

Results of Proficiency Test  
Hydraulic Fluid (used)  
November 2011

Organised by: Institute for Interlaboratory Studies  
Spijkenisse, the Netherlands

Author: ing. R.J. Starink  
Corrector: dr. R.G. Visser & ing. L. Sweere  
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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 2011/2012. In this interlaboratory study, 38 laboratories from 25 different countries have participated. See appendix 2 for the number of participants per country. In this report, the test results of the 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 H576 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 ISO guide 43, ILAC-G13:2007 and ISO17043:2010. 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 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 present 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 45 litre of the first bulk material was homogenised in

a precleaned drum. After homogenisation, 43 subsamples were transferred to 1 litre amber glass bottles, and labelled #11102. The homogeneity of the subsamples #11102 was checked by determination of Density in accordance with ASTM D4052:09 and Water according to ASTM D6304:07 on 8 stratified randomly selected samples.

	<i>Density @ 15 °C in kg/L</i>	<i>Water in %M/M</i>
Sample #11102-1	0.87084	0.0107
Sample #11102-2	0.87084	0.0112
Sample #11102-3	0.87087	0.0111
Sample #11102-4	0.87087	0.0111
Sample #11102-5	0.87087	0.0111
Sample #11102-6	0.87087	0.0109
Sample #11102-7	0.87085	0.0110
Sample #11102-8	0.87087	0.0111

table 1: homogeneity test results of subsamples #11102

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:

	<i>Density @ 15 °C in kg/L</i>	<i>Water in %M/M</i>
r (Observed)	0.00004	0.0004
reference method	D4052:09	D6304:07
0.3 * R (ref. method)	0.00015	0.0085

table 2: repeatabilities of subsamples #11102

The approximately 5 litre of the second bulk material, positive on wear metals, was homogenised and was transferred to 40 subsamples of 100 mL HDPE containers, labelled #11103. The homogeneity of the subsamples #11103 was checked by determination of Phosphorus in accordance with ASTM D5185:09 on 8 stratified randomly selected samples.

	<i>Phosphorus in mg/kg</i>
Sample #11103-1	434
Sample #11103-2	437
Sample #11103-3	437
Sample #11103-4	439
Sample #11103-5	434
Sample #11103-6	428
Sample #11103-7	432
Sample #11103-8	428

table 3: homogeneity test results of subsamples #11103

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:

	<i>Phosphorus in mg/kg</i>
r (Observed)	11.5
reference method	D5185:09
0.3* R (ref. method)	26.9

table 4: repeatability of subsamples #11103

Each calculated repeatability was equal or less than 0.3 times the corresponding reproducibility of the respective test method. Therefore, homogeneity of the subsamples #11102 and #11103 was assumed.

To each of the participating laboratories was dispatched: One 1 litre amber glass bottle, labelled #11102 and one 100 mL HDPE container, labelled #11103 on October 26, 2011.

## 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, Extractable Organic Halogens, Total Organic Halogens and Water on sample #11102 and 20 elements (17 wear metals and 3 additives) on sample #11103.

To get comparable results a detailed report form, on which the units were prescribed, was sent together with each set of samples. In addition, a letter of instructions and a SDS were added to the 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, nr.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:

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

The  $Z_{(\text{target})}$  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 received the samples late. Two laboratories reported the results after the final reporting date and two other laboratories did not report any results at all.

The 36 reporting participants sent in 667 numerical results. Observed were 65 outlying results, which is 9.7% 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 abnormal distribution was found for the following determination: Acid Number, Density, Aluminium, Lead, Magnesium, Sodium, Silicon and Tin. In this case the statistical evaluation 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 ware that the sample "metals only" (#11103) contained a large number of elements, spectral interferences might explain the large spread found for some elements.*

Acid Number (Total): This determination was very problematic. Only one statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of ASTM D664:11a (or ASTM D974:11). *The large spread may be explained by the difference in volume found by using inflection-point titration or by end-point titration (see ASTM D664:11a, fig.1 and 13.1.2).*

Density @ 15°C: This determination was problematic for a number of laboratories. Seven statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ASTM D4052:09.

Flash Point PMcc: This determination was not problematic. Only one statistical outlier was observed and the calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of ASTM D93:11 method B.

Kin.Visco.@ 40°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 D445:11a.

Kin.Visco.@ 100°C: 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 D445:11a.

Extractable Organic Halogens: For this determination, only one numerical result was reported. Therefore no significant conclusions were drawn.

Water: This determination was not problematic. Three statistical outliers were observed. However, 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).



- Aluminium: This determination was not problematic. Only two statistical outliers were observed and the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D5185:09.
- Barium: As the average concentration found by the group (29.7 mg/kg) is above the application range given in ASTM D5185:09 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 D5185:09. This determination may not be problematic. Only one statistical outlier was observed and the calculated reproducibility after rejection of the statistical outlier is in good agreement with the estimated requirements calculated using the Horwitz equation.
- Chromium: This determination was not problematic. Only two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D5185:09.
- Copper: This determination was not problematic. Only one statistical outlier was observed and the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D5185:09.
- Iron: This determination was not problematic. One statistical outlier was observed and the calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of ASTM D5185:09.
- Lead: This determination was not problematic at this low level (6.3 mg/kg). Although the consensus value found is below the application range of the test method, no statistical outliers were observed and the calculated reproducibility is in full agreement with the requirements of ASTM D5185:09.
- Lithium: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with the estimated requirements, calculated using the Horwitz equation.
- Magnesium: This determination was not problematic. Two statistical outliers were detected. The calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ASTM D5185:09.
- Manganese: This determination was problematic. Two statistical outliers were detected and the calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D5185:09.
- Molybdenum This determination was problematic for a number of laboratories. Three statistical outliers were observed. However, the calculated reproducibility

after rejection of the statistical outliers is in good agreement with the requirements of ASTM D5185:09

- Nickel: This determination was not problematic. Five statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D5185:09.
- Sodium: 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 D5185:09.
- Silicon: This determination was not problematic at this low level (7.2 mg/kg). Although the consensus value found is below the application range of the test method, one false negative and three statistical outliers were observed. However, the calculated reproducibility is in good agreement with the requirements of ASTM D5185:09.
- Silver: This determination was problematic for a number of laboratories. Five statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D5185:09.
- Tin: This determination was not problematic at this low level (6.0 mg/kg). Although the consensus value found is below the application range of the test method, three statistical outliers were observed. However, the calculated reproducibility is in good agreement with the requirements of ASTM D5185:09.
- Titanium: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D5185:09.
- Vanadium: This determination was problematic for a number of laboratories. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outlier was almost in agreement with the requirements of ASTM D5185:09.
- Calcium: As the average concentration found by the group (23.5 mg/kg) is below the application range given in ASTM D5185:09 table 3 (40-9000 mg/kg), it was decided to use the estimated reproducibility calculated using the Horwitz equation instead of the reproducibility of ASTM D5185:09. This determination was problematic for a number of laboratories. Three statistical outliers were observed. However, the calculated reproducibility

after rejection of the statistical outliers is in agreement with the estimated requirements, calculated using the Horwitz equation.

Phosphorus:

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 D5185:09.

Zinc:

As the average concentration found by the group (29.4 mg/kg) is below the application range given in ASTM D5185:09 table 3 (60-1600 mg/kg), it was decided to use the estimated reproducibility calculated using the Horwitz equation instead of the reproducibility of ASTM D5185:09. This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full agreement with the estimated requirements, calculated using the Horwitz equation.

#### 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).

<i>Parameter</i>	<i>Unit</i>	<i>n</i>	<i>Average</i>	<i>2.8 * sd</i>	<i>R(lit)</i>
Total Acid Number	mg KOH/g	29	0.103	0.111	0.045
Density @ 15 °C	kg/L	23	0.8709	0.0005	0.0005
Flash Point PMcc	°C	28	199.95	9.91	10.00
Kinematic viscosity @ 40 °C	mm/s <sup>2</sup>	31	30.489	0.447	0.232
Kinematic viscosity @ 100 °C	mm/s <sup>2</sup>	30	5.3214	0.1078	0.0404
Organic Halogens Extractable	mg/kg	1	n.a.	n.a.	n.a.
Water	%M/M	25	0.0091	0.0095	0.0253

table 5: reproducibilities of results of sample #11102.

<i>Parameter</i>	<i>Unit</i>	<i>n</i>	<i>average</i>	<i>2.8 * sd</i>	<i>R (lit)</i>
Aluminium as Al	mg/kg	26	5.55	4.21	5.93
Barium as Ba	mg/kg	21	29.69	6.89	(7.99)
Chromium as Cr	mg/kg	25	5.75	1.86	2.35
Copper as Cu	mg/kg	27	22.64	4.76	5.43
Iron as Fe	mg/kg	26	24.94	6.93	6.82
Lead as Pb	mg/kg	26	6.26	6.08	(5.81)
Lithium as Li	mg/kg	9	31.45	7.49	8.38
Magnesium as Mg	mg/kg	23	5.71	2.67	2.75
Manganese as Mn	mg/kg	19	5.81	1.82	1.07
Molybdenum as Mo	mg/kg	18	5.41	1.96	2.12
Nickel as Ni	mg/kg	21	5.84	2.06	3.62
Silicon as Si	mg/kg	20	7.16	4.18	(6.25)
Silver as Ag	mg/kg	15	3.51	1.33	1.32
Sodium as Na	mg/kg	20	7.41	7.97	4.56
Tin as Sn	mg/kg	18	5.97	3.38	(6.36)
Titanium as Ti	mg/kg	18	5.68	1.37	5.66
Vanadium as V	mg/kg	20	5.72	2.16	1.91
Calcium as Ca	mg/kg	26	23.49	7.15	(6.54)
Phosphorus as P	mg/kg	24	446.42	102.78	90.85
Zinc as Zn	mg/kg	27	29.36	7.92	(7.91)

table 6: reproducibilities of results of sample #11103

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 2011 WITH THE PREVIOUS PTs.

	<i>November 2011</i>	<i>November 2010</i>	<i>November 2009</i>	<i>November 2008</i>
Number of reporting labs	36	30	30	25
Number of results reported	667	434	520	418
Statistical outliers	65	36	35	32
Percentage outliers	9.7%	8.3%	6.7%	7.7%

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	<i>November 2011</i>	<i>November 2010</i>	<i>November 2009</i>	<i>November 2008</i>
Total Acid Number	--	+	++	++
Density @ 15 °C	+/-	--	--	--
Flash Point PMcc	+/-	++	--	--
Kinematic viscosity @ 40 °C	--	--	--	--
Kinematic viscosity @ 100 °C	--	--	--	--
Organic Halogens Extractable	n.e.	n.e.	n.e.	n.e.
Water	++	++	++	++
Aluminium as Al	+	++	++	++
Barium as Ba	(+)	(++)	(++)	(++)
Calcium as Ca	(+/-)	(+)	++	--
Chromium as Cr	++	-	-	++
Copper as Cu	+	--	++	+/-
Iron as Fe	+/-	++	+	++
Lead as Pb	(+/-)	++	++	++
Lithium as Li	+	++	+	(++)
Magnesium as Mg	+/-	++	++	++
Manganese as Mn	--	-	++	++
Molybdenum	+	--	++	n.e.
Nickel as Ni	++	++	+	+/-
Phosphorus as P	(-)	--	++	-
Silicon as Si	(++)	++	++	++
Silver as Ag	+/-	++	++	n.e.
Sodium as Na	(--)	++	++	++
Tin as Sn	(++)	++	(++)	++
Titanium as Ti	++	++	++	n.e.
Vanadium as V	(-)	++	++	++
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
- n.e.: not evaluated

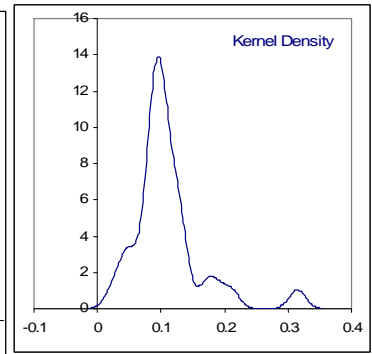
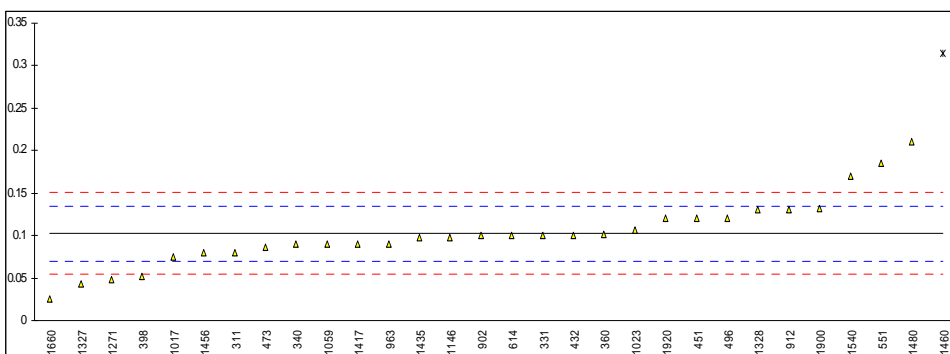
**APPENDIX 1**

Determination of Acid Number (Total) on sample #11102; results in mg KOH/g.

lab	method	value	mark	z(targ)	remarks
255		-----		-----	
311	D664	0.08		-1.40	
331	D664	0.10		-0.16	
340	D664	0.09		-0.78	
360	D664	0.101		-0.10	
398	D664	0.052		-3.14	
432	D664	0.100		-0.16	
451	D664	0.12		1.08	
473	D664	0.0864		-1.00	
496	D664	0.121		1.15	
551	D664	0.185		5.12	
613		-----		-----	
614	D664	0.10		-0.16	
902	D664	0.10		-0.16	
912	D664	0.131		1.77	
963	D664	0.0903		-0.76	
1017	D974	0.0754		-1.68	
1023	D664	0.107		0.28	
1059	ISO6619	0.09		-0.78	
1146	D664	0.098		-0.28	
1243		-----		-----	
1271	D664	0.0487		-3.34	
1295		-----		-----	
1327	D664	0.043		-3.70	
1328	INH-7304	0.130		1.70	
1417	in house	0.09		-0.78	
1435	D664	0.098		-0.28	
1456	D974	0.08		-1.40	
1460	D664	0.315	G(0.01)	13.18	
1480	D664	0.21		6.67	
1540	D664	0.170		4.19	
1628		-----		-----	
1630		-----		-----	
1660	D664	0.025		-4.81	
1722		-----		-----	
1900	D664	0.132		1.83	
1920	D664	0.120		1.08	
7003		-----		-----	

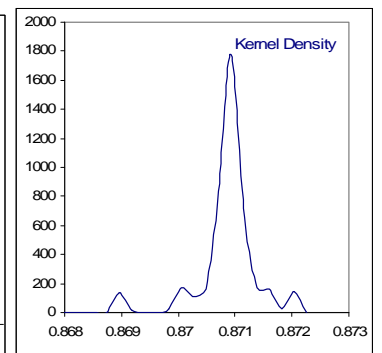
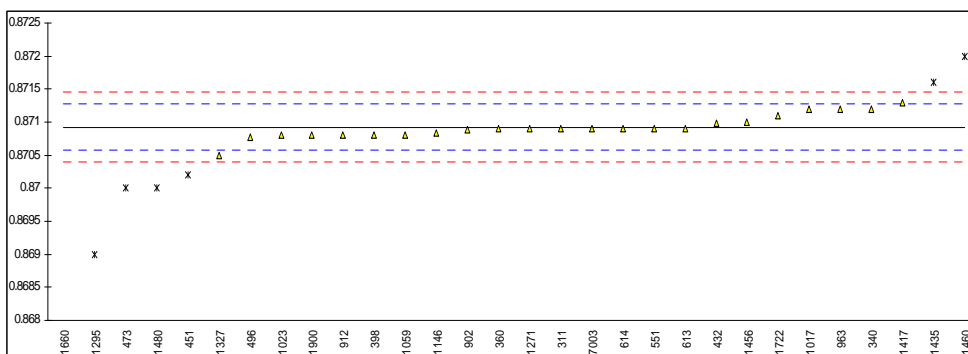
normality not OK  
n 29  
outliers 1  
mean (n) 0.1025  
st.dev. (n) 0.03965  
R(calc.) 0.1110  
R(D664:11a) 0.0451

Compare R(D974) = 0.0400



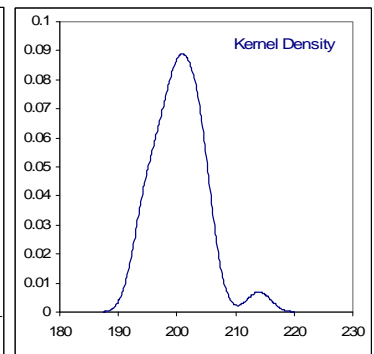
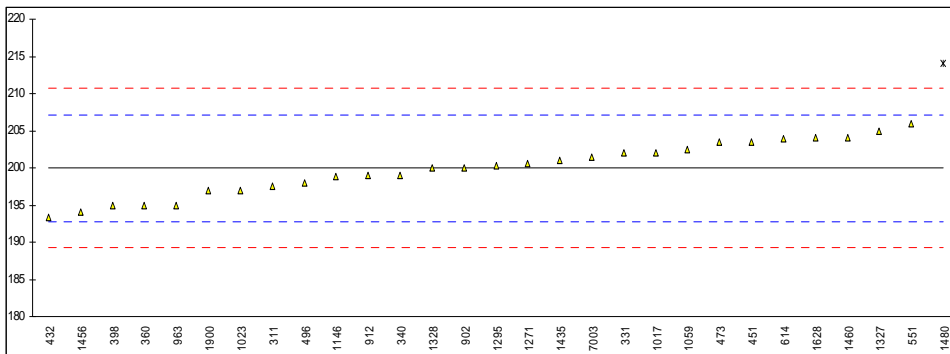
Determination of Density @ 15°C on sample #11102; results in kg/L.

lab	method	value	mark	z(targ)	remarks
255		-----		-----	
311	D4052	0.8709		-0.14	
331		-----		-----	
340	D4052	0.8712	C	1.54	First reported 871.21
360	D4052	0.8709		-0.14	
398	D4052	0.8708		-0.70	
432	D4052	0.87099		0.36	
451	D4052	0.8702	G(0.05)	-4.06	
473	D4052	0.8700	G(0.05)	-5.18	
496	D4052	0.87077		-0.87	
551	D4052	0.8709		-0.14	
613	D4052	0.87091		-0.09	
614	D4052	0.8709		-0.14	
902	D4052	0.87089		-0.20	
912	D4052	0.8708		-0.70	
963	D4052	0.8712		1.54	
1017	D4052	0.8712		1.54	
1023	D4052	0.8708		-0.70	
1059	D4052	0.8708		-0.70	
1146	D4052	0.87083		-0.54	
1243		-----		-----	
1271	D4052	0.8709	C	-0.14	First reported 0.8697
1295	ISO3675	0.869	G(0.01)	-10.78	
1327	D4052	0.8705		-2.38	
1328		-----		-----	
1417	in house	0.8713		2.10	
1435	D4052	0.8716	DG(0.05)	3.78	
1456	D4052	0.8710		0.42	
1460	D7042	0.8720	DG(0.05)	6.02	
1480	D4052	0.870	G(0.01)	-5.18	
1540		-----		-----	
1628		-----		-----	
1630		-----		-----	
1660	D4052	0.8164	G(0.01)	-305.34	
1722	D4052	0.8711		0.98	
1900	D4052	0.8708	C	-0.70	First reported 870.8
1920		-----		-----	
7003	D4052	0.8709		-0.14	
normality		not OK			
n		23			
outliers		7			
mean (n)		0.87093			
st.dev. (n)		0.000179			
R(calc.)		0.00050			
R(D4052:09)		0.00050			



Determination of Flash Point PMcc on sample #11102; results in °C.

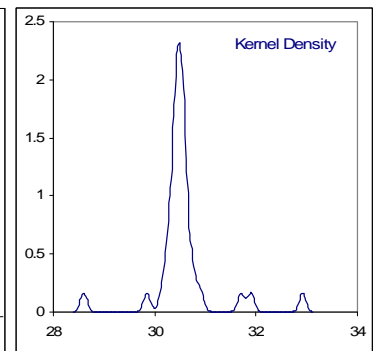
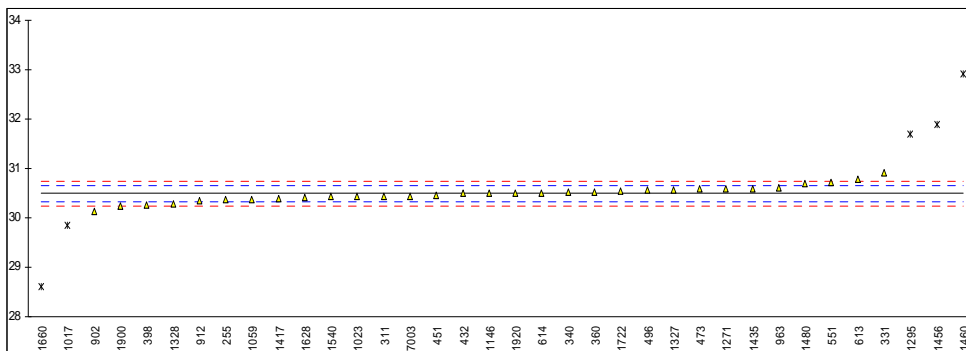
lab	method	value	mark	z(targ)	remarks
255		-----		-----	
311	D93-AE	197.5		-0.68	
331	D93-AE	202		0.58	
340	D93-AF	199.0		-0.26	
360	D93-AE	195.0		-1.38	
398	D93-AE	195		-1.38	
432	D93-AE	193.3		-1.86	
451	D93-AE	203.5		1.00	
473	D93-AE	203.5		1.00	
496	D93-AE	198		-0.54	
551	D93-AF	206		1.70	
613		-----		-----	
614	D93-MF	203.9		1.11	
902	D93-AE	200		0.02	
912	D93-AE	199	C	-0.26	First reported 209
963	D93-MF	195.0		-1.38	
1017	D93-AE	202.0		0.58	
1023	D93-AE	197		-0.82	
1059	ISO2719-AE	202.5		0.72	
1146	D93-AE	198.9		-0.29	
1243		-----		-----	
1271	D93-AF	200.6		0.18	
1295	ISO2719	200.275	C	0.09	First reported 224.77
1327	D93-AF	205.0		1.42	
1328	INH-261	200.0		0.02	
1417		-----		-----	
1435	D93-AE	201		0.30	
1456	D93-MF	194.0		-1.66	
1460	D93-AE	204.0		1.14	
1480	D93-MF	214.00	G(0.05)	3.94	
1540		-----		-----	
1628	ISO2719-AE	204.0		1.14	
1630		-----		-----	
1660		-----		-----	
1722		-----		-----	
1900	D3828-AF	197		-0.82	
1920		-----		-----	
7003	D93	201.5		0.44	
	normality	OK			
	n	28			
	outliers	1			
	mean (n)	199.95			
	st.dev. (n)	3.538			
	R(calc.)	9.91			
	R(D93:11-B)	10.00			





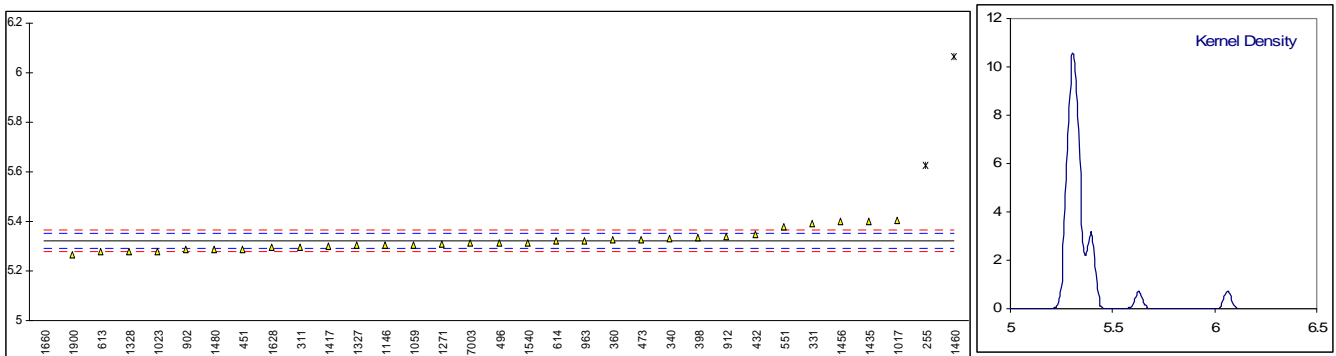
Determination of Kinematic Viscosity @ 40°C on sample #11102; results in mm/s<sup>2</sup>.

lab	method	value	mark	z(targ)	remarks
255	D7279	30.3688		-1.45	
311	D445	30.43		-0.72	
331	D7279	30.91		5.09	
340	D445	30.515		0.31	
360	D445	30.517		0.34	
398	D445	30.260		-2.77	
432	D445	30.490		0.01	
451	D445	30.46		-0.35	
473	D445	30.581		1.11	
496	D445	30.559		0.84	
551	D445	30.7125		2.70	
613	D445	30.79		3.64	
614	D445	30.5		0.13	
902	D445	30.14		-4.22	
912	D445	30.35		-1.68	
963	D445	30.61		1.46	
1017	D445	29.8475	G(0.05)	-7.75	
1023	D445	30.43		-0.72	
1059	D445	30.38		-1.32	
1146	D445	30.497		0.09	
1243		-----		-----	
1271	ISO3104	30.59		1.22	
1295	ISO3104	31.705	G(0.01)	14.69	
1327	D445	30.56		0.86	
1328	INH-265	30.28		-2.53	
1417	in house	30.4		-1.08	
1435	D7042	30.593		1.25	
1456	D445	31.9	G(0.01)	17.05	
1460	D445	32.91999	G(0.01)	29.37	
1480	D445	30.686		2.38	
1540	D445	30.429		-0.73	
1628	ISO3104	30.403		-1.04	
1630		-----		-----	
1660	D445	28.6	G(0.05)	-22.83	
1722	D445	30.5370		0.58	
1900	D445	30.25		-2.89	
1920	D445	30.498		0.11	
7003	D7042	30.438		-0.62	
normality		OK			
n		31			
outliers		5			
mean (n)		30.489			
st.dev. (n)		0.1597			
R(calc.)		0.447			
R(D445:11a)		0.232			



Determination of Kinematic Viscosity @ 100°C on sample #11102; results in mm/s<sup>2</sup>

lab	method	value	mark	z(targ)	remarks
255	D7279	5.628	C,G(0.01)	21.23	First reported 5.5477
311	D445	5.297		-1.69	
331	D7279	5.39		4.75	
340	D445	5.3315		0.70	
360	D445	5.3269		0.38	
398	D445	5.3361		1.02	
432	D445	5.3500		1.98	
451	D445	5.289		-2.24	
473	D445	5.327		0.39	
496	D445	5.3139		-0.52	
551	D445	5.3781		3.93	
613	D445	5.28		-2.86	
614	D445	5.32		-0.09	
902	D445	5.287		-2.38	
912	D445	5.337		1.08	
963	D445	5.321		-0.02	
1017	D445	5.4022		5.60	
1023	D445	5.28		-2.86	
1059	D445	5.304		-1.20	
1146	D445	5.3032		-1.26	
1243		-----		-----	
1271	ISO3104	5.31		-0.79	
1295		-----		-----	
1327	D445	5.303		-1.27	
1328	INH-265	5.280		-2.86	
1417	in house	5.3		-1.48	
1435	D7042	5.4		5.45	
1456	D445	5.4		5.45	
1460	D445	6.063893	G(0.01)	51.41	
1480	D445	5.2874		-2.35	
1540	D445	5.3145		-0.47	
1628	ISO3104	5.295		-1.82	
1630		-----		-----	
1660	D445	4.8	G(0.05)	-36.10	
1722		-----		-----	
1900	D445	5.265		-3.90	
1920		-----		-----	
7003	D7042	5.3118		-0.66	
	normality	OK			
	n	30			
	outliers	3			
	mean (n)	5.3214			
	st.dev. (n)	0.03851			
	R(calc.)	0.1078			
	R(D445:11a)	0.0404			

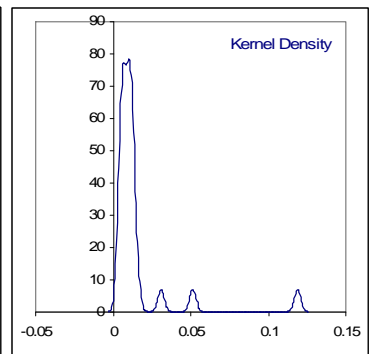
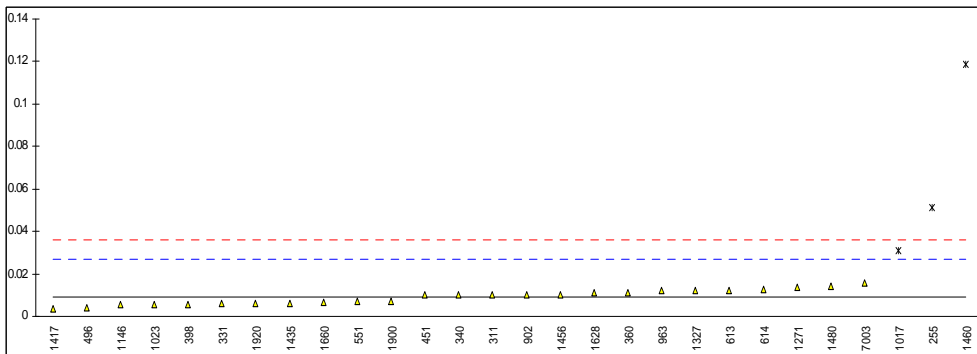


## Determination of Extractable Organic Chloride on sample #11102; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255		----		----	
311		----		----	
331		----		----	
340		----		----	
360		----		----	
398		----		----	
432		----		----	
451		----		----	
473		----		----	
496		----		----	
551		----		----	
613		----		----	
614		----		----	
902		----		----	
912		----		----	
963		----		----	
1017		----		----	
1023		----		----	
1059		----		----	
1146		----		----	
1243		----		----	
1271		----		----	
1295		----		----	
1327		----		----	
1328		----		----	
1417		----		----	
1435		----		----	
1456		----		----	
1460		----		----	
1480		----		----	
1540		----		----	
1628		----		----	
1630		----		----	
1660		----		----	
1722		----		----	
1900	in house	105		----	
1920		----		----	
7003		----		----	
	normality	n.a.			
	n	1			
	outliers	0			
	mean (n)	n.a.			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(lit)	n.a.			

Determination of Water on sample #11102; results in %M/M.

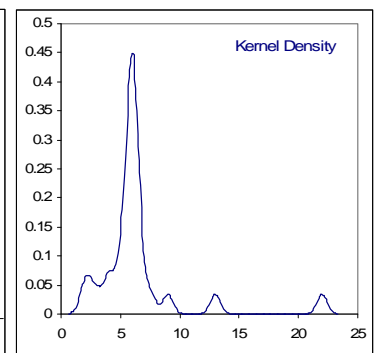
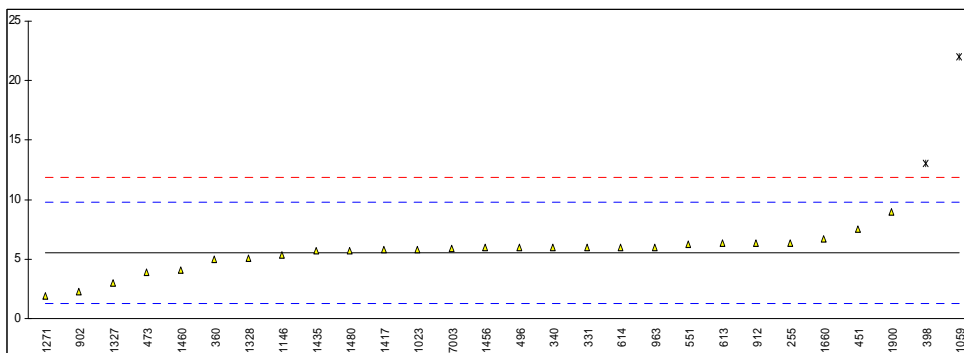
lab	method	Value	mark	z(targ)	remarks
255	ISO9114	0.0513	C,G(0.01)	4.66	First reported 0.22 (%V/V)
311	D6304A	0.01		0.10	
331	D6304C	0.0060		-0.35	
340	D6304A	0.010		0.10	
360	D6304A	0.011		0.21	
398	D6304A	0.0056		-0.39	
432		-----		-----	
451	D6304A	0.01	C	0.10	First reported 56
473	D6304C	<0.01	C	<0.10	First reported 52.4
496	D6304C	0.0043		-0.53	
551	D6304A	0.00698		-0.24	
613	D6304A	0.0122		0.34	
614	D6304C	0.0127		0.40	
902	D6304A	0.01		0.10	
912		-----		-----	
963	D6304A	0.012		0.32	
1017	D6304A	0.03096	G(0.01)	2.41	
1023	D6304A	0.0055		-0.40	
1059	D6304mod	<0.003		<-0.68	
1146	D6304C	0.0054		-0.41	
1243		-----		-----	
1271	ISO12937	0.0139		0.53	
1295		-----		-----	
1327	D6304A	0.0121		0.33	
1328		-----		-----	
1417	in house	0.0035		-0.62	
1435	D1744	0.0063	C	-0.31	First reported 63
1456	D1744mod	0.01		0.10	
1460	D6304A	0.1189	G(0.01)	12.13	
1480	D6304A	0.0142		0.56	
1540		-----		-----	
1628	in house	0.0110		0.21	
1630		-----		-----	
1660	IEC60814	0.0068	C	-0.26	First reported 68
1722		-----		-----	
1900	D6304C	0.0071		-0.22	
1920	D6304C	0.00603		-0.34	
7003	E1064	0.0155		0.70	
					<u>Only ASTM D6304-C</u>
normality	OK			OK	
n	25			6	
outliers	3			0	
mean (n)	0.0091			0.0069	
st.dev. (n)	0.00339			0.00298	
R(calc.)	0.0095			0.0083	
R(D6304:07)	0.0253			0.0214	



Determination of Aluminium (Al) on sample #11103; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255	in house	6.3745		0.39	
311		----		----	
331	D5185	6		0.21	
340	D5185	6		0.21	
360	D5185	4.98		-0.27	
398	D6595	13.0	G(0.01)	3.52	
432		----		----	
451	D5185	7.55		0.95	
473	D5185	3.9266		-0.76	
496	D5185	6.00		0.21	
551	D5185	6.282		0.35	
613	D5185	6.3		0.36	
614	D5185	6.0		0.21	
902	D5185	2.3		-1.53	
912	D5185	6.36		0.38	
963	D5185	6.0		0.21	
1017		----		----	
1023	D5185	5.84		0.14	
1059	in house	22	G(0.01)	7.77	
1146	D5185	5.299		-0.12	
1243		----		----	
1271	in house	1.935		-1.70	
1295		----		----	
1327	D5185	3.00		-1.20	
1328	D5185	5.09		-0.21	
1417	in house	5.83		0.13	
1435	D5185	5.69		0.07	
1456	D5185	6		0.21	
1460	D5185	4.117335		-0.67	
1480	D5185	5.7		0.07	
1540		----		----	
1628		----		----	
1630		----		----	
1660	D5185	6.70		0.55	
1722		----		----	
1900	D6595	9		1.63	
1920		----		----	
7003	D3919	5.900		0.17	
normality		not OK			
n		26			
outliers		2			
mean (n)		5.55			
st.dev. (n)		1.505			
R(calc.)		4.21			
R(D5185:09)		5.93			

Application range ASTM D5185:09: 6 – 40 mg/kg

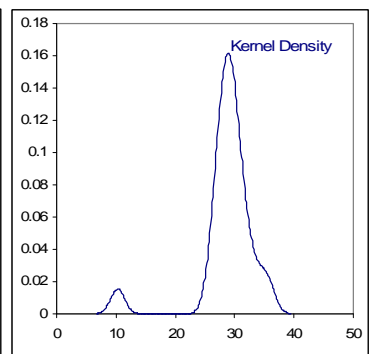
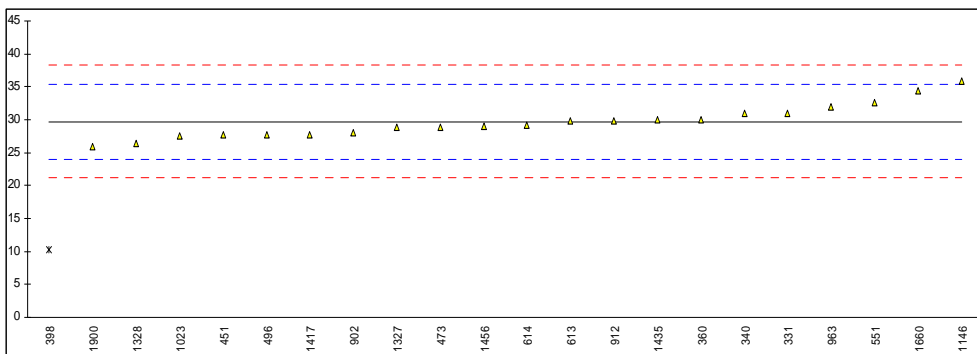


Determination of Barium (Ba) on sample #11103; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255		----		----	
311		----		----	
331	D5185	31	C	0.46	First reported 3
340	D5185	31		0.46	
360	D5185	30.0		0.11	
398	D6595	10.3	G(0.01)	-6.80	
432		----		----	
451	D5185	27.7		-0.70	
473	D5185	28.8609		-0.29	
496	D5185	27.72		-0.69	
551	D5185	32.5905		1.02	
613	D5185	29.8		0.04	
614	D5185	29.24		-0.16	
902	D5185	28.1		-0.56	
912	D5185	29.84		0.05	
963	D5185	32.0		0.81	
1017		----		----	
1023	D5185	27.6		-0.73	
1059		----		----	
1146	D5185	35.81		2.14	
1243		----		----	
1271		----		----	
1295		----		----	
1327	D5185	28.79	C	-0.32	First reported 8.27
1328	D5185	26.47		-1.13	
1417	in house	27.76		-0.68	
1435	D5185	29.94		0.09	
1456	D5185	29		-0.24	
1460		----		----	
1480		----		----	
1540		----		----	
1628		----		----	
1630		----		----	
1660	D5185	34.34		1.63	
1722		----		----	
1900	D6595	26		-1.30	
1920		----		----	
7003		----		----	

normality OK  
n 21  
outliers 1  
mean (n) 29.69  
st.dev. (n) 2.460  
R(calc.) 6.89  
R(Horwitz) 7.99

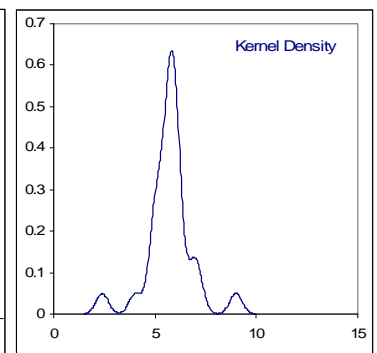
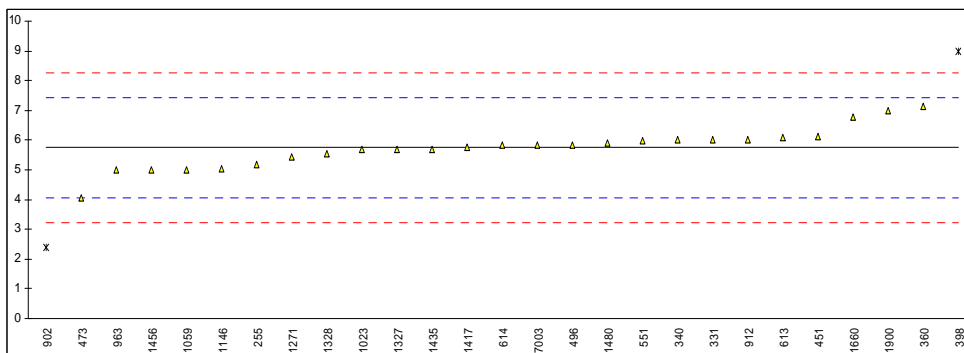
Application range ASTM D5185:09: 0.5 – 4 mg/kg



Determination of Chromium (Cr) on sample #11103; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255	in house	5.1792		-0.67	
311		----		----	
331	D5185	6		0.30	
340	D5185	6		0.30	
360	D5185	7.14		1.66	
398	D6595	9.0	G(0.05)	3.87	
432		----		----	
451	D5185	6.12		0.45	
473	D5185	4.0478		-2.02	
496	D5185	5.84		0.11	
551	D5185	5.9915		0.29	
613	D5185	6.1		0.42	
614	D5185	5.82		0.09	
902	D5185	2.4	G(0.05)	-3.98	
912	D5185	6.01		0.32	
963	D5185	5.0		-0.89	
1017		----		----	
1023	D5185	5.68		-0.08	
1059	in house	5		-0.89	
1146	D5185	5.045		-0.83	
1243		----		----	
1271	in house	5.45		-0.35	
1295		----		----	
1327	D5185	5.70		-0.05	
1328	D5185	5.55		-0.23	
1417	in house	5.76		0.02	
1435	D5185	5.70		-0.05	
1456	D5185	5		-0.89	
1460		----		----	
1480	D5185	5.9		0.18	
1540		----		----	
1628		----		----	
1630		----		----	
1660	D5185	6.77		1.22	
1722		----		----	
1900	D6595	7		1.49	
1920		----		----	
7003	D3919	5.828		0.10	
normality		OK			
n		25			
outliers		2			
mean (n)		5.75			
st.dev. (n)		0.666			
R(calc.)		1.86			
R(D5185:09)		2.35			

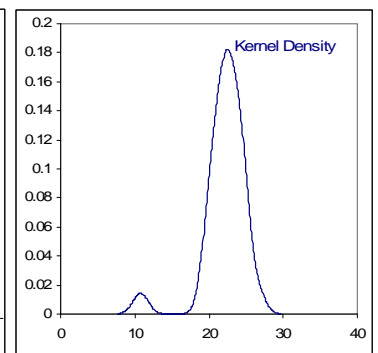
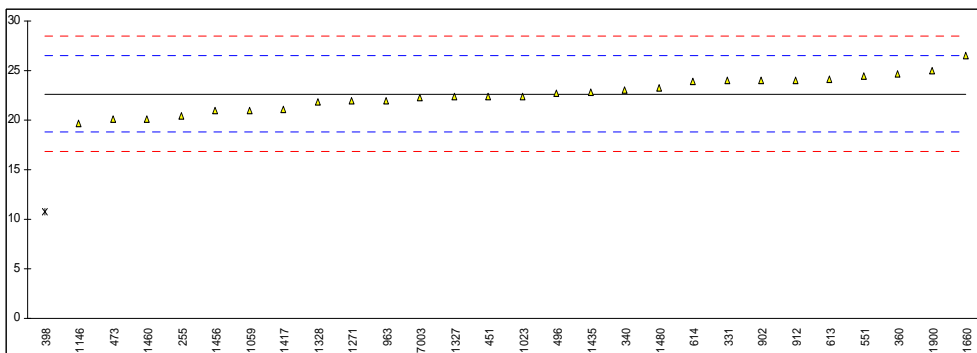
Application range ASTM D5185:09: 1 – 40 mg/kg



Determination of Copper (Cu) on sample #11103; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255	in house	20.4166		-1.14	
311		-----		-----	
331	D5185	24		0.70	
340	D5185	23		0.19	
360	D5185	24.7		1.06	
398	D6595	10.8	G(0.01)	-6.10	
432		-----		-----	
451	D5185	22.4		-0.12	
473	D5185	20.0730		-1.32	
496	D5185	22.7		0.03	
551	D5185	24.4805		0.95	
613	D5185	24.1		0.75	
614	D5185	23.89		0.65	
902	D5185	24	C	0.70	First reported 18
912	D5185	24.05		0.73	
963	D5185	22		-0.33	
1017		-----		-----	
1023	D5185	22.4		-0.12	
1059	in house	21		-0.84	
1146	D5185	19.68		-1.52	
1243		-----		-----	
1271	in house	21.95	C	-0.35	First reported 17.07
1295		-----		-----	
1327	D5185	22.4		-0.12	
1328	D5185	21.86		-0.40	
1417	in house	21.04		-0.82	
1435	D5185	22.81		0.09	
1456	D5185	21		-0.84	
1460	D5185	20.106303		-1.30	
1480	D5185	23.3		0.34	
1540		-----		-----	
1628		-----		-----	
1630		-----		-----	
1660	D5185	26.57		2.03	
1722		-----		-----	
1900	D6595	25		1.22	
1920		-----		-----	
7003	D4691	22.29		-0.18	
	normality	OK			
	n	27			
	outliers	1			
	mean (n)	22.64			
	st.dev. (n)	1.699			
	R(calc.)	4.76			
	R(D5185:09)	5.43			

Application range ASTM D5185:09: 2 – 160 mg/kg

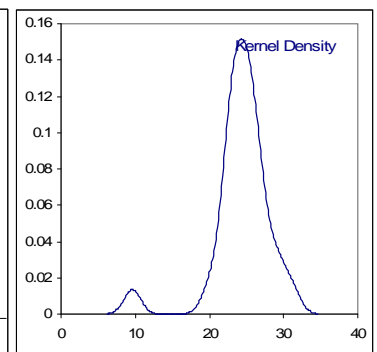
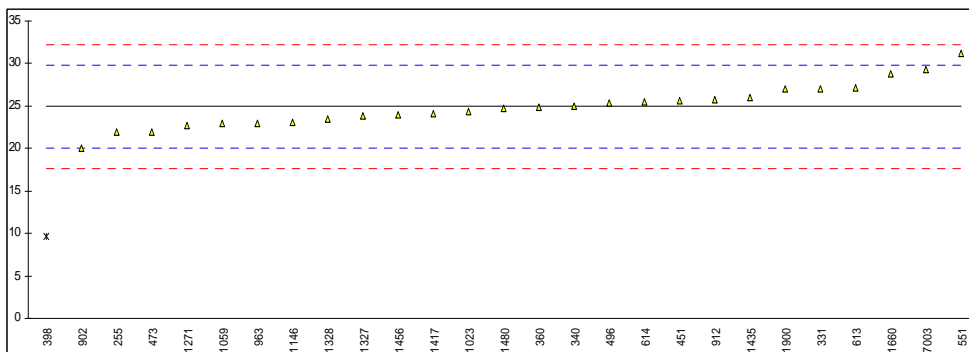




Determination of Iron (Fe) on sample #11103; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255	in house	21.883		-1.26	
311		----		----	
331	D5185	27		0.85	
340	D5185	25		0.02	
360	D5185	24.9		-0.02	
398	D6595	9.6	G(0.01)	-6.30	
432		----		----	
451	D5185	25.6		0.27	
473	D5185	21.9968		-1.21	
496	D5185	25.3		0.15	
551	D5185	31.159		2.55	
613	D5185	27.1		0.89	
614	D5185	25.45		0.21	
902	D5185	20		-2.03	
912	D5185	25.77		0.34	
963	D5185	23		-0.80	
1017		----		----	
1023	D5185	24.3		-0.26	
1059	in house	23		-0.80	
1146	D5185	23.07		-0.77	
1243		----		----	
1271	in house	22.65	C	-0.94	First reported 11.55
1295		----		----	
1327	D5185	23.89		-0.43	
1328	D5185	23.44		-0.62	
1417	in house	24.10		-0.35	
1435	D5185	26.02		0.44	
1456	D5185	24		-0.39	
1460		----		----	
1480	D5185	24.7		-0.10	
1540		----		----	
1628		----		----	
1630		----		----	
1660	D5185	28.82		1.59	
1722		----		----	
1900	D6595	27		0.85	
1920		----		----	
7003	D4691	29.35	C	1.81	First reported 35.30
normality	OK				
n	26				
outliers	1				
mean (n)	24.94				
st.dev. (n)	2.475				
R(calc.)	6.93				
R(D5185:09)	6.82				

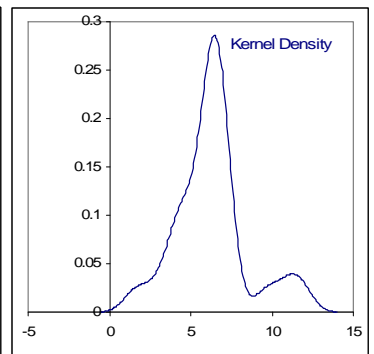
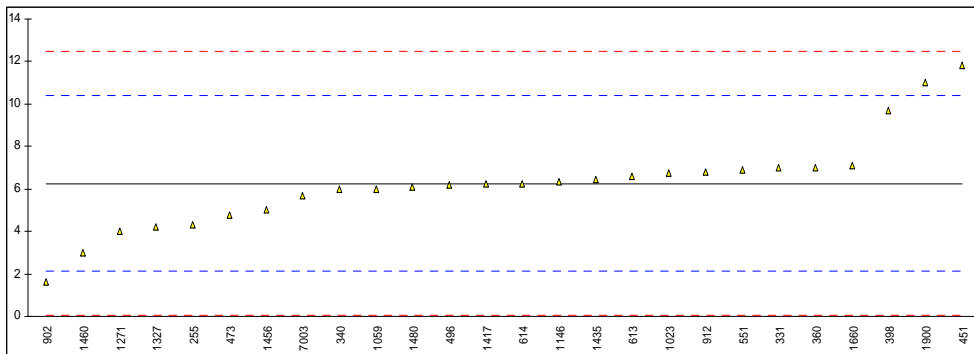
Application range ASTM D5185:09: 2 – 140 mg/kg



Determination of Lead (Pb) on sample #11103; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255	in house	4.3156		-0.94	
311		----		----	
331	D5185	7		0.36	
340	D5185	6		-0.13	
360	D5185	7.01		0.36	
398	D6595	9.7		1.66	
432		----		----	
451	D5185	11.8		2.67	
473	D5185	4.7796		-0.72	
496	D5185	6.19		-0.03	
551	D5185	6.9145		0.31	
613	D5185	6.6		0.16	
614	D5185	6.23		-0.02	
902	D5185	1.6		-2.25	
912	D5185	6.82		0.27	
963		----		----	
1017		----		----	
1023	D5185	6.74		0.23	
1059	in house	6		-0.13	
1146	D5185	6.328		0.03	
1243		----		----	
1271	in house	4		-1.09	
1295		----		----	
1327	D5185	4.23		-0.98	
1328		----		----	
1417	in house	6.22		-0.02	
1435	D5185	6.46		0.10	
1456	D5185	5		-0.61	
1460	D5185	2.971374		-1.59	
1480	D5185	6.1		-0.08	
1540		----		----	
1628		----		----	
1630		----		----	
1660	D5185	7.12		0.41	
1722		----		----	
1900	D6595	11		2.28	
1920		----		----	
7003	D3919	5.695		-0.27	
normality		not OK			
n		26			
outliers		0			
mean (n)		6.26			
st.dev. (n)		2.171			
R(calc.)		6.08			
R(D5185:09)		(5.81)			

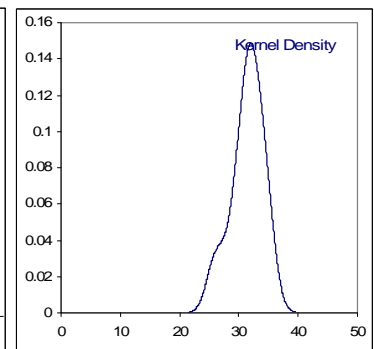
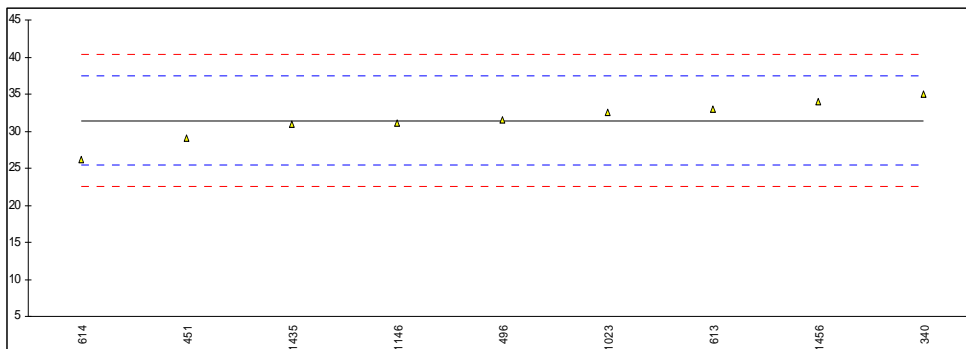
Application range ASTM D5185:09: 10 – 160 mg/kg



Determination of Lithium (Li) on sample #11103; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255		----		----	
311		----		----	
331		----		----	
340	D5185	35		1.19	
360		----		----	
398		----		----	
432		----		----	
451	D5185	29.0		-0.82	
473		----		----	
496	D5185	31.5		0.02	
551		----		----	
613	D5185	32.9		0.48	
614	D5185	26.1		-1.79	
902		----		----	
912		----		----	
963		----		----	
1017		----		----	
1023	D5185	32.5		0.35	
1059		----		----	
1146	D5185	31.09		-0.12	
1243		----		----	
1271		----		----	
1295		----		----	
1327		----		----	
1328		----		----	
1417		----		----	
1435	D5185	30.95		-0.17	
1456	D5185	34		0.85	
1460		----		----	
1480		----		----	
1540		----		----	
1628		----		----	
1630		----		----	
1660		----		----	
1722		----		----	
1900		----		----	
1920		----		----	
7003		----		----	

normality OK  
n 9  
outliers 0  
mean (n) 31.45  
st.dev. (n) 2.677  
R(calc.) 7.49  
R(Horwitz) 8.38

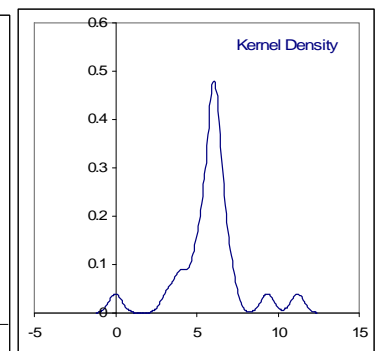
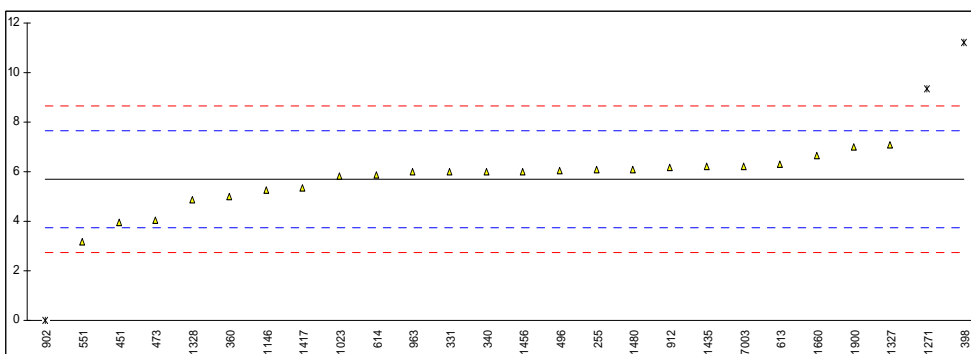


Determination of Magnesium (Mg) on sample #11103; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255	in house	6.0903		0.39	
311		-----		-----	
331	D5185	6		0.30	
340	D5185	6		0.30	
360	D5185	4.99		-0.73	
398	D6595	11.2	G(0.01)	5.59	
432		-----		-----	
451	D5185	3.97		-1.77	
473	D5185	4.0529		-1.68	
496	D5185	6.035		0.33	
551	D5185	3.1865		-2.56	
613	D5185	6.3		0.60	
614	D5185	5.85		0.15	
902	D5185	0.00	ex	-5.81	Result excluded, zero not a real result
912	D5185	6.17		0.47	
963	D5185	6.00		0.30	
1017		-----		-----	
1023	D5185	5.84		0.13	
1059	in house	<50		-----	
1146	D5185	5.255		-0.46	
1243		-----		-----	
1271	in house	9.35	G(0.05)	3.71	
1295		-----		-----	
1327	D5185	7.07		1.39	
1328	D5185	4.89		-0.83	
1417	in house	5.35		-0.36	
1435	D5185	6.21		0.51	
1456	D5185	6		0.30	
1460		-----		-----	
1480	D5185	6.1		0.40	
1540		-----		-----	
1628		-----		-----	
1630		-----		-----	
1660	D5185	6.67		0.98	
1722		-----		-----	
1900	D6595	7		1.31	
1920		-----		-----	
7003	D4691	6.239		0.54	

normality not OK  
n 23  
outliers 2  
mean (n) 5.71  
st.dev. (n) 0.953  
R(calc.) 2.67  
R(D5185:09) 2.75

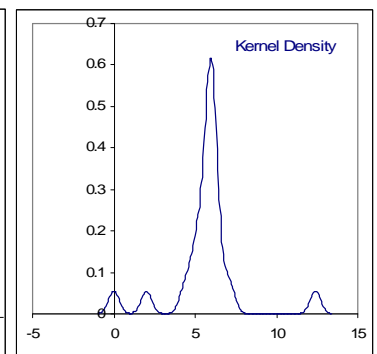
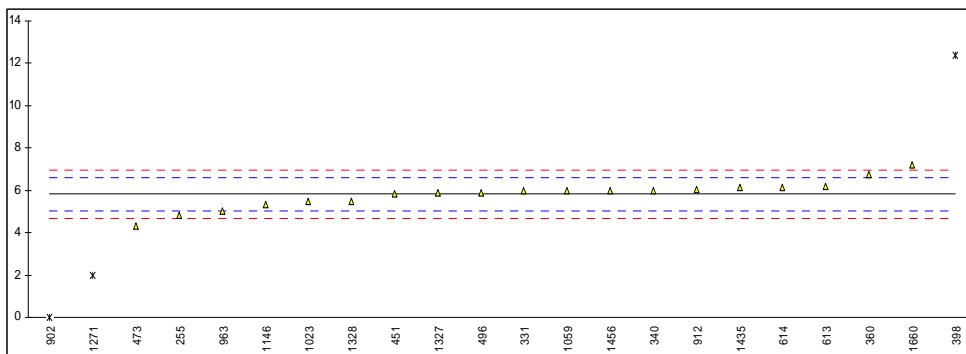
Application range ASTM D5185:09: 5 – 1700 mg/kg



Determination of Manganese (Mn) on sample #11103; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255	in house	4.8396		-2.54	
311		----		----	
331	D5185	6		0.49	
340	D5185	6		0.49	
360	D5185	6.74		2.41	
398	D6595	12.4	G(0.01)	17.16	
432		----		----	
451	D5185	5.82		0.02	
473	D5185	4.3188		-3.89	
496	D5185	5.89		0.20	
551		----		----	
613	D5185	6.2		1.01	
614	D5185	6.15		0.88	
902	D5185	0.0	ex	-15.15	Result excluded, zero not a real result
912	D5185	6.03		0.56	
963	D5185	5.0		-2.12	
1017		----		----	
1023	D5185	5.47		-0.89	
1059	in house	6		0.49	
1146	D5185	5.313		-1.30	
1243		----		----	
1271	in house	2	G(0.01)	-9.94	
1295		----		----	
1327	D5185	5.87		0.15	
1328	D5185	5.49		-0.84	
1417		----		----	
1435	D5185	6.14		0.85	
1456	D5185	6		0.49	
1460		----		----	
1480		----		----	
1540		----		----	
1628		----		----	
1630		----		----	
1660	D5185	7.18		3.56	
1722		----		----	
1900		----		----	
1920		----		----	
7003		----		----	
normality	OK				
n	19				
outliers	2				
mean (n)	5.81				
st.dev. (n)	0.650				
R(calc.)	1.82				
R(D5185:09)	1.07				

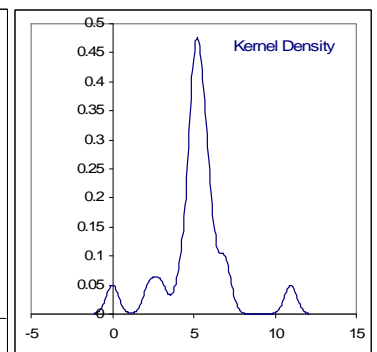
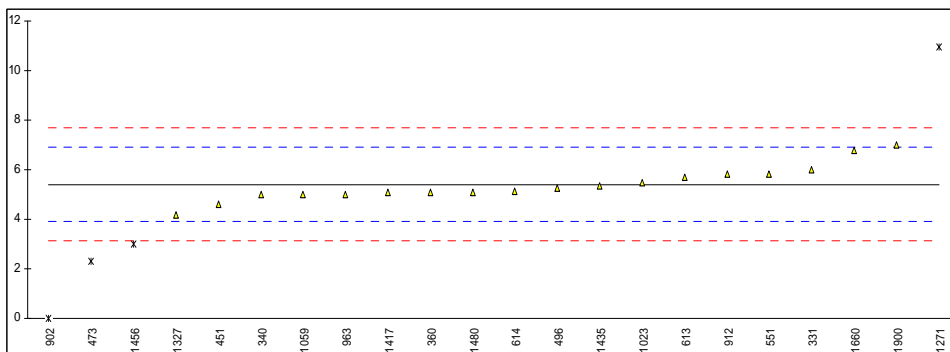
Application range ASTM D5185:09: 5 – 700 mg/kg



Determination of Molybdenum (Mo) on sample #11103; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255		----		----	
311		----		----	
331	D5185	6		0.78	
340	D5185	5		-0.54	
360	D5185	5.09		-0.42	
398		----		----	
432		----		----	
451	D5185	4.59		-1.08	
473	D5185	2.2832	DG(0.05)	-4.13	
496	D5185	5.27		-0.19	
551	D5185	5.8365		0.56	
613	D5185	5.7		0.38	
614	D5185	5.14		-0.36	
902	D5185	0.0	ex	-7.14	Result excluded, zero not a real result
912	D5185	5.83		0.55	
963	D5185	5.0		-0.54	
1017		----		----	
1023	D5185	5.48		0.09	
1059	in house	5		-0.54	
1146		----		----	
1243		----		----	
1271	in house	10.96	CG(0.01)	7.32	First reported 14
1295		----		----	
1327	D5185	4.17		-1.64	
1328		----		----	
1417	in house	5.07		-0.45	
1435	D5185	5.34		-0.09	
1456	D5185	3	DG(0.05)	-3.18	
1460		----		----	
1480	D5185	5.1		-0.41	
1540		----		----	
1628		----		----	
1630		----		----	
1660	D5185	6.79		1.82	
1722		----		----	
1900	D6595	7		2.10	
1920		----		----	
7003		----		----	
normality		OK			
n		18			
outliers		3			
mean (n)		5.41			
st.dev. (n)		0.701			
R(calc.)		1.96			
R(D5185:09)		2.12			

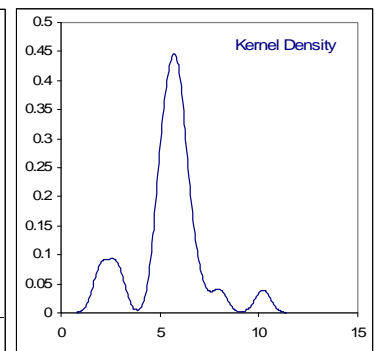
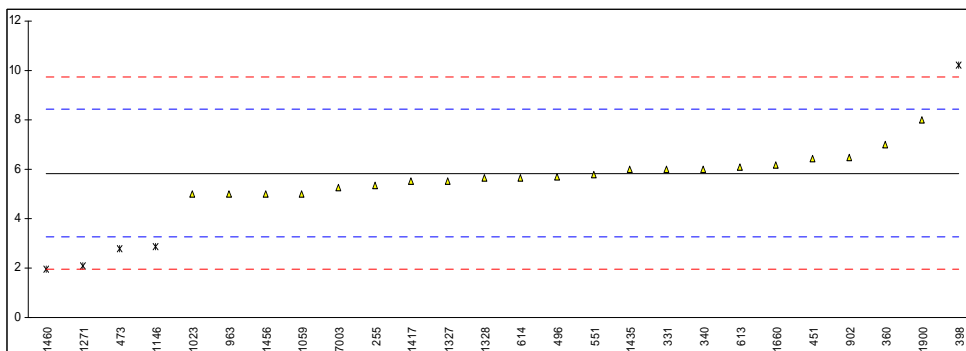
Application range ASTM D5185:09: 5 – 200 mg/kg



Determination of Nickel (Ni) on sample #11103; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255	in house	5.3593		-0.37	
311		----		----	
331	D5185	6		0.12	
340	D5185	6		0.12	
360	D5185	6.99		0.89	
398	D6595	10.2	G(0.05)	3.37	
432		----		----	
451	D5185	6.45		0.47	
473	D5185	2.7946	G(0.05)	-2.35	
496	D5185	5.69		-0.12	
551	D5185	5.7885		-0.04	
613	D5185	6.1		0.20	
614	D5185	5.67		-0.13	
902	D5185	6.5	C	0.51	First reported 1.6
912		----		----	
963	D5185	5.0		-0.65	
1017		----		----	
1023	D5185	4.98		-0.66	
1059	in house	5		-0.65	
1146	D5185	2.856	G(0.05)	-2.30	
1243		----		----	
1271	in house	2.09	G(0.05)	-2.90	
1295		----		----	
1327	D5185	5.51		-0.25	
1328	D5185	5.67		-0.13	
1417	in house	5.51		-0.25	
1435	D5185	6.00		0.12	
1456	D5185	5		-0.65	
1460	D5185	1.95714	G(0.05)	-3.00	
1480		----		----	
1540		----		----	
1628		----		----	
1630		----		----	
1660	D5185	6.16		0.25	
1722		----		----	
1900	D6595	8		1.67	
1920		----		----	
7003	D3919	5.253		-0.45	
normality	OK				
n	21				
outliers	5				
mean (n)	5.84				
st.dev. (n)	0.734				
R(calc.)	2.06				
R(D5185:09)	3.62				

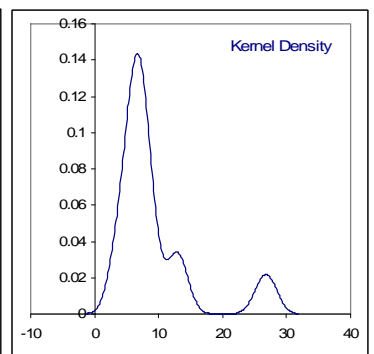
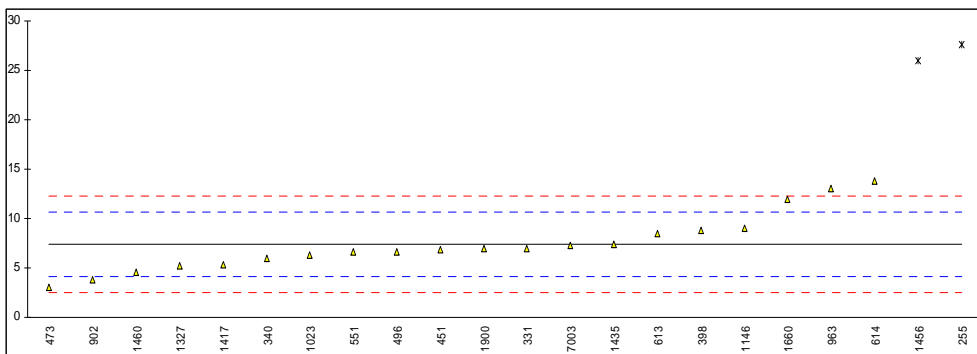
Application range ASTM D5185:09: 5 – 40 mg/kg



Determination of Sodium (Na) on sample #11103; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255	in house	27.5728	G(0.05)	12.38	
311		-----		-----	
331	D5185	7		-0.25	
340	D5185	6		-0.87	
360	D5185	<7.0		<-0.25	
398	D6595	8.8		0.85	
432		-----		-----	
451	D5185	6.88		-0.32	
473	D5185	3.0012		-2.71	
496	D5185	6.63		-0.48	
551	D5185	6.6255		-0.48	
613	D5185	8.5		0.67	
614	D5185	13.8		3.92	
902	D5185	3.8		-2.22	
912		-----		-----	
963	D5185	13		3.43	
1017		-----		-----	
1023	D5185	6.33		-0.66	
1059		-----		-----	
1146	D5185	9.063		1.02	
1243		-----		-----	
1271		-----		-----	
1295		-----		-----	
1327	D5185	5.20		-1.36	
1328		-----		-----	
1417	in house	5.31		-1.29	
1435	D5185	7.44		0.02	
1456	D5185	26	G(0.01)	11.42	
1460	D5185	4.54740		-1.76	
1480		-----		-----	
1540		-----		-----	
1628		-----		-----	
1630		-----		-----	
1660	D5185	12		2.82	
1722		-----		-----	
1900	D6595	7		-0.25	
1920		-----		-----	
7003	D4691	7.253		-0.10	
normality		not OK			
n		20			
outliers		2			
mean (n)		7.41			
st.dev. (n)		2.847			
R(calc.)		7.97			
R(D5185:09)		4.56			

Application range ASTM D5185:09: 7 – 70 mg/kg



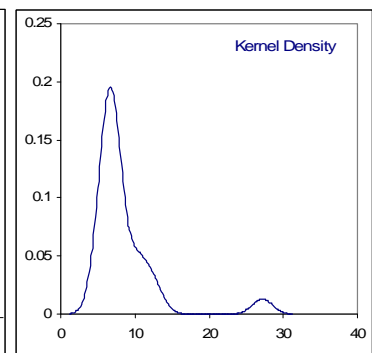
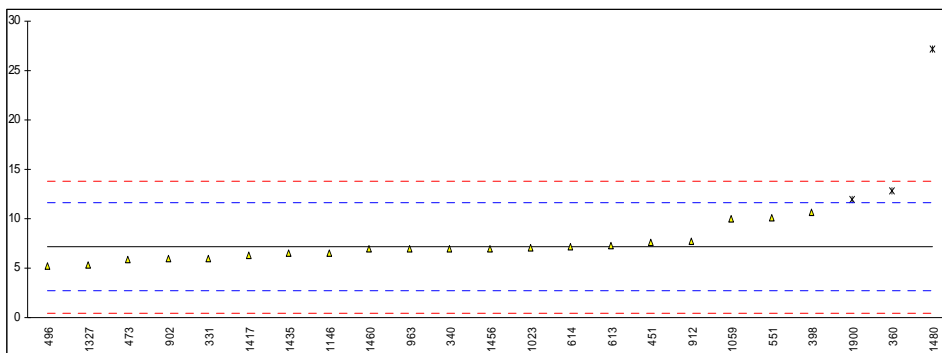


Determination of Silicon (Si) on sample #11103; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255		----		----	
311		----		----	
331	D5185	6		-0.52	
340	D5185	7		-0.07	
360	D5185	12.8	DG(0.05)	2.53	
398	D6595	10.6		1.54	
432		----		----	
451	D5185	7.58		0.19	
473	D5185	5.8525		-0.59	
496	D5185	5.25		-0.86	
551	D5185	10.151		1.34	
613	D5185	7.3		0.06	
614	D5185	7.15		0.00	
902	D5185	6.0		-0.52	
912	D5185	7.70		0.24	
963	D5185	7		-0.07	
1017		----		----	
1023	D5185	7.03		-0.06	
1059	in house	10		1.27	
1146	D5185	6.537		-0.28	
1243		----		----	
1271		----		----	
1295		----		----	
1327	D5185	5.37		-0.80	
1328		----		----	
1417	in house	6.28		-0.39	
1435	D5185	6.47		-0.31	
1456	D5185	7		-0.07	
1460	D5185	6.92528		-0.11	
1480	D5185	27.2	G(0.01)	8.98	
1540		----		----	
1628		----		----	
1630		----		----	
1660	D5185	<1		<-2.76	False negative?
1722		----		----	
1900	D6595	12	DG(0.05)	2.17	
1920		----		----	
7003		----		----	

normality not OK  
n 20  
outliers 3  
mean (n) 7.16  
st.dev. (n) 1.493  
R(calc.) 4.18  
R(D5185:09) (6.25)

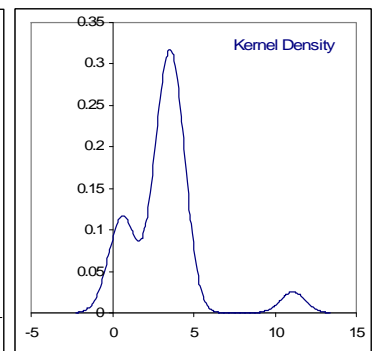
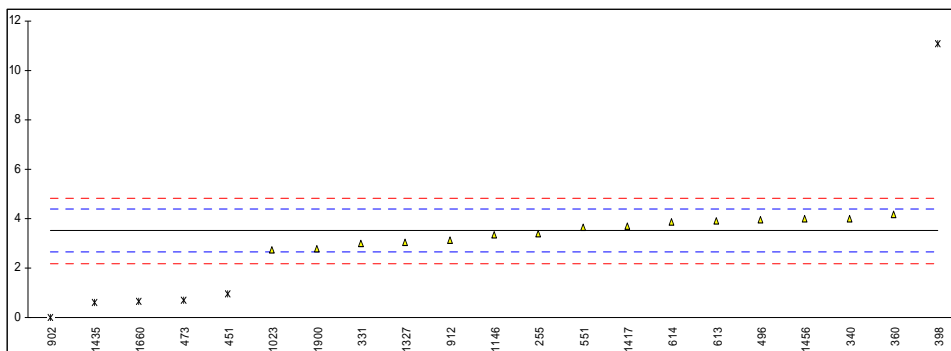
Application range ASTM D5185:09: 8 – 50 mg/kg



Determination of Silver (Ag) on sample #11103; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255	in house	3.4005		-0.26	
311		----		----	
331	D5185	3		-1.17	
340	D5185	4		1.11	
360	D5185	4.17		1.50	
398	D6595	11.1	G(0.01)	17.28	
432		----		----	
451	D5185	0.97	G(0.01)	-5.79	
473	D5185	0.6829	G(0.01)	-6.44	
496	D5185	3.97		1.04	
551	D5185	3.631		0.27	
613	D5185	3.9		0.88	
614	D5185	3.85		0.77	
902	D5185	0.0	ex	-8.00	Result excluded, zero not a real result
912	D5185	3.13		-0.87	
963		----		----	
1017		----		----	
1023	D5185	2.76		-1.71	
1059		----		----	
1146	D5185	3.331		-0.41	
1243		----		----	
1271		----		----	
1295		----		----	
1327	D5185	3.05		-1.05	
1328		----		----	
1417	in house	3.70		0.43	
1435	D5185	0.60	G(0.01)	-6.63	
1456	D5185	4		1.11	
1460		----		----	
1480		----		----	
1540		----		----	
1628		----		----	
1630		----		----	
1660	D5185	0.65	G(0.01)	-6.52	
1722		----		----	
1900	D6595	2.8		-1.62	
1920		----		----	
7003		----		----	
normality	OK				
n	15				
outliers	5				
mean (n)	3.51				
st.dev. (n)	0.476				
R(calc.)	1.33				
R(D5185:09)	1.23				

Application range ASTM D5185:09: 0.5 – 50 mg/kg

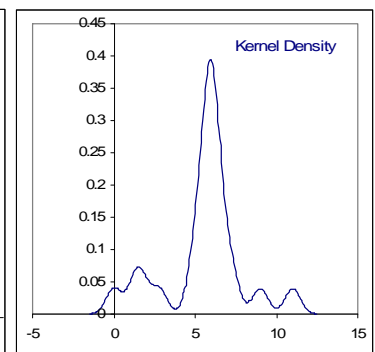
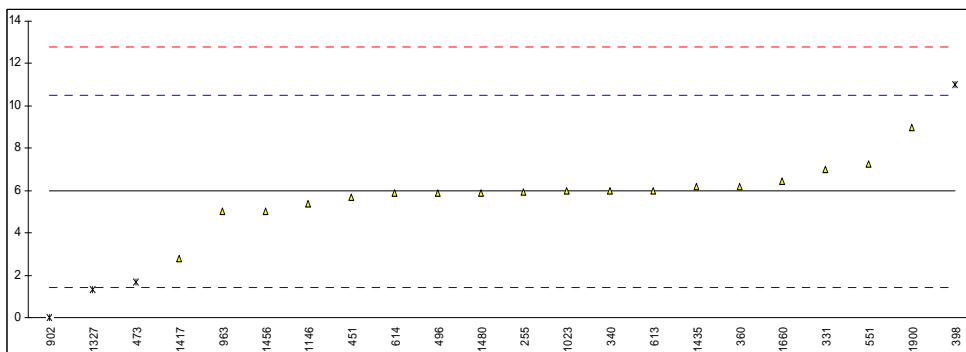


Determination of Tin (Sn) on sample #11103; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255	in house	5.939		-0.02	
311		----		----	
331	D5185	7		0.45	
340	D5185	6		0.01	
360	D5185	6.21		0.10	
398	D6595	11.0	G(0.05)	2.21	
432		----		----	
451	D5185	5.70		-0.12	
473	D5185	1.6989	G(0.01)	-1.88	
496	D5185	5.88		-0.04	
551	D5185	7.2515		0.56	
613	D5185	6.0		0.01	
614	D5185	5.86		-0.05	
902	D5185	0.0	ex	-2.63	Result excluded, zero is not a real result
912		----		----	
963	D5185	5		-0.43	
1017		----		----	
1023	D5185	5.99		0.01	
1059	in house	<6		<0.01	
1146	D5185	5.399		-0.25	
1243		----		----	
1271		----		----	
1295		----		----	
1327	D5185	1.30	G(0.01)	-2.06	
1328		----		----	
1417	in house	2.78		-1.41	
1435	D5185	6.17		0.09	
1456	D5185	5		-0.43	
1460		----		----	
1480	D5185	5.9		-0.03	
1540		----		----	
1628		----		----	
1630		----		----	
1660	D5185	6.46		0.21	
1722		----		----	
1900	D6595	9		1.33	
1920		----		----	
7003		----		----	

normality not OK  
n 18  
outliers 3  
mean (n) 5.97  
st.dev. (n) 1.206  
R(calc.) 3.38  
R(D5185:09) (6.36)

Application range ASTM D5185:09: 10 – 40 mg/kg

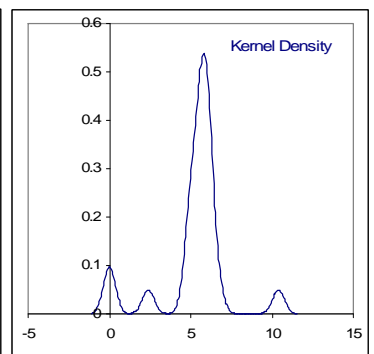
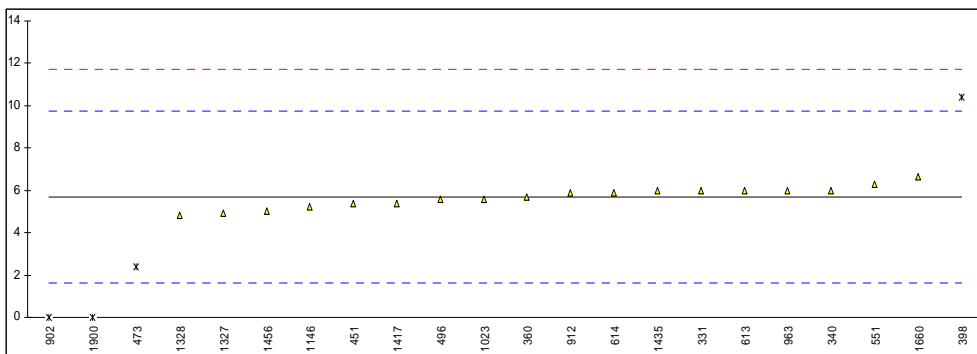


Determination of Titanium (Ti) on sample #11103; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255		----		----	
311		----		----	
331	D5185	6		0.16	
340	D5185	6		0.16	
360	D5185	5.70		0.01	
398	D6595	10.4	G(0.01)	2.34	
432		----		----	
451	D5185	5.39		-0.14	
473	D5185	2.3935	G(0.01)	-1.63	
496	D5185	5.56		-0.06	
551	D5185	6.2695		0.29	
613	D5185	6.0		0.16	
614	D5185	5.9		0.11	
902	D5185	0.0	ex	-2.81	Result excluded, zero not a real result
912	D5185	5.87		0.09	
963	D5185	6		0.16	
1017		----		----	
1023	D5185	5.60		-0.04	
1059		----		----	
1146	D5185	5.212		-0.23	
1243		----		----	
1271		----		----	
1295		----		----	
1327	D5185	4.90		-0.39	
1328	D5185	4.83		-0.42	
1417	in house	5.39		-0.14	
1435	D5185	5.98		0.15	
1456	D5185	5		-0.34	
1460		----		----	
1480		----		----	
1540		----		----	
1628		----		----	
1630		----		----	
1660	D5185	6.65		0.48	
1722		----		----	
1900	D6595	0	ex	-2.81	Result excluded zero not a real result
1920		----		----	
7003		----		----	

normality OK  
n 18  
outliers 2  
mean (n) 5.68  
st.dev. (n) 0.490  
R(calc.) 1.37  
R(D5185:09) 5.66

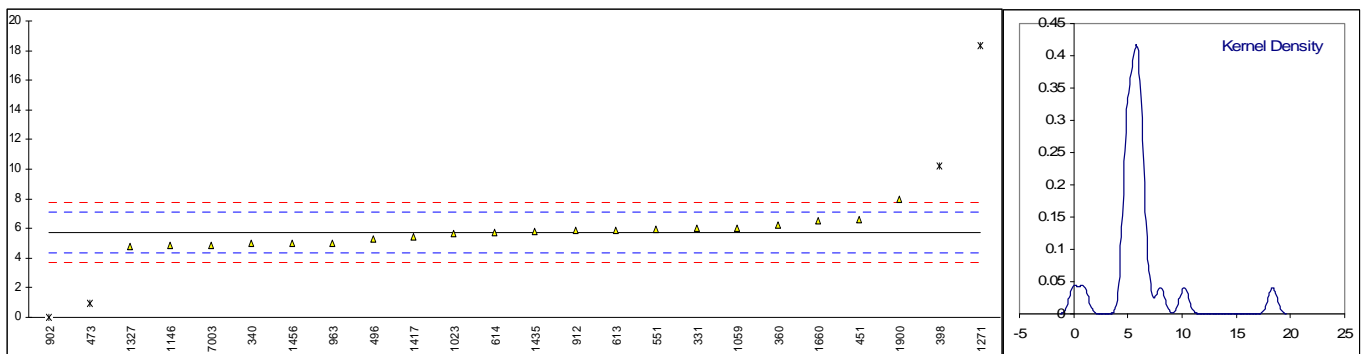
Application range ASTM D5185:09: 5 – 40 mg/kg



Determination of Vanadium (V) on sample #11103; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255		----		----	
311		----		----	
331	D5185	6		0.42	
340	D5185	5		-1.05	
360	D5185	6.20		0.71	
398	D6595	10.2	G(0.05)	6.59	
432		----		----	
451	D5185	6.57		1.26	
473	D5185	0.9485	G(0.05)	-7.01	
496	D5185	5.29		-0.63	
551	D5185	5.9345		0.32	
613	D5185	5.9		0.27	
614	D5185	5.7		-0.02	
902	D5185	0.0	ex	-8.40	Result excluded, zero not a real result
912	D5185	5.88		0.24	
963	D5185	5		-1.05	
1017		----		----	
1023	D5185	5.62		-0.14	
1059	in house	6		0.42	
1146	D5185	4.822		-1.31	
1243		----		----	
1271	in house	18.35	C,G(0.01)	18.57	First reported 23.25
1295		----		----	
1327	D5185	4.80		-1.35	
1328		----		----	
1417	in house	5.42		-0.43	
1435	D5185	5.79		0.11	
1456	D5185	5		-1.05	
1460		----		----	
1480		----		----	
1540		----		----	
1628		----		----	
1630		----		----	
1660	D5185	6.54		1.21	
1722		----		----	
1900	D6595	8		3.36	
1920		----		----	
7003	D3919	4.853		-1.27	
normality		OK			
n		20			
outliers		3			
mean (n)		5.72			
st.dev. (n)		0.772			
R(calc.)		2.16			
R(D5185:09)		1.91			

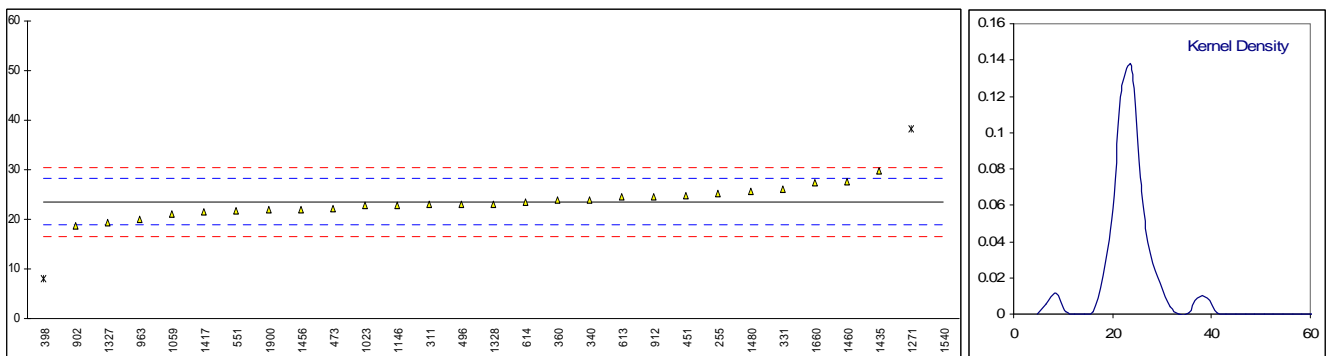
Application range ASTM D5185:09: 1 – 50 mg/kg



Determination of Calcium (Ca) on sample #11103; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255	in house	25.184		0.73	
311	D5185	23		-0.21	
331	D5185	26		1.08	
340	D5185	24		0.22	
360	D5185	24.0		0.22	
398	D6595	8.1	G(0.05)	-6.58	
432		-----		-----	
451	D5185	24.8		0.56	
473	D5185	22.1644		-0.57	
496	D5185	23.14		-0.15	
551	D5185	21.707		-0.76	
613	D5185	24.6		0.48	
614	D5185	23.55		0.03	
902	D5185	18.8		-2.01	
912	D5185	24.6		0.48	
963	D5185	20		-1.49	
1017		-----		-----	
1023	D5185	22.8		-0.29	
1059	in house	21		-1.06	
1146	D5185	22.90		-0.25	
1243		-----		-----	
1271	in house	38.21	CG(0.05)	6.30	First reported 14
1295		-----		-----	
1327	D5185	19.33		-1.78	
1328	D5185	23.15		-0.14	
1417	in house	21.51		-0.85	
1435	D5185	29.84		2.72	
1456	D5185	22		-0.64	
1460	D5185	27.61793		1.77	
1480	D5185	25.6		0.90	
1540	D6481	490	G(0.01)	199.64	
1628		-----		-----	
1630		-----		-----	
1660	D5185	27.37		1.66	
1722		-----		-----	
1900	D6595	22		-0.64	
1920		-----		-----	
7003		-----		-----	
normality	OK				
n	26				
outliers	3				
mean (n)	23.49				
st.dev. (n)	2.554				
R(calc.)	7.15				
R(Horwitz)	6.54				

Application range ASTM D5185:09: 40 – 9000 mg/kg

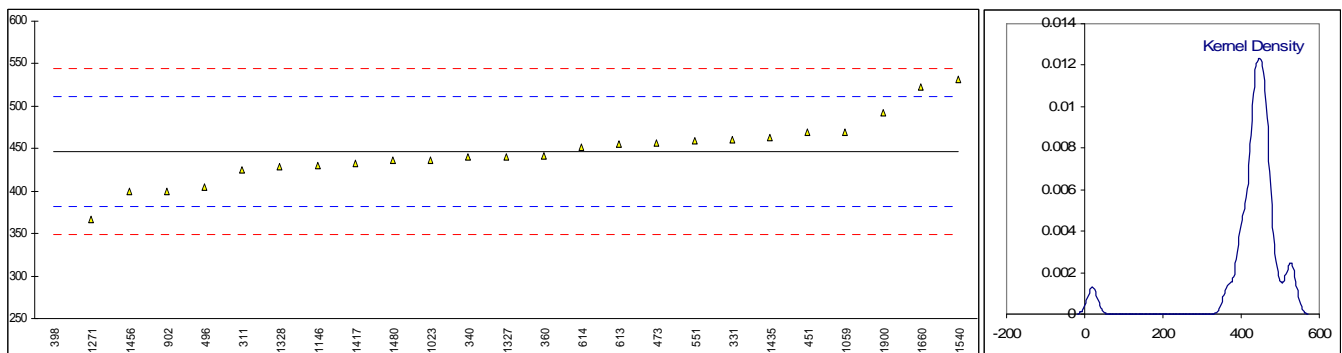


Determination of Phosphorus (P) on sample #11103; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255		----		----	
311	D5185	425		-0.66	
331	D5185	460		0.42	
340	D5185	440		-0.20	
360	D5185	441		-0.17	
398	D6595	19.6	G(0.01)	-13.15	
432		----		----	
451	D5185	469.6		0.71	
473	D5185	456.429		0.31	
496	D5185	404.1		-1.30	
551	D5185	459.8555		0.41	
613	D5185	456		0.30	
614	D5185	451		0.14	
902	D5185	400		-1.43	
912		----		----	
963		----		----	
1017		----		----	
1023	D5185	437		-0.29	
1059	in house	470		0.73	
1146	D5185	429.6		-0.52	
1243		----		----	
1271	D6481	367	C	-2.45	First reported 0.0367
1295		----		----	
1327	D5185	440.3		-0.19	
1328	D5185	429		-0.54	
1417	in house	432.7		-0.42	
1435	D5185	462.5		0.50	
1456	D5185	400		-1.43	
1460		----		----	
1480	D5185	436.8		-0.30	
1540	D6481	532		2.64	
1628		----		----	
1630		----		----	
1660	D5185	522.11		2.33	
1722		----		----	
1900	D6595	492		1.40	
1920		----		----	
7003		----		----	

normality OK  
n 24  
outliers 1  
mean (n) 446.42  
st.dev. (n) 36.707  
R(calc.) 102.78  
R(D5185:09) 90.85

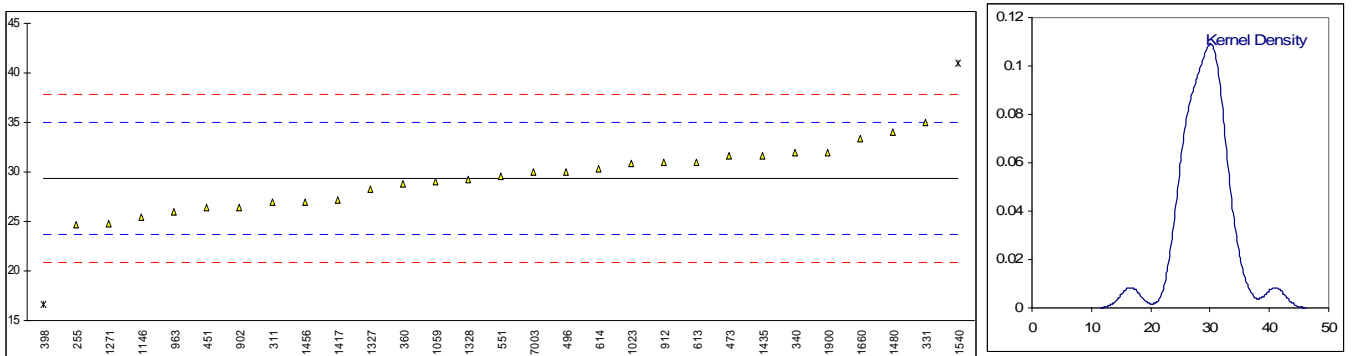
Application range ASTM D5185:09: 10 – 1000 mg/kg



Determination of Zinc (Zn) on sample #11103; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
255	in house	24.664		-1.66	
311	D5185	27		-0.83	
331	D5185	35		2.00	
340	D5185	32		0.94	
360	D5185	28.8		-0.20	
398	D6595	16.6	G(0.05)	-4.52	
432		-----		-----	
451	D5185	26.4		-1.05	
473	D5185	31.6347		0.81	
496	D5185	30.05		0.24	
551	D5185	29.568		0.07	
613	D5185	31.0		0.58	
614	D5185	30.3		0.33	
902	D5185	26.4		-1.05	
912	D5185	31.0		0.58	
963	D5185	26		-1.19	
1017		-----		-----	
1023	D5185	30.9		0.55	
1059	in house	29		-0.13	
1146	D5185	25.42		-1.39	
1243		-----		-----	
1271	in house	24.83	C	-1.60	First reported 15.05
1295		-----		-----	
1327	D5185	28.25	C	-0.39	First reported 21.10
1328	D5185	29.22		-0.05	
1417	in house	27.18		-0.77	
1435	D5185	31.66		0.82	
1456	D5185	27		-0.83	
1460		-----		-----	
1480	D5185	34.0		1.64	
1540	D6481	41	G(0.01)	4.12	
1628		-----		-----	
1630		-----		-----	
1660	D5185	33.40		1.43	
1722		-----		-----	
1900	D6595	32		0.94	
1920		-----		-----	
7003	D4691	29.99		0.22	
	normality	OK			
	n	27			
	outliers	2			
	mean (n)	29.36			
	st.dev. (n)	2.829			
	R(calc.)	7.92			
	R(Horwitz)	7.91			

Application range ASTM D5185: 60-1600 mg/kg





**APPENDIX 2****Number of participants per country**

3 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 ESTONIA  
2 laboratories in FRANCE  
2 laboratories in GERMANY  
2 laboratories in INDIA  
1 laboratory in IRAN  
2 laboratories in ITALY  
3 laboratories in NORWAY  
2 laboratories in P.R. of CHINA  
1 laboratory in ROMANIA  
2 laboratories in SAUDI ARABIA  
1 laboratory in SERBIA  
1 laboratory in SLOVENIA  
1 laboratory in SPAIN  
1 laboratory in SWEDEN  
1 laboratory in TANZANIA  
1 laboratory in THAILAND  
2 laboratories in THE NETHERLANDS  
1 laboratory in TURKEY  
2 laboratories in UNITED KINGDOM

**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/>).