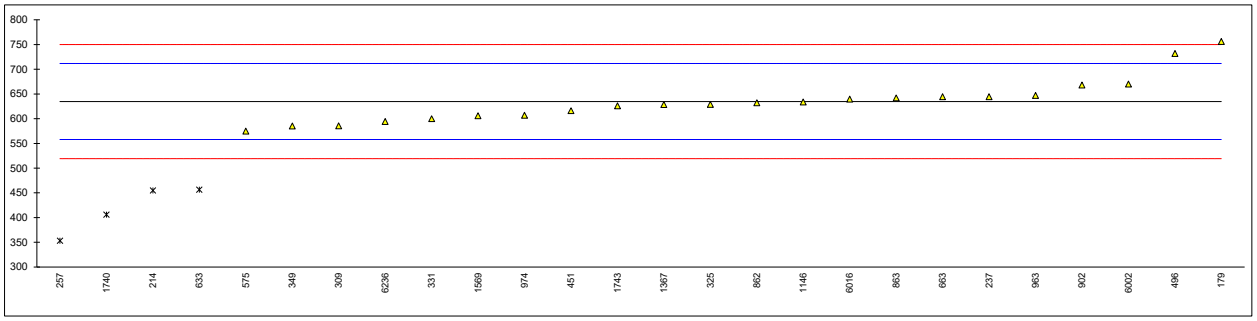


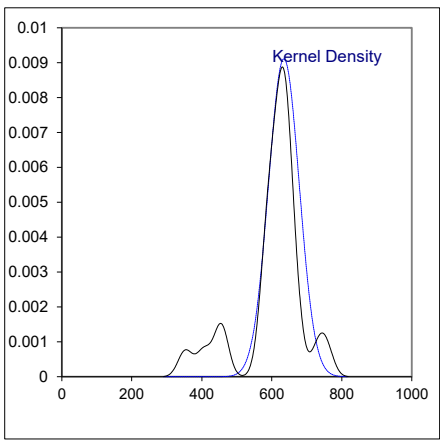


Determination of Calcium as Ca on sample #21032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D5185	756		3.16	
214	D6595	454.75	C,DG(0.01)	-4.68	first reported 6.12
237	D5185	644.4		0.25	
257	D6595	353.09	DG(0.01)	-7.32	
309	D5185	585.5		-1.28	
325	D5185	629		-0.15	
331	D5185	600		-0.90	
349	D5185	585		-1.29	
451	D5185	616		-0.48	
496	D5185	731.9		2.53	
575	D6595	575		-1.55	
633	D6595	456.5	DG(0.01)	-4.63	
663	D5185	644.4		0.25	
862	D5185	632		-0.07	
863	D5185	642		0.19	
902	D5185	668		0.87	
962		-----		-----	
963	D5185	647.06		0.32	
974		607		-0.72	
1146	D5185	633.78		-0.02	
1367	D5185	628.5		-0.16	
1448		-----		-----	
1569	D5185	606		-0.74	
1740	D6595	406	C,DG(0.01)	-5.95	first reported 436
1743		626		-0.22	
6002	D5185	670.0		0.92	
6016	D5185	639.8		0.13	
6236	D5185	594.2		-1.05	

	normality	not OK
	n	22
	outliers	4
	mean (n)	634.615
	st.dev. (n)	43.8239
	R(calc.)	122.707
	st.dev.(Horwitz)	38.4424
	R(Horwitz)	107.639
Compare:		
	R(D5185:18)	65.971

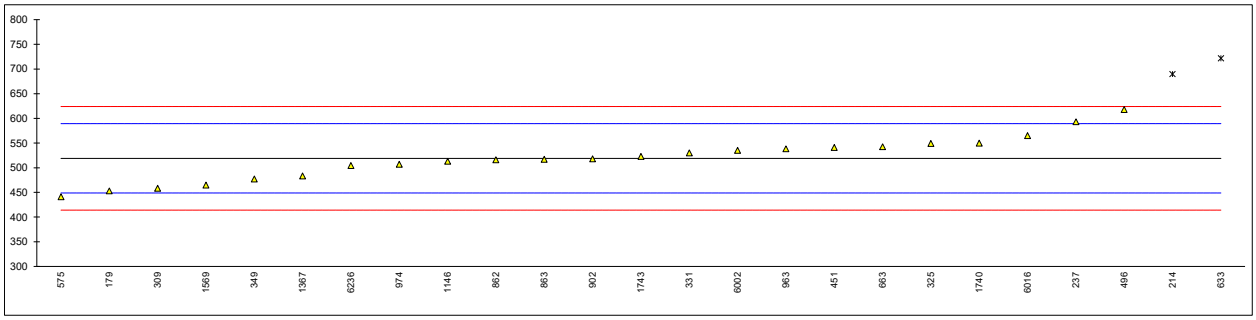


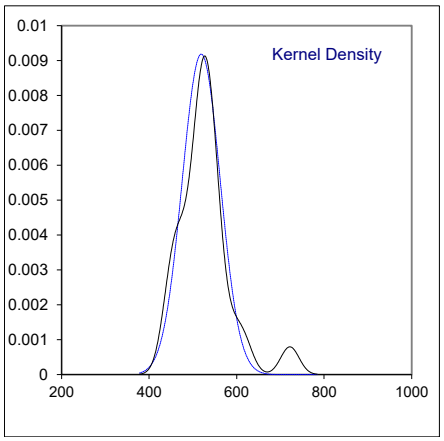


Determination of Phosphorus as P on sample #21032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D5185	453		-1.89	
214	D6595	689.82	R(0.05)	4.88	
237	D5185	593.1		2.12	
257		-----		-----	
309	D5185	458.4		-1.73	
325	D5185	549		0.86	
331	D5185	530		0.31	
349	D5185	477		-1.20	
451	D5185	541		0.63	
496	D5185	617.7		2.82	
575	D6595	441		-2.23	
633	D6595	721.7	R(0.05)	5.79	
663	D5185	542.5		0.67	
862	D5185	516		-0.09	
863	D5185	517		-0.06	
902	D5185	518		-0.03	
962		-----		-----	
963	D5185	538.28		0.55	
974		507		-0.34	
1146	D5185	513.01		-0.17	
1367	D5185	483.3		-1.02	
1448		-----		-----	
1569	D5185	465		-1.54	
1740	D6595	550		0.89	
1743		523		0.11	
6002	D5185	535		0.46	
6016	D5185	565		1.31	
6236	D5185	504.3		-0.42	

normality	OK
n	23
outliers	2
mean (n)	519.026
st.dev. (n)	43.4256
R(calc.)	121.592
st.dev.(D5185:18)	34.9868
R(D5185:18)	97.963

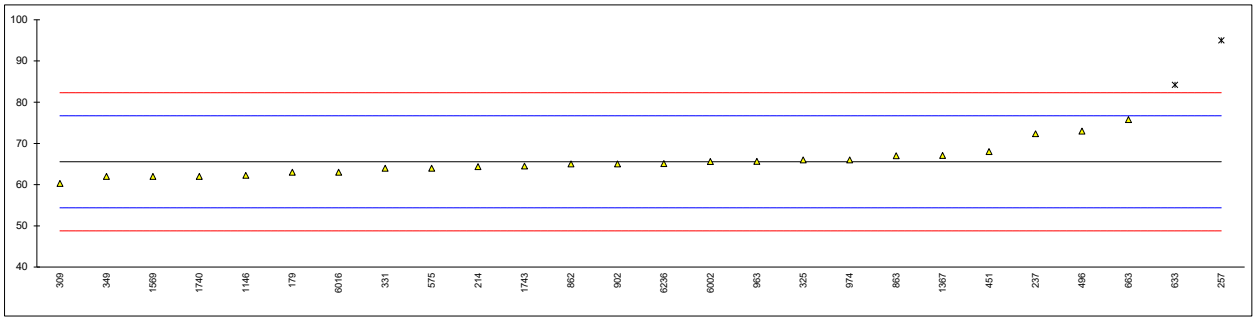


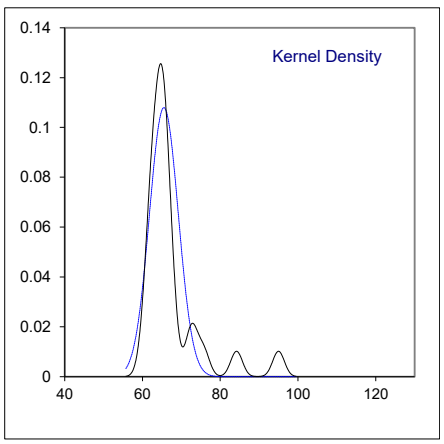


Determination of Zinc as Zn on sample #21032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D5185	63		-0.45	
214	D6595	64.35		-0.21	
237	D5185	72.35		1.22	
257	D6595	94.98	R(0.01)	5.27	
309	D5185	60.31		-0.94	
325	D5185	66		0.08	
331	D5185	64		-0.28	
349	D5185	62		-0.63	
451	D5185	68		0.44	
496	D5185	72.98		1.33	
575	D6595	64	C	-0.28	first reported 48
633	D6595	84.2	R(0.01)	3.34	
663	D5185	75.77		1.83	
862	D5185	65		-0.10	
863	D5185	67		0.26	
902	D5185	65		-0.10	
962		-----		-----	
963	D5185	65.67		0.02	
974		66		0.08	
1146	D5185	62.25		-0.59	
1367	D5185	67.09		0.28	
1448		-----		-----	
1569	D5185	62		-0.63	
1740	D6595	62		-0.63	
1743		64.5		-0.19	
6002	D5185	65.6		0.01	
6016	D5185	63		-0.45	
6236	D5185	65.1		-0.08	

	normality	not OK
	n	24
	outliers	2
	mean (n)	65.540
	st.dev. (n)	3.6955
	R(calc.)	10.347
	st.dev.(Horwitz)	5.5875
	R(Horwitz)	15.645
Compare:		
	R(D5185:18)	8.265





APPENDIX 2

Reported test results of other elements on sample #21032; results in mg/kg

lab	Cadmium as Cd	Lead as Pb	Nickel as Ni	Silver as Ag	Tin as Sn	Titanium as Ti	Vanadium as V
178	-----	-----	-----	-----	-----	-----	-----
179	-----	1	<1	<0.10	<1	-----	<1
214	0.00	0.00	0.46	0.00	0.00	0.34	0.30
237	0.1658	0.4091	0.7088	<0.1	<0.1	0.1474	<0.1
257	0.38	0.00	0.14	0.00	0.55	0.24	0.88
309	0.013	0	0	0.087	0.073	0.126	0.020
325	-----	<1	<1	<1	<1	<1	<1
331	0	0	0	0	0	0	0
349	0	0	0	0	1	0	0
451	<1	<1	<1	<1	<1	<1	<1
496	0.28	0.94	0.29	<0.1	1.27	0.09	<0.1
575	-----	-----	-----	-----	-----	-----	-----
633	0	0	0.59	0.16	0.88	0	0.09
663	-----	<1	<1	<0.5	<1	<1	<1
862	<1	<1	<1	<1	<1	<1	<1
863	<1	<1	<1	<1	<1	<1	<1
902	-----	<10	<5	<0.5	<10	<5	<1
962	-----	-----	-----	-----	-----	-----	-----
963	<0.10	0.65	0.40	<0.10	0.45	0.11	<0.10
974	<1	<1	<1	<1	1	<1	<1
1146	-----	<2	<1	<1	<1	<1	<1
1367	0.079	1.35	0.175	0.102	0	0.131	0
1448	-----	-----	-----	-----	-----	-----	-----
1569	-----	<1	<1	<1	2	-----	<1
1740	-----	<1	<1	<1	<1	<1	<1
1743	0.0	0.4	0.3	0.0	0.0	0.1	0.0
6002	0.0	0.37	0.387	0.126	0.236	0.125	0.05
6016	-----	1.9	0.76	-----	7.3	<0.5	<1
6236	-----	0.4	0.4	0	0.7	0.1	0

APPENDIX 3 Analytical details of the Level of Contamination

lab	Sample rolled	Sample diluted	Dilution ratio	Manufacturer and the model of test equipment
178	---	---	---	
179	---	---	---	
214	Yes	No	---	PAMAS N500
237	---	---	---	
257	---	---	---	
309	---	---	---	
325	---	---	---	
331	---	---	---	
349	---	---	---	
451	No	No	---	Pamas
496	No	No	---	
575	Yes	No	---	PAMAS S40
633	No	Yes	10:90 Sample:Solvent	PAMAS S-40
663	Yes	Yes	1:1	PAMAS SBSS-C
862	Yes	No	---	Manufacturer: PAMAS ; Model: SBSS-C
863	Yes	Yes	---	Manufacturer PAMAS Model:SBSS
902	Yes	Yes	---	CINRG Full Autoating Dilution System Particle Counter
962	---	---	---	
963	---	---	---	
974	---	---	---	
1146	---	---	---	
1367	Yes	Yes	Dilution factor 3.0	PAMAS SBSS
1448	---	---	---	
1569	No	No	---	PAMAS SBSS
1740	No	Yes	1:2	manufacturer:PAMAS MODEL: SBSS
1743	---	---	---	
6002	Yes	Yes	1:1	CINGR CS-APC-2
6016	Yes	No	---	Particle shape classifier and particle counter SpectroLNF Q200, Model No.: Q230
6236	Yes	Yes	30% sample in solvent. Solvent: 25V% IPA/ 75V% Tolueen	CINRG. Laser LDS 4188

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
1 lab in	GERMANY
1 lab in	GREECE
1 lab in	KAZAKHSTAN
1 lab in	MALAYSIA
2 labs in	NETHERLANDS
1 lab in	NIGERIA
1 lab in	PHILIPPINES
1 lab in	POLAND
2 labs in	SAUDI ARABIA
2 labs in	SPAIN
1 lab in	TANZANIA
1 lab in	THAILAND
1 lab in	TURKEY
1 lab in	UNITED ARAB EMIRATES
2 labs in	UNITED KINGDOM
2 labs in	UNITED STATES OF AMERICA

APPENDIX 5

Abbreviations

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

Literature

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