

# Results of Proficiency Test Used Lubricating Oil April 2009

Organised by: Institute for Interlaboratory Studies  
Spijkenisse, the Netherlands

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## 1 INTRODUCTION

Since 1997, a proficiency test for Lubricating Oil is organized every year by the Institute for Interlaboratory Studies. During the annual proficiency testing program 2008/2009, it was decided to continue the round robin for the analyses of Lubricating Oil. In this international interlaboratory study 79 laboratories in 48 different countries have participated, but not all laboratories reported results for the used oil analysis. See appendix 2 for a list of participants in alphabetical country order. In this report the results of the Lubricating Oil (used oil) proficiency test are presented and discussed.

## 2 SET UP

The Institute for Interlaboratory Studies (i.i.s.) in Spijkenisse, The Netherlands, was the organizer of this proficiency test. It was decided to send 2 different samples (1\*500 mL (labelled #0937) and 1\*50 mL (labelled #0938)) of used Lubricating Oil that was donated by one of the participating laboratories. Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

### 2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO guide 43 and ILAC-G13:2007, (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This ensures 100% confidentiality of participant's data. Also customer's satisfaction is measured on a regular basis by sending questionnaires.

### 2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'i.i.s. Interlaboratory Studies: Protocol for the Organization, Statistics and Evaluation' of November 2008 (i.i.s.-protocol, version 3.1).

### 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 of 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

In this proficiency test two different samples were used.

The necessary bulk material for the first sample, used Lubricating Oil, was donated by a third party laboratory. The necessary 60 litre bulk material was homogenised in a drum. After homogenisation, 110 subsamples were transferred to 0.5 L brown glass bottles, labelled #0937. The homogeneity of the subsamples #0937 was checked by determination of Density @ 20°C in

accordance with ASTM D4052:02e1 and Water in accordance with ASTM D6304:07 on 6 stratified randomly selected samples.

The analyses were subcontracted to an accredited laboratory.

	Density @ 20°C In kg/L	Water in mg/kg
sample #0937-1	0.8925	1815
sample #0937-2	0.8925	1870
sample #0937-3	0.8925	1840
sample #0937-4	0.8925	1810
sample #0937-5	0.8926	1835
sample #0937-6	0.8925	1840

Table 1: homogeneity test of subsamples #0937

The second bulk material, used Lubricating Oil fortified with several wear metals, was also obtained from a third party laboratory. The approximately 5 L bulk material was homogenised in a precleaned can. After homogenisation, 119 subsamples were transferred to 50 mL PE bottles, each filled with approximately 40 mL material and labelled #0938. The homogeneity of the subsamples #0938 was checked by the determination of Phosphorus and Zinc in accordance with ASTM D5185:09 on 4 stratified randomly selected samples.

	Phosphorus in mg/kg	Zinc in mg/kg
sample #0938-1	770	825
sample #0938-2	775	825
sample #0938-3	780	816
sample #0938-4	785	821

Table 2: homogeneity test of subsamples #0938

From the results in tables 1 and 2 the repeatabilities of the results were calculated by multiplication of the standard deviations by 2.8:

	Density @ 20°C in kg/L	Water in mg/kg	Phosporus in mg/kg	Zinc in mg/kg
r (sample #0937)	0.0001	60	---	---
r (sample #0938)	---	---	18	12
reference method	ASTM D4052:02e1	ASTM D6304:04	ASTM D5185:09	ASTM D5185:09
r (reference)	0.0001	80	62	55

Table 3: repeatability of subsamples #0937 and #0938

The repeatabilities of the results of the homogeneity tests for Density @ 20°C and water for sample #0937 are both in agreement with the repeatabilities required by respective standards. Therefore, the homogeneity of the prepared subsamples #0937 was assumed.

The repeatabilities of the results of the homogeneity tests for Phosphorus and Zinc for sample #0938 are both in agreement with the repeatabilities required in ASTM D5185:2009. Therefore homogeneity of the prepared subsamples #0938 was assumed.

To each of the participating laboratories 2 samples of Lubricating Oil (1\*0.5 L brown glass bottle labelled #0937, 1\*50 mL PE bottle labelled #0938) were sent on April 1<sup>st</sup>, 2009.

## 2.5 ANALYSES

The participants were requested to determine on sample #0937: Acid Number (Total), Base Number (Total), Density @ 15°C, Flash Point PMcc, Kinematic Viscosity @ 40°C and @ 100°C and Water.

On sample #0938 the participants were requested to determine 20 elements (Wear metals: Ag, Al, Ba, Cr, Cu, Fe, Pb, Li, Mg, Mn, Mo, Ni, Na, Si, Sn, Ti and V and the additives Ca, P and Zn).

To get comparable results a detailed report form, on which the units were prescribed as well as some of the required standards, was sent together with each set of samples. Also 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 data are tabulated per determination in the appendix of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder fax was sent to those laboratories that had not reported results at that moment.

Shortly 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 results are used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

### 3.1 STATISTICS

Statistical calculations were performed as described in the report 'i.i.s. Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' (i.i.s.-protocol, version 3.1) of November 2008. 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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.

In accordance to 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, by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test, by

G(0.05) or DG(0.05) for the Grubbs test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

### 3.2 GRAPHICS

In order to visualise 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 results are plotted. The corresponding laboratory numbers are under the X-axis. A straight line presents the average of the reported data. Two striped lines present the reproducibility limits of the selected standard, calculated as mean  $\pm$  target reproducibility, parallel to the average line. 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.12 and 13).

### 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 target standard deviation was calculated from the literature reproducibility by division with 2.8. The z-scores were calculated according to:

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

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

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

$ z  < 1$	good
$1 <  z  < 2$	satisfactory
$2 <  z  < 3$	questionable
$ z  > 3$	unsatisfactory

## 4 EVALUATION

In this interlaboratory study some problems were encountered with the dispatch of the samples to laboratories in Ghana, Jordan and Russia. Eleven participants reported after the final reporting date, and five participants did not report any results at all. Not all laboratories were able to report all analyses requested. In total 79 participants reported 1125 results. Observed were 72 outlying results, which is 6.4% 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. Non-Gaussian distributions were found for the following determinations: Density @ 15°C, Kinematic Viscosity @ 100°C, water, Aluminium and Lead. In these cases 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, which 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.

Acid Number (Total): This determination was problematic for this sample. Four results were outside the reproducibility limits. After rejection of the statistical outliers, the calculated reproducibility is not in agreement with the requirements of ASTM D664:09. When the test results determined by ASTM D664 are evaluated separately, the calculated reproducibility is not changed significantly. Problems due to the dark colour of the used lubrication oil may partly explain the large spread.

Base Number (Total): This determination was problematic for this sample. Five results were outside the reproducibility limits. The calculated reproducibility is, after rejection of the statistical outliers, not in agreement with the requirements of ASTM D2896:07a. Problems due to the dark colour of the used lubrication oil may partly explain the large spread.

Density @ 15°C: This determination was very problematic. Thirteen results were outside the reproducibility limits. After rejection of the statistical outliers, the reproducibility is not at all in agreement with the requirements of ASTM D4052:02e1.

Flash Point PMcc: This determination was problematic. Six results were out the reproducibility limits. The calculated reproducibility is, after rejection of the statistical outliers, not at all in agreement with the requirements of ASTM D93:08 procedure B. When the results for the different modes used (ie, automatic or manual, flame or electrically) were evaluated separately, no significant differences were observed.

Kin.Visco.@ 40°C: This determination was very problematic. Ten results were outside the reproducibility limits. After rejection of the statistical outliers, the calculated reproducibility is not at all in agreement with the requirements of ASTM D445:06. The large spread may be explained by the dark colour of the used lubrication oil colour. Especially the automated viscosity apparatus would be sensitive to this problem. Also the critical temperature of the water bath may be a problem.

Kin.Visco.@ 100°C: This determination was very problematic. Nineteen results were outside the reproducibility limits. After rejection of the statistical outliers the calculated



reproducibility is not at all in agreement with the requirements of ASTM D445:06. The large spread may be explained by the dark colour of the used lubrication oil colour. Especially the automated viscosity apparatus would be sensitive to this problem. Also the critical temperature of the water bath may be a problem.

Water: This determination was problematic. Four results were outside the reproducibility limits. The calculated reproducibility is, after rejection of the statistical outlier, not in agreement with the requirements of ASTM D6304:04ae1. One participant used method ASTM D1744 that was discontinued in 2000. The preferred method to use may be ASTM D6304:04ae1 method C. This method is applicable for oils with difficult matrix interferences (presence of additives). Coulometric titration could be problematic due to the alkalinity of the sample. The high alkalinity may cause a drifting end point, thus yielding an incorrect (too high) water content.

Aluminium: The determination was problematic for one laboratory. Only one result was outside the reproducibility limits and the calculated reproducibility is, after rejection of a statistical outlier, in good agreement with the requirements of ASTM D5185:09.

Barium: This determination was problematic for two laboratories. Two results were outside the reproducibility limits. Although all reported results are above the application range (0.5-4 mg/kg), the calculated reproducibility is, after the rejection of the statistical outliers, in good agreement with the estimated extrapolated requirements of ASTM D5185:09. The calculated reproducibility is in agreement when compared with the strict estimated reproducibility limits of the Horwitz function.

Chromium: This determination was problematic for three laboratories. Three results were outside the reproducibility limits and the calculated reproducibility is, after rejection of the statistical outliers, in good agreement with the requirements of ASTM D5185:09.

Copper: This determination was problematic to three laboratories. Three results were outside the reproducibility limits, and the calculated reproducibility is, after rejection of the statistical outliers, in good agreement with the requirements of ASTM D5185:09.

Iron: This determination was problematic for one laboratory. Only one result was outside the reproducibility limits and the calculated reproducibility is, after rejection of the statistical outlier, in full agreement with the requirements of ASTM D5185:09.

- Lead: This determination was problematic for one laboratory. Only one result was outside the reproducibility limits and the calculated reproducibility is, after rejection of the statistical outlier, in good agreement with the requirements of ASTM D5185:09.
- Lithium: Unfortunately, for this element no test method with precision data was available, therefore the Horwitz equation was used to calculate reproducibility limits.  
This determination was not problematic. No results were outside the estimated reproducibility limits and the calculated reproducibility is in agreement with the strict estimated requirements calculated using the Horwitz equation.
- Magnesium: This determination was problematic. Six results were outside the reproducibility limits and the calculated reproducibility is, after rejection of the statistical outliers, not in agreement with the requirements of ASTM D5185:09.
- Manganese: This determination was problematic for four laboratories. Three results were outside the reproducibility limits, and the calculated reproducibility is, after rejection of the statistical outliers, in good agreement with the requirements of ASTM D5185:09.
- Molybdenum This determination was not problematic. Only two laboratories were outside the reproducibility limits and the calculated reproducibility is, after rejection of the statistical outliers, in full agreement with ASTM D5185:09.
- Nickel: This determination was problematic for three laboratories. Three results were outside the reproducibility limits and the calculated reproducibility is, after rejection of the statistical outliers, in good agreement with the requirements of ASTM D5185:09.
- Sodium: This determination was problematic. Four results were outside the reproducibility limits and the calculated reproducibility is, after rejection of the statistical outliers, not in agreement with the requirements of ASTM D5185:09.
- Silicon: This determination was problematic for one laboratory. Only one result was outside the reproducibility limits and the calculated reproducibility is, after rejection of the statistical outlier, in agreement with the requirements of ASTM D5185:09.
- Silver: This determination was problematic. Four results were outside the reproducibility limits and the calculated reproducibility is, after rejection of the statistical outlier, not in agreement with the requirements of ASTM D5185:09.
- Tin: This determination was not problematic. No results were outside the reproducibility limits and the calculated reproducibility is, after rejection of the

statistical outliers, in good agreement with the requirements of ASTM D5185:09.

- Titanium: This determination was not problematic. No results were outside the reproducibility limits and the calculated reproducibility is in full agreement with the requirements of ASTM D5185:09.
- Vanadium: This determination was problematic for two laboratories. Only two results were outside the reproducibility limits and the calculated reproducibility is, after rejection of the statistical outliers, in agreement with the requirements of ASTM D5185:09.
- Calcium: This determination was problematic. Four results were outside the reproducibility limits, and the calculated reproducibility is, after rejection of the statistical outliers, not in agreement with the requirements of ASTM D5185:09.
- Phosphorus: This determination was problematic. Seven results were outside the reproducibility limits, and the calculated reproducibility is, after rejection of the statistical outliers, not at all in agreement with the requirements of ASTM D5185:09.
- Zinc: This determination was problematic. Four results were outside the reproducibility limits, and the calculated reproducibility is, after rejection of the statistical outliers, not 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 the reproducibility as found for the group of participating laboratories that participated. The average results, calculated reproducibilities and reproducibilities derived from literature standards (in casu ASTM and IP standards), are compared in the next table.

Parameter	Unit	n	Average	2.8 * sd	R (lit)
Acid Number (Total)	mg KOH/g	33	2.34	1.54	1.03
Base Number (Total)	mg KOH/g	36	10.14	0.81	0.71
Density @ 15°C	kg/L	53	0.89583	0.00162	0.00050
Flash Point PMcc	°C	47	206.7	17.65	10.0
Kinematic Viscosity @ 40 °C	mm <sup>2</sup> /s	55	124.782	1.777	0.948
Kinematic Viscosity @ 100°C	mm <sup>2</sup> /s	51	13.4841	0.3435	0.1045
Water	mg/kg	43	1103.03	1792.37	1130.38

Table 5: reproducibilities of results of sample #0937

Parameter	Unit	n	Average	2.8 * sd	R (lit)
Aluminium as Al	mg/kg	41	13.50	5.75	7.48
Barium as Ba	mg/kg	33	41.78	11.62	18.29
Chromium as Cr	mg/kg	45	12.41	2.91	3.76
Copper as Cu	mg/kg	44	52.73	12.21	12.65
Iron as Fe	mg/kg	45	22.46	6.15	6.27
Lead as Pb	mg/kg	45	14.17	5.86	7.79
Lithium as Li	mg/kg	7	42.28	9.84	10.78
Magnesium as Mg	mg/kg	40	59.82	20.60	16.81
Manganese as Mn	mg/kg	36	12.49	3.26	2.69
Molybdenum as Mo	mg/kg	31	15.54	3.45	4.49
Nickel as Ni	mg/kg	37	11.82	2.81	5.16
Sodium as Na	mg/kg	33	17.15	11.11	8.27
Silicon as Si	mg/kg	39	17.41	8.20	8.84
Silver as Ag	mg/kg	34	12.38	6.57	4.33
Tin as Sn	mg/kg	37	11.97	4.01	9.79
Titanium as Ti	mg/kg	35	12.02	4.86	8.04
Vanadium as V	mg/kg	38	11.89	4.35	4.26
Calcium as Ca	mg/kg	41	3085.4	643.6	515.5
Phosphorus as P	mg/kg	36	802.7	210.2	121.8
Zinc as Zn	mg/kg	45	925.1	174.0	152.0

Table 6: reproducibilities of results of sample #0938

Results between brackets were above the application range of the method, therefore the results should be evaluated with care

Without further statistical calculations it can be concluded that for a number of tests there is a not a good compliance of the group of participants with the relevant standards. The tests that are problematic have been discussed in paragraph 4.1.

### 4.3 COMPARISON OF THE PROFICIENCY TEST OF APRIL 2009 WITH PREVIOUS PTS

	April 2009	April 2008	April 2007 *)	April 2006 *)
Number of reporting participants	79	86	71	65
Number of results reported	1125	963	1644	1344
Statistical outliers	74	64	84	104
Percentage outliers	6.6%	6.6%	5.1%	7.7%

Table 7: comparison with previous proficiency tests

\*) both used and unused lubricating oil

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	April 2009	April 2008	April 2007 *)	April 2006 *)
Total Acid Number	-	--	+/-	--
Total Base Number	+/-	--	--	++
Density @ 15 °C	--	--	--	--
Flash Point PMcc	--	+/-	--	--
Kinematic Viscosity @ 40 °C	--	--	--	--
Kinematic Viscosity @ 100 °C	--	--	--	--
Water	--	+	--	--
Metals (20 elements)	+	-	+	+

Table 8: comparison determinations against the standard

\*) both used and unused lubricating oil

The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

- ++: 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.d.: not determined
- n.e.: not evaluated

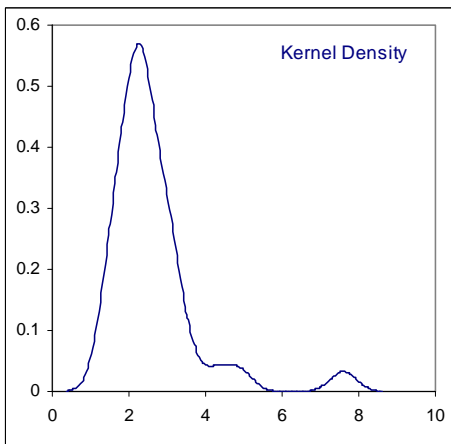
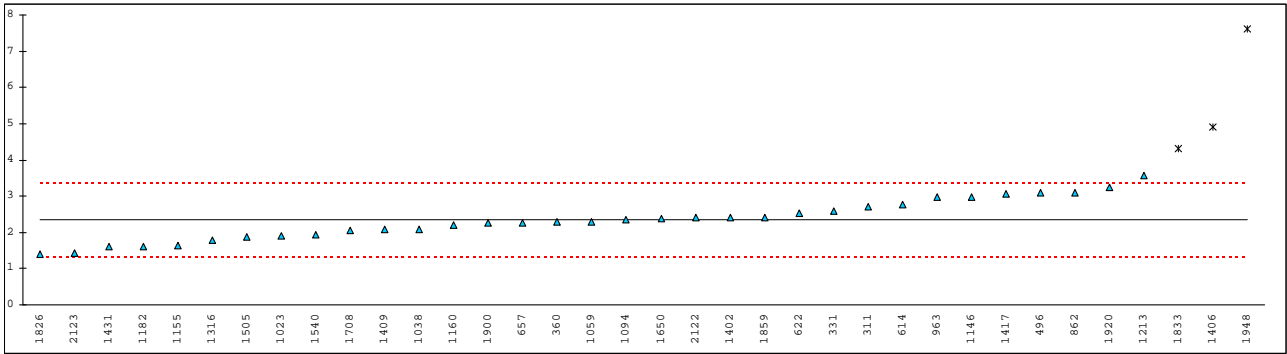
**APPENDIX 1**

Determination of Acid Number (Total) on sample #0937; results in mg KOH/g

lab	method	value	mark	Z(targ)	remarks
233		----		----	
236		----		----	
252		----		----	
254		----		----	
255	D664	<0.05		-6.23	False negative?
311	D664	2.70		0.98	
318		----		----	
331	D664	2.58		0.66	
333		----		----	
343		----		----	
360	D664	2.295		-0.12	
450		----		----	
451		----		----	
473		----		----	
496	D664	3.08		2.02	
562		----		----	
593		----		----	
605		----		----	
614	D664	2.77		1.17	
622	D664	2.52		0.49	
657	D664	2.26		-0.21	
663		----		----	
850		----		----	
862	D664	3.095		2.06	
875		----		----	
912		----		----	
963	D664	2.98		1.75	
994		----		----	
1013		----		----	
1017		----		----	
1023	D664	1.9152		-1.15	
1038	D664	2.095		-0.66	
1047		----		----	
1059	ISO6619	2.30		-0.10	
1094	D664	2.3520		0.04	
1106		----		----	
1128		----		----	
1146	D664	2.986		1.76	
1155	ISO6619	1.628		-1.93	
1160	D664	2.19		-0.40	
1173		----		----	
1182	D664	1.61		-1.98	
1183		----		----	
1184		----		----	
1203		----		----	
1213	D664	3.58		3.38	
1231		----		----	
1316	D664	1.79		-1.49	
1402	D664	2.4		0.17	
1406	D664	4.90	G(0.05)	6.97	
1407		----		----	
1409	D664	2.07		-0.73	
1417	In house	3.05		1.94	
1428		----		----	
1431	D664	1.61		-1.98	
1505	D664	1.863		-1.29	
1526		----		----	
1535		----		----	
1540	D664	1.9195		-1.14	
1613		----		----	
1648		----		----	
1650	D664	2.37		0.09	
1660		----		----	
1708	D664	2.06	C	-0.76	First reported 1.257
1722		----		----	
1730		----		----	
1826	D664	1.39		-2.58	
1827		----		----	
1833	D664	4.3	G(0.05)	5.34	
1842		----		----	
1850		----		----	
1859	D664	2.4070		0.19	
1861		----		----	
1864		----		----	

1900	D664	2.251		-0.24	
1920	D664	3.235		2.44	
1948	D664	7.602	C,G(0.01)	14.32	First reported 3.693
2122	IP177	2.3995		0.17	
2123	D664	1.42		-2.50	

Only D664 results		
normality	OK	OK
n	33	32
outliers	3	3
mean (n)	2.34	2.32
st.dev. (n)	0.550	0.543
R(calc.)	1.54	1.52
R(D664:09)	1.03	1.02

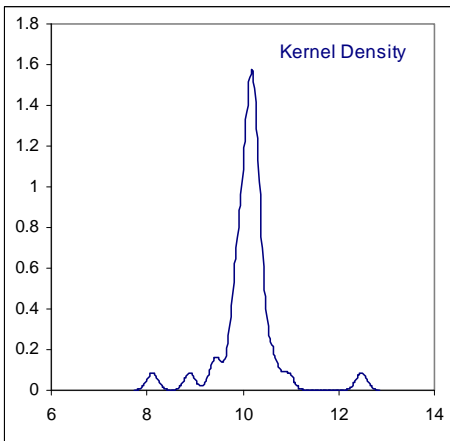
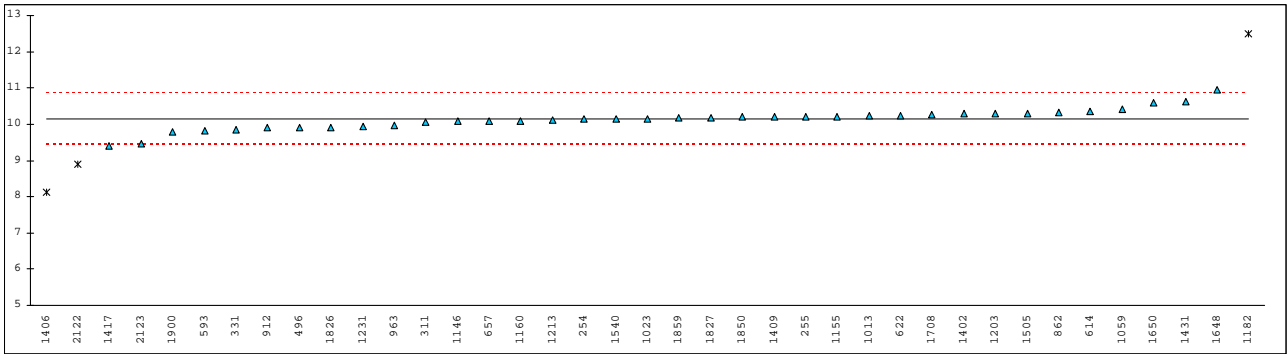


## Determination of Base Number (Total) on sample #0937; results in mg KOH/g

lab	method	value	mark	Z(targ)	remarks
233		----		----	
236		----		----	
252		----		----	
254	D2896	10.14		0.02	
255	D2896	10.2		0.26	
311	D2896	10.06		-0.30	
318		----		----	
331	D2896	9.86		-1.09	
333		----		----	
343		----		----	
360		----		----	
450		----		----	
451		----		----	
473		----		----	
496	D2896	9.90		-0.93	
562		----		----	
593	In house	9.82		-1.24	
605		----		----	
614	D2896	10.36		0.89	
622	D2896	10.24		0.41	
657	D2896	10.08		-0.22	
663		----		----	
850		----		----	
862	D2896	10.309		0.69	
875		----		----	
912	D2896	9.90	C	-0.93	First reported 11.030
963	D2896	9.98	C	-0.61	First reported 8.99
994		----		----	
1013	D2896	10.23		0.37	
1017		----		----	
1023	D2896	10.15		0.06	
1038		----		----	
1047		----		----	
1059	ISO3371	10.4		1.04	
1094		----		----	
1106		----		----	
1128		----		----	
1146	D2896	10.078		-0.23	
1155	ISO3771	10.21		0.29	
1160	D2896	10.1		-0.14	
1173		----		----	
1182	D2896	12.48	G(0.01)	9.25	
1183		----		----	
1184		----		----	
1203	ISO3771	10.3		0.65	
1213	D2896	10.12		-0.06	
1231	D2896	9.94		-0.77	
1316		----		----	
1402	D2896	10.3		0.65	
1406	D2896	8.11	C,G(0.05)	-7.99	First reported 11.02
1407		----		----	
1409	D2896	10.2	C	0.26	First reported 11.69
1417	In house	9.4		-2.90	
1428		----		----	
1431	D2896	10.63		1.95	
1505	D2896	10.3		0.65	
1526		----		----	
1535		----		----	
1540	D2896	10.148		0.05	
1613		----		----	
1648	D2896	10.94		3.18	
1650	D2896	10.59		1.79	
1660		----		----	
1708	D2896	10.26		0.49	
1722		----		----	
1730		----		----	
1826	D2896	9.92		-0.85	
1827	D2896	10.187		0.20	
1833		----		----	
1842		----		----	
1850	ISO3771	10.19		0.22	
1859	D2896	10.1861		0.20	
1861		----		----	
1864		----		----	
1900	D2896	9.775		-1.42	
1920		----		----	



1948	-----	-----		
2122	IP400	8.89	G(0.01)	-4.91
2123	D2896	9.47	C	-2.63
				First reported 9.04
normality	OK			
n	36			
outliers	3			
mean (n)	10.14			
st.dev. (n)	0.288			
R(calc.)	0.81			
R(D2896:07a)	0.71			

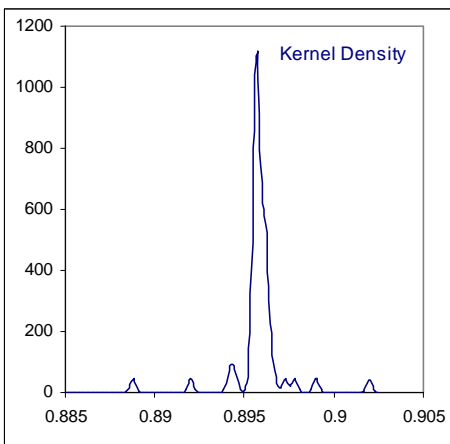
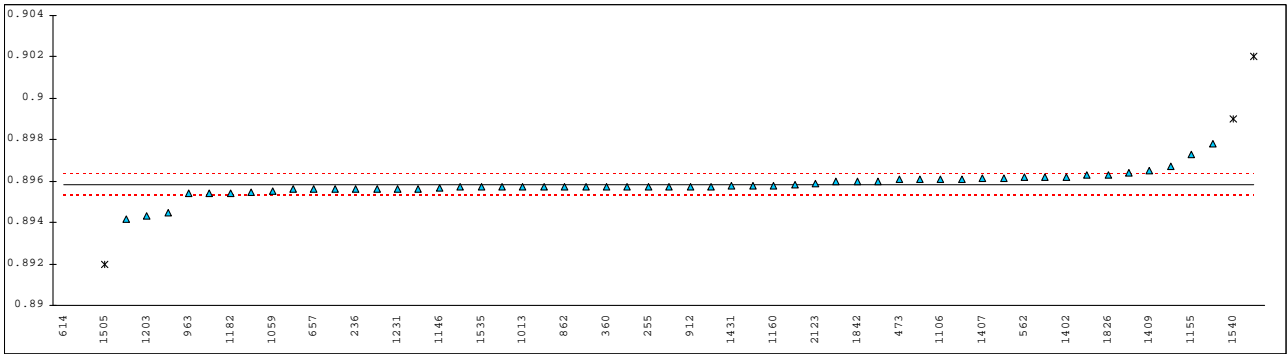


## Determination of Density @ 15°C on sample #0937; results in kg/L

lab	method	value	mark	Z(targ)	remarks
233		-----		-----	
236	D1298	0.8956		-1.27	
252		-----		-----	
254		-----		-----	
255	D1296	0.8957		-0.71	
311	D4052	0.8957		-0.71	
318		-----		-----	
331	D4052	0.89783	C	11.21	First reported 0.89683
333	D4052	0.8958		-0.15	
343	D4052	0.89614		1.75	
360	D4052	0.8957		-0.71	
450	D4052	0.8962		2.09	
451		-----		-----	
473	D4052	0.8961		1.53	
496	D4052	0.89547	C	-2.00	First reported 0.89447
562	D4052	0.8962		2.09	
593	D4052	0.8967		4.89	
605	D4052	0.8957		-0.71	
614	D4052	0.8692	C,G(0.01)	-149.11	First reported 869.2
622	D4052	0.8956		-1.27	
657	D4052	0.8956		-1.27	
663	D4052	0.8956		-1.27	
850	D4052	0.8960		0.97	
862	D4052	0.89570		-0.71	
875		-----		-----	
912	D4052	0.8957		-0.71	
963	D4052	0.8954		-2.39	
994	D4052	0.8957		-0.71	
1013	D4052	0.8957		-0.71	
1017		-----		-----	
1023	D4052	0.8956		-1.27	
1038	D4052	0.8960		0.97	
1047	D4052	0.8961		1.53	
1059	D4052	0.8955		-1.83	
1094	D4052	0.89572		-0.60	
1106	D4052	0.89610		1.53	
1128		-----		-----	
1146	D4052	0.89567		-0.88	
1155	ISO3675	0.8973		8.25	
1160	D4052	0.8958		-0.15	
1173		-----		-----	
1182	D7042	0.8954		-2.39	
1183		-----		-----	
1184		-----		-----	
1203	ISO12185	0.8943		-8.55	
1213		-----		-----	
1231	D4052	0.8956	U	-1.27	Reported in wrong unit (895.6)
1316	D4052	0.8956		-1.27	
1402	IP365	0.8962		2.09	
1406	ISO12185	0.89610	C	1.53	First reported 896.10
1407	ISO12185	0.89614		1.75	
1409	D4052	0.8965		3.77	
1417		-----		-----	
1428		-----	W	-----	Withdrawn, first reported 894.6
1431	D4052	0.8958		-0.15	
1505	D1298	0.892	G(0.01)	-21.43	
1526	In house	0.902	G(0.01)	34.57	
1535	ISO3675	0.8957	C	-0.71	First reported, 0.8943
1540	D4052	0.8990	G(0.01)	17.77	
1613		-----		-----	
1648	D1298	0.89584		0.07	
1650	D4052	0.8957		-0.71	
1660		-----		-----	
1708		-----		-----	
1722	D4052	0.8964		3.21	
1730		-----		-----	
1826	D4052	0.8963		2.65	
1827		-----		-----	
1833	D4052	0.8957		-0.71	
1842	IP365	0.8960		0.97	
1850	D4052	0.8945		-7.43	
1859	D4052	0.8963		2.65	
1861		-----		-----	
1864	ISO12185	0.8954	C	-2.39	First reported 896.2
1900	In house	0.8888	G(0.01)	-39.35	
1920		-----		-----	

1948	D4052	0.8957	C	-0.71	First reported 895.7
2122	In house	0.89415		-9.39	
2123	ISO3838	0.8959		0.41	

normality not OK  
 n 53  
 outliers 5  
 mean (n) 0.89583  
 st.dev. (n) 0.000579  
 R(calc.) 0.00162  
 R(D4052:02e1) 0.00050



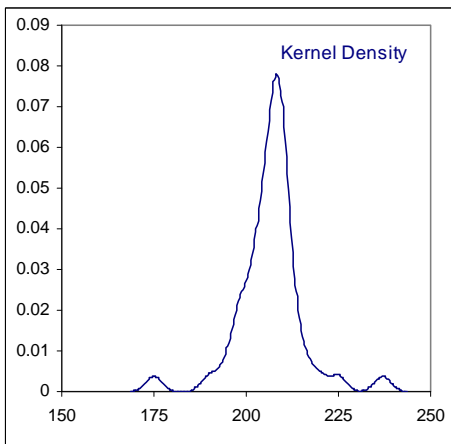
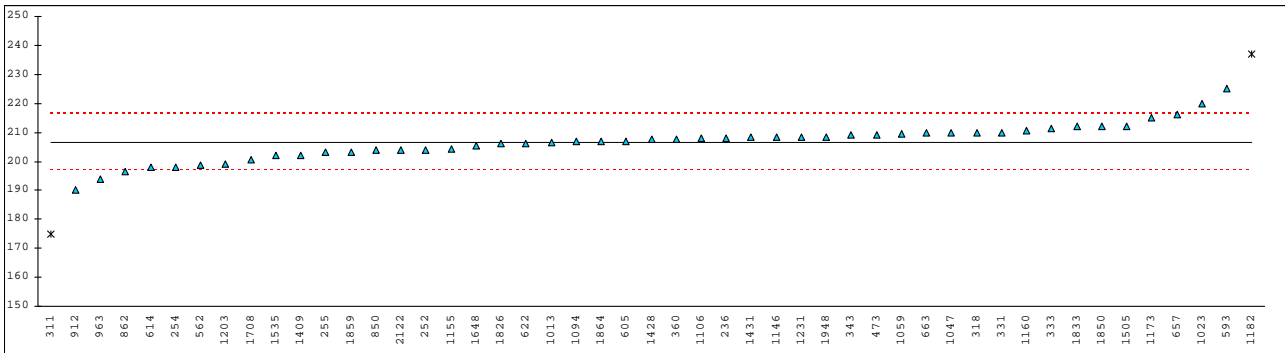
## Determination of Flash Point PMcc on sample #0937; results in °C

lab	method	value	mark	Z(targ)	remarks
233		----		----	
236	D93-MF	208.0		0.38	
252	D93-ME	204.0		-0.74	
254	D93-MF	198.0		-2.42	
255	D93-	203		-1.02	
311	D93-AF	175.0	G(0.01)	-8.86	
318	INH-CMA	210.0		0.94	
331	D93-	210		0.94	
333	D93-AF	211.5		1.36	
343	D93-AE	209		0.66	
360	D93-AE	207.8	C	0.32	First reported 191.8
450		----		----	
451		----		----	
473	D93-AE	209		0.66	
496		----		----	
562	D93-AF	198.75		-2.21	
593	D93-	225		5.14	
605	D3828-MF	207.0		0.10	
614	D93-MF	198		-2.42	
622	D93-MF	206.0		-0.18	
657	D93-MF	216.0		2.62	
663	D93-	210.0		0.94	
850	D93-MF	203.9		-0.77	
862	D93-MF	196.65		-2.80	
875		----		----	
912	D93-AF	190.0	C	-4.66	First reported 186.0
963	D93-AE	194		-3.54	
994		----		----	
1013	D93-	206.5		-0.04	
1017		----		----	
1023	D6450-AE	220		3.74	
1038		----		----	
1047	ISO2719-AE	210		0.94	
1059	ISO2719-AE	209.5		0.80	
1094	D93-	207.0		0.10	
1106	D93-AE	208.0		0.38	
1128		----		----	
1146	D93-AE	208.48		0.51	
1155	ISO2719-MF	204.22		-0.68	
1160	D93-AE	210.5		1.08	
1173	IP34-MF	215.2		2.39	
1182	D92	237	ex	8.50	Deviating method used
1183		----		----	
1184		----		----	
1203	ISO2719-AF	199.0		-2.14	
1213		----		----	
1231	D93-AE	208.5		0.52	
1316		----		----	
1402		----		----	
1406		----		----	
1407		----		----	
1409	D93-AF	202.0		-1.30	
1417		----		----	
1428	ISO2719-AE	207.5		0.24	
1431	D93-AF	208.3		0.46	
1505	D93-MF	212		1.50	
1526	In house	>55		----	
1535	ISO2719-ME	202.0		-1.30	
1540		----		----	
1613		----		----	
1648	D93-	205.5		-0.32	
1650		----		----	
1660		----		----	
1708	D93-AE	200.5		-1.72	
1722		----		----	
1730		----		----	
1826	D93-AE	206		-0.18	
1827		----		----	
1833	D93-	212		1.50	
1842		----		----	
1850	ISO2719	212		1.50	
1859	ISO2719-MF	203		-1.02	
1861		----		----	
1864	D93-	207.0		0.10	
1900		----		----	
1920		----		----	

1948	D93-	208.5	0.52
2122	D93-MF	204	-0.74
2123		----	----

		<u>Only AE results</u>	<u>Only AF results</u>	<u>Only ME results</u>	<u>Only MF results</u>
normality	OK	not OK	OK	not OK	OK
n	47	15	6	3	14
outliers	2	0	1	0	0
mean (n)	206.7	207.9	201.6	203.0	205.9
st.dev. (n)	6.30	5.47	7.65	na	6.06
R(calc.)	17.7	15.3	21.4	na	17.0
R(D93:08)	10.0	10.0	10.0	10.0	10.0

- A = automated mode
- AE = automated mode / electric ignition
- AF = automated mode / flame ignition
- M = manual mode
- ME = manual mode / electric ignition
- MF = manual mode / flame ignition

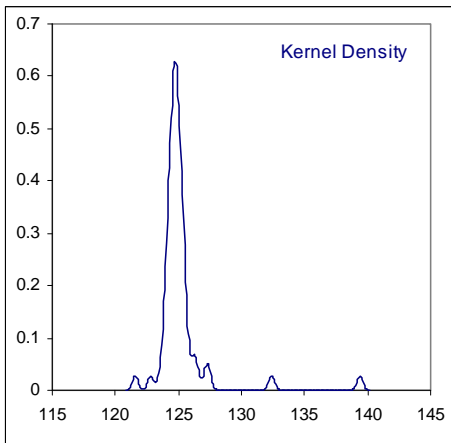
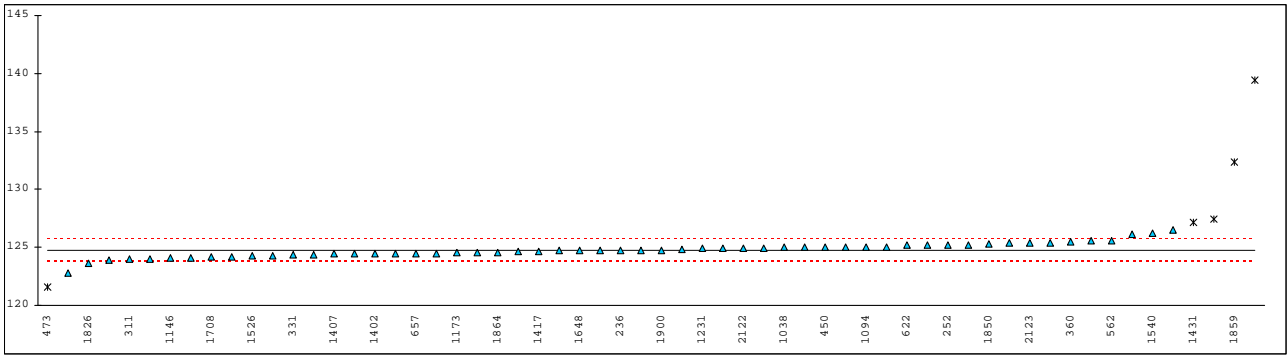


Determination of Kinematic Viscosity @ 40°C on sample #0937; results in mm<sup>2</sup>/s

lab	method	value	mark	Z(targ)	remarks
233		----		----	
236	D445	124.74		-0.12	
252	D445	125.23		1.32	
254		----		----	
255	D445	127.39	G(0.05)	7.70	
311	D445	124.0		-2.31	
318		----		----	
331	HOUILLON	124.39		-1.16	
333	D445	124.5		-0.83	
343	D445	124.76		-0.07	
360	D445	125.49		2.09	
450	D445	125.0		0.64	
451		----		----	
473	D445 mod	121.591	G(0.01)	-9.42	
496	D445	124.4		-1.13	
562	D445	125.599		2.41	
593	D445	124.25		-1.57	
605		----		----	
614	D445	126.5		5.07	
622	D445	125.2		1.23	
657	D445	124.5		-0.83	
663		----		----	
850	D445	124.48		-0.89	
862	D445	125.05		0.79	
875		----		----	
912	D445	122.80		-5.85	
963	D445	125.4		1.82	
994	D445	125.25		1.38	
1013		----		----	
1017		----		----	
1023	D445	124.225		-1.65	
1038	D445	125.00		0.64	
1047	D445	124.0		-2.31	
1059	ISO3104	125.4		1.82	
1094	D445	125.037		0.75	
1106		----		----	
1128		----		----	
1146	D445	124.05		-2.16	
1155	ISO3104	125.567		2.32	
1160	D445	124.1		-2.01	
1173	IP71	124.59		-0.57	
1182	D445	126.09		3.86	
1183		----		----	
1184		----		----	
1203	ISO3104	125.2		1.23	
1213	D445	125.0		0.64	
1231	D445	124.9		0.35	
1316	D445	124.5		-0.83	
1402	D445	124.5		-0.83	
1406	D445	124.63		-0.45	
1407	ISO3104	124.42		-1.07	
1409	D445	124.6		-0.54	
1417	In house	124.69		-0.27	
1428	ISO3104	123.9		-2.60	
1431	D7042	127.2	G(0.05)	7.14	
1505	D445	139.40	G(0.01)	43.16	
1526	In house	124.23		-1.63	
1535	ISO3104	124.733		-0.15	
1540	D445	126.24		4.30	
1613		----		----	
1648	D445	124.72		-0.18	
1650	D445	124.83		0.14	
1660		----		----	
1708	D445	124.18		-1.78	
1722		----		----	
1730		----		----	
1826	D445	123.6		-3.49	
1827	D445	125.013		0.68	
1833	D445	124.7		-0.24	
1842		----		----	
1850	ISO3104	125.3		1.53	
1859	ISO3104	132.3897	C,G(0.01)	22.46	First reported 131.4783
1861		----		----	
1864	ISO3104	124.6		-0.54	
1900	D445	124.78	C	-0.01	First reported 113.83
1920	D445	124.95		0.50	

1948	D445	124.90	0.35
2122	In house	124.91	0.38
2123	D445	125.4	1.82

normality OK  
 n 55  
 outliers 5  
 mean (n) 124.782  
 st.dev. (n) 0.6345  
 R(calc.) 1.777  
 R(D445:06) 0.948



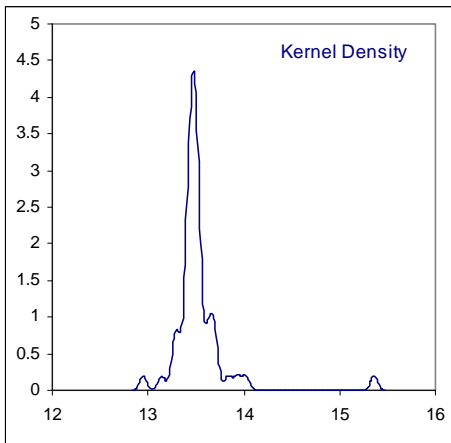
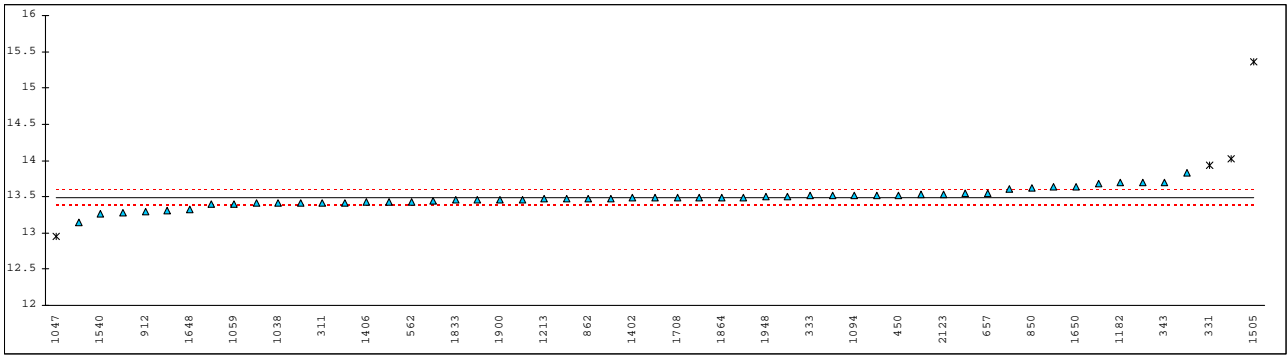
Determination of Kinematic Viscosity @ 100°C on sample #0937; results in mm<sup>2</sup>/s

lab	method	value	mark	Z(targ)	remarks
233		----		----	
236	D445	13.518		0.93	
252	D445	13.412		-1.97	
254		----		----	
255	D445	13.685	C	5.49	First reported 14.95
311	D445	13.42		-1.75	
318		----		----	
331	HOUILLON	13.93	G(0.05)	12.18	
333	D445	13.51		0.71	
343	D445	13.70		5.90	
360		----		----	
450	D445	13.52		0.98	
451		----		----	
473		----		----	
496	D445	13.45		-0.93	
562	D445	13.433		-1.40	
593	D445	13.28		-5.58	
605		----		----	
614	D445	13.835		9.59	
622	D445	13.46		-0.66	
657	D445	13.55		1.80	
663		----		----	
850	D445	13.624		3.82	
862	D445	13.475		-0.25	
875		----		----	
912	D445	13.30		-5.03	
963	D445	13.31		-4.76	
994		----		----	
1013		----		----	
1017		----		----	
1023	D445	13.416		-1.86	
1038	D445	13.416		-1.86	
1047	D445	12.95	G(0.05)	-14.59	
1059	ISO3104	13.40		-2.30	
1094	D445	13.515		0.85	
1106		----		----	
1128		----		----	
1146	D445	13.470		-0.38	
1155	ISO3104	13.479		-0.14	
1160	D445	13.14		-9.40	
1173		----		----	
1182	D445	13.69		5.63	
1183		----		----	
1184		----		----	
1203	ISO3104	13.5		0.44	
1213	D445	13.47		-0.38	
1231	D445	13.53		1.26	
1316	D445	13.42		-1.75	
1402	D445	13.48		-0.11	
1406	D445	13.424		-1.64	
1407	ISO3104	13.492		0.22	
1409	D445	13.43		-1.48	
1417	In house	13.61		3.44	
1428	ISO3104	13.48		-0.11	
1431	D7042	14.02	G(0.05)	14.64	
1505	D445	15.36	G(0.01)	51.26	
1526		----		----	
1535	ISO3104	13.3921		-2.51	
1540	D445	13.258		-6.18	
1613		----		----	
1648	D445	13.326		-4.32	
1650	D445	13.641		4.29	
1660		----		----	
1708	D445	13.480		-0.11	
1722		----		----	
1730		----		----	
1826	D445	13.51		0.71	
1827	D445	13.447		-1.01	
1833	D445	13.45		-0.93	
1842		----		----	
1850	ISO3104	13.48		-0.11	
1859	ISO3104	13.6350		4.12	
1861		----		----	
1864	ISO3104	13.49		0.16	
1900	D445	13.46		-0.66	
1920	D445	13.545		1.66	



1948	D445	13.499	0.41
2122	In house	13.70	5.90
2123	D445	13.53	1.26

normality not OK  
 n 51  
 outliers 4  
 mean (n) 13.4841  
 st.dev. (n) 0.12269  
 R(calc.) 0.3435  
 R(D445:06) 0.1025

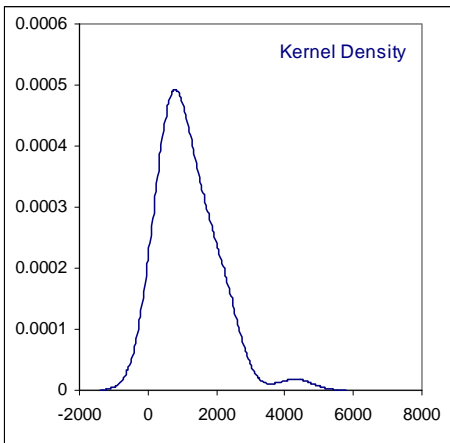
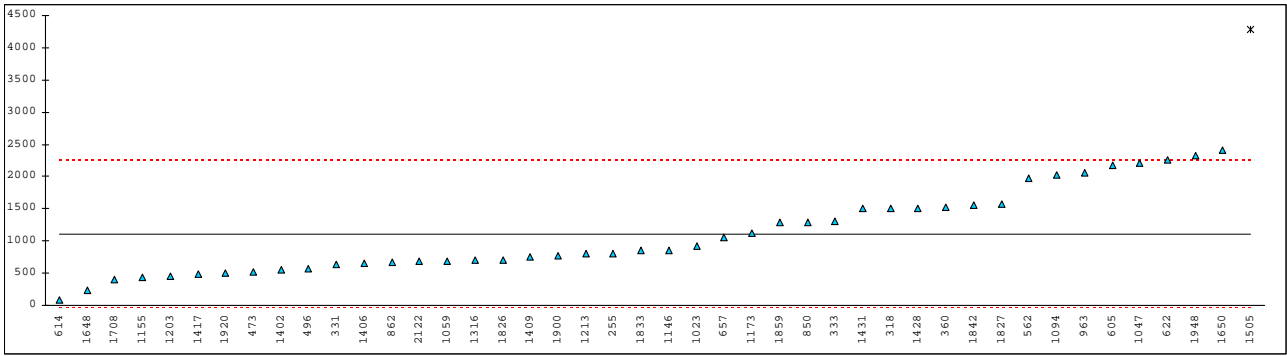


## Determination of Water on sample #0937; results in mg/kg

lab	method	value	mark	Z(targ)	remarks
233		----		----	
236		----		----	
252		----		----	
254		----		----	
255	D95	800		-0.75	
311		----		----	
318	INH-CMA	1500		0.98	
331	D6304	635.13		-1.16	
333	D6304	1310		0.51	
343		----		----	
360	D6304	1528.6		1.05	
450		----		----	
451		----		----	
473	D6304 mod	525.2		-1.43	
496	D6304	573.3		-1.31	
562	In house	1976.7		2.16	
593		----		----	
605	D6304	2180		2.67	
614	D4928	90	C	-2.51	First reported 0.0871
622	D6304	2262		2.87	
657	D6304	1048.8		-0.13	
663		----		----	
850	D6304	1286.0		0.45	
862	D6304	677.5		-1.05	
875		----		----	
912		----		----	
963	D6304	2056		2.36	
994		----		----	
1013		----		----	
1017		----		----	
1023	D6304	914.05		-0.47	
1038		----		----	
1047	ISO12937	2200		2.72	
1059	In house	690		-1.02	
1094	ISO760	2022		2.28	
1106		----		----	
1128		----		----	
1146	D6304	858		-0.61	
1155	D6304	434.2		-1.66	
1160		----		----	
1173	In house	1115		0.03	
1182		----		----	
1183		----		----	
1184		----		----	
1203	ISO12937	460		-1.59	
1213	D6304	800		-0.75	
1231		----		----	
1316	D6304	710		-0.97	
1402	D6304	558		-1.35	
1406	D1744	660		-1.10	
1407		----		----	
1409	D6304	755		-0.86	
1417	In house	482.8		-1.54	
1428	ISO12937	1506		1.00	
1431	D6304	1498		0.98	
1505	D6304	4280	G(0.01)	7.87	
1526	In house	<5000		----	
1535		----		----	
1540		----		----	
1613		----		----	
1648	D95	240		-2.14	
1650	D6304 mod	2413		3.24	
1660		----		----	
1708	D6304	400		-1.74	
1722		----		----	
1730		----		----	
1826	D6304	710		-0.97	
1827	D6304	1575.6		1.17	
1833	D6304	850.22		-0.63	
1842	IP386	1559		1.13	
1850		----		----	
1859	ISO10337	1284.84		0.45	
1861		----		----	
1864		----		----	
1900	D6304-C	771		-0.82	
1920	D6304	510.0		-1.47	

1948	D6304	2317	3.01
2122	KF	687.33	-1.03
2123		-----	-----

normality	not OK
n	43
outliers	1
mean (n)	1103.03
st.dev. (n)	640.131
R(calc.)	1792.37
R(D6304:04ae1)	1130.38

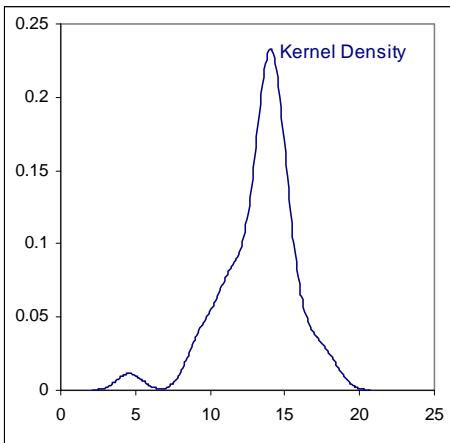
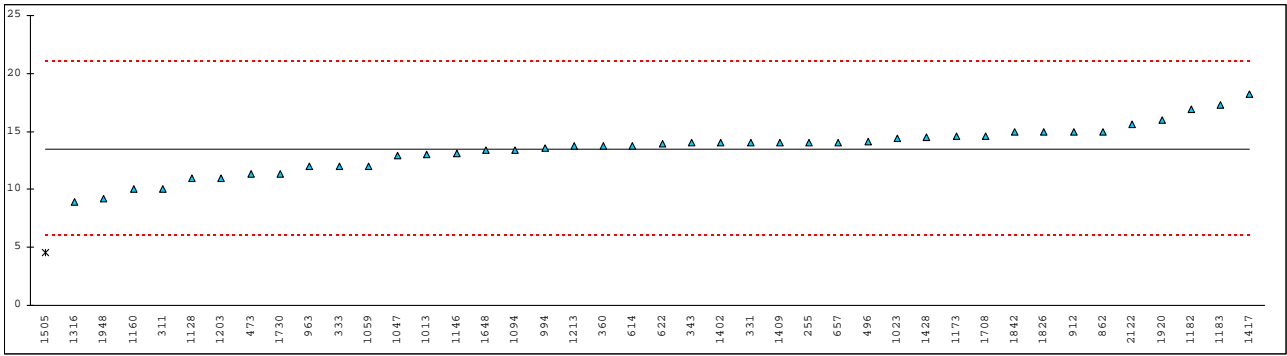


## Determination of Aluminium (Al) on sample #0938; results in mg/kg

lab	method	value	mark	Z(targ)	remarks
233		----		----	
236		----		----	
252		----		----	
254		----		----	
255	AAS	14.00		0.19	
311	D5185	10		-1.31	
318		----		----	
331	D5185	14.0		0.19	
333	D5185	12		-0.56	
343	D5185	14		0.19	
360	D5185	13.8	C	0.11	First reported 4.79
450		----		----	
451		----		----	
473	D5185 mod	11.313		-0.82	
496	D5185	14.16		0.25	
562		----		----	
593		----		----	
605		----		----	
614	D5185	13.8		0.11	
622	D5185	13.93		0.16	
657	D5185	14.016		0.19	
663		----		----	
850		----		----	
862	D5185	15		0.56	
875		----		----	
912	D5185	15		0.56	
963	D5185	11.95		-0.58	
994	D5185	13.55		0.02	
1013	D5185	13		-0.19	
1017		----		----	
1023	D5185	14.36		0.32	
1038		----		----	
1047	D5185	12.94		-0.21	
1059	In house	12		-0.56	
1094	D5185	13.4		-0.04	
1106		----		----	
1128	In house	11		-0.94	
1146	In house	13.10		-0.15	
1155		----		----	
1160	D5185	10		-1.31	
1173	In house	14.6		0.41	
1182	D6595	16.9		1.27	
1183	D6595	17.3		1.42	
1184		----		----	
1203	In house	11		-0.94	
1213	In house	13.78		0.10	
1231		----		----	
1316	D5185	8.9		-1.72	
1402	D5185	14		0.19	
1406		----		----	
1407		----		----	
1409	D5185	14		0.19	
1417	In house	18.217		1.77	
1428	D5185	14.5		0.37	
1431		----		----	
1505	D5185	4.60	G(0.01)	-3.33	
1526		----		----	
1535		----		----	
1540		----		----	
1613		----		----	
1648	D5185	13.40		-0.04	
1650		----		----	
1660		----		----	
1708	D5185	14.6		0.41	
1722		----		----	
1730	D5185	11.314		-0.82	
1826	D5185	15		0.56	
1827		----		----	
1833		----		----	
1842	In house	15		0.56	
1850		----		----	
1859		----		----	
1861		----	W	----	Withdrawn, first reported 36.6
1864		----		----	
1900		----		----	
1920	D5185	16.0		0.94	

1948	D5185	9.155	C	-1.63	First reported 0.329
2122	D5185	15.65		0.80	
2123		-----		-----	

normality	not OK
n	41
outliers	1
mean (n)	13.50
st.dev. (n)	2.055
R(calc.)	5.75
R(D5185:09)	7.48



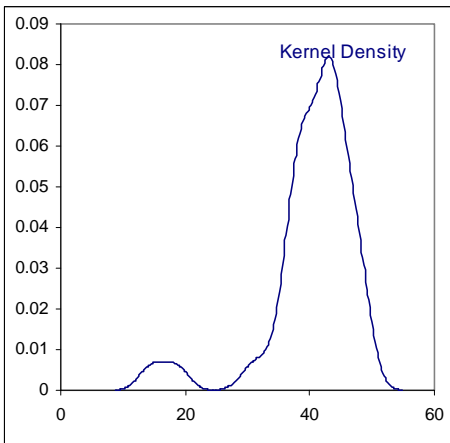
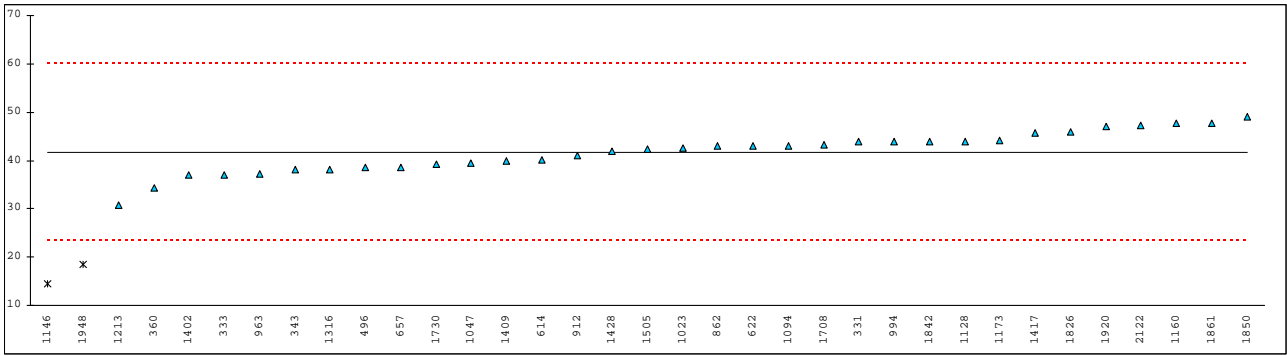
## Determination of Barium (Ba) on sample #0938; results in mg/kg

lab	method	value	mark	Z(targ)	remarks
233		----		----	
236		----		----	
252		----		----	
254		----		----	
255		----		----	
311	D5185	>4		----	
318		----		----	
331	D5185	43.8		0.31	
333	D5185	37		-0.73	
343	D5185	38		-0.58	
360	D5185	34.4		-1.13	
450		----		----	
451		----		----	
473		----		----	
496	D5185	38.49		-0.50	
562		----		----	
593		----		----	
605		----		----	
614	D5185	40.08		-0.26	
622	D5185	43.03		0.19	
657	D5185	38.657		-0.48	
663		----		----	
850		----		----	
862	D5185	43		0.19	
875		----		----	
912	D5185	41		-0.12	
963	D5185	37.28		-0.69	
994	D5185	43.82		0.31	
1013		----		----	
1017		----		----	
1023	D5185	42.52		0.11	
1038		----		----	
1047	D5185	39.40		-0.36	
1059		----		----	
1094	D5185	43.1		0.20	
1106		----		----	
1128	In house	44		0.34	
1146	In house	14.53	G(0.01)	-4.17	
1155		----		----	
1160	D5185	47.6		0.89	
1173	In house	44.2		0.37	
1182		----		----	
1183		----		----	
1184		----		----	
1203		----	W	----	Withdrawn, first reported 11
1213	In house	30.80		-1.68	
1231		----		----	
1316	D5185	38.1		-0.56	
1402	D5185	37		-0.73	
1406		----		----	
1407		----		----	
1409	D5185	40		-0.27	
1417	In house	45.798		0.62	
1428	D5185	41.9		0.02	
1431		----		----	
1505	D5185	42.30		0.08	
1526		----		----	
1535		----		----	
1540		----		----	
1613		----		----	
1648		----		----	
1650		----		----	
1660		----		----	
1708	D5185	43.2		0.22	
1722		----		----	
1730	D5185	39.321		-0.38	
1826	D5185	46		0.65	
1827		----		----	
1833		----		----	
1842	In house	44		0.34	
1850	In house	49		1.11	
1859		----		----	
1861	D5185	47.6		0.89	
1864		----		----	
1900		----		----	
1920	D5185	47.1		0.81	

1948	D5185	18.58	C,G(0.01)	-3.55	First reported 0.319
2122	D5185	47.25		0.84	
2123		-----		-----	

normality	OK
n	33
outliers	2
mean (n)	41.78
st.dev. (n)	4.151
R(calc.)	11.62
R(D5185:09)	18.29

R(Horwitz); 10.7



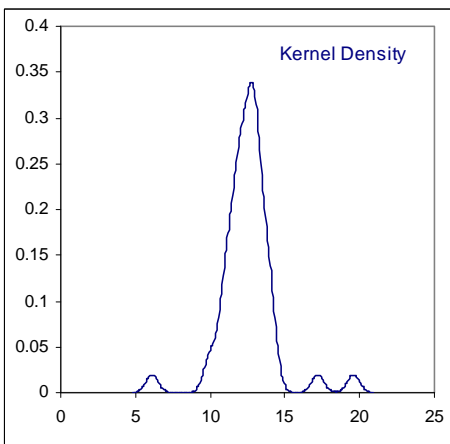
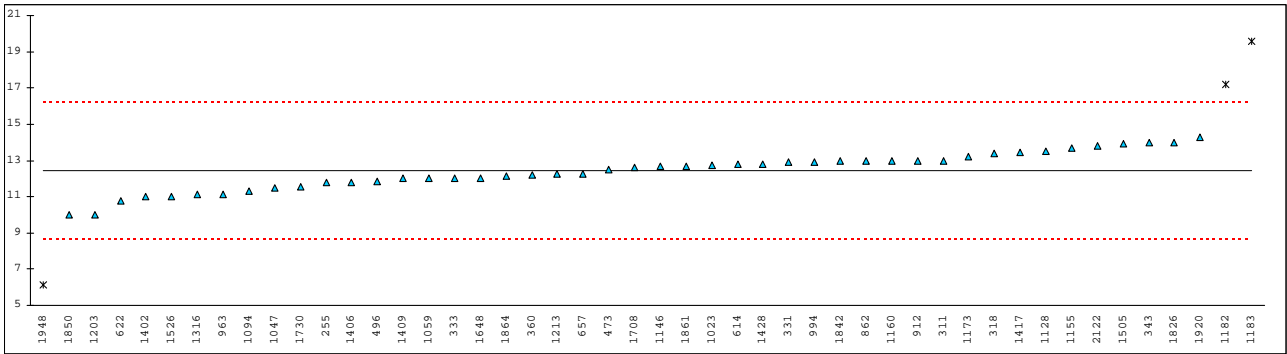
## Determination of Chromium (Cr) on sample #0938; results in mg/kg

lab	method	value	mark	Z(targ)	remarks
233		----		----	
236		----		----	
252		----		----	
254		----		----	
255	AAS	11.76		-0.48	
311	D5185	13		0.44	
318	INH-CMA	13.37		0.72	
331	D5185	12.9		0.37	
333	D5185	12		-0.30	
343	D5185	14		1.18	
360	D5185	12.2	C	-0.15	First reported 5.14
450		----		----	
451		----		----	
473	D5185 mod	12.496		0.07	
496	D5185	11.85		-0.42	
562		----		----	
593		----		----	
605		----		----	
614	D5185	12.8		0.29	
622	D5185	10.77		-1.22	
657	D5185	12.271		-0.10	
663		----		----	
850		----		----	
862	D5185	13		0.44	
875		----		----	
912	D5185	13		0.44	
963	D5185	11.14		-0.94	
994	D5185	12.90		0.37	
1013		----		----	
1017		----		----	
1023	D5185	12.76		0.26	
1038		----		----	
1047	D5185	11.50		-0.68	
1059	In house	12		-0.30	
1094	D5185	11.3		-0.82	
1106		----		----	
1128	In house	13.5		0.81	
1146	In house	12.66		0.19	
1155	DIN51397 mod	13.688		0.95	
1160	D5185	13		0.44	
1173	In house	13.2		0.59	
1182	D6595	17.2	G(0.01)	3.56	
1183	D6595	19.6	G(0.01)	5.35	
1184		----		----	
1203	In house	10		-1.79	
1213	In house	12.25		-0.12	
1231		----		----	
1316	D5185	11.1		-0.97	
1402	D5185	11		-1.05	
1406	D4628	11.8		-0.45	
1407		----		----	
1409	D5185	12		-0.30	
1417	In house	13.423		0.75	
1428	D5185	12.8		0.29	
1431		----		----	
1505	D5185	13.90		1.11	
1526	D5185	11		-1.05	
1535		----		----	
1540		----		----	
1613		----		----	
1648	D5185	12.01		-0.30	
1650		----		----	
1660		----		----	
1708	D5185	12.6		0.14	
1722		----		----	
1730	D5185	11.517		-0.66	
1826	D5185	14		1.18	
1827		----		----	
1833		----		----	
1842	In house	13		0.44	
1850	In house	10		-1.79	
1859		----		----	
1861	D5185	12.7		0.22	
1864	D5185	12.11		-0.22	
1900		----		----	
1920	D5185	14.3		1.41	



1948	D5185	6.115	C,G(0.05)	-4.68	First reported 0.181
2122	D5185	13.8		1.04	
2123		-----		-----	

normality	OK
n	45
outliers	3
mean (n)	12.41
st.dev. (n)	1.038
R(calc.)	2.91
R(D5185:09)	3.76

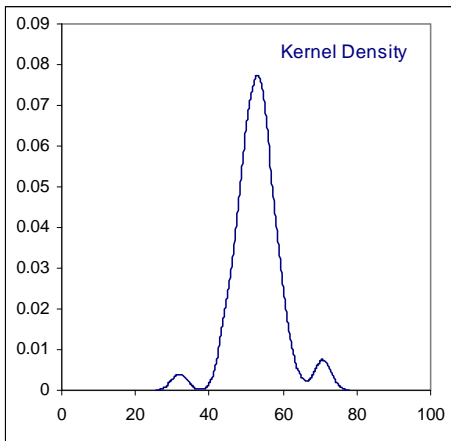
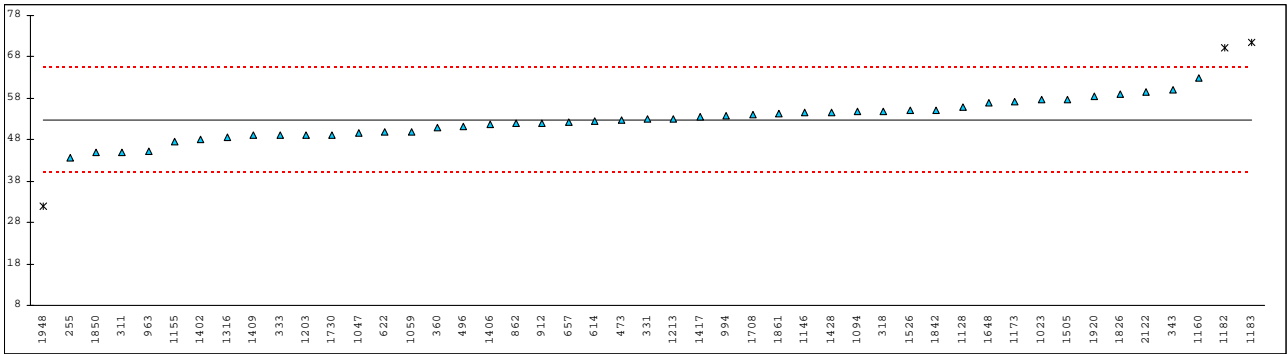


## Determination of Copper (Cu) on sample #0938; results in mg/kg

lab	method	value	mark	Z(targ)	remarks
233		----		----	
236		----		----	
252		----		----	
254		----		----	
255	AAS	43.675		-2.00	
311	D5185	45		-1.71	
318	INH-CMA	54.92		0.49	
331	D5185	52.9		0.04	
333	D5185	49		-0.82	
343	D5185	60		1.61	
360	D5185	50.9		-0.40	
450		----		----	
451		----		----	
473	D5185 mod	52.697		-0.01	
496	D5185	51.13		-0.35	
562		----		----	
593		----		----	
605		----		----	
614	D5185	52.6		-0.03	
622	D5185	49.91		-0.62	
657	D5185	52.353		-0.08	
663		----		----	
850		----		----	
862	D5185	52		-0.16	
875		----		----	
912	D5185	52		-0.16	
963	D5185	45.31		-1.64	
994	D5185	53.82		0.24	
1013		----		----	
1017		----		----	
1023	D5185	57.71		1.10	
1038		----		----	
1047	D5185	49.52		-0.71	
1059	In house	50		-0.60	
1094	D5185	54.8		0.46	
1106		----		----	
1128	In house	56		0.72	
1146	In house	54.55		0.40	
1155	DIN51397 mod	47.568		-1.14	
1160	D5185	63		2.27	
1173	In house	57.1		0.97	
1182	D6595	70.1	G(0.05)	3.84	
1183	D6595	71.5	G(0.05)	4.15	
1184		----		----	
1203	In house	49		-0.82	
1213	In house	52.96		0.05	
1231		----		----	
1316	D5185	48.6		-0.91	
1402	D5185	48		-1.05	
1406	D4628	51.6		-0.25	
1407		----		----	
1409	D5185	49		-0.82	
1417	In house	53.590		0.19	
1428	D5185	54.7		0.44	
1431		----		----	
1505	D5185	57.80		1.12	
1526	D5185	55		0.50	
1535		----		----	
1540		----		----	
1613		----		----	
1648	D5185	56.84		0.91	
1650		----		----	
1660		----		----	
1708	D5185	54.1		0.30	
1722		----		----	
1730	D5185	49.098		-0.80	
1826	D5185	59		1.39	
1827		----		----	
1833		----		----	
1842	In house	55		0.50	
1850	In house	45		-1.71	
1859		----		----	
1861	D5185	54.3		0.35	
1864		----		----	
1900		----		----	
1920	D5185	58.5		1.28	

1948	D5185	31.96	C,G(0.05)	-4.60	First reported 1.21
2122	D5185	59.45		1.49	
2123		-----		-----	

normality	OK
n	44
outliers	3
mean (n)	52.73
st.dev. (n)	4.361
R(calc.)	12.21
R(D5185:09)	12.65

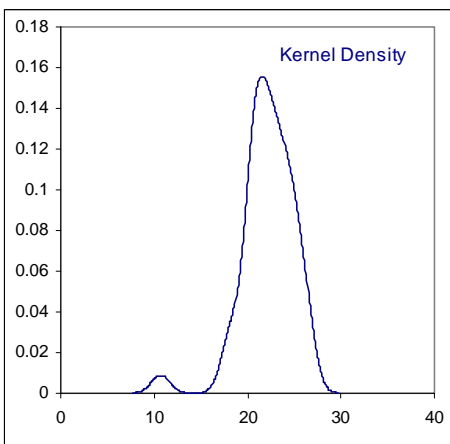
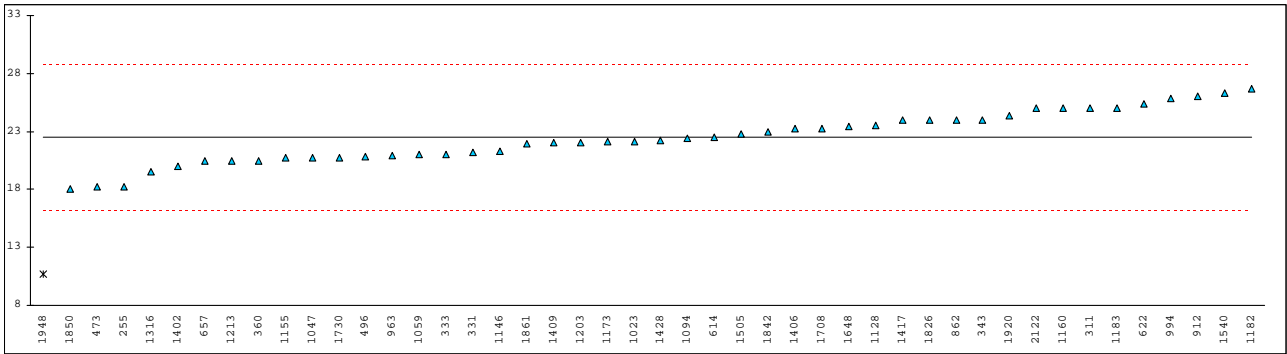


## Determination of Iron (Fe) on sample #0938; results in mg/kg

lab	method	value	mark	Z(targ)	remarks
233		----		----	
236		----		----	
252		----		----	
254		----		----	
255	AAS	18.233		-1.89	
311	D5185	25		1.13	
318		----		----	
331	D5185	21.2		-0.56	
333	D5185	21		-0.65	
343	D5185	24		0.69	
360	D5185	20.5	C	-0.88	First reported 13.9
450		----		----	
451		----		----	
473	D5185 mod	18.212		-1.90	
496	D5185	20.82		-0.73	
562		----		----	
593		----		----	
605		----		----	
614	D5185	22.49		0.01	
622	D5185	25.35		1.29	
657	D5185	20.437		-0.90	
663		----		----	
850		----		----	
862	D5185	24		0.69	
875		----		----	
912	D5185	26		1.58	
963	D5185	20.95		-0.67	
994	D5185	25.84		1.51	
1013		----		----	
1017		----		----	
1023	D5185	22.13		-0.15	
1038		----		----	
1047	D5185	20.75		-0.76	
1059	In house	21		-0.65	
1094	D5185	22.4		-0.03	
1106		----		----	
1128	In house	23.5		0.46	
1146	In house	21.29		-0.52	
1155	DIN51397	20.703		-0.79	
1160	D5185	25		1.13	
1173	In house	22.1		-0.16	
1182	D6595	26.7		1.89	
1183	D6595	25		1.13	
1184		----		----	
1203	In house	22		-0.21	
1213	In house	20.48		-0.88	
1231		----		----	
1316	D5185	19.5		-1.32	
1402	D5185	20		-1.10	
1406	D4628	23.2		0.33	
1407		----		----	
1409	D5185	22		-0.21	
1417	In house	23.990		0.68	
1428	D5185	22.2		-0.12	
1431		----		----	
1505	D5185	22.80		0.15	
1526		----		----	
1535		----		----	
1540	D6481	26.3		1.71	
1613		----		----	
1648	D5185	23.40		0.42	
1650		----		----	
1660		----		----	
1708	D5185	23.2		0.33	
1722		----		----	
1730	D5185	20.773		-0.75	
1826	D5185	24		0.69	
1827		----		----	
1833		----		----	
1842	In house	23		0.24	
1850	In house	18		-1.99	
1859		----		----	
1861	D5185	21.9		-0.25	
1864		----		----	
1900		----		----	
1920	D5185	24.4		0.87	

1948	D5185	10.70	C,G(0.01)	-5.25	First reported 0.176
2122	D5185	25		1.13	
2123		-----		-----	

normality	OK
n	45
outliers	1
mean (n)	22.46
st.dev. (n)	2.196
R(calc.)	6.15
R(D5185:09)	6.27

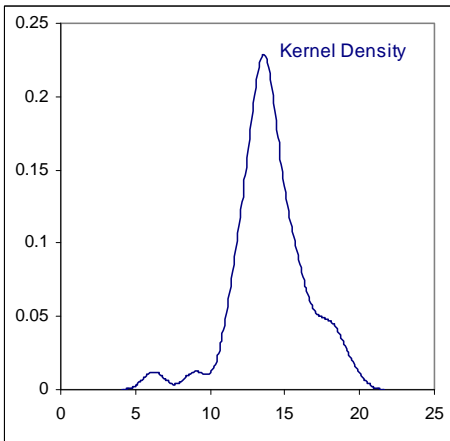
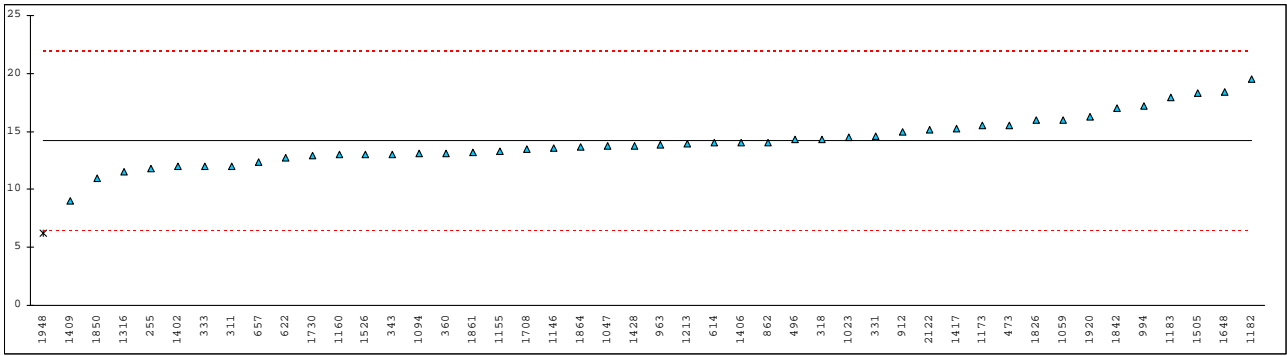


## Determination of Lead (Pb) on sample #0938; results in mg/kg

lab	method	value	mark	Z(targ)	remarks
233		----		----	
236		----		----	
252		----		----	
254		----		----	
255	AAS	11.800		-0.85	
311	D5185	12		-0.78	
318	INH-CMA	14.31		0.05	
331	D5185	14.6		0.15	
333	D5185	12		-0.78	
343	D5185	13		-0.42	
360	D5185	13.1	C	-0.39	First reported 5.56
450		----		----	
451		----		----	
473	D5185 mod	15.523		0.48	
496	D5185	14.27		0.03	
562		----		----	
593		----		----	
605		----		----	
614	D5185	14.0		-0.06	
622	D5185	12.72		-0.52	
657	D5185	12.335		-0.66	
663		----		----	
850		----		----	
862	D5185	14		-0.06	
875		----		----	
912	D5185	15		0.30	
963	D5185	13.86		-0.11	
994	D5185	17.20		1.09	
1013		----		----	
1017		----		----	
1023	D5185	14.52		0.12	
1038		----		----	
1047	D5185	13.77		-0.15	
1059	In house	16		0.66	
1094	D5185	13.1		-0.39	
1106		----		----	
1128		----		----	
1146	In house	13.59		-0.21	
1155	DIN51397 mod	13.297		-0.32	
1160	D5185	13		-0.42	
1173	In house	15.5		0.48	
1182	D6595	19.5		1.91	
1183	D6595	17.9		1.34	
1184		----		----	
1203		----	W	----	First reported 5
1213	In house	13.96		-0.08	
1231		----		----	
1316	D5185	11.5		-0.96	
1402	D5185	12		-0.78	
1406	D4628	14.0		-0.06	
1407		----		----	
1409	D5185	9		-1.86	
1417	In house	15.199		0.37	
1428	D5185	13.8		-0.13	
1431		----		----	
1505	D5185	18.30		1.48	
1526	D5185	13		-0.42	
1535		----		----	
1540		----		----	
1613		----		----	
1648	D5185	18.42		1.53	
1650		----		----	
1660		----		----	
1708	D5185	13.5		-0.24	
1722		----		----	
1730	D5185	12.963		-0.44	
1826	D5185	16		0.66	
1827		----		----	
1833		----		----	
1842	In house	17		1.02	
1850	In house	11		-1.14	
1859		----		----	
1861	D5185	13.2		-0.35	
1864	D5185	13.63		-0.20	
1900		----		----	
1920	D5185	16.3		0.76	

1948	D5185	6.214	C,G(0.05)	-2.86	First reported 0.184
2122	D5185	15.15		0.35	
2123		-----		-----	

normality	not OK
n	45
outliers	1
mean (n)	14.17
st.dev. (n)	2.093
R(calc.)	5.86
R(D5185:09)	7.79



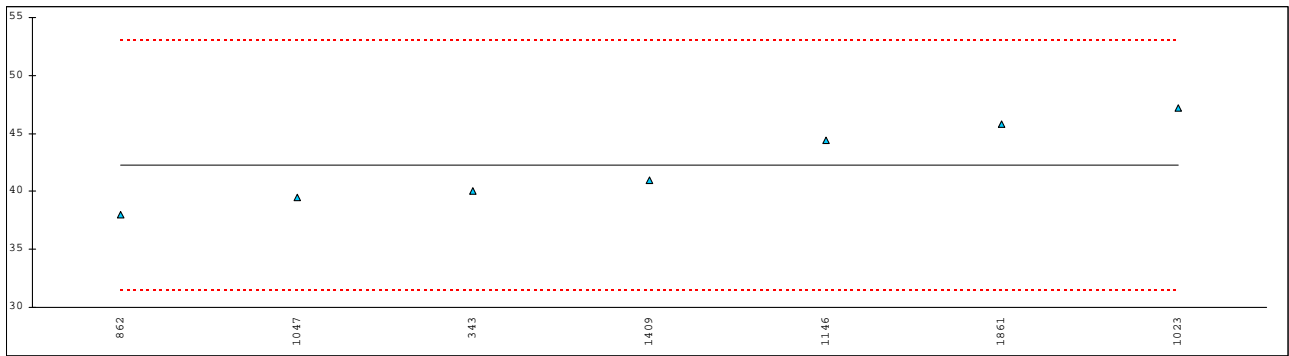
Determination of Lithium (Li) on sample #0938; results in mg/kg

lab	method	value	mark	Z(targ)	remarks
233		----		----	
236		----		----	
252		----		----	
254		----		----	
255		----		----	
311		----		----	
318		----		----	
331		----		----	
333		----		----	
343	D5185 mod	40		-0.59	
360		----		----	
450		----		----	
451		----		----	
473		----		----	
496		----		----	
562		----		----	
593		----		----	
605		----		----	
614		----		----	
622		----		----	
657		----		----	
663		----		----	
850		----		----	
862	D5185	38		-1.11	
875		----		----	
912		----		----	
963		----		----	
994		----		----	
1013		----		----	
1017		----		----	
1023	D5185	47.2		1.28	
1038		----		----	
1047	D5185	39.51		-0.72	
1059		----		----	
1094		----		----	
1106		----		----	
1128		----		----	
1146	In house	44.45		0.56	
1155		----		----	
1160		----		----	
1173		----		----	
1182		----		----	
1183		----		----	
1184		----		----	
1203		----		----	
1213		----		----	
1231		----		----	
1316		----		----	
1402		----		----	
1406		----		----	
1407		----		----	
1409	D5185	41		-0.33	
1417		----		----	
1428		----		----	
1431		----		----	
1505		----		----	
1526		----		----	
1535		----		----	
1540		----		----	
1613		----		----	
1648		----		----	
1650		----		----	
1660		----		----	
1708		----		----	
1722		----		----	
1730		----		----	
1826		----		----	
1827		----		----	
1833		----		----	
1842		----		----	
1850		----		----	
1859		----		----	
1861	D5185	45.8		0.91	
1864		----		----	
1900		----		----	
1920		----		----	



1948 -----  
 2122 -----  
 2123 -----

normality OK  
 n 7  
 outliers 0  
 mean (n) 42.28  
 st.dev. (n) 3.515  
 R(calc.) 9.84  
 R(Horwitz) 10.78

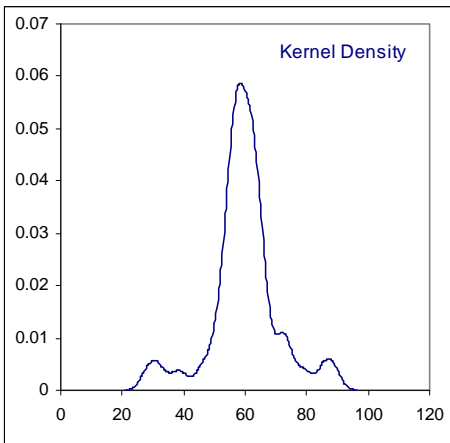
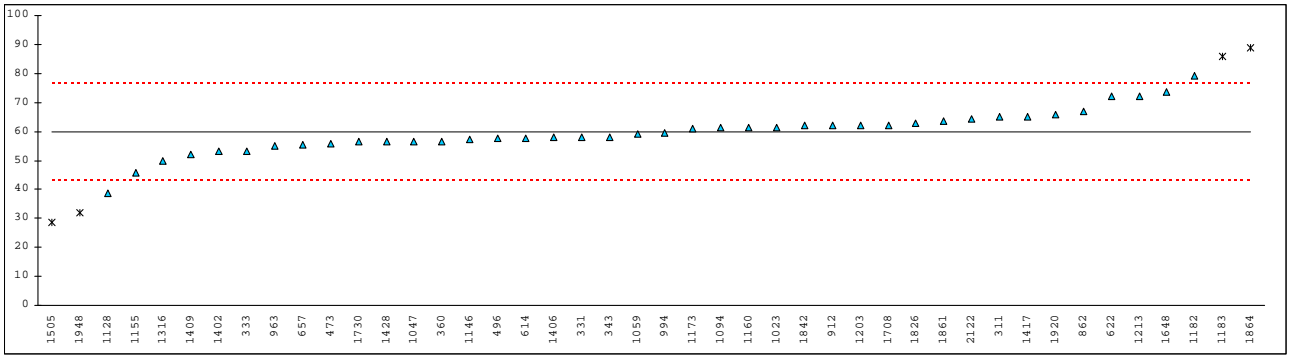


## Determination of Magnesium (Mg) on sample #0938; results in mg/kg

lab	method	value	mark	Z(targ)	remarks
233		----		----	
236		----		----	
252		----		----	
254		----		----	
255		----		----	
311	D5185	65		0.86	
318		----		----	
331	D5185	58.0		-0.30	
333	D5185	53		-1.14	
343	D5185	58		-0.30	
360	D5185	56.65		-0.53	
450		----		----	
451		----		----	
473	D5185 mod	55.863		-0.66	
496	D5185	57.51		-0.38	
562		----		----	
593		----		----	
605		----		----	
614	D5185	57.52		-0.38	
622	D5185	72.01		2.03	
657	D5185	55.292		-0.75	
663		----		----	
850		----		----	
862	D5185	67		1.20	
875		----		----	
912	D5185	62		0.36	
963	D5185	54.94		-0.81	
994	D5185	59.48		-0.06	
1013		----		----	
1017		----		----	
1023	D5185	61.52		0.28	
1038		----		----	
1047	D5185	56.53		-0.55	
1059	In house	59		-0.14	
1094	D5185	61.2		0.23	
1106		----		----	
1128	In house	38.5		-3.55	
1146	In house	57.39		-0.40	
1155	DIN51397 mod	45.861		-2.33	
1160	D5185	61.3		0.25	
1173	In house	61.0		0.20	
1182	D6595	79.1		3.21	
1183	D6595	85.7	DG(0.01)	4.31	
1184		----		----	
1203	In house	62		0.36	
1213	In house	72.10		2.05	
1231		----		----	
1316	D5185	49.8		-1.67	
1402	D5185	53		-1.14	
1406	D4628	57.9		-0.32	
1407		----		----	
1409	D5185	52		-1.30	
1417	In house	65.168		0.89	
1428	D5185	56.5		-0.55	
1431		----		----	
1505	D5185	28.60	DG(0.05)	-5.20	
1526		----		----	
1535		----		----	
1540		----		----	
1613		----		----	
1648	D5185	73.62		2.30	
1650		----		----	
1660		----		----	
1708	D5185	62.1		0.38	
1722		----		----	
1730	D5185	56.331		-0.58	
1826	D4951	63		0.53	
1827		----		----	
1833		----		----	
1842	In house	62		0.36	
1850		----		----	
1859		----		----	
1861	D5185	63.6		0.63	
1864	D5185	88.68	DG(0.01)	4.81	
1900		----		----	
1920	D5185	65.8		1.00	

1948	D5185	32.05	C,DG(0.05)	-4.63	First reported 0.945
2122	D5185	64.2		0.73	
2123		-----		-----	

normality	OK
n	40
outliers	4
mean (n)	59.82
st.dev. (n)	7.358
R(calc.)	20.60
R(D5185:09)	16.81

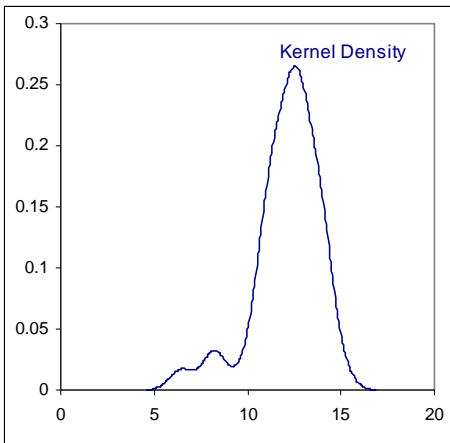
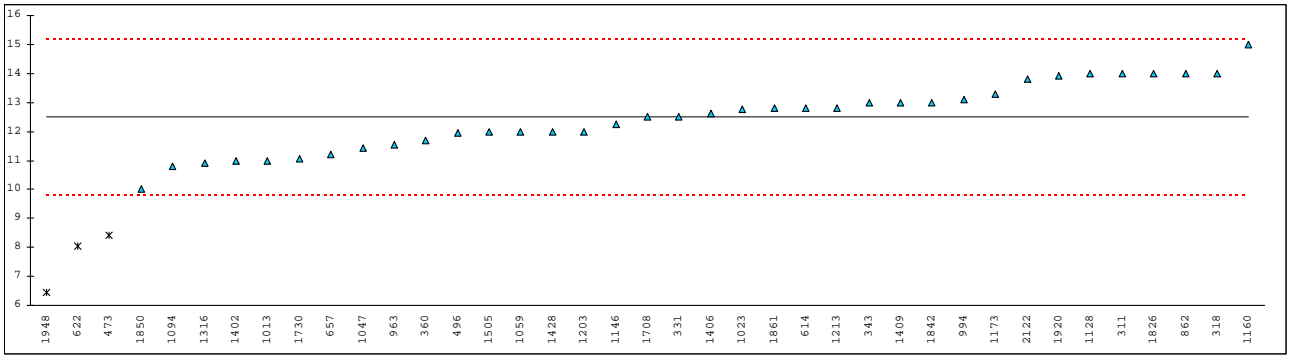


## Determination of Manganese (Mn) on sample #0938; results in mg/kg

lab	method	value	mark	Z(targ)	remarks
233		----		----	
236		----		----	
252		----		----	
254		----		----	
255		----		----	
311	D5185	14		1.57	
318	INH-CMA	14.01		1.58	
331	D5185	12.5		0.01	
333	D5185	<1		-11.96	False negative?
343	D5185	13		0.53	
360	D5185	11.7	C	-0.82	First reported 7.49
450		----		----	
451		----		----	
473	D5185 mod	8.4096	C,DG(0.05)	-4.25	First reported 10.145
496	D5185	11.94		-0.57	
562		----		----	
593		----		----	
605		----		----	
614	D5185	12.8		0.32	
622	D5185	8.06	DG(0.05)	-4.61	
657	D5185	11.202		-1.34	
663		----		----	
850		----		----	
862	D5185	14		1.57	
875		----		----	
912		----		----	
963	D5185	11.55		-0.98	
994	D5185	13.11		0.64	
1013	D5185	11		-1.55	
1017		----		----	
1023	D5185	12.76		0.28	
1038		----		----	
1047	D5185	11.42		-1.11	
1059	In house	12		-0.51	
1094	D5185	10.8		-1.76	
1106		----		----	
1128	In house	14		1.57	
1146	In house	12.23		-0.27	
1155		----		----	
1160	D5185	15		2.61	
1173	In house	13.3		0.84	
1182		----		----	
1183		----		----	
1184		----		----	
1203	In house	12		-0.51	
1213	In house	12.80		0.32	
1231		----		----	
1316	D5185	10.9		-1.66	
1402	D5185	11		-1.55	
1406	D4628	12.6		0.11	
1407		----		----	
1409	D5185	13		0.53	
1417		----		----	
1428	D5185	12.0		-0.51	
1431		----		----	
1505	D5185	12.00		-0.51	
1526		----		----	
1535		----		----	
1540		----		----	
1613		----		----	
1648		----		----	
1650		----		----	
1660		----		----	
1708	D5185	12.5		0.01	
1722		----		----	
1730	D5185	11.063		-1.49	
1826	D5185	14		1.57	
1827		----		----	
1833		----		----	
1842	In house	13		0.53	
1850	In house	10		-2.59	
1859		----		----	
1861	D5185	12.8		0.32	
1864		----		----	
1900		----		----	
1920	D5185	13.9		1.47	

1948 D5185 6.458 C,G(0.05) -6.28 First reported 0.116  
 2122 D5185 13.8 1.36  
 2123 -----

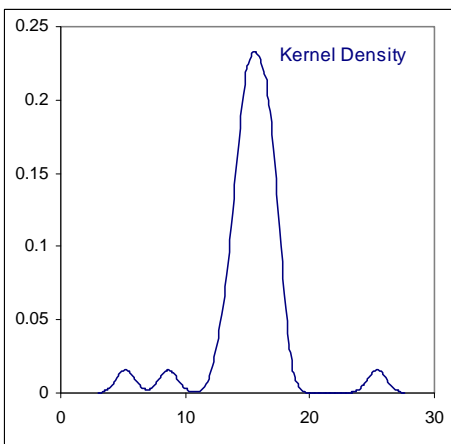
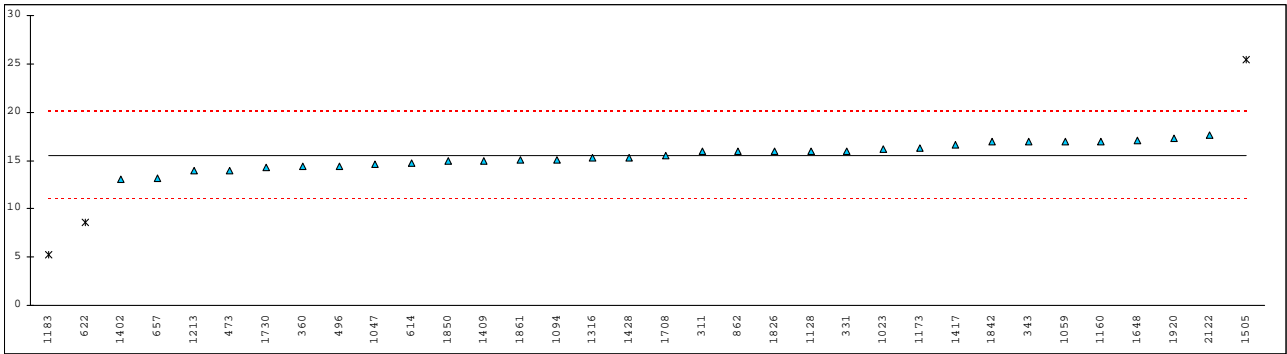
normality OK  
 n 36  
 outliers 3  
 mean (n) 12.49  
 st.dev. (n) 1.163  
 R(calc.) 3.26  
 R(D5185:09) 2.69



Determination of Molybdenum (Mo) on sample #0938; results in mg/kg

lab	method	value	mark	Z(targ)	remarks
233		----		----	
236		----		----	
252		----		----	
254		----		----	
255		----		----	
311	D5185	16		0.28	
318		----		----	
331	D5185	16.0		0.28	
333		----		----	
343	D5185	17		0.91	
360	D5185	14.4	C	-0.71	First reported 4.86
450		----		----	
451		----		----	
473	D5185 mod	13.968		-0.98	
496	D5185	14.44		-0.69	
562		----		----	
593		----		----	
605		----		----	
614	D5185	14.7		-0.53	
622	D5185	8.64	G(0.01)	-4.31	
657	D5185	13.110		-1.52	
663		----		----	
850		----		----	
862	D5185	16		0.28	
875		----		----	
912		----		----	
963		----		----	
994		----		----	
1013		----		----	
1017		----		----	
1023	D5185	16.22		0.42	
1038		----		----	
1047	D5185	14.63		-0.57	
1059	In house	17		0.91	
1094	D5185	15.1		-0.28	
1106		----		----	
1128	In house	16		0.28	
1146		----		----	
1155		----		----	
1160	D5185	17		0.91	
1173	In house	16.3		0.47	
1182		----		----	
1183	D6595	5.2	G(0.01)	-6.45	
1184		----		----	
1203		----		----	
1213	In house	13.92		-1.01	
1231		----		----	
1316	D5185	15.3		-0.15	
1402	D5185	13		-1.59	
1406		----		----	
1407		----		----	
1409	D5185	15		-0.34	
1417	In house	16.578		0.64	
1428	D5185	15.3		-0.15	
1431		----		----	
1505	D5185	25.40	G(0.01)	6.15	
1526		----		----	
1535		----		----	
1540		----		----	
1613		----		----	
1648	D5185	17.09		0.96	
1650		----		----	
1660		----		----	
1708	D5185	15.5		-0.03	
1722		----		----	
1730	D5185	14.327		-0.76	
1826	D5185	16		0.28	
1827		----		----	
1833		----		----	
1842	In house	17		0.91	
1850	In house	15		-0.34	
1859		----		----	
1861	D5185	15.1		-0.28	
1864		----		----	
1900		----		----	
1920	D5185	17.3		1.09	

1948		----	----
2122	D5185	17.6	1.28
2123		----	----
normality		OK	
n		31	
outliers		3	
mean (n)		15.54	
st.dev. (n)		1.232	
R(calc.)		3.45	
R(D5185:09)		4.49	



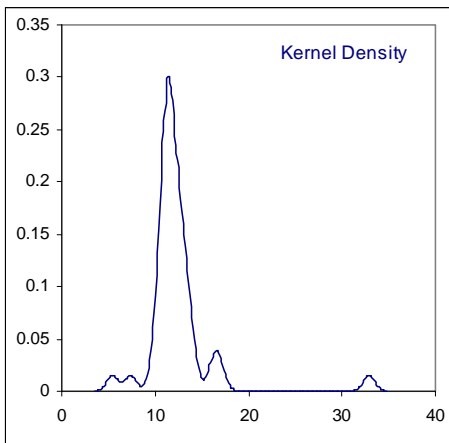
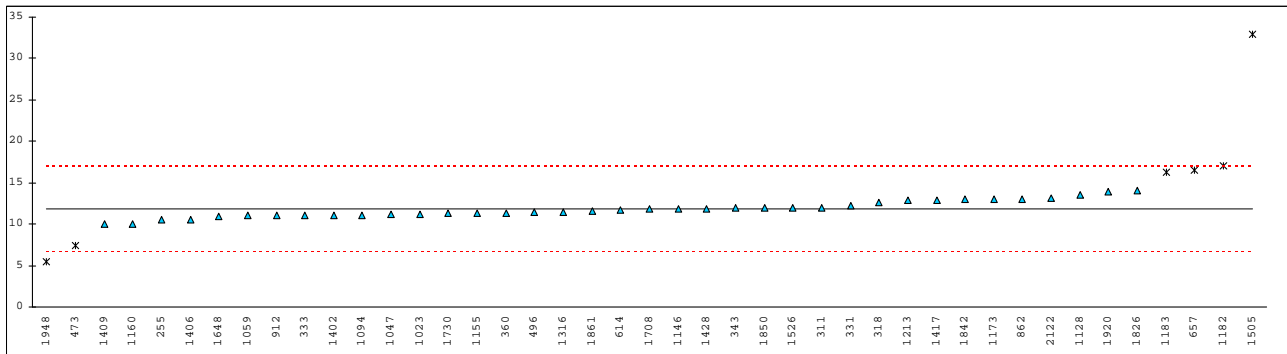
## Determination of Nickel (Ni) on sample #0938; results in mg/kg

lab	method	value	mark	Z(targ)	remarks
233		----		----	
236		----		----	
252		----		----	
254		----		----	
255	AAS	10.60		-0.66	
311	D5185	12		0.10	
318	INH-CMA	12.57		0.41	
331	D5185	12.2		0.21	
333	D5185	11		-0.45	
343	D5185	12		0.10	
360	D5185	11.3	C	-0.28	First reported 2.61
450		----		----	
451		----		----	
473	D5185 mod	7.379	G(0.05)	-2.41	
496	D5185	11.41		-0.22	
562		----		----	
593		----		----	
605		----		----	
614	D5185	11.7		-0.07	
622		----		----	
657	D5185	16.501	DG(0.05)	2.54	
663		----		----	
850		----		----	
862	D5185	13		0.64	
875		----		----	
912	D5185	11		-0.45	
963		----		----	
994		----		----	
1013		----		----	
1017		----		----	
1023	D5185	11.25		-0.31	
1038		----		----	
1047	D5185	11.25		-0.31	
1059	In house	11		-0.45	
1094	D5185	11.1		-0.39	
1106		----		----	
1128	In house	13.5		0.91	
1146	In house	11.85		0.02	
1155	DIN51397 mod	11.287		-0.29	
1160	D5185	10		-0.99	
1173	In house	13.0		0.64	
1182	D6595	17.0	DG(0.05)	2.81	
1183	D6595	16.2	G(0.05)	2.38	
1184		----		----	
1203		----	W	----	Withdrawn, first reported 6
1213	In house	12.84		0.55	
1231		----		----	
1316	D5185	11.5		-0.17	
1402	D5185	11		-0.45	
1406	D4628	10.6		-0.66	
1407		----		----	
1409	D5185	10		-0.99	
1417	In house	12.929		0.60	
1428	D5185	11.9		0.04	
1431		----		----	
1505	D5185	32.90	G(0.01)	11.44	
1526	D5185	12		0.10	
1535		----		----	
1540		----		----	
1613		----		----	
1648	D5185	10.92		-0.49	
1650		----		----	
1660		----		----	
1708	D5185	11.8		-0.01	
1722		----		----	
1730	D5185	11.271		-0.30	
1826	D5185	14		1.18	
1827		----		----	
1833		----		----	
1842	In house	13		0.64	
1850	In house	12		0.10	
1859		----		----	
1861	D5185	11.6		-0.12	
1864		----		----	
1900		----		----	
1920	D5185	13.9		1.13	



1948 D5185 5.433 C,G(0.05) -3.47 First reported 0.223  
 2122 D5185 13.1 0.69  
 2123 -----

normality OK  
 n 37  
 outliers 6  
 mean (n) 11.82  
 st.dev. (n) 1.004  
 R(calc.) 2.81  
 R(D5185:09) 5.16

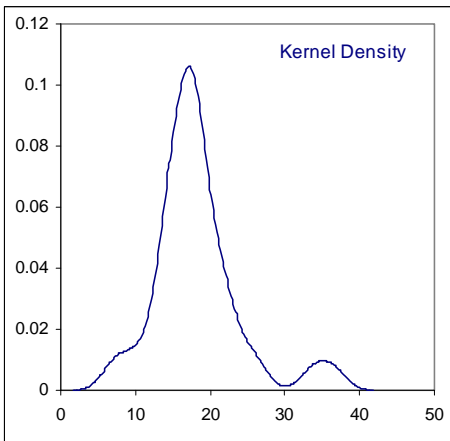
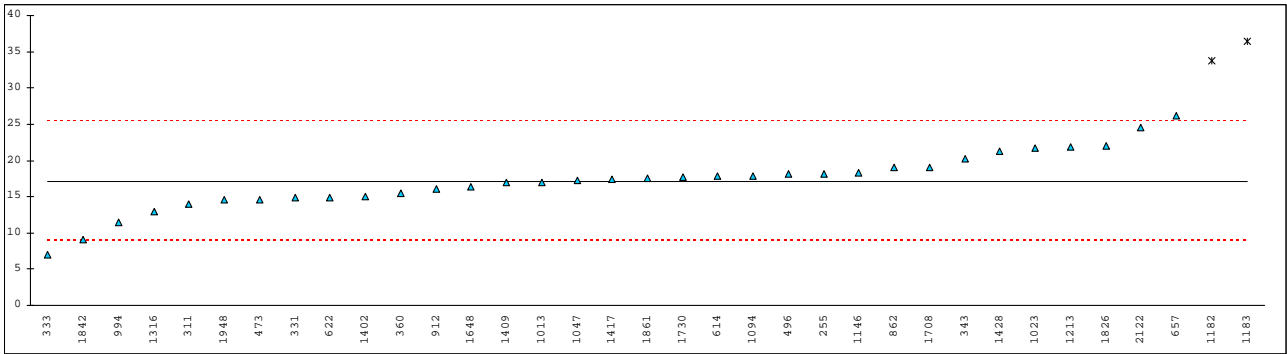


## Determination of Sodium (Na) on sample #0938; results in mg/kg

lab	method	value	mark	Z(targ)	remarks
233		----		----	
236		----		----	
252		----		----	
254		----		----	
255	AAS	18.10		0.32	
311	D5185	14		-1.07	
318		----		----	
331	D5185	14.8		-0.79	
333	D5185	7		-3.43	
343	INH-1555	20.175		1.02	
360	D5185	15.4		-0.59	
450		----		----	
451		----		----	
473	D5185 mod	14.620		-0.86	
496	D5185	18.07		0.31	
562		----		----	
593		----		----	
605		----		----	
614	D5185	17.8		0.22	
622	D5185	14.83		-0.78	
657	D5185	26.244		3.08	
663		----		----	
850		----		----	
862	D5185	19		0.63	
875		----		----	
912	D5185	16		-0.39	
963		----		----	
994	D5185	11.40		-1.95	
1013	D5185	17		-0.05	
1017		----		----	
1023	D5185	21.74		1.55	
1038		----		----	
1047	D5185	17.27		0.04	
1059		----		----	
1094	D5185	17.9		0.25	
1106		----		----	
1128		----		----	
1146	In house	18.29		0.39	
1155		----		----	
1160	D5185	<1		-5.46	False negative?
1173		----		----	
1182	D6595	33.8	G(0.01)	5.64	
1183	D6595	36.5	G(0.05)	6.55	
1184		----		----	
1203		----		----	
1213	In house	21.8	C	1.57	First reported 29.18
1231		----		----	
1316	D5185	13.0		-1.40	
1402	D5185	15		-0.73	
1406		----		----	
1407		----		----	
1409	D5185	17		-0.05	
1417	In house	17.466		0.11	
1428	D5185	21.2		1.37	
1431		----		----	
1505		----		----	
1526		----		----	
1535		----		----	
1540		----		----	
1613		----		----	
1648	D5185	16.34		-0.27	
1650		----		----	
1660		----		----	
1708	D5185	19.1	C	0.66	First reported 26.7
1722		----		----	
1730	D5185	17.632		0.16	
1826	D5185	22		1.64	
1827		----		----	
1833		----		----	
1842	In house	9		-2.76	
1850		----		----	
1859		----		----	
1861	D5185	17.6		0.15	
1864		----		----	
1900		----		----	
1920		----	W	----	Withdrawn, first reported 31.3

1948	D5185	14.58	C	-0.87	First reported 1.45
2122	D5185	24.5		2.49	
2123		-----		-----	

normality	OK
n	33
outliers	2
mean (n)	17.15
st.dev. (n)	3.967
R(calc.)	11.11
R(D5185:09)	8.27

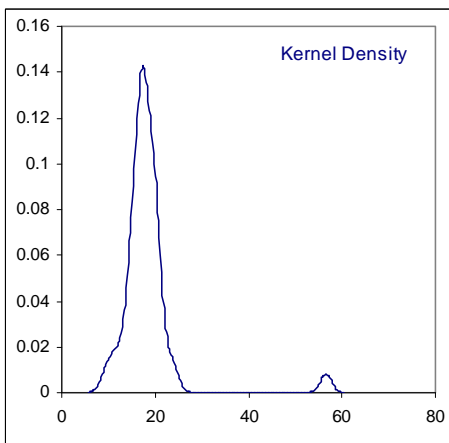
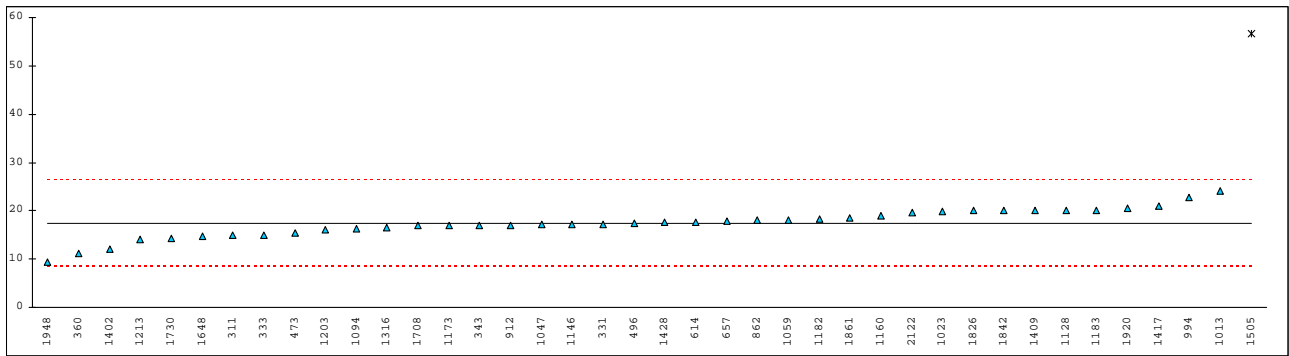


## Determination of Silicon (Si) on sample #0938; results in mg/kg

lab	method	value	mark	Z(targ)	remarks
233		----		----	
236		----		----	
252		----		----	
254		----		----	
255		----		----	
311	D5185	15		-0.76	
318		----		----	
331	D5185	17.2		-0.07	
333	D5185	15		-0.76	
343	D5185	17		-0.13	
360	D5185	11.2		-1.97	
450		----		----	
451		----		----	
473	D5185 mod	15.334		-0.66	
496	D5185	17.39		-0.01	
562		----		----	
593		----		----	
605		----		----	
614	D5185	17.6		0.06	
622		----		----	
657	D5185	17.792		0.12	
663		----		----	
850		----		----	
862	D5185	18		0.19	
875		----		----	
912	D5185	17		-0.13	
963		----		----	
994	D5185	22.75		1.69	
1013	D5185	24		2.09	
1017		----		----	
1023	D5185	19.76		0.75	
1038		----		----	
1047	D5185	17.08		-0.10	
1059	In house	18		0.19	
1094	D5185	16.2		-0.38	
1106		----		----	
1128	In house	20		0.82	
1146	In house	17.18		-0.07	
1155		----		----	
1160	D5185	19	C	0.51	First reported 1.9
1173	In house	16.9		-0.16	
1182	D6595	18.2		0.25	
1183	D6595	20.1		0.85	
1184		----		----	
1203	In house	16		-0.45	
1213	In house	14.01		-1.08	
1231		----		----	
1316	D5185	16.4		-0.32	
1402	D5185	12		-1.71	
1406		----		----	
1407		----		----	
1409	D5185	20	C	0.82	First reported <1
1417	In house	20.979		1.13	
1428	D5185	17.6		0.06	
1431		----		----	
1505	D5185	56.60	G(0.01)	12.42	
1526		----		----	
1535		----		----	
1540		----		----	
1613		----		----	
1648	D5185	14.74		-0.84	
1650		----		----	
1660		----		----	
1708	D5185	16.9		-0.16	
1722		----		----	
1730	D5185	14.292		-0.99	
1826	D5185	20		0.82	
1827		----		----	
1833		----		----	
1842	In house	20		0.82	
1850		----		----	
1859		----		----	
1861	D5185	18.6		0.38	
1864		----		----	
1900		----		----	
1920	D5185	20.5		0.98	

1948	D5185	9.419	C	-2.53	First reported 0.151
2122	D5185	19.7		0.73	
2123		-----		-----	

normality	OK
n	39
outliers	1
mean (n)	17.41
st.dev. (n)	2.929
R(calc.)	8.20
R(D5185:09)	8.84

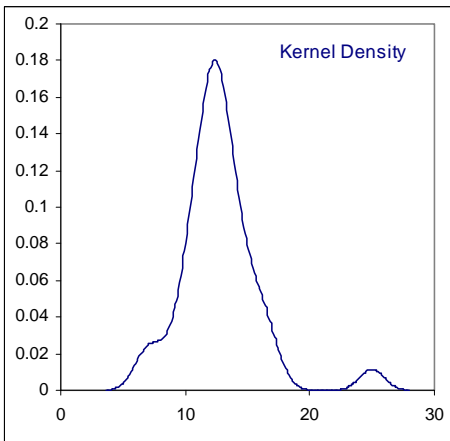
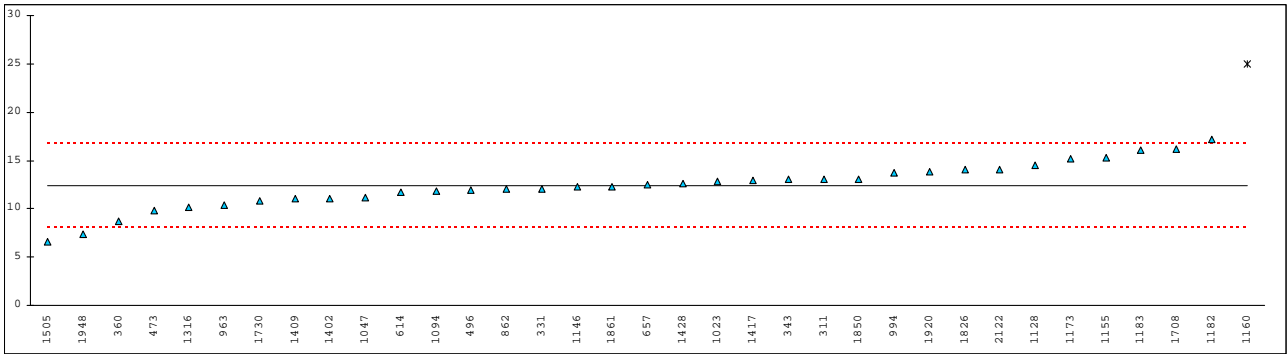


Determination of Silver (Ag) on sample #0938; results in mg/kg

lab	method	value	mark	Z(targ)	remarks
233		----		----	
236		----		----	
252		----		----	
254		----		----	
255		----		----	
311	D5185	13		0.40	
318		----		----	
331	D5185	12.0		-0.24	
333		----		----	
343	D5185	13		0.40	
360	D5185	8.67		-2.40	
450		----		----	
451		----		----	
473	D5185 mod	9.790		-1.67	
496	D5185	11.91		-0.30	
562		----		----	
593		----		----	
605		----		----	
614	D5185	11.7		-0.44	
622		----		----	
657	D5185	12.546		0.11	
663		----		----	
850		----		----	
862	D5185	12		-0.24	
875		----		----	
912		----		----	
963	D5185	10.37		-1.30	
994	D5185	13.74		0.88	
1013		----		----	
1017		----		----	
1023	D5185	12.84		0.30	
1038		----		----	
1047	D5185	11.17		-0.78	
1059		----		----	
1094	D5185	11.8		-0.37	
1106		----		----	
1128	In house	14.5		1.37	
1146	In house	12.24		-0.09	
1155	DIN51397 mod	15.3085		1.89	
1160	D5185	25	G(0.01)	8.16	
1173	In house	15.2		1.82	
1182	D6595	17.2		3.12	
1183	D6595	16.1		2.41	
1184		----		----	
1203		----		----	
1213		----		----	
1231		----		----	
1316	D5185	10.1		-1.47	
1402	D5185	11		-0.89	
1406		----		----	
1407		----		----	
1409	D5185	11		-0.89	
1417	In house	12.925		0.35	
1428	D5185	12.6		0.14	
1431		----		----	
1505	D5185	6.60		-3.73	
1526		----		----	
1535		----		----	
1540		----		----	
1613		----		----	
1648		----		----	
1650		----		----	
1660		----		----	
1708	D5185	16.2	C	2.47	First reported 46.5
1722		----		----	
1730	D5185	10.786		-1.03	
1826	D5185	14		1.05	
1827		----		----	
1833		----		----	
1842		----		----	
1850	In house	13		0.40	
1859		----		----	
1861	D5185	12.3		-0.05	
1864		----		----	
1900		----		----	
1920	D5185	13.8		0.92	

1948	D5185	7.336	C	-3.26	First reported 0.179
2122	D5185	14.1		1.11	
2123		-----		-----	

normality	OK
n	34
outliers	1
mean (n)	12.38
st.dev. (n)	2.346
R(calc.)	6.57
R(D5185:09)	4.33



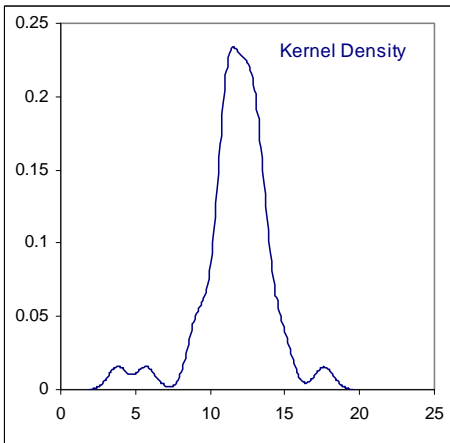
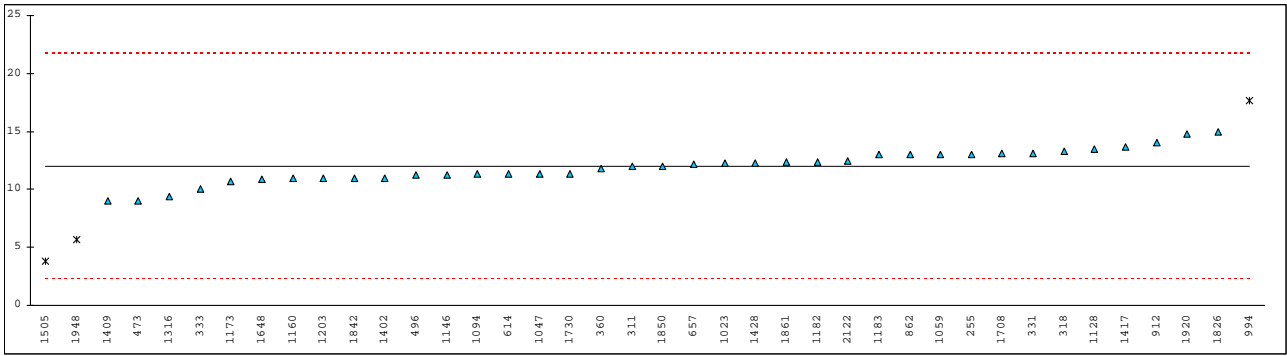
## Determination of Tin (Sn) on sample #0938; results in mg/kg

lab	method	value	mark	Z(targ)	remarks
233		----		----	
236		----		----	
252		----		----	
254		----		----	
255	AAS	13.00		0.29	
311	D5185	12		0.01	
318	INH-CMA	13.29		0.38	
331	D5185	13.1		0.32	
333	D5185	10		-0.56	
343	D5185	<10		----	
360	D5185	11.8	C	-0.05	First reported 2.78
450		----		----	
451		----		----	
473	D5185 mod	9.001		-0.85	
496	D5185	11.21		-0.22	
562		----		----	
593		----		----	
605		----		----	
614	D5185	11.3		-0.19	
622		----		----	
657	D5185	12.219		0.07	
663		----		----	
850		----		----	
862	D5185	13		0.29	
875		----		----	
912	D5185	14		0.58	
963		----		----	
994	D5185	17.63	G(0.05)	1.62	
1013		----		----	
1017		----		----	
1023	D5185	12.24		0.08	
1038		----		----	
1047	D5185	11.31		-0.19	
1059	In house	13		0.29	
1094	D5185	11.3		-0.19	
1106		----		----	
1128	In house	13.5		0.44	
1146	In house	11.25		-0.21	
1155		----		----	
1160	D5185	11		-0.28	
1173	In house	10.7		-0.36	
1182	D6595	12.4		0.12	
1183	D6595	13		0.29	
1184		----		----	
1203	In house	11		-0.28	
1213		----		----	
1231		----		----	
1316	D5185	9.4		-0.74	
1402	D5185	11		-0.28	
1406		----		----	
1407		----		----	
1409	D5185	9		-0.85	
1417	In house	13.705		0.50	
1428	D5185	12.3		0.09	
1431		----		----	
1505	D5185	3.80	G(0.01)	-2.34	
1526		----		----	
1535		----		----	
1540		----		----	
1613		----		----	
1648	D5185	10.84		-0.32	
1650		----		----	
1660		----		----	
1708	D5185	13.1		0.32	
1722		----		----	
1730	D5185	11.335		-0.18	
1826	D5185	15		0.87	
1827		----		----	
1833		----		----	
1842	In house	11		-0.28	
1850	In house	12		0.01	
1859		----		----	
1861	D5185	12.4		0.12	
1864		----		----	
1900		----		----	
1920	D5185	14.8		0.81	



1948 D5185 5.707 C,G(0.05) -1.79 First reported 1.41  
 2122 D5185 12.5 0.15  
 2123 -----

normality OK  
 n 37  
 outliers 3  
 mean (n) 11.97  
 st.dev. (n) 1.431  
 R(calc.) 4.01  
 R(D5185:09) 9.79

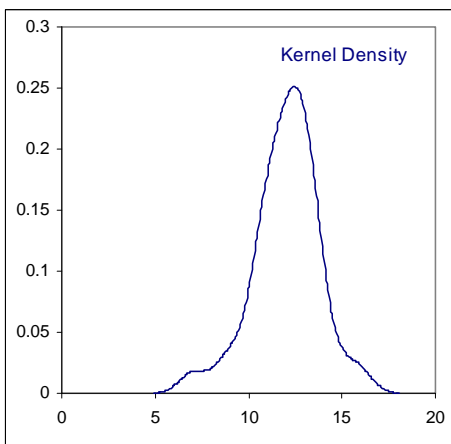
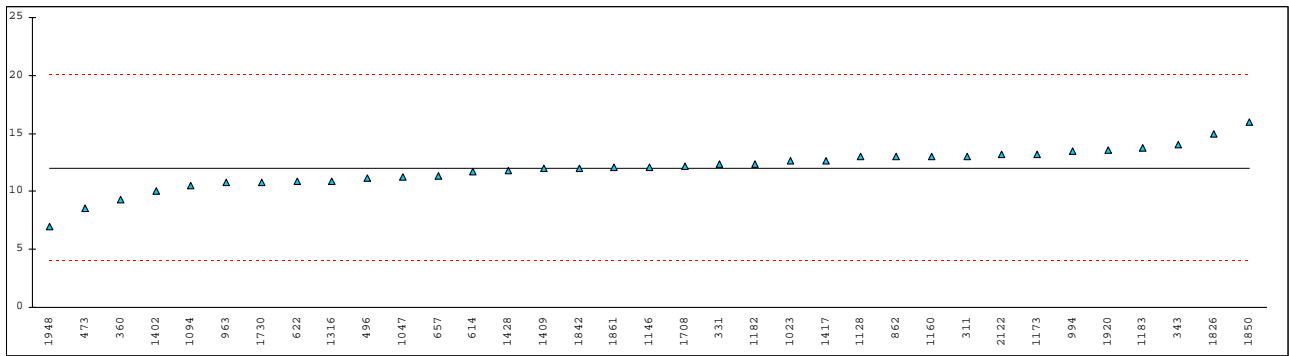


Determination of Titanium (Ti) on sample #0938; results in mg/kg

lab	method	value	mark	Z(targ)	remarks
233		----		----	
236		----		----	
252		----		----	
254		----		----	
255		----		----	
311	D5185	13		0.34	
318		----		----	
331	D5185	12.4		0.13	
333		----		----	
343	D5185	14		0.69	
360	D5185	9.30		-0.95	
450		----		----	
451		----		----	
473	D5185 mod	8.566		-1.20	
496	D5185	11.19		-0.29	
562		----		----	
593		----		----	
605		----		----	
614	D5185	11.7		-0.11	
622	D5185	10.83		-0.41	
657	D5185	11.334		-0.24	
663		----		----	
850		----		----	
862	D5185	13		0.34	
875		----		----	
912		----		----	
963	D5185	10.80		-0.43	
994	D5185	13.47		0.50	
1013		----		----	
1017		----		----	
1023	D5185	12.64		0.22	
1038		----		----	
1047	D5185	11.25		-0.27	
1059		----		----	
1094	D5185	10.5		-0.53	
1106		----		----	
1128	In house	13		0.34	
1146	In house	12.11		0.03	
1155		----		----	
1160	D5185	13		0.34	
1173	In house	13.2		0.41	
1182	D6595	12.4		0.13	
1183	D6595	13.8		0.62	
1184		----		----	
1203		----		----	
1213		----		----	
1231		----		----	
1316	D5185	10.9		-0.39	
1402	D5185	10		-0.70	
1406		----		----	
1407		----		----	
1409	D5185	12		-0.01	
1417	In house	12.648		0.22	
1428	D5185	11.8		-0.08	
1431		----		----	
1505		----		----	
1526		----		----	
1535		----		----	
1540		----		----	
1613		----		----	
1648		----		----	
1650		----		----	
1660		----		----	
1708	D5185	12.2		0.06	
1722		----		----	
1730	D5185	10.818		-0.42	
1826	D5185	15		1.04	
1827		----		----	
1833		----		----	
1842	In house	12		-0.01	
1850	In house	16		1.38	
1859		----		----	
1861	D5185	12.1		0.03	
1864		----		----	
1900		----		----	
1920	D5185	13.6		0.55	

1948	D5185	6.998	C	-1.75	First reported 0.074
2122	D5185	13.2		0.41	
2123		-----		-----	

normality	OK
n	35
outliers	0
mean (n)	12.02
st.dev. (n)	1.737
R(calc.)	4.86
R(D5185:09)	8.04

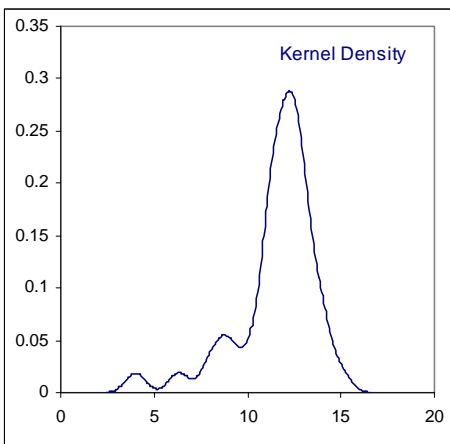
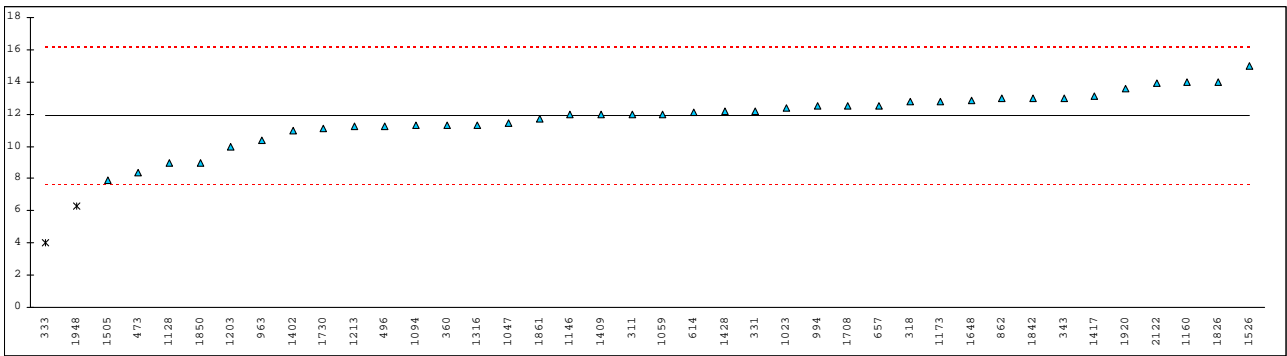


## Determination of Vanadium (V) on sample #0938; results in mg/kg

lab	method	value	mark	Z(targ)	remarks
233		----		----	
236		----		----	
252		----		----	
254		----		----	
255		----		----	
311	D5185	12		0.07	
318	INH-CMA	12.79		0.59	
331	D5185	12.2		0.21	
333	D5185	4	G(0.01)	-5.18	
343	D5185	13		0.73	
360	D5185	11.3	C	-0.39	First reported 5.58
450		----		----	
451		----		----	
473	D5185 mod	8.382		-2.30	
496	D5185	11.27		-0.41	
562		----		----	
593		----		----	
605		----		----	
614	D5185	12.1		0.14	
622		----		----	
657	D5185	12.504		0.41	
663		----		----	
850		----		----	
862	D5185	13		0.73	
875		----		----	
912		----		----	
963	D5185	10.38		-0.99	
994	D5185	12.49		0.40	
1013		----		----	
1017		----		----	
1023	D5185	12.39		0.33	
1038		----		----	
1047	D5185	11.46		-0.28	
1059	In house	12		0.07	
1094	D5185	11.3		-0.39	
1106		----		----	
1128	In house	9		-1.90	
1146	In house	11.97		0.05	
1155		----		----	
1160	D5185	14		1.39	
1173	In house	12.8		0.60	
1182		----		----	
1183		----		----	
1184		----		----	
1203	In house	10		-1.24	
1213	In house	11.21		-0.44	
1231		----		----	
1316	D5185	11.3		-0.39	
1402	D5185	11		-0.58	
1406		----		----	
1407		----		----	
1409	D5185	12		0.07	
1417	In house	13.120		0.81	
1428	D5185	12.2		0.21	
1431		----		----	
1505	D5185	7.90		-2.62	
1526	D5185	15		2.04	
1535		----		----	
1540		----		----	
1613		----		----	
1648	D5185	12.82		0.61	
1650		----		----	
1660		----		----	
1708	D5185	12.5		0.40	
1722		----		----	
1730	D5185	11.132		-0.50	
1826	D5185	14		1.39	
1827		----		----	
1833		----		----	
1842	In house	13		0.73	
1850	In house	9		-1.90	
1859		----		----	
1861	D5185	11.7		-0.12	
1864		----		----	
1900		----		----	
1920	D5185	13.6		1.12	

1948	D5185	6.299	C,G(0.05)	-3.67	First reported 0.096
2122	D5185	13.9		1.32	
2123		-----		-----	

normality	OK
n	38
outliers	2
mean (n)	11.89
st.dev. (n)	1.552
R(calc.)	4.35
R(D5185:09)	4.26

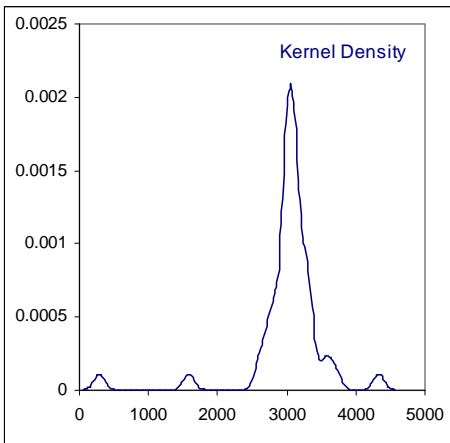
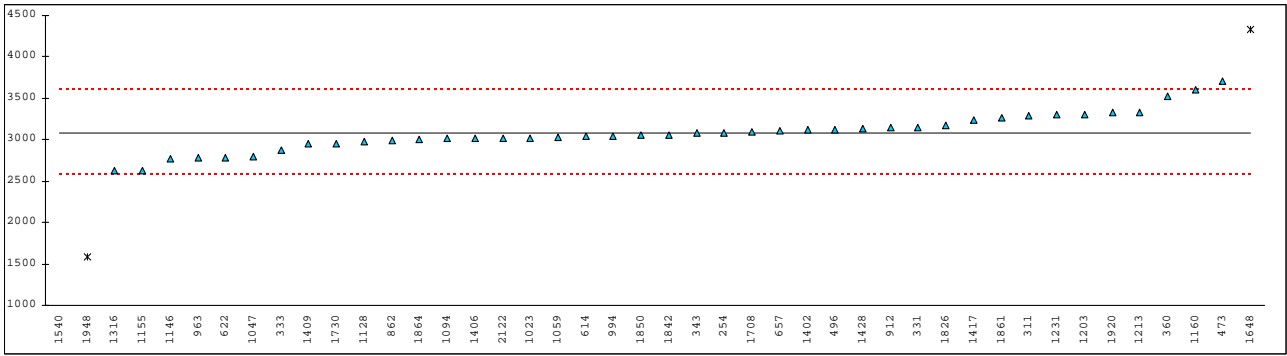


## Determination of Calcium (Ca) on sample #0938; results in mg/kg

lab	method	value	mark	Z(targ)	remarks
233		----		----	
236		----		----	
252		----		----	
254	IP308	3082.07		-0.02	
255		----		----	
311	D5185	3294		1.13	
318		----		----	
331	D5185	3152.7		0.37	
333	D5185	2880		-1.12	
343	D5185	3079		-0.03	
360	D5185	3527		2.40	
450		----		----	
451		----		----	
473	D5185 mod	3712.07		3.40	
496	D5185	3115.5		0.16	
562		----		----	
593		----		----	
605		----		----	
614	D5185	3038		-0.26	
622	D5185	2786.24		-1.63	
657	D5185	3104.6		0.10	
663		----		----	
850		----		----	
862	D5185	2986		-0.54	
875		----		----	
912	D5185	3149		0.35	
963	D5185	2777		-1.68	
994	D5185	3044.2		-0.22	
1013		----		----	
1017		----		----	
1023	D5185	3023		-0.34	
1038		----		----	
1047	D5185	2796.0		-1.57	
1059	In house	3025		-0.33	
1094	D5185	3012		-0.40	
1106		----		----	
1128	In house	2974.5		-0.60	
1146	In house	2769.0		-1.72	
1155	DIN51397 mod	2630.25		-2.47	
1160	D5185	3600		2.80	
1173		----		----	
1182		----		----	
1183		----		----	
1184		----		----	
1203	In house	3301		1.17	
1213	D4628	3330		1.33	
1231	D4951	3300		1.17	
1316	D5185	2620		-2.53	
1402	D5185	3115		0.16	
1406	D4628	3012		-0.40	
1407		----		----	
1409	D5185	2946		-0.76	
1417	In house	3231.943		0.80	
1428	D5185	3136		0.27	
1431		----		----	
1505		----		----	
1526		----		----	
1535		----		----	
1540	D6481	287.7	G(0.01)	-15.20	
1613		----		----	
1648	D5185	4328.60	G(0.05)	6.75	
1650		----		----	
1660		----		----	
1708	D5185	3096		0.06	
1722		----		----	
1730	D5185	2951.132		-0.73	
1826	D5185	3170		0.46	
1827		----		----	
1833		----		----	
1842	In house	3060		-0.14	
1850	In house	3056		-0.16	
1859		----		----	
1861	D5185	3269.0		1.00	
1864	D5185	3003		-0.45	
1900		----		----	
1920	D5185	3330		1.33	

1948	D5185	1584	C,G(0.01)	-8.16	First reported 48.2
2122	D5185	3018.5		-0.36	
2123		-----		-----	

normality	OK
n	41
outliers	3
mean (n)	3085.4
st.dev. (n)	229.84
R(calc.)	643.6
R(D5185:09)	515.5



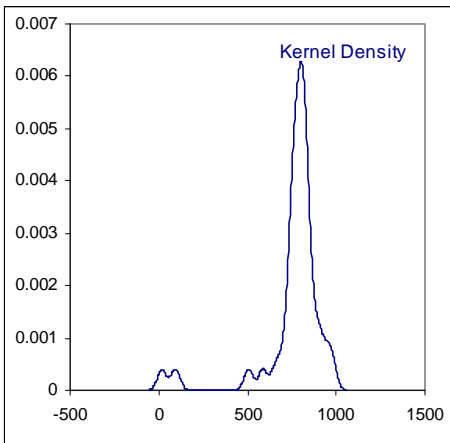
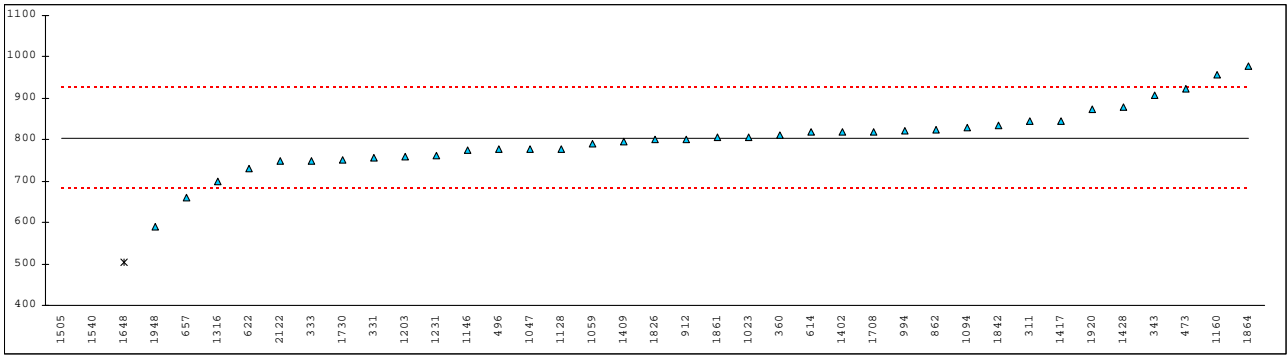
## Determination of Phosphorus (P) on sample #0938; results in mg/kg

lab	method	value	mark	Z(targ)	remarks
233		----		----	
236		----		----	
252		----		----	
254		----		----	
255		----		----	
311	D5185	845		0.97	
318		----		----	
331	D5185	757.2		-1.05	
333	D5185	750		-1.21	
343	D5185	908		2.42	
360	D5185	812		0.21	
450		----		----	
451		----		----	
473	D5185 mod	922.047		2.74	
496	D5185	776.7		-0.60	
562		----		----	
593		----		----	
605		----		----	
614	D5185	818.2		0.36	
622	D5185	730.05		-1.67	
657	D5185	660.14		-3.28	
663		----		----	
850		----		----	
862	D5185	823		0.47	
875		----		----	
912	D5185	802		-0.02	
963		----		----	
994	D5185	820.4		0.41	
1013		----		----	
1017		----		----	
1023	D5185	807		0.10	
1038		----		----	
1047	D5185	776.8		-0.60	
1059	In house	790		-0.29	
1094	D5185	830		0.63	
1106		----		----	
1128	In house	778.5		-0.56	
1146	In house	773.6		-0.67	
1155		----		----	
1160	D5185	958		3.57	
1173		----		----	
1182		----		----	
1183		----		----	
1184		----		----	
1203	In house	758		-1.03	
1213		----		----	
1231	D4951	762		-0.94	
1316	D5185	700		-2.36	
1402	D5185	819		0.37	
1406		----		----	
1407		----		----	
1409	D5185	795		-0.18	
1417	In house	845.757		0.99	
1428	D5185	879		1.75	
1431		----		----	
1505	D5185	18	G(0.01)	-18.04	
1526		----		----	
1535		----		----	
1540	D6481	96.3	G(0.01)	-16.24	
1613		----		----	
1648	D5185	505.35	G(0.05)	-6.83	
1650		----		----	
1660		----		----	
1708	D5185	819.5		0.39	
1722		----		----	
1730	D5185	750.708		-1.20	
1826	D5185	800		-0.06	
1827		----		----	
1833		----		----	
1842	In house	835		0.74	
1850		----		----	
1859		----		----	
1861	D5185	804.8		0.05	
1864	D5185	978.7		4.04	
1900		----		----	
1920	D5185	873		1.62	



1948	D5185	588.7	C	-4.92	First reported 0.836
2122	D5185	750		-1.21	
2123		-----		-----	

normality	OK
n	36
outliers	3
mean (n)	802.7
st.dev. (n)	75.06
R(calc.)	210.2
R(D5185:09)	121.8

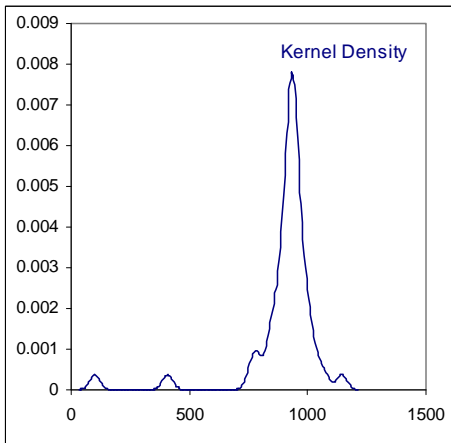
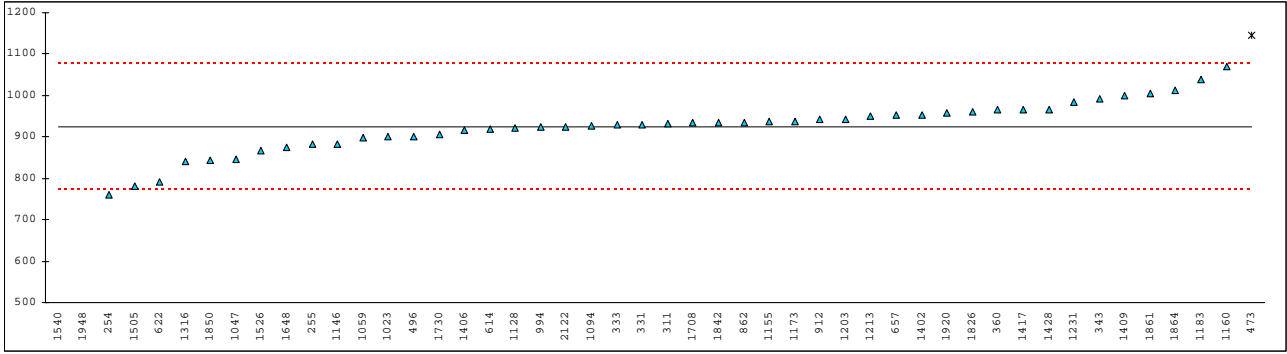


## Determination of Zinc (Zn) on sample #0938; results in mg/kg

lab	method	value	mark	Z(targ)	remarks
233		----		----	
236		----		----	
252		----		----	
254	IP308	760.18		-3.04	
255	AAS	883.00		-0.77	
311	D5185	931		0.11	
318		----		----	
331	D5185	930.4		0.10	
333	D5185	930		0.09	
343	D5185	993		1.25	
360	D5185	965		0.74	
450		----		----	
451		----		----	
473	D5185 mod	1145.16	G(0.05)	4.05	
496	D5185	901.3		-0.44	
562		----		----	
593		----		----	
605		----		----	
614	D5185	919.4		-0.10	
622	D5185	790.61		-2.48	
657	D5185	952.17		0.50	
663		----		----	
850		----		----	
862	D5185	935		0.18	
875		----		----	
912	D5185	942		0.31	
963		----		----	
994	D5185	923.5		-0.03	
1013		----		----	
1017		----		----	
1023	D5185	901		-0.44	
1038		----		----	
1047	D5185	844.9		-1.48	
1059	In house	898		-0.50	
1094	D5185	926	C	0.02	First reported 1296
1106		----		----	
1128	In house	921		-0.07	
1146	In house	883.6		-0.76	
1155	DIN51397 mod	935.94	C	0.20	First reported 779.27
1160	D5185	1070		2.67	
1173	In house	936.7		0.21	
1182		----		----	
1183	D6595	1038		2.08	
1184		----		----	
1203	In house	943		0.33	
1213	D4628	951		0.48	
1231	D4951	984		1.09	
1316	D5185	840		-1.57	
1402	D5185	953		0.51	
1406	D4628	916		-0.17	
1407		----		----	
1409	D5185	1000		1.38	
1417	In house	965.535		0.75	
1428	D5185	967		0.77	
1431		----		----	
1505	D5185	781.6		-2.64	
1526	D5185	867		-1.07	
1535		----		----	
1540	D6481	100.0	G(0.01)	-15.20	
1613		----		----	
1648	D5185	874.07		-0.94	
1650		----		----	
1660		----		----	
1708	D5185	933.5		0.16	
1722		----		----	
1730	D5185	905.234		-0.37	
1826	D5185	960		0.64	
1827		----		----	
1833		----		----	
1842	In house	934		0.16	
1850	In house	844		-1.49	
1859		----		----	
1861	D5185	1003.7		1.45	
1864	D5185	1013		1.62	
1900		----		----	
1920	D5185	957		0.59	

1948	D5185	407.7	C,G(0.01)	-9.53	First reported 12.87
2122	D5185	923.65		-0.03	
2123		-----		-----	

normality	OK
n	45
outliers	3
mean (n)	925.1
st.dev. (n)	62.15
R(calc.)	174.0
R(D5185:09)	152.0



**APPENDIX 2****Number of participants on (alphabetical) country order**

2 laboratories in AUSTRALIA  
1 laboratory in AZERBAIJAN  
2 laboratories in BELGIUM  
1 laboratory in BULGARIA  
1 laboratory in CAMEROUN  
1 laboratory in CHILE  
1 laboratory in CROATIA  
1 laboratory in ECUADOR  
2 laboratories in FRANCE  
1 laboratory in GERMANY  
1 laboratory in GHANA  
8 laboratories in GREECE  
1 laboratory in HONG KONG  
1 laboratory in HUNGARY  
1 laboratory in INDIA  
1 laboratory in INDONESIA  
1 laboratory in ITALY  
1 laboratory in JORDAN  
2 laboratories in KENYA  
1 laboratory in LATVIA  
2 laboratories in MALAYSIA  
1 laboratory in NEGARA BRUNEI DARUSSALAM  
3 laboratories in NORWAY  
1 laboratory in P.R. of CHINA  
3 laboratories in POLAND  
1 laboratory in PORTUGAL  
1 laboratory in REPUBLIC OF MACEDONIA  
2 laboratories in ROMANIA  
1 laboratory in RUSSIA  
1 laboratory in SAUDI ARABIA  
2 laboratories in SERBIA  
1 laboratory in SINGAPORE  
1 laboratory in SLOVENIA  
1 laboratory in SOUTH KOREA  
2 laboratories in SPAIN  
2 laboratories in SWEDEN  
1 laboratory in TANZANIA  
3 laboratories in THAILAND  
5 laboratories in THE NETHERLANDS  
4 laboratories in TURKEY  
8 laboratories in UNITED KINGDOM  
1 laboratory in VIETNAM

### 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
U	= reported indifferent unit
W	= results withdrawn on request of the participants
fr.	= first reported
S	= scope of the reported method is not applicable
n.a.	= not applicable
SDS	= Safety Data Sheet

#### Literature:

- 1 i.i.s. Interlaboratory Studies, Protocol for the Organisation, Statistics and Evaluation, November 2008 (version 3.1)
- 2 ASTM E178-89
- 3 ASTM E1301-89
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- 5 ISO 5725, parts 1-6, 1994
- 6 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
- 7 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 8 IP 367/84
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- 10 P.L. Davies, First reported Z. Anal. Chem, 331, 513, (1988)
- 11 J.N. Miller, Analyst, 118, 455, (1993)
- 12 Analytical Methods Committee Technical brief, No4 January 2001.
- 13 The Royal Society of Chemistry 2002, Analyst 2002, 127 pages 1359-1364, P.J. Lowthian and M. Thompson (see <http://www.rsc.org/suppdata/an/b2/b205600n/>).