

Results of Proficiency Test
Vacuum Gasoil
December 2014

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Report: iis14G06

April 2015

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1 INTRODUCTION

In 2014, a proficiency test was organized on request of several participants for Vacuum Gas Oil (VGO) by the Institute for Interlaboratory Studies. During the annual proficiency testing program 2014/2015, it was decided to continue the round robin for the analysis of VGO. In this interlaboratory study 52 laboratories in 27 different countries have participated. See appendix 3 for the number of participants per country. In this report, the results of the 2014 VGO proficiency test are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

2 SETUP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test. It was decided to send one bottle of 1L VGO (labelled #14255) that was purchased from a local supplier. The analyses for fit-for-use and homogeneity were subcontracted. Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010 (R007). This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Also customer's satisfaction is measured on regular basis by the distribution of questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol can be downloaded from the iis website www.iisnl.com.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved

2.4 SAMPLES

The necessary bulk material was obtained from a local supplier. The 100 litre bulk material was transferred after homogenizing into 90 brown glass bottles of 1 litre (labelled #14255). The homogeneity of the subsamples #14255 was checked by determination of Density @15°C in accordance with ISO12185 on 8 stratified randomly selected samples.

	Density @ 15 °C in kg/m ³
Sample #14255-1	933.4
Sample #14255-2	933.4
Sample #14255-3	933.3
Sample #14255-4	933.3
Sample #14255-5	933.4
Sample #14255-6	933.4
Sample #14255-7	933.3
Sample #14255-8	933.3

Table 1: homogeneity test results of subsamples #14255

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

	Density @ 15 °C in kg/m ³
r (sample #14255)	0.15
reference test	ISO12185:96
0.3 x R(reference test)	0.45

Table 2: evaluation of the repeatabilities of the subsamples #14255

The calculated repeatability is less than 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneity of the subsamples #14255 was assumed.

To each of the participating laboratories, 1 sample of 1 L in a brown glass bottle (labelled #14255) was sent on Nov 19, 2014.

2.5 ANALYSES

The participants were requested to determine on sample #14255: Acid Number (Total), Aniline Point, Asphaltenes, Carbon Residue (Micro method), Density @ 15°C, Distillation and Simulated Distillation (IBP, 10% rec, 30% rec, 50% rec, 70% rec, 90% rec and FBP), Flash Point PMcc, Kinematic Viscosity @ 50°C and @ 100°C, Micro Carbon Residue, Nitrogen, Total Sulphur and Metals (Arsenic, Calcium, Copper, Iron, Nickel, Silicon, Sodium, Vanadium).

To get comparable results a detailed report form, on which the units were prescribed as well as the required standards and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The detailed report form was also made available

for download on the iis website www.iisnl.com. A SDS and a form to confirm receipt of the samples were added to the sample package.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were received. The original reported results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after deadline, a reminder fax was sent to those laboratories that had not yet reported. Shortly after the deadline, the available results were screened for suspect data. A result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the (raw data of the) reported results.

Additional or corrected results have been used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' (iis-protocol, April 2014 version 3.3). For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. Results reported as '<...>' or '>...>' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test 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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.

In accordance to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon, Grubbs and Rosner outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test and by R(0.01) for the Rosner General ESD test (see appendix 4, no.15). Stragglers are marked by D(0.05) for the Dixon 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 the averages and the standard deviations.

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

3.2 GRAPHICS

In order to visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for each determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are under the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected standard. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 4; nos.13 and 14). 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 target standard deviation was calculated from the literature reproducibility by division with 2.8.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate the fit-for-useness of the reported test result.

The z-scores were calculated according to:

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

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

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

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

4 EVALUATION

In this interlaboratory study, problems with sample despatch were encountered due to several reasons. Ten participants reported the test results after the final reporting date and one participant did not report any test results at all. Not all laboratories were able to report all analyses requested. In total 51 participants reported 785 test results. Observed were 35 outlying results, which is 4.5% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

4.1 EVALUATION PER TEST

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

In the iis PT reports, ASTM methods are referred to with a number (e.g. D2086) and an added designation for the year that the method was adopted or revised (e.g. D2086-08). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D2086-08 (2013)). In the results tables of Appendix 1 only the method number and year of adoption or revision will be used.

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

The IP-PM-CW standard is a specific method for the determination of metallic elements in vacuum gasoil in the concentration range 0.1 mg/kg to 10 mg/kg. Regretfully IP-PM-CW does not mention precision data. The precision data of this method will be determined in 2015.

Acid Number (total): 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 D664:11a.

Aniline Point: This determination was very problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements of ASTM D611:12.
The test results of the blanc, the aniline point of n-heptane, vary over a small range (69.2 – 69.5).

Asphaltenes: No significant conclusions were drawn because the precision data of IP143:04 are applicable to values between 0.50% M/M and 30.0% M/M.

- Carbon Residue: (Micro method) This determination was very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of ASTM D4530:11. It should be noted that the precision data for the method has been obtained for the 2 ml capacity vials. Differences in vial size and/or sample intake may (partially) be the cause of the large spread.
- Density @ 15°C: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in full agreement with the requirements of ISO 12185:96.
- Flash Point PMcc: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with requirements of ASTM D93-B:13.
- Kin.Visco. @ 50°C: This determination may be very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of ASTM D445:14e2 for residual oils.
- Kin.Visco. @ 100°C: This determination may be not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D445:14e2 for residual oils.
- Nitrogen: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with ASTM D5762:12.
- Total Sulphur: This determination was problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in not in agreement with the requirements of ASTM D4294:10.
- Metals:**
- Arsenic (As): Only four participants reported a numerical result. Therefore no significant conclusions were drawn.
- Calcium (Ca): This determination may be very problematic. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of IP501:05.
- Copper (Cu): All results were near or below the application range of method IP-PM-CW (0.1 – 10 mg/kg). Therefore no statistical conclusions were drawn.

- Iron (Fe): This determination may not be problematic. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is full agreement with the requirements of IP501:05.
- Nickel (Ni): This determination may not be problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of IP501:05.
- Silicon (Si): The consensus value was far below the application range of method IP501:05 (10 – 250 mg/kg). Therefore no statistical conclusions were drawn.
- Sodium (Na): This determination may not be problematic. Six statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of IP501:05.
- Vanadium (V): This determination may not be problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of IP501:05.
- Sim. Dist. The Simulated Distillation according to ASTM D6352:14 was partly problematic. In total two statistical outliers were observed. The calculated reproducibilities were in agreement for IBP, 50% rec and 70% rec with the requirements of ASTM D6352:14. For 10% rec, 30% rec, 90% rec and FBP the calculated reproducibilities were not in agreement with the requirements of ASTM D6352:14.
- Distillation
Acc. D1160 The distillation according to ASTM D1160:13 was partly problematic. In total two statistical outliers were observed. The calculated reproducibilities were in agreement for 10% rec, 30% rec, 50% rec and 90% rec with the requirements of ASTM D1160:13. For IBP, 70% rec and FBP the calculated reproducibilities were not in agreement with the requirements of ASTM D1160:13.
Note: Care should be taken in pressure and cooling temperature.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and the reproducibility as found for the group of participating laboratories that participated. The average results, calculated reproducibilities and reproducibilities derived from literature standards (in casu ASTM standards), are compared in the next table.

Parameter	unit	n	Average	2.8 * sd	R(lit)
Acid Number (Total)	mg KOH/g	28	0.43	0.12	0.20
Aniline Point	°C	20	82.64	1.96	1.00
Asphaltenes	%M/M	24	0.102	0.298	(0.020)
Carbon Residue, micro method	%M/M	40	0.85	0.45	0.18
Density @ 15 °C	kg/m ³	46	933.6	1.58	1.50
Flash Point PMcc	°C	29	196.3	10.5	10.0
Kinematic Viscosity @ 50 °C	cSt	36	74.91	10.75	5.54
Kinematic Viscosity @ 100 °C	cSt	39	11.14	0.20	0.77
Nitrogen	%M/M	32	0.13	0.05	0.04
Total Sulphur Content	%M/M	45	2.18	0.14	0.12
Arsenic (As)	mg/kg	7	<1	n.a	n.a
Calcium (Ca)	mg/kg	21	0.32	0.76	0.30
Copper (Cu)	mg/kg	18	<0.1	n.a	n.a
Iron (Fe)	mg/kg	21	0.27	0.43	0.45
Nickel (Ni)	mg/kg	23	0.32	0.75	0.90
Silicon (Si)	mg/kg	18	0.78	2.65	(0.26)
Sodium (Na)	mg/kg	24	0.40	0.69	0.65
Vanadium (V)	mg/kg	26	0.42	0.37	1.00
<u>Simdist D6352</u>					
-IBP	°C	15	291.3	51.8	49.1
-10% rec	°C	15	391.0	9.4	7.1
-30% rec	°C	15	434.0	7.4	5.9
-50% rec	°C	15	467.4	7.0	6.4
-70% rec	°C	15	504.2	7.4	7.2
-90% rec	°C	15	552.6	12.9	10.5
-FBP	°C	13	611.8	64.7	38.1
<u>Distillation D1160</u>					
-IBP	°C	22	295.1	95.0	49.4
-10% rec	°C	23	408.3	15.2	17.9
-30% rec	°C	23	443.2	11.8	11.2
-50% rec	°C	23	471.4	11.3	11.2
-70% rec	°C	23	506.3	11.5	9.7
-90% rec	°C	21	553.5	11.9	22.8
-FBP	°C	20	575.1	34.0	26.9

Table 3: reproducibilities of results of sample #14255

results between brackets should be used with care, because the average was below the application range

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

4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2014 WITH PREVIOUS PTS

	<i>December 2014</i>	<i>November 2013</i>
Number of reporting labs	51	32
Number of results reported	785	474
Statistical outliers	35	12
Percentage outliers	4.5%	2.5%

Table 4: Comparison with previous proficiency tests.

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

The performance of the determinations of the proficiency tests was compared against the requirements of the respective standards. The conclusions are given the following table:

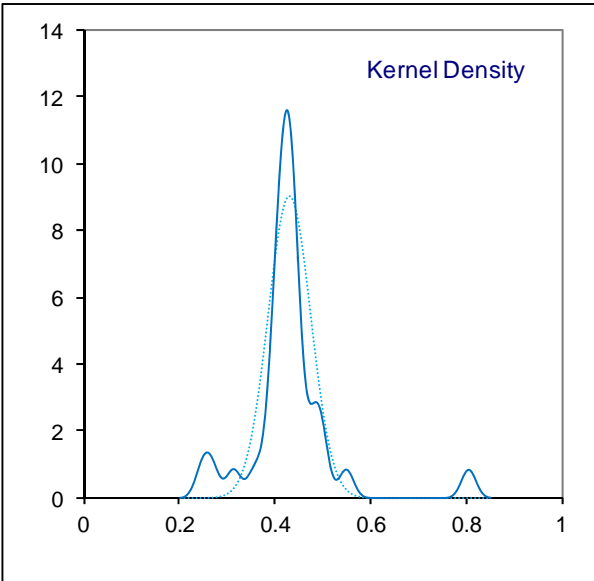
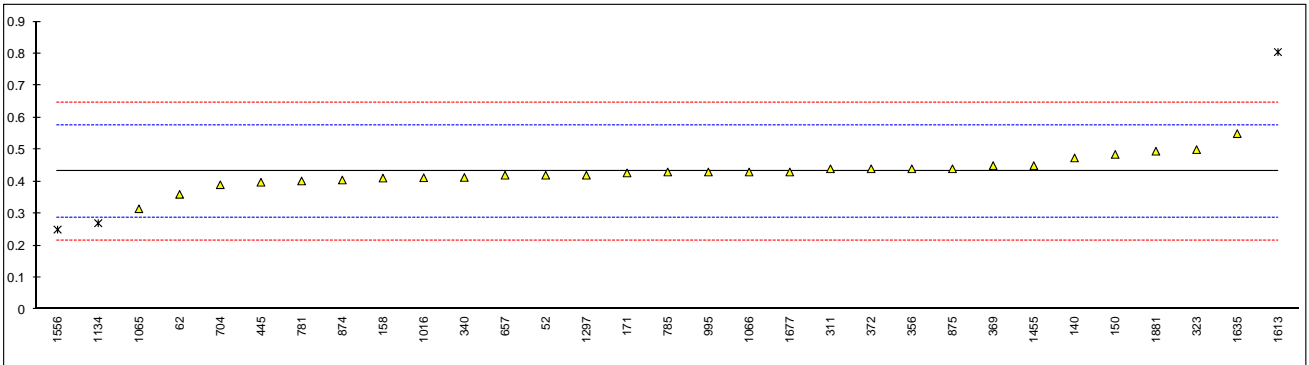
Determination	<i>December 2014</i>	<i>November 2013</i>
Acid Number (Total)	+	++
Aniline Point	--	--
Asphaltenes	(--)	(--)
Carbon Residue, micro method	--	+
Density @ 15 °C	+/-	+/-
Flash Point PMcc	+/-	+
Kinematic Viscosity @ 50 °C	--	++
Kinematic Viscosity @ 100 °C	++	++
Nitrogen	-	+
Total Sulphur Content	+/-	+/-
-Arsenic (As)	n.e.	n.e.
-Calcium (Ca)	--	-
-Copper (Cu)	n.e.	n.e.
-Nickel (Ni)	+/-	++
-Iron (Fe)	+	++
-Silicon (Si)	(--)	(--)
-Sodium (Na)	+/-	+/-
-Vanadium (V)	++	++
<u>Distillation D6352</u>		
-IBP	+/-	+
-10% rec	-	+
-30% rec	-	-
-50% rec	-	-
-70% rec	+/-	-
-90% rec	+/-	+
-FBP	-	+
<u>Distillation D1160</u>		
-IBP	--	--
-10% rec	+	+
-30% rec	+/-	+
-50% rec	+/-	+
-70% rec	-	+
-90% rec	++	++
-FBP	-	-

Table 5: comparison determinations against the standard

APPENDIX 1

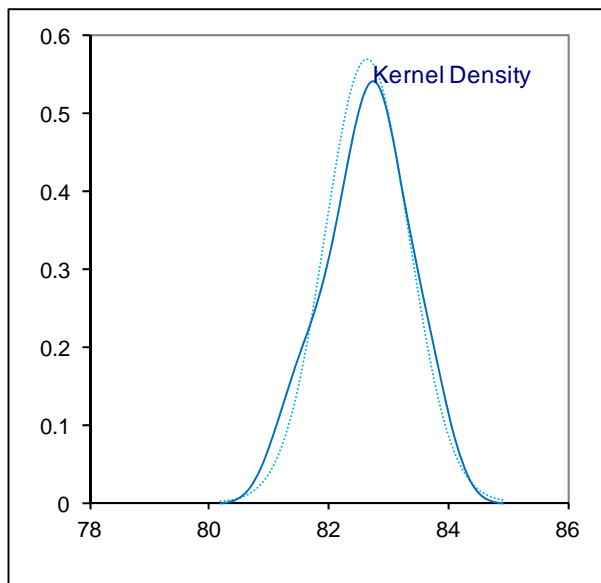
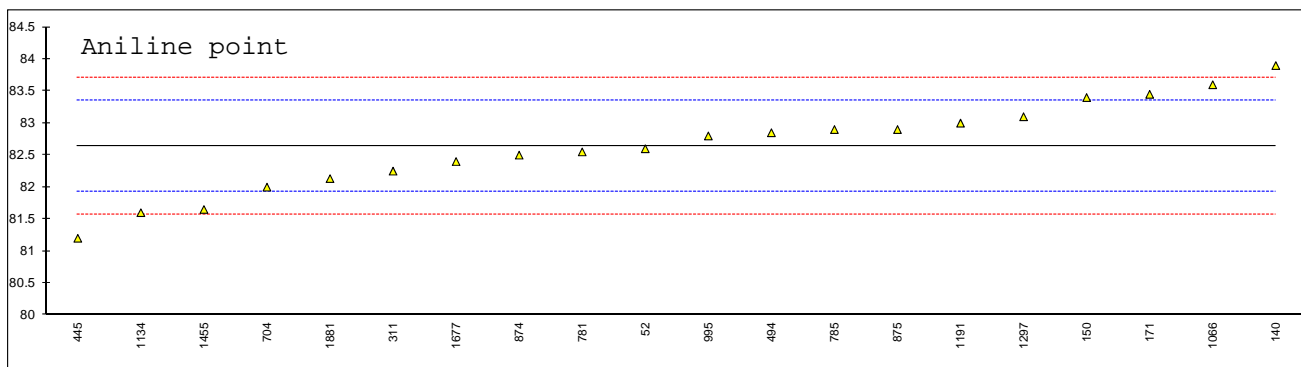
Determination of Acid Number (Total) on sample #14255; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
52	D664	0.42		-0.16	
62	D664	0.36		-0.99	
120		----		----	
131		----		----	
133		----		----	
140	D664	0.474		0.59	
150	D664	0.485	C	0.74	first reported:0.98
158	D664	0.411		-0.28	
171	D664	0.427		-0.06	
228		----		----	
311	D664	0.44		0.12	
313		----		----	
323	D664	0.50		0.95	
333		----		----	
334		----		----	
336		----		----	
337		----		----	
340	D664	0.413		-0.25	
356	D664	0.440		0.12	
369	D664	0.45		0.26	
372	D664	0.44		0.12	
445	D664	0.398		-0.46	
492		----		----	
494		----		----	
657	D664	0.42		-0.16	
704	D664	0.390		-0.57	
705		----		----	
781	D664	0.402		-0.41	
785	D664	0.43		-0.02	
791		----		----	
874	D664	0.405		-0.37	
875	D664	0.44		0.12	
966		----		----	
995	D664	0.43		-0.02	
1016	D664	0.412		-0.27	
1065	D664	0.315		-1.61	
1066	D664	0.43		-0.02	
1095		----		----	
1134	D664	0.27	R(0.05)	-2.24	
1191		----		----	
1201		----		----	
1297	D664	0.42		-0.16	
1340		----		----	
1397		----		----	
1455	D664	0.45		0.26	
1521		----		----	
1543		----		----	
1556	D664	0.25	C,R(0.05)	-2.52	first reported:1.07
1613	D664	0.805	R(0.01)	5.18	
1635	D664	0.55		1.65	
1677	D664	0.43		-0.02	
1881	D664	0.495		0.88	
	normality	not OK			
	n	28			
	outliers	3			
	mean (n)	0.4313			
	st.dev. (n)	0.04434			
	R(calc.)	0.1241			
	R(D664:11a)	0.2018			



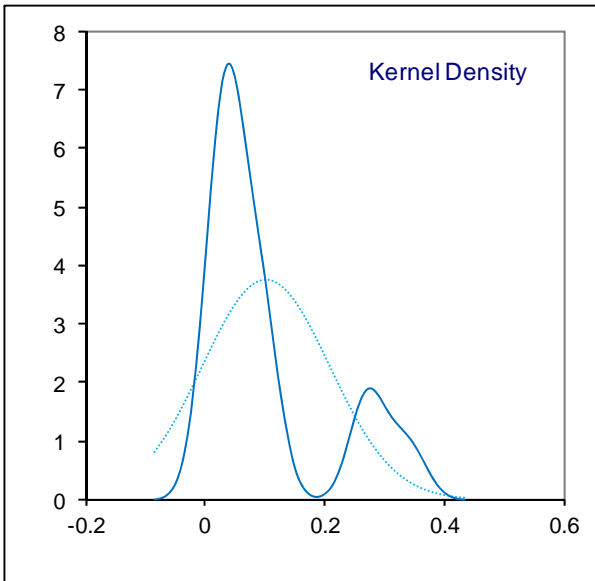
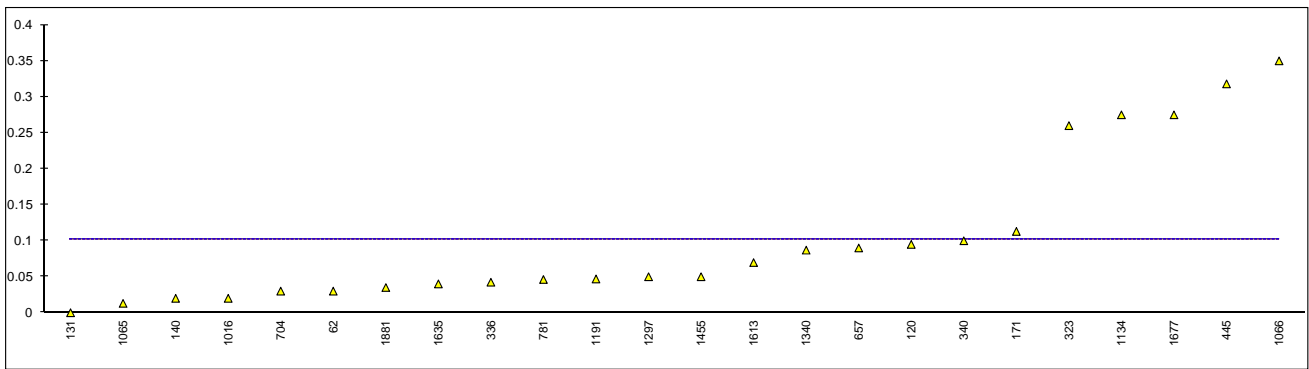
Determination of Aniline Point on sample #14255; results in °C

lab	method	Aniline Point	mark	z(targ)	Aniline Point n-heptane	remarks
52	D611-E	82.60		-0.11	69.40	
62		----		----	----	
120		----		----	----	
131		----		----	----	
133		----		----	----	
140	D611	83.9	C	3.53	69.25	first reported:85.4
150	D611-E	83.4		2.13	----	
158		----		----	----	
171	D611-E	83.45		2.27	----	
228		----		----	----	
311	D611-B	82.25		-1.09	69.30	
313		----		----	----	
323		----		----	----	
333		----		----	----	
334		----		----	----	
336		----		----	----	
337		----		----	----	
340		----		----	----	
356		----		----	----	
369	D611-A	----		----	69.4	
372	D611-E	----		----	69.30	
445	D611-B	81.2		-4.03	69.4	
492		----		----	----	
494	D611-B	82.85		0.59	69.3	
657		----		----	----	
704	D611-B	82.00		-1.79	69.20	
705		----		----	----	
781	D611-E	82.55		-0.25	69.25	
785	D611-E	82.9		0.73	69.2	
791		----		----	----	
874	D611-E	82.5		-0.39	69.3	
875	D611-E	82.90		0.73	69.25	
966		----		----	----	
995	D611-B	82.8	C	0.45	----	first reported:85.8
1016		----		----	----	
1065		----		----	----	
1066	D611-B	83.60		2.69	69.40	
1095		----	W	----	----	first reported:>170
1134	IP2-B	81.6		-2.91	69.5	
1191	D611-E	83.0		1.01	----	
1201		----		----	----	
1297		83.1		1.29	69.3	
1340		----		----	----	
1397		----		----	----	
1455	D611-E	81.65		-2.77	69.20	
1521		----		----	----	
1543		----		----	----	
1556		----		----	----	
1613		----		----	----	
1635		----		----	----	
1677	D611-A	82.4		-0.67	----	
1881	D611-B	82.133		-1.42	69.30	
	normality	OK				
	n	20				
	outliers	0				
	mean (n)	82.639				
	st.dev. (n)	0.7007				
	R(calc.)	1.962				
	R(D611:12)	1.000				



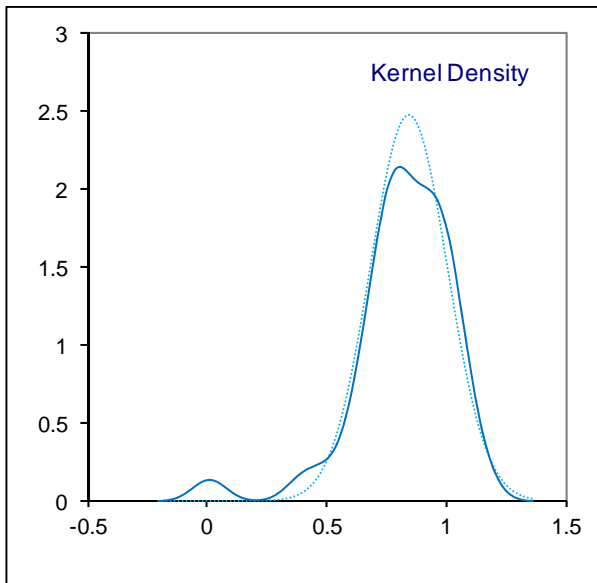
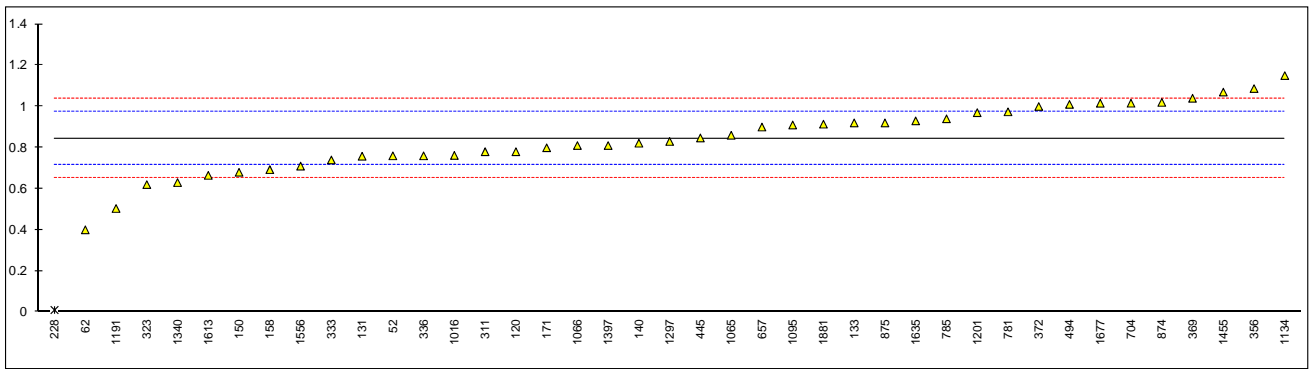
Determination of Asphaltenes on sample #14255; results in %M/M

lab	method	value	mark	z(targ)	remarks
52		----		----	
62	D6560	0.03		----	
120	IP143	0.095		----	
131	D6560	0.00		----	
133		----		----	
140	IP143	0.02		----	
150	IP143	<0.1		----	
158		----		----	
171	IP143	0.113		----	
228		----		----	
311		----		----	
313		----		----	
323	IP143	0.26		----	
333		----		----	
334		----		----	
336	INH-642	0.0424		----	
337		----		----	
340	IP143	0.10		----	
356	IP143	<0.5		----	
369		----		----	
372	IP143	<0.50		----	
445	IP143	0.318		----	
492		----		----	
494	IP143	<0.05		----	
657	IP143	0.09		----	
704	IP143	0.03		----	
705		----		----	
781	INH-642	0.0462		----	
785		----		----	
791		----		----	
874	IP143	<0.5		----	
875		----		----	
966		----		----	
995	IP143	<0.5		----	
1016	IP143	0.02		----	
1065	UOP614	0.013		----	
1066	IP143	0.35		----	
1095		----		----	
1134	IP143	0.275		----	
1191	INH-9313	0.0469		----	
1201		----		----	
1297	D6560	0.05		----	
1340	D6560	0.087		----	
1397		----		----	
1455	IP143	0.05		----	
1521		----		----	
1543		----		----	
1556		----	W	----	first reported:424.4
1613	D6560	0.0697		----	
1635	D6560	0.04		----	
1677	IP143	0.275		----	
1881	INH-642	0.035		----	
	normality	suspect			
	n	24			
	outliers	0			
	mean (n)	0.1023			
	st.dev. (n)	0.10635			
	R(calc.)	0.2978			
	R(IP143:04)	(0.0205)			precision is applicable between 0.50% M/M and 30.0% M/M



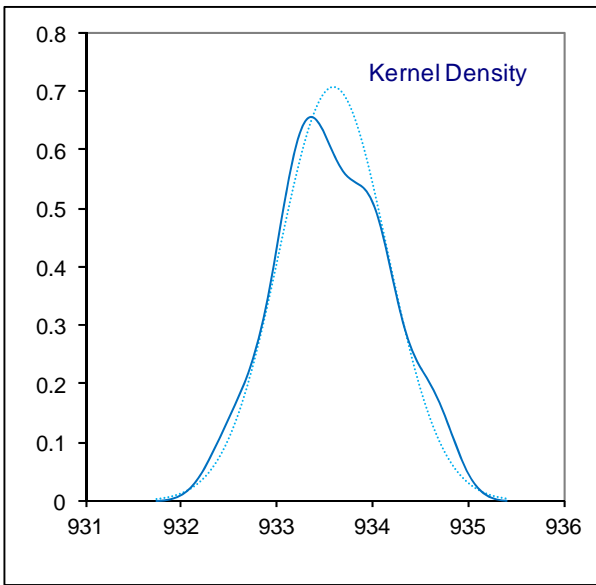
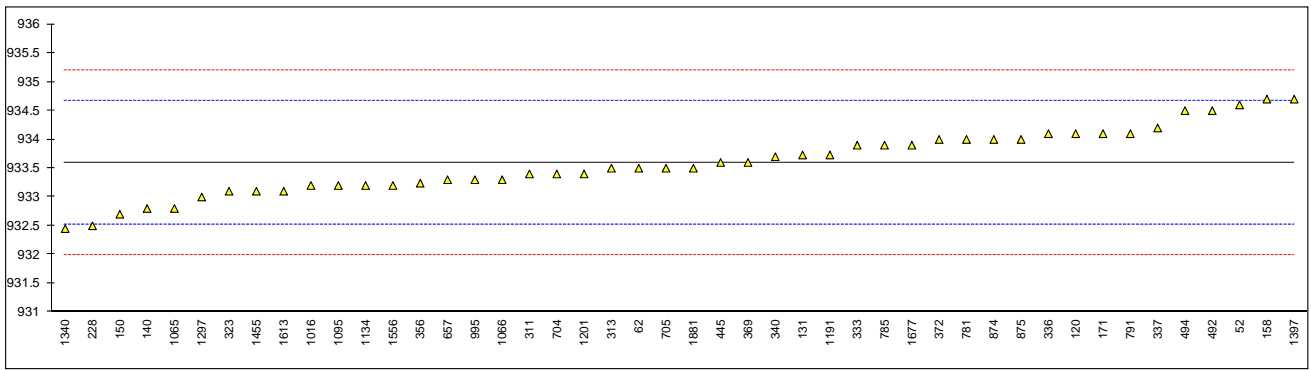
Determination of Carbon Residue, Micro method on sample #14255; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	D4530	0.76		-1.32	
62	D4530	0.40		-6.92	
120	D4530	0.78		-1.01	
131	D4530	0.758		-1.36	
133	D4530	0.92		1.16	
140	D4530	0.8215		-0.37	
150	D4530	0.68		-2.57	
158	D4530	0.693		-2.37	
171	D4530	0.799		-0.72	
228	D189	0.01	R(0.01)	-12.99	
311	D4530	0.78		-1.01	
313		----		----	
323	D4530	0.62		-3.50	
333	D4530	0.74		-1.64	
334		----		----	
336	D4530	0.76		-1.32	
337		----		----	
340		----		----	
356	D4530	1.087		3.76	
369	D4530	1.04		3.03	
372	D4530	1.00		2.41	
445	D4530	0.847		0.03	
492		----		----	
494	D4530	1.01		2.56	
657	D4530	0.90		0.85	
704	D4530	1.016		2.66	
705		----		----	
781	D4530	0.974		2.00	
785	D4530	0.94		1.48	
791		----		----	
874	D4530	1.02		2.72	
875	D4530	0.92		1.16	
966		----		----	
995		----		----	
1016	ISO10370	0.762		-1.29	
1065	D4530	0.86		0.23	
1066	D4530	0.81		-0.55	
1095	D4530	0.91		1.01	
1134	IP13	1.15		4.74	
1191	ISO10370	0.5039	C	-5.31	first reported:0
1201	D4530	0.97		1.94	
1297	D4530	0.83		-0.24	
1340	ISO10370	0.63		-3.35	
1397	D4530	0.81		-0.55	
1455	D4530	1.07		3.50	
1521		----		----	
1543		----		----	
1556	ISO10370	0.71		-2.10	
1613	D4530	0.665		-2.80	
1635	D4530	0.93		1.32	
1677	D4530	1.0158		2.65	
1881	D4530	0.914		1.07	
	normality	OK			
	n	40			
	outliers	1			
	mean (n)	0.8452			
	st.dev. (n)	0.16160			
	R(calc.)	0.4525			
	R(D4530:11)	0.1800			Compare R(ISO10370:93) = 0.058



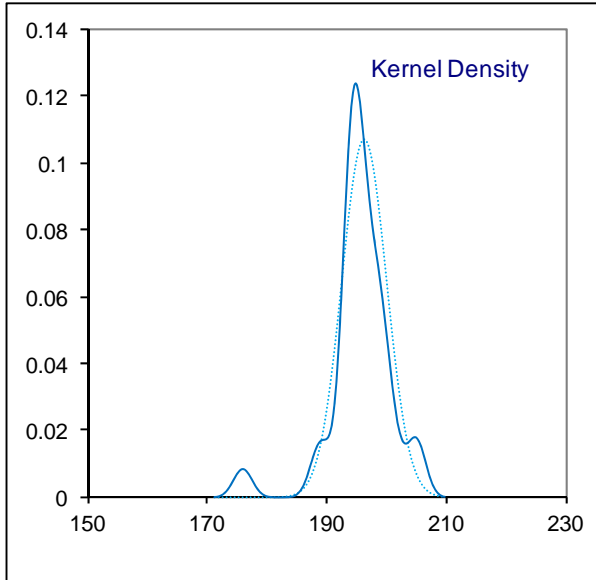
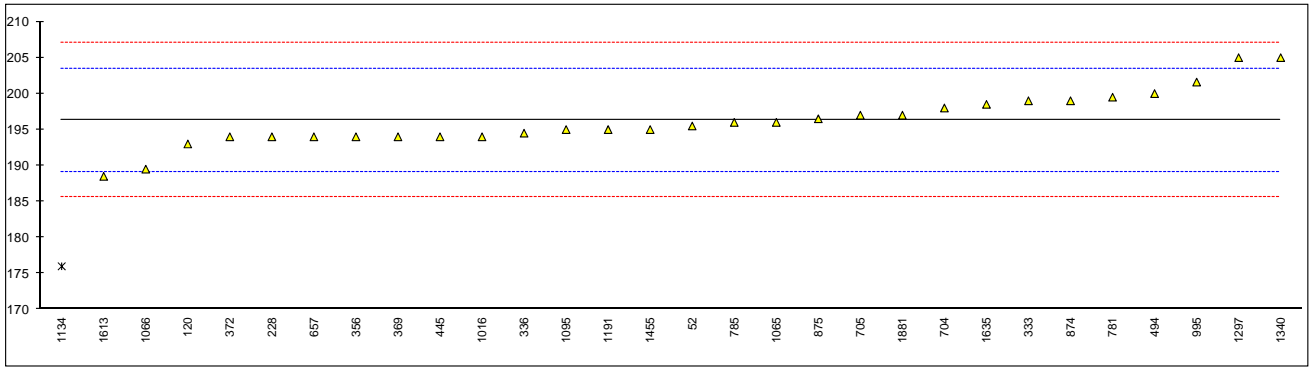
Determination of Density @ 15°C on sample #14255; results in kg/m³

lab	method	value	mark	z(targ)	remarks
52	D4052	934.6		1.88	
62	D4052	933.5		-0.18	
120	D4052	934.1		0.94	
131	D4052	933.73		0.25	
133		-----		-----	
140	ISO12185	932.8		-1.48	
150	D4052	932.7		-1.67	
158	D287	934.7		2.06	
171	ISO12185	934.1		0.94	
228	D1298	932.5		-2.04	
311	ISO12185	933.4		-0.36	
313	ISO12185	933.5		-0.18	
323	ISO12185	933.1		-0.92	
333	D4052	933.9		0.57	
334		-----		-----	
336	D4052	934.1		0.94	
337	ISO12185	934.2		1.13	
340	ISO12185	933.7		0.20	
356	ISO12185	933.24		-0.66	
369	D4052	933.6		0.01	
372	ISO12185	934.0		0.76	
445	ISO12185	933.6		0.01	
492	ISO12185	934.5		1.69	
494	ISO12185	934.5		1.69	
657	D4052	933.3		-0.55	
704	ISO12185	933.4		-0.36	
705	D1298	933.5		-0.18	
781	ISO12185	934.0		0.76	
785	D1298	933.9		0.57	
791	D1298	934.1		0.94	
874	ISO12185	934.0		0.76	
875	D1298	934.0		0.76	
966		-----		-----	
995	ISO12185	933.3		-0.55	
1016	ISO12185	933.2		-0.74	
1065	D1298	932.8		-1.48	
1066	ISO12185	933.3		-0.55	
1095	ISO12185	933.2		-0.74	
1134	IP365	933.2		-0.74	
1191	ISO12185	933.73		0.25	
1201	ISO12185	933.4		-0.36	
1297	D4052	933.0		-1.11	
1340	ISO3675	932.45		-2.14	
1397	D1298	934.7		2.06	
1455	ISO12185	933.1		-0.92	
1521		-----		-----	
1543		-----		-----	
1556	ISO12185	933.2		-0.74	
1613	D4052	933.1		-0.92	
1635		-----		-----	
1677	D4052	933.90		0.57	
1881	D4052	933.5		-0.18	
	normality	OK			
	n	46			
	outliers	0			
	mean (n)	933.59			
	st.dev. (n)	0.563			
	R(calc.)	1.58			
	R(ISO12185:96)	1.50			



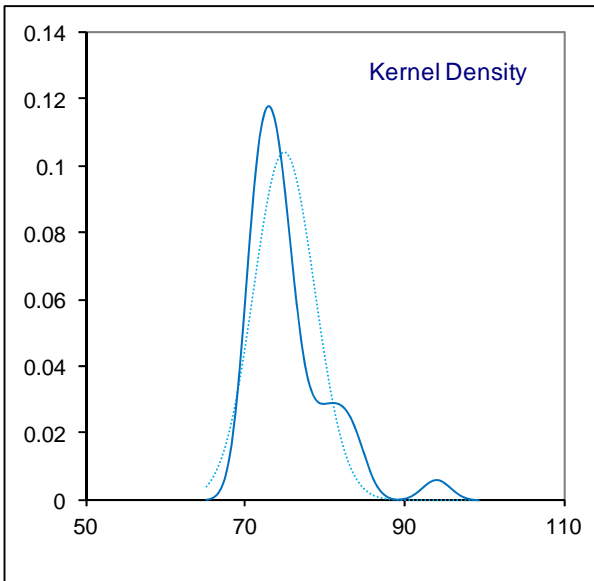
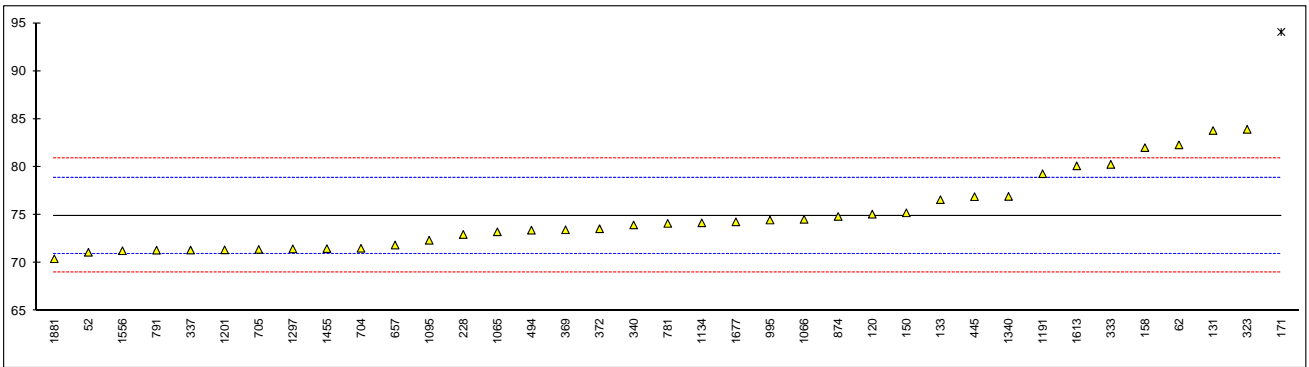
Determination of Flash Point PMcc on sample #14255; results in °C

lab	method	value	mark	z(targ)	remarks
52	D93-A	195.5		-0.22	
62	D93-B	>150		----	
120	D93-B	193		-0.92	
131		----		----	
133		----		----	
140		----		----	
150	D93-B	>230		>9.44	false positive test result?
158	D93-B	>110		----	
171		----		----	
228	D93-B	194.0		-0.64	
311	D93-B	>180		----	
313	D93-B	>160		----	
323		----		----	
333	D93-B	199.0		0.76	
334		----		----	
336	D93-B	194.5		-0.50	
337		----		----	
340		----		----	
356	D93-B	194.0		-0.64	
369	D93-B	194.0		-0.64	
372	D93-B	194.0		-0.64	
445	D93-B	194.0		-0.64	
492		----		----	
494	D93-B	200.0		1.04	
657	D93-B	194.0		-0.64	
704	D93-B	198.0		0.48	
705	D93-B	197.0		0.20	
781	D93-B	199.5		0.90	
785	D93-B	196.0		-0.08	
791		----		----	
874	D93-B	199.0		0.76	
875	D93-B	196.5		0.06	
966		----		----	
995	D93-B	201.6		1.49	
1016	D93-A	194		-0.64	
1065	D93-B	196		-0.08	
1066	D93-A	189.5		-1.90	
1095	D93-B	195.0		-0.36	
1134	IP523-B	176.0	R(0.01)	-5.68	
1191	ISO2719	195.0		-0.36	
1201		----		----	
1297	D93-B	205		2.44	
1340	D93-B	205		2.44	
1397	D93-B	----		----	Sample burned at 237 °C but instrument did not detect the flash point
1455	D93-A	195.0		-0.36	
1521		----		----	
1543		----		----	
1556	ISO2719-B	>210		>3.84	false positive test result?
1613	D93-B	188.5		-2.18	
1635	D93-B	198.5		0.62	
1677	D93-B	>200		----	
1881	D93-B	197.0		0.20	
	normality	OK			
	n	29			
	outliers	1			
	mean (n)	196.28			
	st.dev. (n)	3.736			
	R(calc.)	10.46			
	R(D93-B:13)	10.00			



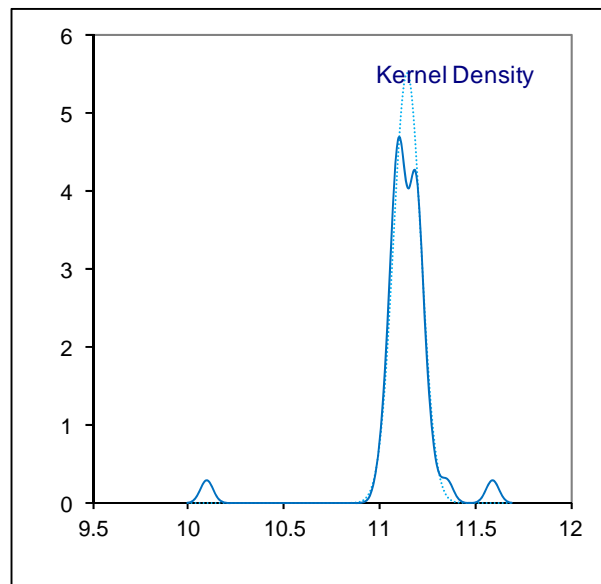
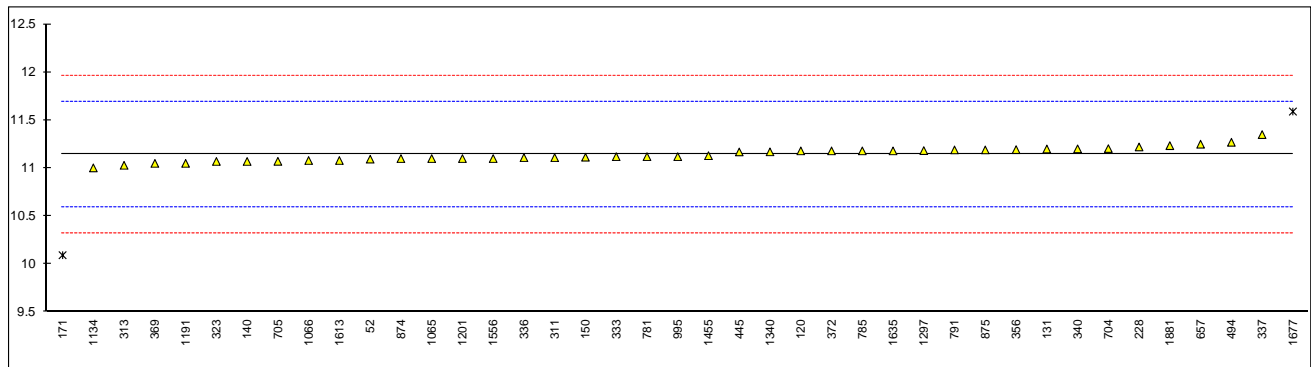
Determination of Kinematic Viscosity @ 50°C on sample #14255; results in mm²/s

lab	method	value	mark	z(targ)	remarks
52	D445	71.1162		-1.92	
62	D445	82.311		3.74	
120	D445	75.09		0.09	
131	D445	83.81		4.50	
133	D445	76.6		0.85	
140		----		----	
150	D445	75.23		0.16	
158	D445	82.022		3.59	
171	D445	94.077	R(0.01)	9.68	
228	D445	72.974		-0.98	
311		----		----	
313		----		----	
323	D445	83.932		4.56	
333	D445	80.28		2.71	
334		----		----	
336		----		----	
337	D445	71.35		-1.80	
340	D445	73.965		-0.48	
356		----		----	
369	D445	73.46		-0.73	
372	D445	73.57		-0.68	
445	D445	76.915		1.01	
492		----		----	
494	D445	73.42		-0.75	
657	D445	71.87		-1.54	
704	D445	71.532		-1.71	
705	D445	71.415		-1.77	
781	D445	74.13		-0.39	
785		----		----	
791	D445	71.34		-1.80	
874	D445	74.85		-0.03	
875		----		----	
966		----		----	
995	D445	74.48		-0.22	
1016		----		----	
1065	D445	73.25		-0.84	
1066	D445	74.55		-0.18	
1095	D445	72.37		-1.28	
1134	D445	74.185		-0.37	
1191	ISO3104	79.306		2.22	
1201	D445	71.37		-1.79	
1297	D7042	71.477		-1.73	
1340	ISO3104	76.945		1.03	
1397		----		----	
1455	D445	71.50		-1.72	
1521		----		----	
1543		----		----	
1556	ISO3104	71.276		-1.84	
1613	D445	80.125		2.63	
1635		----		----	
1677	D445	74.29		-0.31	
1881	D445	70.441		-2.26	
	normality	suspect			
	n	36			
	outliers	1			
	mean (n)	74.910			
	st.dev. (n)	3.8399			
	R(calc.)	10.752			
	R(D445:14e2)	5.543			



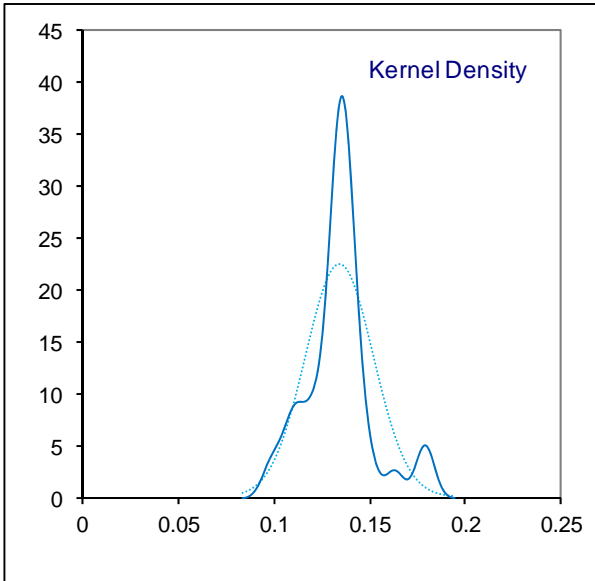
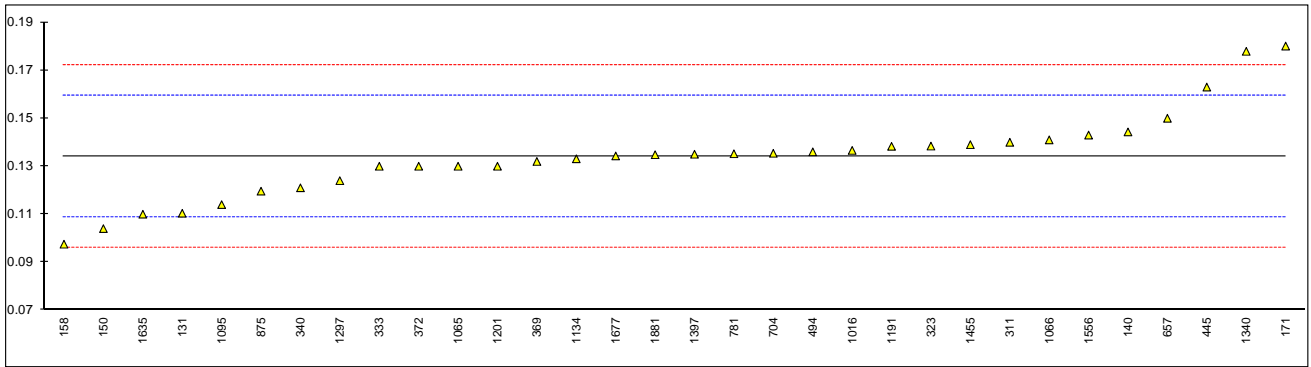
Determination of Kinematic Viscosity @ 100°C on sample #14255; results in mm²/s

lab	method	value	mark	z(targ)	remarks
52	D445	11.0938		-0.18	
62		----		----	
120	D445	11.18		0.14	
131	D445	11.20		0.21	
133		----		----	
140	D445	11.07		-0.27	
150	D445	11.114		-0.11	
158		----		----	
171	D445	10.093	R(0.01)	-3.84	
228	D445	11.221		0.29	
311	D445	11.11		-0.12	
313	D445	11.03		-0.41	
323	D445	11.07		-0.27	
333	D445	11.12		-0.08	
334		----		----	
336	D445	11.11		-0.12	
337	D445	11.35		0.76	
340	D445	11.201		0.21	
356	D445	11.193		0.18	
369	D445	11.05		-0.34	
372	D445	11.18		0.14	
445	D445	11.169		0.10	
492		----		----	
494	D445	11.27		0.47	
657	D445	11.25		0.39	
704	D445	11.204		0.22	
705	D445	11.071		-0.26	
781	D445	11.12		-0.08	
785	D445	11.18		0.14	
791	D445	11.19		0.17	
874	D445	11.10		-0.16	
875	D445	11.19		0.17	
966		----		----	
995	D445	11.12		-0.08	
1016		----		----	
1065	D445	11.10		-0.16	
1066	D445	11.08		-0.23	
1095		----		----	
1134	D445	11.003		-0.51	
1191	ISO3104	11.05		-0.34	
1201	D445	11.10		-0.16	
1297	D7042	11.183		0.15	
1340	ISO3104	11.17		0.10	
1397		----		----	
1455	D445	11.13		-0.05	
1521		----		----	
1543		----		----	
1556	ISO3104	11.10		-0.16	
1613	D445	11.08		-0.23	
1635	D445	11.18		0.14	
1677	D445	11.59	R(0.01)	1.64	
1881	D445	11.235		0.34	
	normality	OK			
	n	39			
	outliers	2			
	mean (n)	11.143			
	st.dev. (n)	0.0723			
	R(calc.)	0.202			
	R(D445:14e2)	0.766			



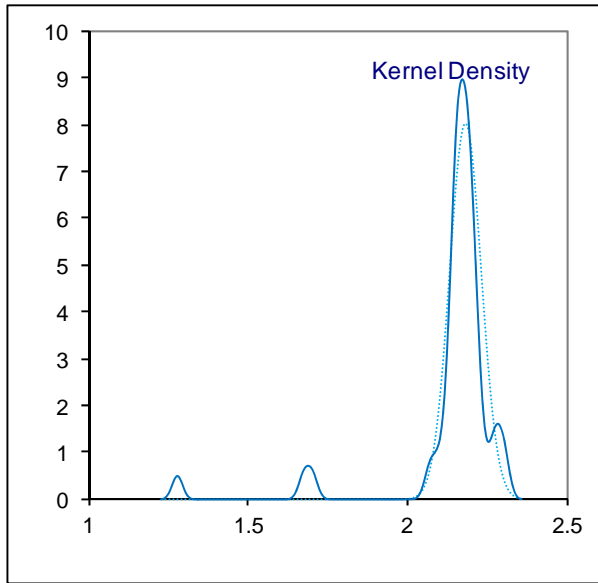
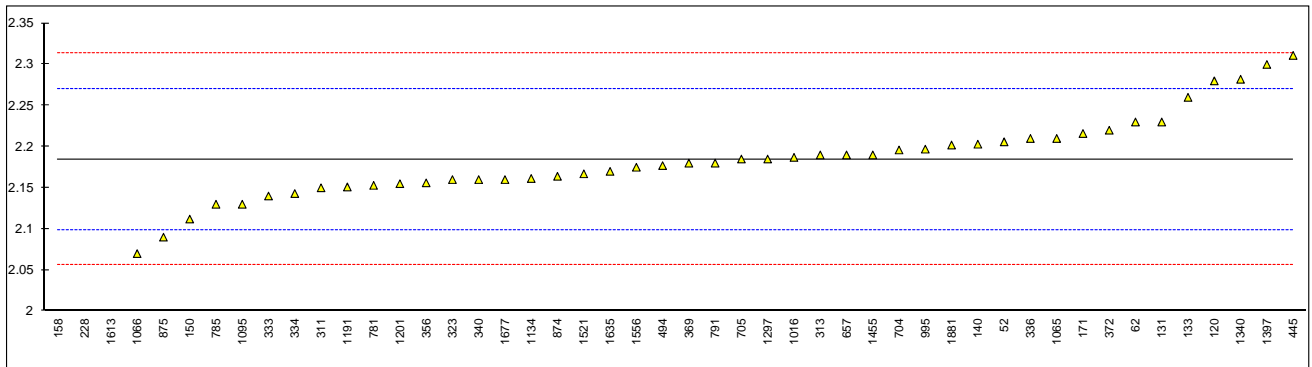
Determination of Nitrogen on sample #14255; results in %M/M

lab	method	value	mark	z(targ)	remarks
52		----		----	
62		----		----	
120		----		----	
131	D4629	0.1104	C	-1.85	first reported:1104.02
133		----		----	
140	D5762	0.1443	C	0.81	first reported:1443
150	D5762	0.1040		-2.36	
158	D5762	0.0975	C	-2.87	first reported:974.8
171	D5762	0.1801		3.62	
228		----		----	
311	D5762	0.14		0.47	
313		----		----	
323	D5762	0.1384		0.35	
333	D5762	0.13		-0.31	
334		----		----	
336		----		----	
337		----		----	
340	INH-10006	0.121		-1.02	
356		----		----	
369	D3228	0.132		-0.16	
372	D5762	0.13		-0.31	
445	D5762	0.163		2.28	
492		----		----	
494	D5762	0.136	C	0.16	first reported: 1363
657	D5762	0.15		1.26	
704	D5762	0.1354		0.11	
705		----		----	
781	D5762	0.1352		0.09	
785		----		----	
791		----		----	
874		----		----	
875	D5762	0.1196		-1.13	
966		----		----	
995		----		----	
1016	D5762	0.1366	C	0.20	first reported:1366
1065	D5762	0.130		-0.31	
1066	D5762	0.141		0.55	
1095	D5762	0.114		-1.57	
1134	D5762	0.133101	C	-0.07	probably unit error, reported:1331.01
1191	D5762	0.1383		0.34	
1201	D5762	0.13		-0.31	
1297	D4629	0.124		-0.79	
1340	D5762	0.17791		3.45	
1397	UOP384	0.135		0.08	
1455	D5762	0.139		0.39	
1521		----		----	
1543		----		----	
1556	D5762	0.143		0.71	
1613		----		----	
1635	D5762	0.11		-1.89	
1677	D5762	0.1343		0.02	
1881	D5762	0.1348		0.06	
	normality	suspect			
	n	32			
	outliers	0			
	mean (n)	0.1340			
	st.dev. (n)	0.01779			
	R(calc.)	0.0498			
	R(D5762:12)	0.0356			



Determination of Total Sulphur on sample #14255; results in %M/M

lab	method	value	mark	z(targ)	remarks
52	D4294	2.2059		0.49	
62	D4294	2.23		1.05	
120	D4294	2.28		2.22	
131	D4294	2.23		1.05	
133	D4294	2.26		1.75	
140	D4294	2.20303		0.43	
150	D4294	2.112		-1.69	
158	D4294	1.2815	R(0.01)	-21.03	
171	D4294	2.216		0.73	
228	D4294	1.678	R(0.01)	-11.80	
311	D4294	2.15		-0.81	
313	ISO8754	2.19		0.12	
323	D4294	2.16		-0.58	
333	D2622	2.14		-1.04	
334	D4294	2.143		-0.97	
336	D4294	2.21		0.59	
337		-----		-----	
340	D4294	2.16		-0.58	
356	D4294	2.156		-0.67	
369	D2622	2.18		-0.11	
372	D4294	2.22		0.82	
445	D4294	2.311		2.94	
492		-----		-----	
494	D4294	2.177		-0.18	
657	D4294	2.19		0.12	
704	D4294	2.196		0.26	
705	D4294	2.185		0.01	
781	D4294	2.153		-0.74	
785	D4294	2.13		-1.28	
791	D4294	2.18		-0.11	
874	D4294	2.164		-0.48	
875	D4294	2.09		-2.21	
966		-----		-----	
995	D4294	2.197		0.28	
1016	in house	2.187		0.05	
1065	IP336	2.21		0.59	
1066	D2622	2.07		-2.67	
1095	D4294	2.13		-1.28	
1134	IP336	2.1613		-0.55	
1191	ISO8754	2.151		-0.79	
1201	D4294	2.155		-0.69	
1297	D4294	2.185		0.01	
1340	ISO8754	2.282		2.26	
1397	D2622	2.3		2.68	
1455	D2622	2.19		0.12	
1521	ISO14596	2.167		-0.41	
1543		-----		-----	
1556	ISO8754	2.175		-0.23	
1613	D1551	1.705	R(0.01)	-11.17	
1635	D4294	2.17		-0.34	
1677	D4294	2.160		-0.58	
1881	D4294	2.202		0.40	
	normality	OK			
	n	45			
	outliers	3			
	mean (n)	2.185			
	st.dev. (n)	0.0496			
	R(calc.)	0.139			
	R(D4294:10)	0.120			



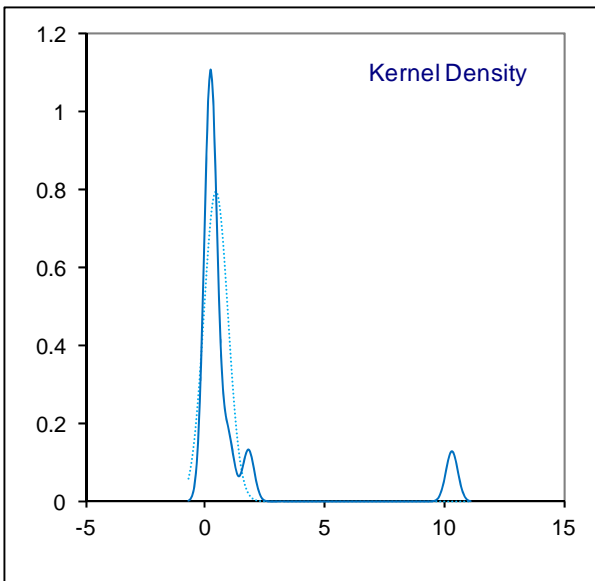
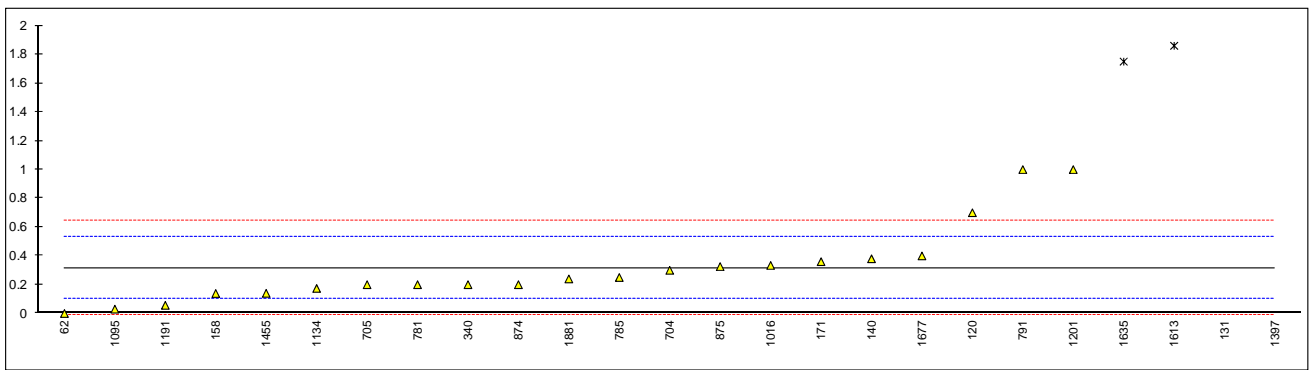
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Determination of Arsenic on sample #14255; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52		----		----	
62		----		----	
120	D5708B	<1		----	
131	IP501	0.0		----	
133		----		----	
140	D5708B	0.004		----	
150		----		----	
158		----		----	
171	IP501	0.034		----	
228		----		----	
311	UOP986	<0.05		----	
313		----		----	
323		----		----	
333		----		----	
334		----		----	
336		----		----	
337		----		----	
340		----		----	
356		----		----	
369		----		----	
372		----		----	
445	D5185	<1		----	
492		----		----	
494		----		----	
657		----		----	
704		----		----	
705		----		----	
781		----		----	
785		----		----	
791		----		----	
874		----		----	
875		----		----	
966		----		----	
995		----		----	
1016		----		----	
1065		----		----	
1066		----		----	
1095		----		----	
1134		----		----	
1191		----		----	
1201		----		----	
1297	in house	0.026		----	
1340		----		----	
1397		----		----	
1455		----		----	
1521		----		----	
1543		----		----	
1556		----		----	
1613		----		----	
1635		----		----	
1677		----		----	
1881		----		----	
	normality	n.a			
	n	7			
	outliers	n.a			
	mean (n)	<1			
	st.dev. (n)	n.a			
	R(calc.)	n.a			
	R(IP PM CW/04)	unknown			Application range: 0.1 – 10 mg/kg

Determination of Calcium on sample #14255; results in mg/kg

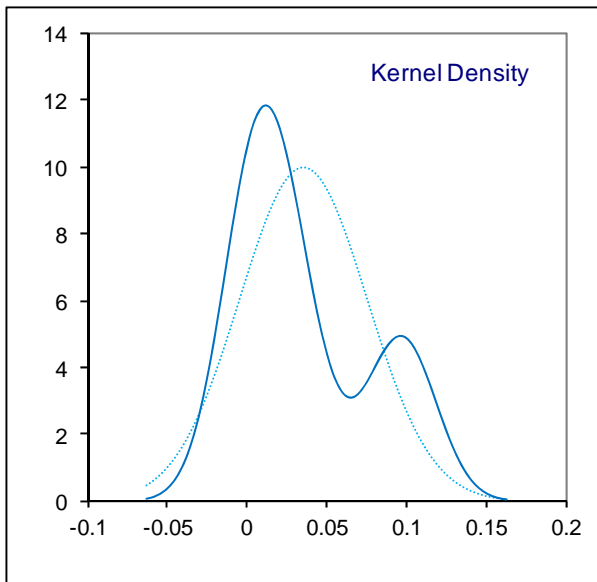
lab	method	value	mark	z(targ)	remarks
52		----		----	
62	IP470	0		-2.90	
120	IP501	0.7		3.54	
131	IP501	10.24	R(0.01)	91.30	
133		----		----	
140	IP501	0.38		0.59	
150	IP501	<1		----	
158	IP501	0.139		-1.63	
171	IP501	0.360		0.41	
228		----		----	
311	D7111	<0.10		----	
313		----		----	
323	IP501	<3		----	
333		----		----	
334		----		----	
336		----		----	
337		----		----	
340	IP501	0.2		-1.06	
356	IP501	<3		----	
369	IP501	<3		----	
372	IP470	<3		----	
445	IP501	<3		----	
492		----		----	
494	IP501	<1		----	
657	IP501	<3		----	
704	IP470	0.3		-0.14	
705	IP470	0.20		-1.06	
781	IP PM CW	0.2		-1.06	
785	NEN6966	0.25		-0.60	
791	IP470	1		6.30	
874	IP470	0.2		-1.06	
875	IP501	0.326		0.09	
966		----		----	
995		----		----	
1016	NEN6966	0.334		0.17	
1065		----		----	
1066		----		----	
1095	D5708	0.030		-2.63	
1134	IP501	0.174		-1.30	
1191	D5185	0.0566		-2.38	
1201	IP501	1		6.30	
1297		----		----	
1340		----		----	
1397	ISO11885	10.4	R(0.01)	92.77	
1455	IP PM CW	0.14		-1.62	
1521		----		----	
1543		----		----	
1556		----		----	
1613	IP470	1.86	R(0.01)	14.21	false positive test result?
1635	IP501	1.75	R(0.01)	13.19	false positive test result?
1677	IP501	0.4		0.78	
1881	IP PM CW	0.24		-0.70	
	normality	not OK			
	n	21			
	outliers	4			
	mean (n)	0.3157			
	st.dev. (n)	0.27316			
	R(calc.)	0.7648			
	R(IP501:05)	0.3044			Application range : 3 – 100 mg/kg
	compare				
	R(IP470:05)	3.5395			Application range : 3 - 100mg/kg
	R(IP PM CW/04)	unknown			Application range: 0.1 – 10 mg/kg



Determination of Copper on sample #14255; results in mg/kg

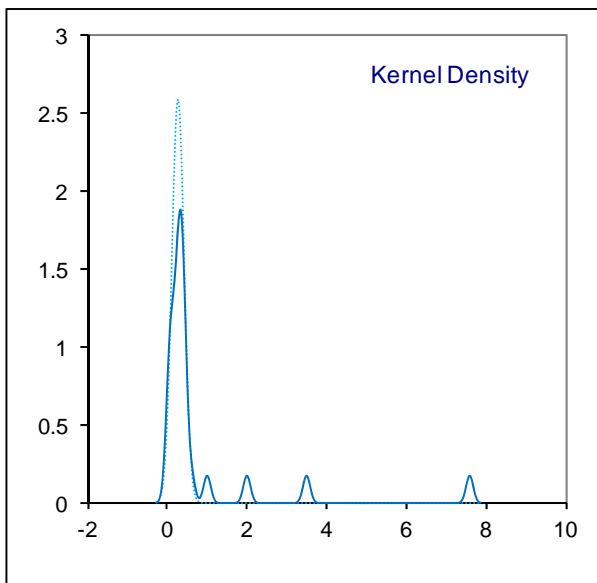
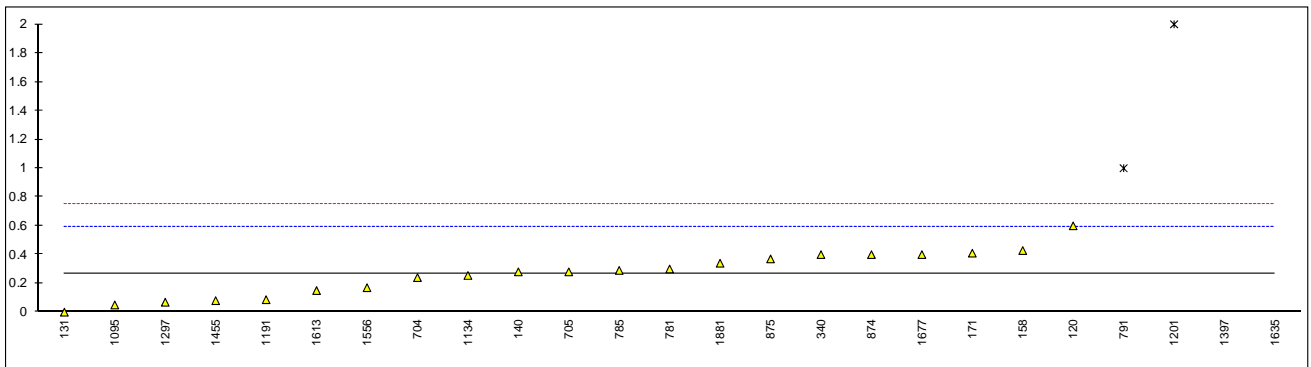
lab	method	value	mark	z(targ)	remarks
52		----		----	
62		----		----	
120	IP501	0.1		----	
131	IP501	0.0		----	
133		----		----	
140	D5708B	0.03		----	
150	IP501	<1		----	
158		----		----	
171	IP501	0.086		----	
228		----		----	
311	D7111	<0.10		----	
313		----		----	
323		----		----	
333		----		----	
334		----		----	
336		----		----	
337		----		----	
340		----		----	
356		----		----	
369		----		----	
372	IP470	<1		----	
445	D5185	<2		----	
492		----		----	
494		----		----	
657	IP501	<1		----	
704	IP PM CW	0.02		----	
705	IP PM CW	0.02		----	
781	IP PM CW	0.0		----	
785	in house	0.02		----	
791		<1		----	
874	IP470	0.1		----	
875		0.039		----	
966		----		----	
995		----		----	
1016		----		----	
1065		----		----	
1066		----		----	
1095	D5708	0.010		----	
1134		----		----	
1191	D5185	0.0078		----	
1201	IP501	0		----	
1297		----		----	
1340		----		----	
1397	ISO11885	<0.003		----	
1455	IP PM CW	<0.1		----	
1521		----		----	
1543		----		----	
1556	INH-1020	<0.1		----	
1613		----		----	
1635		----		----	
1677	IP501	0.1		----	
1881	IP PM CW	0.00		----	
	normality	n.a			
	n	18			
	outliers	n.a			
	mean (n)	<0.1			
	st.dev. (n)	n.a			
	R(calc.)	n.a			
	R(IP PM CW/04)	unknown			Application range: 0.1 – 10 mg/kg





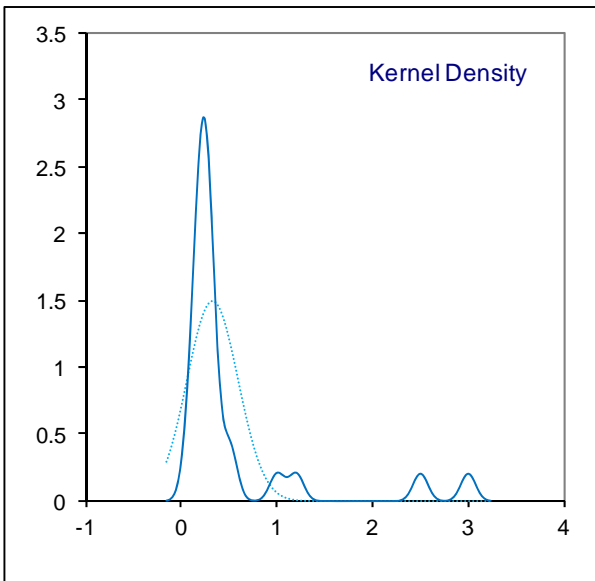
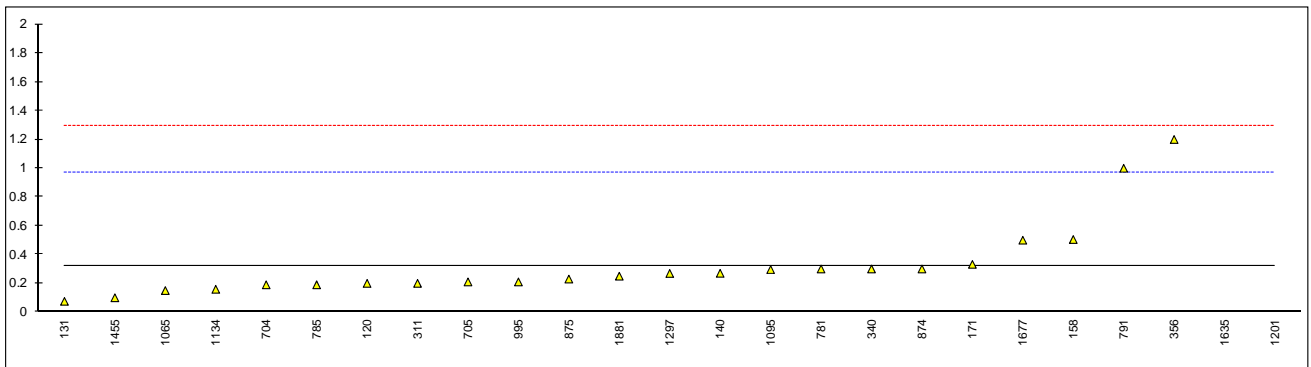
Determination of Iron on sample #14255; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52		----		----	
62	IP470	<1		----	
120	IP501	0.6		2.06	
131	IP501	0.0		-1.65	
133		----		----	
140	IP501	0.28		0.08	
150	IP501	<1		----	
158	IP501	0.428		1.00	
171	IP501	0.410		0.89	
228		----		----	
311	D7111	<0.10		----	
313		----		----	
323	IP501	<2		----	
333		----		----	
334		----		----	
336		----		----	
337		----		----	
340	IP501	0.4		0.82	
356	IP501	<2		----	
369	IP501	<2		----	
372	IP470	<2		----	
445	IP501	<2		----	
492		----		----	
494	IP501	<1		----	
657	IP501	<1		----	
704	IP PM CW	0.24		-0.16	
705	IP PM CW	0.28		0.08	
781	IP PM CW	0.3		0.21	
785	IP470	0.29		0.14	
791	IP470	1	R(0.01)	4.53	
874	IP470	0.4		0.82	
875	IP501	0.37		0.64	
966		----		----	
995		----		----	
1016	in house	<2		----	
1065		----		----	
1066		----		----	
1095	D5708	0.050		-1.34	
1134	IP501	0.255		-0.07	
1191	D5185	0.0869		-1.11	
1201	IP501	2	R(0.01)	10.71	
1297	D5708	0.069		-1.22	
1340		----		----	
1397	ISO11885	3.50	R(0.01)	19.98	
1455	IP PM CW	0.08		-1.15	
1521		----		----	
1543		----		----	
1556	INH-1020	0.170		-0.60	
1613	IP470	0.15		-0.72	
1635	IP501	7.6	R(0.01)	45.31	
1677	IP501	0.4		0.82	
1881	IP PM CW	0.34		0.45	
	normality	OK			
	n	21			
	outliers	4			
	mean (n)	0.2666			
	st.dev. (n)	0.15446			
	R(calc.)	0.4325			
	R(IP501:05)	0.4532			Application range : 2 mg/kg – 60 mg/kg
	compare				
	R(IP470:05)	0.8537			Application range : 2 mg/kg – 60 mg/kg
	R(IP PM CW/04)	unknown			Application range: 0.1 – 10 mg/kg



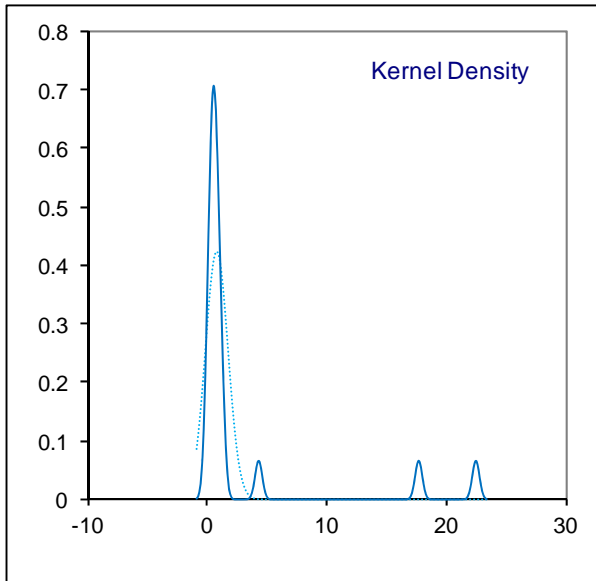
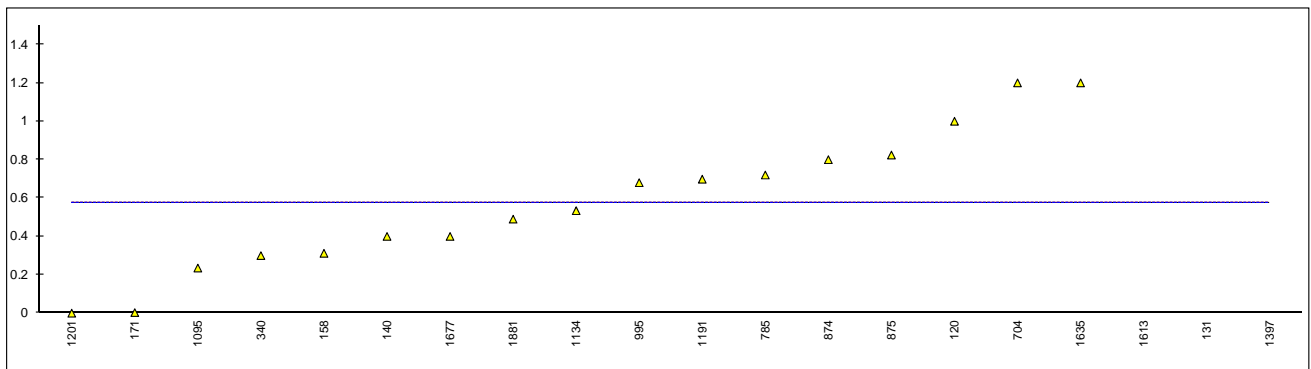
Determination of Nickel on sample #14255; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52		----		----	
62	IP470	<1		----	
120	IP501	0.2		-0.38	
131	IP501	0.075		-0.77	
133		----		----	
140	IP501	0.27		-0.17	
150	IP501	<1		----	
158	IP501	0.505		0.56	
171	IP501	0.332		0.03	
228		----		----	
311	D7111	0.20		-0.38	
313		----		----	
323	IP501	<1		----	
333		----		----	
334		----		----	
336		----		----	
337		----		----	
340	IP501	0.3		-0.07	
356	IP501	1.2		2.72	
369	IP501	<1		----	
372		----		----	
445	IP501	<1		----	
492		----		----	
494	IP501	<1		----	
657	IP501	<1		----	
704	IP PM CW	0.19		-0.41	
705	IP PM CW	0.21		-0.35	
781	IP PM CW	0.3		-0.07	
785	IP470	0.19		-0.41	
791	IP470	1		2.10	
874	IP470	0.3		-0.07	
875	IP501	0.23		-0.29	
966		----		----	
995	D5863	0.21		-0.35	
1016	in house	<2		----	
1065	in house	0.15		-0.54	
1066		----		----	
1095	D5708	0.296		-0.08	
1134	IP501	0.159		-0.51	
1191		----		----	
1201	IP501	3	R(0.01)	8.29	
1297	D5708	0.269		-0.17	
1340		----		----	
1397	ISO11885	<0.015		----	
1455	IP PM CW	0.1		-0.69	
1521		----		----	
1543		----		----	
1556	INH-1020	<0.1		----	
1613	IP470	<1.0		----	
1635	IP501	2.5	R(0.01)	6.75	
1677	IP501	0.5		0.55	
1881	IP PM CW	0.25		-0.23	
	normality	not OK			
	n	23			
	outliers	2			
	mean (n)	0.3233			
	st.dev. (n)	0.26742			
	R(calc.)	0.7488			
	R(IP501:05)	0.9036			Application range : 1 – 100 mg/kg
	compare				
	R(IP470:05)	1.3265			Application range : 1 – 100 mg/kg
	R(IP PM CW/04)	unknown			Application range: 0.1 – 10 mg/kg



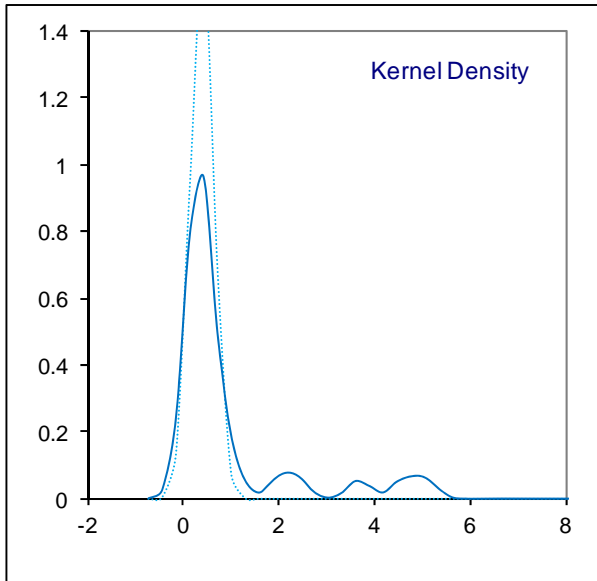
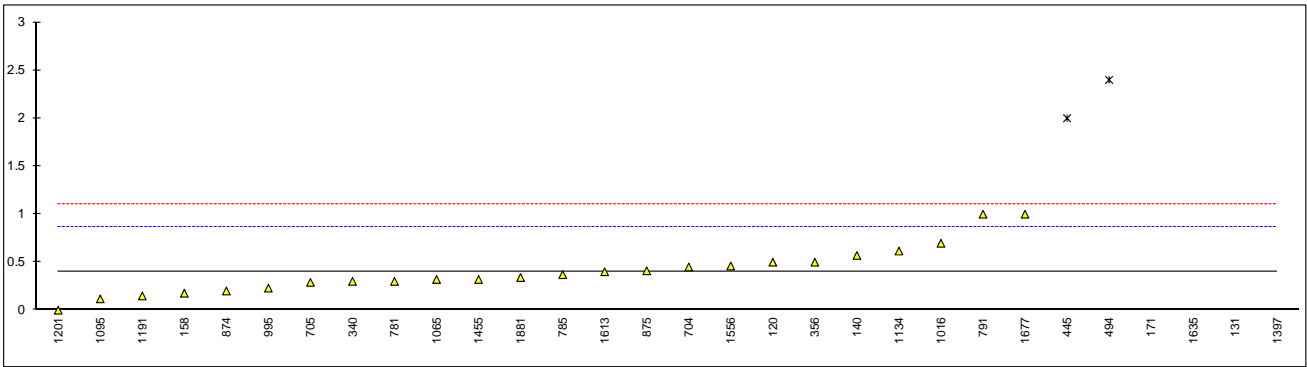
Determination of Silicon on sample #14255; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52		----		----	
62	IP470	<1		----	
120	IP501	1.0		----	
131	IP501	17.71	R(0.01)	----	false positive test result?
133		----		----	
140	IP501	0.40		----	
150	IP501	<1		----	
158	IP501	0.312		----	
171	IP501	0.003		----	
228		----		----	
311	UOP796	<1.0		----	
313		----		----	
323	IP501	<10		----	
333		----		----	
334		----		----	
336		----		----	
337		----		----	
340	IP501	0.3		----	
356	IP501	<5		----	
369	IP501	<10		----	
372	IP470	<10		----	
445	IP501	<10		----	
492		----		----	
494	IP501	<10		----	
657	IP501	<10		----	
704	IP470	1.2		----	
705		----		----	
781		----		----	
785	IP470	0.72		----	
791		----		----	
874	IP470	0.8		----	
875	IP501	0.824		----	
966		----		----	
995	IP377	0.68		----	
1016		----		----	
1065		----		----	
1066		----		----	
1095	D5708	0.235		----	
1134	IP501	0.534		----	
1191	D5185	0.698		----	
1201	IP501	0		----	
1297		----		----	
1340		----		----	
1397	ISO11885	22.5	R(0.01)	----	false positive test result?
1455		----		----	
1521		----		----	
1543		----		----	
1556		----		----	
1613	IP470	4.3		----	
1635	IP501	1.2		----	
1677	IP501	0.4		----	
1881	IP470	0.49		----	
	normality	not OK			
	n	18			
	outliers	2			
	mean (n)	0.7831			
	st.dev. (n)	0.94577			
	R(calc.)	2.6481			
	R(IP501:05)	(0.2600)			Application range : 10 – 250 mg/kg
	compare				
	R(IP470:05)	1.1783			Application range : 10 – 250 mg/kg
	R(IP PM CW/04)	unknown			Application range: 0.1 – 10 mg/kg



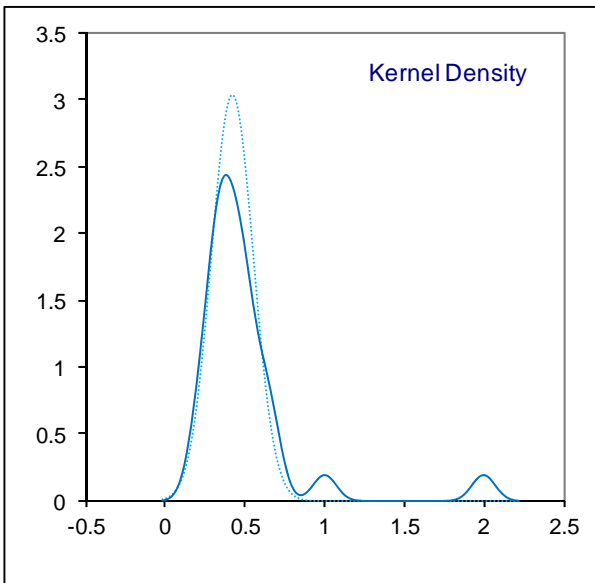
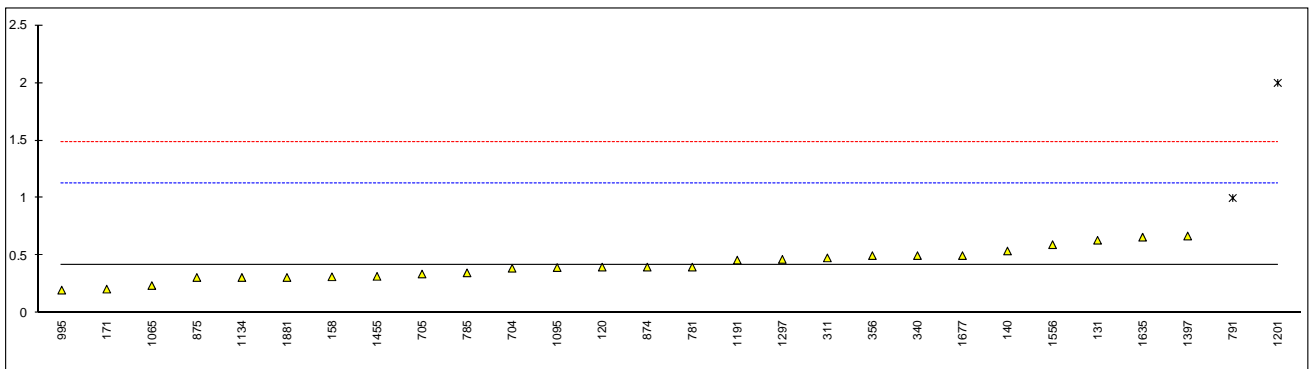
Determination of Sodium on sample #14255; results in mg/kg

lab	method	value	mark	z(targ)	remarks
52		----		----	
62	IP470	<1		----	
120	IP501	0.5		0.41	
131	IP501	5.084	R(0.01)	20.20	
133		----		----	
140	IP501	0.57		0.71	
150	IP501	<1		----	
158	IP501	0.177		-0.98	
171	IP501	3.686	R(0.01)	14.16	
228		----		----	
311	D7111	<0.10		----	
313		----		----	
323	IP501	<1		----	
333		----		----	
334		----		----	
336		----		----	
337		----		----	
340	IP501	0.3		-0.45	
356	IP501	0.5		0.41	
369	IP501	<1		----	
372		----		----	
445	IP501	2.0	R(0.01)	6.89	
492		----		----	
494	IP501	2.4	R(0.01)	8.61	
657	IP501	<1		----	
704	IP PM CW	0.45		0.19	
705	IP PM CW	0.29		-0.50	
781	IP PM CW	0.3		-0.45	
785	IP470	0.37		-0.15	
791	IP470	1		2.57	
874	IP470	0.2		-0.88	
875	IP501	0.41		0.02	
966		----		----	
995	D5863	0.23		-0.75	
1016	NEN6966	0.697		1.26	
1065	in house	0.32		-0.37	
1066		----		----	
1095	D5708	0.118		-1.24	
1134	IP501	0.616		0.91	
1191	D5185	0.1483		-1.11	
1201	IP501	0		-1.75	
1297		----		----	
1340		----		----	
1397	ISO11885	72.1	R(0.01)	309.45	
1455	IP PM CW	0.32		-0.37	
1521		----		----	
1543		----		----	
1556	INH-1020	0.460		0.24	
1613	IP470	0.4		-0.02	
1635	IP501	4.6	R(0.01)	18.11	
1677	IP501	1.0		2.57	
1881	IP PM CW	0.34		-0.28	
	normality	not OK			
	n	24			
	outliers	6			
	mean (n)	0.4048			
	st.dev. (n)	0.24498			
	R(calc.)	0.6859			
	R(IP501:05)	0.6487			Application range : 1 – 100 mg/kg
	compare				
	R(IP470:05)	0.7574			Application range : 1 – 100 mg/kg
	R(IP PM CW/04)	unknown			Application range: 0.1 – 10 mg/kg



Determination of Vanadium on sample #14255; results in mg/kg

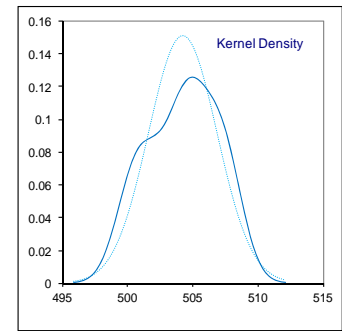
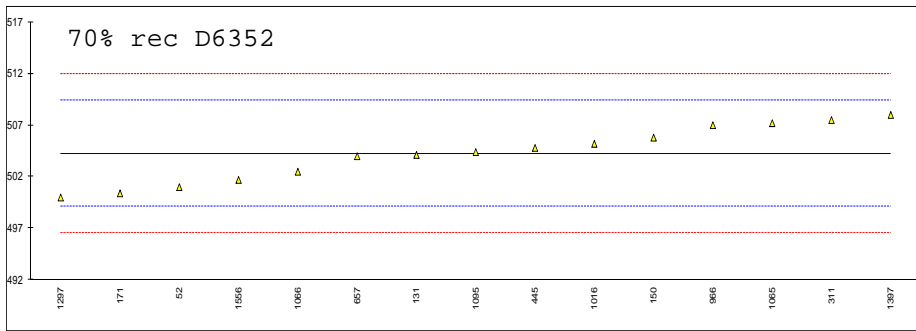
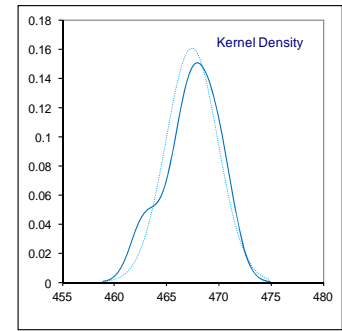
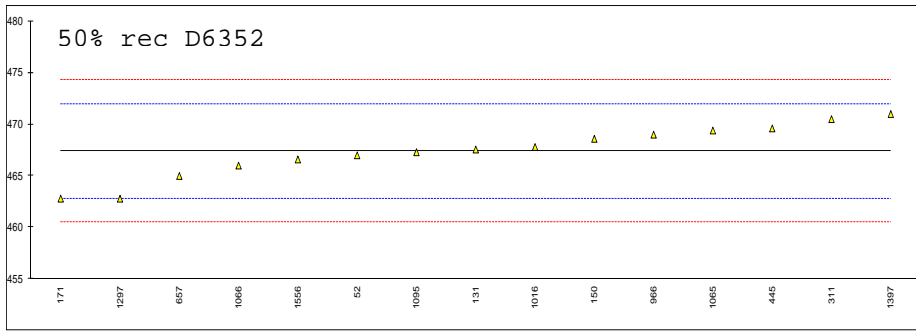
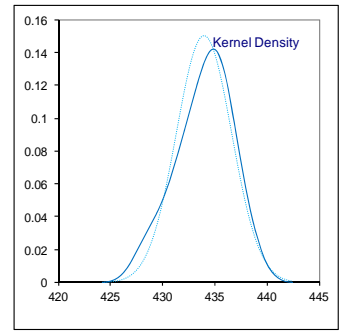
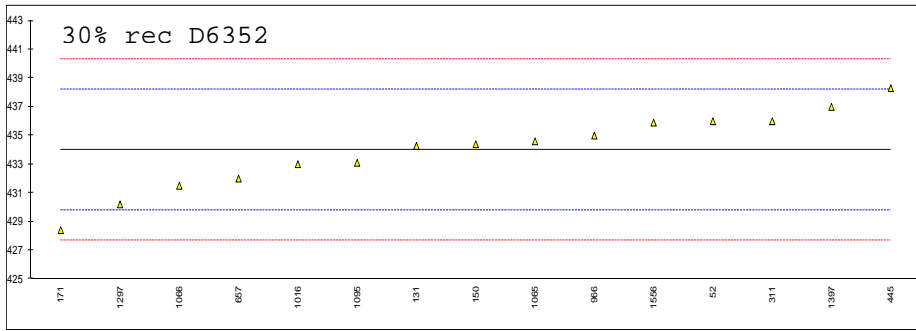
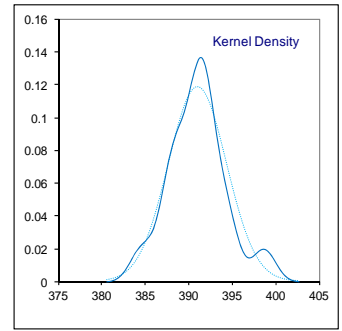
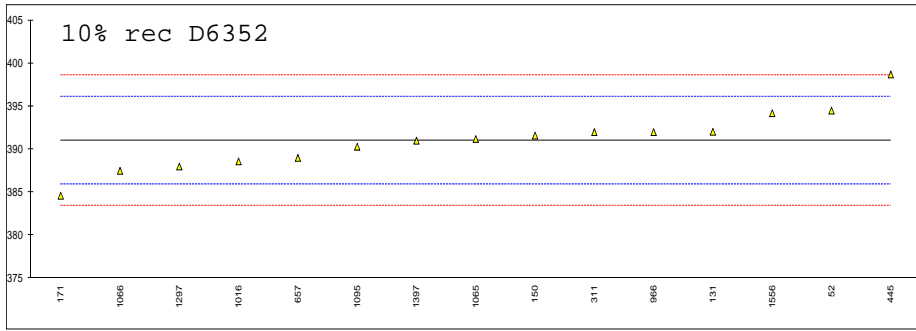
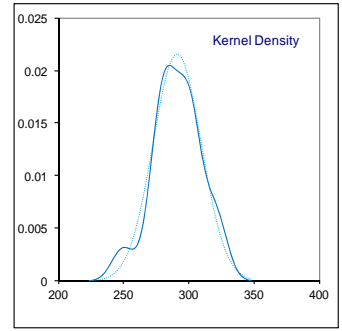
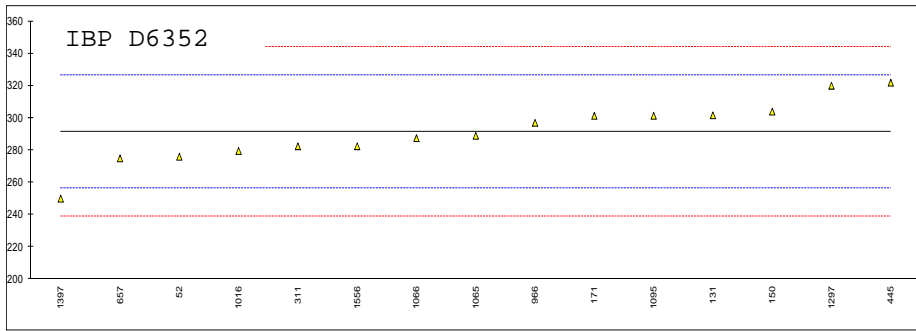
lab	method	value	mark	z(targ)	remarks
52		----		----	
62	IP470	<1		----	
120	IP501	0.4		-0.05	
131	IP501	0.633		0.60	
133		----		----	
140	IP501	0.54		0.34	
150	IP501	<1		----	
158	IP501	0.316		-0.29	
171	IP501	0.209		-0.59	
228		----		----	
311	D7111	0.48		0.17	
313		----		----	
323	IP501	<1		----	
333		----		----	
334		----		----	
336		----		----	
337		----		----	
340	IP501	0.5		0.23	
356	IP501	0.5		0.23	
369	IP501	<1		----	
372		----		----	
445	IP501	<1		----	
492		----		----	
494	IP501	<1		----	
657	IP501	<1		----	
704	IP PM CW	0.39		-0.08	
705	IP PM CW	0.34		-0.22	
781	IP PM CW	0.4		-0.05	
785	IP470	0.35		-0.19	
791	IP470	1	R(0.01)	1.63	
874	IP470	0.4		-0.05	
875	IP501	0.31		-0.31	
966		----		----	
995	D5863	0.20		-0.62	
1016	in house	<2		----	
1065	in house	0.24		-0.50	
1066		----		----	
1095	D5708	0.396		-0.06	
1134	IP501	0.310		-0.31	
1191	D5185	0.461		0.12	
1201	IP501	2	R(0.01)	4.44	
1297	D5708	0.467		0.13	
1340		----		----	
1397	ISO11885	0.67		0.70	
1455	IP PM CW	0.32		-0.28	
1521		----		----	
1543		----		----	
1556	INH-1020	0.595		0.49	
1613		----		----	
1635	IP501	0.66		0.68	
1677	IP501	0.5		0.23	
1881	IP PM CW	0.31		-0.31	
	normality	OK			
	n	26			
	outliers	2			
	mean (n)	0.4191			
	st.dev. (n)	0.13169			
	R(calc.)	0.3687			
	R(IP501:05)	0.9970			Application range: 1 – 400 mg/kg
	compare				
	R(IP470:05)	2.110			Application range: 1 – 400 mg/kg
	R(IP PM CW/04)	unknown			Application range: 0.1 – 10 mg/kg

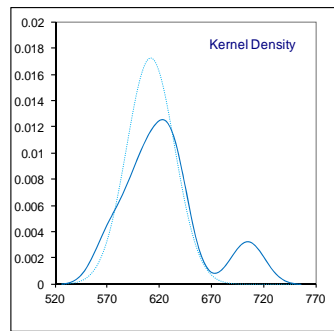
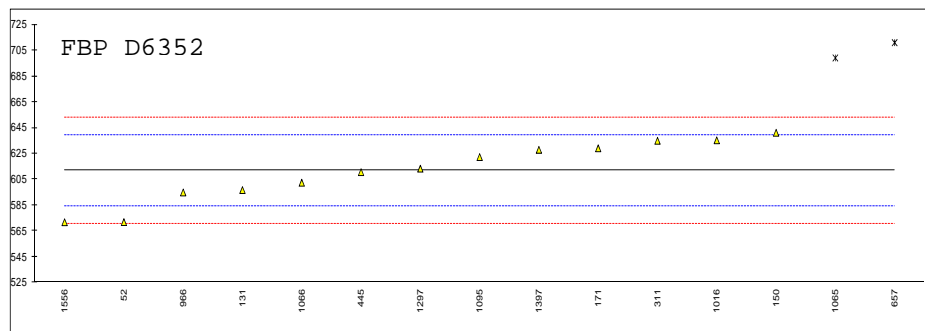
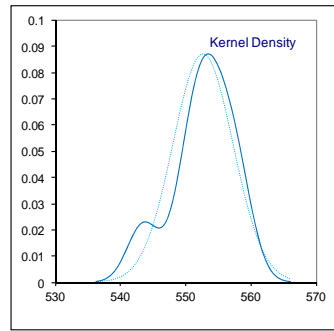
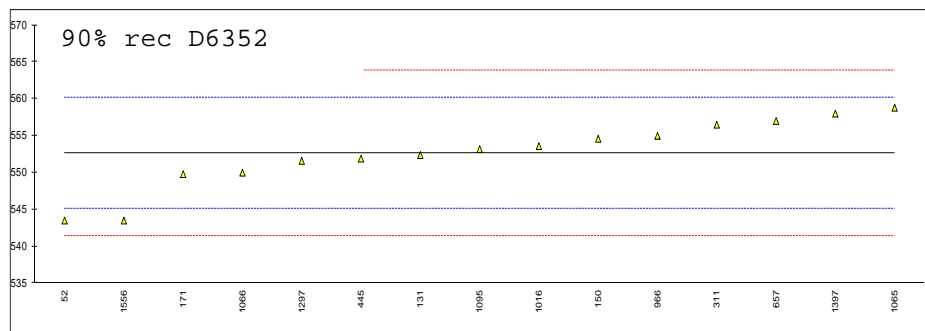


Determination of Sim. Dist. D6352 on sample #14255; result in °C

lab	method	IBP	10% rec	30% rec	50% rec	70% rec	90% rec	FBP
52	D7213	276.0	394.5	436.0	467.0	501.0	543.5	572.0
62		----	----	----	----	----	----	----
120		----	----	----	----	----	----	----
131	D6352	301.67	392.04	434.28	467.56	504.12	552.39	596.83
133		----	----	----	----	----	----	----
140		----	----	----	----	----	----	----
150	D7169	304.0	391.6	434.4	468.6	505.8	554.6	641.2
158		----	----	----	----	----	----	----
171	D6352	301.4	384.6	428.4	462.8	500.4	549.8	629.2
228		----	----	----	----	----	----	----
311	D6352	282.5	392.0	436.0	470.5	507.5	556.5	635.0
313		----	----	----	----	----	----	----
323		----	----	----	----	----	----	----
333		----	----	----	----	----	----	----
334		----	----	----	----	----	----	----
336		----	----	----	----	----	----	----
337		----	----	----	----	----	----	----
340		----	----	----	----	----	----	----
356		----	----	----	----	----	----	----
369		----	----	----	----	----	----	----
372		----	----	----	----	----	----	----
445	D2887	321.9	398.7	438.3	469.6	504.8	551.9	610.7
492		----	----	----	----	----	----	----
494		----	----	----	----	----	----	----
657	D6352	275.0	389.0	432.0	465.0	504.0	557.0	<u>711.0</u>
704		----	----	----	----	----	----	----
705		----	----	----	----	----	----	----
781		----	----	----	----	----	----	----
785		----	----	----	----	----	----	----
791		----	----	----	----	----	----	----
874		----	----	----	----	----	----	----
875		----	----	----	----	----	----	----
966	D2887	297.00	392.00	435.00	469.00	507.00	555.00	595.00
995		----	----	----	----	----	----	----
1016	D7169	279.6	388.6	433.0	467.8	505.2	553.6	635.4
1065	D6352	289.0	391.2	434.6	469.4	507.2	558.8	<u>699.2</u>
1066	D6352	287.5	387.5	431.5	466.0	502.5	550.0	602.5
1095	D7169	301.4	390.3	433.1	467.3	504.4	553.2	622.4
1134		----	----	----	----	----	----	----
1191		----	----	----	----	----	----	----
1201		----	----	----	----	----	----	----
1297	D6352	320.0	388.0	430.2	462.8	500.0	551.6	613.4
1340		----	----	----	----	----	----	----
1397	D6352	250	391	437	471	508	558	628
1455		----	----	----	----	----	----	----
1521		----	----	----	----	----	----	----
1543		----	----	----	----	----	----	----
1556	ISO3924	282.5	394.2	435.9	466.6	501.7	543.5	571.9
1613		----	----	----	----	----	----	----
1635		----	----	----	----	----	----	----
1677		----	----	----	----	----	----	----
1881		----	----	----	----	----	----	----
	normality	OK	suspect	OK	OK	OK	OK	OK
	n	15	15	15	15	15	15	13
	outliers	0	0	0	0	0	0	2
	mean (n)	291.30	391.02	433.98	467.40	504.24	552.63	611.81
	st.dev. (n)	18.482	3.356	2.653	2.485	2.640	4.591	23.112
	R(calc.)	51.75	9.40	7.43	6.96	7.39	12.85	64.71
	R(D6352:14)	49.10	7.10	5.90	6.40	7.20	10.50	38.10

Test results underlined and bold belong to the statistical outliers acc. to Rosner/Grubbs/Dixon outlier test





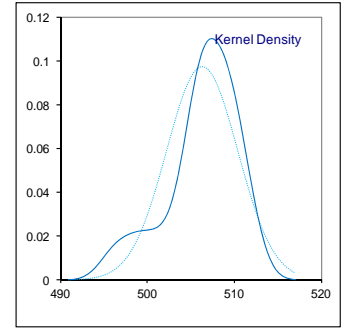
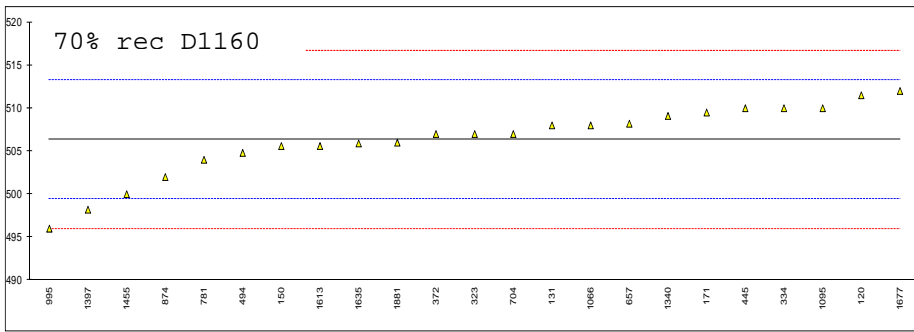
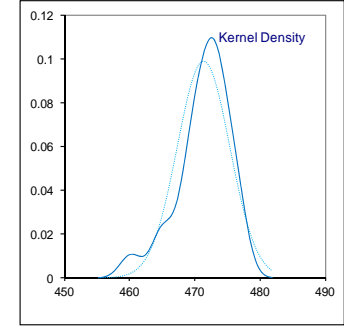
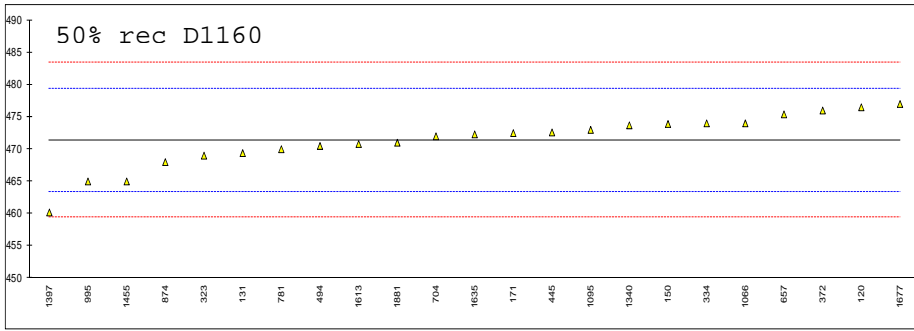
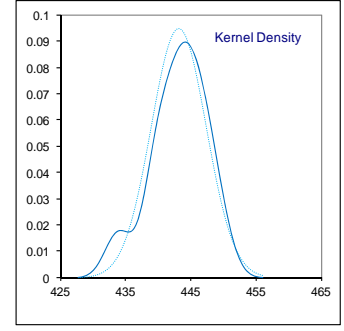
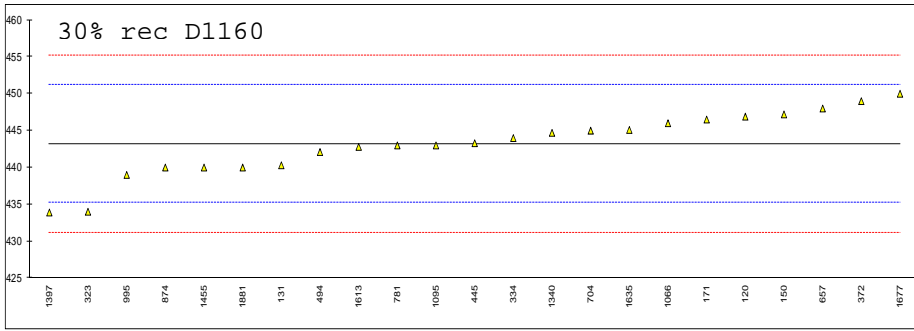
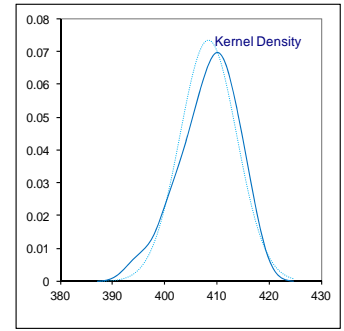
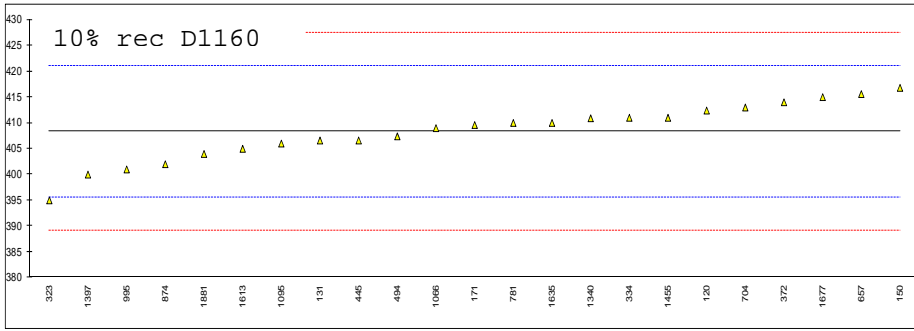
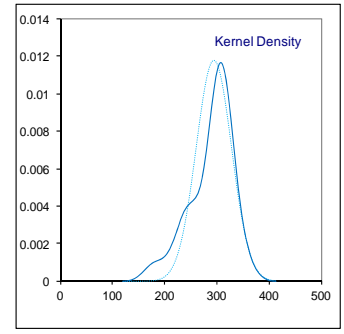
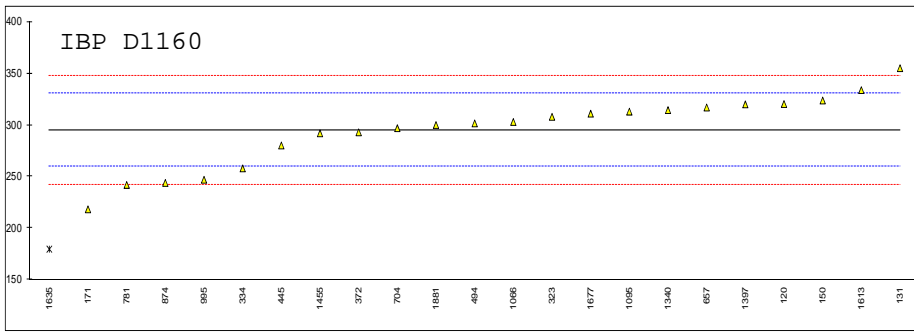
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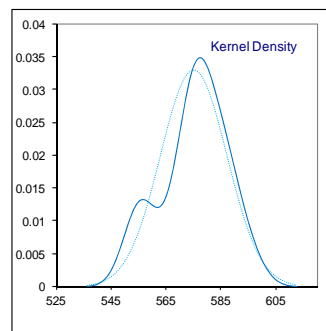
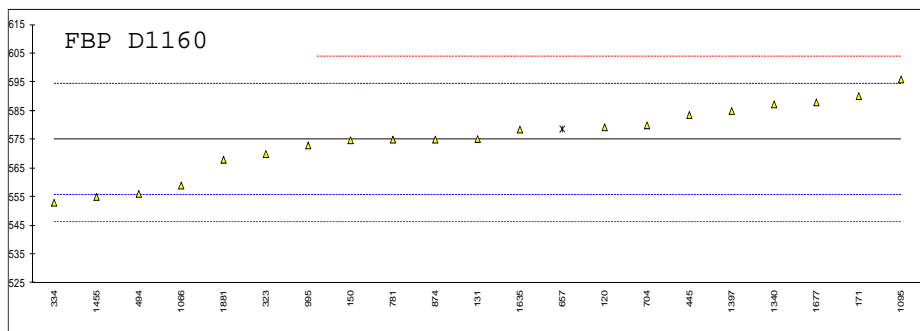
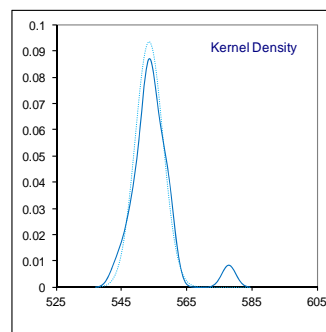
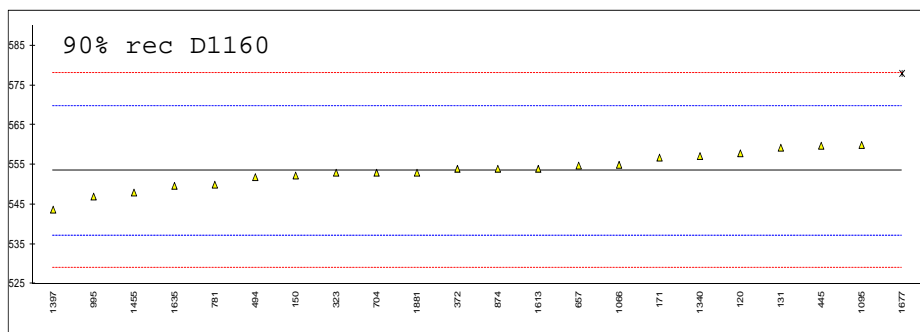
Determination of Distillation D1160 on sample #14255; result in °C

lab	method	IBP	10% rec	30% rec	50% rec	70% rec	90% rec	FBP
52		----	----	----	----	----	----	----
62		----	----	----	----	----	----	----
120	D1160	320.4	412.4	446.9	476.5	511.5	557.9	579.3
131	D1160	355.1	406.6	440.3	469.4	508.0	559.3	575.2
133		----	----	----	----	----	----	----
140		----	----	----	----	----	----	----
150	D1160	323.8	416.8	447.2	473.9	505.6	552.3	574.8
158		----	----	----	----	----	----	----
171	D1160	218.4	409.6	446.5	472.5	509.5	556.8	590.2
228		----	----	----	----	----	----	----
311		----	----	----	----	----	----	----
313		----	----	----	----	----	----	----
323	D1160	308	395	434	469	507	553	570
333		----	----	----	----	----	----	----
334	D1160	258	411	444	474	510	----	553
336		----	----	----	----	----	----	----
337		----	----	----	----	----	----	----
340		----	----	----	----	----	----	----
356		----	----	----	----	----	----	----
369		----	----	----	----	----	----	----
372	D1160	293	414	449	476	507	554	----
445	D1160	280.2	406.6	443.3	472.6	510.0	559.8	583.6
492		----	----	----	----	----	----	----
494	D1160	301.6	407.4	442.1	470.5	504.8	551.9	556.1
657	D1160	317.0	415.6	448.0	475.4	508.2	554.8	<u>578.7</u>
704	D1160	297	413	445	472	507	553	580
705		----	----	----	----	----	----	----
781	D1160	242	410	443	470	504	550	575
785		----	----	----	----	----	----	----
791		----	----	----	----	----	----	----
874	D1160	244	402	440	468	502	554	575
875		----	----	----	----	----	----	----
966		----	----	----	----	----	----	----
995	D1160	247.0	401.0	439.0	465.0	496.0	547.0	573.0
1016		----	----	----	----	----	----	----
1065		----	----	----	----	----	----	----
1066	D1160	303	409	446	474	508	555	559
1095	D1160	313	406	443	473	510	560	596
1134		----	----	----	----	----	----	----
1191		----	----	----	----	----	----	----
1201		----	----	----	----	----	----	----
1297		----	----	----	----	----	----	----
1340	D1160	314.5	410.9	444.7	473.7	509.1	557.2	587.3
1397	D1160	320	400	433.9	460.2	498.2	543.7	585
1455	D1160	292	411	440	465	500	548	555
1521		----	----	----	----	----	----	----
1543		----	----	----	----	----	----	----
1556		----	----	----	----	----	----	----
1613	D1160	333.8	405.0	442.8	470.8	505.6	554.0	----
1635	D1160	179.8	410.0	445.1	472.3	505.9	549.7	578.5
1677	D1160	311	415	450	477	512	<u>578</u>	588
1881	D1160	300	404	440	471	506	553	568
	normality	OK	OK	OK	suspect	OK	OK	OK
	n	22	23	23	23	23	21	20
	outliers	1	0	0	0	0	1	0 + 1 excl.
	mean (n)	295.13	408.34	443.21	471.38	506.32	553.54	575.10
	st.dev. (n)	33.911	5.440	4.213	4.034	4.091	4.267	12.133
	R(calc.)	94.95	15.23	11.80	11.29	11.45	11.95	33.97
	R(D1160:13)	49.45	17.88	11.21	11.22	9.66	22.85	26.89

Test result underlined and bold belongs to the statistical outliers acc. to Rosner/Grubbs/Dixon outlier test

Bold, Underlined and Italic test result excluded; lab 675 reported FBP as stop point 96% vol





APPENDIX 2

z-scores Simulated Distillation according to ASTM D6352 on sample #14255

lab	IBP	10%	30%	50%	70%	90%	FBP
52	-0.87	1.37	0.96	-0.17	-1.26	-2.43	-2.93
62	----	----	----	----	----	----	----
120	----	----	----	----	----	----	----
131	0.59	0.40	0.14	0.07	-0.05	-0.06	-1.10
133	----	----	----	----	----	----	----
140	----	----	----	----	----	----	----
150	0.72	0.23	0.20	0.53	0.61	0.53	2.16
158	----	----	----	----	----	----	----
171	0.58	-2.53	-2.65	-2.01	-1.49	-0.75	1.28
228	----	----	----	----	----	----	----
311	-0.50	0.39	0.96	1.36	1.27	1.03	1.70
313	----	----	----	----	----	----	----
323	----	----	----	----	----	----	----
333	----	----	----	----	----	----	----
334	----	----	----	----	----	----	----
336	----	----	----	----	----	----	----
337	----	----	----	----	----	----	----
340	----	----	----	----	----	----	----
356	----	----	----	----	----	----	----
369	----	----	----	----	----	----	----
372	----	----	----	----	----	----	----
445	1.75	3.03	2.05	0.96	0.22	-0.19	-0.08
492	----	----	----	----	----	----	----
494	----	----	----	----	----	----	----
657	-0.93	-0.80	-0.94	-1.05	-0.09	1.17	<u>7.29</u>
704	----	----	----	----	----	----	----
705	----	----	----	----	----	----	----
781	----	----	----	----	----	----	----
785	----	----	----	----	----	----	----
791	----	----	----	----	----	----	----
874	----	----	----	----	----	----	----
875	----	----	----	----	----	----	----
966	0.33	0.39	0.48	0.70	1.07	0.63	-1.24
995	----	----	----	----	----	----	----
1016	-0.67	-0.95	-0.46	0.18	0.37	0.26	1.73
1065	-0.13	0.07	0.29	0.88	1.15	1.65	<u>6.42</u>
1066	-0.22	-1.39	-1.18	-0.61	-0.68	-0.70	-0.68
1095	0.58	-0.28	-0.42	-0.04	0.06	0.15	0.78
1134	----	----	----	----	----	----	----
1191	----	----	----	----	----	----	----
1201	----	----	----	----	----	----	----
1297	1.64	-1.19	-1.79	-2.01	-1.65	-0.27	0.12
1340	----	----	----	----	----	----	----
1397	-2.36	-0.01	1.43	1.58	1.46	1.43	1.19
1455	----	----	----	----	----	----	----
1521	----	----	----	----	----	----	----
1543	----	----	----	----	----	----	----
1556	-0.50	1.26	0.91	-0.35	-0.99	-2.43	-2.93
1613	----	----	----	----	----	----	----
1635	----	----	----	----	----	----	----
1677	----	----	----	----	----	----	----
1881	----	----	----	----	----	----	----

Z-scores underlined and bold belong to the statistical outliers acc. to Rosner/Grubbs/Dixon outlier test.

z-scores Distillation according to ASTM D1160 on sample #14255

lab	IBP	10%	30%	50%	70%	90%	FBP
52	----	----	----	----	----	----	----
62	----	----	----	----	----	----	----
120	1.43	0.64	0.92	1.28	1.50	0.53	0.44
131	3.40	-0.27	-0.73	-0.49	0.49	0.71	0.01
133	----	----	----	----	----	----	----
140	----	----	----	----	----	----	----
150	1.62	1.32	1.00	0.63	-0.21	-0.15	-0.03
158	----	----	----	----	----	----	----
171	-4.34	0.20	0.82	0.28	0.92	0.40	1.57
228	----	----	----	----	----	----	----
311	----	----	----	----	----	----	----
313	----	----	----	----	----	----	----
323	0.73	-2.09	-2.30	-0.59	0.20	-0.07	-0.53
333	----	----	----	----	----	----	----
334	-2.10	0.42	0.20	0.65	1.07	----	-2.30
336	----	----	----	----	----	----	----
337	----	----	----	----	----	----	----
340	----	----	----	----	----	----	----
356	----	----	----	----	----	----	----
369	----	----	----	----	----	----	----
372	-0.12	0.89	1.45	1.15	0.20	0.06	----
445	-0.85	-0.27	0.02	0.30	1.07	0.77	0.88
492	----	----	----	----	----	----	----
494	0.37	-0.15	-0.28	-0.22	-0.44	-0.20	-1.98
657	1.24	1.14	1.20	1.00	0.54	0.15	<i>0.37</i>
704	0.11	0.73	0.45	0.15	0.20	-0.07	0.51
705	----	----	----	----	----	----	----
781	-3.01	0.26	-0.05	-0.35	-0.67	-0.43	-0.01
785	----	----	----	----	----	----	----
791	----	----	----	----	----	----	----
874	-2.90	-0.99	-0.80	-0.84	-1.25	0.06	-0.01
875	----	----	----	----	----	----	----
966	----	----	----	----	----	----	----
995	-2.73	-1.15	-1.05	-1.59	-2.99	-0.80	-0.22
1016	----	----	----	----	----	----	----
1065	----	----	----	----	----	----	----
1066	0.45	0.10	0.70	0.65	0.49	0.18	-1.68
1095	1.01	-0.37	-0.05	0.40	1.07	0.79	2.18
1134	----	----	----	----	----	----	----
1191	----	----	----	----	----	----	----
1201	----	----	----	----	----	----	----
1297	----	----	----	----	----	----	----
1340	1.10	0.40	0.37	0.58	0.80	0.45	1.27
1397	1.41	-1.31	-2.33	-2.79	-2.35	-1.21	1.03
1455	-0.18	0.42	-0.80	-1.59	-1.83	-0.68	-2.09
1521	----	----	----	----	----	----	----
1543	----	----	----	----	----	----	----
1556	----	----	----	----	----	----	----
1613	2.19	-0.52	-0.10	-0.15	-0.21	0.06	----
1635	<u>-6.53</u>	0.26	0.47	0.23	-0.12	-0.47	0.35
1677	0.90	1.04	1.70	1.40	1.65	<u>3.00</u>	1.34
1881	0.28	-0.68	-0.80	-0.10	-0.09	-0.07	-0.74

Z-scores underlined and bold belong to the statistical outliers acc. to Rosner/Grubbs/Dixon outlier test.

Z-score underlined, bold and italic: Lab 657 excluded: result reported as stop point 96% vol.

APPENDIX 3

Number of participants per country

1 lab in BELGIUM
1 lab in BOSNIA and HERZEGOVINA
2 labs in CANADA
2 labs in CROATIA
2 labs in ESTONIA
1 lab in FINLAND
5 labs in FRANCE
1 lab in GEORGIA
2 labs in GERMANY
1 lab in GREECE
1 lab in ISRAEL
1 lab in JORDAN
1 lab in LATVIA
1 lab in LITHUANIA
1 lab in MALTA
6 labs in NETHERLANDS
1 lab in POLAND
1 lab in PORTUGAL
5 labs in RUSSIAN FEDERATION
1 lab in SAUDI ARABIA
1 lab in SINGAPORE
1 lab in SUDAN
1 lab in SWEDEN
1 lab in TOGO
2 labs in UKRAINE
2 labs in UNITED KINGDOM
7 labs in UNITED STATES OF AMERICA

APPENDIX 4

Abbreviations:

C	= final result after checking of first reported suspect result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner outlier test
R(0.05)	= straggler in Rosner outlier test
ex	= excluded from calculations
E	= error in calculations
n.a.	= not applicable

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