

**Results of Proficiency Test
Vacuum Gasoil (VGO)
December 2020**

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2013 the Institute for Interlaboratory Studies (iis) organizes a proficiency test (PT) for Vacuum Gasoil (VGO) every year. During the annual proficiency testing program 2020/2021 it was decided to continue the round robin for the analysis of Vacuum Gasoil based on the scope of the latest version of ISO8217.

In this interlaboratory study 67 laboratories in 30 different countries registered for participation. See appendix 2 for the number of participants per country. In this report the results of the Vacuum Gasoil proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.

It was decided to send two different samples of Vacuum Gasoil: a 1L bottle labelled #20235 for various analyzes and a 100mL PE bottle labelled #20236 specifically prepared for metal determinations.

The participants were requested to report rounded and unrounded test results. The unrounded test 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/IEC17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. 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 organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

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

For the first sample a batch of approximately 90 liters of Vacuum Gasoil was obtained from a third party. After homogenization 80 amber glass bottles of 1L were filled and labelled #20235.

The homogeneity of the subsamples was checked by determination of Density at 15 °C in accordance with ISO12185 on 8 stratified randomly selected subsamples.

	Density at 15°C in kg/m ³
Sample #20235-1	914.9
Sample #20235-2	914.9
Sample #20235-3	914.9
Sample #20235-4	914.9
Sample #20235-5	914.9
Sample #20235-6	914.9
Sample #20235-7	914.9
Sample #20235-8	914.9

Table 1: homogeneity test results of subsamples #20235

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2 in the next table.

	Density at 15°C in kg/m ³
r (observed)	0.00
reference test method	ISO12185:96
0.3 x R (reference test method)	0.45

Table 2: evaluation of the repeatability of subsamples #20235

The calculated repeatability is in agreement with 0.3 times the reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

For the second sample a batch of approximately 10 liters of Vacuum Gasoil was obtained from a third party and made positive on the metals Aluminum, Silicon, Copper and Nickel. After homogenization 80 PE bottles of 100 mL were filled and labelled #20236.

The homogeneity of the subsamples was checked by determination of Copper and Nickel in accordance with IP501 on 7 stratified randomly selected subsamples.

	Copper in mg/kg	Nickel in mg/kg
Sample #20236-1	8	11
Sample #20236-2	8	10
Sample #20236-3	8	11
Sample #20236-4	8	10
Sample #20236-5	9	9
Sample #20236-6	8	9
Sample #20236-7	8	10

Table 3: homogeneity test results of subsamples #20236

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

	Copper in mg/kg	Nickel in mg/kg
r (observed)	1	2
reference test method	IP621:16	IP501:05
0.3 x R (reference test method)	1	2

Table 4: evaluation of the repeatability of subsamples #20236

The calculated repeatabilities are in agreement with 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one sample labelled #20235 and one sample labelled #20236 were sent on November 4, 2020. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Vacuum Gasoil packed in amber glass bottles and PE bottles was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYZES

The participants were requested to determine on sample #20235: Total Acid Number, Aniline Point, Asphaltenes, Carbon Residue (micro method), Density at 15°C, Flash Point PMcc, Kinematic Viscosity (50°C and 100°C), Nitrogen, Pour Point (manual and automatic), Total Sulfur, Simulated Distillation and Distillation at 10 mmHg (IBP, 10% rec, 30% rec, 50% rec, 70% rec, 90% rec and FBP).

On sample #20236 was requested to determine: Aluminum, Silicon, Sum of Aluminum and Silicon, Arsenic, Copper, Iron, Nickel, Sodium, Vanadium, Calcium, Phosphorus and Zinc.

It was also requested to report some analytical details about Total Acid Number determination.

It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis/. The reported test results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test 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 reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5).

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test 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, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

According to ISO5725 the original test results per determination were submitted to Dixon's, Grubbs' or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's 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. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

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 for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on 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 reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve was projected over the Kernel Density Graph for reference.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation of this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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 whether the reported test result is fit-for-use.

The z-scores were calculated according to:

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

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

Absolute values for $z < 2$ are very common and absolute values for $z > 3$ are very rare.

The usual interpretation of z-scores is as follows:

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

4 EVALUATION

In this interlaboratory study some problems were encountered with the dispatch of the samples due to the COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with another three weeks. When considering the test results of the two samples together two participants reported test results after the final reporting date and three participants did not report any test results. Not all participants were able to report all tests requested.

In total 64 reporting laboratories submitted 1172 numerical test results. Observed were 37 outlying test results, which is 3.2%. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

Not all data sets proved to have a normal Gaussian distribution. These are referred to as “not OK” or “suspect”. The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

4.1 EVALUATION PER SAMPLE AND PER TEST

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

In the iis PT reports ASTM methods are referred to with a number (e.g. D611) and an added designation for the year that the method was adopted or revised (e.g. D611:12).

If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D611:12(2016)). In the results tables of appendix 1 only the method number and year of adoption or revision (e.g. D611:12) will be used.

Although VGO is an important feedstock for cracking installations, there are only a few analytical test methods specifically designed for the analysis of VGO. Most parameters are to be determined by using methods that are intended for residual fuel oil and blending components. Where applicable, precision data for Fuel Oil were used.

Sample #20235

Total Acid Number: This determination may be problematic dependent on the test method used. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of Inflection Point at titration volume 125 mL of ASTM D664-A:18e2. However, the calculated reproducibility is in agreement with the requirements for Inflection Point at titration volume 60 mL and Buffer End Point at titration volume 60 mL but not for Buffer End Point at 125 mL of ASTM D664-A:18e2.

When the test results reported for Inflection Point (60 and 125 mL) were evaluated separately, the calculated reproducibility is in agreement with the requirements of both IP 60 mL and 125 mL.

The majority of laboratories reported to use IP 125 mL, therefore the z-scores were calculated with this method reproducibility.

Aniline Point: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D611:12(2016). When test method D611-E and the group where n-heptane was in specification were evaluated separately the calculated reproducibilities were in agreement with the requirements for n-heptane of ASTM D611:12(2016), but still not for the group that used D611-E.

Asphaltenes: This determination was not problematic. All reporting participants agreed on a level <0.50 %M/M which is below the application range of test method IP143:04. Therefore, no z-scores were calculated.

Carbon Residue (micro method): 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 D4530:15(2020).

Density at 15°C: This determination was not problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO12185:96.

Flash Point PMcc: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with requirements of ASTM D93-B:20.

Kinematic Viscosity at 50°C: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D445:19a.

Kinematic Viscosity at 100°C: This determination was not problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D445:19a.

Nitrogen: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with ASTM D5762:18a. When the test results of ASTM D5762 Volumetric and Gravimetric test methods were evaluated separately, the calculated reproducibilities are again not in agreement with the requirements of ASTM D5762:18a.

Pour Point Manual: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with ASTM D97:17b.

Pour Point Automated: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full agreement with ASTM D5950:14(2020).

Total Sulfur: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ASTM D4294:16e1.

Simulated Distillation: This determination was problematic. In total, over seven parameters, two statistical outliers were observed. The calculated reproducibilities after rejection of the statistical outliers are in agreement with the requirements of ASTM D6352:19e1 for 10% and 90% recovered. For Initial Boiling Point the calculated reproducibility is not at all in agreement with the requirements of ASTM D6352:19e1, therefore no z-scores were calculated. For 30%, 50%, 70% recovered and Final Boiling Point the calculated reproducibilities are also not in agreement with the requirements of ASTM D6352:19e1.

Distillation at 10 mmHg as AET: This determination was not problematic. In total, over seven parameters, three statistical outliers were observed and one other test result was excluded. The calculated reproducibilities after rejection of the suspect data are in agreement with the requirements of ASTM D1160:18 for 10%, 30%, 50%, 70% recovered and Final Boiling Point. The calculated reproducibilities for Initial Boiling Point and 90% recovered are not in agreement with the requirements of ASTM D1160:18.

Sample #20236

Unfortunately, a suitable reference test method, providing the precision data, is not available for all determinations. For these tests the calculated reproducibility was compared against the estimated reproducibility calculated with the Horwitz equation.

Aluminum: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of IP501:05 and IP470:05.

- Silicon: This determination may be problematic depending on the test method used. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the strict requirements of IP501:05 but is in full agreement with the requirements of IP470:05.
- Sum of Aluminum and Silicon: This determination may be problematic dependent on the test method used. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of IP501:05 but is in agreement with the requirements of IP470:05.
- Iron: This determination may be problematic dependent on the test method used. Six statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of IP501:05 and IP470:05.
- Nickel: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of IP501:05 and IP470:05.
- Sodium: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of IP501:05 and in agreement with the requirements of IP470:05.
- Vanadium: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of IP501:05 and IP470:05.
- Calcium: This determination was not problematic. Almost all reporting participants agreed on a level <3 mg/kg which is below the application range of test method IP501:05. Therefore, no z-scores were calculated.
- Phosphorus: This determination was not problematic. Almost all of the reporting participants agreed on a level <1 mg/kg which is below the application range of test method IP501:05. Therefore, no z-scores were calculated.
- Zinc: This determination was not problematic. Almost all of the reporting participants agreed on a level <1 mg/kg which is below the application range of test method IP501:05. Therefore, no z-scores were calculated.
- Arsenic: This determination was not problematic. Most of the reporting participants agreed on a level <1 mg/kg. Therefore, no z-scores were calculated.
- Copper: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of IP621:16.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method or as declared by the estimated target reproducibility calculated with the Horwitz equation and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from reference test methods (in casu ASTM and IP test methods) or estimated by using the Horwitz equation are presented in the next table.

Parameter	unit	n	average	2.8 * sd	R(lit)
Total Acid Number	mg KOH/g	44	0.25	0.08	0.05
Aniline Point	°C	18	85.1	1.8	1.0
Asphaltenes	%M/M	34	<0.50	n.e.	n.e.
Carbon Residue (micro method)	%M/M	46	0.60	0.19	0.17
Density at 15°C	kg/m ³	57	915.2	1.3	1.5
Flash Point PMcc	°C	52	77.9	7.3	10
Kinematic Viscosity at 50°C	mm ² /s	55	36.71	1.109	3.106
Kinematic Viscosity at 100°C	mm ² /s	41	7.409	0.188	0.894
Nitrogen	mg/kg	35	1276	432	339
Pour Point Manual	°C	46	35.9	3.9	9
Pour Point Automated, 3°C interval	°C	20	34.5	6.6	6.1
Total Sulfur	%M/M	57	1.17	0.08	0.08
Simulated Distillation					
Initial Boiling Point	°C	14	(198.9)	(238)	(49.1)
Temp 10% recovered	°C	15	361.1	7.1	7.1
Temp 30% recovered	°C	15	408.2	8.5	5.9
Temp 50% recovered	°C	14	441.2	7.8	6.4
Temp 70% recovered	°C	14	478.2	8.8	7.2
Temp 90% recovered	°C	14	531.1	11.0	10.5
Final Boiling Point	°C	14	620.9	87.5	38.1
Distillation at 10 mmHg as AET					
Initial Boiling Point	°C	26	282.2	71.3	49.5
Temp 10% recovered	°C	27	380.5	15.9	17.2
Temp 30% recovered	°C	27	417.2	11.0	11.5
Temp 50% recovered	°C	27	447.5	11.9	11.3
Temp 70% recovered	°C	27	481.3	10.9	9.9
Temp 90% recovered	°C	24	534.9	16.0	10.1
Final Boiling Point	°C	24	549.1	24.0	26.9

Table 5: reproducibilities of tests on sample #20235

() results between brackets should be used with care

Parameter	unit	n	average	2.8 * sd	R(lit)
Aluminum as Al	mg/kg	33	11.4	4.1	3.8
Silicon as Si	mg/kg	23	5.7	4.8	1.9
Sum of Aluminum and Silicon	mg/kg	21	16.9	5.7	4.3
Iron as Fe	mg/kg	37	6.7	3.0	2.7
Nickel as Ni	mg/kg	46	10.0	3.9	6.0
Sodium as Na	mg/kg	41	6.0	2.6	2.9
Vanadium as V	mg/kg	47	3.5	1.7	3.5
Calcium as Ca	mg/kg	35	<3	n.e.	n.e.
Phosphorus as P	mg/kg	22	<1	n.e.	n.e.
Zinc as Zn	mg/kg	31	<1	n.e.	n.e.
Arsenic as As	mg/kg	6	<1	n.e.	n.e.
Copper as Cu	mg/kg	26	8.5	3.1	2.9

Table 6: reproducibilities of tests on sample #20236

Without further statistical calculations, it can be concluded that for many tests there is a good compliance of the group of participants with the reference test methods. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2020 WITH PREVIOUS PTS

	December 2020	December 2019	December 2018	December 2017	December 2016
Number of reporting laboratories	64	63	66	74	73
Number of test results	1172	1211	1113	1275	1241
Number of statistical outliers	37	69	39	61	53
Percentage of statistical outliers	3.2%	5.7%	3.5%	4.8%	4.3%

Table 7: comparison with previous proficiency tests

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

The performance of the determinations of the proficiency tests was compared to the requirements of the reference test methods. The conclusions are given the following table.

Determination	December 2020	December 2019	December 2018	December 2017	December 2016
Total Acid Number	-	-	++	+	++
Aniline Point	-	--	--	--	-
Asphaltenes	n.e.	n.e.	(--)*	(--)*	(--)*
Carbon Residue (micro method)	-	+	-	+/-	-
Density at 15°C	+	+/-	+/-	+/-	+/-
Flash Point PMcc	+	+	+/-	+	++
Kinematic Viscosity at 50°C	++	++	++	++	++
Kinematic Viscosity at 100°C	++	++	++	++	++

Determination	December 2020	December 2019	December 2018	December 2017	December 2016
Nitrogen	-	+	+	-	+/-
Pour Point Manual	++	+	++	++	++
Pour Point Automated 3°C interval	+/-	-	+	++	++
Total Sulfur	+/-	+/-	+/-	+	+
Simulated Distillation	-	-	+	-	+
Distillation at 10 mmHg as AET	+/-	+/-	+	+	-
Aluminum as Al	+/-	+/-	n.a.	n.a.	n.a.
Silicon as Si	--	-	n.e.	n.e.	n.e.
Sum of Aluminum and Silicon	-	+/-	n.a.	n.a.	n.a.
Iron as Fe	+/-	+/-	+	+	-
Nickel as Ni	+	+	+	++	++
Sodium as Na	+/-	+/-	++	++	+
Vanadium as V	++	+/-	+	++	++
Calcium as Ca	n.e.	+/-	-	+/-	+/-
Phosphorus as P	n.e.	+	n.a.	n.a.	n.a.
Zinc as Zn	n.e.	-	n.a.	n.a.	n.a.
Arsenic as As	n.e.	--	n.e.	n.e.	n.e.
Copper as Cu	+/-	++	n.e.	n.e.	n.e.

Table 8: comparison determinations against the reference test methods

()* results between brackets should be used with care because the assigned value was below the application range

The following performance categories were used:

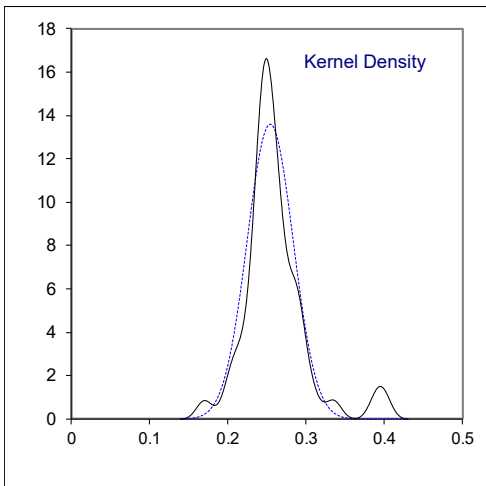
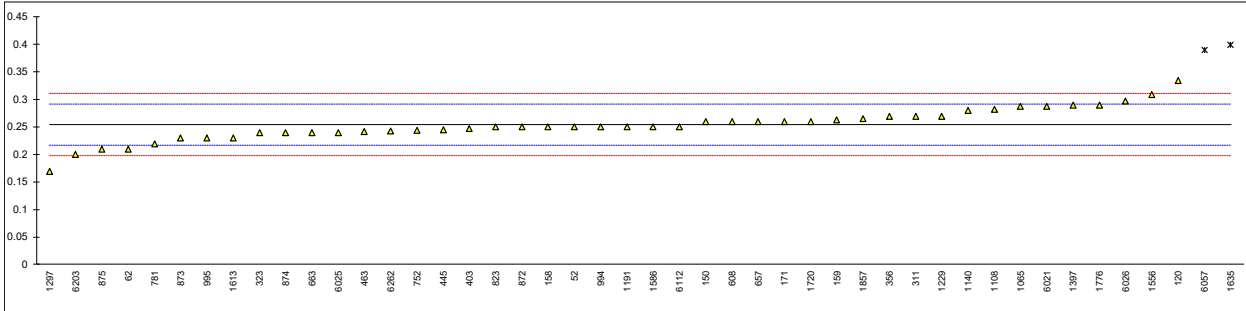
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance equals the reference test method
- : group performed worse than the reference test method
- : group performed much worse than the reference test method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

APPENDIX 1

Determination of Total Acid Number on sample #20235; results in mg KOH/g

lab	method	value	mark	z(targ)	end point	volume	remarks
52	D664-A	0.25		-0.22	Inflection Point	125 mL	
62	D664-A	0.21		-2.35	----	----	
120	D664-A	0.335	C	4.33	Buffer End Point pH 10	125 mL	fr. 0.136
140		----		----	Inflection Point	125 mL	
150	D664-A	0.26		0.32	Inflection Point	60 mL	
154		----		----	----	----	
158	D664-A	0.25		-0.22	Inflection Point	125 mL	
159	D664-A	0.263	C	0.48	----	----	fr. 0.33082
171	D664-A	0.26		0.32	----	----	
225		----		----	----	----	
311	D664-A	0.27		0.85	Buffer End Point pH 10	125 mL	
313		----		----	----	----	
317		----		----	----	----	
323	D664-A	0.24		-0.75	Inflection Point	125 mL	
333		----		----	----	----	
356	D664-B	0.27		0.85	Inflection Point	125 mL	
381		----		----	----	----	
403	D664-A	0.247		-0.38	Inflection Point	125 mL	
445	D664-A	0.245		-0.48	Inflection Point	60 mL	
463	D664-A	0.242		-0.64	Buffer End Point pH 11	125 mL	
541		----		----	----	----	
608	D664-A	0.260		0.32	Inflection Point	125 mL	
657	D664-A	0.26		0.32	Inflection Point	125 mL	
663	D664-A	0.24		-0.75	Inflection Point	125 mL	
732		----		----	----	----	
750		----		----	----	----	
752	D664-A	0.244		-0.54	Inflection Point	60 mL	
778		----		----	----	----	
781	D664-A	0.22		-1.82	Inflection Point	125 mL	
785		----		----	----	----	
798		----		----	----	----	
823	D664-A	0.25		-0.22	Inflection Point	125 mL	
872	D664-A	0.25		-0.22	Inflection Point	125 mL	
873	D664-A	0.23		-1.29	Buffer End Point pH 10	125 mL	
874	D664-A	0.24		-0.75	----	----	
875	D664-A	0.21		-2.35	----	----	
994	D664-A	0.25		-0.22	Inflection Point	125 mL	
995	D664-A	0.23		-1.29	Inflection Point	125 mL	
1011		----		----	----	----	
1016		----		----	----	----	
1065	D664-A	0.288		1.81	----	----	
1081		----		----	----	----	
1108	D664-A	0.2829	C	1.54	Inflection Point	125 mL	fr. 0.3251
1140	IP177	0.28		1.39	Inflection Point	125 mL	
1191	ISO6618	0.25		-0.22	----	----	
1205		----		----	----	----	
1229	ISO6618	0.27		0.85	----	----	
1297	D664-A	0.17		-4.49	Inflection Point	125 mL	
1397	D664-A	0.29		1.92	Inflection Point	125 mL	
1510		----		----	----	----	
1556	D664-A	0.309		2.94	Buffer End Point pH 10	125 mL	
1586	D664-A	0.25		-0.22	Inflection Point	125 mL	
1613	D664-A	0.23		-1.29	Inflection Point	125 mL	
1635	D664-A	0.40	R(0.01)	7.80	----	----	
1720	D664-A	0.26		0.32	----	----	
1776	D664-A	0.29		1.92	Buffer End Point pH 10	125 mL	
1857	D664-A	0.265		0.58	Inflection Point	125 mL	
1881		----		----	----	----	
6021	D664-A	0.288		1.81	Inflection Point	125 mL	
6024		----		----	----	----	
6025	D664-A	0.24		-0.75	Inflection Point	125 mL	
6026	D664-A	0.297		2.29	Buffer End Point pH 10	125 mL	
6057	D664-A	0.39	R(0.01)	7.26	----	----	
6112	D664-A	0.25		-0.22	Inflection Point	125 mL	
6203	D664-A	0.2		-2.89	Inflection Point	125 mL	
6238		----		----	----	----	
6262	D664-A	0.2427		-0.61	----	----	

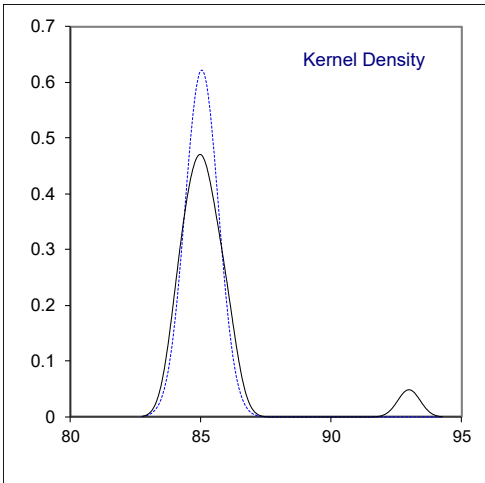
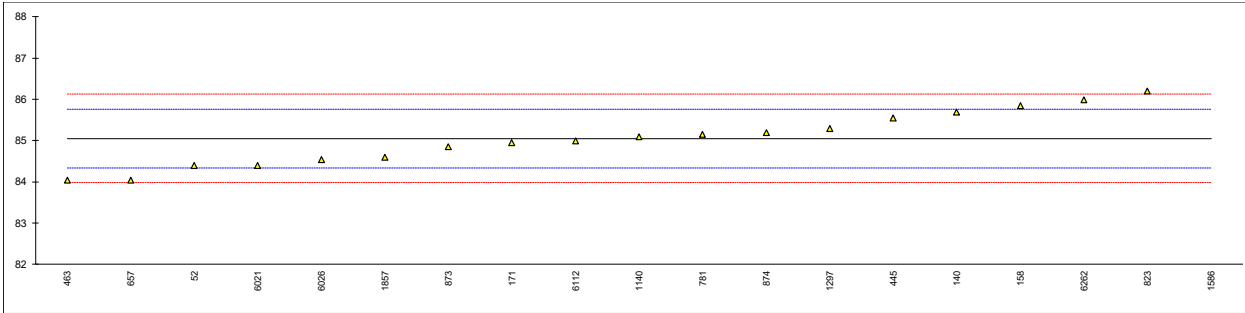
		<u>IP (60+125 mL) only</u>
normality	suspect	OK
n	44	26
outliers	2	1
mean (n)	0.2541	0.2516
st.dev. (n)	0.02939	0.02045
R(calc.)	0.0823	0.0573
st.dev.(D664-A:18e2 IP 125 mL)	0.01871	0.01853
R(D664-A:18e2 IP 125 mL)	0.0524	0.0519
Compare		
R(D664-A:18e2 BEP 125 mL)	0.0748	-----
R(D664-A:18e2 IP 60 mL)	0.1308	0.1297
R(D664-A:18e2 BEP 60 mL)	0.1455	-----



Determination of Aniline Point on sample #20235; results in °C

lab	method	value	mark	z(targ)	of n-heptane	remarks
52	D611-E	84.40		-1.82	69.50	
62		----		----	----	
120		----		----	----	
140	D611-B	85.70		1.82	----	
150		----		----	----	
154		----		----	----	
158	D611-E	85.85		2.24	----	
159		----		----	----	
171	D611-E	84.95		-0.28	----	
225		----		----	----	
311		----		----	----	
313		----		----	----	
317		----		----	----	
323		----		----	----	
333		----		----	----	
356		----		----	----	
381		----		----	----	
403		----		----	----	
445	IP2-B	85.55		1.40	69.30	test method IP2-A used for Aniline Point
463	D611-B	84.05		-2.80	69.15	test method D611-A used for Aniline Point
541		----		----	----	
608		----		----	----	
657	D611-B	84.05		-2.80	69.40	
663		----		----	----	
732		----		----	----	
750		----		----	----	
752		----		----	----	
778		----		----	----	
781	D611-E	85.15		0.28	69.30	
785		----		----	----	
798		----		----	----	
823	D611-E	86.2		3.22	----	
872		----		----	----	
873	D611-E	84.85		-0.56	69.30	
874	D611-E	85.2	C	0.42	69.3	first reported 83.2
875		----		----	----	
994		----		----	----	
995		----		----	----	
1011		----		----	----	
1016		----		----	----	
1065		----		----	----	
1081		----		----	----	
1108		----		----	----	
1140	D611-E	85.1		0.14	----	
1191		----		----	----	
1205		----		----	----	
1229		----		----	----	
1297	D611-E	85.3		0.70	69.3	
1397		----		----	----	
1510		----		----	----	
1556		----		----	----	
1586	D611-A	93.0	C,G(0.01)	22.26	----	first reported 83.0
1613		----	W	----	----	test result withdrawn, first reported 81.0
1635		----		----	----	
1720		----		----	----	
1776		----		----	----	
1857	D611-E	84.60		-1.26	69.15	
1881		----		----	----	
6021	D611-B	84.40		-1.82	----	
6024		----		----	----	
6025		----		----	----	
6026	D611-E	84.55		-1.40	69.3	
6057		----		----	----	
6112	D611-E	85.0		-0.14	----	
6203		----		----	----	
6238		----		----	----	
6262	D611-A	86.00	C	2.66	69.50	first reported 83.3

		<u>of n-heptane only</u>	<u>D611-E only</u>
normality	OK	OK	OK
n	18	11	12
outliers	1	0	0
mean (n)	85.050	69.318	85.096
st.dev. (n)	0.6410	0.1146	0.5189
R(calc.)	1.795	0.321	1.453
st.dev.(D611:12)	0.3571	0.1786	0.3571
R(D611:12)	1.0	1.0	1.0
Compare			
R(iis19G07)	1.998 at a mean of 80.746		



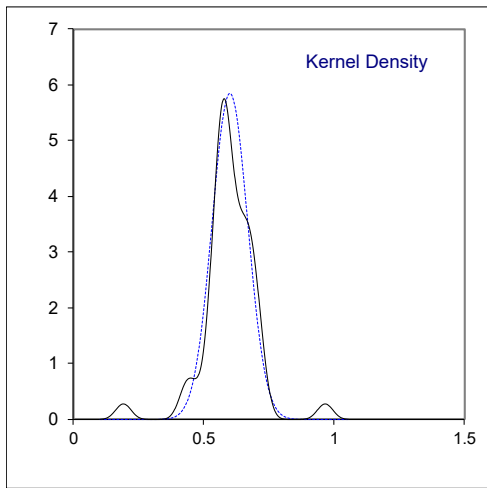
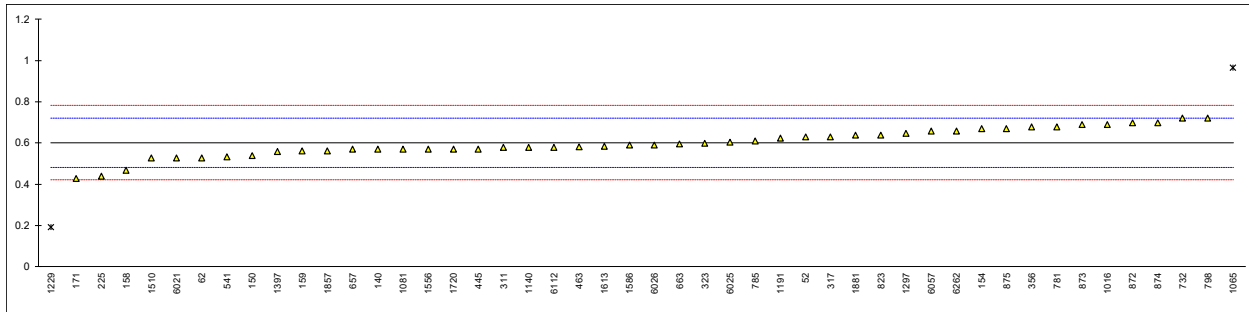
Determination of Asphaltenes on sample #20235; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	IP143	0.10		----	
62		----		----	
120		----		----	
140	IP143	0.10		----	
150	IP143	0.20		----	
154	IP143	0.05		----	
158		----		----	
159		----		----	
171	IP143	0.10		----	
225		----		----	
311		----		----	
313		----		----	
317		----		----	
323		----		----	
333		----		----	
356	IP143	Below 0.50		----	
381		----		----	
403		----		----	
445	IP143	0.082		----	
463	IP143	0.092		----	
541		----		----	
608	D6560	0.1544		----	
657	IP143	<0.50		----	
663		----		----	
732		----		----	
750		----		----	
752	INH-642	0.1364		----	
778		----		----	
781	INH-642	0.1453		----	
785		----		----	
798	INH-642	0.1483	C	----	first reported 1483 %M/M
823		----		----	
872	IP143	0.10		----	
873	IP143	0.10		----	
874	IP143	0.07		----	
875	IP143	<0.5		----	
994	IP143	0.16		----	
995	IP143	0.16		----	
1011		----		----	
1016	IP143	0.11		----	
1065	D6560	0.298		----	
1081		----		----	
1108		----		----	
1140	IP143	0.09		----	
1191		----		----	
1205		----		----	
1229		----		----	
1297		----		----	
1397	D6560	<0,5		----	
1510	IP143	0.05		----	
1556	IP143	0.09		----	
1586	IP143	0.136		----	
1613	D6560	0.05		----	
1635	D6560	0.04		----	
1720		----		----	
1776		----		----	
1857	INH-642	0.154		----	
1881	INH-642	0.1558		----	
6021		----		----	
6024		----		----	
6025		----		----	
6026	INH-642	0.1595		----	
6057	IP143	0.11		----	
6112		----		----	
6203	IP143	0.066		----	
6238		----		----	
6262	IP143	<0.50	C	----	first reported 0.53
n		34			
mean (n)		<0.50			precision applicable between 0.50 – 30 %M/M

Determination of Carbon Residue (micro method) on sample #20235; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	D4530	0.63		0.47	
62	D4530	0.53		-1.19	
120		----		----	
140	D4530	0.57		-0.52	
150	D4530	0.54		-1.02	
154	D4530	0.67		1.14	
158	D4530	0.47		-2.18	
159	D4530	0.56145		-0.66	
171	D4530	0.43		-2.85	
225	D4530	0.44		-2.68	
311	D4530	0.58		-0.36	
313		----		----	
317	D4530	0.632		0.51	
323	D4530	0.60		-0.02	
333		----		----	
356	D4530	0.68		1.31	
381		----		----	
403		----		----	
445	D4530	0.5724		-0.48	
463	D4530	0.583		-0.31	
541	D4530	0.535		-1.10	
608		----		----	
657	D4530	0.57		-0.52	
663	D4530	0.598		-0.06	
732	D4530	0.721		1.99	
750		----		----	
752		----		----	
778		----		----	
781	D4530	0.68		1.31	
785	D4530	0.61		0.14	
798	D4530	0.7211		1.99	
823	ISO10370	0.64	C	0.64	first reported 0.32
872	D4530	0.70		1.64	
873	D4530	0.69		1.47	
874	D4530	0.70		1.64	
875	D4530	0.67		1.14	
994		----		----	
995		----		----	
1011		----		----	
1016	ISO10370	0.69		1.47	
1065	D4530	0.9673	C,R(0.01)	6.08	first reported 0.298
1081	ISO10370	0.57		-0.52	
1108		----		----	
1140	D4530	0.58		-0.36	
1191	ISO10370	0.62465		0.39	
1205		----		----	
1229	ISO10370	0.1922949	R(0.01)	-6.80	
1297	D4530	0.649		0.79	
1397	D4530	0.56		-0.69	
1510	D4530	0.528		-1.22	
1556	ISO10370	0.57		-0.52	
1586	D4530	0.59		-0.19	
1613	D4530	0.585		-0.27	
1635		----		----	
1720	D4530	0.57		-0.52	
1776		----		----	
1857	D4530	0.562		-0.65	
1881	ISO10370	0.639		0.62	
6021	D4530	0.529		-1.20	
6024		----		----	
6025	D4530	0.6049		0.06	
6026	ISO10370	0.59		-0.19	
6057	D4530	0.66		0.97	
6112	ISO10370	0.58		-0.36	
6203		----		----	
6238		----		----	
6262	D4530	0.66		0.97	

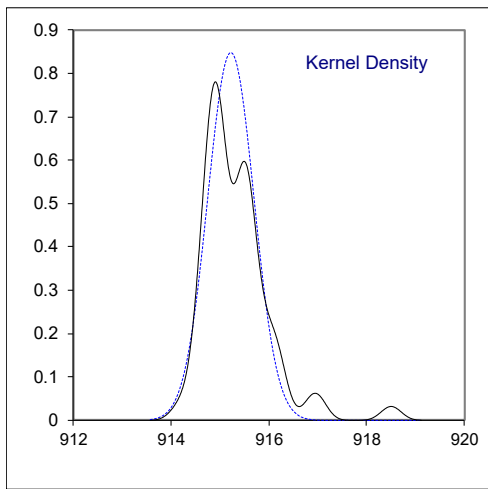
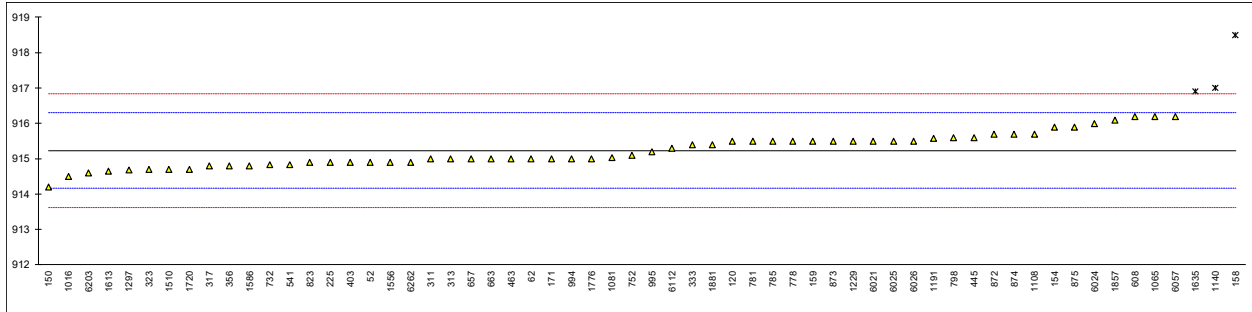
normality	OK
n	46
outliers	2
mean (n)	0.6014
st.dev. (n)	0.06822
R(calc.)	0.1910
st.dev.(D4530:15)	0.06021
R(D4530:15)	0.1686
Compare	
R(ISO10370)	0.0549



Determination of Density at 15°C on sample #20235; results in kg/m³

lab	method	value	mark	z(targ)	remarks
52	D4052	914.9		-0.61	
62	D4052	915.0		-0.43	
120	D4052	915.5		0.51	
140		-----		-----	
150	D1298	914.2		-1.92	
154	D1298	915.9		1.25	
158	D4052	918.5	G(0.01)	6.11	
159	D4052	915.5		0.51	
171	ISO12185	915.0		-0.43	
225	D4052	914.9		-0.61	
311	ISO12185	915.0		-0.43	
313	ISO12185	915.0		-0.43	
317	ISO12185	914.8		-0.80	
323	ISO12185	914.7		-0.99	
333	ISO12185	915.4		0.32	
356	ISO12185	914.8		-0.80	
381		-----		-----	
403	ISO12185	914.9		-0.61	
445	IP365	915.6		0.69	
463	ISO12185	915.0		-0.43	
541	ISO12185	914.83		-0.74	
608	D4052	916.19		1.80	
657	D4052	915.0		-0.43	
663	D4052	915.00		-0.43	
732	ISO12185	914.83		-0.74	
750		-----		-----	
752	ISO3675	915.1		-0.24	
778	ISO12185	915.5		0.51	
781	ISO12185	915.5		0.51	
785	ISO12185	915.5		0.51	
798	D1298	915.6		0.69	
823	ISO12185	914.9		-0.61	
872	ISO12185	915.7		0.88	
873	ISO12185	915.5		0.51	
874	ISO12185	915.7		0.88	
875	ISO12185	915.9		1.25	
994	ISO12185	915.0		-0.43	
995	ISO12185	915.2		-0.05	
1011		-----		-----	
1016	ISO12185	914.5		-1.36	
1065	D4052	916.2		1.81	
1081	D4052	915.04		-0.35	
1108	D1298	915.7		0.88	
1140	IP365	917.0	DG(0.01)	3.31	
1191	ISO12185	915.58		0.66	
1205		-----		-----	
1229	ISO12185	915.5		0.51	
1297	D4052	914.69		-1.00	
1397		-----		-----	
1510	D4052	914.7		-0.99	
1556	ISO12185	914.9		-0.61	
1586	D4052	914.8		-0.80	
1613	D4052	914.65		-1.08	
1635	ISO3675	916.9	DG(0.01)	3.12	
1720	D4052	914.7		-0.99	
1776	ISO12185	915.0		-0.43	
1857	ISO12185	916.1		1.63	
1881	ISO12185	915.4		0.32	
6021	ISO12185	915.5		0.51	
6024	D1298	916.0		1.44	
6025	D1298	915.5		0.51	
6026	ISO12185	915.5		0.51	
6057	ISO12185	916.2		1.81	
6112	ISO12185	915.3		0.13	
6203	D4052	914.6		-1.17	
6238		-----		-----	
6262	D4052	914.9		-0.61	

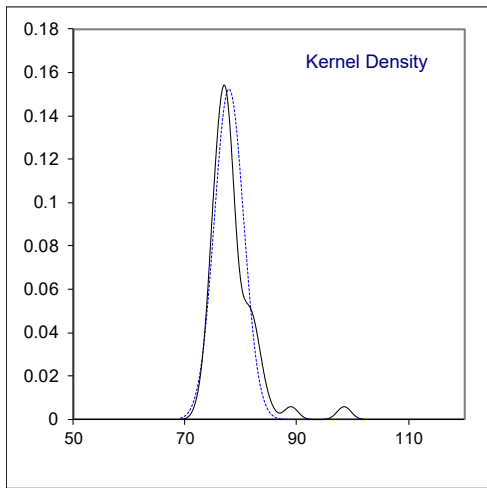
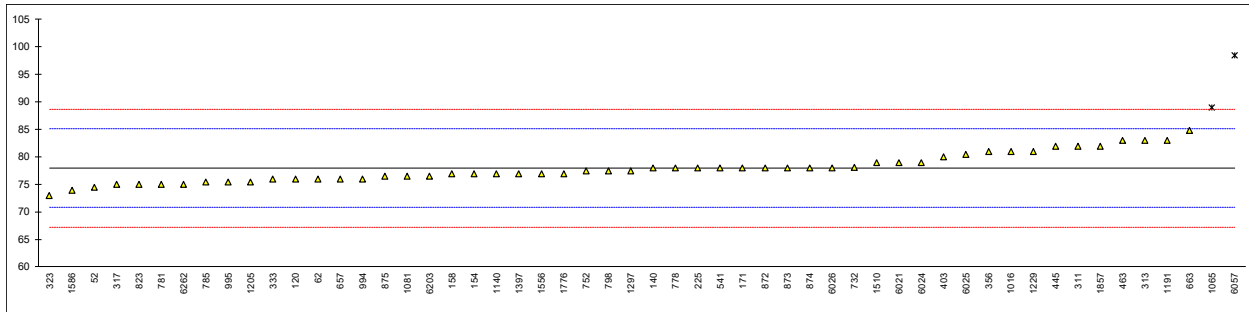
normality	OK
n	57
outliers	3
mean (n)	915.23
st.dev. (n)	0.471
R(calc.)	1.32
st.dev.(ISO12185:96)	0.536
R(ISO12185:96)	1.5



Determination of Flash Point PMcc on sample #20235; results in °C

lab	method	value	mark	z(targ)	remarks
52	D93-B	74.5		-0.96	
62	D93-B	76.0	C	-0.54	first reported 97.5
120	D93-B	76.0		-0.54	
140	D93-B	78.0		0.02	
150		----		----	
154	D93-A	77.0		-0.26	
158	D93-B	77.0		-0.26	
159		----		----	
171	D93-B	78.0		0.02	
225	D93-A	78.0		0.02	
311	D93-B	82.0		1.14	
313	D93-B	83.0		1.42	
317	D93-B	75.0		-0.82	
323	D93-B	73.0		-1.38	
333	D93-B	76.0	C	-0.54	first reported D93-A: 88.0
356	D93-B	81.0		0.86	
381		----		----	
403	D93-A	80.0		0.58	
445	D93-B	82.0		1.14	
463	D93-B	83.0		1.42	
541	ISO2719	78.00		0.02	
608		----		----	
657	D93-B	76.0		-0.54	
663	D93-B	84.8		1.92	
732	ISO2719	78.1		0.05	
750		----		----	
752	ISO2719	77.50		-0.12	
778	D93-B	78.0		0.02	
781	D93-B	75.0		-0.82	
785	D93-B	75.5		-0.68	
798	D93-B	77.5		-0.12	
823	ISO2719	75.0		-0.82	
872	D93-B	78		0.02	
873	D93-B	78.0		0.02	
874	D93-B	78.0		0.02	
875	D93-B	76.5		-0.40	
994	D93-B	76.0		-0.54	
995	D93-B	75.5		-0.68	
1011		----		----	
1016	D93-A	81.0		0.86	
1065	D93-A	89	R(0.01)	3.10	
1081	D93-B	76.5		-0.40	
1108		----		----	
1140	D93-B	77.0		-0.26	
1191	ISO2719	83.0		1.42	
1205	D93-B	75.5		-0.68	
1229	ISO2719	81		0.86	
1297	D93-B	77.5		-0.12	
1397	D93-B	77.0		-0.26	
1510	D93-B	79.0		0.30	
1556	ISO2719	77.0		-0.26	
1586	D93-B	74.0		-1.10	
1613	D93-B	--		----	
1635		----		----	
1720		----		----	
1776	ISO2719	77.0		-0.26	
1857	D93-A	82.0		1.14	
1881		----		----	
6021	D93-B	79.0		0.30	
6024	D93-B	79.0		0.30	
6025	D93-B	80.5		0.72	
6026	ISO2719	78.0		0.02	
6057	D93-B	98.5	R(0.01)	5.76	
6112		----		----	
6203	D93-B	76.5		-0.40	
6238		----		----	
6262	D93-B	75.0		-0.82	

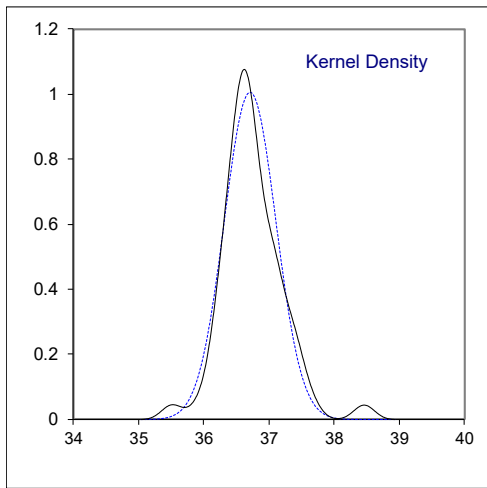
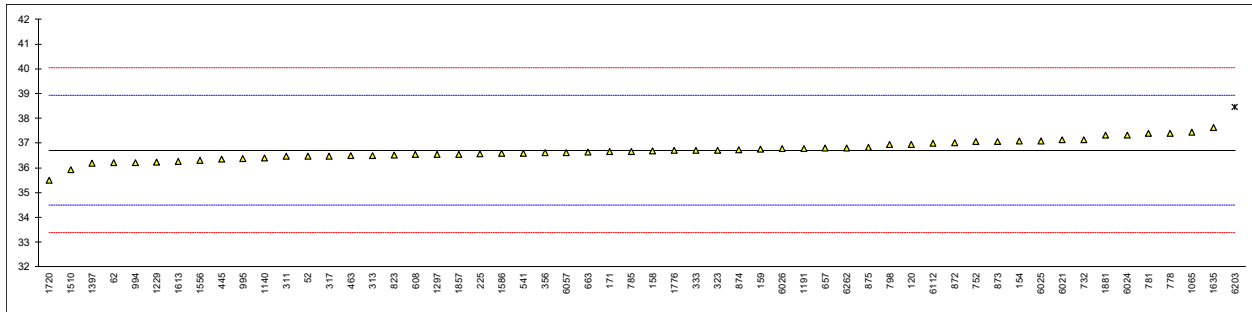
normality	OK
n	52
outliers	2
mean (n)	77.93
st.dev. (n)	2.622
R(calc.)	7.34
st.dev.(D93-B:20)	3.571
R(D93-B:20)	10
Compare	
R(ISO2719:16)	10



Determination of Kinematic Viscosity at 50°C on sample #20235; results in mm²/s

lab	method	value	mark	z(targ)	remarks
52	D445	36.47		-0.22	
62	D445	36.21		-0.45	
120	D445	36.951		0.22	
140		----		----	
150		----		----	
154	D445	37.09		0.34	
158	D445	36.70		-0.01	
159	D445	36.75		0.04	
171	D445	36.66		-0.04	
225	D445	36.58		-0.12	
311	D445	36.47		-0.22	
313	D445	36.50		-0.19	
317	D445	36.48		-0.21	
323	D445	36.72		0.01	
333	D445	36.72		0.01	
356	D445	36.61		-0.09	
381		----		----	
403		----		----	
445	D445	36.369		-0.31	
463	ISO3104	36.495		-0.19	
541	D445	36.602		-0.10	
608	D445	36.545		-0.15	
657	D445	36.80		0.08	
663	D445	36.648		-0.05	
732	D445	37.136		0.39	
750		----		----	
752	ISO3104	37.075		0.33	
778	D445	37.40		0.62	
781	D445	37.39		0.61	
785	D445	36.66		-0.04	
798	D445	36.9394		0.21	
823	ISO3104	36.52		-0.17	
872	D445	37.02		0.28	
873	D445	37.08		0.33	
874	D445	36.74		0.03	
875	D445	36.84		0.12	
994	D445	36.21		-0.45	
995	D445	36.38		-0.30	
1011		----		----	
1016		----		----	
1065	D445	37.44		0.66	
1081		----		----	
1108		----		----	
1140	D445	36.3956		-0.28	
1191	ISO3104	36.794		0.08	
1205		----		----	
1229	ISO3104	36.25		-0.41	
1297	D7042	36.546		-0.15	
1397	D7042	36.19		-0.47	
1510	D445	35.94		-0.69	
1556	ISO3104	36.314		-0.36	
1586	D445	36.59		-0.11	
1613	D445	36.27		-0.40	
1635	D445	37.64		0.84	
1720	D445	35.51		-1.08	
1776	ISO3104	36.710		0.00	
1857	D445	36.554		-0.14	
1881	D445	37.32		0.55	
6021	D445	37.130		0.38	
6024	D445	37.32		0.55	
6025	D445	37.10		0.35	
6026	ISO3104	36.790		0.07	
6057	D445	36.62		-0.08	
6112	D445	37.00		0.26	
6203	D7042	38.46	R(0.01)	1.58	
6238		----		----	
6262	D445	36.805		0.09	

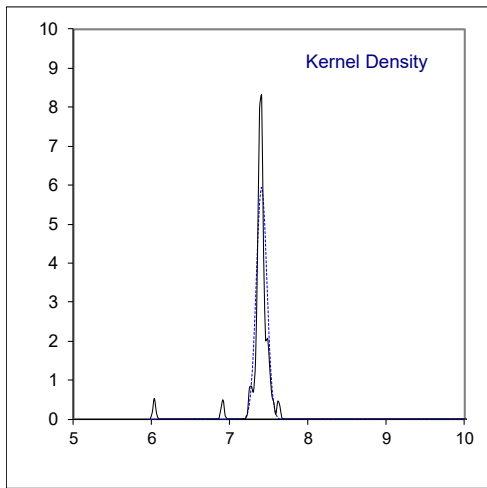
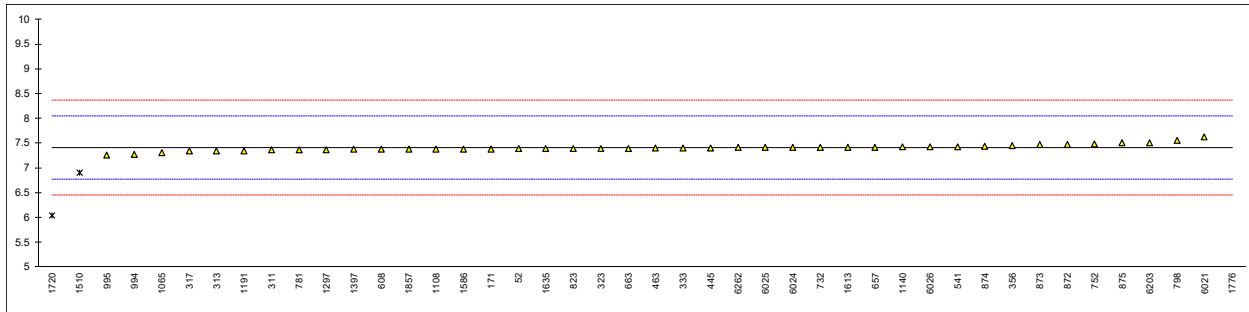
normality	OK
n	55
outliers	1
mean (n)	36.7089
st.dev. (n)	0.39624
R(calc.)	1.1095
st.dev.(D445:19a)	1.10926
R(D445:19a)	3.1059
Compare	
R(ISO3104:20)	3.1059



Determination of Kinematic Viscosity at 100°C on sample #20235; results in mm²/s

lab	method	value	mark	z(targ)	remarks
52	D445	7.388		-0.07	
62		----		----	
120		----		----	
140		----		----	
150		----		----	
154		----		----	
158		----		----	
159		----		----	
171	D445	7.386		-0.07	
225		----		----	
311	D445	7.368		-0.13	
313	D445	7.347		-0.19	
317	D445	7.345		-0.20	
323	D445	7.397		-0.04	
333	D445	7.404		-0.02	
356	D445	7.454		0.14	
381		----		----	
403		----		----	
445	D445	7.409		0.00	
463	ISO3104	7.4030		-0.02	
541	D445	7.4290		0.06	
608	D445	7.377		-0.10	
657	D445	7.421		0.04	
663	D445	7.3977		-0.03	
732	D445	7.4147		0.02	
750		----		----	
752	ISO3104	7.482		0.23	
778		----		----	
781	D445	7.369		-0.12	
785		----		----	
798	D445	7.5522		0.45	
823	D445	7.395		-0.04	
872	D445	7.481		0.23	
873	D445	7.479		0.22	
874	D445	7.445		0.11	
875	D445	7.507		0.31	
994	D445	7.272		-0.43	
995	D445	7.259		-0.47	
1011		----		----	
1016		----		----	
1065	D445	7.310		-0.31	
1081		----		----	
1108	D445	7.385		-0.07	
1140	D445	7.4240		0.05	
1191	ISO3104	7.3491		-0.19	
1205		----		----	
1229		----		----	
1297	D7042	7.3727		-0.11	
1397	D7042	7.375		-0.11	
1510	D445	6.911	R(0.01)	-1.56	
1556		----		----	
1586	D445	7.385		-0.07	
1613	D445	7.415		0.02	
1635	D445	7.392		-0.05	
1720	D445	6.039	R(0.01)	-4.29	
1776	ISO3104	12.529	R(0.01)	16.05	
1857	D445	7.3786		-0.09	
1881		----		----	
6021	D445	7.6254		0.68	
6024	D445	7.412		0.01	
6025	D445	7.411		0.01	
6026	ISO3104	7.4269		0.06	
6057		----		----	
6112		----		----	
6203	D7042	7.510		0.32	
6238		----		----	
6262	D445	7.4103		0.00	

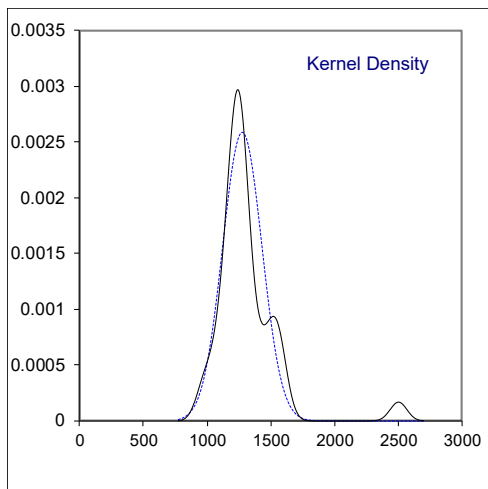
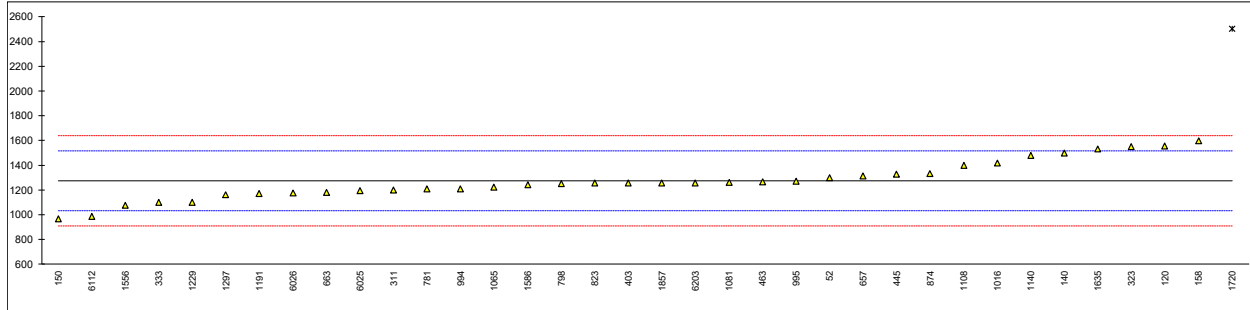
normality	not OK
n	41
outliers	3
mean (n)	7.4089
st.dev. (n)	0.06714
R(calc.)	0.1880
st.dev.(D445:19a)	0.31911
R(D445:19a)	0.8935
Compare	
R(ISO3104:20)	0.8935



Determination of Nitrogen on sample #20235; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	D4629	1300		0.20	
62		----		----	
120	D4629	1556.932		2.32	
140	D5762 Volumetric	1500		1.85	
150	D5762 Volumetric	970		-2.52	
154		----		----	
158	D5762 Gravimetric	1600		2.67	
159		----		----	
171		----		----	
225		----		----	
311	D5762 Volumetric	1200		-0.63	
313		----		----	
317		----		----	
323	D5762 Gravimetric	1554		2.29	
333	D5762 Volumetric	1100		-1.45	
356		----		----	
381		----		----	
403	D5762 Gravimetric	1260.0		-0.13	
445	D5762 Gravimetric	1330		0.44	
463	D5762 Gravimetric	1268		-0.07	
541		----		----	
608		----		----	
657	D5762 Gravimetric	1316.22		0.33	
663	D5762 Gravimetric	1183		-0.77	
732		----		----	
750		----		----	
752		----		----	
778		----		----	
781	D3228	1210		-0.55	
785		----		----	
798	D5762	1255		-0.17	
823	D5762 Gravimetric	1260		-0.13	
872		----		----	
873		----		----	
874	D5762 Volumetric	1336		0.49	
875		----		----	
994	D5762 Volumetric	1210		-0.55	
995	D3228	1275		-0.01	
1011		----		----	
1016	D5762 Gravimetric	1421		1.20	
1065	D5762 Gravimetric	1227		-0.40	
1081	D5762 Volumetric	1264		-0.10	
1108	D5762 Gravimetric	1402		1.04	
1140	D4629	1482.7154		1.70	
1191	D5762 Gravimetric	1174		-0.84	
1205		----		----	
1229	D4629	1100		-1.45	
1297	D4629	1164.1		-0.92	
1397		----	W	----	test result withdrawn, first reported 900
1510		----		----	
1556	D5762 Volumetric	1080		-1.62	
1586	D5762 Volumetric	1246		-0.25	
1613		----		----	
1635	D5762 Gravimetric	1535		2.14	
1720	D4629	2503.3	R(0.01)	10.12	
1776		----		----	
1857	D5762 Gravimetric	1260		-0.13	
1881		----		----	
6021		----		----	
6024		----		----	
6025	D5762 Volumetric	1196		-0.66	
6026	D5762 Gravimetric	1177		-0.82	
6057		----		----	
6112	D5762 Volumetric	990		-2.36	
6203	D4629	1260		-0.13	
6238		----		----	
6262		----		----	

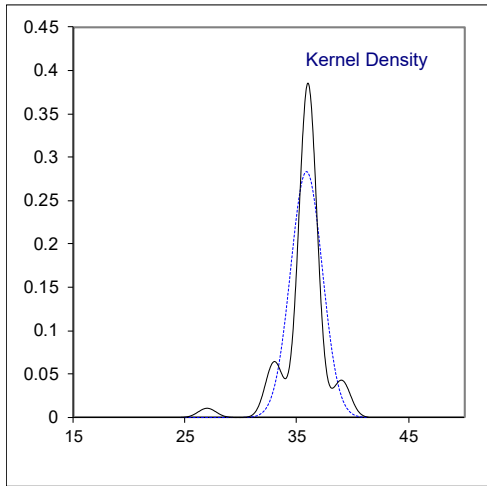
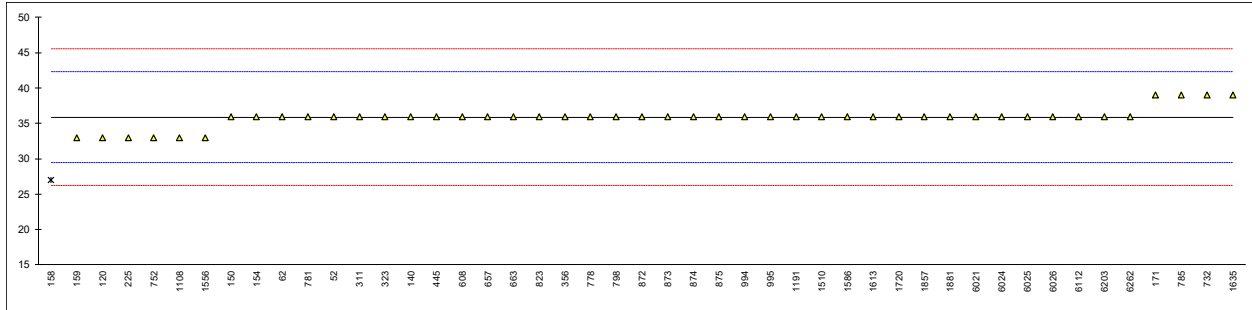
		<u>D5762 Volumetric only</u>	<u>D5762 Gravimetric only</u>
normality	OK	OK	OK
n	35	11	15
outliers	1	0	0
mean (n)	1276.08	1190.18	1331.15
st.dev. (n)	154.332	153.350	140.723
R(calc.)	432.13	429.38	394.03
st.dev.(D5762:18a)	121.228	113.067	126.459
R(D5762:18a)	339.44	316.59	354.09



Determination of Pour Point Manual on sample #20235; results in °C

lab	method	value	mark	z(targ)	remarks
52	D97	36		0.04	
62	D97	36		0.04	
120	D97	33		-0.89	
140	D97	36		0.04	
150	D97	36		0.04	
154	D97	36		0.04	
158	D97	27	R(0.01)	-2.76	
159	D97	33.0		-0.89	
171	D97	39		0.97	
225	D97	33		-0.89	
311	D97	36		0.04	
313		----		----	
317		----		----	
323	D97	36		0.04	
333		----		----	
356	D97	36		0.04	
381		----		----	
403		----		----	
445	D97	36		0.04	
463		----		----	
541		----		----	
608	D97	36		0.04	
657	D97	36		0.04	
663	D97	36		0.04	
732	D97	39		0.97	
750		----		----	
752	D97	33		-0.89	
778	D97	36		0.04	
781	D97	36		0.04	
785	D97	39		0.97	
798	D97	36		0.04	
823	ISO3016	36		0.04	
872	D97	36		0.04	
873	D97	36		0.04	
874	D97	36		0.04	
875	D97	36		0.04	
994	D97	36		0.04	
995	D97	36		0.04	
1011		----		----	
1016		----		----	
1065		----		----	
1081		----		----	
1108	D97	33	C	-0.89	first reported 27
1140		----		----	
1191	ISO3016	36		0.04	
1205		----		----	
1229		----		----	
1297		----		----	
1397		----		----	
1510	D97	36		0.04	
1556	ISO3016	33.0		-0.89	
1586	D97	36		0.04	
1613	D97	36		0.04	
1635	D97	39		0.97	
1720		36		0.04	
1776		----		----	
1857	D97	36		0.04	
1881	D97	36		0.04	
6021	D97	36		0.04	
6024	D97	36		0.04	
6025	D97	36		0.04	
6026	ISO3016	36		0.04	
6057		----		----	
6112	D97	36		0.04	
6203	D97	36		0.04	
6238		----		----	
6262	D97	36		0.04	

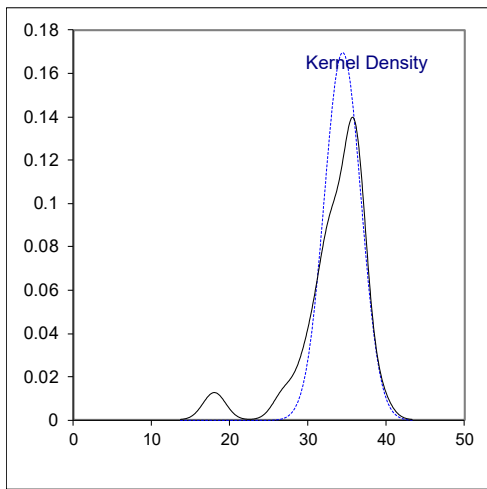
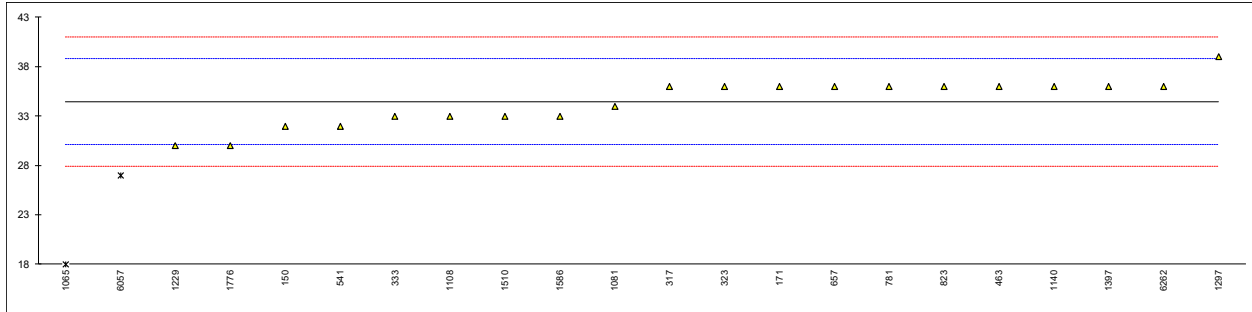
normality	suspect
n	46
outliers	1
mean (n)	35.87
st.dev. (n)	1.408
R(calc.)	3.94
st.dev.(D97:17b)	3.214
R(D97:17b)	9
Compare	
R((ISO3016:19)	9



Determination of Pour Point Automated 3°C interval on sample #20235; results in °C

lab	method	value	mark	z(targ)	remarks
52		----		----	
62		----		----	
120		----		----	
140		----		----	
150	D5950	32		-1.12	
154		----		----	
158		----		----	
159		----		----	
171	D5950	36		0.71	
225		----		----	
311		----		----	
313		----		----	
317	D6749	36		0.71	
323	D5950	36		0.71	
333	D5950	33		-0.67	
356		----		----	
381		----		----	
403		----		----	
445		----		----	
463	D6892	36		0.71	
541	D5950	32		-1.12	
608		----		----	
657	D5950	36		0.71	
663		----		----	
732		----		----	
750		----		----	
752		----		----	
778		----		----	
781	D5950	36		0.71	
785		----		----	
798		----		----	
823	D5950	36		0.71	
872		----		----	
873		----		----	
874		----		----	
875		----		----	
994		----		----	
995		----		----	
1011		----		----	
1016		----		----	
1065	D5950	18	C,R(0.01)	-7.55	first reported 27
1081	D5950	34		-0.21	
1108	D5950	33	C	-0.67	first reported 27
1140	D5950	36		0.71	
1191		----		----	
1205		----		----	
1229	D5950	30		-2.04	
1297	D5949	39.0		2.09	
1397	D5950	36		0.71	
1510	D5950	33		-0.67	
1556		----		----	
1586	D5950	33		-0.67	
1613		----		----	
1635		----		----	
1720		----		----	
1776	D5950	30		-2.04	
1857		----		----	
1881		----		----	
6021		----		----	
6024		----		----	
6025		----		----	
6026		----		----	
6057	D5950	27	R(0.05)	-3.42	
6112		----		----	
6203		----		----	
6238		----		----	
6262	D5950	36.0		0.71	

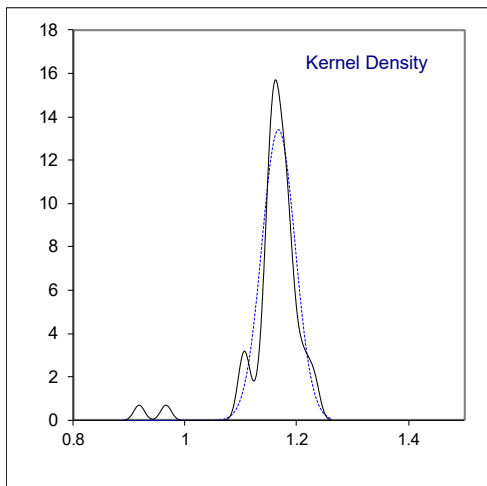
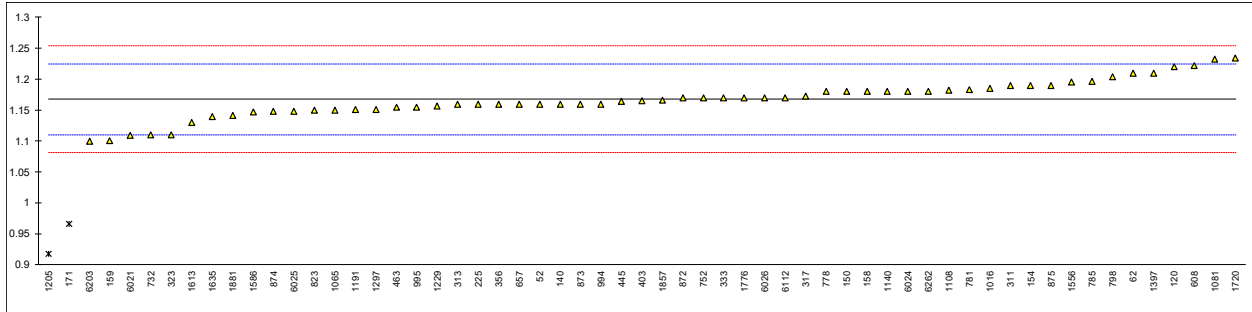
normality	OK
n	20
outliers	2
mean (n)	34.450
st.dev. (n)	2.3503
R(calc.)	6.581
st.dev.(D5950:14)	2.1786
R(D5950:14)	6.1



Determination of Total Sulfur on sample #20235; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	D4294	1.16		-0.26	
62	D4294	1.21		1.48	
120	D4294	1.22033		1.84	
140	D4294	1.16		-0.26	
150	D4294	1.18		0.43	
154	D4294	1.19		0.78	
158	D4294	1.18		0.43	
159	D4294	1.10092		-2.32	
171	D4294	0.966	R(0.01)	-7.03	
225	D4294	1.16		-0.26	
311	ISO8754	1.19		0.78	
313	ISO8754	1.16		-0.26	
317	D2622	1.173		0.19	
323	D4294	1.11		-2.01	
333	D4294	1.17		0.09	
356	D4294	1.16		-0.26	
381		----		----	
403	D4294	1.165		-0.09	
445	D4294	1.164		-0.12	
463	D4294	1.155		-0.44	
541		----		----	
608	D4294	1.2221		1.90	
657	D4294	1.16		-0.26	
663		----		----	
732	D4294	1.110		-2.01	
750		----		----	
752	ISO8754	1.17		0.09	
778	D4294	1.18		0.43	
781	D4294	1.183		0.54	
785	D4294	1.197		1.03	
798	D4294	1.204		1.27	
823	ISO8754	1.15		-0.61	
872	D4294	1.17		0.09	
873	D4294	1.16		-0.26	
874	D4294	1.148		-0.68	
875	D4294	1.19		0.78	
994	D4294	1.16		-0.26	
995	D4294	1.155		-0.44	
1011		----		----	
1016		1.185		0.61	
1065	D4294	1.15		-0.61	
1081	D4294	1.233		2.28	
1108	D4294	1.1823		0.52	
1140	D2622	1.180		0.43	
1191	ISO8754	1.151		-0.58	
1205	ISO14596	0.918	R(0.01)	-8.70	
1229	ISO8754	1.157		-0.37	
1297	D4294	1.151		-0.58	
1397	D2622	1.21		1.48	
1510	D4294	<17		----	
1556	ISO8754	1.196		0.99	
1586	D4294	1.147		-0.72	
1613	D4294	1.13		-1.31	
1635	D4294	1.14		-0.96	
1720	D4294	1.2341		2.32	
1776	ISO8754	1.17		0.09	
1857	D4294	1.166		-0.05	
1881	ISO8754	1.142		-0.89	
6021	D4294	1.109		-2.04	
6024	D4294	1.18		0.43	
6025	D4294	1.148		-0.68	
6026	IP336	1.17		0.09	
6057		----		----	
6112	D4294	1.17		0.09	
6203	D2622	1.1		-2.36	
6238		----		----	
6262	D4294	1.1804		0.45	

normality	OK
n	57
outliers	2
mean (n)	1.1675
st.dev. (n)	0.02973
R(calc.)	0.0832
st.dev.(D4294:16e1)	0.02867
R(D4294:16e1)	0.0803



Determination of Simulated Distillation on sample #20235; result in °C

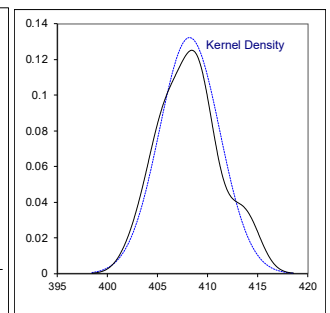
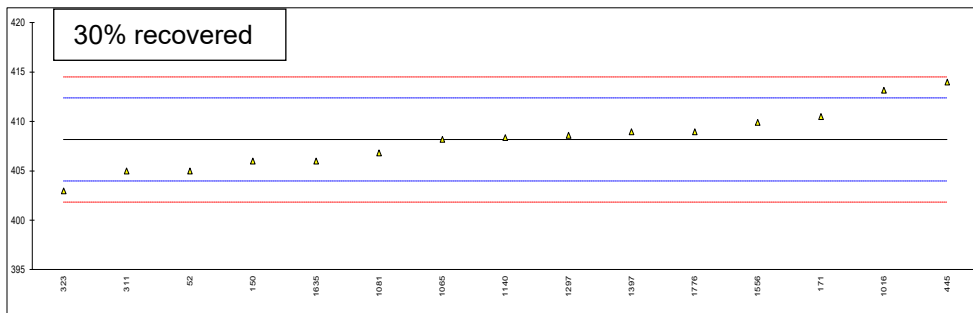
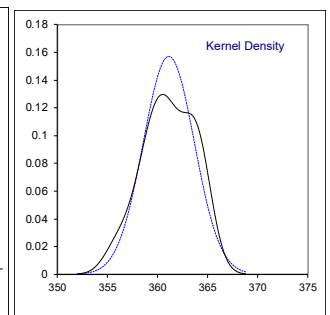
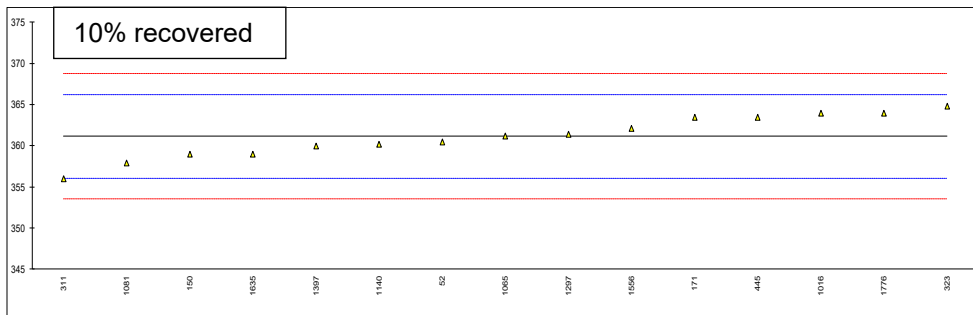
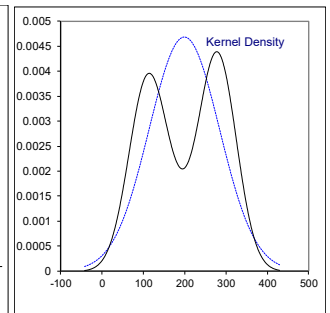
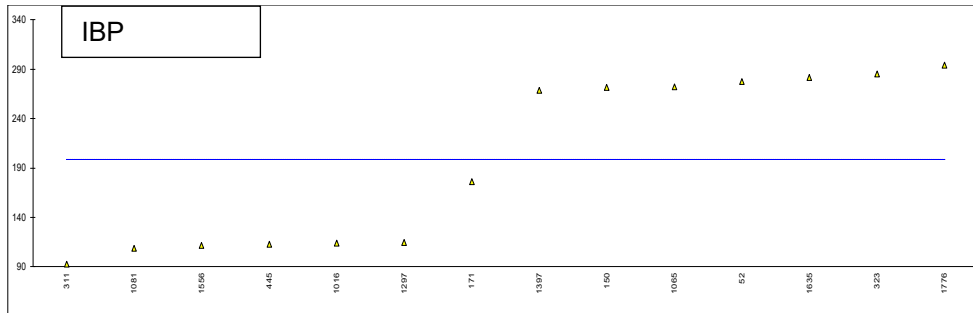
lab	method	IBP	10%	30%	50%	70%	90%	FBP
52	D6352	278.0	360.5	405.0	436.5	472.5	525.5	608.0
62		----	----	----	----	----	----	----
120		----	----	----	----	----	----	----
140		----	----	----	----	----	----	----
150	D6352	271.5	359.0	406.0	440.0	477.0	530.5	660.0
154		----	----	----	----	----	----	----
158		----	----	----	----	----	----	----
159		----	----	----	----	----	----	----
171	D6352	176.5	363.5	410.5	443.5	481.0	536.0	626.0
225		----	----	----	----	----	----	----
311	D6352	93.0	356.0	405.0	439.0	476.5	529.0	614.0
313		----	----	----	----	----	----	----
317		----	----	----	----	----	----	----
323	D6352	285.7	364.8	403.0	437.3	473.0	526.0	566.2
333		----	----	----	----	----	----	----
356		----	----	----	----	----	----	----
381		----	----	----	----	----	----	----
403		----	----	----	----	----	----	----
445	D7169	113.0	363.5	414.0	452.5 G(0.05)	500.0 G(0.01)	>720.0 f+?	>720.0 f+?
463		----	----	----	----	----	----	----
541		----	----	----	----	----	----	----
608		----	----	----	----	----	----	----
657		----	----	----	----	----	----	----
663		----	----	----	----	----	----	----
732		----	----	----	----	----	----	----
750		----	----	----	----	----	----	----
752		----	----	----	----	----	----	----
778		----	----	----	----	----	----	----
781		----	----	----	----	----	----	----
785		----	----	----	----	----	----	----
798		----	----	----	----	----	----	----
823		----	----	----	----	----	----	----
872		----	----	----	----	----	----	----
873		----	----	----	----	----	----	----
874		----	----	----	----	----	----	----
875		----	----	----	----	----	----	----
994		----	----	----	----	----	----	----
995		----	----	----	----	----	----	----
1011		----	----	----	----	----	----	----
1016	D7169	114.1	364.0	413.2	447.7	485.3	540.2	628.6
1065	D6352	272.2	361.2	408.2	441.6	478.8	531.4	660.6
1081	IP480	109.18	357.92	406.83	440.69	478.42	532.88	663.85
1108		----	----	----	----	----	----	----
1140		----	360.2	408.4	442.4	480.0	532.2	663
1191		----	----	----	----	----	----	----
1205		----	----	----	----	----	----	----
1229		----	----	----	----	----	----	----
1297	D7213	114.6	361.4	408.6	441.8	478.0	530.2	597.8
1397		269	360	409	442	479	532	615
1510		----	----	----	----	----	----	----
1556	ISO3924	112.0	362.1	409.9	443.6	479.2	527.1	581.8
1586		----	----	----	----	----	----	----
1613		----	----	----	----	----	----	----
1635	D2887	282	359	406	440	478	533	606
1720		----	----	----	----	----	----	----
1776	D6352	294	364	409	441	478	529	602
1857		----	----	----	----	----	----	----
1881		----	----	----	----	----	----	----
6021		----	----	----	----	----	----	----
6024		----	----	----	----	----	----	----
6025		----	----	----	----	----	----	----
6026		----	----	----	----	----	----	----
6057		----	----	----	----	----	----	----
6112		----	----	----	----	----	----	----
6203		----	----	----	----	----	----	----
6238		----	----	----	----	----	----	----
6262		----	----	----	----	----	----	----

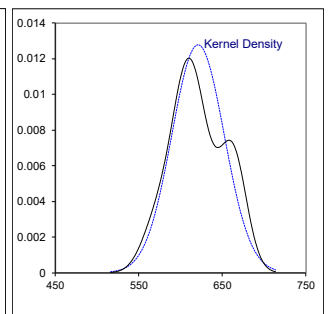
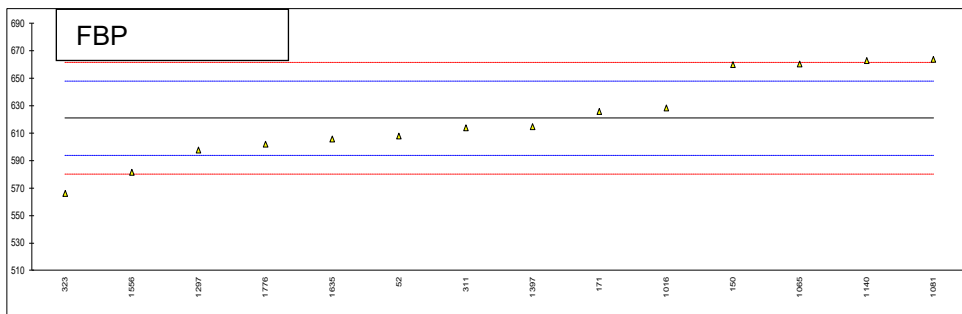
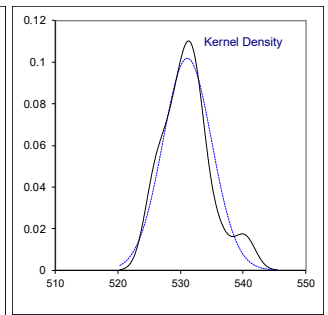
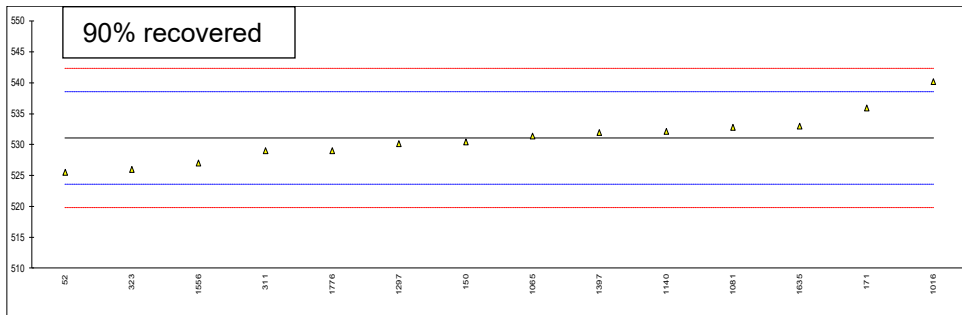
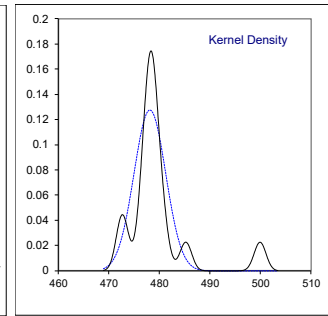
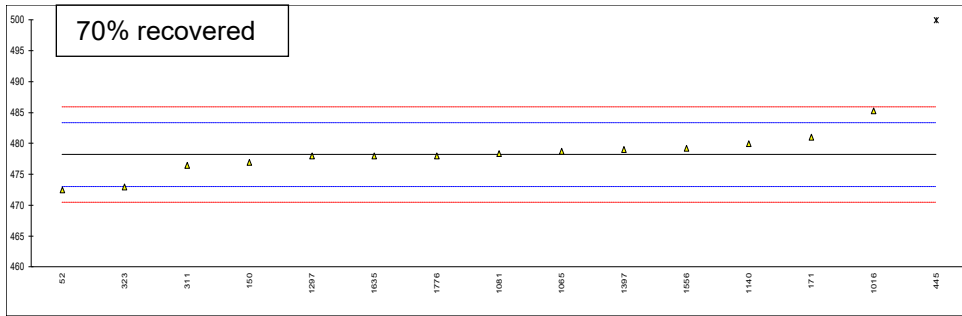
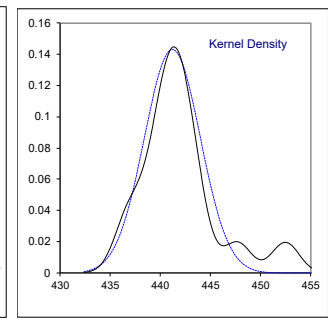
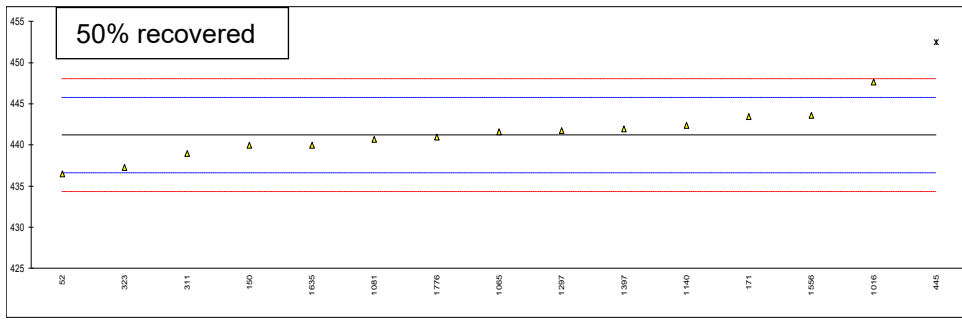
normality	OK	OK	OK	suspect	suspect	suspect	OK
n	14	15	15	14	14	14	14
outliers	0	0	0	1	1	0	0
mean (n)	(198.91)	361.14	408.18	441.22	478.19	531.07	620.92
st.dev. (n)	(85.166)	2.541	3.016	2.790	3.132	3.913	31.243
R(calc.)	(238.47)	7.12	8.45	7.81	8.77	10.96	87.48
st.dev.(D6352:19e1)	(17.536)	2.536	2.107	2.286	2.571	3.750	13.607
R(D6352:19e1)	(49.1)	7.1	5.9	6.4	7.2	10.5	38.1

Lab 445 possibly a false positive test result at 90% and FBP?
 Results between brackets must be used with due care.

Z-scores

lab	IBP	10%	30%	50%	70%	90%	FBP
52	----	-0.25	-1.51	-2.07	-2.21	-1.49	-0.95
150	----	-0.84	-1.03	-0.53	-0.46	-0.15	2.87
171	----	0.93	1.10	1.00	1.09	1.31	0.37
311	----	-2.03	-1.51	-0.97	-0.66	-0.55	-0.51
323	----	1.44	-2.46	-1.72	-2.02	-1.35	-4.02
445	----	0.93	2.76	4.93	8.48	>50.38	>7.28
1016	----	1.13	2.38	2.83	2.76	2.43	0.56
1065	----	0.02	0.01	0.17	0.24	0.09	2.92
1081	----	-1.27	-0.64	-0.23	0.09	0.48	3.16
1140	----	-0.37	0.11	0.52	0.70	0.30	3.09
1297	----	0.10	0.20	0.25	-0.08	-0.23	-1.70
1397	----	-0.45	0.39	0.34	0.31	0.25	-0.43
1556	----	0.38	0.82	1.04	0.39	-1.06	-2.87
1635	----	-0.84	-1.03	-0.53	-0.08	0.51	-1.10
1776	----	1.13	0.39	-0.10	-0.08	-0.55	-1.39





Determination of Distillation at 10 mmHg as AET on sample #20235; result in °C

lab	method	IBP	10%	30%	50%	70%	90%	FBP
52		----	----	----	----	----	----	----
62		----	----	----	----	----	----	----
120		----	----	----	----	----	----	----
140		----	----	----	----	----	----	----
150	D1160	297	390	422	451	484	538	554
154		----	----	----	----	----	----	----
158		----	----	----	----	----	----	----
159		----	----	----	----	----	----	----
171	D1160	320	391	424	453	486	541	586
225		----	----	----	----	----	----	----
311		----	----	----	----	----	----	----
313		----	----	----	----	----	----	----
317		----	----	----	----	----	----	----
323		----	----	----	----	----	----	----
333		----	----	----	----	----	----	----
356	D1160	288	389	422	452	487	544	564
381		----	----	----	----	----	----	----
403		----	----	----	----	----	----	----
445	D1160	279.0	371.2	413.4	444.4	479.7	533.0	543.6
463	D1160	271.0	379.0	C 418.0	444.0	477.0	526.0	548.0
541		----	----	----	----	----	----	----
608		----	----	----	----	----	----	----
657	D1160	275.8	378.8	416.5	445.7	480.6	536.4	561.1
663		----	----	----	----	----	----	----
732	D1160	260	378.5	417	446.5	481.5	536.5	545
750		----	----	----	----	----	----	----
752	D1160	261	382	420	451	486	543	543
778		----	----	----	----	----	----	----
781	D1160	281	379	415	446	476	532	544
785	D1160	269.8	379.6	419.6	451.1	485.0	----	540.1
798		----	----	----	----	----	----	----
823	D1160	239	C 380	417	448	485	536	552
872	D1160	220	375	410	440	475	530	543
873	D1160	268	377	413	443	478	532	545
874	D1160	280	380	421	449	480	532	546
875	D1160	265	379	415	450	479	528	537
994	D1160	271.0	380.0	419.0	443.0	477.0	525.0	548.0
995	D1160	273.5	377.0	415.5	444.5	477.5	526.0	552.0
1011		----	----	----	----	----	----	----
1016		----	----	----	----	----	----	----
1065	D1160	295.7	365.6	408.1	438.9	476.3	----	----
1081		----	----	----	----	----	----	----
1108	D1160	312.6	378.7	414.6	443.5	478.9	539.8	551.0
1140		----	----	----	----	----	----	----
1191		----	----	----	----	----	----	----
1205		----	----	----	----	----	----	----
1229		----	----	----	----	----	----	----
1297		----	----	----	----	----	----	----
1397		----	----	----	----	----	----	----
1510		----	----	----	----	----	----	----
1556		----	----	----	----	----	----	----
1586	D1160	308	388	424	452	486	0	ex 532
1613	D1160	335.9	384.6	415.3	457.0	487.0	535.6	554.7
1635		----	----	----	----	----	----	----
1720		----	----	----	----	----	----	----
1776		----	----	----	----	----	----	----
1857	D1160	298.5	384.5	418.8	447.6	481.3	536.8	550.6
1881		----	----	----	----	----	----	----
6021	D1160	274	380	415	445	477	533	548
6024		----	----	----	----	----	----	----
6025		----	----	----	----	----	----	----
6026	D1160	271	383	416	450	481	535	551
6057	D1160	180.5	R(0.05) 374.1	414.6	445.9	481.9	534.0	552.5
6112	D1160	312.8	383.2	419.2	448.3	482.8	535.8	572.2
6203	D1160	309.6	386.7	421.7	452.6	487.5	547.6	588.4
6238		----	----	----	----	----	----	----
6262		----	----	----	----	----	----	----

normality	OK	OK	OK	OK	OK	OK	suspect
n	26	27	27	27	27	24	24
outliers	1	0	0	0	0	0 +1ex	2
mean (n)	282.20	380.54	417.23	447.52	481.26	534.85	549.08
st.dev. (n)	25.452	5.680	3.925	4.265	3.905	5.721	8.572
R(calc.)	71.27	15.90	10.99	11.94	10.93	16.02	24.00
st.dev.(D1160:18)	17.660	6.150	4.104	4.029	3.539	3.589	9.605
R(D1160:18)	49.45	17.22	11.49	11.28	9.91	10.05	26.89

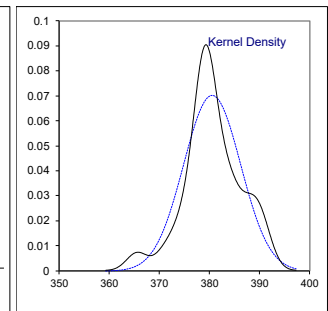
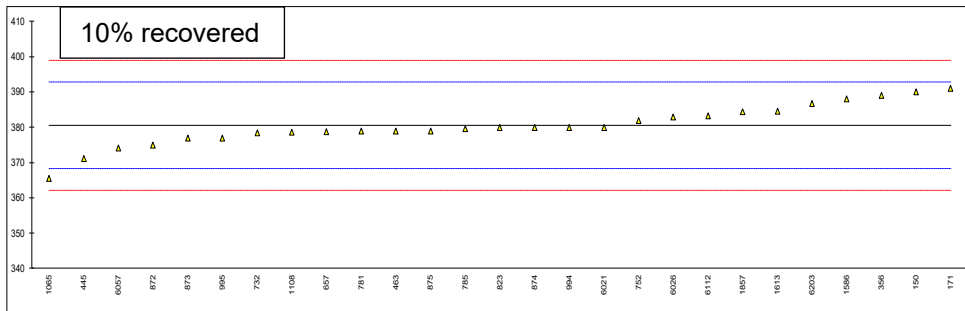
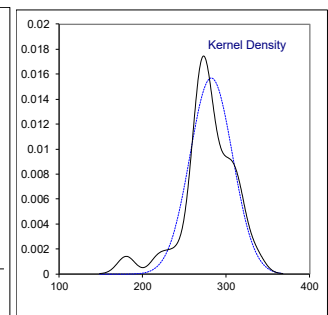
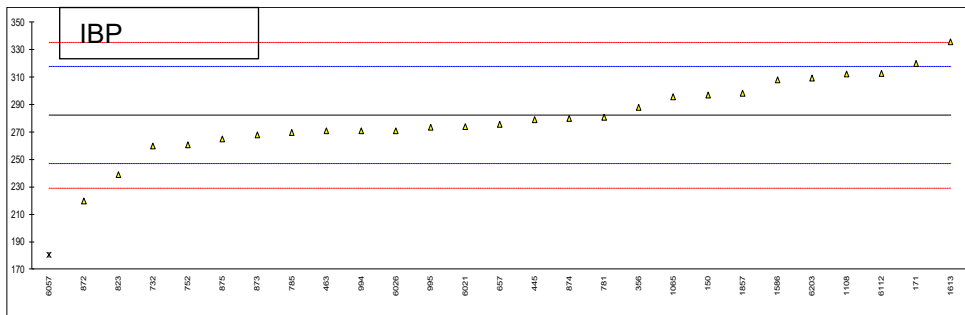
Lab 463 first reported 357.0

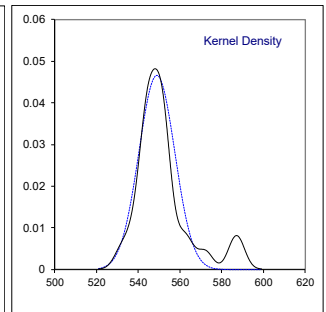
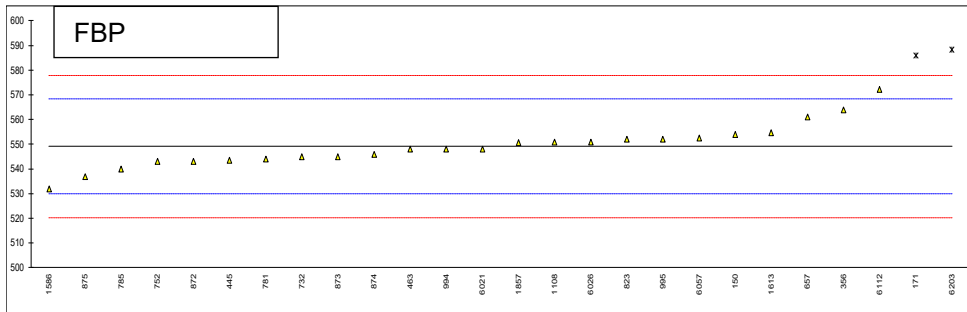
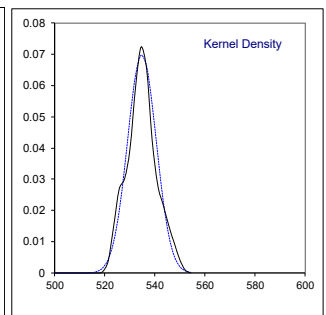
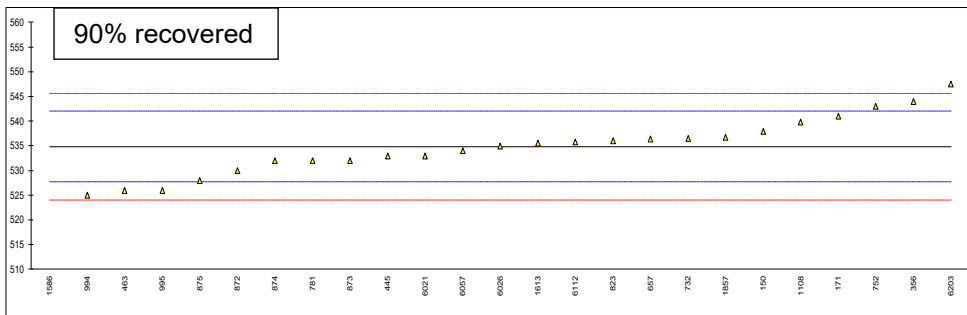
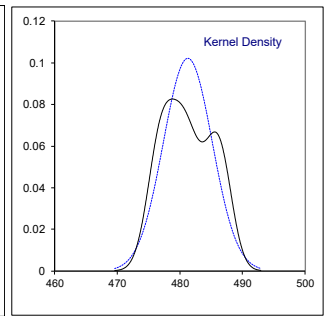
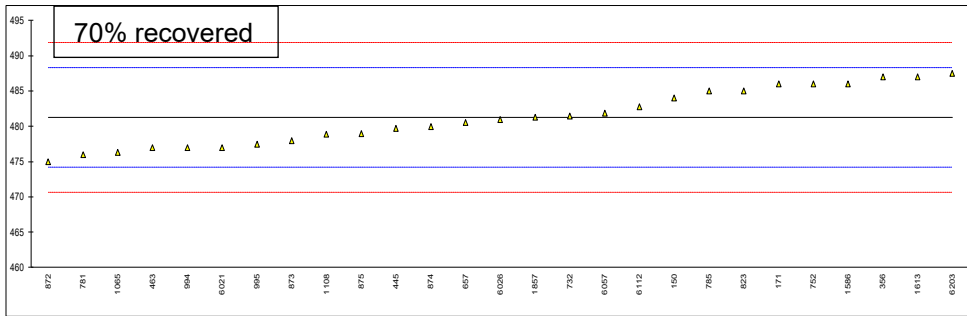
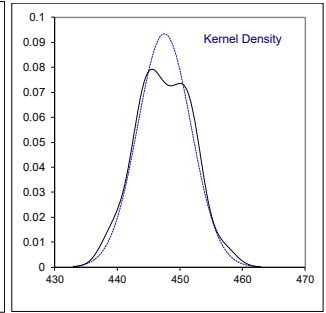
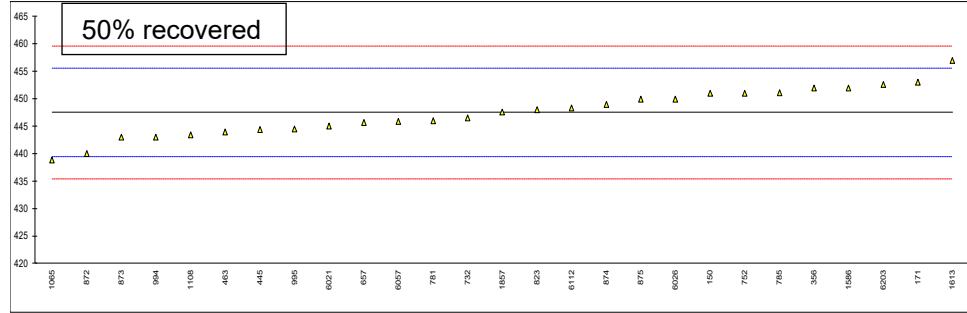
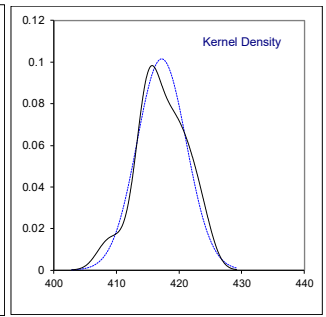
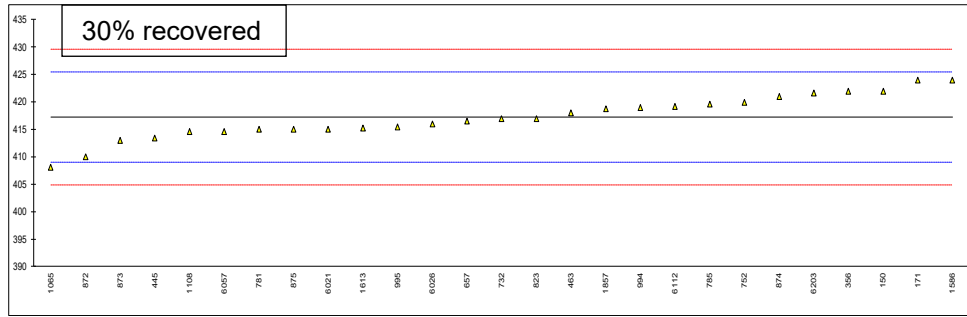
Lab 823 first reported 141

Lab 1586 90% rec. test result excluded as zero is not a real test value

Z-scores

lab	IBP	10%	30%	50%	70%	90%	FBP
150	0.84	1.54	1.16	0.86	0.77	0.88	0.51
171	2.14	1.70	1.65	1.36	1.34	1.71	3.84
356	0.33	1.38	1.16	1.11	1.62	2.55	1.55
445	-0.18	-1.52	-0.93	-0.77	-0.44	-0.52	-0.57
463	-0.63	-0.25	0.19	-0.87	-1.20	-2.47	-0.11
657	-0.36	-0.28	-0.18	-0.45	-0.19	0.43	1.25
732	-1.26	-0.33	-0.06	-0.25	0.07	0.46	-0.42
752	-1.20	0.24	0.67	0.86	1.34	2.27	-0.63
781	-0.07	-0.25	-0.54	-0.38	-1.49	-0.80	-0.53
785	-0.70	-0.15	0.58	0.89	1.06	-----	-0.93
823	-2.45	-0.09	-0.06	0.12	1.06	0.32	0.30
872	-3.52	-0.90	-1.76	-1.87	-1.77	-1.35	-0.63
873	-0.80	-0.58	-1.03	-1.12	-0.92	-0.80	-0.42
874	-0.12	-0.09	0.92	0.37	-0.36	-0.80	-0.32
875	-0.97	-0.25	-0.54	0.62	-0.64	-1.91	-1.26
994	-0.63	-0.09	0.43	-1.12	-1.20	-2.75	-0.11
995	-0.49	-0.58	-0.42	-0.75	-1.06	-2.47	0.30
1065	0.76	-2.43	-2.23	-2.14	-1.40	-----	-----
1108	1.72	-0.30	-0.64	-1.00	-0.67	1.38	0.20
1586	1.46	1.21	1.65	1.11	1.34	-149.01	-1.78
1613	3.04	0.66	-0.47	2.35	1.62	0.21	0.59
1857	0.92	0.64	0.38	0.02	0.01	0.54	0.16
6021	-0.46	-0.09	-0.54	-0.63	-1.20	-0.52	-0.11
6026	-0.63	0.40	-0.30	0.62	-0.07	0.04	0.20
6057	-5.76	-1.05	-0.64	-0.40	0.18	-0.24	0.36
6112	1.73	0.43	0.48	0.19	0.44	0.26	2.41
6203	1.55	1.00	1.09	1.26	1.76	3.55	4.09



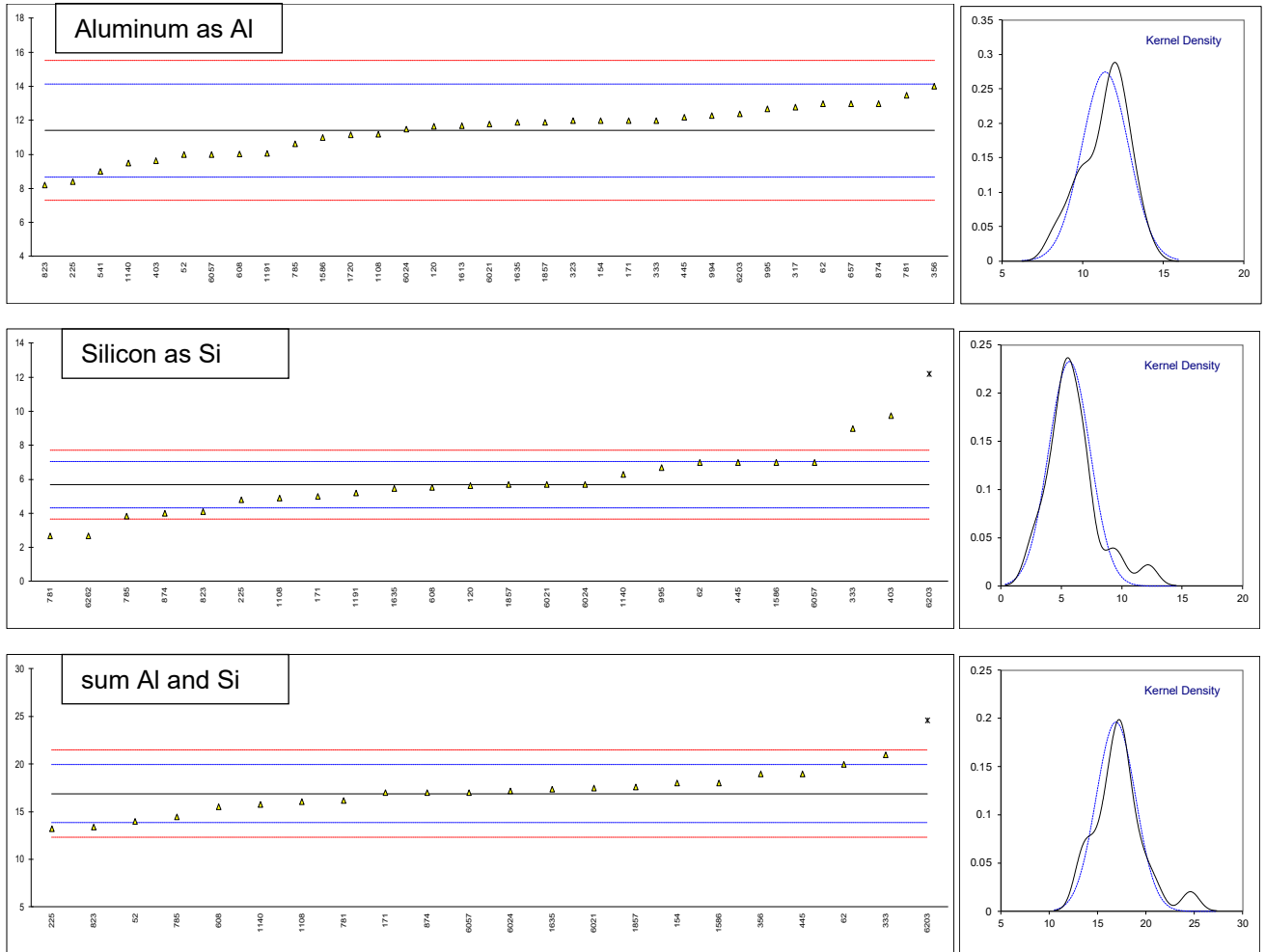


Determination of Aluminum as Al, Silicon as Si and sum Al and Si on sample #20236; results in mg/kg

lab	method	Al	mark	z(targ)	Si	mark	z(targ)	Sum Al+Si	mark	z(targ)
52	IP501	10		-1.02	<10		----	14		-1.88
62	IP501	13		1.17	7		1.95	20		2.04
120	IP501	11.66		0.19	5.644		-0.06	----		----
140		----		----	----		----	----		----
150		----		----	<10		----	----		----
154	IP501	12		0.44	<10		----	18		0.73
158		----		----	<10		----	----		----
159		----		----	----		----	----		----
171	IP501	12		0.44	5		-1.02	17		0.08
225	IP501	8.41		-2.18	4.81		-1.30	13.22		-2.39
311		----		----	----		----	----		----
313		----		----	----		----	----		----
317	IP501	12.8		1.02	<10		----	----		----
323	IP501	12		0.44	<10		----	<22		----
333	IP501	12		0.44	9		4.91	21		2.69
356	IP501	14		1.90	<10		----	19		1.39
381		----		----	----		----	----		----
403	D7111	9.64		-1.28	9.76		6.04	----		----
445	IP501	12.2		0.58	7.0		1.95	19.0		1.39
463		----		----	----		----	----		----
541	IP501	9.0		-1.75	<10		----	<15		----
608	IP501	10.019		-1.01	5.549		-0.20	15.568		-0.86
657	IP501	13		1.17	<10		----	<15		----
663		----		----	----		----	----		----
732		----		----	----		----	----		----
750		----		----	----		----	----		----
752		----		----	----		----	----		----
778		----		----	----		----	----		----
781	IP501	13.5		1.53	2.7		-4.43	16.2		-0.45
785	IP470	10.64		-0.55	3.86		-2.71	14.5		-1.56
798		----		----	----		----	----		----
823	IP501	8.2	C	-2.33	4.1		-2.35	13.4	C	-2.28
872		----		----	----		----	----		----
873		----		----	----		----	----		----
874	IP501	13		1.17	4		-2.50	17		0.08
875		----		----	----		----	----		----
994	IP501	12.3		0.66	<10		----	----		----
995	IP470	12.7		0.95	6.7		1.50	----		----
1011		----		----	----		----	----		----
1016		----		----	----		----	----		----
1065		----		----	----		----	----		----
1081		----		----	----		----	----		----
1108	IP470	11.2		-0.15	4.9		-1.17	16.1		-0.51
1140	IP501	9.5		-1.38	6.3		0.91	15.8		-0.71
1191	IP501	10.06		-0.98	5.22		-0.69	----		----
1205		----		----	----		----	----		----
1229		----		----	----		----	----		----
1297		----		----	----		----	----		----
1397		----		----	----		----	----		----
1510		----		----	----		----	----		----
1556		----		----	----		----	----		----
1586	IP470	11		-0.29	7		1.95	18		0.73
1613	IP501	11.7		0.22	----	W	----	----	W	----
1635	IP501	11.9		0.36	5.46		-0.34	17.4		0.34
1720	D5708	11.150		-0.18	----		----	----		----
1776		----		----	----		----	----		----
1857	IP501	11.9		0.36	5.7		0.02	17.6		0.47
1881		----		----	----		----	----		----
6021	IP501	11.8		0.29	5.7		0.02	17.5		0.41
6024	IP470	11.5		0.07	5.7		0.02	17.2		0.21
6025		----		----	----		----	----		----
6026		----		----	----		----	----		----
6057	IP501	10		-1.02	7		1.95	17		0.08
6112		----		----	----		----	----		----
6203	IP501	12.4		0.73	12.2	R(0.05)	9.66	24.6	R(0.05)	5.05
6238		----		----	----		----	----		----
6262		----	W	----	2.7		-4.43	----	W	----

normality	OK	OK	OK
n	33	23	21
outliers	0	1	1
mean (n)	11.40	5.69	16.88
st.dev. (n)	1.451	1.713	2.037
R(calc.)	4.06	4.80	5.70
st.dev.(IP501:05)	1.372	0.674	1.529
R(IP501:05)	3.84	1.89	4.28
Compare			
R(IP470:05)	7.09	4.45	8.37

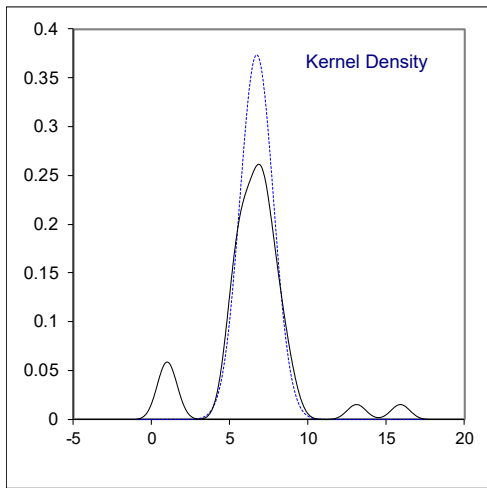
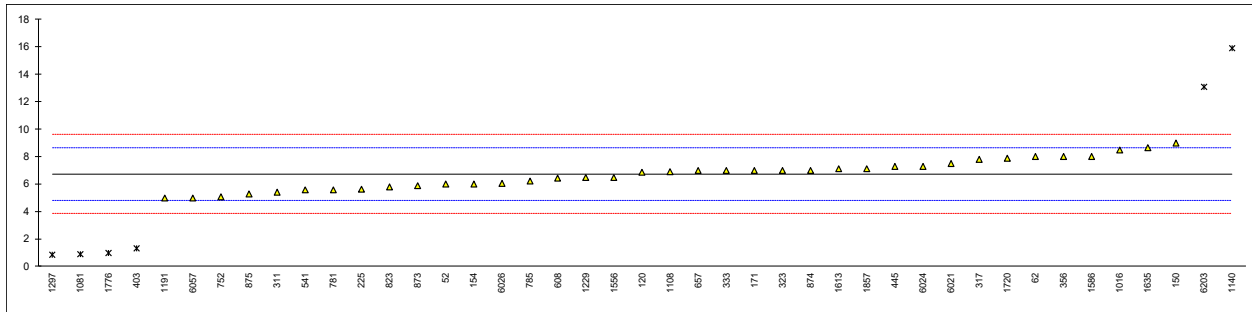
Lab 823 first reported 6.3 for Al and 10.4 for sum Al+Si
 Lab 1613 test result withdrawn for Si and sum Al+Si, first reported 11.0 and 22.7 respectively
 Lab 6262 test result withdrawn for Al and sum Al+Si, first reported 35 and 6.2 respectively



Determination of Iron as Fe on sample #20236; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	IP501	6		-0.76	
62	IP501	8		1.33	
120	IP501	6.869		0.15	
140		----		----	
150	IP501	9		2.38	
154	IP501	6		-0.76	
158	IP501	<2	f-?	<-4.95	possibly a false negative test result?
159		----		----	
171	IP501	7		0.29	
225	IP501	5.63		-1.15	
311	IP621	5.4		-1.39	
313		----		----	
317	IP501	7.8		1.12	
323	IP501	7		0.29	
333	IP501	7		0.29	
356	IP501	8		1.33	
381		----		----	
403	D7111	1.33	R(0.01)	-5.65	
445	IP501	7.3		0.60	
463		----		----	
541	IP501	5.6		-1.18	
608	IP501	6.437		-0.30	
657	IP501	7		0.29	
663		----		----	
732		----		----	
750		----		----	
752	IP621	5.094		-1.71	
778		----		----	
781	IP501	5.6		-1.18	
785	IP470	6.23		-0.52	
798		----		----	
823	IP501	5.8		-0.97	
872		----		----	
873	IP621	5.9		-0.87	
874	IP501	7		0.29	
875	IP501	5.3		-1.49	
994		----		----	
995		----		----	
1011		----		----	
1016	In house	8.5		1.86	
1065		----		----	
1081	In house	0.891	R(0.05)	-6.11	
1108	IP470	6.9		0.18	
1140	IP501	15.9	R(0.01)	9.60	
1191	ISO10478	4.97		-1.84	
1205		----		----	
1229		6.5		-0.24	
1297	In house	0.873	R(0.05)	-6.13	
1397		----		----	
1510		----		----	
1556	IP621	6.5		-0.24	
1586	IP470	8		1.33	
1613	IP501	7.1		0.39	
1635	IP501	8.65		2.01	
1720	D5708	7.904		1.23	
1776	IP621	1.0	R(0.05)	-5.99	
1857	IP501	7.1		0.39	
1881		----		----	
6021	IP501	7.5		0.81	
6024	IP470	7.3		0.60	
6025		----		----	
6026	IP621	6.0352		-0.72	
6057	IP501	5		-1.81	
6112		----		----	
6203	IP501	13.1	R(0.05)	6.67	
6238		----		----	
6262		----	W	----	test result withdrawn, first reported 1.8

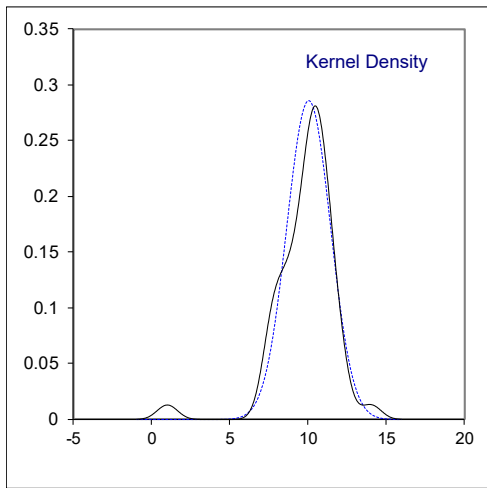
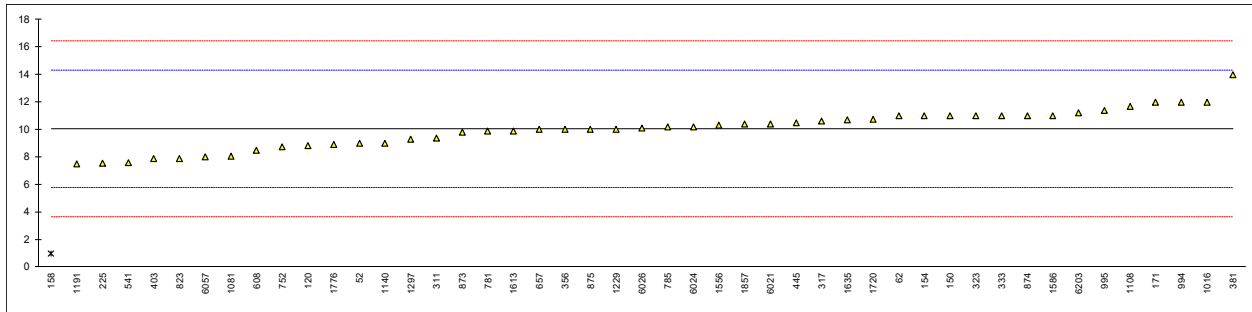
normality	OK
n	37
outliers	6
mean (n)	6.73
st.dev. (n)	1.069
R(calc.)	2.99
st.dev.(IP501:05)	0.955
R(IP501:05)	2.68
Compare	
R(IP470:05)	5.92



Determination of Nickel as Ni on sample #20236; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	IP501	9		-0.48	
62	IP501	11		0.46	
120	IP501	8.845		-0.55	
140		----		----	
150	IP501	11		0.46	
154	IP501	11		0.46	
158	IP501	1	R(0.01)	-4.23	
159		----		----	
171	IP501	12		0.93	
225	IP501	7.53		-1.17	
311	IP621	9.4		-0.29	
313		----		----	
317	IP501	10.6		0.27	
323	IP501	11		0.46	
333	IP501	11		0.46	
356	IP501	10		-0.01	
381	IP501	14.0		1.86	
403	D7111	7.90		-1.00	
445	IP501	10.5		0.22	
463		----		----	
541	IP501	7.6		-1.14	
608	IP501	8.474		-0.73	
657	IP501	10		-0.01	
663		----		----	
732		----		----	
750		----		----	
752	IP621	8.741		-0.60	
778		----		----	
781	IP501	9.9		-0.06	
785	IP470	10.18		0.07	
798		----		----	
823	IP501	7.9		-1.00	
872		----		----	
873	IP621	9.8		-0.11	
874	IP501	11		0.46	
875	IP501	10		-0.01	
994	IP501	12.0		0.93	
995	IP470	11.4		0.64	
1011		----		----	
1016	In house	12.0		0.93	
1065		----		----	
1081	In house	8.046		-0.93	
1108	D5708	11.7		0.78	
1140	IP501	9.0		-0.48	
1191	ISO10478	7.51		-1.18	
1205		----		----	
1229		10		-0.01	
1297	In house	9.315		-0.33	
1397		----		----	
1510		----		----	
1556	IP621	10.3		0.13	
1586	IP470	11		0.46	
1613	IP501	9.9		-0.06	
1635	IP501	10.7		0.32	
1720	D5708	10.740		0.33	
1776	IP621	8.9		-0.53	
1857	IP501	10.4		0.18	
1881		----		----	
6021	IP501	10.4		0.18	
6024	IP470	10.2		0.08	
6025		----		----	
6026	IP621	10.1237		0.05	
6057	IP501	8		-0.95	
6112		----		----	
6203	IP501	11.2		0.55	
6238		----		----	
6262		----	W	----	test result withdrawn, first reported 3.2

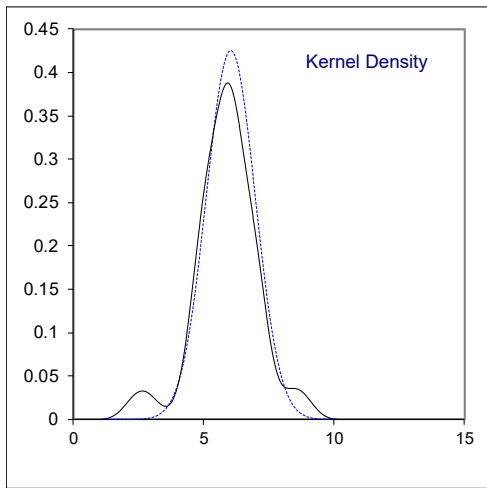
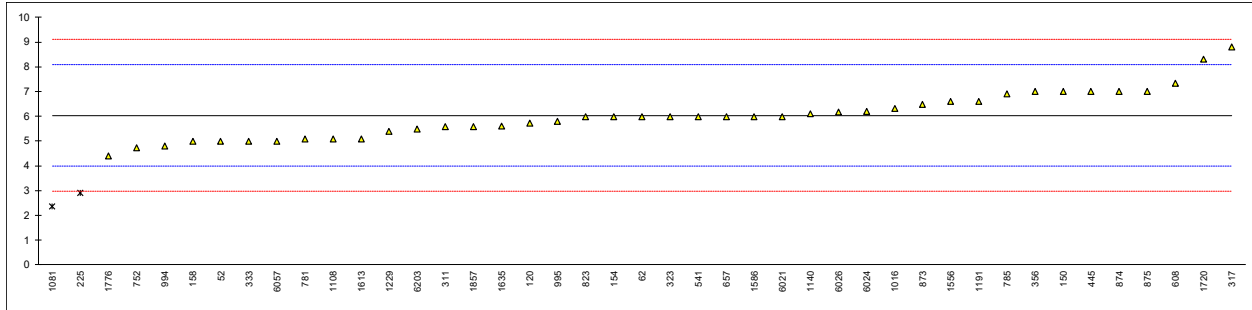
normality	OK
n	46
outliers	1
mean (n)	10.03
st.dev. (n)	1.394
R(calc.)	3.90
st.dev.(IP501:05)	2.134
R(IP501:05)	5.97
Compare	
R(IP470:05)	8.29



Determination of Sodium as Na on sample #20236; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	IP501	5		-1.01	
62	IP501	6		-0.03	
120	IP501	5.734		-0.29	
140		----		----	
150	IP501	7		0.94	
154	IP501	6		-0.03	
158	IP501	5		-1.01	
159		----		----	
171		----		----	
225	IP501	2.92	DG(0.05)	-3.04	
311	IP621	5.6		-0.42	
313		----		----	
317	IP501	8.8		2.70	
323	IP501	6		-0.03	
333	IP501	5		-1.01	
356	IP501	7		0.94	
381		----		----	
403		----		----	
445	IP501	7.0		0.94	
463		----		----	
541	IP501	6.0		-0.03	
608	IP501	7.336		1.27	
657	IP501	6		-0.03	
663		----		----	
732		----		----	
750		----		----	
752	IP621	4.738		-1.26	
778		----		----	
781	IP501	5.1		-0.91	
785	IP470	6.91		0.86	
798		----		----	
823	IP501	6.0		-0.03	
872		----		----	
873	IP470	6.5		0.46	
874	IP501	7		0.94	
875	IP501	7		0.94	
994	IP501	4.8		-1.20	
995	IP470	5.8		-0.23	
1011		----		----	
1016	NEN6966	6.323		0.28	
1065		----		----	
1081	In house	2.369	DG(0.05)	-3.58	
1108	D5708	5.1		-0.91	
1140	IP501	6.1		0.07	
1191	ISO10478	6.61		0.56	
1205		----		----	
1229		5.4		-0.62	
1297		----		----	
1397		----		----	
1510		----		----	
1556	IP621	6.6		0.55	
1586	IP470	6		-0.03	
1613	IP501	5.1		-0.91	
1635	IP501	5.61		-0.41	
1720	D5708	8.314		2.23	
1776	IP621	4.4		-1.60	
1857	IP501	5.6		-0.42	
1881		----		----	
6021	IP501	6.0		-0.03	
6024	IP470	6.2		0.16	
6025		----		----	
6026	IP621	6.1726		0.14	
6057	IP501	5		-1.01	
6112		----		----	
6203	IP501	5.5		-0.52	
6238		----		----	
6262		----	W	----	test result withdrawn, first reported 1.7

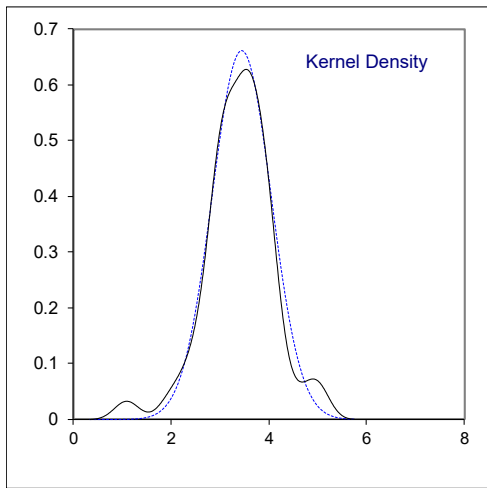
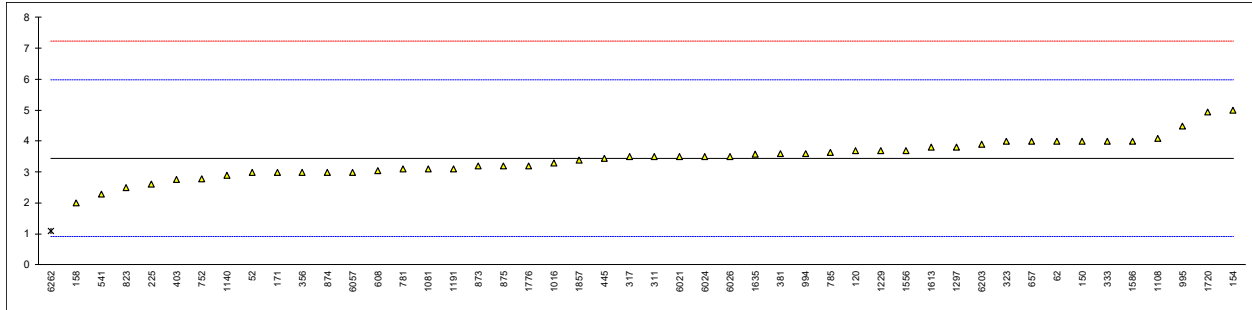
normality	suspect
n	41
outliers	2
mean (n)	6.03
st.dev. (n)	0.937
R(calc.)	2.62
st.dev.(IP501:05)	1.024
R(IP501:05)	2.87
Compare	
R(IP470:05)	3.83



Determination of Vanadium as V on sample #20236; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	IP501	3		-0.36	
62	IP501	4		0.44	
120	IP501	3.686		0.19	
140		----		----	
150	IP501	4		0.44	
154	IP501	5		1.23	
158	IP501	2		-1.15	
159		----		----	
171	IP501	3		-0.36	
225	IP501	2.61		-0.66	
311	IP621	3.5		0.04	
313		----		----	
317	IP501	3.5		0.04	
323	IP501	4		0.44	
333	IP501	4		0.44	
356	IP501	3		-0.36	
381	IP501	3.6		0.12	
403	D7111	2.76		-0.55	
445	IP501	3.45		0.00	
463		----		----	
541	IP501	2.3		-0.91	
608	IP501	3.056		-0.31	
657	IP501	4		0.44	
663		----		----	
732		----		----	
750		----		----	
752	IP621	2.79		-0.52	
778		----		----	
781	IP501	3.1		-0.28	
785	IP470	3.64		0.15	
798		----		----	
823	IP501	2.5		-0.75	
872		----		----	
873	IP621	3.2		-0.20	
874	IP470	3		-0.36	
875	IP501	3.2		-0.20	
994	IP501	3.6		0.12	
995	IP470	4.5		0.83	
1011		----		----	
1016	In house	3.3		-0.12	
1065		----		----	
1081	In house	3.102		-0.27	
1108	D5708	4.1		0.52	
1140	IP501	2.9		-0.43	
1191	ISO10478	3.11		-0.27	
1205		----		----	
1229		3.7		0.20	
1297	In house	3.817		0.29	
1397		----		----	
1510		----		----	
1556	IP621	3.7		0.20	
1586	IP470	4		0.44	
1613	IP501	3.8		0.28	
1635	IP501	3.58		0.10	
1720	D5708	4.941		1.18	
1776	IP621	3.2		-0.20	
1857	IP501	3.4		-0.04	
1881		----		----	
6021	IP501	3.5		0.04	
6024	IP470	3.5		0.04	
6025		----		----	
6026	IP621	3.5076		0.05	
6057	IP501	3		-0.36	
6112		----		----	
6203	IP501	3.9		0.36	
6238		----		----	
6262	IP501	1.1	R(0.05)	-1.86	

normality	OK
n	47
outliers	1
mean (n)	3.45
st.dev. (n)	0.603
R(calc.)	1.69
st.dev.(IP501:05)	1.261
R(IP501:05)	3.53
Compare	
R(IP470:05)	6.05



Determination of Calcium as Ca on sample #20236; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	IP501	<3		----	
62	IP501	2		----	
120	IP501	2.396		----	
140		----		----	
150	IP501	3		----	
154	IP501	<3		----	
158	IP501	<3		----	
159		----		----	
171	IP501	1		----	
225	IP501	1.34		----	
311		----		----	
313		----		----	
317	IP501	<3		----	
323	IP501	< 3		----	
333	IP501	<3		----	
356	IP501	Below 3		----	
381		----		----	
403	D7111	0.51		----	
445	IP501	2.9		----	
463		----		----	
541	IP501	<3		----	
608	IP501	2.339		----	
657	IP501	3		----	
663		----		----	
732		----		----	
750		----		----	
752		----		----	
778		----		----	
781	IP501	2.2		----	
785		----		----	
798		----		----	
823	IP501	2.3		----	
872		----		----	
873		----		----	
874	IP501	2		----	
875	IP501	2		----	
994	IP501	<3		----	
995	IP470	0.09		----	
1011		----		----	
1016	NEN6966	1.992		----	
1065		----		----	
1081	In house	1.504		----	
1108	IP470	2.8		----	
1140	IP501	2.5		----	
1191	ISO0478	1.72		----	
1205		----		----	
1229		1.9		----	
1297		----		----	
1397		----		----	
1510		----		----	
1556		----		----	
1586	IP470	2		----	
1613	IP501	2.4		----	
1635	IP501	2.45		----	
1720	D5708	4.894		----	possibly false positive test result?
1776		----		----	
1857	IP501	1.9		----	
1881		----		----	
6021		----		----	
6024		----		----	
6025		----		----	
6026		----		----	
6057	IP501	<1		----	
6112		----		----	
6203	IP501	9.1		----	possibly false positive test result?
6238		----		----	
6262	IP501	0.6		----	
n		35			
mean (n)		<3			application range: 3 – 100 mg/kg

Determination of Phosphorus as P on sample #20236; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	IP501	<1		----	
62	IP501	0.4		----	
120	IP501	<1		----	
140		----		----	
150	IP501	<1		----	
154	IP501	1		----	
158	IP501	<1		----	
159		----		----	
171	IP501	<1		----	
225		----		----	
311		----		----	
313		----		----	
317	IP501	<1		----	
323	IP501	< 1		----	
333	IP501	<1		----	
356	IP501	1		----	
381		----		----	
403		----		----	
445	IP501	0.42		----	
463		----		----	
541	IP501	<1		----	
608		----		----	
657	IP501	<1		----	
663		----		----	
732		----		----	
750		----		----	
752		----		----	
778		----		----	
781	IP501	1.5		----	
785		----		----	
798		----		----	
823	IP501	0.3		----	
872		----		----	
873		----		----	
874		----		----	
875		----		----	
994		----		----	
995		----		----	
1011		----		----	
1016		----		----	
1065		----		----	
1081		----		----	
1108		----		----	
1140	IP501	0.5		----	
1191	IP501	0.61		----	
1205		----		----	
1229		----		----	
1297		----		----	
1397		----		----	
1510		----		----	
1556		----		----	
1586	IP501	5		----	possibly false positive test result?
1613		----		----	
1635	IP501	<1		----	
1720		----		----	
1776		----		----	
1857	IP501	0.5		----	
1881		----		----	
6021		----		----	
6024		----		----	
6025		----		----	
6026		----		----	
6057	IP501	<1		----	
6112		----		----	
6203	IP501	0.44		----	
6238		----		----	
6262	IP501	0.3		----	
n		22			
mean (n)		<1			application range: 1 – 60 mg/kg

Determination of Zinc as Zn on sample #20236; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	IP501	1		----	
62	IP501	1		----	
120	IP501	0.754		----	
140		----		----	
150	IP501	<1		----	
154	IP501	1		----	
158	IP501	<1		----	
159		----		----	
171	IP501	<1		----	
225	IP501	0.47		----	
311		----		----	
313		----		----	
317	IP501	1.3		----	
323	IP501	< 1		----	
333	IP501	<1		----	
356	IP501	1		----	
381		----		----	
403	D7111	0.76		----	
445	IP501	0.71		----	
463		----		----	
541	IP501	<1		----	
608	IP501	0.515		----	
657	IP501	<1		----	
663		----		----	
732		----		----	
750		----		----	
752		----		----	
778		----		----	
781	IP501	0.4		----	
785	IP470	0.70		----	
798		----		----	
823	IP501	0.8		----	
872		----		----	
873	IP470	0.61		----	
874	IP501	0.8		----	
875	IP501	< 1		----	
994		----		----	
995		----		----	
1011		----		----	
1016		----		----	
1065		----		----	
1081		----		----	
1108	IP470	0.4		----	
1140	IP501	0.7		----	
1191	IP501	0.28		----	
1205		----		----	
1229		----		----	
1297		----		----	
1397		----		----	
1510		----		----	
1556		----		----	
1586	IP470	0.8		----	
1613	IP501	0.6		----	
1635	IP501	<1		----	
1720		----		----	
1776		----		----	
1857	IP501	0.7		----	
1881		----		----	
6021		----		----	
6024		----		----	
6025		----		----	
6026		----		----	
6057	IP501	<1		----	
6112		----		----	
6203	IP501	1.56		----	
6238		----		----	
6262	IP501	0.2		----	
n		31			
mean (n)		<1			application range: 1 – 70 mg/kg

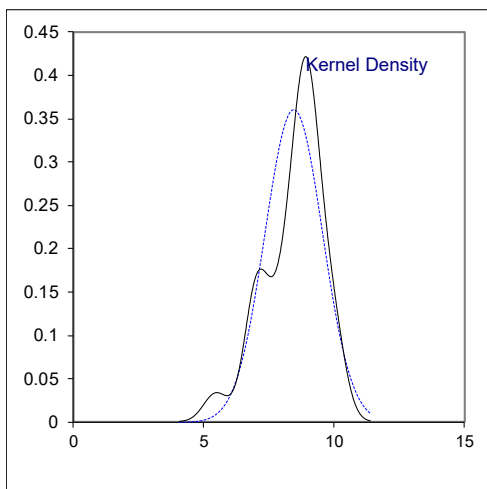
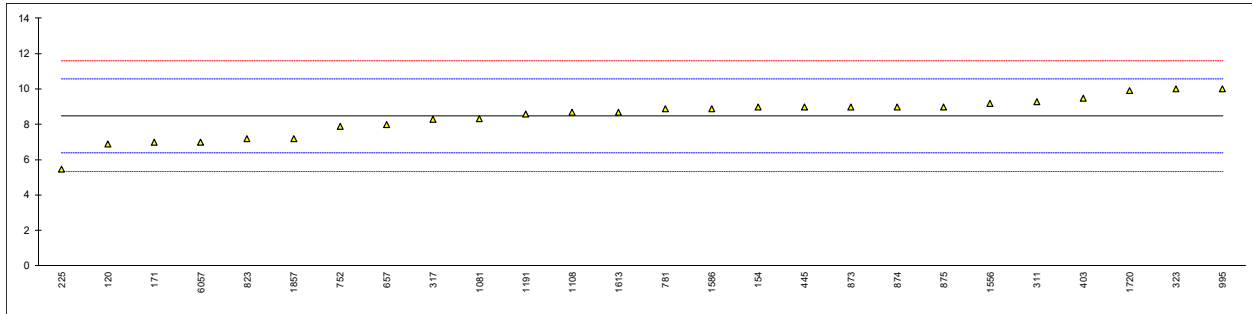
Determination of Arsenic as As on sample #20236; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52		----		----	
62		----		----	
120		----		----	
140		----		----	
150		----		----	
154		----		----	
158		----		----	
159		----		----	
171	IP501	<1		----	
225		----		----	
311		----		----	
313		----		----	
317		----		----	
323	IP501	< 1		----	
333		----		----	
356		----		----	
381		----		----	
403		----		----	
445		----		----	
463		----		----	
541		----		----	
608		----		----	
657		----		----	
663		----		----	
732		----		----	
750		----		----	
752		----		----	
778		----		----	
781	UOP986	0.0729		----	
785		----		----	
798		----		----	
823	IP501	0.1		----	
872		----		----	
873		----		----	
874		----		----	
875		----		----	
994		----		----	
995		----		----	
1011		----		----	
1016		----		----	
1065		----		----	
1081	In house	0.035		----	
1108		----		----	
1140		----		----	
1191		----		----	
1205		----		----	
1229		----		----	
1297	In house	0.032		----	
1397		----		----	
1510		----		----	
1556		----		----	
1586		----		----	
1613		----		----	
1635		----		----	
1720	D5708	1.834		----	
1776		----		----	
1857		----		----	
1881		----		----	
6021		----		----	
6024		----		----	
6025		----		----	
6026		----		----	
6057		----		----	
6112		----		----	
6203		----		----	
6238		----		----	
6262		----		----	
	n	6			
	mean (n)	<1			

Determination of Copper as Cu on sample #20236; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52		----		----	
62		----		----	
120	IP501	6.9	C	-1.49	first reported 5.595
140		----		----	
150		----		----	
154	IP501	9		0.52	
158	IP501	<1		<-7.14	possibly false negative test result?
159		----		----	
171	IP501	7		-1.40	
225	IP501	5.46		-2.87	
311	IP621	9.3		0.80	
313		----		----	
317	IP501Mod.	8.3		-0.15	
323	IP501	10		1.47	
333		----		----	
356		----		----	
381		----		----	
403	D7111	9.48		0.98	
445	IP501	9.0		0.52	
463		----		----	
541		----		----	
608		----		----	
657	IP501	8		-0.44	
663		----		----	
732		----		----	
750		----		----	
752	IP621	7.883		-0.55	
778		----		----	
781	IP501	8.9		0.42	
785		----		----	
798		----		----	
823	IP501	7.2		-1.21	
872		----		----	
873	IP621	9.0		0.52	
874	IP501	9		0.52	
875	IP501	9		0.52	
994		----		----	
995	IP621	10		1.47	
1011		----		----	
1016		----		----	
1065		----		----	
1081	In house	8.314		-0.14	
1108	D5708	8.7		0.23	
1140		----		----	
1191	ISO10478	8.58		0.12	
1205		----		----	
1229		----		----	
1297		----		----	
1397		----		----	
1510		----		----	
1556	IP621	9.2		0.71	
1586	IP501	8.9		0.42	
1613	IP501	8.7		0.23	
1635		----		----	
1720	D5708	9.925		1.40	
1776		----		----	
1857	IP501	7.2		-1.21	
1881		----		----	
6021		----		----	
6024		----		----	
6025		----		----	
6026		----		----	
6057	IP501	7		-1.40	
6112		----		----	
6203		----		----	
6238		----		----	
6262		----	W	----	test result withdrawn, first reported 2.9

normality	OK
n	26
outliers	0
mean (n)	8.46
st.dev. (n)	1.107
R(calc.)	3.10
st.dev.(IP621:16)	1.045
R(IP621:16)	2.93



APPENDIX 2**Number of participants per country**

1 lab in ARGENTINA
1 lab in AZERBAIJAN
3 labs in BELGIUM
2 labs in CANADA
1 lab in CONGO Brazzaville
1 lab in COTE D'IVOIRE
2 labs in CROATIA
2 labs in FINLAND
1 lab in FRANCE
1 lab in GEORGIA
1 lab in GERMANY
3 labs in GREECE
1 lab in ISRAEL
1 lab in JORDAN
1 lab in KAZAKHSTAN
1 lab in LITHUANIA
1 lab in MALAYSIA
2 labs in MALTA
5 labs in NETHERLANDS
1 lab in PORTUGAL
2 labs in ROMANIA
15 labs in RUSSIAN FEDERATION
1 lab in SERBIA
1 lab in SINGAPORE
1 lab in SOUTH KOREA
1 lab in SUDAN
3 labs in SWEDEN
1 lab in THAILAND
3 labs in UNITED KINGDOM
7 labs in UNITED STATES OF AMERICA

APPENDIX 3

Abbreviations

C	= final test result after checking of first reported suspect test 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
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
fr.	= first reported
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?
SDS	= Safety Data Sheet

Literature

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