Results of Proficiency Test
Hydraulic Fluid (used)
November 2013

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1   INTRODUCTION

Since 2003, the Institute for Interlaboratory Studies organized a proficiency test for the analysis of used Hydraulic Fluid every year. It was decided to continue this interlaboratory study during the annual program 2013/2014. In this interlaboratory study, 45 laboratories from 32 different countries have participated. See appendix 2 for the number of participants per country. In this report, the test results of the 2013 interlaboratory study on used hydraulic fluid are presented and discussed.

2   SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, The Netherlands, was the organiser of this proficiency test. Analysis for fit-for-use and homogeneity testing were subcontracted.
It was decided to send two different samples of used oils: one sample of 1 litre used H515 Hydraulic Fluid and one sample of 0.1 litre used H515 Hydraulic Fluid especially for wear metals. Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

2.1   QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system on IEC/ISO17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially 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 organisation 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 January 2010 (iis-protocol, version 3.2).

2.3   CONFIDENTIALITY STATEMENT

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

2.4 SAMPLES

The necessary bulk materials for the two samples of used Hydraulic Fluid were obtained from a participating laboratory. The approximately 60 litre of the first bulk material was homogenised in a precleaned drum. After homogenisation, 60 subsamples were transferred to 1 litre amber glass bottles, and labelled #13204. The homogeneity of the subsamples #13204 was checked by determination of Density in accordance with ASTM D4052:02e1 and Viscosity at 40ºC according to ASTM D445:12 on 8 stratified randomly selected samples.

	Density @15 ^{o}C in kg/L	Viscosity @40ºC in cSt
Sample #13204-1	0.87441	13.08
Sample #13204-2	0.87440	13.11
Sample #13204-3	0.87440	13.11
Sample #13204-4	0.87440	13.12
Sample #13204-5	0.87441	13.11
Sample #13204-6	0.87440	13.11
Sample #13204-7	0.87440	13.11
Sample #13204-8	0.87440	13.11

Table 1: homogeneity test results of subsamples #13204

From the test results of table 1, the repeatabilities were calculated and compared with 0.3 times the corresponding target reproducibility in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Density @15 ^{o}C in kg/L	Viscosity @40ºC in cSt
r (Observed)	0.00001	0.03
reference method	ISO12185:96	D445:12
0.3 * R (ref. method)	0.00015	0.03

Table 2: repeatabilities of subsamples #13204

The approximately 10 litre of the second bulk material, positive on a number of metals, was homogenised and was transferred to 100 subsamples of 100 mL HDPE containers, labelled #13205. The homogeneity of the subsamples #13205 was checked by determination of Density in accordance with ASTM D4052:11 and Zinc in accordance with ASTM D5185:13 on 8 stratified randomly selected samples.

	Density @ 15 ^{o}C in kg/L	Zinc in mg/kg
Sample #13205-1	0.88052	1190
Sample #13205-2	0.88052	1205
Sample #13205-3	0.88053	1200
Sample #13205-4	0.88052	1215
Sample #13205-5	0.88054	1215
Sample #13205-6	0.88053	1230
Sample #13205-7	0.88052	1225
Sample #13205-8	0.88053	1235

Table 3: homogeneity test results of subsamples #13205

From the test results of table 3, the repeatability was calculated and compared with 0.3 times the corresponding target reproducibility in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	Density @ 15 ^{o}C in kg/L	Zinc in mg/kg
r (Observed)	0.00002	43
reference method	ISO12185:96	D5185:13
0.3* R (ref. method)	0.00015	62

Table 4: repeatability of subsamples #13205

Each calculated repeatability was equal or less than 0.3 times the corresponding reproducibility of the respective test method. Therefore, homogeneity of the subsamples #13204 and #13205 was assumed.

To each of the participating laboratories was dispatched: One 1 litre amber glass bottle, labelled #13204 and one 100 mL HDPE container, labelled #13205 on October 23, 2013.

2.5 STABILITY OF THE SAMPLES

The stability of Hydraulic Fluid, packed in the brown glass bottles or in HDPE containers, was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYSES

The participants were asked to determine Total Acid Number, Density @ 15°C, Flash Point PMcc, Kinematic Viscosity @ 40°C and @ 100°C and Water on sample #13204 and 20 elements (17 wear metals and 3 additives) on sample #13205.

To get comparable results a detailed report form, on which the units were prescribed as well as some of the required standards and a letter of instructions were prepared and made available for download on the iis website (www.iisnl.com).
A SDS and a form to confirm receipt of the samples were added to the sample package.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were gathered. The original results are tabulated per determination in the appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline the available results were screened for suspect data. A 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 results. Additional or corrected data are put under 'Remarks' in the result tables in appendix 1.

Results that came in after deadline were not taken into account in the screening for suspect data and thus these participants were not requested for checks.

### 3.1	STATISTICS

The protocol followed in the organisation 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 January 2010 (iis-protocol, version 3.2).
For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. 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. After removal of outliers, this check was repeated. In case a data set does not have a normal distribution, the results of the statistical evaluation should be used with due care.
In accordance with ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests. Outliers are marked by D(0.01) for the Dixon test and by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test and by G(0.05) or DG(0.05) for the Grubbs test. Both outliers and stragglers were not included in the calculations of the averages and the 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. When the uncertainty passed the evaluation no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

Finally, the reproducibilities were calculated from the standard deviations by multiplying these 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 analysis results are plotted. The corresponding laboratory numbers are under 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 standard. 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 (see appendix 3, nos.13-14).

### 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 spread of this interlaboratory study.

The target standard deviation was calculated from the target reproducibility (preferably taken from a standardized test method) by division with 2.8.

The z-scores were calculated in accordance with:

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

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

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 the fit-for-useness of the reported test result.

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 proficiency test, no serious problems were encountered during dispatch and execution. Only one laboratory (Brazil) received the samples late. Six laboratories reported the results after the final reporting date and three other laboratories did not report any results at all.

The 42 reporting participants sent in 776 numerical results. Observed were 41 outlying results, which is 5.3% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

Not all original data sets proved to have a normal distribution. An anormal distribution was found for the following determinations: Density, Kinematic Viscosity at 40ºC, Barium, Chromium, Lead and Calcium. In these cases the statistical evaluations should be used with due care.

4.1   EVALUATION PER TEST

In this section, the results are discussed per test. The methods that are used by the various laboratories are taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the original data. The abbreviations, used in these tables, are listed in appendix 3.

One should be aware that the sample "metals only" (#13205) contained a large number of elements, spectral interferences might explain the large spread found for some elements.

Acid Number (Total): Regretfully, the consensus value of the group was below the application range (0.1 – 150 mg KOH/g) of ASTM D664:11a. Therefore, no significant conclusions were drawn.

Density @ 15°C: This determination was problematic for a number of laboratories. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO12185:96.

Flash Point PMcc: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D93:13 method B.

Kin.Visco.@ 40°C: This determination was very problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements of ASTM D445:12. When the ASTM D445 results are evaluated separately, the calculated reproducibility is again not at all in agreement with the requirements of the standard.

Kin.Visco.@ 100°C: This determination was very problematic. Five statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ASTM D445:12. When the ASTM D445 results are evaluated separately, the calculated reproducibility is somewhat smaller, but again not at all in agreement with the requirements of the standard.

Water: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility, after rejection of the statistical outliers is in good agreement with the requirements of ASTM D6304:07. The preferred method to use is ASTM D6304:07 method C. This method is applicable for oils with difficult matrix interferences (presence of additives). When the test results for ASTM D6304 method C were evaluated separately, the calculated reproducibility is somewhat smaller and again in agreement with the requirements of the test method.

Aluminium: Regretfully, the consensus value of the group was below the application range (6 – 40 mg/kg) of ASTM D5185:13. Therefore, no significant conclusions were drawn.

Barium: As the average concentration found by the group (254 mg/kg) is far above the application range given in ASTM D5185:13 table 3 (0.5 – 4 mg/kg), it was decided to use the estimated reproducibility calculated using the Horwitz equation instead of the reproducibility of ASTM D5185:13. This determination may not be problematic. Only one statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated requirements calculated using the Horwitz equation.

Chromium: This determination was problematic. Only one statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D5185:13.

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 D5185:13.

Iron: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D5185:13.

Lead: Regretfully, the consensus value of the group was below the application range (10 – 160 mg/kg) of ASTM D5185:13. Therefore, no significant conclusions were drawn.

Lithium: This determination was not problematic. One statistical outlier was observed and the calculated reproducibility after rejection of the statistical outlier is in agreement with the estimated requirements, calculated using the Horwitz equation.

Magnesium: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ASTM D5185:13.

Manganese: Regretfully, the consensus value of the group was below the application range (5 – 700 mg/kg) of ASTM D5185:13. Therefore, no significant conclusions were drawn.

Molybdenum   Regretfully, the consensus value of the group was below the application range (5 – 200 mg/kg) of ASTM D5185:13. Therefore, no significant conclusions were drawn.

Nickel: Regretfully, the consensus value of the group was below the application range (5 – 40 mg/kg) of ASTM D5185:13. Therefore, no significant conclusions were drawn.

Sodium: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D5185:13.

Silicon: Regretfully, the consensus value of the group was below the application range (8 – 50 mg/kg) of ASTM D5185:13. Therefore, no significant conclusions were drawn.

Silver: This determination was very problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ASTM D5185:13.

Tin: Regretfully, the consensus value of the group was below the application range (10 – 40 mg/kg) of ASTM D5185:13. Therefore, no significant conclusions were drawn.

Titanium: Regretfully, the consensus value of the group was below the application range (5 – 40 mg/kg) of ASTM D5185:13. Therefore, no significant conclusions were drawn.

Vanadium: This determination was very problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ASTM D5185:13.

Calcium: This determination was very problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ASTM D5185:13.

Phosphorus: This determination was very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of ASTM D5185:13.

Zinc: This determination was problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D5185:13.

4.2   PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and these parameters as found for the group of participating laboratories. The average results and the calculated reproducibilities are compared in the next tables with the reproducibilities, derived from literature standards (in casu the ASTM, IP, ISO and EN standards, see tables in appendix 1).

Parameter	unit	n	average	2.8 * sd	R(lit)
Total Acid Number	mg KOH/g	27	0.063	0.054	(0.028)
Density @ 15°C	kg/L	32	0.8743	0.0005	0.0005
Flash Point PMcc	^{o}C	32	94.4	6.9	10.0
Kinematic viscosity @ 40°C	mm/s^2	41	13.12	0.20	0.10
Kinematic viscosity @ 100°C	mm/s^2	32	4.806	0.065	0.037
Water	mg/kg	29	85.0	50.8	242.9

Table 5: reproducibilities of results of sample #13204.

Parameter	Unit	n	average	2.8 * sd	R (lit)
Aluminium as Al	mg/kg	29	2.70	1.97	(4.92)
Barium as Ba	mg/kg	28	90.6	22.9	20.6
Chromium as Cr	mg/kg	30	3.11	2.33	1.62
Copper as Cu	mg/kg	31	29.7	5.9	7.1
Iron as Fe	mg/kg	31	6.11	2.87	2.21
Lead as Pb	mg/kg	29	6.11	3.41	(5.76)
Lithium as Li	mg/kg	8	7.08	1.09	2.36
Magnesium as Mg	mg/kg	28	5.49	2.52	2.67
Manganese as Mn	mg/kg	24	2.45	1.92	(0.38)
Molybdenum as Mo	mg/kg	24	2.50	1.91	(1.23)
Nickel as Ni	mg/kg	28	2.30	1.59	(2.28)
Sodium as Na	mg/kg	25	7.74	5.23	4.70
Silicon as Si	mg/kg	27	4.78	4.53	(5.34)
Silver as Ag	mg/kg	26	2.86	2.43	1.00
Tin as Sn	mg/kg	25	2.32	2.67	(3.54)
Titanium as Ti	mg/kg	25	2.22	1.40	(3.64)
Vanadium as V	mg/kg	27	2.32	1.59	0.71
Calcium as Ca	mg/kg	30	389	125	35
Phosphorus as P	mg/kg	31	1169	328	147
Zinc as Zn	mg/kg	34	1272	250	216

Table 6: reproducibilities of results of sample #13205
results between brackets to be used with care, result was lower (or above) than application range of reference method

Without further statistical calculations, it could be concluded that for many tests there is a good compliance of the group of participating laboratories with the relevant standards. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2013 WITH THE PREVIOUS PTS.

	November 2013	November 2012	November 2011	November 2010
Number of reporting labs	42	40	36	30
Number of results reported	776	754	667	434
Statistical outliers	41	46	65	36
Percentage outliers	5.3%	6.1%	9.7%	8.3%

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 against the requirements of the respective standards. The conclusions are given the following table:

Determination	November 2013	November 2012	November 2011	November 2010
Total Acid Number	(--)	+/-	--	+
Density @ 15°C	+/-	--	+/-	--
Flash Point PMcc	++	-	+/-	++
Kinematic viscosity @ 40°C	--	--	--	--
Kinematic viscosity @ 100°C	--	--	--	--
Water	++	++	++	++
Aluminium as Al	(++)	++	+	++
Barium as Ba	-	(-)	(+)	(++)
Chromium as Cr	--	++	++	-
Copper as Cu	+	+/-	+	--
Iron as Fe	-	+/-	+/-	++
Lead as Pb	(++)	++	(+/-)	++
Lithium as Li	++	++	+	++
Magnesium as Mg	+	++	+/-	++
Manganese as Mn	(--)	--	--	-
Molybdenum	(--)	++	+	--
Nickel as Ni	(++)	++	++	++
Sodium as Na	-	--	(--)	++
Silicon as Si	+	++	(++)	++
Silver as Ag	--	--	+/-	++
Tin as Sn	(+)	++	(++)	++
Titanium as Ti	(++)	++	++	++
Vanadium as V	--	++	(-)	++
Calcium as Ca	--	--	(+/-)	(+)
Phosphorus as P	--	--	(-)	--
Zinc as Zn	-	--	(+/-)	--

Table 8: comparison determinations against the standard
results between brackets to be used with care, result was lower (or above) than application range of reference method.

++:   group performed much better than the standard
+ :   group performed better than the standard
+/-:   group performance equals the standard
- :   group performed worse than the standard
-- :   group performed much worse than the standard

APPENDIX 1

Determination of Acid Number (Total) on sample #13204; results in mg KOH/g.

lab	method	Value	mark	z(targ)	remarks
233		-----		-----	
255		-----		-----	
271		-----		-----	
311	D664	0.10		-----	
325	D664	0.055		-----	
331	D664	0.09		-----	
335	D664	0.06		-----	
340	D664	0.06		-----	
349	D664	0.03		-----	
360	D974	0.083		-----	
398	D664	0.0309		-----	
432		-----		-----	
451	D664	0.019		-----	
473	D664	0.0655		-----	
495	D664	0.074		-----	
496	D664	0.030		-----	
541	D664	<0.1		-----	
551		-----		-----	
562		-----		-----	
603	D664	0.0314		-----	
614	D664	0.12		-----	False positive result?
633	D664	0.083		-----	
634	D974	0.06		-----	
902	D664	0.073		-----	
912	D664	0.060		-----	
963	D664	0.06		-----	
1017		-----		-----	
1023	in house	<0.07		-----	
1059	ISO6619	0.07		-----	
1146	D664	0.055		-----	
1201	D664	0.05		-----	
1243	D664	0.08		-----	
1271	ISO6618	0.042		-----	
1328		-----		-----	
1403		-----		-----	
1628		-----		-----	
1660	IEC62021-1	0.064		-----	
1686		-----		-----	
1748	D664	0.093		-----	
1752		-----		-----	
1768	ISO6618	0.06190		-----	
1842		-----		-----	
1900	D664	0.08		-----	
1915		-----		-----	
7011	D664	0.0505		-----	

normality	OK	
n	29	
outliers	0	
mean (n)	0.063	
st.dev. (n)	0.0231	
R(calc.)	0.065	Compare R(D974:12) = 0.04
R(D664:11a)	(0.028)	Application range: 0.1 – 150 mg KOH/g
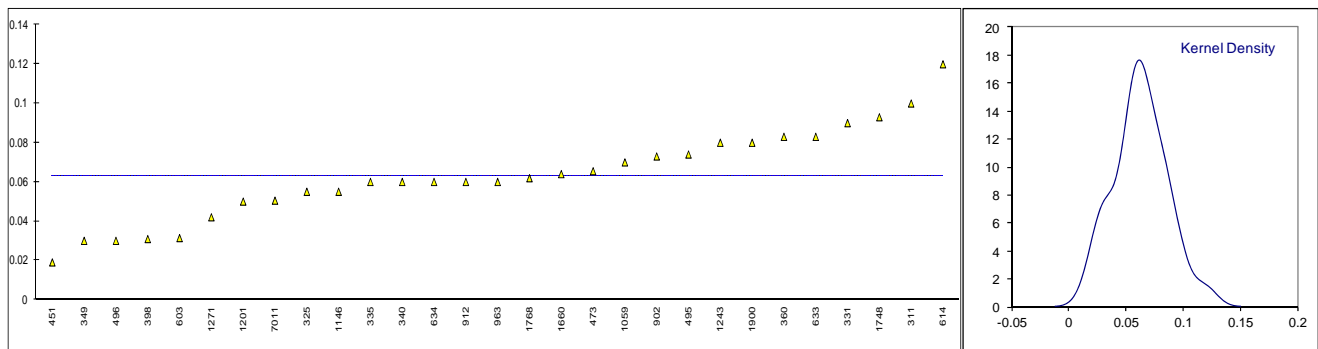
Determination of Density @ 15°C on sample #13204; results in kg/L.

lab	method	value	mark	z(targ)	remarks
233		-----		-----	
255		-----		-----	
271		-----		-----	
311	D4052	0.8744		0.48	
325	D4052	0.87438		0.36	
331	ISO12185	0.8745	C	1.04	First reported 874.5
335	D4052	0.8743		-0.08	
340	D4052	0.8744	C	0.48	First reported 874.4
349	D4052	0.8734	C,G(0.01)	-5.12	First reported 873.4
360	D4052	0.8743		-0.08	
398	D4052	0.8741		-1.20	
432	D4052	0.87455	C	1.32	First reported 0.84755
451	D4052	0.87415		-0.92	
473	D4052	0.8741		-1.20	
495	D4052	0.8742		-0.64	
496	D4052	0.87428		-0.20	
541	D4052	0.8743		-0.08	
551		-----		-----	
562	D4052	0.8743		-0.08	
603	D4052	0.87428		-0.20	
614	D4052	0.87403		-1.60	
633	D4052	0.8743		-0.08	
634	D4052	0.8744	C	0.48	First reported 0.8674
902	D4052	0.87430		-0.08	
912	D4052	0.8743		-0.08	
963	D4052	0.8744		0.48	
1017		-----		-----	
1023	D4052	0.8744		0.48	
1059	D4052	0.8742		-0.64	
1146	D4052	0.87438		0.36	
1201	D4052	0.8742		-0.64	
1243	D4052	0.8746	C	1.60	First reported 874.6
1271	D7042	0.8743	C	-0.08	First reported 874.3
1328	D4052	0.87444		0.70	
1403		-----		-----	
1628		-----		-----	
1660	D7042	0.8752	G(0.05)	4.96	
1686	D4052	0.8699	G(0.01)	-24.72	
1748	D4052	0.8740	C	-1.76	First reported 874.0
1752		-----		-----	
1768	ISO3675	0.8771	C,G(0.05)	15.60	First reported 0.873483
1842	D4052	0.8742		-0.64	
1900	D4052	0.8743		-0.08	
1915		-----		-----	
7011	D7042	0.8748		2.72	

normality	not OK	
n	32	
outliers	4	
mean (n)	0.87432	
st.dev. (n)	0.000163	
R(calc.)	0.00046	
R(ISO12185:96)	0.00050	
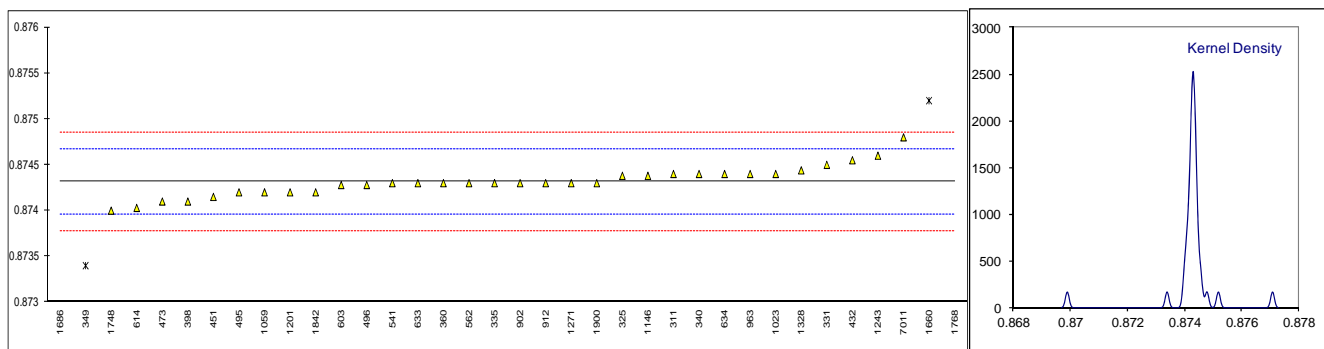
Determination of Flash Point PMcc on sample #13204; results in °C.

lab	method	value	mark	z(targ)	remarks
233		-----		-----	
255		-----		-----	
271	D93-A	95.8	C	0.38	First reported 83.8
311		-----		-----	
325	D93-A	93.0		-0.40	
331	D93-A	96.6		0.60	
335		-----		-----	
340	D93-A	112.5	G(0.01)	5.06	
349	D93-A	90		-1.24	
360	D93-A	97.0		0.72	
398	D93-A	94.6		0.04	
432	D93-A	94.7		0.07	
451	D93-A	98		1.00	
473	D93-A	-----	C	-----	First reported >230
495	D93-A	93.0		-0.40	
496	D93-A	90.8		-1.02	
541	D93-A	96.0		0.44	
551		-----		-----	
562	D93-A	93.15		-0.36	
603	D3828-B	94.0		-0.12	
614	D93-A	97.9		0.97	
633	D93-A	95.8075		0.38	
634	D93-A	91.0		-0.96	
902	D93-A	92.5		-0.54	
912		-----		-----	
963	D93-A	94.0		-0.12	
1017		-----		-----	
1023	D93-A	94		-0.12	
1059	ISO2719-A	100.0		1.56	
1146	in house-A	90.98		-0.97	
1201	D93-A	93.0		-0.40	
1243	D93	95		0.16	
1271	ISO2719-A	93.6		-0.24	
1328	D93-A	93.5		-0.26	
1403		-----		-----	
1628	ISO2719	99.0		1.28	
1660	D93-B	92.5		-0.54	
1686	D93-A	92.2		-0.63	
1748	D93-A	97		0.72	
1752		-----		-----	
1768		-----	W	-----	Result withdrawn, reported 104
1842	D93-A	92.5		-0.54	
1900	in house	97		0.72	
1915		-----		-----	
7011	D93-A	94		-0.12	

normality	OK	
n	32	
outliers	1	
mean (n)	94.44	
st.dev. (n)	2.479	
R(calc.)	6.94	
R(D93-B:13)	10.00	
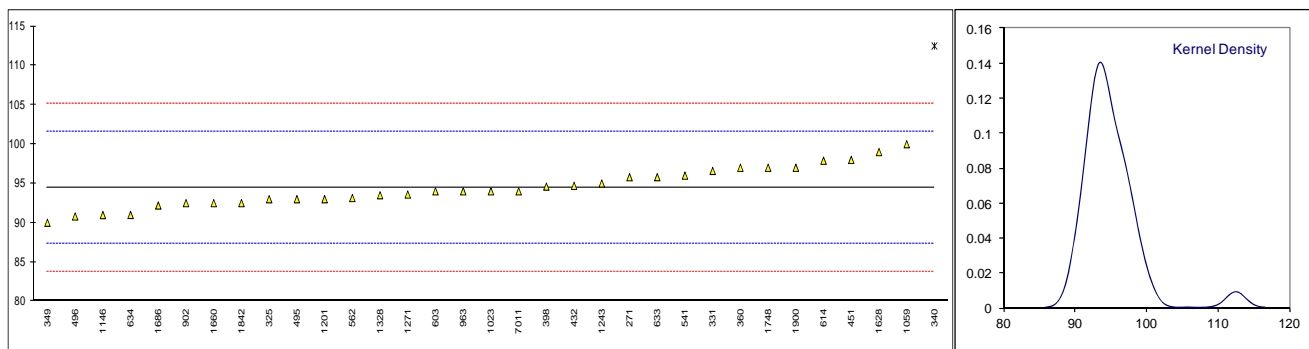
Determination of Kinematic Viscosity @ 40°C on sample #13204; results in mm/s^2.

lab	method	value	mark	z(targ)	remarks
233	D7279	13.00		-3.43	
255	D7279	13.232		3.09	
271	D445	13.11		-0.34	
311	D445	13.13		0.22	
325	D445	13.10		-0.62	
331	D7279	13.14		0.50	
335	D445	13.020		-2.86	
340	D445	13.145		0.64	
349	D445	13.27	C	4.15	First reported 12.27
360	D445	13.131		0.25	
398	D445	13.10		-0.62	
432	D445	13.11		-0.34	
451	D7279	13.16		1.07	
473	D445	13.254		3.71	
495	D445	13.16		1.07	
496	D445	13.000		-3.43	
541	D445	13.07		-1.46	
551		-----		-----	
562	D7279	13.07		-1.46	
603	D7042	13.077		-1.26	
614	D445	13.2		2.19	
633	D445	12.9846	C	-3.86	First reported 4.6432
634	D445	13.12		-0.06	
902	D7279	13.12		-0.06	
912	D445	13.09		-0.90	
963	D445	13.14		0.50	
1017		-----		-----	
1023	D445	13.11		-0.34	
1059	ISO3104	13.13		0.22	
1146	D445	13.139		0.48	
1201	D445	13.11		-0.34	
1243	D445	13.18		1.63	
1271	D7042	13.124		0.06	
1328	D445	13.07		-1.46	
1403		-----		-----	
1628	ISO3104	13.143		0.59	
1660	D7042	13.088		-0.96	
1686	D445	13.14		0.50	
1748	D445	13.32		5.56	
1752	D445	13.24		3.31	
1768	ISO3104	13.077521		-1.25	
1842	IP71	13.11		-0.34	
1900	D445	13		-3.43	
1915		-----		-----	
7011	D7042	13.088		-0.96	

				Only ASTM D445 / ISO3104 data
normality	not OK			not OK
n	41			31
outliers	0			0
mean (n)	13.122			13.126
st.dev. (n)	0.0725			0.0762
R(calc.)	0.203			0.213
R(D445:12)	0.100			0.100
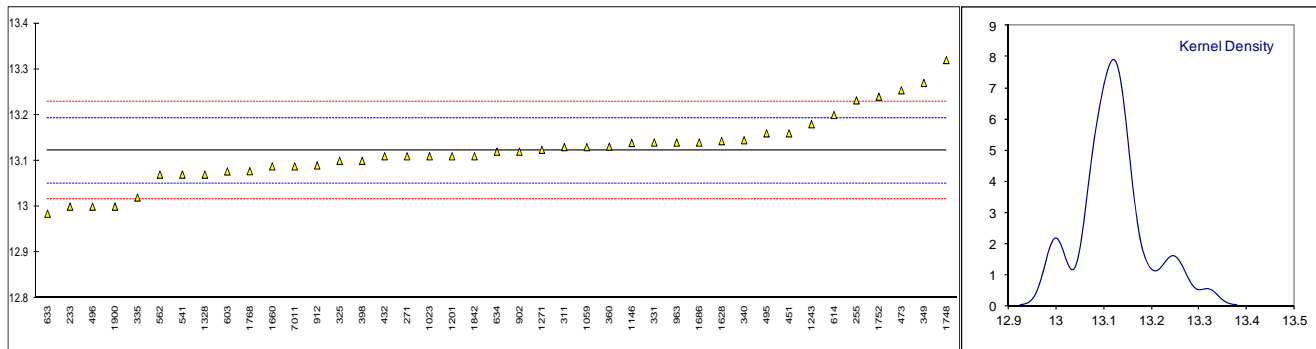
Determination of Kinematic Viscosity @ 100°C on sample #13204; results in mm/s^2

lab	method	value	mark	z(targ)	remarks
233	D7279	4.70	C,DG(0.05)	-8.13	First reported 4.70
255	D7279	4.829		1.76	
271	D445	4.790		-1.23	
311	D445	4.789		-1.31	
325	D445	4.800		-0.46	
331	D7279	4.86		4.14	
335		-----		-----	
340		-----		-----	
349	D445	4.846		3.06	
360	D445	4.8256		1.50	
398	D445	4.753	C	-4.07	First reported 4.645
432	D445	4.802		-0.31	
451	D7279	4.84		2.60	
473	D445	4.830		1.84	
495	D445	4.8116		0.43	
496	D445	4.7980		-0.62	
541	D445	4.779		-2.07	
551		-----		-----	
562	D7279	4.529	G(0.01)	-21.24	
603	D7042	4.8236		1.35	
614		-----		-----	
633	D445	4.6432	C,G(0.01)	-12.48	First reported 12.9846
634	D445	4.822		1.22	
902	D7279	4.897	G(0.05)	6.97	
912	D445	4.801		-0.39	
963	D445	4.774		-2.46	
1017		-----		-----	
1023	D445	4.78		-2.00	
1059	ISO3104	4.790		-1.23	
1146	D445	4.8091		0.23	
1201	D445	4.781		-1.92	
1243	D445	4.81		0.30	
1271	D7042	4.7976		-0.65	
1328	D445	4.807		0.07	
1403		-----		-----	
1628	ISO3104	4.806		0.00	
1660	D7042	4.829		1.76	
1686	D445	4.83		1.84	
1748	D445	4.804		-0.16	
1752	D445	4.804		-0.16	
1768		-----		-----	
1842	IP71	4.784		-1.69	
1900	D445	4.71	DG(0.05)	-7.36	
1915		-----		-----	
7011	D7042	4.788		-1.38	

					Only ASTM D445 / ISO3104 data:
normality	OK				OK
n	32				25
outliers	5				2
mean (n)	4.8060				4.8011
st.dev. (n)	0.02313				0.02055
R(calc.)	0.0648				0.0575
R(D445:12)	0.0365				0.0365
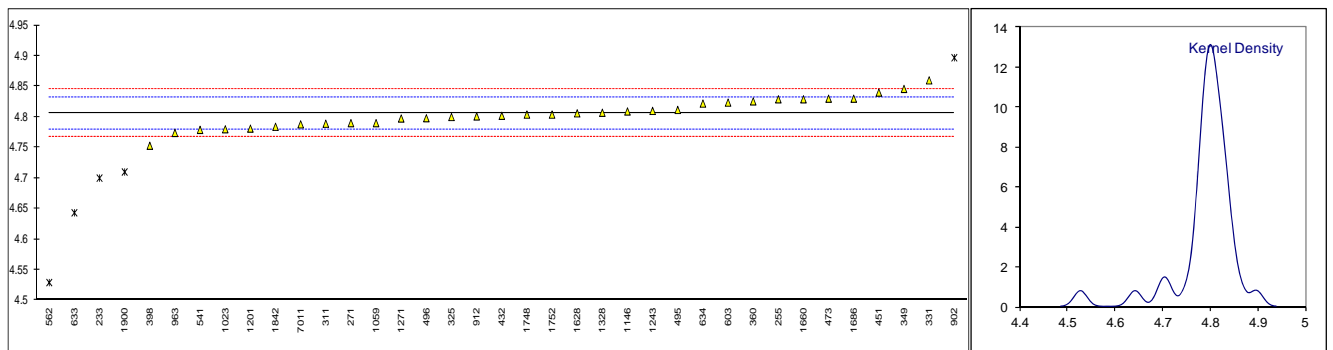
Determination of Water on sample #13204; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233		-----		-----	
255		-----		-----	
271	D6304	104.2		0.22	
311		-----		-----	
325	D6304-A	74.5		-0.12	
331	D6304-A	75		-0.12	
335	D6304	85		0.00	
340	D6304-A	95		0.11	
349	D6304-A	151	C,DG(0.05)	0.76	First reported 511
360	D6304-A	110.0		0.29	
398	D6304-C	86.7		0.02	
432		-----		-----	
451	D6304-A	146	DG(0.05)	0.70	
473	D6304-C	80.6		-0.05	
495	D6304-C	41		-0.51	
496	D6304-C	79		-0.07	
541		-----		-----	
551		-----		-----	
562		-----		-----	
603	D6304-C	62.4		-0.26	
614	D6304-C	74		-0.13	
633	E203	106.0	C	0.24	First reported 160.0
634	D6304-A	102		0.20	
902	D6304-A	102.50		0.20	
912	D6304-C	85		0.00	
963	D6304-A	96		0.13	
1017		-----		-----	
1023	D6304	70		-0.17	
1059	D6304-A	70		-0.17	
1146	D6304-C	90		0.06	
1201	D6304-A	102		0.20	
1243	D6304	100		0.17	
1271		-----		-----	
1328		-----		-----	
1403		-----		-----	
1628	in house	93.4		0.10	
1660	IEC60814	84.2		-0.01	
1686	D6304-C	58		-0.31	
1748		-----		-----	
1752		-----		-----	
1768	ISO3733	46	C	-0.45	First reported 0.0
1842	IP386	89		0.05	
1900	D6304-C	92		0.08	
1915		-----		-----	
7011	D6304-A	112.3		0.31	

		Only ASTM D6304-C data:
normality	OK	OK
n	29	10
outliers	2	0
mean (n)	85.03	74.87
st.dev. (n)	18.134	16.340
R(calc.)	50.78	45.75
R(D6304:07)	242.89	225.04
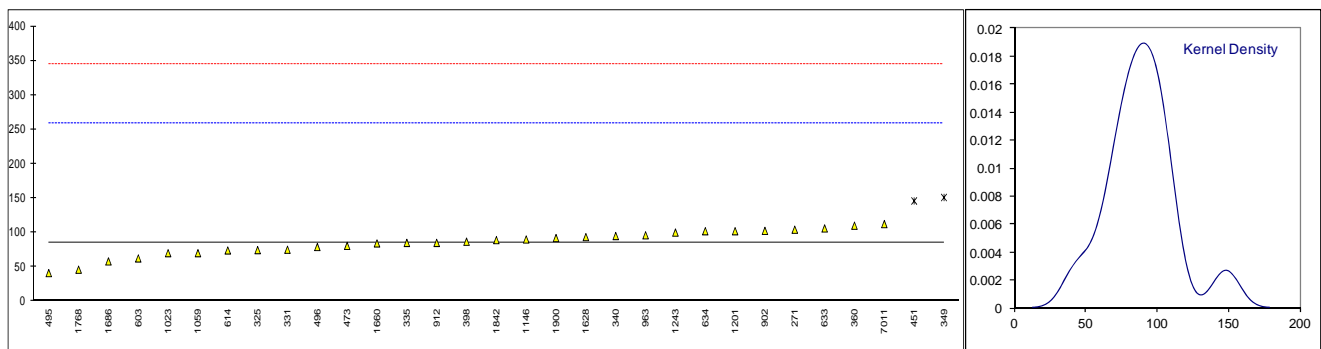
Determination of Aluminum (Al) on sample #13205; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	4		-----	
255	INH-OLI	1.76175		-----	
271	D5185	3.59		-----	
311		-----		-----	
325	D5185	2.5		-----	
331	D5185	2.8		-----	
335		-----		-----	
340	D5185	2.6		-----	
349	D5185	2.46		-----	
360	D5185	2.66		-----	
398		-----		-----	
432		-----		-----	
451	D5185	3		-----	
473	D5185	<1		-----	
495	D5185	3.1		-----	
496	D5185	2.50		-----	
541	D5185	<6		-----	
551		-----		-----	
562	D6595	3.1		-----	
603	D5185	2.434		-----	
614	D5185	2.90		-----	
633	D6595	3.639		-----	
634	D6595	3.889		-----	
902	D5185	1.78		-----	
912	D5185	2		-----	
963	D5185	2.257		-----	
1017		-----		-----	
1023	D5185	2.43		-----	
1059	in house	2.8		-----	
1146	in house	2.485		-----	
1201	D5185	1		-----	
1243	D5185	1.3		-----	
1271	D5185	3.50		-----	
1328	D5185	2.68		-----	
1403		-----		-----	
1628		-----		-----	
1660	D5185	3.20		-----	
1686		-----		-----	
1748		-----		-----	
1752		-----		-----	
1768		-----		-----	
1842	INH-01	2.9		-----	
1900	D6595	3		-----	
1915		-----		-----	
7011		-----		-----	

normality	OK
n	29
outliers	0
mean (n)	2.699
st.dev. (n)	0.7038
R(calc.)	1.971
R(D5185:13)	(4.919) Application range ASTM D5185: 6 – 40 mg/kg
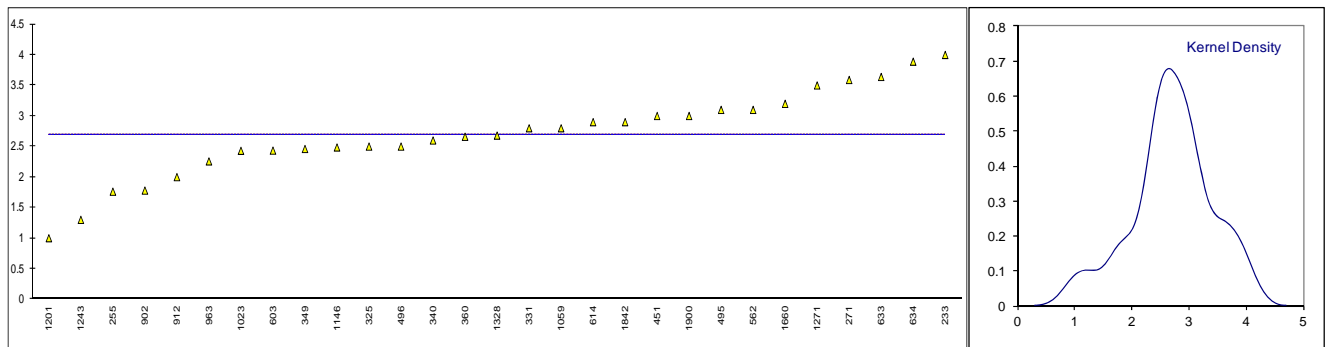
Determination of Barium (Ba) on sample #13205; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	90		-0.08	
255		-----		-----	
271	D5185	110.7	C	2.74	First reported 131.1
311		-----		-----	
325	D5185	88.0		-0.35	
331	D5185	94.3		0.51	
335		-----		-----	
340	D5185	89.2		-0.19	
349	D5185	90.34		-0.03	
360		-----		-----	
398		-----		-----	
432		-----		-----	
451	D5185	86		-0.62	
473	D5185	84.4073		-0.84	
495	D5185	93.9		0.45	
496	D5185	86.0		-0.62	
541	D5185	85.5		-0.69	
551		-----		-----	
562	D6595	73.2		-2.36	
603	D5185	90.42		-0.02	
614	D5185	94.1		0.48	
633	D6595	102.6	C	1.64	First reported 107.74
634	D6595	103.26		1.73	
902	D5185	87.8275		-0.37	
912	D5185	90		-0.08	
963	D5185	89.27		-0.18	
1017		-----		-----	
1023	D5185	82.7		-1.07	
1059	in house	86.7		-0.53	
1146	in house	85.03		-0.75	
1201	D5185	105		1.96	
1243	D5185	89.3		-0.17	
1271	D5185	103.75		1.79	
1328		-----		-----	
1403		-----		-----	
1628		-----		-----	
1660	D5185	0.00	G(0.01)	-12.31	
1686		-----		-----	
1748		-----		-----	
1752	in house	80		-1.44	
1768		-----		-----	
1842	INH-01	86.2		-0.59	
1900	D6595	88		-0.35	
1915		-----		-----	
7011		-----		-----	

normality	not OK	
n	28	
outliers	1	
mean (n)	90.56	
st.dev. (n)	8.171	
R(calc.)	22.88	
R(Horwitz)	20.59	R (D5185) = 37.26. Application range ASTM D5185: 0.5 – 4 mg/kg
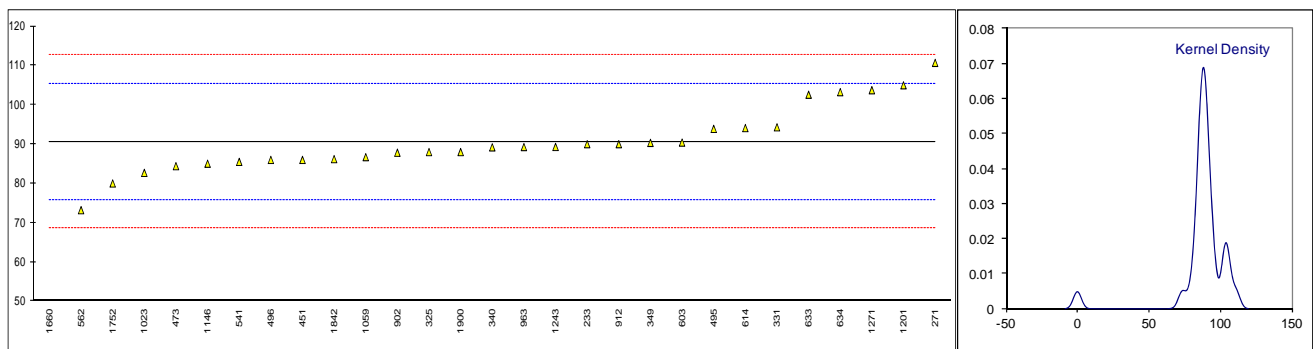
Determination of Chromium (Cr) on sample #13205; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	4		1.54	
255	INH-OLI	2.2127		-1.55	
271	D5185	2.87		-0.42	
311		-----		-----	
325	D5185	3.0		-0.19	
331	D5185	3.5		0.67	
335		-----		-----	
340	D5185	3.3		0.33	
349	D5185	0.85		-3.91	
360	D5185	3.36		0.43	
398		-----		-----	
432		-----		-----	
451	D5185	3		-0.19	
473	D5185	1.7337	C	-2.38	First reported 1.8783
495	D5185	3.3		0.33	
496	D5185	3.05		-0.11	
541	D5185	3.1		-0.02	
551		-----		-----	
562	D6595	1.6		-2.61	
603	D5185	3.134		0.04	
614	D5185	3.33		0.38	
633	D6595	4.326		2.10	
634	D6595	4.277		2.02	
902	D5185	2.2485		-1.49	
912	D5185	3		-0.19	
963	D5185	2.786		-0.56	
1017		-----		-----	
1023	D5185	3.08		-0.05	
1059	in house	3.0		-0.19	
1146	in house	3.146		0.06	
1201	D5185	3		-0.19	
1243	D5185	<2	C	-----	First reported 1.8
1271	D5185	3.80		1.19	
1328	D5185	3.17		0.10	
1403	ISO11885	5.15		3.53	
1628		-----		-----	
1660	D5185	7.00	G(0.01)	6.73	
1686		-----		-----	
1748		-----		-----	
1752		-----		-----	
1768		-----		-----	
1842	INH-01	3.4		0.50	
1900	D6595	3.6		0.85	
1915		-----		-----	
7011		-----		-----	

normality	not OK	
n	30	
outliers	1	
mean (n)	3.111	
st.dev. (n)	0.8303	
R(calc.)	2.325	
R(D5185:13)	1.619	Application range ASTM D5185: 1 – 40 mg/kg
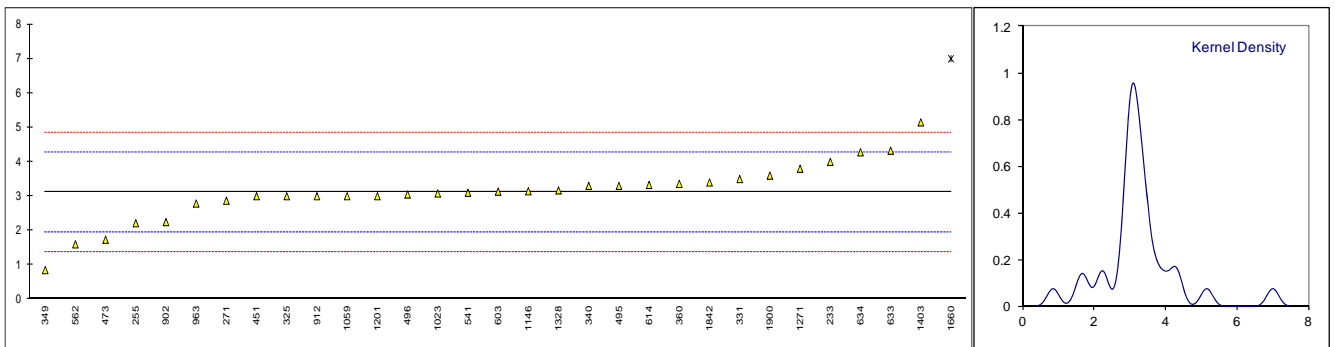
Determination of Copper (Cu) on sample #13205; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	31		0.50	
255	INH-OLI	26.330		-1.34	
271	D5185	37.9	C,G(0.05)	3.20	First reported 43.5
311		-----		-----	
325	D5185	30		0.10	
331	D5185	30.8		0.42	
335		-----		-----	
340	D5185	30.9		0.46	
349	D5185	32.10		0.93	
360	D5185	30.2		0.18	
398		-----		-----	
432		-----		-----	
451	D5185	29		-0.29	
473	D5185	29.4405		-0.12	
495	D5185	30.7		0.38	
496	D5185	29.5		-0.09	
541	D5185	29		-0.29	
551		-----		-----	
562	D6595	12.8	G(0.01)	-6.64	
603	D5185	32.56		1.11	
614	D5185	31.6		0.73	
633	D6595	25.710		-1.58	
634	D6595	25.350		-1.72	
902	D5185	28.345		-0.55	
912	D5185	30		0.10	
963	D5185	27.63		-0.83	
1017		-----		-----	
1023	D5185	27.6		-0.84	
1059	in house	26.3		-1.35	
1146	in house	29.31		-0.17	
1201	D5185	30		0.10	
1243	D5185	29.7		-0.01	
1271	D5185	30.10	C	0.14	First reported 37.50
1328	D5185	29.73		0.00	
1403	ISO11885	30.26		0.21	
1628		-----		-----	
1660	D5185	33.0		1.28	
1686		-----		-----	
1748		-----		-----	
1752	in house	35		2.07	
1768		-----		-----	
1842	INH-01	29.6		-0.05	
1900	D6595	31		0.50	
1915		-----		-----	
7011		-----		-----	

normality	OK	
n	31	
outliers	2	
mean (n)	29.73	
st.dev. (n)	2.114	
R(calc.)	5.92	
R(D5185:13)	7.14	Application range ASTM D5185: 2 – 160 mg/kg
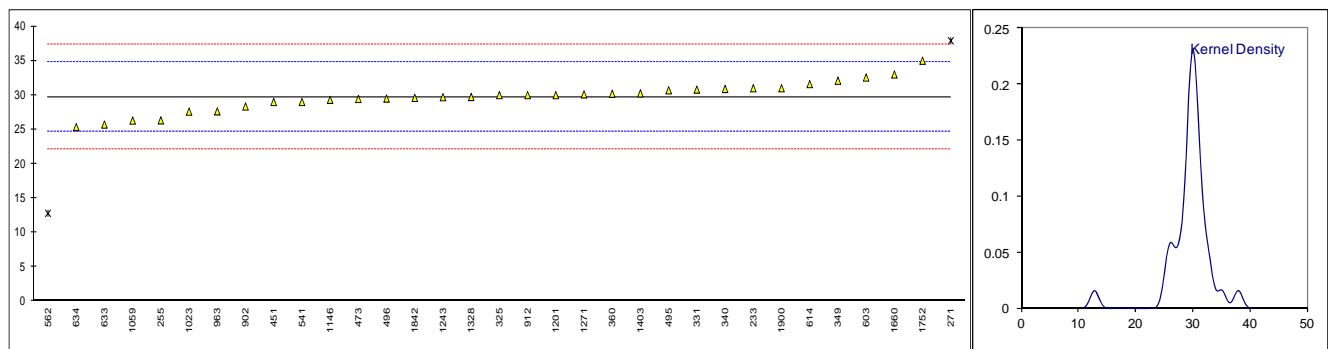
Determination of Iron (Fe) on sample #13205; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	7		1.13	
255	INH-OLI	6.1118		0.00	
271	D5185	7.84		2.19	
311		-----		-----	
325	D5185	5.75		-0.46	
331	D5185	6.4		0.37	
335		-----		-----	
340	D5185	6.1		-0.01	
349	D5185	4.06		-2.60	
360	D5185	6.41		0.38	
398		-----		-----	
432		-----		-----	
451	D5185	6		-0.14	
473	D5185	1.9605	C,G(0.05)	-5.25	First reported 2.4597
495	D5185	7.0		1.13	
496	D5185	5.85		-0.33	
541	D5185	5.9		-0.27	
551		-----		-----	
562	D6595	4.2		-2.42	
603	D5185	5.256		-1.08	
614	D5185	6.72		0.77	
633	D6595	7.743		2.07	
634	D6595	6.125		0.02	
902	D5185	5.3608		-0.95	
912	D5185	6		-0.14	
963	D5185	5.155		-1.21	
1017		-----		-----	
1023	D5185	5.58		-0.67	
1059	in house	7.4		1.63	
1146	in house	5.529		-0.74	
1201	D5185	4		-2.67	
1243	D5185	5.5		-0.77	
1271	D5185	7.40		1.63	
1328	D5185	6.01		-0.13	
1403	ISO11885	11.37	G(0.01)	6.66	
1628		-----		-----	
1660	D5185	5.63		-0.61	
1686		-----		-----	
1748		-----		-----	
1752	in house	7		1.13	
1768		-----		-----	
1842	INH-01	6.4		0.37	
1900	D6595	8		2.39	
1915		-----		-----	
7011		-----		-----	

normality	OK
n	31
outliers	2
mean (n)	6.111
st.dev. (n)	1.0258
R(calc.)	2.872
R(D5185:13)	2.212

Application range ASTM D5185: 2 – 140 mg/kg
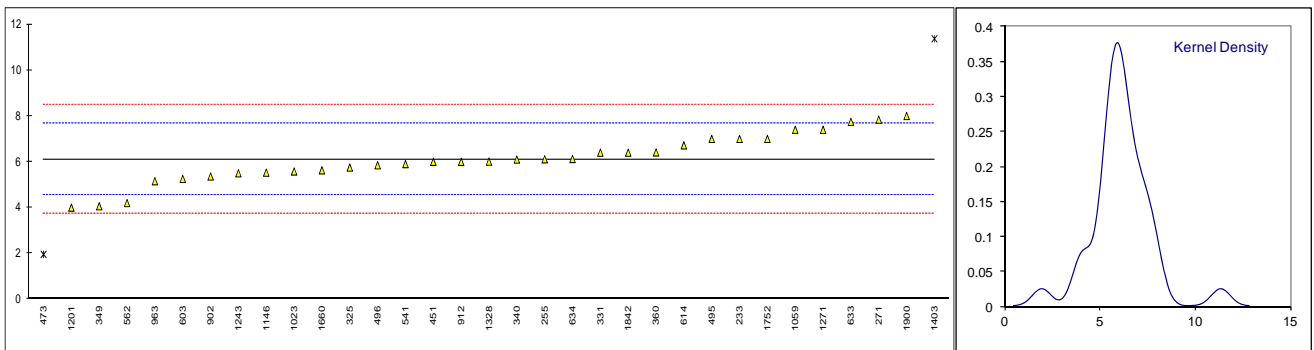
Determination of Lead (Pb) on sample #13205; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	6		-----	
255	INH-OLI	4.9749		-----	
271	D5185	6.86		-----	
311		-----		-----	
325	D5185	5.75		-----	
331	D5185	6.1		-----	
335		-----		-----	
340	D5185	5.9		-----	
349	D5185	6.73		-----	
360	D5185	5.79		-----	
398		-----		-----	
432		-----		-----	
451	D5185	8		-----	
473	D5185	3.9185	C	-----	First reported 3.2373
495	D5185	5.9		-----	
496	D5185	4.85		-----	
541	D5185	<10		-----	
551		-----		-----	
562	D6595	1.4	DG(0.05)	-----	
603	D5185	5.602		-----	
614	D5185	5.85		-----	
633	D6595	5.609		-----	
634	D6595	5.880		-----	
902	D5185	4.957		-----	
912	D5185	6		-----	
963	D5185	4.913		-----	
1017		-----		-----	
1023	D5185	5.57		-----	
1059	in house	8.2		-----	
1146	in house	5.284		-----	
1201	D5185	7		-----	
1243	D5185	5.2		-----	
1271	D5185	6.30		-----	
1328		-----		-----	
1403	ISO11885	8.54		-----	
1628		-----		-----	
1660	D5185	9.28		-----	
1686		-----		-----	
1748		-----		-----	
1752	in house	5		-----	
1768		-----		-----	
1842	INH-01	2.6	DG(0.05)	-----	
1900	D6595	7.3		-----	
1915		-----		-----	
7011		-----		-----	

normality	not OK
n	29
outliers	2
mean (n)	6.112
st.dev. (n)	1.2179
R(calc.)	3.410
R(D5185:13)	(5.756)

Application range ASTM D5185: 10 – 160 mg/kg
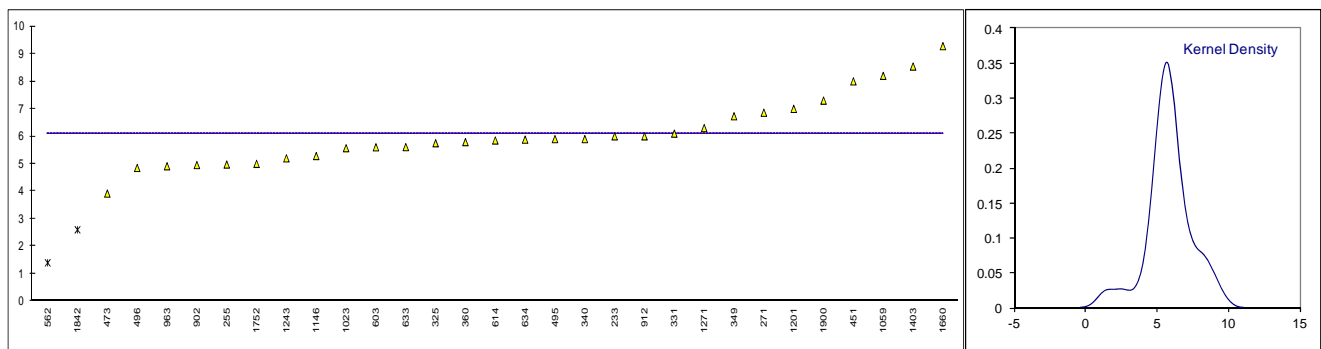
Determination of Lithium (Li) on sample #13205; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233		-----		-----	
255		-----		-----	
271		-----		-----	
311		-----		-----	
325		-----		-----	
331	D5185	7.4		0.38	
335		-----		-----	
340		-----		-----	
349		-----		-----	
360		-----		-----	
398		-----		-----	
432		-----		-----	
451		7		-0.09	
473		-----		-----	
495		-----		-----	
496	D5185	7.55		0.56	
541		-----		-----	
551		-----		-----	
562		-----		-----	
603		-----		-----	
614	D5185	7.0		-0.09	
633	D6595	20.17	C,G(0.01)	15.52	First reported 18.480
634		-----		-----	
902		6.436		-0.76	
912		-----		-----	
963	D5185	7.524		0.53	
1017		-----		-----	
1023	D5185	6.80		-0.33	
1059		-----		-----	
1146	in house	6.903		-0.21	
1201		-----		-----	
1243		-----		-----	
1271		-----		-----	
1328		-----		-----	
1403		-----		-----	
1628		-----		-----	
1660		-----		-----	
1686		-----		-----	
1748		-----		-----	
1752		-----		-----	
1768		-----		-----	
1842		-----		-----	
1900		-----		-----	
1915		-----		-----	
7011		-----		-----	

normality	OK	
n	8	
outliers	1	
mean (n)	7.077	
st.dev. (n)	0.3887	
R(calc.)	1.088	
R(Horwitz)	2.362	
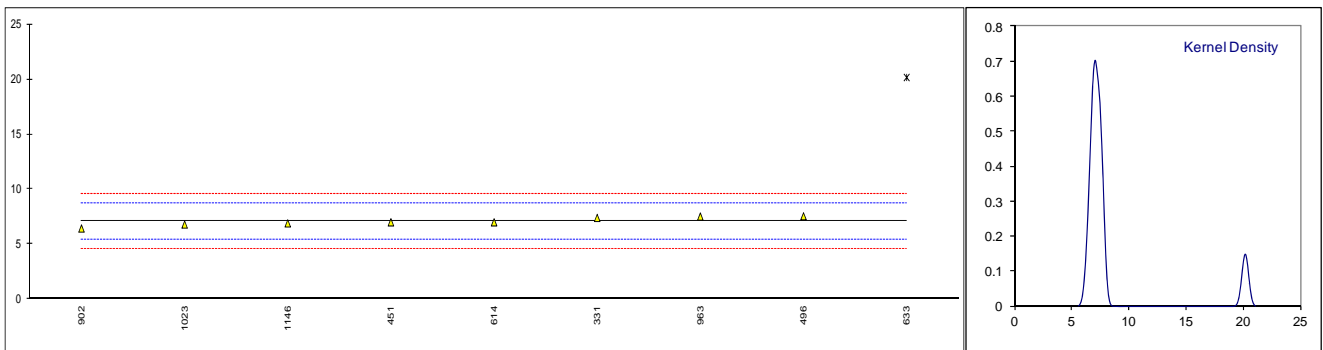
Determination of Magnesium (Mg) on sample #13205; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	6		0.53	
255	INH-OLI	5.2306		-0.27	
271	D5185	6.712		1.28	
311		-----		-----	
325	D5185	5.00		-0.51	
331	D5185	4.8		-0.72	
335		-----		-----	
340	D5185	5.5		0.01	
349	D5185	5.80		0.32	
360	D5185	5.91		0.44	
398		-----		-----	
432		-----		-----	
451	D5185	5		-0.51	
473	D5185	1.8683	C,G(0.05)	-3.80	First reported 2.1710
495	D5185	6.2		0.74	
496	D5185	5.185		-0.32	
541	D5185	<5		-----	
551		-----		-----	
562	D6595	6.4		0.95	
603	D5185	5.969		0.50	
614	D5185	6.2		0.74	
633	D6595	6.054		0.59	
634	D6595	5.527		0.04	
902	D5185	4.615		-0.92	
912	D5185	6		0.53	
963	D5185	5.328		-0.17	
1017		-----		-----	
1023	D5185	5.15		-0.36	
1059		-----		-----	
1146	in house	5.853		0.38	
1201	D5185	4		-1.56	
1243	D5185	3.0	C	-2.61	First reported 3.5
1271	D5185	6.35		0.90	
1328	D5185	5.57		0.08	
1403		-----		-----	
1628		-----		-----	
1660	D5185	0.00	G(0.01)	-5.75	
1686		-----		-----	
1748		-----		-----	
1752	in house	3.6		-1.98	
1768		-----		-----	
1842	INH-01	5.8		0.32	
1900	D6595	7		1.58	
1915		-----		-----	
7011		-----		-----	

normality	OK	
n	28	
outliers	2	
mean (n)	5.491	
st.dev. (n)	0.9015	
R(calc.)	2.524	
R(D5185:13)	2.672	Application range ASTM D5185: 5 – 1700 mg/kg
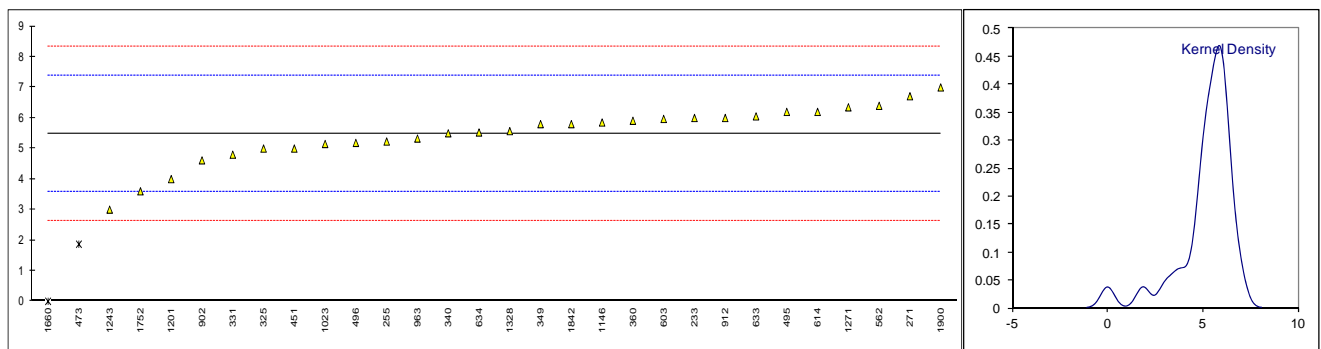
Determination of Manganese (Mn) on sample #13205; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233		-----		-----	
255	INH-OLI	2.0282		-----	
271	D5185	1.36		-----	
311		-----		-----	
325	D5185	2.25		-----	
331	D5185	2.6		-----	
335		-----		-----	
340	D5185	2.6		-----	
349		-----		-----	
360	D5185	2.50		-----	
398		-----		-----	
432		-----		-----	
451	D5185	1		-----	
473	D5185	<1		-----	
495		-----		-----	
496	D5185	2.30		-----	
541	D5185	<5		-----	
551		-----		-----	
562		-----		-----	
603	D5185	2.205		-----	
614	D5185	2.8		-----	
633	D6595	3.782		-----	
634	D6595	3.687	C	-----	First reported 4.216
902	D5185	1.579		-----	
912	D5185	2		-----	
963	D5185	2.272		-----	
1017		-----		-----	
1023	D5185	2.40		-----	
1059	in house	2.7		-----	
1146	in house	2.474		-----	
1201	D5185	2		-----	
1243	D5185	2.0		-----	
1271	D5185	2.90		-----	
1328	D5185	2.56		-----	
1403		-----		-----	
1628		-----		-----	
1660	D5185	0.00	G(0.05)	-----	
1686		-----		-----	
1748		-----		-----	
1752	in house	3.6		-----	
1768		-----		-----	
1842	INH-01	3.3		-----	
1900		-----		-----	
1915		-----		-----	
7011		-----		-----	

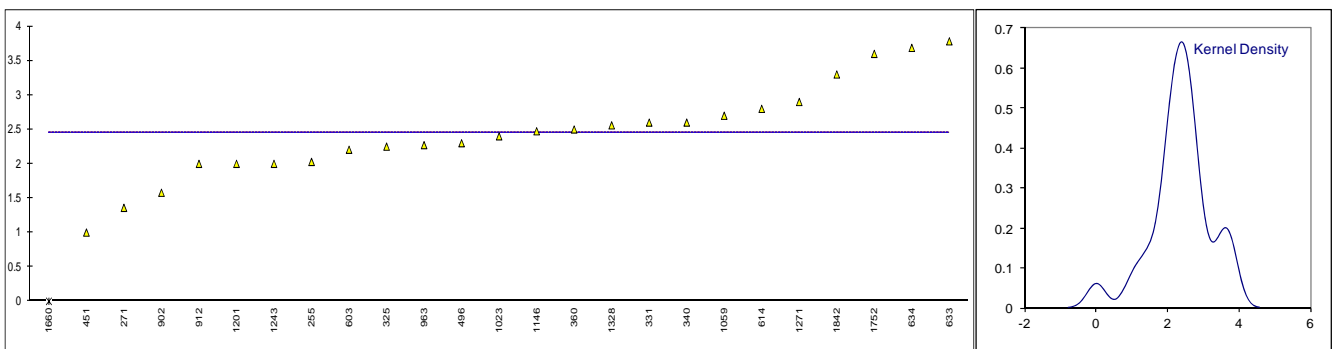|		
normality	OK	
n	24	
outliers	1	
mean (n)	2.454	
st.dev. (n)	0.6849	
R(calc.)	1.918	
R(D5185:13)	(0.382)	Application range ASTM D5185: 5 – 700 mg/kg

Determination of Molybdenum (Mo) on sample #13205; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	3		-----	
255		-----		-----	
271	D5185	1.79		-----	
311		-----		-----	
325	D5185	2.0		-----	
331	D5185	2.7		-----	
335		-----		-----	
340	D5185	2.4		-----	
349	D5185	1.04		-----	
360	D5185	2.68		-----	
398		-----		-----	
432		-----		-----	
451	D5185	3		-----	
473	D5185	<1		-----	
495	D5185	2.7		-----	
496	D5185	2.05		-----	
541	D5185	<5		-----	
551		-----		-----	
562	D6595	2.0		-----	
603	D5185	2.251		-----	
614	D5185	2.41		-----	
633	D6595	4.052		-----	
634	D6595	2.573		-----	
902	D5185	1.387		-----	
912	D5185	3		-----	
963	D5185	2.280		-----	
1017		-----		-----	
1023	D5185	2.37		-----	
1059	in house	4.5	G(0.05)	-----	
1146		-----		-----	
1201	D5185	3		-----	
1243	D5185	<1.0		-----	
1271	D5185	2.95		-----	
1328		-----		-----	
1403	ISO11885	3.89		-----	
1628		-----		-----	
1660	D5185	0.00	G(0.01)	-----	
1686		-----		-----	
1748		-----		-----	
1752		-----		-----	
1768		-----		-----	
1842	INH-01	2.4		-----	
1900	D6595	2		-----	
1915		-----		-----	
7011		-----		-----	

|		
normality	OK	
n	24	
outliers	2	
mean (n)	2.497	
st.dev. (n)	0.6803	
R(calc.)	1.905	
R(D5185:13)	(1.226)	Application range ASTM D5185: 5 – 200 mg/kg
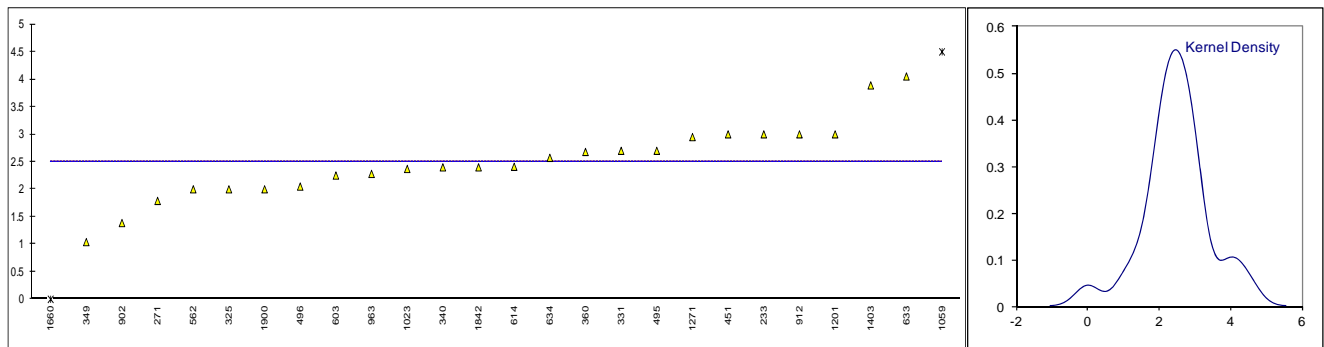
Determination of Nickel (Ni) on sample #13205; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	3	C	-----	First reported 4
255	INH-OLI	1.9870		-----	
271	D5185	2.30		-----	
311		-----		-----	
325	D5185	2.75		-----	
331	D5185	2.8		-----	
335		-----		-----	
340	D5185	2.5		-----	
349	D5185	1.29		-----	
360	D5185	2.54		-----	
398		-----		-----	
432		-----		-----	
451	D5185	2		-----	
473	D5185	<1		-----	
495	D5185	2.6		-----	
496	D5185	2.25		-----	
541	D5185	<5		-----	
551		-----		-----	
562	D6595	1.6		-----	
603	D5185	1.410		-----	
614	D5185	2.68		-----	
633	D6595	3.426		-----	
634	D6595	3.401		-----	
902	D5185	1.488		-----	
912	D5185	2.0	C	-----	First reported 1
963	D5185	2.229		-----	
1017		-----		-----	
1023	D5185	2.14		-----	
1059	in house	2.2		-----	
1146	in house	2.533		-----	
1201	D5185	2		-----	
1243	D5185	<1.0		-----	
1271	D5185	2.80		-----	
1328	D5185	2.51		-----	
1403	ISO11885	4.81	G(0.01)	-----	
1628		-----		-----	
1660	D5185	2.37		-----	
1686		-----		-----	
1748		-----		-----	
1752		-----		-----	
1768		-----		-----	
1842	INH-01	2.5		-----	
1900	D6595	1.2		-----	
1915		-----		-----	
7011		-----		-----	

normality	OK	
n	28	
outliers	1	
mean (n)	2.304	
st.dev. (n)	0.5686	
R(calc.)	1.592	
R(D5185:13)	(2.277)	Application range ASTM D5185: 5 – 40 mg/kg
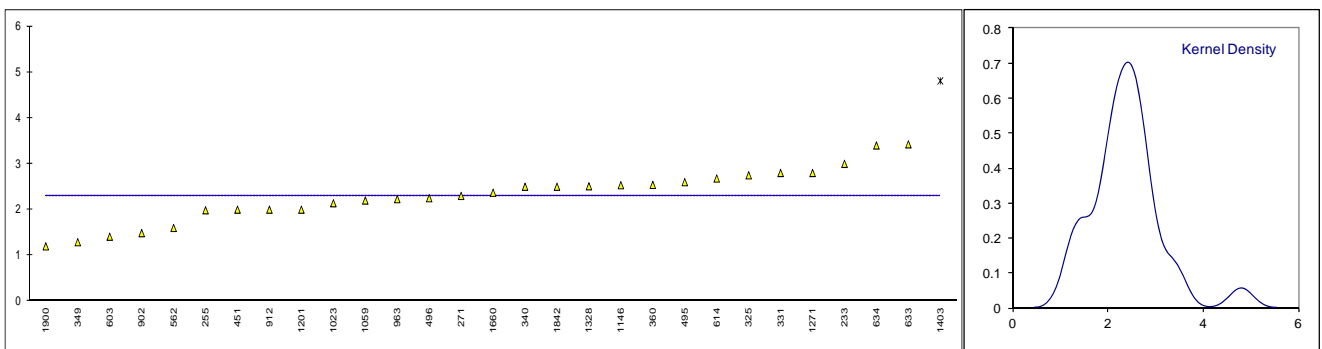
Determination of Sodium (Na) on sample #13205; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	8		0.16	
255	INH-OLI	5.5233		-1.32	
271	D5185	10.6		1.70	
311		-----		-----	
325	D5185	5.25		-1.48	
331	D5185	6.0		-1.03	
335		-----		-----	
340	D5185	9.7		1.17	
349	D5185	23.56	G(0.01)	9.42	
360	D5185	7.80		0.04	
398		-----		-----	
432		-----		-----	
451	D5185	9		0.75	
473	D5185	3.7478		-2.38	
495	D5185	9.8		1.23	
496	D5185	6.65		-0.65	
541	D5185	<7		-----	
551		-----		-----	
562	D6595	8.2		0.28	
603	D5185	7.947		0.13	
614	D5185	6.8		-0.56	
633	D6595	9.773		1.21	
634	D6595	8.715		0.58	
902	D5185	7.322		-0.25	
912		-----		-----	
963	D5185	5.706		-1.21	
1017		-----		-----	
1023	D5185	6.24		-0.89	
1059		-----		-----	
1146	in house	6.501		-0.74	
1201	D5185	12		2.54	
1243		-----		-----	
1271	D5185	8.85		0.66	
1328		-----		-----	
1403		-----		-----	
1628		-----		-----	
1660	D5185	7.00		-0.44	
1686		-----		-----	
1748		-----		-----	
1752		-----		-----	
1768		-----		-----	
1842	INH-01	8.3		0.34	
1900	D6595	8		0.16	
1915		-----		-----	
7011		-----		-----	

normality	OK	
n	25	
outliers	1	
mean (n)	7.737	
st.dev. (n)	1.8688	
R(calc.)	5.233	
R(D5185:13)	4.702	Application range ASTM D5185: 7 – 70 mg/kg
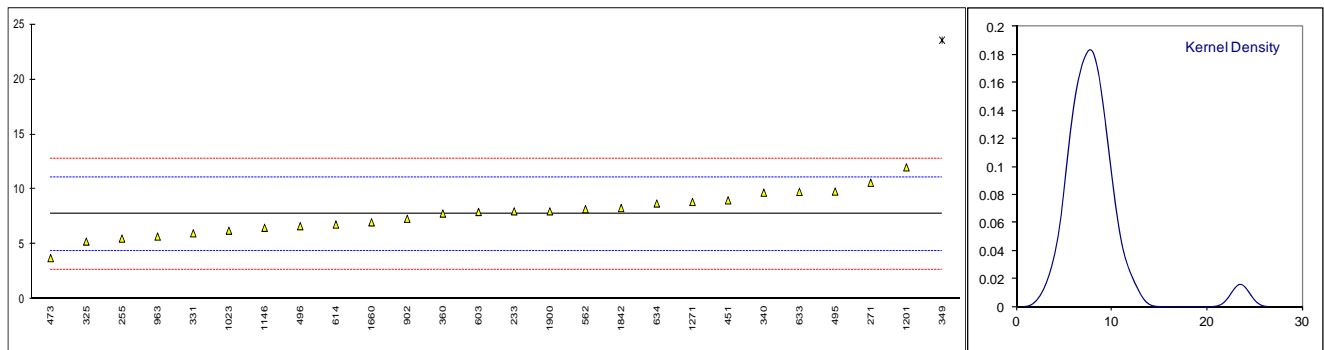
Determination of Silicon (Si) on sample #13205; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	4		-----	
255		-----		-----	
271	D5185	7.21		-----	
311		-----		-----	
325	D5185	5.0		-----	
331	D5185	4.5		-----	
335		-----		-----	
340	D5185	4.3		-----	
349	D5185	3.95		-----	
360	D5185	6.85		-----	
398		-----		-----	
432		-----		-----	
451	D5185	6		-----	
473	D5185	1.3771		-----	
495	D5185	5.2		-----	
496	D5185	5.10		-----	
541	D5185	<8		-----	
551		-----		-----	
562	D6595	5.1		-----	
603	D5185	5.116		-----	
614	D5185	5.27		-----	
633	D6595	5.796		-----	
634	D6595	4.996		-----	
902	D5185	1.829		-----	
912	D5185	4		-----	
963	D5185	2.480		-----	
1017		-----		-----	
1023	D5185	3.91		-----	
1059	in house	7.8		-----	
1146	in house	6.344		-----	
1201	D5185	2		-----	
1243	D5185	4.0		-----	
1271	D5185	5.75		-----	
1328		-----		-----	
1403		-----		-----	
1628		-----		-----	
1660	D5185	0.00	G(0.01)	-----	
1686		-----		-----	
1748		-----		-----	
1752		-----		-----	
1768		-----		-----	
1842	INH-01	4.3		-----	
1900	D6595	7		-----	
1915		-----		-----	
7011		-----		-----	

normality	OK
n	27
outliers	1
mean (n)	4.784
st.dev. (n)	1.6183
R(calc.)	4.531
R(D5185:13)	(5.340)

Application range ASTM D5185: 8 – 50 mg/kg
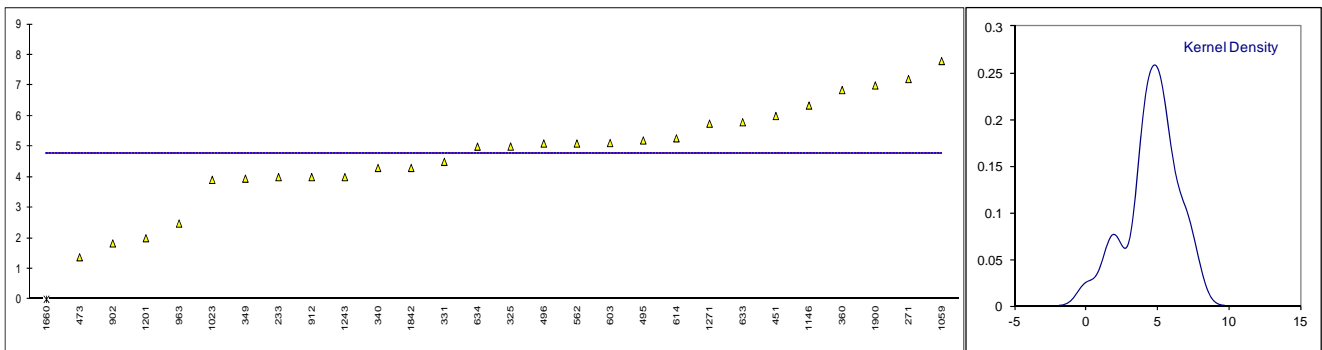
Determination of Silver (Ag) on sample #13205; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	3		0.39	
255	INH-OLI	1.8825		-2.74	
271	D5185	1.71		-3.22	
311		-----		-----	
325	D5185	2.25		-1.71	
331	D5185	2.3		-1.57	
335		-----		-----	
340	D5185	3.3		1.23	
349	D5185	4.26		3.91	
360	D5185	3.70		2.34	
398		-----		-----	
432		-----		-----	
451	D5185	3		0.39	
473	D5185	0.3647	G(0.05)	-6.98	
495	D5185	2.8		-0.17	
496	D5185	3.35		1.36	
541	D5185	3.8		2.62	
551		-----		-----	
562	D6595	1.9		-2.69	
603	D5185	3.117		0.71	
614	D5185	2.8		-0.17	
633	D6595	2.444		-1.17	
634	D6595	2.359		-1.41	
902	D5185	4.241		3.86	
912	D5185	3		0.39	
963	D5185	2.244		-1.73	
1017		-----		-----	
1023	D5185	2.37		-1.37	
1059	in house	2.2		-1.85	
1146	in house	2.399		-1.29	
1201	D5185	32	G(0.01)	81.45	
1243		-----		-----	
1271	D5185	2.95		0.25	
1328		-----		-----	
1403		-----		-----	
1628		-----		-----	
1660	D5185	1.73		-3.16	
1686		-----		-----	
1748		-----		-----	
1752		-----		-----	
1768		-----		-----	
1842	INH-01	5.3		6.82	
1900	in house	39.0	G(0.01)	101.02	
1915		-----		-----	
7011		-----		-----	

normality	OK	
n	26	
outliers	3	
mean (n)	2.862	
st.dev. (n)	0.8680	
R(calc.)	2.431	
R(D5185:13)	1.002	Application range ASTM D5185: 0.5 – 50 mg/kg
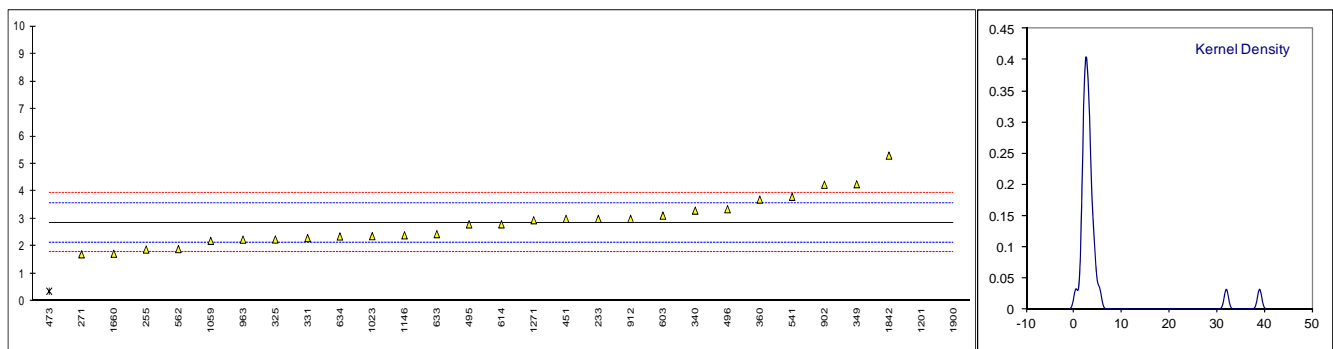
Determination of Tin (Sn) on sample #13205; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	2		-----	
255		-----		-----	
271	D5185	1.63		-----	
311		-----		-----	
325	D5185	2.25		-----	
331	D5185	2.7		-----	
335		-----		-----	
340	D5185	2.7		-----	
349	D5185	3.22		-----	
360	D5185	3.61		-----	
398		-----		-----	
432		-----		-----	
451	D5185	2		-----	
473	D5185	0.0280	G(0.05)	-----	
495	D5185	2.6		-----	
496	D5185	2.35		-----	
541	D5185	<10		-----	
551		-----		-----	
562	D6595	0.3		-----	
603	D5185	1.141		-----	
614	D5185	2.52		-----	
633	D6595	0.033	G(0.05)	-----	
634	D6595	0.67		-----	
902	D5185	1.450		-----	
912	D5185	3		-----	
963	D5185	2.200		-----	
1017		-----		-----	
1023	D5185	2.68		-----	
1059	in house	1.5		-----	
1146	in house	2.613		-----	
1201	D5185	5		-----	
1243	D5185	1.9		-----	
1271	D5185	2.95		-----	
1328		-----		-----	
1403		-----		-----	
1628		-----		-----	
1660	D5185	2.70		-----	
1686		-----		-----	
1748		-----		-----	
1752		-----		-----	
1768		-----		-----	
1842	INH-01	2.4		-----	
1900	D6595	0	ex	-----	Result excluded, zero is not a real result
1915		-----		-----	
7011		-----		-----	

normality	OK	
n	25	
outliers	3	
mean (n)	2.323	
st.dev. (n)	0.9528	
R(calc.)	2.668	
R(D5185:13)	(3.542)	Application range ASTM D5185: 10 – 40 mg/kg
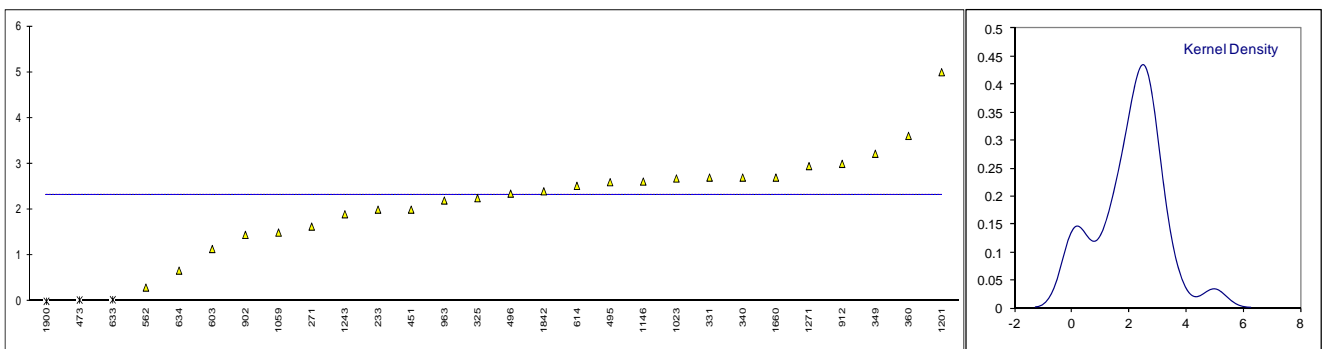
Determination of Titanium (Ti) on sample #13205; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	3		-----	
255		-----		-----	
271	D5185	1.89		-----	
311		-----		-----	
325		-----		-----	
331	D5185	2.5		-----	
335		-----		-----	
340	D5185	2.4		-----	
349	D5185	1.87		-----	
360	D5185	2.53		-----	
398		-----		-----	
432		-----		-----	
451	D5185	2		-----	
473	D5185	<1		-----	
495	D5185	2.4		-----	
496	D5185	2.05		-----	
541	D5185	<5		-----	
551		-----		-----	
562	D6595	1.1		-----	
603	D5185	1.242		-----	
614	D5185	2.4		-----	
633	D6595	2.077		-----	
634	D6595	2.368		-----	
902	D5185	1.602		-----	
912	D5185	2		-----	
963	D5185	2.146		-----	
1017		-----		-----	
1023	D5185	2.26		-----	
1059		-----		-----	
1146	in house	2.292		-----	
1201	D5185	2		-----	
1243		-----		-----	
1271	D5185	2.70		-----	
1328	D5185	2.40		-----	
1403		-----		-----	
1628		-----		-----	
1660	D5185	2.58		-----	
1686		-----		-----	
1748		-----		-----	
1752		-----		-----	
1768		-----		-----	
1842	INH-01	2.3		-----	
1900	in house	3.5		-----	
1915		-----		-----	
7011		-----		-----	

normality	OK	
n	25	
outliers	0	
mean (n)	2.224	
st.dev. (n)	0.4987	
R(calc.)	1.396	
R(D5185:13)	(3.640)	Application range ASTM D5185: 5 – 40 mg/kg
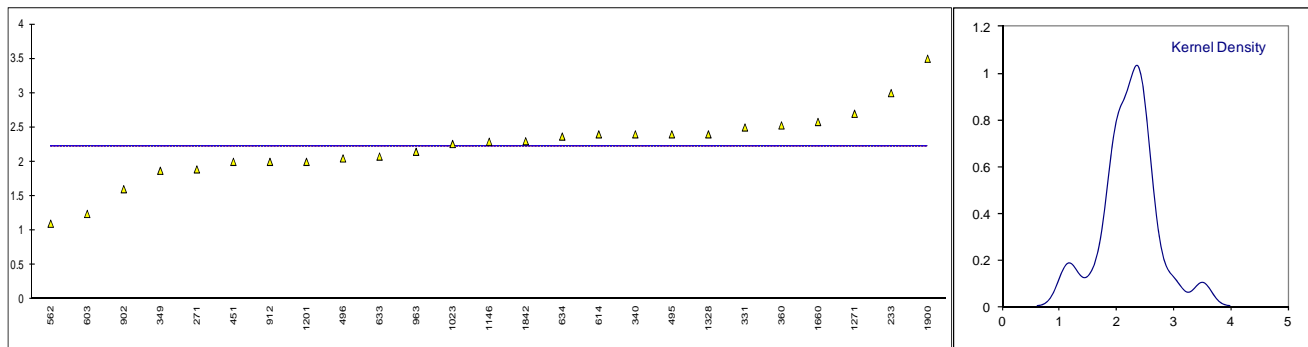
Determination of Vanadium (V) on sample #13205; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	3		2.70	
255		-----		-----	
271	D5185	1.95		-1.46	
311		-----		-----	
325	D5185	2.0		-1.26	
331	D5185	2.5		0.72	
335		-----		-----	
340	D5185	2.5		0.72	
349	D5185	1.54		-3.09	
360	D5185	2.50		0.72	
398		-----		-----	
432		-----		-----	
451	D5185	2		-1.26	
473	D5185	0.2481	C,G(0.05)	-8.21	First reported 0.5595
495	D5185	2.4		0.32	
496	D5185	2.20		-0.47	
541	D5185	2.3		-0.08	
551		-----		-----	
562	D6595	0.9		-5.63	
603	D5185	2.256		-0.25	
614	D5185	2.6		1.11	
633	D6595	3.18	C	3.41	First reported 3.685
634	D6595	3.286	C	3.83	First reported 3.904
902	D5185	1.448		-3.45	
912	D5185	2		-1.26	
963	D5185	2.129		-0.75	
1017		-----		-----	
1023	D5185	2.28		-0.15	
1059	in house	2.4		0.32	
1146	in house	2.296		-0.09	
1201	D5185	2		-1.26	
1243	D5185	<1.0	C	<-5.23	First reported 0.6, False negative?
1271	D5185	2.85		2.10	
1328		-----		-----	
1403		-----		-----	
1628		-----		-----	
1660	D5185	6.61	G(0.01)	17.01	
1686		-----		-----	
1748		-----		-----	
1752	in house	3.6		5.08	
1768		-----		-----	
1842	INH-01	2.5		0.72	
1900	D6595	2		-1.26	
1915		-----		-----	
7011		-----		-----	

normality	OK	
n	27	
outliers	2	
mean (n)	2.319	
st.dev. (n)	0.5673	
R(calc.)	1.588	
R(D5185:13)	0.706	Application range ASTM D5185: 1 – 50 mg/kg
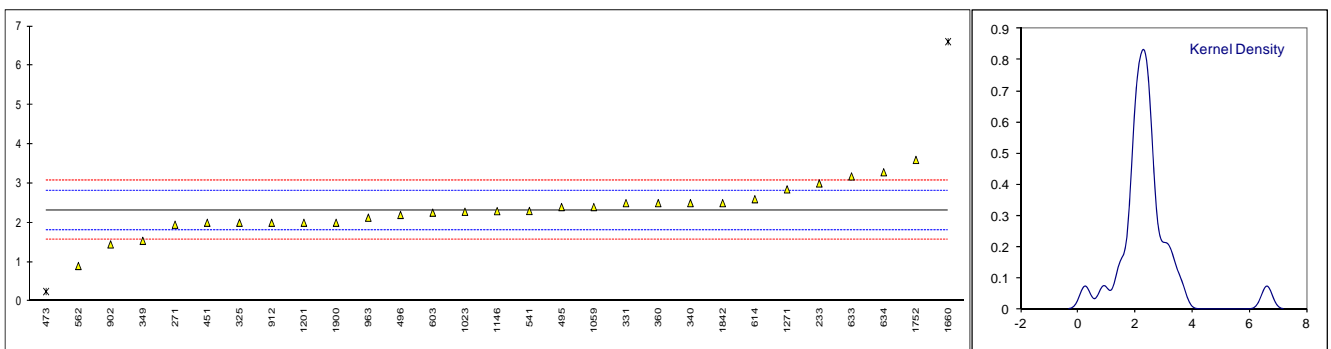
Determination of Calcium (Ca) on sample #13205; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233		-----		-----	
255	INH-OLI	310.305	C	-6.32	First reported 348.24
271	D5185	502.1	C,G(0.05)	9.06	First reported 600.7
311	D5185	420		2.48	
325	D5185	402.75		1.09	
331	D5185	426.4		2.99	
335		-----		-----	
340	D5185	407.0		1.43	
349	D5185	405.43		1.31	
360	D5185	411.4		1.79	
398		-----		-----	
432		-----		-----	
451	D5185	388		-0.09	
473	D5185	437.988		3.92	
495	D5185	447.0		4.64	
496	D5185	389.5		0.03	
541	D5185	408		1.51	
551		-----		-----	
562	D6595	308.9		-6.43	
603	D5185	409.5		1.63	
614	D5185	418		2.32	
633	D6595	281.10	C	-8.66	First reported 295.77
634	D6595	280.80	C	-8.68	First reported 289.06
902	D5185	400		0.87	
912	D5185	400		0.87	
963	D5185	386.0		-0.25	
1017		-----		-----	
1023	D5185	388		-0.09	
1059	in house	390		0.07	
1146	in house	419.7		2.45	
1201	D5185	430		3.28	
1243	D5185	415.1		2.08	
1271	D5185	394.45		0.43	
1328		-----		-----	
1403		-----		-----	
1628		-----		-----	
1660	D5185	200	G(0.05)	-15.16	
1686		-----		-----	
1748	D4927	353	C	-2.89	First reported 0.0353
1752	in house	426		2.96	
1768		-----		-----	
1842	INH-01	399		0.79	
1900	D6595	320		-5.54	
1915		-----		-----	
7011		-----		-----	

|		
normality	not OK	
n	30	
outliers	2	
mean (n)	389.11	
st.dev. (n)	44.761	
R(calc.)	125.33	Compare R(Horwitz) = 71.04
R(D5185:13)	34.93	Application range ASTM D5185: 40 – 9000 mg/kg
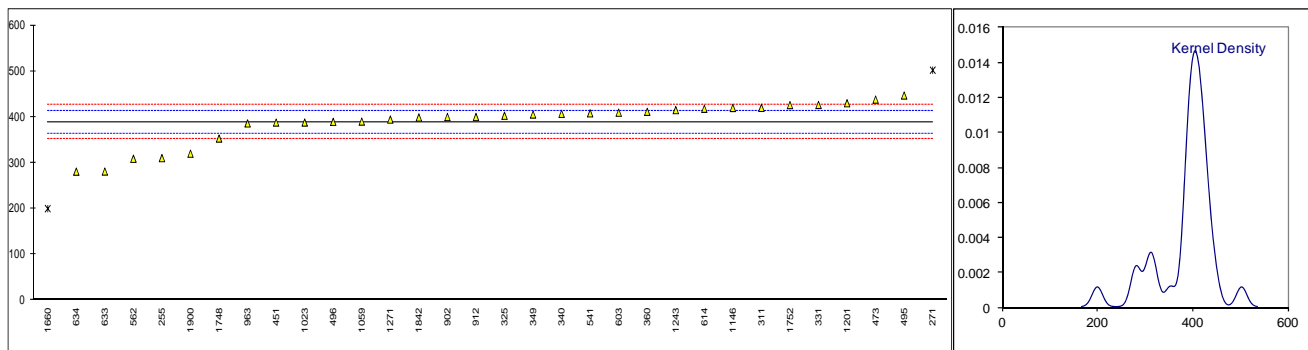
Determination of Phosphorus (P) on sample #13205; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	1179		0.19	
255		-----		-----	
271	D5185	1362.1	C	3.67	First reported 1626
311	D5185	1110		-1.13	
325	D5185	1087.5		-1.56	
331	D5185	1183.5		0.27	
335		-----		-----	
340	D5185	1089		-1.53	
349	D5185	1125.98		-0.82	
360	D5185	1208		0.74	
398		-----		-----	
432		-----		-----	
451	D5185	1076		-1.78	
473	D5185	1237.40		1.30	
495	D5185	1169.0		-0.01	
496	D5185	1015		-2.94	
541	D5185	1120		-0.94	
551		-----		-----	
562	D6595	962.2		-3.94	
603	D5185	1565	G(0.05)	7.54	
614	D5185	1148		-0.41	
633	D6595	1376.8		3.95	
634	D6595	1346.7		3.38	
902	D5185	1318		2.83	
912	D5185	1146		-0.44	
963	D5185	946.9		-4.23	
1017		-----		-----	
1023	D5185	1034		-2.58	
1059	in house	1044		-2.39	
1146	in house	1147		-0.42	
1201	D5185	1175		0.11	
1243	D5185	1087		-1.57	
1271	D5185	1274.45		2.00	
1328	D5185	1139		-0.58	
1403		-----		-----	
1628		-----		-----	
1660	D5185	1340		3.25	
1686		-----		-----	
1748	D4927	1316	C	2.79	First reported 0.1316
1752		-----		-----	
1768		-----		-----	
1842	INH-01	1170		0.01	
1900	D6595	1314		2.76	
1915		-----		-----	
7011		-----		-----	

normality	OK	
n	31	
outliers	1	
mean (n)	1169.28	
st.dev. (n)	117.182	
R(calc.)	328.11	Compare R(Horwitz) = 180.90
R(D5185:13)	147.04	Application range ASTM D5185: 10 – 1000 mg/kg
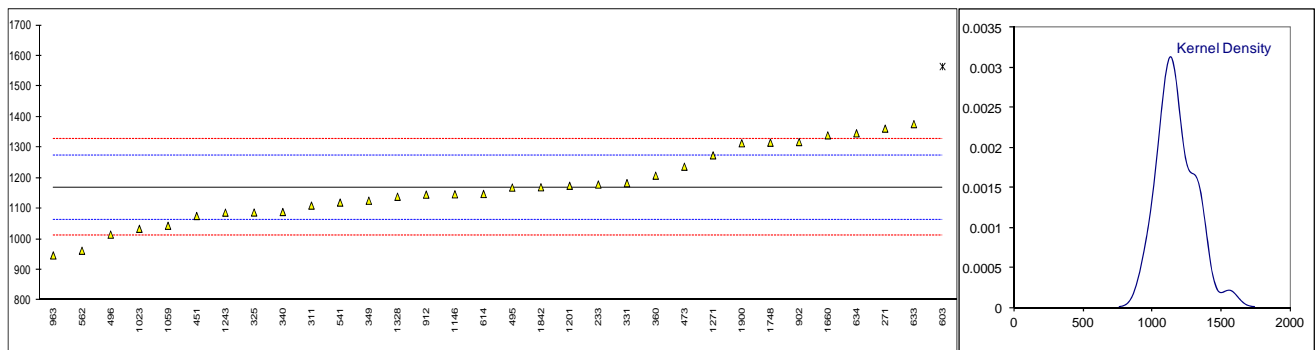
Determination of Zinc (Zn) on sample #13205; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	1354		1.07	
255	INH-OLI	1173.50		-1.27	
271	D5185	1438.4	C	2.17	First reported 1711
311	D5185	1210		-0.80	
325	D5185	1210.5		-0.79	
331	D5185	1355.6		1.09	
335		-----		-----	
340	D5185	1229		-0.55	
349	D5185	1200.11		-0.93	
360	D5185	1234		-0.49	
398		-----		-----	
432		-----		-----	
451	D5185	1161		-1.43	
473	D5185	1300.15		0.37	
495	D5185	1379.7		1.40	
496	D5185	1160		-1.45	
541	D5185	1240		-0.41	
551		-----		-----	
562	D6595	1381.2		1.42	
603	D5185	1249		-0.29	
614	D5185	1258		-0.18	
633	D6595	1448.7	C	2.30	First reported 1573.8
634	D6595	1439.7		2.18	
902	D5185	1177		-1.23	
912	D5185	1281		0.12	
963	D5185	1275		0.05	
1017		-----		-----	
1023	D5185	1128		-1.86	
1059	in house	1171		-1.30	
1146	in house	1293		0.28	
1201	D5185	1225		-0.60	
1243	D5185	1237		-0.45	
1271	D5185	1420.35		1.93	
1328	D5185	1189		-1.07	
1403	ISO11885	1865.09	G(0.01)	7.71	
1628		-----		-----	
1660	D5185	1300		0.37	
1686		-----		-----	
1748	D4927	1277	C	0.07	First reported 0.1277
1752	in house	1318		0.60	
1768		-----		-----	
1842	INH-01	1190		-1.06	
1900	D6595	1328		0.73	
1915		-----		-----	
7011		-----		-----	

normality	OK	
n	34	
outliers	1	
mean (n)	1271.53	
st.dev. (n)	89.285	
R(calc.)	250.00	Compare R(Horwitz) = 194.25
R(D5185:13)	215.69	Application range ASTM D5185: 60 – 1600 mg/kg
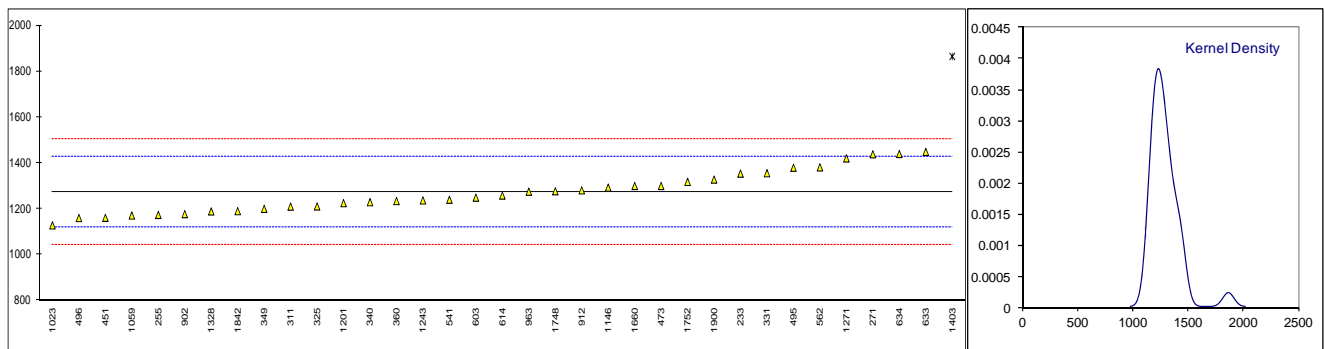
APPENDIX 2

Number of participants per country

    2 laboratories in　AUSTRALIA
    1 laboratory in　AUSTRIA
    2 laboratories in　BELGIUM
    1 laboratory in　BOSNIA and HERZEGOVIN
    1 laboratory in　BRAZIL
    1 laboratory in　BULGARIA
    1 laboratory in　CHILE
    1 laboratory in　ESTONIA
    3 laboratories in　FRANCE
    3 laboratories in　GERMANY
    1 laboratories in　GHANA
    1 laboratory in　INDIA
    1 laboratory in　IRAN
    2 laboratories in　ITALY
    1 laboratory in　JORDAN
    1 laboratory in　MALAYSIA
    3 laboratories in　NORWAY
    1 laboratory in　P.R. of CHINA
    2 laboratories in　PHILIPPINES
    1 laboratory in　POLAND
    2 laboratories in　SAUDI ARABIA
    1 laboratory in　SERBIA
    1 laboratory in　SLOVENIA
    1 laboratory in　SOUTH AFRICA
    1 laboratory in　SPAIN
    1 laboratory in　SWEDEN
    1 laboratory in　TANZANIA
    3 laboratories in　THE NETHERLANDS
    1 laboratory in　TURKEY
    2 laboratories in　UNITED KINGDOM
    1 laboratory in　UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations:

C   = final result after checking of first reported suspect 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
ex   = excluded from calculations
n.a.   = not applicable
W   = withdrawn result on request of participant
SDS   = Safety Data Sheet

Literature:

1   iis Interlaboratory Studies, Protocol for the Organization, Statistics and Evaluation, January 2010
2   ASTM E178-89
3   ASTM E1301-89
4   ISO 5725-86
5   ISO 5725, parts 1-6, 1994
6   ISO 13528-05
7   M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
8   W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
9   IP 367/84
10   DIN 38402 T41/42
11   P.L. Davies, First reported Z. Anal. Chem, 331, 513, (1988)
12   J.N. Miller, Analyst, 118, 455, (1993)
13   Analytical Methods Committee Technical Brief, No4 January 2001
14   The Royal Society of Chemistry 2002, Analyst 2002, 127 page1359-1364, P.J. Lowthian and M. Thompson (see http://www.rsc.org/suppdata/an/b2/b205600n/).