Results of Proficiency Test
Vacuum Gasoil (VGO)
December 2019

Organised 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 the analysis of Vacuum Gasoil (VGO) every year. During the annual proficiency testing program 2019/2020 it was decided to continue the round robin for the analysis of Vacuum Gasoil in accordance with the latest version of ISO8217.
In this interlaboratory study 66 laboratories in 29 different countries registered for participation. See appendix 2 for the number of participants per country. In this report, the results of the 2019 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 analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send to all participants one bottle of 1L VGO labelled #19265 and one bottle of 0.1L VGO labelled #19266 specifically prepared for metal determinations.
Participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for the 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 organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of 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

A batch of Vacuum Gasoil (VGO) was obtained from a third party. Approximately 100 liters bulk material was homogenised in a pre-cleaned drum. After homogenisation 90 amber glass bottles of 1L were filled and labelled #19265. The homogeneity of the subsamples #19265 was checked by determination of Density in accordance with ISO12185 on 8 stratified randomly selected subsamples.

	Density at 15°C in kg/m^3
Sample #19265-1	916.6
Sample #19265-2	916.6
Sample #19265-3	916.6
Sample #19265-4	916.6
Sample #19265-5	916.6
Sample #19265-6	916.6
Sample #19265-7	916.5
Sample #19265-8	916.6

Table 1: homogeneity test results of subsamples #19265

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)	0.10
reference test method	ISO12185:96
0.3 * R (ref. test method)	0.45

Table 2: evaluation of the repeatability of the subsamples #19265

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

Another batch of 10L VGO was obtained from a third party which were selected for the preparation of the metal determination sample. After homogenisation 90 plastic PE bottles of 100 mL were filled and labelled #19266. The homogeneity of the subsamples #19266 was checked by determination of Iron in accordance with IP501 on 8 stratified randomly selected subsamples.

	Iron in mg/kg
Sample #19266-1	15.98
Sample #19266-2	14.40
Sample #19266-3	14.65
Sample #19266-4	13.31
Sample #19266-5	13.76
Sample #19266-6	13.98
Sample #19266-7	14.30
Sample #19266-8	12.85

Table 3: homogeneity test results of subsamples #19266

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.

	Iron in mg/kg
r (observed)	2.7
reference test method	IP501:05
0.3 * R (ref. test method)	2.7

Table 4: evaluation of the repeatability of the subsamples #19266

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

To each of the participating laboratories a set of one bottle of 1L VGO labelled #19265 and one bottle of 0.1L VGO labelled #19266 were sent on November 20, 2019. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Vacuum Gasoil (VGO) 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 ANALYSES

The participants were asked to determine the following parameters:
On sample #19265: Acid Number (Total), Aniline Point, Asphaltenes, Carbon Residue (Micro method), Density at 15°C, Flash Point PMcc, Kinematic Viscosity at 50°C and at 100°C, Nitrogen, Pour Point (manual or automatic), Sulfur Total, Simulated Distillation and Distillation at 10 mmHg (IBP, 10% rec, 30% rec, 50% rec, 70% rec, 90% rec and FBP).
On sample #19266: Aluminum, Silicon, Sum of Aluminum and Silicon, Arsenic, Calcium, Zinc, Copper, Iron, Nickel, Sodium, Vanadium and Phosphorus.
Also, some extra questions regarding the Acid Number determination were asked.

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 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 reanalysis). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the test 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' and/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 in 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. In some cases, a reproducibility based on former iis proficiency tests could be used.
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:

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

The $z_{(target)}$ 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 proficiency test some problems were encountered with the dispatch of the samples to laboratories in Russian Federation. Eight laboratories reported after the final reporting date and three participants did not report any test results. Not all laboratories were able to report all requested parameters. In total 63 participants reported 1211 test results. Observed were 69 outlying test results, which is 5.7% of the numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

Not all original 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 table 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 intented for residual fuel oil and blending components. Where applicable, precision data for Fuel Oil were used.

Sample #19265

Acid Number (Total): 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 ASTM D664:18e2 mode IP 125mL but it is in agreement with the requirements of all other modes of ASTM D664:18e2.
When the data reported for Inflection Point (60 and 125 mL) were evaluated separately, the calculated reproducibility is in agreement with the requirements of IP 60 mL but not with 125 mL. The majority of laboratories reported to use 125 mL IP, therefore the z-scores were calculated with this method reproducibility.

Aniline Point: This determination was problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D611:12(2016). When method E and the group were the Heptane was in specification were evaluated separately the calculated reproducibilities were again not in agreement with the requirements of ASTM D611:12(2016).
Laboratories should consider the accuracy of the sample intake. Samples with a pour point above ambient temperature could be better weighed in. The used density to convert 10 mL into weight should be the density at ambient temperature.

Asphaltenes: No z-scores were calculated because the precision data of IP143:04 is applicable to values between 0.50 %M/M and 30.0 %M/M.

Carbon Residue (micro method): 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 D4530:15.

Density at 15°C: 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 ISO12185:96.

Flash Point PMcc: This determination was not problematic. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with requirements of ASTM D93-B:19.

Kinematic Viscosity at 50°C: This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D445:19.

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

Nitrogen: This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with ASTM D5762:18a.
When the test results of ASTM D5762 Volumetric and Gravimetric test methods were evaluated separately, the calculated reproducibility over the Volumetric test results was much smaller than the calculated reproducibility over the Gravimetric test results. The calculated reproducibility over the Volumetric test results is 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 problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with ASTM D5950:14.

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

Simulated Distillation: The Simulated Distillation over seven parameters was problematic. In total one statistical outlier was observed. The calculated reproducibilities after rejection of the statistical outlier for Initial Boiling Point and Final Boiling Point are in agreement with the requirements of ASTM D6352:15. For 10%, 30%, 50%, 70% and 90% recovered the calculated reproducibilities are not in agreement with the requirements of ASTM D6352:15.

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

Sample #19266

It was observed that the mean of most elements was below the application range of IP501:05. Nevertheless, it was decided to calculated z-scores as the laboratories were able to measure lower than these limits.

Unfortunately, a suitable reference test method providing the precision data is not available for the determination of all elements. For the tests that have no available precision data the calculated reproducibility was compared against the reproducibility estimated from the Horwitz equation.

Aluminum: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical ouliers is in agreement with the requirements of IP501:05 and IP470:05.

Silicon: This determination was problematic dependent on test method used. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the strict requirements of IP501:05 but in agreement with the requirements of IP470:05.

Sum of Aluminum and Silicon: This determination was not problematic. Two statistical outliers were observed and one other test result was excluded. However, the calculated reproducibility after rejection of the suspect data is in agreement with the requirements of IP501:05 and IP470:05.

Arsenic: This determination may be problematic at the low level of 0.03 mg/kg. No statistical outliers were observed but one test result was excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the estimated reproducibility using the Horwitz equation. Arsenic is an element that will be difficult to analyse by dry ashing or acid destruction at low levels. A more appropiate method might be UOP 986.

Calcium: This determination was not problematic. Five statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical ouliers is in full agreement with the requirements of IP501:05 and with IP470:05.

Phosphorus: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical oulier is in agreement with the requirements of IP501:05.

Zinc: This determination was problematic dependent on test method used. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical ouliers is not in agreement with the requirements of IP501:05 but it is in agreement with IP470:05.

Copper: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of IP621:16.

Iron: This determination was not problematic. Five statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical ouliers is in full 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 oulier is in agreement with the requirements of IP501:05 and IP470:05.

Sodium: This determination was not problematic. Five statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical ouliers is in full agreement with the requirements of IP501:05 and with IP470:05.

Vanadium: This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of IP501:05 and IP470:05.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

Parameter	unit	n	average	2.8 * sd	R(lit)
Acid Number (Total)	mg KOH/g	37	0.21	0.05	0.04
Aniline Point	°C	25	80.7	2.0	1.0
Asphaltenes	%M/M	34	<0.50	n.e.	n.e.
Carbon Residue (micro method)	%M/M	43	0.18	0.11	0.15
Density at 15°C	kg/m^3	57	916.8	1.5	1.5
Flash Point PMcc	°C	40	201.0	8.6	10
Kinematic Viscosity at 50°C	mm^2/s	54	34.42	0.530	2.912
Kinematic Viscosity at 100°C	mm^2/s	46	6.927	0.086	0.835
Nitrogen	mg/kg	37	1065	278	283
Pour Point, Manual	°C	43	31.8	5.5	9
Pour Point, Automated, 3°C interval	°C	22	29.9	8.8	6.1
Sulfur Total	%M/M	57	0.89	0.07	0.07
Simulated Distillation					
- Initial Boiling Point	°C	17	291.9	31.6	49.1
- Temp 10% recovered	°C	17	361.0	11.7	7.1
- Temp 30% recovered	°C	17	403.5	11.5	5.9
- Temp 50% recovered	°C	17	431.0	14.4	6.4
- Temp 70% recovered	°C	17	462.4	12.8	7.2
- Temp 90% recovered	°C	17	508.2	12.9	10.5
- Final Boiling Point	°C	15	580.5	36.7	38.1
Distillation at 10 mmHg as AET					
- Initial Boiling Point	°C	32	319.1	41.1	49.5
- Temp 10% recovered	°C	32	379.5	13.4	14.8
- Temp 30% recovered	°C	32	411.6	11.5	10.6
- Temp 50% recovered	°C	31	434.7	11.1	10.3
- Temp 70% recovered	°C	31	462.5	14.2	9.2
- Temp 90% recovered	°C	31	507.5	20.4	22.2
- Final Boiling Point	°C	31	538.7	31.6	26.9

Table 5: reproducibilities of results of sample #19265

Parameter	unit	n	average	2.8 * sd	R(lit)
Aluminum as Al	mg/kg	19	3.4	1.1	1.1
Silicon as Si	mg/kg	19	5.4	2.4	1.8
Sum of Aluminum and Silicon	mg/kg	16	8.8	2.4	2.1
Arsenic as As	mg/kg	5	0.03	0.04	0.02
Calcium as Ca	mg/kg	24	4.5	1.7	1.7
Phosphorus as P	mg/kg	16	1.0	0.9	1.3
Zinc as Zn	mg/kg	22	1.1	0.8	0.5
Copper as Cu	mg/kg	18	0.1	0.3	1.6
Iron as Fe	mg/kg	35	13.5	4.0	3.9
Nickel as Ni	mg/kg	42	2.2	1.5	2.6
Sodium as Na	mg/kg	37	11.4	4.0	4.1
Vanadium as V	mg/kg	41	5.7	2.2	4.8

Table 6: reproducibilities of results of sample #19266

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2019 WITH PREVIOUS PTS

	December 2019	December 2018	December 2017	December 2016	December 2015
Number of reporting labs	63	66	74	73	54
Number of test results	1211	1113	1275	1241	897
Number of statistical outliers	69	39	61	53	22
Percentage outliers	5.7%	3.5%	4.8%	4.3%	2.5%

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 reference test methods. The conclusions are given the following table.

Determination	December 2019	December 2018	December 2017	December 2016	December 2015
Acid Number (Total)	-	++	+	++	+
Aniline Point	--	--	--	-	--
Asphaltenes	n.e.	(--)*	(--)*	(--)*	(--)*
Carbon Residue (micro method)	+	-	+/-	-	+/-
Density at 15°C	+/-	+/-	+/-	+/-	+/-
Flash Point PMcc	+	+/-	+	++	+
Kinematic Viscosity at 50°C	++	++	++	++	++
Kinematic Viscosity at 100°C	++	++	++	++	++
Nitrogen	+	+	-	+/-	+/-
Pour Point, Manual	+	++	++	++	n.e.
Pour Point, Automated 3°C interval	-	+	++	++	n.e.
Sulfur Total	+/-	+/-	+	+	+
Simulated Distillation	-	+	-	+	-
Distillation at 10 mmHg as AET	+/-	+	+	-	+/-
Aluminum as Al	+/-	n.a.	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.	n.a.
Arsenic as As	--	n.e.	n.e.	n.e.	n.e.
Calcium as Ca	+/-	-	+/-	+/-	+/-
Phosphorus as P	+	n.a.	n.a.	n.a.	n.a.
Zinc as Zn	-	n.a.	n.a.	n.a.	n.a.
Copper as Cu	++	n.e.	n.e.	n.e.	n.e.
Iron as Fe	+/-	+	+	-	+/-
Nickel as Ni	+	+	++	++	++
Sodium as Na	+/-	++	++	+	+/-
Vanadium as V	+/-	+	++	++	+

Table 8: comparison determinations against the reference test methods

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

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

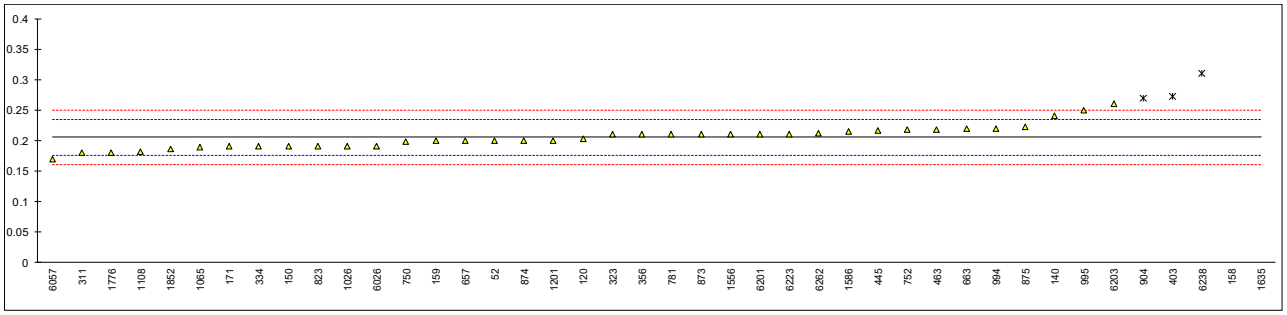
- ++: group performed much better than the reference test method
- + : group performed better than the reference test method
- +/-: group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test method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

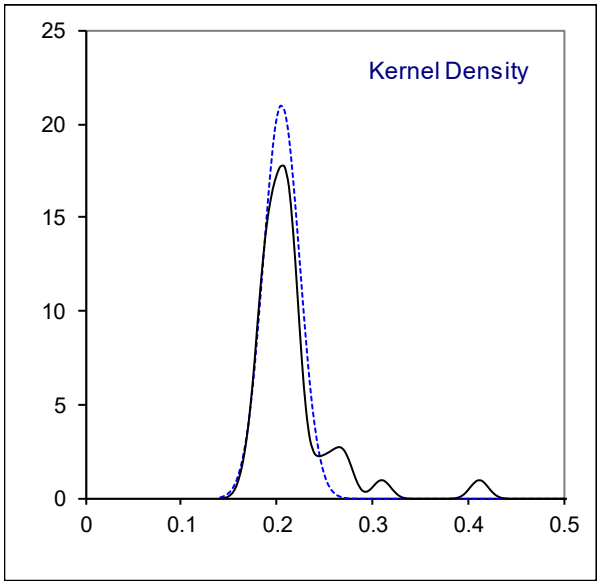
APPENDIX 1

Determination of Acid Number (Total) on sample #19265; results in mg KOH/g

lab	method	value	mark	z(targ)	end point	volume	remarks
52	D664-A	0.20		-0.35	Inflection Point	125mL	
62		-----		-----	---	---	
120	D664-A	0.203		-0.15	Buffer End Point pH 10	125mL	
140	D664-B	0.24		2.32	---	---	
150	D664-A	0.19		-1.02	---	---	
154		-----		-----	---	---	
158	D664-A	0.412	R(0.01)	13.80	Inflection Point	60mL	
159	D664-A	0.20		-0.35	Inflection Point	---	
171	D664-A	0.19		-1.02	---	---	
225		-----		-----	---	---	
311	D664-A	0.18		-1.68	---	---	
313		-----		-----	---	---	
317		-----		-----	---	---	
323	D664-A	0.21		0.32	Inflection Point	125mL	
333		-----		-----	---	---	
334	D664-A	0.19		-1.02	Buffer End Point pH 11	125mL	
356	D664-B	0.21		0.32	Inflection Point	125mL	
381		-----		-----	---	---	
403	D664-A	0.273	R(0.01)	4.52	Inflection Point	125mL	
445	D664-A	0.217		0.79	Inflection Point	60mL	
463	D664-A	0.218		0.85	Buffer End Point pH 11	125mL	
608		-----		-----	---	---	
657	D664-A	0.20		-0.35	Inflection Point	125mL	
663	D664-A	0.22		0.99	Inflection Point	125mL	
732		-----		-----	---	---	
750	D664-A	0.199		-0.42	Inflection Point	60mL	
752	D664-A	0.218		0.85	---	---	
778		-----		-----	---	---	
781	D664-A	0.21		0.32	Inflection Point	60mL	
785		-----		-----	---	---	
798		-----		-----	---	---	
823	D664-A	0.19		-1.02	Inflection Point	125mL	
873	D664-A	0.21		0.32	Buffer End Point pH 10	125 mL	
874	D664-A	0.20		-0.35	Buffer End Point pH 10	125 mL	
875	D664-A	0.223		1.19	---	---	
904	D664-B	0.27	R(0.01)	4.32	Inflection Point	125mL	
994	D664-A	0.22		0.99	Inflection Point	60mL	
995	D664-A	0.25		2.99	Inflection Point	125mL	
997		-----		-----	---	---	
1011		-----		-----	---	---	
1026	D664-A	0.19		-1.02	Buffer End Point pH 10	125mL	
1065	D664-A	0.189		-1.08	Inflection Point	125mL	
1081		-----		-----	---	---	
1108	D664-A	0.182		-1.55	Inflection Point	125mL	
1134		-----		-----	---	---	
1191		-----		-----	---	---	
1201	D664-A	0.20		-0.35	---	---	
1229		-----		-----	---	---	
1297		-----		-----	---	---	
1510		-----		-----	---	---	
1556	D664-A	0.21		0.32	Buffer End Point pH 10	125mL	
1586	D664-B	0.215		0.65	Inflection Point	125mL	
1635	D664-A	1.2541	R(0.01)	70.02	---	---	
1776	D664-A	0.18		-1.68	Buffer End Point pH 10	125mL	
1852	ISO6618	0.186		-1.28	Inflection Point	125mL	
1881		-----		-----	---	---	
6020		-----		-----	---	---	
6026	D664-A	0.1910		-0.95	Buffer End Point pH 10	125mL	
6057	D664-A	0.17		-2.35	Inflection Point	60mL	
6112		-----		-----	---	---	
6198		-----		-----	---	---	
6201		0.21		0.32	Inflection Point	125mL	
6203	D664-A	0.260		3.66	Inflection Point	125mL	
6223	D664-A	0.21		0.32	Inflection Point	125mL	
6238	D664-A	0.31	R(0.01)	6.99	Inflection Point	60mL	
6262	D664-A	0.2124		0.48	Inflection Point	125mL	

		IP (60 + 125 mL) only
normality	suspect	suspect
N	37	21
outliers	5	4
mean (n)	0.2052	0.2076
st.dev. (n)	0.01898	0.02060
R(calc.)	0.0531	0.0577
st.dev.(D664:18e2 IP 125 mL)	0.01498	0.01498
R(D664:18e2 IP 125 mL)	0.0419	0.0424
Compare		
R(D664:18e2 BEP 60 mL)	0.1182	---
R(D664:18e2 IP 60 mL)	0.1098	0.1108
R(D664:18e2 BEP 125 mL)	0.0598	---





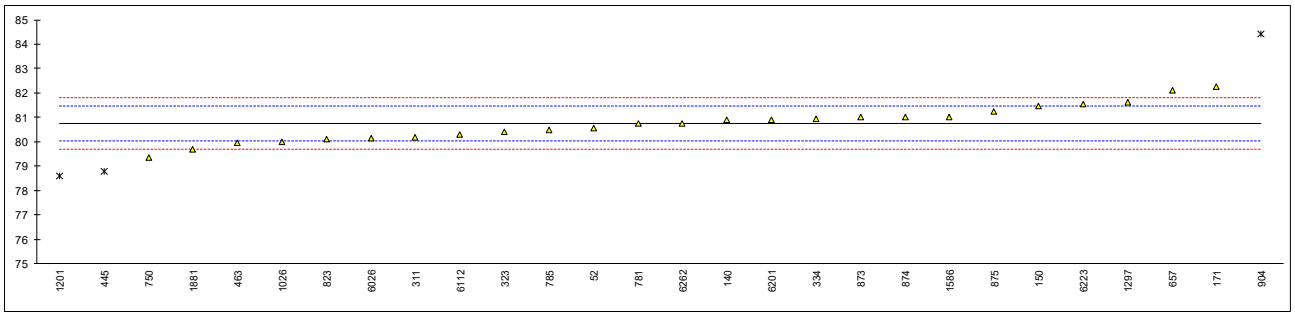
Determination of Aniline Point on sample #19265; results in °C

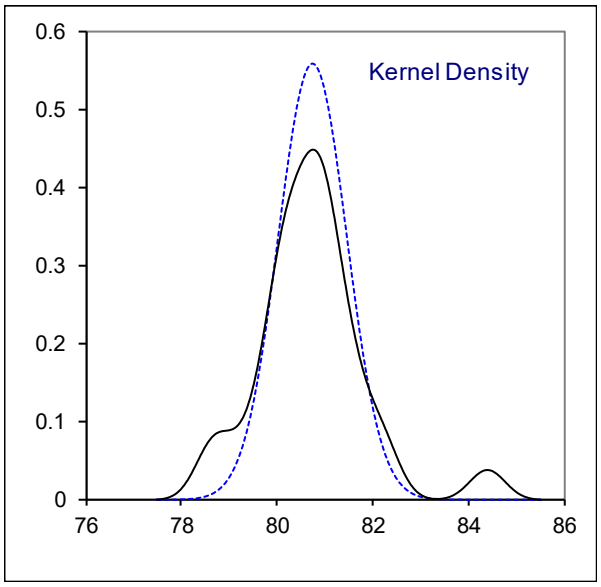
lab	method	value	mark	z(targ)	n-Heptane	remarks
52	D611-E	80.55		-0.55	69.25	
62		-----		-----	-----	
120		-----		-----	-----	
140	D611-B	80.9		0.43	-----	
150	D611-E	81.45		1.97	-----	
154		-----		-----	-----	
158		-----		-----	-----	
159		-----		-----	-----	
171	D611-E	82.25		4.21	-----	
225		-----		-----	-----	
311	D611-E	80.20		-1.53	69.20	
313		-----		-----	-----	
317		-----		-----	-----	
323	D611-B	80.4		-0.97	-----	
333		-----		-----	-----	
334	D611-B	80.95		0.57	69.4	
356		-----		-----	-----	
381		-----		-----	-----	
403		-----		-----	-----	
445	D611-D	78.80	R(0.05)	-5.45	69.40	
463	D611-B	79.95		-2.23	69.25	
608		-----		-----	-----	
657	D611-B	82.10	C	3.79	69.30 C	first reported 78.50 and 71.00
663		-----		-----	-----	
732		-----		-----	-----	
750	D611	79.35		-3.91	-----	
752		-----		-----	-----	
778		-----		-----	-----	
781	D611-E	80.75		0.01	69.2	
785	D611-E	80.5		-0.69	69.2	
798		-----		-----	-----	
823	D611-E	80.1		-1.81	-----	
873	D611-E	81.00		0.71	69.30	
874	D611-E	81.00		0.71	-----	
875	D611-E	81.25		1.41	-----	
904	D611-A	84.4	R(0.01)	10.23	-----	
994		-----		-----	-----	
995		-----		-----	-----	
997		-----		-----	-----	
1011		-----		-----	-----	
1026	D611-E	80.0		-2.09	69.1	
1065		-----		-----	-----	
1081		-----		-----	-----	
1108		-----		-----	-----	
1134		-----		-----	-----	
1191		-----		-----	-----	
1201	D611-A	78.6	R(0.05)	-6.01	-----	
1229		-----		-----	-----	
1297	D611-E	81.6		2.39	68.8	
1510		-----		-----	-----	
1556		-----		-----	-----	
1586	D611-A	81		0.71	-----	
1635		-----		-----	-----	
1776		-----		-----	-----	
1852		-----		-----	-----	
1881	D611-B	79.70		-2.93	69.20	
6020		-----		-----	-----	
6026	D611-A	80.15		-1.67	69.2	
6057		-----		-----	-----	
6112	D611-E	80.3		-1.25	-----	
6198		-----		-----	-----	
6201		80.90		0.43	69.15	
6203		-----		-----	-----	
6223	D611-E	81.55		2.25	69.3 C	first reported 70.65
6238		-----		-----	-----	
6262	D611-E	80.75		0.01	69.45	

		Only D611-E	Only Heptane OK
normality	OK	OK	suspect
n	25	16	15
outliers	3	0	0
mean (n)	80.746	80.959	80.523
st.dev. (n)	0.7137	0.6924	0.7878
R(calc.)	1.998	1.939	2.206
st.dev.(D611:12)	0.3571	0.3571	0.3571
R(D611:12)	1.0	1.0	1.0

Compare

R(iis18G07) 3.603 at a mean of 81.630





Determination of Asphaltenes on sample #19265; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	IP143	<0.5		-----	
62		-----		-----	
120		-----		-----	
140	IP143	0.30		-----	
150	IP143	<0.05		-----	
154	IP143	<0.05		-----	
158		-----		-----	
159		-----		-----	
171	IP143	0.10		-----	
225		-----		-----	
311		-----		-----	
313		-----		-----	
317		-----		-----	
323	IP143	<0.50		-----	
333		-----		-----	
334		-----		-----	
356	IP143	<0.50		-----	
381	INH-642	0.021		-----	
403		-----		-----	
445	IP143	0.11		-----	
463	IP143	0.024		-----	
608	D6560	0.0816		-----	
657	IP143	<1.00		-----	
663		-----		-----	
732		-----		-----	
750	D6560	0.020		-----	
752	INH-642	0.0519		-----	
778		-----		-----	
781	INH-642	0.0412		-----	
785	INH-642	0.040		-----	
798		-----		-----	
823	IP143	0.033		-----	
873	IP143	0.045		-----	
874	IP143	0.055		-----	
875	INH-642	0.0455		-----	
904	IP143	0.048		-----	
994		-----		-----	
995	IP143	0.033		-----	
997		-----		-----	
1011		-----		-----	
1026	IP143	0.30		-----	
1065	D6560	0.02		-----	
1081		-----		-----	
1108		-----		-----	
1134		-----		-----	
1191		-----		-----	
1201		-----		-----	
1229		-----		-----	
1297	D6560	0.1490		-----	
1510	IP143	0.13		-----	
1556		-----		-----	
1586	IP143	0.02		-----	
1635	D6560	0.013		-----	
1776		-----		-----	
1852		-----		-----	
1881		0.0591		-----	
6020		-----		-----	
6026	In house	0.0468		-----	
6057	IP143	0.03		-----	
6112		-----		-----	
6198		-----		-----	
6201		0.06		-----	
6203	IP143	0.935		-----	
6223	IP143	0.169		-----	
6238		-----		-----	
6262	IP143	0.3		-----	
	n	34			
	mean(n)	<0.50			Precision applicable between 0.50 – 30 %M/M

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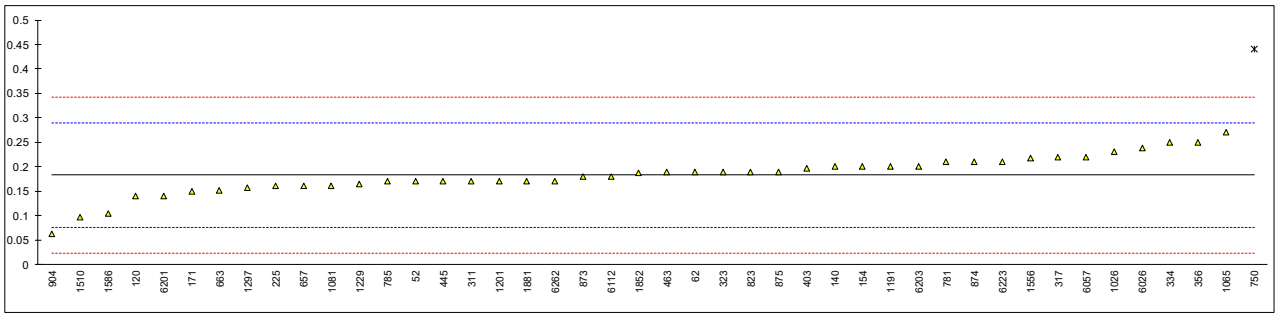
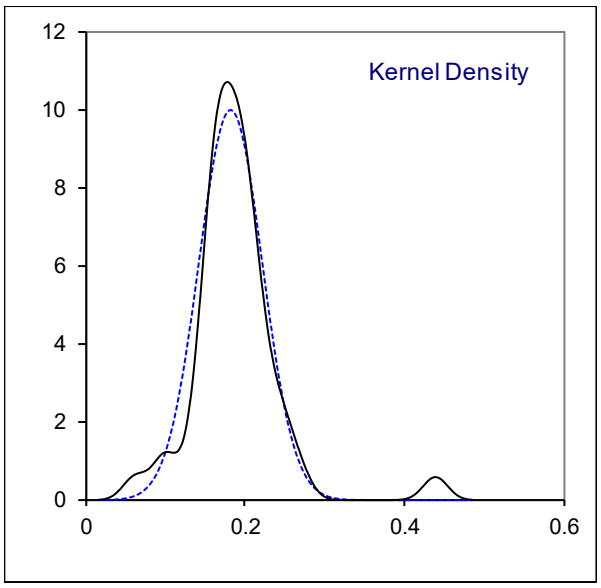
Determination of Carbon Residue (micro method) on sample #19265; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	D4530	0.17		-0.24	
62	D4530	0.19	C	0.14	first reported 0.49
120	D4530	0.1395		-0.81	
140	D4530	0.20		0.32	
150	D4530	<0.10		-----	
154	D4530	0.20		0.32	
158		-----		-----	
159		-----		-----	
171	D4530	0.15		-0.62	
225	D4530	0.16		-0.43	
311	D4530	0.17		-0.24	
313		-----		-----	
317	D4530	0.22		0.70	
323	D4530	0.19		0.14	
333		-----		-----	
334	D4530	0.25		1.26	
356	D4530	0.25		1.26	
381		-----		-----	
403	ISO10370	0.196		0.25	
445	D4530	0.17		-0.24	
463	D4530	0.189		0.12	
608		-----		-----	
657	D4530	0.16		-0.43	
663	D4530	0.152		-0.58	
732		-----		-----	
750	D189	0.44	R(0.01)	4.84	
752		-----		-----	
778		-----		-----	
781	D4530	0.21		0.51	
785	D4530	0.17		-0.24	
798		-----		-----	
823	ISO10370	0.19		0.14	
873	D4530	0.18		-0.05	
874	D4530	0.21		0.51	
875	D4530	0.19		0.14	
904	D4530	0.063		-2.25	
994		-----		-----	
995		-----		-----	
997		-----		-----	
1011		-----		-----	
1026	D4530	0.23		0.89	
1065	D4530	0.27		1.64	
1081	ISO10370	0.16		-0.43	
1108		-----		-----	
1134		-----		-----	
1191	ISO10370	0.200		0.32	
1201	D4530	0.17		-0.24	
1229	ISO10370	0.164		-0.35	
1297	D4530	0.158		-0.46	
1510	ISO10370	0.097		-1.61	
1556	ISO10370	0.217		0.64	
1586	D4530	0.105		-1.46	
1635		-----		-----	
1776		-----		-----	
1852	ISO10370	0.187		0.08	
1881	D4530	0.17		-0.24	
6020		-----		-----	
6026	D4530	0.2390		1.06	
6057	D4530	0.22		0.70	
6112	D4530	0.18		-0.05	
6198		-----		-----	
6201		0.14		-0.80	
6203	ISO10370	0.201		0.34	
6223	D4530	0.21		0.51	
6238		-----		-----	
6262	D4530	0.17		-0.24	

normality	suspect
n	43
outliers	1
mean (n)	0.1827
st.dev. (n)	0.03995
R(calc.)	0.1119
st.dev.(D4530:15)	0.05321
R(D4530:15)	0.1490
Compare	
R(ISO10370)	0.0248

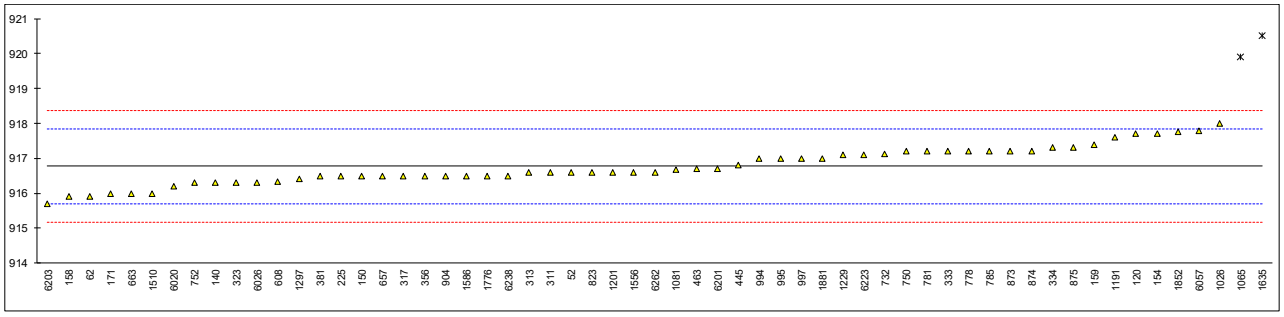


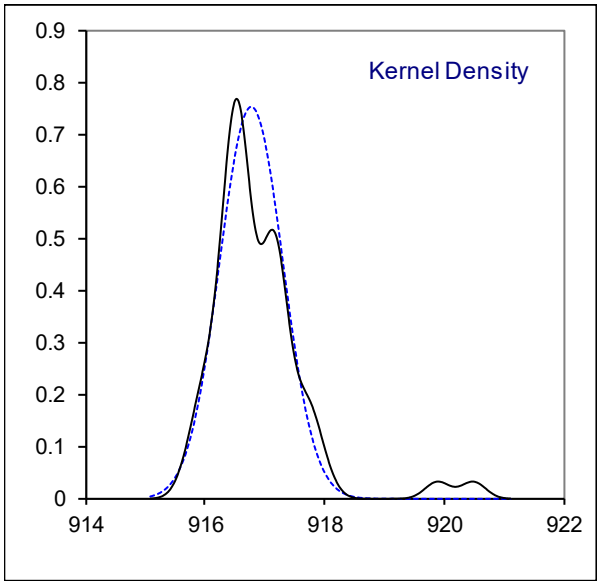


Determination of Density at 15°C on sample #19265; results in kg/m^3

lab	method	value	mark	z(targ)	remarks
52	D4052	916.6		-0.32	
62	D4052	915.9		-1.63	
120	D4052	917.7		1.73	
140	D4052	916.3		-0.88	
150	D1298	916.5		-0.51	
154	D4052	917.7		1.73	
158	D4052	915.9		-1.63	
159	D4052	917.4		1.17	
171	ISO12185	916.0		-1.44	
225	D4052	916.5		-0.51	
311	ISO12185	916.6		-0.32	
313	ISO12185	916.6		-0.32	
317	ISO12185	916.5		-0.51	
323	ISO12185	916.3		-0.88	
333	ISO12185	917.2		0.80	
334	ISO12185	917.3		0.99	
356	ISO12185	916.5		-0.51	
381	ISO12185	916.5		-0.51	
403		-----		-----	
445	ISO12185	916.8		0.05	
463	ISO12185	916.7		-0.13	
608	D4052	916.33		-0.82	
657	D4052	916.5		-0.51	
663	D4052	916.00		-1.44	
732	ISO12185	917.12		0.65	
750	D1298	917.2		0.80	
752	D1298	916.3		-0.88	
778	ISO12185	917.2		0.80	
781	ISO12185	917.2		0.80	
785	D1298	917.2		0.80	
798		-----		-----	
823	ISO12185	916.6		-0.32	
873	ISO12185	917.2		0.80	
874	ISO12185	917.2		0.80	
875	ISO12185	917.3		0.99	
904	D1298	916.5	C	-0.51	first reported 0.9265 kg/L
994	ISO12185	917.0		0.43	
995	ISO12185	917.0		0.43	
997	D4052	917.0		0.43	
1011		-----		-----	
1026	D4052	918.0		2.29	
1065	D4052	919.9	R(0.01)	5.84	
1081	D4052	916.68		-0.17	
1108		-----		-----	
1134		-----		-----	
1191	ISO12185	917.6		1.55	
1201	ISO12185	916.6		-0.32	
1229	ISO12185	917.1		0.61	
1297	D4052	916.4		-0.69	
1510	ISO12185	916.0		-1.44	
1556	ISO12185	916.6		-0.32	
1586	D4052	916.5	C	-0.51	first reported 0.9165 kg/m^3
1635	ISO3675	920.5	R(0.01)	6.96	
1776	ISO12185	916.5		-0.51	
1852	ISO12185	917.77		1.86	
1881	ISO12185	917.0		0.43	
6020	ISO12185	916.2		-1.07	
6026	D1298	916.3	C	-0.88	first reported 0.9163 no unit
6057	ISO12185	917.8		1.92	
6112		-----		-----	
6198		-----	W	-----	Test result withdrawn. First reported 918.8
6201		916.7		-0.13	
6203	D7042	915.7		-2.00	
6223	ISO12185	917.1		0.61	
6238	D4052	916.5		-0.51	
6262	ISO12185	916.6		-0.32	

normality	OK
n	57
outliers	2
mean (n)	916.77
st.dev. (n)	0.530
R(calc.)	1.48
st.dev.(ISO12185:96)	0.536
R(ISO12185:96)	1.5

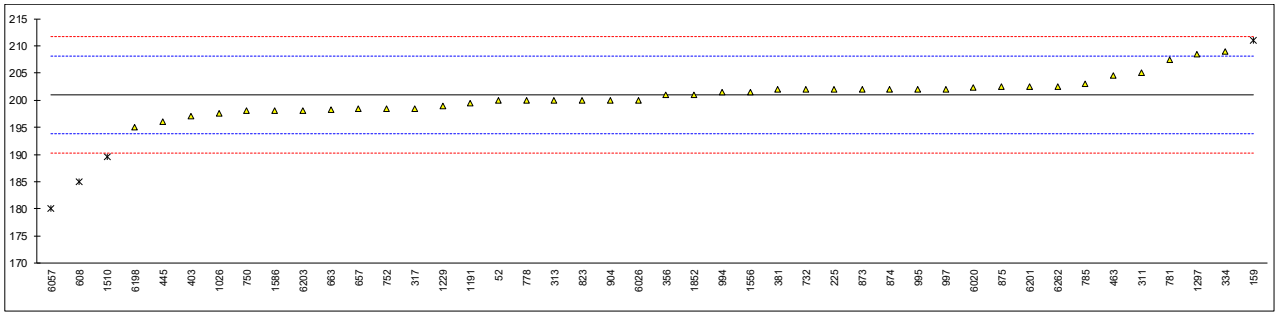


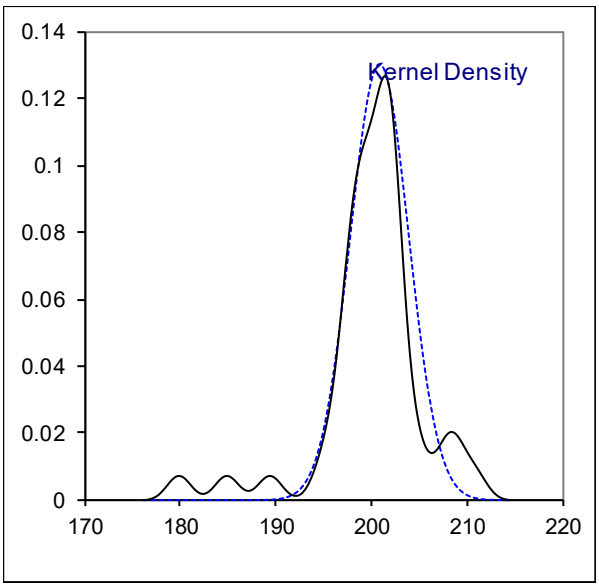


Determination of Flash Point PMcc on sample #19265; results in °C

lab	method	value	mark	z(targ)	remarks
52	D93-B	200.0		-0.27	
62		-----		-----	
120		-----		-----	
140	D93-B	>230		>8.13	possibly a false positive test result?
150	D93-B	>110.0		-----	
154	D93-B	>121.0		-----	
158	D93-B	>210		-----	
159	D93-A	211.0	R(0.05)	2.81	
171	D93-B	>110		-----	
225	D93-B	202.0		0.29	
311	D93-B	205.0		1.13	
313	D93-B	200.0		-0.27	
317	D93-B	198.5		-0.69	
323		-----		-----	
333		-----		-----	
334	D93-B	209.0		2.25	
356	D93-B	201.0		0.01	
381	ISO2719	202		0.29	
403	D93-A	197.0		-1.11	
445	D93-B	196.0		-1.39	
463	D93-B	204.5		0.99	
608	D93-A	185	R(0.01)	-4.47	
657	D93-B	198.5		-0.69	
663	D93-B	198.3		-0.74	
732	ISO2719	202.0		0.29	
750	D93-B	198.0		-0.83	
752	INH-6356	198.5		-0.69	
778	D93-B	200.0		-0.27	
781	D93-B	207.5		1.83	
785	D93-B	203.0		0.57	
798		-----		-----	
823	D93-B	200.0		-0.27	
873	D93-B	202.0		0.29	
874	D93-B	202.0		0.29	
875	D93-B	202.5		0.43	
904	D93-B	200		-0.27	
994	D93-B	201.5		0.15	
995	D93-B	202.0		0.29	
997	D93-B	202.0		0.29	
1011		-----		-----	
1026	ISO2719	197.5		-0.97	
1065		-----		-----	
1081	D93-A	>220		>5.33	possibly a false positive test result?
1108		-----		-----	
1134		-----		-----	
1191	ISO2719	199.5		-0.41	
1201		-----		-----	
1229	ISO2719	199.0		-0.55	
1297	D93-B	208.5		2.11	
1510	D93-B	189.5	R(0.05)	-3.21	
1556	ISO2719	201.5		0.15	
1586	D93-B	198.0		-0.83	
1635		-----		-----	
1776	ISO2719	>120		-----	
1852	ISO2719	201		0.01	
1881		-----		-----	
6020	D93-B	202.4		0.40	
6026	D93-B	200.0		-0.27	
6057	D93-B	180.0	R(0.01)	-5.87	
6112		-----		-----	
6198	D93-B	195.0		-1.67	
6201		202.5	C	0.43	first reported 179.5
6203	ISO2719	198.0		-0.83	
6223		-----		-----	
6238		-----		-----	
6262	D93-B	202.5		0.43	

normality	OK
n	40
outliers	4
mean (n)	200.95
st.dev. (n)	3.061
R(calc.)	8.57
st.dev.(D93-B:19)	3.571
R(D93-B:19)	10
Compare	
R(ISO2719)	10

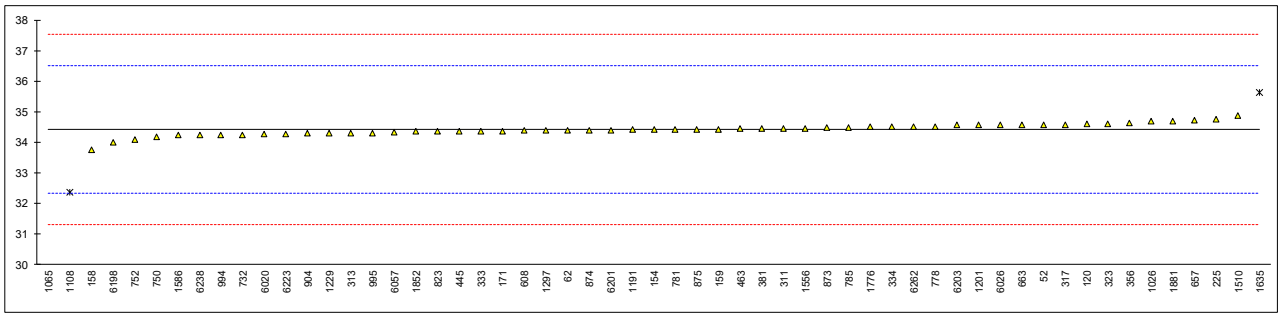


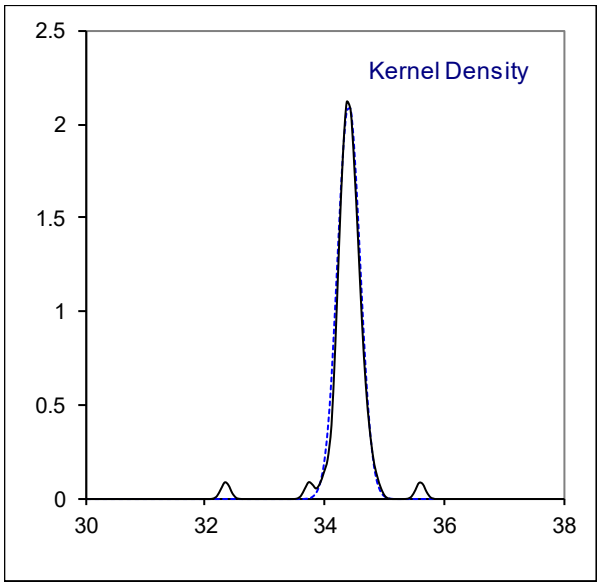


Determination of Kinematic Viscosity at 50°C on sample #19265; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
52	D445	34.58		0.15	
62	D445	34.398		-0.02	
120	D445	34.597		0.17	
140		-----		-----	
150		-----		-----	
154	D445	34.41		-0.01	
158	D445	33.76125		-0.63	
159	D445	34.4296	C	0.01	first reported 28.8079
171	D445	34.37		-0.05	
225	D445	34.76		0.33	
311	D445	34.46		0.04	
313	D445	34.31		-0.11	
317	D445	34.58		0.15	
323	D445	34.61		0.18	
333	D445	34.37		-0.05	
334	ISO3104	34.50		0.08	
356	D445	34.62		0.19	
381	D445	34.46	C	0.04	first reported as K.V. at 100°C
403		-----		-----	
445	D445	34.35		-0.07	
463	D445	34.440		0.02	
608	D445	34.38		-0.04	
657	D445	34.72		0.29	
663	D445	34.571		0.14	
732	D445	34.25		-0.16	
750	D445	34.19		-0.22	
752	D445	34.10		-0.31	
778	D445	34.52		0.10	
781	D445	34.42		0.00	
785	D445	34.49		0.07	
798		-----		-----	
823	D445	34.35		-0.07	
873	D445	34.48		0.06	
874	D445	34.40		-0.02	
875	D445	34.42		0.00	
904	D445	34.29		-0.13	
994	D445	34.24		-0.17	
995	D445	34.31		-0.11	
997		-----		-----	
1011		-----		-----	
1026	ISO3104	34.7		0.27	
1065	D445	19.82	R(0.01)	-14.04	
1081		-----		-----	
1108	D445	32.36	R(0.01)	-1.98	
1134		-----		-----	
1191	ISO3104	34.406		-0.01	
1201	D445	34.56		0.13	
1229	ISO3104	34.29		-0.13	
1297	D7042	34.39		-0.03	
1510	D445	34.88		0.44	
1556	ISO3104	34.460		0.04	
1586	D445	34.23		-0.18	
1635	ISO3104	35.62	R(0.01)	1.15	
1776	ISO3104	34.495		0.07	
1852	ISO3104	34.344		-0.07	
1881	D445	34.70		0.27	
6020	D445	34.255		-0.16	
6026	D445	34.56		0.13	
6057	D445	34.32		-0.10	
6112		-----		-----	
6198	D445	34.00		-0.40	
6201		34.40		-0.02	
6203	D7042	34.557		0.13	
6223	D445	34.27		-0.14	
6238	D445	34.236		-0.18	
6262	D445	34.511		0.09	

normality	not OK
n	54
outliers	3
mean (n)	34.4204
st.dev. (n)	0.18918
R(calc.)	0.5297
st.dev.(D445:19)	1.04011
R(D445:19)	2.9123
Compare	
R(ISO3104)	2.5471

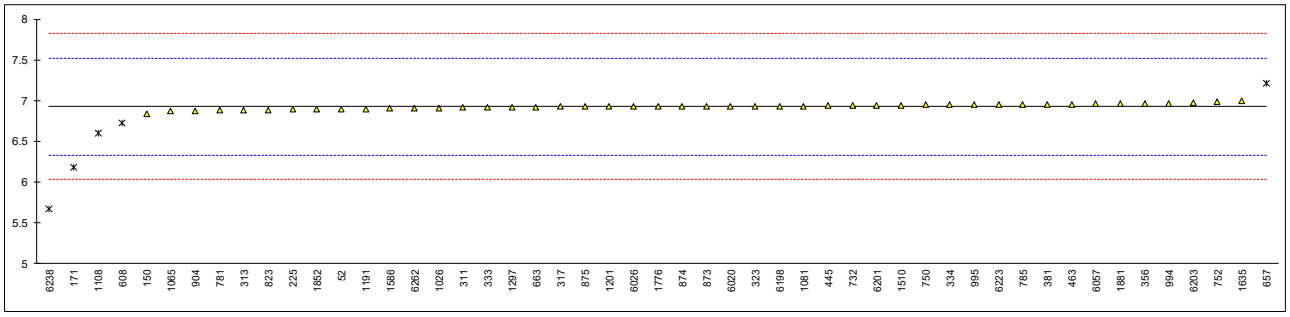


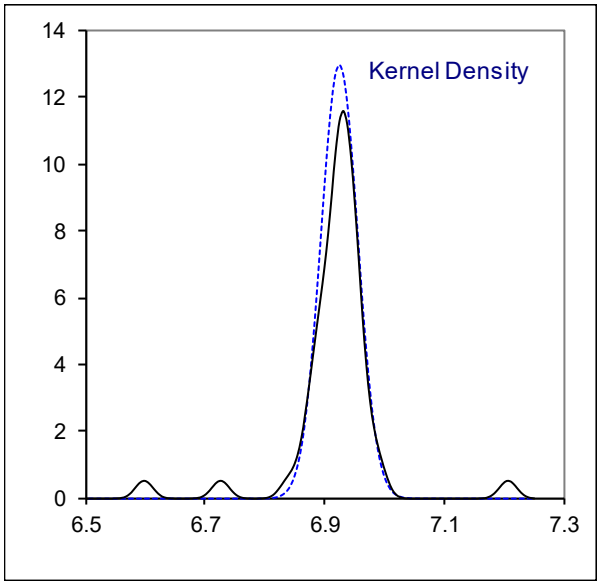


Determination of Kinematic Viscosity at 100°C on sample #19265; results in mm^2/s

lab	method	value	mark	z(targ)	remarks
52	D445	6.899		-0.09	
62		-----		-----	
120		-----		-----	
140		-----		-----	
150	D445	6.842		-0.28	
154		-----		-----	
158		-----		-----	
159		-----		-----	
171	D445	6.178	R(0.01)	-2.51	
225	D445	6.895		-0.11	
311	D445	6.915		-0.04	
313	D445	6.886		-0.14	
317	D445	6.926		0.00	
323	D445	6.931		0.01	
333	D445	6.915		-0.04	
334	D445	6.947		0.07	
356	D445	6.961		0.12	
381	D445	6.950	C	0.08	first reported as K.V. at 50°C
403		-----		-----	
445	D445	6.936		0.03	
463	D445	6.9541		0.09	
608	D445	6.728	R(0.01)	-0.67	
657	D445	7.209	R(0.01)	0.95	
663	D445	6.9230		-0.01	
732	D445	6.937		0.03	
750	D445	6.947		0.07	
752	D445	6.988		0.21	
778		-----		-----	
781	D445	6.881		-0.15	
785	D445	6.948		0.07	
798		-----		-----	
823	D445	6.888		-0.13	
873	D445	6.929		0.01	
874	D445	6.927		0.00	
875	D445	6.926		0.00	
904	D445	6.873		-0.18	
994	D445	6.965		0.13	
995	D445	6.947		0.07	
997		-----		-----	
1011		-----		-----	
1026	D445	6.91		-0.06	
1065	D445	6.869		-0.19	
1081	D445	6.933		0.02	
1108	D445	6.600	R(0.01)	-1.09	
1134		-----		-----	
1191	ISO3104	6.8997		-0.09	
1201	D445	6.926		0.00	
1229		-----		-----	
1297	D7042	6.920		-0.02	
1510	D445	6.945		0.06	
1556		-----		-----	
1586	D445	6.902		-0.08	
1635	ISO3104	6.993		0.22	
1776	ISO3104	6.9261		0.00	
1852	ISO3104	6.896		-0.10	
1881	D445	6.960		0.11	
6020	D445	6.930		0.01	
6026	D445	6.926		0.00	
6057	D445	6.958		0.11	
6112		-----		-----	
6198	D445	6.932		0.02	
6201		6.942		0.05	
6203	D7042	6.9703		0.15	
6223	ISO3104	6.947		0.07	
6238	D445	5.667	R(0.01)	-4.22	
6262	D445	6.9057		-0.07	

normality	OK
n	46
outliers	5
mean (n)	6.9267
st.dev. (n)	0.03083
R(calc.)	0.0863
st.dev.(D445:19)	0.29834
R(D445:19)	0.8354
Compare	
R(ISO3104)	0.5971

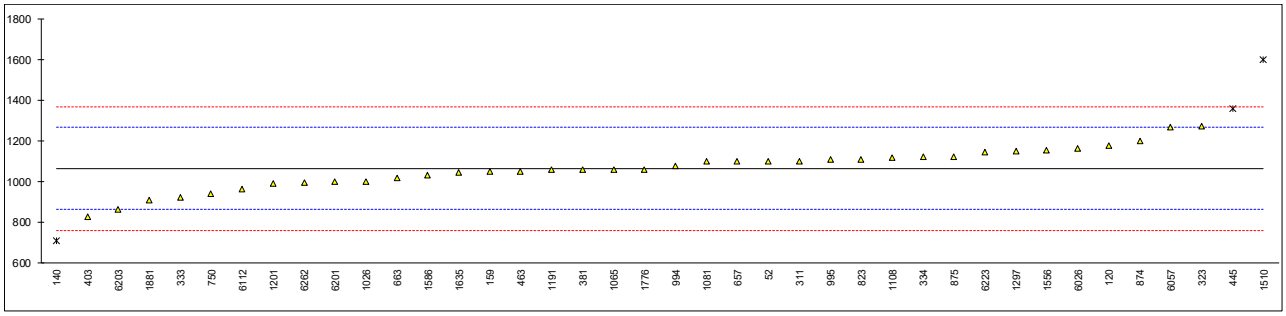


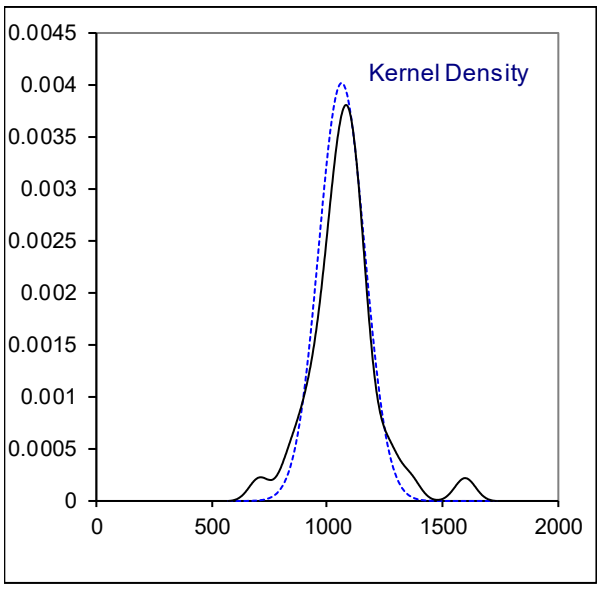


Determination of Nitrogen on sample #19265; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	D4629	1100		0.34	
62		-----		-----	
120	D4629	1176.449		1.10	
140	D5762 Volumetric	710	R(0.01)	-3.51	
150		-----		-----	
154		-----		-----	
158		-----		-----	
159	D4629	1048.7		-0.16	
171		-----		-----	
225		-----		-----	
311	D5762 Volumetric	1100		0.34	
313		-----		-----	
317		-----		-----	
323	D5762 Gravimetric	1270	C	2.02	first reported 1470
333	D5762 Volumetric	921		-1.43	
334	D5762 Gravimetric	1120		0.54	
356		-----		-----	
381	D4629	1060		-0.05	
403	D5762 Gravimetric	829.80		-2.33	
445	D5762 Gravimetric	1360	R(0.01)	2.91	
463	D5762 Gravimetric	1050		-0.15	
608		-----		-----	
657	D5762 Gravimetric	1100		0.34	
663	D5762 Gravimetric	1016.5		-0.48	
732		-----		-----	
750	D5762	940		-1.24	
752		-----		-----	
778		-----		-----	
781		-----		-----	
785		-----		-----	
798		-----		-----	
823	D5762 Volumetric	1110		0.44	
873		-----		-----	
874	D5762	1200		1.33	
875	D5762 Gravimetric	1123		0.57	
904		-----		-----	
994	D5762 Volumetric	1075		0.10	
995	D3228	1107		0.41	
997		-----		-----	
1011		-----		-----	
1026		1000		-0.65	
1065	D4629	1060		-0.05	
1081	D4629	1097.64		0.32	
1108	D5762 Gravimetric	1117		0.51	
1134		-----		-----	
1191	D5762 Volumetric	1059		-0.06	
1201	D5762 Gravimetric	990		-0.74	
1229		-----		-----	
1297	D4629	1150		0.84	
1510	D5291	1600	R(0.01)	5.28	
1556	D5762 Volumetric	1156		0.90	
1586	D5762 Gravimetric	1031		-0.34	
1635	D5762 Gravimetric	1046		-0.19	
1776	D5762 Volumetric	1060		-0.05	
1852		-----		-----	
1881	D5762 Volumetric	907		-1.56	
6020		-----		-----	
6026	D5762 Gravimetric	1163.87		0.97	
6057	D5762 Gravimetric	1268		2.00	
6112	D5762 Volumetric	963		-1.01	
6198		-----		-----	
6201		999		-0.66	
6203	D5762 Gravimetric	863.5		-1.99	
6223	D5762 Volumetric	1145	C	0.79	first reported 1383
6238		-----		-----	
6262	D4629	993.278		-0.71	

		D5762 Volumetric	D5762 Gravimetric
normality	OK	OK	OK
n	37	10	14
outliers	3	1	1
mean (n)	1065.32	1049.60	1070.62
st.dev. (n)	99.268	89.334	126.651
R(calc.)	277.95	250.13	354.62
st.dev.(D5762:18a)	101.205	101.205	101.205
R(D5762:18a)	283.37	279.19	284.78

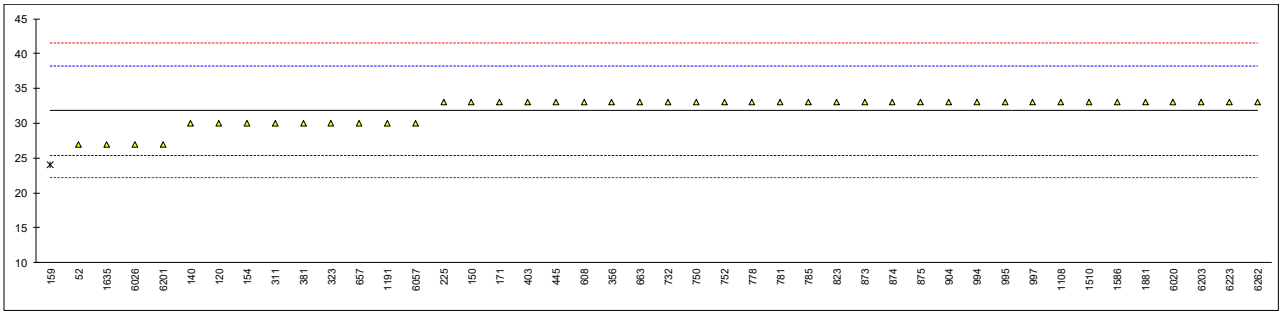


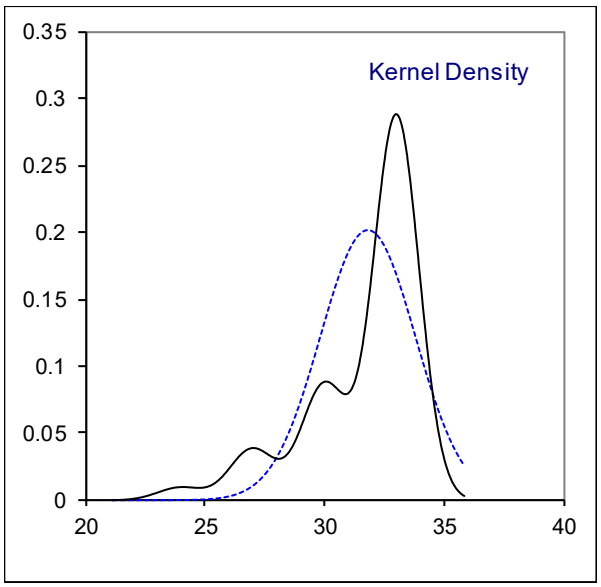


Determination of Pour Point, Manual on sample #19265; results in °C

lab	method	value	mark	z(targ)	remarks
52	D97	27		-1.50	
62		-----		-----	
120	D97	30		-0.56	
140	D97	30		-0.56	
150	D97	33		0.37	
154	D97	30		-0.56	
158		-----		-----	
159	D97	24	R(0.05)	-2.43	
171	D97	33		0.37	
225	D97	33		0.37	
311	D97	30		-0.56	
313		-----		-----	
317		-----		-----	
323	D97	30.0		-0.56	
333		-----		-----	
334		-----		-----	
356	D97	33		0.37	
381	D97	30		-0.56	
403	D97	33		0.37	
445	D97	33		0.37	
463		-----		-----	
608	D97	33		0.37	
657	D97	30		-0.56	
663	D97	33		0.37	
732	D97	33		0.37	
750	D97	33		0.37	
752	INH-20287-A	33		0.37	
778	D97	33		0.37	
781	D97	33		0.37	
785	D97	33		0.37	
798		-----		-----	
823	D97	33		0.37	
873	D97	33		0.37	
874	D97	33		0.37	
875	D97	33		0.37	
904	D97	33		0.37	
994	D97	33		0.37	
995	D97	33		0.37	
997	D97	33		0.37	
1011		-----		-----	
1026		-----		-----	
1065		-----		-----	
1081		-----		-----	
1108	D97	33		0.37	
1134		-----		-----	
1191	ISO3016	30		-0.56	
1201		-----		-----	
1229		-----		-----	
1297		-----		-----	
1510	D97	33		0.37	
1556		-----		-----	
1586	D97	33		0.37	
1635	D97	27		-1.50	
1776		-----		-----	
1852		-----		-----	
1881	D97	33		0.37	
6020	D97	33		0.37	
6026	D97	27		-1.50	
6057	D97	30		-0.56	
6112		-----		-----	
6198		-----		-----	
6201		27		-1.50	
6203	D97	33		0.37	
6223	D97	33		0.37	
6238		-----		-----	
6262	D97	33		0.37	

normality	OK
n	43
outliers	1
mean (n)	31.81
st.dev. (n)	1.979
R(calc.)	5.54
st.dev.(D97:17b)	3.214
R(D97:17b)	9
Compare	
R(ISO3016)	9

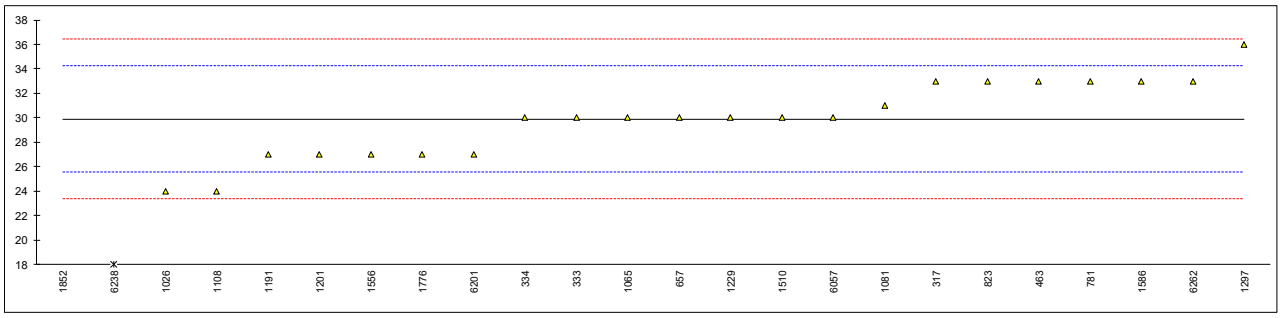


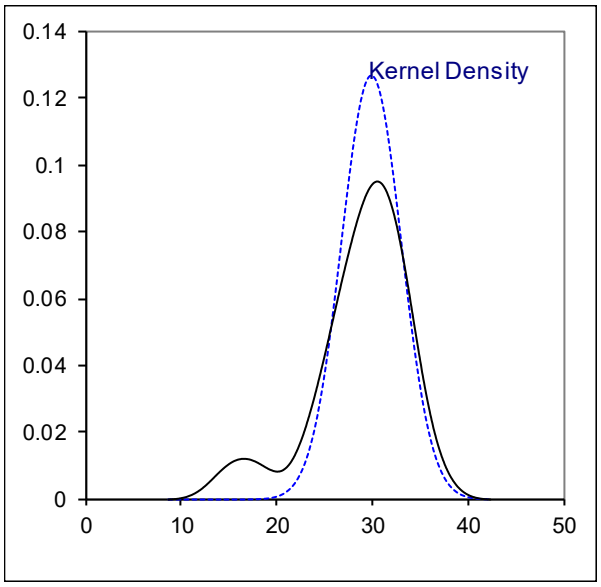


Determination of Pour Point, Automated, 3°C interval on sample #19265; results in °C

lab	method	value	mark	z(targ)	remarks
52		-----		-----	
62		-----		-----	
120		-----		-----	
140		-----		-----	
150		-----		-----	
154		-----		-----	
158		-----		-----	
159		-----		-----	
171		-----		-----	
225		-----		-----	
311		-----		-----	
313		-----		-----	
317	D6749	33		1.42	
323		-----		-----	
333	D5950	30		0.04	
334	D5950	30		0.04	
356		-----		-----	
381		-----		-----	
403		-----		-----	
445		-----		-----	
463	D6892	33		1.42	
608		-----		-----	
657	D5950	30		0.04	
663		-----		-----	
732		-----		-----	
750		-----		-----	
752		-----		-----	
778		-----		-----	
781	D5950	33		1.42	
785		-----		-----	
798		-----		-----	
823	D5950	33		1.42	
873		-----		-----	
874		-----		-----	
875		-----		-----	
904		-----		-----	
994		-----		-----	
995		-----		-----	
997		-----		-----	
1011		-----		-----	
1026	D5950	24		-2.71	
1065	D5950	30		0.04	
1081	D5950	31		0.50	
1108	D5950	24		-2.71	
1134		-----		-----	
1191	D5950	27		-1.34	
1201	D5950	27		-1.34	
1229	D5950	30		0.04	
1297	D5949	36.0		2.80	
1510	D5950	30		0.04	
1556	ISO3016	27		-1.34	
1586	D5950	33		1.42	
1635		-----		-----	
1776	D5950	27		-1.34	
1852	D5950	15	C,R(0.05)	-6.84	first reported 17.5
1881		-----		-----	
6020		-----		-----	
6026		-----		-----	
6057	D5950	30		0.04	
6112		-----		-----	
6198		-----		-----	
6201		27		-1.34	
6203		-----		-----	
6223		-----		-----	
6238		18	R(0.05)	-5.47	
6262	D5950	33		1.42	

normality	OK
n	22
outliers	2
mean (n)	29.909
st.dev. (n)	3.1458
R(calc.)	8.808
st.dev.(D5950:14)	2.1786
R(D5950:14)	6.1

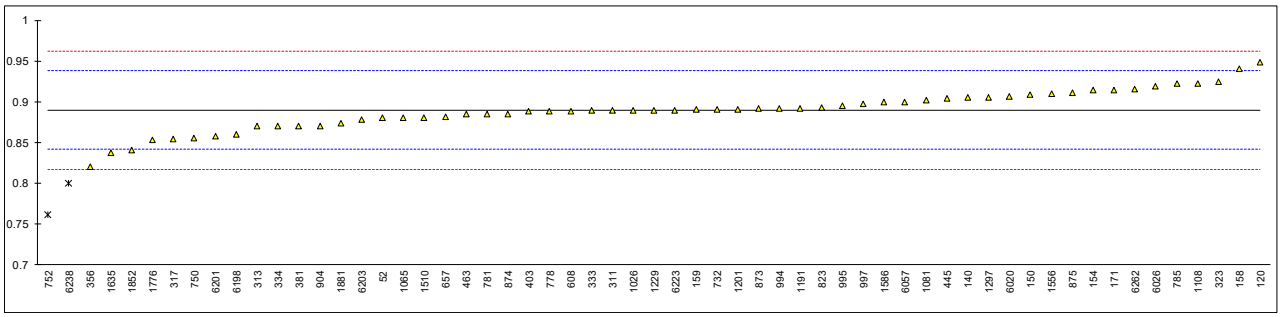


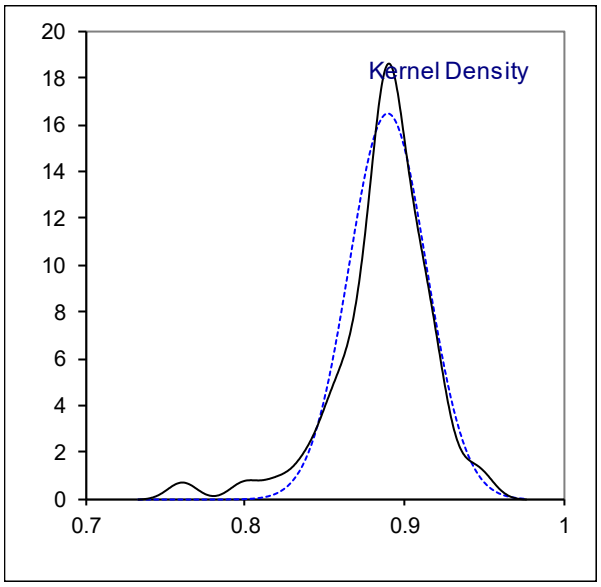


Determination of Sulfur, Total on sample #19265; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	D4294	0.880		-0.40	
62		-----		-----	
120	D4294	0.94889		2.46	
140	D4294	0.905		0.64	
150	D4294	0.909		0.80	
154	D4294	0.914		1.01	
158	D4294	0.94081		2.13	
159	D4294	0.8910		0.06	
171	D4294	0.914		1.01	
225		-----		-----	
311	D4294	0.89		0.01	
313	D4294	0.87		-0.82	
317	D2622	0.854	C	-1.48	first reported 0.807
323	D4294	0.925		1.47	
333	D4294	0.890		0.01	
334	ISO8754	0.87		-0.82	
356	D4294	0.82		-2.89	
381	ISO8754	0.87		-0.82	
403	ISO8754	0.888		-0.07	
445	D4294	0.904		0.60	
463	D4294	0.885		-0.19	
608	D4294	0.8883		-0.06	
657	D4294	0.882		-0.32	
663		-----		-----	
732	D4294	0.891		0.06	
750	D4294	0.856		-1.40	
752	D4294	0.761	R(0.01)	-5.35	
778	D4294	0.888		-0.07	
781	D4294	0.885		-0.19	
785	D4294	0.922		1.34	
798		-----		-----	
823	ISO8754	0.893	C	0.14	first reported 0.632
873	D4294	0.892		0.10	
874	D4294	0.885		-0.19	
875	D4294	0.911		0.89	
904	D4294	0.87		-0.82	
994	D4294	0.892		0.10	
995	D4294	0.895		0.22	
997	D4294	0.897		0.31	
1011		-----		-----	
1026	D2622	0.89		0.01	
1065	D4294	0.88		-0.40	
1081	D4294	0.9017		0.50	
1108	D4294	0.922		1.34	
1134		-----		-----	
1191	ISO8754	0.892		0.10	
1201	D4294	0.891		0.06	
1229	ISO8754	0.89		0.01	
1297	D4294	0.905		0.64	
1510	D4294	0.88		-0.40	
1556	ISO8754	0.910		0.85	
1586	D4294	0.900		0.43	
1635	ISO8754	0.837		-2.19	
1776	ISO8754	0.853		-1.52	
1852	ISO8754	0.841		-2.02	
1881	ISO8754	0.874		-0.65	
6020	D4294	0.9070		0.72	
6026	D4294	0.919		1.22	
6057	D4294	0.90		0.43	
6112		-----		-----	
6198	D2622	0.86		-1.23	
6201		0.858		-1.32	
6203	D2622	0.878		-0.48	
6223	D4294	0.890		0.01	
6238	D4294	0.80	R(0.05)	-3.73	
6262	D4294	0.91576		1.08	

normality	OK
n	57
outliers	2
mean (n)	0.8897
st.dev. (n)	0.02422
R(calc.)	0.0678
st.dev.(D4294:16e1)	0.02407
R(D4294:16e1)	0.0674





Determination of Simulated Distillation on sample #19265; result in °C

lab	method	IBP	10%	30%	50%	70%	90%	FBP
52		-----	-----	-----	-----	-----	-----	-----
62		-----	-----	-----	-----	-----	-----	-----
120		-----	-----	-----	-----	-----	-----	-----
140		-----	-----	-----	-----	-----	-----	-----
150	D6352	292.5	357.0	400.5	428.5	460.0	506.5	589.0
154		-----	-----	-----	-----	-----	-----	-----
158		-----	-----	-----	-----	-----	-----	-----
159		-----	-----	-----	-----	-----	-----	-----
171	D6352	275.2 C	357.5 C	401.2 C	421.7	461.5 C	506.5	606.0 C
225		-----	-----	-----	-----	-----	-----	-----
311	D6352	295.5	359.0	402.0	429.5	461.5	507.0	578.0
313		-----	-----	-----	-----	-----	-----	-----
317	D6352	299.0	359.5	402.5	431.0	461.0	506.5	590.5
323	D6352	268.6	358.3	399.6	425.5	454.0	497.0	556.5
333		-----	-----	-----	-----	-----	-----	-----
334		-----	-----	-----	-----	-----	-----	-----
356		-----	-----	-----	-----	-----	-----	-----
381		-----	-----	-----	-----	-----	-----	-----
403		-----	-----	-----	-----	-----	-----	-----
445	D7169	295.7	361.5	403.8	432.3	464.2	512.0	>720 **f+?**
463		-----	-----	-----	-----	-----	-----	-----
608		-----	-----	-----	-----	-----	-----	-----
657	D6352	299	369	410	437	468	515	595
663		-----	-----	-----	-----	-----	-----	-----
732		-----	-----	-----	-----	-----	-----	-----
750		-----	-----	-----	-----	-----	-----	-----
752		-----	-----	-----	-----	-----	-----	-----
778		-----	-----	-----	-----	-----	-----	-----
781		-----	-----	-----	-----	-----	-----	-----
785		-----	-----	-----	-----	-----	-----	-----
798		-----	-----	-----	-----	-----	-----	-----
823		-----	-----	-----	-----	-----	-----	-----
873		-----	-----	-----	-----	-----	-----	-----
874		-----	-----	-----	-----	-----	-----	-----
875		-----	-----	-----	-----	-----	-----	-----
904		-----	-----	-----	-----	-----	-----	-----
994		-----	-----	-----	-----	-----	-----	-----
995		-----	-----	-----	-----	-----	-----	-----
997		-----	-----	-----	-----	-----	-----	-----
1011		-----	-----	-----	-----	-----	-----	-----
1026		-----	-----	-----	-----	-----	-----	-----
1065		300.0	360.6	403.0	430.8	462.2	507.2	573.8
1081	IP480	303.39	364.23	405.59	433.39	464.69	510.76	583.54
1108		-----	-----	-----	-----	-----	-----	-----
1134		-----	-----	-----	-----	-----	-----	-----
1191		-----	-----	-----	-----	-----	-----	-----
1201	D2887	267.5	357.5	400.5	428.0	459.5	507.5	**629.5**
1229		-----	-----	-----	-----	-----	-----	-----
1297	D7213	298.2	362.8	405.0	433.0	464.0	510.0	580.8
1510		-----	-----	-----	-----	-----	-----	-----
1556	ISO3924	301.4	367.5	409.2	437.8	468.7	514.3	579.4
1586		-----	-----	-----	-----	-----	-----	-----
1635		-----	-----	-----	-----	-----	-----	-----
1776	D6352	296	366	412	442	472	513	558
1852		-----	-----	-----	-----	-----	-----	-----
1881		-----	-----	-----	-----	-----	-----	-----
6020		-----	-----	-----	-----	-----	-----	-----
6026		-----	-----	-----	-----	-----	-----	-----
6057		-----	-----	-----	-----	-----	-----	-----
6112		-----	-----	-----	-----	-----	-----	-----
6198	D7213	300.8	365.7	407.3	435.2	465.3	512.3	588.5
6201		284.5	354.0	397.0	425.0	456.0	503.5	580.0
6203	D6352	294.5	358.5	400.5	428.0	460.5	507.0	581.5
6223		-----	-----	-----	-----	-----	-----	-----
6238		-----	-----	-----	-----	-----	-----	-----
6262	D6352	291.0	359.0	400.5	428.0	458.0	502.5	567.5

	IBP	10%	30%	50%	70%	90%	FBP
normality	OK	OK	OK	OK	OK	OK	OK
n	17	17	17	17	17	17	15
outliers	0	0	0	0	0	0	1
mean (n)	291.93	361.04	403.54	430.98	462.42	508.15	580.54
st.dev. (n)	11.268	4.188	4.111	5.127	4.575	4.614	13.123
R(calc.)	31.55	11.73	11.51	14.36	12.81	12.92	36.74
st.dev.(D6352:15)	17.536	2.536	2.107	2.286	2.571	3.750	13.607
R(D6352:15)	49.1	7.1	5.9	6.4	7.2	10.5	38.1

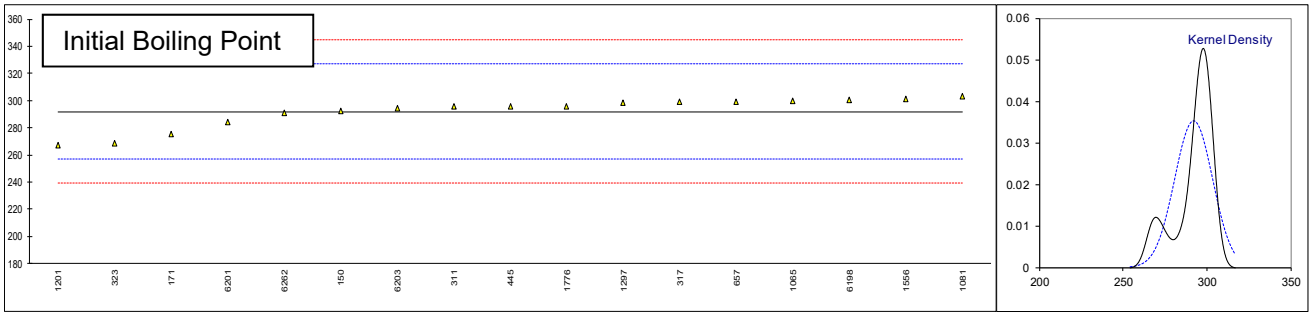
The reported result underlined and bold is a statistical outlier

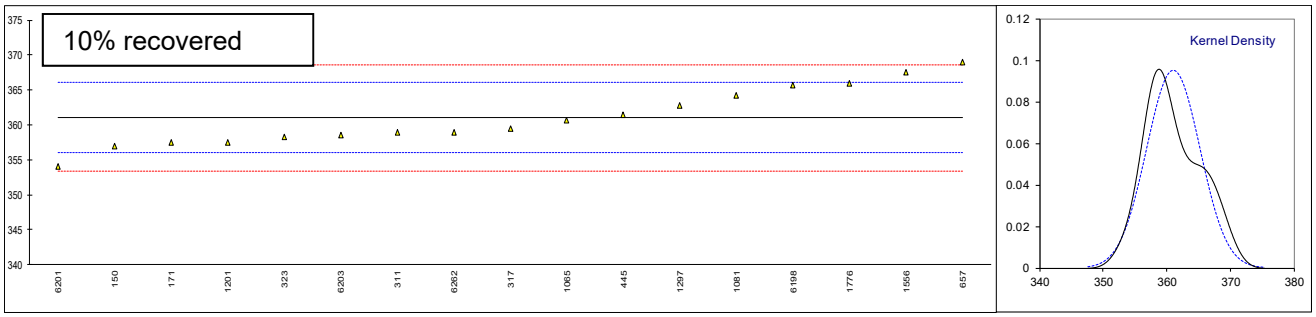
Lab 171 first reported: 117, 168.3, 387.4, 440.9 and 645.7 respectively

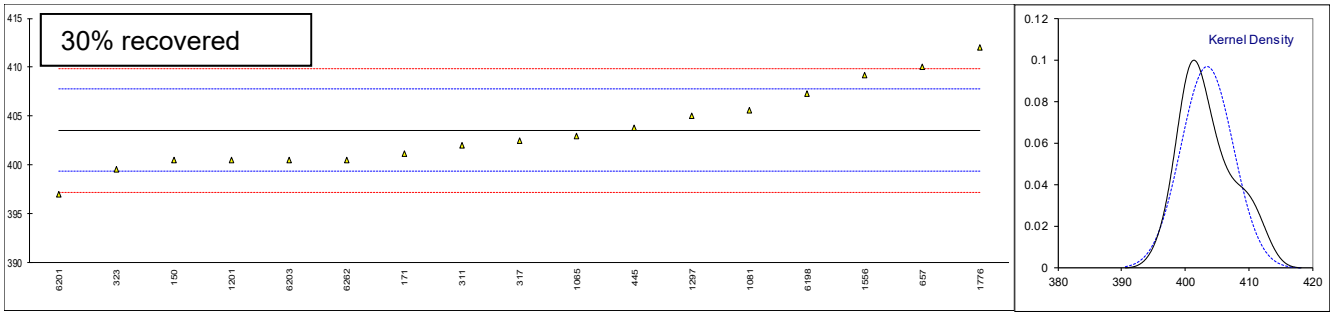
f+? = reported possibly a false positive test result (lab 445)

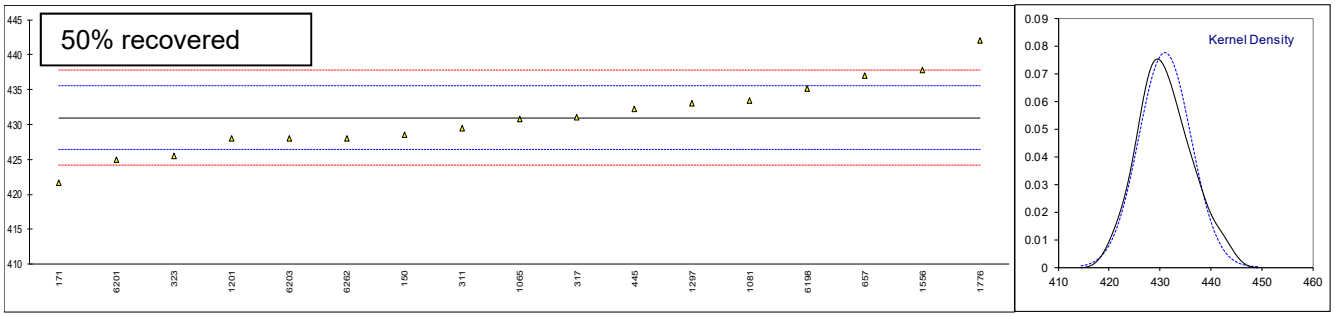
z-scores

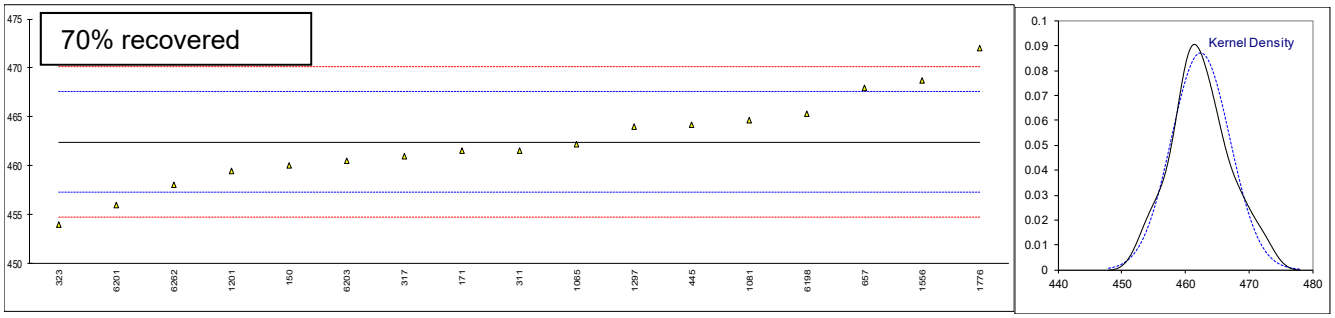
lab	IBP	10%	30%	50%	70%	90%	FBP
150	0.03	-1.59	-1.44	-1.09	-0.94	-0.44	0.62
171	-0.95	-1.39	-1.11	-4.06	-0.36	-0.44	1.87
311	0.20	-0.80	-0.73	-0.65	-0.36	-0.31	-0.19
317	0.40	-0.61	-0.49	0.01	-0.55	-0.44	0.73
323	-1.33	-1.08	-1.87	-2.40	-3.27	-2.97	-1.77
445	0.22	0.18	0.12	0.58	0.69	1.03	---
657	0.40	3.14	3.07	2.63	2.17	1.83	1.06
1065	0.46	-0.17	-0.26	-0.08	-0.08	-0.25	-0.50
1081	0.65	1.26	0.97	1.05	0.88	0.70	0.22
1201	-1.39	-1.39	-1.44	-1.30	-1.13	-0.17	3.60
1297	0.36	0.70	0.69	0.88	0.62	0.49	0.02
1556	0.54	2.55	2.69	2.98	2.44	1.64	-0.08
1776	0.23	1.96	4.01	4.82	3.73	1.29	-1.66
6198	0.51	1.84	1.78	1.85	1.12	1.11	0.59
6201	-0.42	-2.78	-3.10	-2.62	-2.50	-1.24	-0.04
6203	0.15	-1.00	-1.44	-1.30	-0.75	-0.31	0.07
6262	-0.05	-0.80	-1.44	-1.30	-1.72	-1.51	-0.96

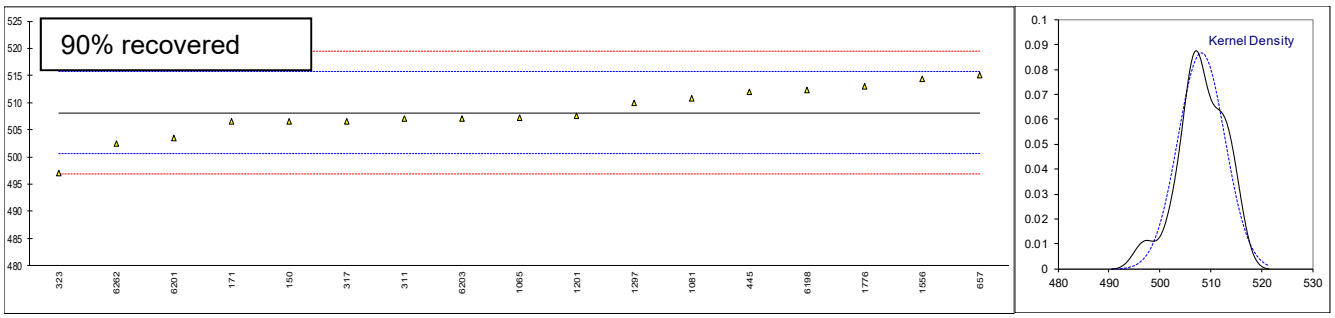


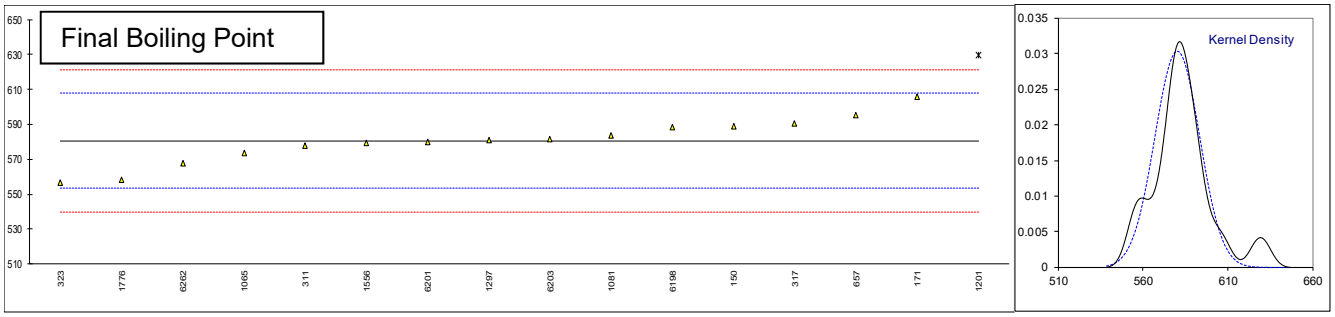



30% recovered
Kernel Density


50% recovered
Kernel Density


70% recovered
Kernel Density


90% recovered
Kernel Density


Final Boiling Point
Kernel Density

Determination of Distillation at 10 mmHg as AET on sample #19265; result in °C

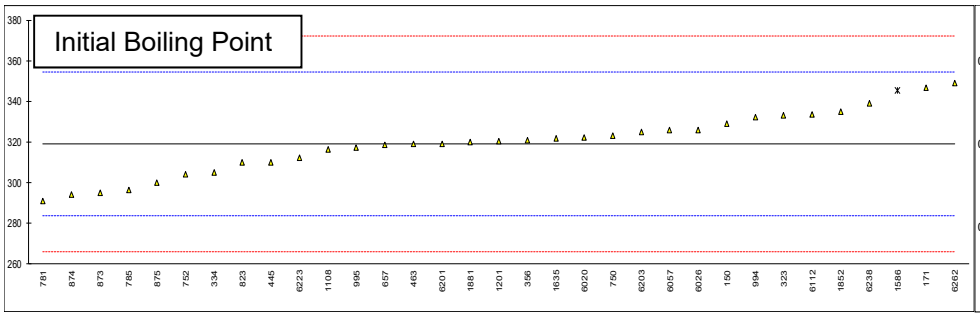
lab	method	IBP	10%	30%	50%	70%	90%	FBP
52		-----	-----	-----	-----	-----	-----	-----
62		-----	-----	-----	-----	-----	-----	-----
120		-----	-----	-----	-----	-----	-----	-----
140		-----	-----	-----	-----	-----	-----	-----
150	D1160	329	384	410	434	462	507	520
154		-----	-----	-----	-----	-----	-----	-----
158		-----	-----	-----	-----	-----	-----	-----
159		-----	-----	-----	-----	-----	-----	-----
171	D1160	346.8 C	389.5 C	422 C	**449.1** C	473.8 C	514.3 C	518.7 C
225		-----	-----	-----	-----	-----	-----	-----
311		-----	-----	-----	-----	-----	-----	-----
313		-----	-----	-----	-----	-----	-----	-----
317		-----	-----	-----	-----	-----	-----	-----
323	D1160	333	378	412	435	463	502	550
333		-----	-----	-----	-----	-----	-----	-----
334	D1160	305	386	415	438	464	507	531
356	D1160	321	384	413	434	466	509	537
381		-----	-----	-----	-----	-----	-----	-----
403		-----	-----	-----	-----	-----	-----	-----
445	D1160	310.1	375.3	409.1	433.2	462.1	506.7	547.6
463	D1160	319.0	379.0	409.0	430.0	456.0	496.0	535.0
608		-----	-----	-----	-----	-----	-----	-----
657	D1160	318.5	381.3	412.6	436.1	462.5 C	506.8	535.3
663		-----	-----	-----	-----	-----	-----	-----
732		-----	-----	-----	-----	-----	-----	-----
750	D1160	323	378	411	434	463	504	540
752	D1160	304	378	412	435	463	508	546
778		-----	-----	-----	-----	-----	-----	-----
781	D1160	291	375	410	434	467	511	539
785	D1160	296.5	375.6	411.3	434.7	462.8	510.0	526.4
798		-----	-----	-----	-----	-----	-----	-----
823	D1160	310	378	410	434	462	505	528
873	D1160	295	377	411	434	465	510	532
874	D1160	294	378	409	433	465	509	528
875	D1160	300	371	409	434	463	510	532
904		-----	-----	-----	-----	-----	-----	-----
994	D1160	332.0	385.0 C	419.0 C	445.0 C	470.0 C	529.0	558.0
995	D1160	317.0	390.5	421.0	441.5 C	**482.5**	521.0	548.0
997		-----	-----	-----	-----	-----	-----	-----
1011		-----	-----	-----	-----	-----	-----	-----
1026		-----	-----	-----	-----	-----	-----	-----
1065		-----	-----	-----	-----	-----	-----	-----
1081		-----	-----	-----	-----	-----	-----	-----
1108	D1160	316.1	374.8	407.1	430.8	456.9	500.4	521.8
1134		-----	-----	-----	-----	-----	-----	-----
1191		-----	-----	-----	-----	-----	-----	-----
1201	D1160	320.4	382.7	415.3	439.5	468.6	517.3	551.5
1229		-----	-----	-----	-----	-----	-----	-----
1297		-----	-----	-----	-----	-----	-----	-----
1510		-----	-----	-----	-----	-----	-----	-----
1556		-----	-----	-----	-----	-----	-----	-----
1586	D1160	345.1 ex	**396.5**	**428.2**	**452.5**	475.9 ex	516.5 ex	534.5 ex
1635	D1160	321.9	372.9	405.5	428.7	454.9	**480.9**	**482.1**
1776		-----	-----	-----	-----	-----	-----	-----
1852	D1160	335.0	385.6	413.5	439.6	464.1	510.2	553.0
1881	D1160	320	375	405	427	451	500	550
6020	D1160	322	382	415	440	468	515	547
6026	D1160	326	383	415	438	467	512	546
6057	D1160	325.6	378.2	410.8	435.3	465.2	504.8	528.1
6112		333.7	382.7	414.4	436.8	464.0	508.0	556.2
6198		-----	-----	-----	-----	-----	-----	-----
6201		319.1	379.7	410.3	433.7	459.7	503.0	544.8
6203	D1160	325.1	374.2	404.9	427.5	455.2	498.2	522.3
6223	D1160	312	374	409	436	453	494	538
6238	D1160	339	378	408	431	458	498	536
6262	D1160	349.0	379.1	410.8	433.4	461.3	506.2	551.3

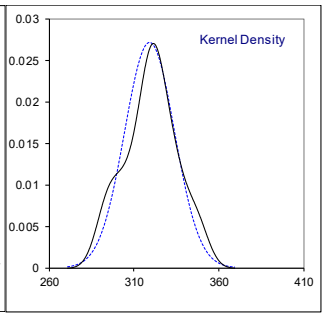
	IBP	10%	30%	50%	70%	90%	FBP
normality	OK	OK	OK	OK	OK	suspect	OK
n	32	32	32	31	31	31	31
outliers	0+1ex	1	1	2	1+1ex	1+1ex	1+1ex
mean (n)	319.06	379.53	411.58	434.74	462.49	507.51	538.65
st.dev. (n)	14.674	4.768	4.088	3.946	5.064	7.267	11.289
R(calc.)	41.09	13.35	11.45	11.05	14.18	20.35	31.61
st.dev.(D1160:18)	17.660	5.269	3.792	3.680	3.284	7.914	9.605
R(D1160:18)	49.45	14.75	10.62	10.30	9.20	22.16	26.89

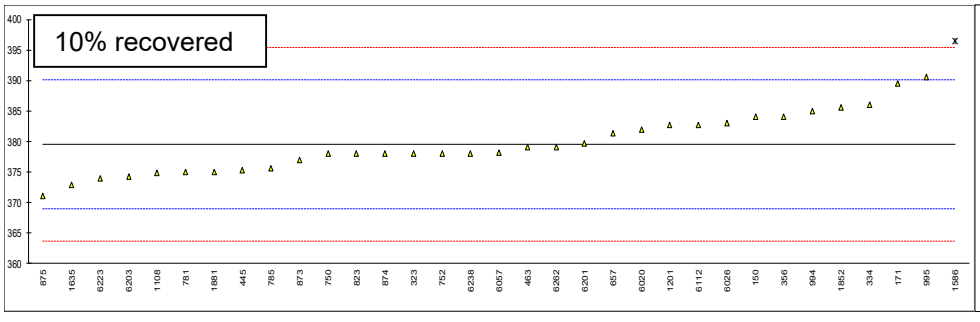
The reported results underlined and bold are statistical outliers
Lab 171 first reported: 656, 733, 792, 840, 885, 958 and 966 respectively
Lab 657 first reported: 162.5
Lab 994 first reported: 405, 436, 462 and 490 respectively
Lab 995 first reported: 458
Lab 1586 test results are excluded because the other related test results were statistical outliers

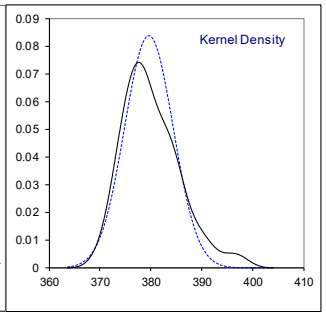
z-scores

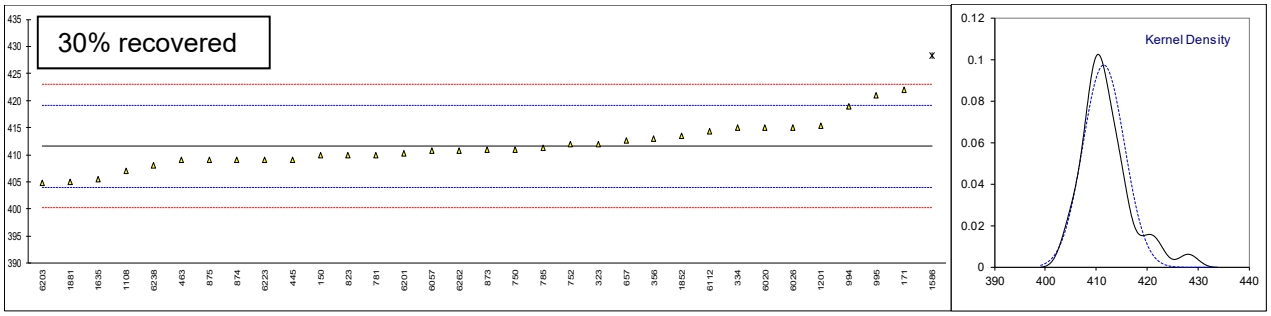
lab	IBP	10%	30%	50%	70%	90%	FBP
150	0.56	0.85	-0.42	-0.20	-0.15	-0.06	-1.94
171	1.57	1.89	2.75	3.90	3.44	0.86	-2.08
323	0.79	-0.29	0.11	0.07	0.16	-0.70	1.18
334	-0.80	1.23	0.90	0.89	0.46	-0.06	-0.80
356	0.11	0.85	0.37	-0.20	1.07	0.19	-0.17
445	-0.51	-0.80	-0.65	-0.42	-0.12	-0.10	0.93
463	0.00	-0.10	-0.68	-1.29	-1.98	-1.45	-0.38
657	-0.03	0.34	0.27	0.37	0.00	-0.09	-0.35
750	0.22	-0.29	-0.15	-0.20	0.16	-0.44	0.14
752	-0.85	-0.29	0.11	0.07	0.16	0.06	0.77
781	-1.59	-0.86	-0.42	-0.20	1.37	0.44	0.04
785	-1.28	-0.75	-0.07	-0.01	0.10	0.31	-1.27
823	-0.51	-0.29	-0.42	-0.20	-0.15	-0.32	-1.11
873	-1.36	-0.48	-0.15	-0.20	0.77	0.31	-0.69
874	-1.42	-0.29	-0.68	-0.47	0.77	0.19	-1.11
875	-1.08	-1.62	-0.68	-0.20	0.16	0.31	-0.69
994	0.73	1.04	1.96	2.79	2.29	2.72	2.02
995	-0.12	2.08	2.48	1.84	6.09	1.70	0.97
1108	-0.17	-0.90	-1.18	-1.07	-1.70	-0.90	-1.75
1201	0.08	0.60	0.98	1.29	1.86	1.24	1.34
1586	1.47	3.22	4.38	4.83	4.08	1.14	-0.43
1635	0.16	-1.26	-1.60	-1.64	-2.31	-3.36	-5.89
1852	0.90	1.15	0.51	1.32	0.49	0.34	1.49
1881	0.05	-0.86	-1.74	-2.10	-3.50	-0.95	1.18
6020	0.17	0.47	0.90	1.43	1.68	0.95	0.87
6026	0.39	0.66	0.90	0.89	1.37	0.57	0.77
6057	0.37	-0.25	-0.21	0.15	0.83	-0.34	-1.10
6112	0.83	0.60	0.74	0.56	0.46	0.06	1.83
6201	0.00	0.03	-0.34	-0.28	-0.85	-0.57	0.64
6203	0.34	-1.01	-1.76	-1.97	-2.22	-1.18	-1.70
6223	-0.40	-1.05	-0.68	0.34	-2.89	-1.71	-0.07
6238	1.13	-0.29	-0.94	-1.02	-1.37	-1.20	-0.28
6262	1.70	-0.08	-0.21	-0.36	-0.36	-0.17	1.32

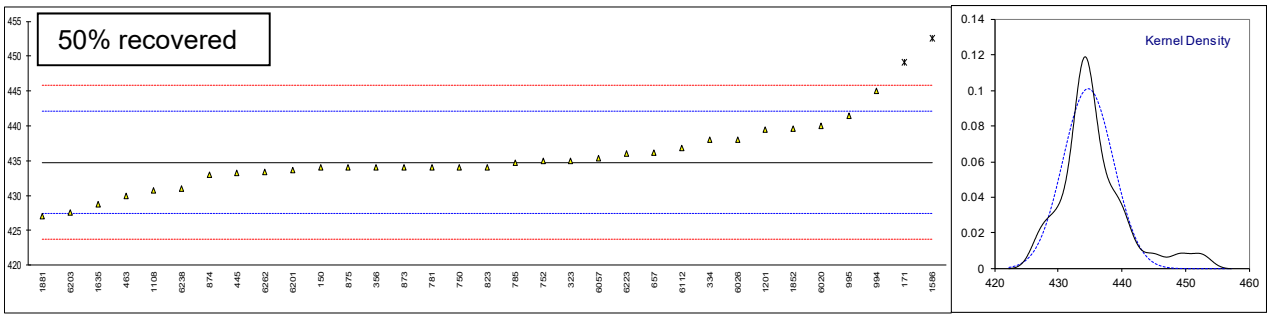


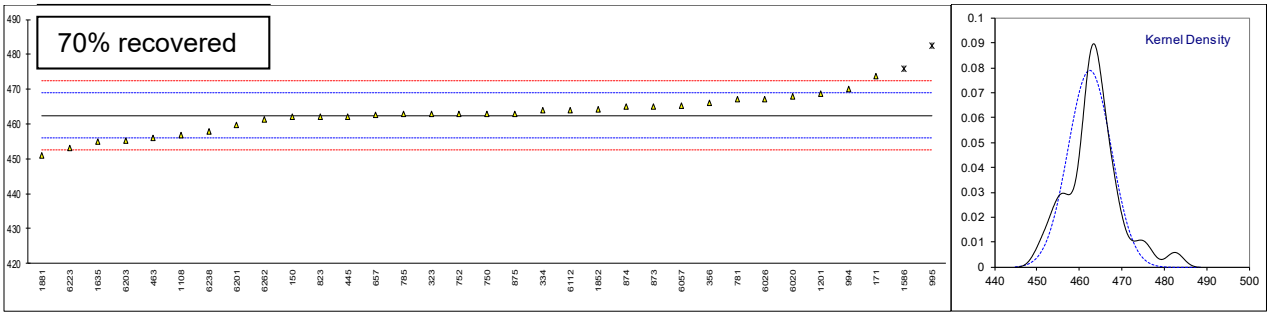


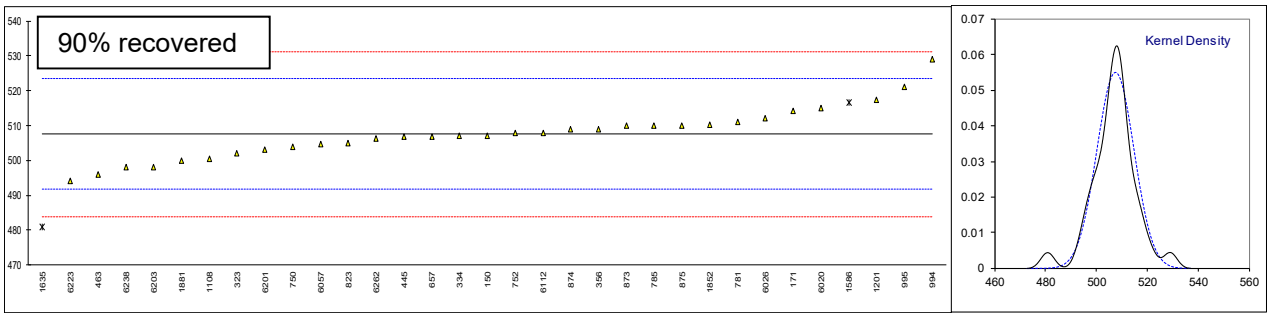


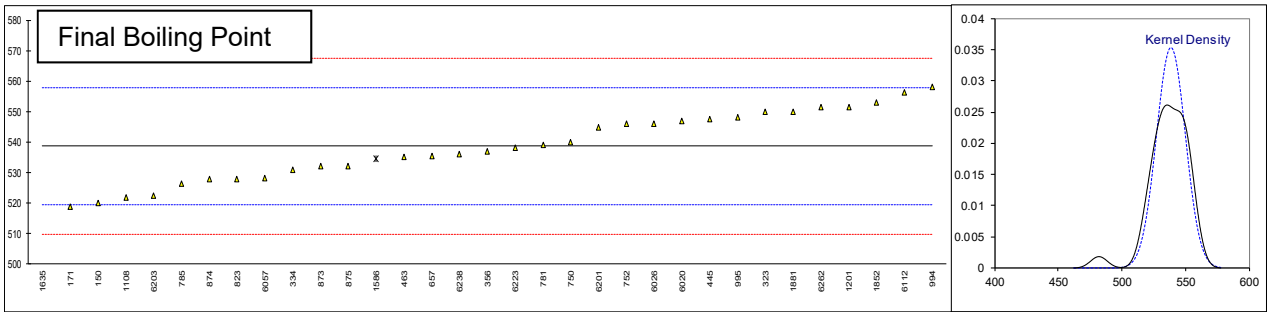



30% recovered
Kernel Density


50% recovered
Kernel Density


70% recovered
Kernel Density


90% recovered
Kernel Density

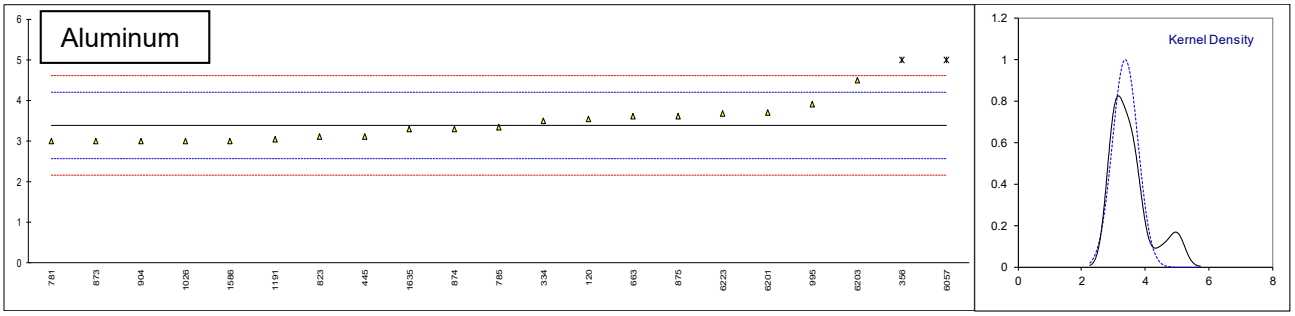

Final Boiling Point
Kernel Density

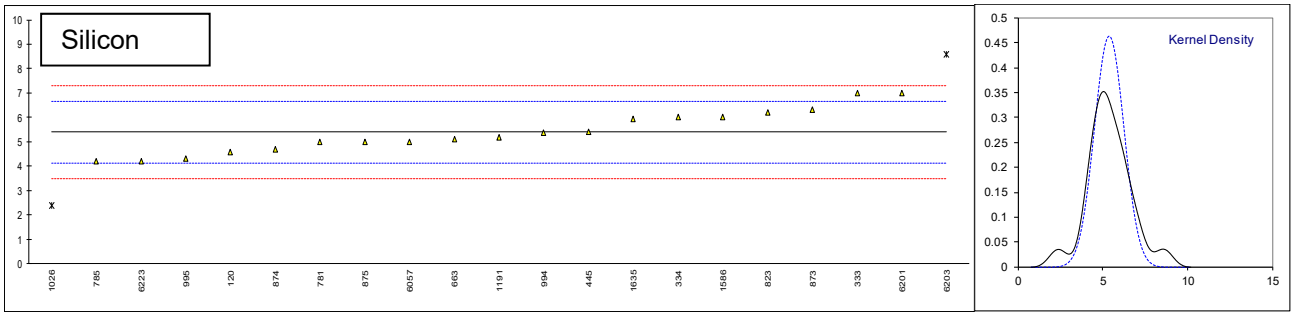
Determination of Aluminum as Al, Silicon as Si and total Al+Si on sample #19266; results in mg/kg

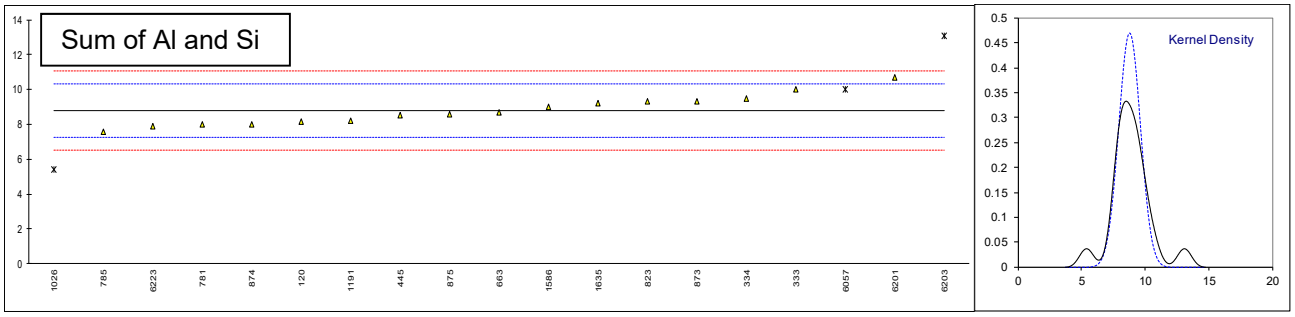
lab	method	Al	mark	z(targ)	Si	mark	z(targ)	Sum Al+Si	mark	z(targ)
52	IP501	<5		-----	<10		-----	<15		-----
62		-----		-----	-----		-----	-----		-----
120	IP501	3.550		0.42	4.591		-1.26	8.141		-0.86
140		-----		-----	-----		-----	-----		-----
150	IP501	<5		-----	<10		-----	-----		-----
154	IP501	<5		-----	<10		-----	<15		-----
158		-----		-----	-----		-----	-----		-----
159	IP501	<5		-----	<10		-----	<15		-----
171		-----		-----	-----		-----	-----		-----
225		-----		-----	-----		-----	-----		-----
311		-----		-----	-----		-----	-----		-----
313		-----		-----	-----		-----	-----		-----
317	IP501	<5		-----	<10		-----	<10		-----
323	IP501	<5		-----	<10		-----	<15		-----
333	IP501	<5		-----	7	C	2.51	10	C	1.60
334	IP501	3.5		0.30	6.0		0.95	9.5		0.94
356	IP501	5	R(0.05)	3.99	<10		-----	<15		-----
381		-----		-----	-----		-----	-----		-----
403		-----		-----	-----		-----	-----		-----
445	IP501	3.1		-0.69	5.4		0.01	8.5		-0.38
463		-----		-----	-----		-----	-----		-----
608		-----		-----	-----		-----	-----		-----
657	IP501	<5		-----	<10		-----	<15		-----
663	IP501	3.6		0.54	5.1		-0.46	8.7		-0.12
732		-----		-----	-----		-----	-----		-----
750		-----		-----	-----		-----	-----		-----
752		-----		-----	-----		-----	-----		-----
778		-----		-----	-----		-----	-----		-----
781	IP501	3		-0.93	5		-0.62	8		-1.05
785	IP470	3.34		-0.10	4.21		-1.85	7.55		-1.64
798		-----		-----	-----		-----	-----		-----
823	IP501	3.1		-0.69	6.2		1.26	9.3		0.68
873	IP470	3.0		-0.93	6.3		1.41	9.3		0.68
874	IP501	3.3		-0.19	4.7		-1.09	8.0		-1.05
875	IP501	3.6		0.54	5.0		-0.62	8.6		-0.25
904	IP470	3		-0.93	<5		-----	<10		-----
994	IP501	<5		-----	5.38		-0.02	-----		-----
995	IP470	3.9	C	1.28	4.3		-1.71	-----		-----
997		-----		-----	-----		-----	-----		-----
1011		-----		-----	-----		-----	-----		-----
1026	IP501	3.0		-0.93	2.4	R(0.01)	-4.68	5.4	D(0.05)	-4.49
1065		-----		-----	-----		-----	-----		-----
1081		-----		-----	-----		-----	-----		-----
1108		-----		-----	-----		-----	-----		-----
1134		-----		-----	-----		-----	-----		-----
1191	IP501	3.04		-0.83	5.19		-0.32	8.23		-0.74
1201		-----		-----	-----		-----	-----		-----
1229		-----		-----	-----		-----	-----		-----
1297		-----		-----	-----		-----	-----		-----
1510		-----		-----	-----		-----	-----		-----
1556		-----		-----	-----		-----	-----		-----
1586	IP470	3		-0.93	6		0.95	9		0.28
1635	IP501	3.29		-0.22	5.93		0.84	9.22		0.57
1776		-----		-----	-----		-----	-----		-----
1852		-----		-----	-----		-----	-----		-----
1881		-----		-----	-----		-----	-----		-----
6020		-----		-----	-----		-----	-----		-----
6026		-----		-----	-----		-----	-----		-----
6057	IP501	5	R(0.05)	3.99	5		-0.62	10	ex	1.60
6112		-----		-----	-----		-----	-----		-----
6198		-----		-----	-----		-----	-----		-----
6201	IP501	3.7		0.79	7.0		2.51	10.7		2.53
6203	IP470	4.5		2.76	8.6	R(0.01)	5.01	13.1	D(0.01)	5.71
6223	IP470	3.68		0.74	4.21		-1.85	7.89		-1.19
6238		-----		-----	-----		-----	-----		-----
6262		-----		-----	-----		-----	-----		-----

	Al	Si	Sum Al+Si
normality	not OK	OK	OK
n	19	19	16
outliers	2	2	2+1ex
mean (n)	3.38	5.40	8.79
st.dev. (n)	0.399	0.862	0.849
R(calc.)	1.12	2.41	2.38
st.dev.(IP501:05)	0.407	0.640	0.755
R(IP501:05)	1.14	1.79	2.11
Compare			
R(IP470:05)	3.14	4.29	5.30

Lab 333 first reported: 20 and 23 respectively
Lab 995 first reported: 5.9
Lab 6057 test result is excluded because of a statistical outlier in the Al determination





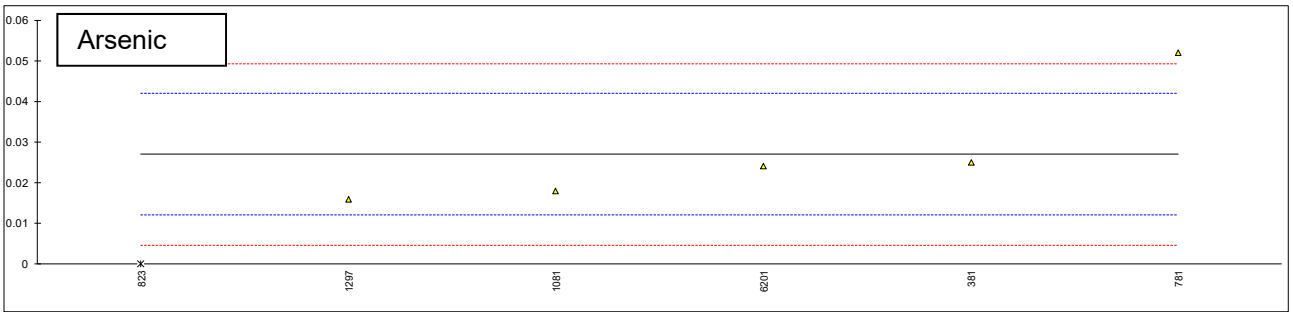


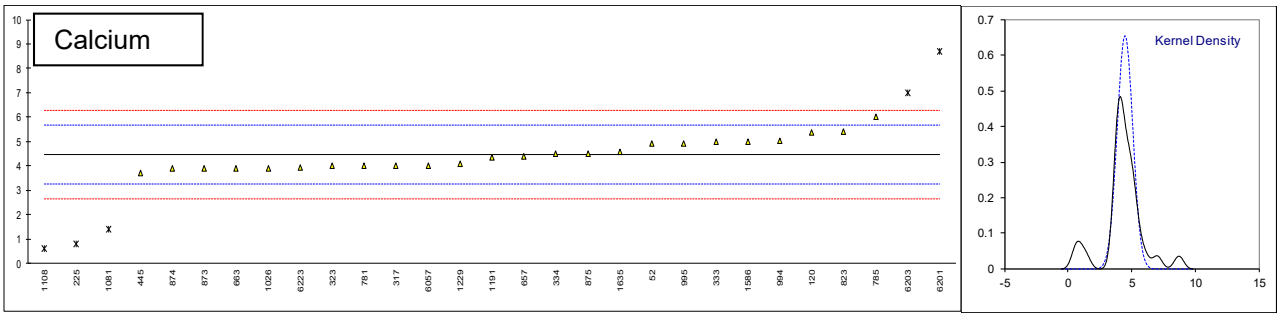
Determination of Arsenic as As, Calcium as Ca and Phosphorus as P on sample #19266; results in mg/kg

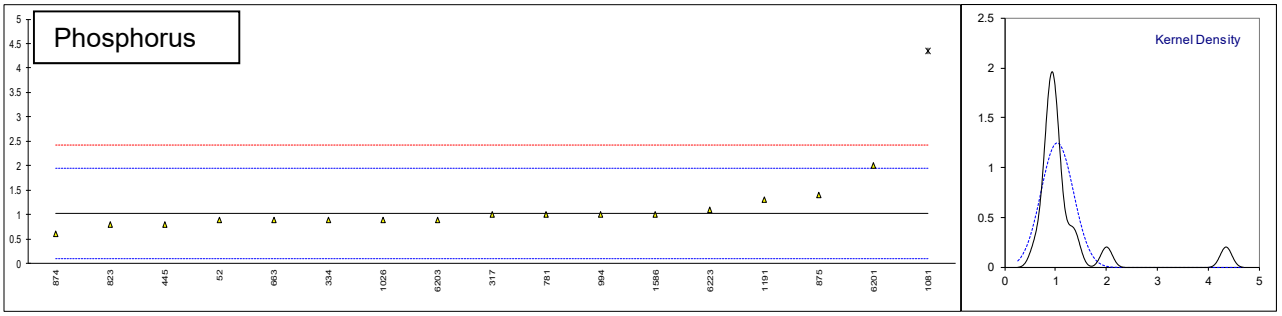
lab	method	As	mark	z(targ)	Ca	mark	z(targ)	P	mark	z(targ)
52	IP501	-----		-----	4.9		0.71	0.9		-0.28
62		-----		-----	-----		-----	-----		-----
120	IP501	-----		-----	5.380		1.50	-----		-----
140		-----		-----	-----		-----	-----		-----
150	IP501	-----		-----	<3		-----	<1		-----
154	IP501	-----		-----	<3		-----	<1		-----
158		-----		-----	-----		-----	-----		-----
159	IP501	-----		-----	<3		-----	<1		-----
171		-----		-----	-----		-----	-----		-----
225	IP501	-----		-----	0.81	R(0.05)	-6.01	-----		-----
311		-----		-----	-----		-----	-----		-----
313		-----		-----	-----		-----	-----		-----
317	IP501	-----		-----	4		-0.77	1		-0.07
323	IP501	-----		-----	4		-0.77	<1		-----
333	IP501	-----		-----	5		0.87	<1		-----
334	IP501	-----		-----	4.5		0.05	0.9		-0.28
356	IP501	-----		-----	<3		-----	<1		-----
381		0.025		-0.27	-----		-----	-----		-----
403		-----		-----	-----		-----	-----		-----
445	IP501	< 1		-----	3.7		-1.26	0.80		-0.50
463		-----		-----	-----		-----	-----		-----
608		-----		-----	-----		-----	-----		-----
657	IP501	-----		-----	4.4		-0.11	<1		-----
663	IP501	-----		-----	3.9		-0.94	0.9		-0.28
732		-----		-----	-----		-----	-----		-----
750		-----		-----	-----		-----	-----		-----
752		-----		-----	-----		-----	-----		-----
778		-----		-----	-----		-----	-----		-----
781	IP501	0.052		3.36	4		-0.77	1		-0.07
785	IP470	-----		-----	6.01		2.53	-----		-----
798		-----		-----	-----		-----	-----		-----
823	IP501	0	ex	-3.63	5.4		1.53	0.8		-0.50
873	IP470	-----		-----	3.9		-0.94	-----		-----
874	IP501	-----		-----	3.9		-0.94	0.6		-0.93
875	IP501	-----		-----	4.5		0.05	1.4		0.80
904	IP470	-----		-----	<1		<-5.70	-----		-----
994	IP501	-----		-----	5.02	C	0.90	1.0		-0.07
995	IP470	-----		-----	4.9		0.71	-----		-----
997		-----		-----	-----		-----	-----		-----
1011		-----		-----	-----		-----	-----		-----
1026	IP501	-----		-----	3.9		-0.94	0.9		-0.28
1065		-----		-----	-----		-----	-----		-----
1081	In house	0.018		-1.21	1.395	R(0.05)	-5.05	4.348	D(0.01)	7.15
1108	D5708	-----		-----	0.6	R(0.05)	-6.36	-----		-----
1134		-----		-----	-----		-----	-----		-----
1191	IP501	-----		-----	4.36		-0.18	1.3		0.58
1201		-----		-----	-----		-----	-----		-----
1229	In house	-----		-----	4.1		-0.61	-----		-----
1297		0.016		-1.48	-----		-----	-----		-----
1510		-----		-----	-----		-----	-----		-----
1556		-----		-----	-----		-----	-----		-----
1586	IP470	-----		-----	5		0.87	1		-0.07
1635	IP501	-----		-----	4.57		0.17	<1		-----
1776		-----		-----	-----		-----	-----		-----
1852		-----		-----	-----		-----	<10		-----
1881		-----		-----	-----		-----	-----		-----
6020		-----		-----	-----		-----	-----		-----
6026		-----		-----	-----		-----	-----		-----
6057	IP501	<1		-----	4		-0.77	<1		-----
6112		-----		-----	-----		-----	-----		-----
6198		-----		-----	-----		-----	-----		-----
6201	IP501	0.024		-0.40	8.7	R(0.05)	6.95	2.0		2.09
6203	IP470	-----		-----	7.0	R(0.05)	4.16	0.9		-0.28
6223	IP470	-----		-----	3.92		-0.90	1.1		0.15
6238		-----		-----	-----		-----	-----		-----
6262		-----		-----	-----		-----	-----		-----

	As	Ca	P
normality	unknown	OK	not OK
n	5	24	16
outliers	0+1ex	5	1
mean (n)	0.03	4.47	1.03
st.dev. (n)	0.014	0.609	0.320
R(calc.)	0.04	1.71	0.90
st.dev.(IP501:05)	0.007 (Horwitz)	0.609	0.464
R(IP501:05)	0.02 (Horwitz)	1.70	1.30
Compare			
R(IP470:05)		4.06	

Lab 823 test result excluded as zero is not a real test result
Lab 994 first reported 8.35





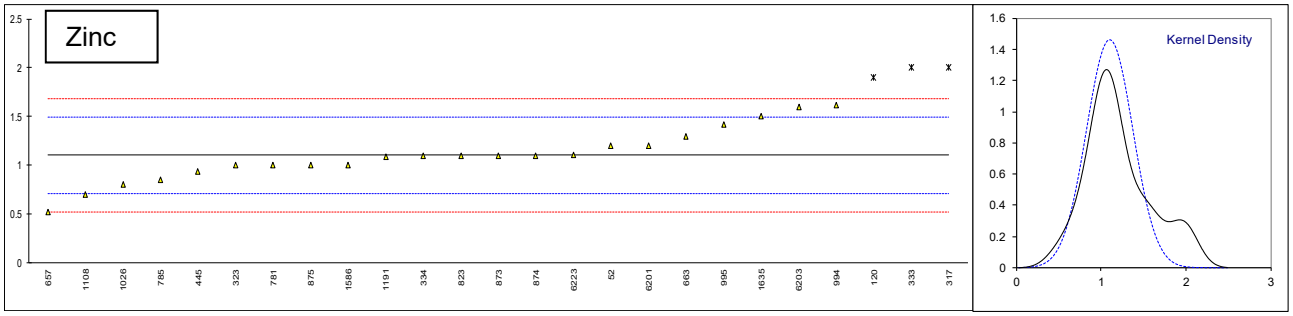


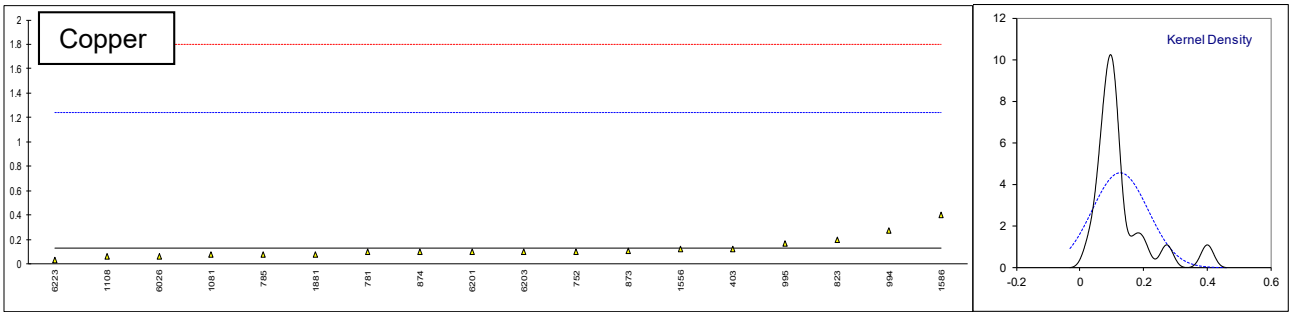
Determination of Zinc as Zn, Copper as Cu and Iron as Fe on sample #19266; results in mg/kg

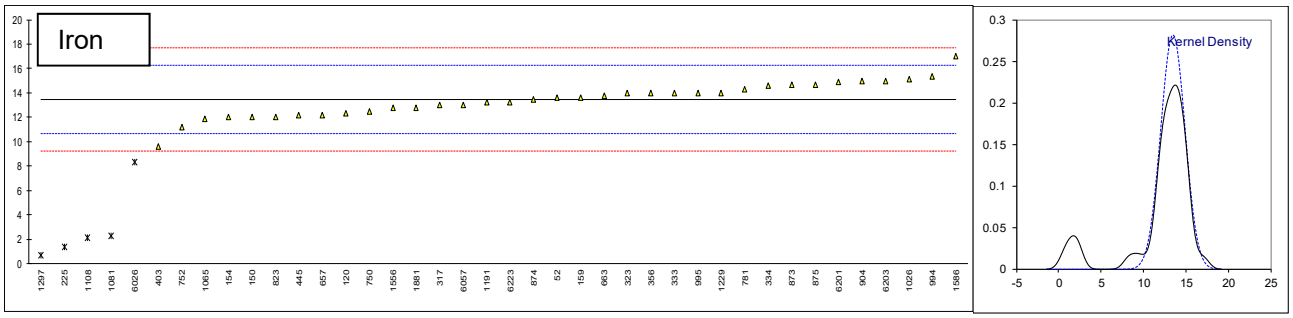
lab	method	Zn	mark	z(targ)	Cu	mark	z(targ)	Fe	mark	z(targ)
52	IP501	1.2		0.50	-----		-----	13.6		0.10
62		-----		-----	-----		-----	-----		-----
120	IP501	1.904	R(0.01)	4.13	-----		-----	12.36		-0.79
140		-----		-----	-----		-----	-----		-----
150	IP501	<1		-----	-----		-----	12		-1.04
154	IP501	<1		-----	<1		-----	12	C	-1.04
158		-----		-----	-----		-----	-----		-----
159	IP501	<1		-----	-----		-----	13.6		0.10
171		-----		-----	-----		-----	-----		-----
225	IP501	-----		-----	-----		-----	1.35	R(0.01)	-8.66
311		-----		-----	-----		-----	-----		-----
313		-----		-----	-----		-----	-----		-----
317	IP501	2	R(0.01)	4.62	-----		-----	13		-0.33
323	IP501	1		-0.53	<1		-----	14		0.38
333	IP501	2	R(0.01)	4.62	-----		-----	14		0.38
334	IP501	1.1		-0.01	-----		-----	14.6		0.81
356	IP501	<1		-----	-----		-----	14		0.38
381		-----		-----	-----		-----	-----		-----
403		-----		-----	0.12		-0.01	9.60		-2.76
445	IP501	0.94		-0.84	< 1		-----	12.2		-0.90
463		-----		-----	-----		-----	-----		-----
608		-----		-----	-----		-----	-----		-----
657	IP501	0.52		-3.00	<1		-----	12.2		-0.90
663	IP501	1.3		1.02	-----		-----	13.8		0.24
732		-----		-----	-----		-----	-----		-----
750		-----		-----	<0.1		-----	12.5		-0.69
752		-----		-----	0.102		-0.04	11.181		-1.63
778		-----		-----	-----		-----	-----		-----
781	IP501	1		-0.53	0.1		-0.05	14.3		0.60
785	IP470	0.85		-1.30	0.08		-0.08	-----		-----
798		-----		-----	-----		-----	-----		-----
823	IP501	1.1		-0.01	0.2		0.13	12.0		-1.04
873	IP470	1.1		-0.01	0.11		-0.03	14.7		0.89
874	IP501	1.1		-0.01	0.1		-0.05	13.5		0.03
875	IP501	1.0		-0.53	-----		-----	14.7		0.89
904	IP470	<2		-----	<1		-----	15		1.10
994	IP501	1.62		2.66	0.273		0.26	15.39		1.38
995	IP470	1.42		1.64	0.17		0.08	14		0.38
997		-----		-----	-----		-----	-----		-----
1011		-----		-----	-----		-----	-----		-----
1026	IP501	0.8		-1.56	-----		-----	15.1		1.17
1065		-----		-----	-----		-----	11.9		-1.12
1081	In house	-----		-----	0.077		-0.09	2.253	R(0.01)	-8.01
1108	D5708	0.7		-2.07	0.06		-0.12	2.1	R(0.01)	-8.12
1134		-----		-----	-----		-----	-----		-----
1191	IP501	1.09		-0.06	-----		-----	13.21		-0.18
1201		-----		-----	-----		-----	-----		-----
1229	In house	-----		-----	-----		-----	14		0.38
1297		-----		-----	-----		-----	0.728	R(0.01)	-9.10
1510		-----		-----	-----		-----	-----		-----
1556		-----		-----	0.119		-0.01	12.775		-0.49
1586	IP470	1		-0.53	0.4		0.49	17		2.53
1635	IP501	1.50		2.05	-----		-----	<0.2	C	<-9.48
1776		-----		-----	-----		-----	-----		-----
1852		-----		-----	-----		-----	-----		-----
1881		-----		-----	0.08		-0.08	12.8		-0.47
6020		-----		-----	-----		-----	-----		-----
6026		-----		-----	0.0657		-0.11	8.346	C,R(0.05)	-3.66
6057	IP501	<1		-----	<1		-----	13		-0.33
6112		-----		-----	-----		-----	-----		-----
6198		-----		-----	-----		-----	-----		-----
6201	IP501	1.2		0.50	0.1		-0.05	14.9		1.03
6203	IP470	1.6		2.56	0.1		-0.05	15.0		1.10
6223	IP470	1.11		0.04	0.03		-0.17	13.24		-0.16
6238		-----		-----	-----		-----	-----		-----
6262		-----		-----	-----		-----	-----		-----

	Zn	Cu	Fe
normality	OK	not OK	OK
n	22	18	35
outliers	3	0	5
mean (n)	1.10	0.13	13.46
st.dev. (n)	0.272	0.088	1.416
R(calc.)	0.76	0.25	3.96
st.dev.(IP501:05)	0.194	0.557 (IP621:16)	1.399
R(IP501:05)	0.54	1.56 (IP:621:16)	3.92
Compare			
R(IP470:05)	3.64		8.98

Lab 154 first reported: 3
Lab 1635 first reported: 18.9
Lab 6026 first reported: 7.346





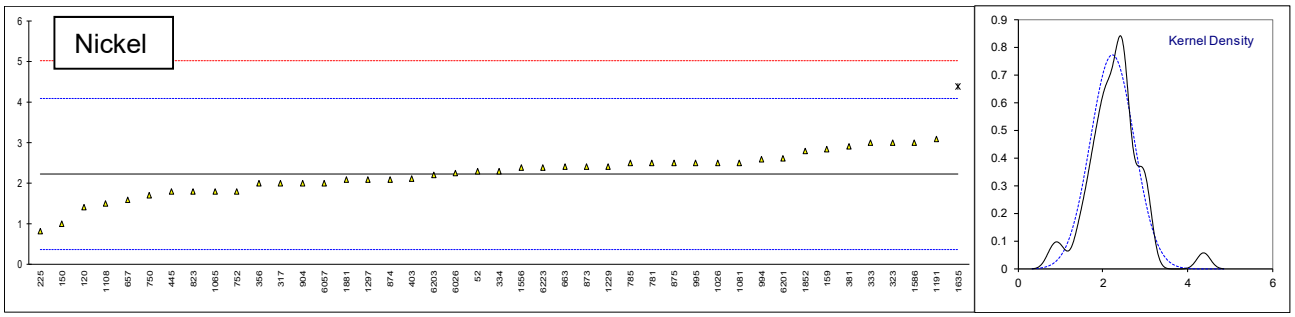


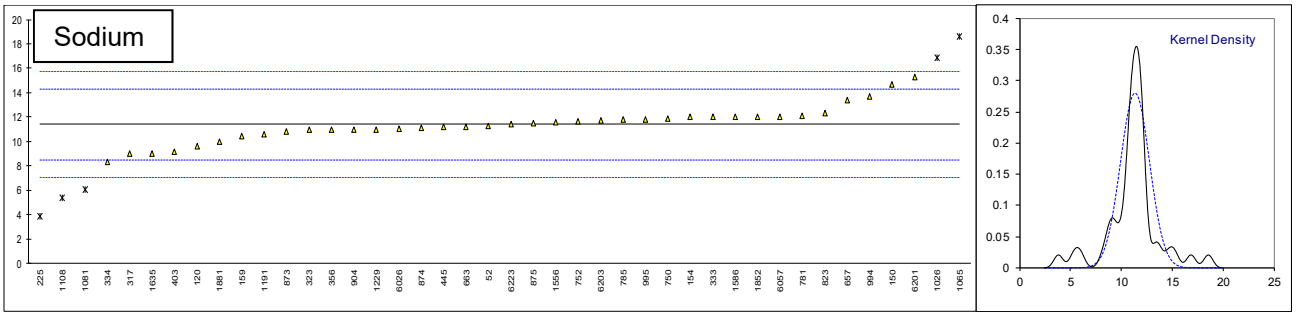
Determination of Nickel as Ni, Sodium as Na and Vanadium as V on sample #19266; results in mg/kg

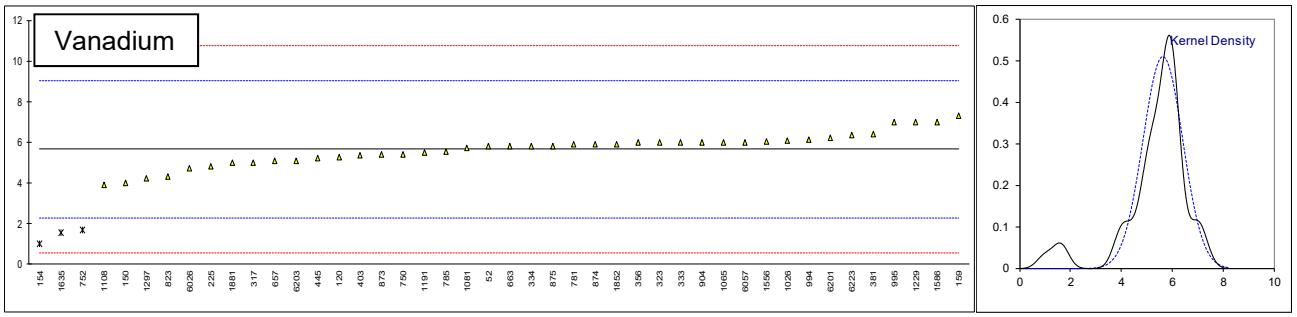
lab	method	Ni	mark	z(targ)	Na	mark	z(targ)	V	mark	z(targ)
52	IP501	2.3		0.08	11.3		-0.06	5.8		0.08
62		-----		-----	-----		-----	-----		-----
120	IP501	1.412		-0.87	9.624		-1.22	5.260		-0.23
140		-----		-----	-----		-----	-----		-----
150	IP501	1		-1.31	14.7	C	2.28	4		-0.98
154	IP501	<1		-----	12	C	0.42	1	R(0.01)	-2.74
158		-----		-----	-----		-----	-----		-----
159	IP501	2.84		0.66	10.42		-0.67	7.32		0.98
171		-----		-----	-----		-----	-----		-----
225	IP501	0.81		-1.52	3.86	R(0.05)	-5.19	4.82		-0.49
311		-----		-----	-----		-----	-----		-----
313		-----		-----	-----		-----	-----		-----
317	IP501	2		-0.24	9		-1.65	5		-0.39
323	IP501	3		0.83	11		-0.27	6		0.20
333	IP501	3		0.83	12		0.42	6		0.20
334	IP501	2.3		0.08	8.3		-2.13	5.8		0.08
356	IP501	2		-0.24	11		-0.27	6		0.20
381	INH-407	2.9		0.72	-----		-----	6.4	C	0.44
403	IP621	2.11		-0.12	9.15		-1.55	5.35		-0.18
445	IP501	1.8		-0.46	11.2		-0.13	5.2		-0.27
463		-----		-----	-----		-----	-----		-----
608		-----		-----	-----		-----	-----		-----
657	IP501	1.6		-0.67	13.4		1.38	5.1		-0.33
663	IP501	2.4		0.19	11.2		-0.13	5.8		0.08
732		-----		-----	-----		-----	-----		-----
750	IP621	1.7		-0.56	11.9		0.35	5.4		-0.15
752	IP621	1.803		-0.45	11.619		0.15	1.707	R(0.01)	-2.33
778		-----		-----	-----		-----	-----		-----
781	IP621	2.5		0.30	12.1		0.49	5.9		0.14
785	IP470	2.50		0.30	11.8		0.28	5.52		-0.08
798		-----		-----	-----		-----	-----		-----
823	IP501	1.8		-0.46	12.3		0.62	4.3		-0.80
873	IP621	2.4		0.19	10.8		-0.41	5.4		-0.15
874	IP501	2.1		-0.13	11.1		-0.20	5.9		0.14
875	IP501	2.5		0.30	11.5		0.07	5.8		0.08
904	IP470	2		-0.24	11		-0.27	6		0.20
994	IP501	2.59		0.39	13.68		1.57	6.135		0.28
995	IP470	2.5		0.30	11.8		0.28	7.0		0.79
997		-----		-----	-----		-----	-----		-----
1011		-----		-----	-----		-----	-----		-----
1026	IP501	2.5		0.30	16.9	R(0.05)	3.79	6.1		0.26
1065	UOP389	1.8		-0.46	18.6	R(0.05)	4.96	6.0		0.20
1081	In house	2.504		0.30	6.029	R(0.05)	-3.69	5.709		0.03
1108	D5708	1.5		-0.78	5.4	R(0.05)	-4.13	3.9		-1.04
1134		-----		-----	-----		-----	-----		-----
1191	IP501	3.08		0.92	10.61		-0.54	5.51		-0.09
1201		-----		-----	-----		-----	-----		-----
1229	In house	2.4		0.19	11		-0.27	7.0		0.79
1297	In house	2.090		-0.14	-----		-----	4.216		-0.85
1510		-----		-----	-----		-----	-----		-----
1556	IP621	2.387		0.17	11.608		0.15	6.022		0.22
1586	IP470	3		0.83	12		0.42	7		0.79
1635	IP501	4.38	R(0.01)	2.31	9.00		-1.65	1.57	R(0.01)	-2.41
1776		-----		-----	-----		-----	-----		-----
1852	DIN51790-4	2.8		0.62	12		0.42	5.9		0.14
1881	IP621	2.08		-0.16	10.0		-0.96	4.98		-0.40
6020		-----		-----	-----		-----	-----		-----
6026	IP621	2.2400		0.02	11.0705		-0.22	4.7277		-0.55
6057	IP501	2		-0.24	12		0.42	6		0.20
6112		-----		-----	-----		-----	-----		-----
6198		-----		-----	-----		-----	-----		-----
6201	IP501	2.6		0.40	15.3		2.69	6.2		0.32
6203	IP470	2.2		-0.03	11.7		0.21	5.1		-0.33
6223	IP470	2.39		0.18	11.40		0.00	6.34		0.40
6238		-----		-----	-----		-----	-----		-----
6262		-----		-----	-----		-----	-----		-----

	Ni	Na	V
normality	OK	suspect	OK
n	42	37	41
outliers	1	5	3
mean (n)	2.22	11.39	5.66
st.dev. (n)	0.516	1.427	0.782
R(calc.)	1.45	4.00	2.19
st.dev.(IP501:05)	0.932	1.452	1.697
R(IP501:05)	2.61	4.07	4.75
Compare			
R(IP470:05)	3.62	5.61	7.75

Lab 150 first reported: 2
Lab 154 first reported: 3
Lab 381 first reported: 28







APPENDIX 2

Number of participants per country

1 lab in	AZERBAIJAN
2 labs in	BELGIUM
2 labs in	CANADA
1 lab in	CHINA, People's Republic
1 lab in	CONGO Brazzaville
1 lab in	COTE D'IVOIRE
1 lab in	CROATIA
2 labs in	FINLAND
2 labs in	FRANCE
2 labs in	GEORGIA
2 labs in	GERMANY
3 labs in	GREECE
1 lab in	ISRAEL
1 lab in	KAZAKHSTAN
1 lab in	LITHUANIA
1 lab in	MALAYSIA
2 labs in	MALTA
7 labs in	NETHERLANDS
1 lab in	PORTUGAL
2 labs in	ROMANIA
12 labs in	RUSSIAN FEDERATION
1 lab in	SERBIA
1 lab in	SINGAPORE
1 lab in	SOUTH KOREA
3 labs in	SWEDEN
1 lab in	THAILAND
1 lab in	TURKEY
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 = possibly an error in calculations
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
SDS = Safety Data Sheet

Literature

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