



Institute for  
Interlaboratory Studies

## Results of Proficiency Test Vacuum Gasoil (VGO) November 2022

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 scheme for the analysis of Vacuum Gasoil (VGO) based on the latest version of ISO8217 every year. During the annual proficiency testing program 2022/2023 it was decided to continue the round robin for the analysis of Vacuum Gasoil (VGO).

In this interlaboratory study registered for participation:

- 60 laboratories in 26 countries for regular analyzes in VGO iis22G08
- 45 laboratories in 24 countries on metal analyzes in VGO iis22C08M

In total 60 laboratories in 26 countries registered for participation in one or two proficiency tests, see appendix 2 for the number of participants per country. In this report the results of the Vacuum Gasoil (VGO) proficiency tests are presented and discussed. This report is also electronically available through the iis website [www.iisnl.com](http://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.

In this proficiency test the participants received, depending on the registration, from one up to two different samples of Vacuum Gasoil, see table below.

Sample ID	PT ID	Quantity	Purpose
#22235	iis22G08	1x 1 L	Regular analyzes
#22236	iis22G08M	1x 100 mL PE	Metal analyzes

Table 1: Vacuum Gasoil samples used in iis22G08 and iis22G08M PTs

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](http://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 preparation of the sample for the regular analyzes in Vacuum Gasoil a batch of approximately 100 liters of Vacuum Gasoil was obtained from a third party. After homogenization 80 amber glass bottles of 1 L were filled and labelled #22235.

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 <sup>3</sup>
sample #22235-1	942.7
sample #22235-2	942.7
sample #22235-3	942.7
sample #22235-4	942.7
sample #22235-5	942.7
sample #22235-6	942.7
sample #22235-7	942.7
sample #22235-8	942.7

Table 2: homogeneity test results of subsamples #22235

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 <sup>3</sup>
r (observed)	0.0
reference test method	ISO12185:96
0.3 x R (reference test method)	0.5

Table 3: evaluation of the repeatability of subsamples #22235

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 preparation of the sample for the metal analyzes in Vacuum Gasoil a batch of approximately 10 liters of Vacuum Gasoil was obtained from a third party. To this batch detectable levels of Aluminum, Silicon, Iron, Nickel, Sodium and Vanadium were added. After homogenization 75 PE bottles of 100 mL were filled and labelled #22236.

The homogeneity of the subsamples was checked by determination of Nickel in accordance with IP501 on 8 stratified randomly selected subsamples.

	Nickel in mg/kg
sample #22236-1	7
sample #22236-2	6
sample #22236-3	7
sample #22236-4	7
sample #22236-5	6
sample #22236-6	6
sample #22236-7	6
sample #22236-8	4 D(0.05)

Table 4: homogeneity test results of subsamples #22236

Subsample 8 is a Dixon outlier and therefore excluded from statistical evaluation of the homogeneity.

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.

	Nickel in mg/kg
r (observed)	1.5
reference test method	IP621:16
0.3 x R (reference test method)	1.5

Table 5: evaluation of the repeatabilities of subsamples #22236

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

Depending on the registration of the participant the appropriate set of PT samples was sent on November 2, 2022. 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 #22235: Total Acid Number, Aniline Point, Asphaltenes, Carbon Residue (Micro method), Density at 15 °C, Flash Point PMcc, Kinematic Viscosity at 50 °C and 100 °C, Nitrogen, Pour Point (Manual and Automated), Total Sulfur, Simulated Distillation and Distillation at 10 mmHg (IBP, 10% rec, 30% rec, 50% rec, 70% rec, 90% rec and FBP).

On sample #22236 it was requested to determine: Aluminum, Silicon, Sum Aluminum and Silicon, Arsenic, Copper, Iron, Nickel, Sodium, Vanadium and Calcium.

It was also requested to report some analytical details on the determination of Total Acid Number.

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/](http://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](http://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/](http://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.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. 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 (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

### 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 (derived from e.g. ISO or ASTM test methods), 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, 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. Therefore, 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. The reporting time on the data entry portal was extended with another week.

In the Vacuum Gasoil regular round robin ten participants reported test results after the extended reporting date and three other participants did not report any test results.

In the Vacuum Gasoil Metals round robin six participants reported test results after the extended reporting date and five other participants did not report any test results.

Not all participants were able to report all tests requested.

In total 57 participants reported 1048 numerical test results. Observed were 47 outlying test results, which is 4.5%. In proficiency tests 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 in appendix 1. The abbreviations, used in these tables, are explained in appendix 3.

In the iis PT reports ASTM test methods are referred to with a number (e.g. D611) and an added designation for the year that the test method was adopted or revised (e.g. D611:12). When a method has been reapproved an “R” will be added and the year of approval (e.g. D611:12R16).

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 is used.

##### **sample #22235**

Total Acid Number: The majority of the laboratories reported to use Inflection Point with titration volume of 125 mL. Therefore, the z-scores are calculated with the reproducibility of ASTM D664-A:18e2 for Inflection Point at titration volume 125 mL.

This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of ASTM D664-A:18e2 for IP at 125 mL and is also in agreement with the requirements of ASTM D664-A:18e2 for IP at 60 mL, BEP at 60 mL and BEP at 125 mL.

Aniline Point: 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 D611:12R16. According to §7.1 of test method ASTM D611:12R16 the Aniline should be sufficiently pure that when tested with n-Heptane the Aniline Point shall be  $69.3 \pm 0.2$  °C. When evaluated over the test results where this criterium is met the calculated reproducibility is still not in agreement with the requirements of ASTM D611:12R16.

Asphaltenes: This determination was not problematic. Almost all reporting participants agreed on a level <0.50 %M/M which is below the application range of test method IP143:04R21. Therefore, no z-scores are calculated.

Carbon Residue (Micro method): This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D4530:15R20.

Density at 15 °C: This determination was problematic for a number of laboratories. Five 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. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D445:21e2.

Kinematic Viscosity at 100 °C: This determination was not problematic. Five statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D445:21e2.

Nitrogen: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with ASTM D5762:18a.

Pour Point Manual: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with ASTM D97:17bR22.

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 D5950:14R20.

Total Sulfur: 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 D4294:21.

Simulated Distillation: This determination may be problematic. Over seven parameters eight statistical outliers were observed and two other test results were excluded. The calculated reproducibilities for IBP, 50%, 70% and 90% recovered after rejection of the suspect data are in agreement with the requirements of ASTM D6352:19e1, 10% and 30% recovered and FBP are not in agreement.

Distillation at 10 mmHg as AET: This determination was not problematic. Over seven parameters four statistical outliers were observed. Allmost all calculated reproducibilities after rejection of the statistical outliers are in agreement with the requirements of ASTM D1160:18, except for 70% and 90% recovered.

**sample #22236**

Aluminum: 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 IP501:05R19.

Silicon: 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 IP501:05R19.

Sum of Aluminum and Silicon: This determination was not problematic. One statistical outlier was observed and two other test results were excluded. The calculated reproducibility after rejection of the suspect data is in agreement with the requirements of IP501:05R19.

Arsenic: This determination was not problematic. All reporting participants agreed on a level <1 mg/kg. Therefore, no z-scores are calculated.

Copper: This determination was not problematic. All reporting participants agreed on a level <1 mg/kg. Therefore, no z-scores are calculated.

Iron: 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 IP621:16.

Nickel: This determination was not problematic. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of IP621:16.

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

Vanadium: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of IP621:16.

Calcium: This determination was not problematic. All reporting participants agreed on a level <3 mg/kg. Therefore, no z-scores are calculated.

## 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility ( $2.8 * \text{standard deviation}$ ) and the target reproducibility derived from reference methods are presented in the next table.

Parameter	unit	n	average	$2.8 * \text{sd}$	R(lit)
Total Acid Number	mg KOH/g	40	0.59	0.12	0.13
Aniline Point	°C	19	79.8	1.6	1
Asphaltenes	%M/M	33	<0.50	n.e.	n.e.
Carbon Residue (Micro method)	%M/M	44	0.76	0.31	0.18
Density at 15 °C	kg/m <sup>3</sup>	50	943.1	1.3	1.5
Flash Point PMcc	°C	38	203.4	6.9	10
Kinematic Viscosity at 50 °C	mm <sup>2</sup> /s	51	97.517	2.683	8.251
Kinematic Viscosity at 100 °C	mm <sup>2</sup> /s	44	13.033	0.218	1.572
Nitrogen	mg/kg	35	1720	278	457
Pour Point Manual	°C	45	39.4	10.1	9
Pour Point Automated 3 °C int.	°C	16	39.1	10.2	6.1
Total Sulfur	%M/M	51	2.52	0.19	0.13
Simulated Distillation					
Initial Boiling Point	°C	13	290.4	18.5	49.1
Temp 10% recovered	°C	15	398.2	7.9	7.1
Temp 30% recovered	°C	15	447.3	7.1	5.9
Temp 50% recovered	°C	15	477.6	6.8	6.4
Temp 70% recovered	°C	15	505.3	7.6	7.2
Temp 90% recovered	°C	14	542.2	8.5	10.5
Final Boiling Point	°C	15	614.9	58.3	38.1
Distillation at 10 mmHg as AET					
Initial Boiling Point	°C	26	298.4	46.0	49.4
Temp 10% recovered	°C	26	415.8	16.0	18.4
Temp 30% recovered	°C	26	457.3	11.5	11.4
Temp 50% recovered	°C	25	481.1	8.3	10.0
Temp 70% recovered	°C	26	504.8	9.2	8.3
Temp 90% recovered	°C	24	537.5	11.1	8.9
Final Boiling Point	°C	23	555.9	11.5	26.9

Table 6: reproducibilities of tests on sample #22235

Parameter	unit	n	average	2.8 * sd	R(lit)
Aluminum as Al	mg/kg	32	9.9	2.4	3.3
Silicon as Si	mg/kg	24	4.6	3.8	1.5
Sum Aluminum and Silicon	mg/kg	21	14.6	3.8	3.7
Arsenic as As	mg/kg	8	<1	n.e.	n.e.
Copper as Cu	mg/kg	30	<1	n.e.	n.e.
Iron as Fe	mg/kg	34	10.0	4.0	9.7
Nickel as Ni	mg/kg	32	6.5	2.2	5.1
Sodium as Na	mg/kg	35	8.3	3.7	5.5
Vanadium as V	mg/kg	36	9.0	2.4	3.9
Calcium as Ca	mg/kg	33	<3	n.e.	n.e.

Table 7: reproducibilities of tests on sample #22236

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 NOVEMBER 2022 WITH PREVIOUS PTS

	November 2022	December 2021	December 2020	December 2019	December 2018
Number of reporting laboratories	57	63	64	63	66
Number of test results	1048	1276	1172	1211	1113
Number of statistical outliers	47	35	37	69	39
Percentage of statistical outliers	4.5%	2.7%	3.2%	5.7%	3.5%

Table 8: 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.

Parameter	November 2022	December 2021	December 2020	December 2019	December 2018
Total Acid Number	+/-	-	-	-	++
Aniline Point	-	--	-	--	--
Asphaltenes	n.e.	n.e.	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	++	++	++	++	++
Nitrogen	+	+	-	+	+
Pour Point Manual	-	+	++	+	++

Parameter	November 2022	December 2021	December 2020	December 2019	December 2018
Pour Point Automated 3 °C int.	-	+/-	+/-	-	+
Total Sulfur	-	+	+/-	+/-	+/-
Simulated Distillation	+	+	-	-	+
Distillation at 10 mmHg as AET	+	+	+/-	+/-	+
Aluminum as Al	+	+	+/-	+/-	n.a.
Silicon as Si	--	--	--	-	n.e.
Sum Aluminum and Silicon	+/-	--	-	+/-	n.a.
Arsenic as As	n.e.	n.e.	n.e.	--	n.e.
Copper as Cu	n.e.	n.e.	+/-	++	n.e.
Iron as Fe	++	+/-	+/-	+/-	+
Nickel as Ni	++	+	+	+	+
Sodium as Na	+	+	+/-	+/-	++
Vanadium as V	+	++	++	+/-	+
Calcium as Ca	n.e.	-	n.e.	+/-	-

Table 9: comparison of determinations to the reference test methods

For results between brackets no z-scores are calculated.

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 #22235; results in mg KOH/g

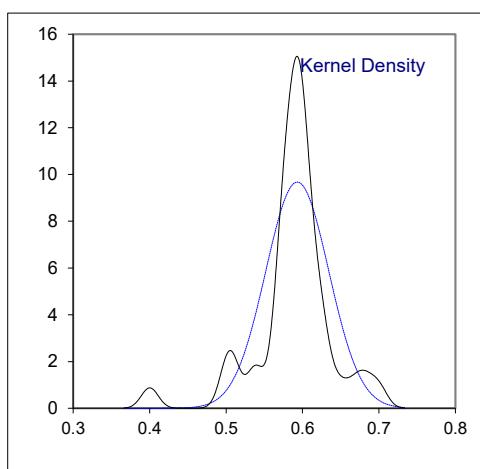
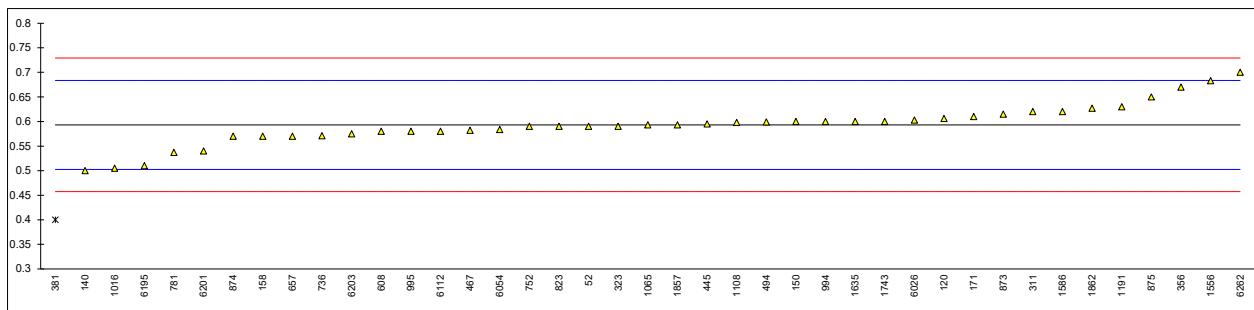
lab	method	value	mark	z(targ)	end point	volume	remarks
52	D664-A	0.59		-0.07	---	---	
62		----		----	---	---	
120	D664-A	0.606	C	0.28	Inflection Point	60 mL	fr. 0.902
140	D664-A	0.50		-2.06	Inflection Point	125 mL	
150	D664-A	0.60		0.15	---	---	
154		----		----	---	---	
158	D664-A	0.57		-0.51	Inflection Point	125 mL	
159		----		----	---	---	
171	D664-A	0.61		0.37	---	---	
225		----		----	---	---	
311	D664-A	0.62		0.59	---	---	
313		----		----	---	---	
323	D664-A	0.59		-0.07	Inflection Point	125 mL	
333		----		----	---	---	
356	D664-A	0.67	C	1.70	Inflection Point	125 mL	fr. 0.42
381	D664-A	0.4	C,R(0.01)	-4.26	Buffer End Point pH 10	125 mL	fr. 0.29
445	D664-A	0.595		0.04	Inflection Point	60 mL	
467	D664-A	0.582		-0.25	Buffer End Point pH 11	125 mL	
494	D664-A	0.599		0.13	Inflection Point	60 mL	
608	D664-A	0.58		-0.29	Inflection Point	125 mL	
657	D664-A	0.57		-0.51	Inflection Point	125 mL	
663		----		----	---	---	
710		----		----	---	---	
736	D664-A	0.5710		-0.49	Inflection Point	125 mL	
750		----		----	---	---	
752	D664-A	0.59		-0.07	Inflection Point	60 mL	
753		----		----	---	---	
778		----		----	---	---	
781	D664-A	0.537		-1.24	Inflection Point	125 mL	
785		----		----	---	---	
798		----		----	---	---	
823	D664-A	0.59		-0.07	Inflection Point	125 mL	
872		----		----	---	---	
873	D664-A	0.615		0.48	Inflection Point	125 mL	
874	D664-A	0.57		-0.51	Buffer End Point pH 10	125 mL	
875	D664-A	0.65		1.25	---	---	
994	D664-A	0.60		0.15	Buffer End Point pH 10	125 mL	
995	D664-A	0.58		-0.29	Inflection Point	125 mL	
1016	D664-A	0.505		-1.94	---	---	
1065	D664-A	0.593		0.00	---	---	
1081		----		----	---	---	
1108	D664-A	0.5980		0.11	Inflection Point	125 mL	
1191	ISO6618	0.63		0.81	---	---	
1205		----		----	---	---	
1556	D664-A	0.683		1.98	Inflection Point	---	
1586	D664-A	0.62		0.59	Inflection Point	125 mL	
1635	D664-A	0.6		0.15	Inflection Point	60 mL	
1743	D664-A	0.60		0.15	Buffer End Point pH 11	60 mL	
1857	D664-A	0.593		0.00	Inflection Point	125 mL	
1862	D664-A	0.627		0.75	Inflection Point	125 mL	
6026	D664-A	0.6029		0.22	Inflection Point	125 mL	
6054	D974	0.58363	C	-0.21	---	---	fr. 0.94634
6112	D664-A	0.58		-0.29	---	---	
6114		----		----	---	125 mL	
6195	D664-A	0.51		-1.83	Inflection Point	125 mL	
6201	D664-A	0.54		-1.17	Inflection Point	125 mL	
6203	D664-A	0.575		-0.40	Inflection Point	125 mL	
6262	D664-A	0.70		2.36	---	---	
6447		----		----	---	---	
6496		----		----	---	---	

normality  
n  
outliers  
mean (n)  
st.dev. (n)  
R(calc.)  
st.dev.(D664-A:18e2 IP 125 mL)  
R(D664-A:18e2 IP 125 mL)

suspect  
40  
1  
0.5931  
0.04126  
0.1155  
0.04532  
0.1269  
0.2621  
0.3329  
0.1825

Compare:

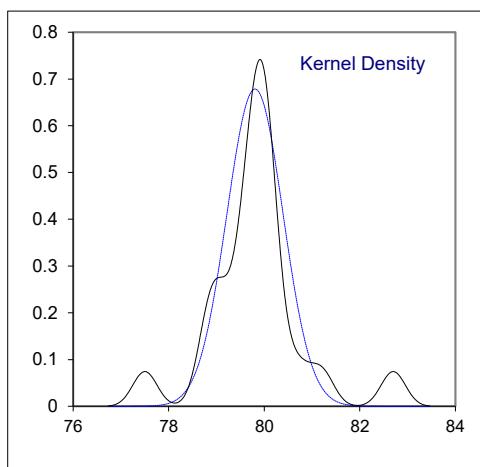
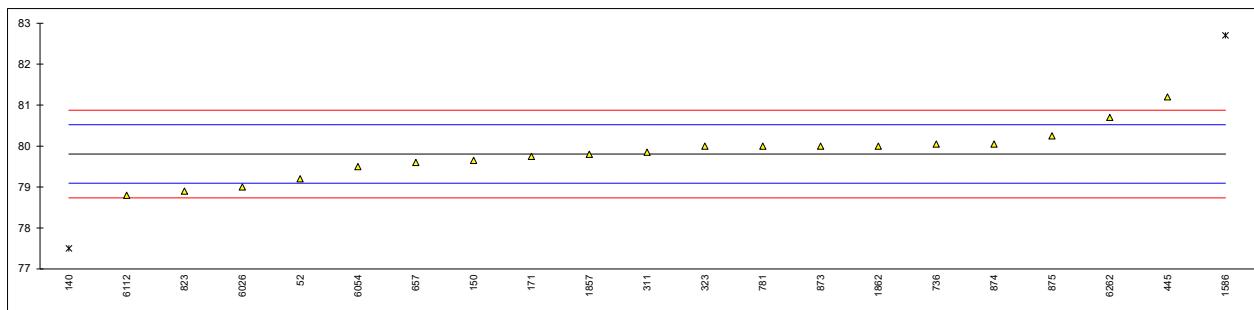
R(D664-A:18e2 IP 60 mL)  
R(D664-A:18e2 BEP 60 mL)  
R(D664-A:18e2 BEP 125 mL)



## Determination of Aniline Point on sample #22235; results in °C

lab	method	value	mark	z(targ)	of n-Heptane	mark	remarks
52	D611-E	79.20		-1.69	69.25		
62		----		----	----		
120		----		----	----		
140	D611-E	77.5	R(0.05)	-6.45	----		
150	D611-E	79.65	C	-0.43	----		first reported 78.45
154		----		----	----		
158		----		----	----		
159		----		----	----		
171	D611-E	79.75		-0.15	----		
225		----		----	----		
311	D611-E	79.85		0.13	69.35		
313		----		----	----		
323	D611-A	80.0		0.55	----		
333		----		----	----		
356		----		----	----		
381		----		----	----		
445	D611-A	81.20		3.91	69.40		
467		----		----	----		
494		----		----	----		
608		----		----	----		
657	D611-B	79.60		-0.57	69.4		
663		----		----	----		
710		----		----	----		
736	D611-B	80.05		0.69	70.35	C	first reported 70.15
750		----		----	----		
752		----		----	----		
753		----		----	----		
778		----		----	----		
781	D611-E	80.00		0.55	69.2		
785		----		----	----		
798		----		----	----		
823	D611-E	78.9	C	-2.53	69.3		first reported 82.1
872		----		----	----		
873	D611-E	80.00		0.55	69.30		
874	D611-E	80.05		0.69	69.30		
875	D611-E	80.25		1.25	69.3		
994		----		----	----		
995		----		----	----		
1016		----		----	----		
1065		----		----	----		
1081		----		----	----		
1108		----		----	----		
1191		----		----	----		
1205		----		----	----		
1556		----		----	----		
1586	D611-A	82.7	C,R(0.05)	8.11	----		first reported 78.2
1635		----		----	----		
1743		----		----	----		
1857	D611-E	79.800		-0.01	69.35		
1862	D611-B	80.00		0.55	69.30		
6026	D611-E	79.00		-2.25	----		
6054	D611-E	79.5		-0.85	69.2		
6112	D611-E	78.8	C	-2.81	----		first reported 77.8
6114		----		----	----		
6195		----		----	----		
6201		----		----	----		
6203		----		----	----		
6262	D611-D	80.70		2.51	----		
6447		----		----	----		
6496		----		----	----		
normality		OK	Aniline Point with n-heptane between 69.3 °C ± 0.2 °C only *:				
n		19	12				
outliers		2	0				
mean (n)		79.805	79.862				
st.dev. (n)		0.5881	0.5721				
R(calc.)		1.647	1.602				
st.dev.(D611:12R16)		0.3571	0.3571				
R(D611:12R16)		1	1				

\* According to §7.1 of ASTM D611:12R16

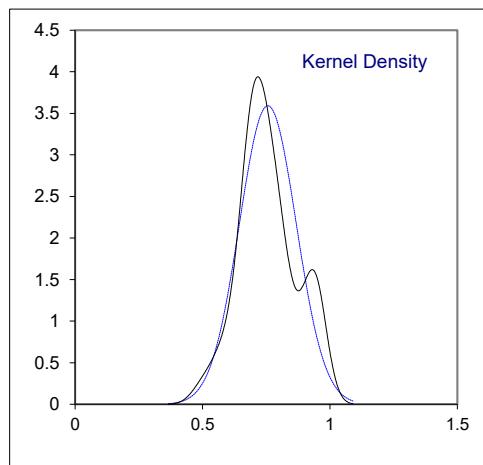
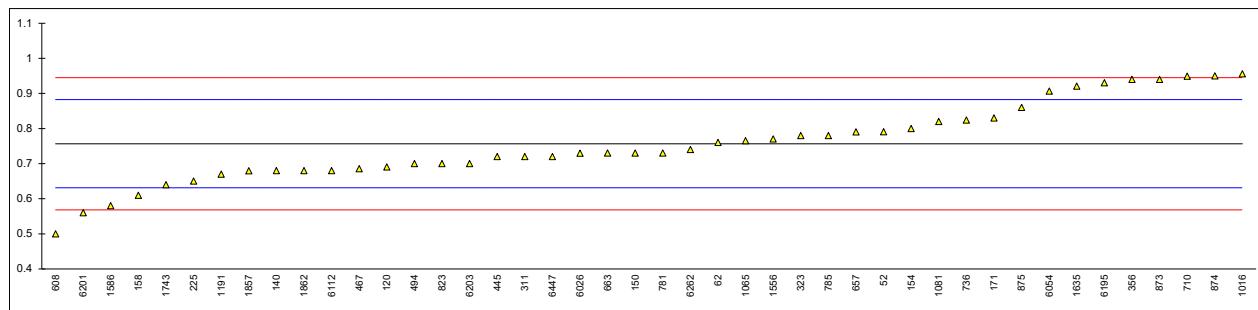


## Determination of Asphaltenes on sample #22235; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	IP143	0.109		----	
62		----		----	
120		----		----	
140	IP143	0.50		----	
150	IP143	0.15		----	
154	IP143	0.05		----	
158	D6560	0.45		----	
159		----		----	
171	IP143	0.02		----	
225		----		----	
311		----		----	
313		----		----	
323		----		----	
333		----		----	
356	IP143	Below 0.50		----	
381	INH-642	0.008		----	
445	IP143	0.14		----	
467	IP143	0.02		----	
494	D6560	0.63		----	
608	D6560	0.36		----	
657	IP143	0.07		----	
663	IP143	<0.50		----	
710		----		----	
736		----		----	
750		----		----	
752	INH-642	0.01744		----	
753		----		----	
778		----		----	
781	IP143	0.12		----	
785	IP143	0.05		----	
798		----		----	
823	IP143	<0.50		----	
872		----		----	
873	IP143	<0.5		----	
874	In house	<0.50		----	
875	IP143	<0.5		----	
994	D6560	<0.50		----	
995	IP143	<0.5		----	
1016	IP143	0.16		----	
1065		----		----	
1081		----		----	
1108		----		----	
1191		----		----	
1205		----		----	
1556		----		----	
1586	IP143	0.01		----	
1635	IP143	0.08		----	
1743		----		----	
1857	In house	0.0239		----	
1862	In house	0.0235		----	
6026	In house	0.0202		----	
6054	D6560	0.06401	C		first reported 1.36385
6112		----		----	
6114	IP143	<0.50		----	
6195	IP143	0.15		----	
6201	IP143	0.15		----	
6203	IP143	0.45		----	
6262	IP143	0.0317		----	
6447		----		----	
6496		----		----	
n		33			
mean (n)		<0.50			application range IP143:04R21 between 0.50-30 %M/M

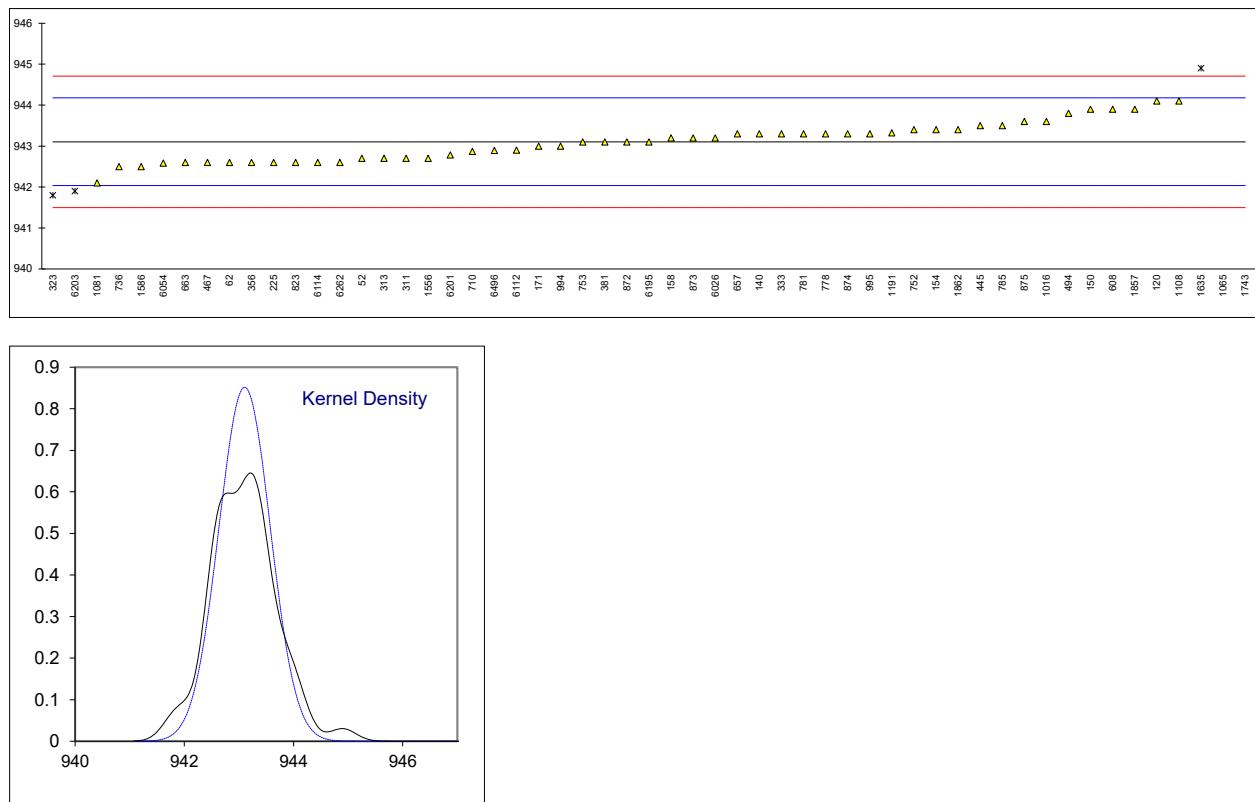
## Determination of Carbon Residue (Micro method) on sample #22235; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	D4530	0.791		0.55	
62	D4530	0.76		0.06	
120	ISO10370	0.69		-1.06	
140	D4530	0.68		-1.22	
150	D4530	0.73		-0.42	
154	D4530	0.80		0.69	
158	D4530	0.61		-2.33	
159		----		----	
171	D4530	0.83		1.17	
225	D4530	0.65		-1.70	
311	D4530	0.72		-0.58	
313		----		----	
323	D4530	0.78		0.37	
333		----		----	
356	D4530	0.94		2.92	
381		----		----	
445	D4530	0.72		-0.58	
467	D4530	0.685		-1.14	
494	D4530	0.70		-0.90	
608	D4530	0.50		-4.08	
657	D4530	0.79	C	0.53	first reported 1.22
663	D4530	0.73		-0.42	
710	ISO10370	0.949		3.07	
736	D4530	0.824	C	1.07	first reported 0.963
750		----		----	
752		----		----	
753		----		----	
778		----		----	
781	D4530	0.73		-0.42	
785	D4530	0.78		0.37	
798		----		----	
823	ISO10370	0.70		-0.90	
872		----		----	
873	D4530	0.94		2.92	
874	D4530	0.95		3.08	
875	D4530	0.86		1.65	
994		----		----	
995		----		----	
1016	ISO10370	0.9561		3.18	
1065	D4530	0.7651		0.14	
1081	ISO10370	0.82		1.01	
1108		----		----	
1191	ISO10370	0.66982		-1.38	
1205		----		----	
1556	ISO10370	0.77		0.21	
1586	ISO10370	0.58		-2.81	
1635	D4530	0.921		2.62	
1743	ISO10370	0.64		-1.86	
1857	D4530	0.679		-1.23	
1862	D4530	0.680		-1.22	
6026	ISO10370	0.7294		-0.43	
6054	D4530	0.906525		2.39	
6112	ISO10370	0.68		-1.22	
6114		----		----	
6195	D4530	0.93		2.76	
6201	D4530	0.56		-3.13	
6203	ISO10370	0.70		-0.90	
6262	D4530	0.74		-0.26	
6447	D4530	0.72		-0.58	
6496		----		----	
	normality	OK			
	n	44			
	outliers	0			
	mean (n)	0.7565			
	st.dev. (n)	0.11111			
	R(calc.)	0.3111			
	st.dev.(D4530:15R20)	0.06280			
	R(D4530:15R20)	0.1758			
Compare:	R(ISO10370:14)	0.0639			



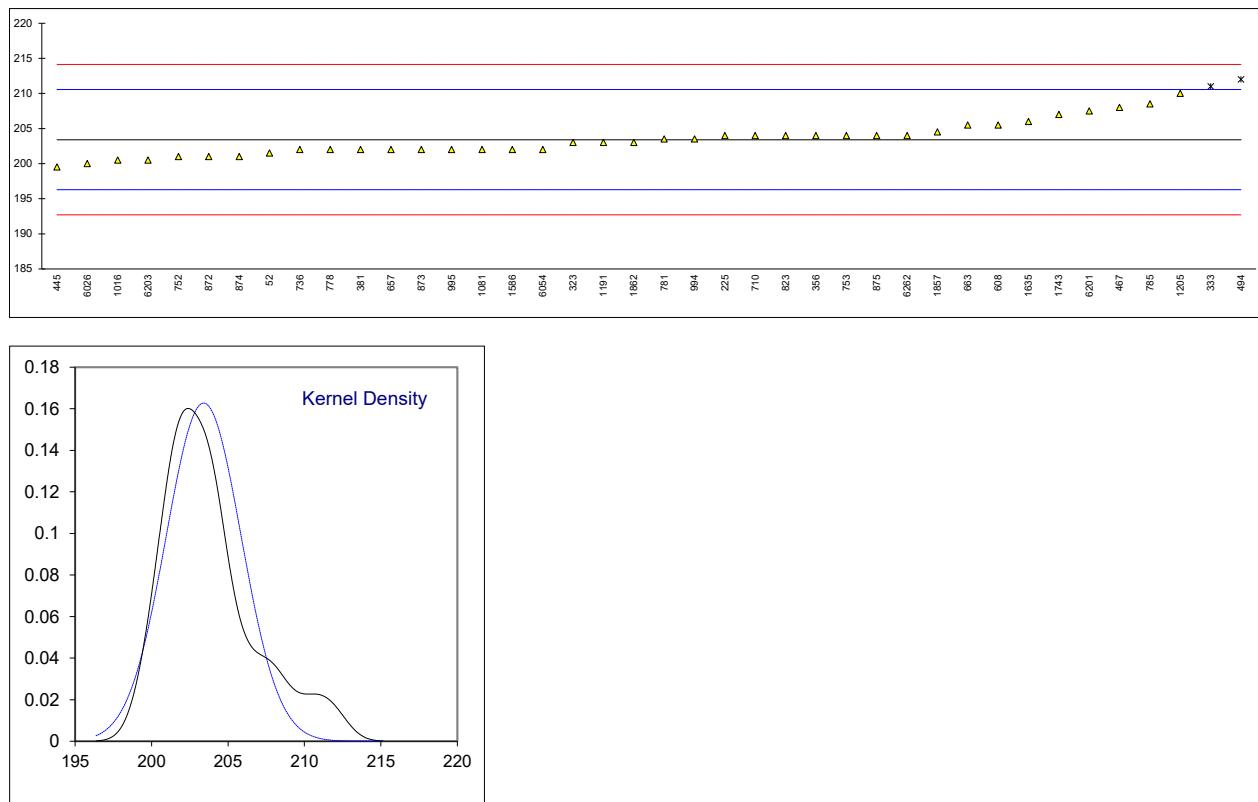
Determination of Density at 15 °C on sample #22235; results in kg/m<sup>3</sup>

lab	method	value	mark	z(targ)	remarks
52	D4052	942.7		-0.76	
62	D4052	942.6		-0.94	
120	D4052	944.1		1.86	
140	D4052	943.3		0.36	
150	D4052	943.9		1.48	
154	D4052	943.4		0.55	
158	D4052	943.2		0.18	
159		----		----	
171	ISO12185	943.0		-0.20	
225	D4052	942.6		-0.94	
311	ISO12185	942.7		-0.76	
313	ISO12185	942.7		-0.76	
323	ISO12185	941.8	R(0.05)	-2.44	
333	ISO12185	943.3		0.36	
356	ISO12185	942.6		-0.94	
381	D4052	943.1	C	-0.01	first reported 937.0
445	D4052	943.5		0.74	
467	ISO12185	942.6		-0.94	
494	ISO12185	943.8		1.30	
608	D4052	943.9		1.48	
657	D4052	943.3		0.36	
663	D4052	942.6		-0.94	
710	ISO12185	942.87		-0.44	
736	ISO12185	942.5		-1.13	
750		----		----	
752	D1298	943.4		0.55	
753	ISO12185	943.1		-0.01	
778	ISO12185	943.3		0.36	
781	ISO12185	943.3		0.36	
785	D1298	943.5		0.74	
798		----		----	
823	ISO12185	942.6		-0.94	
872	ISO12185	943.1		-0.01	
873	ISO12185	943.2		0.18	
874	ISO12185	943.3		0.36	
875	ISO12185	943.6		0.92	
994	ISO12185	943.0		-0.20	
995	ISO12185	943.3		0.36	
1016	ISO12185	943.6		0.92	
1065	D1298	952.3	C,R(0.01)	17.16	first reported 0.9515 kg/L
1081	D4052	942.1		-1.88	
1108	D1298	944.1	C	1.86	first reported 941.1
1191	ISO12185	943.32		0.40	
1205		----		----	
1556	ISO12185	942.7		-0.76	
1586	ISO12185	942.5		-1.13	
1635	ISO3675	944.9	R(0.05)	3.35	
1743	ISO12185	955.0	R(0.01)	22.20	
1857	ISO12185	943.9		1.48	
1862	ISO3675	943.4		0.55	
6026	D1298	943.2		0.18	
6054	D4052	942.58		-0.98	
6112	ISO12185	942.9		-0.38	
6114	ISO12185	942.6		-0.94	
6195	IP365	943.1		-0.01	
6201	ISO12185	942.78		-0.61	
6203	D7042	941.9	R(0.05)	-2.25	
6262	D4052	942.6		-0.94	
6447		----		----	
6496	ISO12185	942.89		-0.40	
	normality	OK			
	n	50			
	outliers	5			
	mean (n)	943.10			
	st.dev. (n)	0.468			
	R(calc.)	1.31			
	st.dev.(ISO12185:96)	0.536			
	R(ISO12185:96)	1.5			



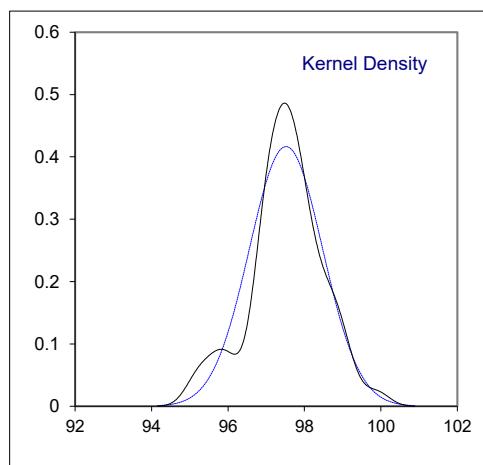
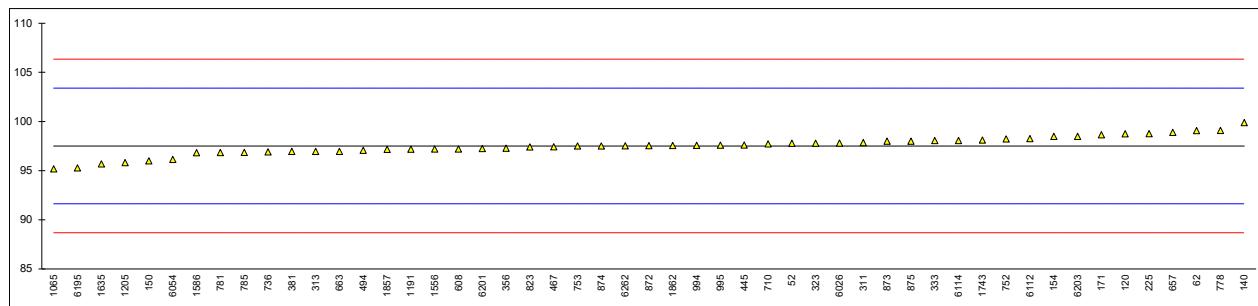
## Determination of Flash Point PMcc on sample #22235; results in °C

lab	method	value	mark	z(targ)	remarks
52	D93-A	201.5		-0.53	
62		----		----	
120		----		----	
140	D93-B	>110		----	
150	D93-B	>110		----	
154	D93-B	>130	C	----	first reported 113
158	D93-B	>110		----	
159		----		----	
171	D93-B	>110		----	
225	D93-A	204.0		0.17	
311	D93-B	>200		----	
313	D93-B	>200		----	
323	D93-B	203.0		-0.11	
333	D93-B	211	DG(0.05)	2.13	
356	D93-B	204.0		0.17	
381	D93-B	202		-0.39	
445	D93-B	199.5		-1.09	
467	D93-B	208.0		1.29	
494	D93-B	212.0	DG(0.05)	2.41	
608	D93-B	205.5		0.59	
657	D93-B	202.0		-0.39	
663	D93-B	205.5		0.59	
710	D93-B	204.0		0.17	
736	D93-B	202.0		-0.39	
750		----		----	
752	D93-B	201.0		-0.67	
753	D93-B	204.0		0.17	
778	D93-B	202.0		-0.39	
781	D93-B	203.5		0.03	
785	D93-B	208.5		1.43	
798		----		----	
823	ISO2719	204	C	0.17	first reported 194
872	D93-B	201.0		-0.67	
873	D93-B	202		-0.39	
874	D93-B	201		-0.67	
875	D93-B	204.0		0.17	
994	D93-B	203.5		0.03	
995	D93-B	202.0		-0.39	
1016	D93-B	200.5		-0.81	
1065		----		----	
1081	D93-B	202		-0.39	
1108		----		----	
1191	ISO2719	203		-0.11	
1205	D93-B	210.0		1.85	
1556	ISO2719	>110		----	
1586	D93-B	202.0		-0.39	
1635	D93-B	206		0.73	
1743	ISO2719	207		1.01	
1857	D93-A	204.5		0.31	
1862	D93-B	203.0		-0.11	
6026	D93-B	200.0		-0.95	
6054	D93-B	202.0		-0.39	
6112		----		----	
6114		----		----	
6195	D93-B	>110		----	
6201	D93-B	207.5		1.15	
6203	D93-B	200.5		-0.81	
6262	D93-B	204.0		0.17	
6447		----		----	
6496		----		----	
	normality	OK			
	n	38			
	outliers	2			
	mean (n)	203.408			
	st.dev. (n)	2.4518			
	R(calc.)	6.865			
	st.dev.(D93-B:20)	3.5714			
	R(D93-B:20)	10			



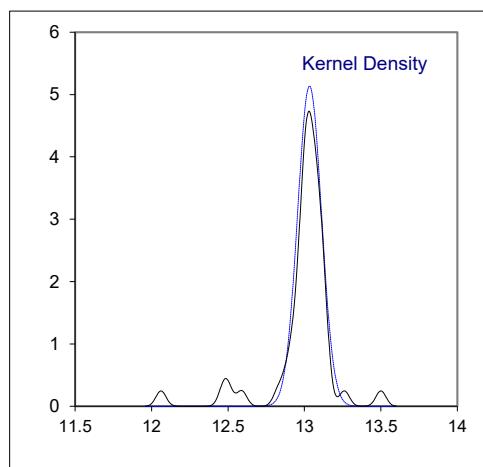
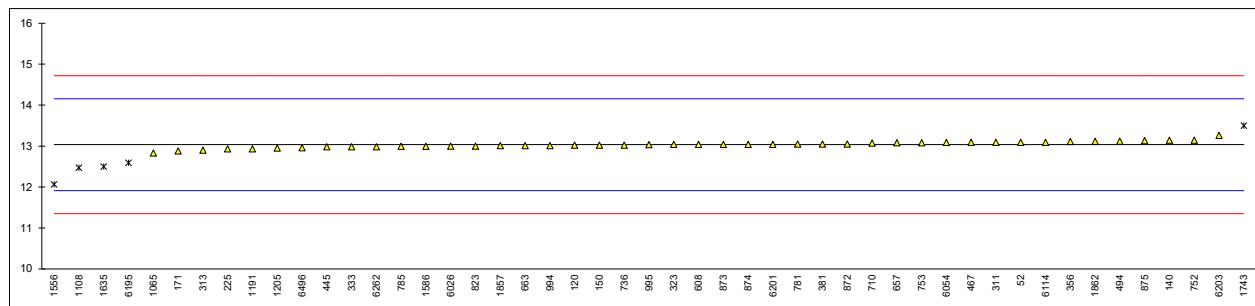
Determination of Kinematic Viscosity at 50 °C on sample #22235; results in mm<sup>2</sup>/s

lab	method	value	mark	z(targ)	remarks
52	D445	97.78		0.09	
62	D445	99.06		0.52	
120	D445	98.73		0.41	
140	D445	99.88		0.80	
150	D445	95.98		-0.52	
154	D445	98.47		0.32	
158		----		----	
159		----		----	
171	D445	98.65		0.38	
225	D445	98.77		0.43	
311	D445	97.84		0.11	
313	D445	96.95		-0.19	
323	D445	97.78		0.09	
333	D445	98.05		0.18	
356	D445	97.27		-0.08	
381	D445	96.95		-0.19	
445	D445	97.596		0.03	
467	ISO3104	97.423		-0.03	
494	D445	97.07		-0.15	
608	D445	97.19		-0.11	
657	D445	98.89		0.47	
663	D445	96.95		-0.19	
710	D445	97.720		0.07	
736	D445	96.91		-0.21	
750		----		----	
752	D445	98.22		0.24	
753	D445	97.51		0.00	
778	D445	99.08		0.53	
781	D445	96.836		-0.23	
785	D445	96.85		-0.23	
798		----		----	
823	ISO3104	97.40		-0.04	
872	D445	97.54		0.01	
873	D445	97.99		0.16	
874	D445	97.51		0.00	
875	D445	97.99		0.16	
994	D445	97.57		0.02	
995	D445	97.58		0.02	
1016		----		----	
1065	D445	95.17		-0.80	
1081		----		----	
1108		----		----	
1191	ISO3104	97.171		-0.12	
1205	ISO3104	95.79915		-0.58	
1556	ISO3104	97.189	C	-0.11	first reported 971.89
1586	ISO3104	96.81		-0.24	
1635	D445	95.68		-0.62	
1743		98.1		0.20	
1857	D445	97.167		-0.12	
1862	D445	97.555		0.01	
6026	D445	97.7961		0.09	
6054	D445	96.134031		-0.47	
6112	D445	98.25		0.25	
6114	D445	98.05		0.18	
6195	D445	95.28		-0.76	
6201	D445	97.24		-0.09	
6203	D7042	98.484		0.33	
6262	D445	97.530		0.00	
6447		----		----	
6496		----		----	
normality		OK			
n		51			
outliers		0			
mean (n)		97.5175			
st.dev. (n)		0.95809			
R(calc.)		2.6826			
st.dev.(D445:21e2)		2.94677			
R(D445:21e2)		8.2510			



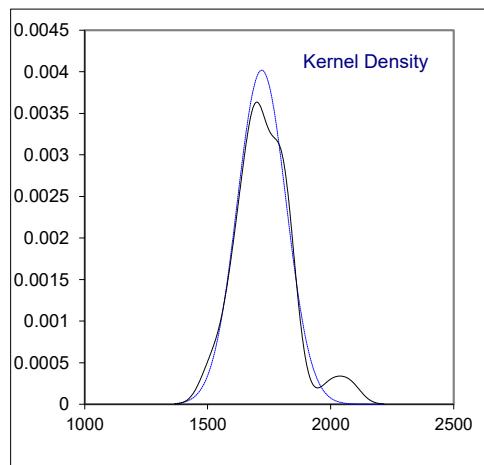
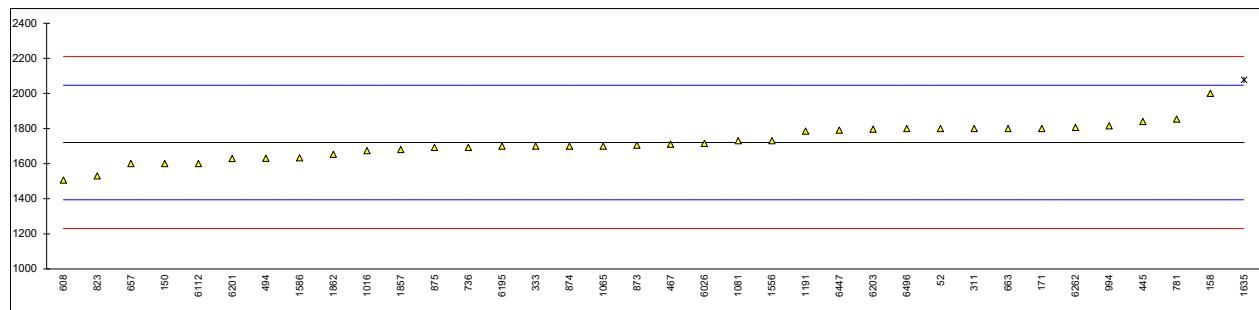
## Determination of Kinematic Viscosity at 100 °C on sample #22235; results in mm<sup>2</sup>/s

lab	method	value	mark	z(targ)	remarks
52	D445	13.09		0.10	
62		-----		-----	
120	D7042	13.02		-0.02	
140	D445	13.14		0.19	
150	D445	13.02		-0.02	
154		-----		-----	
158		-----		-----	
159		-----		-----	
171	D445	12.88		-0.27	
225	D445	12.93		-0.18	
311	D445	13.09		0.10	
313	D445	12.90		-0.24	
323	D445	13.04		0.01	
333	D445	12.98		-0.09	
356	D445	13.11		0.14	
381	D445	13.05		0.03	
445	D445	12.98		-0.09	
467	ISO3104	13.089		0.10	
494	D445	13.12		0.15	
608	D445	13.04		0.01	
657	D445	13.08		0.08	
663	D445	13.01		-0.04	
710	D445	13.072		0.07	
736	D445	13.02		-0.02	
750		-----		-----	
752	D445	13.14		0.19	
753	D445	13.08		0.08	
778		-----		-----	
781	D445	13.05		0.03	
785	D445	12.99		-0.08	
798		-----		-----	
823	D445	13.00		-0.06	
872	D445	13.05		0.03	
873	D445	13.04		0.01	
874	D445	13.04		0.01	
875	D445	13.13		0.17	
994	D445	13.01		-0.04	
995	D445	13.03		-0.01	
1016		-----		-----	
1065	D445	12.829	C	-0.36	first reported 37.002
1081		-----		-----	
1108	D445	12.470	C,R(0.01)	-1.00	first reported 16.285
1191	ISO3104	12.931		-0.18	
1205	ISO3104	12.952255		-0.14	
1556	ISO3104	12.062	R(0.01)	-1.73	
1586	ISO3104	12.99		-0.08	
1635	D445	12.50	R(0.01)	-0.95	
1743	D7279 corrected to D445	13.5	R(0.01)	0.83	
1857	D445	13.008		-0.05	
1862	D445	13.116		0.15	
6026	ISO3104	12.9984		-0.06	
6054	D445	13.087		0.10	
6112		-----		-----	
6114	D445	13.09		0.10	
6195	D445	12.59	R(0.01)	-0.79	
6201	D445	13.04		0.01	
6203	D7042	13.261		0.41	
6262	D445	12.981		-0.09	
6447		-----		-----	
6496	ISO3104	12.96		-0.13	
normality					
n					
outliers					
mean (n)					
st.dev. (n)					
R(calc.)					
st.dev.(D445:21e2)					
R(D445:21e2)					



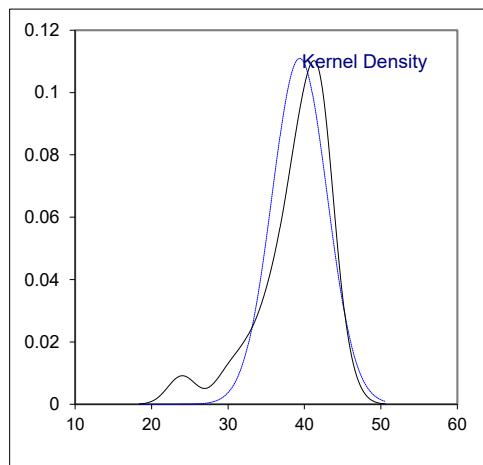
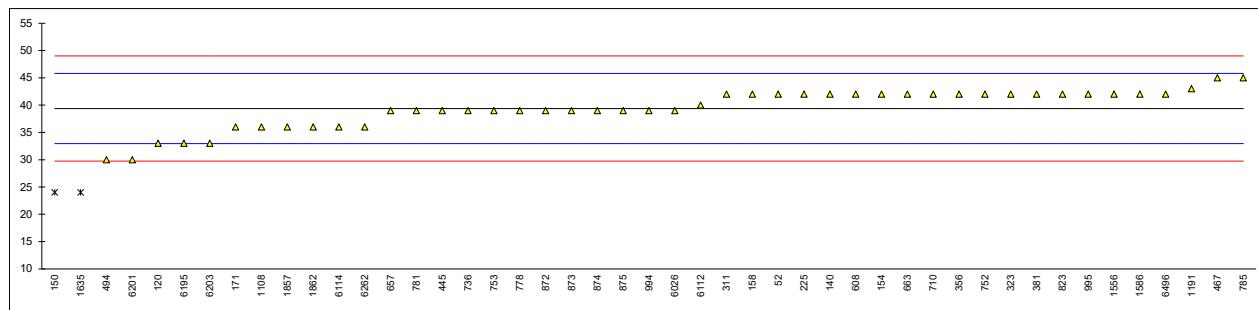
## Determination of Nitrogen on sample #22235; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	D4629	1800		0.49	
62		----		----	
120		----		----	
140		----		----	
150	D5762 Gravimetric	1600		-0.73	
154		----		----	
158	D5762 Gravimetric	2000		1.71	
159		----		----	
171	D5762 Gravimetric	1800		0.49	
225		----		----	
311	D5762 Volumetric	1800		0.49	
313		----		----	
323		----		----	
333	D5762 Volumetric	1700		-0.12	
356		----		----	
381		----		----	
445	D5762 Gravimetric	1840		0.74	
467	D5762 Gravimetric	1710		-0.06	
494	D5762 Gravimetric	1630		-0.55	
608	D5762 Gravimetric	1506.21		-1.31	
657	D5762 Gravimetric	1600		-0.73	
663	D5762 Gravimetric	1800		0.49	
710		----		----	
736	D5762 Volumetric	1692.52		-0.17	
750		----		----	
752		----		----	
753		----		----	
778		----		----	
781	D5762 Gravimetric	1854		0.82	
785		----		----	
798		----		----	
823	D5762 Gravimetric	1530		-1.16	
872		----		----	
873	D4629 Volumetric	1705		-0.09	
874	D5762 Gravimetric	1700		-0.12	
875	D5762	1692		-0.17	
994	D5762 Volumetric	1815		0.58	
995		----		----	
1016	D5762 Gravimetric	1673.08		-0.29	
1065	D4629	1700		-0.12	
1081	D4629	1730		0.06	
1108		----		----	
1191	D5762 Gravimetric	1785		0.40	
1205		----		----	
1556	D5762 Gravimetric	1730.242		0.06	
1586	D5762 Volumetric	1633		-0.53	
1635	D5762 Gravimetric	2077	R(0.05)	2.19	
1743		----		----	
1857	D5762 Gravimetric	1680		-0.24	
1862	D5762 Gravimetric	1653		-0.41	
6026	D5762 Gravimetric	1714.56		-0.03	
6054		----		----	
6112	D4629	1600		-0.73	
6114		----		----	
6195	D4629	1698.5		-0.13	
6201	D5762 Gravimetric	1629.87		-0.55	
6203	D5762 Gravimetric	1796		0.47	
6262	D5762 Gravimetric	1806		0.53	
6447	D4629	1790		0.43	
6496		1799.5		0.49	
	normality	OK			
	n	35			
	outliers	1			
	mean (n)	1719.81			
	st.dev. (n)	99.239			
	R(calc.)	277.87			
	st.dev.(D5762:18a)	163.382			
	R(D5762:18a)	457.47			



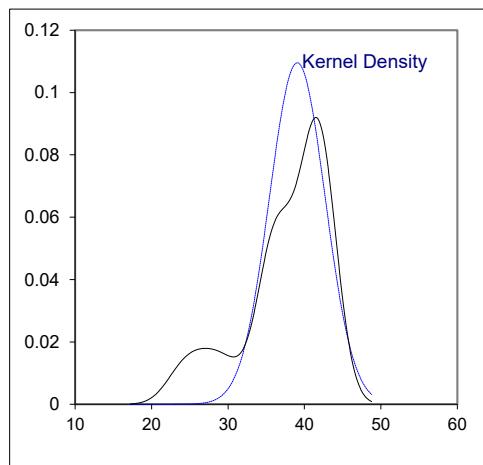
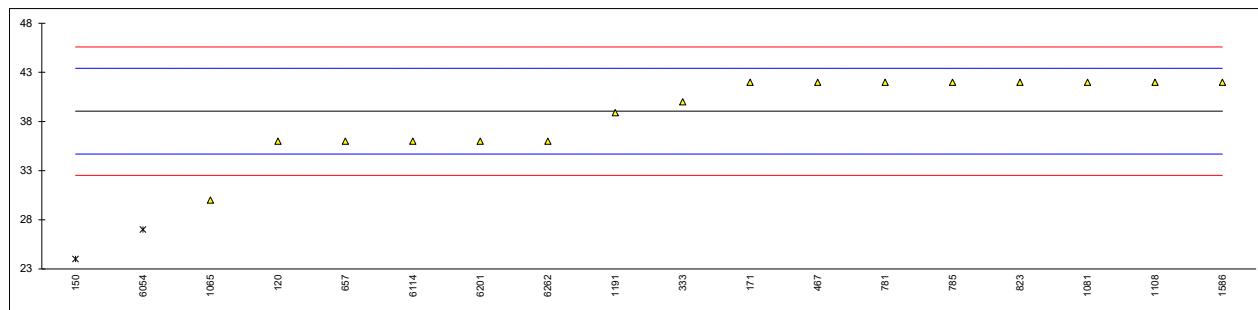
## Determination of Pour Point Manual on sample #22235; results in °C

lab	method	value	mark	z(targ)	remarks
52	D97	42		0.82	
62		----		----	
120	D97	33		-1.98	
140	D97	42		0.82	
150	D97	24	C,R(0.01)	-4.78	first reported 21
154	D97	42		0.82	
158	D97	42		0.82	
159		----		----	
171	D97	36		-1.05	
225	D97	42		0.82	
311	D97	42		0.82	
313		----		----	
323	D97	42		0.82	
333		----		----	
356	D97	42		0.82	
381	ISO3016	42		0.82	
445	D97	39		-0.12	
467	ISO3016	45		1.75	
494	D97	30.0		-2.92	
608	D97	42		0.82	
657	D97	39		-0.12	
663	D97	42	C	0.82	first reported 30
710	D97	42		0.82	
736	D97	39		-0.12	
750		----		----	
752	D97	42		0.82	
753	D97	39		-0.12	
778	D97	39		-0.12	
781	D97	39		-0.12	
785	D97	45.0		1.75	
798		----		----	
823	ISO3016	42		0.82	
872	D97	39		-0.12	
873	D97	39		-0.12	
874	D97	39		-0.12	
875	D97	39		-0.12	
994	D97	39		-0.12	
995	D97	42		0.82	
1016		----		----	
1065		----		----	
1081		----		----	
1108	D97	36		-1.05	
1191	ISO3016	43		1.13	
1205		----		----	
1556	ISO3016	42.0		0.82	
1586	ISO3016	42		0.82	
1635	D97	24	R(0.01)	-4.78	
1743		----		----	
1857	D97	36		-1.05	
1862	D97	36	C	-1.05	first reported 30
6026	ISO3016	39		-0.12	
6054		----		----	
6112	D97	40		0.19	
6114	D97	36		-1.05	
6195	D97	33		-1.98	
6201	D97	30		-2.92	
6203	D97	33		-1.98	
6262	D97	36.0		-1.05	
6447		----		----	
6496		42		0.82	
normality		OK			
n		45			
outliers		2			
mean (n)		39.38			
st.dev. (n)		3.595			
R(calc.)		10.07			
st.dev.(D97:17bR22)		3.214			
R(D97:17bR22)		9			



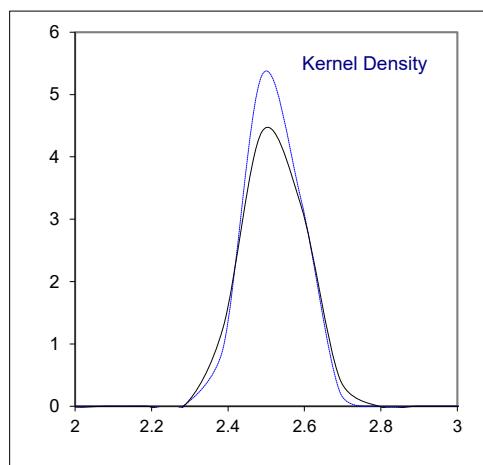
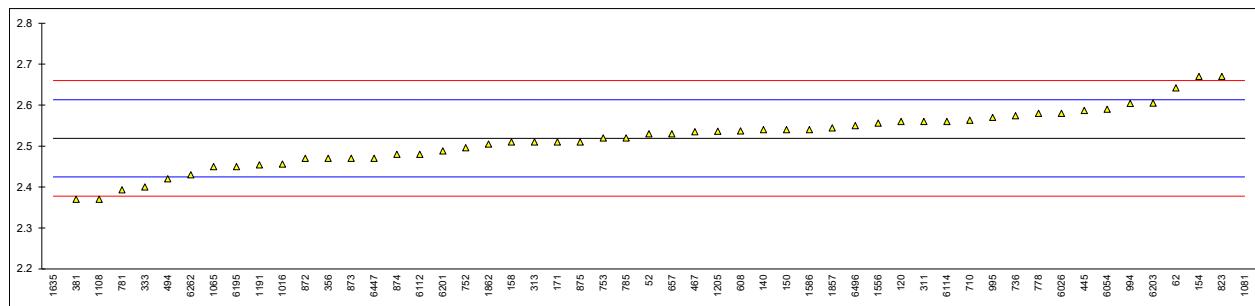
## Determination of Pour Point Automated 3 °C interval on sample #22235; results in °C

lab	method	value	mark	z(targ)	remarks
52		----		----	
62		----		----	
120	D5949	36		-1.40	
140		----		----	
150	D5950	24	C,DG(0.05)	-6.91	first reported 19
154		----		----	
158		----		----	
159		----		----	
171	D5950	42		1.35	
225		----		----	
311		----		----	
313		----		----	
323		----		----	
333	D5950	40		0.43	
356		----		----	
381		----		----	
445		----		----	
467	D6892	42		1.35	
494		----		----	
608		----		----	
657	D5950	36		-1.40	
663		----		----	
710		----		----	
736		----		----	
750		----		----	
752		----		----	
753		----		----	
778		----		----	
781	D5950	42		1.35	
785	D6749	42.0		1.35	
798		----		----	
823	D5950	42		1.35	
872		----		----	
873		----		----	
874		----		----	
875		----		----	
994		----		----	
995		----		----	
1016		----		----	
1065	D5950	30		-4.16	
1081	D5950	42		1.35	
1108	D5950	42		1.35	
1191	D5950	38.9		-0.07	
1205		----		----	
1556		----		----	
1586	D5950	42		1.35	
1635		----		----	
1743		----		----	
1857		----		----	
1862		----		----	
6026		----		----	
6054	D5950	27.0	DG(0.05)	-5.53	
6112		----		----	
6114	D5950	36		-1.40	
6195		N/A		----	
6201	D5950	36		-1.40	
6203		----		----	
6262	D5950	36.0		-1.40	
6447		----		----	
6496		----		----	
normality		OK			
n		16			
outliers		2			
mean (n)		39.06			
st.dev. (n)		3.642			
R(calc.)		10.20			
st.dev.(D5950:14R20)		2.179			
R(D5950:14R20)		6.1			



## Determination of Total Sulfur on sample #22235; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	D4294	2.53		0.24	
62	D4294	2.642		2.62	
120	D4294	2.56		0.88	
140	D4294	2.54		0.45	
150	D4294	2.54		0.45	
154	D4294	2.67		3.21	
158	D4294	2.51		-0.19	
159		----		----	
171	D4294	2.51	C	-0.19	first reported 2.19
225		----		----	
311	ISO8754	2.56		0.88	
313	ISO8754	2.51		-0.19	
323		----		----	
333	D4294	2.40		-2.52	
356	D4294	2.47		-1.04	
381	ISO8754	2.37		-3.16	
445	D4294	2.587		1.45	
467	D4294	2.535		0.35	
494	D4294	2.420		-2.10	
608	D4294	2.537		0.39	
657	D4294	2.53		0.24	
663		----		----	
710	D4294	2.563		0.94	
736	D4294	2.574		1.17	
750		----		----	
752	D4294	2.496		-0.48	
753	D4294	2.52		0.03	
778	D4294	2.58		1.30	
781	D4294	2.393		-2.67	
785	D4294	2.52		0.03	
798		----		----	
823	ISO8754	2.67		3.21	
872	D4294	2.47		-1.04	
873	D4294	2.47		-1.04	
874	D4294	2.48		-0.82	
875	D4294	2.51		-0.19	
994	D4294	2.604		1.81	
995	D4294	2.57		1.09	
1016	In house	2.456		-1.33	
1065	D4294	2.45		-1.46	
1081	D4294	27.66	C,R(0.01)	534.13	first reported 2.77
1108	D4294	2.370	C	-3.16	first reported 2.766
1191	ISO8754	2.454		-1.38	
1205	ISO14596	2.536		0.37	
1556	ISO8754	2.556		0.79	
1586	ISO8754	2.54		0.45	
1635	D4294	1.86	R(0.01)	-13.99	
1743		----		----	
1857	D4294	2.544		0.54	
1862	D4294	2.505		-0.29	
6026	IP336	2.58		1.30	
6054	D4294	2.59		1.51	
6112	D4294	2.48		-0.82	
6114	D4294	2.56		0.88	
6195	D4294	2.45		-1.46	
6201	D4294	2.488		-0.65	
6203	D4294	2.605		1.83	
6262	D2622	2.43		-1.89	
6447	D2622	2.47		-1.04	
6496	ISO8754	2.55		0.66	
normality					
n					
outliers					
mean (n)					
st.dev. (n)					
R(calc.)					
st.dev.(D4294:21)					
R(D4294:21)					



## Determination of Simulated Distillation on sample #22235; result in °C

lab	method	IBP	10%	30%	50%	70%	90%	FBP
52	D6352	276.5	C	395.0	C	443.0	C	472.5
62		----	----	----	----	----	----	----
120		----	----	----	----	----	----	----
140		----	----	----	----	----	----	----
150	D2887	286.0		395.5		445.0		475.5
154		----	----	----	----	----	----	----
158		----	----	----	----	----	----	----
159		----	----	----	----	----	----	----
171	D6352	290.0		398.5		447.5		477.0
225		----	----	----	----	----	----	----
311	D7169	292.5		397.5		446.0		476.5
313		----	----	----	----	----	----	----
323	D6352	255.8	G(1)	393.9		445.5		475.3
333		----	----	----	----	----	----	----
356		----	----	----	----	----	----	----
381		----	----	----	----	----	----	----
445	D7169	291.7		401.0		449.5		479.1
467		----	----	----	----	----	----	----
494		----	----	----	----	----	----	----
608		----	----	----	----	----	----	----
657	D6352	286.5		396.5		446.5		476.5
663		----	----	----	----	----	----	----
710		----	----	----	----	----	----	----
736		----	----	----	----	----	----	----
750		----	----	----	----	----	----	----
752		----	----	----	----	----	----	----
753		----	----	----	----	----	----	----
778		----	----	----	----	----	----	----
781	D6352	298.4		400.7		447.1		480.0
785		----	----	----	----	----	----	----
798		----	----	----	----	----	----	----
823		----	----	----	----	----	----	----
872		----	----	----	----	----	----	----
873		----	----	----	----	----	----	----
874		----	----	----	----	----	----	----
875		----	----	----	----	----	----	----
994		----	----	----	----	----	----	----
995		----	----	----	----	----	----	----
1016	D7169	304.5		399.5		447.0		478.0
1065		289.6	ex	385.8	G(5)	434.8	G(1)	465.6
1081		290		398		447		477
1108		----	----	----	----	----	----	----
1191		----	----	----	----	----	----	----
1205		----	----	----	----	----	----	----
1556	ISO3924	291.3		403.8		453.4		481.4
1586		----	----	----	----	----	----	----
1635	D2887	232	G(1)	395		446		479
1743		----	----	----	----	----	----	----
1857		----	----	----	----	----	----	----
1862		----	----	----	----	----	----	----
6026		----	----	----	----	----	----	----
6054		----	----	----	----	----	----	----
6112		----	----	----	----	----	----	----
6114		----	----	----	----	----	----	----
6195		----	----	----	----	----	----	----
6201		290.5		398.0		448.5		480.0
6203	D6352	285.5		397.5		446.0		475.5
6262		----	----	----	----	----	----	----
6447	D2887Mod.	291.6		402.1		451.0		480.6
6496		----	----	----	----	----	----	----
	normality	not OK		OK		suspect		OK
	n	13		15		15		15
	outliers	2 +1ex		1		1		2
	mean (n)	290.38		398.17		447.27		477.59
	st.dev. (n)	6.604		2.836		2.538		2.429
	R(calc.)	18.49		7.94		7.11		6.80
	st.dev.(D6352:19e1)	17.536		2.536		2.107		2.286
	R(D6352:19e1)	49.1		7.1		5.9		6.4

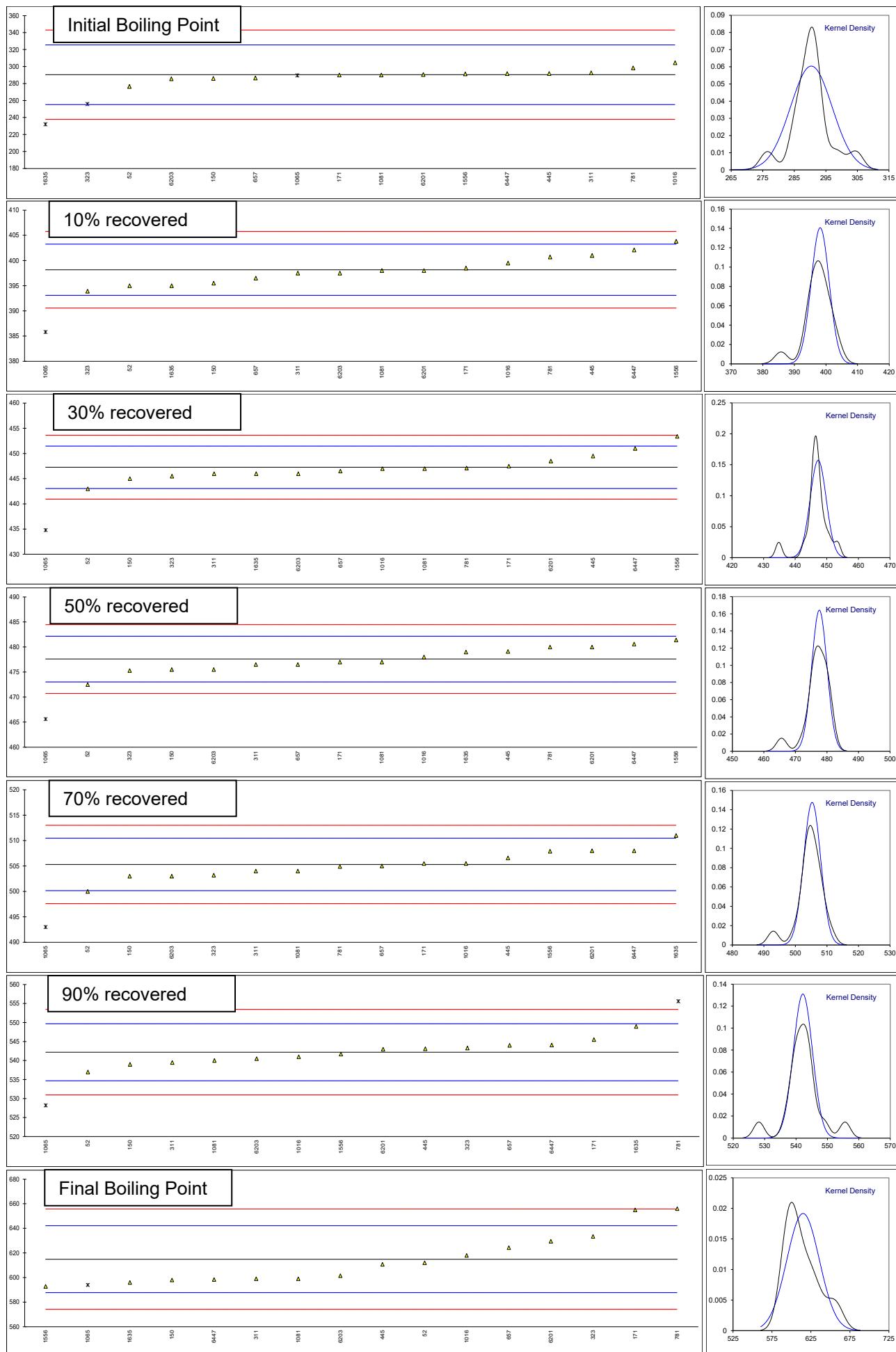
Lab 52 first reported respectively 262.5, 392.5, 442.0, 471.5, 499.5, 536.5, 608.0

Lab 657 first reported 653.5

Lab 1065 test results excluded for IBP and FBP as statistical outliers in related distillation parameters

## z-scores for Simulated Distillation

lab	IBP	10%	30%	50%	70%	90%	FBP
52	-0.79	-1.25	-2.02	-2.23	-2.06	-1.38	-0.21
62	----	----	----	----	----	----	----
120	----	----	----	----	----	----	----
140	----	----	----	----	----	----	----
150	-0.25	-1.05	-1.08	-0.92	-0.90	-0.85	-1.24
154	----	----	----	----	----	----	----
158	----	----	----	----	----	----	----
159	----	----	----	----	----	----	----
171	-0.02	0.13	0.11	-0.26	0.08	0.88	2.95
225	----	----	----	----	----	----	----
311	0.12	-0.26	-0.60	-0.48	-0.51	-0.72	-1.17
313	----	----	----	----	----	----	----
323	-1.97	-1.68	-0.84	-1.00	-0.82	0.30	1.36
333	----	----	----	----	----	----	----
356	----	----	----	----	----	----	----
381	----	----	----	----	----	----	----
445	0.08	1.12	1.06	0.66	0.50	0.24	-0.31
467	----	----	----	----	----	----	----
494	----	----	----	----	----	----	----
608	----	----	----	----	----	----	----
657	-0.22	-0.66	-0.36	-0.48	-0.12	0.48	0.69
663	----	----	----	----	----	----	----
710	----	----	----	----	----	----	----
736	----	----	----	----	----	----	----
750	----	----	----	----	----	----	----
752	----	----	----	----	----	----	----
753	----	----	----	----	----	----	----
778	----	----	----	----	----	----	----
781	0.46	1.00	-0.08	1.05	-0.16	3.58	3.02
785	----	----	----	----	----	----	----
798	----	----	----	----	----	----	----
823	----	----	----	----	----	----	----
872	----	----	----	----	----	----	----
873	----	----	----	----	----	----	----
874	----	----	----	----	----	----	----
875	----	----	----	----	----	----	----
994	----	----	----	----	----	----	----
995	----	----	----	----	----	----	----
1016	0.80	0.53	-0.13	0.18	0.08	-0.32	0.23
1065	-0.04	-4.88	-5.92	-5.25	-4.79	-3.73	-1.54
1081	-0.02	-0.07	-0.13	-0.26	-0.51	-0.58	-1.17
1108	----	----	----	----	----	----	----
1191	----	----	----	----	----	----	----
1205	----	----	----	----	----	----	----
1556	0.05	2.22	2.91	1.67	1.01	-0.13	-1.62
1586	----	----	----	----	----	----	----
1635	-3.33	-1.25	-0.60	0.62	2.21	1.82	-1.39
1743	----	----	----	----	----	----	----
1857	----	----	----	----	----	----	----
1862	----	----	----	----	----	----	----
6026	----	----	----	----	----	----	----
6054	----	----	----	----	----	----	----
6112	----	----	----	----	----	----	----
6114	----	----	----	----	----	----	----
6195	----	----	----	----	----	----	----
6201	0.01	-0.07	0.59	1.05	1.05	0.22	1.07
6203	-0.28	-0.26	-0.60	-0.92	-0.90	-0.45	-0.99
6262	----	----	----	----	----	----	----
6447	0.07	1.55	1.77	1.32	1.05	0.51	-1.21
6496	----	----	----	----	----	----	----



## Determination of Distillation at 10 mmHg as AET on sample #22235; result in °C

lab	method	IBP	10%	30%	50%	70%	90%	FBP
52		----	----	----	----	----	----	----
62		----	----	----	----	----	----	----
120		----	----	----	----	----	----	----
140		----	----	----	----	----	----	----
150	D1160	332	415	C	464	486	511	541
154		----	----	----	----	----	----	----
158		----	----	----	----	----	----	----
159		----	----	----	----	----	----	----
171	D1160	301	422		461	485	507	541
225		----	----	----	----	----	----	----
311	D1160	300	421		461	482	506	538
313		----	----	----	----	----	----	----
323		----	----	----	----	----	----	----
333		----	----	----	----	----	----	----
356	D1160	299	419		455	479	500	541
381		----	----	----	----	----	----	----
445	D1160	300.8	416.0		457.6	483.5	508.1	545.0
467	D1160	292	416		458	481	503	535
494	D1160	291.4	414.9		461.9	467.1	R(1)	508.0
608		----	----	----	----	----	----	----
657		----	----	----	----	----	----	----
663		----	----	----	----	----	----	----
710	D1160	292.8	416.5		458.6	480.0	503.6	536.7
736	D1160	309.3	417.4		462.3	484.4	504.5	541.0
750		----	----	----	----	----	----	----
752		----	----	----	----	----	----	----
753		----	----	----	----	----	----	----
778		----	----	----	----	----	----	----
781	D1160	274	407		450	476	500	535
785	D1160	272.3	417.7		462.8	488.2	512.2	----
798		----	----	----	----	----	----	----
823		----	----	----	----	----	----	----
872		----	----	----	----	----	----	----
873	D1160	281	410		453	479	503	535
874	D1160	278.9	412.4		455.4	482.2	505.0	537.1
875	D1160	281.0	409.0		450.5	479.0	504.7	536.7
994	D1160	296.0	415.0		457.0	480.0	504.0	538.0
995	D1160	297	C	410.0		452.0	478.0	502.0
1016		----	----	----	----	----	----	----
1065		----	----	----	----	----	----	----
1081		----	----	----	----	----	----	----
1108	D1160	312.6	415.4		457.6	479.4	502.0	C
1191		----	----	----	----	----	----	----
1205		----	----	----	----	----	----	----
1556		----	----	----	----	----	----	----
1586	D1160	324	402		449	477	500	528
1635	D1160	277.0	410.4		452.6	476.5	499.3	529.0
1743		----	----	----	----	----	----	----
1857	D1160	326.1	419.9		460.4	483.5	506.1	538.6
1862	D1160	300	420		460	482	505	536
6026	D1160	280	415		458	481	507	539
6054		----	----	----	----	----	----	----
6112	D1160	317.4	419.1		459.0	481.5	506.0	538.6
6114		----	----	----	----	----	----	----
6195	D1160	313.5	423.6		458.1	483.0	508.6	542.9
6201	D1160	307.4	430.1		457.3	481.0	505.6	541.9
6203	D1160	301.4	417.0		456.5	480.1	503.2	534.7
6262		----	----	----	----	----	----	----
6447		----	----	----	----	----	----	----
6496		----	----	----	----	----	----	----
normality		OK	suspect	OK	OK	OK	OK	OK
n		26	26	26	25	26	24	23
outliers		0	0	0	1	0	1	2
mean (n)		298.38	415.82	457.25	481.13	504.80	537.50	555.88
st.dev. (n)		16.440	5.726	4.092	2.971	3.286	3.963	4.095
R(calc.)		46.03	16.03	11.46	8.32	9.20	11.10	11.46
st.dev.(D1160:18)		17.660	6.573	4.062	3.568	2.958	3.196	9.605
R(D1160:18)		49.45	18.40	11.37	9.99	8.28	8.95	26.89

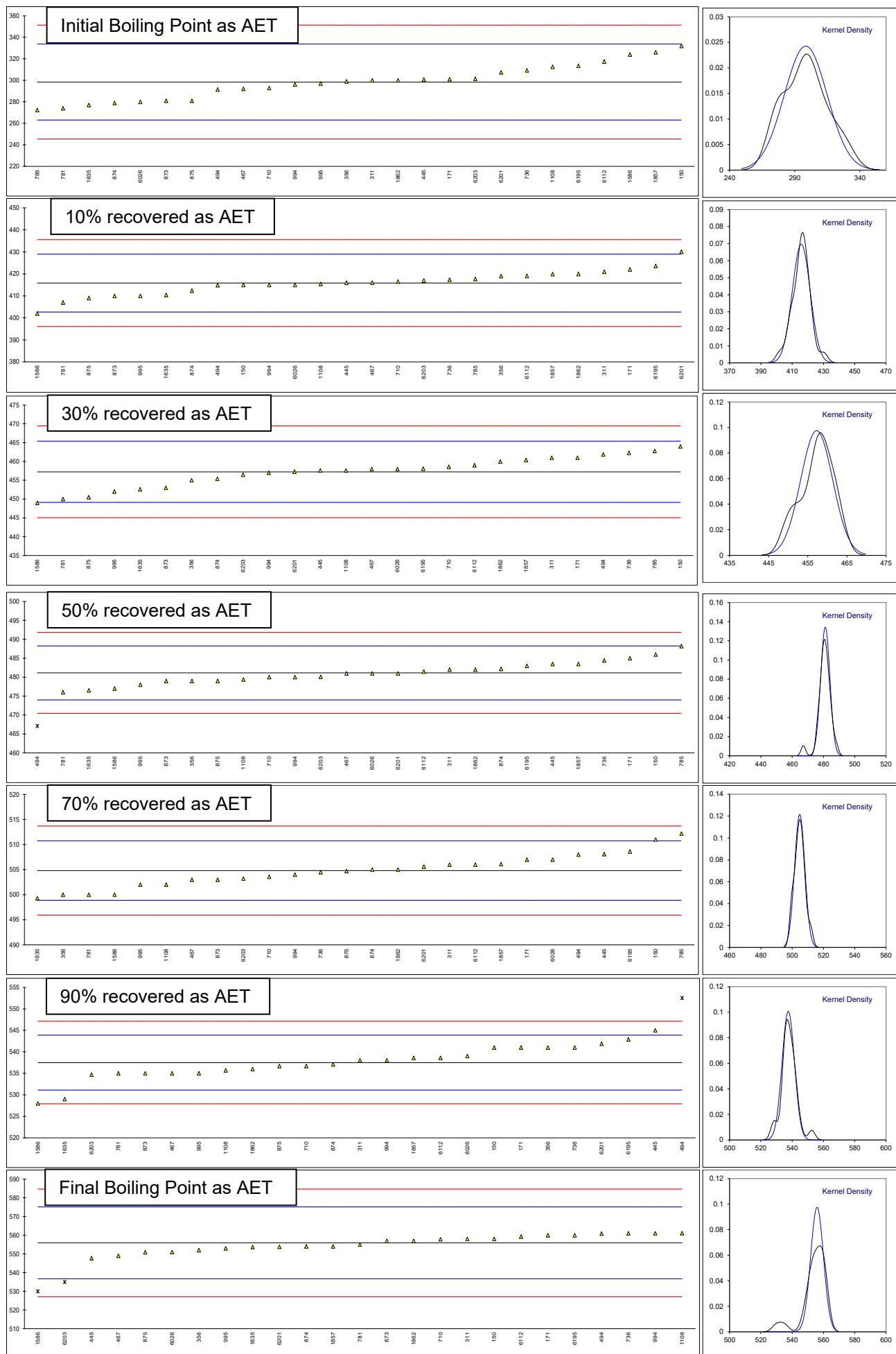
Lab 150 first reported 429

Lab 995 first reported 257.0

Lab 1108 first reported 349.5, 377.3 and 400.0 respectively

## z-scores Distillation at 10 mmHg as AET

lab	IBP	10%	30%	50%	70%	90%	FBP
52	----	----	----	----	----	----	----
62	----	----	----	----	----	----	----
120	----	----	----	----	----	----	----
140	----	----	----	----	----	----	----
150	1.90	-0.13	1.66	1.36	2.09	1.10	0.22
154	----	----	----	----	----	----	----
158	----	----	----	----	----	----	----
159	----	----	----	----	----	----	----
171	0.15	0.94	0.92	1.08	0.74	1.10	0.43
225	----	----	----	----	----	----	----
311	0.09	0.79	0.92	0.24	0.40	0.16	0.22
313	----	----	----	----	----	----	----
323	----	----	----	----	----	----	----
333	----	----	----	----	----	----	----
356	0.04	0.48	-0.55	-0.60	-1.62	1.10	-0.40
381	----	----	----	----	----	----	----
445	0.14	0.03	0.09	0.66	1.11	2.35	-0.85
467	-0.36	0.03	0.18	-0.04	-0.61	-0.78	-0.72
494	-0.40	-0.14	1.14	-3.93	1.08	4.69	0.52
608	----	----	----	----	----	----	----
657	----	----	----	----	----	----	----
663	----	----	----	----	----	----	----
710	-0.32	0.10	0.33	-0.32	-0.41	-0.25	0.20
736	0.62	0.24	1.24	0.92	-0.10	1.10	0.53
750	----	----	----	----	----	----	----
752	----	----	----	----	----	----	----
753	----	----	----	----	----	----	----
778	----	----	----	----	----	----	----
781	-1.38	-1.34	-1.79	-1.44	-1.62	-0.78	-0.09
785	-1.48	0.29	1.37	1.98	2.50	----	----
798	----	----	----	----	----	----	----
823	----	----	----	----	----	----	----
872	----	----	----	----	----	----	----
873	-0.98	-0.89	-1.05	-0.60	-0.61	-0.78	0.12
874	-1.10	-0.52	-0.46	0.30	0.07	-0.12	-0.20
875	-0.98	-1.04	-1.66	-0.60	-0.04	-0.25	-0.52
994	-0.13	-0.13	-0.06	-0.32	-0.27	0.16	0.53
995	-0.08	-0.89	-1.29	-0.88	-0.95	-0.78	-0.30
1016	----	----	----	----	----	----	----
1065	----	----	----	----	----	----	----
1081	----	----	----	----	----	----	----
1108	0.81	-0.06	0.09	-0.49	-0.95	-0.56	0.54
1191	----	----	----	----	----	----	----
1205	----	----	----	----	----	----	----
1556	----	----	----	----	----	----	----
1586	1.45	-2.10	-2.03	-1.16	-1.62	-2.97	-2.69
1635	-1.21	-0.83	-1.15	-1.30	-1.86	-2.66	-0.23
1743	----	----	----	----	----	----	----
1857	1.57	0.62	0.77	0.66	0.44	0.35	-0.20
1862	0.09	0.64	0.68	0.24	0.07	-0.47	0.12
6026	-1.04	-0.13	0.18	-0.04	0.74	0.47	-0.51
6054	----	----	----	----	----	----	----
6112	1.08	0.50	0.43	0.10	0.40	0.35	0.36
6114	----	----	----	----	----	----	----
6195	0.86	1.18	0.21	0.52	1.28	1.69	0.43
6201	0.51	2.17	0.01	-0.04	0.27	1.38	-0.22
6203	0.17	0.18	-0.19	-0.29	-0.54	-0.87	-2.17
6262	----	----	----	----	----	----	----
6447	----	----	----	----	----	----	----
6496	----	----	----	----	----	----	----



Determination of Aluminum as Al, Silicon as Si and sum Al and Si on sample #22236; results in mg/kg

lab	method	Al	mark	z(targ)	Si	mark	z(targ)	Sum Al+Si	mark	z(targ)
52	IP501	10		0.07	<10		----	15		0.32
62	IP501	10.4		0.41	7		4.36	17		1.84
120	IP501	9.827		-0.07	3.177		-2.63	13.004		-1.20
150	IP501	<5	f?	<-4.12	<10		----	<15		----
154	IP501	11		0.91	<10		----	<15		----
158	IP501	8	C	-1.61	<10		----	----		----
171	IP501	9		-0.77	2		-4.78	12		-1.97
225	IP501	10.80		0.74	4.51		-0.19	16		1.08
311		----		----	----		----	----		----
323	IP501	11		0.91	< 10		----	< 21		----
333	IP501	10		0.07	6		2.54	16		1.08
356		----		----	----		----	----		----
381	D5185	9.5		-0.35	----		----	----		----
445		----		----	----		----	----		----
494	IP501	11		0.91	2		-4.78	13		-1.21
608	IP501	9		-0.77	4		-1.12	13		-1.21
657	IP501	6.0	C,R(1)	-3.28	<10	C	----	6.0	C,R(1)	-6.54
736	IP501	9.94		0.02	4.1		-0.94	----		----
752		----		----	----		----	----		----
778		----		----	----		----	----		----
781	IP501	9.9		-0.01	4.9		0.53	14.8		0.17
785	IP470	9.7		-0.18	5.4		1.44	15.1		0.39
823	IP501	8.8		-0.94	3.2		-2.58	12		-1.97
873	IP470	10.6		0.57	5.4		1.44	16		1.08
874	IP501	10.4		0.41	5.3		1.26	15		0.32
875	IP501	10.6		0.57	3.6		-1.85	14.2		-0.29
994	IP501	10.50		0.49	<10		----	----		----
995	IP470	11		0.91	7		4.36	----		----
1016		----		----	----		----	----		----
1065		----		----	----		----	----		----
1191	IP501	9.46		-0.38	4.55		-0.11	----		----
1556		----		----	----		----	----		----
1586	IP470	11		0.91	4		-1.12	15		0.32
1635		8.44		-1.24	11.8	R(1)	13.14	20.2	ex	4.28
1743	NF T60-106	8.6		-1.10	10.4	R(1)	10.58	19	ex	3.36
1857	IP501	11.0		0.91	4.3		-0.57	15.3		0.55
1862	IP501	9.9		-0.01	3.9		-1.30	13.8		-0.60
6026	IP470	9.5071		-0.34	----		----	----		----
6054		----		----	4.86298		0.46	----		----
6114		----		----	----		----	----		----
6195		----		----	----		----	----		----
6201	IP501	9.039		-0.74	6.888		4.16	16		1.08
6203	IP501	10.78		0.72	3.75		-1.58	14.53		-0.04
6262	IP501	9.2		-0.60	5.4		1.44	14.6		0.01
6496	IP470	9.43		-0.41	5.47		1.57	14.9		0.24
normality		OK		OK			OK			
n		32		24			21			
outliers		1		2			1 +2ex			
mean (n)		9.916		4.613			14.583			
st.dev. (n)		0.8607		1.3645			1.3590			
R(calc.)		2.410		3.821			3.805			
st.dev.(IP501:05R19)		1.1935		0.5470			1.3129			
R(IP501:05R19)		3.342		1.531			3.676			

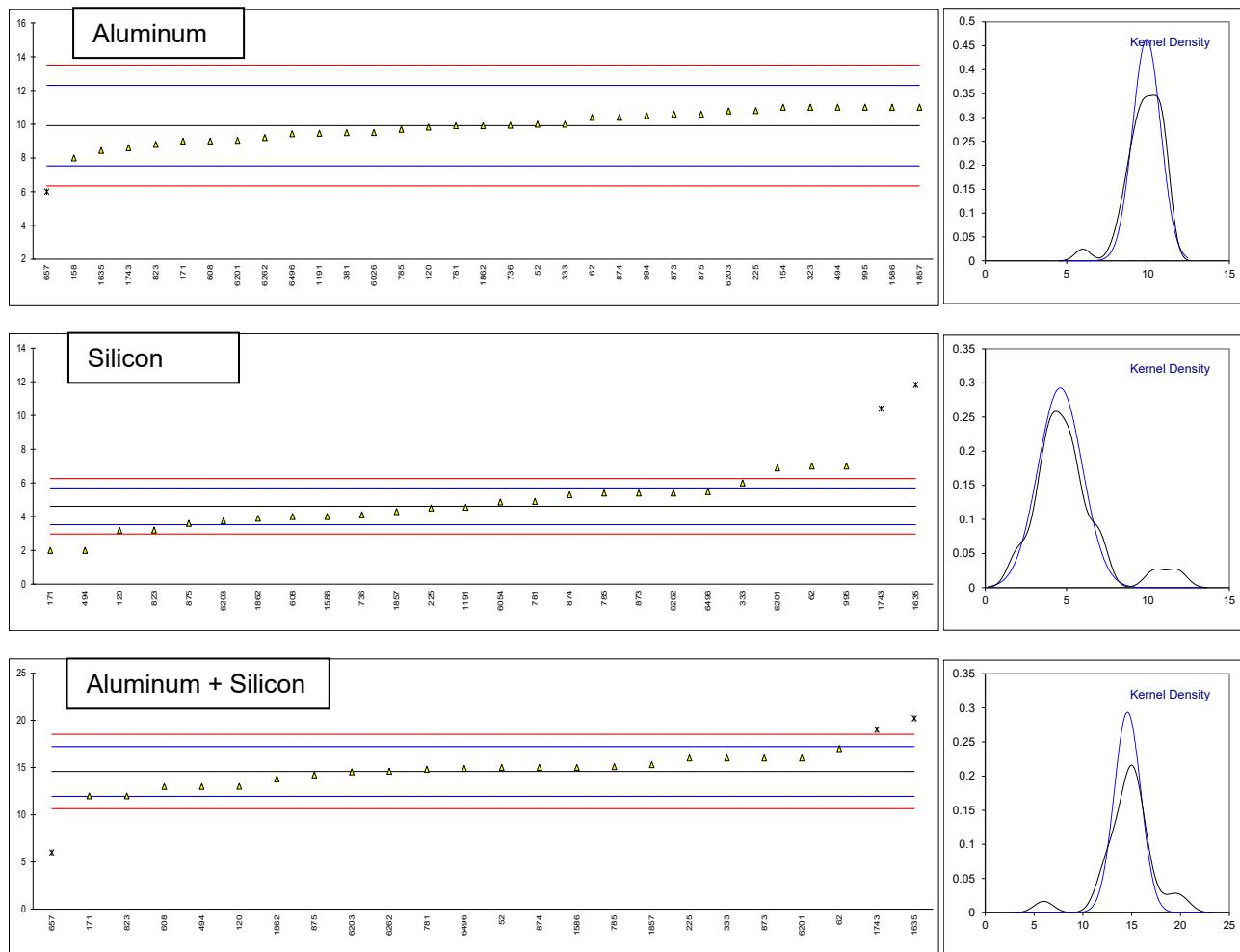
Lab 150 possibly a false negative test result?

Lab 158 first reported 5

Lab 657 first reported 0.3, 1.0 and 1.3 respectively

Lab 1635 excluded as statistical outlier in Si

Lab 1743 excluded as statistical outlier in Si



## Determination of Arsenic as As on sample #22236; results in mg/kg

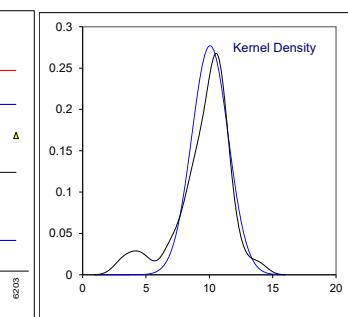
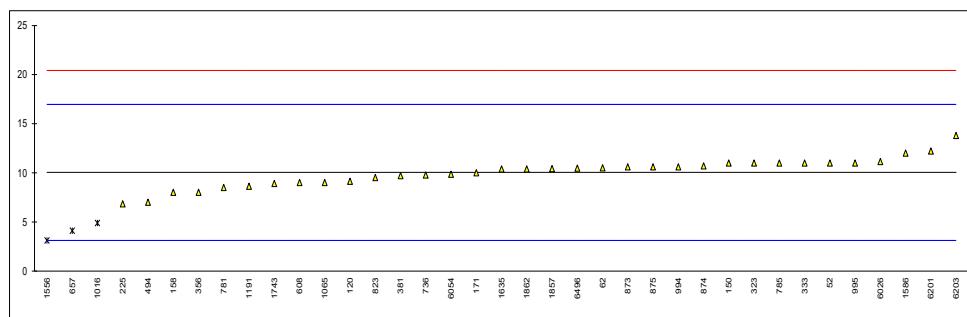
lab	method	value	mark	z(targ)	remarks
52		----		----	
62		----		----	
120	IP501	0.00		----	
150	IP501	<1		----	
154		----		----	
158		----		----	
171	IP501	<1		----	
225		----		----	
311		----		----	
323		----		----	
333		----		----	
356		----		----	
381	INH-118	0.037		----	
445		----		----	
494		----		----	
608	IP501	0		----	
657		----		----	
736	UOP992	0.042		----	
752		----		----	
778		----		----	
781		----		----	
785		----		----	
823	IP501	<1		----	
873		----		----	
874		----		----	
875		----		----	
994		----		----	
995		----		----	
1016		----		----	
1065		----		----	
1191		----		----	
1556		----		----	
1586		----		----	
1635		----		----	
1743		----		----	
1857	UOP986	<0.050		----	
1862		----		----	
6026		----		----	
6054		----		----	
6114		----		----	
6195		----		----	
6201		----		----	
6203		----		----	
6262		----		----	
6496		----		----	
n		8			
mean (n)		<1			

## Determination of Copper as Cu on sample #22236; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52		----		----	
62		----		----	
120	IP501	0.093		----	
150	IP501	<1		----	
154		----		----	
158	IP501	<1		----	
171	IP501	<1		----	
225		----		----	
311	IP621	<1		----	
323	IP501	< 1		----	
333		----		----	
356	IP621	0.2		----	
381	INH-118	0.111		----	
445		----		----	
494	IP501	<1		----	
608	IP501	0		----	
657	IP501	<1		----	
736	IP621	0.14		----	
752		----		----	
778		----		----	
781	IP621	0.1		----	
785	IP470	0.83		----	
823	IP501	<1		----	
873	IP621	0.1		----	
874	IP621	0.1		----	
875	IP501	0.5		----	
994	IP501	<1		----	
995	IP621	0.3		----	
1016		----		----	
1065		----		----	
1191	In house	0.10		----	
1556	IP621	0.01		----	
1586	IP470	0.05		----	
1635		0.17		----	
1743	NF T60-106	0.1		----	
1857	IP621	0.10		----	
1862	IP501	<0.1		----	
6026		----		----	
6054	IP470	0.104246		----	
6114		----		----	
6195		----		----	
6201	IP621	0.155		----	
6203		----		----	
6262		----		----	
6496	IP621	0.17		----	
n		30			
mean (n)		<1			

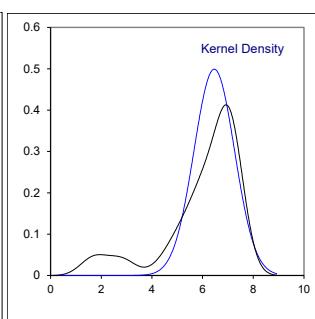
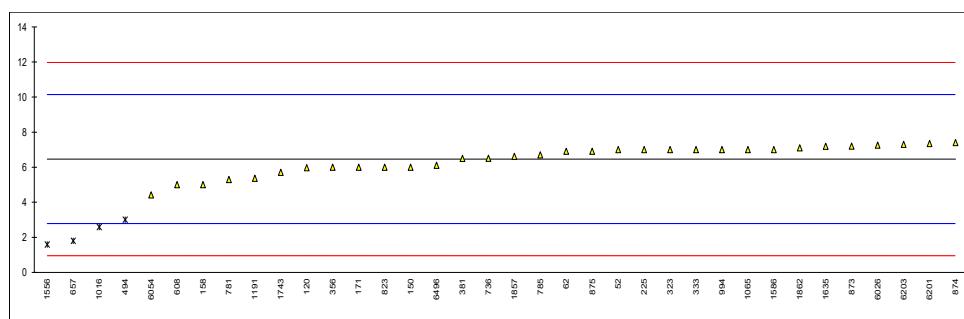
## Determination of Iron as Fe on sample #22236; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	IP501	11		0.27	
62	IP501	10.5		0.13	
120	IP501	9.141		-0.26	
150	IP501	11	C	0.27	first reported 5
154		----		----	
158	IP501	8	C	-0.59	first reported 5
171	IP501	10		-0.01	
225	IP501	6.84		-0.93	
311		----		----	
323	IP501	11		0.27	
333	IP501	11		0.27	
356	IP621	8		-0.59	
381	D5185	9.7		-0.10	
445		----		----	
494	IP501	7		-0.88	
608	IP501	9		-0.30	
657	IP501	4.1	C,R(0.05)	-1.72	first reported <2
736	IP621	9.78		-0.08	
752		----		----	
778		----		----	
781	IP621	8.5		-0.45	
785	IP470	11.0		0.27	
823	IP501	9.51		-0.16	
873	IP470	10.6		0.16	
874	IP501	10.7		0.19	
875	IP501	10.6		0.16	
994	IP501	10.6		0.16	
995	IP470	11		0.27	
1016	In house	4.886	R(0.05)	-1.49	
1065	IP470	9.0		-0.30	
1191	ISO10478	8.62		-0.41	
1556	IP621	3.11	R(0.05)	-2.01	
1586	IP470	12		0.56	
1635	D5708	10.4		0.10	
1743	NF T60-106	8.9		-0.33	
1857	IP621	10.42		0.11	
1862	IP501	10.4		0.10	
6026	IP470	11.1336		0.31	
6054	IP470	9.84815		-0.06	
6114		----		----	
6195		----		----	
6201	IP621	12.201		0.62	
6203	IP501	13.79		1.08	
6262		----		----	
6496	IP470	10.47		0.12	
normality					
n		OK			
outliers		34			
mean (n)		3			
st.dev. (n)		10.049			
R(calc.)		1.4401			
st.dev.(IP621:16)		4.032			
R(IP621:16)		3.4599			
		9.688			



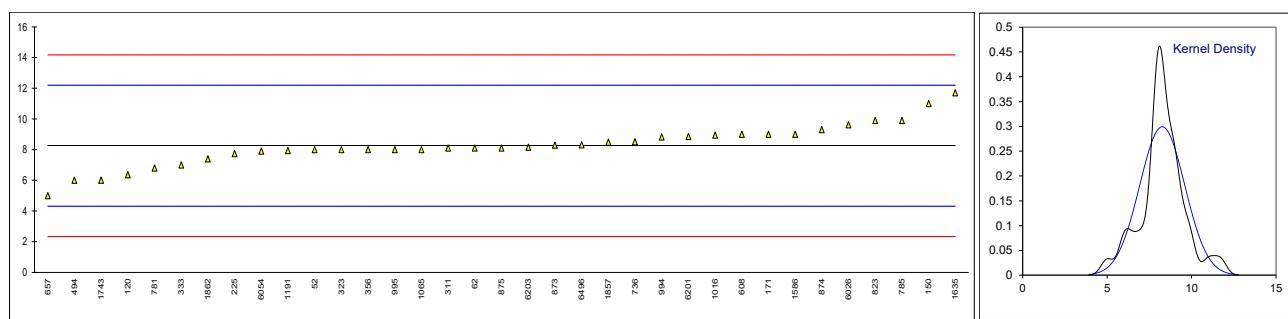
## Determination of Nickel as Ni on sample #22236; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	IP501	7		0.29	
62	IP501	6.9		0.24	
120	IP501	5.962		-0.27	
150	IP501	6	C	-0.25	first reported 3
154		----		----	
158	IP501	5	C	-0.79	first reported 3
171	IP501	6		-0.25	
225	IP501	7.00		0.29	
311		----		----	
323	IP501	7		0.29	
333	IP501	7		0.29	
356	IP621	6		-0.25	
381	D5185	6.5		0.02	
445		----		----	
494	IP501	3	R(0.01)	-1.88	
608	IP501	5		-0.79	
657	IP501	1.8	C,R(0.05)	-2.54	first reported <1
736	IP621	6.51		0.03	
752		----		----	
778		----		----	
781	IP621	5.3		-0.63	
785	IP470	6.7		0.13	
823	IP501	6.0		-0.25	
873	IP621	7.2		0.40	
874	IP501	7.4		0.51	
875	IP501	6.9		0.24	
994	IP501	7.0		0.29	
995		----		----	
1016	In house	2.576	R(0.05)	-2.11	
1065	IP470	7.0		0.29	
1191	ISO10478	5.36		-0.60	
1556	IP621	1.59	R(0.05)	-2.65	
1586	IP470	7		0.29	
1635	D5708	7.19		0.40	
1743	NF T60-108	5.7		-0.41	
1857	IP621	6.62		0.09	
1862	IP501	7.1		0.35	
6026	IP470	7.2571		0.43	
6054	IP470	4.41684		-1.11	
6114		----		----	
6195		----		----	
6201	IP621	7.346		0.48	
6203	IP501	7.30		0.46	
6262		----		----	
6496	IP621	6.11		-0.19	
<hr/>					
normality					
n		OK			
outliers		32			
mean (n)		4			
st.dev. (n)		6.462			
R(calc.)		0.7993			
st.dev.(IP621:16)		2.238			
R(IP621:16)		1.8385			
		5.148			



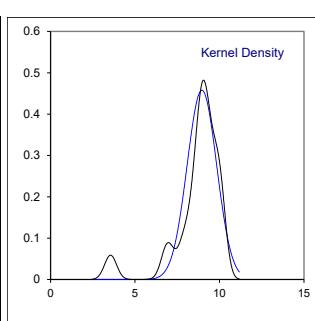
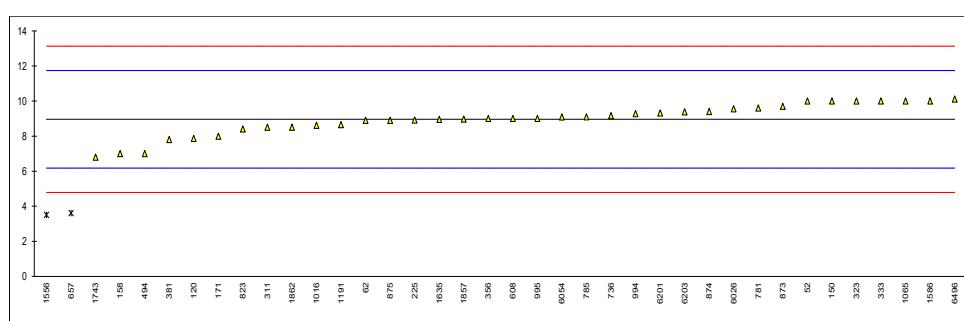
## Determination of Sodium as Na on sample #22236; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	IP501	8		-0.13	
62	IP501	8.1		-0.08	
120	IP501	6.359		-0.97	
150	IP501	11	C	1.39	first reported 5
154		----		----	
158		----		----	
171	IP501	9		0.37	
225	IP501	7.74		-0.27	
311	IP621	8.1		-0.08	
323	IP501	8		-0.13	
333	IP501	7		-0.64	
356	IP621	8		-0.13	
381		----		----	
445		----		----	
494	IP501	6		-1.15	
608	IP501	9		0.37	
657	IP501	5.0	C	-1.66	first reported 1.1
736	IP621	8.51		0.12	
752		----		----	
778		----		----	
781	IP621	6.8		-0.74	
785	IP470	9.9		0.83	
823	IP501	9.9		0.83	
873	IP621	8.3		0.02	
874	IP501	9.3		0.52	
875	IP501	8.1		-0.08	
994	IP501	8.83		0.29	
995	IP470	8		-0.13	
1016	NEN6966	8.944		0.34	
1065	IP470	8.0		-0.13	
1191	ISO10478	7.94		-0.16	
1556		----		----	
1586	IP470	9		0.37	
1635	D5708	11.7		1.74	
1743	NF T60-106	6.0		-1.15	
1857	IP621	8.49		0.11	
1862	IP501	7.4		-0.44	
6026	IP470	9.6105		0.68	
6054	IP470	7.91345		-0.18	
6114		----		----	
6195		----		----	
6201	IP621	8.859		0.30	
6203	IP501	8.15		-0.06	
6262		----		----	
6496	IP621	8.32		0.03	
normality					
n		35			
outliers		0			
mean (n)		8.265			
st.dev. (n)		1.3326			
R(calc.)		3.731			
st.dev.(IP621:16)		1.9720			
R(IP621:16)		5.522			



## Determination of Vanadium as V on sample #22236; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	IP501	10		0.75	
62	IP501	8.9		-0.04	
120	IP501	7.869		-0.78	
150	IP501	10	C	0.75	first reported 4
154		----		----	
158	IP501	7	C	-1.41	first reported 5
171	IP501	8		-0.69	
225	IP501	8.92		-0.03	
311	IP621	8.5		-0.33	
323	IP501	10		0.75	
333	IP501	10		0.75	
356	IP621	9		0.03	
381	D5185	7.8		-0.83	
445		----		----	
494	IP501	7		-1.41	
608	IP501	9		0.03	
657	IP501	3.6	C,R(0.01)	-3.85	first reported <1
736	IP621	9.16		0.15	
752		----		----	
778		----		----	
781	IP621	9.6		0.46	
785	IP470	9.1		0.10	
823	IP501	8.4		-0.40	
873	IP470	9.7		0.53	
874	IP501	9.4		0.32	
875	IP501	8.9		-0.04	
994	IP501	9.28		0.23	
995	IP470	9		0.03	
1016	In house	8.616		-0.25	
1065	IP470	10.0		0.75	
1191	ISO10478	8.65		-0.22	
1556	IP621	3.50	R(0.01)	-3.92	
1586	IP470	10		0.75	
1635	D5708	8.95		-0.01	
1743	NF T60-106	6.8		-1.55	
1857	IP621	8.97		0.01	
1862	IP501	8.5		-0.33	
6026	IP470	9.5545		0.43	
6054	IP470	9.08518		0.09	
6114		----		----	
6195		----		----	
6201	IP621	9.317		0.26	
6203	IP501	9.38		0.30	
6262		----		----	
6496	IP621	10.11		0.83	
normality					
n		36			
outliers		2			
mean (n)		8.957			
st.dev. (n)		0.8713			
R(calc.)		2.440			
st.dev.(IP621:16)		1.3907			
R(IP621:16)		3.894			



## Determination of Calcium as Ca on sample #22236; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52	IP501	<3		----	
62	IP501	0.8		----	
120	IP501	0.00		----	
150	IP501	<3		----	
154		----		----	
158	IP501	<3		----	
171	IP501	<1		----	
225	IP501	0.52		----	
311		----		----	
323	IP501	< 3		----	
333	IP501	<3		----	
356		----		----	
381	D5185	<1		----	
445		----		----	
494	IP501	<1		----	
608	IP501	1		----	
657	IP501	<3		----	
736	IP501	0.44		----	
752		----		----	
778		----		----	
781	IP501	0.4		----	
785	IP470	1.5		----	
823	IP501	1.6		----	
873	IP470	0.6		----	
874	IP501	1.1		----	
875	IP501	0.6		----	
994	IP501	<3		----	
995	IP470	1		----	
1016	NEN6966	0.42		----	
1065		----		----	
1191	In house	0.47		----	
1556		----		----	
1586	IP470	2		----	
1635	D5708	0.47		----	
1743	NF T60-106	0.32		----	
1857	IP501	0.50		----	
1862	IP501	0.4		----	
6026		----		----	
6054	IP470	0.485675		----	
6114		----		----	
6195		----		----	
6201	IP501	0		----	
6203	IP501	1.7		----	
6262		----		----	
6496	IP621	<3		----	
n		33			
mean (n)		<3			

**APPENDIX 2****Number of participants per country**

1 lab in AZERBAIJAN  
3 labs in BELGIUM  
2 labs in CANADA  
2 labs in COTE D'IVOIRE  
1 lab in CROATIA  
1 lab in DENMARK  
1 lab in FINLAND  
2 labs in FRANCE  
1 lab in GEORGIA  
1 lab in GERMANY  
2 labs in GREECE  
1 lab in ISRAEL  
2 labs in KAZAKHSTAN  
1 lab in KOREA, Republic of  
1 lab in MALAYSIA  
2 labs in MALTA  
6 labs in NETHERLANDS  
2 labs in ROMANIA  
14 labs in RUSSIAN FEDERATION  
1 lab in SERBIA  
1 lab in SINGAPORE  
2 labs in SWEDEN  
1 lab in THAILAND  
1 lab in UNITED ARAB EMIRATES  
1 lab 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) / D(1)	= outlier in Dixon's outlier test
D(0.05) / D(5)	= straggler in Dixon's outlier test
G(0.01) / G(1)	= outlier in Grubbs' outlier test
G(0.05) / G(5)	= straggler in Grubbs' outlier test
DG(0.01) / DG(1)	= outlier in Double Grubbs' outlier test
DG(0.05) / DG(5)	= straggler in Double Grubbs' outlier test
R(0.01) / R(1)	= outlier in Rosner's outlier test
R(0.05) / R(5)	= 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
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?
SDS	= Safety Data Sheet

**Literature**

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- 3 ISO5725 parts 1-6:94
- 4 ISO13528:05
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- 7 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
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- 9 Analytical Methods Committee, Technical Brief, No 4, January 2001
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- 11 W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
- 12 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)