Results of Proficiency Test
Hydraulic Fluid (used)
November 2012

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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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 2012/2013. In this interlaboratory study, 43 laboratories from 27 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 V968 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% 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 #12141. The homogeneity of the subsamples #12141 was checked by determination of Density in accordance with ASTM D4052:02e1 and Water according to ASTM D6304:07 on 8 stratified randomly selected samples.

	Density @ 15 ^{o}C in kg/L	*Water in %M/M*
Sample #12141-1	0.87877	0.0150
Sample #12141-2	0.87877	0.0142
Sample #12141-3	0.87878	0.0122
Sample #12141-4	0.87878	0.0140
Sample #12141-5	0.87876	0.0150
Sample #12141-6	0.87877	0.0120
Sample #12141-7	0.87877	0.0140
Sample #12141-8	0.87877	0.0145

Table 1: homogeneity test results of subsamples #12141

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	*Water in %M/M*
r (Observed)	0.00002	0.0032
reference method	D4052:02e1	D6304:07
0.3 * R (ref. method)	0.00015	0.0098

Table 2: repeatabilities of subsamples #12141

The approximately 6 litre of the second bulk material, positive on wear metals, was homogenised and was transferred to 60 subsamples of 100 mL HDPE containers, labelled #12142. The homogeneity of the subsamples #12142 was checked by determination of Density in accordance with ASTM D4052:02e1 and Calcium in accordance with ASTM D5185:09 on 8 stratified randomly selected samples.

	Density @ 15 °C in kg/L	*Calcium in mg/kg*
Sample #12142-1	0.87414	197
Sample #12142-2	0.87412	197
Sample #12142-3	0.87412	198
Sample #12142-4	0.87414	197
Sample #12142-5	0.87414	197
Sample #12142-6	0.87413	194
Sample #12142-7	0.87414	197
Sample #12142-8	0.87412	197

Table 3: homogeneity test results of subsamples #12142

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 °C in kg/L	*Calcium in mg/kg*
r (Observed)	0.00003	3.3
reference method	D4052:02e1	D5185:09
0.3* R (ref. method)	0.00015	4.3

Table 4: repeatability of subsamples #12142

Each calculated repeatability was equal or less than 0.3 times the corresponding reproducibility of the respective test method. Therefore, homogeneity of the subsamples #12141 and #12142 was assumed.

To each of the participating laboratories was dispatched: One 1 litre amber glass bottle, labelled #12141 and one 100 mL HDPE container, labelled #12142 on October 24, 2012.

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 #12141 and 20 elements (17 wear metals and 3 additives) on sample #12142.

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:

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

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:

$\lvert z \rvert < 1$	good
$1 < \lvert z \rvert < 2$	satisfactory
$2 < \lvert z \rvert < 3$	questionable
$3 < \lvert z \rvert$	unsatisfactory

4 EVALUATION

In this proficiency test, no serious problems were encountered during dispatch and execution. Only two laboratories (Brazil and Serbia) received the samples late. Eleven laboratories reported the results after the final reporting date and three other laboratories did not report any results at all.
The 40 reporting participants sent in 754 numerical results. Observed were 46 outlying results, which is 6.1% 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 determination: Density, Silver and Phosphorus. 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” (#12142) contained a large number of elements, spectral interferences might explain the large spread found for some elements.
For the metals on sample #12142, 15 of the 19 test results reported by laboratory 473 appeared to be either false negative or statistical outlier. Therefore it was decided to exclude also the other reported results for statistical evaluation.

Acid Number (Total): This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in full agreement with the requirements of ASTM D664:11a.

Density @ 15°C: This determination was very problematic. Four statistical outliers were observed and the calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ASTM D4052:02E1.

Flash Point PMcc: This determination was problematic. Two results were excluded as the used test method is technically not equivalent. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D93:12 method B. Nineteen participants reported to have used procedure A of ASTM D93:12, which is not intended for used oil.

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 somewhat smaller, but again not at all in agreement with the requirements of the standard.

Kin.Visco.@ 100°C: 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 D445:12. When the ASTM D445 results are evaluated separately, the calculated reproducibility is not changed and thus again not at all in agreement with the requirements of the standard.

Water: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility 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 much smaller and again in agreement with the requirements of the test method.

Aluminium: This determination was not problematic. Only one statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D5185:09.

Barium: As the average concentration found by the group (254 mg/kg) is far 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. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outlier is in full agreement with the estimated requirements calculated using the Horwitz equation.

Chromium: 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 good agreement with the requirements of ASTM D5185:09.

Copper: This determination was not problematic. Only one statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is almost in 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. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ASTM D5185:09.

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. Three statistical outliers were observed. However, 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. 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.

Molybdenum This determination was not problematic. Only one statistical outlier was observed and the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D5185:09

Nickel: 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 D5185:09.

Sodium: This determination was problematic. Three statistical outliers were observed and the calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D5185:09.

Silicon: 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.

Silver: 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 D5185:09.

Tin: 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.

Titanium: 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.

Vanadium: 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.

Calcium: 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 D5185:09.

Phosphorus: 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 D5185:09.

Zinc: 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 D5185:09.

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	31	0.414	0.183	0.182
Density @ 15°C	kg/L	29	0.8789	0.0009	0.0005
Flash Point PMcc	^{o}C	31	208.5	11.2	10.0
Kinematic viscosity @ 40°C	mm/s^2	38	62.304	1.239	0.474
Kinematic viscosity @ 100°C	mm/s^2	35	9.720	0.196	0.074
Water	%M/M	31	0.009	0.014	0.025

Table 5: reproducibilities of results of sample #12141.

Parameter	*Unit*	*n*	*average*	*2.8 * sd*	*R (lit)*
Aluminium as Al	mg/kg	28	9.14	3.55	6.76
Barium as Ba	mg/kg	26	254.4	52.7	(49.5)
Chromium as Cr	mg/kg	27	9.51	2.37	3.20
Copper as Cu	mg/kg	29	31.64	8.40	7.59
Iron as Fe	mg/kg	28	14.63	4.42	4.45
Lead as Pb	mg/kg	27	10.69	2.57	7.04
Lithium as Li	mg/kg	9	6.20	1.87	2.11
Magnesium as Mg	mg/kg	26	15.90	4.67	6.06
Manganese as Mn	mg/kg	25	9.62	2.75	1.97
Molybdenum as Mo	mg/kg	27	9.47	2.58	3.16
Nickel as Ni	mg/kg	25	8.85	2.35	4.46
Sodium as Na	mg/kg	22	11.18	8.30	6.11
Silicon as Si	mg/kg	26	12.32	5.35	7.72
Silver as Ag	mg/kg	24	8.14	4.23	2.85
Tin as Sn	mg/kg	27	9.99	3.14	8.75
Titanium as Ti	mg/kg	22	9.28	2.28	7.12
Vanadium as V	mg/kg	27	9.63	2.84	3.38
Calcium as Ca	mg/kg	28	194.3	57.9	14.2
Phosphorus as P	mg/kg	26	861.0	207.2	126.2
Zinc as Zn	mg/kg	30	360.5	92.8	53.9

Table 6: reproducibilities of results of sample #12142
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 2012 WITH THE PREVIOUS PTS.

	November 2012	*November 2011*	*November 2010*	*November 2009*
Number of reporting labs	40	36	30	30
Number of results reported	754	667	434	520
Statistical outliers	46	65	36	35
Percentage outliers	6.1%	9.7%	8.3%	6.7%

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	*November 2012*	*November 2011*	*November 2010*	*November 2009*
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.

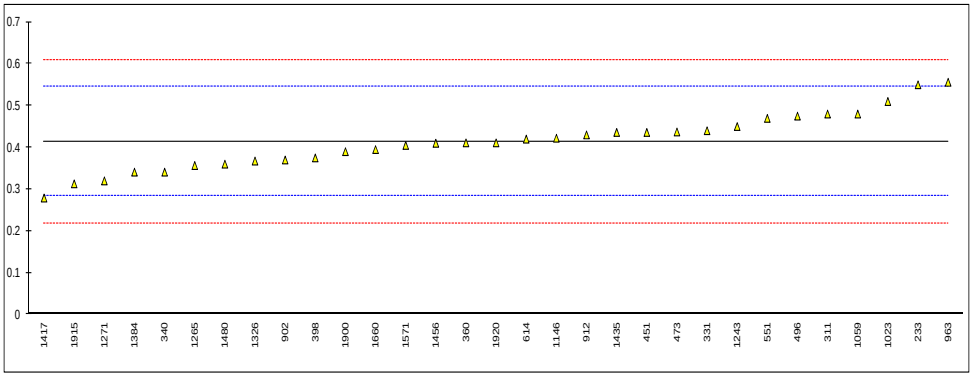
++: group performed much better than the standard
+ : group performed better than the standard
+/-: group performance equals the standard
- : group performed worse than the standard
-- : group performed much worse than the standard
n.e.: not evaluated

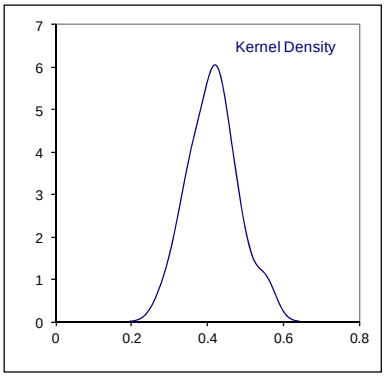
APPENDIX 1

Determination of Acid Number (Total) on sample #12141; results in mg KOH/g.

lab	method	value	mark	z(targ)	remarks
233	D664	0.55	C	2.09	First reported 0.85
255		-----		-----	
311	D664	0.48		1.01	
325		-----		-----	
331	D664	0.440		0.40	
340	D664	0.341		-1.12	
360	D664	0.411		-0.05	
398	D664	0.375		-0.60	
432		-----		-----	
451	D664	0.436		0.34	
473	D664	0.4371		0.35	
496	D664	0.475		0.94	
551	D664	0.4696		0.85	
562		-----		-----	
614	D664	0.42		0.09	
902	D664	0.370		-0.68	
912	D664	0.43		0.24	
963	D664	0.556		2.18	
1017		-----		-----	
1023	D664	0.51		1.47	
1059	ISO6619	0.48		1.01	
1146	D664	0.422		0.12	
1150		-----		-----	
1155		-----		-----	
1174		-----		-----	
1243	D664	0.45		0.55	
1244		-----		-----	
1265	D664	0.3568		-0.88	
1271	D664	0.32		-1.45	
1326	D664	0.3675		-0.72	
1384	ISO6618	0.341		-1.12	
1417	in house	0.279		-2.08	
1435	D664	0.436		0.34	
1456	D974	0.41		-0.06	
1480	D664	0.360	C	-0.83	First reported 0.120
1571	D664	0.405		-0.14	
1628		-----		-----	
1660	IEC62021-1	0.395		-0.29	
1842		-----		-----	
1900	D664	0.39		-0.37	
1915	D974	0.3129		-1.56	
1920	D664	0.411		-0.05	
2160		-----		-----	

normality	OK
n	31
outliers	0
mean (n)	0.4141
st.dev. (n)	0.06533
R(calc.)	0.1829
R(D664:11a)	0.1822

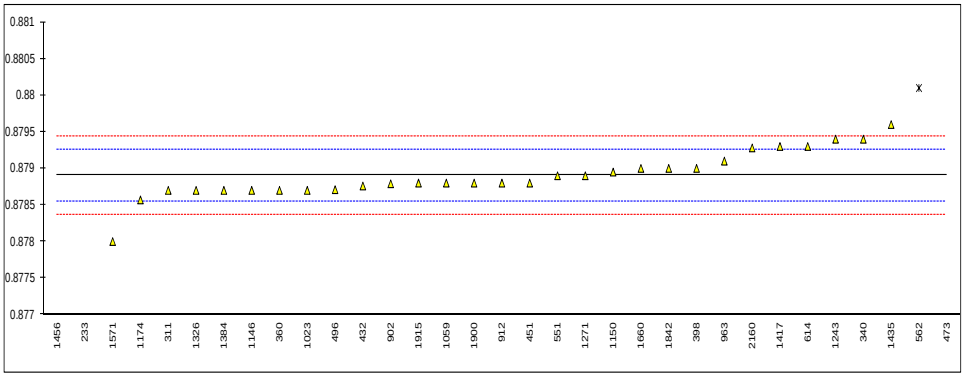


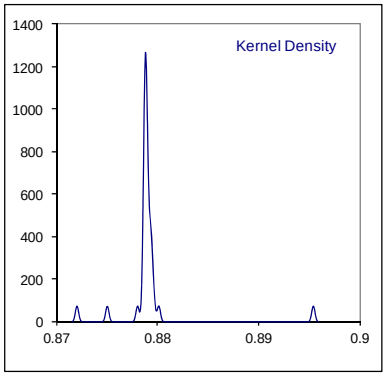


Determination of Density @ 15°C on sample #12141; results in kg/L.

lab	method	value	mark	z(targ)	remarks
233	D4052	0.8750	G(0.01)	-21.01	
255		-----		-----	
311	D4052	0.8787		-1.09	
325		-----		-----	
331		-----		-----	
340	D4052	0.8794		2.68	
360	D4052	0.8787		-1.09	
398	D4052	0.879		0.53	
432	D4052	0.87876		-0.77	
451	D4052	0.8788		-0.55	
473	D4052	0.8954	G(0.01)	88.83	
496	D4052	0.87871		-1.03	
551	D4052	0.8789		-0.01	
562	D4052	0.8801	G(0.05)	6.45	
614	D4052	0.8793		2.14	
902	D4052	0.87879		-0.60	
912	D4052	0.8788		-0.55	
963	D4052	0.8791		1.07	
1017		-----		-----	
1023	D4052	0.8787		-1.09	
1059	D4052	0.8788		-0.55	
1146	D4052	0.87870		-1.09	
1150	ISO12185	0.87895	C	0.26	Reported 878.95
1155		-----		-----	
1174	ISO3675	0.87857	C	-1.79	First reported 0.87785
1243	ISO12185	0.8794		2.68	
1244		-----		-----	
1265		-----		-----	
1271	D4052	0.8789		-0.01	
1326	D4052	0.8787		-1.09	
1384	ISO3675	0.8787		-1.09	
1417	IP365	0.8793		2.14	
1435	D4052	0.8796		3.76	
1456	D4052	0.8720	G(0.01)	-37.17	
1480		-----		-----	
1571	D7042	0.878		-4.86	
1628		-----		-----	
1660	D7042	0.8790		0.53	
1842	D4052	0.8790	C	0.53	Reported 879.0
1900	D4052	0.8788		-0.55	
1915	D4052	0.8788		-0.55	
1920		-----		-----	
2160	D4052	0.87928		2.03	

normality	not OK
n	29
outliers	4
mean (n)	0.87890
st.dev. (n)	0.000316
R(calc.)	0.00089
R(D4052:02E1)	0.00052



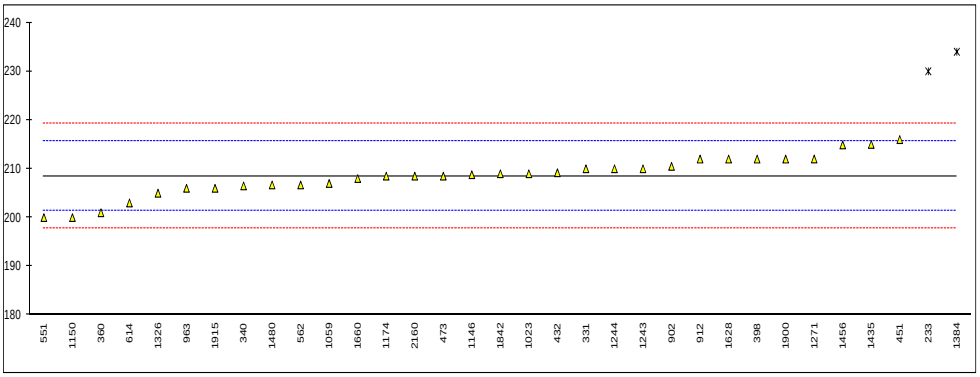


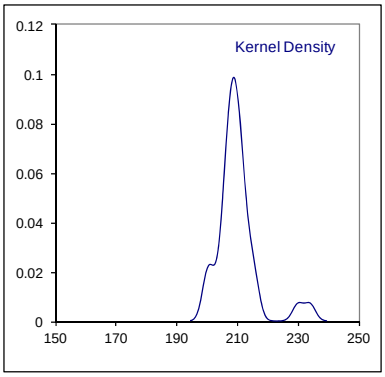
Determination of Flash Point PMcc on sample #12141; results in °C.

lab	method	value	mark	z(targ)	remarks
233	D92	230	ex	6.02	Result excluded, used test method is not equivalent to ASTM D93
255		-----		-----	
311		-----		-----	
325		-----		-----	
331	D93A	210.0		0.42	
340	D93A	206.5		-0.56	
360	D93A	201.0		-2.10	
398	D93A	212.0		0.98	
432	D93B	209.2		0.19	
451	D93A	216		2.10	
473	D93B	208.5		0.00	
496		-----		-----	
551	D93	200.0		-2.38	
562	D93B	206.7		-0.51	
614	D93A	203.0		-1.54	
902	D93A	210.5		0.56	
912	D93A	212		0.98	
963	D93A	206.0		-0.70	
1017		-----		-----	
1023	D93A	209		0.14	
1059	ISO2719A	207.0		-0.42	
1146	in house	208.80		0.08	
1150	ISO2719A	200.0		-2.38	
1155		-----		-----	
1174	ISO2719A	208.49		-0.01	
1243	ISO2719	210		0.42	
1244	D93A	210.0		0.42	
1265		-----		-----	
1271	ISO2719A	212.02		0.98	
1326	D93A	205.0		-0.98	
1384	ISO2592	234	ex	7.14	Result excluded, used test method is not equivalent to ASTM D93
1417		-----		-----	
1435	D93A	215.00		1.82	
1456	D93	214.9		1.79	
1480	D93	206.70		-0.51	
1571		-----		-----	
1628	ISO2719	212.0		0.98	
1660	D93A	208.0		-0.14	
1842	D93A	209		0.14	
1900	in house	212		0.98	
1915	D93	206		-0.70	
1920		-----		-----	
2160	D93A	208.50		0.00	
	normality	OK			
	n	31			
	outliers	0			
	mean (n)	208.51			
	st.dev. (n)	4.006			
	R(calc.)	11.22			
	R(D93:12-B)	10.00			Compare R(ASTM D93:12-A) = 14.80

A= used procedure A of the test method

B= used procedure B of the test method

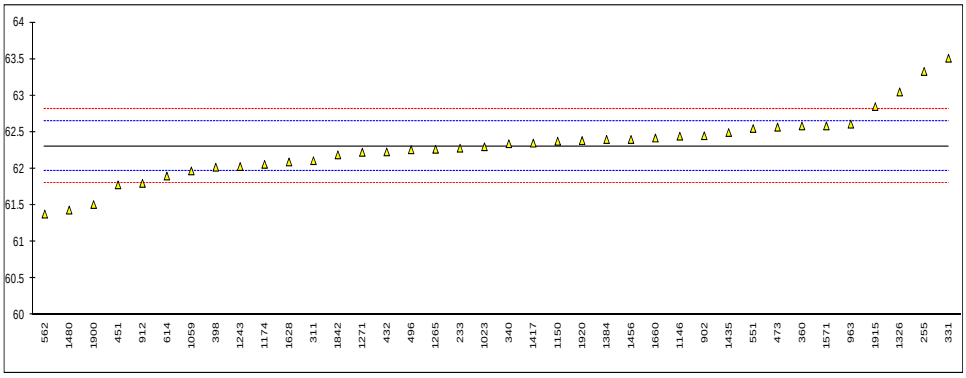


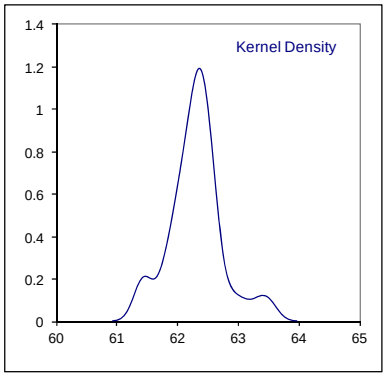


Determination of Kinematic Viscosity @ 40°C on sample #12141; results in mm/s^2.

lab	method	value	mark	z(targ)	remarks
233	D7279	62.28		-0.14	
255	D7279	63.33		6.07	
311	D445	62.11		-1.15	
325		-----		-----	
331	D7279	63.51		7.13	
340	D445	62.342		0.23	
360	D445	62.585		1.66	
398	D445	62.021		-1.67	
432	D445	62.230		-0.44	
451	D445	61.78		-3.10	
473	D445	62.5685		1.57	
496	D445	62.260		-0.26	
551	D7279	62.55		1.46	
562	D445	61.38		-5.46	
614	D445	61.9		-2.39	
902	D7279	62.451		0.87	
912	D445	61.80		-2.98	
963	D445	62.61		1.81	
1017		-----		-----	
1023	D445	62.30		-0.02	
1059	ISO3104	61.97		-1.97	
1146	D445	62.445		0.84	
1150	ISO3104	62.37597		0.43	
1155		-----		-----	
1174	ISO3104	62.05964		-1.44	
1243	D7279	62.03		-1.62	
1244		-----		-----	
1265	D445	62.26535		-0.23	
1271	D445	62.225		-0.47	
1326	D445	63.05		4.41	
1384	ISO3104	62.40		0.57	
1417	in house	62.35		0.27	
1435	D7042	62.495		1.13	
1456	D7042	62.4		0.57	
1480	D445	61.4346		-5.14	
1571	D445	62.586		1.67	
1628	ISO3104	62.092		-1.25	
1660	D7042	62.42		0.69	
1842	IP71	62.19		-0.67	
1900	D445	61.51		-4.69	
1915	D7042	62.85		3.23	
1920	D445	62.385		0.48	
2160		-----		-----	

		Only ASTM D445 / ISO3104 data:	
normality	OK	OK	
n	38	28	
outliers	0	0	
mean (n)	62.304	62.187	
st.dev. (n)	0.4423	0.3767	
R(calc.)	1.239	1.055	
R(D445:12)	0.474	0.473	Compare R(D7279) = 1.869

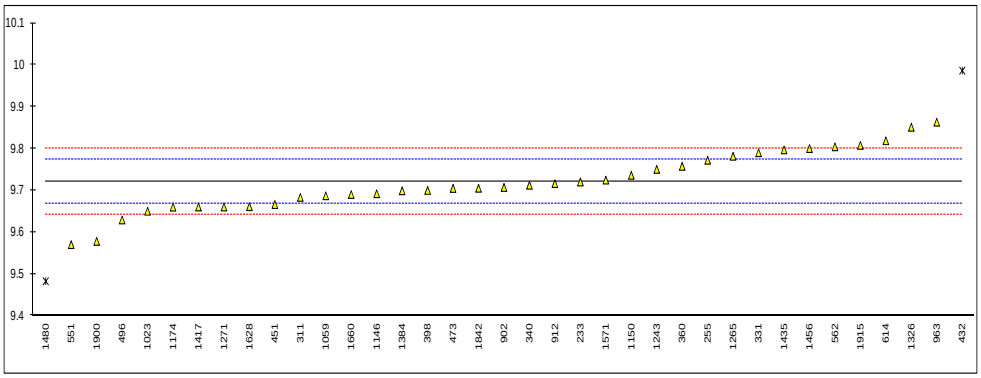


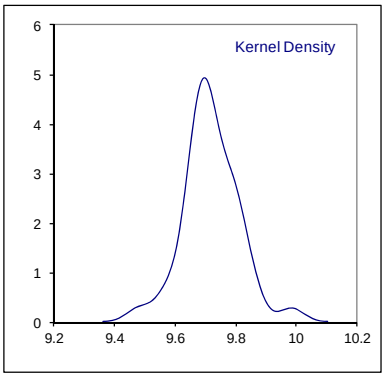


Determination of Kinematic Viscosity @ 100°C on sample #12141; results in mm/s^2

lab	method	value	mark	z(targ)	remarks
233	D7279	9.72		0.00	
255	D7279	9.772		1.97	
311	D445	9.683		-1.40	
325		-----		-----	
331	D7279	9.79		2.65	
340	D445	9.7122		-0.30	
360	D445	9.7575		1.42	
398	D445	9.6998		-0.77	
432	D445	9.9865	G(0.05)	10.10	
451	D445	9.666		-2.05	
473	D445	9.7047		-0.58	
496	D445	9.6290		-3.45	
551	D7279	9.570		-5.69	
562	D445	9.804		3.18	
614	D445	9.8185	C	3.73	First reported 9.583
902	D7279	9.707		-0.49	
912	D445	9.716		-0.15	
963	D445	9.863		5.42	
1017		-----		-----	
1023	D445	9.65		-2.66	
1059	ISO3104	9.687		-1.25	
1146	D445	9.6920		-1.06	
1150	ISO3104	9.73591		0.60	
1155		-----		-----	
1174	ISO3104	9.65961		-2.29	
1243	D7279	9.75		1.14	
1244		-----		-----	
1265	D445	9.78157		2.33	
1271	D445	9.66		-2.28	
1326	D445	9.851		4.96	
1384	ISO3104	9.699		-0.80	
1417	in house	9.66		-2.28	
1435	D7042	9.7969		2.91	
1456	D7042	9.8		3.03	
1480	D445	9.48234	G(0.05)	-9.01	
1571	D7042	9.72455		0.17	
1628	ISO3104	9.6610		-2.24	
1660	D7042	9.69		-1.14	
1842	IP71	9.705		-0.57	
1900	D445	9.5779		-5.39	
1915	D7042	9.8075		3.31	
1920		-----		-----	
2160		-----		-----	
					Only ASTM D445 / ISO3104 data:
	normality	OK			not OK
	n	35			24
	outliers	2			2
	mean (n)	9.7200			9.7114
	st.dev. (n)	0.07011			0.07018
	R(calc.)	0.1963			0.1965
	R(D445:12)	0.0739			0.0738

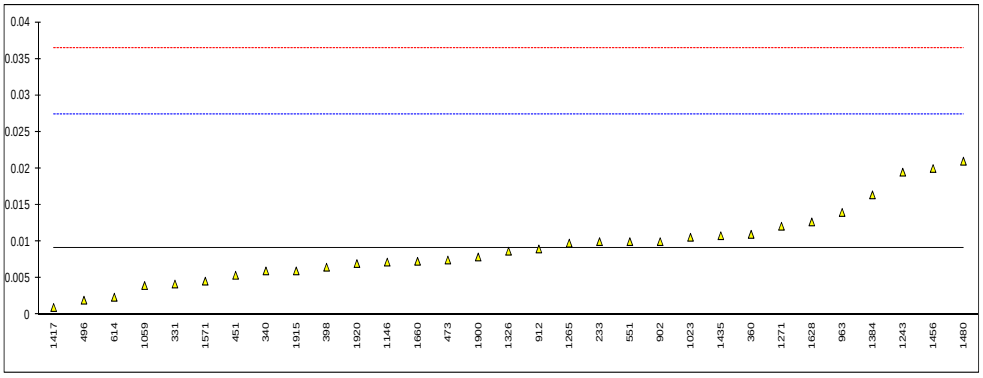
Compare R(D7279) = 0.5443

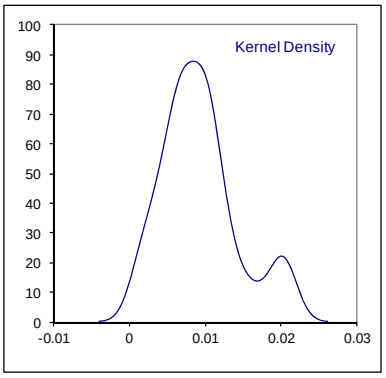




Determination of Water on sample #12141; results in %M/M.

lab	method	value	mark	z(targ)	remarks
233	D7358	0.01		0.09	
255		-----		-----	
311		-----		-----	
325		-----		-----	
331	D6304A	0.0042	C	-0.55	First reported 41.7
340	D6304A	0.0060		-0.35	
360	D6304A	0.011		0.20	
398	D6304C	0.0065		-0.30	
432		-----		-----	
451	D6304C	0.0054		-0.42	
473	D6304C	0.00749	C	-0.19	Reported 74.9
496	D6304C	0.0020		-0.79	
551	D6304A	0.010		0.09	
562		-----		-----	
614	D6304C	0.0024	C	-0.75	First reported 23.7
902	D6304A	0.01		0.09	
912	D6304C	0.009		-0.02	
963	D6304A	0.014		0.53	
1017		-----		-----	
1023	D6304A	0.0106		0.16	
1059	D6304Mod.A	0.0040		-0.57	
1146	D6304C	0.0072		-0.22	
1150		-----		-----	
1155		-----		-----	
1174		-----		-----	
1243	DIN51777	0.0195		1.14	
1244		-----		-----	
1265	D6304A	0.0098		0.07	
1271	ISO12937	0.0121		0.32	
1326	INH-207	0.00869		-0.05	
1384	ISO760	0.0164		0.79	
1417	in house	0.001		-0.90	
1435	D1744	0.01081	C	0.18	Reported 108.1
1456	D1744	0.02		1.19	
1480	D6304A	0.0210		1.30	
1571	D6304C	0.0046		-0.50	
1628	in house	0.0127		0.39	
1660	IEC60814	0.007312	C	-0.21	Reported 73.12
1842		-----		-----	
1900	D6304C	0.0079		-0.14	
1915	D1533A	0.0060		-0.35	
1920	D6304C	0.00700		-0.24	
2160		-----		-----	
					Only ASTM D6304-C
	normality	OK			OK
	n	31			11
	outliers	0			0
	mean (n)	0.00918			0.00577
	st.dev. (n)	0.005052			0.002285
	R(calc.)	0.01415			0.00640
	R(D6304:07)	0.02543			0.01925



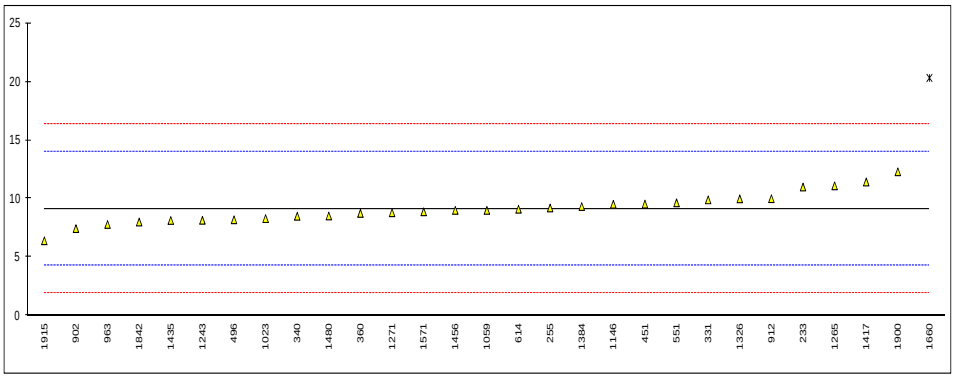


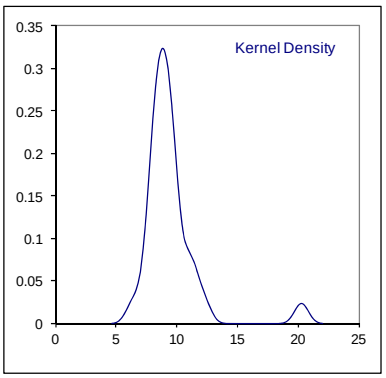
Determination of Aluminum (Al) on sample #12142; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	11		0.77	
255	INH-1021	9.2045		0.03	
311		-----		-----	
325		-----		-----	
331	D5185	9.9		0.31	
340	D5185	8.5		-0.27	
360	D5185	8.75		-0.16	
398		-----		-----	
432		-----		-----	
451	D5185	9.54		0.17	
473	D5185	<0.1		<-3.74	False negative?
496	D5185	8.2		-0.39	
551	D5185	9.65		0.21	
562		-----		-----	
614	D5185	9.1		-0.02	
902	D5185	7.44		-0.71	
912	D5185	10		0.36	
963	D5185	7.8		-0.56	
1017		-----		-----	
1023	D5185	8.3		-0.35	
1059	in house	9		-0.06	
1146	D5185	9.52		0.16	
1150		-----		-----	
1155		-----		-----	
1174		-----		-----	
1243	DIN51396	8.15		-0.41	
1244		-----		-----	
1265	D6595	11.1		0.81	
1271	D5185	8.8		-0.14	
1326	D5185	9.99		0.35	
1384	D5185	9.3307		0.08	
1417	in house	11.43		0.95	
1435	D5185	8.137		-0.42	
1456	D5185	9		-0.06	
1480	D5185	8.52		-0.26	
1571	D5185	8.8822		-0.11	
1628		-----		-----	
1660	D5185	20.32	G(0.01)	4.63	
1842	INH-01	8		-0.47	
1900	D6595	12.30		1.31	
1915	D5185	6.4003		-1.14	
1920		-----		-----	
2160		-----		-----	

normality	OK
n	28
outliers	1
mean (n)	9.14
st.dev. (n)	1.268
R(calc.)	3.55
R(D5185:09)	6.76

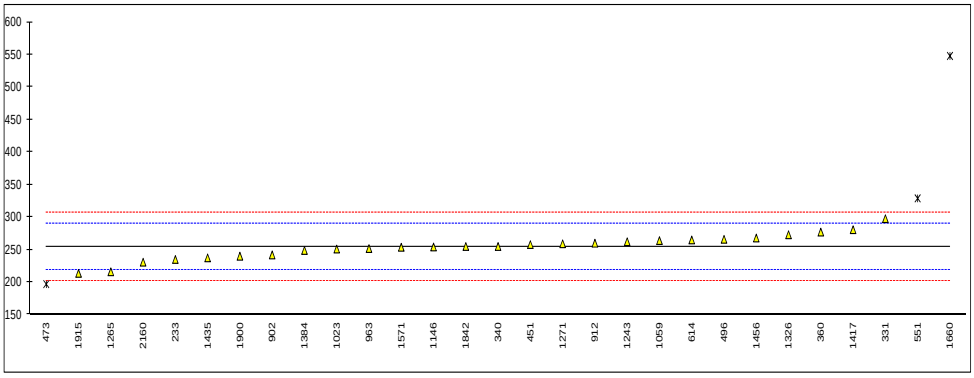
Application range ASTM D5185: 6 – 40 mg/kg

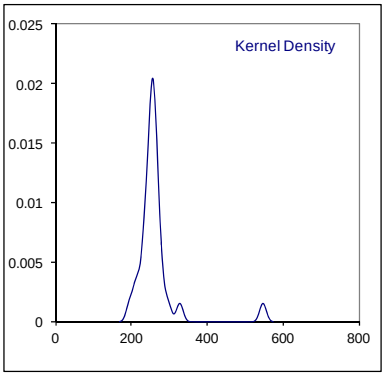




Determination of Barium (Ba) on sample #12142; results in mg/kg.

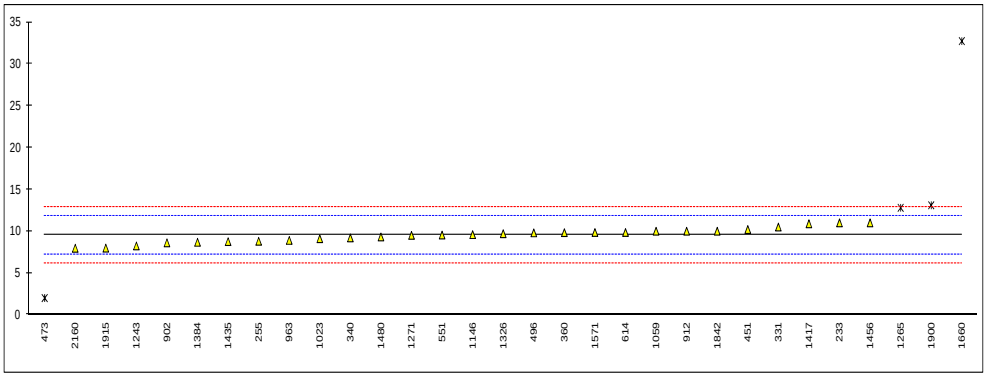
lab	method	value	mark	z(targ)	remarks
233	D6595	235		-1.10	
255		-----		-----	
311		-----		-----	
325		-----		-----	
331	D5185	297.7		2.45	
340	D5185	255.0		0.03	
360	D5185	277		1.28	
398		-----		-----	
432		-----		-----	
451	D5185	257.8		0.19	
473	D5185	197	G(0.05)	-3.25	
496	D5185	266.0		0.65	
551	D5185	328.92	G(0.05)	4.21	
562		-----		-----	
614	D5185	265	C	0.60	First reported 99.2
902	D5185	242		-0.70	
912	D5185	260		0.32	
963	D5185	251.8		-0.15	
1017		-----		-----	
1023	D5185	251		-0.19	
1059	in house	264		0.54	
1146	D5185	254.1		-0.02	
1150		-----		-----	
1155		-----		-----	
1174		-----		-----	
1243	DIN31391	262.3		0.45	
1244		-----		-----	
1265	D6595	216		-2.17	
1271	D5185	259.1		0.26	
1326	D5185	273		1.05	
1384	D5185	248.8667		-0.31	
1417	in house	280.7		1.49	
1435	D5185	237.4		-0.96	
1456	D5185	268		0.77	
1480		-----		-----	
1571	D5185	253.76		-0.04	
1628		-----		-----	
1660	D5185	548	G(0.01)	16.60	
1842	INH-01	255		0.03	
1900	D6595	240	C	-0.82	First reported 393.00
1915	D5185	213.549		-2.31	
1920		-----		-----	
2160	in house	230.8		-1.34	
	normality	OK			
	n	26			
	outliers	3			
	mean (n)	254.42			
	st.dev. (n)	18.817			
	R(calc.)	52.69			
	R(Horwitz)	49.52			Application range ASTM D5185: 0.5 – 4 mg/kg

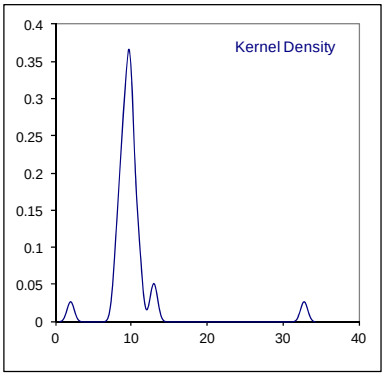




Determination of Chromium (Cr) on sample #12142; results in mg/kg.

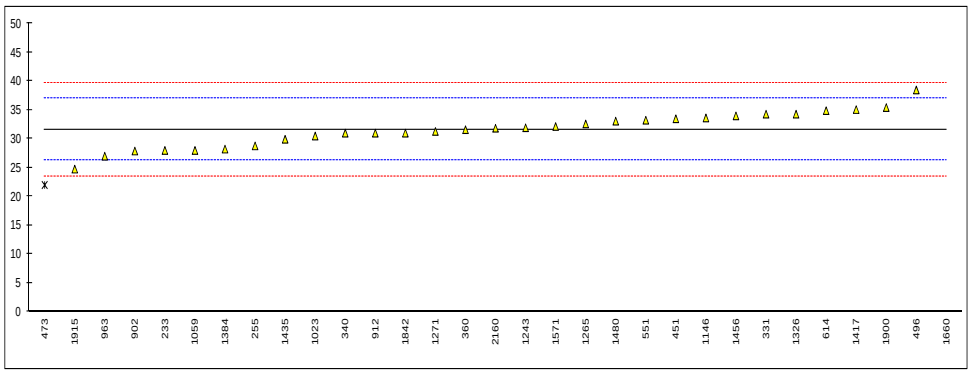
lab	method	value	mark	z(targ)	remarks
233	D6595	11		1.30	
255	INH-1021	8.794		-0.63	
311		-----		-----	
325		-----		-----	
331	D5185	10.5		0.87	
340	D5185	9.2		-0.27	
360	D5185	9.83		0.28	
398		-----		-----	
432		-----		-----	
451	D5185	10.2		0.60	
473	D5185	2	G(0.01)	-6.57	
496	D5185	9.8		0.25	
551	D5185	9.54		0.03	
562		-----		-----	
614	D5185	9.85		0.30	
902	D5185	8.61		-0.79	
912	D5185	10		0.43	
963	D5185	8.9		-0.53	
1017		-----		-----	
1023	D5185	9.1		-0.36	
1059	in house	10		0.43	
1146	D5185	9.59		0.07	
1150		-----		-----	
1155		-----		-----	
1174		-----		-----	
1243	DIN51396	8.24		-1.11	
1244		-----		-----	
1265	D6595	12.8	DG(0.01)	2.88	
1271	D5185	9.5		-0.01	
1326	D5185	9.70		0.17	
1384	D5185	8.6707		-0.73	
1417	in house	10.89		1.21	
1435	D5185	8.750		-0.66	
1456	D5185	11		1.30	
1480	D5185	9.31		-0.17	
1571	D5185	9.8484		0.30	
1628		-----		-----	
1660	D5185	32.74	G(0.01)	20.33	
1842	INH-01	10		0.43	
1900	D6595	13.10	DG(0.01)	3.14	
1915	D5185	7.9781		-1.34	
1920		-----		-----	
2160	D6052	7.96		-1.36	
	normality	OK			
	n	27			
	outliers	4			
	mean (n)	9.51			
	st.dev. (n)	0.846			
	R(calc.)	2.37			
	R(D5185:09)	3.20			Application range ASTM D5185: 1 – 40 mg/kg

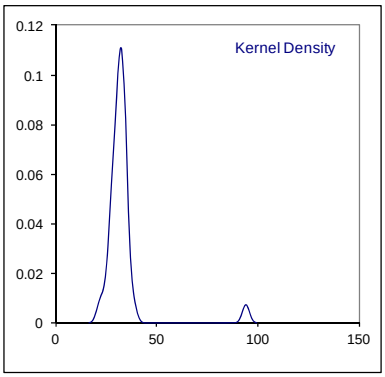




Determination of Copper (Cu) on sample #12142; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	28		-1.34	
255	INH-1021	28.79		-1.05	
311		-----		-----	
325		-----		-----	
331	D5185	34.3		0.98	
340	D5185	31.0		-0.24	
360	D5185	31.6		-0.02	
398		-----		-----	
432		-----		-----	
451	D5185	33.5		0.68	
473	D5185	22	ex	-3.56	Result excluded see §4.1
496	D5185	38.5		2.53	
551	D5185	33.25		0.59	
562		-----		-----	
614	D5185	34.91		1.20	
902	D5185	27.9		-1.38	
912	D5185	31		-0.24	
963	D5185	27		-1.71	
1017		-----		-----	
1023	D5185	30.5		-0.42	
1059	in house	28		-1.34	
1146	D5185	33.65		0.74	
1150		-----		-----	
1155		-----		-----	
1174		-----		-----	
1243	DIN51396	31.94		0.11	
1244		-----		-----	
1265	D6595	32.6		0.35	
1271	D5185	31.3		-0.13	
1326	D5185	34.3		0.98	
1384	D5185	28.2331		-1.26	
1417	in house	35.10		1.27	
1435	D5185	29.94		-0.63	
1456	D5185	34		0.87	
1480	D5185	33.09		0.53	
1571	D5185	32.1624		0.19	
1628		-----		-----	
1660	D5185	93.90	G(0.01)	22.95	
1842	INH-01	31		-0.24	
1900	D6595	35.45		1.40	
1915	D5185	24.762		-2.54	
1920		-----		-----	
2160	in house	31.85		0.08	
	normality	OK			
	n	29			
	outliers	1			
	mean (n)	31.64			
	st.dev. (n)	3.000			
	R(calc.)	8.40			
	R(D5185:09)	7.59			Application range ASTM D5185: 2 – 160 mg/kg

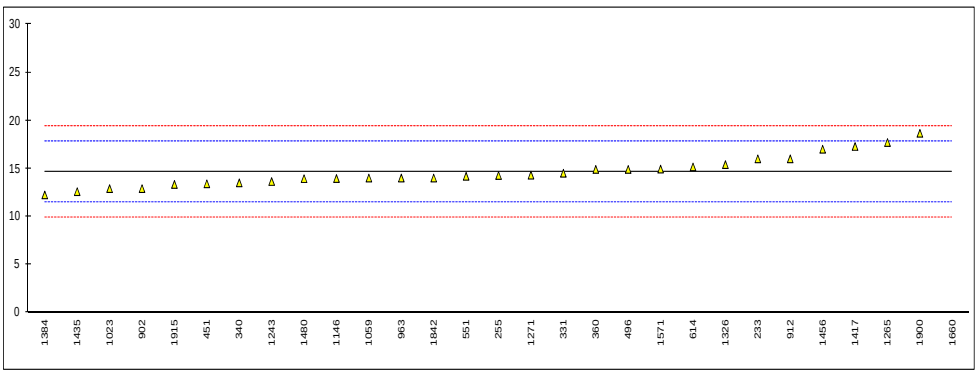


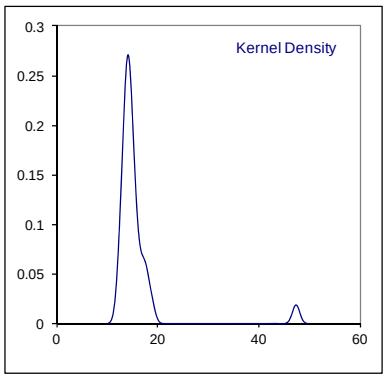


Determination of Iron (Fe) on sample #12142; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	16		0.86	
255	INH-1021	14.267		-0.23	
311		-----		-----	
325		-----		-----	
331	D5185	14.5		-0.08	
340	D5185	13.5		-0.71	
360	D5185	14.9		0.17	
398		-----		-----	
432		-----		-----	
451	D5185	13.4		-0.77	
473	D5185	<0.1		<-9.14	False negative?
496	D5185	14.9		0.17	
551	D5185	14.18		-0.28	
562		-----		-----	
614	D5185	15.16		0.34	
902	D5185	12.9		-1.09	
912	D5185	16		0.86	
963	D5185	14		-0.40	
1017		-----		-----	
1023	D5185	12.9		-1.09	
1059	in house	14		-0.40	
1146	D5185	13.95		-0.43	
1150		-----		-----	
1155		-----		-----	
1174		-----		-----	
1243	DIN51396	13.64		-0.62	
1244		-----		-----	
1265	D6595	17.7		1.93	
1271	D5185	14.3		-0.21	
1326	D5185	15.4		0.49	
1384	D5185	12.2394		-1.50	
1417	in house	17.29		1.68	
1435	D5185	12.58		-1.29	
1456	D5185	17		1.49	
1480	D5185	13.94		-0.43	
1571	D5185	14.937		0.19	
1628		-----		-----	
1660	D5185	47.27	G(0.01)	20.55	
1842	INH-01	14		-0.40	
1900	D6595	18.66		2.54	
1915	D5185	13.335		-0.81	
1920		-----		-----	
2160		-----		-----	

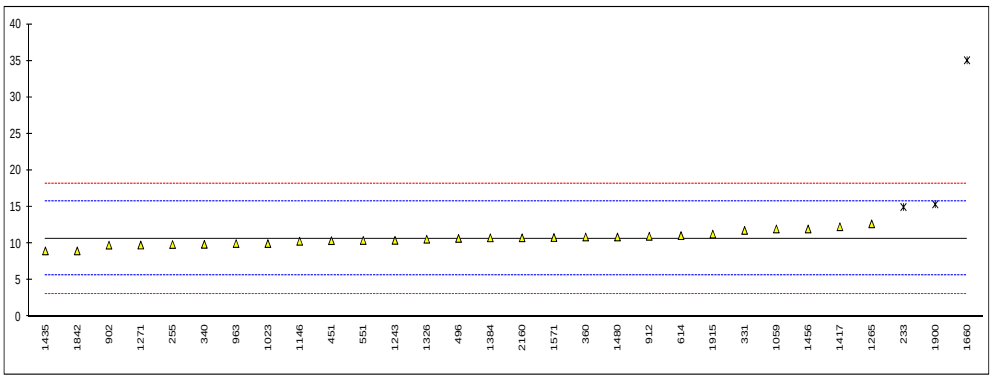
normality	OK	
n	28	
outliers	1	
mean (n)	14.63	
st.dev. (n)	1.577	
R(calc.)	4.42	
R(D5185:09)	4.45	Application range ASTM D5185: 2 – 140 mg/kg

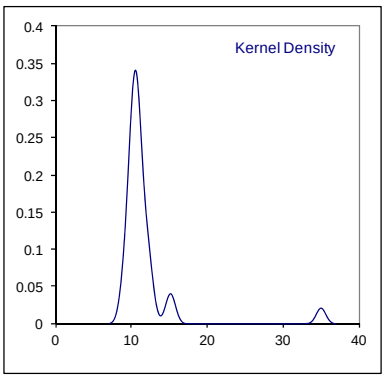




Determination of Lead (Pb) on sample #12142; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	15	G(0.01)	1.71	
255	INH-1021	9.87		-0.33	
311		-----		-----	
325		-----		-----	
331	D5185	11.8		0.44	
340	D5185	9.9		-0.31	
360	D5185	10.9		0.08	
398		-----		-----	
432		-----		-----	
451	D5185	10.4		-0.12	
473	D5185	<0.1		<-4.29	False negative?
496	D5185	10.7		0.00	
551	D5185	10.43		-0.10	
562		-----		-----	
614	D5185	11.09		0.16	
902	D5185	9.78		-0.36	
912	D5185	11		0.12	
963	D5185	10		-0.27	
1017		-----		-----	
1023	D5185	10.0		-0.27	
1059	in house	12		0.52	
1146	D5185	10.32		-0.15	
1150		-----		-----	
1155		-----		-----	
1174		-----		-----	
1243	DIN51396	10.44		-0.10	
1244		-----		-----	
1265	D6595	12.7		0.80	
1271	D5185	9.8		-0.35	
1326	D5185	10.6		-0.04	
1384	D5185	10.7840		0.04	
1417	in house	12.30		0.64	
1435	D5185	9.000		-0.67	
1456	D5185	12		0.52	
1480	D5185	10.90		0.08	
1571	D5185	10.827		0.05	
1628		-----		-----	
1660	D5185	35.06	G(0.01)	9.69	
1842	INH-01	9		-0.67	
1900	D6595	15.37	G(0.05)	1.86	
1915	D5185	11.2902		0.24	
1920		-----		-----	
2160	D6052	10.79		0.04	
	normality	OK			
	n	27			
	outliers	3			
	mean (n)	10.69			
	st.dev. (n)	0.919			
	R(calc.)	2.57			
	R(D5185:09)	7.04			Application range ASTM D5185: 10 – 160 mg/kg

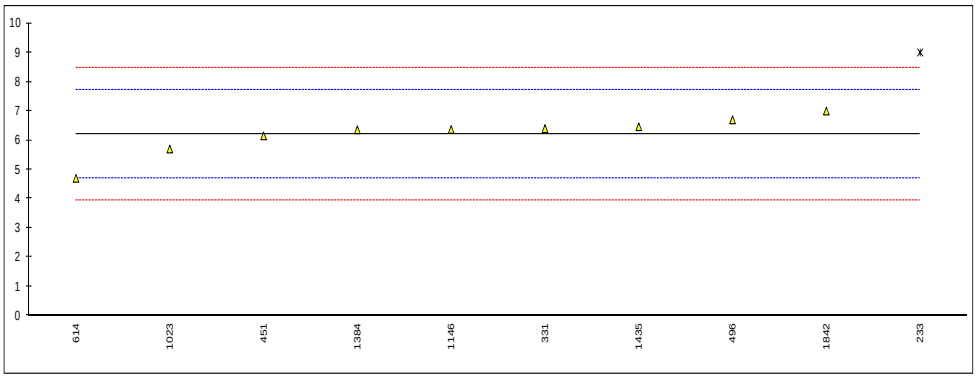


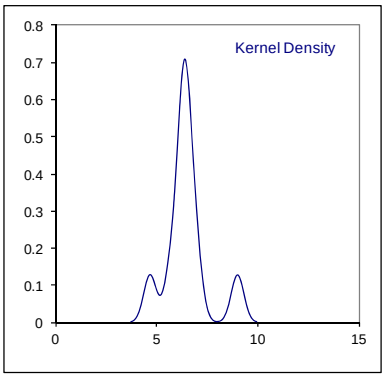


Determination of Lithium (Li) on sample #12142; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	9	G(0.05)	3.71	
255		-----		-----	
311		-----		-----	
325		-----		-----	
331	D5185	6.4		0.26	
340		-----		-----	
360		-----		-----	
398		-----		-----	
432		-----		-----	
451		6.15		-0.07	
473		-----		-----	
496	D5185	6.7		0.66	
551		-----		-----	
562		-----		-----	
614	D5185	4.7		-2.00	
902		-----		-----	
912		-----		-----	
963		-----		-----	
1017		-----		-----	
1023	D5185	5.7		-0.67	
1059		-----		-----	
1146		6.37		0.22	
1150		-----		-----	
1155		-----		-----	
1174		-----		-----	
1243		-----		-----	
1244		-----		-----	
1265		-----		-----	
1271		-----		-----	
1326		-----		-----	
1384	INH-04	6.3601		0.21	
1417		-----		-----	
1435		6.463		0.34	
1456		-----		-----	
1480		-----		-----	
1571		-----		-----	
1628		-----		-----	
1660		-----		-----	
1842	INH-01	7		1.05	
1900		-----		-----	
1915		-----		-----	
1920		-----		-----	
2160		-----		-----	

normality	OK
n	9
outliers	1
mean (n)	6.20
st.dev. (n)	0.667
R(calc.)	1.87
R(Horwitz)	2.11



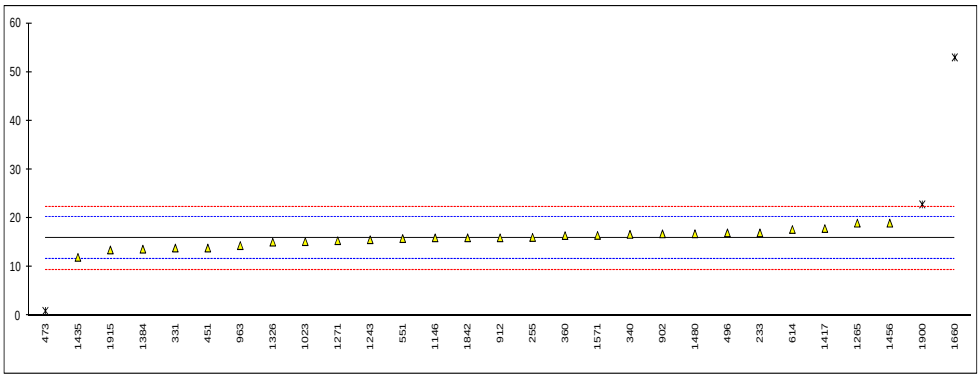


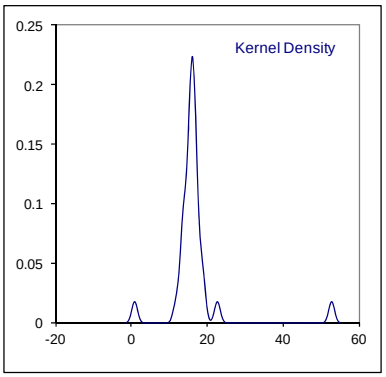
Determination of Magnesium (Mg) on sample #12142; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	17		0.51	
255	INH-1021	16.07		0.08	
311		-----		-----	
325		-----		-----	
331	D5185	13.9		-0.92	
340	D5185	16.7		0.37	
360	D5185	16.46		0.26	
398		-----		-----	
432		-----		-----	
451	D5185	13.9		-0.92	
473	D5185	1	G(0.01)	-6.89	
496	D5185	17.0		0.51	
551	D5185	15.84		-0.03	
562		-----		-----	
614	D5185	17.7		0.83	
902	D5185	16.8		0.42	
912	D5185	16		0.05	
963	D5185	14.4		-0.69	
1017		-----		-----	
1023	D5185	15.2		-0.32	
1059	in house	<50		-----	
1146	D5185	15.99		0.04	
1150		-----		-----	
1155		-----		-----	
1174		-----		-----	
1243	DIN51396	15.60		-0.14	
1244		-----		-----	
1265	D6595	19.0		1.43	
1271	D5185	15.4		-0.23	
1326	D5185	15.1		-0.37	
1384	D5185	13.6667		-1.03	
1417	in house	17.90		0.92	
1435	D5185	11.99		-1.81	
1456	D5185	19		1.43	
1480	D5185	16.83		0.43	
1571	D5185	16.480		0.27	
1628		-----		-----	
1660	D5185	53.0	G(0.01)	17.14	
1842	INH-01	16		0.05	
1900	D6595	22.84	G(0.05)	3.21	
1915	D5185	13.5022		-1.11	
1920		-----		-----	
2160		-----		-----	

normality	OK
n	26
outliers	3
mean (n)	15.90
st.dev. (n)	1.669
R(calc.)	4.67
R(D5185:09)	6.06

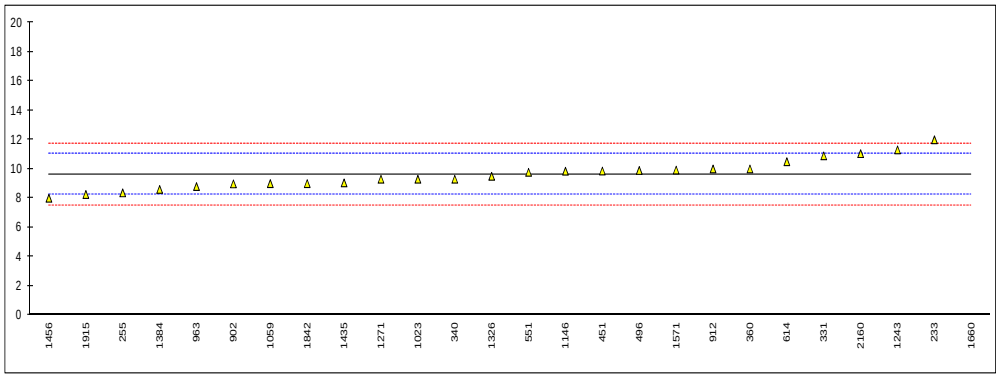
Application range ASTM D5185: 5 – 1700 mg/kg

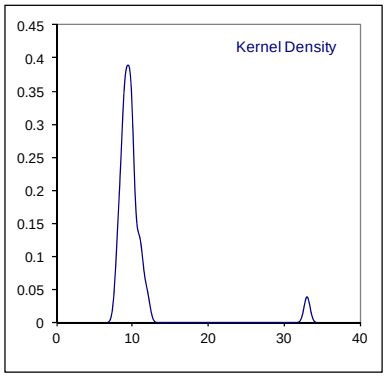




Determination of Manganese (Mn) on sample #12142; results in mg/kg.

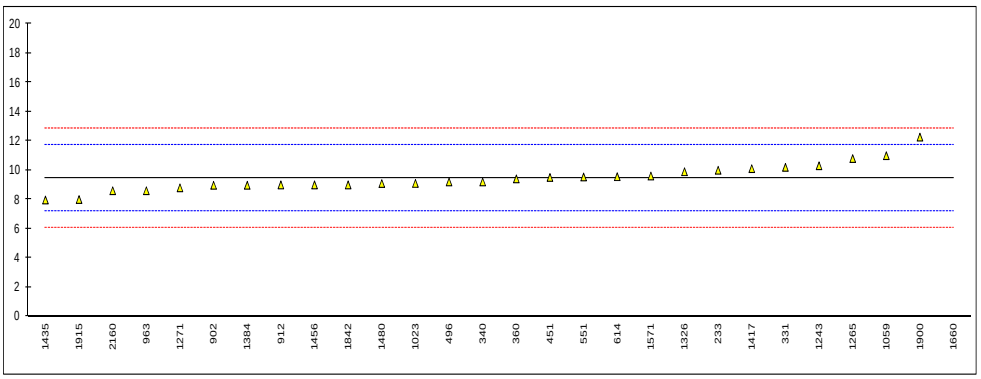
lab	method	value	mark	z(targ)	remarks
233	D6595	12		3.39	
255	INH-1021	8.366		-1.79	
311		-----		-----	
325		-----		-----	
331	D5185	10.9		1.82	
340	D5185	9.3		-0.46	
360	D5185	10.0		0.54	
398		-----		-----	
432		-----		-----	
451	D5185	9.86		0.34	
473	D5185	<0.1		<-13.53	False negative?
496	D5185	9.9		0.40	
551	D5185	9.77		0.21	
562		-----		-----	
614	D5185	10.5		1.25	
902	D5185	8.98		-0.91	
912	D5185	10		0.54	
963	D5185	8.8		-1.17	
1017		-----		-----	
1023	D5185	9.3		-0.46	
1059	in house	9		-0.88	
1146	D5185	9.85		0.33	
1150		-----		-----	
1155		-----		-----	
1174		-----		-----	
1243	DIN51396	11.31		2.40	
1244		-----		-----	
1265		-----		-----	
1271	D5185	9.3		-0.46	
1326	D5185	9.50		-0.17	
1384	D5185	8.5990		-1.45	
1417		-----		-----	
1435	D5185	9.048		-0.82	
1456	D5185	8		-2.31	
1480		-----		-----	
1571	D5185	9.9259		0.43	
1628		-----		-----	
1660	D5185	33.10	G(0.01)	33.42	
1842	INH-01	9		-0.88	
1900		-----		-----	
1915	D5185	8.2561		-1.94	
1920		-----		-----	
2160	in house	11.05		2.03	
	normality	OK			
	n	25			
	outliers	1			
	mean (n)	9.62			
	st.dev. (n)	0.983			
	R(calc.)	2.75			
	R(D5185:09)	1.97			Application range ASTM D55185: 5 – 700 mg/kg

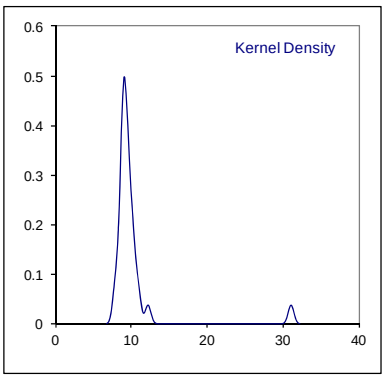




Determination of Molybdenum (Mo) on sample #12142; results in mg/kg.

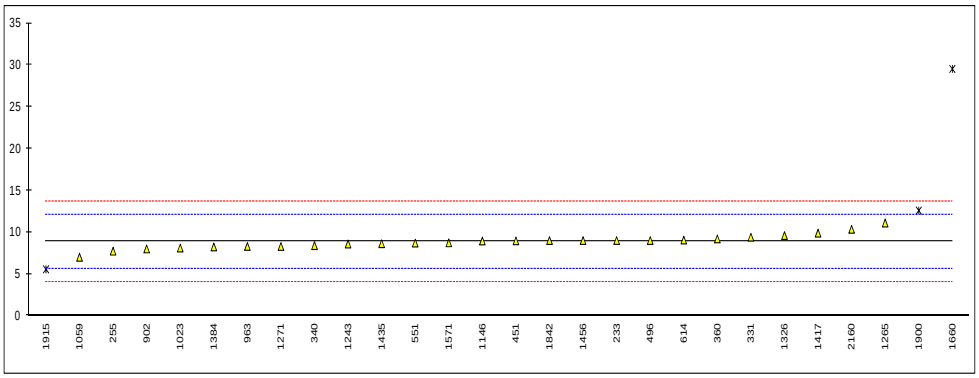
lab	method	value	mark	z(targ)	remarks
233	D6595	10		0.47	
255		-----		-----	
311		-----		-----	
325		-----		-----	
331	D5185	10.2		0.65	
340	D5185	9.2		-0.24	
360	D5185	9.41		-0.05	
398		-----		-----	
432		-----		-----	
451	D5185	9.51		0.03	
473	D5185	<0.1		<-8.30	False negative?
496	D5185	9.2		-0.24	
551	D5185	9.54		0.06	
562		-----		-----	
614	D5185	9.56		0.08	
902	D5185	8.97		-0.44	
912	D5185	9		-0.42	
963	D5185	8.6		-0.77	
1017		-----		-----	
1023	D5185	9.1		-0.33	
1059	in house	11		1.36	
1146		-----		-----	
1150		-----		-----	
1155		-----		-----	
1174		-----		-----	
1243	DIN51396	10.31		0.74	
1244		-----		-----	
1265	D6595	10.8		1.18	
1271	D5185	8.8		-0.59	
1326	D5185	9.90		0.38	
1384	D5185	8.9746		-0.44	
1417	in house	10.11		0.57	
1435	D5185	7.964		-1.34	
1456	D5185	9		-0.42	
1480	D5185	9.09		-0.34	
1571	D5185	9.6054		0.12	
1628		-----		-----	
1660	D5185	31.13	G(0.01)	19.20	
1842	INH-01	9		-0.42	
1900	D6595	12.27		2.48	
1915	D5185	7.9986		-1.31	
1920		-----		-----	
2160	in house	8.60		-0.77	
	normality	OK			
	n	27			
	outliers	1			
	mean (n)	9.47			
	st.dev. (n)	0.922			
	R(calc.)	2.58			
	R(D5185:09)	3.16			Application range ASTM D5185: 5 – 200 mg/kg

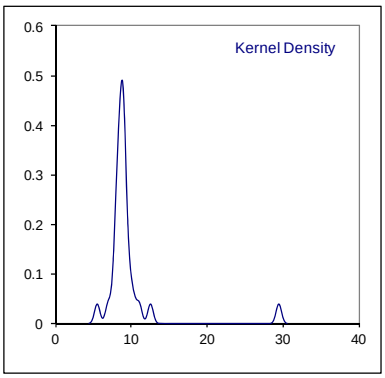




Determination of Nickel (Ni) on sample #12142; results in mg/kg.

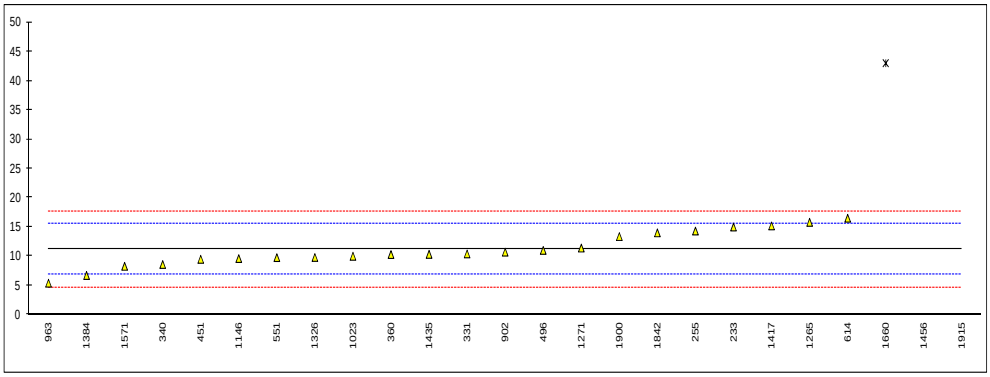
lab	method	value	mark	z(targ)	remarks
233	D6595	9		0.10	
255	INH-1021	7.731		-0.70	
311		-----		-----	
325		-----		-----	
331	D5185	9.4		0.35	
340	D5185	8.4		-0.28	
360	D5185	9.19		0.21	
398		-----		-----	
432		-----		-----	
451	D5185	8.96		0.07	
473	D5185	<0.1		<-5.49	False negative?
496	D5185	9.0		0.10	
551	D5185	8.70		-0.09	
562		-----		-----	
614	D5185	9.06		0.13	
902	D5185	8.00		-0.53	
912		-----		-----	
963	D5185	8.3		-0.34	
1017		-----		-----	
1023	D5185	8.1		-0.47	
1059	in house	7		-1.16	
1146	D5185	8.95		0.06	
1150		-----		-----	
1155		-----		-----	
1174		-----		-----	
1243	DIN51396	8.59		-0.16	
1244		-----		-----	
1265	D6595	11.1		1.41	
1271	D5185	8.3		-0.34	
1326	D5185	9.60		0.47	
1384	D5185	8.2349		-0.38	
1417	in house	9.89		0.65	
1435	D5185	8.621		-0.14	
1456	D5185	9		0.10	
1480		-----		-----	
1571	D5185	8.7279		-0.08	
1628		-----		-----	
1660	D5185	29.53	G(0.01)	12.98	
1842	INH-01	9		0.10	
1900	D6595	12.59	G(0.05)	2.35	
1915	D5185	5.5554	G(0.05)	-2.07	
1920		-----		-----	
2160	in house	10.35		0.94	
	normality	OK			
	n	25			
	outliers	3			
	mean (n)	8.85			
	st.dev. (n)	0.838			
	R(calc.)	2.35			
	R(D5185:09)	4.46			Application range ASTM D5185: 5 – 40 mg/kg

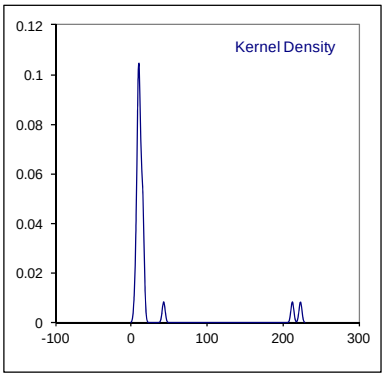




Determination of Sodium (Na) on sample #12142; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	15		1.75	
255	INH-1021	14.3065		1.43	
311		-----		-----	
325		-----		-----	
331	D5185	10.4		-0.36	
340	D5185	8.6		-1.18	
360	D5185	10.3		-0.40	
398		-----		-----	
432		-----		-----	
451	D5185	9.48		-0.78	
473	D5185	<0.1		<-5.08	False negative?
496	D5185	11.0		-0.08	
551	D5185	9.77		-0.65	
562		-----		-----	
614	D5185	16.5		2.44	
902	D5185	10.7		-0.22	
912		-----		-----	
963	D5185	5.4		-2.65	
1017		-----		-----	
1023	D5185	10.0		-0.54	
1059		-----		-----	
1146	D5185	9.60		-0.72	
1150		-----		-----	
1155		-----		-----	
1174		-----		-----	
1243		-----		-----	
1244		-----		-----	
1265	D6595	15.8		2.12	
1271	D5185	11.4		0.10	
1326	D5185	9.80		-0.63	
1384	D5185	6.7170		-2.05	
1417	in house	15.19		1.84	
1435	D5185	10.33		-0.39	
1456	D5185	213	G(0.01)	92.55	
1480		-----		-----	
1571	D5185	8.2826		-1.33	
1628		-----		-----	
1660	D5185	43.0	G(0.01)	14.59	
1842	INH-01	14		1.29	
1900	D6595	13.37		1.00	
1915	D5185	223.698	G(0.01)	97.45	
1920		-----		-----	
2160		-----		-----	
	normality	OK			
	n	22			
	outliers	3			
	mean (n)	11.18			
	st.dev. (n)	2.963			
	R(calc.)	8.30			
	R(D5185:09)	6.11			Application range ASTM D5185: 7 – 70 mg/kg

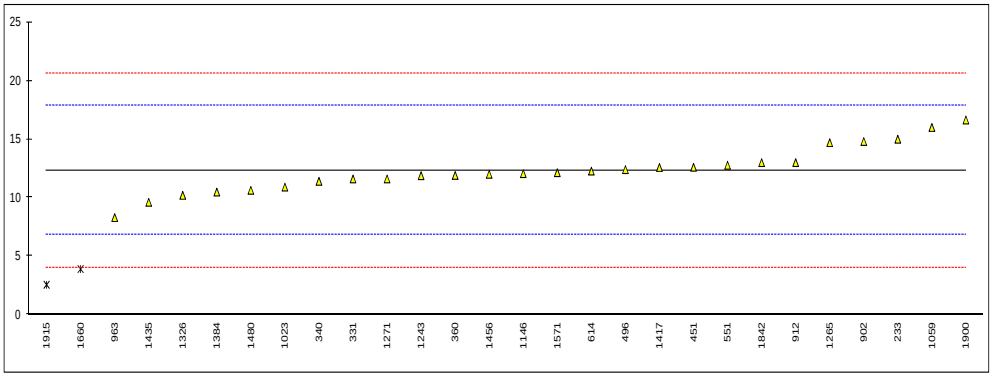


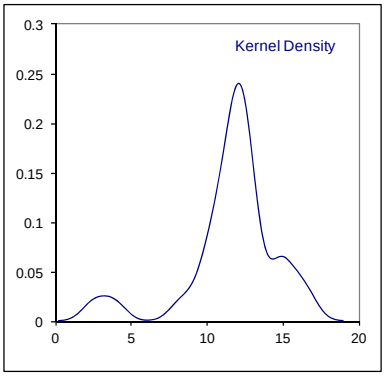


Determination of Silicon (Si) on sample #12142; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	15		0.97	
255		-----		-----	
311		-----		-----	
325		-----		-----	
331	D5185	11.6		-0.26	
340	D5185	11.4		-0.34	
360	D5185	11.9		-0.15	
398		-----		-----	
432		-----		-----	
451	D5185	12.6		0.10	
473	D5185	<0.1		<-4.43	False negative?
496	D5185	12.4		0.03	
551	D5185	12.77		0.16	
562		-----		-----	
614	D5185	12.27		-0.02	
902	D5185	14.8		0.90	
912	D5185	13		0.24	
963	D5185	8.3		-1.46	
1017		-----		-----	
1023	D5185	10.9		-0.52	
1059	in house	16		1.33	
1146	D5185	12.05		-0.10	
1150		-----		-----	
1155		-----		-----	
1174		-----		-----	
1243	DIN51396	11.88		-0.16	
1244		-----		-----	
1265	D6595	14.7		0.86	
1271	D5185	11.6		-0.26	
1326	D5185	10.2		-0.77	
1384	D5185	10.4767		-0.67	
1417	in house	12.58		0.09	
1435	D5185	9.608		-0.98	
1456	D5185	12		-0.12	
1480	D5185	10.63		-0.61	
1571	D5185	12.1383		-0.07	
1628		-----		-----	
1660	D5185	3.90	G(0.01)	-3.05	
1842	INH-01	13		0.24	
1900	D6595	16.64		1.56	
1915	D5185	2.5568	G(0.05)	-3.54	
1920		-----		-----	
2160		-----		-----	

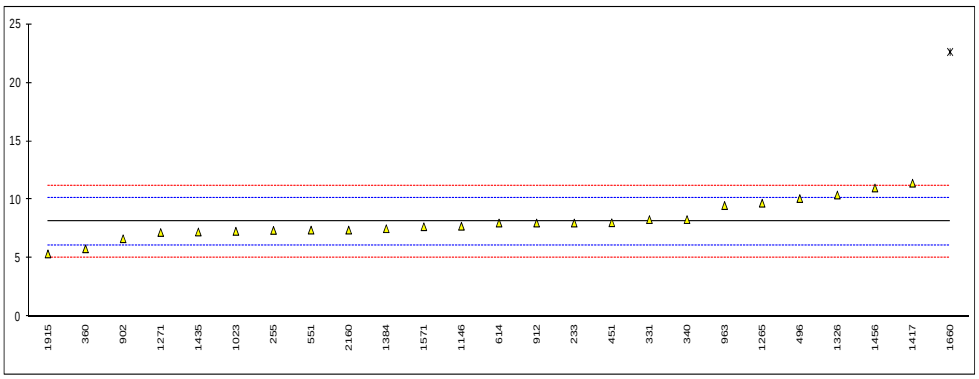
normality	OK	
n	26	
outliers	2	
mean (n)	12.32	
st.dev. (n)	1.910	
R(calc.)	5.35	
R(D5185:09)	7.72	Application range ASTM D5185: 8 – 50 mg/kg

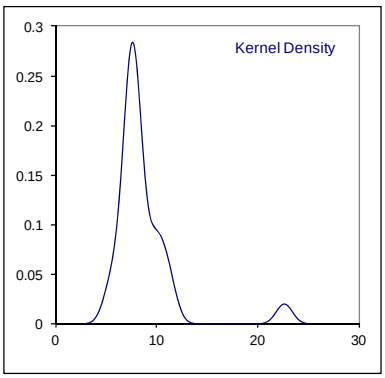




Determination of Silver (Ag) on sample #12142; results in mg/kg.

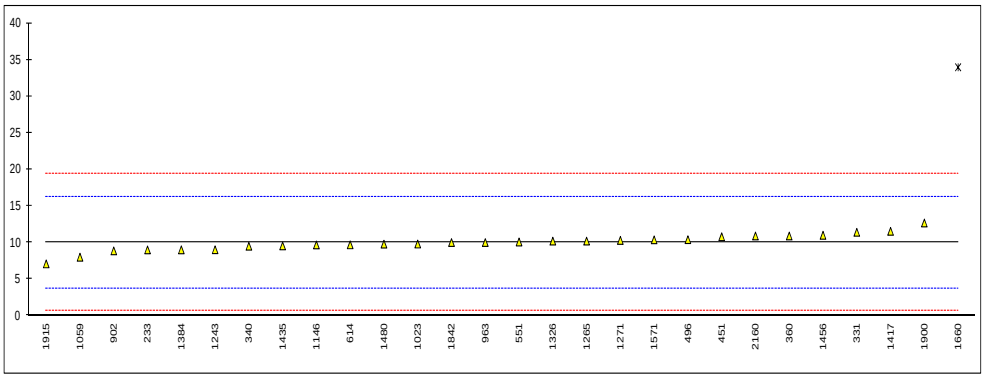
lab	method	value	mark	z(targ)	remarks
233	D6595	8		-0.14	
255	INH-1021	7.373		-0.76	
311		-----		-----	
325		-----		-----	
331	D5185	8.3		0.15	
340	D5185	8.3		0.15	
360	D5185	5.80		-2.30	
398		-----		-----	
432		-----		-----	
451	D5185	8.03		-0.11	
473	D5185	<0.1		<-7.90	False negative?
496	D5185	10.1		1.92	
551	D5185	7.40		-0.73	
562		-----		-----	
614	D5185	8.0		-0.14	
902	D5185	6.66		-1.46	
912	D5185	8		-0.14	
963	D5185	9.5		1.33	
1017		-----		-----	
1023	D5185	7.3		-0.83	
1059		-----		-----	
1146	D5185	7.73		-0.41	
1150		-----		-----	
1155		-----		-----	
1174		-----		-----	
1243		-----		-----	
1244		-----		-----	
1265	D6595	9.7		1.53	
1271	D5185	7.2		-0.93	
1326	D5185	10.4		2.22	
1384	D5185	7.5191		-0.61	
1417	in house	11.41		3.21	
1435	D5185	7.241		-0.89	
1456	D5185	11		2.81	
1480		-----		-----	
1571	D5185	7.6898		-0.44	
1628		-----		-----	
1660	D5185	22.62	G(0.01)	14.22	
1842		-----		-----	
1900		-----		-----	
1915	D5185	5.3682		-2.73	
1920		-----		-----	
2160	in house	7.40		-0.73	
	normality	not OK			
	n	24			
	outliers	1			
	mean (n)	8.14			
	st.dev. (n)	1.510			
	R(calc.)	4.23			
	R(D5185:09)	2.85			Application range ASTM D5185: 0.5 – 50 mg/kg

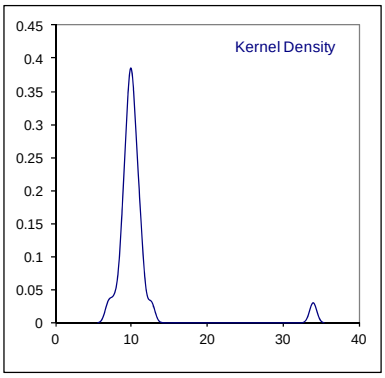




Determination of Tin (Sn) on sample #12142; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	9		-0.32	
255		-----		-----	
311		-----		-----	
325		-----		-----	
331	D5185	11.4		0.45	
340	D5185	9.5		-0.16	
360	D5185	10.9		0.29	
398		-----		-----	
432		-----		-----	
451	D5185	10.8		0.26	
473	D5185	<0.1		<-3.16	False negative?
496	D5185	10.4		0.13	
551	D5185	10.08		0.03	
562		-----		-----	
614	D5185	9.69		-0.10	
902	D5185	8.86		-0.36	
912		-----		-----	
963	D5185	10.0		0.00	
1017		-----		-----	
1023	D5185	9.8		-0.06	
1059	in house	8		-0.64	
1146	D5185	9.67		-0.10	
1150		-----		-----	
1155		-----		-----	
1174		-----		-----	
1243	DIN51396	9.02		-0.31	
1244		-----		-----	
1265	D6595	10.2		0.07	
1271	D5185	10.3		0.10	
1326	D5185	10.2		0.07	
1384	D5185	9.0073		-0.31	
1417	in house	11.53		0.49	
1435	D5185	9.539		-0.14	
1456	D5185	11		0.32	
1480	D5185	9.78		-0.07	
1571	D5185	10.3723		0.12	
1628		-----		-----	
1660	D5185	33.97	G(0.01)	7.68	
1842	INH-01	10		0.00	
1900	D6595	12.68		0.86	
1915	D5185	7.0859		-0.93	
1920		-----		-----	
2160	in house	10.90		0.29	
	normality	OK			
	n	27			
	outliers	1			
	mean (n)	9.99			
	st.dev. (n)	1.122			
	R(calc.)	3.14			
	R(D5185:09)	8.75			Application range ASTM D5185: 10 – 40 mg/kg

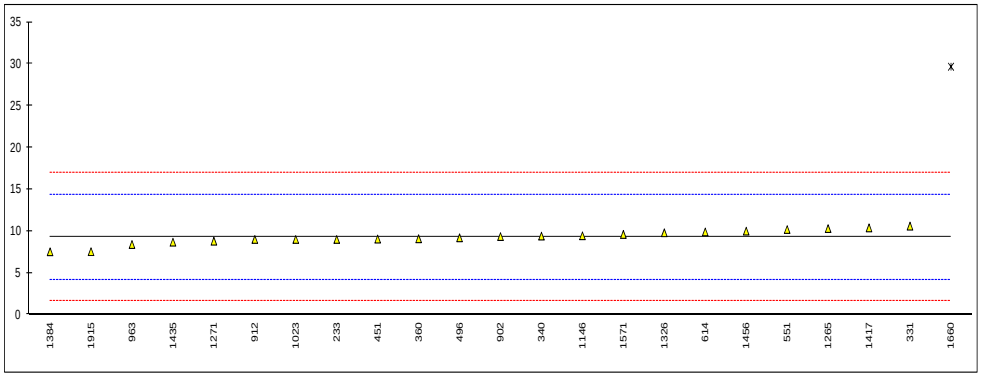


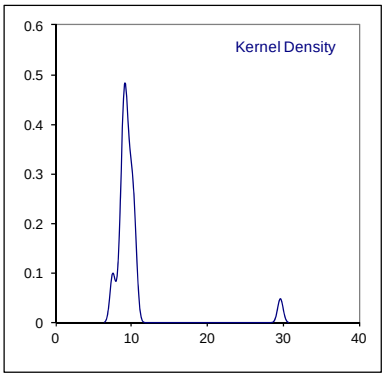


Determination of Titanium (Ti) on sample #12142; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	9		-0.11	
255		-----		-----	
311		-----		-----	
325		-----		-----	
331	D5185	10.6		0.52	
340	D5185	9.4		0.05	
360	D5185	9.09		-0.08	
398		-----		-----	
432		-----		-----	
451	D5185	9.05		-0.09	
473	D5185	<0.1		<-3.61	False negative?
496	D5185	9.2		-0.03	
551	D5185	10.19		0.36	
562		-----		-----	
614	D5185	9.9		0.24	
902	D5185	9.35		0.03	
912	D5185	9		-0.11	
963	D5185	8.4		-0.35	
1017		-----		-----	
1023	D5185	9.0		-0.11	
1059		-----		-----	
1146	D5185	9.44		0.06	
1150		-----		-----	
1155		-----		-----	
1174		-----		-----	
1243		-----		-----	
1244		-----		-----	
1265	D6595	10.3		0.40	
1271	D5185	8.8		-0.19	
1326	D5185	9.80		0.20	
1384	D5185	7.5267		-0.69	
1417	in house	10.39		0.43	
1435	D5185	8.674		-0.24	
1456	D5185	10		0.28	
1480		-----		-----	
1571	D5185	9.6068		0.13	
1628		-----		-----	
1660	D5185	29.67	G(0.01)	8.01	
1842		-----		-----	
1900		-----		-----	
1915	D5185	7.5397		-0.69	
1920		-----		-----	
2160		-----		-----	

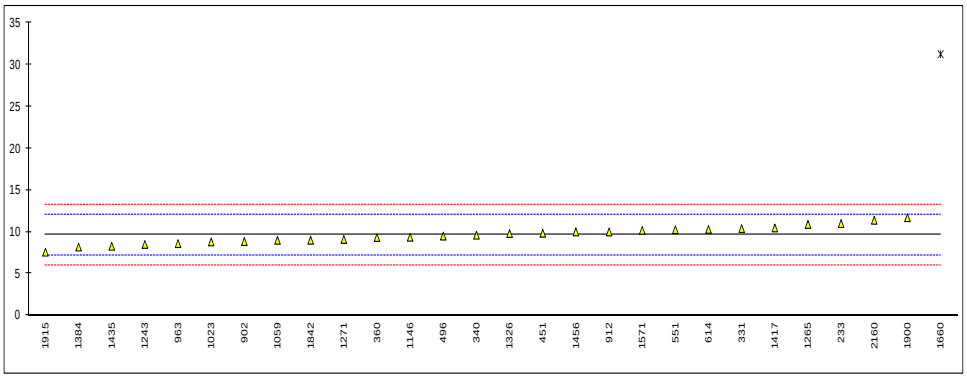
normality	OK	
n	22	
outliers	1	
mean (n)	9.28	
st.dev. (n)	0.816	
R(calc.)	2.28	
R(D5185:09)	7.12	Application range ASTM D5185: 5 - 40 mg/kg

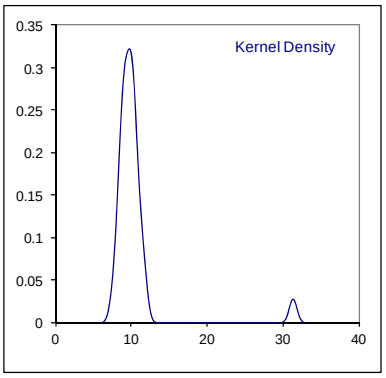




Determination of Vanadium (V) on sample #12142; results in mg/kg.

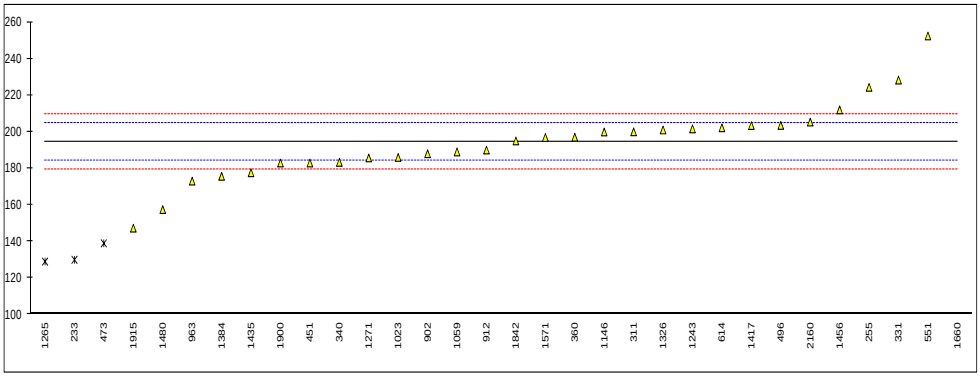
lab	method	value	mark	z(targ)	remarks
233	D6595	11		1.14	
255		-----		-----	
311		-----		-----	
325		-----		-----	
331	D5185	10.4		0.64	
340	D5185	9.6		-0.02	
360	D5185	9.32		-0.25	
398		-----		-----	
432		-----		-----	
451	D5185	9.84		0.18	
473	D5185	<0.1		<-7.89	False negative?
496	D5185	9.5		-0.10	
551	D5185	10.26		0.52	
562		-----		-----	
614	D5185	10.3		0.56	
902	D5185	8.85		-0.64	
912	D5185	10		0.31	
963	D5185	8.6		-0.85	
1017		-----		-----	
1023	D5185	8.8		-0.68	
1059	in house	9		-0.52	
1146	D5185	9.36		-0.22	
1150		-----		-----	
1155		-----		-----	
1174		-----		-----	
1243	DIN51396	8.50		-0.93	
1244		-----		-----	
1265	D6595	10.9		1.06	
1271	D5185	9.1		-0.44	
1326	D5185	9.80		0.14	
1384	D5185	8.1808		-1.20	
1417	in house	10.47		0.70	
1435	D5185	8.277		-1.12	
1456	D5185	10		0.31	
1480		-----		-----	
1571	D5185	10.1840		0.46	
1628		-----		-----	
1660	D5185	31.28	G(0.01)	17.94	
1842	INH-01	9		-0.52	
1900	D6595	11.70		1.72	
1915	D5185	7.5684		-1.70	
1920		-----		-----	
2160	in house	11.40		1.47	
	normality	OK			
	n	27			
	outliers	1			
	mean (n)	9.63			
	st.dev. (n)	1.015			
	R(calc.)	2.84			
	R(D5185:09)	3.38			Application range ASTM D5185: 1 – 50 mg/kg

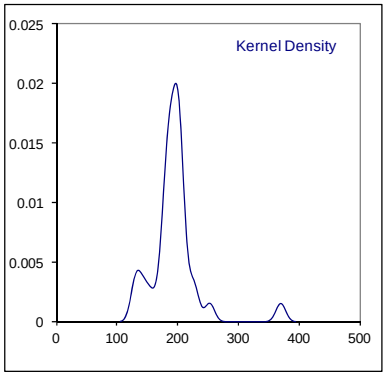




Determination of Calcium (Ca) on sample #12142; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	130	DG(0.05)	-12.72	
255	INH-1021	224.301		5.92	
311	D5185	200		1.12	
325		-----		-----	
331	D5185	228.3		6.71	
340	D5185	183.3		-2.18	
360	D5185	197		0.53	
398		-----		-----	
432		-----		-----	
451	D5185	182.9		-2.26	
473	D5185	139	ex	-10.94	Result excluded, see §4.1
496	D5185	203.5		1.81	
551	D5185	252.43		11.48	
562		-----		-----	
614	D5185	202.2		1.55	
902	D5185	188		-1.25	
912	D5185	190		-0.86	
963	D5185	173		-4.22	
1017		-----		-----	
1023	D5185	186		-1.65	
1059	in house	189		-1.06	
1146	D5185	199.9		1.10	
1150		-----		-----	
1155		-----		-----	
1174		-----		-----	
1243	DIN31391	201.6		1.43	
1244		-----		-----	
1265	D6595	129	DG(0.05)	-12.92	
1271	D5185	185.7		-1.71	
1326	D5185	201		1.32	
1384	D5185	175.6778		-3.69	
1417	in house	203.4		1.79	
1435	D5185	177.6		-3.31	
1456	D5185	212		3.49	
1480	D5185	157.46		-7.29	
1571	D5185	196.886		0.50	
1628		-----		-----	
1660	D5185	370	G(0.01)	34.72	
1842	INH-01	195		0.13	
1900	D5185	182.9		-2.26	
1915	D5185	147.260		-9.31	
1920		-----		-----	
2160	in house	205.3		2.17	
	normality	OK			
	n	28			
	outliers	3			
	mean (n)	194.34			
	st.dev. (n)	20.669			
	R(calc.)	57.87			
	R(D5185:09)	14.17			Application range ASTM D5185: 40 – 9000 mg/kg



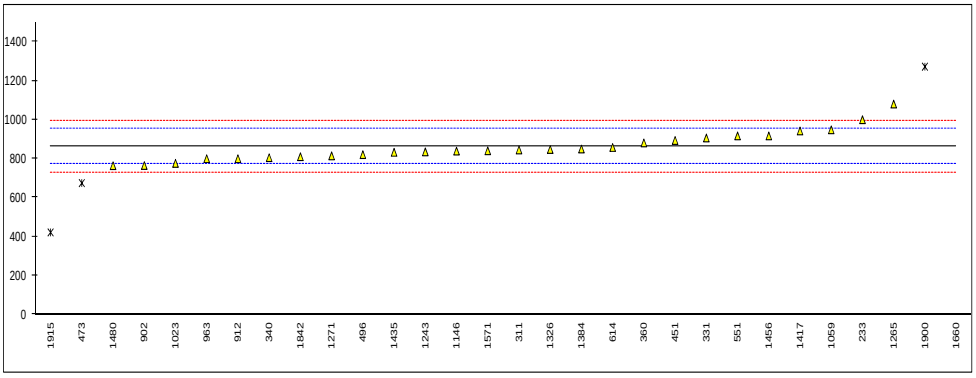


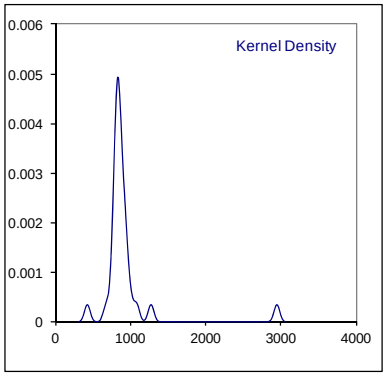
Determination of Phosphorus (P) on sample #12142; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	1000		3.08	
255		-----		-----	
311	D5185	845		-0.36	
325		-----		-----	
331	D5185	906.3		1.00	
340	D5185	805.0		-1.24	
360	D5185	881		0.44	
398		-----		-----	
432		-----		-----	
451	D5185	893.4		0.72	
473	D5185	675	ex	-4.13	Result excluded, see §4.1
496	D5185	821.0		-0.89	
551	D5185	916.92		1.24	
562		-----		-----	
614	D5185	857.4		-0.08	
902	D5185	765		-2.13	
912	D5185	800		-1.35	
963	D5185	800		-1.35	
1017		-----		-----	
1023	D5185	776		-1.89	
1059	in house	948		1.93	
1146	D5185	838.9		-0.49	
1150		-----		-----	
1155		-----		-----	
1174		-----		-----	
1243	DIN31391	835.0		-0.58	
1244		-----		-----	
1265	D6595	1080		4.86	
1271	D5185	814.6		-1.03	
1326	D5185	847		-0.31	
1384	D5185	849.5500		-0.25	
1417	in house	942.1		1.80	
1435	D5185	832.8		-0.63	
1456	D5185	917		1.24	
1480	D5185	764.09		-2.15	
1571	D5185	840.590		-0.45	
1628		-----		-----	
1660	D5185	2950	G(0.01)	46.36	
1842	INH-01	810		-1.13	
1900	D5185	1271.7	G(0.05)	9.11	
1915	D5185	421.89	G(0.05)	-9.74	
1920		-----		-----	
2160		-----		-----	

normality	not OK
n	26
outliers	3
mean (n)	861.03
st.dev. (n)	74.008
R(calc.)	207.22
R(D5185:09)	126.16

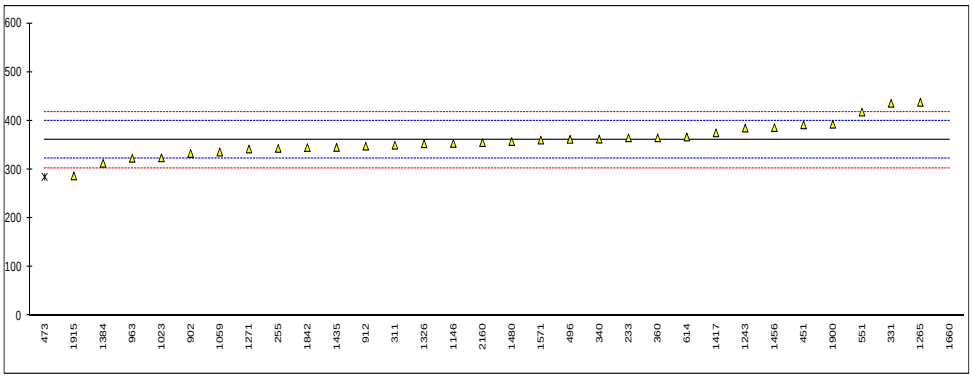
Application range ASTM D5185: 10 – 1000 mg/kg

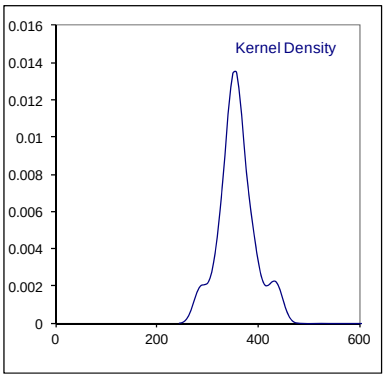




Determination of Zinc (Zn) on sample #12142; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
233	D6595	365		0.24	
255	INH-1021	343.46		-0.88	
311	D5185	350		-0.54	
325		-----		-----	
331	D5185	436.0		3.92	
340	D5185	362.4		0.10	
360	D5185	365		0.24	
398		-----		-----	
432		-----		-----	
451	D5185	391.8		1.63	
473	D5185	285	ex	-3.92	Result excluded, see §4.1
496	D5185	362.0		0.08	
551	D5185	417.89		2.98	
562		-----		-----	
614	D5185	366.9		0.33	
902	D5185	333		-1.43	
912	D5185	348		-0.65	
963	D5185	323		-1.95	
1017		-----		-----	
1023	D5185	324		-1.89	
1059	in house	336		-1.27	
1146	D5185	353.6		-0.36	
1150		-----		-----	
1155		-----		-----	
1174		-----		-----	
1243	DIN31391	385.0		1.27	
1244		-----		-----	
1265	D6595	438		4.03	
1271	D5185	342.1		-0.95	
1326	D5185	353		-0.39	
1384	D5185	312.9545		-2.47	
1417	in house	375.4		0.78	
1435	D5185	345.5		-0.78	
1456	D5185	386		1.33	
1480	D5185	357.49		-0.15	
1571	D5185	360.415		0.00	
1628		-----		-----	
1660	D5185	2000	G(0.01)	85.16	
1842	INH-01	345		-0.80	
1900	D5185	393.0		1.69	
1915	D5185	287.015		-3.82	
1920		-----		-----	
2160	in house	355.2		-0.27	
	normality	OK			
	n	30			
	outliers	1			
	mean (n)	360.47			
	st.dev. (n)	33.142			
	R(calc.)	92.80			
	R(D5185:09)	53.91			Application range ASTM D5185: 60 – 1600 mg/kg





APPENDIX 2

Number of participants per country

2 laboratories in	AUSTRALIA
1 laboratory in	AUSTRIA
3 laboratories in	BELGIUM
1 laboratory in	BOSNIA and HERZEGOVIN
1 laboratory in	BRAZIL
3 laboratories in	BULGARIA
1 laboratory in	CHILE
1 laboratory in	ESTONIA
2 laboratories in	FRANCE
2 laboratories in	GERMANY
1 laboratories in	GHANA
1 laboratory in	INDIA
2 laboratories in	ITALY
1 laboratory in	MALAYSIA
3 laboratories in	NORWAY
1 laboratory in	P.R. of CHINA
1 laboratory in	POLAND
2 laboratories in	SAUDI ARABIA
1 laboratory in	SERBIA
1 laboratory in	SLOVENIA
1 laboratory in	SOUTH KOREA
1 laboratory in	SPAIN
2 laboratories in	SWEDEN
1 laboratory in	TANZANIA
2 laboratories in	THE NETHERLANDS
2 laboratories in	TURKEY
3 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/).