Institute for
Interlaboratory Studies

Results of Proficiency Test
Gear Oil (used)
March 2023

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2017 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of used Gear Oil every year. During the annual proficiency testing program 2022/2023 it was decided to continue the round robin for the analysis of used Gear Oil.

In this interlaboratory study 24 laboratories in 18 countries registered for participation, see appendix 3 for the number of participants per country. In this report the results of the Gear Oil (used) 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 0.5 L bottle labelled #23031 for regular analyzes and one 50 mL PE bottle labelled #23032 for the analyzes of Metals 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 analyzes in used Gear Oil a batch of approximately 25 liters of used Gear Oil was obtained from a third party. After homogenization 40 amber glass bottles of 0.5 L were filled and labelled #23031.
The homogeneity of the subsamples was checked by determination of Density at 15 °C in accordance with ISO12185 and Water in accordance with D6304 procedure C on 8 stratified randomly selected subsamples.

	Density at 15 °C in kg/L	Water in mg/kg
sample #23031-1	0.85483	149
sample #23031-2	0.85483	149
sample #23031-3	0.85483	133
sample #23031-4	0.85483	136
sample #23031-5	0.85482	142
sample #23031-6	0.85483	143
sample #23031-7	0.85483	136
sample #23031-8	0.85483	136

Table 1: homogeneity test results of subsamples #23031

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibility of the reference test methods in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Density at 15 °C in kg/L	Water in mg/kg
r (observed)	0.00001	17
reference test method	ISO12185:96	ASTM D6304-B:20
0.3 x R (reference test method)	0.00015	53

Table 2: evaluation of the repeatabilities of subsamples #23031

The calculated repeatabilities are in agreement with 0.3 times the corresponding reproducibility of the reference test methods. Therefore, homogeneity of the subsamples was assumed.

For the preparation of the sample for the analyzes of Metals in used Gear Oil a batch of approximately 10 liters of Gear Oil (used) was obtained from a third party. After homogenization 40 PE bottles of 50 mL were filled and labelled #23032.
The homogeneity of the subsamples was checked by determination of Iron in accordance with ASTM D5185 on 8 stratified randomly selected subsamples.

	Iron as Fe in mg/kg
sample #23032-1	193.5
sample #23032-2	192.7
sample #23032-3	193.0
sample #23032-4	193.2
sample #23032-5	195.5
sample #23032-6	191.9
sample #23032-7	193.0
sample #23032-8	192.7

Table 3: homogeneity test results of subsamples #23032

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)	2.9
reference test method	ASTM D5185:18
0.3 x R (reference test method)	10.5

Table 4: evaluation of the repeatability of subsamples #23032

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 0.5 L bottle of used Gear Oil labelled #23031 and one 50 mL PE bottle of used Gear Oil labelled #23032 were sent on February 15, 2023. 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 PE 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 #23031: Total Acid Number, Density at 15 °C, Flash Point PMcc, Kinematic Viscosity at 40 °C and 100 °C, Viscosity Index, Membrane Filtration 5.0 µm and Water.
Some extra information was asked about the determination of Total Acid Number.

On sample #23032 it was requested to determine: 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 and 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 appendices 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 appendices 1 and 2. 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 (derived from e.g. ISO or ASTM test methods), 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 some problems were encountered with the dispatch of the samples. Two participants reported test results after the final reporting date and three other participants did not report any test results. Not all participants were able to report all tests requested.
In total 21 participants reported 366 numerical test results. Observed were 17 outlying test results, which is 4.6%. 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 in appendix 1. The abbreviations, used in these tables, are explained in appendix 4.

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). When a method has been reapproved an “R” will be added and the year of approval (e.g. D2270:10R16).

sample #23031

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

Density at 15 °C: 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 ISO12185:96 and ASTM D4052:22.

Flash Point PMcc: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D93:20 procedure A nor with procedure 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 reported to use procedure A.

Kinematic Viscosity at 40 °C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D445:21e2.

Kinematic Viscosity at 100 °C: 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 D445:21e2.

Viscosity Index: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D2270:10R16.

Membrane Filtration 5.0 µm: Only two participants reported a test result. Therefore, no z-scores are calculated.

Water: This determination may be problematic depending on the procedure used. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D6304:20 procedure B, but not in agreement with procedure A and C.
A new version of ASTM D6304 was published in 2020 with major changes. In the 2016 version one precision statement was mentioned for test results based on mass with a broad application range and one based on volume. In the 2020 version all precision statements are based on mass with three different procedures (A - direct injection, B - oven accessory and C - evaporation accessory) each with a different application range. In ASTM D6304:20 the reproducibility for all three procedures is much stricter compared to ASTM D6304:16e1. It was decided to use procedure B for the 2022 PTs of Gear Oil (fresh) and Gear Oil (used).

sample #23032

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.

Boron as B: 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.

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. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5185:18.

Iron as Fe: 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.

Magnesium as Mg: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility 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. 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. Three 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 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.

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

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

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 are calculated for these elements. The test results of these elements 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 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 reference methods are presented in the next tables.

Parameter	unit	n	average	2.8 * sd	R(lit)
Total Acid Number	mg KOH/g	18	0.57	0.21	0.32
Density at 15 °C	kg/L	15	0.8549	0.0003	0.0005
Flash Point PMcc	°C	16	195.0	15.8	13.8
Kinematic Viscosity at 40 °C	mm^2/s	17	48.42	0.45	0.47
Kinematic Viscosity at 100 °C	mm^2/s	14	8.47	0.07	0.08
Viscosity Index		12	152.0	1.5	2
Membrane Filtration 5.0 µm	%M/M	2	n.e.	n.e.	n.e.
Water	mg/kg	18	156	184	185

Table 5: reproducibilities of tests on sample #23031

Parameter	unit	n	average	2.8 * sd	R(lit)
Aluminum as Al	mg/kg	21	12.6	4.0	7.3
Boron as B	mg/kg	17	27.4	11.2	13.4
Chromium as Cr	mg/kg	18	2.3	1.0	1.3
Copper as Cu	mg/kg	21	6.8	1.7	1.6
Iron as Fe	mg/kg	20	198	29	36
Magnesium as Mg	mg/kg	19	7.7	2.7	3.5
Manganese as Mn	mg/kg	16	3.3	1.3	0.5
Molybdenum as Mo	mg/kg	17	4.9	1.1	2.0
Silicon as Si	mg/kg	17	6.8	1.3	6.1
Sodium as Na	mg/kg	20	45.0	10.3	16.4
Calcium as Ca	mg/kg	16	628	80	65
Phosphorus as P	mg/kg	18	518	107	98
Zinc as Zn	mg/kg	18	65.7	10.4	8.3

Table 6: reproducibilities of tests on sample #23032

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 2023 WITH PREVIOUS PTS

	March 2023	March 2022	March 2021	March 2020	April 2019
Number of reporting laboratories	21	23	28	24	24
Number of test results	366	317	616	414	421
Number of statistical outliers	17	11	31	25	22
Percentage of statistical outliers	4.6%	3.5%	5.0%	6.0%	5.2%

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 2023	March 2022	March 2021	March 2020	April 2019
Total Acid Number	+	+/-	-	+/-	-
Density at 15 °C	+	++	+	-	-
Flash Point PMcc	-	+/-	+	+/-	-
Kinematic Viscosity at 40 °C	+/-	+	+/-	++	+
Kinematic Viscosity at 100 °C	+/-	+/-	-	-	-
Viscosity Index	+	+/-	+/-	-	+/-
Membrane Filtration 5.0 µm	n.e.	n.e.	+	++	++
Water	+/-	+	-	+	+

Parameter	March 2023	March 2022	March 2021	March 2020	April 2019
Aluminum as Al	+	+	++	+	+
Barium as Ba	n.e.	n.e.	-	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.e.	n.e.	--	n.a.	n.a.
Magnesium as Mg	+	n.e.	+	n.a.	n.a.
Manganese as Mn	--	n.e.	-	n.a.	n.a.
Molybdenum as Mo	+	n.e.	+	n.a.	n.a.
Potassium as K	n.e.	n.e.	++	n.a.	n.a.
Silicon as Si	++	++	+	++	++
Sodium as Na	+	n.e.	+	+	+
Tin as Sn	n.e	+	n.e.	+	+
Calcium as Ca	-	-	-	(--)	-
Phosphorus as P	+/-	+	-	+	-
Zinc as Zn	-	+/-	+	(--)	(--)

Table 8: comparison of determinations to the reference test methods

For results between brackets no z-scores are calculated

The following performance categories were used:

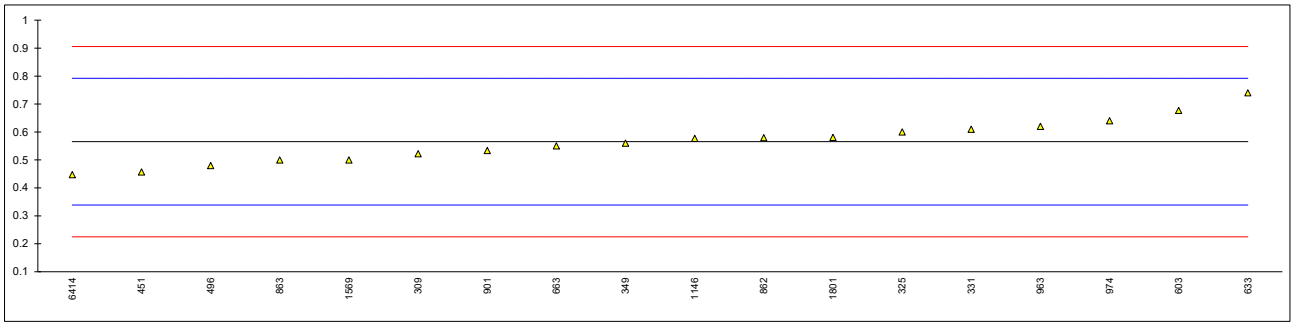
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test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

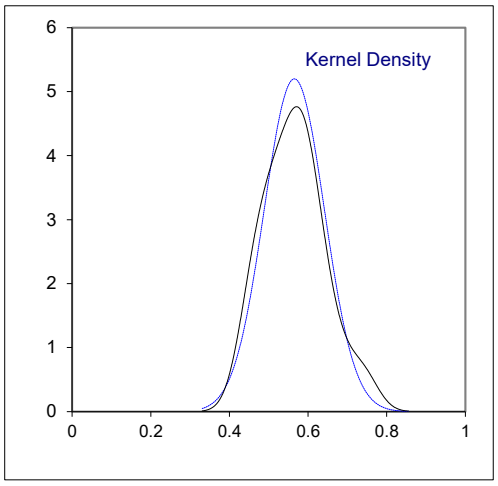
APPENDIX 1

Determination of Total Acid Number on sample #23031; results in mg KOH/g

lab	method	value	mark	z(targ)	end point type	volume (mL)	remarks
178		-----		-----	---	---	
179		-----		-----	---	---	
214		-----		-----	---	---	
237		-----		-----	---	---	
256		-----		-----	---	---	
257		-----		-----	---	---	
309	D664-A	0.522		-0.38	Buffer End Point pH 10	125 mL	
325	D664-A	0.60		0.31	Buffer End Point pH 10	125 mL	
331	D664-A	0.61		0.39	---	---	
349	D664-A	0.56		-0.05	---	---	
451	D664-A	0.457		-0.96	Buffer End Point pH 10	60 mL	
496	D664-A	0.48		-0.75	Buffer End Point pH 10	60 mL	
603	D664-A	0.6770		0.99	Inflection Point	125 mL	
633	D664-A	0.74		1.54	Inflection Point	125 mL	
663	D664-A	0.55		-0.13	---	---	
862	D664-A	0.58		0.13	Inflection Point	60 mL	
863	D664-A	0.50		-0.58	---	---	
901	D664-A	0.534		-0.28	Inflection Point	60 mL	
963	D664-B	0.62		0.48	Inflection Point	60 mL	
974	D664-A	0.64		0.66	Inflection Point	125 mL	
1146	D664-A	0.577		0.10	Buffer End Point pH 10	125 mL	
1569	D664-A	0.50		-0.58	Buffer End Point pH 11	125 mL	
1801	D664-A	0.581		0.14	Buffer End Point pH 11	125 mL	
6414	D664-A	0.447		-1.04	---	---	

normality	OK
n	18
outliers	0
mean (n)	0.5653
st.dev. (n)	0.07670
R(calc.)	0.2148
st.dev.(D664-A:18e2, BEP 60mL)	0.11344
R(D664-A:18e2, BEP 60mL)	0.3176
Compare	
R(D664-A:18e2, BEP 125 mL)	0.1735
R(D664-A:18e2, IP 60 mL)	0.2520
R(D664-A:18e2, IP 125 mL)	0.1207

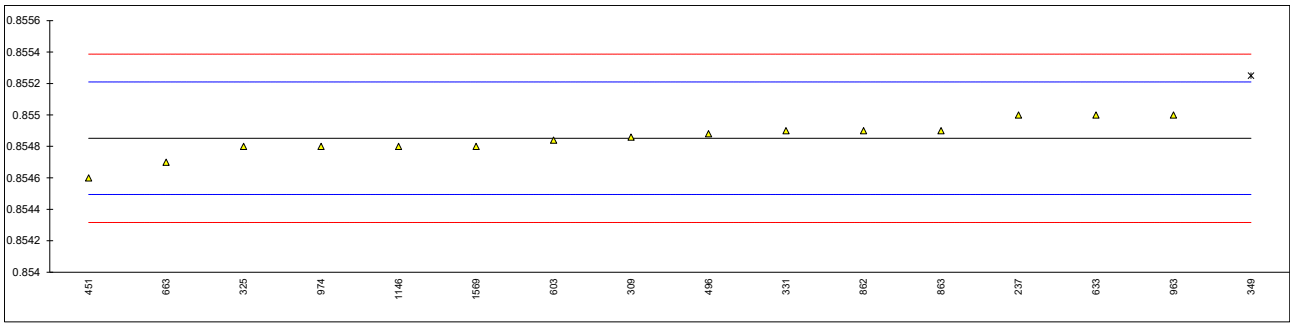


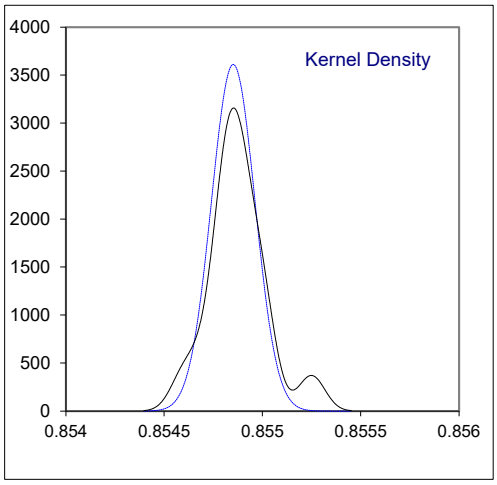


Determination of Density at 15 °C on sample #23031; results in kg/L

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214		-----		-----	
237	D4052	0.8550		0.83	
256		-----		-----	
257		-----		-----	
309	D4052	0.85486		0.05	
325	D4052	0.8548		-0.29	
331	ISO12185	0.8549		0.27	
349	D4052	0.85525	D(0.05)	2.23	
451	D4052	0.8546		-1.41	
496	ISO12185	0.85488		0.16	
603	D4052	0.85484		-0.07	
633	D4052	0.8550		0.83	
663	D4052	0.8547		-0.85	
862	D4052	0.8549		0.27	
863	ISO12185	0.8549		0.27	
901		-----		-----	
963	D4052	0.8550		0.83	
974	D4052	0.8548		-0.29	
1146	D4052	0.8548		-0.29	
1569	D4052	0.8548		-0.29	
1801		-----		-----	
6414		-----		-----	

normality	OK
n	15
outliers	1
mean (n)	0.854852
st.dev. (n)	0.0001105
R(calc.)	0.000309
st.dev.(ISO12185:96)	0.0001786
R(ISO12185:96)	0.0005
compare	
R(D4052:22)	0.00050

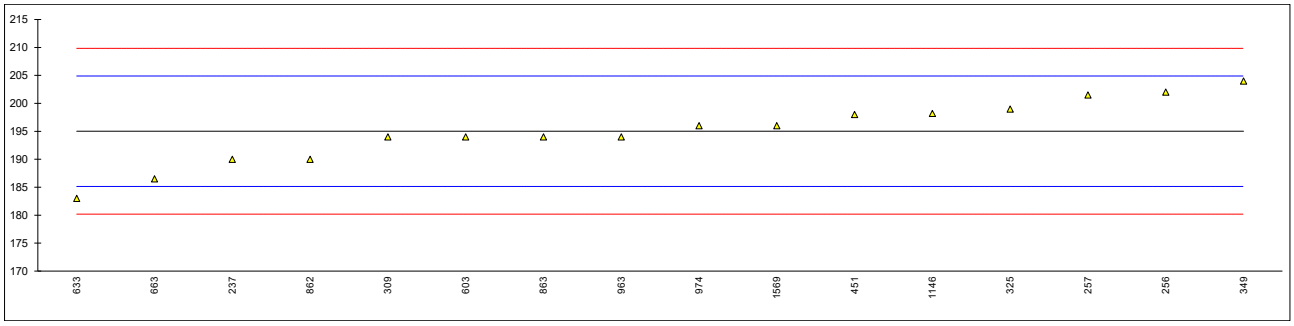


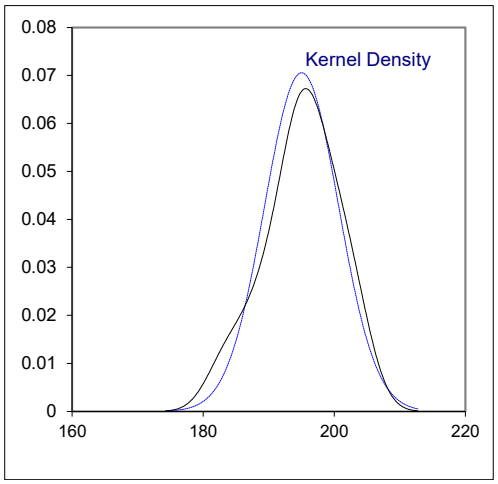


Determination of Flash Point PMcc on sample #23031; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214		-----		-----	
237	D93-B	190.0		-1.01	
256	D3828	202.0		1.41	
257	D3828	201.5		1.31	
309	D93-A	194.0		-0.20	
325	D93-A	199.0		0.81	
331		-----		-----	
349	D93-A	204		1.82	
451	D93-A	198.0		0.60	
496		-----		-----	
603	D93-A	194		-0.20	
633	D93-B	183.0	C	-2.43	first reported 181.50
663	D93-B	186.5		-1.72	
862	D93-A	190		-1.01	
863	D93-A	194.0		-0.20	
901		-----		-----	
963	D93-A	194.0		-0.20	
974	D93-A	196		0.20	
1146	D93-A	198.2		0.64	
1569	D93-A	196		0.20	
1801		-----		-----	
6414		-----		-----	

normality	OK
n	16
outliers	0
mean (n)	195.01
st.dev. (n)	5.655
R(calc.)	15.83
st.dev.(D93-A:20)	4.945
R(D93-A:20)	13.85
compare	
R(D93-B:20)	10

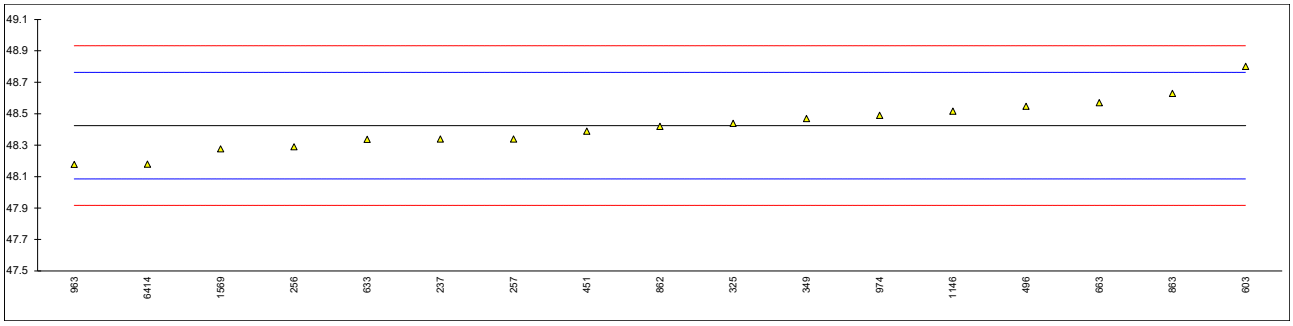


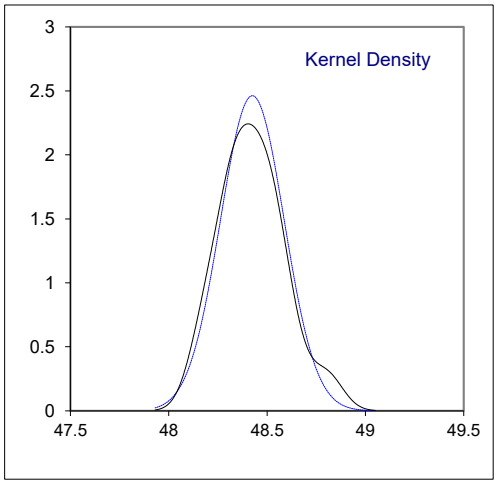


Determination of Kinematic Viscosity at 40 °C on sample #23031; results in mm²/s

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214		-----		-----	
237	D445	48.34		-0.50	
256	D7279 corrected to D445	48.29		-0.80	
257	D7279 corrected to D445	48.34		-0.50	
309		-----		-----	
325	D445	48.44		0.09	
331		-----		-----	
349	D445	48.47		0.27	
451	D7279 corrected to D445	48.39		-0.20	
496	D445	48.548		0.73	
603	D7042	48.802		2.23	
633	D445	48.337		-0.52	
663	D445	48.57	C	0.86	first reported 48.90
862	D445	48.42		-0.03	
863	D445	48.63		1.21	
901		-----		-----	
963	D445	48.178		-1.46	
974	D445	48.49		0.39	
1146	D445	48.517		0.55	
1569	D445	48.277		-0.87	
1801		-----		-----	
6414	D7279 corrected to D445	48.18		-1.45	

normality	OK
n	17
outliers	0
mean (n)	48.4246
st.dev. (n)	0.16199
R(calc.)	0.4536
st.dev.(D445:21e2)	0.16917
R(D445:21e2)	0.4737

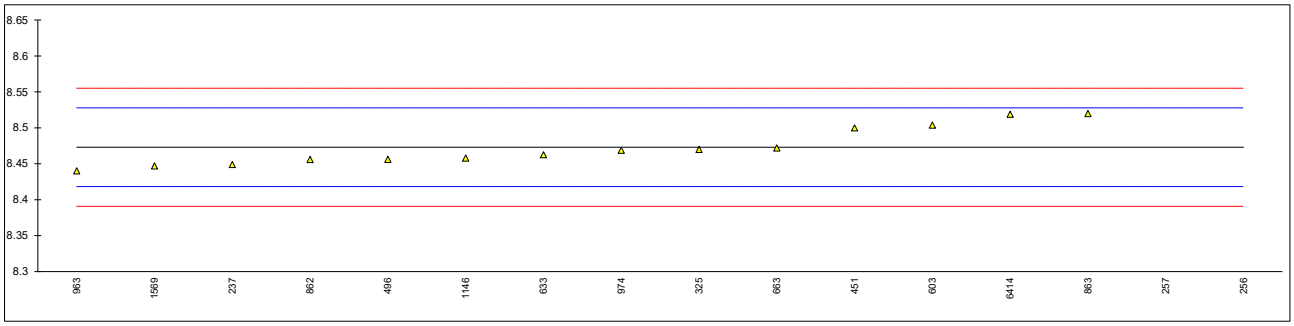


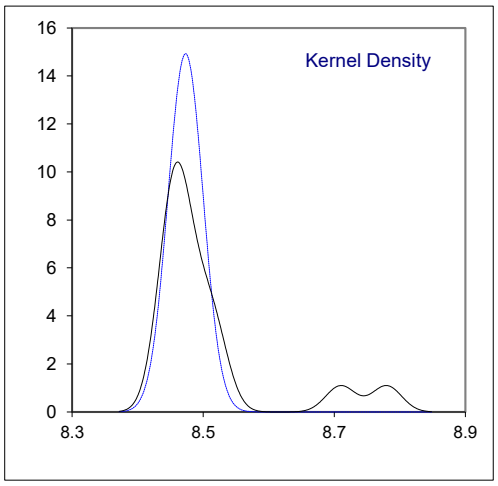


Determination of Kinematic Viscosity at 100 °C on sample #23031; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214		-----		-----	
237	D445	8.449		-0.88	
256	D7279 corrected to D445	8.780	G(0.05)	11.21	
257	D7279 corrected to D445	8.710	G(0.01)	8.66	
309		-----		-----	
325	D445	8.470		-0.11	
331		-----		-----	
349		-----		-----	
451	D7279 corrected to D445	8.50		0.99	
496	D445	8.4561		-0.62	
603	D7042	8.5039		1.13	
633	D445	8.4625		-0.38	
663	D445	8.472		-0.04	
862	D445	8.456		-0.62	
863	D445	8.520		1.72	
901		-----		-----	
963	D445	8.440		-1.21	
974	D445	8.469		-0.15	
1146	D445	8.458		-0.55	
1569	D445	8.447		-0.95	
1801		-----		-----	
6414	D7279 corrected to D445	8.519		1.68	

normality	OK
n	14
outliers	2
mean (n)	8.4730
st.dev. (n)	0.02673
R(calc.)	0.0748
st.dev.(D445:21e2)	0.02737
R(D445:21e2)	0.0766

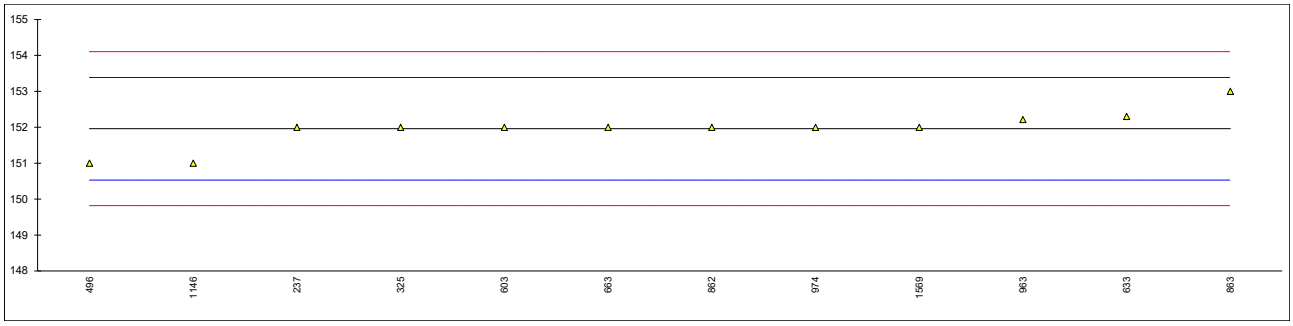


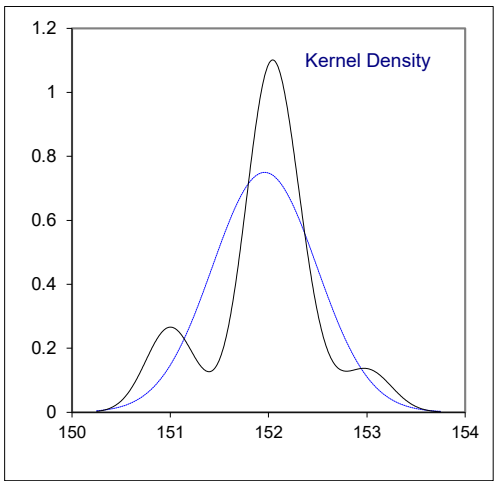


Determination of Viscosity Index on sample #23031

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214		-----		-----	
237	D2270	152		0.06	
256		-----		-----	
257		-----		-----	
309		-----		-----	
325	D2270	152		0.06	
331		-----		-----	
349		-----		-----	
451		-----		-----	
496	D2270	151		-1.34	
603	D2270	152		0.06	
633	D2270	152.3		0.48	
663	D2270	152	C	0.06	first reported 150
862	D2270	152		0.06	
863		153		1.46	
901		-----		-----	
963	D2270	152.213		0.36	
974	D2270	152		0.06	
1146	D2270	151		-1.34	
1569	D2270	152		0.06	
1801		-----		-----	
6414		-----		-----	

normality	suspect
n	12
outliers	0
mean (n)	151.96
st.dev. (n)	0.532
R(calc.)	1.49
st.dev.(D2270:10R16)	0.714
R(D2270:10R16)	2





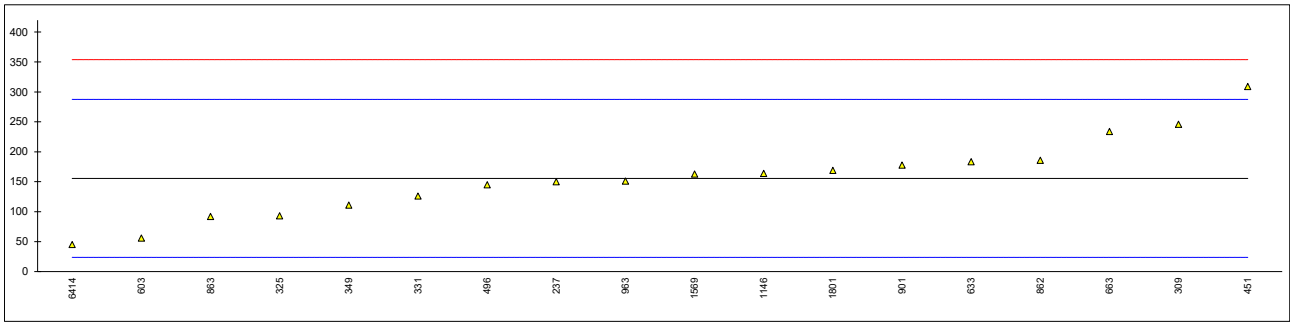
Determination of Membrane Filtration 5.0 µm on sample #23031; results in %M/M

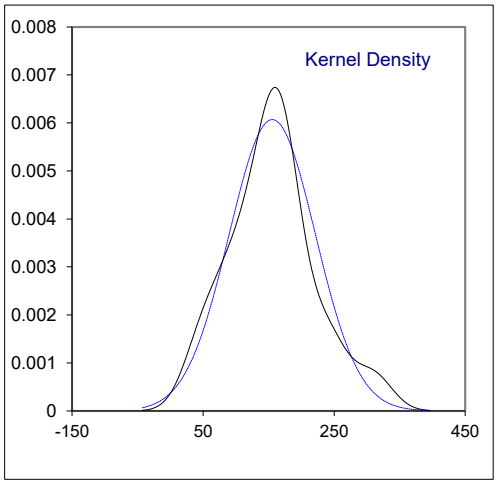
lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214		-----		-----	
237		-----		-----	
256		-----		-----	
257		-----		-----	
309		-----		-----	
325	D4055	0.0170		-----	
331		-----		-----	
349		-----		-----	
451		-----		-----	
496		-----		-----	
603		-----		-----	
633		-----		-----	
663		-----		-----	
862		-----		-----	
863	D4055	<0.01		-----	
901		-----		-----	
963		-----		-----	
974		-----		-----	
1146		-----		-----	
1569		-----		-----	
1801		-----		-----	
6414		-----		-----	

Determination of Water on sample #23031; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214		-----		-----	
237	D6304-C:16e1	150		-0.09	
256		-----		-----	
257		-----		-----	
309	D6304-A:20	246		1.37	
325	D6304-C:20	93		-0.95	
331	D6304-C:20	126.1		-0.45	
349	D6304-C:20	111		-0.68	
451	D6304-B:20	309		2.32	
496	D6304-B:20	145		-0.16	
603	D6304-B:20	55.9		-1.51	
633	D6304-B:20	183.5		0.42	
663	D6304-B:20	234		1.19	
862	D6304-B	186		0.46	
863	D6304-B	92		-0.96	
901	D6304-C:20	177.8		0.34	
963	D6304-B:20	151		-0.07	
974		-----		-----	
1146	D6304-B:20	164		0.13	
1569	D6304-C:16e1	163		0.11	
1801	D6304-A:20	169.0		0.20	
6414	D6304-B:20	45.1	C	-1.67	first reported 5.3

normality	OK
n	18
outliers	0
mean (n)	155.63
st.dev. (n)	65.749
R(calc.)	184.10
st.dev.(D6304-B:20)	66.040
R(D6304-B:20)	184.91
compare	
R(D6304-A:20)	82.232
R(D6304-C:20)	63.779

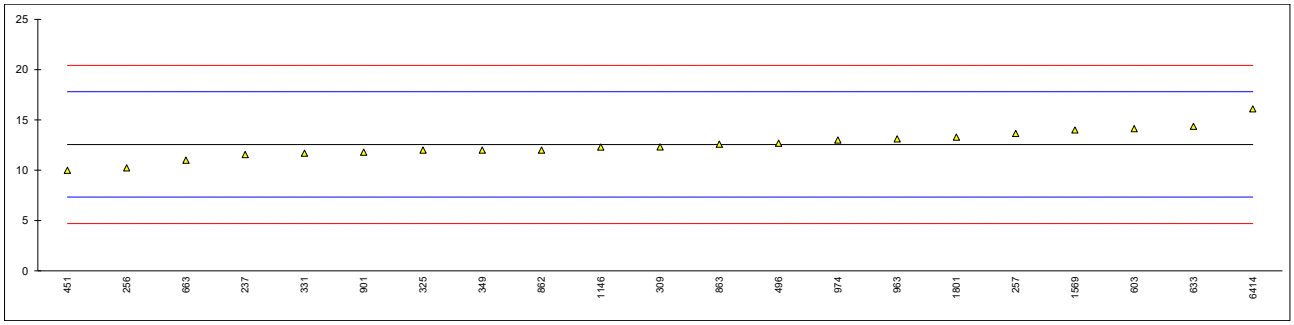


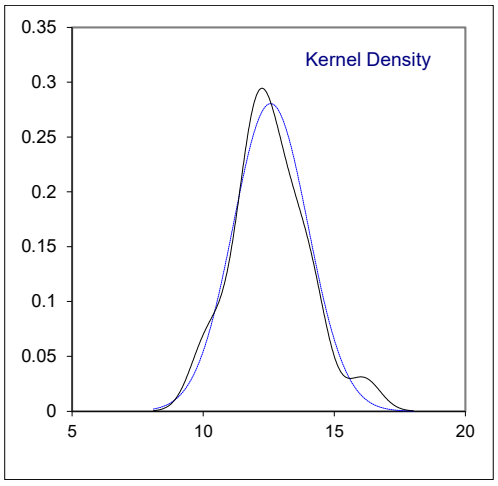


Determination of Aluminum as Al on sample #23032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214		-----		-----	
237	D5185	11.56		-0.39	
256	In house	10.24		-0.89	
257	D6595	13.67		0.42	
309	D5185	12.335		-0.09	
325	D5185	12		-0.22	
331		11.7		-0.33	
349		12		-0.22	
451	D5185	10		-0.98	
496	D5185	12.7		0.05	
603	D5185	14.13		0.60	
633	D6595	14.37		0.69	
663	D5185	11		-0.60	
862		12		-0.22	
863	D5185	12.6		0.01	
901	D5185	11.8		-0.29	
963	D5185	13.12		0.21	
974	D5185	13		0.16	
1146	In house	12.318		-0.10	
1569	D5185	14		0.55	
1801		13.3		0.28	
6414	D6595	16.11		1.35	

normality	OK
n	21
outliers	0
mean (n)	12.569
st.dev. (n)	1.4223
R(calc.)	3.983
st.dev.(D5185:18)	2.6209
R(D5185:18)	7.338

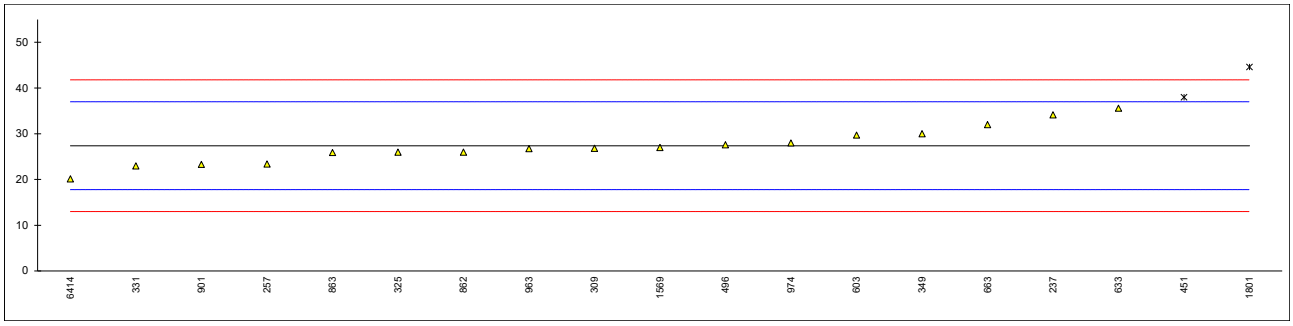


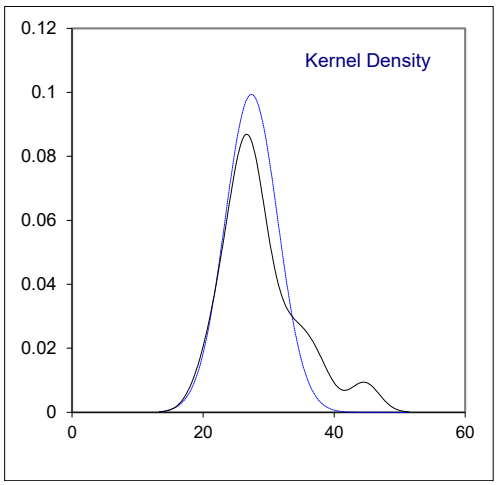


Determination of Boron as B on sample #23032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214		-----		-----	
237	D5185	34.14		1.41	
256		-----		-----	
257	D6595	23.41		-0.83	
309	D5185	26.825		-0.11	
325	D5185	26		-0.29	
331		23.0		-0.91	
349		30		0.55	
451	D5185	38	DG(0.05)	2.21	
496	D5185	27.6		0.05	
603	D5185	29.72		0.49	
633	D6595	35.58		1.71	
663	D5185	32		0.96	
862		26		-0.29	
863	D5185	25.9		-0.31	
901	D5185	23.3		-0.85	
963	D5185	26.78		-0.12	
974	D5185	28		0.13	
1146		-----		-----	
1569	D5185	27		-0.08	
1801		44.6	DG(0.05)	3.59	
6414	D6595	20.13		-1.51	

normality	OK
n	17
outliers	2
mean (n)	27.376
st.dev. (n)	4.0158
R(calc.)	11.244
st.dev.(D5185:18)	4.7991
R(D5185:18)	13.437

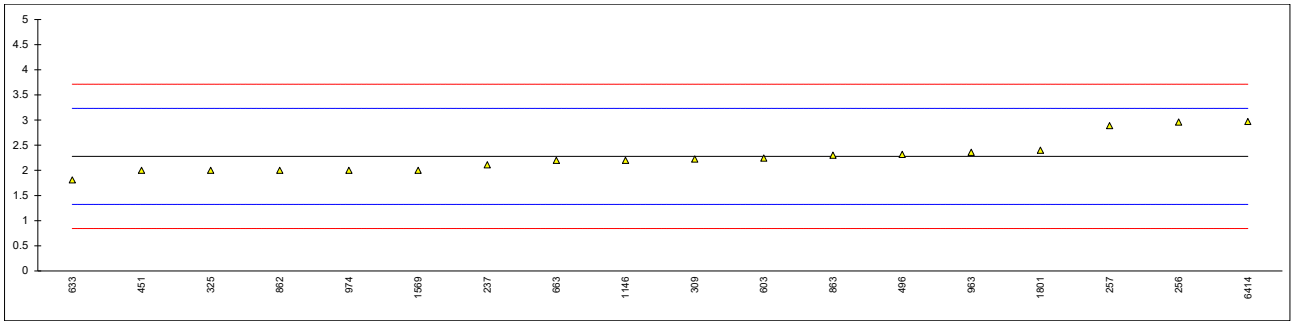


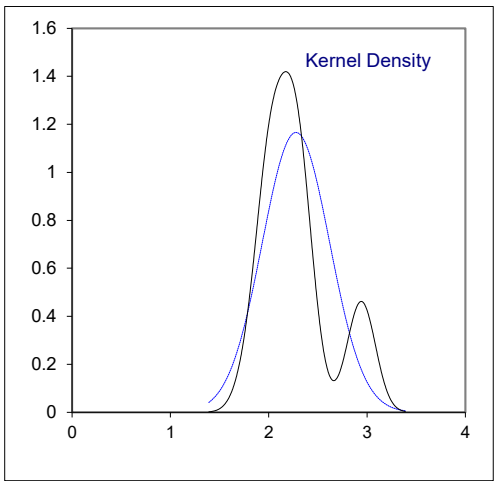


Determination of Chromium as Cr on sample #23032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214		-----		-----	
237	D5185	2.11		-0.35	
256	In house	2.96		1.43	
257	D6595	2.89		1.28	
309	D5185	2.225		-0.11	
325	D5185	2		-0.58	
331		<2	C	-----	first reported 2.0
349		<1.0	C	-----	first reported 1
451	D5185	2		-0.58	
496	D5185	2.32		0.09	
603	D5185	2.245		-0.07	
633	D6595	1.81		-0.98	
663	D5185	2.2		-0.16	
862		2		-0.58	
863	D5185	2.3		0.05	
901		-----		-----	
963	D5185	2.36		0.17	
974	D5185	2		-0.58	
1146	In house	2.2018		-0.16	
1569	D5185	2		-0.58	
1801		2.4		0.26	
6414	D6595	2.97		1.45	

normality	suspect
n	18
outliers	0
mean (n)	2.277
st.dev. (n)	0.3423
R(calc.)	0.958
st.dev.(D5185:18)	0.4779
R(D5185:18)	1.338

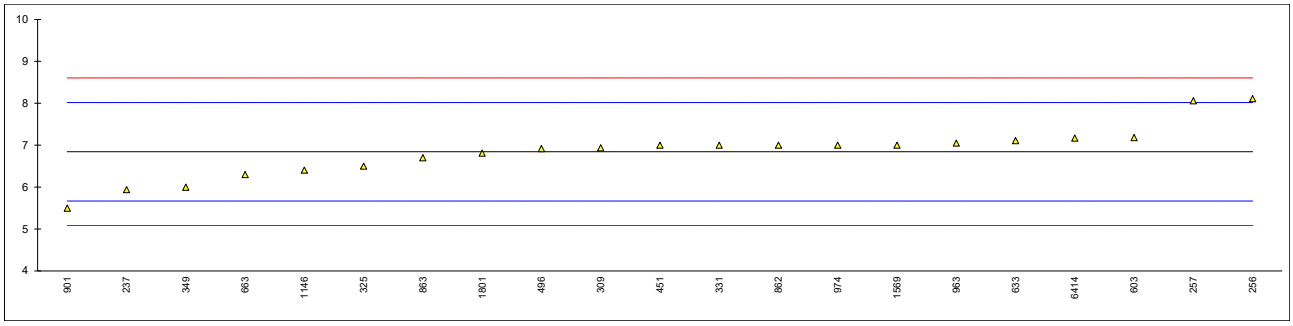


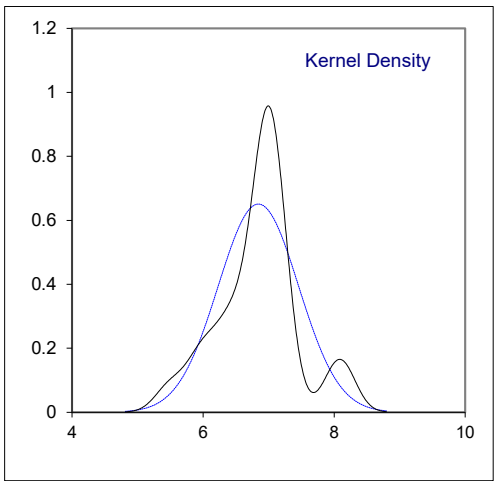


Determination of Copper as Cu on sample #23032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214		-----		-----	
237	D5185	5.94		-1.54	
256	In house	8.11		2.16	
257	D6595	8.06		2.08	
309	D5185	6.939		0.16	
325	D5185	6.5		-0.58	
331		7.0		0.27	
349		6		-1.44	
451	D5185	7		0.27	
496	D5185	6.92		0.13	
603	D5185	7.180		0.58	
633	D6595	7.11		0.46	
663	D5185	6.3		-0.93	
862		7		0.27	
863	D5185	6.7		-0.24	
901	D5185	5.5		-2.29	
963	D5185	7.05		0.35	
974	D5185	7		0.27	
1146	In house	6.4061		-0.74	
1569	D5185	7		0.27	
1801		6.81		-0.06	
6414	D6595	7.17		0.56	

normality	OK
n	21
outliers	0
mean (n)	6.843
st.dev. (n)	0.6129
R(calc.)	1.716
st.dev.(D5185:18)	0.5865
R(D5185:18)	1.642

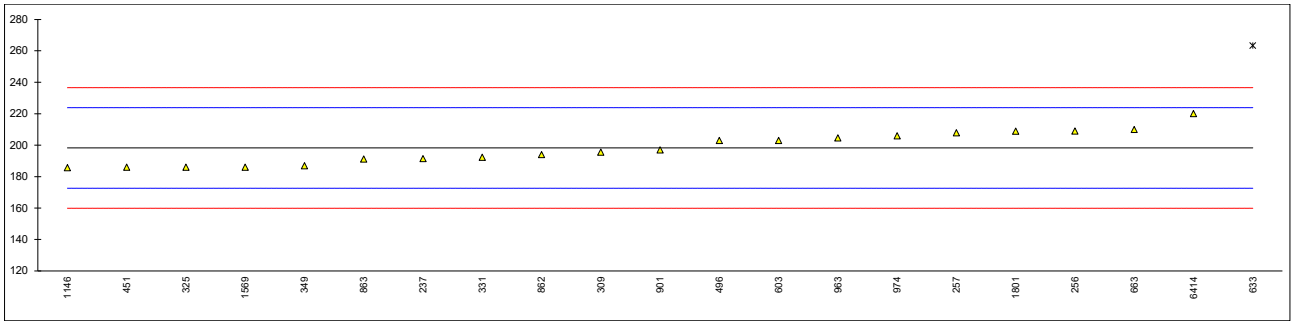


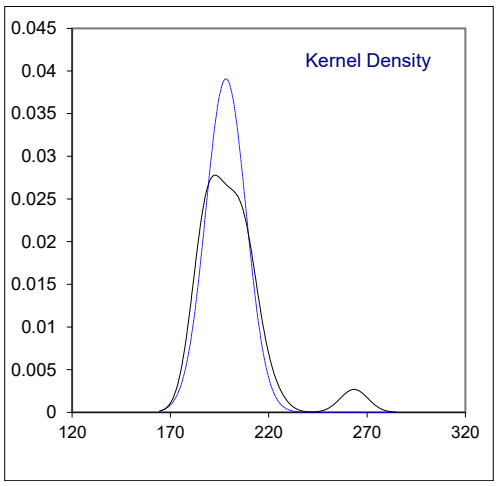


Determination of Iron as Fe on sample #23032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214		-----		-----	
237	D5185	191.5		-0.53	
256	In house	209.10		0.85	
257	D6595	207.9		0.75	
309	D5185	195.577		-0.21	
325	D5185	186		-0.96	
331		192.3		-0.47	
349		187		-0.88	
451	D5185	186		-0.96	
496	D5185	203		0.37	
603	D5185	203.0		0.37	
633	D6595	263.4	C,G(0.01)	5.10	first reported 267.6
663	D5185	210		0.92	
862		194		-0.33	
863	D5185	191.2		-0.55	
901	D5185	197		-0.10	
963	D5185	204.61		0.50	
974	D5185	206		0.61	
1146	In house	185.750		-0.98	
1569	D5185	186		-0.96	
1801		209		0.84	
6414	D6595	220.15		1.71	

normality	OK
n	20
outliers	1
mean (n)	198.254
st.dev. (n)	10.2088
R(calc.)	28.585
st.dev.(D5185:18)	12.7827
R(D5185:18)	35.792

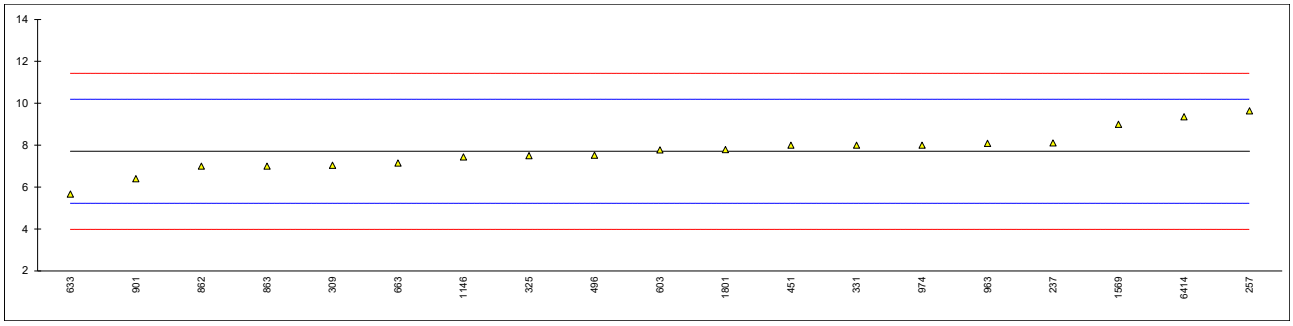


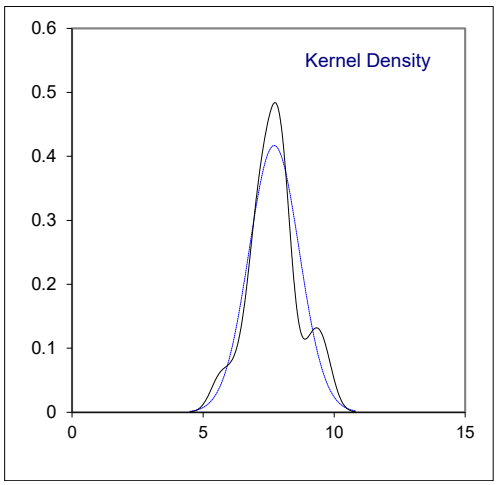


Determination of Magnesium as Mg on sample #23032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214		-----		-----	
237	D5185	8.11		0.32	
256		-----		-----	
257	D6595	9.64		1.56	
309	D5185	7.044		-0.54	
325	D5185	7.5		-0.17	
331		8.0		0.23	
349		<5	C	-----	first reported 3
451	D5185	8		0.23	
496	D5185	7.524		-0.15	
603	D5185	7.777		0.05	
633	D6595	5.67		-1.65	
663	D5185	7.15		-0.45	
862		7		-0.57	
863	D5185	7.0		-0.57	
901	D5185	6.4		-1.06	
963	D5185	8.09		0.31	
974	D5185	8		0.23	
1146	In house	7.4399		-0.22	
1569	D5185	9		1.04	
1801		7.79		0.06	
6414	D6595	9.36		1.33	

normality	OK
n	19
outliers	0
mean (n)	7.710
st.dev. (n)	0.9567
R(calc.)	2.679
st.dev.(D5185:18)	1.2394
R(D5185:18)	3.470

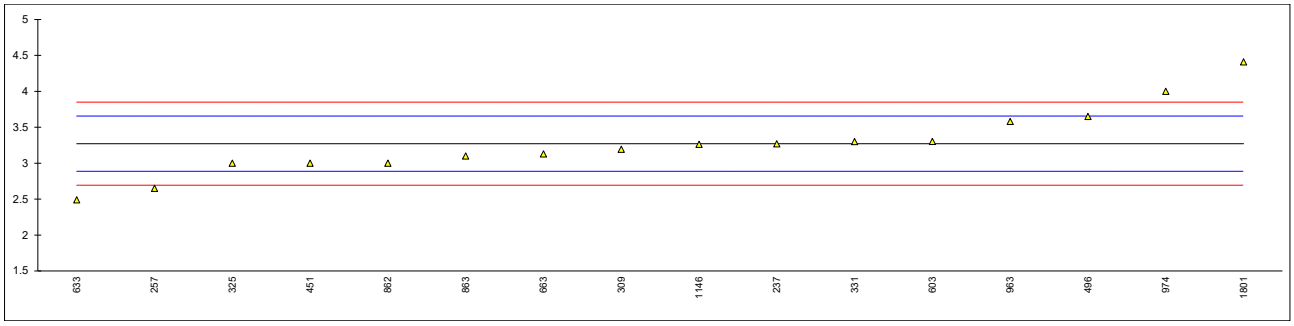


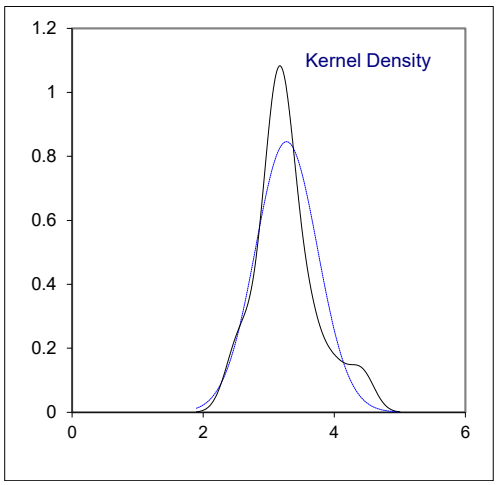


Determination of Manganese as Mn on sample #23032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214		-----		-----	
237	D5185	3.27		-0.01	
256		-----		-----	
257	D6595	2.65		-3.23	
309	D5185	3.193		-0.41	
325	D5185	3		-1.41	
331		3.3		0.15	
349		<5	C	-----	first reported 2
451	D5185	3		-1.41	
496	D5185	3.65		1.97	
603	D5185	3.303		0.17	
633	D6595	2.49		-4.06	
663	D5185	3.13		-0.73	
862		3		-1.41	
863	D5185	3.1		-0.89	
901	D5185	<5		-----	
963	D5185	3.58		1.60	
974	D5185	4		3.79	
1146	In house	3.2621		-0.05	
1569		-----		-----	
1801		4.41		5.92	
6414		-----		-----	

normality	suspect
n	16
outliers	0
mean (n)	3.271
st.dev. (n)	0.4719
R(calc.)	1.321
st.dev.(D5185:18)	0.1925
R(D5185:18)	0.539

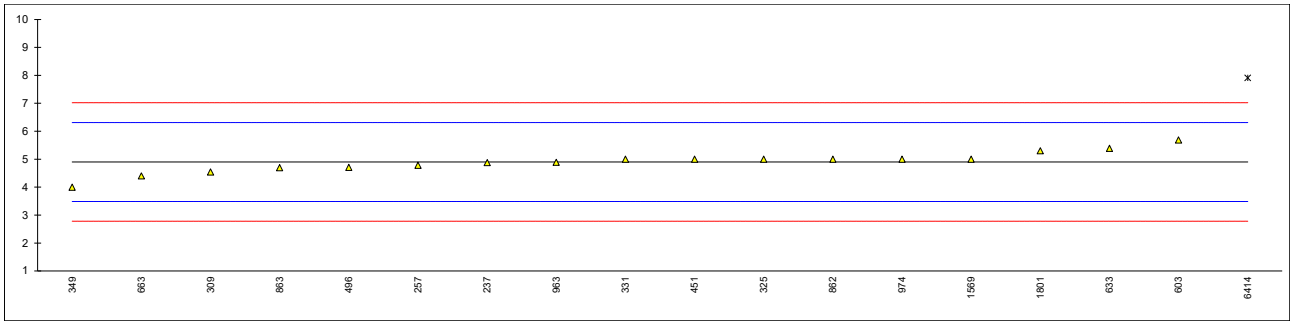


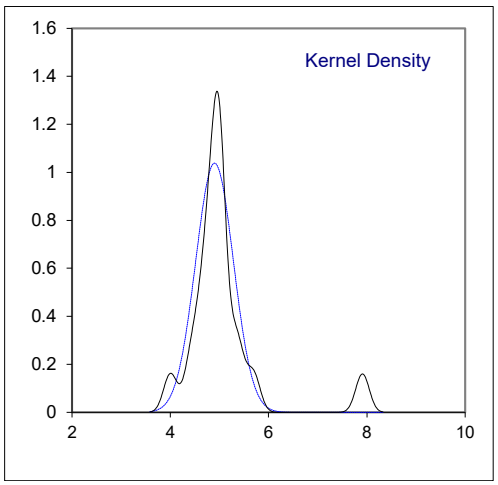


Determination of Molybdenum as Mo on sample #23032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214		-----		-----	
237	D5185	4.88		-0.03	
256		-----		-----	
257	D6595	4.78		-0.17	
309	D5185	4.542		-0.51	
325	D5185	5		0.14	
331		5.0		0.14	
349		4		-1.27	
451	D5185	5		0.14	
496	D5185	4.71		-0.27	
603	D5185	5.689		1.12	
633	D6595	5.39		0.70	
663	D5185	4.4		-0.71	
862		5		0.14	
863	D5185	4.7		-0.28	
901	D5185	<5		-----	
963	D5185	4.89		-0.01	
974	D5185	5		0.14	
1146	In house	<5		-----	
1569	D5185	5		0.14	
1801		5.3		0.57	
6414	D6595	7.91	G(0.01)	4.26	

normality	suspect
n	17
outliers	1
mean (n)	4.899
st.dev. (n)	0.3842
R(calc.)	1.076
st.dev.(D5185:18)	0.7063
R(D5185:18)	1.978

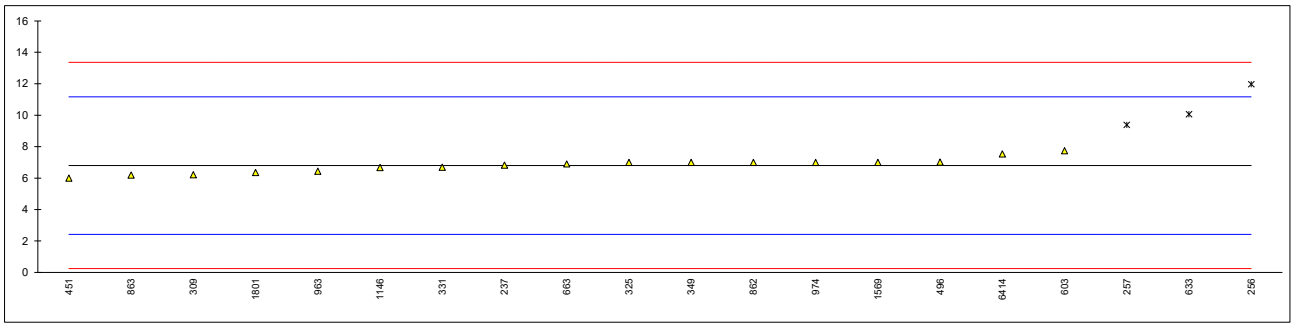


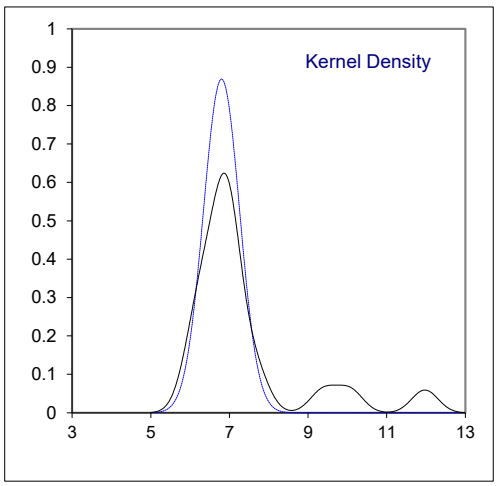


Determination of Silicon as Si on sample #23032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214		-----		-----	
237	D5185	6.83		0.01	
256	In house	11.97	G(0.01)	2.36	
257	D6595	9.39	G(0.01)	1.18	
309	D5185	6.216		-0.27	
325	D5185	7		0.09	
331		6.7	C	-0.05	first reported 44.7
349		7		0.09	
451	D5185	6		-0.37	
496	D5185	7.02		0.10	
603	D5185	7.737		0.43	
633	D6595	10.06	G(0.05)	1.49	
663	D5185	6.9		0.05	
862		7		0.09	
863	D5185	6.2		-0.27	
901	D5185	<8		-----	
963	D5185	6.44		-0.17	
974	D5185	7		0.09	
1146	In house	6.678		-0.06	
1569	D5185	7		0.09	
1801		6.36		-0.20	
6414	D6595	7.54		0.34	

normality	OK
n	17
outliers	3
mean (n)	6.801
st.dev. (n)	0.4592
R(calc.)	1.286
st.dev.(D5185:18)	2.1875
R(D5185:18)	6.125

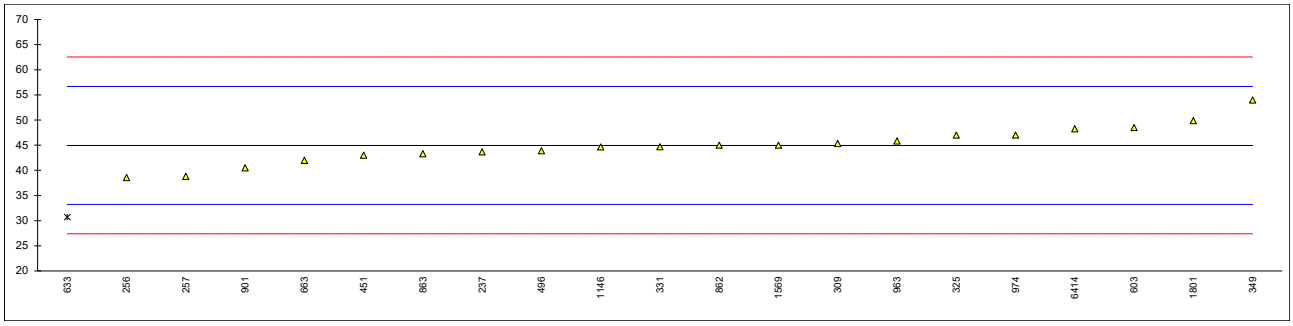


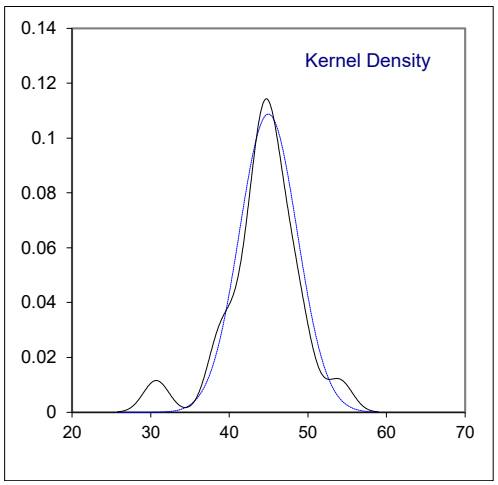


Determination of Sodium as Na on sample #23032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214		-----		-----	
237	D5185	43.7		-0.21	
256	In house	38.58		-1.09	
257	D6595	38.80		-1.05	
309	D5185	45.348		0.07	
325	D5185	47		0.35	
331		44.7	C	-0.04	first reported <2
349		54	C	1.55	first reported 5
451	D5185	43		-0.33	
496	D5185	43.9		-0.18	
603	D5185	48.50		0.61	
633	D6595	30.69	C,G(0.05)	-2.43	first reported 31.42
663	D5185	42		-0.50	
862		45		0.01	
863	D5185	43.3		-0.28	
901	D5185	40.5		-0.76	
963	D5185	45.86		0.16	
974	D5185	47		0.35	
1146	In house	44.649		-0.05	
1569	D5185	45		0.01	
1801		49.9		0.84	
6414	D6595	48.28		0.57	

normality	OK
n	20
outliers	1
mean (n)	44.951
st.dev. (n)	3.6688
R(calc.)	10.273
st.dev.(D5185:18)	5.8570
R(D5185:18)	16.400

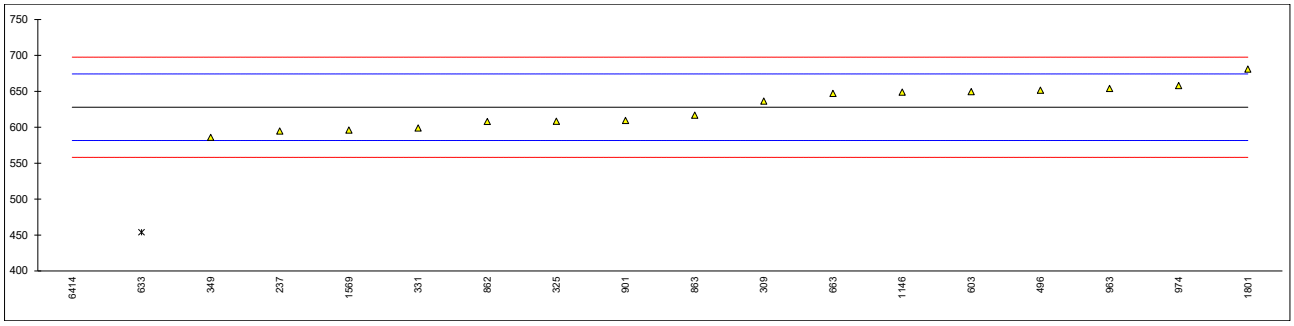


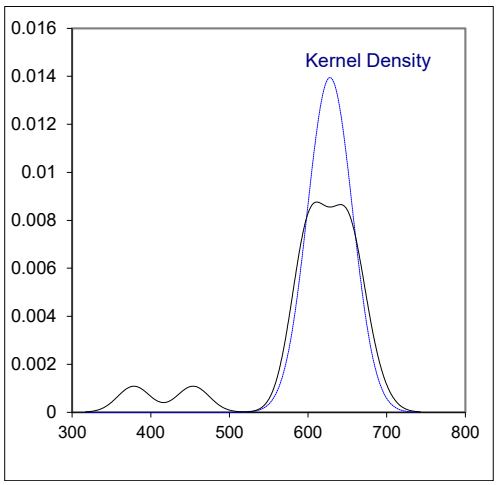


Determination of Calcium as Ca on sample #23032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214		-----		-----	
237	D5185	594.9		-1.42	
256		-----		-----	
257		-----		-----	
309	D5185	636.486		0.37	
325	D5185	608.5		-0.83	
331		599.0		-1.24	
349		586		-1.80	
451		-----		-----	
496	D5185	651.7		1.03	
603	D5185	649.6		0.94	
633	D6595	453.9	C,G(0.01)	-7.49	first reported 454.4
663	D5185	647		0.82	
862		608		-0.85	
863	D5185	616.9		-0.47	
901	D5185	609.4		-0.79	
963	D5185	654.11		1.13	
974	D5185	658		1.30	
1146	In house	648.9		0.91	
1569	D5185	596		-1.37	
1801		681		2.29	
6414	D6595	378.56	G(0.01)	-10.73	

normality	OK
n	16
outliers	2
mean (n)	627.844
st.dev. (n)	28.5919
R(calc.)	80.057
st.dev.(D5185:18)	23.2349
R(D5185:18)	65.058

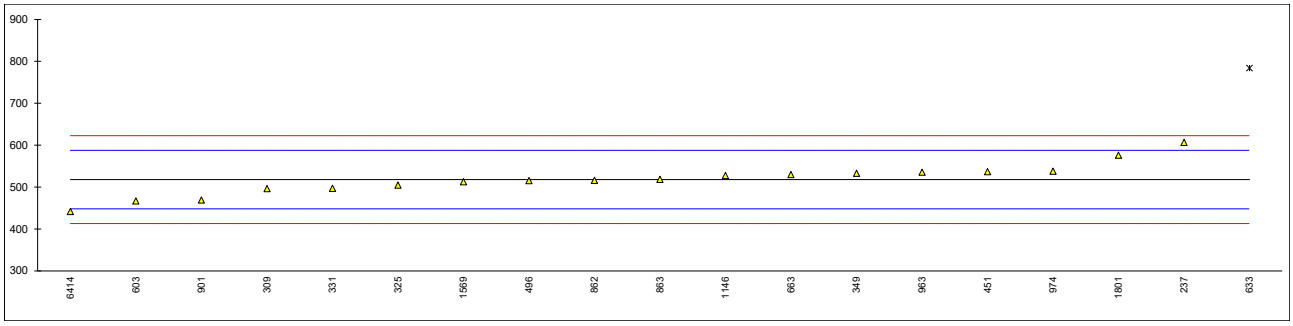


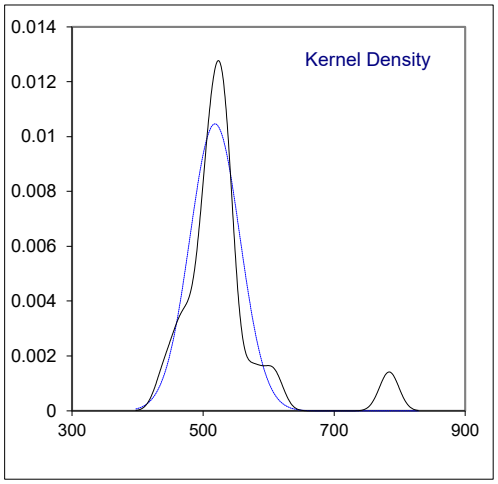


Determination of Phosphorus as P on sample #23032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214		-----		-----	
237	D5185	607	C	2.55	first reported 624.6
256		-----		-----	
257		-----		-----	
309	D5185	496.330		-0.62	
325	D5185	505		-0.37	
331		497.3		-0.59	
349		533		0.43	
451	D5185	537		0.54	
496	D5185	515.5		-0.07	
603	D5185	467.2	C	-1.45	first reported 384.1
633	D6595	783.9	C,G(0.01)	7.61	first reported 783.6
663	D5185	530		0.34	
862		516		-0.06	
863	D5185	518.5		0.01	
901	D5185	469.1		-1.40	
963	D5185	535.53		0.50	
974	D5185	538		0.57	
1146	In house	527.68		0.28	
1569	D5185	513		-0.14	
1801		576		1.66	
6414	D6595	441.68		-2.18	

normality	suspect
n	18
outliers	1
mean (n)	517.990
st.dev. (n)	38.1292
R(calc.)	106.762
st.dev.(D5185:18)	34.9519
R(D5185:18)	97.865

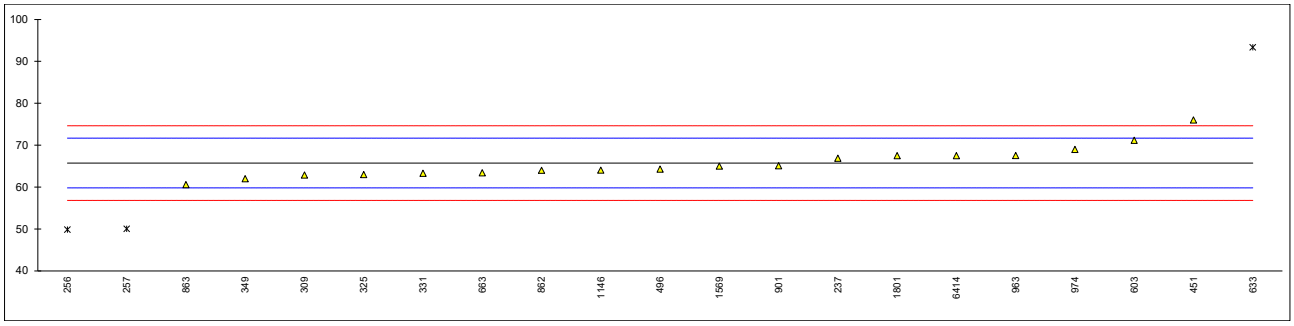


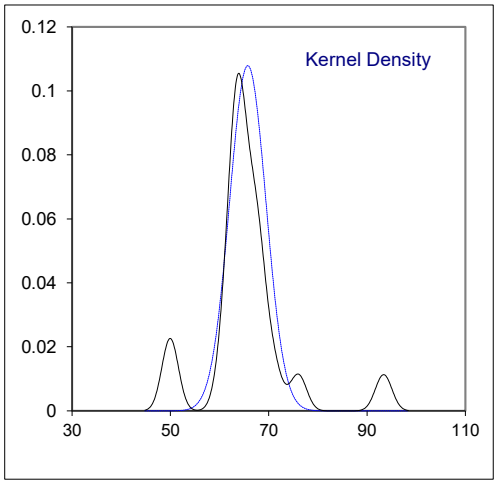


Determination of Zinc as Zn on sample #23032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214		-----		-----	
237	D5185	66.9		0.39	
256	In house	49.87	C,DG(0.01)	-5.36	first reported 102.77
257	D6595	50.04	C,DG(0.01)	-5.30	first reported 101.21
309	D5185	62.870		-0.97	
325	D5185	63		-0.92	
331		63.3		-0.82	
349		62		-1.26	
451	D5185	76		3.47	
496	D5185	64.31		-0.48	
603	D5185	71.16		1.83	
633	D6595	93.36	C,G(0.01)	9.33	first reported 94.60
663	D5185	63.4		-0.79	
862		64		-0.59	
863	D5185	60.6		-1.73	
901	D5185	65.1		-0.21	
963	D5185	67.54		0.61	
974	D5185	69		1.10	
1146	In house	64.058		-0.57	
1569	D5185	65		-0.25	
1801		67.5		0.60	
6414	D6595	67.51		0.60	

normality	not OK
n	18
outliers	3
mean (n)	65.736
st.dev. (n)	3.6984
R(calc.)	10.355
st.dev.(D5185:18)	2.9614
R(D5185:18)	8.292





APPENDIX 2

Reported test results of other elements on sample #23032; results in mg/kg

lab	Barium as Ba	Cadmium as Cd	Lead as Pb	Lithium as Li	Nickel as Ni
178	-----	-----	-----	-----	-----
179	-----	-----	-----	-----	-----
214	-----	-----	-----	-----	-----
237	1	-----	<1	-----	<1
256	-----	-----	0.70	-----	0.0
257	0.72	0.36	0.61	0.65	0.32
309	0.571	<0.01	0.655	0.382	<0.01
325	<1	<1	<1	<1	<1
331	<2	<2	0.0 C	<2	<2
349	<0.5 C	0	0	0	0
451	< 1	< 1	< 1	< 1	< 1
496	<1	<1	<1,1	<1	<1
603	0.848	0.178	0.589	-----	0.646
633	0.547	0.115	0.944	0.954	0.093
663	0.57	-----	<10	-----	<5
862	<1	<1	<1	<1	<1
863	<1.0	<1.0	<1.0	-----	<1.0
901	<0,5	-----	<10	-----	<5
963	0.590	0.05	0.30	0.46	0.27
974	<1	<1	<1	<1	<1
1146	<5	-----	<2	<1	<1
1569	1	-----	<1	-----	1
1801	0.603	0.623	<0.430	-----	0.457
6414	1.5	-----	0.63	-----	0.49

lab	Potassium as K	Silver as Ag	Tin as Sn	Titanium as Ti	Vanadium as V
178	-----	-----	-----	-----	-----
179	-----	-----	-----	-----	-----
214	-----	-----	-----	-----	-----
237	1	<1	<1	<1	<1
256	-----	0.73	0.35	-----	0.76
257	1.24	0.00	0.44	0.00	0.43
309	0.391	0.108	1.045	0.092	0.012
325	3	<1	<1	<1	<1
331	<2	<2 C	<2	<2	<2
349	0	0	0	0	0
451	-----	< 1	< 1	< 1	< 1
496	<1	<1	1.21	<1	<1
603	3.580	0.844	-1.968	0.034	-0.299
633	1.21	0.016	7.47 C	0.334	0
663	-----	<0.5	<10	<5	<1
862	<1	<1	<1	<1	<1
863	1.2	<1.0	<1.0	<1.0	<1.0
901	<40	<0,5	<10	<5	<1
963	<0.10	<0.10	0.07	0.09	<0.10
974	<1	<1	<1	<1	<1
1146	-----	<5	<1	<5	<1
1569	-----	<1	-----	-----	<1
1801	<2.10	1.99	0.212	0.883	1.44
6414	-----	0.03	0.0	0.87	0.0

Lab 331 first reported <2 for Lead and 6.7 for Silver
Lab 349 first reported 0 for Barium
Lab 633 first reported 8.58 for Tin

APPENDIX 3

Number of participants per country

1 lab in ALGERIA
1 lab in BELGIUM
2 labs in CHINA, People's Republic
1 lab in FRANCE
1 lab in GERMANY
1 lab in IRELAND
1 lab in MALAYSIA
2 labs in NETHERLANDS
1 lab in NIGERIA
1 lab in PHILIPPINES
1 lab in SAUDI ARABIA
3 labs in SPAIN
2 labs 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 4

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
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?
SDS	= Safety Data Sheet

Literature

1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2 ISO5725:86
3 ISO5725 parts 1-6:94
4 ISO13528:05
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7 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
8 J.N. Miller, Analyst, 118, 455, (1993)
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10 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)
11 W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)