

Results of Proficiency Test Used Lubricating Oil May 2013

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Report no.: iis13L02 used

July 2013

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1 INTRODUCTION

Since 1997, a proficiency test for used Lubricating Oil is organized every year by the Institute for Interlaboratory Studies. During the annual proficiency testing program 2012/2013, it was decided to continue the round robin for the analyses of used Lubricating Oil.

In this interlaboratory study, 90 laboratories in different countries have participated. See appendix 3 for the number of participants per country. In this report, the results of the 2013 used Lubricating Oil proficiency test are presented and discussed.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test. It was decided to send 2 different samples, 1 bottle of 0.5L (labelled #13063) and 1 bottle of 100 mL, 50% filled (labelled #13064)) of used Lubricating Oil, both donated by one of the participating laboratories. The analyses for fit-for-use and homogeneity were subcontracted. 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/IEC 17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation 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). This protocol can be downloaded via the FAQ page of the iis website <http://www.iisnl.com>.

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

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 precleaned 60L drum. After homogenisation, 105 subsamples were transferred to 0.5 L brown glass bottles and labelled #13063. The homogeneity of the subsamples #13063 was checked by determination of Density @ 15 °C in accordance with ASTM D4052:11 and Water in accordance with ASTM D6304-C on 8 stratified randomly selected samples.

	Density @ 15 °C in kg/L	Water in mg/kg
Sample #13063-1	0.90324	130
Sample #13063-2	0.90322	170
Sample #13063-3	0.90322	120
Sample #13063-4	0.90323	140
Sample #13063-5	0.90321	170
Sample #13063-6	0.90322	150
Sample #13063-7	0.90323	150
Sample #13063-8	0.90322	140

Table 1: homogeneity test results of subsamples #13063

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

	Density @ 15 °C in kg/L	Water in mg/kg
r (sample #13063)	0.00003	49
reference test	ASTM D4052:11	ASTM D6304:07
0.3 * R (reference test)	0.00015	101

Table 2: evaluation of repeatabilities of the subsamples #13063

The second bulk material, used Lubricating Oil, enriched 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, 109 subsamples were transferred to 100 mL PE bottles, each filled with approximately 50 mL material and labelled #13064. The homogeneity of the subsamples #13064 was checked by determination of Copper and Nickel both in accordance with ASTM D5185:09 on 8 stratified randomly selected samples.

	Copper in mg/kg	Nickel in mg/kg
Sample #13064-1	30.1	8.1
Sample #13064-2	30.0	7.9
Sample #13064-3	29.8	7.8
Sample #13064-4	29.6	7.7
Sample #13064-5	30.4	8.0
Sample #13064-6	30.4	7.6
Sample #13064-7	30.2	7.5
Sample #13064-8	30.2	7.5

Table 3: homogeneity test results of subsamples #13064

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

	Copper in mg/kg	Nickel in mg/kg
r (sample #13064)	0.8	0.6
reference test	ASTM D5185:09	ASTM D5185:09
0.3 * R (reference test)	2.2	1.3

Table 4: evaluation of repeatabilities of the subsamples #13064

The calculated repeatabilities are all less than 0.3 times the corresponding reproducibilities of the reference methods. Therefore, homogeneity of the subsamples #13063 and #13064 was assumed.

To each of the participating laboratories 2 samples of Lubricating Oil (1*0.5 L brown glass bottle labelled #13063, 1*100 mL PE bottle labelled #13064) were sent on May 01, 2013.

2.5 STABILITY OF THE SAMPLES

The stability of Lubricating Oil, packed in the brown glass bottles and PE Bottles, was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYSES

The participants were requested to determine on sample #13063: Acid Number (Total), Base Number (Total and Strong), Density @ 15°C, Flash Point PMcc, Analysis run time, Kinematic Viscosity @ 40°C and @ 100°C, Viscosity Index, Kinematic Viscosity by Houillon @ 40°C and @ 100°C and Water.

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) on sample #13064.

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 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 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' (iis-protocol, version 3.2) of January 2010. 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.

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 them with a factor of 2.8.

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data per 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 "x". Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. The Kernel Density 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.

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. 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, problems with sample despatch were encountered due to several problems. Sixteen participants reported after the final reporting date and seven participants did not report any results at all. Not all laboratories were able to report all analyses requested. In total 83 participants reported 1476 results. Observed were 90 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. Non-Gaussian distributions were found for the following determinations: Density @15°C, Flash Point, Kinematic @100°C (ASTM D445), Viscosity Index, Aluminium, Barium, Chromium, Manganese, Nickel, Sodium, Silver, Tin, Titanium, Vanadium, Calcium, Phosphorous and Zinc. 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 4.

Acid Number (Total): This determination was problematic. Three statistical outliers were observed. And two laboratories reported to have used ASTM D974/IP139, a test method that is not equivalent with ASTM D664. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of ASTM D664:11a. This phenomenon has been observed also in previous round robins and therefore the participants were requested to report a number of actual details of the test performed. It appeared that at least seventeen laboratories did not follow ASTM D664 to the letter. These laboratories did either treat the KOH solution not in the correct way and/or did not use the recommended size of test portion, see appendix 2. When the ASTM D664 data were evaluated separately, the average Acid Number of the laboratories that reported to have used an intake of approx 5 grams is lower than the average Acid Number of the laboratories that used an intake of approx 1 gram. Also, the reproducibility of the 'high intake' test results only, now is in agreement with the requirements of ASTM D664:11a.

Base Number (Total): This determination was very problematic. Three statistical outliers were observed. And one test result was excluded from the statistical evaluation as the reported test method is not equivalent with ASTM D2896:11. The calculated reproducibility after rejection of the suspect data is not at all in agreement with the requirements of ASTM D2896:11.

Base Number (Strong): This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D4739:11.

Density @ 15°C: This determination was very problematic. Nine (!) statistical outliers were observed. The calculated reproducibility, after rejection of the statistical outliers is not at all in agreement with the requirements of ASTM D4052:11. The large spread may be explained by possibly not correcting the test result for viscosity properly when using an oscillating densitometer.

Flash Point PMcc: This determination was very problematic. Three statistical outliers were observed. Two laboratories reported to have used ASTM D92 and one laboratory reported to have used ASTM D7094, test methods that are not equivalent to ASTM D93. The calculated reproducibility after rejection of the suspect data is not at all in agreement with the requirements of ASTM D93:11. Remarkably 5 laboratories reported unrealistic run times, that do not match the reported procedure (A or B). Use of procedure A (temperature increase rate of 5° to 6°C per min) should give much shorter run times compared to procedure B (temperature rate 1° to 1.6°C per min). When the test results from ASTM D93 proc.A and ASTM D93 proc.B are evaluated separately then the calculated reproducibility for test method A is much smaller then for test method B and in full agreement with the requirements of ASTM D93:11. The average flash point of D93 proc.B is significantly lower than the average flash point of D93 proc.A.

Kin.Visco.@ 40°C: This determination may be not problematic. Five statistical outliers were observed and five test results were excluded from the statistical evaluation as the reported test method is not equivalent to ASTM D445:12. However, the calculated reproducibility after rejection of the ten suspect data is in good agreement with the requirements of ASTM D445:12.

Kin.Visco.@ 100°C: This determination may be not problematic. Two statistical outliers were observed and five test results were excluded from the statistical evaluation as the reported test method is not equivalent to ASTM D445:12. However, the calculated reproducibility after rejection of the seven suspect data is in good agreement with the requirements of ASTM D445:12.

Viscosity Index This determination was problematic. Three statistical outliers were observed and two suspect results were excluded because these laboratories possibly made a calculation error. The calculated reproducibility after rejection of the five suspect data is not in agreement with the requirements of ASTM D2270:10.

Kin.Visco.@ 40°C: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection statistical outliers is in good agreement with the requirements of ASTM D7279:08. Six laboratories reported to have used ASTM D7042. A mathematical correlation between D7279 and D7042 test results for the determination @40°C is given in ASTM D7279:
 $D7042 = 0.9952 * (\text{Houillon Viscosity Result}).$

Kin.Visco.@ 100°C: This determination was not problematic. No statistical outliers were observed. And the calculated reproducibility is in good agreement with the requirements of ASTM D7279:08. No correlation is mentioned in ASTM D7279 between D7279 and D7042 test results for the determination @100°C. However, the D7042 were not excluded from the statistical evaluation.

Water: This determination was problematic for a number of laboratories. Six statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D6304:07. The preferred method to use for a product containing interfering components may be ASTM D6304 method C. This method is applicable for oils with difficult matrix interferences. When the ASTM D6304 method C data was evaluated separately, the calculated reproducibility is smaller and again in agreement with the requirements of ASTM D6304:07.

Wear metals

Seven laboratories reported to have used ASTM D6595. This test method is used for the quantification of elements from dissolved materials to particles approximately 10 µm in size, while test method ASTM D5185 does not purport to quantitatively determine insoluble particles larger than a few micrometers.

When the ASTM D6595 data for the elements Aluminium, Chromium, Nickel and Manganese were evaluated separately, the D6595 averages were significantly higher than the averages of all data for these elements. This suggests the presence of particles larger than a few micrometers.

Aluminium: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D5185:09.

Barium: This determination may be not problematic. Three statistical outliers and one false negative test result were observed. Although all reported results (except one test result) are above the application range (0.5 - 4 mg/kg), the calculated reproducibility after rejection of the statistical outliers is in good agreement with the estimated extrapolated requirements of ASTM D5185:09.

Chromium: This determination was not problematic. Five statistical outliers and one false negative test result were observed. However, the calculated reproducibility, after rejection of the statistical outliers is in agreement with the requirements of ASTM D5185:09.

Copper: This determination was not problematic. One statistical outlier and one false negative test result were observed. However, the calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of ASTM D5185:09.

- Iron: This determination was not problematic. Three statistical outliers and one false negative test result were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D5185:09.
- Lead: This determination was not problematic. Two statistical outliers and one false negative test result were observed. However, the calculated reproducibility after the rejection of the statistical outliers is in agreement with the requirements of ASTM D5185:09.
- Lithium: Regretfully, for this element no test method with precision data was available, therefore, the Horwitz equation was used to estimate the reproducibility limits. This determination may be problematic. Three statistical outliers were observed. The calculated reproducibility, after rejection of the statistical outliers is not in agreement with the strict estimated requirements, calculated using the Horwitz equation.
- Magnesium: This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after the rejection of the statistical outliers is in agreement with the requirements of ASTM D5185:09.
- Manganese: This determination was very problematic. One statistical outlier and one false negative test result were observed. However, the calculated reproducibility after the rejection of the statistical outlier is in not at all agreement with the requirements of ASTM D5185:09.
- Molybdenum: This determination was problematic for a number of laboratories. Six statistical outliers were observed. However, the calculated reproducibility after the rejection of the statistical outliers is in agreement with the requirements of ASTM D5185:09.
- Nickel: This determination was problematic. One statistical outlier and one false negative test result were observed. The calculated reproducibility after the rejection of the statistical outlier is not in agreement with the requirements of ASTM D5185:09.
- Sodium: This determination was problematic. One false negative test result but no statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D5185:09.
- Silicon: This determination was not problematic. Two statistical outliers and one negative test result were observed. However, the calculated reproducibility, after rejection of the statistical outliers is in agreement with the requirements of ASTM D5185:09.

- Silver: This determination was not problematic. Two statistical outliers and one false negative test result were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D5185:09.
- Tin: This determination was not problematic. One statistical outlier and one false negative test result were observed. However, the calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of ASTM D5185:09. The application range of the method is 10 – 40 mg/kg.
- Titanium: This determination was not problematic. Two statistical outliers and one false negative test result were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D5185:09.
- Vanadium: This determination was not problematic. Four statistical outliers and one false negative test result were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D5185:09.
- Calcium: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D5185:09.
- Phosphorus: This determination was problematic. Five statistical outliers were observed. The calculated reproducibility, after rejection of the statistical outliers is not in agreement with the requirements of D5185:09.
- Zinc: This determination was problematic for a number of laboratories. Six 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.

As used Lubricating Oil is a very difficult matrix to analyze, strict adherence to the test methods with regards to sample preparation, is advised. Improper sample preparation may be the cause of disagreement of the calculated reproducibility with the requirements of the respective reference standard. Also, one should be aware that for each element spectral interferences may occur. And differences may occur in uptake rates between test specimen and standard solutions through viscosity effects.

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. 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	45	2.92	1.38	1.28
Base Number (Total)	mg KOH/g	43	11.71	1.39	0.82
Base Number (Strong)	mg KOH/g	8	9.52	1.70	4.41
Density @ 15°C	kg/L	55	0.9033	0.0011	0.0005
Flash Point PMcc	°C	55	203.6	19.4	10.0
Kinematic Viscosity @ 40°C	mm ² /s	53	140.15	1.94	10.37
Kinematic Viscosity @ 100°C	mm ² /s	53	14.69	0.38	0.91
Viscosity Index		49	103.9	2.8	2.0
Kinematic Viscosity (Houillon) @ 40°C	mm ² /s	14	140.58	1.59	4.22
Kinematic Viscosity (Houillon) @ 100°C	mm ² /s	16	14.74	0.32	0.83
Water	mg/kg	40	381.0	535.9	597.4

Table 5: reproducibilities of test results of sample #13063

Parameter	Unit	n	Average	2.8 * sd	R (lit)
Aluminium as Al	mg/kg	51	9.4	5.9	6.8
Barium as Ba	mg/kg	43	28.5	8.0	12.8
Chromium as Cr	mg/kg	50	8.4	3.2	3.0
Copper as Cu	mg/kg	53	30.5	7.4	7.3
Iron as Fe	mg/kg	52	22.9	6.9	6.4
Lead as Pb	mg/kg	52	12.0	6.4	7.3
Lithium as Li	mg/kg	12	22.8	7.6	6.4
Magnesium as Mg	mg/kg	50	73.6	18.4	19.7
Manganese as Mn	mg/kg	45	8.8	3.7	1.8
Molybdenum as Mo	mg/kg	43	7.9	2.0	2.8
Nickel as Ni	mg/kg	53	8.0	4.9	4.2
Sodium as Na	mg/kg	44	16.4	12.1	8.0
Silicon as Si	mg/kg	49	13.0	5.8	7.9
Silver as Ag	mg/kg	43	8.0	2.3	2.8
Tin as Sn	mg/kg	46	7.6	7.6	7.4
Titanium as Ti	mg/kg	45	7.3	2.7	6.4
Vanadium as V	mg/kg	47	7.9	2.4	2.7
Calcium as Ca	mg/kg	57	3317	782	566
Phosphorus as P	mg/kg	49	858	166	126
Zinc as Zn	mg/kg	51	933	149	153

Table 6: reproducibilities of test results of sample #13064

Without further statistical calculations it can be concluded that for several tests there is a good compliance of the group of participants with the relevant standards. The tests, which are problematic, have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF MAY 2013 WITH PREVIOUS PTS

	May 2013	May 2012	May 2011	May 2010
Number of reporting participants	83	77	75	82
Number of results reported	1476	1216	1257	1409
Statistical outliers	90	53	52	88
Percentage outliers	6.1%	4.4%	4.1%	6.2%

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	May 2013	May 2012	May 2011	May 2010
Total Acid Number	-	-	--	--
Total Base Number	--	-	--	--
Base Number Strong	++	++	n.e.	n.e.
Density @ 15 °C	--	-	--	--
Flash Point PMcc	--	--	--	--
Kinematic Viscosity @ 40 °C	++	++	++	++
Kinematic Viscosity @ 100 °C	++	-	++	++
Viscosity Index	-	n.e.	n.e.	n.e.
Kinematic Viscosity (Houillon) @ 40 °C	++	n.e.	n.e.	n.e.
Kinematic Viscosity (Houillon) @ 100 °C	++	n.e.	n.e.	n.e.
Water	+	-	++	++
Metals (20 elements)	+	+	+/-	+/-

Table 8: comparison determinations against the reference standards

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.e.: not evaluated

APPENDIX 1

Determination of Acid Number (Total) on sample #13063; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
230	D664	2.699		-0.48	
233		----		----	
237	D664	3.12		0.44	
252		----		----	
254		----		----	
255		----		----	
260		----		----	
311	D664	2.56		-0.78	
315		----		----	
325		----		----	
331	D664	2.44		-1.04	
340	D664	2.88		-0.08	
343	D664	2.93		0.03	
349	D664	2.98		0.14	
360	D664	2.875		-0.09	
398	D664	2.901		-0.03	
420		----		----	
432		----		----	
450		----		----	
451	D664Mod.	3.237		0.70	
473		----		----	
496	D664	2.920		0.01	
511		----		----	
541		----		----	
551		----		----	
562	in house	0.15	G(0.05)	-6.04	
575		----		----	
593		----		----	
603	D664	3.42		1.10	
608		----		----	
609	D664	3.2574		0.74	
614		----		----	
633	D664	3.1543		0.52	
657	D664	3.94		2.23	
663	D664	2.499		-0.91	
823	D664	2.60		-0.69	
862	D664	3.1738		0.56	
875	D664	3.068		0.33	
902		----		----	
912	D664	2.29		-1.37	
922		----		----	
963		----		----	
994	D664	3.5		1.27	
1013		----		----	
1017		----		----	
1023	in house	1.73		-2.59	
1059	ISO6619	2.50		-0.91	
1106		----		----	
1146	D664	3.074		0.34	
1155	ISO6619	3.719		1.75	
1161	D664	0.969	G(0.05)	-4.25	
1173		----		----	
1201	D664	4.33		3.08	
1203	D664	2.78		-0.30	
1231	D664	2.662		-0.56	
1243		----		----	
1262	D974	2.475	ex	-0.96	result excluded, first reported: 1.431
1271	D664	2.935		0.04	
1316	D664	2.09		-1.80	
1358		----		----	
1396	IP139	5.557	ex	5.76	result excluded, see §4.1
1402		----		----	
1428		----		----	
1431	D664	2.67		-0.54	
1435		----		----	
1480	D664	3.483		1.24	
1495		----		----	
1569	D664	2.514		-0.88	
1571	D664	2.1284		-1.72	
1579		----		----	
1622	D664	3.9306		2.21	
1648		----		----	
1660	IEC62021	2.912		-0.01	
1680		----		----	

1704	D664	2.419		-1.09
1720	D664	2.92		0.01
1722		----		----
1730		----		----
1740	D664	2.78		-0.30
1800		----		----
1827	D664	6.097	G(0.01)	6.94
1833	D664	2.83953		-0.17
1842		----		----
1850	ISO6619	2.86		-0.12
1854	D664	2.89		-0.06
1900	D664	3.0415		0.27
1903	INH-5088	2.78		-0.30
1915		----		----
2122	IP177	2.823		-0.20
3166		----		----

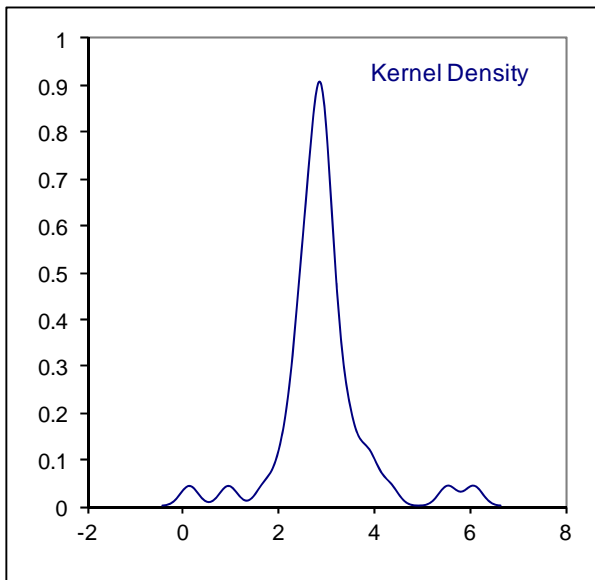
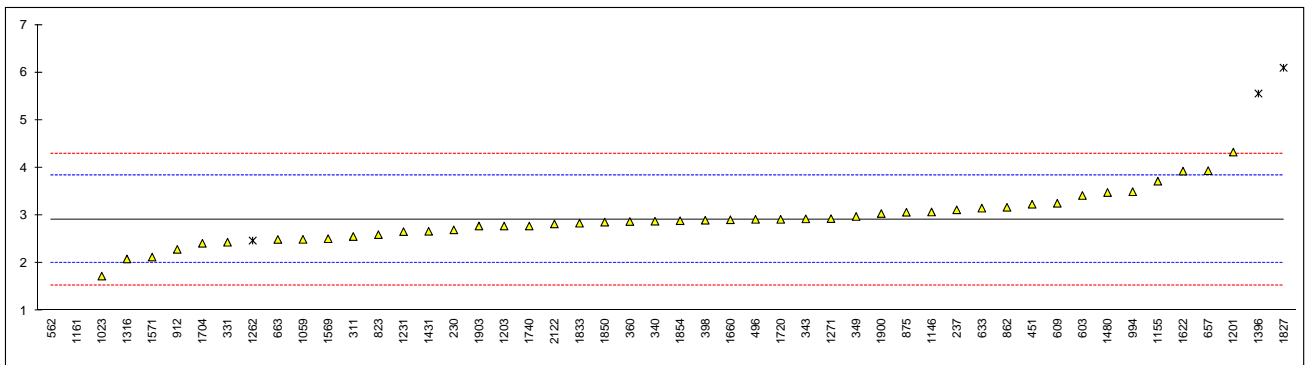
normality	OK
n	45
outliers	3
mean (n)	2.917
st.dev. (n)	0.4920
R(calc.)	1.378
R(D664:11a)	1.283

D664 results with low intake:

OK
12
1
3.064
0.5070
1.420
1.348

D664 results with high intake:

OK
21
0
2.838
0.2940
0.823
1.249



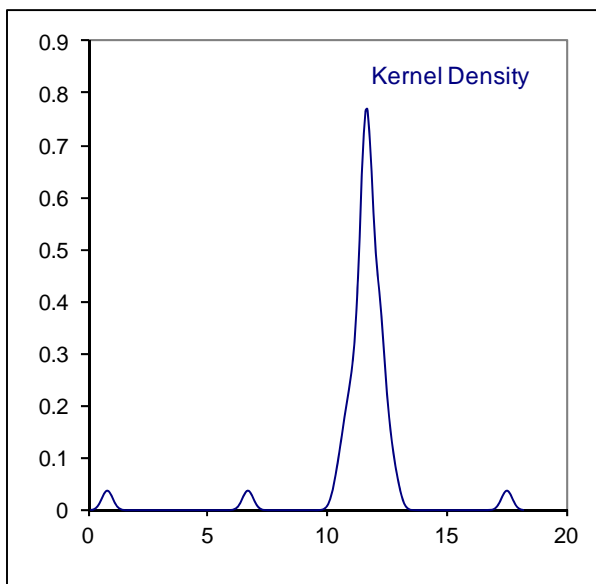
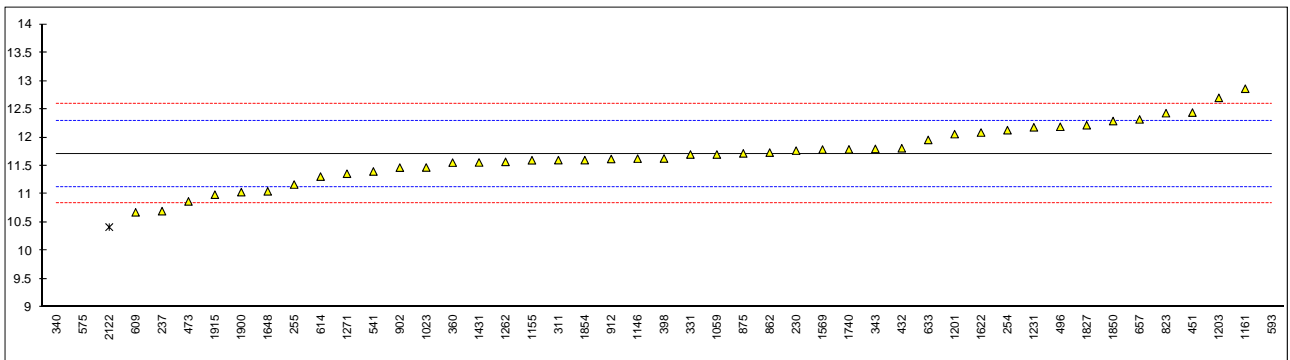
Determination of Base Number (Total) on sample #13063; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
230	D2896	11.769		0.20	
233		----		----	
237	D2896	10.7		-3.45	
252		----		----	
254	D2896	12.13		1.43	
255	D2896	11.17		-1.85	
260		----		----	
311	D2896	11.6		-0.38	
315		----		----	
325		----		----	
331	D2896	11.7		-0.03	
340	D2896	0.83	G(0.01)	-37.16	
343	D2896	11.8		0.31	
349		----		----	
360	D2896	11.557		-0.52	
398	D2896	11.629		-0.28	
420		----		----	
432	D2896	11.81		0.34	
450		----		----	
451	D2896	12.44		2.49	
473	D2896	10.8717		-2.86	
496	D2896	12.19		1.64	
511		----		----	
541	D2896	11.4		-1.06	
551		----		----	
562		----		----	
575	D2896	6.7	C,G(0.01)	-17.11	first reported: 9.91
593	D2896	17.5153	C,G(0.01)	19.83	first reported: 9.1527
603		----		----	
608		----		----	
609	D2896	10.6786		-3.52	
614	D2896	11.31		-1.37	
633	D2896	11.9570		0.84	
657	D2896	12.32		2.08	
663		----		----	
823	D2896	12.43		2.46	
862	D2896	11.7342		0.08	
875	D2896	11.720		0.03	
902	D2896	11.468		-0.83	
912	D2896	11.62		-0.31	
922		----		----	
963		----		----	
994		----		----	
1013		----		----	
1017		----		----	
1023	D2896	11.47		-0.82	
1059	ISO3771	11.7		-0.03	
1106		----		----	
1146	D2896	11.627		-0.28	
1155	ISO3771	11.598		-0.38	
1161	D2896	12.861		3.93	
1173		----		----	
1201	D2896	12.06		1.19	
1203	D2896	12.70		3.38	
1231	D2896	12.18		1.60	
1243		----		----	
1262	D2896	11.57		-0.48	
1271	ISO3771	11.36		-1.20	
1316		----		----	
1358		----		----	
1396		----		----	
1402		----		----	
1428		----		----	
1431	D2896	11.56		-0.51	
1435		----		----	
1480		----		----	
1495		----		----	
1569	D2896	11.788		0.27	
1571		----		----	
1579		----		----	
1622	D2896	12.0884		1.29	
1648	D2896	11.05		-2.26	
1660		----		----	
1680		----		----	
1704		----		----	

1720		----		----
1722		----		----
1730		----		----
1740	D2896	11.79		0.27
1800		----		----
1827	D2896	12.217		1.73
1833		----		----
1842		----		----
1850	ISO3771	12.29		1.98
1854	D2896	11.6		-0.38
1900	D2896	11.035		-2.31
1903		----		----
1915	D2896	10.99	C	-2.46
2122	IP400	10.418	ex	-4.41
3166		----		----

first reported: 7.41
result excluded, see §4.1

normality OK
n 43
outliers 3
mean (n) 11.710
st.dev. (n) 0.4980
R(calc.) 1.394
R(D2896:11) 0.820

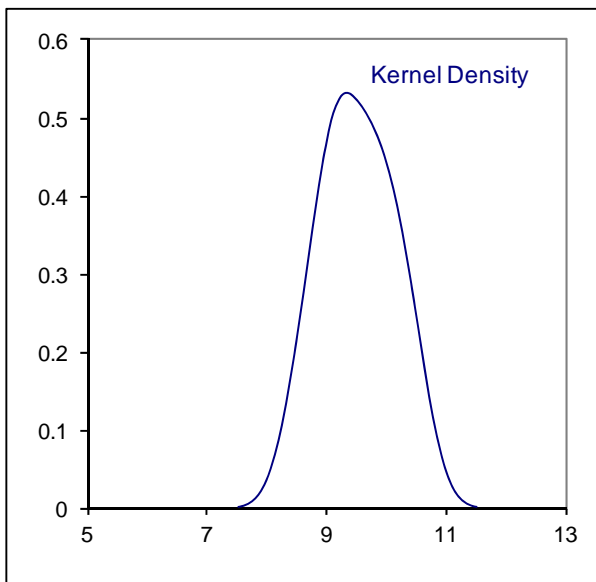
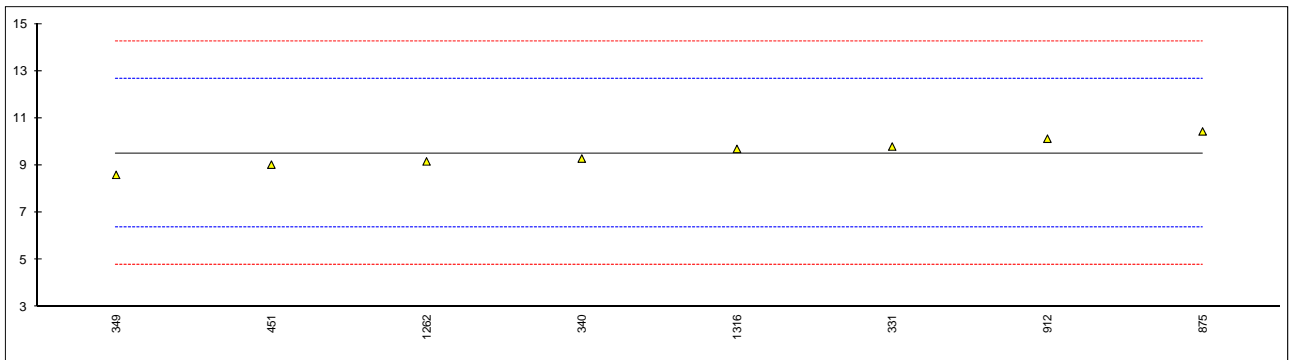


Determination of Base Number (Strong) on sample #13063; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
230		----		----	
233		----		----	
237		----		----	
252		----		----	
254		----		----	
255		----		----	
260		----		----	
311		----		----	
315		----		----	
325		----		----	
331	D4739	9.8		0.18	
340	D4739	9.29		-0.15	
343		----		----	
349	D4739	8.6		-0.58	
360		----		----	
398		----		----	
420		----		----	
432		----		----	
450		----		----	
451	D4739	9.03		-0.31	
473		----		----	
496		----		----	
511		----		----	
541		----		----	
551		----		----	
562		----		----	
575		----		----	
593		----		----	
603		----		----	
608		----		----	
609		----		----	
614		----		----	
633		----		----	
657		----		----	
663		----		----	
823		----		----	
862	D4739	nil		----	
875	D4739	10.44		0.58	
902		----		----	
912	D4739	10.13		0.39	
922		----		----	
963		----		----	
994		----		----	
1013		----		----	
1017		----		----	
1023		----		----	
1059		----		----	
1106		----		----	
1146		----		----	
1155		----		----	
1161		----		----	
1173		----		----	
1201	D4739	nil		----	
1203		----		----	
1231		----		----	
1243		----		----	
1262	D4739	9.17		-0.22	
1271		----		----	
1316	D4739	9.7		0.11	
1358		----		----	
1396		----		----	
1402		----		----	
1428		----		----	
1431		----		----	
1435		----		----	
1480		----		----	
1495		----		----	
1569		----		----	
1571		----		----	
1579		----		----	
1622		----		----	
1648		----		----	
1660		----		----	
1680		----		----	
1704		----		----	

1720	----	----
1722	----	----
1730	----	----
1740	----	----
1800	----	----
1827	----	----
1833	----	----
1842	----	----
1850	----	----
1854	----	----
1900	----	----
1903	----	----
1915	----	----
2122	----	----
3166	----	----

normality OK
 n 8
 outliers 0
 mean (n) 9.520
 st.dev. (n) 0.6084
 R(calc.) 1.704
 R(D4739:11) 4.412

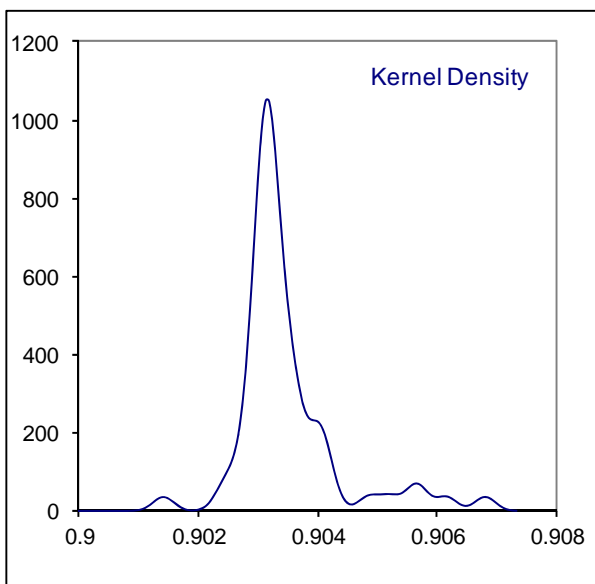
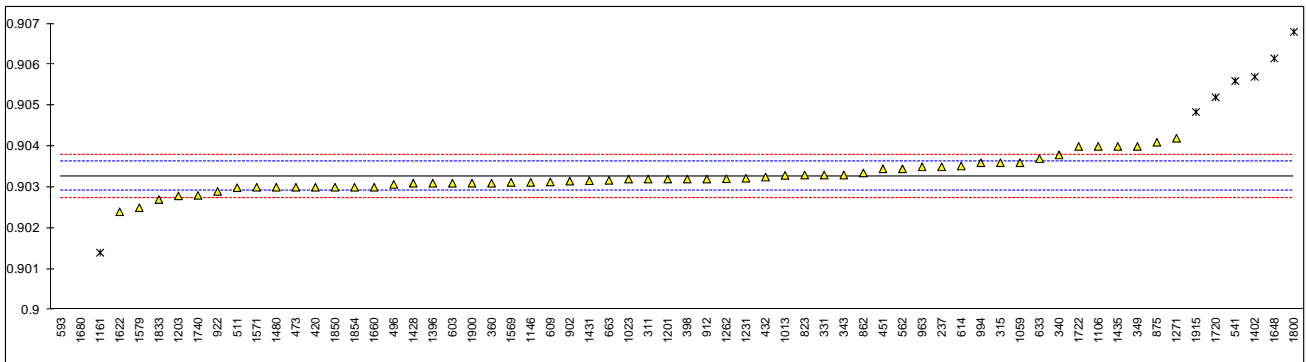


Determination of Density @ 15°C on sample #13063; results in kg/L

lab	method	value	mark	z(targ)	remarks
230		-----		-----	
233		-----		-----	
237	D4052	0.9035		1.30	
252		-----		-----	
254		-----		-----	
255		-----		-----	
260		-----		-----	
311	D4052	0.9032		-0.38	
315	D4052	0.9036		1.86	
325		-----		-----	
331	ISO12185	0.9033		0.18	
340	D4052	0.90379		2.92	
343	D4052	0.9033		0.18	
349	D4052	0.9040		4.10	
360	D4052	0.9031		-0.94	
398	D4052	0.9032		-0.38	
420	D7042	0.903		-1.50	
432	D4052	0.90325		-0.10	
450		-----		-----	
451	D4052	0.90345		1.02	
473	D4052	0.9030		-1.50	
496	D4052	0.90307		-1.11	
511	D4052	0.90299		-1.56	
541	D4052	0.9056	G(0.01)	13.06	
551		-----		-----	
562	D4052	0.90345		1.02	
575		-----		-----	
593	D4052	0.8962	G(0.01)	-39.58	
603	D4052	0.9031		-0.94	
608		-----		-----	
609	D4052	0.90313	C	-0.77	first reported: 903.13 kg/L
614	D4052	0.90352		1.41	
633	D4052	0.9037		2.42	
657		-----		-----	
663	D4052	0.90317		-0.55	
823	D4052	0.9033		0.18	
862	D4052	0.90335		0.46	
875	D4052	0.9041		4.66	
902	D4052	0.90315		-0.66	
912	D4052	0.9032		-0.38	
922	D4052	0.9029		-2.06	
963	D4052	0.9035		1.30	
994	D4052	0.9036		1.86	
1013	D4052	0.90329		0.12	
1017		-----		-----	
1023	D4052	0.9032		-0.38	
1059	D4052	0.9036		1.86	
1106	D5002	0.9040		4.10	
1146	D4052	0.90312		-0.83	
1155		-----		-----	
1161	ISO3675	0.9014	C,G(0.05)	-10.46	probably unit error: reported: 901.4 kg/L
1173		-----		-----	
1201	D4052	0.9032		-0.38	
1203	ISO12185	0.90279		-2.68	
1231	D4052	0.90322		-0.27	
1243		-----		-----	
1262	D4052	0.90321	C	-0.32	first reported: 903.21kg/L
1271	D4052	0.9042	C	5.22	first reported: 904.2 kg/L
1316		-----		-----	
1358		-----		-----	
1396	IP365	0.9031		-0.94	
1402	D4052	0.9057	G(0.05)	13.62	
1428	D4052	0.9031		-0.94	
1431	D4052	0.90316		-0.60	
1435	D4052	0.904		4.10	
1480	D4052	0.9030	C	-1.50	first reported: 0.907 kg/L
1495		-----		-----	
1569	D4052	0.90312		-0.83	
1571	D7042	0.903		-1.50	
1579	ISO3675	0.9025	C	-4.30	first reported: 0.90174 kg/L
1622	D4052	0.9024		-4.86	
1648	D1298	0.90615	G(0.05)	16.14	
1660	D7042	0.9030		-1.50	
1680	in house	0.898	G(0.01)	-29.50	
1704		-----		-----	

1720	D4052	0.9052	C,G(0.05)	10.82	first reported: 0.9050 kg/L
1722	D4052	0.9040		4.10	
1730		----		----	
1740	D4052	0.9028		-2.62	
1800	D4052	0.9068	G(0.01)	19.78	
1827		----		----	
1833	D4052	0.9027		-3.18	
1842		----		----	
1850	D4052	0.9030		-1.50	
1854	D4052	0.9030		-1.50	
1900	D4052	0.9031	C	-0.94	first reported: 903.1 kg/L
1903		----		----	
1915	D4052	0.90484	G(0.01)	8.80	
2122		----		----	
3166		----		----	

normality not OK
 n 55
 outliers 9
 mean (n) 0.90327
 st.dev. (n) 0.000382
 R(calc.) 0.00107
 R(D4052:11) 0.00050



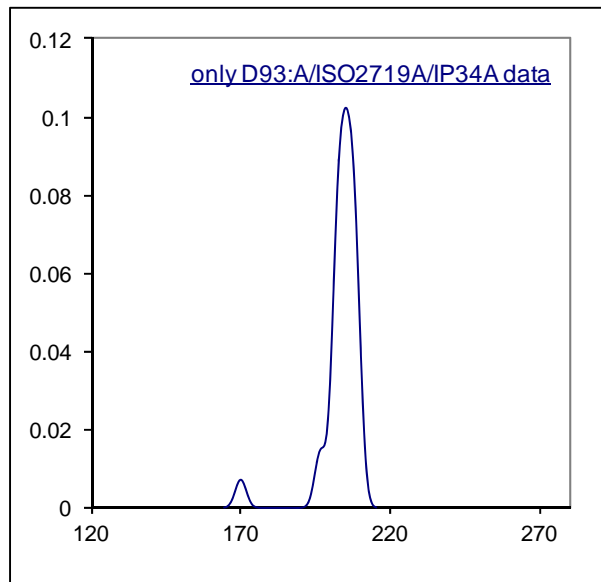
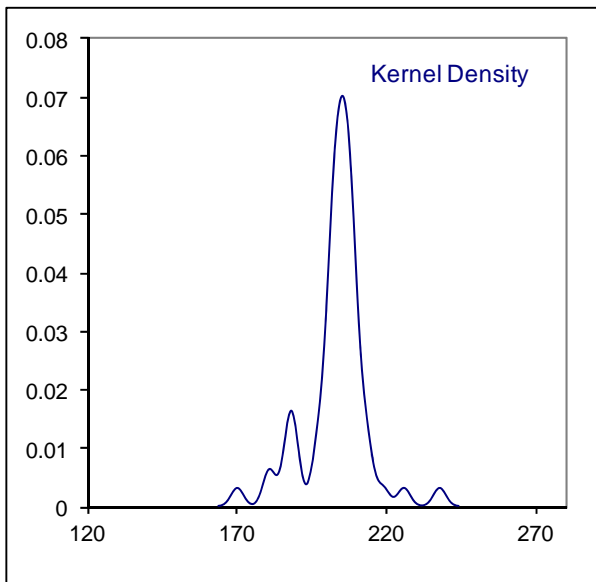
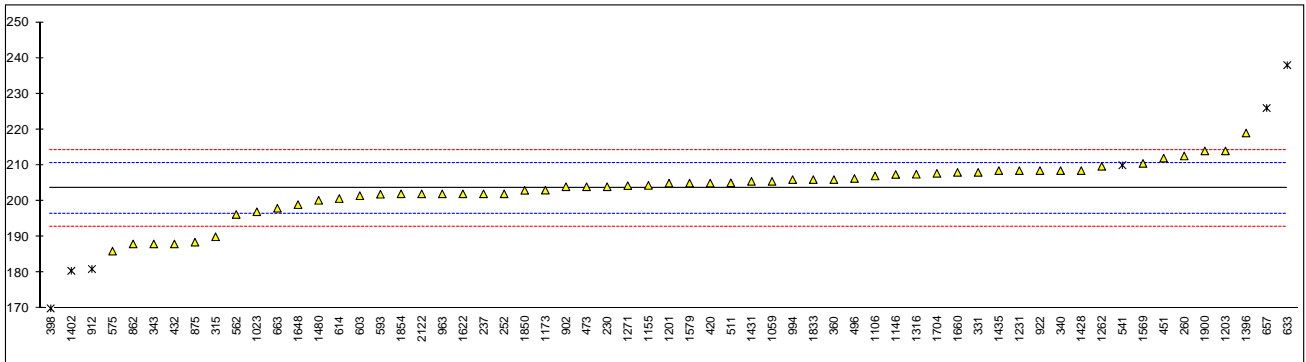
Determination of Flash Point PMcc on sample #13063; results in °C

lab	method	value	mark	z(targ)	Analysis run time	remarks
230	D93A	204.0		0.12	45	
233		----		----	----	
237	D93A	202.0		-0.44	----	
252	D93A	202		-0.44	25	
254		----		----	----	
255		----		----	----	
260	D93	212.6		2.52	----	
311		----		----	----	
315	D93B	190.0		-3.80	132.08	
325		----		----	----	
331	D93B	208.0		1.24	36	run time does not match with procedure B
340	D93A	208.5		1.38	20	
343	D93B	188.0		-4.36	----	
349		----		----	----	
360	D93A	206.0		0.68	37	
398	D93A	170.0	G(0.01)	-9.40	24	
420	ISO2719B	205.0		0.40	----	
432	D93B	188.0		-4.36	139	
450		----		----	----	
451	D93B	212		2.36	45	run time does not match with procedure B
473	D93B	204.0		0.12	----	
496	D93A	206.3		0.76	----	
511	D93A	205.1		0.42	36.35	
541	D7094	210.0	ex	1.80	----	result excluded, see § 4.1
551		----		----	----	
562	D93A	196.25		-2.05	----	
575	D93B	186		-4.92	1'57.3046"	
593	D93A	201.9		-0.47	----	
603	D93A	201.5		-0.58	----	
608		----		----	----	
609		----		----	----	
614	D93B	200.7		-0.81	43	run time does not match with procedure B
633	D92	238.0	ex	9.64	35	result excluded, see § 4.1
657	D92	226	ex	6.28	----	result excluded, see § 4.1
663	D93B	198.0		-1.56	----	
823		----		----	----	
862	D93B	188	C	-4.36	----	first reported: 191
875	D93B	188.5		-4.22	127	
902	D93A	204.0		0.12	38m51s	
912	D93B	181	DG(0.05)	-6.32	120	
922	D93A	208.5		1.38	37	
963	D93A	202		-0.44	----	
994	D93B	206		0.68	35	run time does not match with procedure B
1013		----		----	----	
1017		----		----	----	
1023	D93A	197		-1.84	----	
1059	ISO2719A	205.5		0.54	40	
1106	D93A	207.0		0.96	----	
1146	D93A	207.43		1.08	40m33s	
1155	D93	204.37		0.22	----	
1161		----		----	----	
1173	IP34A	203.05		-0.15	----	
1201	D93A	205.0		0.40	----	
1203	ISO2719	214		2.92	----	
1231	D93A	208.5		1.38	----	
1243		----		----	----	
1262	D93A	209.7		1.71	180.0	run time does not match with procedure A
1271	ISO2719A	204.3		0.20	33	
1316	D93A	207.5		1.10	----	
1358		----		----	----	
1396	IP523	219		4.32	1	reported an unrealistic run time
1402	D93	180.5	DG(0.05)	-6.46	----	
1428	D93A	208.5		1.38	----	
1431	D93A	205.5		0.54	----	
1435	D93	208.5		1.38	----	
1480	D93	200.20		-0.95	15.5	
1495		----		----	----	
1569	D93C	210.5		1.94	17.5	
1571		----		----	----	
1579	ISO2719A	205		0.40	----	
1622	D93A	202.0		-0.44	----	
1648	D93	199	C	-1.28	----	first reported: 190
1660	D93A	208.0		1.24	16	
1680		----		----	----	
1704	D93	207.74		1.16	----	

1720		----		----	----
1722		----		----	----
1730		----		----	----
1740		----	W	----	----
1800		----		----	----
1827		----		----	----
1833	D93	206		0.68	25
1842		----		----	----
1850	ISO2719A	203		-0.16	----
1854	D93B	202	C	-0.44	----
1900	in house	214		2.92	19
1903		----		----	----
1915	D93	>110		----	----
2122	D93AF	202		-0.44	----
3166	D93	>100		----	----
	normality	not OK		<u>only D93:B</u>	<u>only D93:A/ISO2719A/IP34A data</u>
	n	55		OK	OK
	outliers	3		15	30
	mean (n)	203.58		0	1
	st.dev. (n)	6.932		196.35	204.66
	R(calc.)	19.41		9.690	3.299
	R(D93B:12)	10.00		27.13	9.24
				10.00	10.00

result withdrawn, first reported: 190.0

first reported: 190

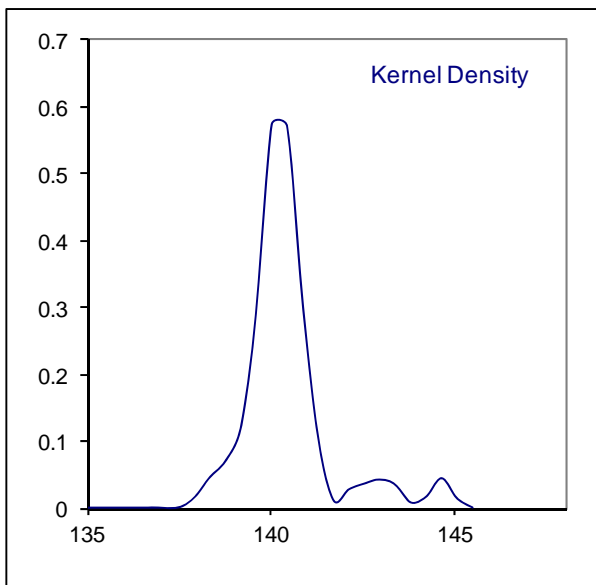
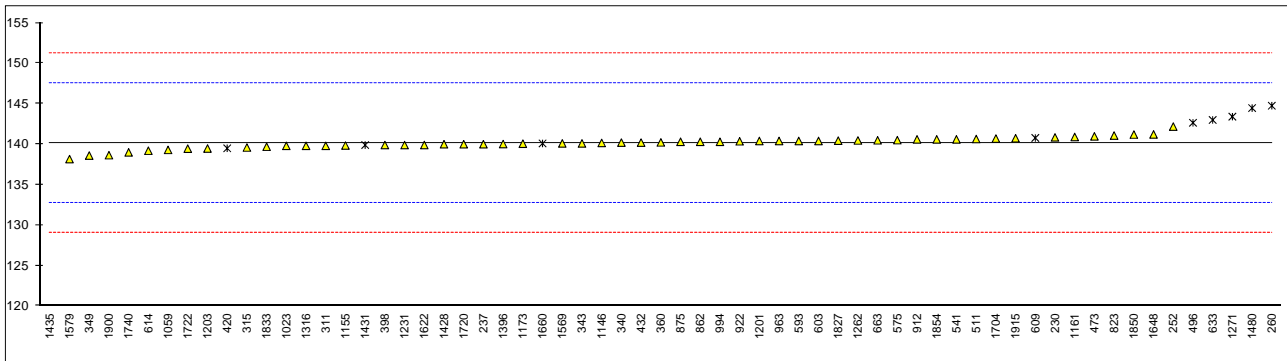


Determination of Kinematic Viscosity @ 40°C (D445) on sample #13063; results in mm²/s

lab	method	value	mark	z(targ)	remarks
230	D445	140.85		0.19	
233		----		----	
237	D445	140.0		-0.04	
252	D445	142.18		0.55	
254		----		----	
255		----		----	
260	D445	144.76	G(0.05)	1.24	
311	D445	139.8		-0.10	
315	D445	139.6		-0.15	
325		----		----	
331		----		----	
340	D445	140.20		0.01	
343	D445	140.1		-0.01	
349	D445	138.6		-0.42	
360	D445	140.23		0.02	
398	D445	139.90		-0.07	
420	D7042	139.5	ex	-0.18	result excluded, see §4.1
432	D445	140.2		0.01	
450		----		----	
451		----		----	
473	D445	140.97		0.22	
496	D445	142.65	G(0.05)	0.67	
511	D445	140.647		0.13	
541	D445	140.6		0.12	
551		----		----	
562		----		----	
575	D445	140.51		0.10	
593	D445	140.40		0.07	
603	D445	140.4		0.07	
608		----		----	
609	D7042	140.775	ex	0.17	result excluded, see §4.1
614	D445	139.2		-0.26	
633	D445	143.0	G(0.05)	0.77	
657		----		----	
663	D445	140.49		0.09	
823	D445	141.074		0.25	
862	D445	140.3		0.04	
875	D445	140.3		0.04	
902		----		----	
912	D445	140.6		0.12	
922	D445	140.37		0.06	
963	D445	140.4		0.07	
994	D445	140.3		0.04	
1013		----		----	
1017		----		----	
1023	D445	139.8		-0.10	
1059	ISO3104	139.3		-0.23	
1106		----		----	
1146	D445	140.16		0.00	
1155	ISO3104	139.84		-0.09	
1161	ISO3104	140.9		0.20	
1173	IP71	140.07		-0.02	
1201	D445	140.4		0.07	
1203	ISO3104	139.475		-0.18	
1231	D445	139.9		-0.07	
1243		----		----	
1262	D445	140.47		0.09	
1271	ISO3104	143.41	G(0.05)	0.88	
1316	D445	139.8		-0.10	
1358		----		----	
1396	IP71	140.02		-0.04	
1402		----		----	
1428	D445	140.0		-0.04	
1431	D7042	139.9	ex	-0.07	result excluded, see §4.1
1435	D7042	39.03	ex	-27.30	result excluded, see §4.1
1480	D445	144.469	C,G(0.01)	1.16	first reported: 128.947
1495		----		----	
1569	D445	140.1	C	-0.01	first reported:14.72
1571		----		----	
1579	ISO3104	138.1622		-0.54	
1622	D445	139.91		-0.07	
1648	D445	141.21		0.28	
1660	D7042	140.10	ex	-0.01	result excluded, see §4.1
1680		----		----	
1704	D445	140.7		0.15	

1720	D445	140.0	-0.04
1722	D445	139.4499	-0.19
1730		-----	-----
1740	D445	139.0	-0.31
1800		-----	-----
1827	D445	140.44	0.08
1833	D445	139.7	-0.12
1842		-----	-----
1850	ISO3104	141.2	0.28
1854	D445	140.6	0.12
1900	D445	138.65	-0.41
1903		-----	-----
1915	D445	140.733	0.16
2122		-----	-----
3166		-----	-----

normality	OK	<u>all data (without exclusions)</u>	OK
n	53		57
outliers	5		6
mean (n)	140.155		140.149
st.dev. (n)	0.6930		0.6795
R(calc.)	1.940		1.903
R(D445:12)	10.371		10.371

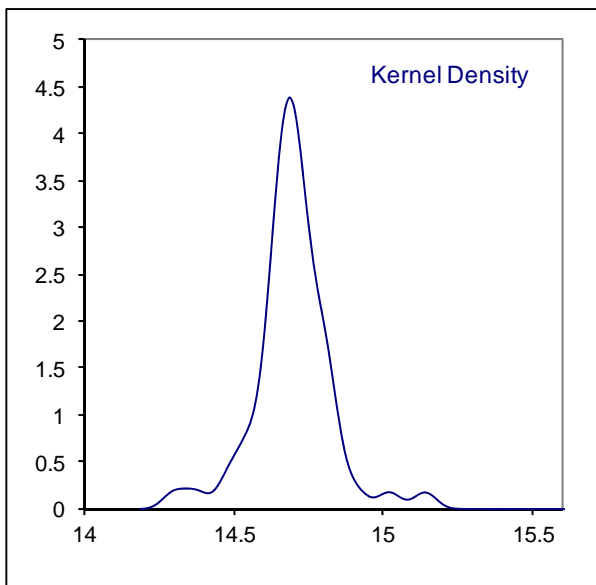
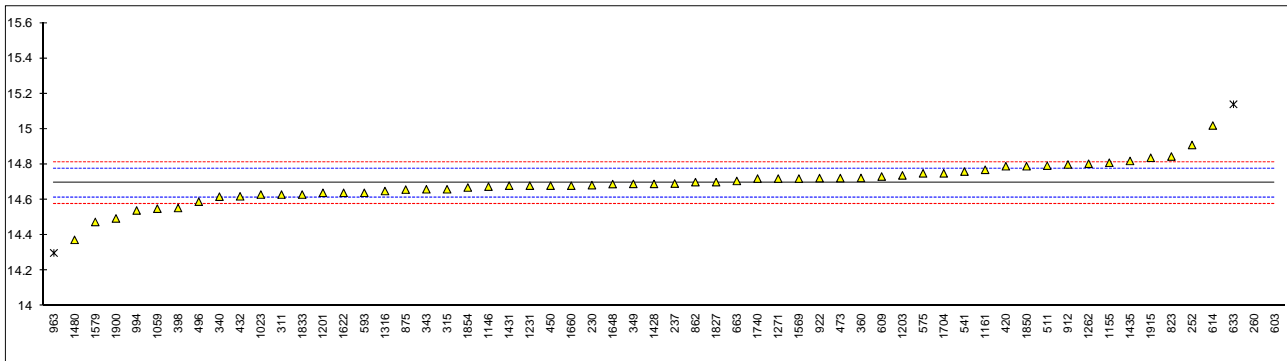


Determination of Kinematic Viscosity @ 100°C on sample #13063; results in mm²/s

lab	method	value	mark	z(targ)	remarks
230	D445	14.683		-0.03	
233		----		----	
237	D445	14.692		0.00	
252	D445	14.91		0.67	
254		----		----	
255		----		----	
260	D445	16.64	G(0.01)	6.01	
311	D445	14.63		-0.19	
315	D445	14.66		-0.10	
325		----		----	
331		----		----	
340	D445	14.618		-0.23	
343	D445	14.66		-0.10	
349	D445	14.69		-0.01	
360	D445	14.724		0.10	
398	D445	14.554		-0.42	
420	D7042	14.79	ex	0.30	result excluded, see §4.1
432	D445	14.62		-0.22	
450	D445	14.68		-0.04	
451		----		----	
473	D445	14.723		0.10	
496	D445	14.59		-0.31	
511	D445	14.793		0.31	
541	D445	14.76		0.21	
551		----		----	
562		----		----	
575	D445	14.750		0.18	
593	D445	14.64		-0.16	
603	D445	18.18	G(0.01)	10.76	
608		----		----	
609	D7042	14.732	ex	0.12	result excluded, see §4.1
614	D445	15.02		1.01	
633	D445	15.14		1.38	
657		----		----	
663	D445	14.707		0.05	
823	D445	14.845		0.47	
862	D445	14.70		0.03	
875	D445	14.658		-0.10	
902		----		----	
912	D445	14.80		0.33	
922	D445	14.723		0.10	
963	D445	14.30		-1.21	
994	D445	14.54		-0.47	
1013		----		----	
1017		----		----	
1023	D445	14.63		-0.19	
1059	ISO3104	14.55		-0.44	
1106		----		----	
1146	D445	14.675		-0.05	
1155	ISO3104	14.81		0.37	
1161	ISO3104	14.77		0.24	
1173		----		----	
1201	D445	14.64		-0.16	
1203	ISO3104	14.738		0.14	
1231	D445	14.68		-0.04	
1243		----		----	
1262	D445	14.804		0.35	
1271	ISO3104	14.72		0.09	
1316	D445	14.65		-0.13	
1358		----		----	
1396		----		----	
1402		----		----	
1428	D445	14.69		-0.01	
1431	D7042	14.68	ex	-0.04	result excluded, see §4.1
1435	D7042	14.82	ex	0.40	result excluded, see §4.1
1480	D445	14.3741		-0.98	
1495		----		----	
1569	D445	14.72	C	0.09	first reported: 140.1
1571		----		----	
1579	ISO3104	14.4752		-0.67	
1622	D445	14.64		-0.16	
1648	D445	14.689		-0.01	
1660	D7042	14.68	ex	-0.04	result excluded, see §4.1
1680		----		----	
1704	D445	14.75		0.18	

1720		----	----
1722		----	----
1730		----	----
1740	D445	14.72	0.09
1800		----	----
1827	D445	14.70	0.03
1833	D445	14.63	-0.19
1842		----	----
1850	ISO3104	14.79	0.30
1854	D445	14.67	-0.07
1900	D445	14.495	-0.61
1903		----	----
1915	D445	14.838	0.45
2122		----	----
3166		----	----

		<u>all data (without exclusions)</u>
normality	not OK	not OK
n	53	58
outliers	2	2
mean (n)	14.692	14.696
st.dev. (n)	0.1340	0.1298
R(calc.)	0.375	0.363
R(D445:12)	0.908	0.908

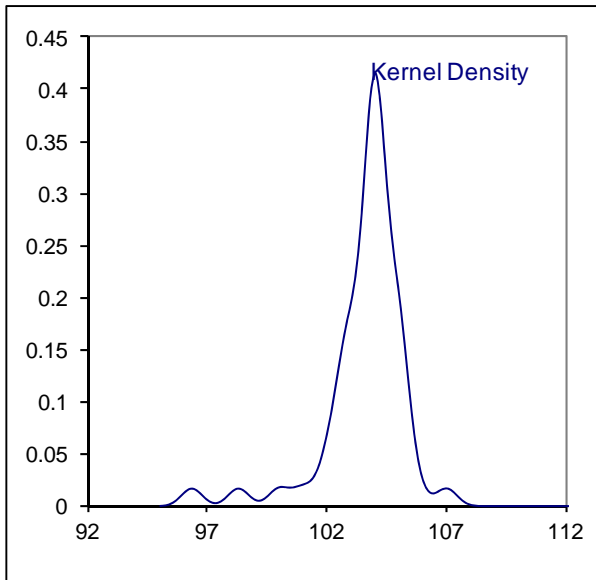
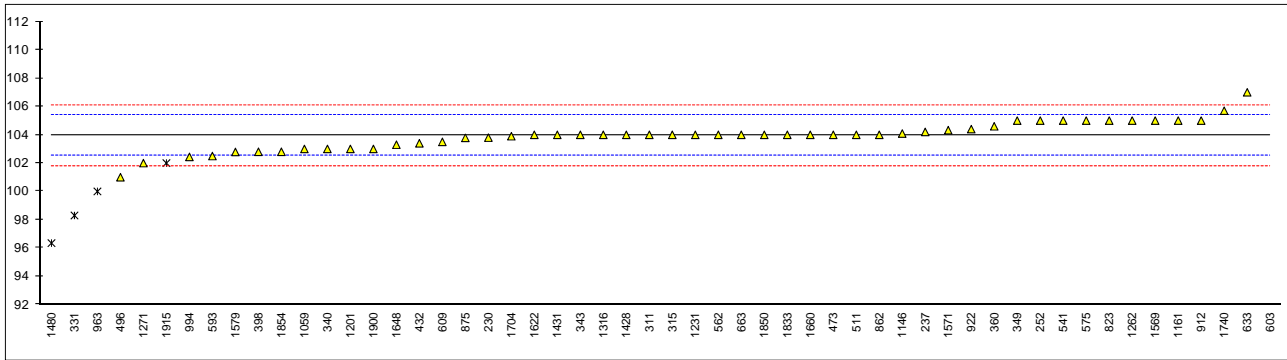


Determination of Viscosity index on sample #13063

lab	method	value	mark	z(targ)	VI calculated by iis	Remarks
230	D2270	103.8		-0.20	103.6	
233		----		----	103.6	
237	D2270	104.2		0.36	104.5	
252	D2270	105		1.48	105	
254		----		----	----	
255		----		----	----	
260		----		----	122	
311	D2270	104		0.08	104	
315	D2270	104		0.08	104	
325		----		----	----	
331	D2270	98.3	G(0.01)	-7.90	----	
340	D2270	103		-1.32	103	
343	D2270	104		0.08	104	
349	D2270	105		1.48	106	
360	D2270	104.6		0.92	104.7	
398	D2270	102.8		-1.60	102.9	
420		----		----	106.1	
432	D2270	103.4		-0.76	103.4	
450		----		----	----	
451		----		----	----	
473	D2270	104.0		0.08	104.0	
496	D2270	101.0		-4.12	100.9	
511	D2270	104		0.08	105	
541	D2270	105		1.48	105	
551		----		----	----	
562	D2279	104		0.08	----	
575	D2270	105		1.48	105	
593	D2270	102.5		-2.02	103.5	
603	D2270	144.67	ex	57.01	142.27	see §4.1
608		----		----	----	
609	D2270	103.5		-0.62	104.3	
614		----		----	109.2	
633	D2270	107		4.28	107	
657		----		----	----	
663	D2270	104		0.08	104	
823	D2270	105		1.48	105	
862	D2270	104		0.08	104	
875	D2270	103.78		-0.23	103.78	
902		----		----	----	
912	D2270	105		1.48	105	
922	D2270	104.4		0.64	104.5	
963	D2270	100	G(0.05)	-5.52	99	
994	D2270	102.44		-2.11	102.33	
1013		----		----	----	
1017		----		----	----	
1023		----		----	103.9	
1059	ISO2909	103		-1.32	103	
1106		----		----	----	
1146	D2270	104.09		0.20	104.11	
1155		----		----	106.0	
1161	D2270	105		1.48	105	
1173		----		----	----	
1201	D2270	103		-1.32	103	
1203		----		----	105.5	
1231	D2270	104		0.08	104	
1243		----		----	----	
1262	D2270	105		1.48	105	
1271	ISO2909	102		-2.72	102	
1316	D2270	104		0.08	104	
1358		----		----	----	
1396		----		----	----	
1402		----		----	----	
1428	D2270	104		0.08	104	
1431	D2270	104		0.08	104	
1435		----		----	394.4	
1480	D2270	96.350	C,G(0.01)	-10.63	96.774	first reported: 111.224
1495		----		----	----	
1569	D2270	105		1.48	105	
1571	D2270	104.327		0.53	----	
1579	ISO2909	102.79		-1.62	103.41	
1622	D2270	104		0.08	104	
1648	D2270	103.3		-0.90	103.4	
1660	D2270	104		0.08	104	
1680		----		----	----	
1704	D2270	103.9		-0.06	104.6	

1720		----		----	----
1722		----		----	----
1730		----		----	----
1740	D2270	105.7		2.46	105.7
1800		----		----	----
1827		----		----	----
1833	D2270	104		0.08	----
1842		----		----	----
1850	ISO2909	104		0.08	----
1854	D2270	102.8		-1.60	----
1900	D2270	103		-1.32	103
1903		----		----	----
1915	D2270	102	ex	-2.72	106 result excluded, see §4.1
2122		----		----	----
3166		----		----	----

normality not OK
n 49
outliers 3
mean (n) 103.95
st.dev. (n) 1.007
R(calc.) 2.82
R(D2270:10) 2.00

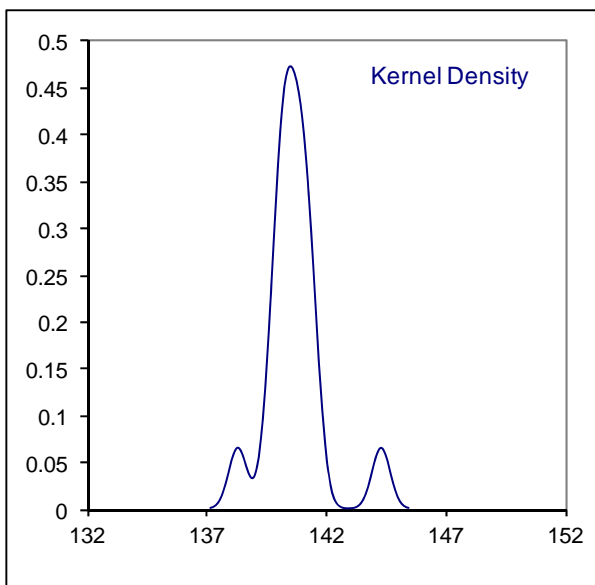
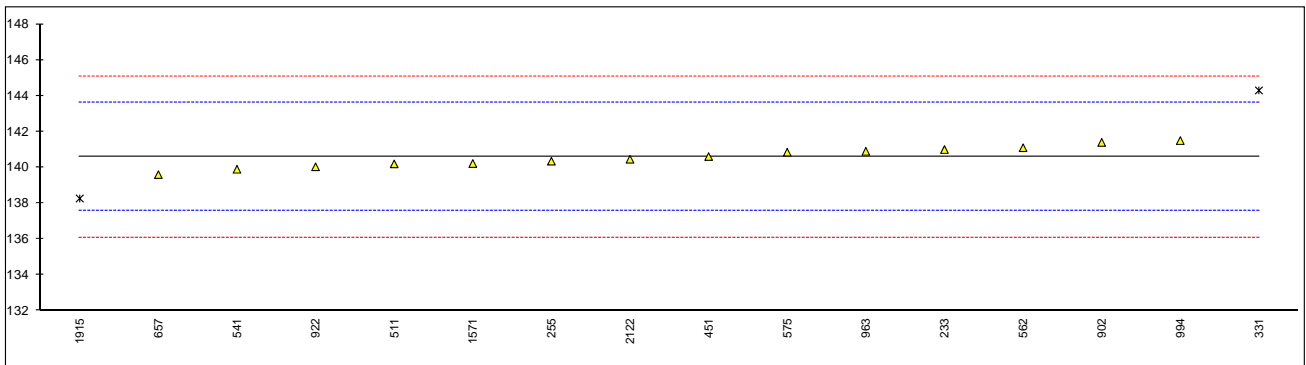


Determination of Kinematic Viscosity @ 40°C (Houillon) on sample #13063; results in mm²/s

lab	method	value	mark	z(targ)	remarks
230		----		----	
233	D7279	141.0		0.28	
237		----		----	
252		----		----	
254		----		----	
255	D7279	140.35		-0.15	
260		----		----	
311		----		----	
315		----		----	
325		----		----	
331	D7279	144.3	G(0.01)	2.47	
340		----		----	
343		----		----	
349		----		----	
360		----		----	
398		----		----	
420		----		----	
432		----		----	
450		----		----	
451	D7279	140.61		0.02	
473		----		----	
496		----		----	
511	D7279	140.20		-0.25	
541	D7042	139.9		-0.45	
551		----		----	
562	D7279	141.1		0.34	
575	D7279	140.85		0.18	
593		----		----	
603		----		----	
608		----		----	
609		----		----	
614		----		----	
633		----		----	
657	D7279	139.6		-0.65	
663		----		----	
823		----		----	
862		----		----	
875		----		----	
902	D7279	141.4		0.54	
912		----		----	
922	D7042	140.04		-0.36	
963	D7042	140.9		0.21	
994	D7042	141.5		0.61	
1013		----		----	
1017		----		----	
1023		----		----	
1059		----		----	
1106		----		----	
1146		----		----	
1155		----		----	
1161		----		----	
1173		----		----	
1201		----		----	
1203		----		----	
1231		----		----	
1243		----		----	
1262		----		----	
1271		----		----	
1316		----		----	
1358		----		----	
1396		----		----	
1402		----		----	
1428		----		----	
1431		----		----	
1435		----		----	
1480		----		----	
1495		----		----	
1569		----		----	
1571	D7042	140.22		-0.24	
1579		----		----	
1622		----		----	
1648		----		----	
1660		----		----	
1680		----		----	
1704		----		----	

1720		----		----
1722		----		----
1730		----		----
1740		----		----
1800		----		----
1827		----		----
1833		----		----
1842		----		----
1850		----		----
1854		----		----
1900		----		----
1903		----		----
1915	D7042	138.27	G(0.05)	-1.53
2122	in house	140.46		-0.08
3166		----		----

normality OK
 n 14
 outliers 2
 mean (n) 140.581
 st.dev. (n) 0.5682
 R(calc.) 1.591
 R(D7279:08) 4.217

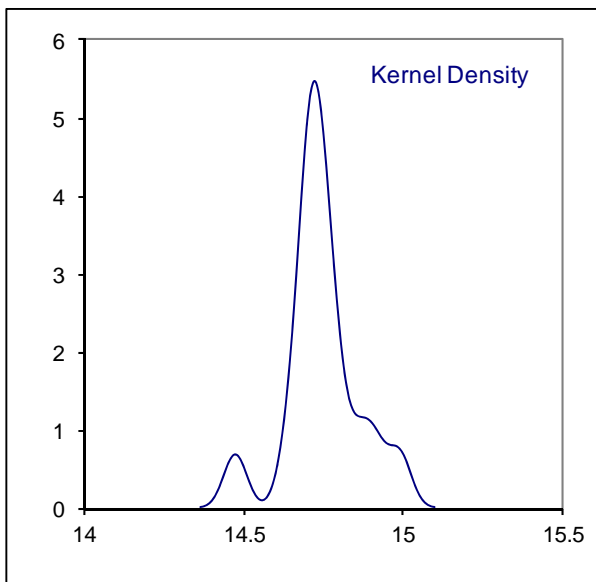
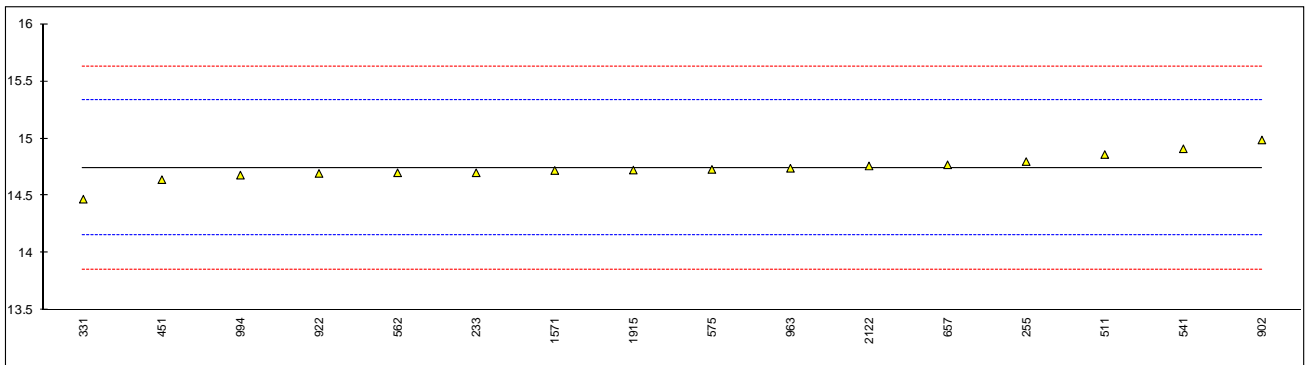


Determination of Kinematic Viscosity @ 100°C (Houillon) on sample #13063; results in mm²/s

lab	method	value	mark	z(targ)	remarks
230		----		----	
233	D7279	14.70		-0.15	
237		----		----	
252		----		----	
254		----		----	
255	D7279	14.798		0.19	
260		----		----	
311		----		----	
315		----		----	
325		----		----	
331	D7279	14.47		-0.93	
340		----		----	
343		----		----	
349		----		----	
360		----		----	
398		----		----	
420		----		----	
432		----		----	
450		----		----	
451	D7279	14.64		-0.35	
473		----		----	
496		----		----	
511	D7279	14.86		0.40	
541	D7042	14.91		0.57	
551		----		----	
562	D7279	14.70		-0.15	
575	D7279	14.73		-0.04	
593		----		----	
603		----		----	
608		----		----	
609		----		----	
614		----		----	
633		----		----	
657	D7279	14.77		0.09	
663		----		----	
823		----		----	
862		----		----	
875		----		----	
902	D7279	14.987		0.83	
912		----		----	
922	D7042	14.695		-0.16	
963	D7042	14.74		-0.01	
994	D7042	14.68		-0.21	
1013		----		----	
1017		----		----	
1023		----		----	
1059		----		----	
1106		----		----	
1146		----		----	
1155		----		----	
1161		----		----	
1173		----		----	
1201		----		----	
1203		----		----	
1231		----		----	
1243		----		----	
1262		----		----	
1271		----		----	
1316		----		----	
1358		----		----	
1396		----		----	
1402		----		----	
1428		----		----	
1431		----		----	
1435		----		----	
1480		----		----	
1495		----		----	
1569		----		----	
1571	D7042	14.7205		-0.08	
1579		----		----	
1622		----		----	
1648		----		----	
1660		----		----	
1680		----		----	
1704		----		----	

1720	----	----	
1722	----	----	
1730	----	----	
1740	----	----	
1800	----	----	
1827	----	----	
1833	----	----	
1842	----	----	
1850	----	----	
1854	----	----	
1900	----	----	
1903	----	----	
1915	D7042	14.724	-0.06
2122	in house	14.76	0.06
3166	----	----	

normality OK
n 16
outliers 0
mean (n) 14.743
st.dev. (n) 0.1158
R(calc.) 0.324
R(D7279:08) 0.826

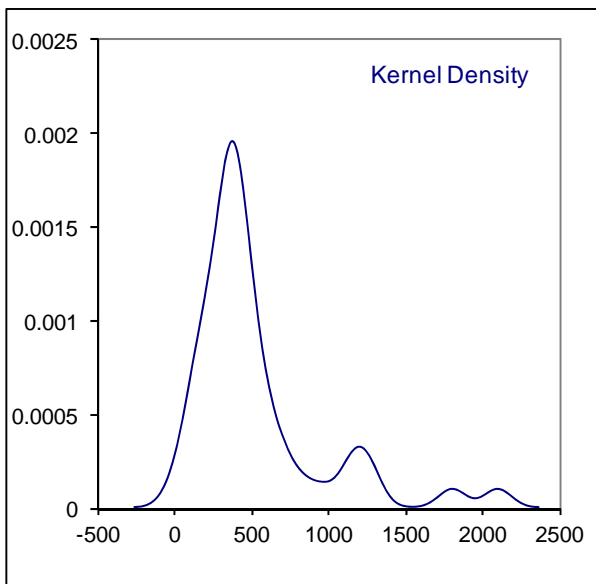
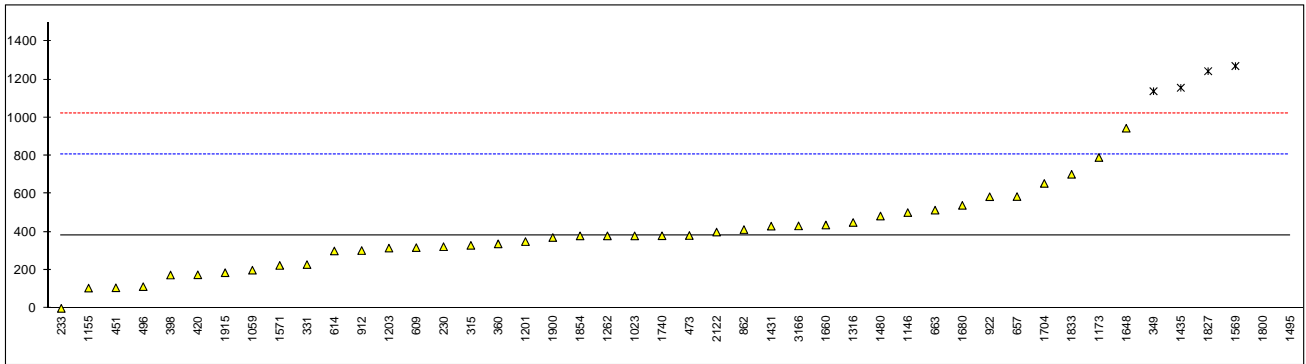


Determination of Water on sample #13063; results in mg/kg

lab	method	value	mark	z(targ)	remarks
230	D6304A	323.1		-0.27	
233	in house	0.02	C	-1.79	first reported: 0.01 mg/kg
237		----		----	
252		----		----	
254		----		----	
255		----		----	
260		----		----	
311		----		----	
315	D6304C	330.34		-0.24	
325		----		----	
331	D6304	229.5		-0.71	
340		----		----	
343		----		----	
349	D6304	1138	DG(0.01)	3.55	
360	D6304C	338.0		-0.20	
398	D6304C	174.6		-0.97	
420	ISO12937	176		-0.96	
432		----		----	
450		----		----	
451	D6304C	108		-1.28	
473	D6304C	381.8		0.00	
496	D6304C	113.5		-1.25	
511		----		----	
541		----		----	
551		----		----	
562		----		----	
575		----		----	
593		----		----	
603		----		----	
608		----		----	
609	D6304C	318.3		-0.29	
614	D6304C	301		-0.38	
633		----		----	
657	D6304C	586		0.96	
663	D6304A	514.9		0.63	
823		----		----	
862	D6304C	412.8		0.15	
875		----		----	
902		----		----	
912	D6304C	303		-0.37	
922	D6304A	585.3		0.96	
963		----		----	
994		----		----	
1013		----		----	
1017		----		----	
1023	D6304A	380		0.00	
1059	D6304A	200		-0.85	
1106		----		----	
1146	D6304C	502		0.57	
1155	D6304C	105.97		-1.29	
1161		----		----	
1173	in house	790.95		1.92	
1201	D6304	350	C	-0.15	probably unit error, reported: 0.035 mg/kg
1203	D6304A	316		-0.30	
1231		----		----	
1243		----		----	
1262	D6304A	380		0.00	
1271		----		----	
1316	D6304C	450		0.32	
1358		----		----	
1396		----		----	
1402		----		----	
1428		----		----	
1431	D6304	431		0.23	
1435	D1744	1156	DG(0.01)	3.63	
1480	D6304A	484		0.48	
1495	E203	2099.7	G(0.01)	8.06	
1569	D6304C	1270	DG(0.05)	4.17	
1571	D6304	225		-0.73	
1579		----		----	
1622		----		----	
1648	D6304	944.0		2.64	
1660	IEC60814	437		0.26	
1680	ISO8534	540		0.75	
1704	D6304	654.75		1.28	

1720		----		----
1722		----		----
1730		----		----
1740	D6304A	380.5		0.00
1800	D6304Mod.	1803.531	G(0.01)	6.67
1827	D6304A	1243	DG(0.05)	4.04
1833	D6304	702.3486		1.51
1842		----		----
1850		----		----
1854	D6304C	380		0.00
1900	D6304C	371.5		-0.04
1903		----		----
1915	D6304	187		-0.91
2122	in house	400		0.09
3166	D4017	432.1		0.24

			<u>Only ASTM D6304C data</u>
normality	OK		OK
n	40		16
outliers	6		1
mean (n)	381.01		323.551
st.dev. (n)	191.384		140.210
R(calc.)	535.87		392.59
R(D6304:07)	597.36		541.55



Determination of Aluminium (Al) on sample #13064; results in mg/kg

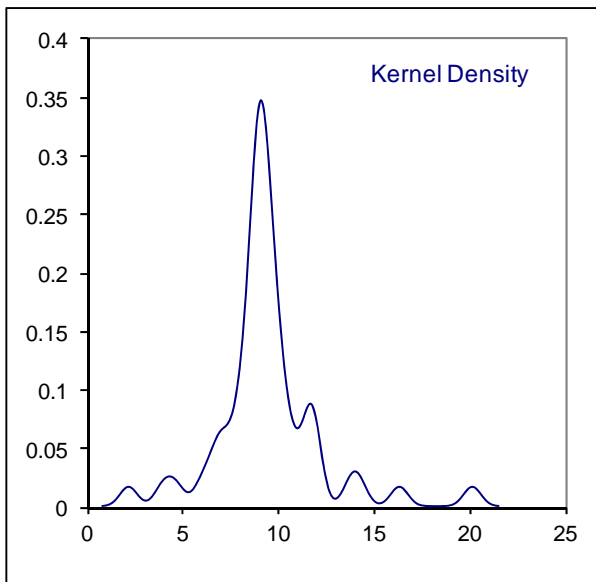
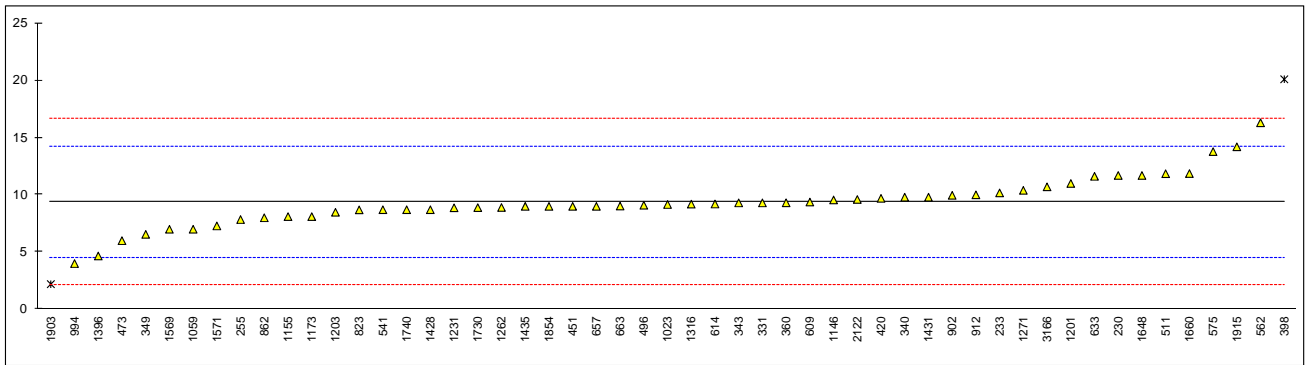
lab	method	value	mark	z(targ)	remarks
230	D6595	11.7		0.97	
233	D6595	10.17		0.34	
237		----		----	
252		----		----	
254		----		----	
255	INH-1	7.845		-0.62	
260		----		----	
311		----		----	
315		----		----	
325		----		----	
331	D5185	9.3		-0.02	
340	D5185	9.8		0.18	
343	D5185	9.3		-0.02	
349	D5185	6.55		-1.16	
360	D5185	9.31		-0.02	
398	D6595	20.1	G(0.01)	4.44	
420	INH-207	9.7		0.14	
432		----		----	
450		----		----	
451	D5185	9		-0.15	
473	D5185	6		-1.39	
496	D5185	9.1		-0.11	
511	D6595	11.85		1.03	
541	D5185	8.7		-0.27	
551		----		----	
562	D6595	16.3		2.87	
575	D6595	13.778		1.83	
593		----		----	
603		----		----	
608		----		----	
609	D5185	9.374		0.01	
614	D5185	9.2		-0.06	
633	D6595	11.62		0.94	
657	D5185	9		-0.15	
663	D5185	9.03		-0.13	
823	D5185	8.693		-0.27	
862	D5185	8		-0.56	
875		----		----	
902	D5185	9.966		0.25	
912	D5185	10		0.27	
922		----		----	
963		----		----	
994	D5185	4.00		-2.22	
1013		----		----	
1017		----		----	
1023	D5185	9.16		-0.08	
1059	in house	7		-0.97	
1106		----		----	
1146	D5185	9.561		0.08	
1155	D5185	8.10		-0.52	
1161		----		----	
1173	in house	8.1		-0.52	
1201	D5185	11		0.68	
1203	D5185	8.48		-0.36	
1231	D5185	8.868		-0.20	
1243		----		----	
1262	D5185	8.9009		-0.19	
1271	D5185	10.4		0.43	
1316	D5185	9.19		-0.07	
1358		----		----	
1396	INH-12	4.66		-1.94	
1402		----		----	
1428	D5185	8.7		-0.27	
1431	in house	9.8		0.18	
1435	D5185	9		-0.15	
1480		----		----	
1495		----		----	
1569	D5185	7		-0.97	
1571	D5185	7.2866		-0.86	
1579		----		----	
1622		----		----	
1648	D5185	11.7		0.97	
1660	D5185	11.86		1.04	
1680		----		----	
1704		----	W	----	result withdrawn, first reported: in house, 20.3

1720		----	----
1722		----	----
1730	D5185	8.884	-0.20
1740	D5185	8.7	-0.27
1800		----	----
1827		----	----
1833		W	----
1842		----	----
1850		----	----
1854	D5185	9	-0.15
1900		----	----
1903	in house	2.2	G(0.05)
1915	D5185	14.2	2.00
2122	D5185	9.6	0.10
3166	EPA6020	10.7	0.56

result withdrawn, first reported: IP 501, 20.4481

normality	not OK	<u>only D6595:05 data</u>	OK
n	51		7
outliers	2		0
mean (n)	9.36		13.65
st.dev. (n)	2.117		3.462
R(calc.)	5.93		9.69
R(D5185:09)	6.77		7.50

application range: 6 - 40 mg/kg



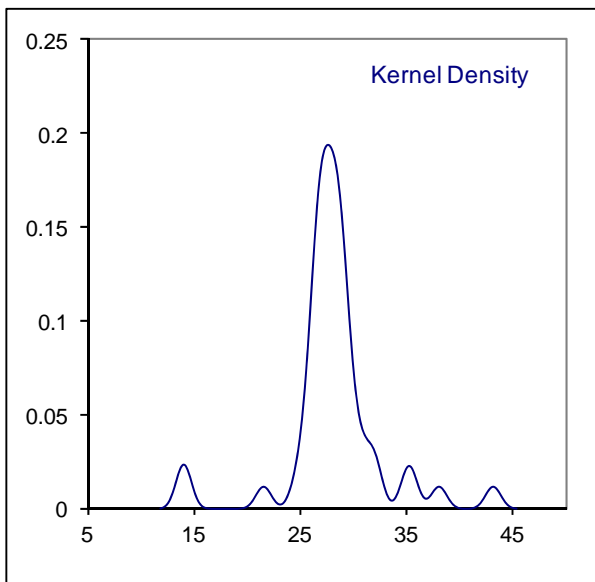
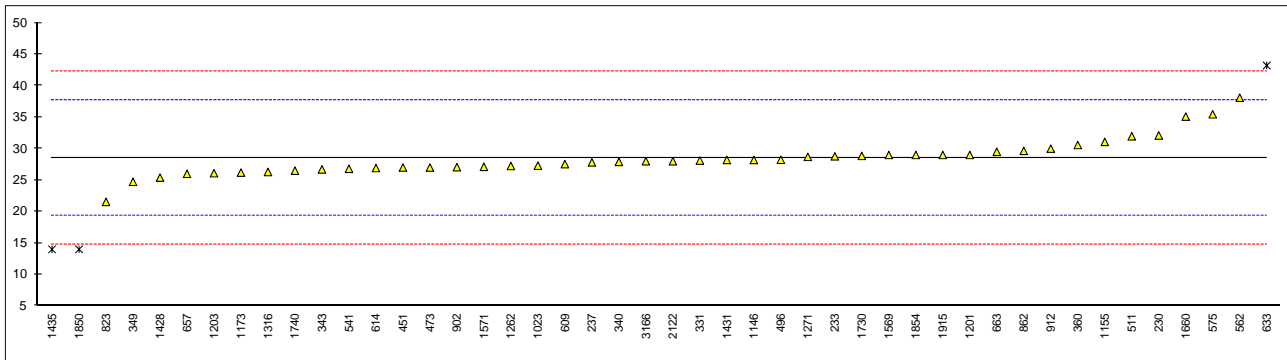
Determination of Barium (Ba) on sample #13064; results in mg/kg

lab	method	value	mark	z(targ)	remarks
230	D6595	32.1		0.79	
233	D6595	28.78		0.07	
237	D5185	27.825		-0.14	
252		----		----	
254		----		----	
255		----		----	
260		----		----	
311		----		----	
315		----		----	
325		----		----	
331	D5185	28.1		-0.08	
340	D5185	27.9		-0.12	
343	D5185	26.7		-0.38	
349	D5185	24.73		-0.81	
360	D5185	30.58		0.46	
398		----		----	
420		----		----	
432		----		----	
450		----		----	
451	D5185	27		-0.32	
473	D5185	27		-0.32	
496	D5185	28.24		-0.05	
511	D6595	31.97		0.77	
541	D5185	26.8		-0.36	
551		----		----	
562	D6595	38.1		2.10	
575	D6595	35.464		1.53	
593		----		----	
603		----		----	
608		----		----	
609	D5185	27.538		-0.20	
614	D5185	26.92		-0.34	
633	D6595	43.21	G(0.05)	3.22	
657	D5185	26		-0.54	
663	D5185	29.50		0.23	
823	D5185	21.54		-1.51	
862	D5185	29.65		0.26	
875		----		----	
902	D5185	27.054		-0.31	
912	D5185	30		0.34	
922		----		----	
963		----		----	
994		----		----	
1013		----		----	
1017		----		----	
1023	D5185	27.3		-0.25	
1059		----		----	
1106		----		----	
1146	D5185	28.21		-0.05	
1155	D5185	31.07		0.57	
1161		----		----	
1173	in house	26.2		-0.49	
1201	D5185	29		0.12	
1203	D5185	26.1		-0.51	
1231		----		----	
1243		----		----	
1262	D5185	27.251		-0.26	
1271	D5185	28.7		0.05	
1316	D5185	26.3		-0.47	
1358		----		----	
1396		----		----	
1402		----		----	
1428	D5185	25.4		-0.67	
1431	in house	28.2		-0.06	
1435	D5185	14	G(0.05)	-3.15	
1480		----		----	
1495		----		----	
1569	D5185	29		0.12	
1571	D5185	27.1156		-0.29	
1579		----		----	
1622		----		----	
1648		----		----	
1660	D5185	35.1		1.45	
1680		----		----	
1704		----		----	

1720		----		----
1722		----		----
1730	D5185	28.848		0.08
1740	D5185	26.5		-0.43
1800		----		----
1827		----		----
1833		----		----
1842		----		----
1850	in house	14	G(0.01)	-3.15
1854	D5185	29		0.12
1900		----		----
1903	in house	<1		<-5.85 false negative?
1915	D5185	29.0		0.12
2122	D5185	28.0		-0.10
3166	EPA6020	28.0		-0.10

normality not OK
n 43
outliers 3
mean (n) 28.46
st.dev. (n) 2.875
R(calc.) 8.05
R(D5185:09) 12.85

application range: 0.5 - 4 mg/kg



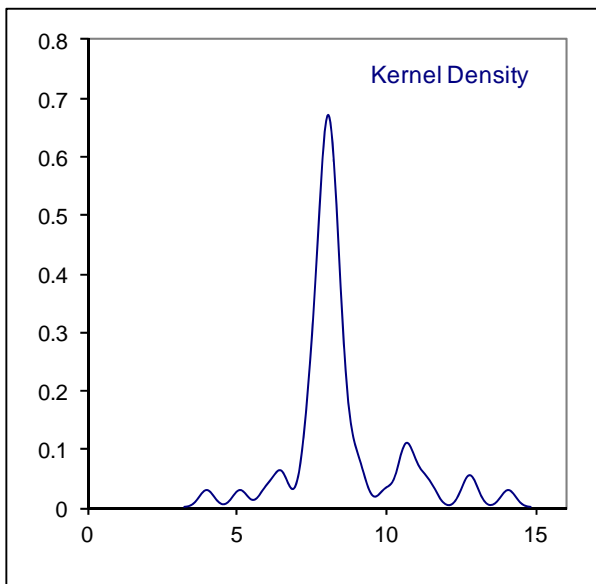
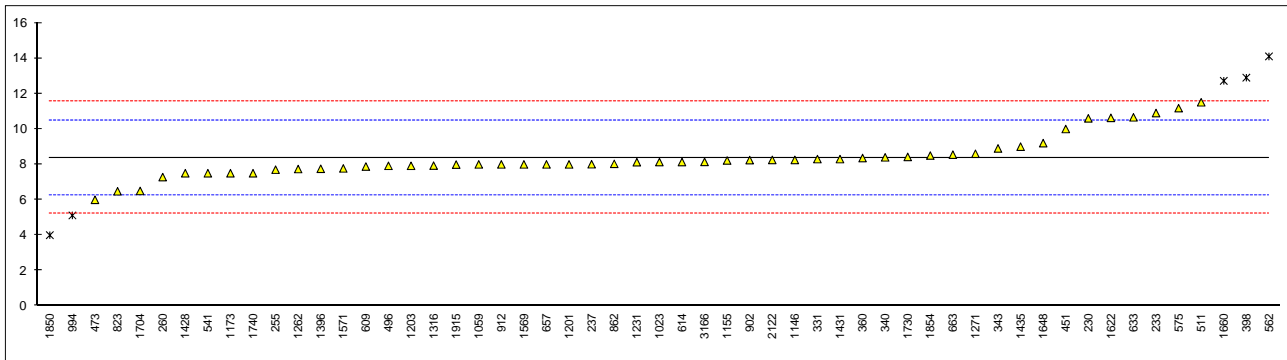
Determination of Chromium (Cr) on sample #13064; results in mg/kg

lab	method	value	mark	z(targ)	remarks
230	D6595	10.6		2.11	
233	D6595	10.90		2.40	
237	D5185	8.009		-0.34	
252		----		----	
254		----		----	
255	INH-1	7.7035		-0.63	
260	D5185	7.28		-1.03	
311		----		----	
315		----		----	
325		----		----	
331	D5185	8.3		-0.06	
340	D5185	8.4		0.03	
343	D5185	8.9		0.50	
349		----		----	
360	D5185	8.36		-0.01	
398	D6595	12.9	DG(0.05)	4.29	
420		----		----	
432		----		----	
450		----		----	
451	D5185	10		1.54	
473	D5185	6		-2.24	
496	D5185	7.92		-0.42	
511	D6595	11.51		2.97	
541	D5185	7.5		-0.82	
551		----		----	
562	D6595	14.1	G(0.05)	5.42	
575	D6595	11.177		2.66	
593		----		----	
603		----		----	
608		----		----	
609	D5185	7.877		-0.46	
614	D5185	8.13		-0.23	
633	D6595	10.66		2.17	
657	D5185	8		-0.35	
663	D5185	8.55		0.17	
823	D5185	6.481		-1.78	
862	D5185	8.03		-0.32	
875		----		----	
902	D5185	8.242		-0.12	
912	D5185	8		-0.35	
922		----		----	
963		----		----	
994	D5185	5.117	C,DG(0.05)	-3.08	first reported: 2.09
1013		----		----	
1017		----		----	
1023	D5185	8.13		-0.23	
1059	in house	8		-0.35	
1106		----		----	
1146	D5185	8.250		-0.11	
1155	D5185	8.22		-0.14	
1161		----		----	
1173	in house	7.5		-0.82	
1201	D5185	8		-0.35	
1203	D5185	7.92		-0.42	
1231	D5185	8.122		-0.23	
1243		----		----	
1262	D5185	7.7376		-0.60	
1271	D5185	8.6		0.22	
1316	D5185	7.93		-0.41	
1358		----		----	
1396	INH-12	7.75		-0.58	
1402		----		----	
1428	D5185	7.5		-0.82	
1431	in house	8.3		-0.06	
1435	D5185	9		0.60	
1480		----		----	
1495		----		----	
1569	D5185	8		-0.35	
1571	D5185	7.7746		-0.56	
1579		----		----	
1622	D5185	10.63		2.14	
1648	D5185	9.20		0.79	
1660	D5185	12.72	DG(0.05)	4.12	
1680		----		----	
1704	in house	6.5		-1.77	

1720		----	----		
1722		----	----		
1730	D5185	8.424	0.05		
1740	D5185	7.5	-0.82		
1800		----	----		
1827		----	----		
1833		----	----		
1842		----	----		
1850	in house	4	DG(0.05)	-4.13	
1854	D5185	8.5		0.12	
1900		----	----		
1903	in house	<1		<-6.83	false negative?
1915	D5185	7.99		-0.36	
2122	D5185	8.25		-0.11	
3166	EPA6020	8.14		-0.22	

normality	not OK	<u>only D6595:05 data</u>
n	50	OK
outliers	5	7
mean (n)	8.368	11.69
st.dev. (n)	1.1455	1.319
R(calc.)	3.207	3.69
R(D5185:09)	2.960	3.62

application range: 1 – 40 mg/kg



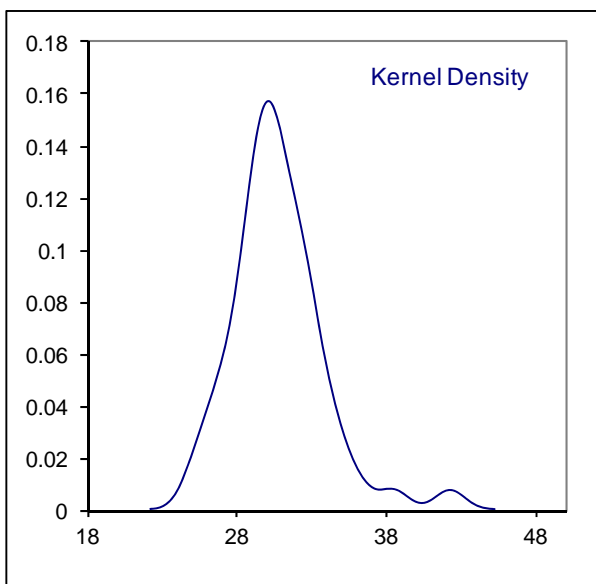
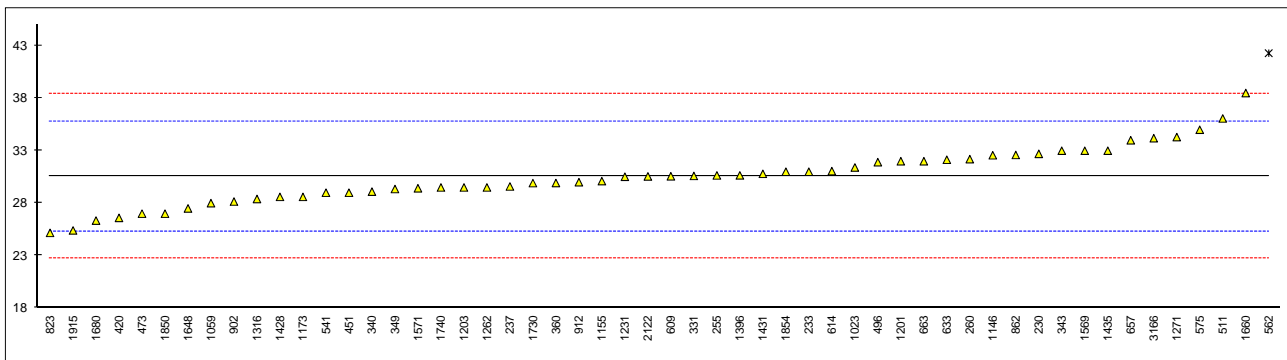
Determination of Copper (Cu) on sample #13064; results in mg/kg

lab	method	value	mark	z(targ)	remarks
230	D6595	32.7		0.82	
233	D6595	31.00		0.18	
237	D5185	29.595		-0.36	
252		----		----	
254		----		----	
255	INH-1	30.649		0.04	
260	D5185	32.20		0.63	
311		----		----	
315		----		----	
325		----		----	
331	D5185	30.6		0.02	
340	D5185	29.1		-0.55	
343	D5185	33		0.94	
349	D5185	29.35		-0.45	
360	D5185	29.92		-0.24	
398		----		----	
420	DIN51404	26.6		-1.51	
432		----		----	
450		----		----	
451	D5185	29		-0.59	
473	D5185	27		-1.35	
496	D5185	31.91		0.52	
511	D6595	36.08		2.12	
541	D5185	29		-0.59	
551		----		----	
562	D6595	42.3	G(0.01)	4.49	
575	D6595	34.999		1.70	
593		----		----	
603		----		----	
608		----		----	
609	D5185	30.563		0.01	
614	D5185	31.06		0.20	
633	D6595	32.14		0.61	
657	D5185	34		1.32	
663	D5185	32.0		0.56	
823	D5185	25.17		-2.05	
862	D5185	32.6		0.79	
875		----		----	
902	D5185	28.157		-0.91	
912	D5185	30		-0.21	
922		----		----	
963		----		----	
994		----		----	
1013		----		----	
1017		----		----	
1023	D5185	31.4		0.33	
1059	in house	28		-0.97	
1106		----		----	
1146	D5185	32.57		0.78	
1155	D5185	30.10		-0.17	
1161		----		----	
1173	in house	28.6		-0.74	
1201	D5185	32		0.56	
1203	D5185	29.5		-0.40	
1231	D5185	30.52		-0.01	
1243		----		----	
1262	D5185	29.500	C	-0.40	first reported: 39.500
1271	D5185	34.3		1.44	
1316	D5185	28.4		-0.82	
1358		----		----	
1396	INH-12	30.65		0.04	
1402		----		----	
1428	D5185	28.6		-0.74	
1431	in house	30.8		0.10	
1435	D5185	33		0.94	
1480		----		----	
1495		----		----	
1569	D5185	33		0.94	
1571	D5185	29.4172		-0.43	
1579		----		----	
1622		----		----	
1648	D5185	27.5		-1.16	
1660	D5185	38.5		3.04	
1680	EN14107	26.34		-1.60	
1704		----	W	----	result withdrawn, first reported: 15.3

1720		----	----
1722		----	----
1730	D5185	29.915	-0.24
1740	D5185	29.5	-0.40
1800		----	----
1827		----	----
1833		----	----
1842		----	----
1850	in house	27	-1.35
1854	D5185	31	0.18
1900		----	----
1903	in house	<1	<-11.08 false negative?
1915	D5185	25.4	-1.96
2122	D5185	30.55	0.00
3166	EPA6020	34.2	1.40

normality OK
n 53
outliers 1
mean (n) 30.541
st.dev. (n) 2.6292
R(calc.) 7.362
R(D5185:09) 7.330

application range: 2 – 160 mg/kg



Determination of Iron (Fe) on sample #13064; results in mg/kg

lab	method	value	mark	z(targ)	remarks
230	D6595	25.5		1.14	
233	D6595	26.18		1.44	
237	D5185	22.245		-0.29	
252		----		----	
254		----		----	
255	INH-1	23.555		0.29	
260	D5185	39.09	G(0.01)	7.12	
311		----		----	
315		----		----	
325		----		----	
331	D5185	21.5		-0.62	
340	D5185	22.9		0.00	
343	D5185	24		0.48	
349	D5185	19.17		-1.64	
360	D5185	23.53		0.27	
398	D6595	36.9	G(0.01)	6.15	
420	DIN51397	22.7		-0.09	
432		----		----	
450		----		----	
451	D5185	23		0.04	
473	D5185	19		-1.72	
496	D5185	22.28		-0.27	
511	D6595	23.80		0.39	
541	D5185	21		-0.84	
551		----		----	
562	D6595	29.5		2.90	
575	D6595	27.089		1.84	
593		----		----	
603		----		----	
608		----		----	
609	D5185	22.433		-0.21	
614	D5185	23.61		0.31	
633	D6595	27.90		2.20	
657	D5185	21		-0.84	
663	D5185	23.9		0.44	
823	D5185	18.14		-2.10	
862	D5185	24.6		0.75	
875		----		----	
902	D5185	25.157		0.99	
912		----		----	
922		----		----	
963		----		----	
994	D5185	22.23	C	-0.30	first reported: 15.94
1013		----	W	----	result withdrawn, first reported: 11
1017		----		----	
1023	D5185	22.1		-0.35	
1059	in house	24		0.48	
1106		----		----	
1146	D5185	22.84		-0.03	
1155	D5185	28.46		2.44	
1161		----		----	
1173	in house	21.0		-0.84	
1201	D5185	23		0.04	
1203	D5185	22.0		-0.40	
1231	D5185	22.23		-0.30	
1243		----		----	
1262	D5185	21.471		-0.63	
1271	D5185	24.6		0.75	
1316	D5185	21.7		-0.53	
1358		----		----	
1396	INH-12	22.43		-0.21	
1402		----		----	
1428	D5185	20.2		-1.19	
1431	in house	24.0		0.48	
1435	D5185	23		0.04	
1480		----		----	
1495		----		----	
1569	D5185	24		0.48	
1571	D5185	22.0296		-0.39	
1579		----		----	
1622		----		----	
1648	D5185	26.2	C	1.45	first reported: 31.4
1660	D5185	32.5	G(0.05)	4.22	
1680	EN14107	19.99		-1.28	
1704		----	W	----	result withdrawn, first reported: 2.9

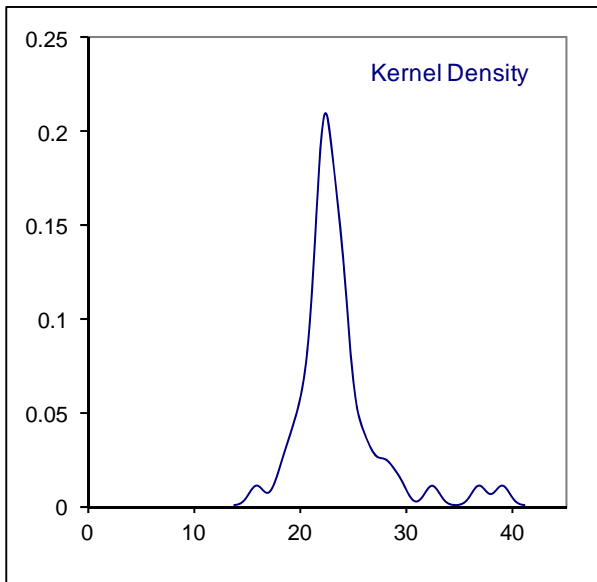
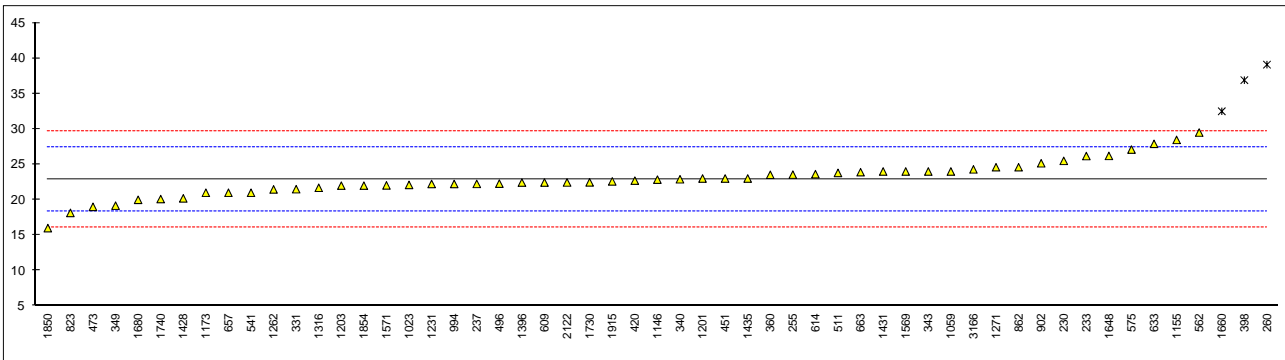
1720		----	----
1722		----	----
1730	D5185	22.454	-0.20
1740	D5185	20.1	-1.23
1800		----	----
1827		----	----
1833		----	----
1842		----	----
1850	in house	16	-3.04
1854	D5185	22	-0.40
1900		----	----
1903	in house	<1	<-9.45
1915	D5185	22.6	-0.13
2122	D5185	22.45	-0.20
3166	EPA6020	24.3	0.61

result withdrawn, first reported: 26.82175

false negative?

normality OK
 n 52
 outliers 3
 mean (n) 22.905
 st.dev. (n) 2.4704
 R(calc.) 6.917
 R(D5185:09) 6.367

application range: 2 – 140 mg/kg



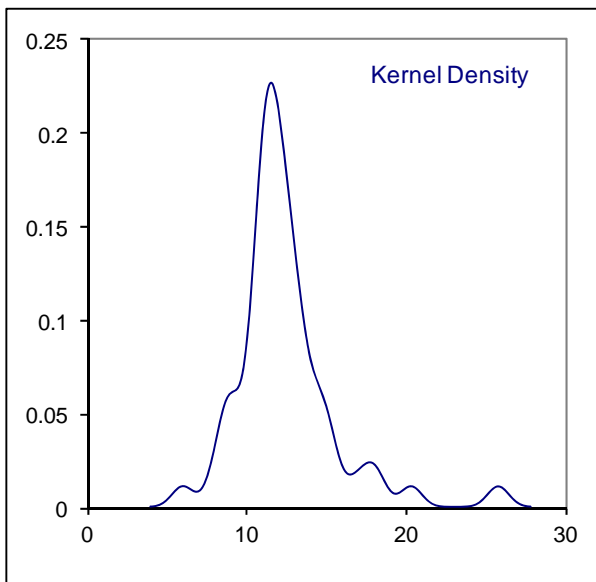
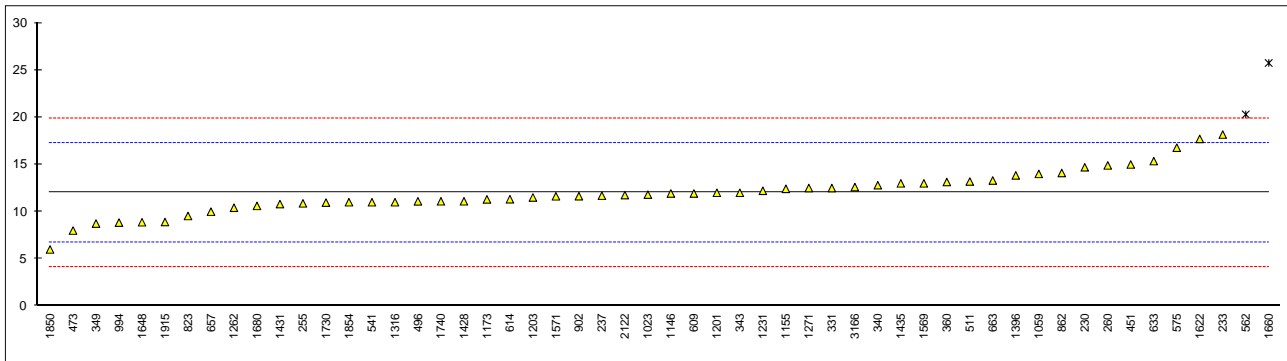
Determination of Lead (Pb) on sample #13064; results in mg/kg

lab	method	value	mark	z(targ)	remarks
230	D6595	14.7		1.03	
233	D6595	18.17		2.35	
237	D5185	11.695		-0.12	
252		----		----	
254		----		----	
255	INH-1	10.882		-0.43	
260	D5185	14.90		1.10	
311		----		----	
315		----		----	
325		----		----	
331	D5185	12.5		0.19	
340	D5185	12.8		0.30	
343	D5185	12		0.00	
349	D5185	8.74		-1.25	
360	D5185	13.14		0.43	
398		----		----	
420		----		----	
432		----		----	
450		----		----	
451	D5185	15		1.14	
473	D5185	8		-1.53	
496	D5185	11.09		-0.35	
511	D6595	13.19		0.45	
541	D5185	11		-0.39	
551		----		----	
562	D6595	20.3	G(0.05)	3.16	
575	D6595	16.777		1.82	
593		----		----	
603		----		----	
608		----		----	
609	D5185	11.921		-0.03	
614	D5185	11.31		-0.27	
633	D6595	15.36		1.28	
657	D5185	10		-0.77	
663	D5185	13.3		0.49	
823	D5185	9.543		-0.94	
862	D5185	14.1		0.80	
875		----		----	
902	D5185	11.633		-0.14	
912		----		----	
922		----		----	
963		----		----	
994	D5185	8.84	C	-1.21	first reported: 2.3
1013		----		----	
1017		----		----	
1023	D5185	11.8		-0.08	
1059	in house	14		0.76	
1106		----		----	
1146	D5185	11.92		-0.03	
1155	D5185	12.42		0.16	
1161		----		----	
1173	in house	11.3		-0.27	
1201	D5185	12		0.00	
1203	D5185	11.5		-0.19	
1231	D5185	12.21		0.08	
1243		----		----	
1262	D5185	10.413		-0.61	
1271	D5185	12.5		0.19	
1316	D5185	11.0		-0.39	
1358		----		----	
1396	INH-12	13.84		0.70	
1402		----		----	
1428	D5185	11.1		-0.35	
1431	in house	10.8		-0.46	
1435	D5185	13		0.38	
1480		----		----	
1495		----		----	
1569	D5185	13		0.38	
1571	D5185	11.6298		-0.15	
1579		----		----	
1622	D5185	17.71		2.17	
1648	D5185	8.88		-1.19	
1660	D5185	25.75	G(0.01)	5.24	
1680	EN14107	10.61		-0.53	
1704		----	W	----	result withdrawn, first reported:35.5

1720		----	----
1722		----	----
1730	D5185	10.956	-0.40
1740	D5185	11.1	-0.35
1800		----	----
1827		----	----
1833		----	----
1842		----	----
1850	in house	6	-2.29
1854	D5185	11	-0.39
1900		----	----
1903	in house	<1	<-4.12 false negative?
1915	D5185	8.9	-1.19
2122	D5185	11.75	-0.10
3166	EPA6020	12.6	0.22

normality OK
n 52
outliers 2
mean (n) 12.010
st.dev. (n) 2.2871
R(calc.) 6.404
R(D5185:09) 7.341

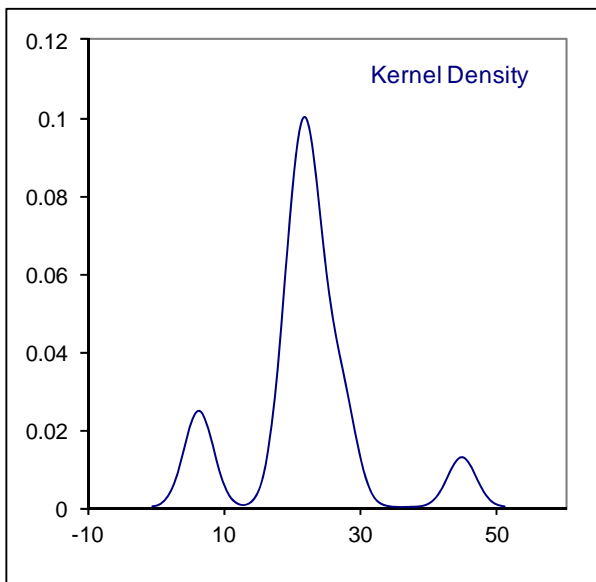
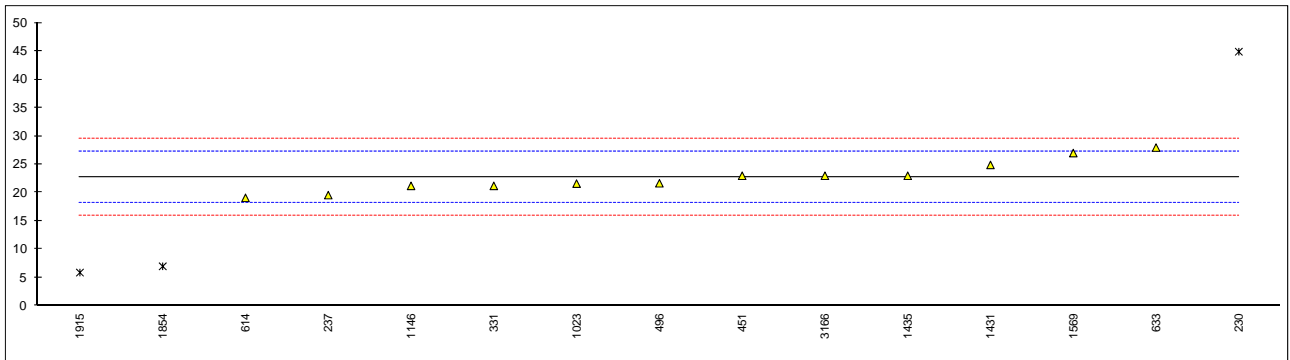
application range: 10 – 160 mg/kg



Determination of Lithium (Li) on sample #13064; results in mg/kg

lab	method	value	mark	z(targ)	remarks
230	D6595	44.9	G(0.05)	9.72	
233		----			
237	D5185	19.58		-1.40	
252		----			
254		----			
255		----			
260		----			
311		----			
315		----			
325		----			
331	D5185	21.2		-0.69	
340		----			
343		----			
349		----			
360		----			
398		----			
420		----			
432		----			
450		----			
451	D5185	23		0.10	
473		----			
496	INH-1824	21.67		-0.48	
511		----			
541		----			
551		----			
562		----			
575		----			
593		----			
603		----			
608		----			
609		----			
614	D5185	19.1		-1.61	
633	D6595	27.97		2.29	
657		----			
663		----			
823		----			
862		----			
875		----			
902		----			
912		----			
922		----			
963		----			
994		----			
1013		----			
1017		----			
1023	D5185	21.6		-0.51	
1059		----			
1106		----			
1146		21.20		-0.69	
1155		----			
1161		----			
1173		----			
1201		----			
1203		----			
1231		----			
1243		----			
1262		----			
1271		----			
1316		----			
1358		----			
1396		----			
1402		----			
1428		----			
1431	in house	24.9		0.94	
1435		23		0.10	
1480		----			
1495		----			
1569	in house	27		1.86	
1571		----			
1579		----			
1622		----			
1648		----			
1660		----			
1680		----			
1704		----			

1720		----	----	
1722		----	----	
1730		----	----	
1740		----	----	
1800		----	----	
1827		----	----	
1833		----	----	
1842		----	----	
1850		----	----	
1854	7	DG(0.01)	-6.93	
1900		----	----	
1903		----	----	
1915	D5184	5.9	DG(0.01)	-7.41
2122		----	----	
3166	EPA6020	23.0		0.10
normality	OK			
n	12			
outliers	3			
mean (n)	22.77			
st.dev. (n)	2.710			
R(calc.)	7.59			
R(Horwitz)	6.37			

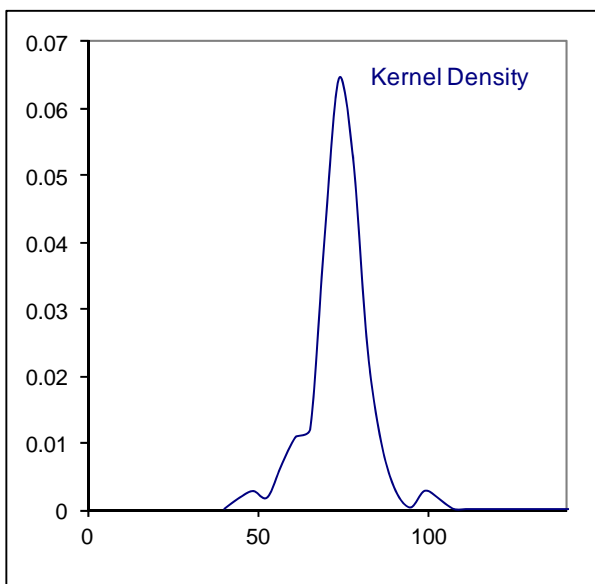
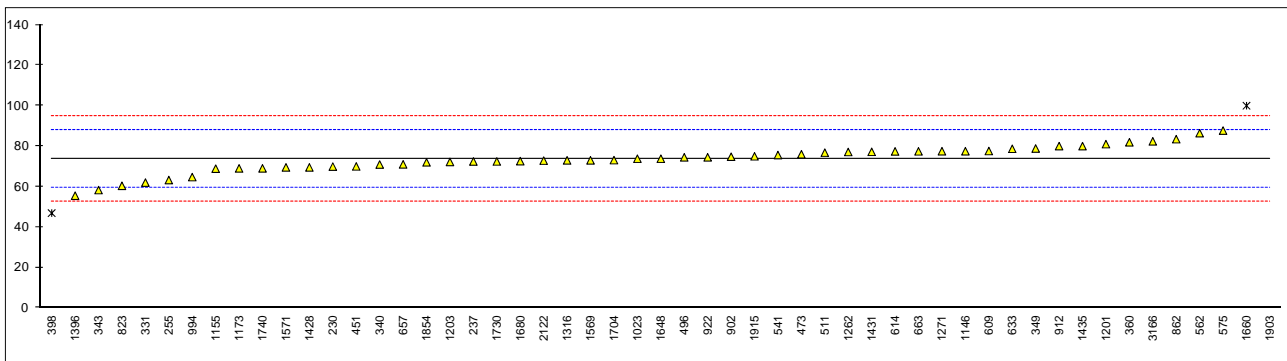


Determination of Magnesium (Mg) on sample #13064; results in mg/kg

lab	method	value	mark	z(targ)	remarks
230	D6595	69.9		-0.53	
233		----		----	
237	D5185	72.44		-0.17	
252		----		----	
254		----		----	
255	INH-1	63.256		-1.48	
260		----		----	
311		----		----	
315		----		----	
325		----		----	
331	D5185	62.0		-1.65	
340	D5185	70.9		-0.39	
343	D5185	58.3		-2.18	
349	D5185	78.85		0.74	
360	D5185	81.90		1.17	
398	D6595	46.9	G(0.05)	-3.80	
420		----		----	
432		----		----	
450		----		----	
451	D5185	70		-0.52	
473	D5185	76		0.33	
496	D5185	74.50		0.12	
511	D6595	76.81		0.45	
541	D5185	75.6		0.28	
551		----		----	
562	D6595	86.4		1.81	
575	D6595	87.687		1.99	
593		----		----	
603		----		----	
608		----		----	
609	D5185	77.602		0.56	
614	D5185	77.4		0.53	
633	D6595	78.70		0.72	
657	D5185	71		-0.38	
663	D5185	77.44		0.54	
823	D5185	60.42		-1.88	
862	D5185	83.45		1.39	
875		----		----	
902	D5185	74.769		0.16	
912	D5185	80		0.90	
922	D4628	74.5		0.12	
963		----		----	
994	D5185	64.71	C	-1.27	first reported: 44.27
1013		----		----	
1017		----		----	
1023	D5185	73.8		0.02	
1059		----		----	
1106		----		----	
1146	D5185	77.50		0.55	
1155	D5185	68.85		-0.68	
1161		----		----	
1173	in house	69.0		-0.66	
1201	D5185	81		1.04	
1203	D5185	72.2		-0.21	
1231		----		----	
1243		----		----	
1262	D5185	77.122		0.49	
1271	D5185	77.5		0.55	
1316	D5185	72.98		-0.09	
1358		----		----	
1396	INH-12	55.51		-2.57	
1402		----		----	
1428	D5185	69.5		-0.59	
1431	in house	77.2		0.50	
1435	D5185	80		0.90	
1480		----		----	
1495		----		----	
1569	D5185	73		-0.09	
1571	D5185	69.4941		-0.59	
1579		----		----	
1622		----	W	----	result withdrawn, first reported: 101.47
1648	D5185	73.8		0.02	
1660	D5185	100.0	G(0.05)	3.74	
1680	EN14107	72.60		-0.15	
1704	in house	73.1		-0.08	

1720		----	----
1722		----	----
1730	D5185	72.454	-0.17
1740	D5185	69	-0.66
1800		----	----
1827		----	----
1833		----	----
1842		----	----
1850		----	----
1854	D5185	72	-0.23
1900		----	----
1903	in house	1101.3	G(0.01) 145.87
1915	D5185	75.0	0.19
2122	D5185	72.85	-0.11
3166	EPA6020	82.4	1.24
normality	OK		
n	50		
outliers	3		
mean (n)	73.65		
st.dev. (n)	6.588		
R(calc.)	18.45		
R(D5185:09)	19.73		

application range: 5 – 1700 mg/kg

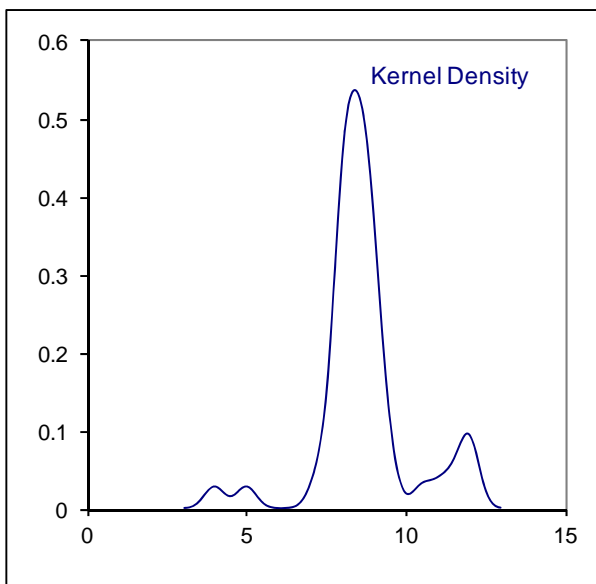
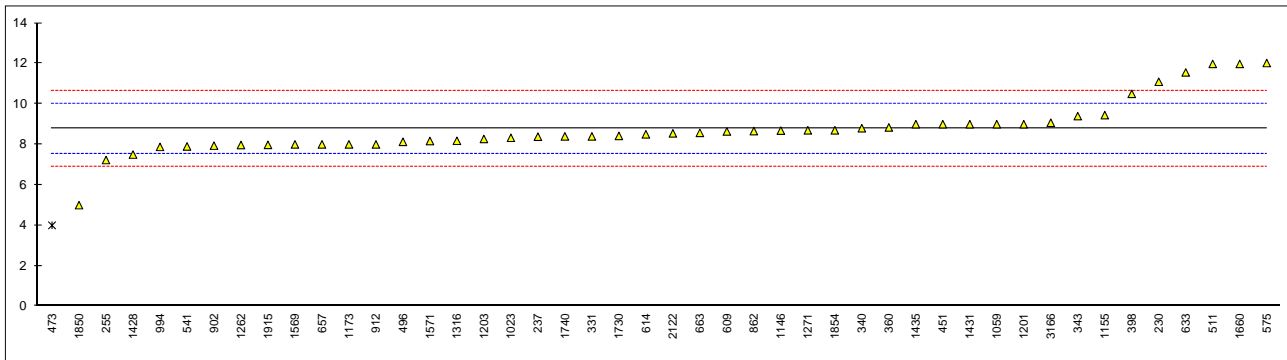


Determination of Manganese (Mn) on sample #13064; results in mg/kg

lab	method	value	mark	z(targ)	remarks
230	D6595	11.1		3.70	
233		----		----	
237	D5185	8.3815		-0.62	
252		----		----	
254		----		----	
255	INH-1	7.23		-2.45	
260		----		----	
311		----		----	
315		----		----	
325		----		----	
331	D5185	8.4		-0.59	
340	D5185	8.8		0.04	
343	D5185	9.4		1.00	
349		----		----	
360	D5185	8.84		0.11	
398	D6595	10.5		2.75	
420		----		----	
432		----		----	
450		----		----	
451	D5185	9		0.36	
473	D5185	4	G(0.05)	-7.59	
496	D5185	8.13		-1.02	
511	D6595	11.98		5.10	
541	D5185	7.9		-1.39	
551		----		----	
562		----		----	
575	D6595	12.025		5.17	
593		----		----	
603		----		----	
608		----		----	
609	D5185	8.64		-0.21	
614	D5185	8.5		-0.43	
633	D6595	11.56		4.43	
657	D5185	8		-1.23	
663	D5185	8.57		-0.32	
823		----		----	
862	D5185	8.66		-0.18	
875		----		----	
902	D5185	7.942		-1.32	
912	D5185	8		-1.23	
922		----		----	
963		----		----	
994	D5185	7.88	C	-1.42	first reported: 2.5
1013		----		----	
1017		----		----	
1023	D5185	8.33		-0.70	
1059	in house	9		0.36	
1106		----		----	
1146	D5185	8.681		-0.15	
1155	D5185	9.45		1.08	
1161		----		----	
1173	in house	8.0		-1.23	
1201	D5185	9		0.36	
1203	D5185	8.27		-0.80	
1231		----		----	
1243		----		----	
1262	D5185	7.9701		-1.28	
1271	D5185	8.7		-0.12	
1316	D5185	8.18		-0.94	
1358		----		----	
1396		----		----	
1402		----		----	
1428	D5185	7.5		-2.02	
1431	in house	9.0		0.36	
1435	D5185	9		0.36	
1480		----		----	
1495		----		----	
1569	D5185	8		-1.23	
1571	D5185	8.1701		-0.96	
1579		----		----	
1622		----	W	----	result withdrawn, first reported: 10.73
1648		----		----	
1660	D5185	11.98		5.10	
1680		----		----	
1704		----	W	----	result withdrawn, first reported: 6.0

1720		----	----
1722		----	----
1730	D5185	8.421	-0.56
1740	D5185	8.4	-0.59
1800		----	----
1827		----	----
1833		----	----
1842		----	----
1850	in house	5	-6.00
1854	D5185	8.7	-0.12
1900		----	----
1903	in house	<1	<-12.09 false negative?
1915	D5185	7.98	-1.26
2122	D5185	8.55	-0.35
3166	EPA6020	9.07	0.47
			<u>only D6595:05 data</u>
normality	not OK		OK
n	45		5
outliers	1		0
mean (n)	8.77		11.43
st.dev. (n)	1.306		0.642
R(calc.)	3.66		1.80
R(D5185:09)	1.76		2.42

application range: 5 -700 mg/kg



Determination of Molybdenum (Mo) on sample #13064; results in mg/kg

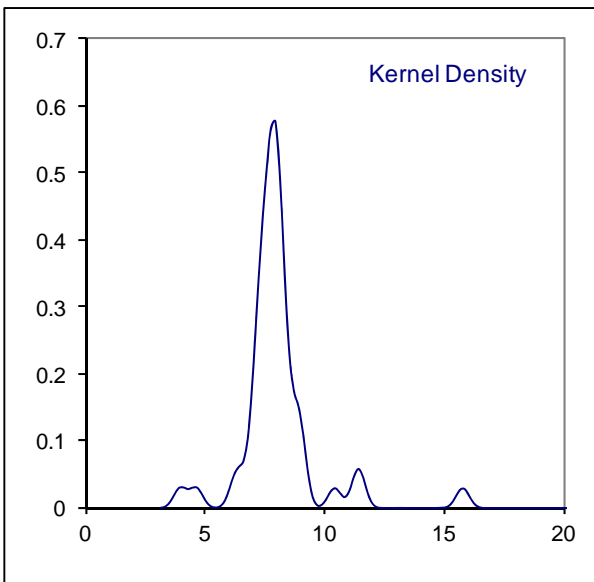
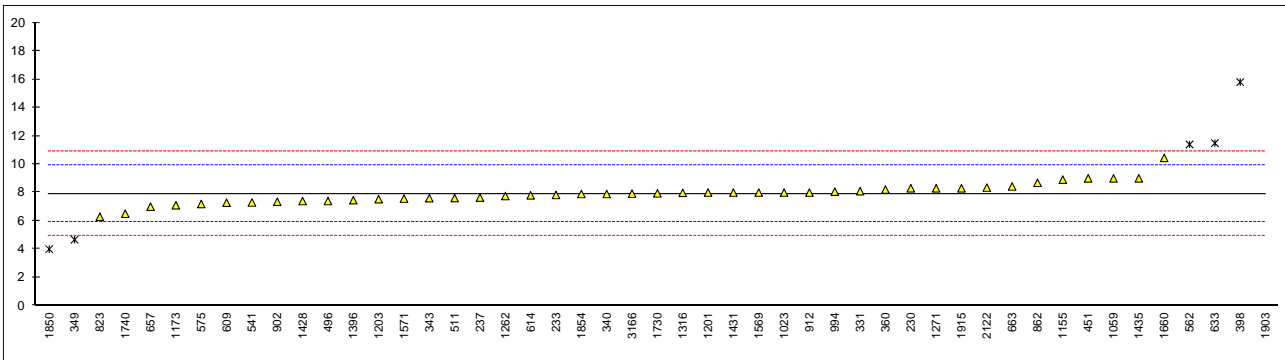
lab	method	value	mark	z(targ)	remarks
230	D6595	8.3		0.39	
233	D6595	7.83		-0.09	
237	D5185	7.641		-0.28	
252		----		----	
254		----		----	
255		----		----	
260		----		----	
311		----		----	
315		----		----	
325		----		----	
331	D5185	8.1		0.19	
340	D5185	7.9		-0.02	
343	D5185	7.6		-0.32	
349	D5185	4.67	G(0.05)	-3.27	
360	D5185	8.21		0.30	
398	D6595	15.8	G(0.01)	7.94	
420		----		----	
432		----		----	
450		----		----	
451	D5185	9		1.09	
473	D5185	<1		<-6.81	false negative?
496	D5185	7.4		-0.52	
511	D6595	7.61		-0.31	
541	D5185	7.3		-0.62	
551		----		----	
562	D6595	11.4	G(0.01)	3.51	
575	D6595	7.192		-0.73	
593		----		----	
603		----		----	
608		----		----	
609	D5185	7.284		-0.64	
614	D5185	7.80		-0.12	
633	D6595	11.49	G(0.05)	3.60	
657	D5185	7		-0.92	
663	D5185	8.43		0.52	
823	D5185	6.289		-1.64	
862	D5185	8.69		0.78	
875		----		----	
902	D5185	7.349		-0.57	
912	D5185	8		0.09	
922		----		----	
963		----		----	
994	D5185	8.063	C	0.15	first reported: 1.94
1013		----		----	
1017		----		----	
1023	D5185	8.00		0.09	
1059	in house	9		1.09	
1106		----		----	
1146		----		----	
1155	D5185	8.91		1.00	
1161		----		----	
1173	in house	7.1		-0.82	
1201	D5185	8		0.09	
1203	D5185	7.53		-0.39	
1231		----		----	
1243		----		----	
1262	D5185	7.7537		-0.16	
1271	D5185	8.3		0.39	
1316	D5185	7.98		0.07	
1358		----		----	
1396	INH-12	7.46		-0.46	
1402		----		----	
1428	D5185	7.4		-0.52	
1431	in house	8.0		0.09	
1435	D5185	9		1.09	
1480		----		----	
1495		----		----	
1569	D5185	8		0.09	
1571	D5185	7.5710		-0.35	
1579		----		----	
1622		----		----	
1648		----		----	
1660	D5185	10.45		2.55	
1680		----		----	
1704		----	W	----	result withdrawn, first reported: 6.0

1720		----		----
1722		----		----
1730	D5185	7.945		0.03
1740	D5185	6.5		-1.43
1800		----		----
1827		----		----
1833		----		----
1842		----		----
1850	in house	4	G(0.05)	-3.94
1854	D5185	7.9		-0.02
1900		----		----
1903	in house	51.4	G(0.01)	43.79
1915	D5185	8.3	C	0.39
2122	D5185	8.35		0.44
3166	EPA6020	7.92		0.00

first reported: 3.98

normality OK
 n 43
 outliers 6
 mean (n) 7.92
 st.dev. (n) 0.719
 R(calc.) 2.01
 R(D5185:09) 2.78

application range: 5 - 200 mg/kg



Determination of Nickel (Ni) on sample #13064; results in mg/kg

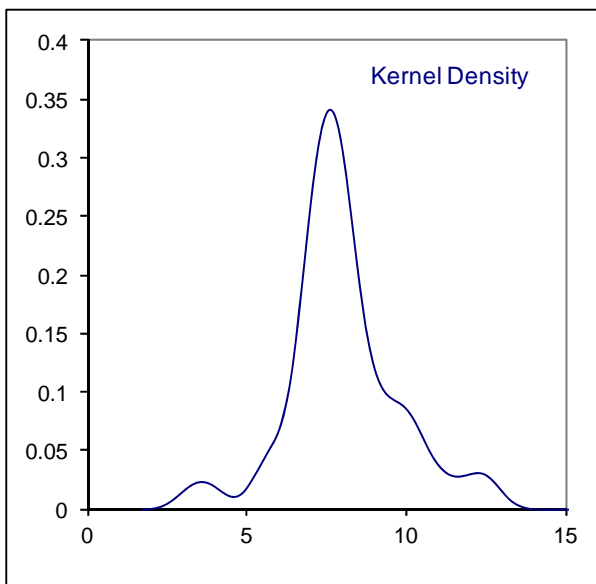
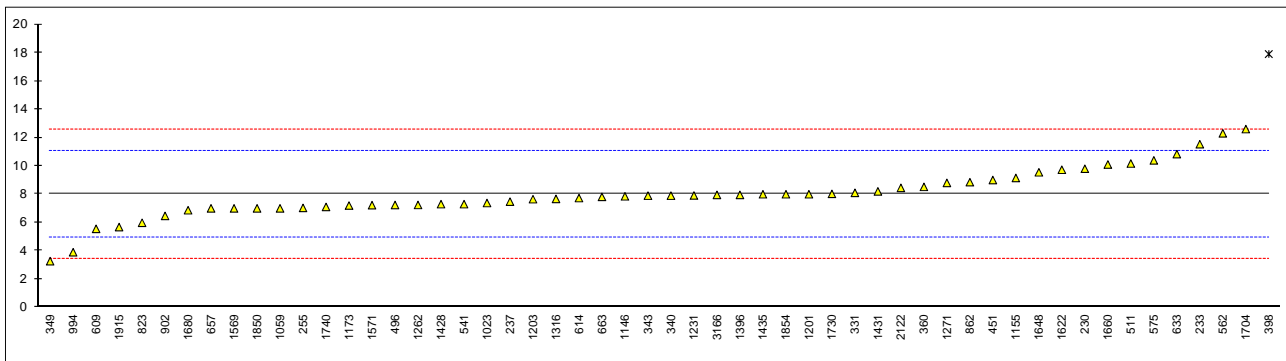
lab	method	value	mark	z(targ)	remarks
230	D6595	9.8		1.18	
233	D6595	11.53		2.32	
237	D5185	7.4735		-0.36	
252		----		----	
254		----		----	
255	INH-1	7.032		-0.65	
260		----		----	
311		----		----	
315		----		----	
325		----		----	
331	D5185	8.1		0.06	
340	D5185	7.9		-0.07	
343	D5185	7.9		-0.07	
349	D5185	3.27		-3.13	
360	D5185	8.52		0.34	
398	D6595	17.9	G(0.01)	6.52	
420		----		----	
432		----		----	
450		----		----	
451	D5185	9		0.65	
473	D5185	<1		<-4.54	false negative?
496	D5185	7.24		-0.51	
511	D6595	10.17		1.42	
541	D5185	7.3		-0.47	
551		----		----	
562	D6595	12.3		2.83	
575	D6595	10.389		1.57	
593		----		----	
603		----		----	
608		----		----	
609	D5185	5.555		-1.62	
614	D5185	7.73		-0.19	
633	D6595	10.83		1.86	
657	D5185	7		-0.67	
663	D5185	7.81		-0.13	
823	D5185	5.981		-1.34	
862	D5185	8.85		0.55	
875		----		----	
902	D5185	6.467		-1.02	
912		----		----	
922		----		----	
963		----		----	
994	D5185	3.90	C	-2.71	first reported: 1.01
1013		----		----	
1017		----		----	
1023	D5185	7.38		-0.42	
1059	in house	7		-0.67	
1106		----		----	
1146	D5185	7.852		-0.11	
1155	D5185	9.15		0.75	
1161		----		----	
1173	in house	7.2		-0.54	
1201	D5185	8		-0.01	
1203	D5185	7.65		-0.24	
1231	D5185	7.912		-0.07	
1243		----		----	
1262	D5185	7.2416		-0.51	
1271	D5185	8.8		0.52	
1316	D5185	7.67		-0.23	
1358		----		----	
1396	INH-12	7.95		-0.04	
1402		----		----	
1428	D5185	7.3		-0.47	
1431	in house	8.2		0.12	
1435	D5185	8		-0.01	
1480		----		----	
1495		----		----	
1569	D5185	7		-0.67	
1571	D5185	7.2273		-0.52	
1579		----		----	
1622	D5185	9.73		1.13	
1648	D5185	9.55		1.01	
1660	D5185	10.10		1.38	
1680	EN14107	6.87		-0.75	
1704	in house	12.6		3.03	

1720		----	----
1722		----	----
1730	D5185	8.025	0.01
1740	D5185	7.1	-0.60
1800		----	----
1827		----	----
1833		----	----
1842		----	----
1850	in house	7	-0.67
1854	D5185	8	-0.01
1900		----	----
1903	in house	<1	<-4.46
1915	D5185	5.68	-1.54
2122	D5185	8.45	0.29
3166	EPA6020	7.95	-0.04
	normality	not OK	<u>only D6595:05 data</u>
	n	53	OK
	outliers	1	6
	mean (n)	8.01	10.84
	st.dev. (n)	1.733	0.932
	R(calc.)	4.85	2.61
	R(D5185:09)	4.25	4.94

result withdrawn, first reported: IP 501, 12.15565

false negative?

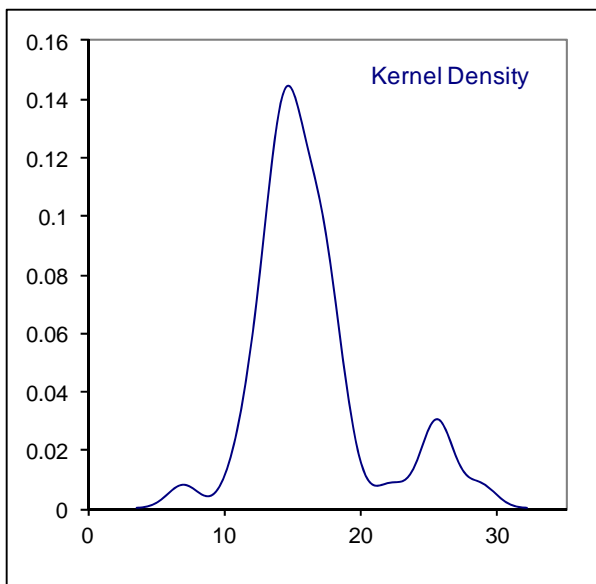
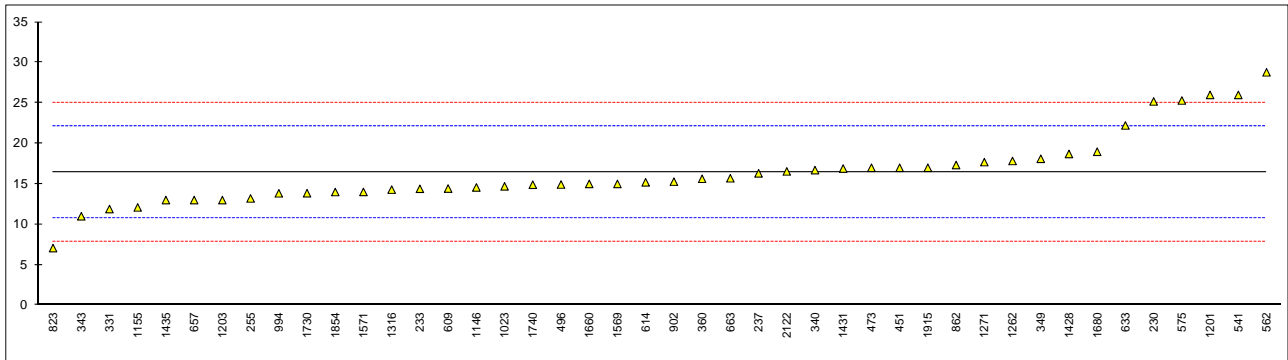
application range: 5 - 40 mg/kg



Determination of Sodium (Na) on sample #13064; results in mg/kg

lab	method	value	mark	z(targ)	remarks
230	D6595	25.2		3.05	
233	D6595	14.40		-0.71	
237	D5185	16.31		-0.05	
252		----		----	
254		----		----	
255	INH-1	13.21		-1.13	
260		----		----	
311		----		----	
315		----		----	
325		----		----	
331	D5185	11.9		-1.59	
340	D5185	16.7		0.09	
343	D5185	11		-1.90	
349	D5185	18.11		0.58	
360	D5185	15.64		-0.28	
398		----		----	
420		----		----	
432		----		----	
450		----		----	
451	D5185	17		0.19	
473	D5185	17		0.19	
496	D5185	14.93		-0.53	
511		----		----	
541	D5185	26		3.33	
551		----		----	
562	D6595	28.8		4.31	
575	D6595	25.317		3.09	
593		----		----	
603		----		----	
608		----		----	
609	D5185	14.427		-0.70	
614	D5185	15.2		-0.43	
633	D6595	22.23		2.02	
657	D5185	13		-1.20	
663	D5185	15.7		-0.26	
823	D5185	7.063		-3.27	
862	D5185	17.34		0.31	
875		----		----	
902	D5185	15.286		-0.40	
912		----		----	
922		----		----	
963		----		----	
994	D5185	13.84		-0.91	
1013		----		----	
1017		----		----	
1023	D5185	14.7		-0.61	
1059		----		----	
1106		----		----	
1146	D5185	14.56		-0.66	
1155	D5185	12.10		-1.52	
1161		----		----	
1173		----		----	
1201	D5185	26		3.33	
1203	D5185	13.0		-1.20	
1231		----		----	
1243		----		----	
1262	D5185	17.839		0.49	
1271	D5185	17.7		0.44	
1316	D5185	14.3		-0.75	
1358		----		----	
1396		----		----	
1402		----		----	
1428	D5185	18.7		0.79	
1431	in house	16.9		0.16	
1435	D5185	13		-1.20	
1480		----		----	
1495		----		----	
1569	D5185	15		-0.50	
1571	D5185	14.0073		-0.85	
1579		----		----	
1622		----		----	
1648		----		----	
1660	D5185	15.0		-0.50	
1680	EN14107	18.98		0.88	
1704		----		----	

1720		----	----
1722		----	----
1730	D5185	13.854	-0.90
1740	D5185	14.9	-0.54
1800		----	----
1827		----	----
1833		W	result withdrawn, first reported: 22.91825
1842		----	----
1850		----	----
1854	D5185	14	-0.85
1900		----	----
1903		----	----
1915	D5185	17.0	0.19
2122	D5185	16.55	0.04
3166	EPA6020	<10	<-2.20 false negative?
normality	not OK		
n	44		
outliers	0		
mean (n)	16.45		
st.dev. (n)	4.327		
R(calc.)	12.11		
R(D5185:09)	8.03		application range: 7 – 70 mg/kg

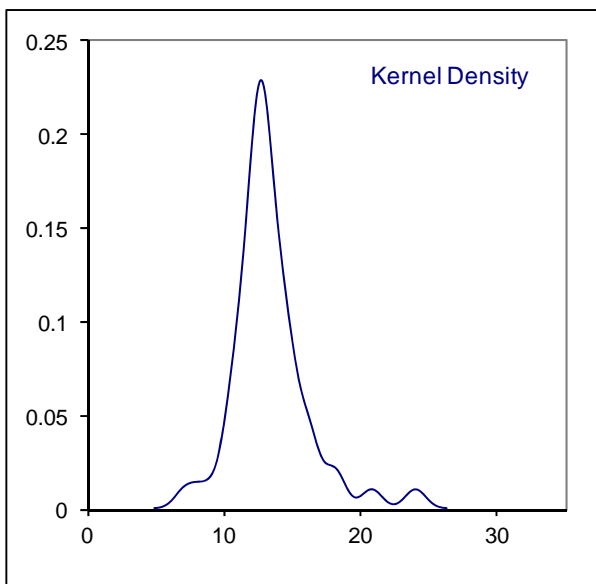
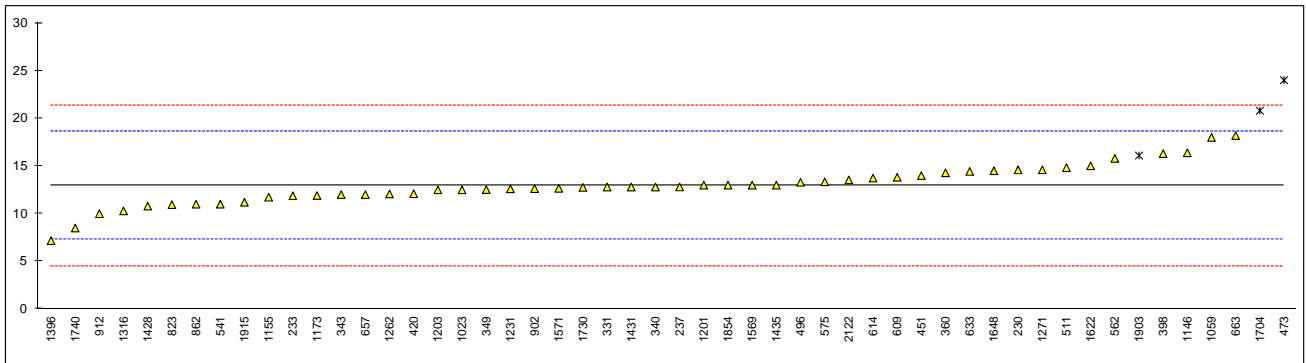


Determination of Silicon (Si) on sample #13064; results in mg/kg

lab	method	value	mark	z(targ)	remarks
230	D6595	14.6		0.59	
233	D6595	11.88		-0.38	
237	D5185	12.805		-0.05	
252		----		----	
254		----		----	
255		----		----	
260		----		----	
311		----		----	
315		----		----	
325		----		----	
331	D5185	12.8		-0.05	
340	D5185	12.8		-0.05	
343	D5185	12		-0.34	
349	D5185	12.53		-0.15	
360	D5185	14.29		0.48	
398	D6595	16.3		1.19	
420	INH-208	12.1		-0.30	
432		----		----	
450		----		----	
451	D5185	14		0.37	
473	D5185	24	G(0.01)	3.93	
496	D5185	13.3		0.12	
511	D6595	14.83		0.67	
541	D5185	11		-0.69	
551		----		----	
562	D6595	15.8		1.01	
575	D6595	13.344		0.14	
593		----		----	
603		----		----	
608		----		----	
609	D5185	13.817		0.31	
614	D5185	13.73		0.28	
633	D6595	14.43		0.53	
657	D5185	12		-0.34	
663	D5185	18.2		1.87	
823	D5185	10.95		-0.71	
862	D5185	11		-0.69	
875		----		----	
902	D5185	12.632		-0.11	
912	D5185	10		-1.05	
922		----		----	
963		----		----	
994		----		----	
1013		----		----	
1017		----		----	
1023	D5185	12.5		-0.16	
1059	in house	18		1.79	
1106		----		----	
1146	D5185	16.39		1.22	
1155	D5185	11.73		-0.43	
1161		----		----	
1173	in house	11.9		-0.37	
1201	D5185	13		0.02	
1203	D5185	12.5		-0.16	
1231	D5185	12.61		-0.12	
1243		----		----	
1262	D5185	12.074		-0.31	
1271	D5185	14.6		0.59	
1316	D5185	10.3		-0.94	
1358		----		----	
1396	INH-12	7.17		-2.06	
1402		----		----	
1428	D5185	10.8		-0.77	
1431	in house	12.8		-0.05	
1435	D5185	13		0.02	
1480		----		----	
1495		----		----	
1569	D5185	13		0.02	
1571	D5185	12.6604		-0.10	
1579		----		----	
1622	D5185	15.02		0.73	
1648	D5185	14.5		0.55	
1660	D5185	<5		----	false negative?
1680		----		----	
1704	in house	20.8	G(0.05)	2.79	

1720		----		----
1722		----		----
1730	D5185	12.757		-0.07
1740	D5185	8.5		-1.58
1800		----		----
1827		----		----
1833		----	W	----- result withdrawn, first reported: 16.8798
1842		----		----
1850		----		----
1854	D5185	13		0.02
1900		----		----
1903	in house	16.1	ex	1.12
1915	D5185	11.2		-0.62
2122	D5185	13.55		0.21
3166	EPA6020	<100		----
normality		OK		
n		49		
outliers		2		
mean (n)		12.95		
st.dev. (n)		2.058		
R(calc.)		5.76		
R(D5185:09)		7.87		

application range: 8 - 50 mg/kg



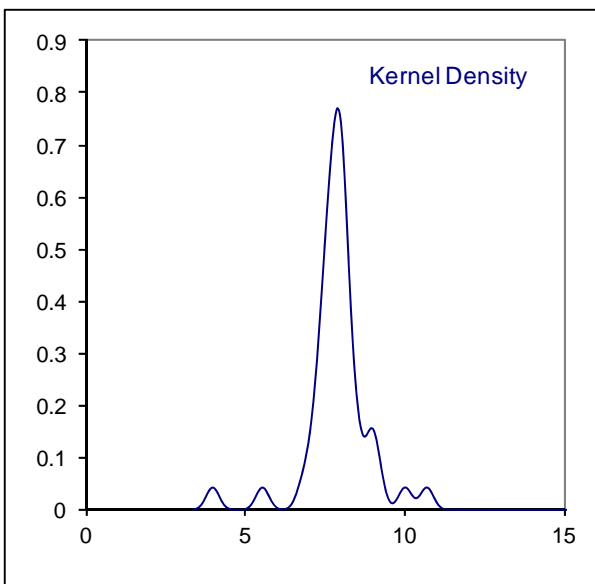
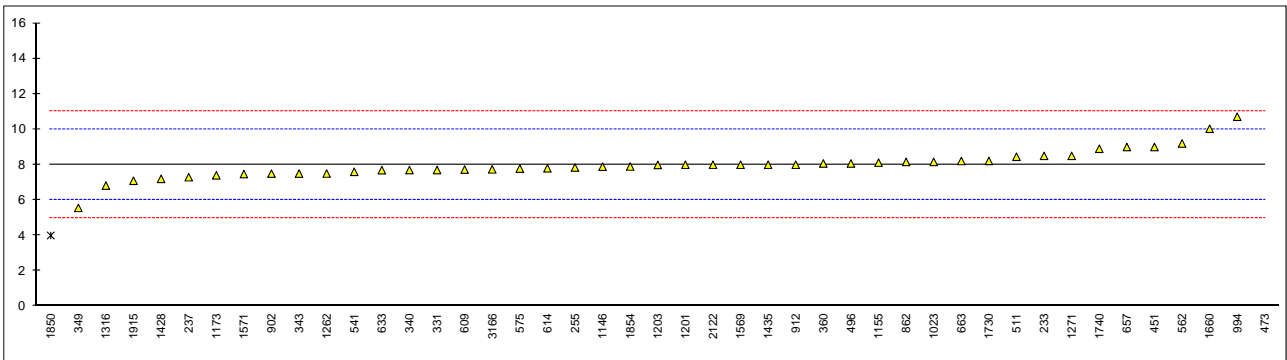
Determination of Silver (Ag) on sample #13064; results in mg/kg

lab	method	value	mark	z(targ)	remarks
230		----		----	
233	D6595	8.50		0.50	
237	D5185	7.295		-0.70	
252		----		----	
254		----		----	
255	INH-1	7.84		-0.16	
260		----		----	
311		----		----	
315		----		----	
325		----		----	
331	D5185	7.7		-0.30	
340	D5185	7.7		-0.30	
343	D5185	7.5		-0.50	
349	D5185	5.56		-2.44	
360	D5185	8.07		0.07	
398		----		----	
420		----		----	
432		----		----	
450		----		----	
451	D5185	9		1.00	
473	D5185	22	G(0.01)	14.00	
496	D5185	8.07		0.07	
511	D6595	8.45		0.45	
541	D5185	7.6		-0.40	
551		----		----	
562	D6595	9.2		1.20	
575	D6595	7.780		-0.22	
593		----		----	
603		----		----	
608		----		----	
609	D5185	7.726		-0.27	
614	D5185	7.8		-0.20	
633	D6595	7.69		-0.31	
657	D5185	9	C	1.00	first reported: 11
663	D5185	8.21		0.21	
823		----		----	
862	D5185	8.16		0.16	
875		----		----	
902	D5185	7.496		-0.50	
912	D5185	8		0.00	
922		----		----	
963		----		----	
994	D5185	10.71	C	2.71	first reported: 1.96
1013		----		----	
1017		----		----	
1023	D5185	8.16		0.16	
1059		----		----	
1106		----		----	
1146	D5185	7.890		-0.11	
1155	D5185	8.117	C	0.12	first reported: 10.93
1161		----		----	
1173	in house	7.4		-0.60	
1201	D5185	8		0.00	
1203	D5185	7.99		-0.01	
1231		----		----	
1243		----		----	
1262	D5185	7.5022		-0.50	
1271	D5185	8.5		0.50	
1316	D5185	6.82		-1.18	
1358		----		----	
1396		----		----	
1402		----		----	
1428	D5185	7.2		-0.80	
1431		----		----	
1435	D5185	8		0.00	
1480		----		----	
1495		----		----	
1569	D5185	8		0.00	
1571	D5185	7.4698		-0.53	
1579		----		----	
1622		----		----	
1648		----		----	
1660	D5185	10.03		2.03	
1680		----		----	
1704		----		----	

1720		----	----
1722		----	----
1730	D5185	8.215	0.22
1740	D5185	8.9	0.90
1800		----	----
1827		----	----
1833		----	----
1842		----	----
1850	in house	4	G(0.01) -4.00
1854	D5185	7.9	-0.10
1900		----	----
1903	in house	<1	<-6.64 false negative?
1915	D5185	7.09	-0.91
2122	D5185	8.0	0.00
3166	EPA6020	7.74	-0.26

normality not OK
n 43
outliers 2
mean (n) 8.00
st.dev. (n) 0.817
R(calc.) 2.29
R(D5185:09) 2.80

application range: 0.5 – 50 mg/kg



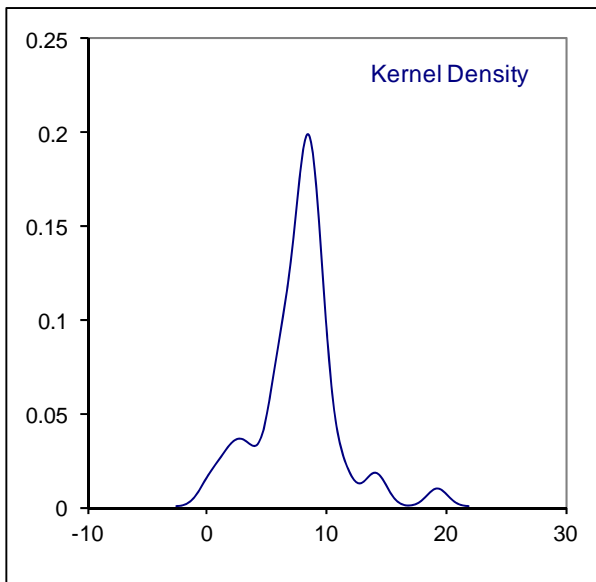
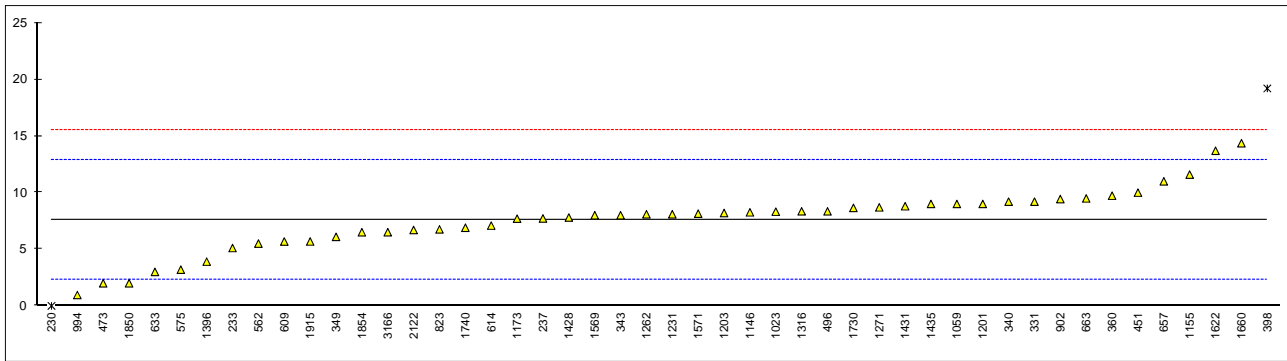
Determination of Tin (Sn) on sample #13064; results in mg/kg

lab	method	value	mark	z(targ)	remarks
230	D6595	0	ex	-2.88	result excluded, zero is not a real value
233	D6595	5.11		-0.94	
237	D5185	7.718		0.05	
252		----		----	
254		----		----	
255		----		----	
260		----		----	
311		----		----	
315		----		----	
325		----		----	
331	D5185	9.2		0.61	
340	D5185	9.2		0.61	
343	D5185	8.0		0.15	
349	D5185	6.09		-0.57	
360	D5185	9.73		0.81	
398	D6595	19.2	G(0.05)	4.40	
420		----		----	
432		----		----	
450		----		----	
451	D5185	10		0.91	
473	D5185	2		-2.12	
496	D5185	8.35		0.29	
511		----		----	
541	D5185	<10		----	
551		----		----	
562	D6595	5.5		-0.80	
575	D6595	3.199		-1.67	
593		----		----	
603		----		----	
608		----		----	
609	D5185	5.682		-0.73	
614	D5185	7.09		-0.19	
633	D6595	3.00		-1.74	
657	D5185	11		1.29	
663	D5185	9.49		0.72	
823	D5185	6.754		-0.32	
862	D5185	<0.01		----	
875		----		----	
902	D5185	9.432		0.70	
912		----		----	
922		----		----	
963		----		----	
994	D5185	0.96		-2.52	
1013		----		----	
1017		----		----	
1023	D5185	8.32		0.27	
1059	in house	9		0.53	
1106		----		----	
1146	D5185	8.261		0.25	
1155	D5185	11.59		1.51	
1161		----		----	
1173	in house	7.7		0.04	
1201	D5185	9		0.53	
1203	D5185	8.20		0.23	
1231	D5185	8.089		0.19	
1243		----		----	
1262	D5185	8.0871		0.19	
1271	D5185	8.7		0.42	
1316	D5185	8.35		0.29	
1358		----		----	
1396	INH-12	3.91		-1.40	
1402		----		----	
1428	D5185	7.8		0.08	
1431	in house	8.8		0.46	
1435	D5185	9		0.53	
1480		----		----	
1495		----		----	
1569	D5185	8		0.15	
1571	D5185	8.1444		0.21	
1579		----		----	
1622	D5185	13.70		2.31	
1648		----		----	
1660	D5185	14.36		2.56	
1680		----		----	
1704		----		----	

1720		----	----
1722		----	----
1730	D5185	8.654	0.40
1740	D5185	6.9	-0.26
1800		----	----
1827		----	----
1833		----	----
1842		----	----
1850	in house	2	-2.12
1854	D5185	6.5	-0.42
1900		----	----
1903	in house	<1	<-2.45 false negative?
1915	D5185	5.69	-0.72
2122	D5185	6.7	-0.34
3166	EPA6020	6.5	-0.42

normality not OK
n 46
outliers 1
mean (n) 7.60
st.dev. (n) 2.716
R(calc.) 7.60
R(D5185:09) 7.38

application range: 10 – 40 mg/kg



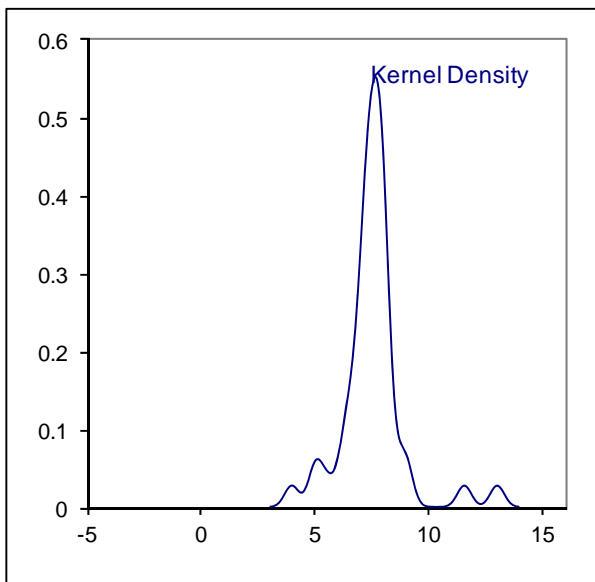
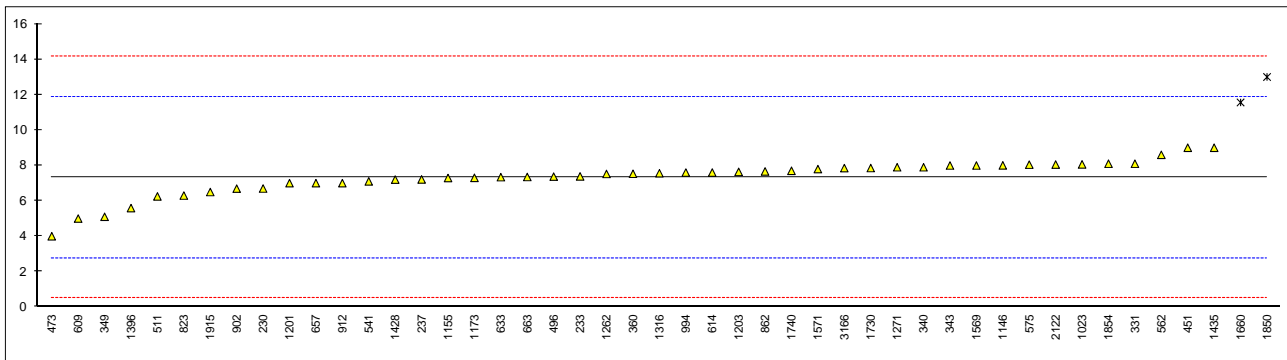
Determination of Titanium (Ti) on sample #13064; results in mg/kg

lab	method	value	mark	z(targ)	remarks
230	D6595	6.7		-0.28	
233	D6595	7.38		0.02	
237	D5185	7.209		-0.05	
252		----		----	
254		----		----	
255		----		----	
260		----		----	
311		----		----	
315		----		----	
325		----		----	
331	D5185	8.1		0.34	
340	D5185	7.9		0.25	
343	D5185	8.0		0.30	
349	D5185	5.10		-0.98	
360	D5185	7.53		0.09	
398		----		----	
420		----		----	
432		----		----	
450		----		----	
451	D5185	9		0.73	
473	D5185	4		-1.46	
496	D5185	7.37		0.02	
511	D6595	6.25		-0.47	
541	D5185	7.1		-0.10	
551		----		----	
562	D6595	8.6		0.56	
575	D6595	8.047		0.32	
593		----		----	
603		----		----	
608		----		----	
609	D5185	5.00		-1.02	
614	D5185	7.6		0.12	
633	D6595	7.34		0.01	
657	D5185	7		-0.14	
663	D5185	7.35		0.01	
823	D5185	6.301		-0.45	
862	D5185	7.66		0.15	
875		----		----	
902	D5185	6.691		-0.28	
912	D5185	7		-0.14	
922		----		----	
963		----		----	
994	D5185	7.597	C	0.12	first reported: 1.38
1013		----		----	
1017		----		----	
1023	D5185	8.06		0.32	
1059		----		----	
1106		----		----	
1146	D5185	8.005		0.30	
1155	D5185	7.29		-0.02	
1161		----		----	
1173	in house	7.3		-0.01	
1201	D5185	7		-0.14	
1203	D5185	7.64		0.14	
1231		----		----	
1243		----		----	
1262	D5185	7.5206		0.08	
1271	D5185	7.9		0.25	
1316	D5185	7.56		0.10	
1358		----		----	
1396	INH-12	5.59		-0.76	
1402		----		----	
1428	D5185	7.2		-0.06	
1431		----		----	
1435	D5185	9		0.73	
1480		----		----	
1495		----		----	
1569	D5185	8		0.30	
1571	D5185	7.7949		0.21	
1579		----		----	
1622		----		----	
1648		----		----	
1660	D5185	11.56	G(0.01)	1.86	
1680		----		----	
1704		----		----	

1720		----	----
1722		----	----
1730	D5185	7.856	0.23
1740	D5185	7.7	0.16
1800		----	----
1827		----	----
1833		----	----
1842		----	----
1850	in house	13	G(0.01) 2.49
1854	D5185	8.1	0.34
1900		----	----
1903	in house	<1	<-2.72 false negative?
1915	D5185	6.5	-0.36
2122	D5185	8.05	0.32
3166	EPA6020	7.85	0.23

normality not OK
n 45
outliers 2
mean (n) 7.33
st.dev. (n) 0.977
R(calc.) 2.73
R(D5185:09) 6.37

application range: 5 – 40 mg/kg



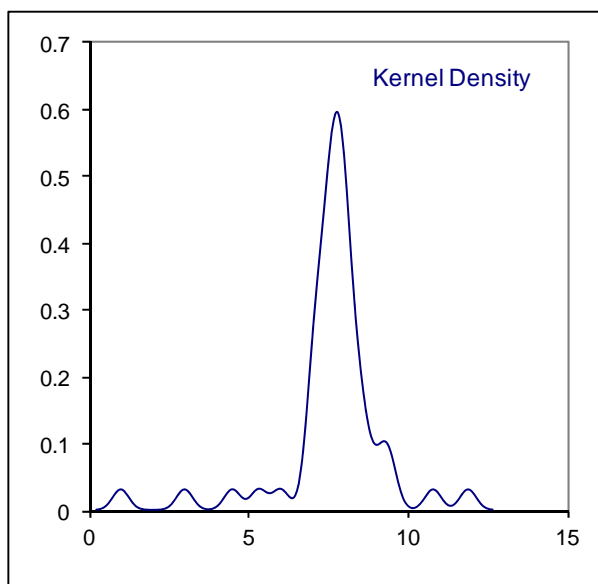
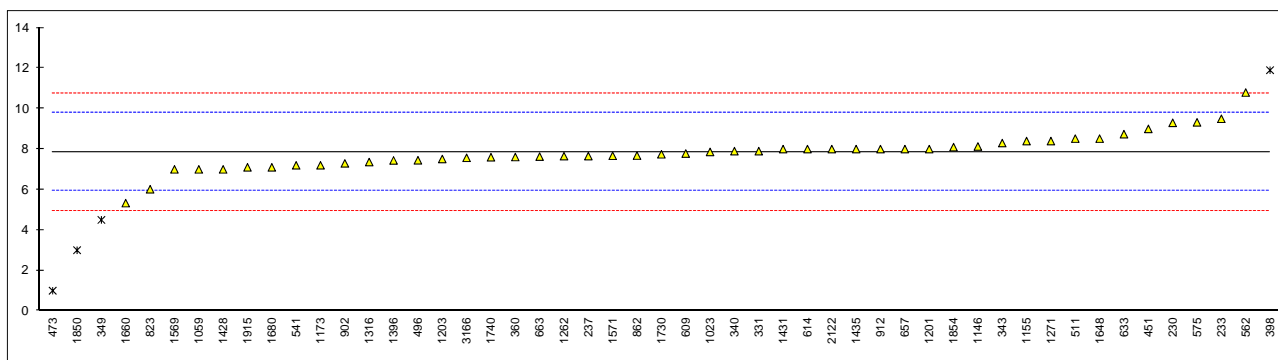
Determination of Vanadium (V) on sample #13064; results in mg/kg

lab	method	value	mark	z(targ)	remarks
230	D6595	9.3		1.49	
233	D6595	9.50		1.70	
237	D5185	7.6525		-0.21	
252		----		----	
254		----		----	
255		----		----	
260		----		----	
311		----		----	
315		----		----	
325		----		----	
331	D5185	7.9		0.04	
340	D5185	7.9		0.04	
343	D5185	8.3		0.46	
349	D5185	4.50	G(0.05)	-3.48	
360	D5185	7.61		-0.26	
398	D6595	11.9	G(0.01)	4.19	
420		----		----	
432		----		----	
450		----		----	
451	D5185	9		1.18	
473	D5185	1	G(0.01)	-7.10	
496	D5185	7.45		-0.42	
511	D6595	8.52		0.69	
541	D5185	7.2		-0.68	
551		----		----	
562	D6595	10.8		3.05	
575	D6595	9.322		1.52	
593		----		----	
603		----		----	
608		----		----	
609	D5185	7.781		-0.08	
614	D5185	8.0		0.15	
633	D6595	8.74		0.91	
657	D5185	8		0.15	
663	D5185	7.63		-0.24	
823	D5185	6.015		-1.91	
862	D5185	7.68		-0.18	
875		----		----	
902	D5185	7.295		-0.58	
912	D5185	8		0.15	
922		----		----	
963		----		----	
994		----		----	
1013		----		----	
1017		----		----	
1023	D5185	7.86		0.00	
1059	in house	7		-0.89	
1106		----		----	
1146	D5185	8.129		0.28	
1155	D5185	8.40		0.56	
1161		----		----	
1173	in house	7.2		-0.68	
1201	D5185	8		0.15	
1203	D5185	7.51		-0.36	
1231		----		----	
1243		----		----	
1262	D5185	7.6515		-0.21	
1271	D5185	8.4		0.56	
1316	D5185	7.36		-0.51	
1358		----		----	
1396	INH-12	7.44		-0.43	
1402		----		----	
1428	D5185	7.0		-0.89	
1431	in house	8.0		0.15	
1435	D5185	8		0.15	
1480		----		----	
1495		----		----	
1569	D5185	7		-0.89	
1571	D5185	7.6725		-0.19	
1579		----		----	
1622		----		----	
1648	D5185	8.52		0.69	
1660	D5185	5.33		-2.62	
1680	EN14107	7.10		-0.78	
1704		----		----	

1720		----	----		
1722		----	----		
1730	D5185	7.745	-0.12		
1740	D5185	7.6	-0.27		
1800		----	----		
1827		----	----		
1833		----	W	-----	result withdrawn, first reported: IP501, 0.7737075
1842		----		-----	
1850	in house	3	G(0.01)	-5.03	
1854	D5185	8.1		0.25	
1900		----		----	
1903	in house	<1		<-6.95	false negative?
1915	D5185	7.1		-0.78	
2122	D5185	8.0		0.15	
3166	EPA6020	7.57		-0.30	

normality not OK
n 47
outliers 4
mean (n) 7.86
st.dev. (n) 0.873
R(calc.) 2.44
R(D5185:09) 2.70

application range: 1 – 50 mg/kg



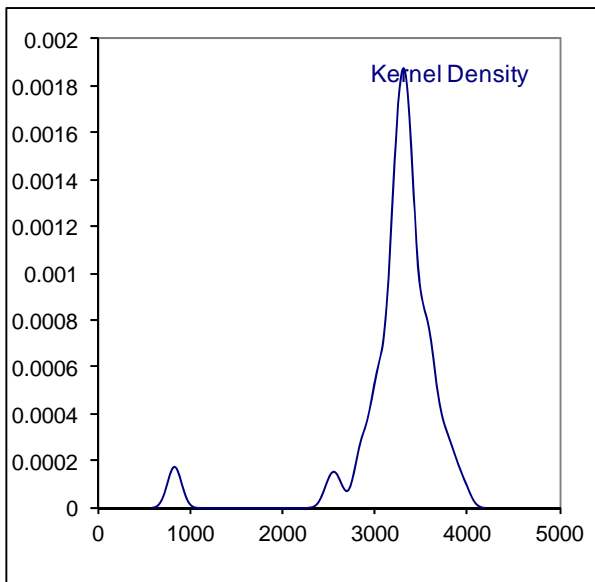
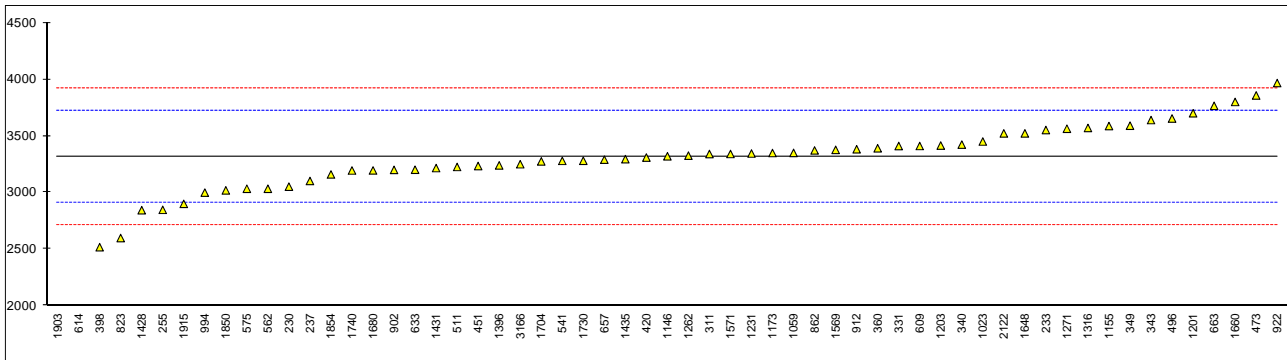
Determination of Calcium (Ca) on sample #13064; results in mg/kg

lab	method	value	mark	z(targ)	remarks
230	D6595	3051.7		-1.31	
233	D6595	3552		1.16	
237	D5185	3102		-1.06	
252		----		----	
254		----		----	
255	INH-1	2847.61		-2.32	
260		----		----	
311	D5185	3340		0.11	
315		----		----	
325		----		----	
331	D5185	3410.8		0.46	
340	D5185	3423		0.52	
343	D5185	3640		1.60	
349	D5185	3590.05		1.35	
360	D5185	3391		0.36	
398	D6595	2517.8		-3.95	
420	DIN51391	3309		-0.04	
432		----		----	
450		----		----	
451	D5185	3234		-0.41	
473	D5185	3857		2.67	
496	D5185	3654		1.66	
511	D6595	3226		-0.45	
541	D5185	3280		-0.18	
551		----		----	
562	D6595	3033.4		-1.40	
575	D6595	3033.3		-1.40	
593		----		----	
603		----		----	
608		----		----	
609	D5185	3411		0.46	
614	D5185	842	G(0.01)	-12.24	
633	D6595	3201.20		-0.57	
657	D5185	3290		-0.13	
663	D5185	3766.1		2.22	
823	D5185	2598		-3.56	
862	D5185	3372		0.27	
875		----		----	
902	D5185	3200		-0.58	
912	D5185	3382		0.32	
922	D4628	3966.82		3.21	
963		----		----	
994	D5185	2999		-1.57	
1013		----		----	
1017		----		----	
1023	D5185	3451		0.66	
1059	in house	3350		0.16	
1106		----		----	
1146	D5185	3320		0.01	
1155	D5185	3586.98	C	1.33	first reported: 3970.5
1161		----		----	
1173	in house	3349		0.16	
1201	D5185	3700		1.89	
1203	D5185	3415		0.48	
1231	D4951	3345		0.14	
1243		----		----	
1262	D5185	3325.7		0.04	
1271	D5185	3563.3	C	1.22	first reported: 0.3504 mg/kg
1316	D5185	3570		1.25	
1358		----		----	
1396	INH-12	3239.43		-0.38	
1402		----		----	
1428	D5185	2844		-2.34	
1431	in house	3216		-0.50	
1435	D5185	3295		-0.11	
1480		----		----	
1495		----		----	
1569	D5185	3377		0.30	
1571	D5185	3340.5525		0.11	
1579		----		----	
1622		----		----	
1648	D5185	3522.6		1.01	
1660	D5185	3800		2.39	
1680	EN14107	3195.26		-0.60	
1704	in house	3275.2		-0.21	

1720		----	----
1722		----	----
1730	D5185	3280.398	-0.18
1740	D5185	3195	-0.60
1800		----	----
1827		----	----
1833		----	----
1842		----	----
1850	in house	3019	-1.47
1854	D5185	3160	-0.78
1900		----	----
1903	in house	818	G(0.01) -12.36
1915	D5185	2900	-2.06
2122	D5185	3522	1.01
3166	EPA6020	3250	-0.33

normality not OK
n 57
outliers 2
mean (n) 3317.30
st.dev. (n) 279.222
R(calc.) 781.82
R(D5185:09) 566.38

application range: 40 – 9000 mg/kg



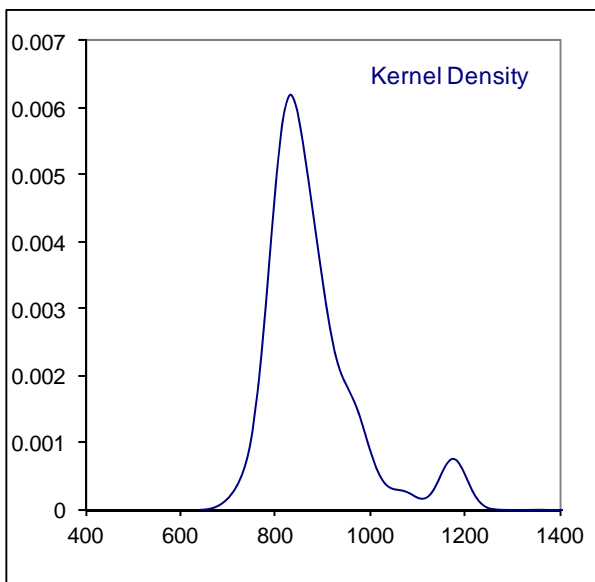
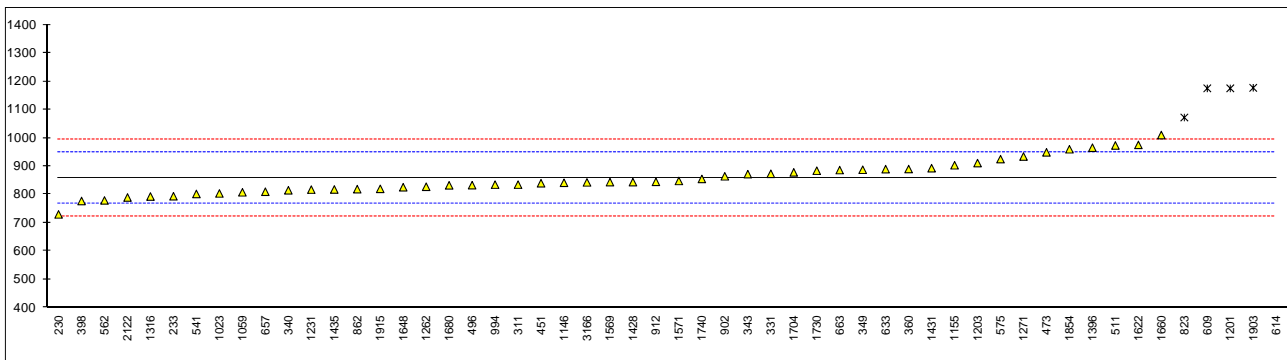
Determination of Phosphorus (P) on sample #13064; results in mg/kg

lab	method	value	mark	z(targ)	remarks
230	D6595	729.9		-2.85	
233	D6595	794		-1.43	
237		----		----	
252		----		----	
254		----		----	
255		----		----	
260		----		----	
311	D5185	835		-0.52	
315		----		----	
325		----		----	
331	D5185	873.3		0.34	
340	D5185	815		-0.96	
343	D5185	872		0.31	
349	D5185	887.69		0.66	
360	D5185	890		0.71	
398	D6595	777.2		-1.80	
420		----		----	
432		----		----	
450		----		----	
451	D5185	840		-0.40	
473	D5185	949		2.02	
496	D5185	833.0		-0.56	
511	D6595	973.25		2.56	
541	D5185	802		-1.25	
551		----		----	
562	D6595	779.2		-1.76	
575	D6595	925.05		1.49	
593		----		----	
603		----		----	
608		----		----	
609	D5185	1175	C,G(0.01)	7.04	first reported: 1362
614	D5185	3773	G(0.01)	64.79	
633	D6595	889.74		0.70	
657	D5185	810		-1.07	
663	D5185	886.4		0.63	
823	D5185	1072	G(0.05)	4.75	
862	D5185	819		-0.87	
875		----		----	
902	D5185	864.5		0.14	
912	D5185	845		-0.29	
922		----		----	
963		----		----	
994	D5185	835	C	-0.52	first reported: 694.8
1013		----	W	----	result withdrawn, first reported: 1230
1017		----		----	
1023	D5185	804		-1.20	
1059	in house	808		-1.12	
1106		----		----	
1146	D5185	841.5		-0.37	
1155	D5185	903.518	C	1.01	first reported: 1019.4
1161		----		----	
1173		----		----	
1201	D5185	1175	DG(0.01)	7.04	
1203	D5185	911		1.17	
1231	D4951	817.5		-0.90	
1243		----		----	
1262	D5185	827.58		-0.68	
1271	D5185	934.3	C	1.69	first reported: 0.0949
1316	D5185	793		-1.45	
1358		----		----	
1396	INH-12	965.46		2.38	
1402		----		----	
1428	D5185	844		-0.32	
1431	in house	893	C	0.77	first reported: 1030
1435	D5185	818		-0.89	
1480		----		----	
1495		----		----	
1569	D5185	844		-0.32	
1571	D5185	848.0211		-0.23	
1579		----		----	
1622	D5185	975.35		2.60	
1648	D5185	825.8		-0.72	
1660	D5185	1010		3.37	
1680	EN14107	832.52		-0.57	
1704	in house	878.4		0.45	

1720		----	----
1722		----	----
1730	D5185	884.214	0.58
1740	D5185	855	-0.07
1800		----	----
1827		----	----
1833		----	----
1842		----	----
1850		----	----
1854	D5185	960	2.26
1900		----	----
1903	in house	1176.9	DG(0.01) 7.08
1915	D5185	820	-0.85
2122	D5185	789.65	-1.52
3166	EPA6020	843	-0.34

normality not OK
n 49
outliers 5
mean (n) 858.21
st.dev. (n) 59.327
R(calc.) 166.11
R(D5185:09) 125.97

application range: 10 – 1000 mg/kg



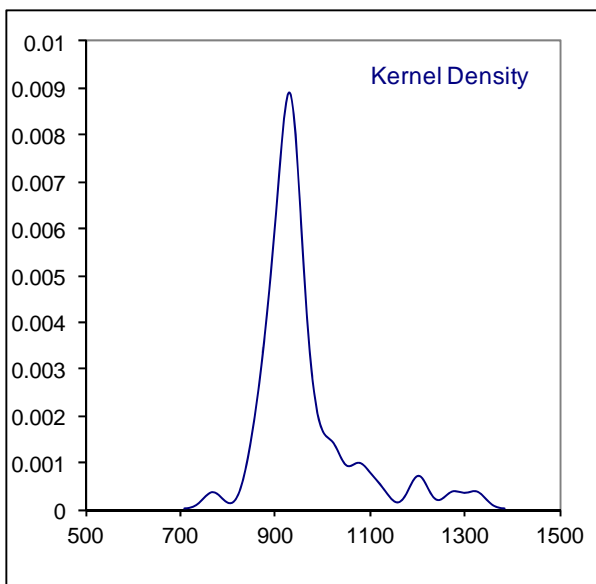
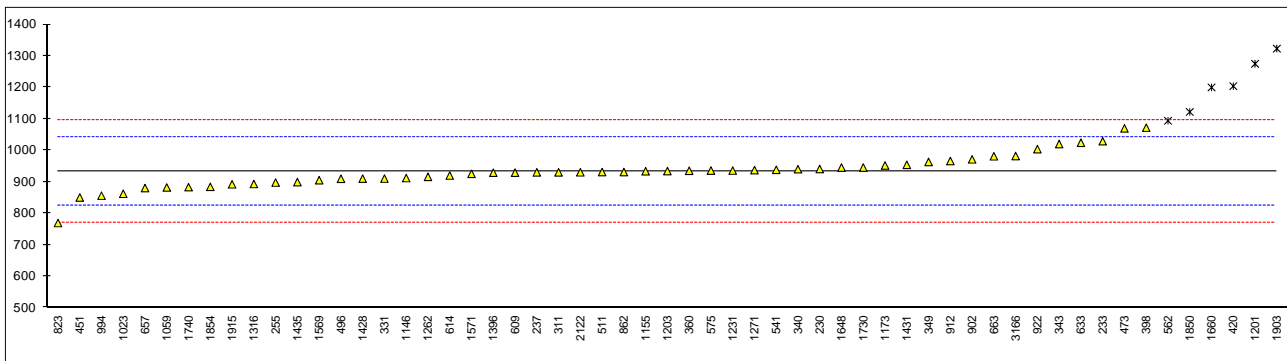
Determination of Zinc (Zn) on sample #13064; results in mg/kg

lab	method	value	mark	z(targ)	remarks
230	D6595	940.8		0.15	
233	D6595	1029		1.76	
237	D5185	930		-0.05	
252		-----		-----	
254		-----		-----	
255	INH-1	897.585		-0.64	
260		-----		-----	
311	D5185	930		-0.05	
315		-----		-----	
325		-----		-----	
331	D5185	910.3		-0.41	
340	D5185	940		0.13	
343	D5185	1020		1.59	
349	D5185	963.15		0.55	
360	D5185	935		0.04	
398	D6595	1072.1		2.54	
420	DIN51391	1204	G(0.05)	4.95	
432		-----		-----	
450		-----		-----	
451	D5185	850		-1.51	
473	D5185	1070		2.50	
496	D5185	909.5		-0.43	
511	D6595	930.87		-0.04	
541	D5185	938		0.09	
551		-----		-----	
562	D6595	1094.0	DG(0.05)	2.94	
575	D6595	935.59		0.05	
593		-----		-----	
603		-----		-----	
608		-----		-----	
609	D5185	929		-0.07	
614	D5185	920		-0.23	
633	D6595	1024.23		1.67	
657	D5185	880		-0.96	
663	D5185	981.1		0.88	
823	D5185	769		-2.99	
862	D5185	931		-0.03	
875		-----		-----	
902	D5185	971.4		0.70	
912	D5185	966		0.61	
922	D4628	1003.86		1.30	
963		-----		-----	
994	D5185	855.9		-1.40	
1013		-----	W	-----	result withdrawn, first reported: 1280
1017		-----		-----	
1023	D5185	862		-1.29	
1059	in house	882		-0.93	
1106		-----		-----	
1146	D5185	911.9		-0.38	
1155	D5185	933.65	C	0.01	first reported: 1087.2
1161		-----		-----	
1173	in house	952		0.35	
1201	D5185	1275	G(0.01)	6.25	
1203	D5185	934		0.02	
1231	D4951	935.8		0.05	
1243		-----		-----	
1262	D5185	915.72		-0.31	
1271	D5185	937	C	0.08	first reported: 0.0949 mg/kg
1316	D5185	893		-0.73	
1358		-----		-----	
1396	INH-12	928.84		-0.07	
1402		-----		-----	
1428	D5185	910		-0.42	
1431	in house	954		0.39	
1435	D5185	899		-0.62	
1480		-----		-----	
1495		-----		-----	
1569	D5185	905		-0.51	
1571	D5185	925.2838		-0.14	
1579		-----		-----	
1622		-----	W	-----	result withdrawn, first reported: 1114.23
1648	D5185	945.2		0.23	
1660	D5185	1200	G(0.01)	4.88	
1680		-----		-----	
1704		-----	W	-----	result withdrawn, first reported: 1165.1

1720		----	----
1722		----	----
1730	D5185	945.246	0.23
1740	D5185	883	-0.91
1800		----	----
1827		----	----
1833		----	----
1842		----	----
1850	in house	1122	DG(0.05) 3.45
1854	D5185	884	-0.89
1900		----	----
1903	in house	1323.3	G(0.01) 7.13
1915	D5185	892	-0.75
2122	D5185	930.25	-0.05
3166	EPA6020	982	0.90

normality not OK
n 51
outliers 6
mean (n) 932.83
st.dev. (n) 53.272
R(calc.) 149.16
R(D5185:09) 153.41

application range: 60 -1600 mg/kg



APPENDIX 2

Analytical details Acid Number determination

lab	method	type of apparatus	KOH solution			type electrodes	difference pH4-pH7 (mV)	drift of elec.	blank titration	sample size (g)	unit reading
			A	B	C						
230	09A	Automatic titrator, 799 GPT titrator	Yes	Yes	Yes	pH electrode	169	1	Yes	5.0555	mV
233											
237	11,A	Titrimo 848plus	Yes	Yes	Yes	Solvotrode, LiCl sat. in ethanol	169	1	Yes	5.0029	mV
252											
254											
255											
260											
311	A	Metrohm 835 titrator	KOH solution from Fisher 0.1 N			pH elec.-Ag/AgCl	174		Yes	5	pH
315											
325											
331	11a,A	Mettler T90	used a commercial KOH solution			Glass electrode Mettler Toledo DC113SC			Yes	3	mV
340	11a, A	Metrohm titino 798 MPT	Yes	Yes	Yes	LiCl	168	2	Yes	5.0237	mV
343	09 ,A	Automatic Titrator	used a commercial react			pH/LiCl3M-EtOH	167	1	Yes	±5	mV
349	11a,A	Automatic Titration System	Yes	Yes	Yes	pH glass electrode	170		Yes	1.28	mV
360	A	MettlerToledo Titrator DL 28	Yes	Yes	Yes	DG 113-SC	165.0	1	Yes	5	mV
398	11a,A	Methrom 636, Titroprocessor	No	Yes	No	Combined Glass	168	1	Yes	1.0127	mV
420											
432											
450											
451											
473											
496	11a,A	Metrohm 730, Metrohm 721, Metrohm 2.2	used a commercial KOH solution			Methrom Pt-Titrode, Metrohm Ag/AgCl	168	2	Yes	1.666/ 1.619	mV
511											
541											
551											
562											
575											
593											
603											
608											
609	11a,A	Metrohm Automatic Potentiometer	No	No	No	Silver-Chloride	209.2	2	Yes	1.0686	mV
614											
633	11a,A	Metrohm Titrimo Plus 848	Yes	Yes	Yes	Metrohm LL solvotrode	167.3	0.1	Yes	5.0079	mV
657	11a,A	Metrohm Titrimo SM 702	0.1 M KOH in IPA was used			pH electrode /AgCl reference electrode	173	2	Yes	1.15	mV
663	11a,A	Automatic Titrator, Metrohm 716 DMS	Yes	Yes	Yes	LiCl sat in ethanol, Metrohm 6.0229.100	170	1	Yes	5	mV
823											
862	11a,A	Metrohm 905 Titrand	Yes	Yes	Yes	Metrohm 6.0279.300	178	<1	Yes	5.0247	mV
875	11,A	AT-500N-1 Kyoto Electronics	Yes	Yes	Yes	H-171 & R-115	167	2	Yes	4.9033/ 4.9411	mV
902											
912	11a,A	Autopotentiometer GT200	Yes	Yes	Yes	Ref elec. GTRS10B/ Detection elec. GTPH1B	171.3	2	Yes	4.6411/ 4.7419/ 4.5330	mV
922											
963											
994	A	Metrohm 848 plus	Yes	Yes	Yes	pH electrode	176	<1	Yes	5	mV
1013											
1017											
1023											
1059											
1106											

1146	11a,A	Applikon equipment	used a commercial KOH solution			pH glass/Ag/AgCl elektrode	166.4	1	Yes	5.11	mV
1155											
1161											
1173											
1201											
1203	11a,A	KEM Automatic Titrator AT-510	Yes	Yes	Yes	Thermo Scientiq 8172 combination pH	250	1	Yes	± 2	mV
1231	A	Automatic titrator	Yes	Yes	Yes	Combine Electrode			Yes	5	mV
1243											
1262											
1271	09,A	Metrohm Titrino 716 DMS	Yes	Yes	Yes	Metrohm LL solvotrode	176	50	No	5 \pm 0.5	mV
1316											
1358											
1396											
1402											
1428											
1431	11,A		No	Yes	Yes	Glass electrode DG113	161		Yes	<u>0.7687</u>	mV
1435											
1480	11,A	Metrohm 702 SM Titrino	used a commercial KOH solution			Ag/AgCl 6.0133.100/LiCl 3M EtOH			Yes	<u>2.00</u>	mV
1495											
1569	11a,A	Metrohm	used a commercial KOH solution			Solvotrode-Metrohm	159		Yes	5.0957	mV
1571	09a	MettlerToledo DL53	used a commercial KOH solution			Combined	170.3	2	Yes	7.0970	mV
1579											
1622	11a,A	Metrohm Titrino 794	Yes	Yes	Yes	Ag/AgCl LiCl sat. in ethanol			Yes	1	mV
1648											
1660											
1680											
1704	11,A	Mettler Toledo T50	Yes	Yes	Yes	Glass electrode with LiCl electrolyte	171		Yes	<u>0.5 to 0.8</u>	mV
1720	07	Metrohm 848 titrino plus	Yes	Yes	Yes	Combined pH electrode 6.0229.100	166		Yes	5	mV
1722											
1730											
1740	11a,A	Automated Metrohm apparatus	used a commercial KOH solution			Ag/AgCl	178		Yes	5	mV
1800											
1827	11a,A	Metrohm, 102 SM Tirino				Solvotrode	182	1	Yes	<u>0.5</u>	mV
1833	A	Automatic Titrator	used a commercial KOH solution			Combination electrode	175		Yes	5	mV
1842											
1850											
1854	11a,A		No	No	No	Metrohm 6.0229.100 LiCl2 mol/l in Ethanol	165		Yes	5.2705	mV
1900	A		used a commercial KOH solution				168	2	Yes	<u>1.026</u>	mV
1903											
1915											
2122											
3166											

A = boiled for 10 minutes

B = stand for 2 days

C = filtered

*) a commercial "ready for use" KOH standard solution was used

APPENDIX 3**Number of participants per country**

1 lab in ARGENTINA
1 lab in AUSTRALIA
1 lab in AUSTRIA
1 lab in AZERBAIJAN
3 labs in BELGIUM
1 lab in BOSNIA and HERZEGOVINA
1 lab in BRAZIL
3 labs in BULGARIA
1 lab in CHILE
1 lab in COLOMBIA
1 lab in CROATIA
1 lab in CZECH REPUBLIC
1 lab in ECUADOR
3 labs in FRANCE
2 labs in GERMANY
1 lab in GHANA
5 labs in GREECE
1 lab in HUNGARY
2 labs in INDIA
1 lab in INDONESIA
2 labs in ITALY
2 labs in KENYA
1 lab in KOREA
4 labs in MALAYSIA
1 lab in MAURITIUS
1 lab in NEGARA BRUNEI DARUSSALAM
1 lab in NIGERIA
3 labs in NORWAY
1 lab in P.R. of CHINA
1 lab in PAKISTAN
1 lab in PERU
1 lab in PHILIPPINES
1 lab in PORTUGAL
1 lab in RUSSIA
2 labs in SAUDI ARABIA
1 lab in SERBIA
1 lab in SINGAPORE
1 lab in SLOVENIA
5 labs in SPAIN
1 lab in SUDAN
2 labs in SWEDEN
1 lab in TANZANIA
3 labs in THAILAND
4 labs in THE NETHERLANDS
4 labs in TURKEY
1 lab in U.S.A.
10 labs in UNITED KINGDOM
1 lab in ZAMBIA

APPENDIX 4

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
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 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 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
- 9 DIN 38402 T41/42
- 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/>).