Institute for
Interlaboratory Studies

Results of Proficiency Test
Gear Oil (used)
March 2022

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. M. Meijer
Correctors: ing. C.M. Nijssen-Wester & ing. R.J. Starink
Approved by: ing. A.S. Noordman-de Neef

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CONTENTS

1 INTRODUCTION 3

2 SET UP 3

2.1 ACCREDITATION 3

2.2 PROTOCOL 3

2.3 CONFIDENTIALITY STATEMENT 3

2.4 SAMPLES 4

2.5 STABILITY OF THE SAMPLES 5

2.6 ANALYZES 5

3 RESULTS 6

3.1 STATISTICS 6

3.2 GRAPHICS 7

3.3 Z-SCORES 8

4 EVALUATION 8

4.1 EVALUATION PER SAMPLE AND PER TEST 9

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES 11

4.3 COMPARISON OF THE PROFICIENCY TEST OF MARCH 2022 WITH PREVIOUS PTS 12

Appendices:

1. Data, statistical and graphic results 14
2. Other reported test results 33
3. Number of participants per country 34
4. Abbreviations and literature 35

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 2021/2022 it was decided to continue the round robin for the analysis of used Gear Oil.

In this interlaboratory study 26 laboratories in 19 countries registered for participation, see appendix 3 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 0.5 L bottle labelled #22031 for the regular analyzes and one 50 mL HDPE bottle labelled #22032 for metal determination.
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 Gear Oil (used) a batch of approximately 22 liters of Gear Oil (used) was obtained from a third party. After homogenization 43 amber glass bottles of 0.5 L were filled and labelled #22031.
The homogeneity of the subsamples was checked by determination of Density at 15 °C in accordance with ISO12185 and Water in accordance with D6304 on 8 stratified randomly selected subsamples.

	Density at 15 °C in kg/L	Water in mg/kg
sample #22031-1	0.89167	263
sample #22031-2	0.89167	248
sample #22031-3	0.89168	240
sample #22031-4	0.89169	225
sample #22031-5	0.89168	243
sample #22031-6	0.89169	241
sample #22031-7	0.89168	214
sample #22031-8	0.89167	216

Table 1: homogeneity test results of subsamples #22031

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)	0.00002	46.9
reference test method	ISO12185:96	ASTM D6304-B:20
0.3 x R (reference test method)	0.00015	66.0

Table 2: evaluation of the repeatabilities of subsamples #22031

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 Metals determination in Gear Oil (used) a batch of approximately 10 liters of Gear Oil (used) was obtained from a third party. This batch was positive for some metals and was spiked with the metals Aluminum, Copper, Silicon and Tin using Conostan standards. After homogenization 48 HDPE bottles of 50 mL were filled and labelled #22032.
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 #22032-1	42.2
sample #22032-2	43.3
sample #22032-3	41.7
sample #22032-4	42.4
sample #22032-5	42.4
sample #22032-6	42.4
sample #22032-7	42.2
sample #22032-8	42.1

Table 3: homogeneity test results of subsamples #22032

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)	1.3
reference test method	ASTM D5185:18
0.3 x R (reference test method)	3.1

Table 4: evaluation of the repeatability of subsamples #22032

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 Gear Oil (used) labelled #22031 and one sample Gear Oil (used) labelled #22032 were sent on February 16, 2022. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Gear Oil (used) 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 #22031: 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, Water and Level of Contamination (counts/mL and scale number).
Also, some extra information was asked about the determination of Total Acid Number.

On sample #22032 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 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

Some problems were encountered with the dispatch of the samples due to COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with another week. One participant reported test results after the extended reporting date and three other participants did not report any test results. Not all participants were able to report all tests requested.
In total 23 participants reported 317 numerical test results. Observed were 11 outlying test results, which is 3.5%. 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.

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 #22031

Total Acid Number: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D664-A:18e2 for the end point mode BEP 60 mL, but is not in agreement for the other end point modes (BEP 125 mL, IP 60 mL and IP 125 mL).
Remarkably, one participant has 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. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers 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 full agreement with the requirements of ASTM D93:20 procedure A, but is not in agreement 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. One statistical outlier was observed and one other test result was excluded. The calculated reproducibility after rejection of the suspect data is in agreement with the requirements of ASTM D445:21e1.

Kinematic Viscosity at 100 °C: This determination may be problematic for a number of laboratories. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ASTM D445:21e1.

Viscosity Index: This determination was not problematic. No statistical outliers were observed but five 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 5.0 µm: Only one laboratory 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).

Level of Contamination: This determination was problematic. No statistical outliers were observed over six parameters. The calculated reproducibilities at ≥ 4 µm are in agreement with the requirements of ASTM D7647:10(2018).
At ≥ 6 µm and ≥ 14 µm the calculated reproducibilities are not in agreement with the respective requirements.

sample #22032

Aluminum as Al: 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.

Boron as B: 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. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D5185:18.

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

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

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

Calcium as Ca: This determination may be problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the estimated reproducibility calculated with the Horwitz equation and also not in agreement with the strict 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 not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the estimated reproducibility calculated with the Horwitz equation but is 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 are 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 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 test methods (in casu ASTM and ISO test methods or Horwitz) are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Total Acid Number	mg KOH/g	19	0.56	0.33	0.32
Density at 15 °C	kg/L	16	0.8916	0.0002	0.0005
Flash Point PMcc	°C	17	193.3	13.2	13.7
Kinematic Viscosity at 40 °C	mm^2/s	17	140.04	2.58	2.95
Kinematic Viscosity at 100 °C	mm^2/s	15	14.07	0.16	0.16
Viscosity Index		11	97.3	2.1	2
Membrane Filtration 5.0 µm	%M/M	1	n.e.	n.e.	n.e.
Water	mg/kg	17	247	187	224
Level of Contamination					
≥ 4 µm (c)	counts/mL	7	54326	50767	61389
≥ 6 µm (c)	counts/mL	7	29264	27990	22241
≥14 µm (c)	counts/mL	7	2384	4578	3218
≥ 4 µm (c)	scale no.	6	22.7	1.4	1.7
≥ 6 µm (c)	scale no.	6	21.8	2.1	1.2

Parameter	unit	n	average	2.8 * sd	R(lit)
≥14 µm (c)	scale no.	6	17.7	3.4	2

Table 5: reproducibilities of tests on sample #22031

Parameter	unit	n	average	2.8 * sd	R(lit)
Aluminum as Al	mg/kg	18	8.3	4.0	6.6
Boron as B	mg/kg	15	5.9	3.5	13.2
Copper as Cu	mg/kg	17	7.9	1.1	1.9
Iron as Fe	mg/kg	17	43.2	4.8	10.6
Silicon as Si	mg/kg	18	9.6	2.2	7.0
Tin as Sn	mg/kg	18	9.0	6.3	8.2
Calcium as Ca	mg/kg	14	2.8	1.9	1.1
Phosphorus as P	mg/kg	15	309	41	76
Zinc as Zn	mg/kg	16	2.7	1.1	1.0

Table 6: reproducibilities of tests on sample #22032

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 2022 WITH PREVIOUS PTS

	March 2022	March 2021	March 2020	April 2019	April 2018
Number of reporting laboratories	23	28	24	24	22
Number of test results	317	616	414	421	391
Number of statistical outliers	11	31	25	22	18
Percentage of statistical outliers	3.5%	5.0%	6.0%	5.2%	4.6%

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 2022	March 2021	March 2020	April 2019	April 2018
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 2022	March 2021	March 2020	April 2019	April 2018
Level of Contamination	-	-	-	-	-
Aluminum as Al	+	++	+	+	+
Barium as Ba	n.e.	-	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.e.	--	n.a.	n.a.	n.a.
Magnesium as Mg	n.e.	+	n.a.	n.a.	n.a.
Manganese as Mn	n.e.	-	n.a.	n.a.	n.a.
Molybdenum as Mo	n.e.	+	n.a.	n.a.	n.a.
Potassium as K	n.e.	++	n.a.	n.a.	n.a.
Silicon as Si	++	+	++	++	++
Sodium as Na	n.e.	+	+	+	+
Tin as Sn	+	n.e.	+	+	+
Calcium as Ca	-	-	(--)	-	-
Phosphorus as P	+	-	+	-	++
Zinc as Zn	+/-	+	(--)	(--)	+/-

Table 8: comparison determinations against the reference test methods

Results between brackets should be used with due care

The following performance categories were used:

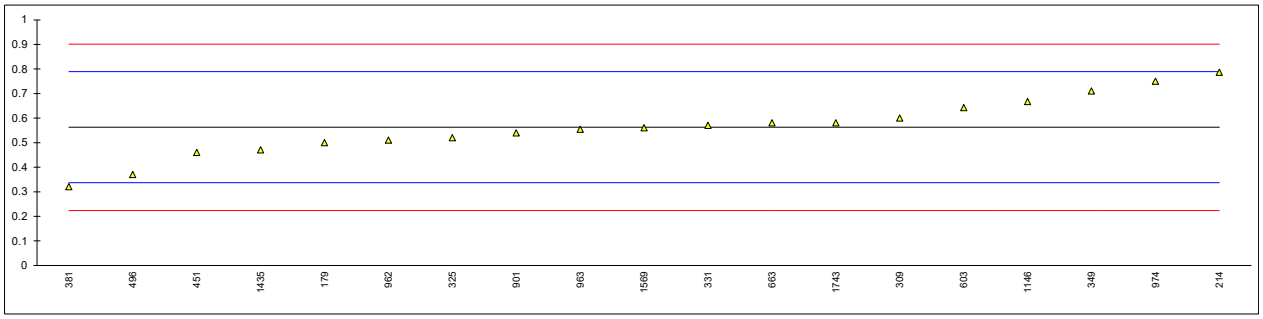
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test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

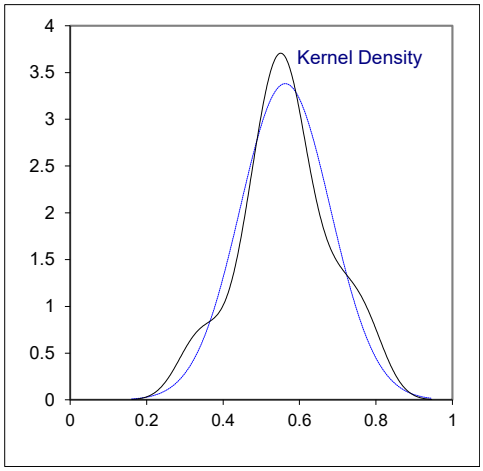
APPENDIX 1

Determination of Total Acid Number on sample #22031; results in mg KOH/g

lab	method	value	mark	z(targ)	end point type	volume (mL)	remarks
178		-----		-----	---	---	
179	D664-A	0.50		-0.55	---	---	
214	D664-A	0.7865		1.98	Inflection Point	125 mL	
237		-----		-----	---	---	
257		-----		-----	---	---	
309	D664-A	0.60		0.33	Buffer End Point pH 10	125 mL	
325	D664-A	0.52		-0.38	---	---	
331	D664Mod.	0.57		0.07	---	---	
349	D664-A	0.71		1.31	Buffer End Point pH 10	125 mL	
381	D664-A	0.32		-2.15	Buffer End Point pH 10	125 mL	
451	D664-A	0.46		-0.91	Buffer End Point pH 10	60 mL	
496	D664-A	0.37		-1.71	Buffer End Point pH 10	60 mL	
603	D664-A	0.6423		0.71	Inflection Point	125 mL	
633		-----		-----	---	---	
663	D664-A	0.58		0.15	Buffer End Point pH 10	60 mL	
862		-----		-----	---	---	
863		-----		-----	---	---	
901	D974	0.54		-0.20	---	---	
962	D974	0.51		-0.47	---	---	
963	D664-B	0.554		-0.08	Inflection Point	60 mL	
974	D664-A	0.75		1.66	Inflection Point	125 mL	
1146	D664-A	0.667		0.92	Buffer End Point pH 10	125 mL	
1435	D664-A	0.47		-0.82	---	---	
1569	D664-A	0.56		-0.02	Inflection Point	125 mL	
1743	D664-A	0.58		0.15	Buffer End Point pH 11	60 mL	
6190		-----		-----	---	---	

	normality	OK
	n	19
	outliers	0
	mean (n)	0.5626
	st.dev. (n)	0.11795
	R(calc.)	0.3303
	st.dev.(D664-A:18e2, BEP 60 mL)	0.11292
	R(D664-A:18e2, BEP 60 mL)	0.3162
Compare		
	R(D664-A:18e2, BEP 125 mL)	0.1726
	R(D664-A:18e2, IP 60 mL)	0.2510
	R(D664-A:18e2, IP 125 mL)	0.1201

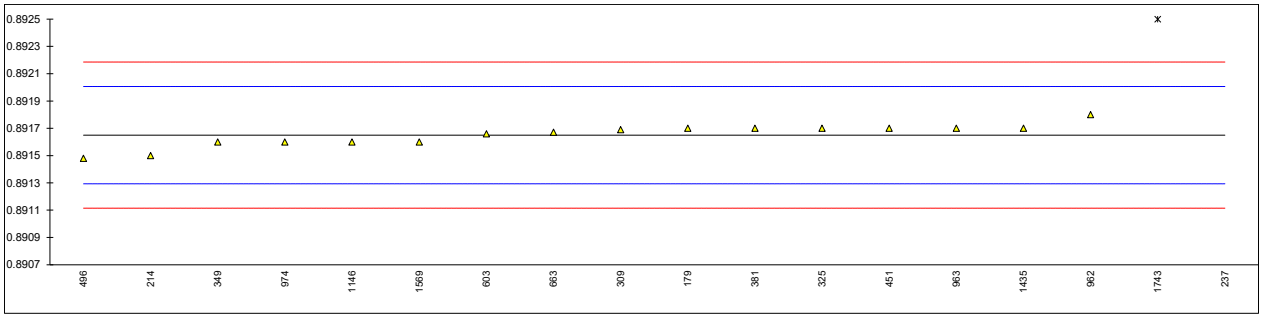


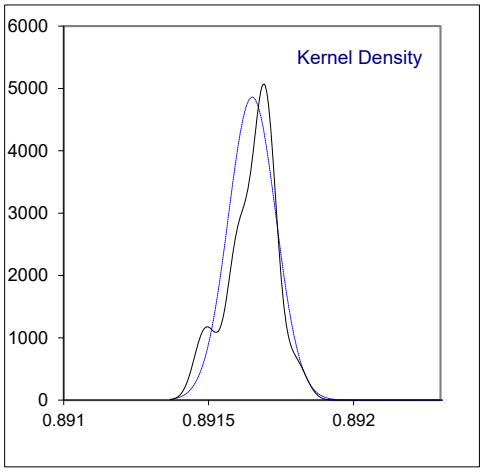


Determination of Density at 15 °C on sample #22031; results in kg/L

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D4052	0.8917		0.28	
214	D7042	0.8915		-0.84	
237	D4052	0.8935	G(0.01)	10.36	
257		-----		-----	
309	D4052	0.89169		0.22	
325	D4052	0.8917		0.28	
331		-----		-----	
349	D4052	0.8916	C	-0.28	first reported 0.8913
381	ISO12185	0.8917		0.28	
451	D4052	0.8917		0.28	
496	ISO12185	0.89148		-0.95	
603	D4052	0.89166		0.06	
633		-----		-----	
663	D4052	0.89167		0.11	
862		-----		-----	
863		-----		-----	
901		-----		-----	
962	D4052	0.8918	C	0.84	first reported 0.8913
963	D4052	0.8917		0.28	
974	D4052	0.8916		-0.28	
1146	D4052	0.8916		-0.28	
1435	D4052	0.8917		0.28	
1569	ISO12185	0.8916	C	-0.28	first reported 895.4 kg/m^3
1743	ISO12185	0.8925	G(0.01)	4.76	
6190		-----		-----	

normality	OK
n	16
outliers	2
mean (n)	0.891650
st.dev. (n)	0.0000821
R(calc.)	0.000230
st.dev.(ISO12185:96)	0.0001786
R(ISO12185:96)	0.0005

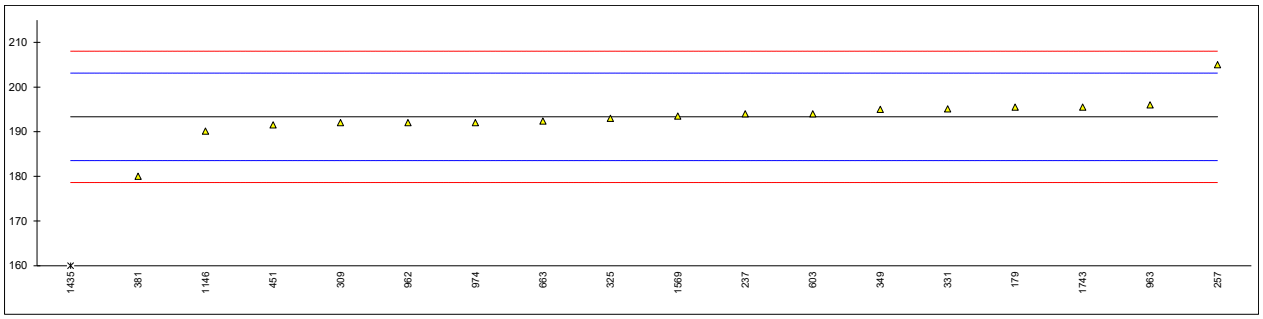


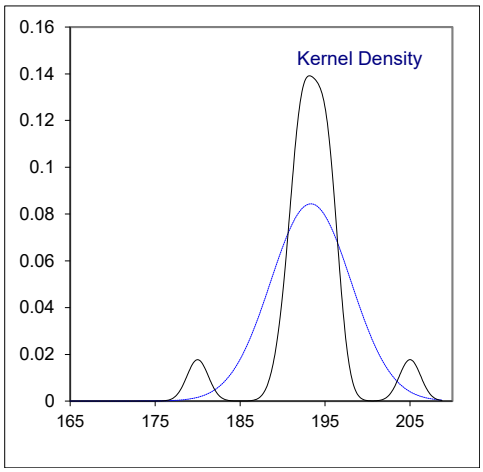


Determination of Flash Point PMcc on sample #22031; results in °C

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D93-A	195.5		0.44	
214		-----		-----	
237	D93	194.0		0.14	
257	D3828	205		2.38	
309	D93-A	192.0		-0.27	
325	D93-A	193	C	-0.07	first reported 181.0
331	D93-A	195.1		0.36	
349	D93-A	195		0.34	
381	ISO2719-B	180	C	-2.72	first reported 178
451	D93-A	191.5		-0.37	
496		-----		-----	
603	D93-A	194		0.14	
633		-----		-----	
663	D93-A	192.38		-0.19	
862		-----		-----	
863		-----		-----	
901		-----		-----	
962	D93-A	192.0		-0.27	
963	D93-A	196.0		0.55	
974	D93-A	192		-0.27	
1146	D93-A	190.1		-0.66	
1435	D93-A	160	G(0.01)	-6.80	
1569	D93-A	193.5		0.04	
1743	ISO2719-A	195.5		0.44	
6190		-----		-----	

	normality	not OK
	n	17
	outliers	1
	mean (n)	193.33
	st.dev. (n)	4.726
	R(calc.)	13.23
	st.dev.(D93-A:20)	4.902
	R(D93-A:20)	13.73
Compare		
	R(D93-B:20)	10

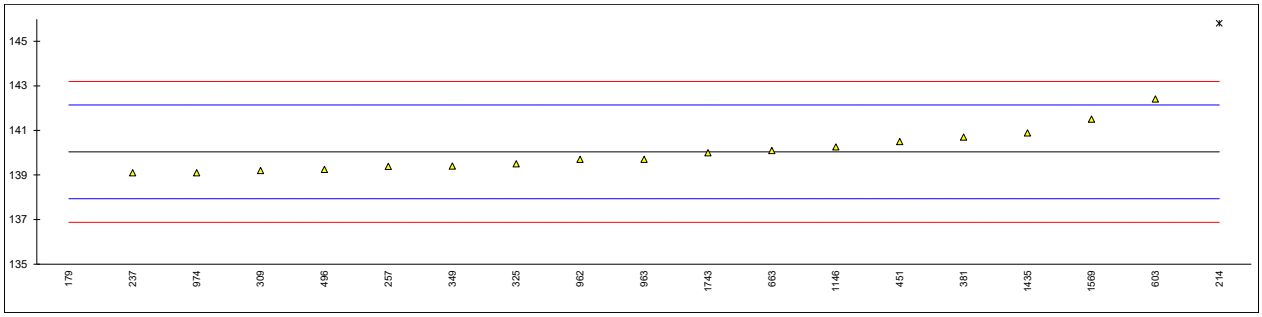


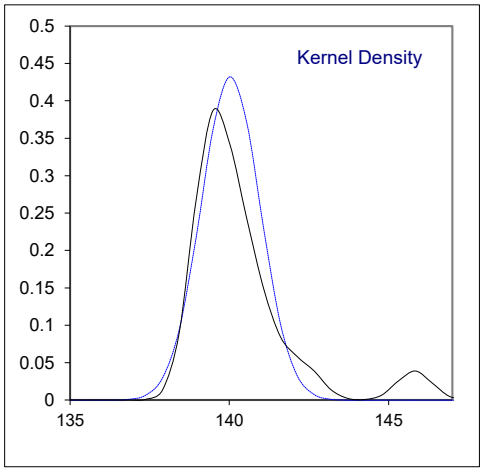


Determination of Kinematic Viscosity at 40 °C on sample #22031; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D445	14.07	ex	-119.62	reported the same test result for KV 40 and 100 °C
214	D7042	145.81	G(0.01)	5.48	
237	D445	139.1		-0.89	
257	D7279 corrected to D445	139.39		-0.62	
309	D445	139.2		-0.80	
325	D445	139.5		-0.51	
331		-----		-----	
349	D445	139.4		-0.61	
381	D445	140.7	C	0.63	first reported 137.0
451	D7279 corrected to D445	140.50		0.44	
496	D445	139.25		-0.75	
603	D7042	142.41		2.25	
633		-----		-----	
663	D445	140.10		0.06	
862		-----		-----	
863		-----		-----	
901		-----		-----	
962	D445	139.7		-0.32	
963	D445	139.7		-0.32	
974	D445	139.1		-0.89	
1146	D445	140.26		0.21	
1435	D7042	140.89		0.81	
1569	D445	141.5		1.39	
1743	D445	140.0		-0.04	
6190		-----		-----	

normality	not OK	
n	17	
outliers	1 +1ex	
mean (n)	140.0412	
st.dev. (n)	0.92275	
R(calc.)	2.5837	
st.dev.(D445:21e1)	1.05313	
R(D445:21e1)	2.9488	R(D445:21e1 - used (in service) formulated oils)

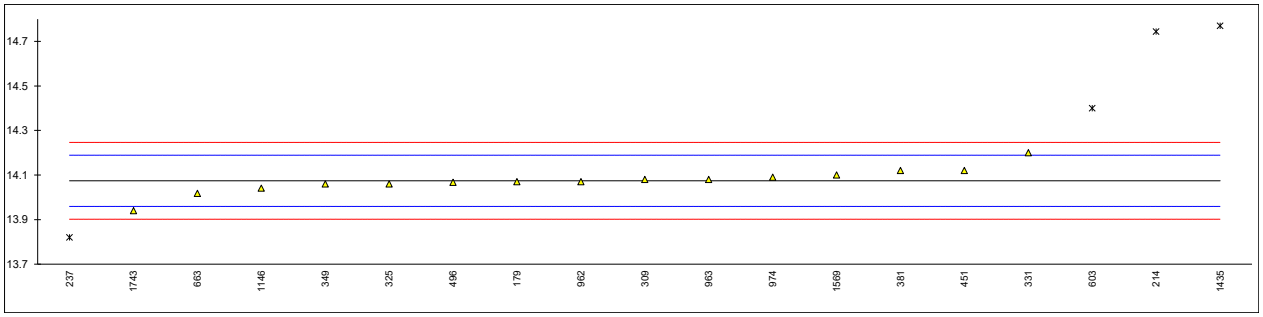


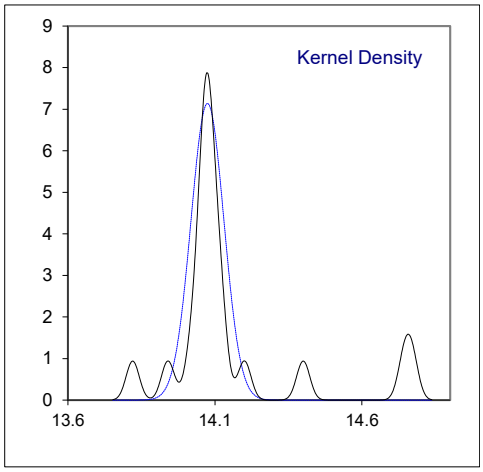


Determination of Kinematic Viscosity at 100 °C on sample #22031; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D445	14.07		-0.08	
214	D7042	14.744	DG(0.01)	11.64	
237	D445	13.82	G(0.01)	-4.42	
257		-----		-----	
309	D445	14.08		0.10	
325	D445	14.06		-0.25	
331	D7279 corrected to D445	14.2	C	2.18	first reported 14.4
349	D445	14.06		-0.25	
381	D445	14.12		0.79	
451	D7279 corrected to D445	14.12		0.79	
496	D445	14.067		-0.13	
603	D7042	14.40	C,G(0.05)	5.66	first reported 14.702
633		-----		-----	
663	D445	14.018		-0.98	
862		-----		-----	
863		-----		-----	
901		-----		-----	
962	D445	14.07		-0.08	
963	D445	14.08		0.10	
974	D445	14.09		0.27	
1146	D445	14.041		-0.58	
1435	D7042	14.77	DG(0.01)	12.09	
1569	D445	14.10		0.45	
1743	D445	13.94		-2.34	
6190		-----		-----	

normality	not OK	
n	15	
outliers	4	
mean (n)	14.0744	
st.dev. (n)	0.05591	
R(calc.)	0.1565	
st.dev.(D445:21e1)	0.05752	
R(D445:21e1)	0.1611	R(D445:21e1 - used (in service) formulated oils)



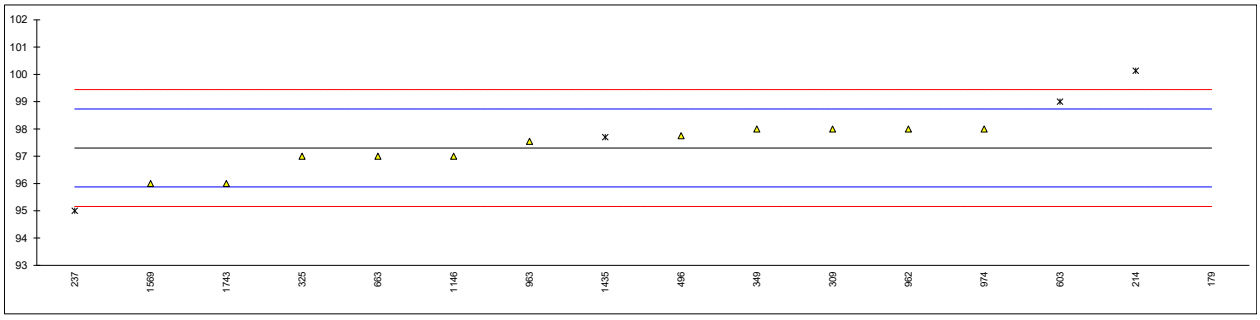


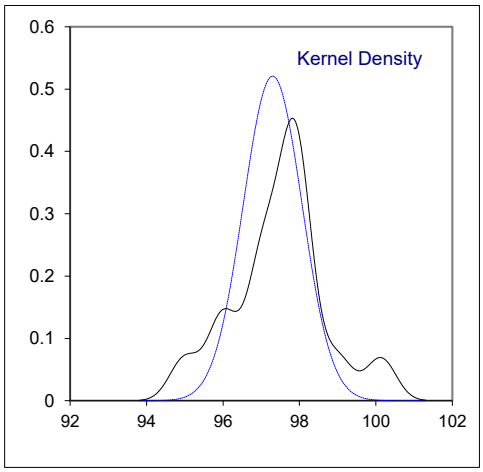
Determination of Viscosity Index on sample #22031

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D2270	971	ex	1223.18	test result excluded as statistical outlier in KV 40 °C
214	D2270	100.131	ex	3.96	test result excluded as statistical outlier in KV 40 and 100 °C
237	D2270	95	ex	-3.22	test result excluded as statistical outlier in KV 100 °C
257		-----		-----	
309	D2270	98		0.98	
325	D2270	97		-0.42	
331		-----		-----	
349	D2270	98		0.98	
381		-----		-----	
451		-----		-----	
496	D2270	97.75		0.63	
603	D2270	99	ex,C	2.38	test result excluded as statistical outlier in KV 100 °C
633		-----		-----	
663	D2270	97		-0.42	
862		-----		-----	
863		-----		-----	
901		-----		-----	
962	D2270	98		0.98	
963	D2270	97.543		0.34	
974	D2270	98		0.98	
1146	D2270	97		-0.42	
1435	D2270	97.70	ex, E	0.56	test result excluded as statistical outlier in KV 100 °C
1569	D2270	96		-1.82	
1743	D2270	96		-1.82	
6190		-----		-----	

normality	OK
n	11
outliers	0 +5ex
mean (n)	97.30
st.dev. (n)	0.766
R(calc.)	2.15
st.dev.(D2270:10)	0.714
R(D2270:10)	2

Lab 603 first reported 102.5
Lab 1435 calculation difference, iis calculated 104.62





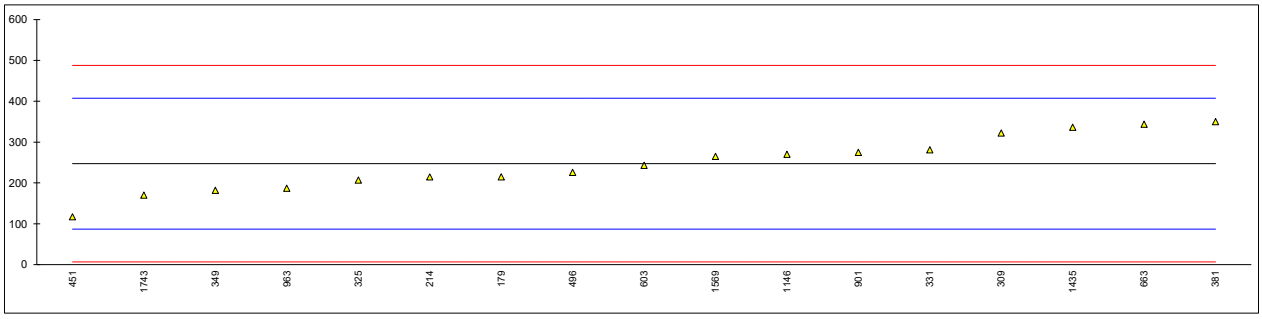
Determination of Membrane Filtration 5.0 µm on sample #22031; results in %M/M

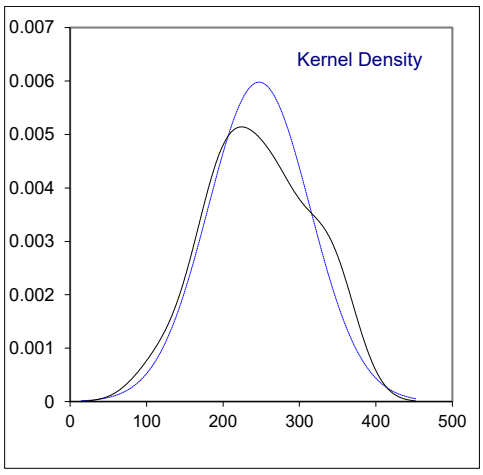
lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214		-----		-----	
237		-----		-----	
257		-----		-----	
309		-----		-----	
325		-----		-----	
331		-----		-----	
349		-----		-----	
381		-----		-----	
451		-----		-----	
496		-----		-----	
603		-----		-----	
633		-----		-----	
663		-----		-----	
862		-----		-----	
863		-----		-----	
901		-----		-----	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1146		-----		-----	
1435	D4055	212		-----	
1569		-----		-----	
1743		-----		-----	
6190		-----		-----	

Determination of Water on sample #22031; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179	D6304-C:20	215		-0.40	
214	ISO12937	214.6		-0.41	
237		-----		-----	
257		-----		-----	
309	D6304-A:20	322.5		0.94	
325	D6304-C:20	207		-0.50	
331	D7279Mod.	281		0.42	
349	D6304-C:20	182		-0.82	
381	D6304-A:20	350		1.28	
451	D6304-B:20	117		-1.63	
496	D6304-B:20	226		-0.27	
603	D6304-C:16e1	242.9		-0.06	
633		-----		-----	
663	D6304-B:20	343.8		1.20	
862		-----		-----	
863		-----		-----	
901	D6304-C:20	275		0.35	
962		-----		-----	
963	D6304-C:16e1	187.0		-0.75	
974		-----		-----	
1146	D6304-B:20	270		0.28	
1435	D6304-A:20	336		1.11	
1569	D6304-C:16e1	265		0.22	
1743	ISO12937	170		-0.97	
6190		-----		-----	

	normality	OK
	n	17
	outliers	0
	mean (n)	247.34
	st.dev. (n)	66.698
	R(calc.)	186.75
	st.dev.(D6304-B:20)	80.133
	R(D6304-B:20)	224.37
Compare		
	R(D6304-A:20)	114.02
	R(D6304-C:20)	92.63

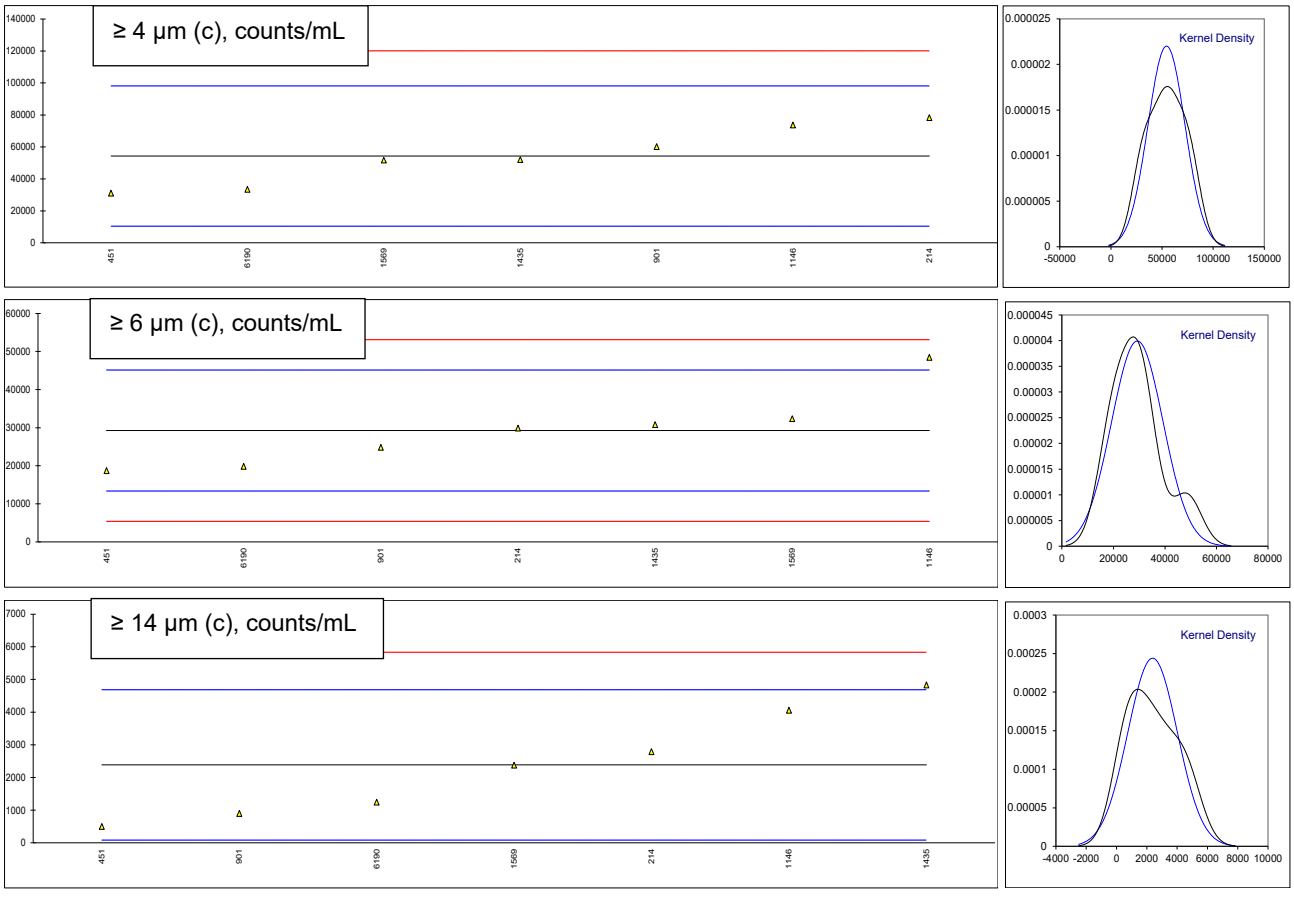




Determination of Level of Contamination on sample #22031; results in counts/mL

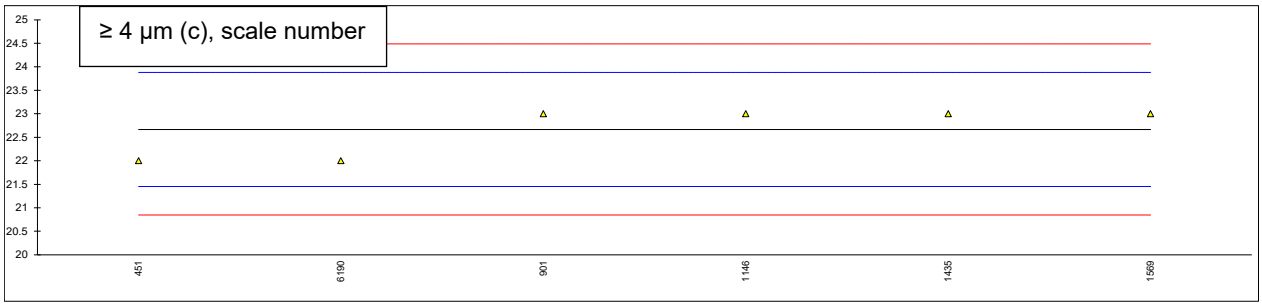
lab	method	≥ 4 µm (c)	mark	z(targ)	≥ 6 µm (c)	mark	z(targ)	≥ 14 µm (c)	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179		-----		-----	-----		-----	-----		-----
214	ISO4406	78240	C	1.09	29867	C	0.08	2787	C	0.35
237		-----		-----	-----		-----	-----		-----
257		-----		-----	-----		-----	-----		-----
309		-----		-----	-----		-----	-----		-----
325		-----		-----	-----		-----	-----		-----
331		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
381		-----		-----	-----		-----	-----		-----
451	ISO11500	31026		-1.06	18710		-1.33	499		-1.64
496		-----		-----	-----		-----	-----		-----
603		-----		-----	-----		-----	-----		-----
633		-----		-----	-----		-----	-----		-----
663		-----		-----	-----		-----	-----		-----
862		-----		-----	-----		-----	-----		-----
863		-----		-----	-----		-----	-----		-----
901	D7647	60192		0.27	24854		-0.56	895		-1.30
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
974		-----		-----	-----		-----	-----		-----
1146		73665		0.88	48444		2.41	4059		1.46
1435	ISO4407	52059		-0.10	30772		0.19	4832		2.13
1569	ISO11500	51684		-0.12	32353		0.39	2374		-0.01
1743		-----		-----	-----		-----	-----		-----
6190		33418.17		-0.95	19847.43		-1.19	1241.70		-0.99
	normality	unknown			unknown			unknown		
	n	7			7			7		
	outliers	0			0			0		
	mean (n)	54326			29264			2384		
	st.dev. (n)	18131.2			9996.3			1634.8		
	R(calc.)	50767			27990			4578		
	st.dev.(D7647:10)	21924.5			7943.1			1149.4		
	R(D7647:10)	61389			22241			3218		

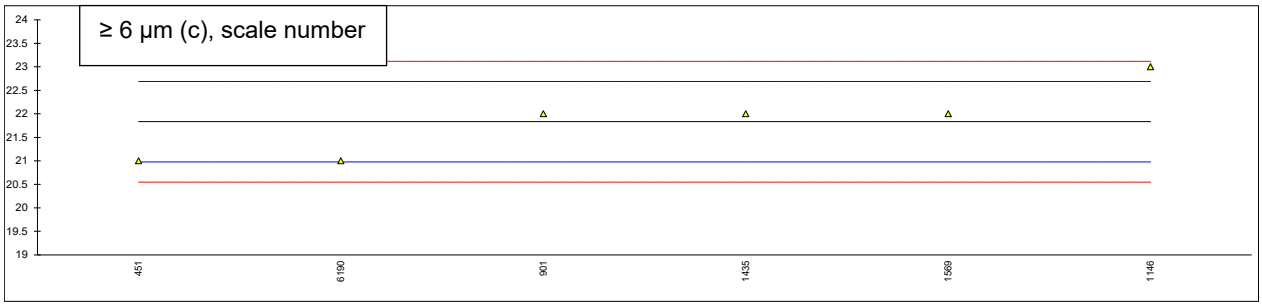
Lab 214 reported test results as scale numbers

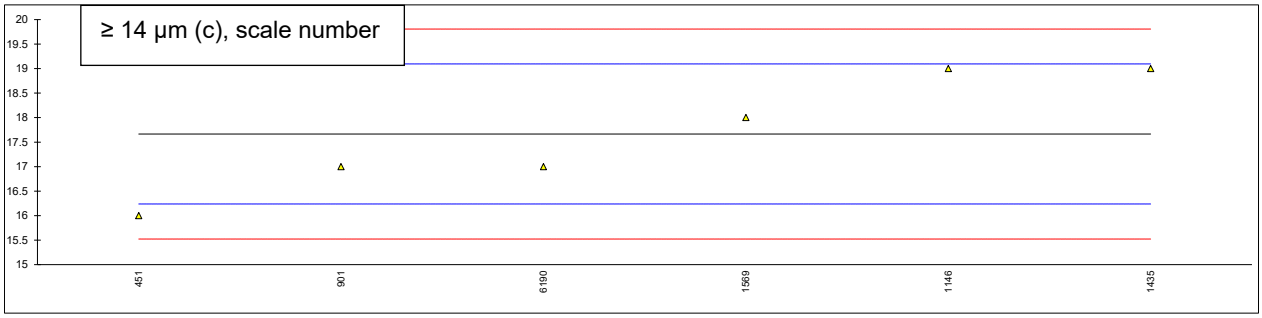


Determination of Level of Contamination acc. to ISO4406 scale on sample #22031; results in scale number

lab	method	≥ 4 µm (c)	mark	z(targ)	≥ 6 µm (c)	mark	z(targ)	≥ 14 µm (c)	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179		-----		-----	-----		-----	-----		-----
214		-----		-----	-----		-----	-----		-----
237		-----		-----	-----		-----	-----		-----
257		-----		-----	-----		-----	-----		-----
309		-----		-----	-----		-----	-----		-----
325		-----		-----	-----		-----	-----		-----
331		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
381		-----		-----	-----		-----	-----		-----
451	ISO4406	22		-1.10	21		-1.94	16		-2.33
496		-----		-----	-----		-----	-----		-----
603		-----		-----	-----		-----	-----		-----
633		-----		-----	-----		-----	-----		-----
663		-----		-----	-----		-----	-----		-----
862		-----		-----	-----		-----	-----		-----
863		-----		-----	-----		-----	-----		-----
901	ISO4406	23		0.55	22		0.39	17		-0.93
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
974		-----		-----	-----		-----	-----		-----
1146	D7647	23		0.55	23		2.72	19		1.87
1435	ISO4406	23		0.55	22		0.39	19		1.87
1569	ISO11500	23		0.55	22		0.39	18		0.47
1743		-----		-----	-----		-----	-----		-----
6190	ISO4406	22		-1.10	21		-1.94	17		-0.93
	normality	unknown			unknown			unknown		
	n	6			6			6		
	outliers	0			0			0		
	mean (n)	22.7			21.8			17.7		
	st.dev. (n)	0.52			0.75			1.21		
	R(calc.)	1.4			2.1			3.4		
	st.dev.(D7647:10)	0.61			0.43			0.71		
	R(D7647:10)	1.7			1.2			2		



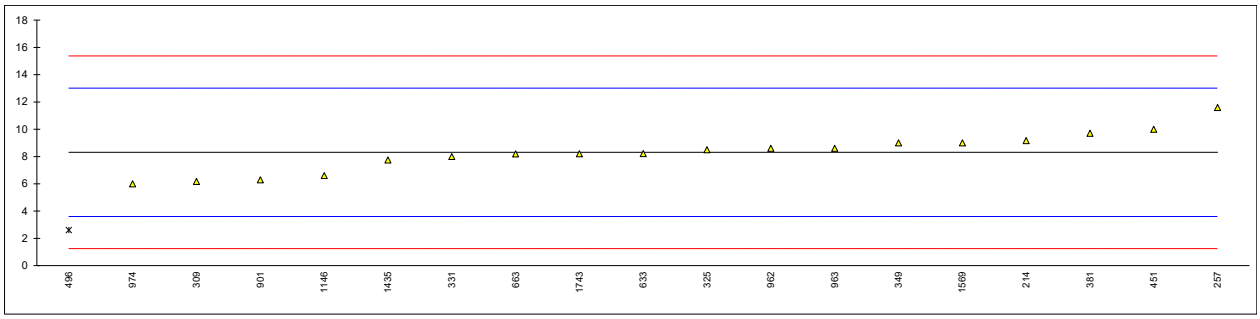


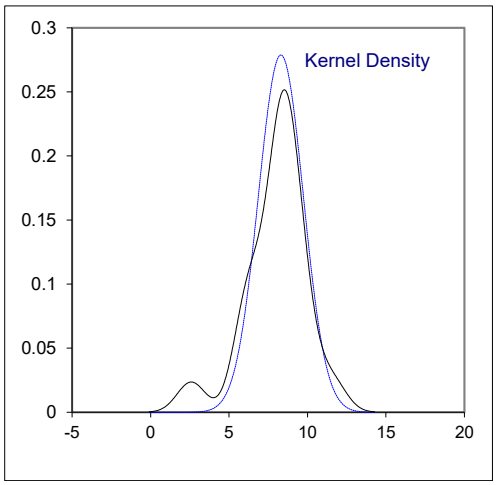


Determination of Aluminum as Al on sample #22032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214	D6595	9.165		0.36	
237		-----		-----	
257	D6595	11.6		1.40	
309	D5185	6.18		-0.91	
325	D5185	8.5	C	0.08	first reported 13
331	D5185	8		-0.13	
349	D5185	9		0.29	
381	D5185	9.70		0.59	
451	D5185	10		0.72	
496	D5185	2.6	G(0.05)	-2.43	
603		-----		-----	
633	D6595	8.222		-0.04	
663	D5185	8.19		-0.05	
862		-----		-----	
863		-----		-----	
901	D5185	6.29		-0.86	
962	D5185	8.6		0.12	
963	D5185	8.60		0.12	
974	D5185	6		-0.98	
1146	D4951	6.6		-0.73	
1435	D5185	7.741		-0.24	
1569	D5185	9		0.29	
1743	D5185	8.2		-0.05	
6190		-----		-----	

normality	OK
n	18
outliers	1
mean (n)	8.310
st.dev. (n)	1.4314
R(calc.)	4.008
st.dev.(D5185:18)	2.3536
R(D5185:18)	6.590

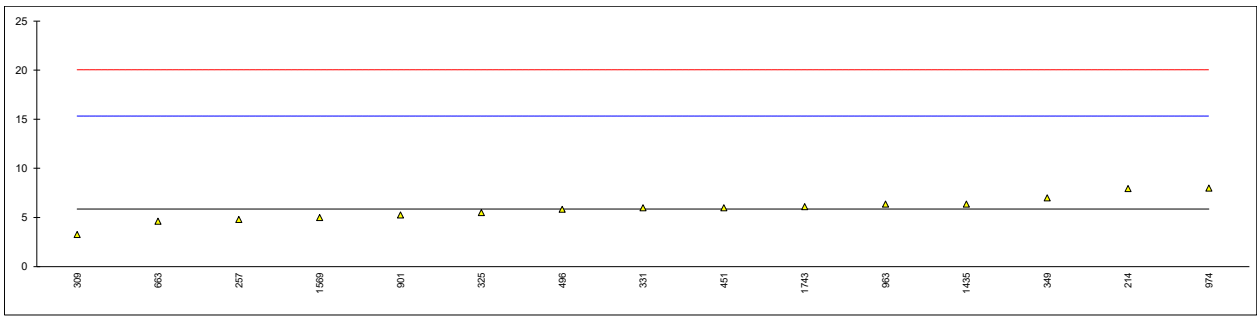


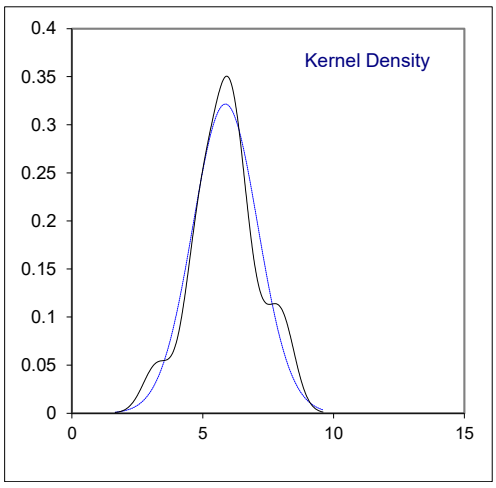


Determination of Boron as B on sample #22032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214	D6595	7.955		0.44	
237		-----		-----	
257	D6595	4.8		-0.23	
309	D5185	3.26		-0.55	
325	D5185	5.5		-0.08	
331	D5185	6		0.03	
349	D5185	7		0.24	
381		-----		-----	
451	D5185	6		0.03	
496	D5185	5.83		-0.01	
603		-----		-----	
633		-----		-----	
663	D5185	4.61		-0.27	
862		-----		-----	
863		-----		-----	
901	D5185	5.26		-0.13	
962		-----		-----	
963	D5185	6.36		0.10	
974	D5185	8		0.45	
1146		-----		-----	
1435	D5185	6.367		0.11	
1569	D5185	5		-0.18	
1743	D5185	6.1		0.05	
6190		-----		-----	

normality	OK
n	15
outliers	0
mean (n)	5.869
st.dev. (n)	1.2406
R(calc.)	3.474
st.dev.(D5185:18)	4.7258
R(D5185:18)	13.232

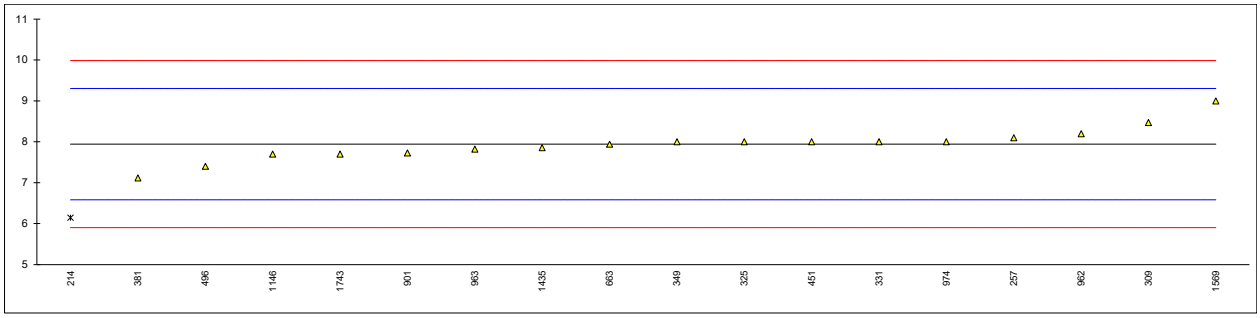


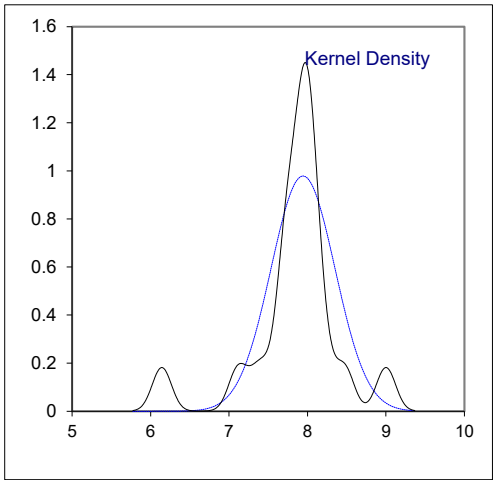


Determination of Copper as Cu on sample #22032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214	D6595	6.145	G(0.05)	-2.64	
237		-----		-----	
257	D6595	8.1		0.23	
309	D5185	8.47		0.77	
325	D5185	8		0.08	
331	D5185	8		0.08	
349	D5185	8		0.08	
381	D5185	7.12		-1.21	
451	D5185	8		0.08	
496	D5185	7.40		-0.80	
603		-----		-----	
633		-----		-----	
663	D5185	7.94		-0.01	
862		-----		-----	
863		-----		-----	
901	D5185	7.73	C	-0.31	first reported 6.59
962	D5185	8.2		0.38	
963	D5185	7.82		-0.18	
974	D5185	8		0.08	
1146	D4951	7.7		-0.36	
1435	D5185	7.858		-0.13	
1569	D5185	9		1.55	
1743	D5185	7.7		-0.36	
6190		-----		-----	

normality	not OK
n	17
outliers	1
mean (n)	7.943
st.dev. (n)	0.4077
R(calc.)	1.142
st.dev.(D5185:18)	0.6809
R(D5185:18)	1.906

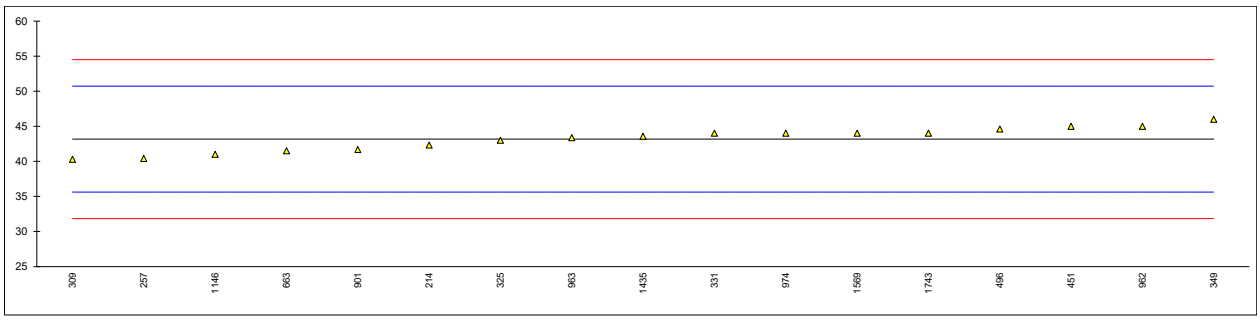


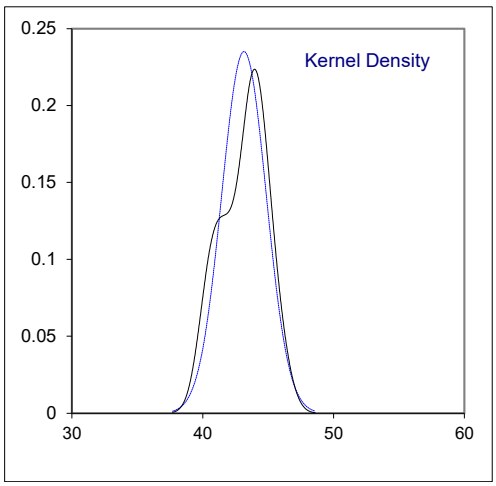


Determination of Iron as Fe on sample #22032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214	D6595	42.33		-0.22	
237		-----		-----	
257	D6595	40.40		-0.73	
309	D5185	40.30		-0.76	
325	D5185	43		-0.04	
331	D5185	44		0.22	
349	D5185	46		0.75	
381		-----		-----	
451	D5185	45		0.49	
496	D5185	44.6		0.38	
603		-----		-----	
633		-----		-----	
663	D5185	41.5		-0.44	
862		-----		-----	
863		-----		-----	
901	D5185	41.68		-0.39	
962	D5185	45		0.49	
963	D5185	43.37		0.06	
974	D5185	44		0.22	
1146	D4951	41.0		-0.57	
1435	D5185	43.571		0.11	
1569	D5185	44		0.22	
1743	D5185	44		0.22	
6190		-----		-----	

normality	OK
n	17
outliers	0
mean (n)	43.162
st.dev. (n)	1.6969
R(calc.)	4.751
st.dev.(D5185:18)	3.7751
R(D5185:18)	10.570

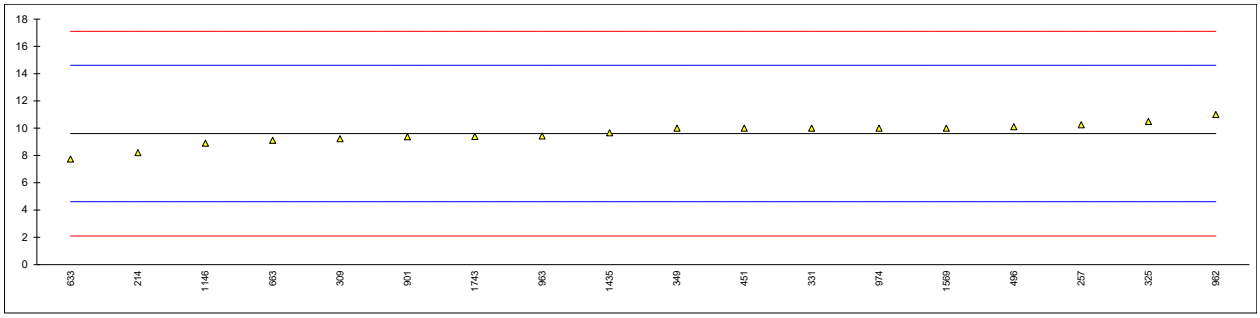


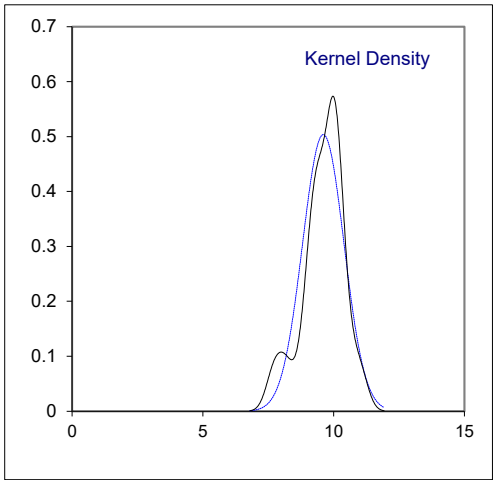


Determination of Silicon as Si on sample #22032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214	D6595	8.205		-0.56	
237		-----		-----	
257	D6595	10.25		0.26	
309	D5185	9.23		-0.15	
325	D5185	10.5		0.36	
331	D5185	10		0.16	
349	D5185	10		0.16	
381		-----		-----	
451	D5185	10		0.16	
496	D5185	10.1		0.20	
603		-----		-----	
633	D6595	7.730		-0.75	
663	D5185	9.11		-0.20	
862		-----		-----	
863		-----		-----	
901	D5185	9.37		-0.09	
962	D5185	11		0.56	
963	D5185	9.43		-0.07	
974	D5185	10		0.16	
1146	D4951	8.9		-0.28	
1435	D5185	9.655		0.02	
1569	D5185	10		0.16	
1743	D5185	9.4		-0.08	
6190		-----		-----	

normality	OK
n	18
outliers	0
mean (n)	9.604
st.dev. (n)	0.7925
R(calc.)	2.219
st.dev.(D5185:18)	2.5027
R(D5185:18)	7.007

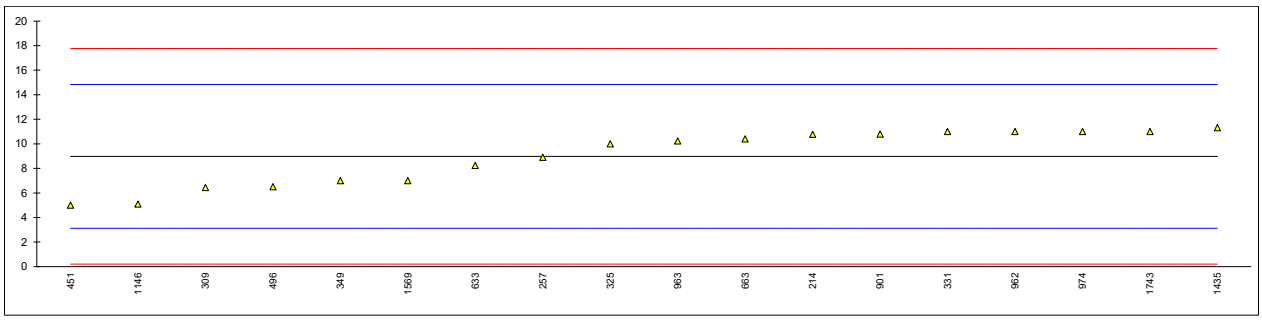


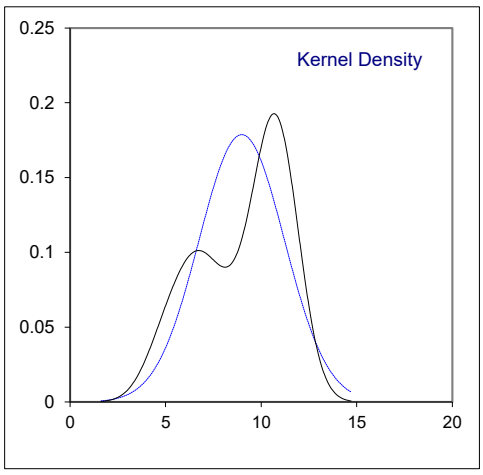


Determination of Tin as Sn on sample #22032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214	D6595	10.77		0.61	
237		-----		-----	
257	D6595	8.90		-0.03	
309	D5185	6.44		-0.87	
325	D5185	10		0.35	
331	D5185	11		0.69	
349	D5185	7		-0.68	
381		-----		-----	
451	D5185	5		-1.36	
496	D5185	6.5		-0.85	
603		-----		-----	
633	D6595	8.2325		-0.26	
663	D5185	10.4		0.48	
862		-----		-----	
863		-----		-----	
901	D5185	10.79		0.62	
962	D5185	11		0.69	
963	D5185	10.23		0.43	
974	D5185	11		0.69	
1146	D4951	5.1		-1.33	
1435	D5185	11.319		0.80	
1569	D5185	7		-0.68	
1743	D5185	11		0.69	
6190		-----		-----	

normality	OK
n	18
outliers	0
mean (n)	8.982
st.dev. (n)	2.2334
R(calc.)	6.253
st.dev.(D5185:18)	2.9252
R(D5185:18)	8.191



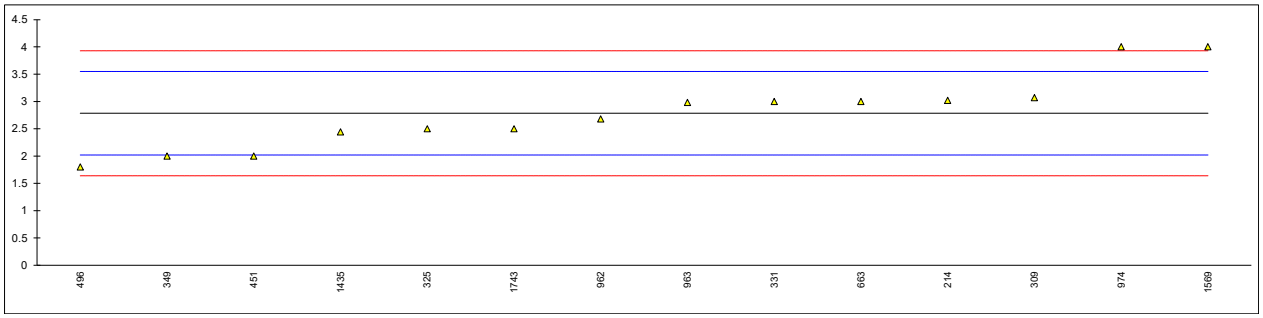


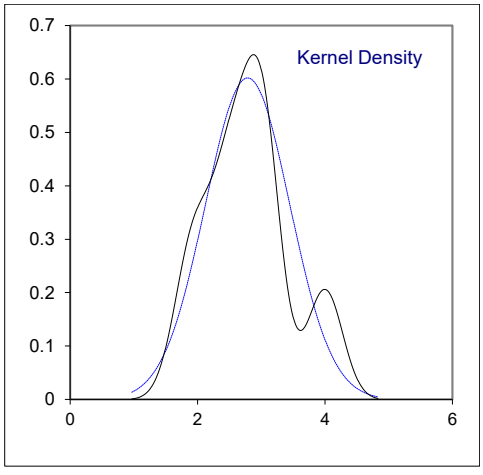
Determination of Calcium as Ca on sample #22032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214	D6595	3.02		0.61	
237		-----		-----	
257		-----		-----	
309	D5185	3.07		0.75	
325	D5185	2.5		-0.75	
331	D5185	3	C	0.56	first reported 5
349	D5185	2		-2.06	
381		-----		-----	
451	D5185	2		-2.06	
496	D5185	1.8		-2.58	
603		-----		-----	
633		-----		-----	
663	D5185	3.00		0.56	
862		-----		-----	
863		-----		-----	
901	D5185	<40		-----	
962	D5185	2.68		-0.28	
963	D5185	2.98		0.51	
974	D5185	4		3.18	
1146	D4951	<5		-----	
1435	D5185	2.442		-0.90	
1569	D5185	4		3.18	
1743	D5185	2.50		-0.75	
6190		-----		-----	

	normality	OK
	n	14
	outliers	0
	mean (n)	2.785
	st.dev. (n)	0.6629
	R(calc.)	1.856
	st.dev.(Horwitz)	0.3820
	R(Horwitz)	1.069
Compare		
	R(D5185:18)	0.057
	R(D6595:17)	1.466

application range D5185:18: 40 - 9000
application range D6595:17: 3.7 - 11460

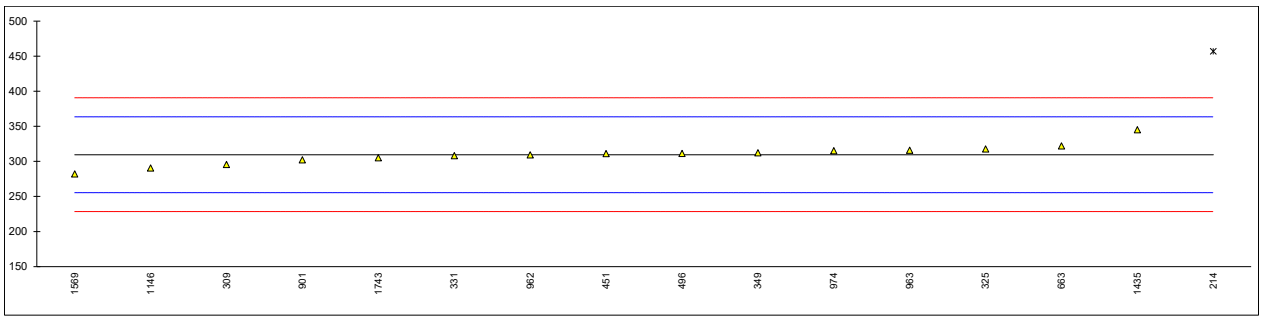


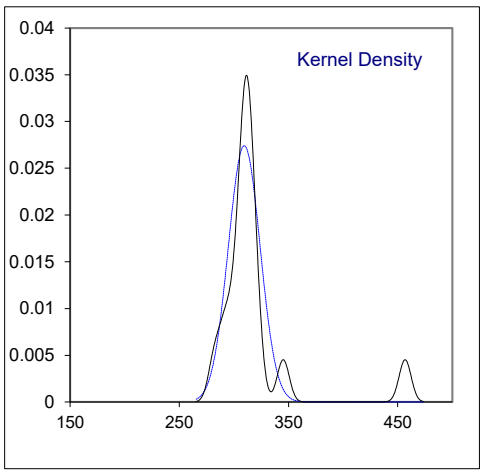


Determination of Phosphorus as P on sample #22032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214	D6595	456.805	G(0.01)	5.46	
237		-----		-----	
257		-----		-----	
309	D5185	295.5		-0.52	
325	D5185	317.5		0.30	
331	D5185	308		-0.05	
349	D5185	312		0.10	
381		-----		-----	
451	D5185	311		0.06	
496	D5185	311.1		0.06	
603		-----		-----	
633		-----		-----	
663	D5185	321.9		0.46	
862		-----		-----	
863		-----		-----	
901	D5185	302		-0.27	
962	D5185	309		-0.02	
963	D5185	315.75		0.23	
974	D5185	315		0.21	
1146	D4951	290.4		-0.70	
1435	D5185	345.127		1.32	
1569	D5185	282		-1.01	
1743	D5185	305		-0.16	
6190		-----		-----	

normality	not OK
n	15
outliers	1
mean (n)	309.418
st.dev. (n)	14.5489
R(calc.)	40.737
st.dev.(D5185:18)	27.0137
R(D5185:18)	75.638



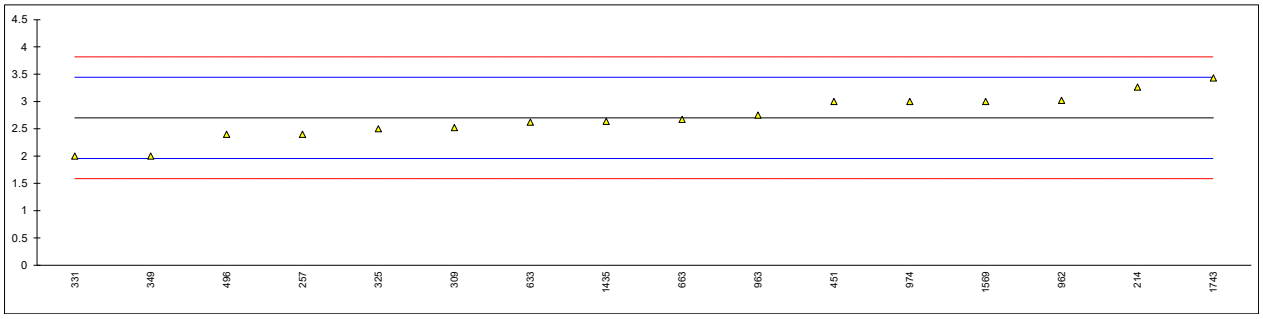


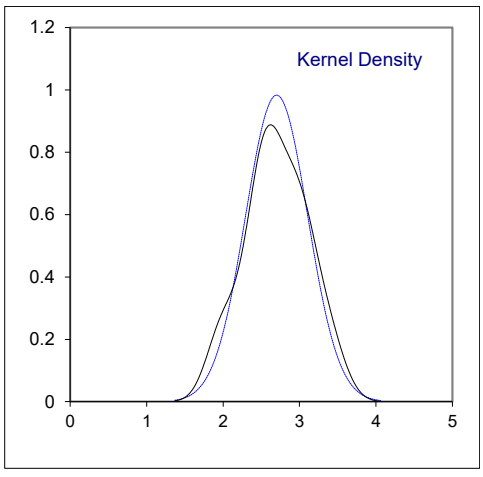
Determination of Zinc as Zn on sample #22032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
214	D6595	3.265		1.52	
237		-----		-----	
257	D6595	2.4		-0.81	
309	D5185	2.52		-0.49	
325	D5185	2.5		-0.54	
331	D5185	2	C	-1.88	first reported 4
349	D5185	2		-1.88	
381		-----		-----	
451	D5185	3		0.80	
496	D5185	2.4		-0.81	
603		-----		-----	
633	D6595	2.6225		-0.21	
663	D5185	2.67		-0.08	
862		-----		-----	
863		-----		-----	
901	D5185	<60		-----	
962	D5185	3.02		0.86	
963	D5185	2.75		0.13	
974	D5185	3		0.80	
1146	D4951	<5		-----	
1435	D5185	2.635		-0.18	
1569	D5185	3		0.80	
1743	D5185	3.43	C	1.96	first reported 4.81
6190		-----		-----	

	normality	OK
	n	16
	outliers	0
	mean (n)	2.701
	st.dev. (n)	0.4058
	R(calc.)	1.136
	st.dev.(Horwitz)	0.3721
	R(Horwitz)	1.042
Compare		
	R(D5185:18)	0.248
	R(D6595:17)	2.072

application range D5185:18: 60 - 1600
application range D6595:17: 5.3 - 1345





APPENDIX 2

Reported test results of other elements on sample #22032; results in mg/kg

lab	Barium as Ba	Cadmium as Cd	Chromium as Cr	Lead as Pb	Lithium as Li	Magnesium as Mg	Manganese as Mn
178	-----	-----	-----	-----	-----	-----	-----
179	-----	-----	-----	-----	-----	-----	-----
214	0.000	0.000	0.045	0.635	0.000	0.235	0.000
237	-----	-----	-----	-----	-----	-----	-----
257	0.1	0.3	0.2	0.0	0.0	0.3	1.5
309	0	0	0.13	0	0	0.14	0.45
325	<1	-----	<1	<1	<1	<1	<1
331	<2	<2	<2	<2	<2	<2	<2
349	0	0	0	0	0	0	1
381	-----	-----	-----	-----	-----	-----	-----
451	< 1	< 1	< 1	< 1	-----	< 1	1
496	<0.5	<1	<1	<1	0.94	<1	<1
603	-----	-----	-----	-----	-----	-----	-----
633	-----	-----	-----	0	0	-----	-----
663	<0.5	-----	<1	<10	-----	<5	<5
862	-----	-----	-----	-----	-----	-----	-----
863	-----	-----	-----	-----	-----	-----	-----
901	<0.5	-----	<1	<10	-----	<5	<5
962	<0.10	-----	0.35	0.15	-----	0.54	0.68
963	<0.100	<0.10	0.30	0.22	<0.10	0.33	0.59
974	<1	<1	<1	<1	<1	1	<1
1146	<5	-----	<1	<2	<1	<1	<1
1435	0.08	0.045	0.312	0.167	0.080	0.167	0.574
1569	<1	-----	1	<1	-----	<1	-----
1743	0.03	0.03	0.3	0.0	0.37	0.00	0.6
6190	-----	-----	-----	-----	-----	-----	-----

lab	Molybdenum as Mo	Nickel as Ni	Potassium as K	Silver as Ag	Sodium as Na		Titanium as Ti	Vanadium as V
178	-----	-----	-----	-----	-----		-----	-----
179	-----	-----	-----	-----	-----		-----	-----
214	0.000	0.200	0.06	0.000	1.81		0.000	0.685
237	-----	-----	-----	-----	-----		-----	-----
257	0.2	0.0	0.1	0.0	0.2		0.1	0.3
309	0	0.26	1.75	0.03	0		0	0
325	<1	<1	<2	<1	<2		<1	<1
331	<2	<2	<2	<2	<2		<2	<2
349	0	0	0	0	0		0	0
381	-----	-----	-----	-----	-----		-----	-----
451	< 1	< 1	< 1	< 1	< 1		< 1	< 1
496	<1	<1	1.4	<1	0.9		<1	<1
603	-----	-----	-----	-----	-----		-----	-----
633	-----	-----	-----	0.075	0.9925		-----	0
663	<5	<5	-----	<0.5	<7		<5	<1
862	-----	-----	-----	-----	-----		-----	-----
863	-----	-----	-----	-----	-----		-----	-----
901	<5	<5	<40	<0.5	<7		<5	<1
962	<0.10	0.23	-----	-----	0.34		<0.10	<0.10
963	0.11	0.14	0.53	<0.10	0.55		<0.10	<0.10
974	<1	<1	<1	<1	<1	C	<1	<1
1146	-----	<1	-----	<1	<4		<1	<1
1435	0.041	0.154	0.449	0.021	0.132		0.044	0.03
1569	<1	1	-----	<1	2		-----	<1
1743	0.1	0.1	0.5	0.0	0.0		0.0	0.0
6190	-----	-----	-----	-----	-----		-----	-----

Lab 974 first reported 8

APPENDIX 3

Number of participants per country

1 lab in	ALGERIA
2 labs in	BELGIUM
2 labs in	CHINA, People's Republic
2 labs in	FRANCE
1 lab in	GERMANY
1 lab in	MALAYSIA
2 labs in	NETHERLANDS
1 lab in	NIGERIA
1 lab in	PHILIPPINES
1 lab in	ROMANIA
2 labs in	SAUDI ARABIA
1 lab in	SERBIA
2 labs in	SPAIN
1 lab in	TANZANIA
1 lab in	THAILAND
1 lab in	TURKEY
1 lab in	UNITED ARAB EMIRATES
1 lab in	UNITED KINGDOM
2 labs in	UNITED STATES OF AMERICA

APPENDIX 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

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