

Results of Proficiency Test
Engine Oil (Fresh)
June 2015

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 1997, the Institute for Interlaboratory Studies organises every year a proficiency test for fresh Lubricating Oil (Engine Oil). In the annual proficiency testing program 2014/2015, it was decided to continue the proficiency test for the analyses of Engine Oil (fresh). In this interlaboratory study, 73 laboratories in 40 different countries have participated. See appendix 2 for the number of participants per country. In this report, the results of the 2015 Engine Oil (fresh) proficiency test are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test. It was decided to send to each laboratory 1 * 1 litre bottle and 1 * 0.5 litre bottle of Engine Oil (Fresh), both labelled #15080.

The analyses for fit-for-use and homogeneity were subcontracted. Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC 17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation 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 is electronically available through the iis internet site 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

The necessary bulk material was obtained from a local supplier. From this 200 litre batch, after homogenizing, 100 brown glass bottles of 1 litre and 100 brown glass bottles of 0.5 litre were filled (both labelled #15080). The homogeneity of the subsamples #15080 was checked by determination of Density at 15°C in accordance with ASTM D4052 and Kinematic Viscosity at 40°C in accordance with ASTM D445 on 8 stratified randomly selected samples.

	Density at 15 °C in kg/L	Kinematic Viscosity at 40°C in mm ² /s
Sample #15080-1	0.86637	76.53
Sample #15080-2	0.86638	76.52
Sample #15080-3	0.86637	76.51
Sample #15080-4	0.86637	76.51
Sample #15080-5	0.86636	76.47
Sample #15080-6	0.86637	76.46
Sample #15080-7	0.86637	76.47
Sample #15080-8	0.86637	76.46

Table 1: homogeneity test results of subsamples #15080

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

	Density at 15 °C in kg/L	Kinematic Viscosity at 40°C in mm ² /s
r (sample #15080)	0.00001	0.08
reference test	ASTM D4052:11	ASTM D445:15
0.3 x R(reference test)	0.00015	0.28

Table 2: evaluation of the repeatabilities of the subsamples #15080

The calculated repeatabilities were less than 0.3 times the corresponding reproducibilities of the reference methods. Therefore, homogeneity of the subsamples #15080 was assumed.

To each of the participating laboratories, one sample of 1 L and one sample of 0.5 L (both labelled, #15080) were sent on May 20, 2015.

2.5 ANALYSES

The participants were requested to determine on sample #15080: Acid Number, Base Number, Color ASTM, Conradson Carbon Residue, Ramsbottom Carbon Residue, Carbon Residue (Micro method), Density at 15°C, Evaporation loss by Noack, Flash Point COC, Flash Point PMcc, Foaming Tendency, Foam Stability, Kinematic Viscosity at 40°C and at 100°C, Viscosity Index, Viscosity Stabinger at 40°C and at 100°C, Viscosity Apparent (CSS) at -20°C, Viscosity HTHS by Tapered Bearing Simulator, Nitrogen, Pour Point (manual, automated), Sulphated Ash, Sulphur, Water, Calcium, Phosphorus and Zinc.

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 gathered. The original data are tabulated per determination in the appendix of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder fax was sent to those laboratories that had not reported results at that moment.

Shortly after the deadline, the available results were screened for suspect data. A result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the results. Additional or corrected results are used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' (iis-protocol, version 3.3) of April 2014.

For 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.

According to ISO 5725 the original results per determination were submitted to Dixon's and/or Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test (ref. 15). 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. When the uncertainty passed the evaluation no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

Finally, the reproducibilities were calculated from the standard deviations by multiplying these 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 analysis 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 standard. Outliers and other data, which were excluded from the calculations, are represented as a "x". Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3; nos.13 and 14). Also a normal Gauss curve was projected over the Kernel Density Graph.

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

The z-scores were calculated according to:

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

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, some problems with sample despatch were encountered by a number of laboratories. Six participants reported after the final reporting date and only one participant did not report any test results at all. Not all laboratories were able to report all analyses requested. In total 72 participants reported 961 test results. Observed were 40 outlying results, which is 4.2%. 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. D189) and an added designation for the year that the method was adopted or revised (e.g. D189-06). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D189-06(2010)). In the results tables of Appendix 1 only the method number and year of adoption or revision will be used.

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

Acid Number : This determination was very problematic. Four statistical outliers were observed. The test results of five laboratories, that reported to have used ASTM D974, were excluded because this test method is not equivalent to ASTM D664. The calculated reproducibility after rejection of the suspect data is not at all agreement with the requirements of ASTM D664:11a. In Table 1 of ASTM D664:11a the recommended size of the test portion is given. Results using smaller sample size may not be equivalent to results obtained with the recommended sample size (See note 13 of ASTM D664:11a).

Base Number : This determination was problematic. One statistical outlier was observed. The test results of two laboratories, that reported to have used resp. ASTM D4739 and IP400, were excluded because these test methods are not equivalent to ASTM D2896. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of ASTM D2896:11. When the reported data of ASTM D2896 were evaluated separately for procedure A and B the calculated reproducibility for procedure B is in good agreement with the requirements of the standard. The calculated reproducibility for procedure A is not in agreement.

- Color ASTM: This determination was not problematic. Only one statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in good agreement with ASTM D1500:12.
- Conradson CR: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D189:06(2010).
- Ramsbottom CR: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D524:10. The low number of results may (partly) explain the observed large spread.
- Carbon Residue (Micro method) This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D4530:11.
- Density at 15°C: This determination was problematic. Seven statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D4052:11.
- Evaporation loss by Noack This determination was problematic for a number laboratories. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D5800:15 procedure A, but not the the requirements of ASTM D5808:15 procedure B.
- Flash Point COC: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D92:12.
- Flash Point PMcc: This determination was problematic for a number of laboratories. One statistical outlier was observed and five results were excluded from statistical calculations as the laboratories reported to have used ASTM D93 procedure B, while procedure A is the only procedure for fresh oils. However, the calculated reproducibility after rejection of the suspect data is in agreement with ASTM D93:15-A.
- Foaming Tendency: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility for the three sequences I and III are in agreement with the requirements of ASTM D892:13. However, the calculated reproducibility of sequence II is not in agreement.
- Foam Stability: None of the reporting laboratories reported a positive result for the settling period after 10 min. Therefore all reporting participants agreed on a result of 0 (Nil).

- Kin.Visco at 40°C: This determination was not problematic. Only one statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D445:15.
- Kin.Visco.at 100°C: This determination was problematic for a number of laboratories. Five statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D445:15.
- Viscosity Index This determination was problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D2270:10. Two calculation errors were observed.
- Visco. Stabinger at 40°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D7042:14.
- Visco. Stabinger at 100°C: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements ASTM D7042:14.
- Viscosity, Apparent at -20°C: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D5293:15.
- Viscosity, HTHS by TBS: This determination may not be problematic. Only four test results were reported. The calculated reproducibility is in agreement with the requirements of ASTM D4683:13.
- Nitrogen: This determination was very problematic. No statistical outliers were observed. Four test results were excluded before the statistical evaluation as the used test method ASTM D4629 is not applicable for high viscosity liquids, nor for liquids containing more than 100 mg/kg nitrogen. The calculated reproducibility after rejection of the suspect data is not at all in agreement with the requirements of ASTM D3228:08 and ASTM D5762:11.
- Pour Point (manual): This determination was not problematic. Only one statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements ASTM D97:12.
- Pour Point (automated): This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with ASTM D5950:14.

- Sulphated Ash:** This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D874:13a.
- Sulphur:** This determination was very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all agreement with the requirements of ASTM D2622:10.
A matrix mismatch between sample and standards (e.g. different C/H ratio and/or the presence of interfering molecules) may (partly) explain the large spread. When the ASTM D2622:10 data was evaluated separately, the calculated reproducibility is much smaller and in agreement with the requirements of ASTM D2622:10. The calculated reproducibility for only ASTM D4294 data is not in agreement with the requirements of the standard.
- Water:** This determination was very problematic for the a number of laboratories. The preferred method to use for a product containing interfering components may be ASTM D6304:07 method C. This method is applicable for oils with difficult matrix interferences only. Sixteen laboratories reported results determined according ASTM D6304 method C. After excluding all results from other test methods the calculated reproducibility is in agreement with the requirements of ASTM D6304:07.
- Calcium:** This determination was very problematic. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ASTM D5185:13e.
- Phosphorus:** This determination was very problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ASTM D5185:13e.
- Zinc:** This determination was very problematic. Two one statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ASTM D5185:13e.

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, ISO and IP standards), are compared in the next table.

Parameter	unit	n	Average	2.8 * sd	R(lit)
Acid Number	mg KOH/g	38	2.62	1.34	0.51
Base Number	mg KOH/g	42	10.28	1.23	0.72
Color ASTM		34	3.8	0.9	1.0
Conradson Carbon Residue	%M/M	13	1.09	0.17	0.25
Ramsbottom Carbon Residue	%M/M	6	0.98	0.23	0.14
Carbon Residue (micro method)	%M/M	27	1.12	0.14	0.19
Density at 15°C	kg/L	58	0.8664	0.0009	0.0005
Evaporation loss by Noack	%M/M	11	12.28	2.17	2.25
Flash Point COC	°C	43	224.9	15.6	18.0
Flash Point PMcc	°C	39	196.4	12.1	13.9
Foaming Tendency, Sequence I	ml	21	1.7	10.2	14.6
Foaming Tendency, Sequence II	ml	21	24.8	46.5	24.8
Foaming Tendency, Sequence III	ml	21	1.7	10.2	14.6
Foam Stability, Sequence I	ml	21	0	n.a.	n.a.
Foam Stability, Sequence II	ml	21	0	n.a.	n.a.
Foam Stability, Sequence III	ml	21	0	n.a.	n.a.
Kinematic Viscosity at 40°C	mm ² /s	59	76.657	1.022	0.935
Kinematic Viscosity at 100°C	mm ² /s	50	11.654	0.114	0.161
Viscosity Index		49	145.5	3.0	2.0
Stabinger Viscosity at 40°C	mm ² /s	15	76.614	1.017	0.948
Stabinger Viscosity at 100°C	mm ² /s	12	11.691	0.120	0.123
Viscosity, Apparent at -20°C	mPa·s	8	3282	204	197
Viscosity, HTHS	mPa·s	4	3.485	0.109	0.151
Nitrogen	mg/kg	8	1218	716	200
Pour Point, manual	°C	33	-31.0	5.5	9.0
Pour Point, automated	°C	18	-32.5	7.9	4.5
Sulphated Ash	%M/M	35	0.97	0.26	0.14
Sulphur	%M/M	27	0.282	0.063	0.025
Water	mg/kg	16	191	263	394
Calcium	mg/kg	41	1261	428	161
Phosphorus	mg/kg	39	947	300	132
Zinc	mg/kg	41	1099	407	184

Table 3: reproducibilities of results of sample #15080 and comparisons with targets

Without further statistical calculations it can be concluded that for a number of tests there is a 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 JUNE 2015 WITH PREVIOUS PTs

	June 2015	June 2014	May 2013	May 2012
Number of reporting labs	72	87	78	78
Number of results reported	961	996	879	804
Statistical outliers	40	20	29	33
Percentage outliers	4.2%	2.0%	3.3%	4.1%

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	June 2015	June 2014	May 2013	May 2012
Acid Number	--	--	--	+/-
Base Number	--	+	-	--
Color ASTM	+	++	++	++
Conradson Carbon Residue	++	+	++	-
Ramsbottom Carbon Residue	--	--	--	-
Carbon Residue (Micro method)	++	-	+	
Density at 15°C	--	--	--	-
Evaporation loss by Noack	+/-			
Flash Point COC	++	+/-	+	+/-
Flash Point PMcc	++	+	++	++
Foaming Tendency	+/-	n.e.	n.e.	n.e.
Kinematic Viscosity at 40°C	+/-	-	-	-
Kinematic Viscosity at 100°C	++	--	--	--
Viscosity Index	--	--	--	n.e.
Stabinger Viscosity at 40°C	+/-	-	--	--
Stabinger Viscosity at 100°C	+/-	+/-	--	--
Viscosity, Apparent (CSS) at -20°C	+/-	+	+	n.e.
Viscosity HTHS by Tapered Bearing	+	n.e.	n.e.	n.e.
Nitrogen	--	--	-	--
Pour Point, manual	++	+	-	+/-
Pour Point, automated	--	+/-	+	-
Sulphated Ash	--	+	-	--
Sulphur	--	--	--	--
Water	++	+	+	++
Calcium	--	+	-	--
Phosphorus	--	-	-	--
Zinc	--	-	-	-

Table 5: comparison determinations against the standard

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

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

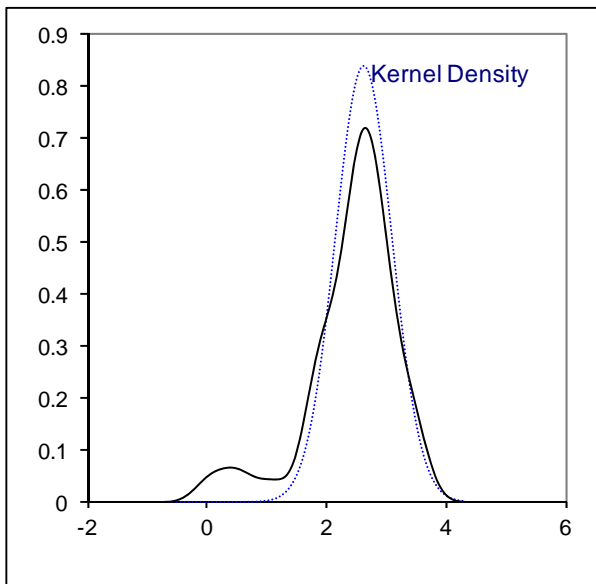
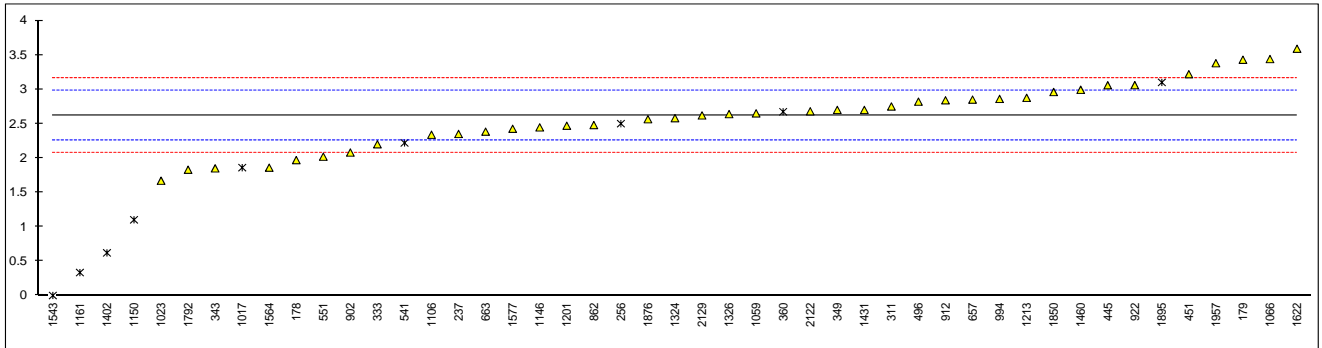
APPENDIX 1

Determination of Acid Number on sample #15080; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
173		----		----	
178	D664Mod.	1.97		-3.57	
179	D664	3.43		4.43	
237	D664	2.35		-1.49	
252		----		----	
254		----		----	
256	D974	2.5	ex	-0.67	result excluded see §4.1
311	D664	2.75		0.70	
315		----		----	
333	D664	2.2		-2.31	
343	D664	1.85		-4.23	
349	D664	2.7		0.43	
360	D974	2.673	ex	0.28	result excluded see §4.1
432		----		----	
445	D664	3.059		2.40	
450		----		----	
451	D664	3.22		3.28	
473		----		----	
496	D664	2.82		1.09	
541	D974	2.22	ex	-2.20	result excluded see §4.1
551	D664	2.02		-3.30	
614		----		----	
657	D664	2.85		1.25	
663	D664	2.384		-1.30	
840		----		----	
862	D664	2.48		-0.78	
875		----		----	
902	D664	2.080		-2.97	
912	D664	2.84		1.20	
922	D664	3.06		2.40	
974		----		----	
994	D664	2.86		1.30	
1017	D974	1.86	ex	-4.18	result excluded see §4.1
1019		----		----	
1023	in house	1.67		-5.22	
1059	ISO6619	2.65		0.15	
1066	D664	3.44		4.48	
1106	D664	2.3386		-1.55	
1146	D664	2.446		-0.97	
1150	INH-1752	1.1014	R(0.05)	-8.34	first reported:0.0014
1161	D664	0.334	R(0.05)	-12.54	first reported:0.150
1173		----		----	
1201	D664	2.47		-0.83	
1213	D664	2.876		1.39	
1235		----		----	
1316		----		----	
1324	D664	2.580		-0.23	
1326	D664	2.639		0.09	
1402	D664	0.62	R(0.05)	-10.98	
1431	D664	2.70		0.43	
1460	D664	2.993		2.03	
1461		----		----	
1543	D664	0.0	R(0.05)	-14.38	
1564	D664	1.86		-4.18	
1577	D664	2.426		-1.07	
1622	D664	3.59		5.31	
1748		----		----	
1792	D664	1.830		-4.34	
1850	ISO6619	2.96		1.85	
1874		----		----	
1876	D664	2.565		-0.31	
1877		----		----	
1895	D974	3.10	ex	2.62	first reported:4.038, result excluded see §4.1
1957	D664	3.38		4.16	
1969		----		----	
1997		----		----	
2122	IP177	2.68		0.32	
2129	D664	2.62		-0.01	
9099		----		----	
9100		----		----	
9101		----		----	
9142		----		----	
9143		----		----	

Only D664/IP177/ISO6619 data

normality	OK	OK
n	38	35
outliers	4 (+5 excl)	3
mean (n)	2.622	2.639
st.dev. (n)	0.4770	0.4661
R(calc.)	1.335	1.305
R(D664:11a)	0.511	0.513



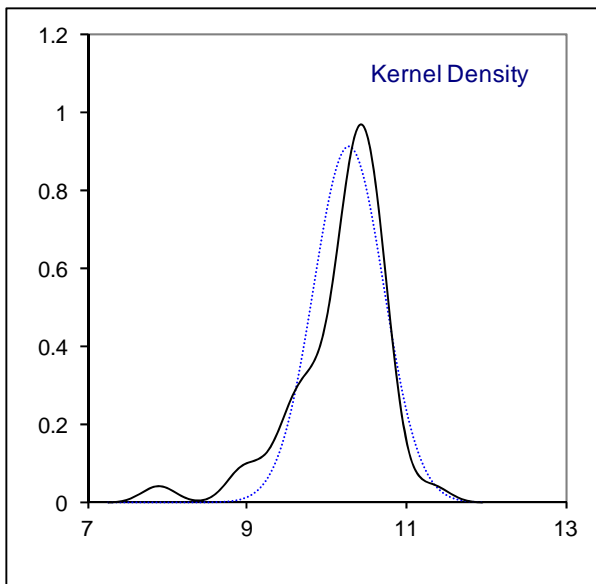
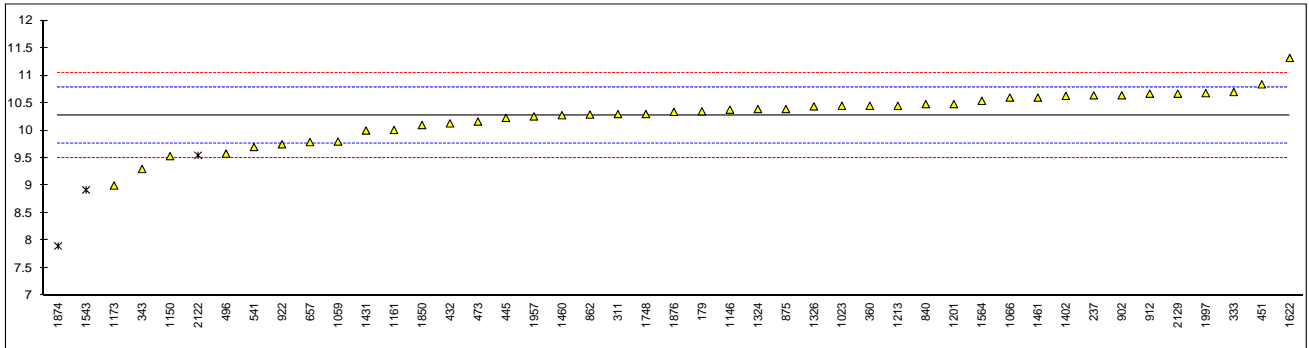
Determination of Base Number on sample #15080; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
173		----		----	
178		----		----	
179	D2896 - B	10.35		0.28	
237	D2896 - A	10.64		1.41	
252		----		----	
254		----		----	
256		----		----	
311	D2896 - A	10.3		0.09	
315		----		----	
333	D2896 - A	10.7		1.65	
343	D2896 - A	9.3		-3.80	
349		----		----	
360	D2896 - B	10.45		0.67	
432	D2896 - B	10.13		-0.57	
445	D2896 - B	10.23		-0.18	
450		----		----	
451	D2896	10.84		2.19	
473	D2896 - B	10.1630		-0.44	
496	D2896 - A	9.58		-2.71	
541	D2896 - B	9.7		-2.25	
551		----		----	
614		----		----	
657	D2896 - B	9.79		-1.90	
663		----		----	
840	D2896 - B	10.48		0.79	
862	D2896 - B	10.29		0.05	
875	D2896 - B	10.3905		0.44	
902	D2896 - B	10.64		1.41	
912	D2896 - B	10.67		1.53	
922	D2896	9.75		-2.05	
974		----		----	
994		----		----	
1017		----		----	
1019		----		----	
1023	D2896 - B	10.45		0.67	
1059	ISO3771	9.8		-1.86	
1066		10.6		1.26	
1106		----		----	
1146	D2896 - A	10.379		0.40	
1150	INH-13727	9.534		-2.89	
1161	ISO3771	10.011	C	-1.04	first reported:11.93
1173	in house	9.0		-4.97	
1201	D2896 - A	10.48		0.79	
1213	D2896	10.450		0.67	
1235		----		----	
1316		----		----	
1324	D2896 - A	10.390		0.44	
1326	D2896	10.44		0.63	
1402	D2896 - A	10.63		1.37	
1431	D2896 - B	10.0		-1.08	
1460	D2896 - B	10.278		0.00	
1461	INH-13727	10.6		1.26	
1543	D4739	8.92	ex	-5.28	result excluded see §4.1
1564	D2896 - B	10.54		1.02	
1577		----		----	
1622	D2896	11.32	C	4.06	first reported:12.80
1748	D2896	10.30		0.09	
1792		----		----	
1850	ISO3771	10.1		-0.69	
1874		7.9	R(0.01)	-9.25	
1876	D2896 - A	10.34		0.24	
1877		----		----	
1895	D974	n.d.		----	False negative, test method is not suitable
1957	D2896 - A	10.256		-0.08	
1969		----		----	
1997	INH-13727	10.68		1.57	
2122	IP400	9.55	ex	-2.83	result excluded see §4.1
2129	D2896 - A	10.67		1.53	
9099		----		----	
9100		----		----	
9101		----		----	
9142		----		----	
9143		----		----	

Only D2896-method A

Only D2896-method B

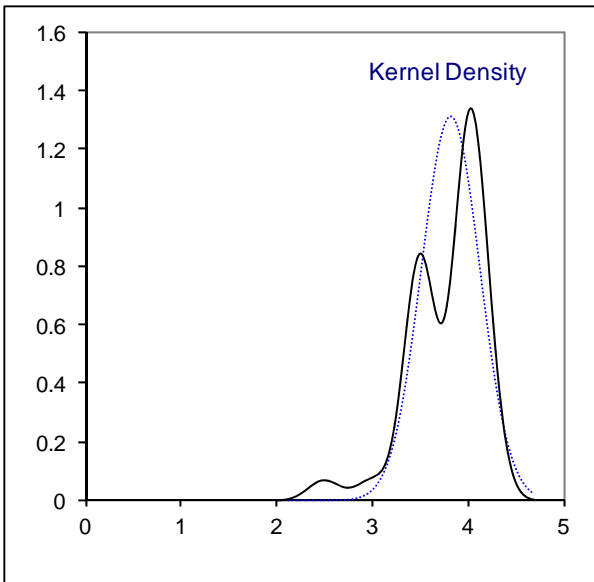
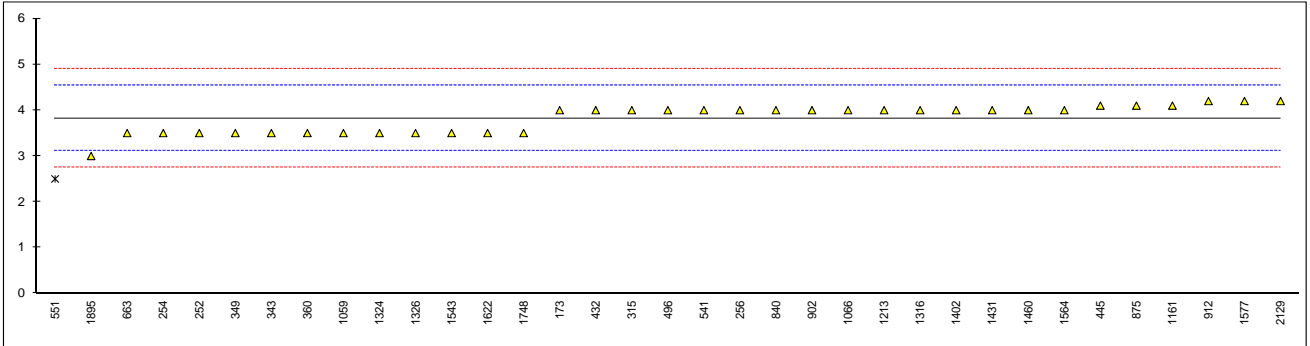
normality	suspect	suspect	OK
n	42	12	16
outliers	1 (+2 excl)	0	0
mean (n)	10.277	10.305	10.284
st.dev. (n)	0.4384	0.4359	0.2778
R(calc.)	1.228	1.220	0.778
R(D2896:11)	0.719	0.721	0.720



Determination of Color ASTM on sample #15080

lab	method	value	mark	z(targ)	remarks
173	D1500	4		0.50	
178		----		----	
179	D1500	L4.5		----	
237	D1500	L 4.5		----	
252	D1500	3.5		-0.90	
254	D1500	3.5		-0.90	
256	D1500	4.0		0.50	
311	D1500	L4.5		----	
315	D1500	4.0		0.50	
333		----		----	
343	D1500	3.5		-0.90	
349	D1500	3.5	C	-0.90	first reported:5.3
360	D1500	3.5		-0.90	
432	D1500	4.0		0.50	
445	D1500	4.1		0.78	
450		----		----	
451		----		----	
473		----		----	
496	D1500	4.0		0.50	
541	D1500	4.0		0.50	
551	D1500	2.5	R(0.01)	-3.70	
614	D1500	L4.5		----	
657	D1500	L4.0		----	
663	D1500	3.5		-0.90	
840	D1500	4.0		0.50	
862	D1500	L4.5		----	
875	D6045	4.1		0.78	
902	D1500	4.00		0.50	
912	D1500	4.2		1.06	
922	D1500	L4.0		----	
974	D1500	L4.0		----	
994	D1500	L4.0		----	
1017		----		----	
1019		----		----	
1023		----		----	
1059	D1500	3.5		-0.90	
1066	D1500	4.0		0.50	
1106		----		----	
1146		----		----	
1150		----		----	
1161	D6045	4.1		0.78	
1173		----		----	
1201	D1500	L4.0		----	
1213	D1500	4.0		0.50	
1235		----		----	
1316	D1500	4.0		0.50	
1324	D1500	3.5		-0.90	
1326	D1500	3.5		-0.90	
1402	D1500	4.0		0.50	
1431	D1500	4.0		0.50	
1460	D1500	4.0		0.50	
1461	ISO2049	L4		----	
1543	D1500	3.5		-0.90	
1564	D1500	4		0.50	
1577	D1500	4.2		1.06	
1622	D1500	3.5		-0.90	
1748	D1500	3.5		-0.90	
1792	D1500	L4,0		----	
1850	D1500	L4.0		----	
1874		----		----	
1876		----		----	
1877	D6045	L4.5		----	
1895	D1500	3		-2.30	
1957		----		----	
1969		----		----	
1997		----		----	
2122		----		----	
2129	D6045	4.2		1.06	
9099		----		----	
9100		----		----	
9101		----		----	
9142		----		----	
9143		----		----	

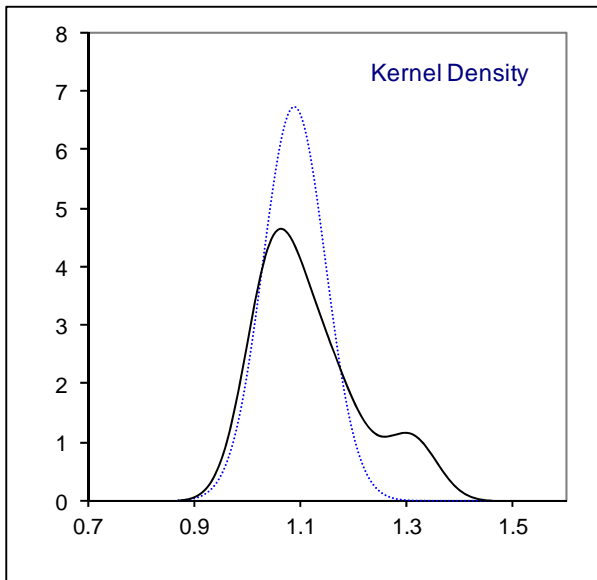
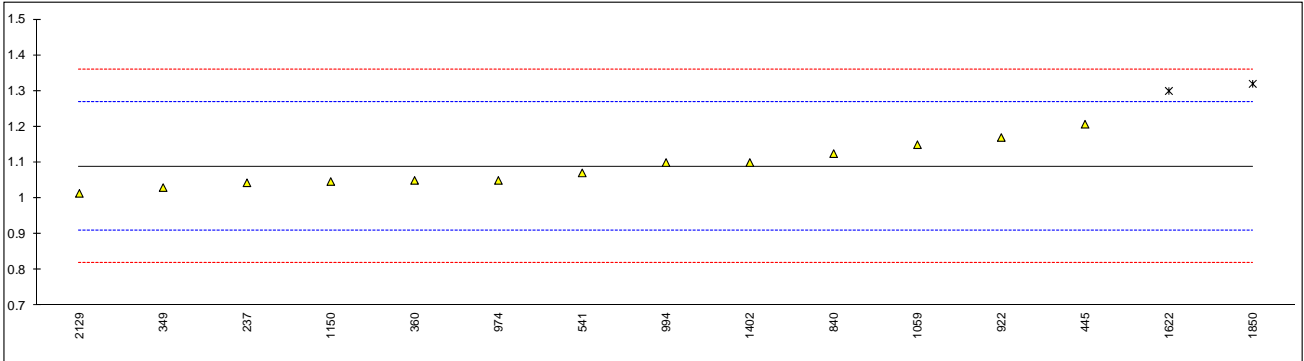
normality OK
 n 34
 outliers 1
 mean (n) 3.82
 st.dev. (n) 0.304
 R(calc.) 0.85
 R(D1500:12) 1.00



Determination of Conradson Carbon Residue on sample #15080; results in %M/M

lab	method	value	mark	z(targ)	remarks
173		----		----	
178		----		----	
179		----		----	
237	D189	1.0435		-0.51	
252		----		----	
254		----		----	
256		----		----	
311		----		----	
315		----		----	
333		----		----	
343		----		----	
349	D189	1.03		-0.66	
360	D189	1.050		-0.43	
432		----		----	
445	D189	1.207		1.31	
450		----		----	
451		----		----	
473		----		----	
496		----		----	
541	D189	1.071		-0.20	
551		----		----	
614		----		----	
657		----		----	
663		----		----	
840	D189	1.125		0.40	
862		----		----	
875		----		----	
902		----		----	
912		----		----	
922	D189	1.17		0.90	
974	D189	1.05		-0.43	
994	D189	1.10		0.12	
1017		----		----	
1019		----		----	
1023		----		----	
1059	D189	1.15		0.68	
1066		----		----	
1106		----		----	
1146		----		----	
1150	ISO6615	1.047		-0.47	
1161		----		----	
1173		----		----	
1201		----		----	
1213		----		----	
1235		----		----	
1316		----		----	
1324		----		----	
1326		----		----	
1402	D189	1.10		0.12	
1431		----		----	
1460		----		----	
1461		----		----	
1543		----		----	
1564		----		----	
1577		----		----	
1622	D189	1.30	DG(0.05)	2.35	
1748		----		----	
1792		----		----	
1850	D189	1.32	DG(0.05)	2.57	
1874		----		----	
1876		----		----	
1877		----		----	
1895		----		----	
1957		----		----	
1969		----		----	
1997		----		----	
2122		----		----	
2129	D189	1.0138		-0.84	
9099		----		----	
9100		----		----	
9101		----		----	
9142		----		----	
9143		----		----	

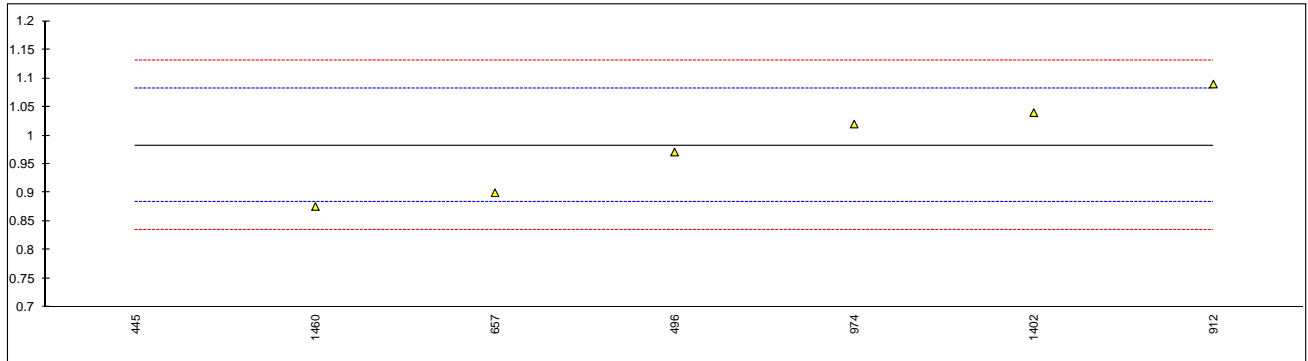
normality	OK
n	13
outliers	2
mean (n)	1.089
st.dev. (n)	0.0592
R(calc.)	0.166
R(D189:06)	0.251



Determination of Ramsbottom Carbon Residue on sample #15080; results in %M/M

lab	method	value	mark	z(targ)	remarks
173		----		----	
178		----		----	
179		----		----	
237		----		----	
252		----		----	
254		----		----	
256		----		----	
311		----		----	
315		----		----	
333		----		----	
343		----		----	
349		----		----	
360		----		----	
432		----		----	
445	D524	0.585	G(0.01)	-8.06	
450		----		----	
451		----		----	
473		----		----	
496	D524	0.9712		-0.24	
541		----		----	
551		----		----	
614		----		----	
657	D524	0.90		-1.68	
663		----		----	
840		----		----	
862		----		----	
875		----		----	
902		----		----	
912	D524	1.09		2.17	
922		----		----	
974	D524	1.02		0.75	
994		----		----	
1017		----		----	
1019		----		----	
1023		----		----	
1059		----		----	
1066		----		----	
1106		----		----	
1146		----		----	
1150		----		----	
1161		----		----	
1173		----		----	
1201		----		----	
1213		----		----	
1235		----		----	
1316		----		----	
1324		----		----	
1326		----		----	
1402	D524	1.04		1.16	
1431		----		----	
1460	D524	0.8760		-2.16	
1461		----		----	
1543		----		----	
1564		----		----	
1577		----		----	
1622		----		----	
1748		----		----	
1792		----		----	
1850		----		----	
1874		----		----	
1876		----		----	
1877		----		----	
1895		----		----	
1957		----		----	
1969		----		----	
1997		----		----	
2122		----		----	
2129		----		----	
9099		----		----	
9100		----		----	
9101		----		----	
9142		----		----	
9143		----		----	

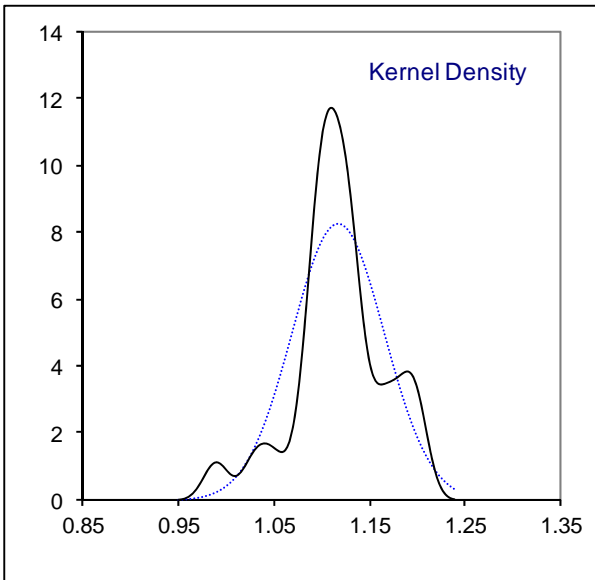
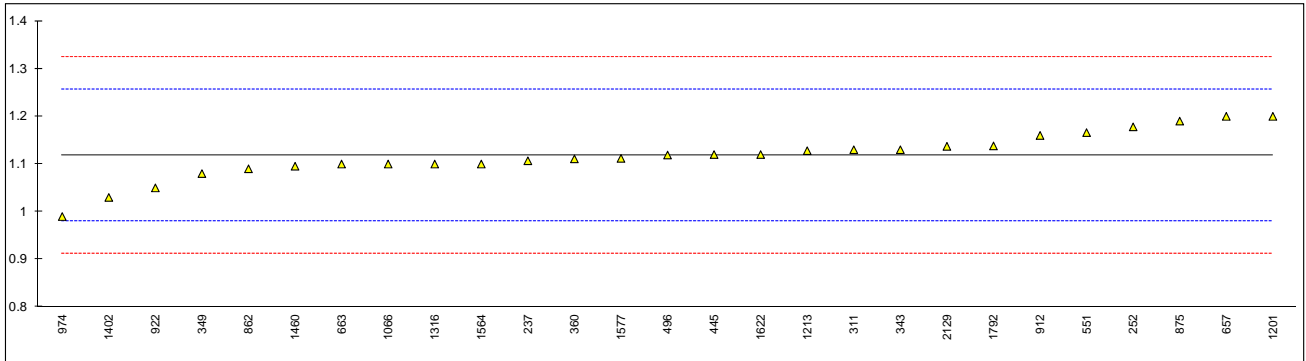
normality	unknown
n	6
outliers	1
mean (n)	0.983
st.dev. (n)	0.0831
R(calc.)	0.233
R(D524:10)	0.138



Determination of Carbon Residue (micro method) on sample #15080; results in %M/M

lab	method	value	mark	z(targ)	remarks
173		----		----	
178		----		----	
179		----		----	
237	D4530	1.1068		-0.16	
252	D4530	1.178		0.87	
254		----		----	
256		----		----	
311	D4530	1.13		0.18	
315		----		----	
333		----		----	
343	D4530	1.13	C	0.18	first reported:0.15
349	D4530	1.08		-0.55	
360	D4530	1.111		-0.10	
432		----		----	
445	D4530	1.12		0.03	
450		----		----	
451		----		----	
473		----		----	
496	D4530	1.1189		0.02	
541		----		----	
551	D4530	1.166		0.70	
614		----		----	
657	D4530	1.20		1.19	
663	D4530	1.10		-0.26	
840		----		----	
862	D4530	1.09	C	-0.40	first reported:0.01
875	D4530	1.19		1.05	
902		----		----	
912	D4530	1.16		0.61	
922	D4530	1.05		-0.99	
974	D4530	0.99		-1.86	
994		----		----	
1017		----		----	
1019		----		----	
1023		----		----	
1059		----		----	
1066	D4530	1.10		-0.26	
1106		----		----	
1146		----		----	
1150		----		----	
1161		----		----	
1173		----		----	
1201	D4530	1.20		1.19	
1213	D4530	1.128		0.15	
1235		----		----	
1316	D4530	1.1	C	-0.26	first reported:0.011
1324		----		----	
1326		----		----	
1402	D4530	1.03		-1.28	
1431		----		----	
1460	D4530	1.0955		-0.32	
1461		----		----	
1543		----		----	
1564	D4530	1.1		-0.26	
1577	D4530	1.112		-0.08	
1622	D4530	1.12		0.03	
1748		----		----	
1792	D4530	1.138		0.29	
1850		----		----	
1874		----		----	
1876		----		----	
1877		----		----	
1895		----		----	
1957		----		----	
1969		----		----	
1997		----		----	
2122		----		----	
2129	ISO10370	1.1372		0.28	
9099		----		----	
9100		----		----	
9101		----		----	
9142		----		----	
9143		----		----	

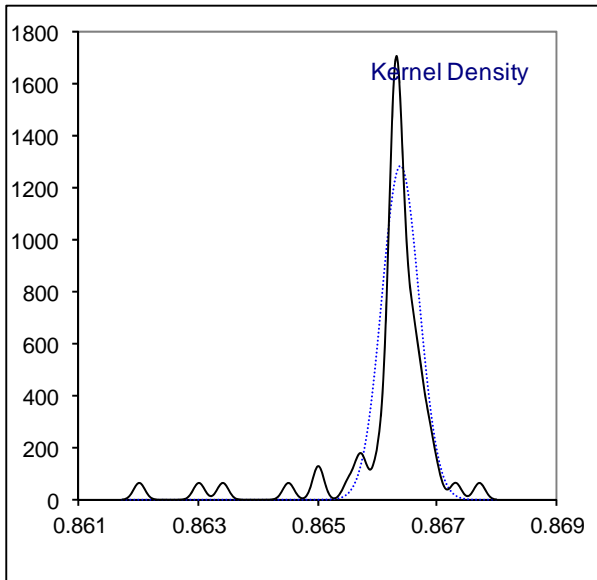
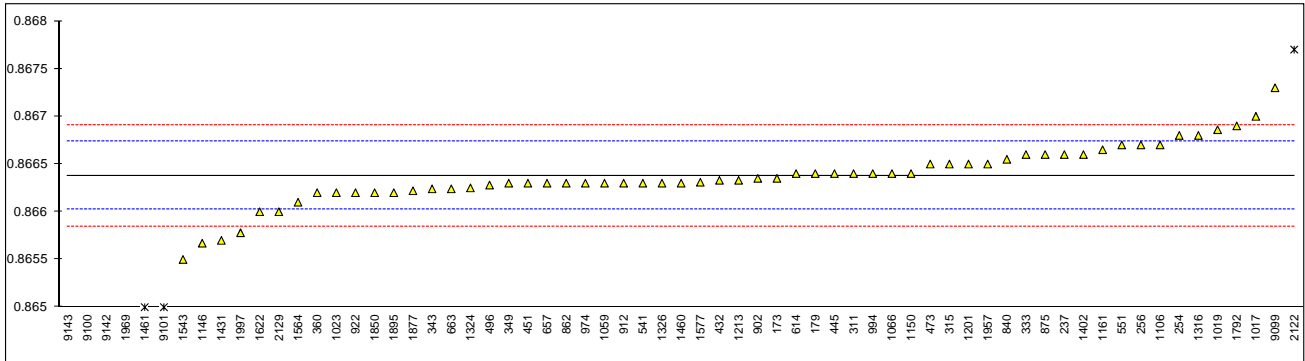
normality	suspect
n	27
outliers	0
mean (n)	1.118
st.dev. (n)	0.0482
R(calc.)	0.135
R(D4530:11)	0.193



Determination of Density at 15°C on sample #15080; results in kg/L

lab	method	value	mark	z(targ)	remarks
173	D4052	0.86635		-0.15	
178		-----		-----	
179	D4052	0.8664		0.13	
237	D4052	0.8666		1.25	
252		-----		-----	
254	D4052	0.8668		2.37	
256	D4052	0.8667		1.81	
311	D4052	0.8664		0.13	
315	D4052	0.8665		0.69	
333	D4052	0.8666		1.25	
343	D4052	0.86624		-0.76	
349	D4052	0.8663		-0.43	
360	D4052	0.8662		-0.99	
432	D4052	0.86633		-0.26	
445	D4052	0.8664		0.13	
450		-----		-----	
451	D4052	0.8663		-0.43	
473	D4052	0.8665		0.69	
496	D4052	0.86628		-0.54	
541	D4052	0.8663		-0.43	
551	D4052	0.8667		1.81	
614	D4052	0.8664		0.13	
657	D4052	0.8663		-0.43	
663	D4052	0.86624		-0.76	
840	D4052	0.86655		0.97	
862	D4052	0.8663		-0.43	
875	D4052	0.8666		1.25	
902	D4052	0.86635		-0.15	
912	D4052	0.8663		-0.43	
922	D4052	0.8662		-0.99	
974	D4052	0.8663		-0.43	
994	D4052	0.8664		0.13	
1017	ISO12185	0.8670		3.49	
1019	D4052	0.86686	C	2.71	first reported: 866.86 kg/L
1023	D4052	0.8662		-0.99	
1059	D4052	0.8663		-0.43	
1066	D4052	0.8664		0.13	
1106	D5002	0.8667		1.81	
1146	ISO12185	0.86567		-3.95	
1150	ISO12185	0.8664		0.13	
1161	ISO3675	0.86665	C	1.53	first reported:867.1 kg/L
1173		-----		-----	
1201	ISO12185	0.8665		0.69	
1213	D4052	0.86633		-0.26	
1235		-----		-----	
1316	D4052	0.8668	C	2.37	first reported:866.8 kg/L
1324	D4052	0.86625	C	-0.71	first reported:866.25 kg/L
1326	D4052	0.8663	C	-0.43	first reported:866.27 kg/L
1402	D4052	0.8666		1.25	
1431	D4052	0.8657		-3.79	
1460	D4052	0.8663		-0.43	
1461	ISO3675	0.8650	C,R(0.01)	-7.71	probably unit error, reported; 865.0 kg/L
1543	D4052	0.8655		-4.91	
1564	D4052	0.8661		-1.55	
1577	D4052	0.86631		-0.37	
1622	D4052	0.8660		-2.11	
1748		-----		-----	
1792	D4052	0.8669		2.93	
1850	D4052	0.8662		-0.99	
1874		-----		-----	
1876		-----		-----	
1877	D4052	0.86622		-0.87	
1895	D4052	0.8662	C	-0.99	first reported:866.2 kg/L
1957	D4052	0.8665		0.69	
1969	ISO3675	0.8645	R(0.01)	-10.51	
1997	ISO3675	0.86578		-3.34	
2122	in house	0.8677	R(0.01)	7.41	
2129	D4052	0.8660		-2.11	
9099	in house	0.8673		5.17	
9100	D1298	0.863	R(0.01)	-18.91	
9101	D1298	0.8650	C,R(0.01)	-7.71	first reported:865.0 kg/L
9142	D1298	0.8634	R(0.01)	-16.67	
9143	D1298	0.862	R(0.01)	-24.51	

normality	suspect
n	58
outliers	7
mean (n)	0.86638
st.dev. (n)	0.000312
R(calc.)	0.00087
R(D4052:11)	0.00050

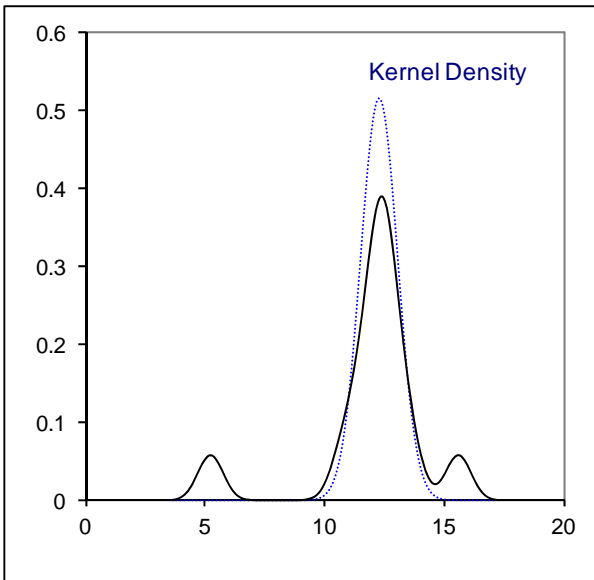
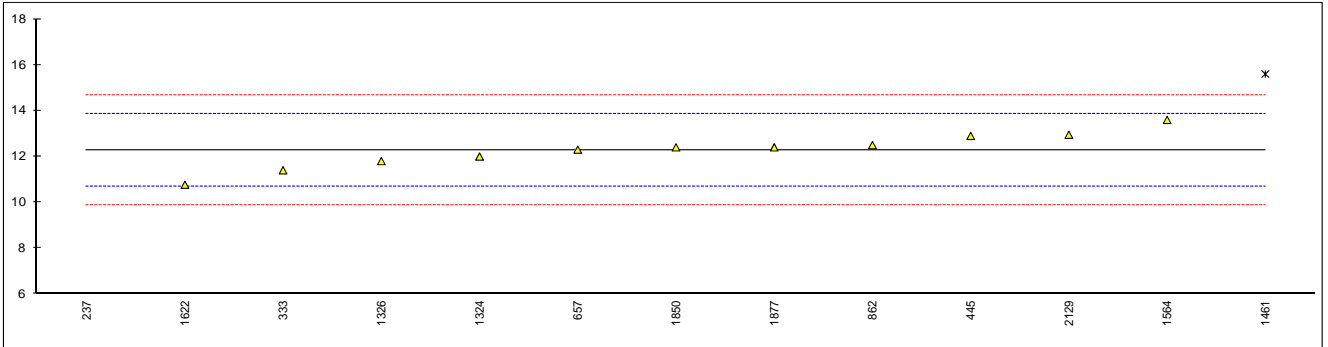


Determination of Evaporation loss by Noack on sample #15080; results in %M/M

lab	method	value	mark	z(targ)	remarks
173		----		----	
178		----		----	
179		----		----	
237	D5800 - A	5.2	C,G(0.01)	-8.82	first reported:2.8
252		----		----	
254		----		----	
256		----		----	
311		----		----	
315		----		----	
333	CEC L-40-93	11.4		-1.09	
343		----		----	
349		----		----	
360		----		----	
432		----		----	
445	D5800 - B	12.9		0.78	
450		----		----	
451		----		----	
473		----		----	
496		----		----	
541		----		----	
551		----		----	
614		----		----	
657	D5800 - B	12.3		0.03	
663		----		----	
840		----		----	
862	D5800 - B	12.5		0.28	
875		----		----	
902		----		----	
912		----		----	
922		----		----	
974		----		----	
994		----		----	
1017		----		----	
1019		----		----	
1023		----		----	
1059		----		----	
1066		----		----	
1106		----		----	
1146		----		----	
1150		----		----	
1161		----		----	
1173		----		----	
1201		----		----	
1213		----		----	
1235		----		----	
1316		----		----	
1324	D5800 - B	12.00		-0.34	
1326	D5800 - A	11.8		-0.59	
1402		----		----	
1431		----		----	
1460		----		----	
1461	INH-8088 - A	15.6	G(0.05)	4.15	
1543		----		----	
1564	DIN 51581	13.6		1.65	
1577		----		----	
1622	D5800 - B	10.77		-1.88	
1748		----		----	
1792		----		----	
1850	D5800 - B	12.4		0.16	
1874		----		----	
1876		----		----	
1877	D5800 - A	12.4		0.16	
1895		----		----	
1957		----		----	
1969		----		----	
1997		----		----	
2122		----		----	
2129	D5800 - A	12.95		0.84	
9099		----		----	
9100		----		----	
9101		----		----	
9142		----		----	
9143		----		----	

Only ASTM D5808-B data:

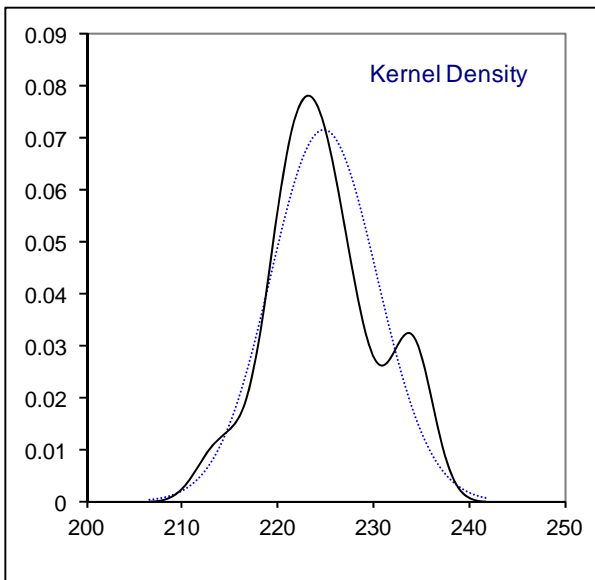
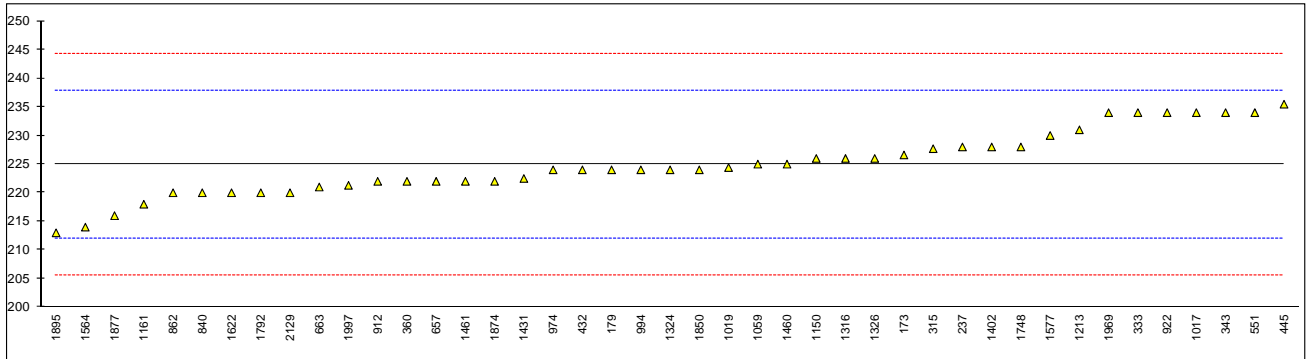
normality	OK	not OK
n	11	6
outliers	2	0
mean (n)	12.275	12.145
st.dev. (n)	0.7756	0.7344
R(calc.)	2.172	2.056
R(D5800:15-A)	2.246	--
comp. R(D5808:15-B)	1.559	1.546



Determination of Flash Point C.O.C. on sample #15080; results in °C

lab	method	value	mark	z(targ)	remarks
173	D92	226.6		0.26	
178		----		----	
179	D92	224		-0.14	
237	D92	228.0		0.48	
252		----		----	
254		----		----	
256		----		----	
311		----		----	
315	D92	227.7		0.43	
333	D92	234		1.41	
343	D92	234	C	1.41	first reported:198
349		----		----	
360	D92	222		-0.45	
432	D92	224		-0.14	
445	D92	235.475		1.64	
450		----		----	
451		----		----	
473		----		----	
496		----		----	
541		----		----	
551	D92	234		1.41	
614		----		----	
657	D92	222.0		-0.45	
663	D92	221.0		-0.61	
840	D92	220		-0.76	
862	D92	220		-0.76	
875		----		----	
902		----		----	
912	D92	222		-0.45	
922	D92	234.0		1.41	
974	D92	224.0		-0.14	
994	D92	224.0		-0.14	
1017	D92	234		1.41	
1019	ISO2592	224.4		-0.08	
1023		----		----	
1059	ISO2592	225		0.01	
1066		----		----	
1106		----		----	
1146		----		----	
1150	ISO2592	226.0		0.17	
1161	D92	218		-1.07	
1173		----		----	
1201		----		----	
1213	D92	231		0.95	
1235		----		----	
1316	D92	226		0.17	
1324	D92	224.0		-0.14	
1326	D92	226		0.17	
1402	D92	228		0.48	
1431	D92	222.5		-0.37	
1460	D92	225		0.01	
1461	ISO2592	222		-0.45	
1543		----		----	
1564	D92	214		-1.70	
1577	D92	230		0.79	
1622	D92	220		-0.76	
1748	D92	228		0.48	
1792	D92	220		-0.76	
1850	ISO2592	224		-0.14	
1874	D92	222		-0.45	
1876		----		----	
1877	D92	216		-1.39	
1895	D92	213		-1.85	
1957		----		----	
1969	ISO2592	233.9875		1.41	
1997	ISO2592	221.3		-0.56	
2122		----		----	
2129	D92	220.0		-0.76	
9099		----		----	
9100		----		----	
9101		----		----	
9142		----		----	
9143		----		----	

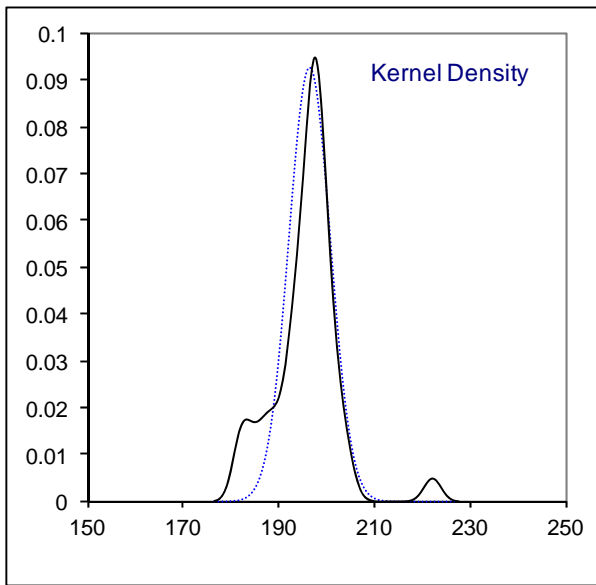
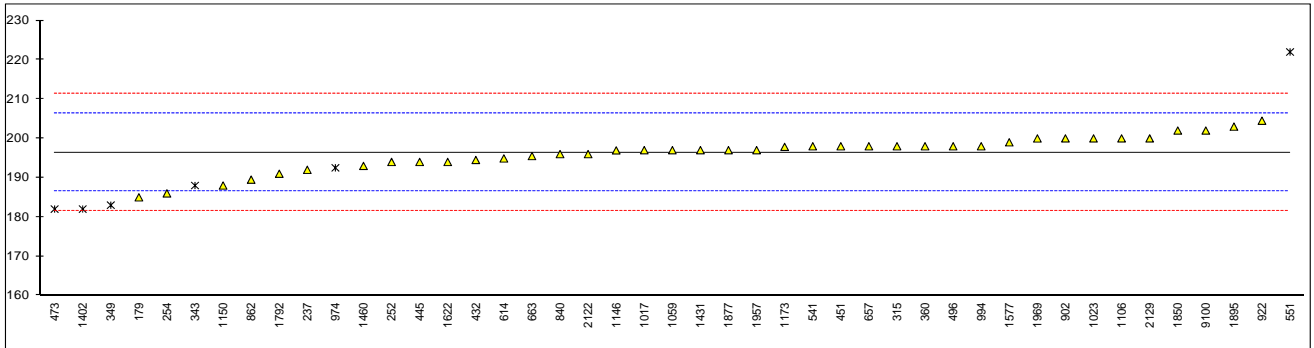
normality	OK
n	43
outliers	0
mean (n)	224.91
st.dev. (n)	5.570
R(calc.)	15.60
R(D92:12)	18.00



Determination of Flash Point PMcc on sample #15080; results in °C

lab	method	value	mark	z(targ)	remarks
173		----		----	
178		----		----	
179	D93-A	185.0		-2.29	
237	D93-A	192.0		-0.88	
252	D93-A	194		-0.48	
254	D93-A	186.0		-2.09	
256		----		----	
311		----		----	
315	D93-A	198.0		0.32	
333		----		----	
343	D93-B	188	C, ex	-1.69	first reported:224, result excluded see §4.1
349	D93-B	183	ex	-2.69	result excluded see §4.1
360	D93-A	198.0		0.32	
432	D93-A	194.5		-0.38	
445	D93-A	194.0		-0.48	
450		----		----	
451	D93-A	198		0.32	
473	D93-B	182.0	ex	-2.89	result excluded see §4.1
496	D93-A	198.0		0.32	
541	D93-A	198.0		0.32	
551	D93-A	222	R(0.01)	5.14	
614	D93-A	194.9		-0.30	
657	D93-A	198.0		0.32	
663	D93	195.5		-0.18	
840	D93-A	196.0		-0.08	
862	D93-A	189.5		-1.39	
875		----		----	
902	D93-A	200.0		0.72	
912		----		----	
922	D93-A	204.5		1.63	
974	D93-B	192.5	ex	-0.78	result excluded see §4.1
994	D93-A	198.0		0.32	
1017	D93-A	197		0.12	
1019		----		----	
1023	D93-A	200		0.72	
1059	ISO2719	197.0		0.12	
1066		----		----	
1106	D93-A	200.0		0.72	
1146	INH-93-A	196.95		0.11	
1150	ISO2719-A	188.0		-1.69	
1161		----		----	
1173	IP34	197.84		0.29	
1201		----	W	----	result withdrawn, reported:157
1213		----		----	
1235		----		----	
1316		----		----	
1324		----		----	
1326		----		----	
1402	D93-B	182.0	ex	-2.89	result excluded see §4.1
1431	D93-A	197		0.12	
1460	D93-A	193		-0.68	
1461		----		----	
1543		----		----	
1564		----		----	
1577	D93-A	199		0.52	
1622	D93-A	194.0		-0.48	
1748		----		----	
1792	D93-A	191.0		-1.08	
1850	ISO2719	202		1.12	
1874		----		----	
1876		----		----	
1877	D93-A	197.0		0.12	
1895	D93	203	C	1.32	first reported:212
1957	D93-A	197		0.12	
1969	ISO2719	199.9875		0.72	
1997		----		----	
2122	D93	196		-0.08	
2129	D93-A	200.0		0.72	
9099		----		----	
9100	D93	202		1.12	
9101		----		----	
9142		----		----	
9143		----		----	

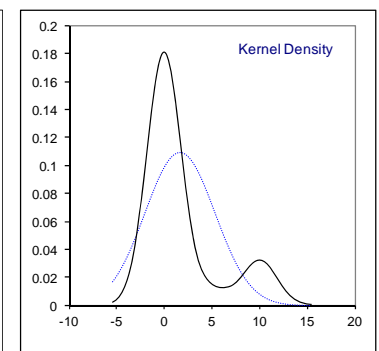
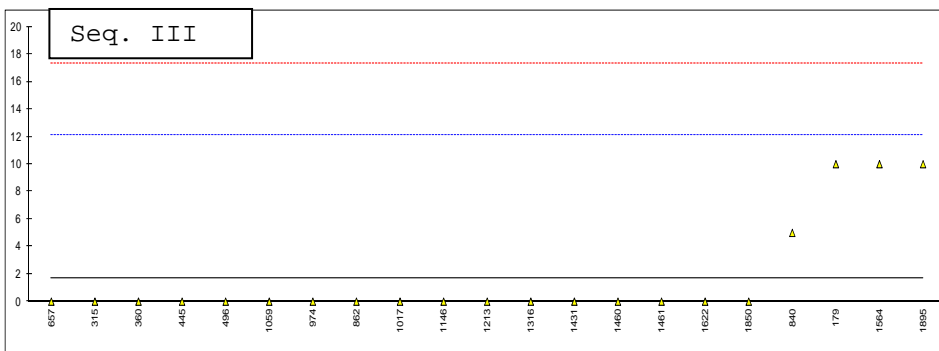
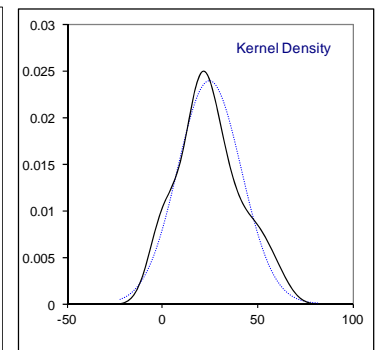
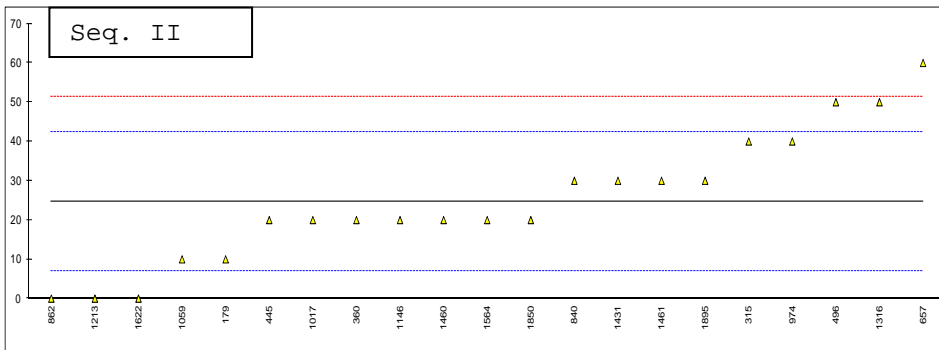
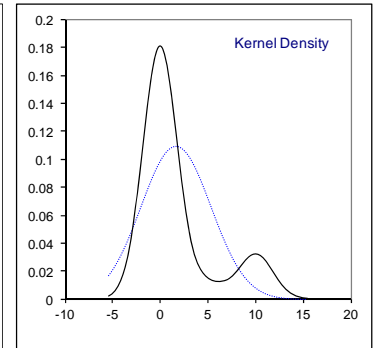
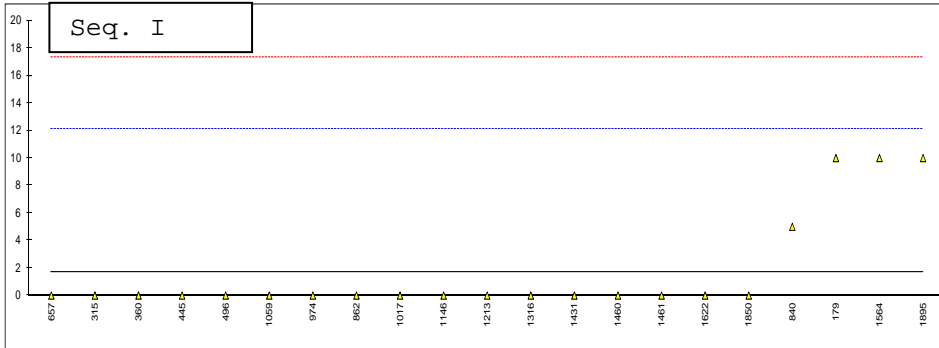
normality	OK
n	39
outliers	1 (+5 excl)
mean (n)	196.40
st.dev. (n)	4.311
R(calc.)	12.07
R(D93-A:15)	13.94



Determination of Foaming Tendency, 5 min blowing period on sample #15080; results in ml

lab	method	Seq. I, Foam	mark	z(targ)	Seq. II, Foam	mark	z(targ)	Seq. III, Foam	mark	z(targ)
173		----		----			----			----
178		----		----			----			----
179	D892	10		1.59	10		-1.67	10		1.59
237		----		----			----			----
252		----		----			----			----
254		----		----			----			----
256		----		----			----			----
311		----		----			----			----
315	D892	0		-0.32	40		1.72	0		-0.32
333		----		----			----			----
343		----		----			----			----
349		----		----			----			----
360	D892	0		-0.32	20		-0.54	0		-0.32
432		----		----			----			----
445	D892	0		-0.32	20		-0.54	0		-0.32
450		----		----			----			----
451		----		----			----			----
473		----		----			----			----
496	D892	0		-0.32	50		2.85	0		-0.32
541		----		----			----			----
551		----		----			----			----
614		----		----			----			----
657	D892	0		-0.32	60		3.98	0		-0.32
663		----		----			----			----
840	D892	5		0.64	30		0.59	5		0.64
862	D892	0		-0.32	0		-2.80	0		-0.32
875		----		----			----			----
902		----		----			----			----
912		----		----			----			----
922		----		----			----			----
974	D892	0		-0.32	40		1.72	0		-0.32
994		----		----			----			----
1017	D892	0		-0.32	20		-0.54	0		-0.32
1019		----		----			----			----
1023		----		----			----			----
1059	D892	0		-0.32	10		-1.67	0		-0.32
1066		----		----			----			----
1106		----		----			----			----
1146	ISO6247	0		-0.32	20		-0.54	0		-0.32
1150		----		----			----			----
1161		----		----			----			----
1173		----		----			----			----
1201		----		----			----			----
1213	D892	0		-0.32	0		-2.80	0		-0.32
1235		----		----			----			----
1316	D892	0		-0.32	50		2.85	0		-0.32
1324		----		----			----			----
1326		----		----			----			----
1402		----		----			----			----
1431	D892	0		-0.32	30		0.59	0		-0.32
1460	D892	0		-0.32	20		-0.54	0		-0.32
1461	ISO6247	0		-0.32	30		0.59	0		-0.32
1543		----		----			----			----
1564	D892	10		1.59	20		-0.54	10		1.59
1577		----		----			----			----
1622	D892	0		-0.32	0		-2.80	0		-0.32
1748		----		----			----			----
1792		----		----			----			----
1850	D892	0		-0.32	20		-0.54	0		-0.32
1874		----		----			----			----
1876		----		----			----			----
1877		----		----			----			----
1895	D892	10		1.59	30		0.59	10		1.59
1957		----		----			----			----
1969		----		----			----			----
1997		----		----			----			----
2122		----		----			----			----
2129		----		----			----			----
9099		----		----			----			----
9100		----		----			----			----
9101		----		----			----			----
9142		----		----			----			----
9143		----		----			----			----

normality	not OK	OK	not OK
n	21	21	21
outliers	0	0	0
mean (n)	1.67	24.76	1.67
st.dev. (n)	3.651	16.619	3.651
R(calc.)	10.22	46.53	10.22
R(D892:13)	14.63	24.80	14.63



Determination of Foam Stability, 10 min settling point on sample #15080; results in ml

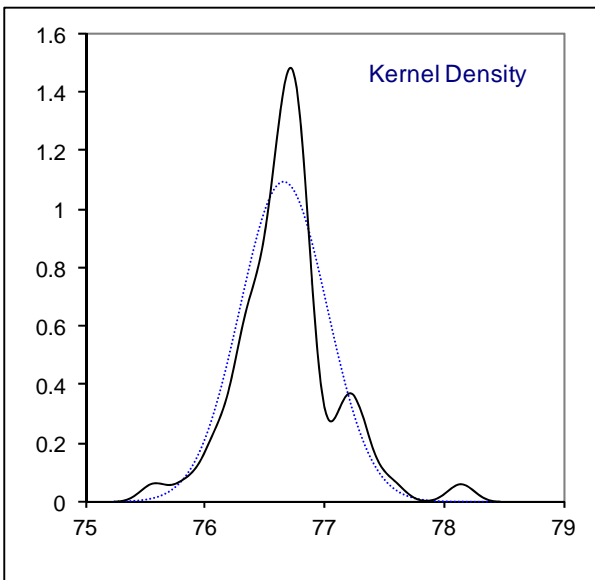
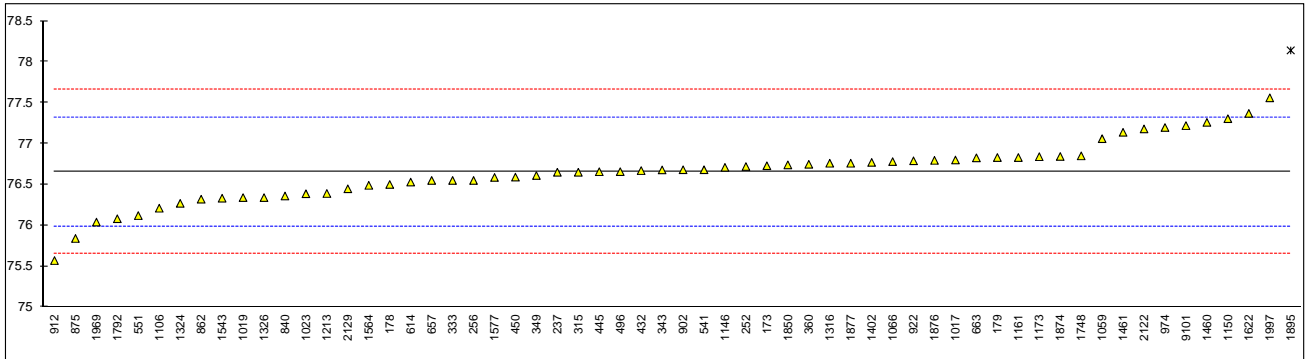
lab	method	Seq. I, Foam	mark	z(targ)	Seq. II, Foam	mark	z(targ)	Seq. III, Foam	mark	z(targ)
173		----		----	----		----	----		----
178		----		----	----		----	----		----
179	D892	0		----	0		----	0		----
237		----		----	----		----	----		----
252		----		----	----		----	----		----
254		----		----	----		----	----		----
256		----		----	----		----	----		----
311		----		----	----		----	----		----
315	D892	0		----	0		----	0		----
333		----		----	----		----	----		----
343		----		----	----		----	----		----
349		----		----	----		----	----		----
360	D892	0		----	0		----	0		----
432		----		----	----		----	----		----
445	D892	0		----	0		----	0		----
450		----		----	----		----	----		----
451		----		----	----		----	----		----
473		----		----	----		----	----		----
496	D892	0		----	0		----	0		----
541		----		----	----		----	----		----
551		----		----	----		----	----		----
614		----		----	----		----	----		----
657	D892	0		----	0		----	0		----
663		----		----	----		----	----		----
840	D892	0		----	0		----	0		----
862	D892	0		----	0		----	0		----
875		----		----	----		----	----		----
902		----		----	----		----	----		----
912		----		----	----		----	----		----
922		----		----	----		----	----		----
974	D892	0		----	0		----	0		----
994		----		----	----		----	----		----
1017	D892	0		----	0		----	0		----
1019		----		----	----		----	----		----
1023		----		----	----		----	----		----
1059	D892	0		----	0		----	0		----
1066		----		----	----		----	----		----
1106		----		----	----		----	----		----
1146	ISO6247	0		----	0		----	0		----
1150		----		----	----		----	----		----
1161		----		----	----		----	----		----
1173		----		----	----		----	----		----
1201		----		----	----		----	----		----
1213	D892	0		----	0		----	0		----
1235		----		----	----		----	----		----
1316	D892	0		----	0		----	0		----
1324		----		----	----		----	----		----
1326		----		----	----		----	----		----
1402		----		----	----		----	----		----
1431	D892	0		----	0		----	0		----
1460	D892	0		----	0		----	0		----
1461	ISO6247	0		----	0		----	0		----
1543		----		----	----		----	----		----
1564	D892	0		----	0		----	0		----
1577		----		----	----		----	----		----
1622	D892	0		----	0		----	0		----
1748		----		----	----		----	----		----
1792		----		----	----		----	----		----
1850	D892	0		----	0		----	0		----
1874		----		----	----		----	----		----
1876		----		----	----		----	----		----
1877		----		----	----		----	----		----
1895	D892	0		----	0		----	0		----
1957		----		----	----		----	----		----
1969		----		----	----		----	----		----
1997		----		----	----		----	----		----
2122		----		----	----		----	----		----
2129		----		----	----		----	----		----
9099		----		----	----		----	----		----
9100		----		----	----		----	----		----
9101		----		----	----		----	----		----
9142		----		----	----		----	----		----
9143		----		----	----		----	----		----

normality	n.a.	n.a.	n.a.
n	21	21	21
outliers	n.a.	n.a.	n.a.
mean (n)	0	0	0
st.dev. (n)	n.a.	n.a.	n.a.
R(calc.)	n.a.	n.a.	n.a.
R(D892:13)	n.a.	n.a.	n.a.

Determination of Kinematic Viscosity at 40°C on sample #15080; results in mm²/s

lab	method	value	mark	z(targ)	remarks
173	D445	76.729		0.22	
178	D445	76.5		-0.47	
179	D445	76.83	C	0.52	first reported:76.32
237	D445	76.65		-0.02	
252	D445	76.72		0.19	
254		-----		-----	
256	D445	76.55		-0.32	
311		-----		-----	
315	D445	76.650		-0.02	
333	D445	76.55		-0.32	
343	D445	76.678		0.06	
349	D445	76.61		-0.14	
360	D445	76.747		0.27	
432	D445	76.67		0.04	
445	D445	76.656		0.00	
450	D445	76.59		-0.20	
451		-----		-----	
473		-----		-----	
496	D445	76.657		0.00	
541	D445	76.68		0.07	
551	D445	76.12		-1.61	
614	D445	76.53		-0.38	
657	D445	76.55		-0.32	
663	D445	76.826		0.51	
840	D445	76.360		-0.89	
862	D445	76.32		-1.01	
875	D445	75.84		-2.44	
902	D445	76.68		0.07	
912	D445	75.57		-3.25	
922	D445	76.79		0.40	
974	D445	77.197		1.62	
994		-----		-----	
1017	D445	76.80		0.43	
1019	ISO3104	76.34		-0.95	
1023	D445	76.387		-0.81	
1059	ISO3104	77.06		1.21	
1066	D445	76.78		0.37	
1106	D445	76.21		-1.34	
1146	D445	76.710		0.16	
1150	ISO3104	77.3046		1.94	
1161	ISO3104	76.83		0.52	
1173	IP71	76.84		0.55	
1201		-----		-----	
1213	D445	76.39		-0.80	
1235		-----		-----	
1316	ISO3104	76.76		0.31	
1324	D445	76.270		-1.16	
1326	D445	76.34		-0.95	
1402	D445	76.77		0.34	
1431		-----		-----	
1460	D445	77.26		1.81	
1461	ISO3104	77.1382	C	1.44	first reported:78.1072
1543	D445	76.33269		-0.97	
1564	D445	76.49		-0.50	
1577	D445	76.586		-0.21	
1622	D445	77.37		2.14	
1748	D445	76.85		0.58	
1792	D445	76.081		-1.72	
1850	ISO3104	76.74		0.25	
1874	D445	76.843		0.56	
1876	D2162	76.795		0.41	
1877	D445	76.76		0.31	
1895	D445	78.14	C,R(0.01)	4.44	first reported:78.37
1957		-----		-----	
1969	ISO3104	76.04		-1.85	
1997	ISO3104	77.56014		2.71	
2122	in house	77.18		1.57	
2129	D445	76.4470		-0.63	
9099		-----		-----	
9100		-----		-----	
9101	D445	77.22		1.69	
9142		-----		-----	
9143		-----		-----	

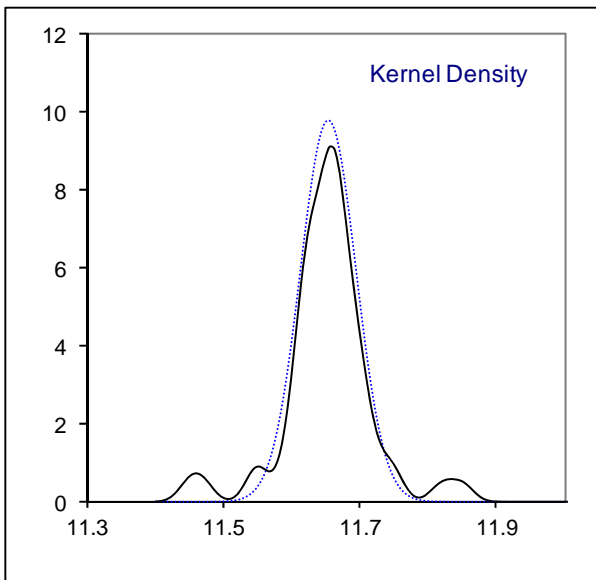
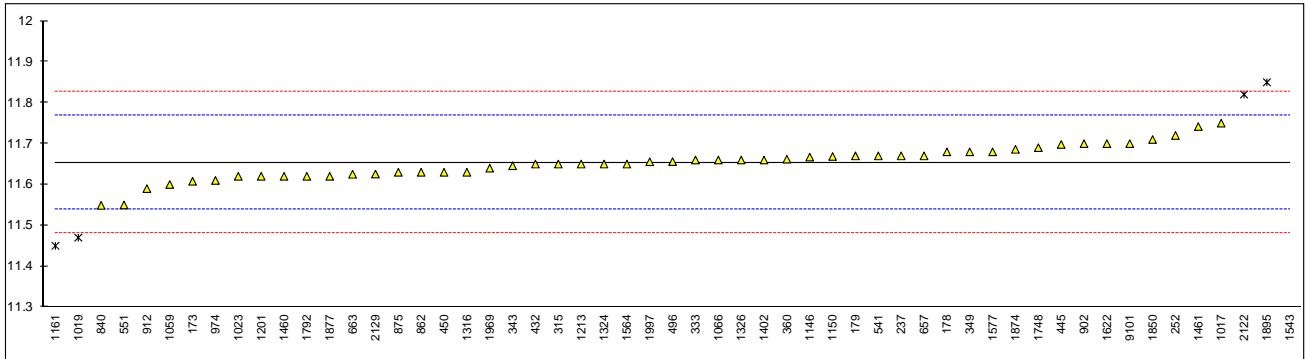
normality	suspect
n	59
outliers	1
mean (n)	76.657
st.dev. (n)	0.3649
R(calc.)	1.022
R(D445:15)	0.935



Determination of Kinematic Viscosity at 100°C on sample #15080; results in mm²/s

lab	method	value	mark	z(targ)	remarks
173	D445	11.608		-0.79	
178	D445	11.68		0.46	
179	D445	11.67	C	0.29	first reported:11.77
237	D445	11.67		0.29	
252	D445	11.72		1.16	
254		----		----	
256		----		----	
311		----		----	
315	D445	11.650		-0.06	
333	D445	11.66		0.11	
343	D445	11.646		-0.13	
349	D445	11.68		0.46	
360	D445	11.662		0.15	
432	D445	11.65		-0.06	
445	D445	11.698		0.77	
450	D445	11.63		-0.41	
451		----		----	
473		----		----	
496	D445	11.656		0.04	
541	D445	11.67		0.29	
551	D445	11.55		-1.80	
614		----		----	
657	D445	11.67		0.29	
663	D445	11.625		-0.50	
840	D445	11.549		-1.82	
862	D445	11.63		-0.41	
875	D445	11.63		-0.41	
902	D445	11.70		0.81	
912	D445	11.59		-1.11	
922		----		----	
974	D445	11.610		-0.76	
994		----		----	
1017	D445	11.75		1.68	
1019	ISO3104	11.47	R(0.01)	-3.20	
1023	D445	11.6200		-0.58	
1059	ISO3104	11.60		-0.93	
1066	D445	11.66		0.11	
1106		----		----	
1146	D445	11.667		0.23	
1150	ISO3104	11.6687		0.26	
1161	ISO3104	11.45	R(0.01)	-3.54	
1173		----		----	
1201	D445	11.62		-0.58	
1213	D445	11.65		-0.06	
1235		----		----	
1316	ISO3104	11.63		-0.41	
1324	D445	11.650		-0.06	
1326	D445	11.66		0.11	
1402	D445	11.66		0.11	
1431		----		----	
1460	D445	11.62		-0.58	
1461	ISO3104	11.7417	C	1.53	first reported:11.7948
1543	D445	13.20939	R(0.01)	27.09	
1564	D445	11.65		-0.06	
1577	D445	11.680		0.46	
1622	D445	11.70		0.81	
1748	D445	11.69	C	0.63	first reported:11.91
1792	D445	11.620		-0.58	
1850	ISO3104	11.71		0.98	
1874	D445	11.686		0.56	
1876		----		----	
1877	D445	11.62		-0.58	
1895	D445	11.85	R(0.01)	3.42	
1957		----		----	
1969	ISO3104	11.64		-0.24	
1997	ISO3104	11.65559		0.04	
2122	in house	11.82	R(0.01)	2.90	
2129	D445	11.6255		-0.49	
9099		----		----	
9100		----		----	
9101	D445	11.70		0.81	
9142		----		----	
9143		----		----	

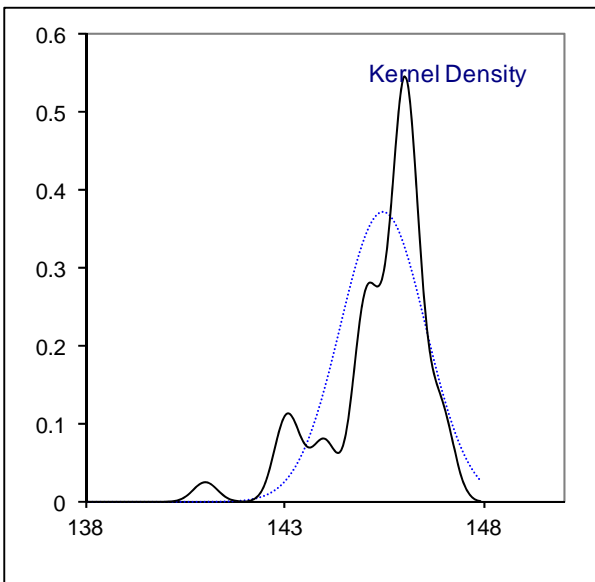
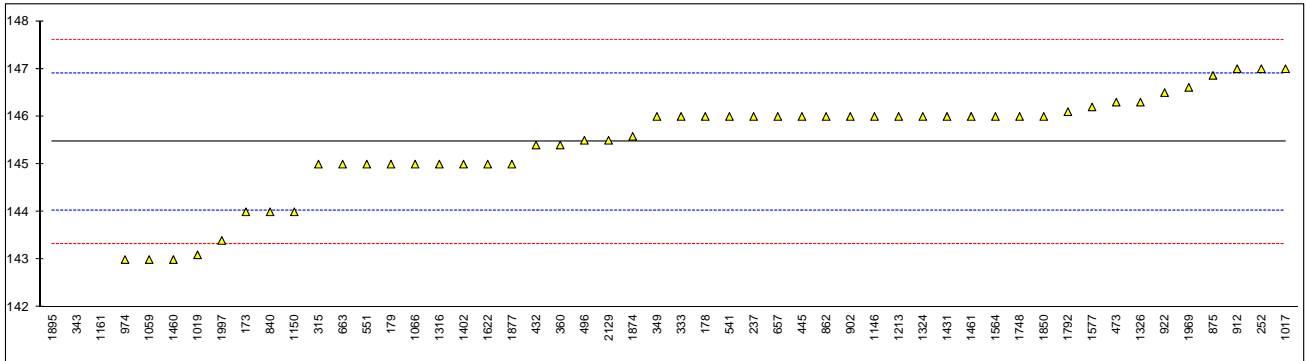
normality OK
 n 50
 outliers 5
 mean (n) 11.654
 st.dev. (n) 0.0408
 R(calc.) 0.114
 R(D445:15) 0.161



Determination of Viscosity Index on sample #15080

lab	method	value	mark	z(targ)	calc. iis	mark	remarks
173	D2270	144		-2.05	144.41		
178	D2270	146		0.75	146.36		
179	D2270	145	C	-0.65	145.41		first reported:149
237	D2270	146.0		0.75	145.82		
252	D2270	147		2.15	146.65		
254		----		----	----		
256		----		----	----		
311		----		----	----		
315	D2270	145		-0.65	145.42		
333	D2270	146		0.75	145.85		
343	D2270	130.25	C, E,R(0.01)	-21.30	145.28		Calc.error? first reported:129.82
349	D2270	146		0.75	146.11		
360	ISO2909	145.4		-0.09	145.44		
432	D2270	145.4		-0.09	145.38		
445	D2270	146		0.75	146.36		
450		----		----	145.16		
451		----		----	----		
473	D2270	146.3		1.17	----		
496	D2270	145.5		0.05	145.53		
541	D2270	146		0.75	145.75		
551	D2270	145		-0.65	144.63		
614		----		----	----		
657	D2270	146		0.75	146.05		
663	D2270	145		-0.65	144.53		
840	D2270	144.0		-2.05	144.06		
862	D2270	146		0.75	145.78		
875	D2270	146.86		1.95	146.88		
902	D2270	146		0.75	146.35		
912	D2270	147		2.15	146.70		
922	D2270	146.5		1.45	----		
974	D2270	143		-3.45	143.40		
994		----		----	----		
1017	D2270	147		2.15	147.06		
1019	D2270	143.1		-3.31	142.51		
1023		----		----	145.42		
1059	ISO2909	143		-3.45	143.51		
1066	D2270	145	C	-0.65	145.33		first reported:130
1106		----		----	----		
1146	D2270	146		0.75	145.63		
1150	ISO2909	144		-2.05	144.32		
1161	D2270	141	R(0.01)	-6.25	141.01	R(0.01)	outlier in viscosity at 100°C
1173		----		----	----		
1201		----		----	----		
1213	D2270	146		0.75	146.01		
1235		----		----	----		
1316	D2270	145		-0.65	144.78		
1324	D2270	146		0.75	146.29		
1326	D2270	146.3		1.17	146.33		
1402	D2270	145		-0.65	145.35		
1431	D2270	146		0.75	----		
1460	D2270	143		-3.45	143.46		
1461	ISO2909	146	C	0.75	146.13		first reported:145
1543		----		----	----		
1564	D2270	146		0.75	145.79		
1577	D2270	146.2		1.03	146.16		
1622	D2270	145		-0.65	144.79		
1748	D2270	146	C	0.75	145.76		first reported:150
1792	D2270	146.1		0.89	146.12		
1850	ISO2909	146		0.75	146.41		
1874	D2270	145.583		0.17	145.70		
1876		----		----	----		
1877	D2270	145		-0.65	144.58		
1895	D2270	130.02	C,E,R(0.01)	-21.62	146.00		Calc. error? first reported:129.76
1957		----		----	----		
1969	ISO2909	146.61		1.60	146.62		
1997	ISO2909	143.4		-2.89	143.49		
2122		----		----	147.57		
2129	D2270	145.5		0.05	145.40		
9099		----		----	----		
9100		----		----	----		
9101		----		----	145.13		
9142		----		----	----		
9143		----		----	----		

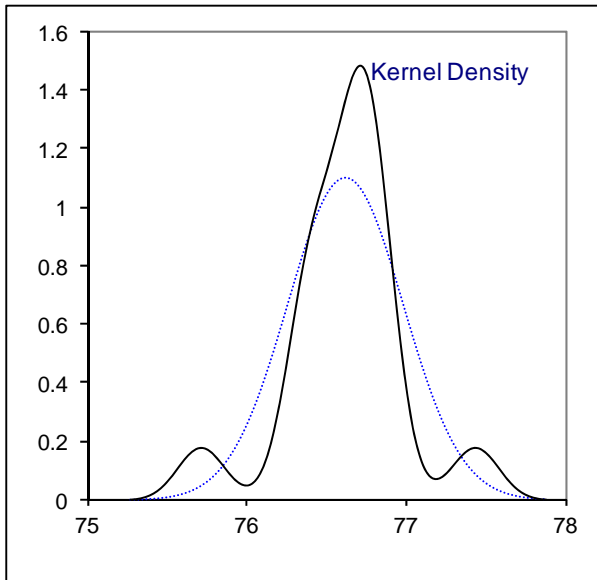
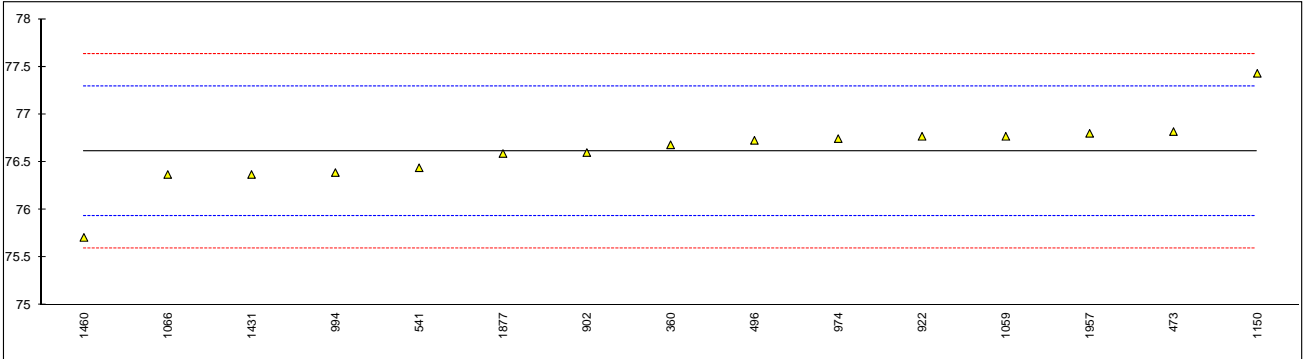
normality	OK	OK
n	49	52
outliers	3	1
mean (n)	145.46	145.48
st.dev. (n)	1.071	1.030
R(calc.)	3.00	2.88
R(D2270:10)	2.00	2.00



Determination of Viscosity Stabinger at 40 °C on sample #15080; results in mm²/s

lab	method	value	mark	z(targ)	remarks
173		----		----	
178		----		----	
179		----		----	
237		----		----	
252		----		----	
254		----		----	
256		----		----	
311		----		----	
315		----		----	
333		----		----	
343		----		----	
349		----		----	
360	D7042	76.681		0.20	
432		----		----	
445		----		----	
450		----		----	
451		----		----	
473	D7042	76.820		0.61	
496	D7042	76.728		0.34	
541	D7042	76.44		-0.52	
551		----		----	
614		----		----	
657		----		----	
663		----		----	
840		----		----	
862		----		----	
875		----		----	
902	D7042	76.60		-0.04	
912		----		----	
922	D7042	76.77		0.46	
974	D7042	76.746		0.39	
994	D7042	76.39		-0.66	
1017		----		----	
1019		----		----	
1023		----		----	
1059	D7042	76.77		0.46	
1066	D7042	76.37		-0.72	
1106		----		----	
1146		----		----	
1150	D7042	77.4305		2.41	
1161		----		----	
1173		----		----	
1201		----		----	
1213		----		----	
1235		----		----	
1316		----		----	
1324		----		----	
1326		----		----	
1402		----		----	
1431	D7042	76.37		-0.72	
1460	D7042	75.71		-2.67	
1461		----		----	
1543		----		----	
1564		----		----	
1577		----		----	
1622		----		----	
1748		----		----	
1792		----		----	
1850		----		----	
1874		----		----	
1876		----		----	
1877	D7042	76.59		-0.07	
1895		----		----	
1957	D7042	76.801		0.55	
1969		----		----	
1997		----		----	
2122		----		----	
2129		----		----	
9099		----		----	
9100		----		----	
9101		----		----	
9142		----		----	
9143		----		----	

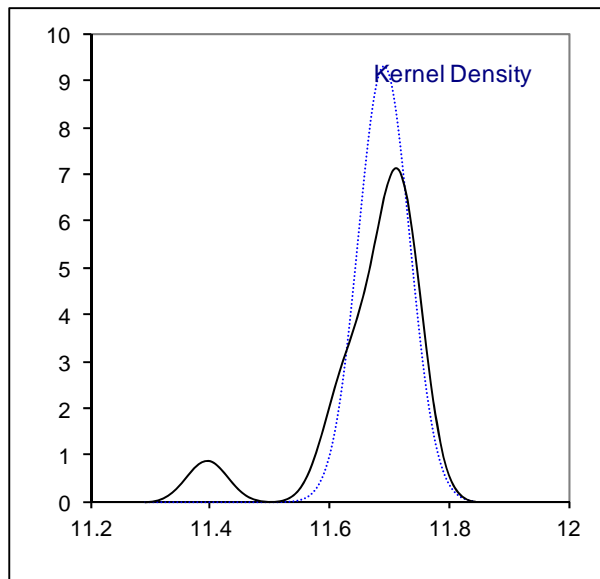
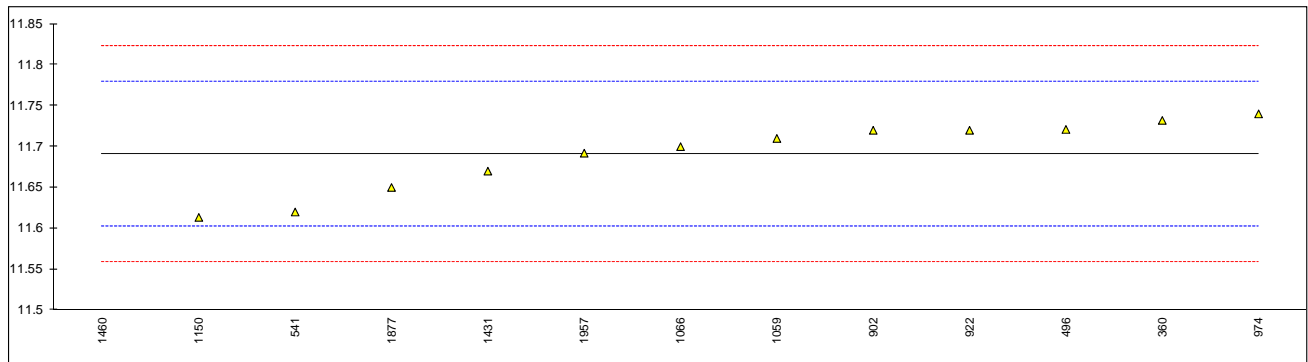
normality	not OK
n	15
outliers	0
mean (n)	76.614
st.dev. (n)	0.3630
R(calc.)	1.017
R(D7042:14)	0.948



Determination of Viscosity Stabinger at 100 °C on sample #15080; results in mm²/s

lab	method	value	mark	z(targ)	remarks
173		----		----	
178		----		----	
179		----		----	
237		----		----	
252		----		----	
254		----		----	
256		----		----	
311		----		----	
315		----		----	
333		----		----	
343		----		----	
349		----		----	
360	D7042	11.732		0.94	
432		----		----	
445		----		----	
450		----		----	
451		----		----	
473		----		----	
496	D7042	11.721		0.69	
541	D7042	11.62		-1.61	
551		----		----	
614		----		----	
657		----		----	
663		----		----	
840		----		----	
862		----		----	
875		----		----	
902	D7042	11.72		0.66	
912		----		----	
922	D7042	11.72		0.66	
974	D7042	11.74		1.12	
994		----		----	
1017		----		----	
1019		----		----	
1023		----		----	
1059	D7042	11.71		0.44	
1066	D7042	11.70		0.21	
1106		----		----	
1146		----		----	
1150	D7042	11.6135		-1.75	
1161		----		----	
1173		----		----	
1201		----	W	----	result withdrawn, first reported: 10.67
1213		----		----	
1235		----		----	
1316		----		----	
1324		----		----	
1326		----		----	
1402		----		----	
1431	D7042	11.67		-0.47	
1460	D7042	11.396	C,G(0.01)	-6.69	first reported:11.39
1461		----		----	
1543		----		----	
1564		----		----	
1577		----		----	
1622		----		----	
1748		----		----	
1792		----		----	
1850		----		----	
1874		----		----	
1876		----		----	
1877	D7042	11.65		-0.92	
1895		----		----	
1957	D7042	11.692		0.03	
1969		----		----	
1997		----		----	
2122		----		----	
2129		----		----	
9099		----		----	
9100		----		----	
9101		----		----	
9142		----		----	
9143		----		----	

normality OK
 n 12
 outliers 1
 mean (n) 11.691
 st.dev. (n) 0.0429
 R(calc.) 0.120
 R(D7042:14) 0.123

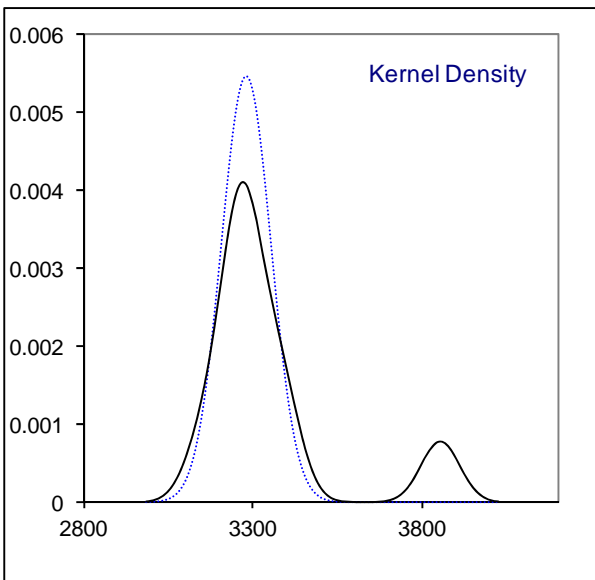
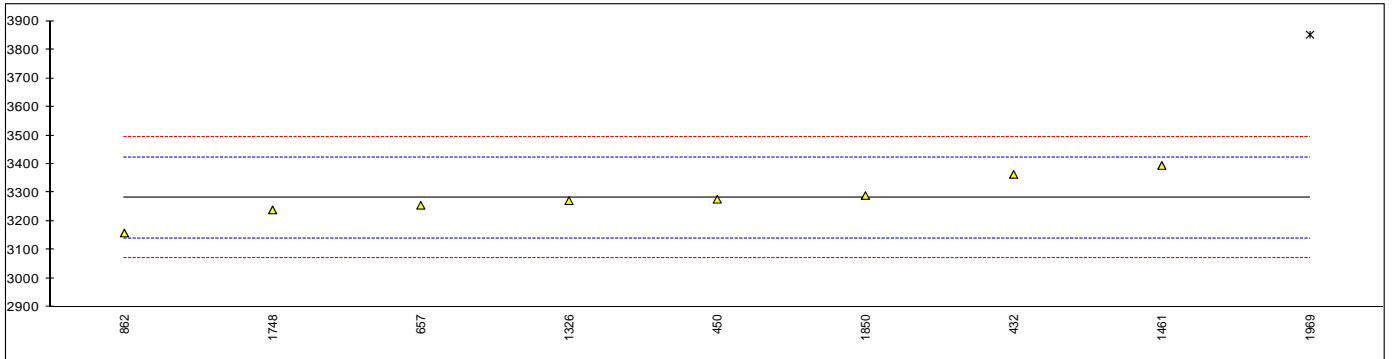


Determination of Viscosity, Apparent (CCS) at -20°C on sample #15080; results in mPa·s

lab	method	value	mark	z(targ)	remarks
173		----		----	
178		----		----	
179		----		----	
237		----		----	
252		----		----	
254		----		----	
256		----		----	
311		----		----	
315		----		----	
333		----		----	
343		----		----	
349		----		----	
360		----		----	
432	D5293	3364		1.17	
445		----		----	
450	D5293	3277		-0.07	
451		----		----	
473		----		----	
496		----		----	
541		----		----	
551		----		----	
614		----		----	
657	D5293	3256		-0.36	
663		----		----	
840		----		----	
862	D5293	3159		-1.74	
875		----		----	
902		----		----	
912		----		----	
922		----		----	
974		----		----	
994		----		----	
1017		----		----	
1019		----		----	
1023		----		----	
1059		----		----	
1066		----		----	
1106		----		----	
1146		----		----	
1150		----		----	
1161		----		----	
1173		----		----	
1201		----		----	
1213		----		----	
1235		----		----	
1316		----		----	
1324		----		----	
1326	D5293	3272		-0.14	
1402		----		----	
1431		----		----	
1460		----		----	
1461	INH-14147	3395		1.61	
1543		----		----	
1564		----		----	
1577		----		----	
1622		----		----	
1748	D5293	3240		-0.59	
1792		----		----	
1850	D5293	3290		0.12	
1874		----		----	
1876		----		----	
1877		----		----	
1895		----		----	
1957		----		----	
1969	D5293	3852	G(0.01)	8.11	
1997		----		----	
2122		----		----	
2129		----		----	
9099		----		----	
9100		----		----	
9101		----		----	
9142		----		----	
9143		----		----	

normality OK
 n 8
 outliers 1
 mean (n) 3281.63
 st.dev. (n) 72.948
 R(calc.) 204.25
 R(D5293:15) 196.90

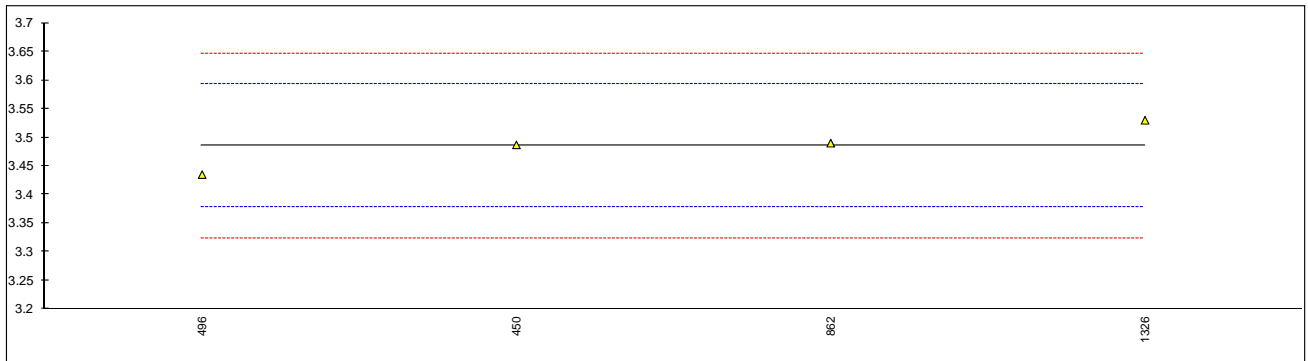
R(D5293:15) constant cooling instrument = 239.6
 R(D5293:15) thermo-electrically cooled instrument = 196.90



Determination of Viscosity HTHS by Tapered Bearing Simulator on sample #15080; results in mPa·s

lab	method	value	mark	z(targ)	remarks
173		----		----	
178		----		----	
179		----		----	
237		----		----	
252		----		----	
254		----		----	
256		----		----	
311		----		----	
315		----		----	
333		----		----	
343		----		----	
349		----		----	
360		----		----	
432		----		----	
445		----		----	
450	D4683	3.487		0.03	
451		----		----	
473		----		----	
496	D4683	3.435		-0.94	
541		----		----	
551		----		----	
614		----		----	
657		----		----	
663		----		----	
840		----		----	
862	D4741	3.49		0.08	
875		----		----	
902		----		----	
912		----		----	
922		----		----	
974		----		----	
994		----		----	
1017		----		----	
1019		----		----	
1023		----		----	
1059		----		----	
1066		----		----	
1106		----		----	
1146		----		----	
1150		----		----	
1161		----		----	
1173		----		----	
1201		----		----	
1213		----		----	
1235		----		----	
1316		----		----	
1324		----		----	
1326	D5481	3.53		0.83	
1402		----		----	
1431		----		----	
1460		----		----	
1461		----		----	
1543		----		----	
1564		----		----	
1577		----		----	
1622		----		----	
1748		----		----	
1792		----		----	
1850		----		----	
1874		----		----	
1876		----		----	
1877		----		----	
1895		----		----	
1957		----		----	
1969		----		----	
1997		----		----	
2122		----		----	
2129		----		----	
9099		----		----	
9100		----		----	
9101		----		----	
9142		----		----	
9143		----		----	

normality	unknown
n	4
outliers	n.a.
mean (n)	3.485
st.dev. (n)	0.0390
R(calc.)	0.109
R(D4683:13)	0.151

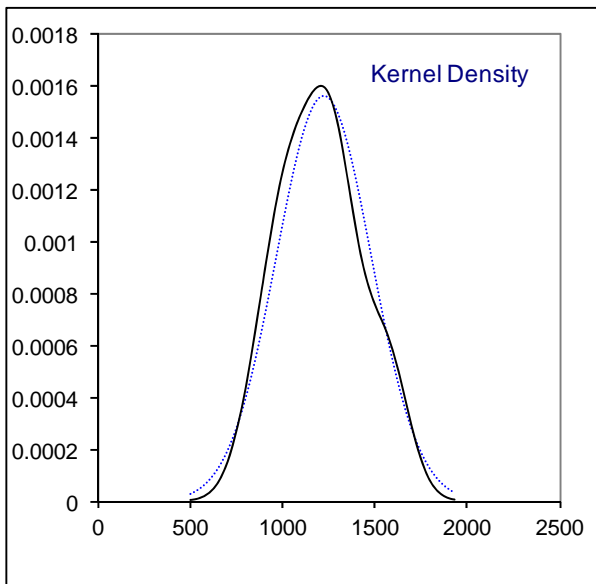
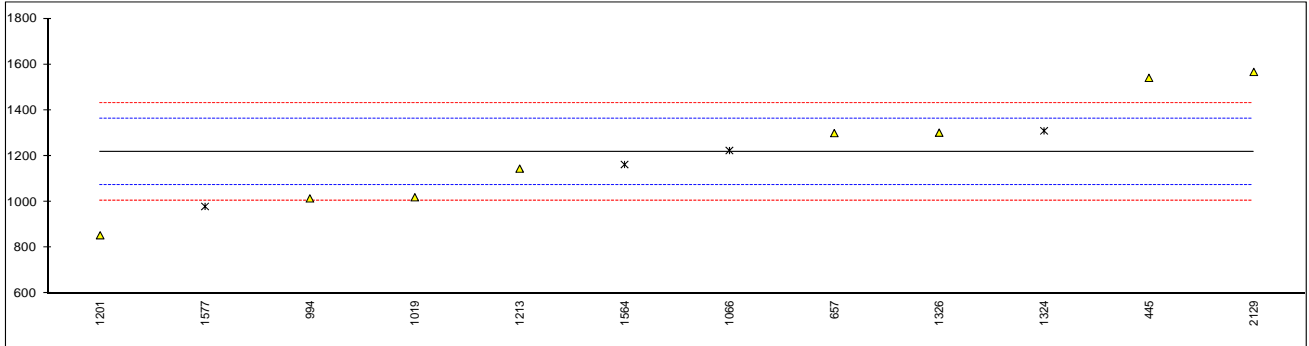


Determination of Nitrogen on sample #15080; results in mg/kg

lab	method	value	mark	z(targ)	remarks
173		----		----	
178		----		----	
179		----		----	
237		----		----	
252		----		----	
254		----		----	
256		----		----	
311		----		----	
315		----		----	
333		----		----	
343		----		----	
349		----		----	
360		----		----	
432		----		----	
445	D5291	1541.2		4.52	
450		----		----	
451		----		----	
473		----		----	
496		----		----	
541		----		----	
551		----		----	
614		----		----	
657	D5762	1300		1.15	
663		----		----	
840		----		----	
862		----		----	
875		----		----	
902		----		----	
912		----		----	
922		----		----	
974		----		----	
994	D5762	1015		-2.84	
1017		----		----	
1019	D5291	1020	C	-2.77	first reported:0.102
1023		----		----	
1059		----		----	
1066	D4629	1224	ex	0.08	Test result excluded, see §4.1
1106		----		----	
1146		----		----	
1150		----		----	
1161		----		----	
1173		----		----	
1201	D3228	854		-5.10	
1213	D3228	1145		-1.02	
1235		----		----	
1316		----		----	
1324	D4629	1310	ex	1.29	Test result excluded, see §4.1
1326	D5762	1302		1.18	
1402		----		----	
1431		----		----	
1460		----		----	
1461		----		----	
1543		----		----	
1564	D4629	1163.0	ex	-0.77	Test result excluded, see §4.1
1577	D4629	980.0	ex	-3.33	Test result excluded, see §4.1
1622		----		----	
1748		----		----	
1792		----		----	
1850		----		----	
1874		----		----	
1876		----		----	
1877		----		----	
1895		----		----	
1957		----		----	
1969		----		----	
1997		----		----	
2122		----		----	
2129	D3228	1567		4.89	
9099		----		----	
9100		----		----	
9101		----		----	
9142		----		----	
9143		----		----	

normality OK
n 8
outliers 0 (+4 excl)
mean (n) 1218.03
st.dev. (n) 255.867
R(calc.) 716.43
R(D3228:08) 200.00

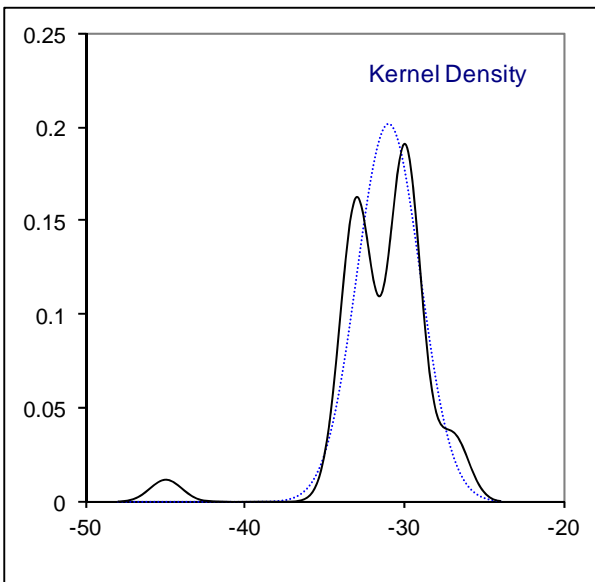
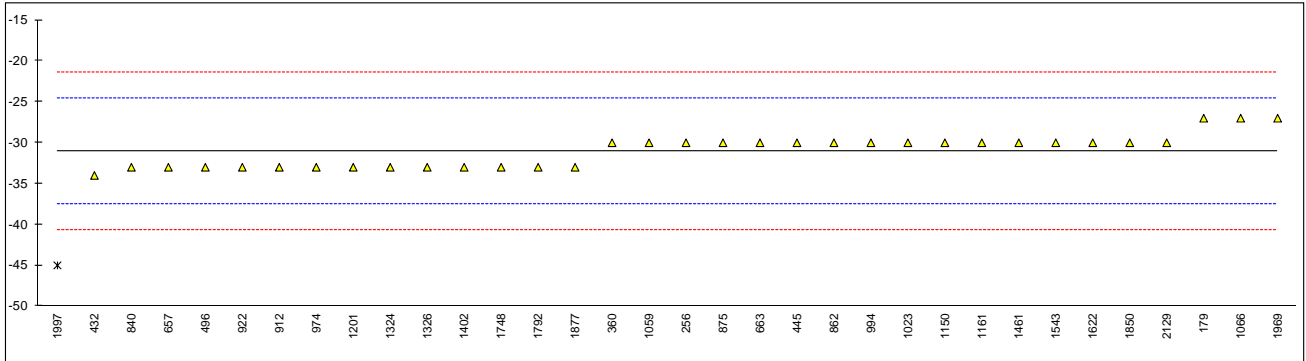
Compare R(D5762:12) = 323.99



Determination of Pour Point, Manual on sample #15080; results in °C

lab	method	value	mark	z(targ)	remarks
173		----		----	
178		----		----	
179	IP15	-27		1.25	
237	D97	<-24		----	
252		----		----	
254	D97	<-27		----	
256	D97	-30		0.32	
311		----		----	
315		----		----	
333		----		----	
343	D97	<-40		<-2.79	False negative?
349		----		----	
360	D97	-30		0.32	
432	D97	-34		-0.92	
445	D97	-30		0.32	
450		----		----	
451		----		----	
473		----		----	
496	D97	-33		-0.61	
541	D97	<-20		----	
551		----		----	
614		----		----	
657	D97	-33		-0.61	
663	D97	-30		0.32	
840	D97	-33		-0.61	
862	D97	-30		0.32	
875	D97	-30		0.32	
902		----		----	
912	D97	-33		-0.61	
922	D97	-33.0		-0.61	
974	D97	-33		-0.61	
994	D97	-30		0.32	
1017		----		----	
1019		----		----	
1023	D97	-30		0.32	
1059	D97	-30		0.32	
1066	D97	-27		1.25	
1106		----		----	
1146		----		----	
1150	ISO3016	-30		0.32	
1161	D97	-30		0.32	
1173		----		----	
1201	D97	-33		-0.61	
1213	D97	<-24		----	
1235		----		----	
1316		----		----	
1324	D97	-33		-0.61	
1326	D97	-33		-0.61	
1402	D97	-33		-0.61	
1431		----		----	
1460		----		----	
1461	ISO3016	-30		0.32	
1543	D97	-30		0.32	
1564		----		----	
1577		----		----	
1622	D97	-30		0.32	
1748	D97	-33		-0.61	
1792	D97	-33		-0.61	
1850	ISO3016	-30		0.32	
1874		----		----	
1876		----		----	
1877	D97	-33		-0.61	
1895		----		----	
1957		----		----	
1969	ISO3016	-27		1.25	
1997	ISO3016	-45	R(0.01)	-4.35	
2122		----		----	
2129	D97	-30		0.32	
9099		----		----	
9100		----		----	
9101		----		----	
9142		----		----	
9143		----		----	

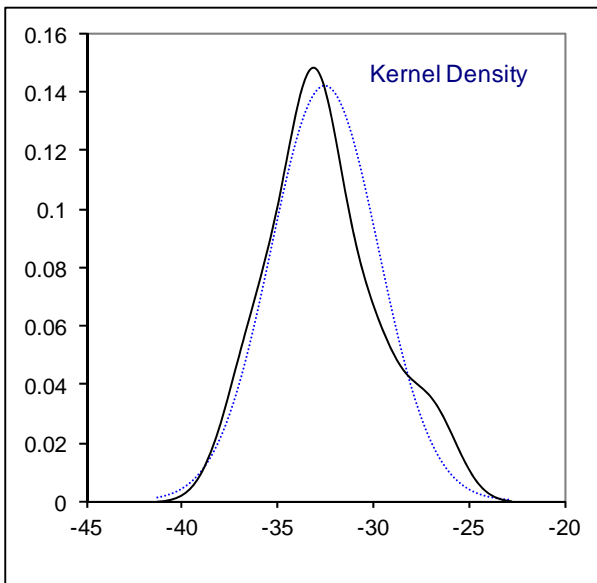
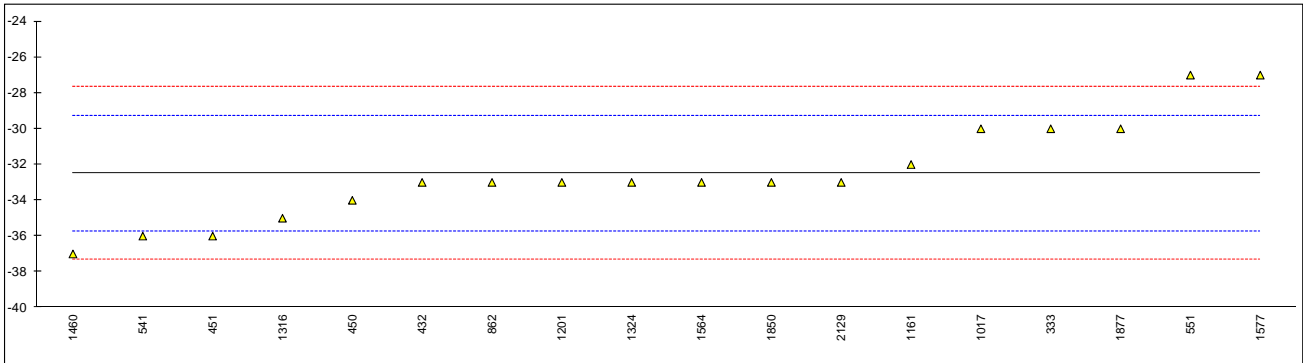
normality	OK
n	33
outliers	1
mean (n)	-31.03
st.dev. (n)	1.976
R(calc.)	5.53
R(D97:12)	9.00



Determination of Pour Point, Automated, 1°C interval on sample #15080; results in °C

lab	method	value	mark	z(targ)	remarks
173		----		----	
178		----		----	
179		----		----	
237		----		----	
252		----		----	
254		----		----	
256		----		----	
311		----		----	
315		----		----	
333	D5950	-30		1.56	
343		----		----	
349		----		----	
360		----		----	
432	D5950	-33		-0.31	
445		----		----	
450	D5950	-34		-0.93	
451	D5950	-36		-2.18	
473		----		----	
496		----		----	
541	D5950	-36		-2.18	
551	D5950	-27		3.42	
614		----		----	
657		----		----	
663		----		----	
840		----		----	
862	D5950	-33		-0.31	
875		----		----	
902		----		----	
912		----		----	
922		----		----	
974		----		----	
994		----		----	
1017	D5950	-30		1.56	
1019		----		----	
1023		----		----	
1059		----		----	
1066		----		----	
1106		----		----	
1146		----		----	
1150		----		----	
1161	D6749	-32		0.31	
1173		----		----	
1201	D5950	-33		-0.31	
1213		----		----	
1235		----		----	
1316	D5950	-35.0		-1.56	
1324	D5949	-33		-0.31	
1326		----		----	
1402		----		----	
1431		----		----	
1460	D5950	-37.0		-2.80	
1461		----		----	
1543		----		----	
1564	D5950	-33		-0.31	
1577	D5950	-27		3.42	
1622		----		----	
1748		----		----	
1792		----		----	
1850	D5950	-33		-0.31	
1874		----		----	
1876		----		----	
1877	D5950	-30		1.56	
1895		----		----	
1957		----		----	
1969		----		----	
1997		----		----	
2122		----		----	
2129	D5950	-33		-0.31	
9099		----		----	
9100		----		----	
9101		----		----	
9142		----		----	
9143		----		----	

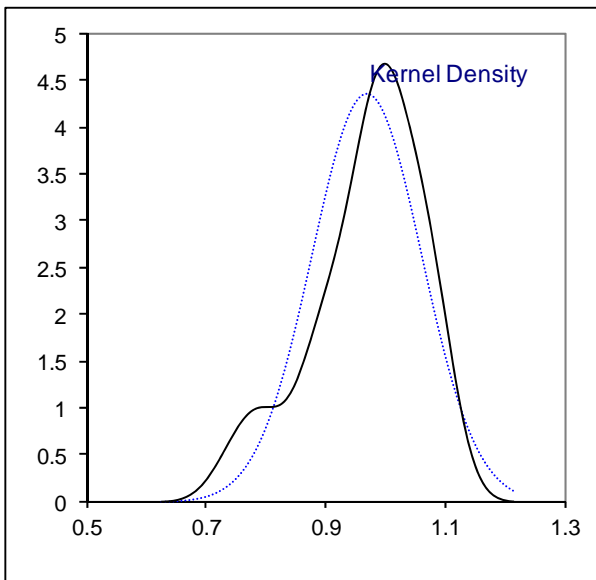
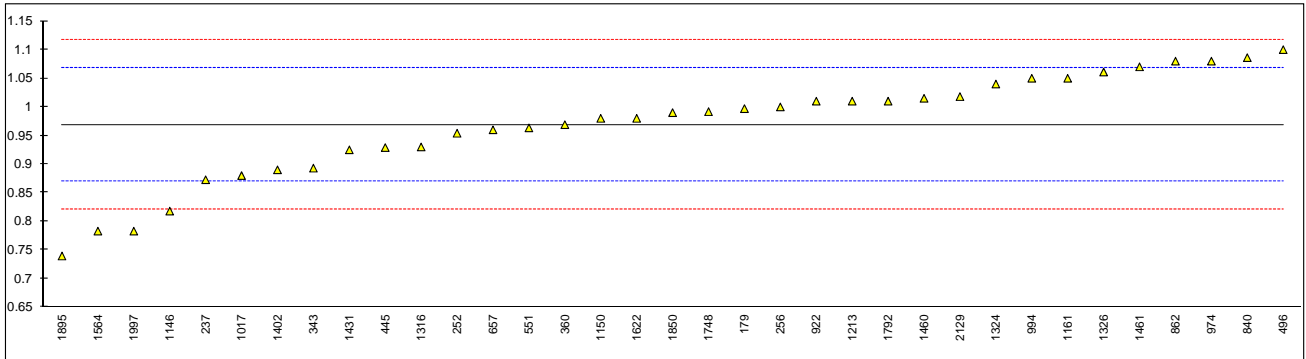
normality OK
n 18
outliers 0
mean (n) -32.50
st.dev. (n) 2.813
R(calc.) 7.88
R(D5950:14) 4.50



Determination of Sulphated Ash on sample #15080; results in %M/M

lab	method	value	mark	z(targ)	remarks
173		----		----	
178		----		----	
179	D874	0.997		0.57	
237	D874	0.87275		-1.94	
252	D874	0.954		-0.30	
254		----		----	
256	D874	1.0		0.63	
311		----		----	
315		----		----	
333		----		----	
343	D874	0.893		-1.53	
349		----		----	
360	D874	0.969		0.00	
432		----		----	
445	D874	0.929		-0.80	
450		----		----	
451		----		----	
473		----		----	
496	D874	1.10		2.65	
541		----		----	
551	D874	0.9633		-0.11	
614		----		----	
657	D874	0.96		-0.18	
663		----		----	
840	D874	1.086		2.37	
862	D874	1.08		2.25	
875		----		----	
902		----		----	
912		----		----	
922	D874	1.01		0.83	
974	D874	1.08		2.25	
994	D874	1.05		1.64	
1017	D874	0.88		-1.79	
1019		----		----	
1023		----		----	
1059		----		----	
1066		----		----	
1106		----		----	
1146	D874	0.81798		-3.05	
1150	ISO3987	0.98		0.23	
1161	ISO3987	1.05		1.64	
1173		----		----	
1201		----		----	
1213	D874	1.01		0.83	
1235		----		----	
1316	D874	0.930		-0.78	
1324	D874	1.040		1.44	
1326	D874	1.061		1.86	
1402	D874	0.89		-1.59	
1431	D874	0.925		-0.88	
1460	D874	1.015		0.93	
1461	ISO3987	1.07		2.04	
1543		----		----	
1564	D874	0.783		-3.75	
1577		----		----	
1622	D874	0.98		0.23	
1748	D874	0.9917		0.46	
1792	D874	1.01		0.83	
1850	ISO3987	0.99		0.43	
1874		----		----	
1876		----		----	
1877		----		----	
1895	D874	0.7395		-4.63	
1957		----		----	
1969		----		----	
1997	ISO3987	0.783		-3.75	
2122		----		----	
2129	D874	1.018		0.99	
9099		----		----	
9100		----		----	
9101		----		----	
9142		----		----	
9143		----		----	

normality OK
 n 35
 outliers 0
 mean (n) 0.969
 st.dev. (n) 0.0914
 R(calc.) 0.256
 R(D874:13a) 0.139



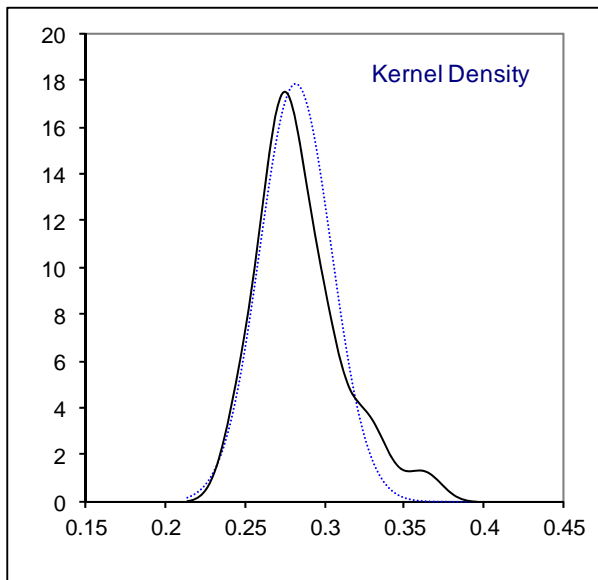
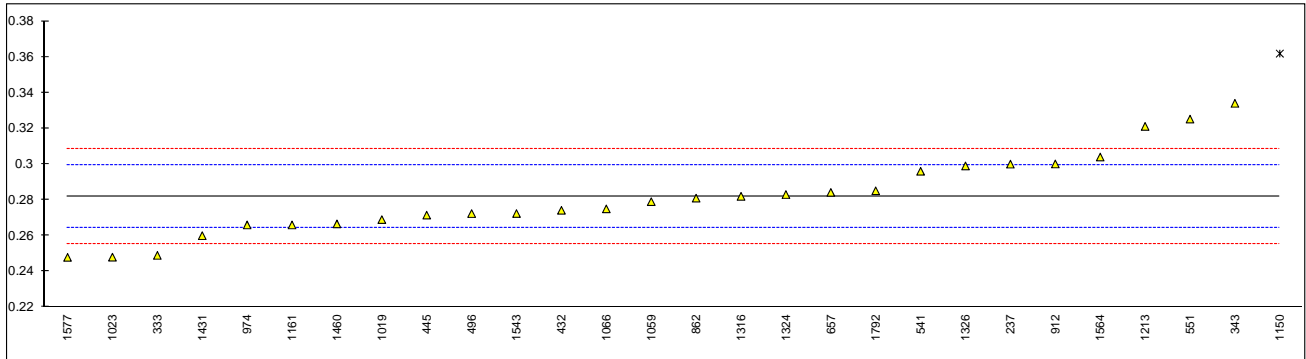
Determination of Sulphur on sample #15080; results in %M/M

lab	method	value	mark	z(targ)	remarks
173		----		----	
178		----		----	
179		----		----	
237	D4294	0.300		2.03	
252		----		----	
254		----		----	
256		----		----	
311		----		----	
315		----		----	
333	D4294	0.249		-3.70	
343	IP336	0.334		5.86	
349		----		----	
360		----		----	
432	D4951	0.2742		-0.87	
445	D2622	0.2715		-1.17	
450		----		----	
451		----		----	
473		----		----	
496	D2622	0.27236		-1.07	
541	D4294	0.296		1.58	
551	D4294	0.3252		4.87	
614		----		----	
657	D4294	0.2842		0.26	
663		----		----	
840		----		----	
862	D2622	0.281		-0.10	
875		----		----	
902		----		----	
912	D4294	0.3001		2.05	
922		----		----	
974	D4294	0.266		-1.79	
994		----		----	
1017		----		----	
1019	D1552	0.269		-1.45	
1023	D4294	0.248		-3.82	
1059	ISO14596	0.279		-0.33	
1066	D2622	0.275		-0.78	
1106		----		----	
1146		----		----	
1150	ISO8754	0.3618	R(0.05)	8.99	
1161	ISO8754	0.266		-1.79	
1173		----		----	
1201		----	W	----	result withdrawn, first reported: 1959
1213	D4294	0.3211		4.41	
1235		----		----	
1316	D7751	0.282		0.01	
1324	D4294	0.283		0.12	
1326	D6481	0.299		1.92	
1402	IP490	>0.0500		----	
1431	in house	0.26		-2.47	
1460	D4294	0.2666		-1.72	
1461		----		----	
1543	D4294	0.2724		-1.07	
1564	D5453	0.304		2.48	
1577	D5453	0.2479		-3.83	
1622		----		----	
1748		----		----	
1792	D4294	0.2851		0.36	
1850		----		----	
1874		----		----	
1876		----		----	
1877		----		----	
1895		----		----	
1957		----		----	
1969		----		----	
1997		----		----	
2122		----		----	
2129		----		----	
9099		----		----	
9100		----		----	
9101		----		----	
9142		----		----	
9143		----		----	

Only ASTM D2622 data

Only ASTM D4294 data

normality	OK	not OK	OK
n	27	4	13
outliers	1	0	0
mean (n)	0.2819	0.2750	0.2844
st.dev. (n)	0.02233	0.00429	0.02418
R(calc.)	0.0625	0.0120	0.0677
R(D2622:10)	0.0249	0.0244	0.0321

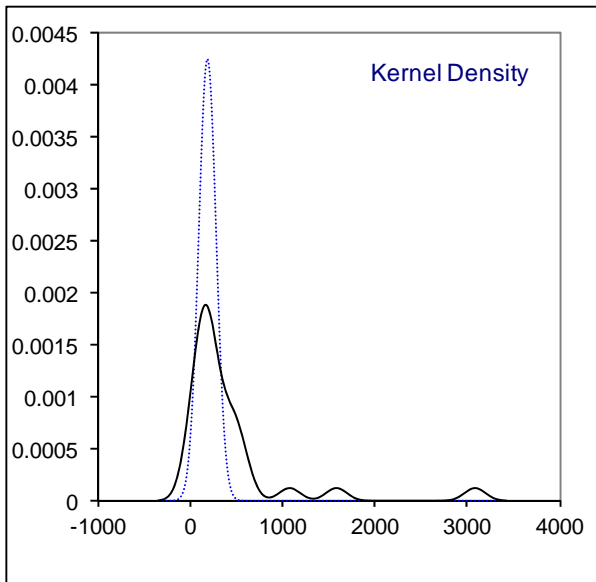
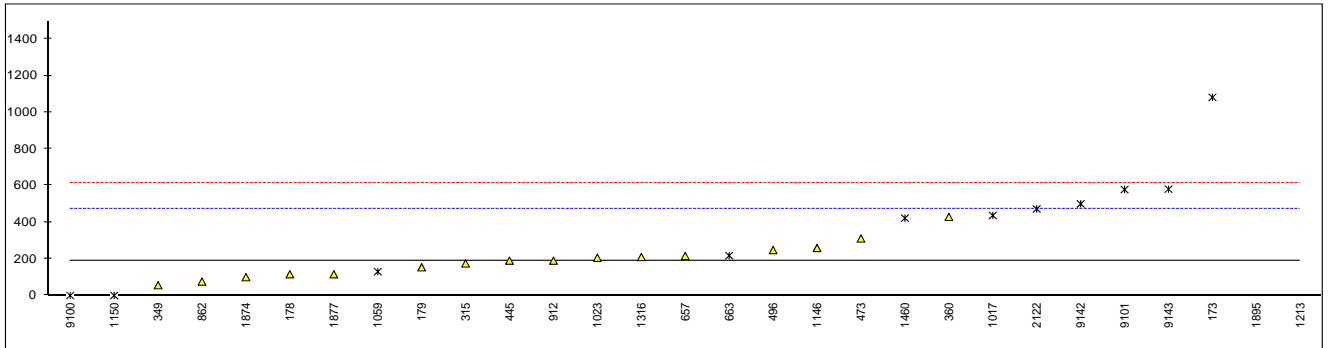


Determination of Water on sample #15080; results in mg/kg

lab	method	value	mark	z(target)	remarks
173	D6304-A	1081	ex	6.32	result excluded, see §4.1
178	D6304-C	116		-0.53	
179	D6304-C	154		-0.26	
237		----		----	
252		----		----	
254		----		----	
256		----		----	
311		----		----	
315	D6304-C	174.95		-0.11	
333		----		----	
343		----		----	
349	D6304-C	56		-0.96	
360	D6304-C	429.2		1.69	
432		----		----	
445	D6304-C	190		-0.01	
450		----		----	
451		----		----	
473	D6304-C	311		0.85	
496	D6304-C	248		0.41	
541		----		----	
551		----		----	
614		----		----	
657	D6304-C	216		0.18	
663	D6304	217.4	ex	0.19	result excluded, see §4.1
840		----		----	
862	D6304-C	76		-0.81	
875		----		----	
902		----		----	
912	D6304-C	190		-0.01	
922	D6304-A	<17.0	ex	----	result excluded, see §4.1
974		----		----	
994		----		----	
1017	D6304-A	436.5	ex	1.74	result excluded, see §4.1
1019		----		----	
1023	D6304-C	206.1		0.11	
1059	D6304-A	130	ex	-0.43	result excluded, see §4.1
1066		----		----	
1106		----		----	
1146	D6304-C	259.0000		0.48	
1150	ISO12937	0.0001	ex	-1.35	result excluded, see §4.1, possibly an unit error?
1161		----		----	
1173		----		----	
1201		----		----	
1213	D6304	3089	ex	20.57	result excluded, see §4.1
1235		----		----	
1316	D6304-C	210		0.14	
1324		----		----	
1326		----		----	
1402		----		----	
1431		----		----	
1460	D6304-A	422	ex	1.64	result excluded, see §4.1
1461		----		----	
1543		----		----	
1564		----		----	
1577		----		----	
1622		----		----	
1748		----		----	
1792		----		----	
1850		----		----	
1874		100		-0.64	result excluded, see §4.1
1876		----		----	
1877	D6304-C	116		-0.53	
1895	D6304	1588.13	ex	9.92	result excluded, see §4.1
1957		----		----	
1969		----		----	
1997		----		----	
2122	in house	472	ex	2.00	result excluded, see §4.1
2129		----		----	
9099	D4006	<20	ex	----	result excluded, see §4.1
9100	D4006	0.00	ex	-1.35	result excluded, see §4.1
9101	D95	578.0345	ex	2.75	result excluded, see §4.1
9142	D4006	500	ex	2.20	result excluded, see §4.1
9143	D95	580.0464	ex	2.76	result excluded, see §4.1

All reported results

normality	suspect	OK
n	16	26
outliers	0 (+13 excl)	3
mean (n)	190.77	245.70
st.dev. (n)	94.105	169.899
R(calc.)	263.49	475.72
R(D6304:07)	394.43	459.11

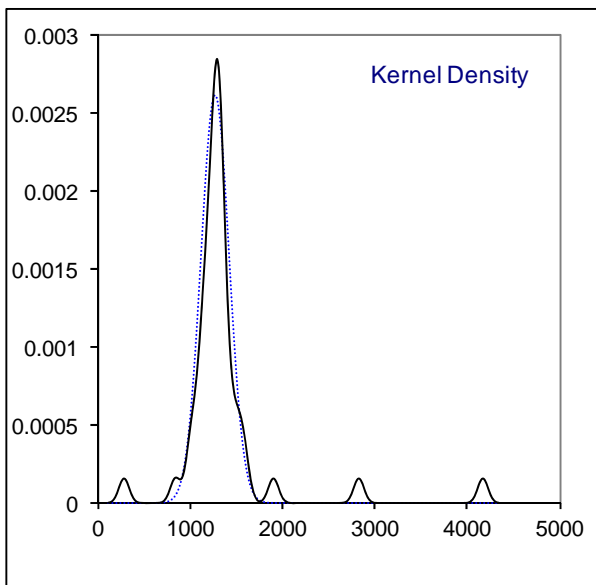
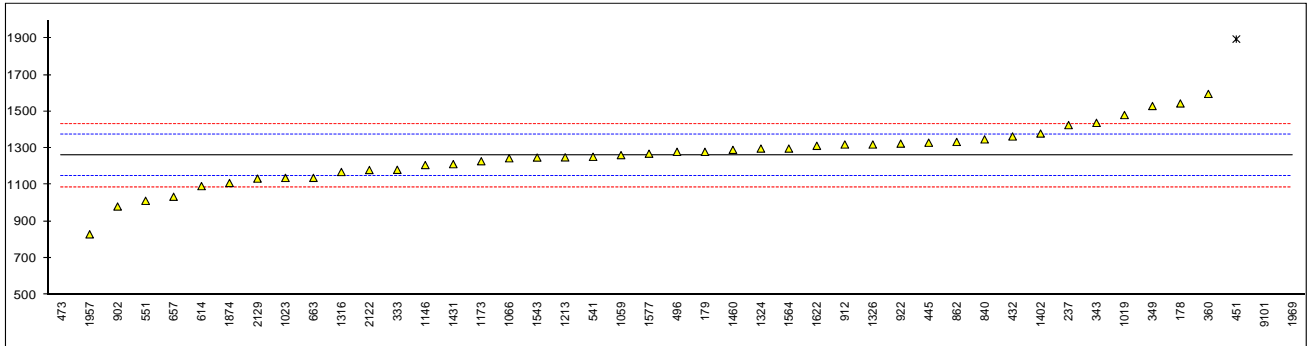


Determination of Calcium (Ca) on sample #15080; results in mg/kg

lab	method	value	mark	z(targ)	remarks
173		----		----	
178	D5185	1544		4.93	
179	D5185	1280		0.34	
237	D5185	1426		2.88	
252		----		----	
254		----		----	
256		----		----	
311		----		----	
315		----		----	
333	D5185	1182		-1.37	
343	D5185	1438.3		3.09	
349	D5185	1530		4.69	
360	D5185	1596		5.83	
432	D4951	1364		1.80	
445	D5185	1330		1.21	
450		----		----	
451	D5185	1895.2	R(0.01)	11.04	
473	D5185	271.1	R(0.01)	-17.21	
496	D5185	1280		0.34	
541	D5185	1253		-0.13	
551	D5185	1013		-4.30	
614	D5185	1093		-2.91	
657	D5185	1035		-3.92	
663	D5185	1138.1		-2.13	
840	D4628	1348		1.52	
862	D5185	1334		1.28	
875		----		----	
902	D5185	981.65		-4.85	
912	D5185	1320		1.03	
922	D5185	1325		1.12	
974		----		----	
994		----		----	
1017		----		----	
1019	D5185	1481		3.83	
1023	D5185	1137.88		-2.13	
1059	in house	1262		0.03	
1066	D4951	1245		-0.27	
1106		----		----	
1146	in house	1208		-0.91	
1150		----		----	
1161		----		----	
1173	in house	1228.92		-0.55	
1201		----		----	
1213	D5185	1250		-0.18	
1235		----		----	
1316	D5185	1170		-1.57	
1324	D5185	1298		0.65	
1326	D5185	1320		1.03	
1402	D5185	1380		2.08	
1431	in house	1213		-0.83	
1460	D5185	1291		0.53	
1461		----		----	
1543	D5185	1249		-0.20	
1564	D4951	1298		0.65	
1577	in house	1270		0.17	
1622	D5185	1313		0.91	
1748		----		----	
1792		----		----	
1850		----		----	
1874	D6595	1110		-2.62	
1876		----		----	
1877		----		----	
1895		----		----	
1957	D5185	830		-7.49	
1969	in house	4171.54	R(0.01)	50.63	
1997		----		----	
2122	D5185	1181.00		-1.38	
2129	D5185	1134		-2.20	
9099		----		----	
9100		----		----	
9101	EPA.3005	2823.047	R(0.01)	27.18	
9142		----		----	
9143		----		----	

normality OK
 n 41
 outliers 4
 mean (n) 1260.51
 st.dev. (n) 152.791
 R(calc.) 427.81
 R(D5185:13e1) 160.99

application range: 40 - 9000 mg/kg

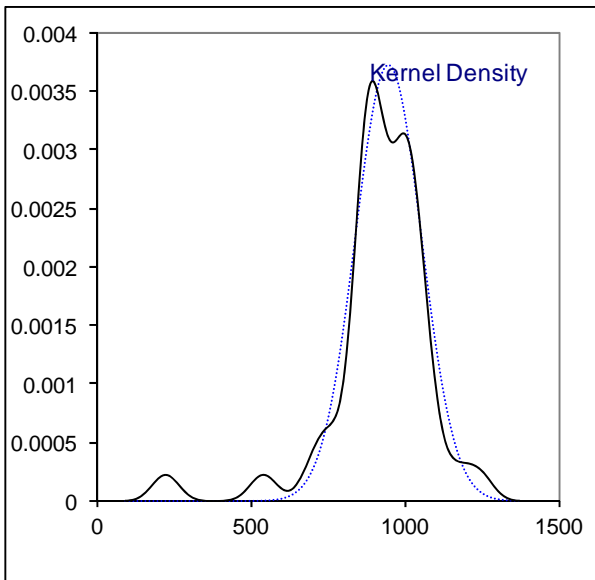
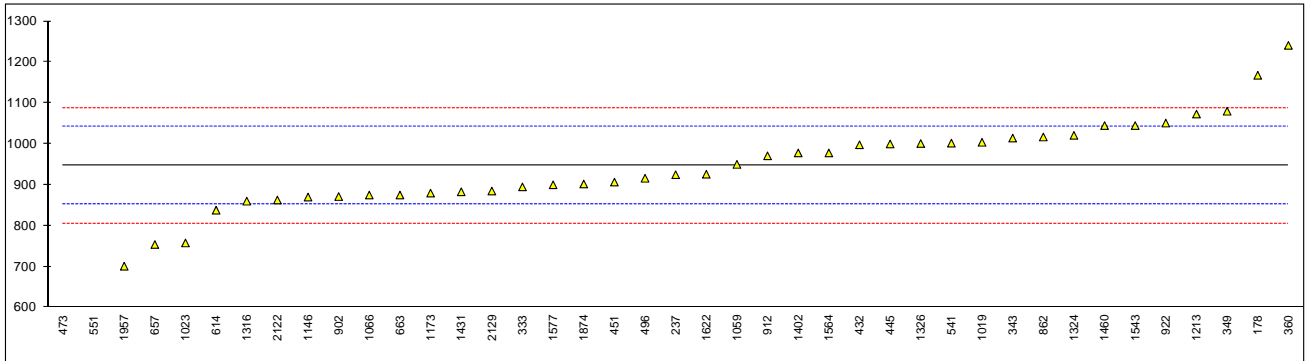


Determination of Phosphorus (P) on sample #15080; results in mg/kg

lab	method	value	mark	z(targ)	remarks
173		----		----	
178	D5185	1168		4.68	
179		----		----	
237	D5185	924.7		-0.47	
252		----		----	
254		----		----	
256		----		----	
311		----		----	
315		----		----	
333	D5185	895		-1.09	
343	D5185	1014.3		1.43	
349	D5185	1080		2.82	
360	D5185	1241		6.23	
432	D4951	998		1.09	
445	D5185	1000		1.13	
450		----		----	
451	D5185	906.8		-0.84	
473	D5185	225.1	R(0.01)	-15.27	
496	D5185	916		-0.65	
541	D5185	1002		1.17	
551	D5185	542	R(0.05)	-8.56	
614	D5185	838		-2.30	
657	D5185	754		-4.08	
663	D5185	875.1		-1.51	
840		----		----	
862	D5185	1017		1.49	
875		----		----	
902	D5185	871.3		-1.60	
912	D5185	971		0.51	
922	D5185	1051		2.21	
974		----		----	
994		----		----	
1017		----		----	
1019	D5185	1004		1.21	
1023	D5185	757.812		-4.00	
1059	in house	950		0.07	
1066	D4951	875		-1.52	
1106		----		----	
1146	in house	870.1		-1.62	
1150		----		----	
1161		----		----	
1173	in house	879.74		-1.42	
1201		----		----	
1213	D5185	1073		2.67	
1235		----		----	
1316	D5185	860		-1.83	
1324	D5185	1021		1.57	
1326	D5185	1001		1.15	
1402	D5185	978		0.66	
1431	in house	883		-1.35	
1460	D5185	1045		2.08	
1461		----		----	
1543	D5185	1045		2.08	
1564	D4951	978		0.66	
1577	in house	900.0		-0.99	
1622	D5185	926		-0.44	
1748		----		----	
1792		----		----	
1850		----		----	
1874	D6595	902		-0.95	
1876		----		----	
1877		----		----	
1895		----		----	
1957	D5185	701		-5.20	
1969		----		----	
1997		----		----	
2122	D5185	862.97		-1.77	
2129	D5185	884.7		-1.31	
9099		----		----	
9100		----		----	
9101		----		----	
9142		----		----	
9143		----		----	

normality OK
 n 39
 outliers 2
 mean (n) 946.68
 st.dev. (n) 107.102
 R(calc.) 299.89
 R(D5185:13e1) 132.30

application range: 10 - 1000 mg/kg

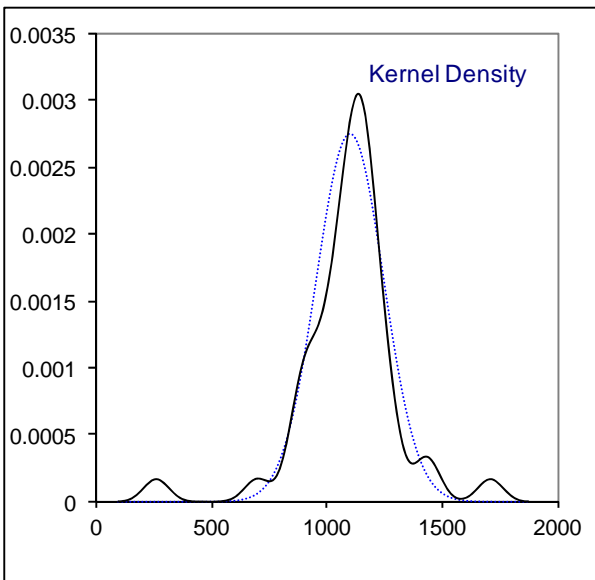
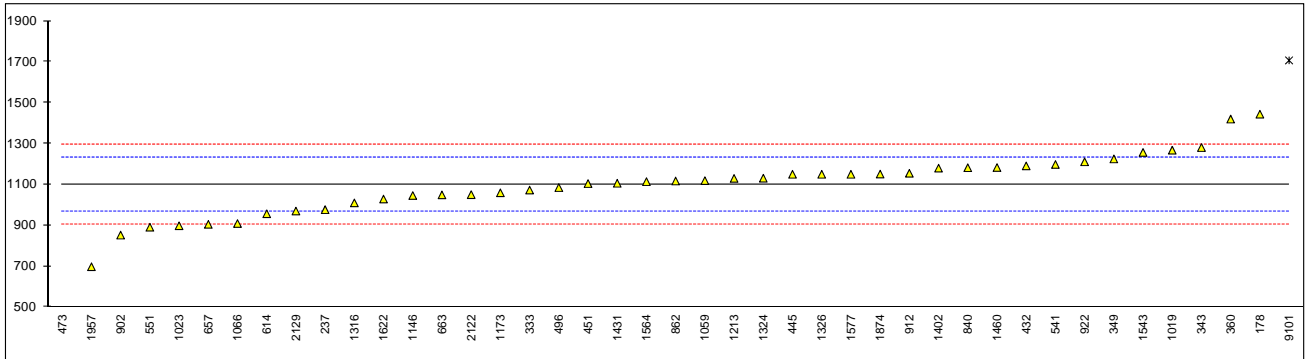


Determination of Zinc (Zn) on sample #15080; results in mg/kg

lab	method	value	mark	z(targ)	remarks
173		----		----	
178	D5185	1444		5.25	
179		----		----	
237	D5185	976.9		-1.87	
252		----		----	
254		----		----	
256		----		----	
311		----		----	
315		----		----	
333	D5185	1073		-0.40	
343	D5185	1280.7		2.76	
349	D5185	1225		1.91	
360	D5185	1420		4.88	
432	D4951	1191		1.39	
445	D5185	1150		0.77	
450		----		----	
451	D5185	1104.5		0.08	
473	D5185	261.2	R(0.01)	-12.77	
496	D5185	1085		-0.22	
541	D5185	1198		1.50	
551	D5185	891		-3.18	
614	D5185	957		-2.17	
657	D5185	905		-2.96	
663	D5185	1049.4		-0.76	
840	D4628	1182		1.26	
862	D5185	1117		0.27	
875		----		----	
902	D5185	852.4		-3.76	
912	D5185	1155		0.85	
922	D5185	1211		1.70	
974		----		----	
994		----		----	
1017		----		----	
1019	D5185	1268		2.57	
1023	D5185	898.082		-3.07	
1059	in house	1119		0.30	
1066	D4951	909		-2.90	
1106		----		----	
1146	in house	1046		-0.81	
1150		----		----	
1161		----		----	
1173	in house	1059.57		-0.61	
1201		----		----	
1213	D5185	1130		0.47	
1235		----		----	
1316	D5185	1010		-1.36	
1324	D5185	1131		0.48	
1326	D5185	1150		0.77	
1402	D5185	1180		1.23	
1431	in house	1106		0.10	
1460	D5185	1183		1.27	
1461		----		----	
1543	D5185	1257		2.40	
1564	D4951	1114		0.22	
1577	in house	1150		0.77	
1622	D5185	1029		-1.07	
1748		----		----	
1792		----		----	
1850		----		----	
1874	D6595	1151		0.79	
1876		----		----	
1877		----		----	
1895		----		----	
1957	D5185	698		-6.12	
1969	in house	n.d.		----	False negative?
1997		----		----	
2122	D5185	1050.33		-0.75	
2129	D5185	970.1		-1.97	
9099		----		----	
9100		----		----	
9101	EPA3005	1707.206	R(0.01)	9.26	
9142		----		----	
9143		----		----	

normality OK
 n 41
 outliers 2
 mean (n) 1099.44
 st.dev. (n) 145.430
 R(calc.) 407.20
 R(D5185:13e1) 183.81

application range: 60 – 1600 mg/kg



APPENDIX 2

Number of participants per country

1 lab in ARGENTINA
1 lab in AUSTRALIA
1 lab in AUSTRIA
1 lab in AZERBAIJAN
2 labs in BELGIUM
1 lab in BRAZIL
1 lab in BRUNEI
4 labs in BULGARIA
3 labs in CHINA, People's Republic
1 lab in CROATIA
1 lab in CZECH REPUBLIC
1 lab in FRANCE
2 labs in GERMANY
1 lab in GREECE
1 lab in INDIA
1 lab in INDONESIA
1 lab in IRELAND
1 lab in JORDAN
2 labs in KENYA
1 lab in MALAYSIA
1 lab in MEXICO
1 lab in MOROCCO
5 labs in NETHERLANDS
6 labs in NIGERIA
2 labs in NORWAY
1 lab in PAKISTAN
1 lab in RUSSIAN FEDERATION
1 lab in SAUDI ARABIA
1 lab in SINGAPORE
1 lab in SLOVENIA
4 labs in SPAIN
2 labs in SUDAN
2 labs in SWEDEN
1 lab in TANZANIA
1 lab in THAILAND
2 labs in TURKEY
1 lab in UNITED ARAB EMIRATES
7 labs in UNITED KINGDOM
3 labs in UNITED STATES OF AMERICA
2 labs in VIETNAM

APPENDIX 3**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
E	= error in calculations
ex	= excluded from calculations
U	= reported in different unit
W	= result withdrawn on request of the participants
fr.	= first reported
S	= scope of the reported method is not applicable
n.a.	= not applicable
n.e.	= not evaluated
SDS	= Safety Data Sheet

Literature:

- 1 iis Interlaboratory Studies, Protocol for the Organization, Statistics and Evaluation, April 2014
- 2 ASTM E178-89
- 3 ASTM E1301-89
- 4 ISO 5725-86
- 5 ISO 5725, parts 1-6, 1994
- 6 ISO13528-05
- 7 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
- 8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 9 IP 367/84
- 10 DIN 38402 T41/42
- 11 P.L. Davies, First reported Z. Anal. Chem, 331, 513, (1988)
- 12 J.N. Miller, Analyst, 118, 455, (1993)
- 13 Analytical Methods Committee Technical brief, No4 January 2001.
- 14 The Royal Society of Chemistry 2002, Analyst 2002, 127 pages 1359-1364, P.J. Lowthian and M. Thompson (see <http://www.rsc.org/suppdata/an/b2/b205600n/>)
- 15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), pp. 165-172, (1983)