

# Results of Proficiency Test Unused Lubricating Oil May 2013

Organised by: Institute for Interlaboratory Studies  
Spijkenisse, the Netherlands

Author: ing. L. Dijkstra  
Correctors: dr. R.G. Visser & ing. L. Sweere  
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## **1 INTRODUCTION**

Since 1997, the Institute for Interlaboratory Studies organises every year a proficiency test for Lubricating Oil. In the annual proficiency testing program 2012/2013, it was decided to continue the proficiency test for the analyses of unused Lubricating Oil. In this interlaboratory study, 85 laboratories in 47 different countries have participated. See appendix 3 for the number of participants per country. In this report, the results of the 2013 Lubricating Oil (unused oil) proficiency test are presented and discussed.

## **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 one bottle of 1L (labelled #13062) of unused Lubricating Oil 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 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 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 January 2010 (iis-protocol, version 3.2). This protocol can be downloaded via the FAQ page of the iis website <http://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 200 litre bulk material (Shell Rimula RT4 15W-40) was transferred, after homogenizing, into 183 brown glass bottles of 1 litre (labelled #13062). The homogeneity of the subsamples #13062 was checked by determination of Density @15°C in accordance with ASTM D4052:11 and Kinematic Viscosity @ 40°C in accordance with ASTM D445:12 on 8 stratified randomly selected samples.

	Density @ 15 °C in kg/L	Viscosity @40°C in mm <sup>2</sup> /s
Sample #13062-1	0.87754	116.5
Sample #13062-2	0.87754	116.4
Sample #13062-3	0.87757	116.4
Sample #13062-4	0.87755	116.4
Sample #13062-5	0.87756	116.4
Sample #13062-6	0.87754	116.4
Sample #13062-7	0.87745	116.4
Sample #13062-8	0.87758	116.1

Table 1: homogeneity test results of subsamples #13062

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 @ 15 °C in kg/L	Viscosity @40°C in mm <sup>2</sup> /s
r (sample #13062)	0.00011	0.3
reference test	ASTM D4052:11	ASTM D445:12
0.3 x R(reference test)	0.00015	0.3

Table 2: evaluation of the repeatabilities of the subsamples #13062

The calculated repeatabilities for Density and Viscosity were respectively less and equal to 0.3 times the corresponding reproducibilities of the reference methods. Therefore, homogeneity of the subsamples #13062 was assumed.

To each of the participating laboratories, 1 sample of 1 L in a brown glass bottle (labelled #13062) was sent on May 01, 2013.

## 2.5 ANALYSES

The participants were requested to determine on sample #13062: Acid Number (Total), Base Number (Total), Color ASTM, Conradson Carbon Residue, Ramsbottom Carbon Residue, Carbon Residue (Micro method), Density @ 15°C, Flash Point COC, Flash Point PMcc, Kinematic Viscosity @ 40°C and @ 100°C, Viscosity Index, Apparent Viscosity @ -20°C , Viscosity Stabinger @ 40°C and @100°C, 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 some of the required standards and a letter of instructions were prepared and made available for download on the iis website [www.iisnl.com](http://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 3.2) of January 2010. 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. 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 and Grubbs 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. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs 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 them with a factor of 2.8.

### 3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported 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 "x". Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. The Kernel Density 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.12 and 13).

### 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 problems. Twenty-two participants reported after the final reporting date and seven participants did not report any test results at all. Not all laboratories were able to report all analyses requested. In total 78 participants reported 879 test results. Observed were 29 outlying results, which is 3.3% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

Not all original data sets proved to have a normal distribution. Non-Gaussian distributions were found for the following determinations: Base Number (Total), Color, Density @ 15°C, Viscosity Index, Viscosity Stabinger @100°C, Pour Point (manual and automated) and Phosphorus. In these cases the statistical evaluation should be used with due care.

### 4.1 EVALUATION PER TEST

In this section, the results are discussed per test. The methods, which are used by the various laboratories, are taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the original data. The abbreviations, used in these tables, are listed in appendix 4.

Acid Number (total): This determination was very problematic. No statistical outliers were observed. However, the results of three laboratories that reported to have used ASTM D974, a test method that is not equivalent with ASTM D664, were excluded prior to the statistical evaluation. The calculated reproducibility after rejection of the suspect data is not at all agreement with the requirements of ASTM D664:11a. This phenomena has been observed also in previous round robins and therefore the participants were requested to report a number of actual details of the test performed. It appeared that at least eleven laboratories did not follow ASTM D664 to the letter. These laboratories did either treat the KOH solution not in the correct way and/or did not use the recommended size of test portion, see appendix 2. When the ASTM D664 data were evaluated separately, the average Acid Number of the laboratories that reported to have used an intake of approx 5 grams is significantly lower than the average Acid Number of the laboratories that used an intake of approx 1 gram. This can only explain for a small part of the observed high spread.

Base Number (total): This determination was problematic. Five statistical outliers were observed. And one test result was excluded from the statistical evaluation, as the reported test method is not equivalent with ASTM D2896. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of ASTM D2896:11.

Color: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with ASTM D1500:07.

- Conradson CR: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D189:10.
- Ramsbottom CR: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements of ASTM D524:10. The low number of results may (partly) explain the observed spread.
- Carbon Residue (Micro method) This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with the requirements of ASTM D4530:11.
- Density @ 15°C: This determination was very problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers, is not at all in agreement with the requirements of ASTM D4052:11. The large spread may be explained by possibly not correcting the test result for viscosity properly.
- Flash Point COC: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in agreement with ASTM D92:12.
- Flash Point PMcc: This determination was not problematic. Two statistical outliers were observed (both laboratories reported to have used procedure B, see below). The calculated reproducibility after rejection of the statistical outliers is in good agreement with ASTM D93:11. Remarkably 7 laboratories reported to have used ASTM D93, procedure B. This method B may not applicable for unused lubricating oils. Also it was remarkable to notice that four laboratories reported unrealistic run times, that do not match the reported procedure (A or B). Use of procedure A (temperature increase rate of 5° to 6°C per min) should give shorter run times than in case that procedure B ( temperature rate 1° to 1.6°C per min) was used.
- Kin.Visco.@ 40°C: This determination was problematic. Six statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the (strict) requirements of ASTM D445:12. A number of laboratories may have used a Canon Fenske Routine Viscometer instead of an Ubbelohde Viscometer. This may (partly) explain the large spread.
- Kin.Visco.@ 100°C: This determination was very problematic. Two statistical outliers were observed and two test results were excluded from the statistical evaluation, as the reported test method is not equivalent with ASTM D445:12. The calculated reproducibility after rejection of the suspected data is not at all in agreement with the strict requirements of ASTM D445:12. A number of laboratories may



have used a Canon Fenske Routine Viscometers instead of Ubbelohde Viscometers. This may partly explain the large spread.

Viscosity Index

This determination was very problematic. Two statistical outliers were observed and two other test results were excluded because these results probably contained a calculation error. The calculated reproducibility after rejection of the four suspect test results is not at all in agreement with the requirements of ASTM D2270:10.

Two originally reported test results (laboratories 657 and 1488) were not replaced by corrected results as the results did not contain calculation errors.

Apparent Viscosity  
@ -20°C

This determination was not problematic. No statistical outliers were observed and the calculated reproducibility in agreement with the requirements of ASTM D5293:10 for both the constant cooling instruments and the electrically cooled instruments. The reproducibility for thermo electrically cooled instruments was used for the statistical evaluation.

Visco. Stabinger  
at 40°C

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 D7042:12a.

Visco. Stabinger  
at 100°C

This determination was very problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements ASTM D7042:12a.

The test results may be divided in to two groups. When the test results from group 1 and group 2 are evaluated separately then the calculated reproducibilities are respectively not in agreement and not at all in agreement with the requirements of ASTM D7042:12a.

Nitrogen:

This determination may be problematic depending on the test method used for the evaluation. No statistical outliers were observed. Three 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 is not in agreement with the requirements of ASTM D3228:08 used by five laboratories, but it is in good agreement with the less strict requirements of ASTM D5762:11, used by four laboratories.

Pour Point (manual): This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility not in agreement with the requirements ASTM D97:12. The applied rounding of the results may (partly) explain the observed spread.

Pour Point (automated): This determination was not problematic. One statistical outlier was observed. Remarkably two laboratories reported to have used ASTM D97 and one laboratory used ISO3016 which are manual test methods. These three

test results were excluded from the statistical evaluation. The calculated reproducibility after rejection of the suspect data is in agreement with ASTM D5950:07.

Sulphated Ash: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D874:07.

Sulphur: This determination was very problematic. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers 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 data was evaluated separately, the calculated reproducibility is much smaller and in agreement with the requirements of ASTM D2622:10. However, the calculated Sulphur content does not change significantly.

Water: 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 D6304:07. 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. When the ASTM D6304 method C data was evaluated separately, the calculated reproducibility is smaller and again in agreement with the requirements of ASTM D6304:07.

Calcium: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D5185:09.

Phosphorus: This determination was problematic. Only one statistical outlier was observed. However, the calculated reproducibility, after rejection of the statistical outlier is not in agreement with the requirements of ASTM D5185:09. The test results appear to be divided into two groups. When the test results of the two groups are evaluated separately, each of the calculated reproducibilities is in good agreement with the requirements of ASTM D5185:08. Causes for the two groups of test results may be different application of correction factors and/or quantification with or without an internal standard.

Zinc: This determination was problematic. Only one statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D5185:09.

## 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)
Total Acid Number	mg KOH/g	44	2.85	1.01	0.54
Total Base Number	mg KOH/g	38	10.7	1.15	0.75
Color		39	3.5	0.4	1.0
Conradson Carbon Residue	%M/M	20	1.12	0.16	0.26
Ramsbottom Carbon Residue	%M/M	5	1.08	0.54	0.15
Carbon Residue (micro method)	%M/M	30	1.17	0.17	0.20
Density @ 15 °C	kg/L	65	0.8778	0.0018	0.0005
Flash Point COC	°C	47	227.2	16.5	18.0
Flash Point PMcc	°C	55	203.3	8.3	14.4
Kinematic Viscosity @ 40 °C	mm <sup>2</sup> /s	60	116.74	1.24	0.89
Kinematic Viscosity @ 100 °C	mm <sup>2</sup> /s	57	15.53	0.24	0.12
Viscosity Index		51	140.1	3.4	2.0
Apparent Viscosity @ -20 °C	mPa·s	12	6185	295	371
Stabinger Viscosity @ 40 °C	mm <sup>2</sup> /s	14	116.14	1.76	0.68
Stabinger Viscosity @ 100 °C	mm <sup>2</sup> /s	12	15.59	0.22	0.06
Nitrogen	mg/kg	10	1140	293	200
Pour Point, manual	°C	36	-32.0	11.7	9.0
Pour Point, automated	°C	15	-35.1	3.8	4.5
Sulphated Ash	%M/M	35	1.08	0.21	0.15
Sulphur	%M/M	32	0.35	0.05	0.03
Water	mg/kg	29	263.6	326.1	478.9
Calcium	mg/kg	42	797.0	124.0	88.7
Phosphorus	mg/kg	40	1209	232	149
Zinc	mg/kg	46	1344	269	229

Table 3: reproducibilities of results of sample #13062 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 MAY 2013 WITH PREVIOUS PT'S

	May 2013	May 2012	May 2011	May 2010
Number of reporting labs	78	78	96	86
Number of results reported	879	804	985	813
Statistical outliers	29	33	52	45
Percentage outliers	3.3%	4.1%	5.3%	5.5%

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	May 2013	May 2012	May 2011	May 2010
Total Acid Number	--	+/-	--	--
Total Base Number	-	--	++	--
Color	++	++	++	++
Conradson Carbon Residue	++	-	++	++
Ramsbottom Carbon Residue	--	-	n.e.	n.e.
Carbo Residue (Micro method)	+			
Density @ 15 °C	--	-	--	--
Flash Point COC	+	+/-	++	++
Flash Point PMcc	++	++	++	++
Kinematic Viscosity @ 40 °C	-	-	--	--
Kinematic Viscosity @ 100 °C	--	--	--	--
Viscosity Index -20 °C	--	n.e.	n.e.	n.e.
Apparent Viscosity	+	n.e.	n.e.	n.e.
Stabinger Viscosity @ 40 °C	--	--	--	--
Stabinger Viscosity @ 100 °C	--	--	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:

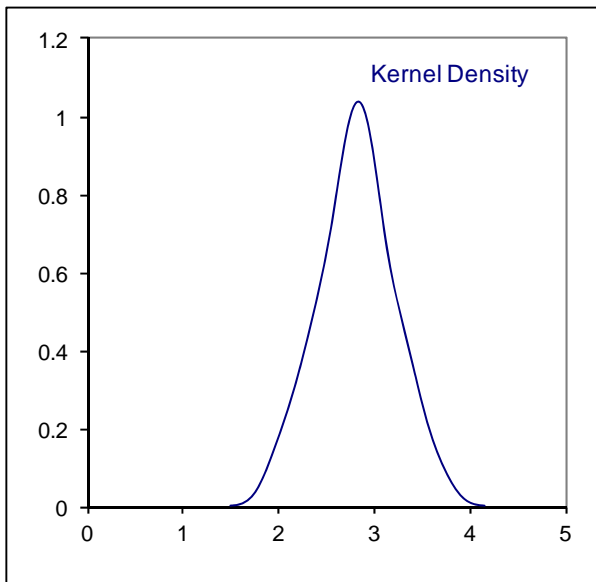
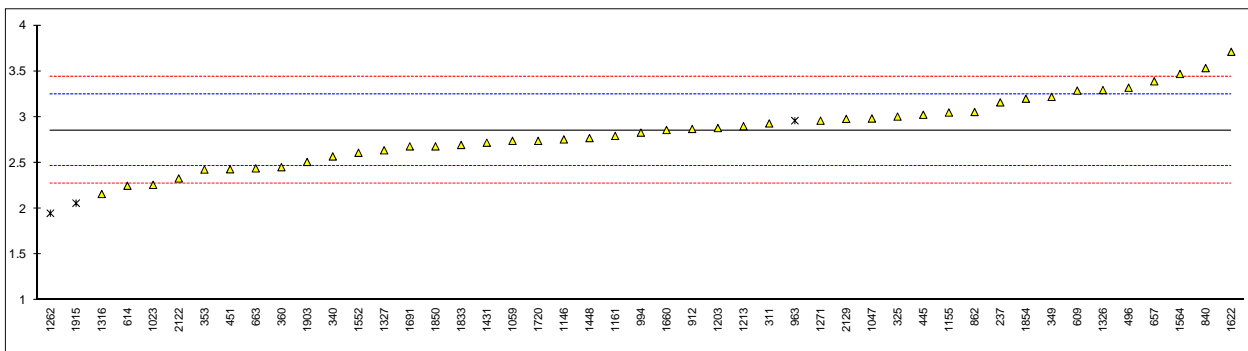
- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- : group performed worse than the standard
- : group performed much worse than the standard
- n.e.: not evaluated

**APPENDIX 1**

Determination of Acid Number (Total) on sample #13062; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
173		----		----	
233		----		----	
237	D664	3.16		1.99	
252		----		----	
254		----		----	
255		----		----	
260		----		----	
311	D664	2.93		0.78	
315		----		----	
325	D664	3.005		1.18	
333		----		----	
337		----		----	
340	D664	2.57		-1.11	
349	D664	3.22		2.31	
353	D664	2.427		-1.86	
360	D664	2.453		-1.72	
396		----		----	
432		----		----	
445	D664	3.025		1.28	
446		----		----	
450		----		----	
451	D664Mod.	2.43		-1.84	
473		----		----	
496	D664	3.320		2.83	
541		----		----	
551		----		----	
603		----		----	
608		----		----	
609	D664	3.2887		2.67	
614	D664	2.25		-2.79	
657	D664	3.39		3.20	
663	D664	2.440		-1.79	
840	D664	3.534		3.95	
862	D664	3.0549		1.44	
875		----		----	
886		----		----	
902		----		----	
912	D664	2.87		0.47	
922		----		----	
963	D974	2.96	ex	0.94	result is excluded, see §4.1
994	D664	2.83		0.26	
1013		----		----	
1017		----		----	
1023	in house	2.26		-2.74	
1047	INH-04049	2.984		1.07	
1059	ISO6619	2.74		-0.22	
1066		----		----	
1106		----		----	
1146	D664	2.756		-0.13	
1155	ISO6619	3.049		1.41	
1161	D664	2.794		0.07	
1173		----		----	
1203	D664	2.88		0.52	
1213	D664	2.90	C	0.63	
1243		----		----	
1244		----		----	
1262	D974	1.95	ex	-4.36	result is excluded, see §4.1
1271	D664	2.96		0.94	
1297		----		----	
1316	D664	2.16		-3.26	
1326	D664	3.2947		2.70	
1327	D664	2.637		-0.76	
1349		----		----	
1402		----		----	
1412		----		----	
1428		----		----	
1431	D664	2.72		-0.32	
1448	D664	2.7708		-0.05	
1488		----		----	
1535		----		----	
1552	D664	2.61		-0.90	
1564	D664	3.47		3.62	

1622	D664	3.7116		4.89	
1660	IEC62021	2.859		0.41	
1691	D664	2.68		-0.53	
1720	D664	2.74		-0.22	
1722		-----		-----	
1833	D664	2.69506		-0.45	
1842		-----		-----	
1850	ISO6619	2.68		-0.53	
1854	D664	3.20		2.20	
1903	INH-5088	2.51		-1.42	
1915	D974	2.06	ex	-3.79	result excluded, see §4.1
2122	IP177	2.33		-2.37	
2129	D664	2.98	C	1.05	result excluded, see §4.1, first reported: 10.61
	normality	OK		OK	
	n	44		7	
	outliers	0		0	
	mean (n)	2.854		3.178	
	st.dev. (n)	0.3615		0.3156	
	R(calc.)	1.012		0.884	
	R(D664:11a)	0.543		0.589	
				<u>D664 results with low intake:</u>	<u>D664 results with high intake:</u>
				OK	OK
				24	24
				0	0
				2.834	2.834
				0.3225	0.3225
				0.903	0.903
				0.541	0.541

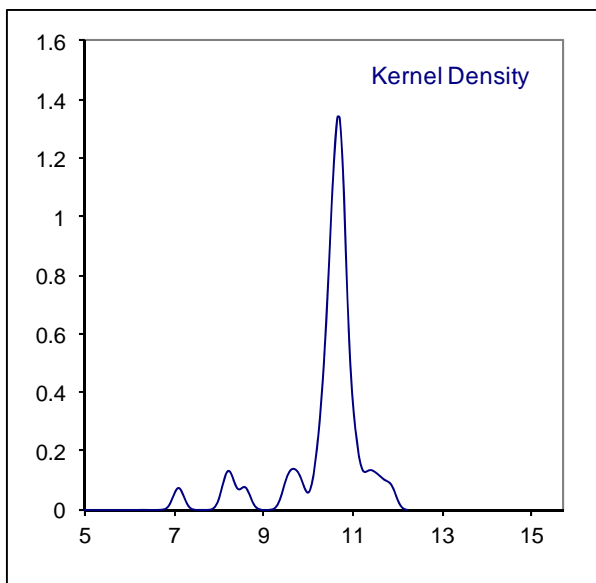
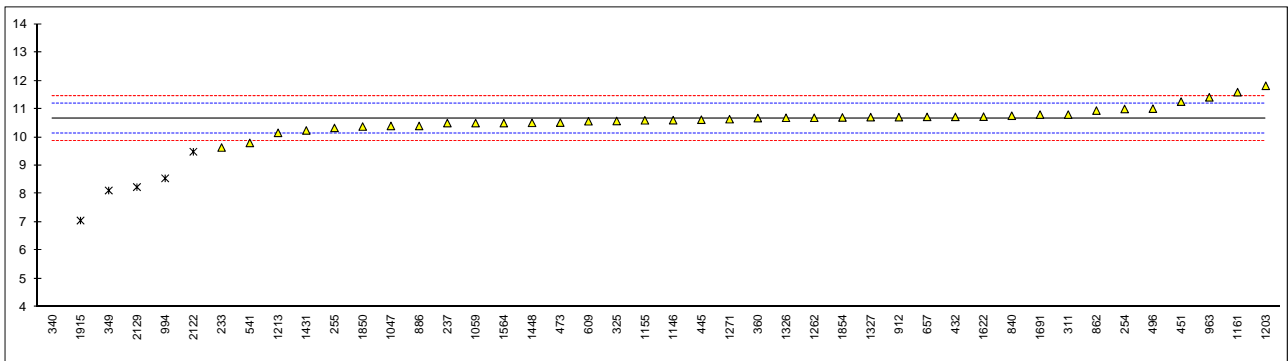


## Determination of Base Number (Total) on sample #13062; results in mg KOH/g

lab	method	value	mark	z(targ)	Remarks
173		----		----	
233	D2896	9.64		-3.84	
237	D2896	10.5		-0.61	
252		----		----	
254	D2896	11.0		1.26	
255	D2896	10.33		-1.25	
260		----		----	
311	D2896	10.8		0.51	
315		----		----	
325	D2896	10.575		-0.33	
333		----		----	
337		----		----	
340	D2896	1.20	G(0.01)	-35.50	
349	D2896	8.12	G(0.05)	-9.54	
353		----		----	
360	D2896	10.677		0.05	
396		----		----	
432	D2896	10.72		0.21	
445	D2896	10.62		-0.16	
446		----		----	
450		----		----	
451	D2896	11.26		2.24	
473	D2896	10.5168		-0.55	
496	D2896	11.01		1.30	
541	D2896	9.8		-3.24	
551		----		----	
603		----		----	
608		----		----	
609	D2896	10.5666		-0.36	
614		----		----	
657	D2896	10.72		0.21	
663		----		----	
840	D2896	10.76		0.36	
862	D2896	10.9433		1.05	
875		----		----	
886	D2896	10.40		-0.99	
902		----		----	
912	D2896	10.71		0.17	
922		----		----	
963	D2896	11.41		2.80	
994	D2896	8.55	G(0.01)	-7.93	
1013		----		----	
1017		----		----	
1023		----		----	
1047	ISO3771	10.40		-0.99	
1059	ISO3771	10.5		-0.61	
1066		----		----	
1106		----		----	
1146	D2896	10.604		-0.22	
1155	ISO3771	10.60		-0.24	
1161	D2896	11.593		3.49	
1173		----		----	
1203	ISO3771	11.82		4.34	
1213	D2896	10.158		-1.90	
1243		----		----	
1244		----		----	
1262	D2896	10.69		0.10	
1271	ISO3771	10.64		-0.09	
1297		----		----	
1316		----		----	
1326	D2896	10.69		0.10	
1327	INH-0251	10.71		0.17	
1349		----		----	
1402		----		----	
1412		----		----	
1428		----		----	
1431	D2896	10.24		-1.59	
1448	D2896	10.5101		-0.58	
1488		----		----	
1535		----		----	
1552		----		----	
1564	D2896	10.5		-0.61	
1622	D2896	10.7274		0.24	
1660		----		----	



1691	D2896	10.8		0.51	
1720		----		----	
1722		----		----	
1833		----		----	
1842		----		----	
1850	ISO3771	10.38		-1.06	
1854	D2896	10.7		0.14	
1903		----		----	
1915	D2896	7.06	G(0.01)	-13.52	
2122	IP400	9.4878	ex	-4.41	result is excluded, as the test method is not equivalent
2129	INH-93	8.24	G(0.01)	-9.09	
normality		not OK			
n		38			
outliers		5			
mean (n)		10.664			
st.dev. (n)		0.4094			
R(calc.)		1.146			
R(D2896:11)		0.746			

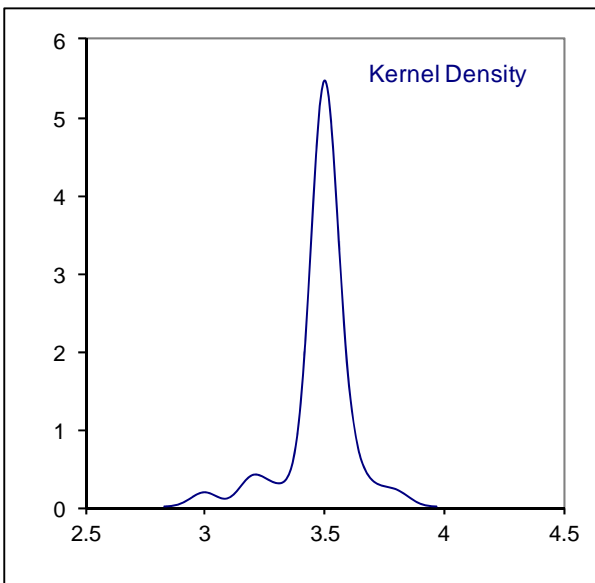
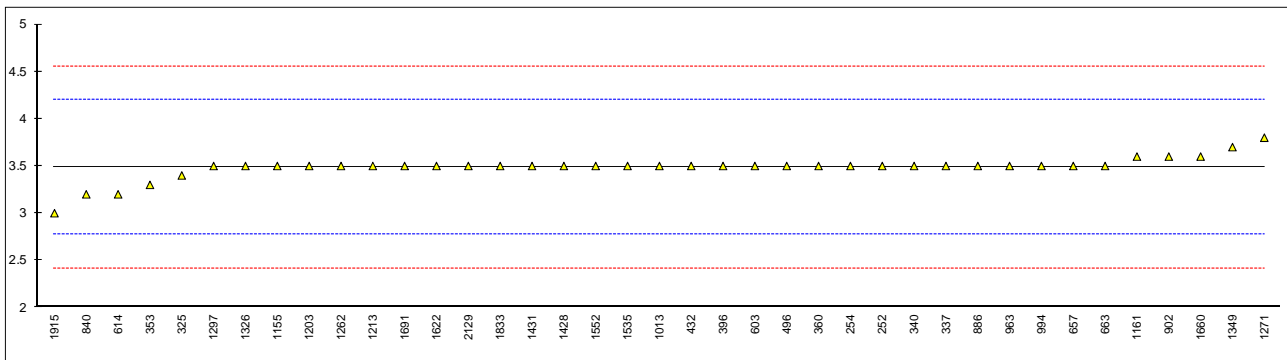


## Determination of Color on sample #13062

lab	method	value	mark	z(targ)	Remarks
173	D1500	L4.0		----	
233				----	
237	D1500	L4.0		----	
252	D1500	3.5		0.04	
254	D1500	3.5		0.04	
255				----	
260				----	
311	D1500	L4.0		----	
315	D1500	L4.0		----	
325	D6045	3.4		-0.24	
333				----	
337	D1500	3.5		0.04	
340	D1500	3.5		0.04	
349				----	
353	D6045	3.3		-0.52	
360	D1500	3.5		0.04	
396	D1500	3.5		0.04	
432	D1500	3.5		0.04	
445	D1500	<3.5		----	
446	D1500	<4.0		----	
450				----	
451				----	
473				----	
496	D1500	3.5		0.04	
541	D1500	L3.5		----	
551				----	
603	D1500	3.5		0.04	
608				----	
609	D1500	L4.0		----	
614	D1500	3.2		-0.80	
657	D1500	3.5		0.04	
663	D1500	3.5		0.04	
840	D1500	3.2		-0.80	
862	D1500	L4.0		----	
875				----	
886	D1500	3.5		0.04	
902	D1500	3.6		0.32	
912	D1500	L4.0		----	
922	D1500	L4.0		----	
963	D1500	3.5		0.04	
994	D1500	3.5		0.04	
1013	D1500	3.5		0.04	
1017				----	
1023				----	
1047				----	
1059	D1500	L3.5		----	
1066				----	
1106				----	
1146				----	
1155	ISO2049	3.5		0.04	
1161	D1500	3.6		0.32	
1173				----	
1203	D1500	3.5		0.04	
1213	D1500	3.5		0.04	
1243				----	
1244				----	
1262	D1500	3.5		0.04	
1271	D1500	3.8		0.88	
1297	D1500	3.5		0.04	
1316				----	
1326	D1500	3.5		0.04	
1327	D1500	<3.5		----	
1349	D1500	3.7		0.60	
1402				----	
1412				----	
1428	D1500	3.5		0.04	
1431	D1500	3.5		0.04	
1448				----	
1488				----	
1535	D1500	3.5		0.04	
1552	D1500	3.50		0.04	
1564	D1500	L3.5		----	
1622	D1500	3.5		0.04	
1660	D1500	3.6		0.32	

1691	D1500	3.5	0.04
1720		----	----
1722		----	----
1833	D1500	3.5	0.04
1842		----	----
1850	ISO2049	L3.5	----
1854	D1500	L3.5	----
1903		----	----
1915	D1500	3.0	-1.36
2122		----	----
2129	D1500	3.5	0.04

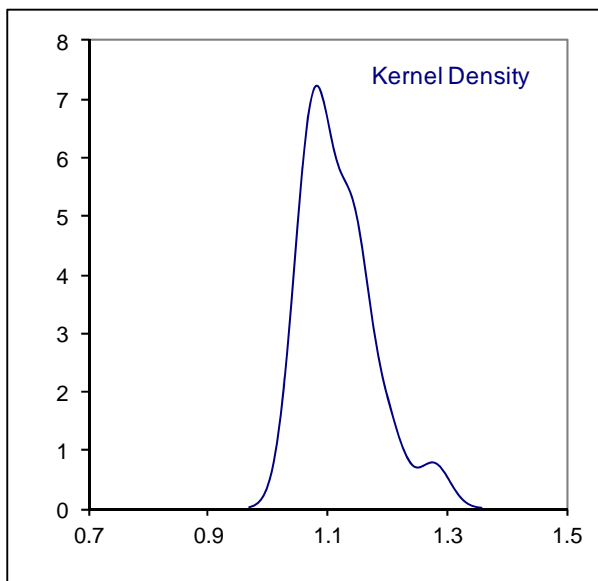
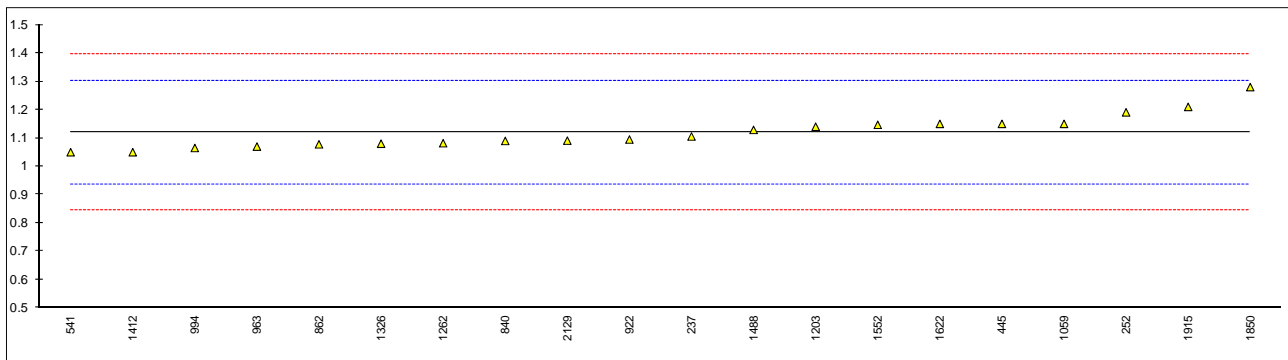
normality not OK  
n 39  
outliers 0  
mean (n) 3.48  
st.dev. (n) 0.129  
R(calc.) 0.36  
R(D1500:07) 1.00



## Determination of Conradson Carbon Residue on sample #13062; results in %M/M

lab	method	value	mark	z(targ)	Remarks
173		----		----	
233		----		----	
237	D189	1.1057		-0.16	
252	D189	1.191		0.77	
254		----		----	
255		----		----	
260		----		----	
311		----		----	
315		----		----	
325		----		----	
333		----		----	
337		----		----	
340		----		----	
349		----		----	
353		----		----	
360		----		----	
396		----		----	
432		----		----	
445	D189	1.15		0.33	
446		----		----	
450		----		----	
451		----		----	
473		----		----	
496		----		----	
541	D189	1.05		-0.77	
551		----		----	
603		----		----	
608		----		----	
609		----		----	
614		----		----	
657		----		----	
663		----		----	
840	D189	1.090		-0.33	
862	D189	1.078		-0.46	
875		----		----	
886		----		----	
902		----		----	
912		----		----	
922	D189	1.095		-0.27	
963	D189	1.07		-0.55	
994	D189	1.065		-0.60	
1013		----		----	
1017		----		----	
1023		----		----	
1047		----		----	
1059	D189	1.15		0.33	
1066		----		----	
1106		----		----	
1146		----		----	
1155		----		----	
1161		----		----	
1173		----		----	
1203	ISO10370	1.14		0.22	
1213		----		----	
1243		----		----	
1244		----		----	
1262	D189	1.082		-0.42	
1271		----		----	
1297		----		----	
1316		----		----	
1326	D189	1.080		-0.44	
1327		----		----	
1349		----		----	
1402		----		----	
1412	D189	1.05		-0.77	
1428		----		----	
1431		----		----	
1448		----		----	
1488	D189	1.129		0.10	
1535		----		----	
1552	D189	1.147		0.29	
1564		----		----	
1622	D189	1.15		0.33	
1660		----		----	

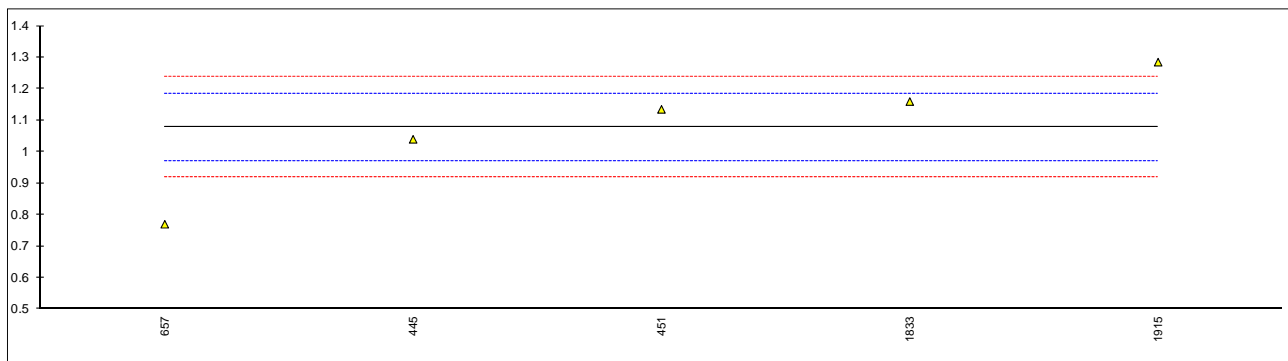
1691		----	----
1720		----	----
1722		----	----
1833		----	----
1842		----	----
1850	ISO6615	1.28	1.74
1854		----	----
1903		----	----
1915	D189	1.210	0.98
2122		----	----
2129	D189	1.091	-0.32
normality		OK	
n		20	
outliers		0	
mean (n)		1.120	
st.dev. (n)		0.0586	
R(calc.)		0.164	
R(D189:10)		0.257	



Determination of Ramsbottom Carbon Residue on sample #13062; results in %M/M

lab	method	value	mark	z(targ)	Remarks
173		----		----	
233		----		----	
237		----		----	
252		----		----	
254		----		----	
255		----		----	
260		----		----	
311		----		----	
315		----		----	
325		----		----	
333		----		----	
337		----		----	
340		----		----	
349		----		----	
353		----		----	
360		----		----	
396		----		----	
432		----		----	
445	D524	1.04		-0.72	
446		----		----	
450		----		----	
451	IP14	1.135		1.07	
473		----		----	
496		----		----	
541		----		----	
551		----		----	
603		----		----	
608		----		----	
609		----		----	
614		----		----	
657	D524	0.77		-5.80	
663		----		----	
840		----		----	
862		----		----	
875		----		----	
886		----		----	
902		----		----	
912		----		----	
922		----		----	
963		----		----	
994		----		----	
1013		----		----	
1017		----		----	
1023		----		----	
1047		----		----	
1059		----		----	
1066		----		----	
1106		----		----	
1146		----		----	
1155		----		----	
1161		----		----	
1173		----		----	
1203		----		----	
1213		----		----	
1243		----		----	
1244		----		----	
1262		----		----	
1271		----		----	
1297		----		----	
1316		----		----	
1326		----		----	
1327		----		----	
1349		----		----	
1402		----		----	
1412		----		----	
1428		----		----	
1431		----		----	
1448		----		----	
1488		----		----	
1535		----		----	
1552		----		----	
1564		----		----	
1622		----		----	
1660		----		----	

1691		----	----
1720		----	----
1722		----	----
1833	D524	1.16	1.54
1842		----	----
1850		----	----
1854		----	----
1903		----	----
1915	D524	1.285	3.90
2122		----	----
2129		----	----
normality		unknown	
n		5	
outliers		0	
mean (n)		1.078	
st.dev. (n)		0.1931	
R(calc.)		0.541	
R(D524:10)		0.149	

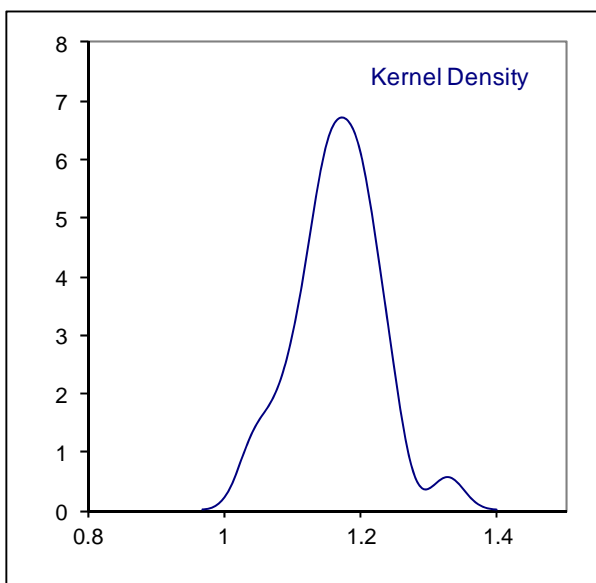
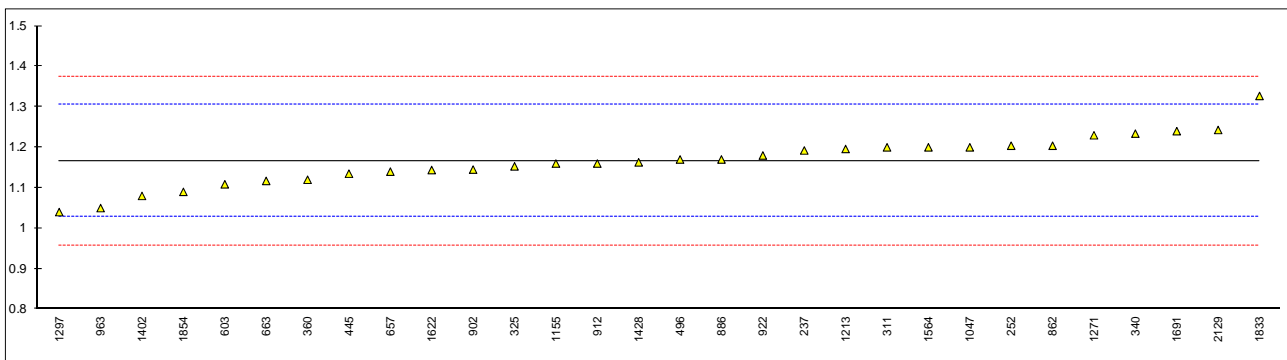


## Determination of Carbon Residue (micro method) on sample #13062; results in %M/M

lab	method	value	mark	z(targ)	Remarks
173		----		----	
233		----		----	
237	D4530	1.1924		0.37	
252	D4530	1.204		0.54	
254		----		----	
255		----		----	
260		----		----	
311	D4530	1.2		0.48	
315		----		----	
325	D4530	1.15305		-0.19	
333		----		----	
337		----		----	
340	ISO10370	1.234		0.97	
349		----		----	
353		----		----	
360	D4530	1.120		-0.67	
396		----		----	
432		----		----	
445	D4530	1.135		-0.45	
446		----		----	
450		----		----	
451		----		----	
473		----		----	
496	D4530	1.170		0.05	
541		----		----	
551		----		----	
603	D4530	1.10865		-0.83	
608		----		----	
609		----		----	
614		----		----	
657	D4530	1.14		-0.38	
663	D4530	1.117		-0.71	
840		----		----	
862	D4530	1.204		0.54	
875		----		----	
886	D4530	1.17		0.05	
902	D4530	1.145		-0.31	
912	D4530	1.16		-0.09	
922	D4530	1.18		0.19	
963	D4530	1.05		-1.67	
994		----		----	
1013		----		----	
1017		----		----	
1023		----		----	
1047	ISO10370	1.20		0.48	
1059		----		----	
1066		----		----	
1106		----		----	
1146		----		----	
1155	D4530	1.16	C	-0.09	first reported: 11.16
1161		----		----	
1173		----		----	
1203		----		----	
1213	D4530	1.196		0.42	
1243		----		----	
1244		----		----	
1262		----		----	
1271	D4530	1.23		0.91	
1297	D4530	1.04		-1.82	
1316		----		----	
1326		----		----	
1327		----		----	
1349		----		----	
1402	D4530	1.08		-1.24	
1412		----		----	
1428	D4530	1.163		-0.05	
1431		----		----	
1448		----		----	
1488		----		----	
1535		----		----	
1552		----		----	
1564	D4530	1.2		0.48	
1622	D4530	1.1440		-0.32	
1660		----		----	



1691	D4530	1.24	1.05
1720		----	----
1722		----	----
1833	D4530	1.327	2.30
1842		----	----
1850		----	----
1854	D4530	1.09	-1.10
1903		----	----
1915		----	----
2122		----	----
2129	IP398	1.243	1.10
normality		OK	
n		30	
outliers		0	
mean (n)		1.167	
st.dev. (n)		0.0608	
R(calc.)		0.170	
R(D4530:11)		0.195	

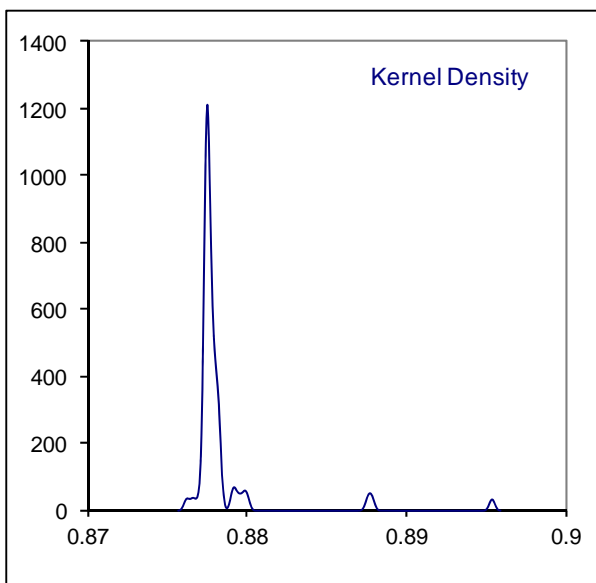
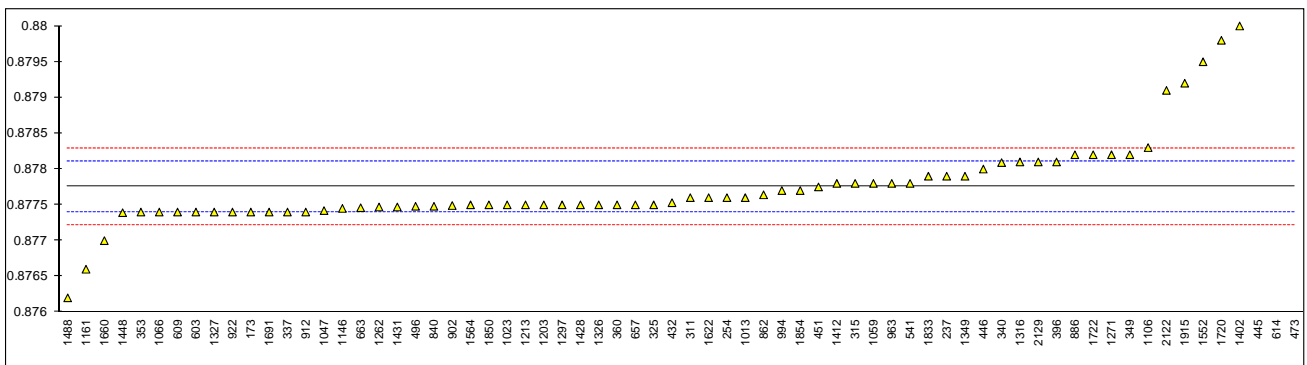


## Determination of Density @ 15°C on sample #13062; results in kg/L

lab	method	value	mark	z(targ)	remarks
173	D4052	0.8774		-1.98	
233		-----		-----	
237	D4052	0.8779		0.82	
252		-----		-----	
254	D4052	0.8776		-0.86	
255		-----		-----	
260		-----		-----	
311	D4052	0.8776		-0.86	
315	D4052	0.8778		0.26	
325	D4052	0.8775		-1.42	
333		-----		-----	
337	D4052	0.8774		-1.98	
340	D4052	0.87809		1.89	
349	D4052	0.8782		2.50	
353	IP365	0.8774	C	-1.98	first reported: 877.4 kg/L
360	D4052	0.8775		-1.42	
396	D4052	0.8781		1.94	
432	D4052	0.87753		-1.25	
445	D4052	0.8876	G(0.01)	55.14	
446	D4052	0.8780		1.38	
450		-----		-----	
451	D4052	0.87775		-0.02	
473	D4052	0.8954	G(0.01)	98.82	
496	D4052	0.87748		-1.53	
541	D4052	0.8778		0.26	
551		-----		-----	
603	D4052	0.8774		-1.98	
608		-----		-----	
609	D4052	0.8774	C	-1.98	first reported: 877.41 kg/L
614	D4052	0.88785	G(0.01)	56.54	
657	D4052	0.8775		-1.42	
663	D4052	0.87746		-1.64	
840	D4052	0.87748		-1.53	
862	D4052	0.87764		-0.63	
875		-----		-----	
886	D4052	0.8782		2.50	
902	D4052	0.87749		-1.47	
912	D4052	0.8774		-1.98	
922	D4052	0.8774		-1.98	
963	D4052	0.8778		0.26	
994	D4052	0.8777		-0.30	
1013	D4052	0.8776		-0.86	
1017		-----		-----	
1023	D4052	0.8775		-1.42	
1047	D4052	0.87742		-1.86	
1059	D4052	0.8778		0.26	
1066	D4052	0.8774		-1.98	
1106	D5002	0.8783		3.06	
1146	D4052	0.87745		-1.70	
1155		-----		-----	
1161	ISO3675	0.8766	C	-6.46	probably unit error, reported 876.6 kg/L
1173		-----		-----	
1203	ISO12185	0.87750		-1.42	
1213	D4052	0.8775		-1.42	
1243		-----		-----	
1244		-----		-----	
1262	D4052	0.87747	C	-1.58	first reported: 877.47 kg/L
1271	D4052	0.8782	C	2.50	first reported: 878.2 kg/L
1297	D4052	0.8775	C	-1.42	probably unit error, reported 877.5 kg/L
1316	D4052	0.8781		1.94	
1326	D4052	0.8775		-1.42	
1327	D4052	0.8774	C	-1.98	first reported: 877.4 kg/L
1349	D4052	0.8779		0.82	
1402	D4052	0.8800		12.58	
1412	D4052	0.8778		0.26	
1428	D4052	0.8775		-1.42	
1431	D4052	0.87747		-1.58	
1448	D4052	0.87739		-2.03	
1488	D1298	0.87620		-8.70	
1535		-----		-----	
1552	D4052	0.87950	C	9.78	probably unit error, reported 879.50 kg/L
1564	D4052	0.8775	C	-1.42	first reported: 877.5 kg/L
1622	D4052	0.8776		-0.86	
1660	D7042	0.8770		-4.22	

1691	D4052	0.8774	C	-1.98	first reported: 877.4 kg/L
1720	D4052	0.8798		11.46	
1722	D4052	0.8782		2.50	
1833	D4052	0.8779		0.82	
1842		----		----	
1850	D4052	0.8775		-1.42	
1854	D4052	0.8777		-0.30	
1903		----		----	
1915	D4052	0.8792		8.10	
2122	in house	0.8791		7.54	
2129	D4052	0.8781	C	1.94	first reported: 878.1 kg/L

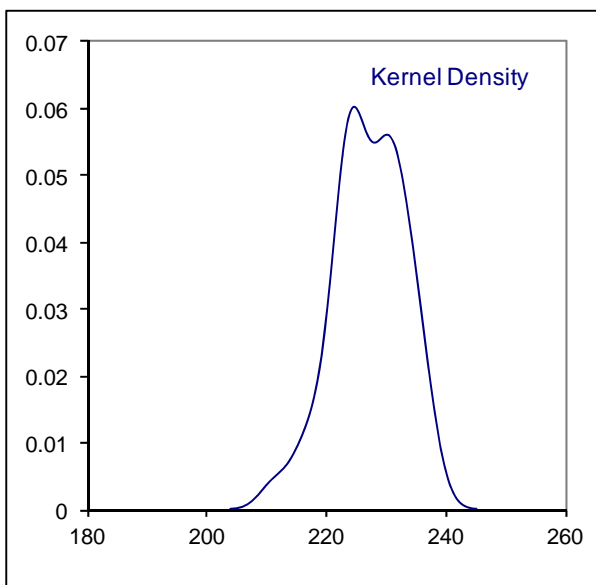
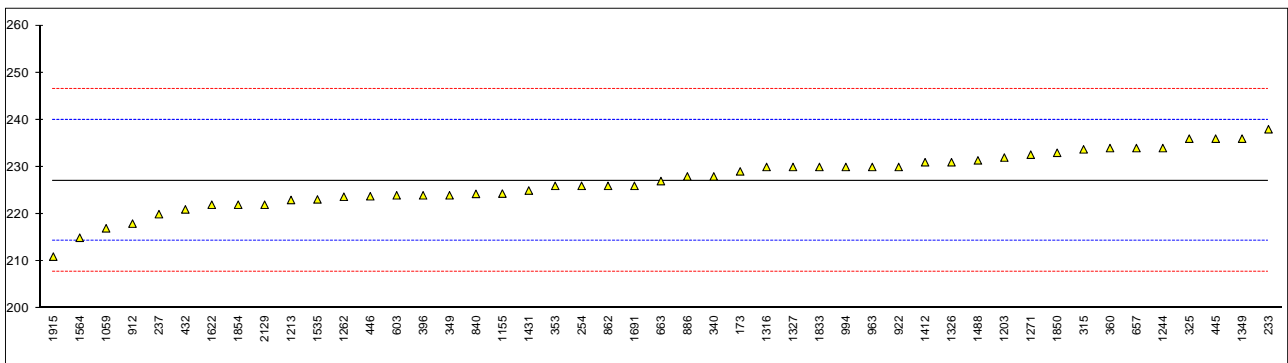
normality not OK  
 n 65  
 outliers 3  
 mean (n) 0.87775  
 st.dev. (n) 0.000627  
 R(calc.) 0.00175  
 R(D4052:11) 0.00050



## Determination of Flash Point C.O.C. on sample #13062; results in °C

lab	method	value	mark	z(targ)	remarks
173	D92	229.06		0.30	
233	D92	238		1.69	
237	D92	220.0		-1.11	
252		-----		-----	
254	D92	226.0		-0.18	
255		-----		-----	
260		-----		-----	
311		-----		-----	
315	D92	233.736		1.02	
325	D92	236.0		1.38	
333		-----		-----	
337		-----		-----	
340	D92	228		0.13	
349	D92	224		-0.49	
353	IP36	226.0		-0.18	
360	D92	234.0		1.07	
396	D92	224		-0.49	
432	D92	221		-0.96	
445	D92	236		1.38	
446	D92	223.8		-0.52	
450		-----		-----	
451		-----		-----	
473		-----		-----	
496		-----		-----	
541		-----		-----	
551		-----		-----	
603	D92	224.0		-0.49	
608		-----		-----	
609		-----		-----	
614		-----		-----	
657	D92	234		1.07	
663	D92	227.0		-0.02	
840	D92	224.3		-0.44	
862	D92	226		-0.18	
875		-----		-----	
886	D92	228.0		0.13	
902		-----		-----	
912	D92	218		-1.42	
922	D92	230		0.44	
963	D92	230		0.44	
994	D92	230.0		0.44	
1013		-----		-----	
1017		-----		-----	
1023		-----		-----	
1047		-----		-----	
1059	ISO2592	217		-1.58	
1066		-----		-----	
1106		-----		-----	
1146		-----		-----	
1155	ISO2592	224.35		-0.44	
1161		-----		-----	
1173		-----		-----	
1203	ISO2592	232		0.75	
1213	D92	223		-0.65	
1243		-----		-----	
1244	D92	234		1.07	
1262	D92	223.7		-0.54	
1271	D92	232.6		0.85	
1297		-----		-----	
1316	D92	230		0.44	
1326	D92	231		0.60	
1327	D92	230.0		0.44	
1349	D92	236		1.38	
1402		-----		-----	
1412	D92	231.0		0.60	
1428		-----	W	-----	result withdrawn, first reported:202
1431	D92	225		-0.33	
1448		-----		-----	
1488	D92	231.4		0.66	
1535	ISO2592	223.125		-0.63	
1552		-----		-----	
1564	D92	215		-1.89	
1622	D92	222		-0.80	
1660		-----		-----	

1691	D92	226	-0.18
1720		-----	-----
1722		-----	-----
1833	D92	230	0.44
1842		-----	-----
1850	ISO2592	233	0.91
1854	D92	222	-0.80
1903		-----	-----
1915	D92	211	-2.51
2122		-----	-----
2129	D92	222.0	-0.80
normality		OK	
n		47	
outliers		0	
mean (n)		227.15	
st.dev. (n)		5.901	
R(calc.)		16.52	
R(D92:12)		18.00	



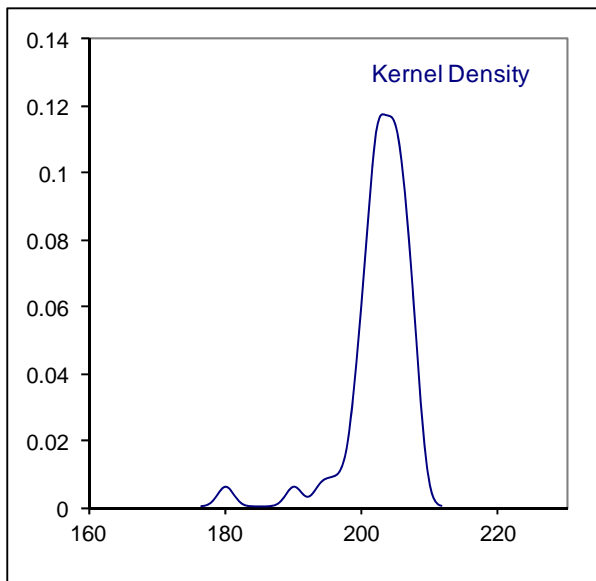
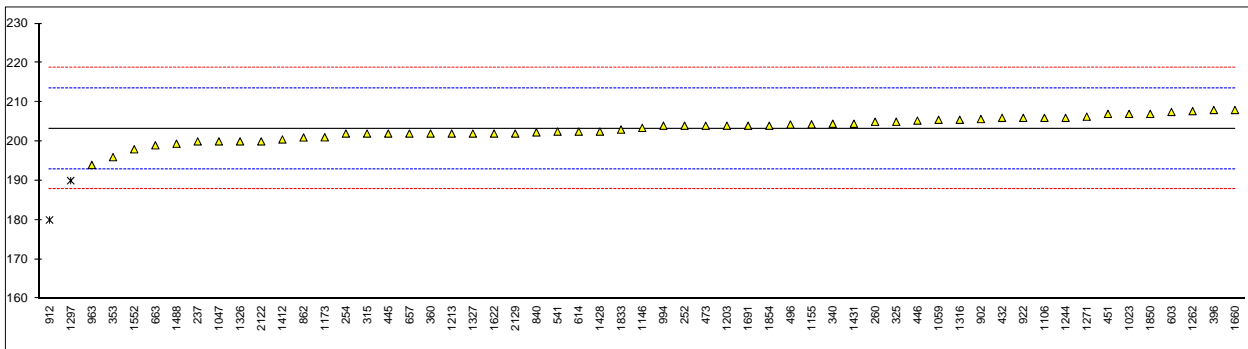
Determination of Flash Point PMcc on sample #13062; results in °C

lab	method	value	mark	z(targ)	Analysis run time	remarks
173		----		----	----	
233		----		----	----	
237	D93A	200.0		-0.63	----	
252	D93A	204		0.14	25	
254	D93A	202.0		-0.24	----	
255		----		----	----	
260	D93	205.0		0.34	----	
311		----		----	----	
315	D93A	202.0		-0.24	39.5	
325	D93	205.0		0.34	----	
333		----		----	----	
337		----		----	----	
340	D93A	204.5		0.24	16	
349		----		----	----	
353	IP34A	196.0		-1.41	30	
360	D93A	202.0		-0.24	36	
396	D93A	208		0.92	----	
432	D93A	206.0		0.53	38	
445	D93A	202.0		-0.24	69	
446	D93A	205.3		0.40	----	
450		----		----	----	
451	IP34B	207		0.73	42	run time does not match with procedure B
473	D93B	204.0		0.14	----	
496	D93A	204.3		0.20	----	
541	D7094	202.5		-0.15	----	
551		----		----	----	
603	D93A	207.5		0.82	----	
608		----		----	----	
609		----		----	----	
614	D93B	202.5		-0.15	47	run time does not match with procedure B
657	D93A	202		-0.24	----	
663	D93A	199.0		-0.83	----	
840	D93A	202.3		-0.19	29	
862	D93A	201.0		-0.44	----	
875		----		----	----	
886		----		----	----	
902	D93A	205.7		0.47	38 min and 45 sec	
912	D93B	180	G(0.01)	-4.51	119	
922	D93A	206		0.53	35	
963	D93A	194.0		-1.80	----	
994	D93B	204.0		0.14	34	run time does not match with procedure B
1013		----		----	----	
1017		----		----	----	
1023	D93A	207		0.73	----	
1047	ISO2719	200.0		-0.63	52	
1059	ISO2719A	205.5		0.43	35	
1066		----		----	----	
1106	D93A	206.0		0.53	----	
1146	in house	203.45		0.04	39 min and 5 sec	
1155	D93A	204.35		0.21	35	
1161		----		----	----	
1173	IP34A	201.05		-0.43	----	
1203	ISO2719	204		0.14	----	
1213	D93A	202		-0.24	33	
1243		----		----	----	
1244	D93A	206		0.53	----	
1262	D93A	207.7		0.86	180.0	run time does not match with procedure A
1271	D93A	206.3		0.59	29	
1297	D93B	190	G(0.01)	-2.57	140	
1316	D93A	205.5		0.43	----	
1326	D93A	200.0		-0.63	----	
1327	D93A	202.0		-0.24	----	
1349		----		----	----	
1402		----		----	----	
1412	D93	200.5		-0.54	----	
1428	D93A	202.5		-0.15	----	
1431	D93A	204.5		0.24	----	
1448		----		----	----	
1488	D93A	199.41		-0.75	31.5	
1535		----		----	----	
1552	D93A	198.00		-1.02	30	
1564		----		----	----	
1622	D93A	202.0		-0.24	----	
1660	D93	208		0.92	18	

1691	D93A	204.0	0.14	----
1720		----	----	----
1722		----	----	----
1833	D93	203	-0.05	15
1842		----	----	----
1850	ISO2719A	207	0.73	----
1854	D93B	204	0.14	----
1903		----	----	----
1915	D93	>110	----	----
2122	D93	200	-0.63	----
2129	D93A	202.0	-0.24	36

only D93:A/ISO2719A/IP34A data:

normality	OK	OK
n	55	41
outliers	2	0
mean (n)	203.26	202.99
st.dev. (n)	2.966	3.117
R(calc.)	8.30	8.73
R(D93-A:12)	14.43	14.41

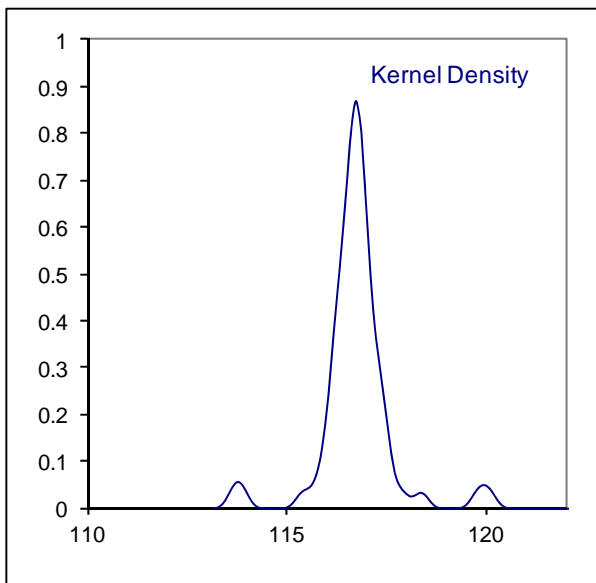
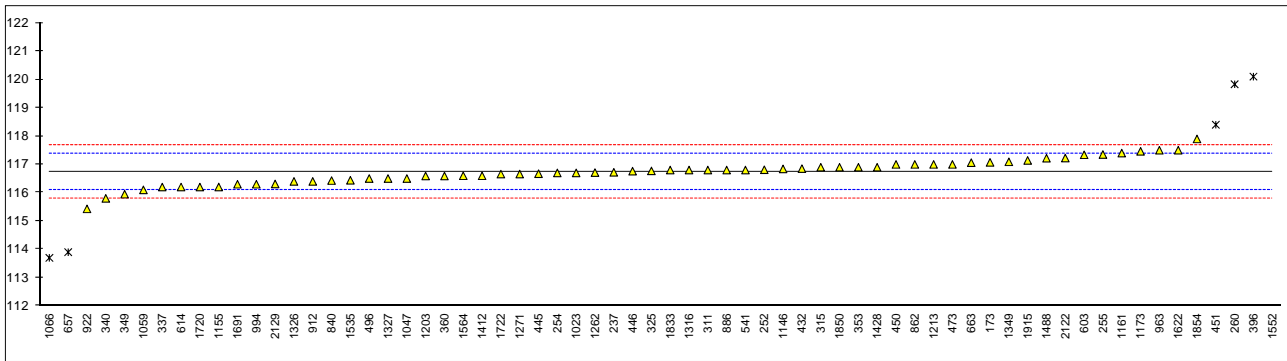


Determination of Kinematic Viscosity @ 40°C on sample #13062; results in mm<sup>2</sup>/s

lab	method	value	mark	z(targ)	remarks
173	D445	117.07		1.05	
233		-----		-----	
237	D445	116.72		-0.05	
252	D445	116.81		0.23	
254	D445	116.7		-0.12	
255	D7279	117.35		1.93	
260	D445	119.83	G(0.01)	9.76	
311	D445	116.8		0.20	
315	D445	116.9		0.51	
325	D445	116.77		0.10	
333		-----		-----	
337	D445	116.2		-1.70	
340	D445	115.80		-2.96	
349	D445	115.95		-2.48	
353	IP71	116.9		0.51	
360	D445	116.59		-0.47	
396	D445	120.1	G(0.01)	10.61	
432	D445	116.85		0.36	
445	D445	116.67		-0.21	
446	D445	116.76		0.07	
450	D445	117.0		0.83	
451	D7279	118.40	G(0.05)	5.25	
473	D445	117.00		0.83	
496	D445	116.50		-0.75	
541	D445	116.8		0.20	
551		-----		-----	
603	D445	117.34		1.90	
608		-----		-----	
609		-----		-----	
614	D445	116.2		-1.70	
657	D445	113.9	G(0.01)	-8.95	
663	D445	117.06		1.02	
840	D445	116.43		-0.97	
862	D445	117.0		0.83	
875		-----		-----	
886	D445	116.8		0.20	
902		-----		-----	
912	D445	116.4		-1.06	
922	D445	115.43		-4.13	
963	D445	117.5		2.41	
994	D445	116.3		-1.38	
1013		-----		-----	
1017		-----		-----	
1023	D445	116.7		-0.12	
1047	D445	116.5		-0.75	
1059	ISO3104	116.1		-2.01	
1066	D445	113.7	G(0.05)	-9.59	
1106		-----		-----	
1146	D445	116.84		0.32	
1155	ISO3104	116.201		-1.69	
1161	ISO3104	117.4		2.09	
1173	IP71	117.46		2.28	
1203	ISO3104	116.590		-0.47	
1213	D445	117.0		0.83	
1243		-----		-----	
1244		-----		-----	
1262	D445	116.71		-0.09	
1271	D445	116.66		-0.24	
1297		-----		-----	
1316	D445	116.8		0.20	
1326	D445	116.4		-1.06	
1327	D445	116.5		-0.75	
1349	D445	117.0918		1.12	
1402		-----		-----	
1412	D445	116.6		-0.43	
1428	D445	116.9		0.51	
1431		-----		-----	
1448		-----		-----	
1488	D445	117.2188		1.52	
1535	ISO3104	116.44		-0.94	
1552	D445	144.29	G(0.01)	86.96	
1564	D445	116.6		-0.43	
1622	D445	117.5		2.41	
1660		-----		-----	



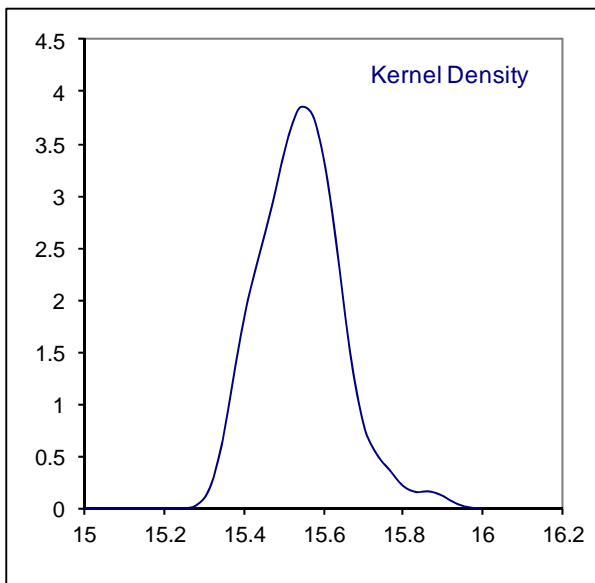
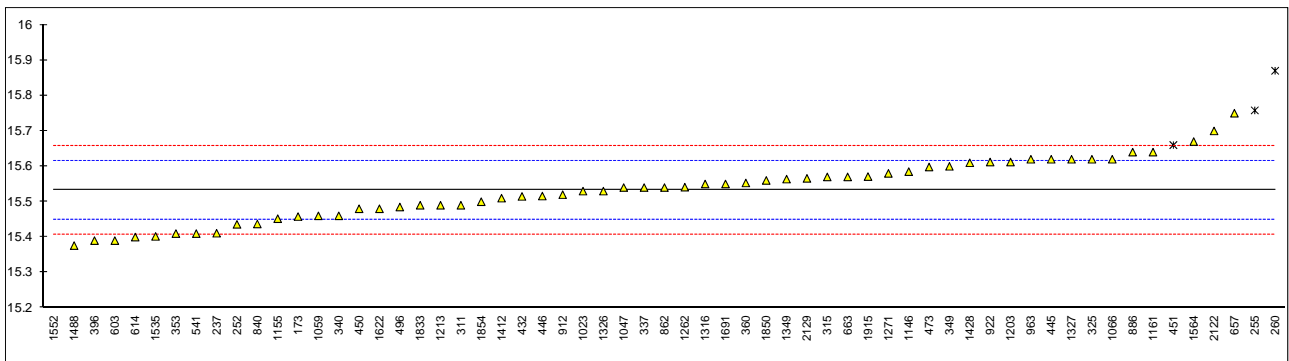
1691	D445	116.3	-1.38
1720	D445	116.2	-1.70
1722	D445	116.6571	-0.25
1833	D445	116.8	0.20
1842		-----	-----
1850	ISO3104	116.9	0.51
1854	D445	117.9	3.67
1903		-----	-----
1915	D445	117.14	1.27
2122	in house	117.225	1.54
2129	D445	116.31	-1.35
normality		OK	
n		60	
outliers		6	
mean (n)		116.737	
st.dev. (n)		0.4440	
R(calc.)		1.243	
R(D445:12)		0.887	



Determination of Kinematic Viscosity @ 100°C on sample #13062; results in mm<sup>2</sup>/s

lab	method	value	mark	z(targ)	remarks
173	D445	15.458		-1.76	
233		-----		-----	
237	D445	15.411		-2.88	
252	D445	15.436		-2.28	
254		-----		-----	
255	D7279	15.758	ex	5.35	result excluded, see §4.1
260	D445	15.87	G(0.05)	8.01	
311	D445	15.49		-1.00	
315	D445	15.57		0.89	
325	D445	15.62		2.08	
333		-----		-----	
337	D445	15.54		0.18	
340	D445	15.460		-1.71	
349	D445	15.60		1.61	
353	IP71	15.41		-2.90	
360	D445	15.553		0.49	
396	D445	15.39		-3.37	
432	D445	15.515		-0.41	
445	D445	15.62		2.08	
446	D445	15.516		-0.39	
450	D445	15.48		-1.24	
451	D7279	15.66	ex	3.03	result excluded, see §4.1
473	D445	15.598		1.56	
496	D445	15.485		-1.12	
541	D445	15.41		-2.90	
551		-----		-----	
603	D445	15.39		-3.37	
608		-----		-----	
609		-----		-----	
614	D445	15.40	C	-3.14	first reported: 15.90
657	D445	15.75		5.16	
663	D445	15.570		0.89	
840	D445	15.437		-2.26	
862	D445	15.54		0.18	
875		-----		-----	
886	D445	15.64		2.56	
902		-----		-----	
912	D445	15.52		-0.29	
922	D445	15.612		1.89	
963	D445	15.62		2.08	
994		-----		-----	
1013		-----		-----	
1017		-----		-----	
1023	D445	15.53		-0.05	
1047	D445	15.54		0.18	
1059	ISO3104	15.46		-1.71	
1066	D445	15.62		2.08	
1106		-----		-----	
1146	D445	15.585		1.25	
1155	ISO3104	15.452		-1.90	
1161	ISO3104	15.64		2.56	
1173		-----		-----	
1203	ISO3104	15.612		1.89	
1213	D445	15.49		-1.00	
1243		-----		-----	
1244		-----		-----	
1262	D445	15.541		0.21	
1271	D445	15.58		1.13	
1297		-----		-----	
1316	D445	15.55		0.42	
1326	D445	15.53		-0.05	
1327	D445	15.62		2.08	
1349	D445	15.5638		0.75	
1402		-----		-----	
1412	D445	15.51		-0.53	
1428	D445	15.61		1.84	
1431		-----		-----	
1448		-----		-----	
1488	D445	15.37613		-3.70	
1535	ISO3104	15.402		-3.09	
1552	D445	7.8447	G(0.01)	-182.35	
1564	D445	15.67		3.27	
1622	D445	15.48		-1.24	
1660		-----		-----	

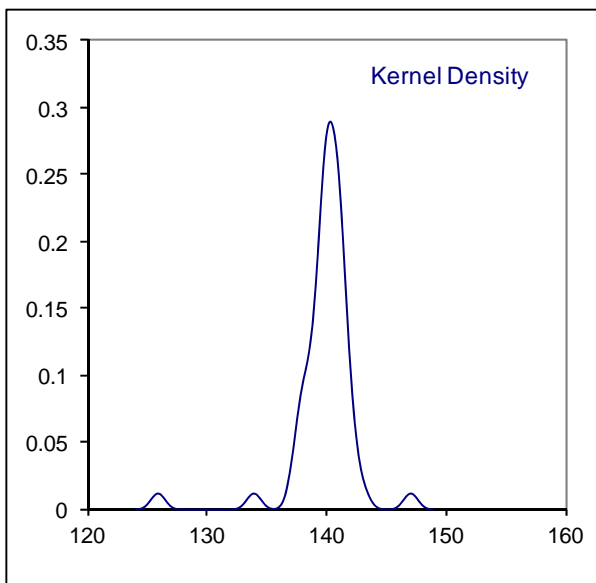
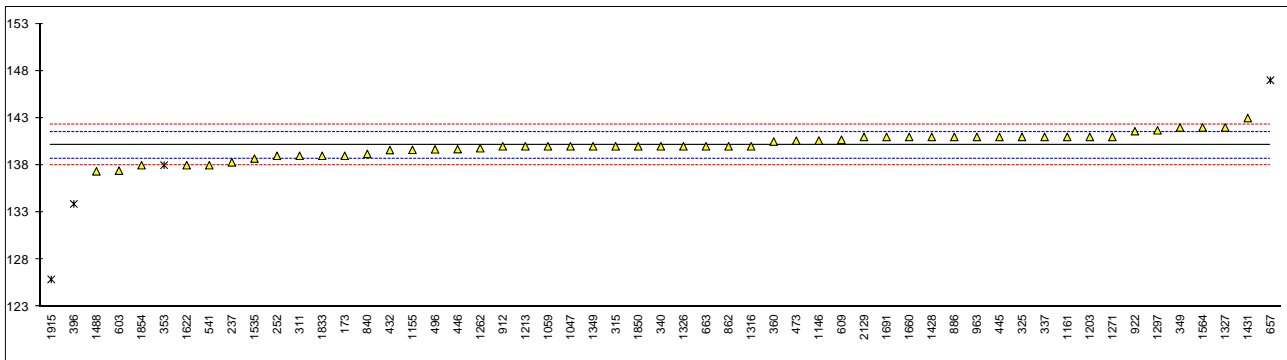
1691	D445	15.55	0.42
1720		-----	-----
1722		-----	-----
1833	D445	15.49	-1.00
1842		-----	-----
1850	ISO3104	15.56	0.66
1854	D445	15.50	-0.77
1903		-----	-----
1915	D445	15.571	0.92
2122	in house	15.7	3.98
2129	D445	15.566	0.80
normality		OK	
n		57	
outliers		2	
mean (n)		15.532	
st.dev. (n)		0.0842	
R(calc.)		0.236	
R(D445:12)		0.118	



## Determination of Viscosity Index on sample #13062

lab	method	Value	mark	z(targ)	VI calculated by iis	Remarks
173	D2270	139		-1.57	139	
233		----		----	----	
237	D2270	138.3		-2.55	138.4	
252	D2270	139		-1.57	139	
254		----		----	----	
255		----		----	142	
260		----		----	139	
311	D2270	139		-1.57	139	
315	D2270	140		-0.17	140	
325	D2270	141		1.23	141	
333		----		----	----	
337	D2270	141		1.23	141	
340	D2270	140		-0.17	140	
349	D2270	142		2.63	142	
353	D2270	138	ex	-2.97	<b>141</b>	see §4.1
360	D2270	140.5		0.53	140.5	
396	D2270	133.9	G(0.01)	-8.71	133.9	
432	D2270	139.6		-0.73	139.7	
445	D2270	141		1.23	141	
446	D2270	139.7		-0.59	139.8	
450		----		----	139	
451		----		----	137	
473	D2270	140.6		0.67	140.6	
496	D2270	139.68		-0.62	139.71	
541	D2270	138		-2.97	138	
551		----		----	----	
603	D2270	137.42		-3.78	137.35	
608		----		----	----	
609	D2270	140.7	C	0.81	----	first reported: 126.2
614		----		----	139	
657	D2270	147	C,G(0.01)	9.63	147	revised result 138 not used
663	D2270	140		-0.17	140	
840	D2270	139.2		-1.29	139.2	
862	D2270	140		-0.17	140	
875		----		----	----	
886	D2270	141		1.23	141	
902		----		----	----	
912	D2270	140		-0.17	140	
922	D2270	141.6		2.07	142.8	
963	D2270	141	C	1.23	140	first reported: 130
994		----		----	----	
1013		----		----	----	
1017		----		----	----	
1023		----		----	140	
1047	D2270	140		-0.17	140	
1059	ISO2909	140		-0.17	140	
1066		----		----	145	
1106		----		----	----	
1146	D2270	140.61		0.69	140.61	
1155	ISO2909	139.63		-0.69	139.65	
1161	D2270	141		1.23	141	
1173		----		----	----	
1203	D2270	141		1.23	141	
1213	D2270	140		-0.17	139	
1243		----		----	----	
1244		----		----	----	
1262	D2270	139.8		-0.45	140.2	
1271	D2270	141		1.23	140	
1297	D2270	141.7		2.21	----	
1316	D2270	140		-0.17	140	
1326	D2270	140		-0.17	140	
1327	D2270	142		2.63	142	
1349	D2270	140.0		-0.17	140.0	
1402		----		----	----	
1412		----		----	140	
1428	D2270	141		1.23	141	
1431	D2270	143		4.03	----	
1448		----		----	----	
1488	D2270	137.35	C	-3.88	137.32	revised result 136.54 not used
1535	ISO2909	138.7		-1.99	138.7	
1552		----		----	-5	
1564	D2270	142		2.63	142	
1622	D2270	138		-2.97	138	
1660	D2270	141		1.23	----	

1691	D2270	141		1.23	141	
1720		----		----	----	
1722		----		----	----	
1833	D2270	139		-1.57	139	
1842		----		----	----	
1850	ISO2909	140		-0.17	140	
1854	D2270	138		-2.97	138	
1903		----		----	----	
1915	D2270	125.9	ex	-19.91	<u>140</u>	see §4.1
2122		----		----	142	
2129	D2270	141		1.23	141	
normality		not OK				
n		51				
outliers		2				
mean (n)		140.12				
st.dev. (n)		1.222				
R(calc.)		3.42				
R(D2270:10)		2.00				



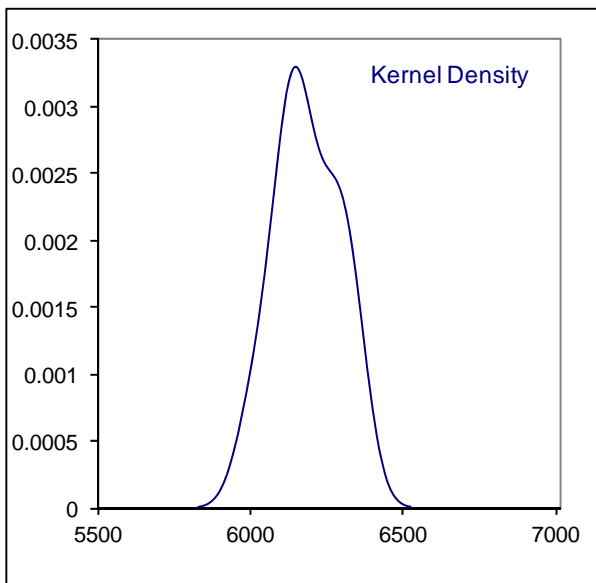
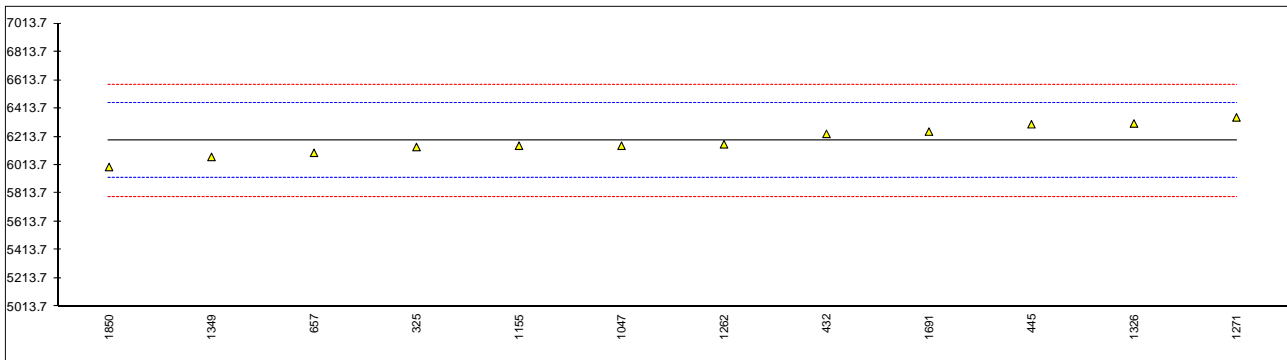
Determination of Apparent viscosity (CCS) @ -20°C on sample #13062; results in mPa·s

lab	method	value	mark	z(targ)	Remarks
173		----		----	
233		----		----	
237		----		----	
252		----		----	
254		----		----	
255		----		----	
260		----		----	
311		----		----	
315		----		----	
325	D5293	6141		-0.33	
333		----		----	
337		----		----	
340		----		----	
349		----		----	
353		----		----	
360		----		----	
396		----		----	
432	D5293	6234		0.37	
445	D5293	6302		0.89	
446		----		----	
450		----		----	
451		----		----	
473		----		----	
496		----		----	
541		----		----	
551		----		----	
603		----		----	
608		----		----	
609		----		----	
614		----		----	
657	D5293	6100		-0.64	
663		----		----	
840		----		----	
862		----		----	
875		----		----	
886		----		----	
902		----		----	
912		----		----	
922		----		----	
963		----		----	
994		----		----	
1013		----		----	
1017		----		----	
1023		----		----	
1047	INH-4150	6150		-0.26	
1059		----		----	
1066		----		----	
1106		----		----	
1146		----		----	
1155	D5293	6150		-0.26	
1161		----		----	
1173		----		----	
1203		----		----	
1213		----		----	
1243		----		----	
1244		----		----	
1262	D5293	6160		-0.19	
1271	D5293	6350		1.25	
1297		----		----	
1316		----		----	
1326	D5293	6307		0.92	
1327		----		----	
1349	D5293	6071		-0.86	
1402		----		----	
1412		----		----	
1428		----		----	
1431		----		----	
1448		----		----	
1488		----		----	
1535		----		----	
1552		----		----	
1564		----	W	----	result withdrawn, first reported: 25575
1622		----		----	
1660		----		----	

1691	D5293	6250	0.49
1720		-----	-----
1722		-----	-----
1833		-----	-----
1842		-----	-----
1850	D5293	6000	-1.39
1854		-----	-----
1903		-----	-----
1915		-----	-----
2122		-----	-----
2129		-----	-----

normality OK  
n 12  
outliers 0  
mean (n) 6184.6  
st.dev. (n) 105.26  
R(calc.) 294.7  
R(D5293:10) 371.1

R(D5293:10) constant cooling instrument = 451.5  
R(D5293:10) thermo-electrically cooled instruments =371.1

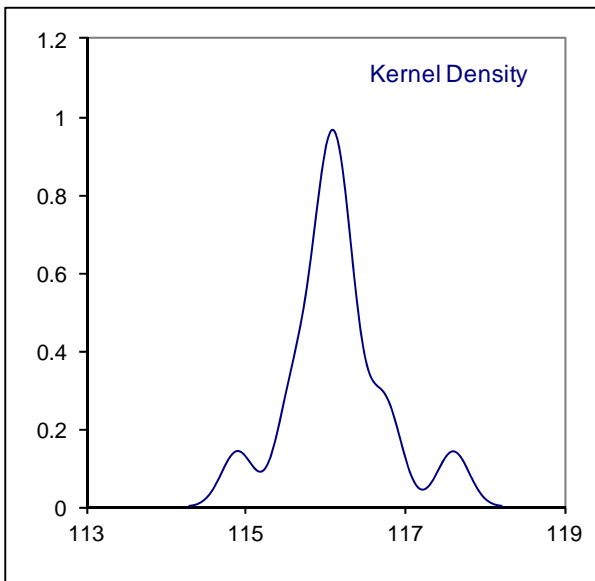
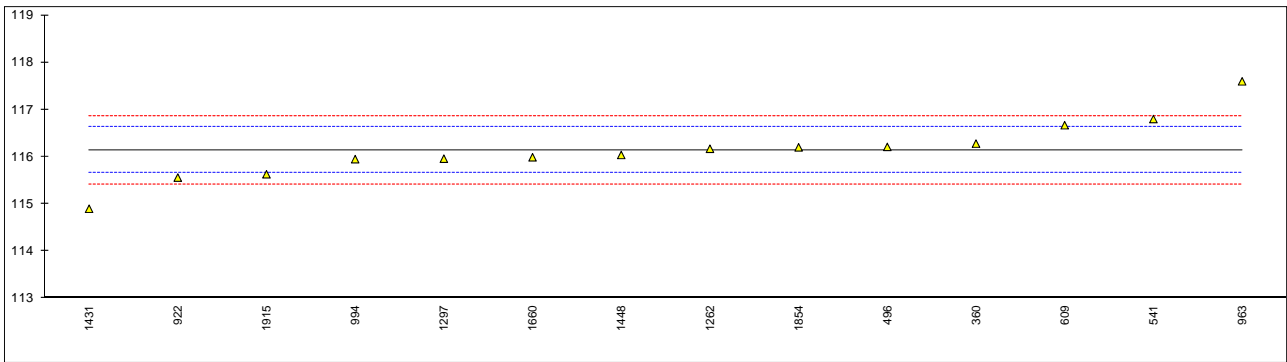


Determination of Viscosity Stabinger @ 40 °C on sample #13062; results in mm<sup>2</sup>/s

lab	method	value	mark	z(targ)	Remarks
173		----		----	
233		----		----	
237		----		----	
252		----		----	
254		----		----	
255		----		----	
260		----		----	
311		----		----	
315		----		----	
325		----		----	
333		----		----	
337		----		----	
340		----		----	
349		----		----	
353		----		----	
360	D7042	116.28		0.58	
396		----		----	
432		----		----	
445		----		----	
446		----		----	
450		----		----	
451		----		----	
473		----		----	
496	D7042	116.21		0.29	
541	D7042	116.8		2.72	
551		----		----	
603		----		----	
608		----		----	
609	D7042	116.67		2.19	
614		----		----	
657		----		----	
663		----		----	
840		----		----	
862		----		----	
875		----		----	
886		----		----	
902		----		----	
912		----		----	
922	D7042	115.56		-2.39	
963	D7042	117.6		6.03	
994	D7042	115.95		-0.78	
1013		----		----	
1017		----		----	
1023		----		----	
1047		----		----	
1059		----		----	
1066		----		----	
1106		----		----	
1146		----		----	
1155		----		----	
1161		----		----	
1173		----		----	
1203		----		----	
1213		----		----	
1243		----		----	
1244		----		----	
1262	D7042	116.1700		0.12	
1271		----		----	
1297	D7042	115.96		-0.74	
1316		----		----	
1326		----		----	
1327		----		----	
1349		----		----	
1402		----		----	
1412		----		----	
1428		----		----	
1431	D7042	114.9		-5.12	
1448	D7042	116.04		-0.41	
1488		----		----	
1535		----		----	
1552		----		----	
1564		----		----	
1622		----		----	
1660	D7042	115.99		-0.62	



1691		----	----
1720		----	----
1722		----	----
1833		----	----
1842		----	----
1850		----	----
1854	D7042	116.2	0.25
1903		----	----
1915	D7042	115.63	-2.11
2122		----	----
2129		----	----
normality		OK	
n		14	
outliers		0	
mean (n)		116.140	
st.dev. (n)		0.6271	
R(calc.)		1.756	
R(D7042:12a)		0.678	

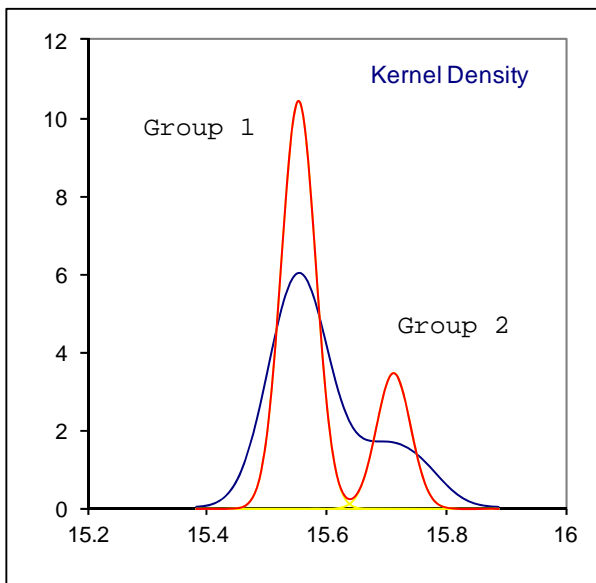
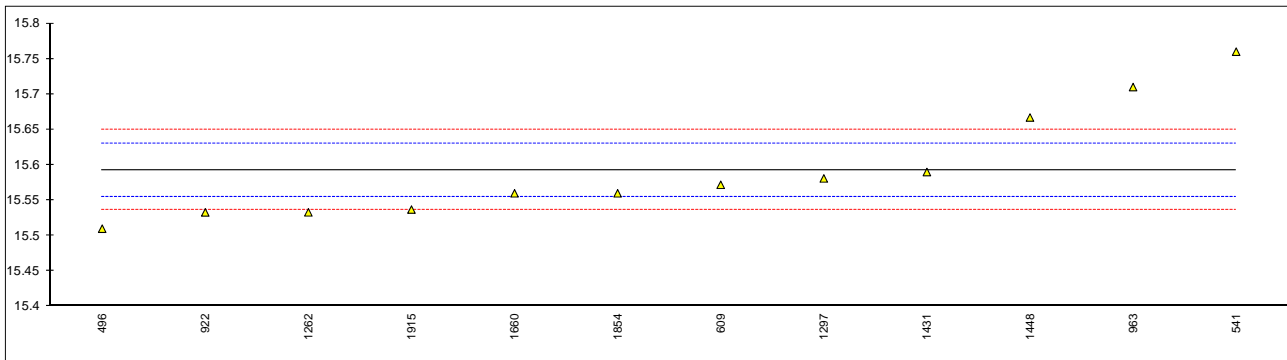


Determination of Viscosity Stabinger @ 100 °C on sample #13062; results in mm<sup>2</sup>/s

lab	method	value	mark	z(targ)	Remarks
173		----		----	
233		----		----	
237		----		----	
252		----		----	
254		----		----	
255		----		----	
260		----		----	
311		----		----	
315		----		----	
325		----		----	
333		----		----	
337		----		----	
340		----		----	
349		----		----	
353		----		----	
360		----		----	
396		----		----	
432		----		----	
445		----		----	
446		----		----	
450		----		----	
451		----		----	
473		----		----	
496	D7042	15.510		-3.89	
541	D7042	15.76		7.87	
551		----		----	
603		----		----	
608		----		----	
609	D7042	15.572		-0.98	
614		----		----	
657		----		----	
663		----		----	
840		----		----	
862		----		----	
875		----		----	
886		----		----	
902		----		----	
912		----		----	
922	D7042	15.533		-2.81	
963	D7042	15.71		5.52	
994		----		----	
1013		----		----	
1017		----		----	
1023		----		----	
1047		----		----	
1059		----		----	
1066		----		----	
1106		----		----	
1146		----		----	
1155		----		----	
1161		----		----	
1173		----		----	
1203		----		----	
1213		----		----	
1243		----		----	
1244		----		----	
1262	D7042	15.5331		-2.81	
1271		----		----	
1297	D7042	15.581		-0.55	
1316		----		----	
1326		----		----	
1327		----		----	
1349		----		----	
1402		----		----	
1412		----		----	
1428		----		----	
1431	D7042	15.59		-0.13	
1448	D7042	15.667		3.49	
1488		----		----	
1535		----		----	
1552		----		----	
1564		----		----	
1622		----		----	
1660	D7042	15.56		-1.54	

1691		----	----
1720		----	----
1722		----	----
1833		----	----
1842		----	----
1850		----	----
1854	D7042	15.56	-1.54
1903		----	----
1915	D7042	15.537	-2.62
2122		----	----
2129		----	----

		<u>Group 1</u>	<u>Group 2</u>
normality	not OK	OK	not OK
n	12	9	3
outliers	0	0	0
mean (n)	15.593	15.553	15.712
st.dev. (n)	0.0781	0.0263	0.0465
R(calc.)	0.219	0.074	0.130
R(D7042:12a)	0.059	0.059	0.060

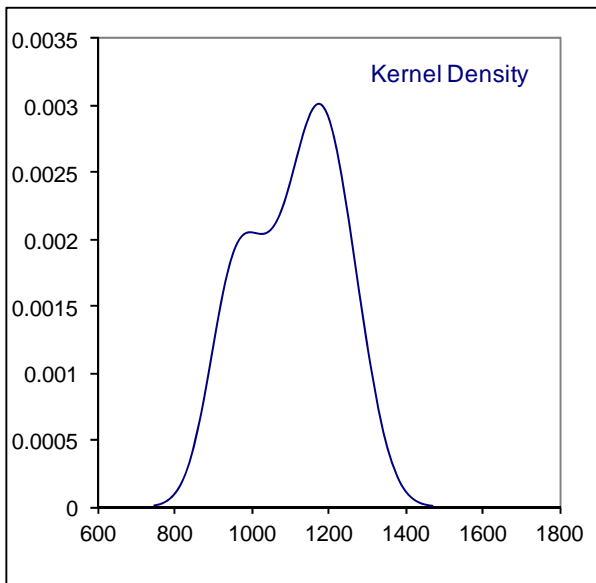
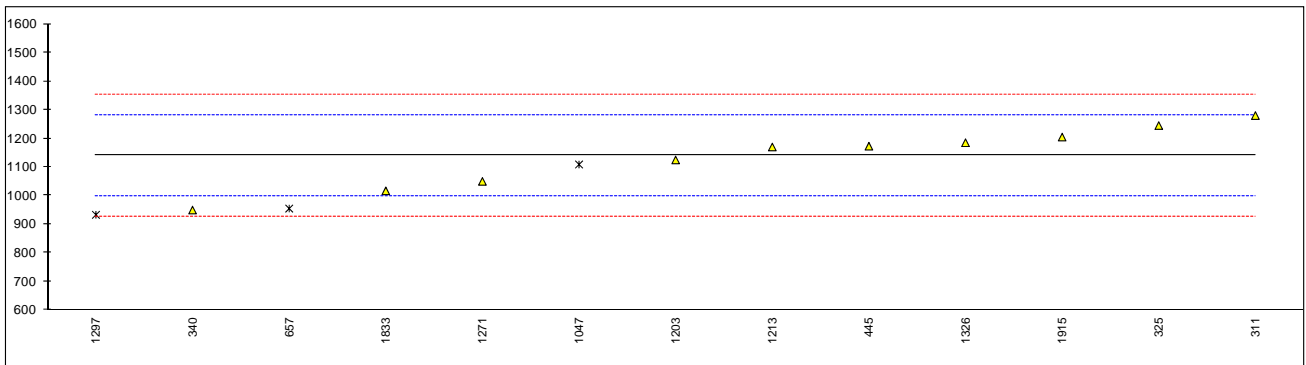


Determination of Nitrogen on sample #13062; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
173		----		----	
233		----		----	
237		----		----	
252		----		----	
254		----		----	
255		----		----	
260		----		----	
311	D5762	1280		1.96	
315		----		----	
325	D5762	1245		1.47	
333		----		----	
337		----		----	
340	INH-10006	950		-2.66	probably unit error, reported: 0.095 mg/kg
349		----		----	
353		----		----	
360		----		----	
396		----		----	
432		----		----	
445	D5762	1173		0.46	
446		----		----	
450		----		----	
451		----		----	
473		----		----	
496		----		----	
541		----		----	
551		----		----	
603		----		----	
608		----		----	
609		----		----	
614		----		----	
657	D4629	955	ex	-2.59	see §4.1
663		----		----	
840		----		----	
862		----		----	
875		----		----	
886		----		----	
902		----		----	
912		----		----	
922		----		----	
963		----		----	
994		----		----	
1013		----		----	
1017		----		----	
1023		----		----	
1047	D4629	1109	ex	-0.43	see §4.1
1059		----		----	
1066		----		----	
1106		----		----	
1146		----		----	
1155		----		----	
1161		----		----	
1173		----		----	
1203	D3228	1125		-0.21	
1213	D3228	1170	C	0.42	first reported: 0.117 mg/kg
1243		----		----	
1244		----		----	
1262		----		----	
1271	D3228	1050	C	-1.26	first reported: 0.122 mg/kg
1297	D4629	933.2	ex	-2.90	see §4.1
1316		----		----	
1326	D5762	1185		0.63	
1327		----		----	
1349		----		----	
1402		----		----	
1412		----		----	
1428		----		----	
1431		----		----	
1448		----		----	
1488		----		----	
1535		----		----	
1552		----		----	
1564		----		----	
1622		----		----	
1660		----		----	

1691		----	----
1720		----	----
1722		----	----
1833	D3228	1017	-1.72
1842		----	----
1850		----	----
1854		----	----
1903		----	----
1915	D3228	1205	0.91
2122		----	----
2129		----	----
normality	OK		
n	10		
outliers	0		
mean (n)	1140.0		
st.dev. (n)	104.57		
R(calc.)	292.8		
R(D3228:08)	200.0		

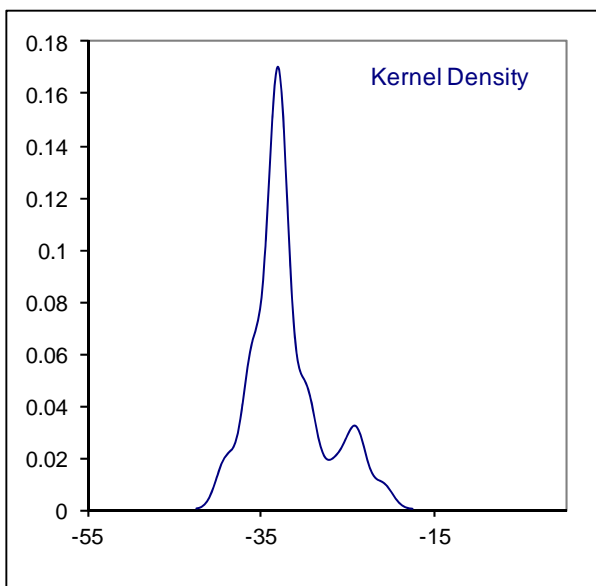
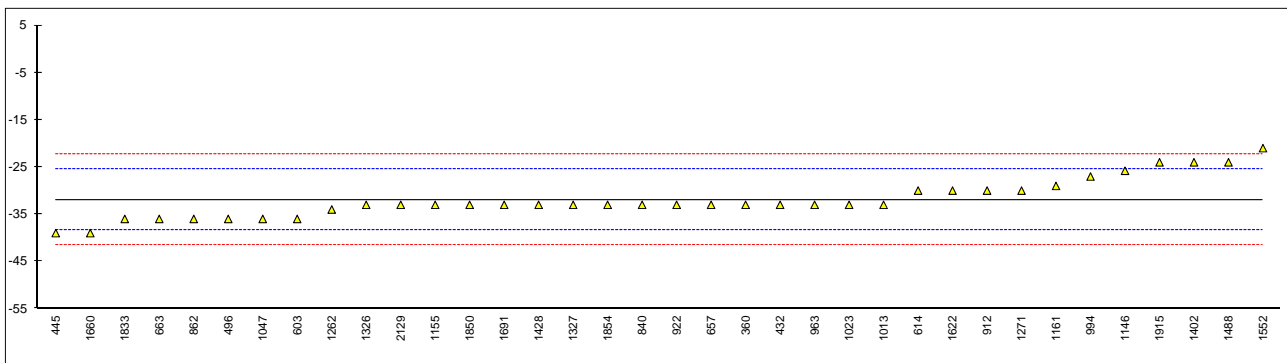
Compare R(D5762:11) = 294.6



Determination of Pour Point (Manual) on sample #13062; results in °C

lab	method	value	mark	z(targ)	Remarks
173		----		----	
233		----		----	
237	D97	<-24		----	
252		----		----	
254	D97	<-6		----	
255		----		----	
260		----		----	
311		----		----	
315		----		----	
325		----		----	
333		----		----	
337		----		----	
340		----		----	
349		----		----	
353		----		----	
360	D97	-33		-0.32	
396		----		----	
432	D97	-33		-0.32	
445	D97	-39		-2.19	
446		----		----	
450		----		----	
451		----		----	
473		----		----	
496	D97	-36.0		-1.25	
541	D97	<21		----	
551		----		----	
603	D97	-36		-1.25	
608		----		----	
609		----		----	
614	D97	-30		0.61	
657	D97	-33		-0.32	
663	D97	-36		-1.25	
840	D97	-33		-0.32	
862	D97	-36		-1.25	
875		----		----	
886		----		----	
902		----		----	
912	D97	-30		0.61	
922	D97	-33		-0.32	
963	D97	-33	C	-0.32	first reported: -21
994	D97	-27		1.55	
1013	D97	-33		-0.32	
1017		----		----	
1023	D97	-33		-0.32	
1047	ISO3016	-36		-1.25	
1059		----		----	
1066		----		----	
1106		----		----	
1146	D97	-25.8		1.92	
1155	ISO3016	-33		-0.32	
1161	D97	-29		0.92	
1173		----		----	
1203		----		----	
1213	D97	<-27		----	
1243		----		----	
1244		----		----	
1262	D97	-34		-0.63	
1271	D97	-30		0.61	
1297		----		----	
1316		----		----	
1326	D97	-33		-0.32	
1327	D97	-33.0		-0.32	
1349		----		----	
1402	D97	-24		2.48	
1412		----		----	
1428	D97	-33		-0.32	
1431		----		----	
1448		----		----	
1488	D97	-24		2.48	
1535		----		----	
1552	D97	-21.0		3.41	
1564	D97	----		----	
1622	D97	-30		0.61	
1660	D97	-39		-2.19	

1691	D97	-33	-0.32
1720		----	----
1722		----	----
1833	D97	-36	-1.25
1842		----	----
1850	ISO3016	-33	-0.32
1854	D97	-33	-0.32
1903		----	----
1915	D97	-24	2.48
2122		----	----
2129	D97	-33	-0.32
normality		not OK	
n		36	
outliers		0	
mean (n)		-31.97	
st.dev. (n)		4.193	
R(calc.)		11.74	
R(D97:12)		9.00	

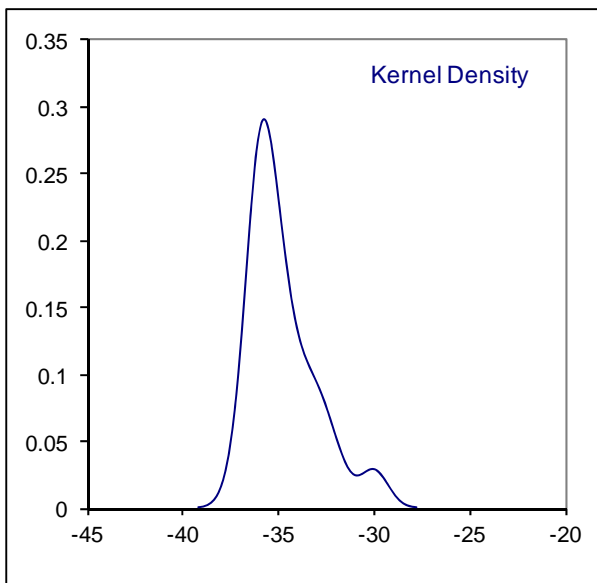
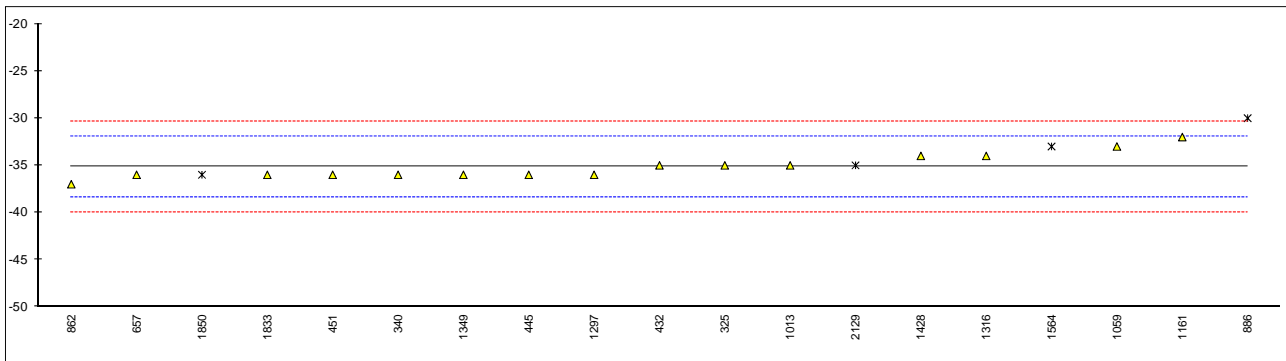


## Determination of Pour Point (Automated, 1°C interval) on sample #13062; results in °C

lab	method	value	mark	z(targ)	Remarks
173		----		----	
233		----		----	
237		----		----	
252		----		----	
254		----		----	
255		----		----	
260		----		----	
311		----		----	
315		----		----	
325	D5950	-35		0.08	
333		----		----	
337		----		----	
340	D5950	-36		-0.54	
349		----		----	
353		----		----	
360		----		----	
396		----		----	
432	D5950	-35		0.08	
445	D5950	-36		-0.54	
446		----		----	
450		----		----	
451	D5949	-36		-0.54	
473		----		----	
496		----		----	
541		----		----	
551		----		----	
603		----		----	
608		----		----	
609		----		----	
614		----		----	
657	D5950	-36		-0.54	
663		----		----	
840		----		----	
862	D5950	-37		-1.16	
875		----		----	
886	D5950	-30	G(0.05)	3.19	
902		----		----	
912		----		----	
922		----		----	
963		----		----	
994		----		----	
1013	D6892	-35		0.08	
1017		----		----	
1023		----		----	
1047		----		----	
1059	D5950	-33		1.33	
1066		----		----	
1106		----		----	
1146		----		----	
1155		----		----	
1161	D6749	-32		1.95	
1173		----		----	
1203		----		----	
1213		----		----	
1243		----		----	
1244		----		----	
1262		----		----	
1271		----		----	
1297	D5950	-36		-0.54	
1316	D5950	-34		0.71	
1326		----		----	
1327		----		----	
1349	D5950	-36		-0.54	
1402		----		----	
1412		----		----	
1428	D6749	-34		0.71	
1431		----		----	
1448		----		----	
1488		----		----	
1535		----		----	
1552		----		----	
1564	D97	-33	ex	1.33	result excluded, see § 4.1
1622		----		----	
1660		----		----	



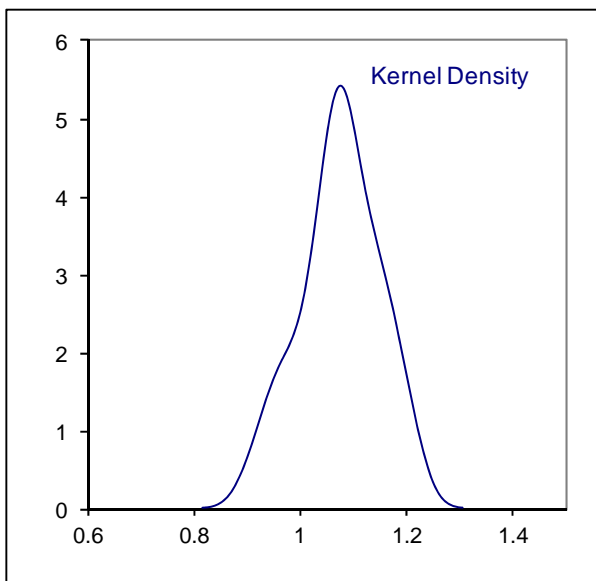
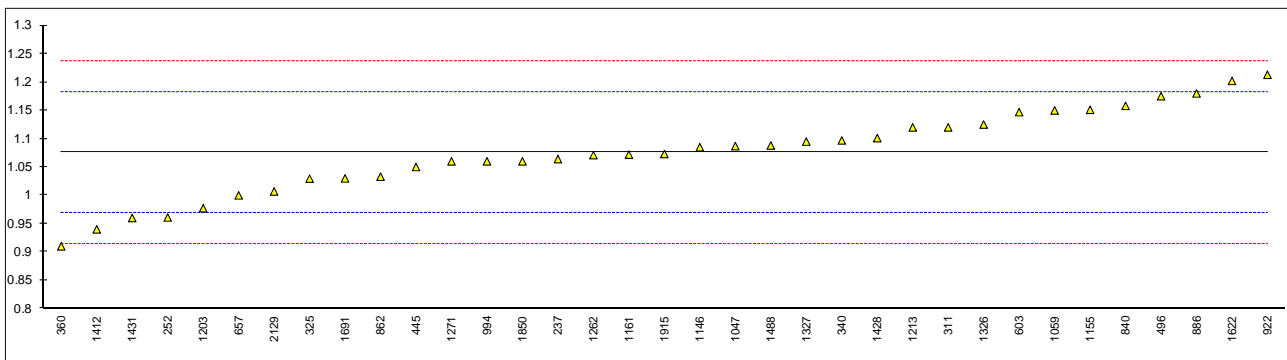
1691		----		----
1720		----		----
1722		----		----
1833	D5950	-36		-0.54
1842		----		----
1850	ISO3016	-36	ex	-0.54
1854		----		----
1903		----		----
1915		----		----
2122		----		----
2129	D97	-35	ex	0.08
	normality	not OK		
	n	15		
	outliers	1		
	mean (n)	-35.13		
	st.dev. (n)	1.356		
	R(calc.)	3.80		
	R(D5950:12)	4.50		



## Determination of Sulphated Ash on sample #13062; results in %M/M

lab	method	value	mark	z(targ)	Remarks
173		----		----	
233		----		----	
237	D874	1.064		-0.22	
252	D874	0.961		-2.14	
254		----		----	
255		----		----	
260		----		----	
311	D874	1.12		0.83	
315		----		----	
325	D874	1.0295		-0.86	
333		----		----	
337		----		----	
340	D874	1.097		0.40	
349		----		----	
353		----		----	
360	D874	0.91		-3.09	
396		----		----	
432		----		----	
445	D874	1.05		-0.48	
446		----		----	
450		----		----	
451		----		----	
473		----		----	
496	D874	1.175		1.85	
541		----		----	
551		----		----	
603	D874	1.147		1.33	
608		----		----	
609		----		----	
614		----		----	
657	D874	1.00		-1.41	
663		----		----	
840	D874	1.158		1.54	
862	D874	1.033		-0.80	
875		----		----	
886	D874	1.18		1.95	
902		----		----	
912		----		----	
922	D874	1.213		2.56	
963		----		----	
994	D874	1.06	C	-0.29	first reported: 0.75 % M/M
1013		----		----	
1017		----		----	
1023		----		----	
1047	ISO3987	1.087		0.21	
1059	ISO3987	1.15		1.39	
1066		----		----	
1106		----		----	
1146	D874	1.085		0.17	
1155	ISO3987	1.151		1.40	
1161	ISO3987	1.072		-0.07	
1173		----		----	
1203	ISO3987	0.9773		-1.84	
1213	D874	1.12		0.83	
1243		----		----	
1244		----		----	
1262	D874	1.071		-0.09	
1271	D874	1.06		-0.29	
1297		----		----	
1316		----		----	
1326	D874	1.125		0.92	
1327	D874	1.095		0.36	
1349		----		----	
1402		----		----	
1412	D874	0.94		-2.53	
1428	D874	1.101		0.47	
1431	D874	0.96		-2.16	
1448		----		----	
1488	D874	1.088		0.23	
1535		----		----	
1552		----		----	
1564		----		----	
1622	D874	1.2021		2.36	
1660		----		----	

1691	D874	1.03	-0.85
1720		----	----
1722		----	----
1833		----	----
1842		----	----
1850	ISO3987	1.06	-0.29
1854		----	----
1903		----	----
1915	D874	1.073	-0.05
2122		----	----
2129	D874	1.007	-1.28
normality		OK	
n		35	
outliers		0	
mean (n)		1.076	
st.dev. (n)		0.0742	
R(calc.)		0.208	
R(D874:07)		0.150	

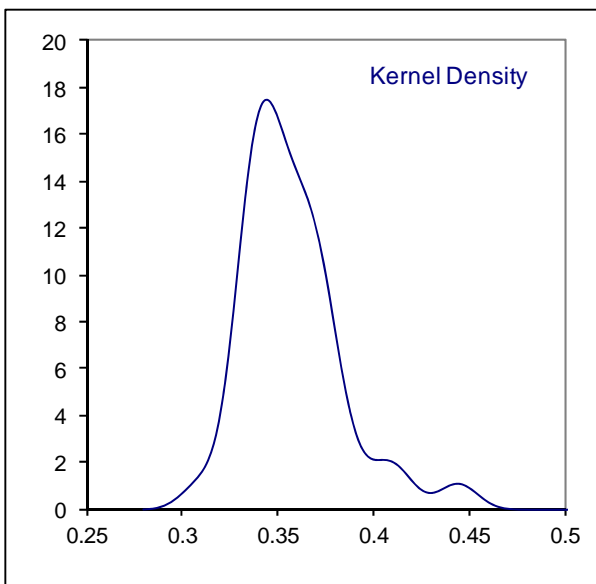
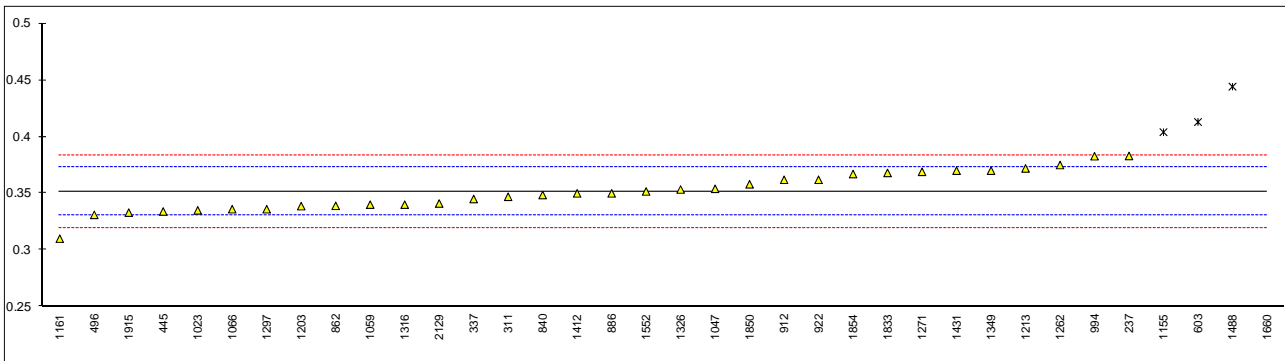


## Determination of Sulphur on sample #13062; results in %M/M

lab	method	value	mark	z(targ)	Remarks
173		----		----	
233		----		----	
237	D4294	0.383		2.96	
252		----		----	
254		----		----	
255		----		----	
260		----		----	
311	D2622	0.347		-0.44	
315		----		----	
325		----		----	
333		----		----	
337	D2622	0.345		-0.62	
340		----		----	
349		----		----	
353		----		----	
360		----		----	
396		----		----	
432		----		----	
445	D2622	0.334		-1.66	
446		----		----	
450		----		----	
451		----		----	
473		----		----	
496	D2622	0.3310		-1.94	
541		----		----	
551		----		----	
603	D4294	0.413	DG(0.05)	5.78	
608		----		----	
609		----		----	
614		----		----	
657		----		----	
663		----		----	
840	D4294	0.3485		-0.29	
862	D2622	0.339		-1.19	
875		----		----	
886	D2622	0.350		-0.15	
902		----		----	
912	D4294	0.362		0.98	
922	D4294	0.362		0.98	
963		----		----	
994	D5453	0.3828	C	2.94	first reported: 0.4194
1013		----		----	
1017		----		----	
1023	D2622	0.335		-1.57	
1047	ISO8754	0.354		0.22	
1059	ISO14596	0.34		-1.10	
1066	D2622	0.336		-1.47	
1106		----		----	
1146		----		----	
1155	D5185	0.404	C,DG(0.05)	4.94	first reported: 4037.36 mg/kg
1161	ISO8754	0.31		-3.92	
1173		----		----	
1203	ISO14596	0.3387		-1.22	
1213	D6481	0.372		1.92	
1243		----		----	
1244		----		----	
1262	D4927	0.3750		2.20	
1271	D4294	0.369		1.64	
1297	D2622	0.3360	C	-1.47	probably unit error, reported 3360 % M/M
1316	D2622	0.340		-1.10	
1326	D6481	0.3534		0.17	
1327		----		----	
1349	D2622	0.370		1.73	
1402		----		----	
1412	D4294	0.350	C	-0.15	first reported: 0.421
1428		----		----	
1431	D4294	0.37		1.73	
1448		----		----	
1488	D4294	0.4442	C,G(0.01)	8.72	first reported: 0.4093
1535		----		----	
1552	D4294	0.3517		0.01	
1564		----	W	----	result withdrawn, first reported: 0.314
1622		----		----	
1660	D5185	0.5600	G(0.01)	19.64	

1691		----	----
1720		----	----
1722		----	----
1833	IP336	0.368	1.54
1842		----	----
1850	ISO8754	0.358	0.60
1854	D2622	0.367	1.45
1903		----	----
1915	D4294	0.333	-1.76
2122		----	----
2129	D4294	0.341	-1.00

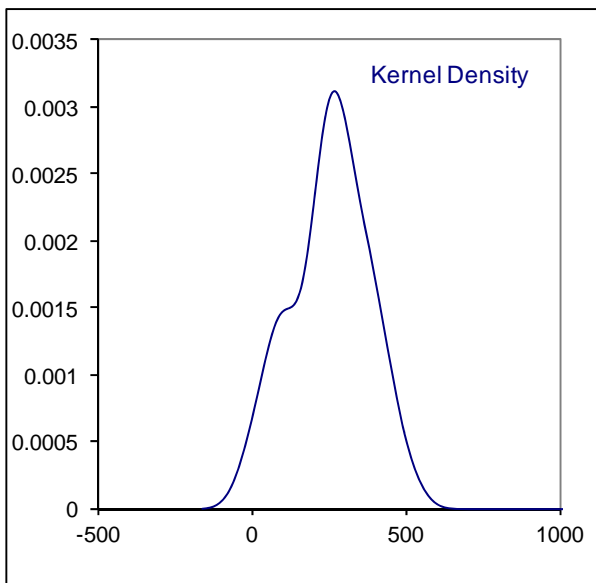
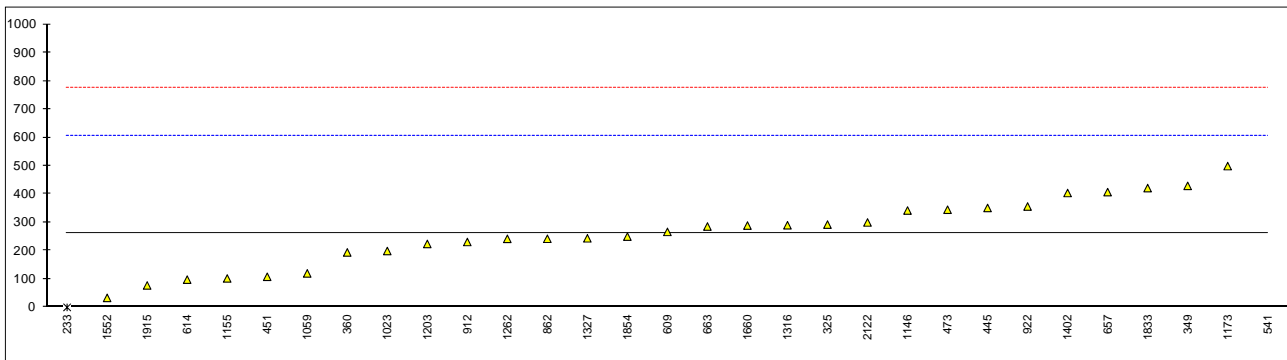
normality	OK	<u>Only ASTM D2622</u>
n	32	OK
outliers	4	1
mean (n)	0.3516	0.3393
st.dev. (n)	0.01707	0.00618
R(calc.)	0.0478	0.0173
R(D2622:10)	0.0297	0.0288



## Determination of Water on sample #13062; results in mg/kg

lab	method	value	mark	z(targ)	remarks
173		----		----	
233	in house	0.001	G(0.05)	-1.58	probably unit error?
237		----		----	
252		----		----	
254		----		----	
255		----		----	
260		----		----	
311		----		----	
315		----		----	
325	D6304A	292.5		0.17	
333		----		----	
337		----		----	
340		----		----	
349	D6304	429		0.99	
353		----		----	
360	D6304C	194.0		-0.42	
396		----		----	
432		----		----	
445	D6304A	351		0.53	
446		----		----	
450		----		----	
451	D6304C	108		-0.94	
473	D6304C	344.8		0.49	
496		----		----	
541	D6304A	2015	G(0.01)	10.53	
551		----		----	
603		----		----	
608		----		----	
609	D6304C	266.7		0.02	
614	D6304C	98		-1.00	
657	D6304C	407		0.86	
663	D6304A	285.6		0.13	
840		----		----	
862	D6304C	242.4		-0.13	
875		----		----	
886		----		----	
902		----		----	
912	D6304C	231		-0.20	
922	D6304	356.2		0.56	
963		----		----	
994		----		----	
1013		----		----	
1017		----		----	
1023	D6304A	199		-0.39	
1047		----		----	
1059	D6304AMod.A	120		-0.86	
1066		----		----	
1106		----		----	
1146	D6304C	342		0.47	
1155	D6304C	102.27		-0.97	
1161		----		----	
1173	in house	499		1.42	
1203	D6304A	224		-0.24	
1213		----		----	
1243		----		----	
1244		----		----	
1262	D6304A	242		-0.13	
1271		----		----	
1297		----		----	
1316	D6304C	290		0.16	
1326		----		----	
1327	D6304C	244		-0.12	
1349		----		----	
1402	D6304C	404		0.84	
1412		----		----	
1428		----		----	
1431		----		----	
1448		----		----	
1488		----		----	
1535		----		----	
1552	D6304	33.50		-1.38	
1564		----		----	
1622		----		----	
1660	IEC60814	289		0.15	

1691		----	----
1720		----	----
1722		----	----
1833	D6304	421.2554	0.95
1842		----	----
1850		----	----
1854	D6304C	250	-0.08
1903		----	----
1915	D6304	77.42	-1.12
2122	in house	300	0.22
2129		----	----
		W	result withdrawn, first reported: 818
			<u>Only ASTM D6304C data</u>
normality	OK		OK
n	29		14
outliers	2		0
mean (n)	263.57		251.73
st.dev. (n)	116.477		102.408
R(calc.)	326.14		286.74
R(D6304:07)	478.87		465.83



## Determination of Calcium (Ca) on sample #13062; results in mg/kg

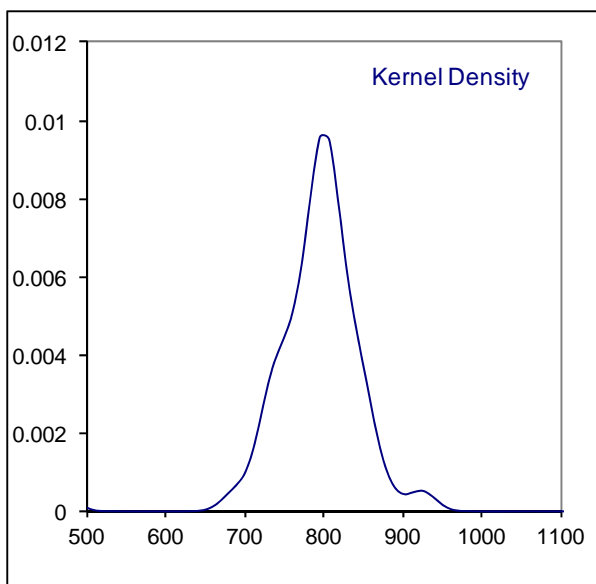
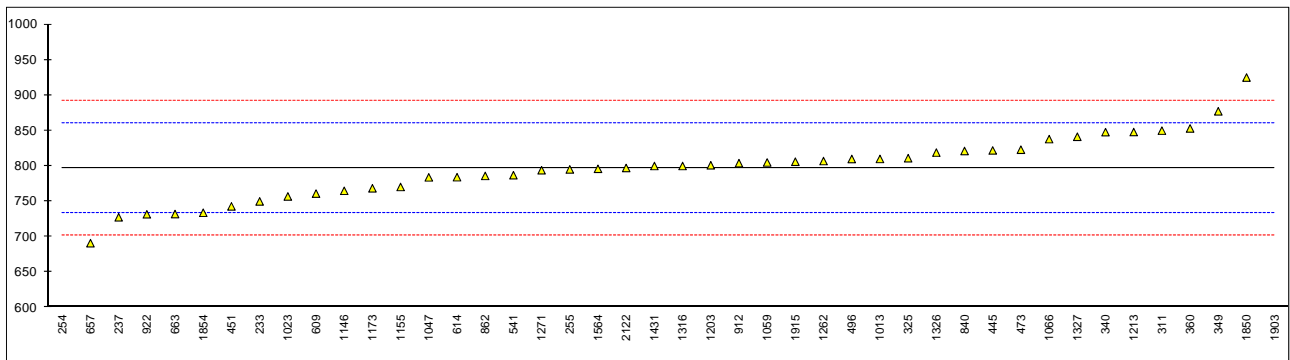
lab	method	value	mark	z(targ)	remarks
173		----		----	
233	D6595	750		-1.48	
237	D5185	727.6	C	-2.19	first reported: 673.6
252		----		----	
254	IP308	468.9	C,G(0.01)	-10.36	first reported: 958.9
255	INH-1	795.173		-0.06	
260		----		----	
311	D5185	850		1.67	
315		----		----	
325	D5185	811		0.44	
333		----		----	
337		----		----	
340	D5185	847.8		1.60	
349	D5185	877.30		2.53	
353		----		----	
360	D5185	853		1.77	
396		----		----	
432		----		----	
445	D5185	822		0.79	
446		----		----	
450		----		----	
451	D5185	743		-1.70	
473	D5185	823		0.82	
496	D5185	809.9		0.41	
541	D5185	787		-0.32	
551		----		----	
603		----		----	
608		----		----	
609	D5185	761		-1.14	
614	D5185	784.2		-0.40	
657	D5185	691	C	-3.35	first reported: 699
663	D5185	732.1		-2.05	
840	UOP389	821.2		0.76	
862	D5185	786		-0.35	
875		----		----	
886		----		----	
902		----		----	
912	D5185	804		0.22	
922	D4628	731.8		-2.06	
963		----		----	
994		----		----	
1013	D5185	810	C	0.41	probably unit error, reported: 0.081 mg/kg
1017		----		----	
1023	D5185	757		-1.26	
1047	D5185	784		-0.41	
1059	in house	805		0.25	
1066	D4951	838	C	1.29	probably unit error, reported: 0.0838 mg/kg
1106		----		----	
1146	D5185	765		-1.01	
1155	D5185	770.39		-0.84	
1161		----		----	
1173	in house	768.5		-0.90	
1203	D5185	801		0.13	
1213	D4628	848		1.61	
1243		----		----	
1244		----		----	
1262	D5185	807		0.32	
1271	D6481	794	C	-0.09	first reported: 0.0794 mg/kg
1297		----		----	
1316	D5185	800		0.09	
1326	D5185	819		0.69	
1327	D5185	841.3		1.40	
1349		----		----	
1402		----		----	
1412		----		----	
1428		----	W	----	result withdrawn: first reported: 650
1431	in house	800		0.09	
1448		----		----	
1488		----		----	
1535		----		----	
1552		----		----	
1564	D4951	796		-0.03	
1622		----		----	
1660	D5185	<5		----	



1691	----	----		
1720	----	----		
1722	----	----		
1833	----	----		
1842	----	----		
1850	in house	925	4.04	
1854	D5185	734	-1.99	
1903	in house	3392.7	G(0.01)	81.93
1915	D5185	806	0.28	
2122	D5185	797.15	0.00	
2129	----	----		

normality OK  
n 42  
outliers 2  
mean (n) 797.01  
st.dev. (n) 44.296  
R(calc.) 124.03  
R(D5185:09) 88.71

Application range 40 - 9000 mg/kg



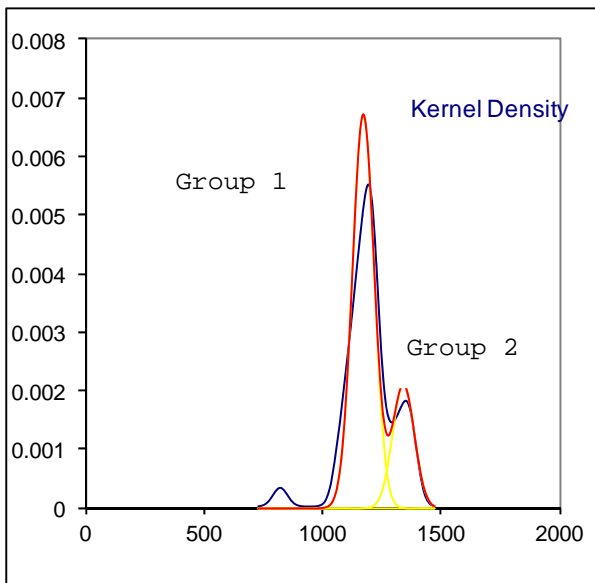
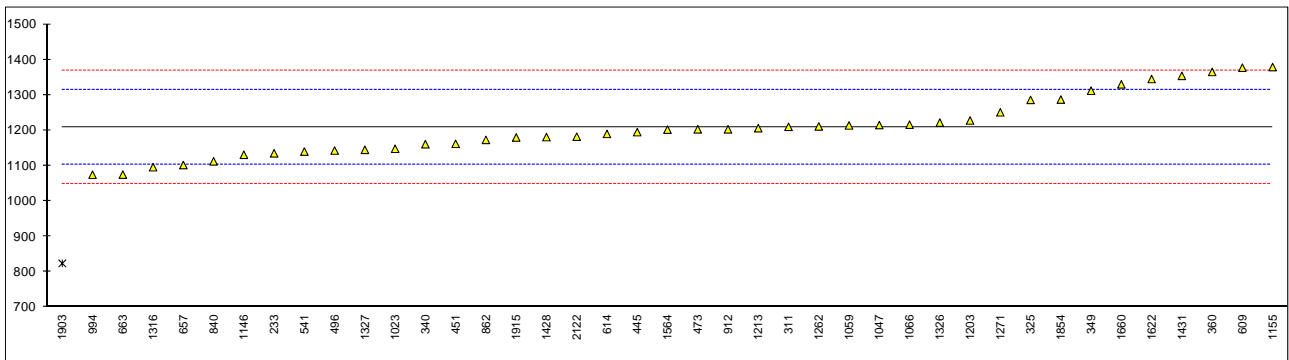
## Determination of Phosphorus (P) on sample #13062; results in mg/kg

lab	method	value	mark	z(targ)	remarks
173		----		----	
233	D6595	1135		-1.38	
237		----		----	
252		----		----	
254		----		----	
255		----		----	
260		----		----	
311	D5185	1210		0.03	
315		----		----	
325	D5185	1286		1.45	
333		----		----	
337		----		----	
340	D5185	1161.0		-0.89	
349	D5185	1312.17		1.94	
353		----		----	
360	D5185	1365		2.93	
396		----		----	
432		----		----	
445	D5185	1195		-0.25	
446		----		----	
450		----		----	
451	D5185	1162		-0.87	
473	D5185	1203		-0.10	
496	D5185	1143		-1.23	
541	D5185	1140		-1.28	
551		----		----	
603		----		----	
608		----		----	
609	D5185	1377	C	3.16	first reported: 1787
614	D5185	1190		-0.35	
657	D5185	1102		-2.00	
663	D5185	1075.1		-2.50	
840	IP500	1112.4		-1.80	
862	D5185	1173		-0.67	
875		----		----	
886		----		----	
902		----		----	
912	D5185	1203		-0.10	
922		----		----	
963		----		----	
994	D5185	1074.68		-2.51	
1013		----		----	
1017		----		----	
1023	D5185	1148		-1.13	
1047	D5185	1215		0.12	
1059	in house	1214		0.10	
1066	D4951	1216		0.14	
1106		----		----	
1146	D5185	1131		-1.45	
1155	D5185	1378.94		3.19	
1161		----		----	
1173		----		----	
1203	D5185	1228		0.36	
1213	D4951	1206		-0.05	
1243		----		----	
1244		----		----	
1262	D5185	1211		0.05	
1271	D6481	1251	C	0.80	first reported: 0.251 mg/kg
1297		----		----	
1316	D5185	1096		-2.11	
1326	D5185	1222		0.25	
1327	D5185	1145		-1.19	
1349		----		----	
1402		----		----	
1412		----		----	
1428	D5185	1181		-0.52	
1431	in house	1354	C	2.72	first reported: 1900
1448		----		----	
1488		----		----	
1535		----		----	
1552		----		----	
1564	D4951	1202		-0.12	
1622	D5185	1345.05		2.56	
1660	D5185	1330		2.27	

1691		----		----
1720		----		----
1722		----		----
1833		----		----
1842		----		----
1850		----		----
1854	D5185	1287		1.47
1903	in house	824.2	G(0.01)	-7.20
1915	D5185	1180		-0.53
2122	D5185	1182.0		-0.50
2129		----		----

		<u>Group1</u>	<u>Group 2</u>
normality	not OK	OK	OK
n	40	29	11
outliers	1	0	0
mean (n)	1208.56	1166.49	1319.47
st.dev. (n)	82.825	44.215	51.146
R(calc.)	231.91	123.80	143.21
R(D5185:09)	149.47	146.86	156.19

Application range: 10 -1000 mg/kg

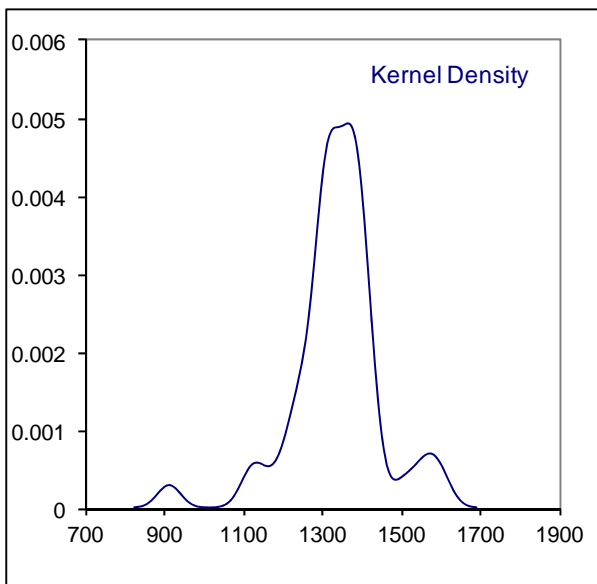
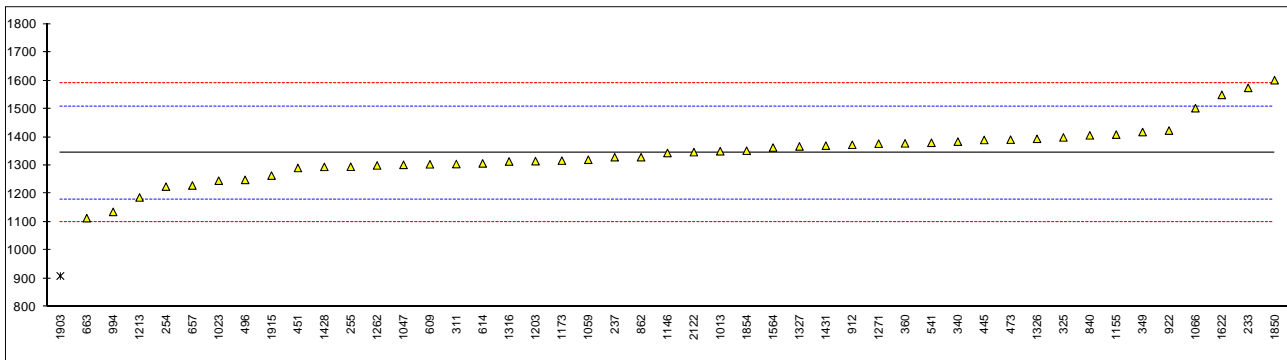


## Determination of Zinc (Zn) on sample #13062; results in mg/kg

lab	method	value	mark	z(targ)	Remarks
173		----		----	
233	D6595	1574	C	2.81	first reported: 1618
237	D5185	1329.0		-0.18	
252		----		----	
254	IP308	1225		-1.45	
255	INH-1	1295.47		-0.59	
260		----		----	
311	D5185	1305		-0.47	
315		----		----	
325	D5185	1399		0.67	
333		----		----	
337		----		----	
340	D5185	1384.0		0.49	
349	D5185	1417.81		0.90	
353		----		----	
360	D5185	1378		0.42	
396		----		----	
432		----		----	
445	D5185	1390		0.56	
446		----		----	
450		----		----	
451	D5185	1291		-0.64	
473	D5185	1391		0.58	
496	D5185	1249		-1.16	
541	D5185	1380		0.44	
551		----		----	
603		----		----	
608		----		----	
609	D5185	1304		-0.49	
614	D5185	1307		-0.45	
657	D5185	1229		-1.40	
663	D5185	1113.9		-2.81	
840	UOP389	1406.4		0.76	
862	D5185	1329		-0.18	
875		----		----	
886		----		----	
902		----		----	
912	D5185	1373		0.36	
922	D4628	1423		0.97	
963		----		----	
994	D5185	1135.94		-2.54	
1013	D5185	1350	C	0.08	probably unit error, reported: 0.135 mg/kg
1017		----		----	
1023	D5185	1246		-1.19	
1047	D5185	1302		-0.51	
1059	in house	1320		-0.29	
1066	D4951	1502	C	1.93	probably unit error, reported: 0.1502 mg/kg
1106		----		----	
1146	D5185	1344		0.00	
1155	D5185	1408.84		0.79	
1161		----		----	
1173	in house	1317.05		-0.33	
1203	D5185	1315		-0.35	
1213	D4628	1187		-1.92	
1243		----		----	
1244		----		----	
1262	D5185	1300		-0.53	
1271	D6481	1377	C	0.41	first reported: 0.1377 mg/kg
1297		----		----	
1316	D5185	1314		-0.36	
1326	D5185	1394		0.61	
1327	D5185	1367		0.28	
1349		----		----	
1402		----		----	
1412		----		----	
1428	D5185	1295		-0.60	
1431	in house	1370		0.32	
1448		----		----	
1488		----		----	
1535		----		----	
1552		----		----	
1564	D4951	1363		0.23	
1622	D5185	1549.02		2.51	
1660	D5185	<5		----	

1691		----		----
1720		----		----
1722		----		----
1833		----		----
1842		----		----
1850	in house	1601		3.14
1854	D5185	1352		0.10
1903	in house	909.6	G(0.01)	-5.30
1915	D5185	1264.2		-0.97
2122	D5185	1346.9		0.04
2129		----		----
normality		OK		
n		46		
outliers		1		
mean (n)		1343.8		
st.dev. (n)		96.12		
R(calc.)		269.1		
R(D5185:09)		229.2		

Application range: 60 - 1600 mg/kg



## APPENDIX 2

## Analytical details acid number determination via ASTM D664

lab	method	type of apparatus	KOH solution			type electrodes	difference pH4-pH7 (mV)	drift of elec.	blank titration	sample size (g)	unit reading
			A	B	C						
173											
233											
237	11, A	848 Titrino plus	Yes	Yes	Yes	LiCl sat. in ethanol	169	1	Yes	5.004	mV
252											
254											
255											
260											
311	A	835 titrindo	*)	*)	*)	Ag/AgCl	174		Yes	5	pH
315											
325	11,A	Metrohm automatic-autosampler	No	Yes	No	LiCl sat in ethanol	175	<30 sec	Yes	<u>±1 gram</u>	mV
333											
337											
340	11, A	Titrimo 798 MPT	Yes	Yes	Yes	LiCl	168	2	Yes	5.0854	mV
349	11a, A	Automatic Titration System	Yes	Yes	Yes	pH glass electrode	175		Yes	<u>1.295</u>	mV
353	11a,A	Metrohm Titrimo Auto Titrator 702 SM	Yes	Yes	Yes	pH with reference electrode	n.a		Yes	5.081	pH
360	A	MettlerToledo Titrator DL 28	Yes	Yes	Yes	DG 113-SC	165.0	1	Yes	5	mV
396											
432											
445	11a,A		*)	*)	*)	Methrom Combination Elektrode	167	n.a	Yes	4.9929	mV
446											
450											
451											
473											
496	11a,A	Metrohm 730/721/ 2.2	*)	*)	*)	Methrom Pt-Titrode, Metrohm Ag/AgCl	172	2	Yes	<u>1.607/</u> <u>1.649</u>	mV
541											
551											
603											
608											
609	11a,A	Metrohm Automatic Potentiometer	*)	*)	*)	Ag/Cl electrode	209.2	2	Yes	<u>1.0204</u>	mV
614	A	702 SM Titrimo	Yes	Yes	No	pH/mV	155		Yes	11.28	mV
657	11a,A	Titrimo Plus 848	*)	*)	*)	Metrohm LL solvotrode	173	1	Yes	5	mV
663	11a,A	Metrohm automatic titrator 716 DMS	Yes	Yes	Yes	LiCl sat in ethanol	170	1	Yes	5	mV
840	11a,A	Metrohm DMP 785 Titrimo	Yes	Yes	Yes	Solvotrode / Metrohm 6.0229.100	177	1	Yes	±5 gram	mV
862	11a,A	Metrohm 905 Titrimo	Yes	Yes	Yes	Metrohm 6.0229.100	178	<1	Yes	5.01215	mV
875											
886											
902											
912	11a,A	Autopotentiometer GT200	Yes	Yes	Yes	Elec. GTRSI0B, Dec, Elec GTPHIB	171.3	2	Yes	<u>4.5726/</u> <u>4.6724</u>	mV
922											
963											
994	A	Metrohm 848 plus	Yes	Yes	Yes	pH electrode	176	<1	Yes	5	mV
1013											
1017											
1023											
1047											
1059											
1066											
1106											
1146	11a,A		*)	*)	*)	Ag/AgCl elektrode	166.4	1	Yes	4.94	mV
1155											
1161	11a,A		*)	*)	*)	DG 116-SC	160		Yes		mV

1173											
1203	11a,A	KEM Titrator AT-510	Yes	Yes	Yes	Thermo Scientiq 8172 comb pH	250	1	Yes	5	mV
1213											
1243											
1244											
1262											
1271	9,A	Titrimo 716 DMS	Yes	Yes	Yes	Metrohm LL solvotrode	176		No	5±0.5	mV
1297											
1316											
1326	11a,A	MettlerToledo T70	Yes	Yes	No	Mettler Toledo DG 113-SC			Yes	5.05	mV
1327	11a,A	Metrohm 862C titrosampler	No	Yes	Yes	Metrohm solvotrode			Yes	5.0	mV
1349											
1402											
1412											
1428											
1431	11,A		No	Yes	Yes	Glass electrode DG113	161		Yes	<u>0.877/</u> <u>0.7687</u>	mV
1448	11a,A	Metrohm 808 titando	*)	*)	*)	Metrohm LiCl sat in ethanol	157.9	1	Yes	5.0	mV
1488											
1535											
1552	09,A	Manual titration + pH Meter	Yes	Yes	Yes	pH Glass electrode	170		Yes	4.9502	pH
1564											
1622	11a,A	Metrohm Titrimo 794	Yes	Yes	Yes	Ag/AgCl LiCl sat. in ethanol			Yes	1	mV
1660											
1691	11a,A	848 titrimo plus	No	Yes	Yes	Metrohm 6.0229.100			Yes	5.0	mV
1720	07	848 titrimo plus	Yes	Yes	Yes	Combined pH electrode 6.0229.100	166		Yes	5	mV
1722											
1833	A	Automatic titrator	*)	*)	*)	Combination electrode	175		Yes	5	mV
1842	09a,A	Metrohm 794 Titrimo	Yes	Yes	Yes	Metrohm 6.0229.100 (LiCl sat in ethanol)		1	Yes	5	mV
1850											
1854	11a,A		No	No	No	Metrohm 6.0229.100 (LiCl sat in ethanol)	165		Yes	5.2972	mV
1903											
1915											
2122											
2129	09a,A		Yes	Yes	Yes	Solvotrode	167		Yes	0.25	mV

A = boiled for 10 minutes

B = stand for 2 days

C = filtered

\*) a commercial "ready for use" KOH standard solution was used

**APPENDIX 3****Number of participants per country**

1 lab in ARGENTINA  
1 lab in AUSTRALIA  
1 lab in AUSTRIA  
2 labs in AZERBAIJAN  
2 labs in BELGIUM  
1 lab in BOSNIA and HERZEGOVINA  
1 lab in BRAZIL  
3 labs in BULGARIA  
1 lab in CAMEROON  
1 lab in CROATIA  
3 labs in FRANCE  
2 labs in GERMANY  
1 lab in GHANA  
3 labs in GREECE  
1 lab in HUNGARY  
1 lab in INDIA  
1 lab in INDONESIA  
1 lab in IRELAND  
2 labs in ITALY  
2 labs in KENYA  
1 lab in LATVIA  
4 labs in MALAYSIA  
1 lab in NEGARA BRUNEI DARUSSALAM  
1 lab in NIGERIA  
2 labs in NORWAY  
4 labs in P.R. of CHINA  
1 lab in PAKISTAN  
1 lab in POLAND  
1 lab in PORTUGAL  
1 lab in RUSSIA  
2 labs in SAUDI ARABIA  
1 lab in SERBIA  
1 lab in SINGAPORE  
1 lab in SLOVENIA  
1 lab in SOUTH KOREA  
2 labs in SPAIN  
1 lab in SUDAN  
1 lab in SWEDEN  
1 lab in TAIWAN R.O.C.  
1 lab in TANZANIA  
2 labs in THAILAND  
4 labs in THE NETHERLANDS  
4 labs in TURKEY  
1 lab in U.S.A.  
11 labs in UNITED KINGDOM  
2 labs in VIETNAM  
1 lab in ZAMBIA



## 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
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, January 2010
- 2 ASTM E178-89
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