Results of Proficiency Test
Gear Oil (used)
March 2020

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2017 the Institute for Laboratory Studies (iis) organizes a proficiency test for used Gear Oil. During the annual proficiency testing program 2019/2020 it was decided to continue the proficiency test for the analysis of used Gear Oil.

In this interlaboratory study 28 laboratories in 20 different countries registered for participation. See appendix 4 for the number of participants per country. In this report the results of the proficiency test for used Gear Oil are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send two different samples of used Gear Oil, one bottle of 0.5L labelled #20031 for various analyzes and one 50mL PE bottle labelled #20032 for metals determination only. The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 ACCREDITATION

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

2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

A batch of approximately 35 liters of used Gear Oil obtained from a third party was used for the preparation of the subsamples for the regular analyzes. After homogenization 50 amber glass bottles of 0.5 liter were filled and labelled #20031. The homogeneity of the subsamples #20031 was checked by determination of Density in accordance with ASTM D4052 and Water according to ASTM D6304 procedure C on 8 stratified randomly selected subsamples.

	Density at 15°C in kg/L	Water in mg/kg
Sample #20031-1	0.89206	190
Sample #20031-2	0.89203	217
Sample #20031-3	0.89202	178
Sample #20031-4	0.89201	193
Sample #20031-5	0.89205	219
Sample #20031-6	0.89205	196
Sample #20031-7	0.89203	186
Sample #20031-8	0.89204	215

Table 1: homogeneity test results of subsamples #20031

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

	Density at 15°C in kg/L	Water in mg/kg
r (observed)	0.00005	43.8
reference test method	ISO12185:96	ASTM D6304:16e1
0.3 * R (reference test method)	0.00015	121.5

Table 2: evaluation of the repeatabilities of subsamples #20031

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

Approximately 3 liters was taken from the remaining part of the batch used Gear Oil and spiked with various metals using Conostan standards: Aluminum, Copper, Silicon and Tin. After homogenization 50 PE bottles of 50mL were filled and labelled #20032.
The homogeneity of the subsamples #20032 was checked by determination of Copper as Cu in accordance with ASTM D5185 on 8 stratified randomly selected subsamples.

	Copper as Cu in mg/kg
Sample #20032-1	13.83
Sample #20032-2	13.41
Sample #20032-3	13.97
Sample #20032-4	13.76
Sample #20032-5	13.72
Sample #20032-6	13.63
Sample #20032-7	13.68
Sample #20032-8	13.41

Table 3: homogeneity test results of subsamples #20032

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.

	Copper as Cu in mg/kg
r (observed)	0.5
reference test method	ASTM D5185:18
0.3 * R (reference test method)	1.0

Table 4: evaluation of the repeatability of subsamples #20032

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

To each of the participating laboratories one sample labelled #20031 and one sample labelled #20032 were sent on February 26, 2020. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

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

2.6 ANALYZES

The participants were requested to determine on sample #20031: Total Acid Number, Density at 15°C, Flash Point PMcc, Kinematic Viscosity at 40°C and at 100°C, Viscosity Index, Membrane Filtration 5µm, Water and Level of Contamination (counts/mL and scale number).
On sample #20032: the elements Aluminum as Al, Barium as Ba, Boron as B, Cadmium as Cd, Chromium as Cr, Copper as Cu, Iron as Fe, Lead as Pb, Lithium as Li, Magnesium as Mg, Manganese as Mn, Molybdenum as Mo, Nickel as Ni, Potassium as K, Silicon as Si, Silver as Ag, Sodium as Na, Tin as Sn, Titanium as Ti, Vanadium as V, Calcium as Ca, Phosphorus as P, Zinc as Zn.

Also, some extra information was asked about the determination of Total Acid Number and level of Contamination.

It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the test results but report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

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

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendix 1 and 2 of this report. The laboratories are presented by their code numbers.

Directly after the deadline a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5).
For the statistical evaluation the unrounded (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers,

this check was repeated. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.
According to ISO5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT, the criterion of ISO13528, paragraph 9.2.1, was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

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

3.2 GRAPHICS

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

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

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

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

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

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

The z-scores were calculated according to:

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

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

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

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

4 EVALUATION

In this interlaboratory study some problems were encountered with the dispatch of the samples due to the COVID-19 pandemic. Two participants reported after the final reporting date and four participants did not report any test results at all. Not all laboratories were able to report all analyzes requested.
In total 24 participants reported 414 numerical test results. Observed were 25 outlying test results, which 6.0% of the numerical test results. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

Not all original data sets proved to have a normal Gaussian distribution. These are referred to as “not OK” or “suspect”. The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1 EVALUATION PER 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 5.

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

Sample #20031

Total Acid Number: This determination may be problematic dependent on the test method used. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D664-A:18e2 BEP 60mL, but is not in agreement with BEP 125mL, IP 60mL and IP 125mL. Remarkably, three participants still used pH 11 for BEP instead of pH 10. In test method ASTM D664:18e2 pH 10 is mentioned.

Density at 15°C: This determination was problematic. Two statistical outliers were observed and two other test results were excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of ISO12185:96.

Flash Point PMcc: Both procedures (A and B) of ASTM D93 may be applicable for this determination (in-use vs used lubricating oil). The majority of the participants used procedure A. Remarkably, one participant used procedure C which is applicable to Biodiesel (B100).
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-A:19.

Kinematic Viscosity at 40°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 ASTM D445:19a.

Kinematic Viscosity at 100°C: 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 D445:19a.

Viscosity Index: This determination was problematic. No statistical outliers were observed but two test results were excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of ASTM D2270:10(2016).

Membrane Filtration 5µm: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D4055:04(2019).

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

Level of Contamination: This determination was problematic. In total five statistical outliers were observed over six parameters (3 in counts/mL and 2 in scale number) and 4 other test results were excluded. The calculated reproducibilities after rejection of the suspect data are not in agreement with the requirements of ASTM D7647:10(2018). No clear conclusion could be drawn from the reported analytical details (appendix 3).

Sample #20032

Aluminum: 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: 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.

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

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

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

Tin: This determination was not problematic. 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: This determination may not be problematic Two statistical outliers were observed and one other test result was excluded. The consensus value was below the application range of ASTM D5185:18. Therefore, no z-scores were calculated.

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

Zinc: This determination may not be problematic. One statistical outlier was observed. The consensus value was below the application range of ASTM D5185:18. Therefore, no z-scores were calculated.

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

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant 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 literature reference test methods (in casu ASTM, EN test methods) or previous proficiency tests are presented in the next tables.

Parameter	unit	n	average	2.8 * sd	R(lit)
Total Acid Number	mg KOH/g	19	0.727	0.427	0.406
Density at 15°C	kg/L	18	0.89207	0.00065	0.0005
Flash Point PMcc	°C	18	193.5	14.3	13.7
Kinematic Viscosity at 40°C	mm^2/s	22	146.18	1.46	3.18
Kinematic Viscosity at 100°C	mm^2/s	21	14.420	0.257	0.167
Viscosity Index		19	96.47	3.08	2
Membrane Filtration 5µm	%M/M	5	0.014	0.013	0.044
Water	mg/kg	21	252	298	466
Level of contamination					
- ≥ 4µm (c)	counts/mL	9	49635	70092	56088
- ≥ 6µm (c)	counts/mL	9	5781	7550	4394
- ≥14µm (c)	counts/mL	9	379	794	512
- ≥ 4µm (c)	scale number	11	22.4	2.9	1.7
- ≥ 6µm (c)	scale number	11	19.3	2.5	1.2
- ≥14µm (c)	scale number	11	14.9	4.4	2.0

Table 5: reproducibilities of tests on sample #20031

Parameter	unit	n	average	2.8 * sd	R(lit)
Aluminum as Al	mg/kg	21	9.0	4.2	6.7
Boron as B	mg/kg	18	6.9	3.9	13.3
Copper as Cu	mg/kg	20	12.7	1.7	3.1
Iron as Fe	mg/kg	21	30.4	5.4	8.0
Silicon as Si	mg/kg	20	12.5	2.9	7.8
Tin as Sn	mg/kg	21	10.4	5.8	9.0
Calcium as Ca	mg/kg	17	2.8	2.9	(0.1)
Phosphorus as P	mg/kg	19	319	59	77
Zinc as Zn	mg/kg	20	3.4	2.8	(0.3)

Table 6: reproducibilities of tests on sample #20032

Without further statistical calculations it can be concluded that for many tests there is a good

compliance of the group of participants with the relevant reference test methods. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF MARCH 2020 WITH PREVIOUS PTS

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

Table 7: comparison with previous proficiency test

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

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

Parameter	March 2020	April 2019	April 2018	April 2017
Total Acid Number	+/-	-	-	-
Density at 15°C	-	-	-	-
Flash Point PMcc	+/-	-	-	+
Kinematic Viscosity at 40°C	++	+	+	++
Kinematic Viscosity at 100°C	-	-	--	+
Viscosity Index	-	+/-	--	+
Membrane Filtration 5µm	++	++	n.e.	n.e.
Water	+	+	++	++
Level of contamination				
counts/mL (≥4 / ≥6 / ≥14µm)	-	-	-	--
scale number (≥4 / ≥6 / ≥14µm)	--	-	-	--
Elements				
Aluminum as Al	+	+	+	n.e.
Boron as B	++	++	+	+
Chromium as Cr	n.e.	n.e.	++	n.e.
Copper as Cu	+	+	+	(-)
Iron as Fe	+	+	+	+
Silicon as Si	++	++	++	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

The following performance categories were used:

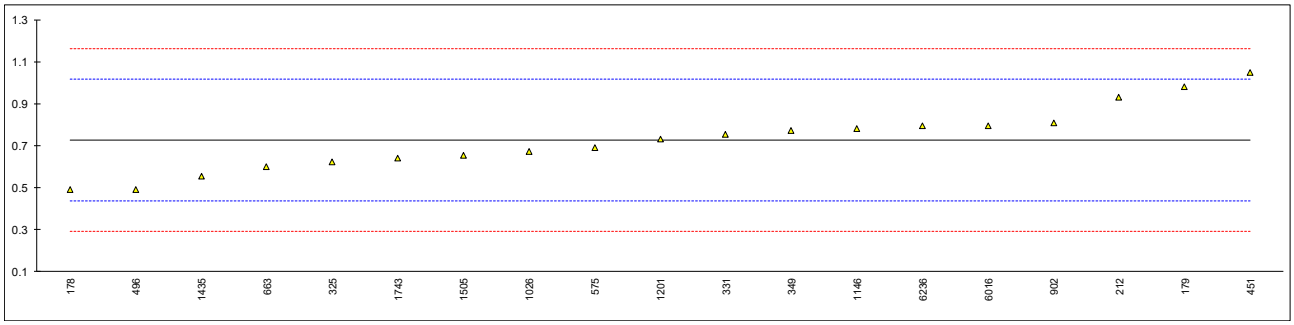
++	:	group performed much better than the reference test method
+	:	group performed better than the reference test method
+/-	:	group performance equals the reference test method
-	:	group performed worse than the reference test method
--	:	group performed much worse than the reference test method
n.e.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

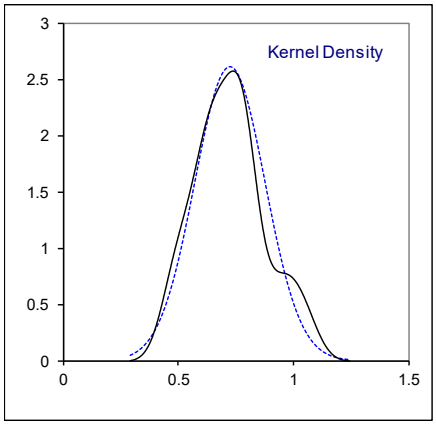
APPENDIX 1

Determination of Total Acid Number on sample #20031; results in mg KOH/g

lab	method	value	mark	z(targ)	End Point Det.	Tit.solv.vol.	remarks
178	D664-A	0.49		-1.63	Buffer End Point pH 11	60 mL	
179	D664-A	0.98	C	1.75	Inflection Point	125 mL	fr. 3.57
212	D664-A	0.93		1.40	Inflection Point	125 mL	
214		-----		-----	---	---	
237		-----		-----	---	---	
257		-----		-----	---	---	
325	D664-A	0.62		-0.74	Buffer End Point pH 10	125 mL	
331	D664Mod.	0.753		0.18	Inflection Point	60 mL	
349	D664-A	0.77		0.30	Buffer End Point pH 10	125 mL	
451	D664-A	1.05		2.23	Buffer End Point pH 10	60 mL	
496	D664-A	0.49		-1.63	Buffer End Point pH 10	60 mL	
575	D664-A	0.69		-0.25	Buffer End Point pH 10	60 mL	
633		-----		-----	---	---	
663	D664-A	0.60		-0.87	Buffer End Point pH 10	60 mL	
862		-----		-----	---	---	
863		-----		-----	---	---	
902	D664-A	0.81		0.58	Inflection Point	60 mL	
962		-----		-----	---	---	
963		-----		-----	---	---	
974		-----		-----	---	---	
1026	D664-A	0.67		-0.39	Buffer End Point pH 10	125 mL	
1146	D664-A	0.783		0.39	Buffer End Point pH 11	125 mL	
1201	D664-A	0.73		0.02	Buffer End Point pH 10	125 mL	
1435	D664-A	0.555		-1.18	---	---	
1505	D974	0.655		-0.49	---	---	
1743	D664-B	0.64		-0.60	Buffer End Point pH 11	60 mL	
6016	D664-A	0.795		0.47	---	---	
6236	D8045	0.793		0.46	Inflection Point	60 mL	

normality	OK
n	19
outliers	0
mean (n)	0.7265
st.dev. (n)	0.15238
R(calc.)	0.4267
st.dev.(D664-A:18e2, BEP 60mL)	0.14492
R(D664-A:18e2, BEP 60mL)	0.4058
compare	
R(D664-A:18e2, BEP 125mL)	0.2259
R(D664-A:18e2, IP 60mL)	0.3095
R(D664-A:18e2, IP 125mL)	0.1568

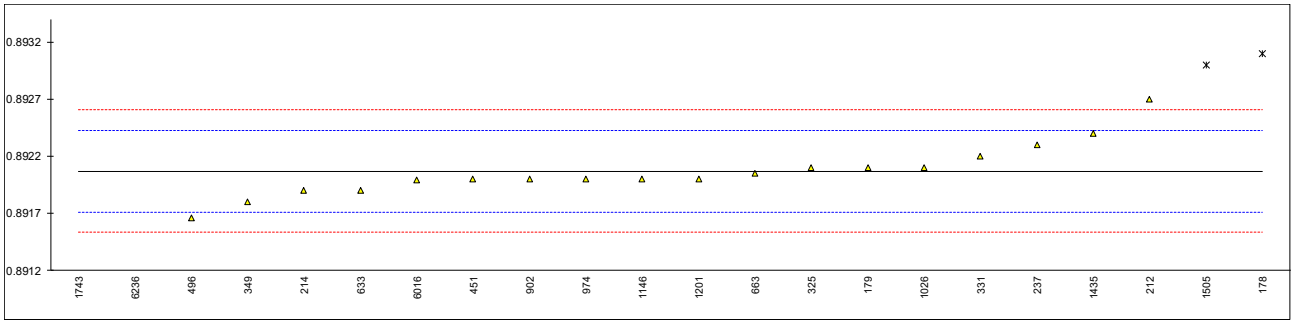


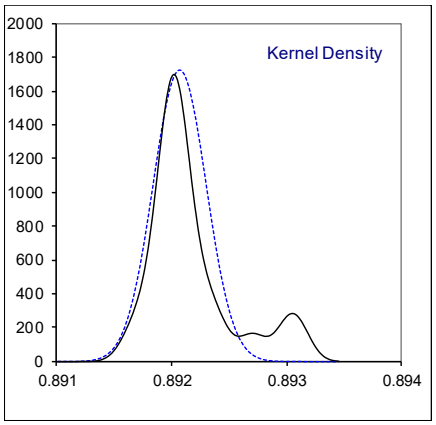


Determination of Density at 15°C on sample #20031; results in kg/L

lab	method	value	mark	z(targ)	remarks
178	D4052	0.8931	R(0.05)	5.79	
179	D4052	0.8921		0.19	
212	ISO12185	0.8927		3.55	
214	D7042	0.8919	C	-0.93	reported 0.8919 kg/m^3
237	D4052	0.8923		1.31	
257		-----		-----	
325	D4052	0.8921		0.19	
331	ISO12185	0.8922	C	0.75	reported 892.2 kg/L
349	D4052	0.8918		-1.49	
451	D4052	0.8920		-0.37	
496	ISO12185	0.89166		-2.28	
575		-----		-----	
633	D4052	0.8919		-0.93	
663	D4052	0.89205		-0.09	
862		-----		-----	
863		-----		-----	
902	D4052	0.8920		-0.37	
962		-----		-----	
963		-----		-----	
974	D4052	0.8920		-0.37	
1026	D4052	0.8921		0.19	
1146	D4052	0.8920		-0.37	
1201	ISO12185	0.8920		-0.37	
1435	D4052	0.8924		1.87	
1505	D7042	0.8930	R(0.05)	5.23	
1743		0.889	ex,C	-17.17	excluded as test result was rounded too much, first reported 891
6016	D4052	0.89199		-0.43	
6236	In house	0.890	ex	-11.57	excluded as test result was rounded too much

normality	not OK
n	18
outliers	2 (+2 ex)
mean (n)	0.892067
st.dev. (n)	0.0002311
R(calc.)	0.000647
st.dev.(ISO12185:96)	0.0001786
R(ISO12185:96)	0.0005

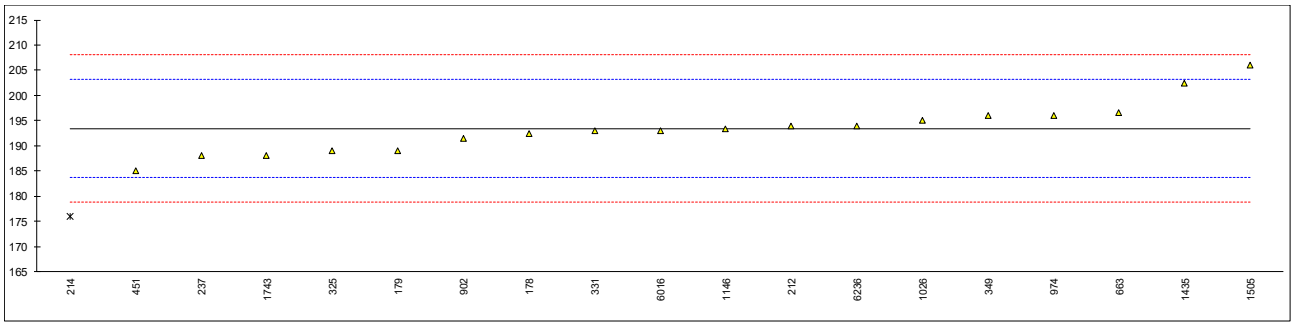


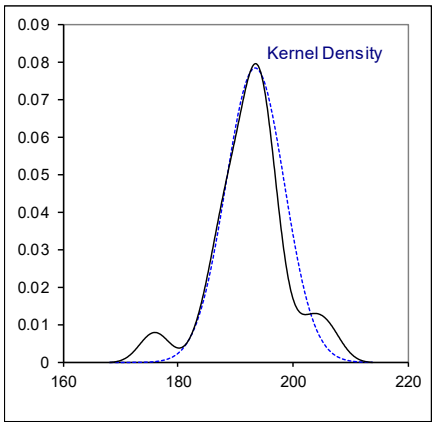


Determination of Flash Point PMcc on sample #20031; results in °C

lab	method	value	mark	z(targ)	remarks
178	D93-A	192.5		-0.20	
179	D93	189.0		-0.91	
212	D93-A	194.0		0.11	
214	D93-A	176	D(0.01)	-3.56	
237	D93-B	188		-1.11	
257		-----		-----	
325	D93-A	189		-0.91	
331	D93-A	192.9		-0.11	
349	D93-A	196		0.52	
451	D93-A	185.0		-1.72	
496		-----		-----	
575		-----		-----	
633		-----		-----	
663	D93-A	196.5		0.62	
862		-----		-----	
863		-----		-----	
902	D93-A	191.5		-0.40	
962		-----		-----	
963		-----		-----	
974	D93-A	196		0.52	
1026	D93-A	195.0		0.31	
1146	D93-A	193.4		-0.01	
1201		-----		-----	
1435	D93-A	202.5		1.84	
1505	D3828	206		2.56	
1743	ISO2719-A	188		-1.11	
6016	D6450	193	C	-0.09	first reported 218.7
6236	D93-C	194		0.11	

normality	suspect
n	18
outliers	1
mean (n)	193.46
st.dev. (n)	5.089
R(calc.)	14.25
st.dev.(D93-A:19)	4.906
R(D93-A:19)	13.74



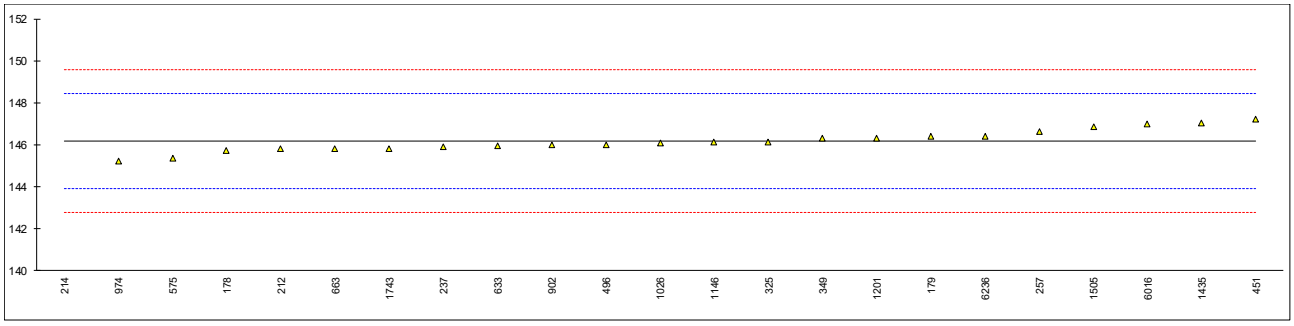


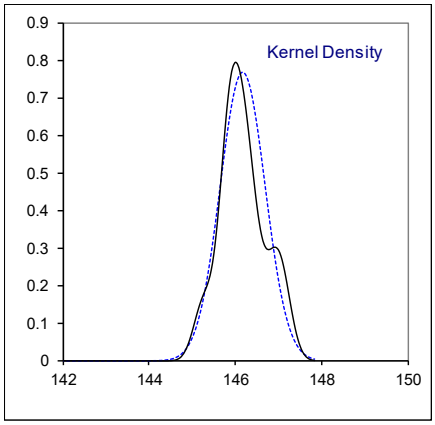
Determination of Kinematic Viscosity at 40°C on sample #20031; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178	D445	145.7		-0.42	
179	D445	146.4		0.19	
212	ISO3104	145.8		-0.34	
214	D7042	134.302	R(0.01)	-10.48	
237	D445	145.9		-0.25	
257	D7279 corrected to D445	146.63		0.40	
325	D445	146.15		-0.03	
331		-----		-----	
349	D445	146.3		0.11	
451	D7279 corrected to D445	147.2		0.90	
496	D445	146.01		-0.15	
575	D7279 corrected to D445	145.35		-0.73	
633	D445	145.952602		-0.20	
663	D445	145.8		-0.34	
862		-----		-----	
863		-----		-----	
902	D445	146.0		-0.16	
962		-----		-----	
963		-----		-----	
974	D445	145.2		-0.86	
1026	D445	146.1		-0.07	
1146	D445	146.13		-0.04	
1201	D445	146.3		0.11	
1435	D7042	147.02		0.74	
1505	D7042	146.84		0.58	
1743	D7279 corrected to D445	145.8		-0.34	
6016	D7042	146.990		0.71	
6236	D7279 corrected to D445	146.40		0.19	

	normality	OK
	n	22
	outliers	1
	mean (n)	146.181
	st.dev. (n)	0.5201
	R(calc.)	1.456
	st.dev.(D445:19a)	1.1339
	R(D445:19a)	3.175

R(D445:19a - used formulated oils)

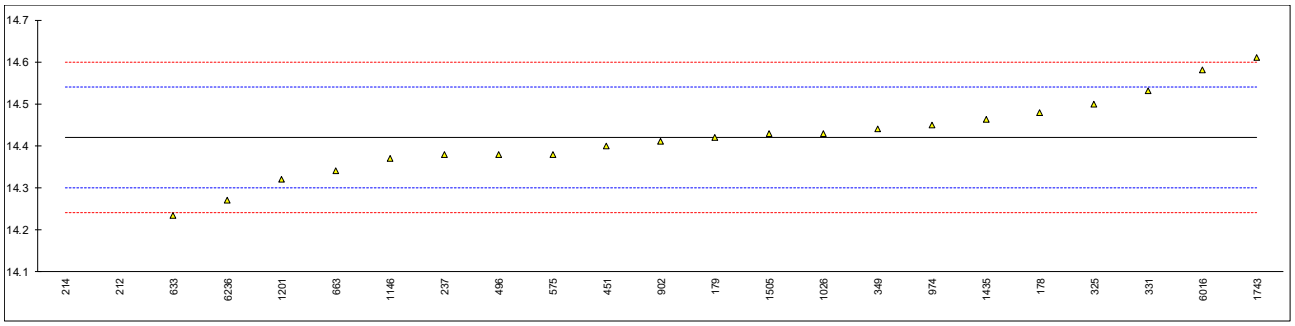


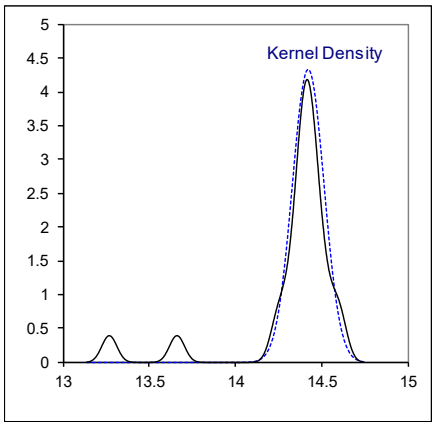


Determination of Kinematic Viscosity at 100°C on sample #20031; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
178	D445	14.48		1.01	
179	D445	14.42		0.00	
212	ISO3104	13.66	R(0.01)	-12.75	
214	D7042	13.268	R(0.01)	-19.33	
237	D445	14.38		-0.67	
257		-----		-----	
325	D445	14.50		1.34	
331	D7279Mod.	14.53		1.85	
349	D445	14.44		0.34	
451	D7279 corrected to D445	14.4		-0.33	
496	D445	14.380		-0.67	
575	D7279 corrected to D445	14.38		-0.67	
633	D445	14.2347964		-3.11	
663	D445	14.34		-1.34	
862		-----		-----	
863		-----		-----	
902	D445	14.41		-0.17	
962		-----		-----	
963		-----		-----	
974	D445	14.45		0.51	
1026	D445	14.43		0.17	
1146	D445	14.37		-0.84	
1201	D445	14.32		-1.68	
1435	D7042	14.462		0.71	
1505	D7042	14.429		0.15	
1743	D7279 corrected to D445	14.61		3.19	
6016	D7042	14.582		2.72	
6236	D7279 corrected to D445	14.27		-2.52	

normality	OK	
n	21	
outliers	2	
mean (n)	14.420	
st.dev. (n)	0.0918	
R(calc.)	0.257	
st.dev.(D445:19a)	0.0596	
R(D445:19a)	0.167	R(D445:19a - used formulated oils)

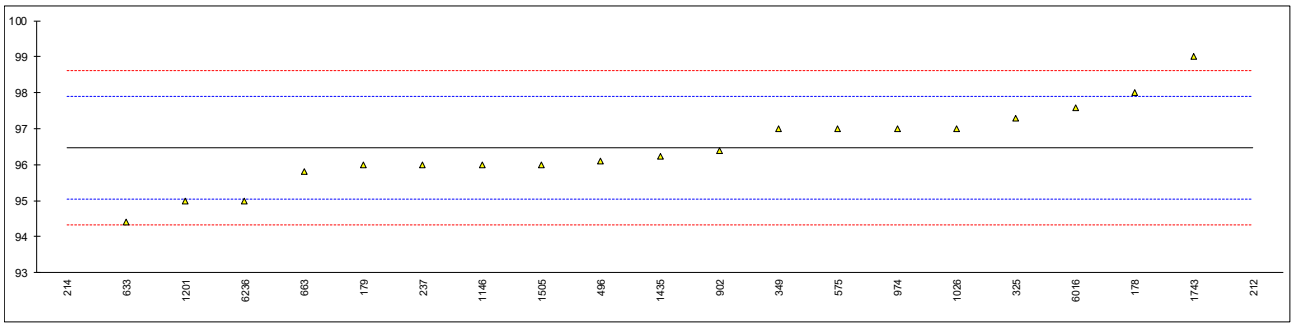


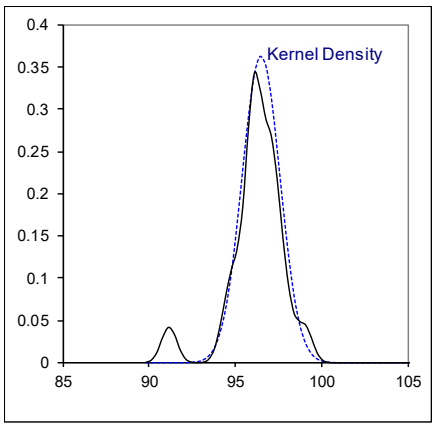


Determination of Viscosity Index on sample #20031

lab	method	value	mark	z(targ)	remarks
178	D2270	98		2.15	
179	D2270	96		-0.65	
212	D2270	173	ex, E	107.15	excluded as statistical outlier in KV 100°C, iis calculated 87
214	D2270	91.13	ex	-7.47	excluded as statistical outliers in KV 40°C and KV 100°C
237	D2270	96		-0.65	
257		-----		-----	
325	D2270	97.3		1.17	
331		-----		-----	
349	D2270	97		0.75	
451		-----		-----	
496	D2270	96.1		-0.51	
575	D2270	97		0.75	
633	D2270	94.415		-2.87	
663	D2270	95.82		-0.90	
862		-----		-----	
863		-----		-----	
902	D2270	96.4		-0.09	
962		-----		-----	
963		-----		-----	
974	D2270	97		0.75	
1026	D2270	97		0.75	
1146	D2270	96		-0.65	
1201	D2270	95		-2.05	
1435	D2270	96.225		-0.34	
1505	D2270	96		-0.65	
1743	ISO2909	99		3.55	
6016	D2270	97.579		1.56	
6236	D2270	95		-2.05	

normality	OK
n	19
outliers	0 (+2 ex)
mean (n)	96.47
st.dev. (n)	1.101
R(calc.)	3.08
st.dev.(D2270:10)	0.714
R(D2270:10)	2

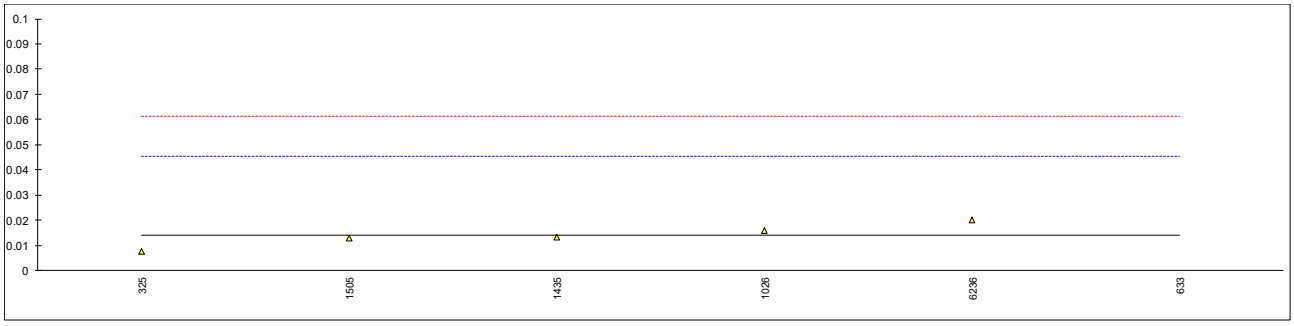




Determination of Membrane Filtration 5µm on sample #20031; results in %M/M

lab	method	value	mark	z(targ)	remarks
178		-----		-----	
179		-----		-----	
212		-----		-----	
214		-----		-----	
237		-----		-----	
257		-----		-----	
325	D4055	0.0077		-0.40	
331		-----		-----	
349		-----		-----	
451		-----		-----	
496		-----		-----	
575		-----		-----	
633	D4055	0.5381	D(0.01)	33.32	
663		-----		-----	
862		-----		-----	
863		-----		-----	
902		-----		-----	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1026		0.016		0.13	
1146		-----		-----	
1201		-----		-----	
1435	D4055	0.0132		-0.05	
1505	D4898	0.013		-0.06	
1743		-----		-----	
6016		-----		-----	
6236	D4055	0.020		0.38	

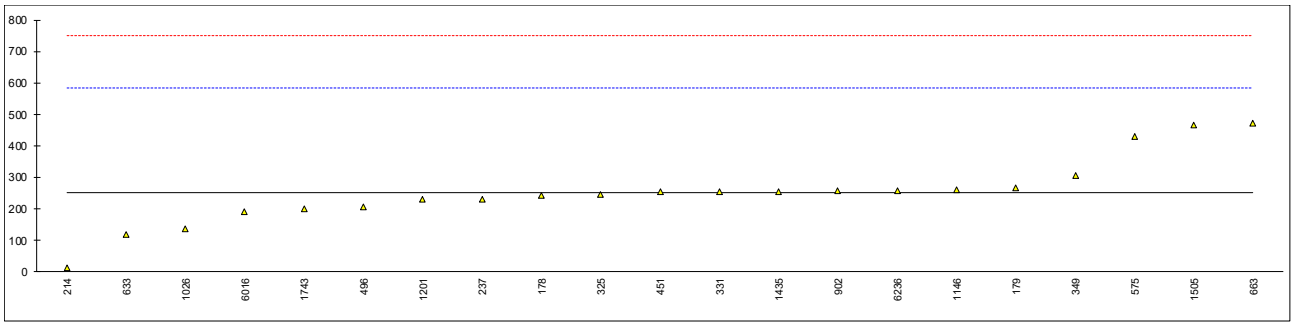
normality	unknown
n	5
outliers	1
mean (n)	0.0140
st.dev. (n)	0.00451
R(calc.)	0.0126
st.dev.(D4055:04)	0.01573
R(D4055:04)	0.0440

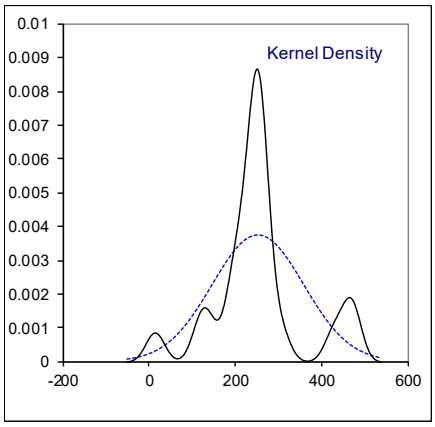


Determination of Water on sample #20031; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D6304-C	242		-0.06	
179	D6304	266		0.09	
212		-----		-----	
214	ISO12937	14		-1.43	
237	D6304-C	230.2		-0.13	
257		-----		-----	
325	D6304-C	245		-0.04	
331	INH-06	253.67		0.01	
349	D6304-C	305		0.32	
451	D6304-C	253		0.01	
496	D6304-C	206		-0.27	
575	D6304-A	428.29		1.06	
633	D6304-C	118.6		-0.80	
663	D6304-C	471		1.32	
862		-----		-----	
863		-----		-----	
902	D6304-C	257.1		0.03	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1026	D6304-C	137		-0.69	
1146	D6304-C	260		0.05	
1201	D6304-C	230		-0.13	
1435	D6304-A	255		0.02	
1505	D6304-A	467		1.29	
1743		200		-0.31	
6016	D6304-A	189.7		-0.37	
6236	D6304-A	258		0.04	

normality	suspect
n	21
outliers	0
mean (n)	251.74
st.dev. (n)	106.273
R(calc.)	297.57
st.dev.(D6304:16e1)	166.375
R(D6304:16e1)	465.85



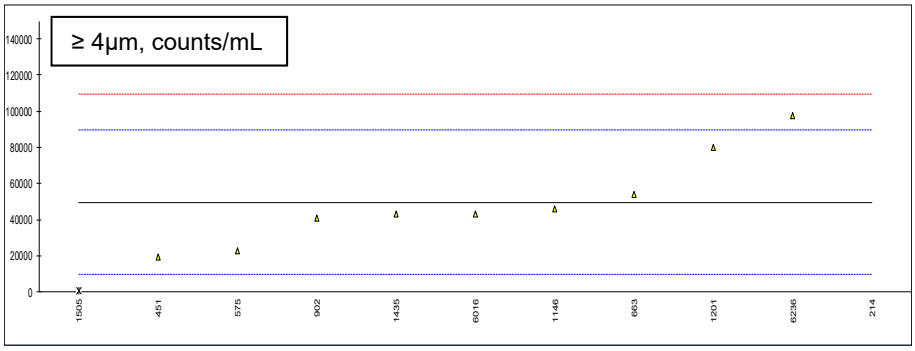


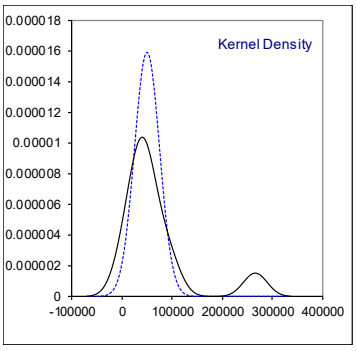
Determination of Level of Contamination on sample #20031; results in counts/mL

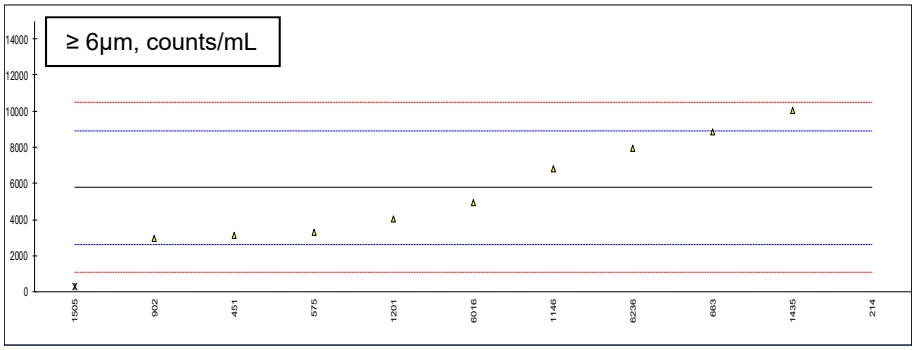
lab	method	≥ 4 µm	mark	z(targ)	≥ 6 µm	mark	z(targ)	≥ 14 µm	mark	z(targ)
178		-----		-----	-----		-----	-----		-----
179		-----		-----	-----		-----	-----		-----
212		-----		-----	-----		-----	-----		-----
214	ISO4406	265652.0	C,D(0.01)	10.78	71595.0	C,D(0.01)	41.94	5927.7	C,D(0.01)	30.35
237		-----		-----	-----		-----	-----		-----
257		-----		-----	-----		-----	-----		-----
325		-----		-----	-----		-----	-----		-----
331		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
451	D7647	19593		-1.50	3126		-1.69	169		-1.15
496		-----		-----	-----		-----	-----		-----
575	D7647	22717		-1.34	3311		-1.57	234		-0.79
633		-----		-----	-----		-----	-----		-----
663	D7647	54144		0.23	8831		1.94	551		0.94
862		-----		-----	-----		-----	-----		-----
863		-----		-----	-----		-----	-----		-----
902	D7647	40634		-0.45	2979		-1.79	120		-1.42
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
974		-----		-----	-----		-----	-----		-----
1026		-----		-----	-----		-----	-----		-----
1146	ISO11500	46000		-0.18	6800		0.65	720		1.86
1201	D7647	79793		1.51	4008		-1.13	80		-1.64
1435	D7647	43152.80		-0.32	10048.80		2.72	898.13		2.84
1505	D4898	749	ex	-2.44	292	ex	-3.50	52	ex	-1.79
1743		-----		-----	-----		-----	-----		-----
6016	ISO4406	43300		-0.32	4960		-0.52	318		-0.33
6236	D7647	97383.3		2.38	7968.8		1.39	322.7		-0.31
	normality	OK			OK			OK		
	n	9			9			9		
	outliers	1 (+1 ex)			1 (+1 ex)			1 (+1 ex)		
	mean (n)	49635			5781			379.2		
	st.dev. (n)	25033.0			2696.3			283.69		
	R(calc.)	70092			7550			794.3		
	st.dev.(D7647:10)	20031.4			1569.2			182.83		
	R(D7647:10)	56088			4394			511.9		

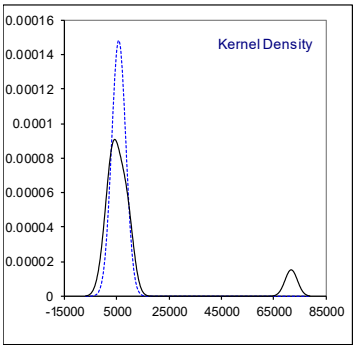
Lab 214: reported test results as scale

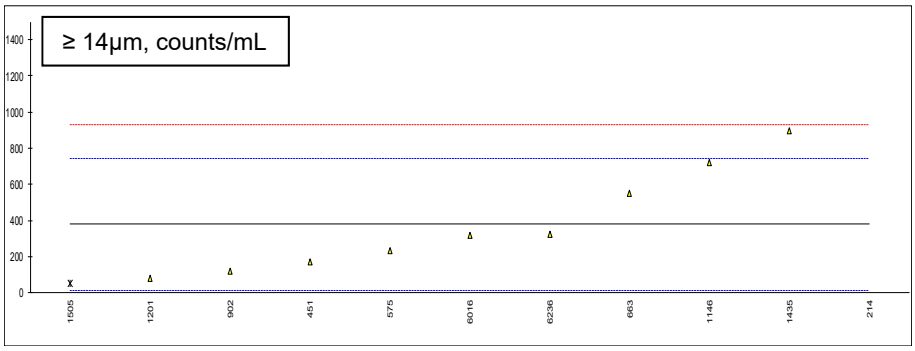
Lab 1505: test results were excluded as there are statistical outliers in scale

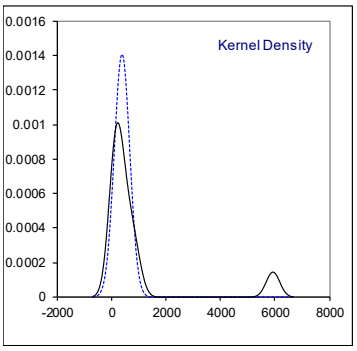

≥ 4µm, counts/mL
140000
120000
100000
80000
60000
40000
20000
0
1505
451
575
902
1435
6016
1146
663
1201
6236
214


Kernel Density
0.000018
0.000016
0.000014
0.000012
0.00001
0.000008
0.000006
0.000004
0.000002
0
-100000
0
100000
200000
300000
400000


≥ 6µm, counts/mL
14000
12000
10000
8000
6000
4000
2000
0
1505
902
451
575
1201
6016
1146
6236
663
1435
214


Kernel Density
0.00016
0.00014
0.00012
0.0001
0.00008
0.00006
0.00004
0.00002
0
-15000
5000
25000
45000
65000
85000


≥ 14µm, counts/mL
1400
1200
1000
800
600
400
200
0
1505
1201
902
451
575
6016
6236
663
1146
1435
214

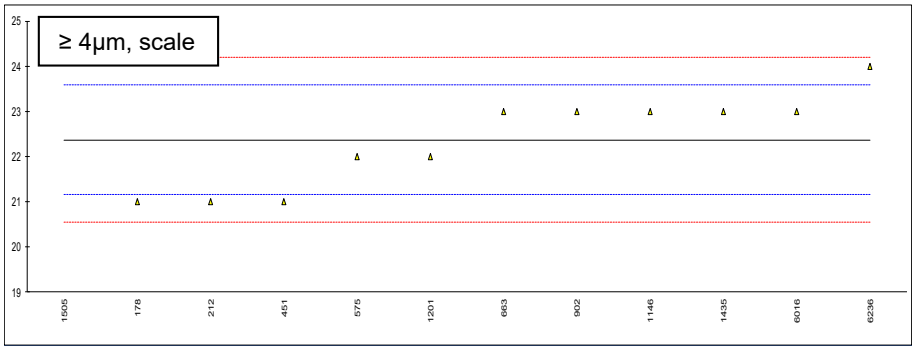

Kernel Density
0.0016
0.0014
0.0012
0.001
0.0008
0.0006
0.0004
0.0002
0
-2000
0
2000
4000
6000
8000

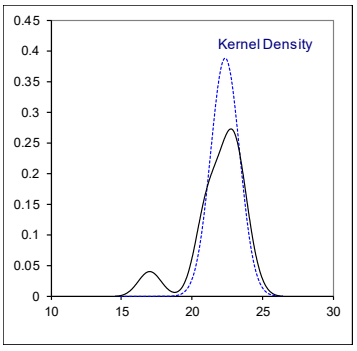
Determination of Level of Contamination acc. to ISO4406 scale on sample #20031; results in scale number

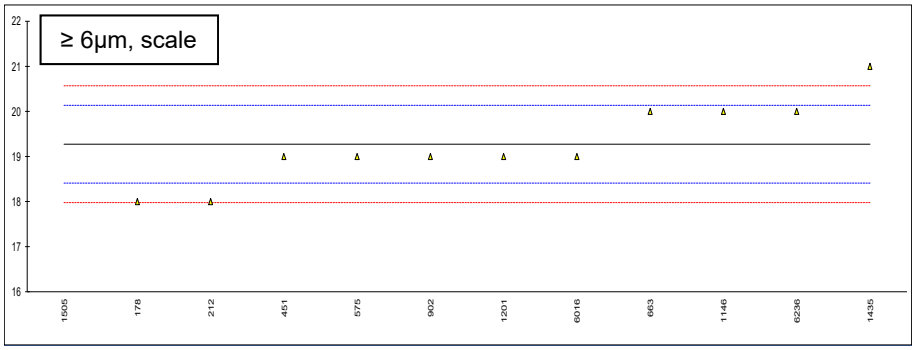
lab	method	≥ 4 µm	mark	z(targ)	≥ 6 µm	mark	z(targ)	≥ 14 µm	mark	z(targ)
178	ISO4406	21		-2.25	18		-2.97	13		-2.67
179		-----		-----	-----		-----	-----		-----
212	ISO4406	21		-2.25	18		-2.97	12		-4.07
214		-----		-----	-----		-----	-----		-----
237		-----		-----	-----		-----	-----		-----
257		-----		-----	-----		-----	-----		-----
325		-----		-----	-----		-----	-----		-----
331		-----		-----	-----		-----	-----		-----
349		-----		-----	-----		-----	-----		-----
451	ISO4406	21		-2.25	19		-0.64	15		0.13
496		-----		-----	-----		-----	-----		-----
575	ISO4406	22		-0.60	19		-0.64	15		0.13
633		-----		-----	-----		-----	-----		-----
663	D7647	23		1.05	20		1.70	16		1.53
862		-----		-----	-----		-----	-----		-----
863		-----		-----	-----		-----	-----		-----
902	ISO4406	23		1.05	19		-0.64	14		-1.27
962		-----		-----	-----		-----	-----		-----
963		-----		-----	-----		-----	-----		-----
974		-----		-----	-----		-----	-----		-----
1026		-----		-----	-----		-----	-----		-----
1146	ISO4406	23		1.05	20		1.70	17		2.93
1201	ISO4406	22	E	-0.60	19		-0.64	14	E	-1.27
1435	ISO4406	23		1.05	21		4.03	17		2.93
1505	ISO4406	17	D(0.01)	-8.83	15	D(0.01)	-9.97	13	ex	-2.67
1743		-----		-----	-----		-----	-----		-----
6016	ISO4406	23		1.05	19		-0.64	15		0.13
6236	ISO4406	24		2.70	20		1.70	16		1.53
	normality	OK			OK			OK		
	n	11			11			11		
	outliers	1			1			0 (+1 ex)		
	mean (n)	22.4			19.3			14.9		
	st.dev. (n)	1.03			0.90			1.58		
	R(calc.)	2.9			2.5			4.4		
	st.dev.(D7647:10)	0.61			0.43			0.71		
	R(D7647:10)	1.7			1.2			2.0		

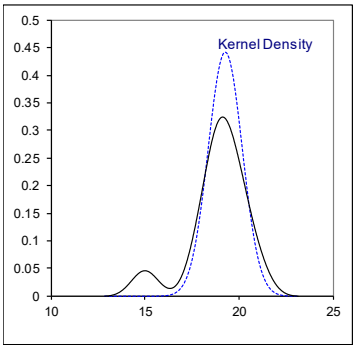
Lab 1201: iis calculated 23 and 13 respectively

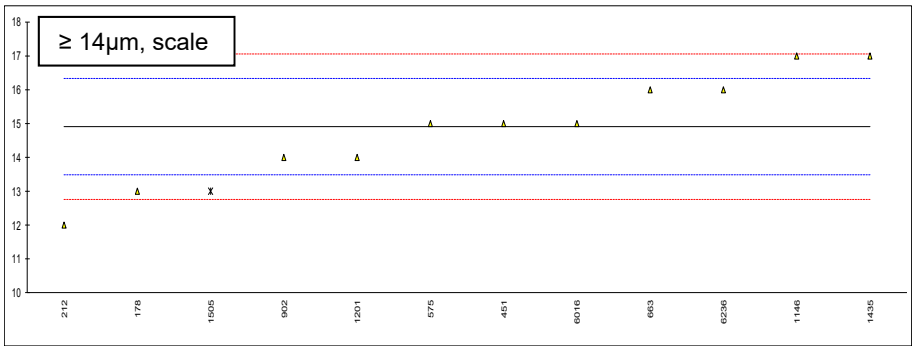
Lab 1505: test result was excluded as other two test results were statistical outliers

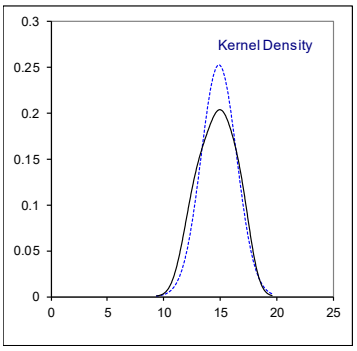

≥ 4µm, scale
1505
178
212
451
575
1201
663
902
1146
1435
6016
6236


Kernel Density


≥ 6µm, scale
1505
178
212
451
575
902
1201
6016
663
1146
6236
1435


Kernel Density

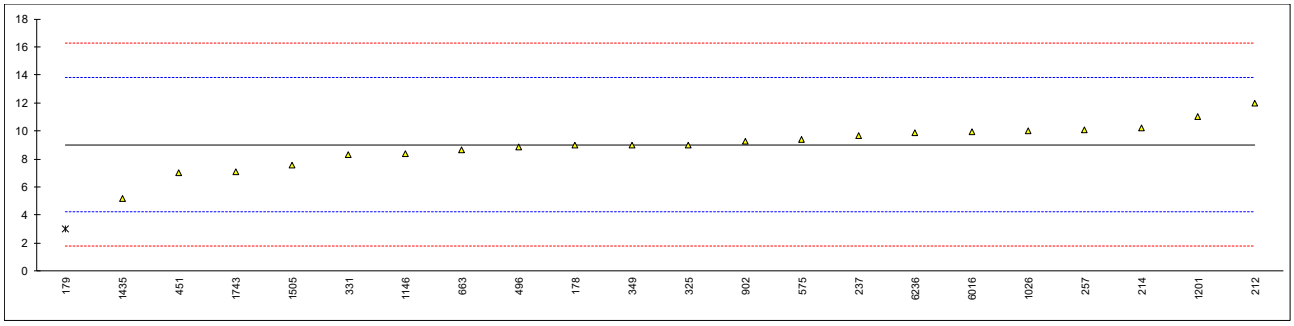

≥ 14µm, scale
212
178
1505
902
1201
575
451
6016
663
6236
1146
1435

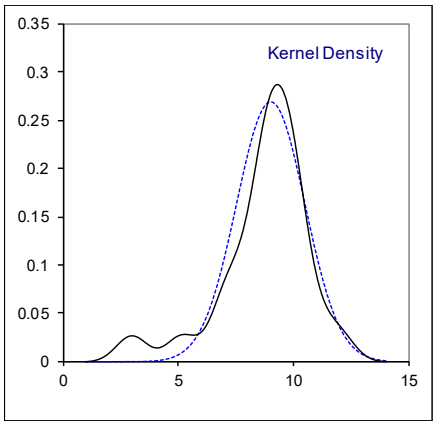

Kernel Density

Determination of Aluminum as Al on sample #20032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	9		-0.01	
179	D5185	3	R(0.05)	-2.51	
212	D5185	12		1.24	
214		10.215		0.50	
237	D5185	9.647		0.26	
257	D6595	10.06		0.43	
325	D5185	9		-0.01	
331	D5185Mod.	8.29		-0.30	
349	D5185	9		-0.01	
451	D5185	7		-0.84	
496	D5185	8.88		-0.06	
575	D6595	9.4		0.16	
633		-----		-----	
663	D5185	8.66		-0.15	
862		-----		-----	
863		-----		-----	
902	D5185	9.24		0.09	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1026	D5185	10		0.41	
1146	D4951	8.403		-0.26	
1201	D5185	11		0.82	
1435	D5185	5.178		-1.60	
1505	D5185	7.56		-0.61	
1743	D5185	7.10		-0.80	
6016	D5185	9.955		0.39	
6236	D5185	9.9		0.36	

normality	suspect
n	21
outliers	1
mean (n)	9.023
st.dev. (n)	1.4871
R(calc.)	4.164
st.dev.(D5185:18)	2.4045
R(D5185:18)	6.733

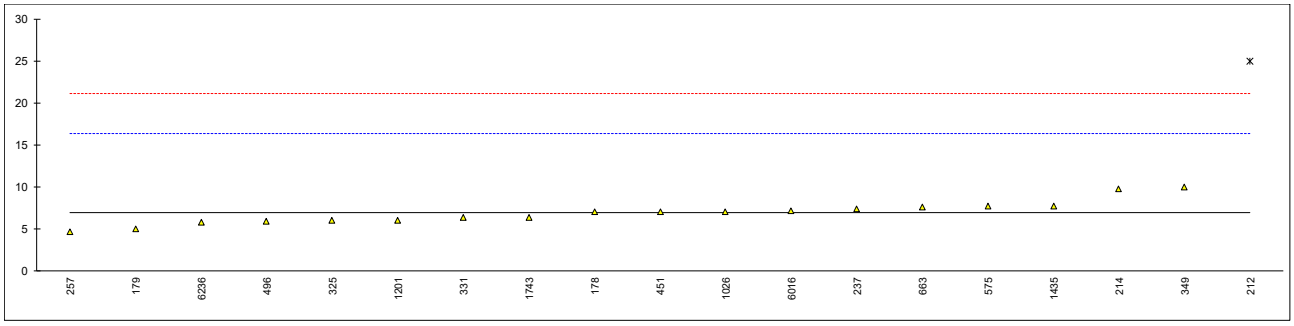


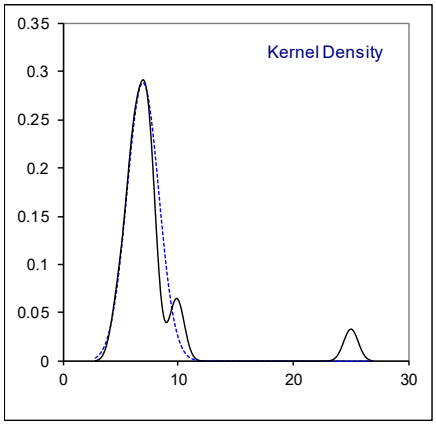


Determination of Boron as B on sample #20032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	7		0.02	
179	D5185	5		-0.40	
212	D5185	25	D(0.01)	3.82	
214		9.719		0.59	
237	D5185	7.362		0.09	
257	D6595	4.68		-0.47	
325	D5185	6		-0.19	
331	D5185Mod.	6.39		-0.11	
349	D5185	10		0.65	
451	D5185	7		0.02	
496	D5185	5.91		-0.21	
575	D6595	7.7		0.17	
633		-----		-----	
663	D5185	7.57		0.14	
862		-----		-----	
863		-----		-----	
902		-----		-----	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1026	D5185	7		0.02	
1146		-----		-----	
1201	D5185	6		-0.19	
1435	D5185	7.712		0.17	
1505		-----		-----	
1743	D5185	6.39		-0.11	
6016	D5185	7.206		0.06	
6236	D5185	5.8		-0.24	

normality	OK
n	18
outliers	1
mean (n)	6.913
st.dev. (n)	1.3828
R(calc.)	3.872
st.dev.(D5185:18)	4.7335
R(D5185:18)	13.254

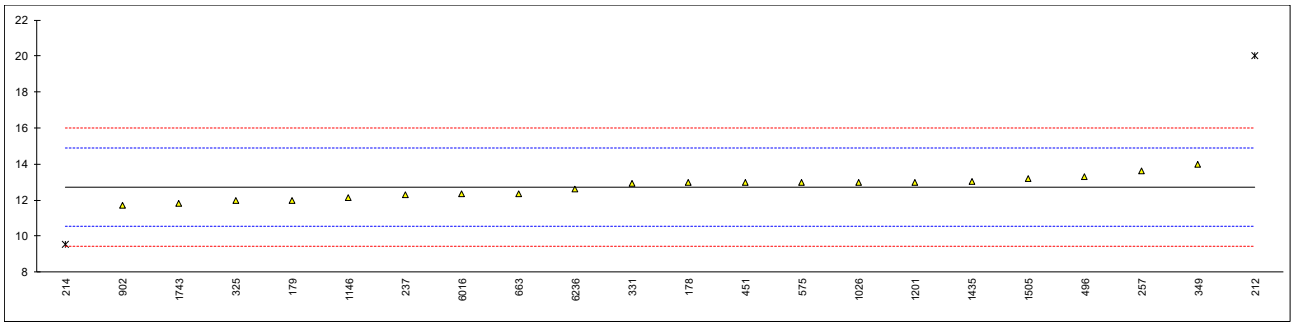


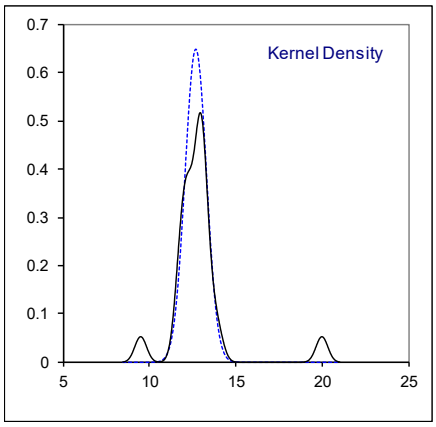


Determination of Copper as Cu on sample #20032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	13		0.26	
179	D5185	12		-0.66	
212	D5185	20	R(0.01)	6.68	
214		9.532	R(0.01)	-2.92	
237	D5185	12.29		-0.39	
257	D6595	13.61		0.82	
325	D5185	12		-0.66	
331	D5185Mod.	12.91		0.18	
349	D5185	14		1.18	
451	D5185	13		0.26	
496	D5185	13.3		0.54	
575	D6595	13		0.26	
633		-----		-----	
663	D5185	12.37		-0.32	
862		-----		-----	
863		-----		-----	
902	D5185	11.71		-0.92	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1026	D5185	13		0.26	
1146	D4951	12.15		-0.52	
1201	D5185	13		0.26	
1435	D5185	13.01		0.27	
1505	D5185	13.2		0.45	
1743	D5185	11.8		-0.84	
6016	D5185	12.343		-0.34	
6236	D5185	12.6		-0.11	

normality	OK
n	20
outliers	2
mean (n)	12.715
st.dev. (n)	0.6156
R(calc.)	1.724
st.dev.(D5185:18)	1.0898
R(D5185:18)	3.052

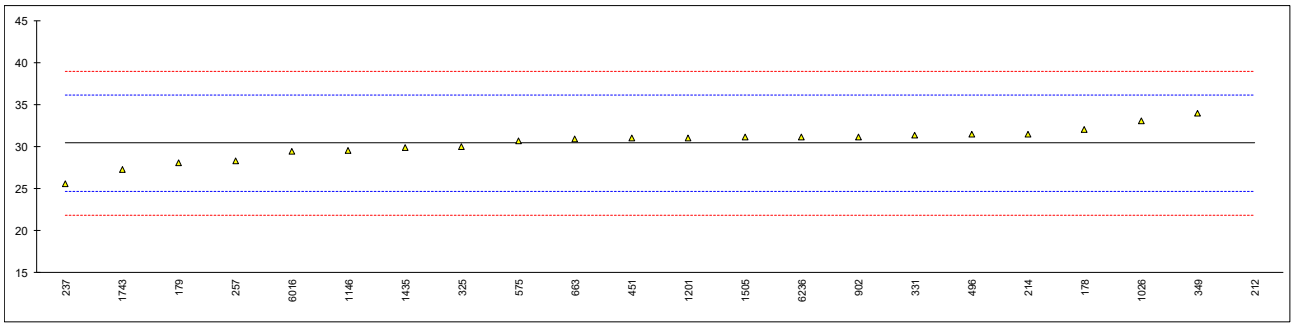


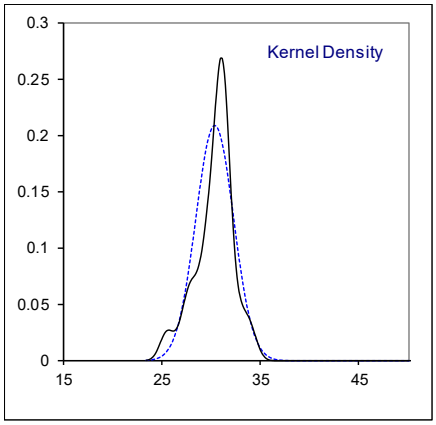


Determination of Iron as Fe on sample #20032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	32		0.56	
179	D5185	28		-0.84	
212	D5185	72	R(0.01)	14.59	
214		31.509		0.39	
237	D5185	25.5675		-1.69	
257	D6595	28.33		-0.73	
325	D5185	30		-0.14	
331	D5185Mod.	31.37		0.34	
349	D5185	34		1.26	
451	D5185	31		0.21	
496	D5185	31.48		0.38	
575	D6595	30.7		0.11	
633		-----		-----	
663	D5185	30.94		0.19	
862		-----		-----	
863		-----		-----	
902	D5185	31.11		0.25	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1026	D5185	33		0.91	
1146	D4951	29.53		-0.30	
1201	D5185	31		0.21	
1435	D5185	29.90		-0.17	
1505	D5185	31.1		0.25	
1743	D5185	27.3		-1.09	
6016	D5185	29.43		-0.34	
6236	D5185	31.1		0.25	

normality	suspect
n	21
outliers	1
mean (n)	30.398
st.dev. (n)	1.9147
R(calc.)	5.361
st.dev.(D5185:18)	2.8518
R(D5185:18)	7.985

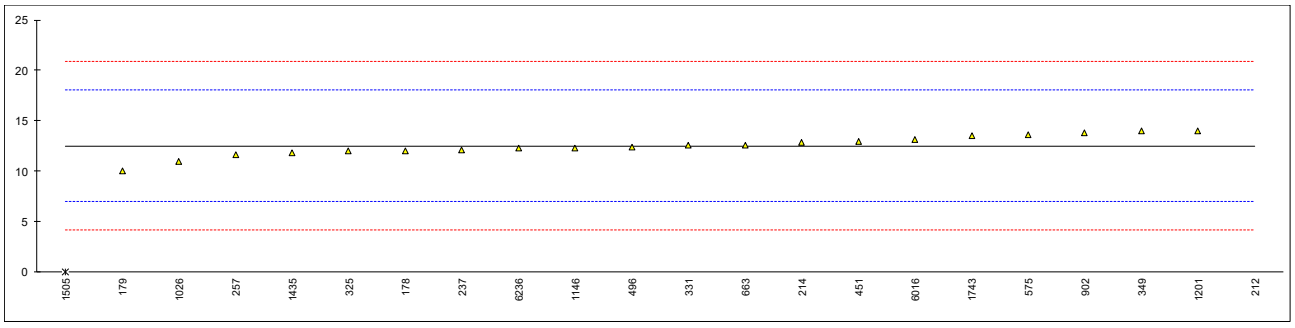


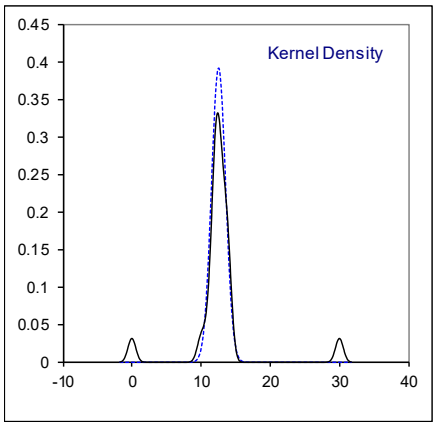


Determination of Silicon as Si on sample #20032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	12		-0.19	
179	D5185	10		-0.91	
212	D5185	30	R(0.01)	6.29	
214		12.833		0.11	
237	D5185	12.14		-0.14	
257	D6595	11.64		-0.32	
325	D5185	12		-0.19	
331	D5185Mod.	12.59		0.02	
349	D5185	14		0.53	
451	D5185	13		0.17	
496	D5185	12.4		-0.05	
575	D6595	13.6		0.38	
633		-----		-----	
663	D5185	12.59		0.02	
862		-----		-----	
863		-----		-----	
902	D5185	13.84		0.47	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1026	D5185	11		-0.55	
1146	D4951	12.31		-0.08	
1201	D5185	14		0.53	
1435	D5185	11.79		-0.27	
1505	D5185	0.0	R(0.01)	-4.51	
1743	D5185	13.5		0.35	
6016	D5185	13.11		0.21	
6236	D5185	12.3		-0.08	

normality	OK
n	20
outliers	2
mean (n)	12.532
st.dev. (n)	1.0181
R(calc.)	2.851
st.dev.(D5185:18)	2.7763
R(D5185:18)	7.774

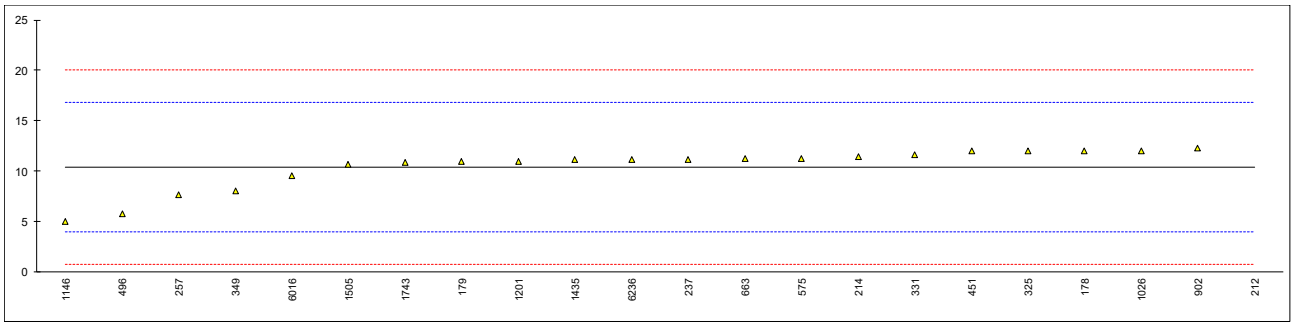


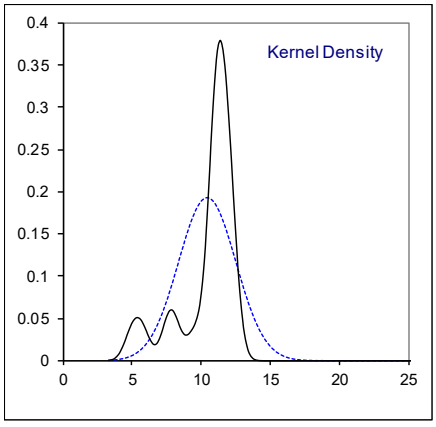


Determination of Tin as Sn on sample #20032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	12		0.49	
179	D5185	11		0.18	
212	D5185	36	R(0.01)	7.97	
214		11.478		0.33	
237	D5185	11.207		0.24	
257	D6595	7.63		-0.87	
325	D5185	12		0.49	
331	D5185Mod.	11.64		0.38	
349	D5185	8		-0.76	
451	D5185	12		0.49	
496	D5185	5.74	C	-1.46	first reported 0.59
575	D6595	11.3		0.27	
633		-----		-----	
663	D5185	11.27		0.26	
862		-----		-----	
863		-----		-----	
902	D5185	12.30		0.58	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1026	D5185	12		0.49	
1146	D4951	4.996		-1.69	
1201	D5185	11		0.18	
1435	D5185	11.17		0.23	
1505	D5185	10.7		0.08	
1743	D5185	10.9		0.15	
6016	D5185	9.518		-0.28	
6236	D5185	11.2		0.24	

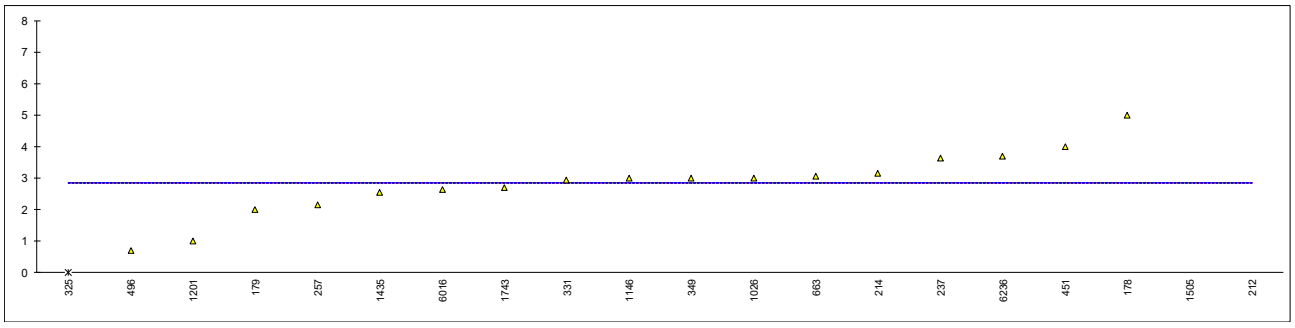
normality	suspect
n	21
outliers	1
mean (n)	10.431
st.dev. (n)	2.0757
R(calc.)	5.812
st.dev.(D5185:18)	3.2094
R(D5185:18)	8.986

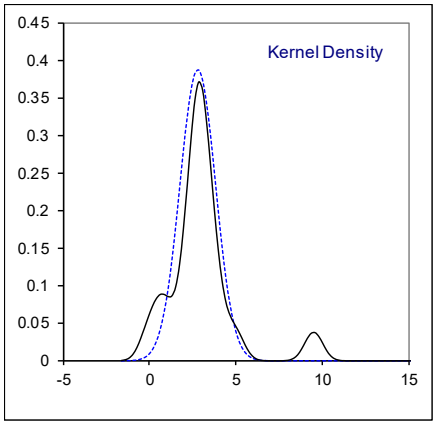




Determination of Calcium as Ca on sample #20032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	5		-----	
179	D5185	2		-----	
212	D5185	54	D(0.01)	-----	
214		3.157		-----	
237	D5185	3.631		-----	
257	D6595	2.15		-----	
325	D5185	0	ex,C	-----	first reported 8, test result excluded as 0 is not a real value
331	D5185Mod.	2.95		-----	
349	D5185	3		-----	
451	D5185	4		-----	
496	D5185	0.69		-----	
575		-----		-----	
633		-----		-----	
663	D5185	3.07		-----	
862		-----		-----	
863		-----		-----	
902	D5185	<40		-----	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1026	D5185	3		-----	
1146	D4951	2.993		-----	
1201	D5185	1		-----	
1435	D5185	2.559		-----	
1505	D5185	9.55	D(0.05)	-----	
1743	D5185	2.70	C	-----	first reported 6.84
6016	D5185	2.623		-----	
6236	D5185	3.7		-----	
	normality	suspect			
	n	17			
	outliers	2 (+1 ex)			
	mean (n)	2.837			
	st.dev. (n)	1.0293			
	R(calc.)	2.882			
	st.dev.(D5185:18)	(0.0208)			
	R(D5185:18)	(0.058)			application range: 40 – 9000 mg/kg
	compare				
	R(Horwitz)	(1.086)			

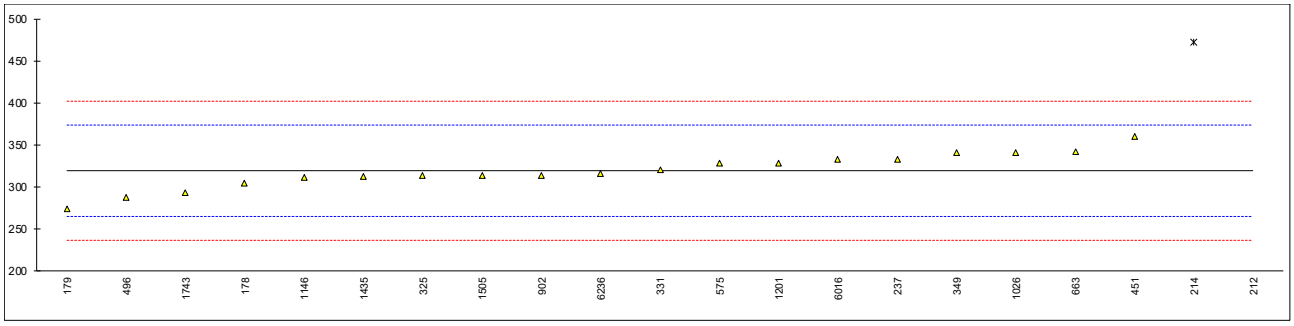


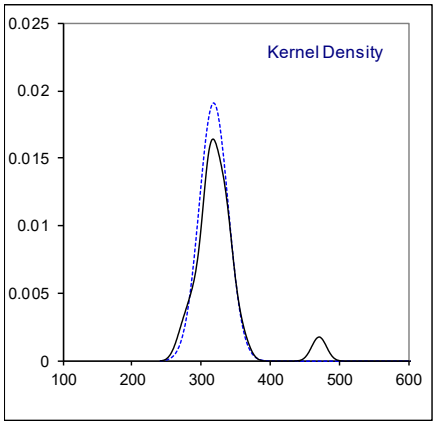


Determination of Phosphorus as P on sample #20032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	304		-0.56	
179	D5185	274		-1.65	
212	D5185	863	R(0.01)	19.81	
214		471.93	R(0.01)	5.56	
237	D5185	333		0.50	
257		-----		-----	
325	D5185	313		-0.23	
331	D5185Mod.	320.88		0.06	
349	D5185	341		0.79	
451	D5185	360		1.48	
496	D5185	287.5		-1.16	
575	D6595	327.97		0.32	
633		-----		-----	
663	D5185	342.38		0.84	
862		-----		-----	
863		-----		-----	
902	D5185	314		-0.19	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1026	D5185	341		0.79	
1146	D4951	311.3		-0.29	
1201	D5185	328		0.32	
1435	D5185	312.5		-0.25	
1505	D5185	313.7		-0.20	
1743	D5185	293		-0.96	
6016	D5185	332.7	C	0.49	first reported 409.6
6236	D5185	316.2		-0.11	

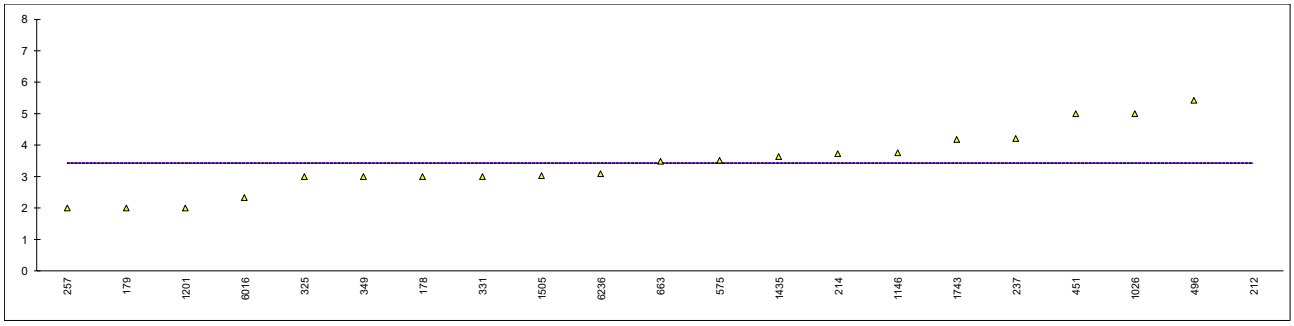
normality	OK
n	19
outliers	2
mean (n)	319.270
st.dev. (n)	20.8961
R(calc.)	58.509
st.dev.(D5185:18)	27.4403
R(D5185:18)	76.833

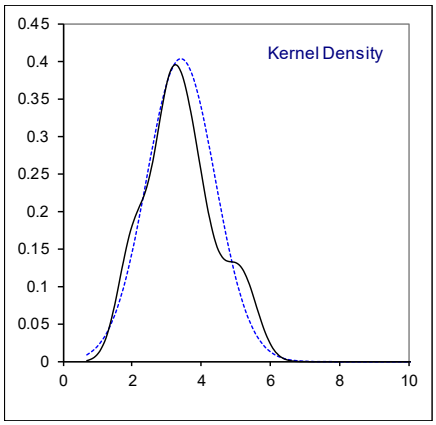




Determination of Zinc as Zn on sample #20032; results in mg/kg

lab	method	value	mark	z(targ)	remarks
178	D5185	3		-----	
179	D5185	2		-----	
212	D5185	21	R(0.01)	-----	
214		3.733		-----	
237	D5185	4.2		-----	
257	D6595	2.0		-----	
325	D5185	3		-----	
331	D5185Mod.	3.01		-----	
349	D5185	3		-----	
451	D5185	5		-----	
496	D5185	5.42	C	-----	first reported 0.31
575	D6595	3.5		-----	
633		-----		-----	
663	D5185	3.47		-----	
862		-----		-----	
863		-----		-----	
902	D5185	<60		-----	
962		-----		-----	
963		-----		-----	
974		-----		-----	
1026	D5185	5		-----	
1146	D4951	3.748		-----	
1201	D5185	2		-----	
1435	D5185	3.625		-----	
1505	D5185	3.04		-----	
1743	D5185	4.18		-----	
6016	D5185	2.335		-----	
6236	D5185	3.1		-----	
	normality	OK			
	n	20			
	outliers	1			
	mean (n)	3.418			
	st.dev. (n)	0.9896			
	R(calc.)	2.771			
	st.dev.(D5185:18)	(0.1146)			
	R(D5185:18)	(0.321)			application range: 60-1600 mg/kg
	compare				
	R(Horwitz)	(1.273)			





APPENDIX 2 Other reported test results

Reported test results of other elements on sample #20032; 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	0	1	0	0	-----	1	1
179	<1	<1	<1	<1	<1	<1	<1
212	10	14	11	26	0	15	22
214	0.000	0.000	0.052	0.630	0.002	0.210	0.000
237	<1	-----	<1	<1	-----	<1	1.10
257	0.1	0.0	0.21	0.0	-----	0.31	0.74
325	0	-----	0	0	-----	0	0
331	0.05	0.02	0.30	0.12	0.06	0.22	0.44
349	0	0	0	1	0	0	0
451	< 1	< 1	< 1	< 1	< 1	< 1	< 1
496	0.31	0.67	0.185	0.38	0.45	0.46	0.69
575	-----	-----	0.4	-----	-----	-----	1.1
633	-----	-----	-----	-----	-----	-----	-----
663	<0.5	-----	<1	<1	-----	<1	<1
862	-----	-----	-----	-----	-----	-----	-----
863	-----	-----	-----	-----	-----	-----	-----
902	<0.5	-----	<1	<10	-----	<5	<5
962	-----	-----	-----	-----	-----	-----	-----
963	-----	-----	-----	-----	-----	-----	-----
974	-----	-----	-----	-----	-----	-----	-----
1026	0	-----	0	0	-----	0	-----
1146	0.4633	-----	0.3243	0.2628	0.1497	0.5578	0.4789
1201	<1	<1	<1	<1	<1	<1	<1
1435	0.2149	0.2081	0.2876	0.2792	0.1944	0.1795	0.5253
1505	0.26	0.22	0.34	0.0	0.27	0.64	0.56
1743	0.05	0.01	0.29	0.11	0.59	0.23	0.40
6016	<1	-----	<1	<1	-----	<1	<1
6236	0.1	-----	0.4	1.0	0.2	0.3	0.4

lab	Molybdenum as Mo	Nickel as Ni	Potassium as K	Silver as Ag	Sodium as Na	Titanium as Ti	Vanadium as V
178	1	-----	0	0	1	0	0
179	<1	<1	<1	<0.10	<3	<1	<1
212	23	19	21	5	2	10	9
214	0.917	1.107	0.105	0.000	1.540	0.350	0.006
237	<1	<1	<1	<1	<1	-----	<1
257	0.0	0.14	0.19	0.07	0.81	0.08	0.02
325	0	0	0	0	0	0	0
331	0.01	0.07	0.44	0.01	0.21	0.04	0
349	0	0	0	0	0	0	0
451	< 1	< 1	1	< 1	1	< 1	< 1
496	<0.1	<0.1	0.93	<0.1	0.108 C	<0.1	<0.1
575	0.4	-----	-----	-----	-----	-----	-----
633	-----	-----	-----	-----	-----	-----	-----
663	<1	<1	1.78	<0.5	<1	<1	<1
862	-----	-----	-----	-----	-----	-----	-----
863	-----	-----	-----	-----	-----	-----	-----
902	<5	<5	-----	-----	-----	<5	<1
962	-----	-----	-----	-----	-----	-----	-----
963	-----	-----	-----	-----	-----	-----	-----
974	-----	-----	-----	-----	-----	-----	-----
1026	0	0	-----	-----	0	0	0
1146	-----	0.1359	-----	0.0476	0.4237	0.0699	0.0682
1201	<1	<1	<1	<1	<1	<1	<1
1435	0.0202	0.1764	0.1251	0.0694	0.2708	0.0696	0.0359
1505	0.0	0.14	-----	0.0	0.56	0.06	0.0
1743	0.08	0.02	0.00	0.00	1.38	0.04	0.01
6016	<1	<1	<1	-----	<1	<1	<1
6236	0.1	0.3	1.7	0.0	0.6	0.1	0.0

Lab 496 first reported 0.64

APPENDIX 3 Analytical details

Level of Contamination

lab	Sample rolled	Sample diluted	Dilution ratio	Manufacturer and the model of your test equipment
178	Yes	Yes	1:1	CINRG
179	---	---		
212	Yes	No		PAMAS Gepruft
214	Yes	No		PAMS 40
237	---	---		
257	---	---		
325	Yes	No		
331	---	---		
349	---	---		
451	No	No		
496	---	---		
575	No	Yes	50:50	PAMAS S40
633	---	---		
663	Yes	Yes	1:1	PAMAS model PAMAS SBSS-C
862	---	---		
863	---	---		
902	Yes	Yes	50 % sample to 50 % diluent, auto-diluting	The CINRG CS-APC-2 fully automated auto-diluting particle counting instrument with Klotz LDS Sensor, Klotz USB counter, Baumer UNKC sample level sensor, Gilson 215 liquid handler and CINRG Systems controller
962	---	---		
963	---	---		
974	---	---		
1026	No	---		
1146	No	---		Pamas SBSS-C HCB-LD-50/50
1201	---	---		
1435	---	---		
1505	---	---		
1743	---	---		
6016	---	---		
6236	Yes	Yes	automated dilution	CINRG LDS 4188 The CINRG equipment makes an automated dilution. it is equipped with a laser that determines the volume of oil in a cup with a max of 30ml. It makes an automated dilution up to 30 ml. Our technicians pour out between 10-15 ml of oil.

APPENDIX 4

Number of participants per country

1 lab in	ALGERIA
3 labs in	BELGIUM
2 labs in	CHINA, People's Republic
1 lab in	COLOMBIA
2 labs in	FRANCE
1 lab in	GERMANY
1 lab in	GREECE
1 lab in	KAZAKHSTAN
1 lab in	MOROCCO
3 labs in	NETHERLANDS
1 lab in	NIGERIA
1 lab in	PHILIPPINES
2 labs in	SAUDI ARABIA
1 lab 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 5

Abbreviations

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

Literature

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