

Results of Proficiency Test Unused Lubricating Oil May 2011

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 Lubricating Oil. In the annual proficiency testing program 2010/2011, it was decided to continue the proficiency test for the analyses of unused Lubricating Oil. In this interlaboratory study, 80 laboratories in 46 different countries have participated. See appendix 2 for the number of participants per country. In this report, the results of the 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 #11041) 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 guide 43 and ILAC-G13:2007, (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This ensures 100% confidentiality of participant's data. Also customer's satisfaction is measured on a regular basis by sending questionnaires.

2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organization, Statistics and Evaluation' of January 2010 (iis-protocol, version 3.2), which can be downloaded from 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 (Heavy duty Engine Oil 10W-40) was transferred after homogenizing into 128 brown glass bottles of 1 litre (labelled #11041). The homogeneity of the subsamples #11041 was checked by determination of Density @ 15°C in accordance with ASTM D4052:09 and Kinematic Viscosity @ 40°C in accordance with ASTM D445:09 on 8 stratified randomly selected samples.

	Density @ 15 °C in kg/L	Viscosity @40°C in mm ² /s
Sample #11041-1	0.86612	87.59
Sample #11041-2	0.86612	87.55
Sample #11041-3	0.86612	87.55
Sample #11041-4	0.86612	87.55
Sample #11041-5	0.86612	87.56
Sample #11041-6	0.86612	87.56
Sample #11041-7	0.86612	87.66
Sample #11041-8	0.86612	87.58

Table 1: homogeneity test results of subsamples #11041

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 ² /s
r (sample #11041)	0.00000	0.10
reference test	ASTM D4052:09	ASTM D445:11a
0.3 x R(reference test)	0.00015	0.20

Table 2: evaluation of the repeatabilities of the subsamples #11041

The calculated repeatabilities are all less than 0.3 times the corresponding reproducibilities of the reference methods. Therefore, homogeneity of the subsamples #11041 was assumed.

To each of the participating laboratories, 1 sample of 1 L in a brown glass bottle (labelled #11041) was sent on April 20, 2011.

2.5 ANALYSES

The participants were requested to determine on sample #11041: Acid Number (Total), Base Number (Total), Colour ASTM, Conradson Carbon Residue, Density @ 15°C, Flash Point PMcc, Flash Point COC, Kinematic Viscosity @ 40°C and @ 100°C, Viscosity Stabinger @ 40°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, was sent together with each set of samples. Also, a letter of instructions and a SDS were added to the 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.

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 3; 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. 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, some problems were encountered with the dispatch of the samples to laboratories in Brazil, Ecuador, Ghana and Russia. Eighteen participants reported after the final reporting date and three participants did not report any test results at all. Not all laboratories were able to report all analyses requested. In total 78 participants reported 804 test results. Observed were 33 outlying results, which is 4.1% 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: Colour, Density @ 15°C, Flash Point PMcc, Kinematic Viscosity @ 100°C, Pour Point (manual) and Calcium. 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 3.

Acid Number (total): This determination was very problematic. Two statistical outliers were observed. Another four test results were excluded from the statistical evaluation, as the reported test method is not equivalent with ASTM D664. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with ASTM D664:09a.

Base Number (total): This determination was not problematic. No statistical outliers were observed. Six test results were excluded from the statistical evaluation, as the reported test method is not equivalent with ASTM D2896. The calculated reproducibility is in full agreement with the requirements of ASTM D2896:07a.

Colour: This determination was not problematic. Only one statistical outlier was observed and the calculated reproducibility after rejection of the statistical outlier is in good agreement with ASTM D1500:07.

CCR: This determination was not problematic. No statistical outliers were observed and two results were excluded for statistical evaluation, as the reported test method is not equivalent with ASTM D189. The calculated reproducibility is in good agreement with the requirements of ASTM D189:06e2.

Density @ 15°C: This determination was problematic. Four statistical outliers were observed and the calculated reproducibility, after rejection of the statistical outliers, is not in agreement with the requirements of ASTM D4052:09. The large spread may be explained by not correcting the test result for viscosity. (see density tables)

Flash Point PMcc: This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D93:08 procedure A.

Flash Point COC: This determination was not problematic. Only one statistical outlier was observed and the calculated reproducibility after rejection of the statistical outlier is in good agreement with ASTM D92:10.

Kin.Visco.@ 40°C: This determination was problematic. Three statistical outliers were observed and the calculated reproducibility after rejection of the statistical outliers is not in agreement with the strict requirements of ASTM D445:11a.

- Kin.Visco.@ 100°C: This determination was very problematic. Three statistical outliers were observed and the calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the strict requirements of ASTM D445:11a.
- Visco. Stabinger at 40°C This determination was very problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is not at all in agreement with ASTM D7042:11.
- Nitrogen: This determination was problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is not at all in agreement with ASTM D3228:08.
- Pour Point (manual): This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with ASTM D97:09.
- Pour Point (automated): This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in full agreement with ASTM D5950:07.
- Sulphated Ash: This determination was not problematic. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D874:07.
- Sulphur: This determination was very problematic. Six statistical outliers were observed and two results were excluded for statistical evaluation. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ASTM D2622:10. When the ASTM D2622 data was evaluated separately, the calculated reproducibility is again not at all in agreement.
- Water: This determination was very problematic for a large 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. At least thirteen laboratories reported results determined according ASTM D6304 method C. These results were low, which suggests that the low average may be more reliable than the higher results, which is in agreement with the low solubility of water in lube oil. After excluding all results, except ASTM D6304-C, the calculated reproducibility is in good agreement with the requirements of ASTM D6304:07.
- Calcium: This determination was not problematic. Only one statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D5185:09.

Phosphorus: This determination was problematic. Only two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ASTM D5185:09.

Zinc: 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.

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

Parameter	unit	n	Average	2.8 * sd	R(lit)
Total Acid Number	mg KOH/g	45	4.26	2.35	0.74
Total Base Number	mg KOH/g	41	16.16	1.02	1.13
Colour		33	2.9	0.5	1.0
Conradson Carbon Residue	%M/M	31	1.77	0.27	0.36
Density @ 15 °C	kg/L	63	0.8662	0.0007	0.0005
Flash Point PMcc	°C	58	203.5	9.1	14.5
Flash Point COC	°C	53	228.0	11.3	18.0
Kinematic Viscosity @ 40 °C	mm ² /s	65	87.19	1.06	0.66
Kinematic Viscosity @ 100 °C	mm ² /s	63	13.32	0.26	0.10
Stabinger Viscosity @ 40 °C	mm ² /s	12	86.88	1.40	0.47
Nitrogen	mg/kg	12	891	352	200
Pour Point, manual	°C	36	-34.0	7.9	9.0
Pour Point, automated	°C	20	-36.9	4.5	4.5
Sulphated Ash	%M/M	30	1.89	0.23	0.32
Sulphur	%M/M	22	0.327	0.049	0.028
Water	mg/kg	13	312.2	332.0	530.1
Calcium	mg/kg	46	4800	995	916
Phosphorus	mg/kg	42	1194	246	149
Zinc	mg/kg	46	1359	302	232

Table 3: reproducibilities of results of sample #11041

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 2011 WITH PREVIOUS PT'S

	May 2011	May 2010	April 2009	April 2008
Number of reporting participants	78	96	86	86
Number of results reported	804	985	813	656
Statistical outliers	33	52	45	48
Percentage outliers	4.1%	5.3%	5.5%	7.3%

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 2011	May 2010	April 2009	April 2008 *)
Total Acid Number	--	--	--	n.e.
Total Base Number	++	--	--	--
Colour	++	++	++	n.e.
Conradson Carbon Residue	++	++	++	++
Density @ 15 °C	--	--	--	--
Flash Point PMcc	++	++	+	--
Flash Point COC	++	++	++	--
Kinematic Viscosity @ 40 °C	--	--	--	--
Kinematic Viscosity @ 100 °C	--	--	--	--
Stabinger Viscosity @ 40 °C	--	--	--	n.e.
Nitrogen	--	--	--	--
Pour Point, manual	++	++	-	--
Pour Point, automated	+/-	++	n.e.	n.e.
Sulphated Ash	++	++	+/-	++
Sulphur	--	--	--	++
Water	++	++	--	n.e.
Calcium	+/-	+/-	+	(++)
Phosphorus	--	--	--	(+/-)
Zinc	--	--	+	(--)

Table 5: comparison determinations against the standard

*) no regular Lubrication Oil but pure Additive

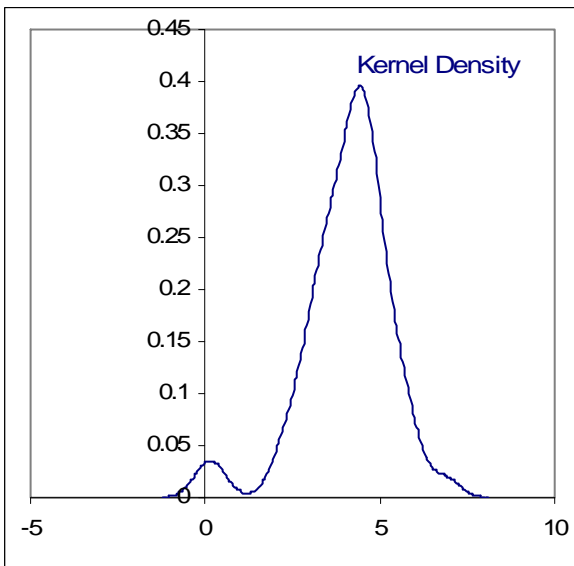
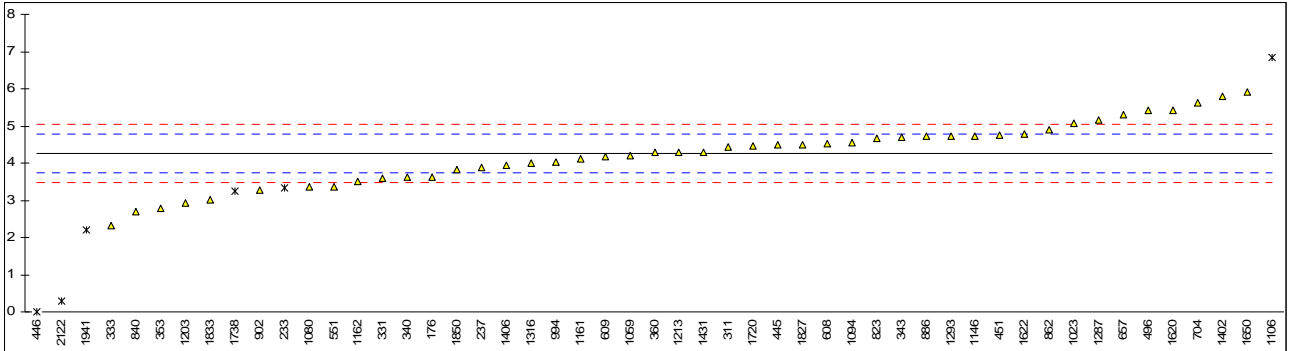
The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

- ++: 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 #11041; results in mg KOH/g**

lab	method	value	mark	z(targ)	remarks
128		----		----	
176	D664	3.6275		-2.39	
233	D974	3.33	ex	-3.51	Result excluded as test method is not equivalent
237	D664	3.87		-1.47	
252		----		----	
254		----		----	
255		----		----	
311	D664	4.44		0.68	
315		----		----	
318		----		----	
331	D664	3.59		-2.53	
333	D664	2.31		-7.36	
340	D664	3.61		-2.46	
343	D664	4.71		1.70	
353	IP177	2.794		-5.54	
357		----		----	
360	D664	4.281		0.08	
396		----		----	
432		----		----	
445	D664	4.48		0.83	
446	D974	0.0078	ex	-16.05	Result excluded as test method is not equivalent
450		----		----	
451	D664mod	4.76		1.89	
473		----		----	
496	D664	5.406		4.32	
551	D664	3.36		-3.40	
593		----		----	
608	D664	4.518		0.97	
609	D664	4.1756		-0.32	
614		----		----	
657	D664	5.30		3.92	
663		----		----	
704	D664	5.625		5.15	
823	D664	4.66		1.51	
840	D664	2.699		-5.89	
862	D664	4.886		2.36	
875		----		----	
886	D664	4.715		1.72	
902	D664	3.28		-3.70	
912		----		----	
963		----		----	
994	D664	4.02		-0.91	
1013		----		----	
1017		----		----	
1023	D664	5.07		3.06	
1059	ISO6619	4.20		-0.23	
1080	D664	3.35		-3.44	
1094	D664	4.5637		1.14	
1106	D664	6.8543	G(0.01)	9.79	
1146	D664	4.73		1.77	
1161	D664	4.115		-0.55	
1162	D664	3.52		-2.80	
1173		----		----	
1203	D664	2.92		-5.06	
1213	D664	4.3		0.15	
1231		----		----	
1235		----		----	
1287	D664	5.16		3.40	
1293	ISO12634	4.721		1.74	
1316	D664	4.01		-0.95	
1349		----		----	
1402	D664	5.8		5.81	
1406	D664	3.95		-1.17	
1428		----		----	
1431	D664	4.30		0.15	
1448		----		----	
1526		----		----	
1613		----		----	
1620	D664	5.41		4.34	
1622	D664	4.78		1.96	
1650	D664	5.91		6.23	
1660		----		----	
1720	D664	4.45	C	0.72	First reported 1.16
1738	D974	3.25	ex	-3.81	Result excluded as test method is not equivalent

1800		-----		-----	
1827	D664	4.481	C	0.83	First reported 7.066
1833	D664	3.02		-4.68	
1850	ISO6619	3.84		-1.59	
1941	ISO6618	2.19	ex	-7.82	Result excluded as test method is not equivalent
2122	IP177	0.28	G(0.01)	-15.03	
normality	OK				
n	45				
outliers	2				
mean (n)	4.260				
st.dev. (n)	0.8407				
R(calc.)	2.354				
R(D664:09a)	0.742				

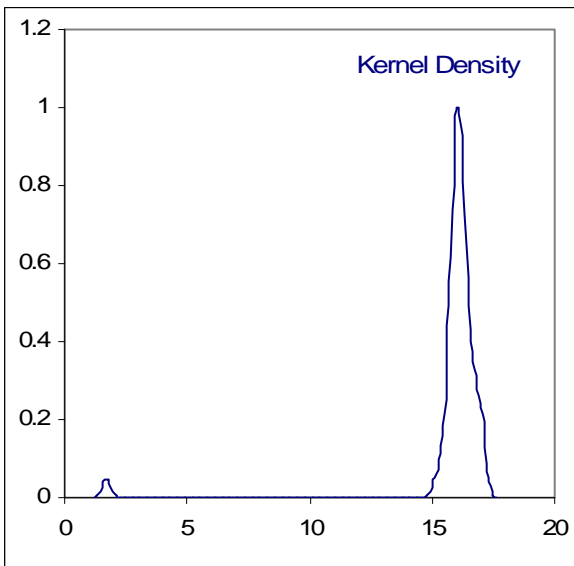
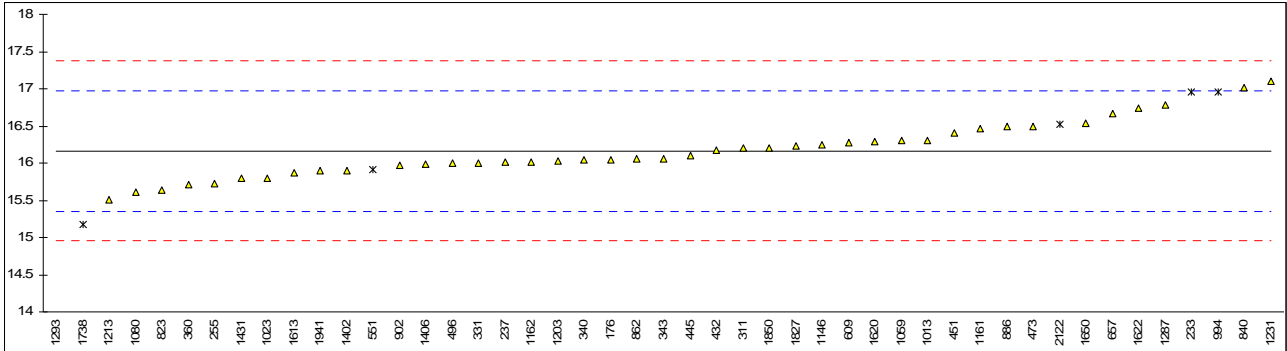


Determination of Base Number (Total) on sample #11041; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
128		----		----	
176	D2896	16.05	C	-0.28	First reported 14.33
233	D974	16.95	ex	1.95	Result excluded as test method is not equivalent
237	D2896	16.01		-0.38	
252		----		----	
254		----		----	
255	D2896	15.73		-1.07	
311	D2896	16.2		0.09	
315		----		----	
318		----		----	
331	D2896	16.0		-0.40	
333		----		----	
340	D2896	16.04		-0.30	
343	D2896	16.06		-0.25	
353		----		----	
357		----		----	
360	D2896	15.717		-1.10	
396		----		----	
432	D2896	16.167		0.01	
445	D2896	16.1		-0.15	
446		----		----	
450		----		----	
451	D2896mod	16.4		0.59	
473	D2896	16.4971		0.83	
496	D2896	15.994		-0.42	
551	D4739	15.91	ex	-0.62	Result excluded as test method is not equivalent
593		----		----	
608		----		----	
609	D2896	16.2716		0.27	
614		----		----	
657	D2896	16.66		1.23	
663		----		----	
704		----		----	
823	D2896	15.64		-1.29	
840	D2896	17.02		2.12	
862	D2896	16.051		-0.28	
875		----		----	
886	D2896	16.491		0.81	
902	D2896	15.97		-0.48	
912		----		----	
963		----		----	
994	D4739	16.96	ex	1.97	Result excluded as test method is not equivalent
1013	D2896	16.3		0.34	
1017		----		----	
1023	D2896	15.8		-0.90	
1059	ISO3771	16.3		0.34	
1080	D2896	15.61		-1.37	
1094		----		----	
1106		----		----	
1146	D2896	16.24		0.19	
1161	D2896	16.46		0.74	
1162	D2896	16.01		-0.38	
1173		----		----	
1203	ISO3771	16.03		-0.33	
1213	D2896	15.51		-1.61	
1231	D2896	17.10		2.32	
1235		----		----	
1287	D2896	16.78		1.53	
1293	ISO12634	1.714	C,ex	-35.76	Fr. 6.882. Result excluded as test method is not equivalent
1316		----		----	
1349		----		----	
1402	D2896	15.9		-0.65	
1406	D2896	15.98		-0.45	
1428		----		----	
1431	D2896	15.79		-0.92	
1448		----		----	
1526		----		----	
1613	D2896	15.87		-0.72	
1620	D2896	16.29	C	0.32	First reported 18.10
1622	D2896	16.74		1.43	
1650	D2896	16.54		0.93	
1660		----		----	
1720		----		----	
1738	D4739	15.18	ex	-2.43	Result excluded as test method is not equivalent

1800		----		----	
1827	D2896	16.232		0.17	
1833		----		----	
1850	ISO3771	16.21		0.12	
1941	ISO3771	15.9		-0.65	
2122	IP400	16.52	ex	0.88	Result excluded as test method is not equivalent

normality OK
 n 41
 outliers 0
 mean (n) 16.162
 st.dev. (n) 0.3637
 R(calc.) 1.018
 R(D2896:07a) 1.131

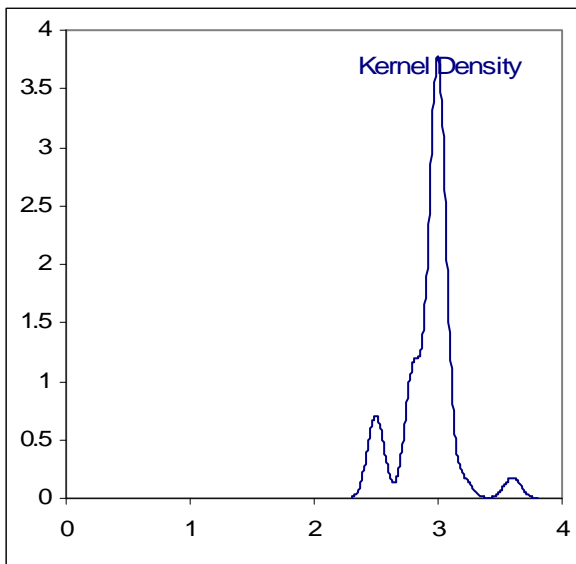
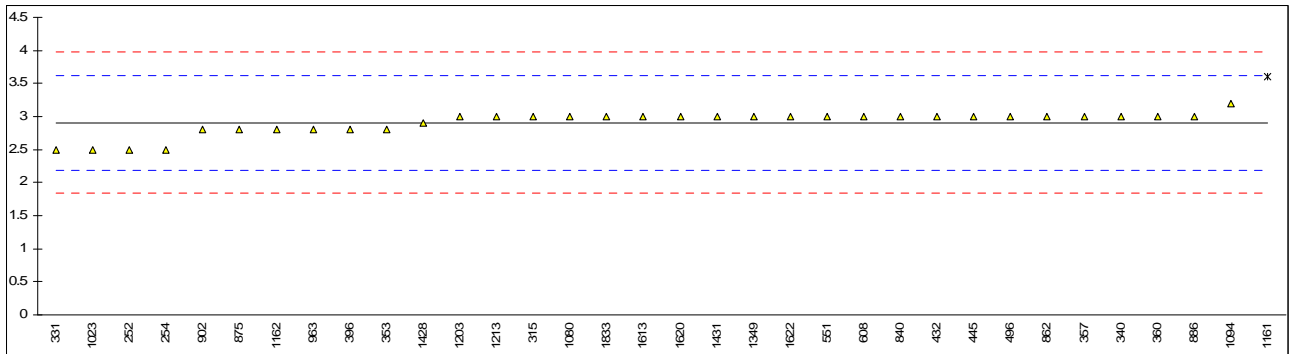


Determination of Colour on sample #11041

lab	method	value	mark	z(targ)	remarks
128		----		----	
176	D1500	L3.0		----	
233		----		----	
237	D1500	L3.0		----	
252	D1500	2.5		-1.14	
254	D1500	2.5		-1.14	
255		----		----	
311	D1500	L3.0		----	
315	D1500	3.0		0.26	
318		----		----	
331	D1500	2.5		-1.14	
333		----		----	
340	D1500	3.0		0.26	
343	D1500	L3		----	
353	D6045	2.8		-0.30	
357	D1500	3.0		0.26	
360	D1500	3.0		0.26	
396	D1500	2.8		-0.30	
432	D1500	3		0.26	
445	D1500	3.0		0.26	
446	D1500	L2.5		----	
450		----		----	
451		----		----	
473		----		----	
496	D1500	3.00		0.26	
551	D1500	3.0		0.26	
593		----		----	
608	D1500	3.0		0.26	
609	D1500	L3.0		----	
614		----		----	
657	D1500	L3.0		----	
663	D1500	L3.0		----	
704	D1500	L3.5		----	
823	D1500	L3.0		----	
840	D1500	3.0		0.26	
862	D1500	3.0		0.26	
875	D6045	2.8		-0.30	
886	D1500	3.0		0.26	
902	D1500	2.8		-0.30	
912	D1500	L3.0		----	
963	D1500	2.8		-0.30	
994	D1500	L3.0		----	
1013	D1500	L3.0		----	
1017		----		----	
1023	D1500	2.5		-1.14	
1059	D1500	L3.0		----	
1080	D1500	3.0		0.26	
1094	In house	3.2		0.82	
1106		----		----	
1146		----		----	
1161	D1500	3.6	G(0.05)	1.94	
1162	D1500	2.8		-0.30	
1173		----		----	
1203	D1500	3.0		0.26	
1213	D1500	3.0		0.26	
1231		----		----	
1235	ISO2049	L3.0		----	
1287		----		----	
1293		----		----	
1316		----		----	
1349	D1500	3.0		0.26	
1402	D1500	L3.5		----	
1406	D1500	L3.0		----	
1428	D6045	2.9		-0.02	
1431	D1500	3.0		0.26	
1448		----		----	
1526		----		----	
1613	D1500	3.0		0.26	
1620	D1500	3.0		0.26	
1622	D1500	3.0		0.26	
1650		----		----	
1660		----		----	
1720		----		----	
1738		----		----	

1800		----	----
1827		----	----
1833	D1500	3.0	0.26
1850	ISO2049	L3.0	----
1941	ISO2049	L3.0	----
2122		----	----

normality not OK
 n 33
 outliers 1
 mean (n) 2.91
 st.dev. (n) 0.177
 R(calc.) 0.49
 R(D1500:07) 1.00

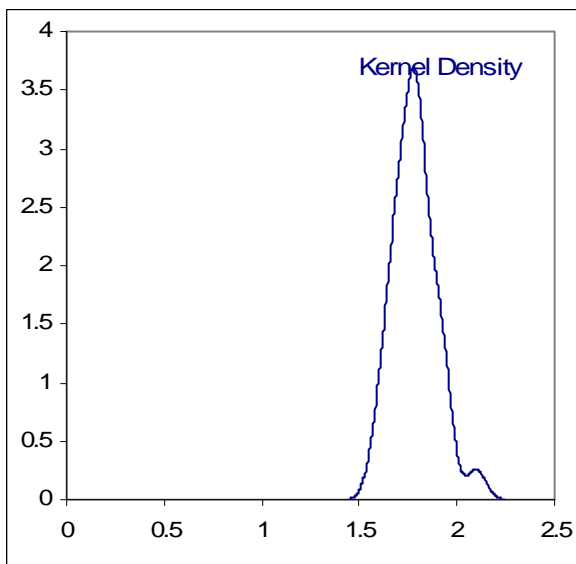
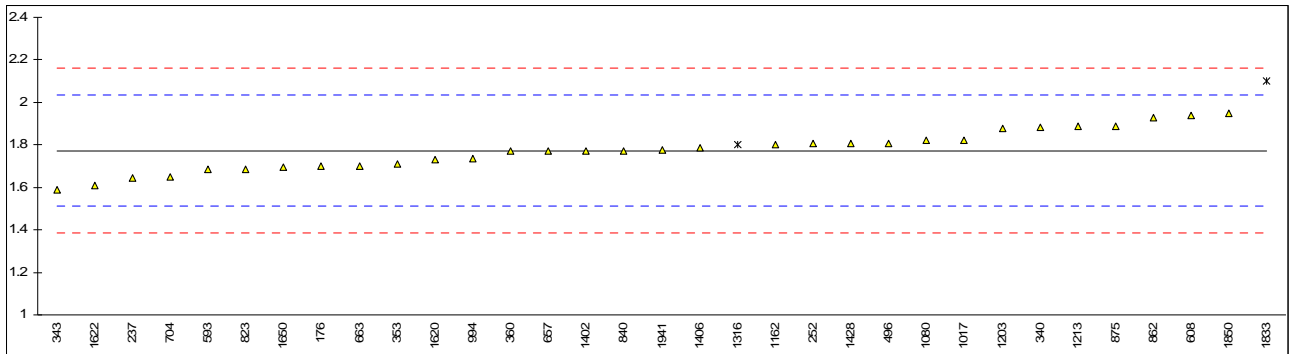


Determination of Conradson Carbon Residue on sample #11041; results in %M/M

lab	method	value	mark	z(targ)	remarks
128		----		----	
176	D189	1.70		-0.57	
233		----		----	
237	D189	1.643		-1.01	
252	D189	1.805		0.24	
254		----		----	
255		----		----	
311		----		----	
315		----		----	
318		----		----	
331		----		----	
333		----		----	
340	D4530	1.882		0.83	
343	D4530	1.587		-1.44	
353	IP13	1.711		-0.49	
357		----		----	
360	D189	1.77		-0.03	
396		----		----	
432		----		----	
445		----		----	
446		----		----	
450		----		----	
451		----		----	
473		----		----	
496	D4530	1.809		0.27	
551		----		----	
593	D189	1.6823		-0.71	
608	D4530	1.937		1.25	
609		----		----	
614		----		----	
657	D4530	1.77		-0.03	
663	D189	1.702		-0.56	
704	D189	1.647		-0.98	
823	D189	1.684		-0.70	
840	D189	1.77		-0.03	
862	D189	1.926		1.17	
875	D4530	1.89		0.89	
886		----		----	
902		----		----	
912		----		----	
963		----		----	
994	D189	1.734		-0.31	
1013		----		----	
1017	D4530	1.82		0.35	
1023		----		----	
1059		----		----	
1080	D4530	1.82		0.35	
1094		----		----	
1106		----		----	
1146		----		----	
1161		----		----	
1162	D4530	1.801		0.21	
1173		----		----	
1203	ISO10370	1.88		0.81	
1213	D4530	1.886		0.86	
1231		----		----	
1235		----		----	
1287		----		----	
1293		----		----	
1316	D524	1.8	C,ex	0.20	First reported 2.3, result excluded as test method is not equivalent
1349		----		----	
1402	D189	1.77		-0.03	
1406	D4530	1.784		0.08	
1428	ISO10370	1.805		0.24	
1431		----		----	
1448		----		----	
1526		----		----	
1613		----		----	
1620	D189	1.73		-0.34	
1622	D189	1.61		-1.27	
1650	D189	1.694		-0.62	
1660		----		----	
1720		----		----	
1738		----		----	

1800	-----			-----	
1827	-----			-----	
1833	D524	2.1	ex	2.51	Result excluded as test method is not equivalent
1850	ISO6615	1.95		1.35	
1941	ISO10370	1.777		0.02	
2122	-----			-----	

		<u>Only ASTM D189</u>	<u>Only ASTM D4530</u>
normality	OK	OK	OK
n	31	19	14
outliers	0	0	0
mean (n)	1.773	1.749	1.818
st.dev. (n)	0.0956	0.0936	0.0836
R(calc.)	0.268	0.262	0.234
R(D189:06e2)	0.363	0.359	0.226

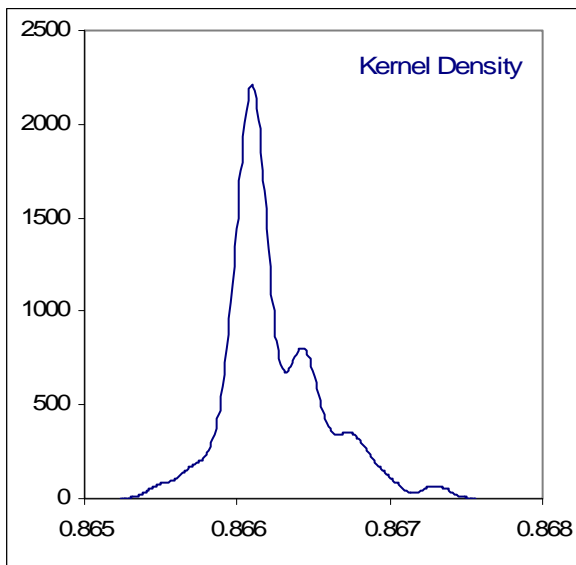
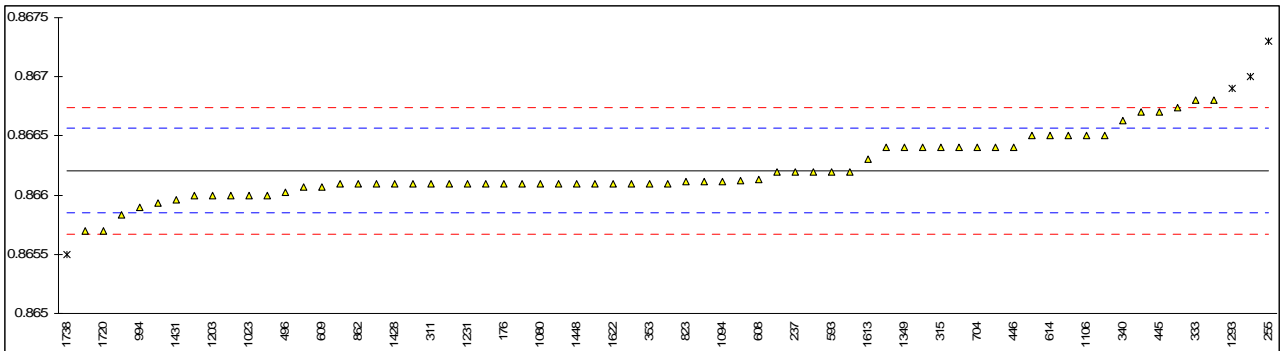


Determination of Density @ 15°C on sample #11041; results in kg/L

lab	method	value	mark	z(targ)	remarks
128		-----		-----	
176	D4052	0.8661		-0.60	
233		-----		-----	
237	D4052	0.8662		-0.04	
252		-----		-----	
254	D4052	0.8660		-1.16	
255	D1298	0.8673	C,G(0.05)	6.12	First reported 0.8682
311	D4052	0.8661		-0.60	
315	D4052	0.8664		1.08	
318		-----		-----	
331		-----		-----	
333	D4052	0.8668		3.32	
340	D4052	0.86663		2.37	
343	D4052	0.86612		-0.49	
353	IP365	0.8661		-0.60	
357	D4052	0.8665		1.64	
360	D4052	0.8662		-0.04	
396	D4052	0.8665		1.64	
432	D4052	0.86674		2.98	
445	D4052	0.8667		2.76	
446	D4052	0.8664		1.08	
450		-----		-----	
451	D4052	0.867	C,G(0.05)	4.44	First reported 867
473		-----		-----	
496	D4052	0.86602		-1.05	
551	D4052	0.8657		-2.84	
593	D4052	0.8662	C	-0.04	First reported 0.8670
608	D4052	0.86613		-0.43	
609	D4052	0.86607		-0.77	
614	D4052	0.8665		1.64	
657	D4052	0.8660		-1.16	
663	D4052	0.8661		-0.60	
704	D4052	0.8664		1.08	
823	D4052	0.86611		-0.54	
840	D4052	0.86607		-0.77	
862	D4052	0.86610		-0.60	
875	D4052	0.8661		-0.60	
886	D4052	0.8667		2.76	
902	D4052	0.86611		-0.54	
912	D4052	0.8661		-0.60	
963	D4052	0.8664		1.08	
994	D4052	0.8659	C	-1.72	First reported 0.8670
1013	D4052	0.8662		-0.04	
1017	D4052	0.8668		3.32	
1023	D4052	0.8660		-1.16	
1059	D4052	0.8664		1.08	
1080	D4052	0.8661		-0.60	
1094	D4052	0.86611		-0.54	
1106	D5002	0.86650		1.64	
1146	D4052	0.86593		-1.55	
1161	D4052	0.8665	C	1.64	First reported 866.48
1162		-----		-----	
1173		-----		-----	
1203	D4052	0.8660		-1.16	
1213	D4052	0.8661		-0.60	
1231	D4052	0.8661	C	-0.60	First reported 866.07
1235	ISO12185	0.86610		-0.60	
1287	D4052	0.86583		-2.11	
1293	D5002/ISO12185	0.8669	C,G(0.05)	3.88	First reported 0.8674
1316	D4052	0.8661		-0.60	
1349	IP365	0.8664	C	1.08	First reported 866.4
1402	D4052	0.8664		1.08	
1406	ISO12185	0.86610	C	-0.60	First reported 866.10
1428	ISO12185	0.8661		-0.60	
1431	D4052	0.86596		-1.38	
1448	D4052	0.86610		-0.60	
1526	D5002	0.866		-1.16	
1613	D4052	0.8663		0.52	
1620	D4052	0.8662		-0.04	
1622	D4052	0.8661		-0.60	
1650	D4052	0.8661	C	-0.60	First reported 866.1
1660		-----		-----	
1720	D4052	0.8657		-2.84	
1738	D1298	0.8655	G(0.05)	-3.96	

1800		----	----
1827		----	----
1833	D4052	0.8661	-0.60
1850	ISO3675	0.8664	1.08
1941	D4052	0.8661	-0.60
2122		----	----

normality not OK
n 63
outliers 4
mean (n) 0.86621
st.dev. (n) 0.000248
R(calc.) 0.00070
R(D4052:09) 0.00050

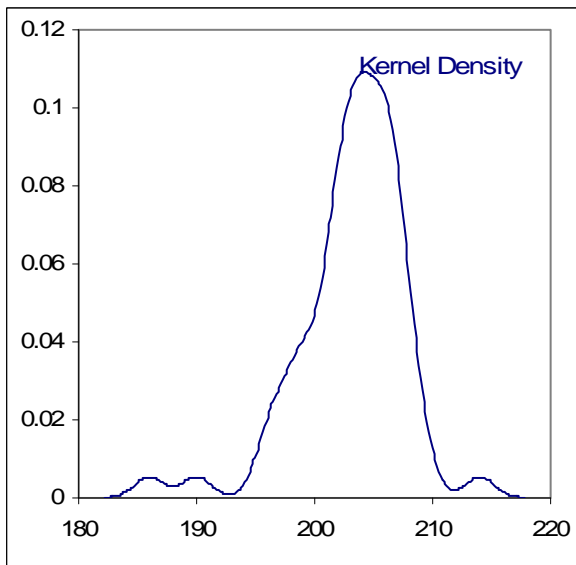
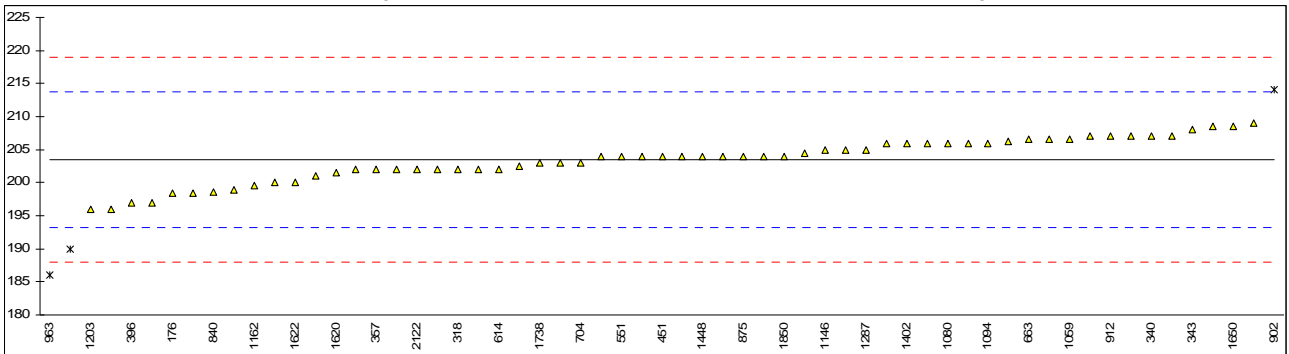


Determination of Flash Point PMcc on sample #11041; results in °C

lab	method	value	mark	z(targ)	remarks
128		----		----	
176	D93-AF	198.5		-0.96	
233		----		----	
237	D93-MF	190.0	G(0.05)	-2.61	
252	D93-ME	196.0		-1.44	
254		----		----	
255		----		----	
311		----		----	
315		----		----	
318	INH-III	202		-0.28	
331	D93-AE	209		1.08	
333	D93-AF	202.5		-0.18	
340	D93-AF	207.0		0.69	
343	D93-AE	208	C	0.88	First reported 190
353	IP34ME	202.0		-0.28	
357	D93-AE	202.0		-0.28	
360	D93-AE	203.0		-0.09	
396	D93-MF	197		-1.25	
432	D93-AE	206		0.49	
445	D93	204.0		0.11	
446	D93-AF	205.9		0.47	
450		----		----	
451	D93-AE	204		0.11	
473	D93-AE	204.0		0.11	
496		----		----	
551	D93-	204		0.11	
593	D93-ME	205		0.30	
608	D93-	206.0		0.49	
609		----		----	
614	D93-MF	202.07		-0.27	
657	D93-AF	204.0		0.11	
663	D93-MF	206.5		0.59	
704	D93-MF	203.0		-0.09	
823	D93-AF	202.0		-0.28	
840	D93-MF	198.6		-0.94	
862	D93-AF	204.0		0.11	
875	D93-MF	204.0		0.11	
886	D93-AF	206.2		0.53	
902	D93-ME	214	C,G(0.05)	2.04	First reported 224
912	D93-AE	207		0.69	
963	D93-MF	186.0	C,G(0.01)	-3.38	First reported 188.0
994	D93-MF	198.5		-0.96	
1013	D93-	197.0		-1.25	
1017	D93-AE	201.0		-0.48	
1023	D93-AE	207		0.69	
1059	ISO2719AE	206.5		0.59	
1080	D93-AE206	206		0.49	
1094	D93-AF	206.0		0.49	
1106	D93-AE	207.0		0.69	
1146	IN HOUSE-AE	205.0		0.30	
1161	D93-AF	206.5		0.59	
1162	D93-MF	199.5		-0.77	
1173	IP34-MF	198.95		-0.87	
1203	D93-	196		-1.44	
1213	D93-MF	202		-0.28	
1231	D93-AE	204.0		0.11	
1235		----		----	
1287	D93-AE	205		0.30	
1293	D6450-MF	207		0.69	
1316		----		----	
1349		----		----	
1402	D93-AE	206	C	0.49	First reported 220
1406		----		----	
1428	D93-A	204.5		0.20	
1431	D93-AF	208.5		0.98	
1448	D93-AE	204.0		0.11	
1526		----		----	
1613		----		----	
1620	D93-ME	201.5		-0.38	
1622	D93-ME	200.0		-0.67	
1650	D93-AE	208.5		0.98	
1660		----		----	
1720	D93-AF	200.0		-0.67	
1738	D93-	203		-0.09	

1800		----	----
1827		----	----
1833	D93-MF	202	-0.28
1850	ISO2719AE	204	0.11
1941		----	----
2122	D93-MF	202	-0.28
normality		not OK	
n		58	
outliers		3	
mean (n)		203.45	
st.dev. (n)		3.258	
R(calc.)		9.12	
R(D93:10, meth A)		14.45	

M	= Manual mode	A	= Automated mode
MF	= Manual mode, flame ignition	AF	= Automated mode, flame ignition
ME	= Manual mode, electric ignition	AE	= Automated mode, electric ignition

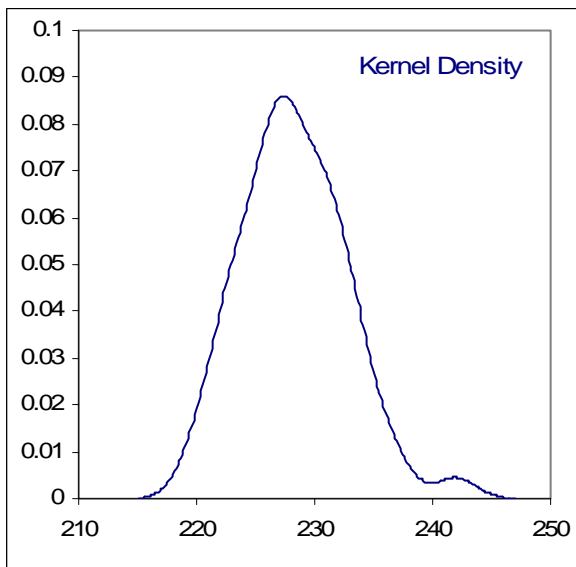
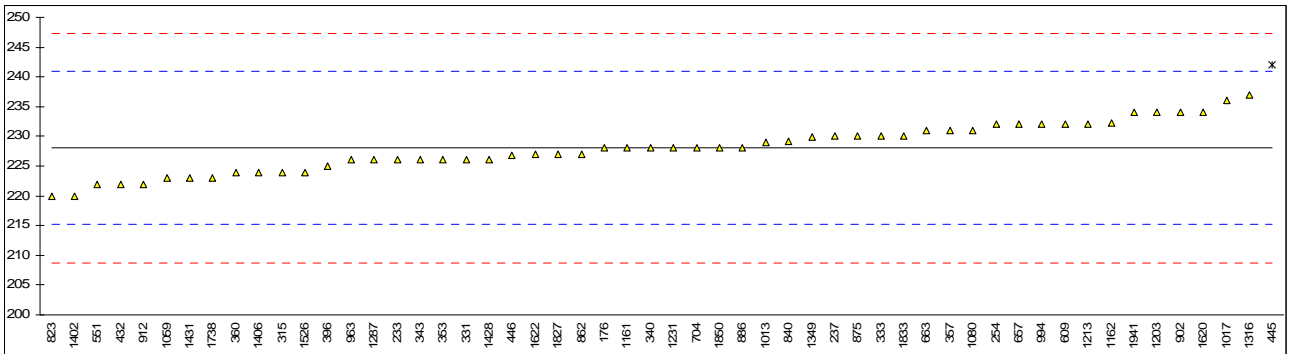


Determination of Flash Point C.O.C. on sample #11041; results in °C

lab	method	value	mark	z(targ)	remarks
128		----		----	
176	D92	228		0.00	
233	D92	226		-0.31	
237	D92	230.0		0.31	
252		----		----	
254	D92	232.0		0.62	
255		----		----	
311		----		----	
315	D92	224		-0.62	
318		----		----	
331	D92	226		-0.31	
333	D92	230		0.31	
340	D92	228		0.00	
343	D92	226		-0.31	
353	IP36	226.0		-0.31	
357	D92	231		0.47	
360	D92	224		-0.62	
396	D92	225		-0.47	
432	D92	222		-0.93	
445	D92	242	G(0.05)	2.18	
446	D92	226.86		-0.18	
450		----		----	
451		----		----	
473		----		----	
496		----		----	
551	D92	222		-0.93	
593		----		----	
608		----		----	
609	D3828	232	C	0.62	First reported 194
614		----		----	
657	D92	232		0.62	
663	D92	231		0.47	
704	D92	228		0.00	
823	D92	220		-1.25	
840	D92	229.2		0.19	
862	D92	227		-0.16	
875	D92	230		0.31	
886	D92	228.0		0.00	
902	D92	234		0.93	
912	D92	222		-0.93	
963	D92	226		-0.31	
994	D92	232.0		0.62	
1013	D92	229		0.15	
1017	D92	236		1.24	
1023		----		----	
1059	ISO2592	223		-0.78	
1080	D92-AF	231		0.47	
1094		----		----	
1106		----		----	
1146		----		----	
1161	D92	228.0		0.00	
1162	D92	232.3		0.67	
1173		----		----	
1203	D92	234		0.93	
1213	D92	232		0.62	
1231	D92	228		0.00	
1235		----		----	
1287	D92	226		-0.31	
1293		----		----	
1316	D92	237		1.40	
1349	D92	229.9		0.29	
1402	D92	220		-1.25	
1406	D92	224		-0.62	
1428	D92	226		-0.31	
1431	D92	223		-0.78	
1448		----		----	
1526	D92-AF	224		-0.62	
1613		----		----	
1620	D92	234		0.93	
1622	D92	227		-0.16	
1650		----		----	
1660		----		----	
1720		----		----	
1738	D92	223		-0.78	

1800		-----	-----
1827	D92	227	-0.16
1833	D92	230	0.31
1850	ISO2592	228	0.00
1941	ISO2592	234	0.93
2122		-----	-----

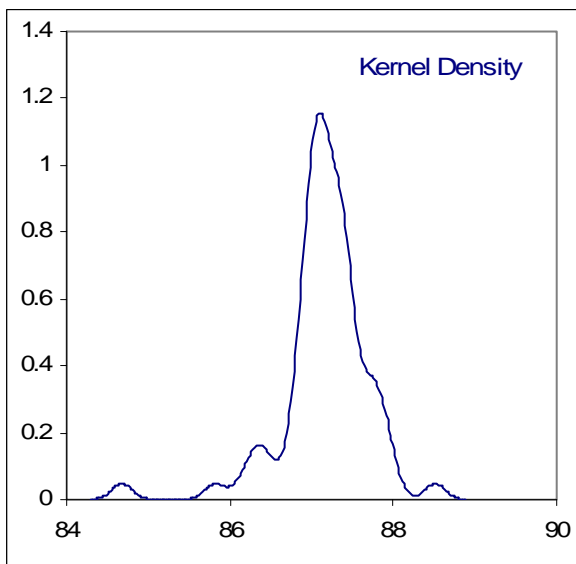
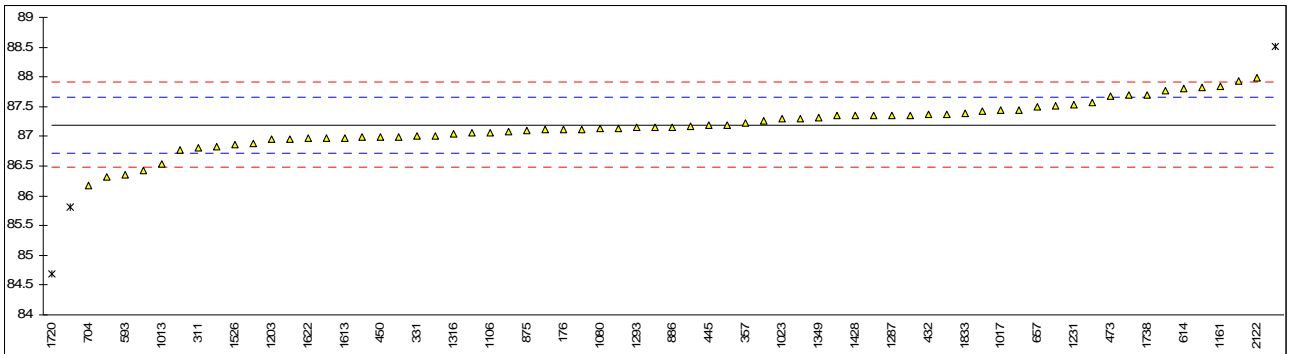
normality	OK
n	53
outliers	1
mean (n)	228.00
st.dev. (n)	4.050
R(calc.)	11.34
R(D92:10)	18.00



Determination of Kinematic Viscosity @ 40°C on sample #11041; results in mm²/s

lab	method	value	mark	z(targ)	remarks
128		----		----	
176	D445	87.12		-0.31	
233	D7279	87.37		0.74	
237	D445	87.18		-0.06	
252	D445	87.6953		2.12	
254		----		----	
255	D445	88.51	C,G(0.01)	5.56	First reported 91.65
311	D445	86.81		-1.62	
315	D445	86.763		-1.82	
318		----		----	
331	D7279	87.00		-0.82	
333	D445	87.17		-0.10	
340	D445	87.081		-0.48	
343	D445	87.434		1.01	
353	IP71	87.936		3.13	
357	D445	87.23		0.15	
360	D445	87.344		0.63	
396		----		----	
432	D445	87.370		0.74	
445	D445	87.18		-0.06	
446	D445	85.820	G(0.05)	-5.81	
450	D445	86.99		-0.86	
451	D7279	87.82		2.64	
473	D445	87.684		2.07	
496	D445	87.156		-0.16	
551	D445	86.82		-1.58	
593	D445	86.35		-3.57	
608	D445	87.123		-0.30	
609		----		----	
614	D445	87.8		2.56	
657	D445	87.49		1.25	
663		----		----	
704	D445	86.170		-4.33	
823	D445	86.885		-1.31	
840	D445	86.998		-0.83	
862	D445	87.138		-0.24	
875	D445	87.10		-0.40	
886	D445	87.16		-0.14	
902	D445	87.00	C	-0.82	First reported 89.29
912	D445	87.56		1.55	
963	D445	86.42		-3.27	
994	D445	87.51		1.33	
1013	D445	86.54		-2.76	
1017	D445	87.4338		1.01	
1023	D445	87.29		0.41	
1059		----		----	
1080	D445	87.13		-0.27	
1094	D445	86.9837		-0.89	
1106	D445	87.07		-0.52	
1146	D445	87.355		0.68	
1161	D445	87.84		2.73	
1162	D445	86.973		-0.93	
1173	IP71	86.31		-3.74	
1203	D445	86.95		-1.03	
1213	D445	86.95		-1.03	
1231	D445	87.54		1.46	
1235		----		----	
1287	D445	87.36		0.70	
1293	ISO3104	87.15	C	-0.19	First reported 89.84
1316	D445	87.04		-0.65	
1349	D445	87.3168		0.52	
1402	D445	87.36		0.70	
1406	D445	87.255		0.26	
1428	ISO3104	87.35		0.66	
1431		----		----	
1448		----		----	
1526	D445	86.865		-1.39	
1613	D445	86.98		-0.90	
1620	D445	87.30		0.45	
1622	D445	86.97		-0.95	
1650	D445	87.777		2.46	
1660		----		----	
1720	D445	84.68	G(0.01)	-10.62	
1738	D445	87.70	C	2.14	First reported 13.27

1800		-----	-----
1827	D445	87.06	-0.57
1833	D445	87.39	0.83
1850	ISO3104	87.42	0.95
1941	ISO3104	87.117	-0.33
2122	in house	87.98	3.32
normality		OK	
n		65	
outliers		3	
mean (n)		87.194	
st.dev. (n)		0.3776	
R(calc.)		1.057	
R(D445:11a)		0.663	



Determination of Kinematic Viscosity @ 100°C on sample #11041; results in mm²/s

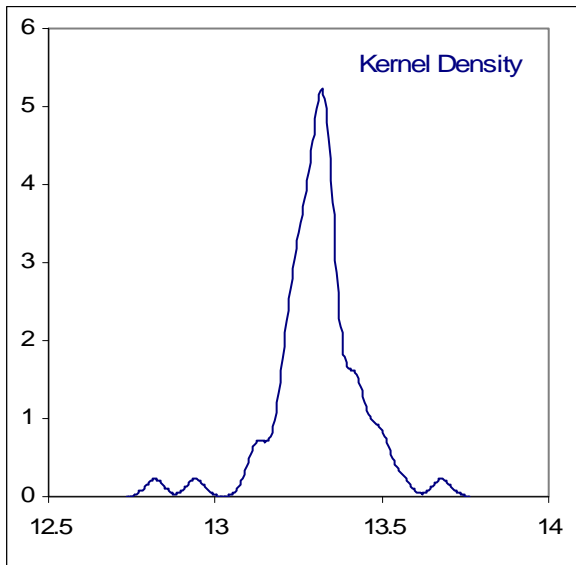
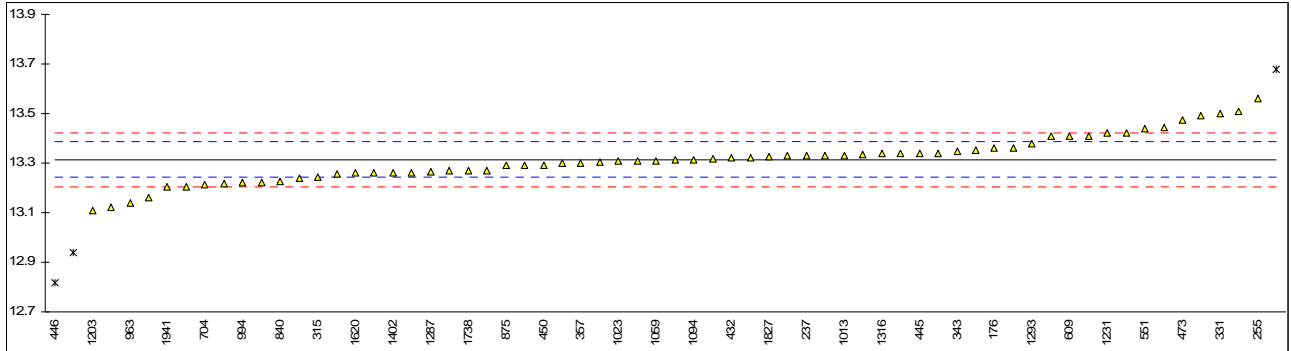
lab	method	value	mark	z(targ)	remarks
128		----		----	
176	D445	13.36		1.26	
233	D7279	12.94	G(0.05)	-10.36	
237	D445	13.33		0.43	
252	D445	13.2575		-1.58	
254		----		----	
255	D445	13.56	C	6.79	First reported 13.7
311	D445	13.26		-1.51	
315	D445	13.245		-1.92	
318		----		----	
331	D7279	13.50		5.13	
333	D445	13.36		1.26	
340	D445	13.239		-2.09	
343	D445	13.349		0.95	
353	IP71	13.262		-1.45	
357	D445	13.30		-0.40	
360	D445	13.305		-0.26	
396		----		----	
432	D445	13.32		0.15	
445	D445	13.34		0.71	
446	D445	12.818	G(0.01)	-13.74	
450	D445	13.29		-0.68	
451	D7279	13.68	G(0.05)	10.11	
473	D445	13.475		4.44	
496	D445	13.313		-0.04	
551	D445	13.44		3.47	
593	D445	13.12	C	-5.38	First reported 13.04
608		----		----	
609	D7042	13.41	C	2.64	First reported 13.539
614		----		----	
657	D445	13.41		2.64	
663		----		----	
704	D445	13.214		-2.78	
823	D445	13.218		-2.67	
840	D445	13.228		-2.39	
862	D445	13.268		-1.29	
875	D445	13.29		-0.68	
886	D445	13.33		0.43	
902	D445	13.34		0.71	
912	D445	13.22		-2.62	
963	D445	13.14	C	-4.83	First reported 13.79
994	D445	13.22		-2.62	
1013	D445	13.33		0.43	
1017	D445	13.4423		3.54	
1023	D445	13.31		-0.12	
1059	ISO3104	13.31		-0.12	
1080	D445	13.32		0.15	
1094	D445	13.3150		0.01	
1106		----		----	
1146	D445	13.333		0.51	
1161	D445	13.35		0.98	
1162	D445	13.163		-4.19	
1173		----		----	
1203	D445	13.11	C	-5.66	First reported 13.00
1213	D445	13.27		-1.23	
1231	D445	13.42		2.92	
1235		----		----	
1287	D445	13.264		-1.40	
1293	ISO3104	13.38		1.81	
1316	D445	13.34		0.71	
1349	D445	13.4207		2.94	
1402	D445	13.26		-1.51	
1406	D445	13.300		-0.40	
1428	ISO3104	13.33		0.43	
1431	D7042	13.41		2.64	
1448	D7042	13.205		-3.03	
1526		----		----	
1613	D445	13.49		4.86	
1620	D445	13.26	C	-1.51	First reported 13.04
1622	D445	13.29		-0.68	
1650	D445	13.310		-0.12	
1660		----		----	
1720		----		----	
1738	D445	13.27	C	-1.23	First reported 87.70

1800		-----	-----
1827	D445	13.325	0.29
1833	D445	13.318	0.10
1850	ISO3104	13.34	0.71
1941	ISO3104	13.205	-3.03
2122	in house	13.51	5.41

normality	not OK
n	63
outliers	3
mean (n)	13.315
st.dev. (n)	0.0920
R(calc.)	0.258
R(D445:11a)	0.101

Only ASTM D445 data:

normality	not OK
n	58
outliers	1
mean (n)	13.307
st.dev. (n)	0.0856
R(calc.)	0.240
R(D445:11a)	0.101

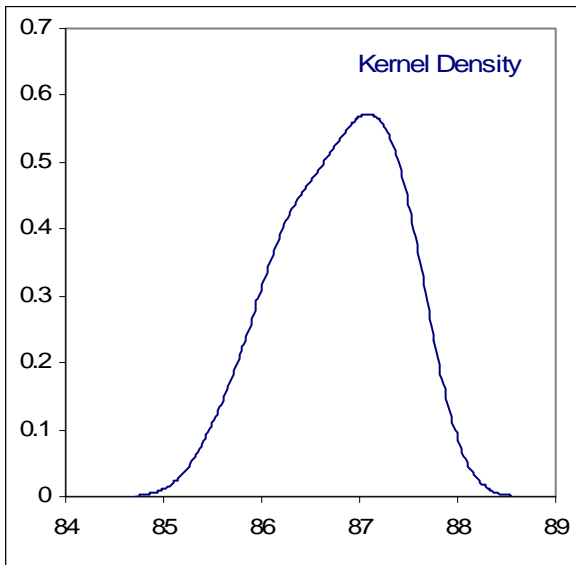
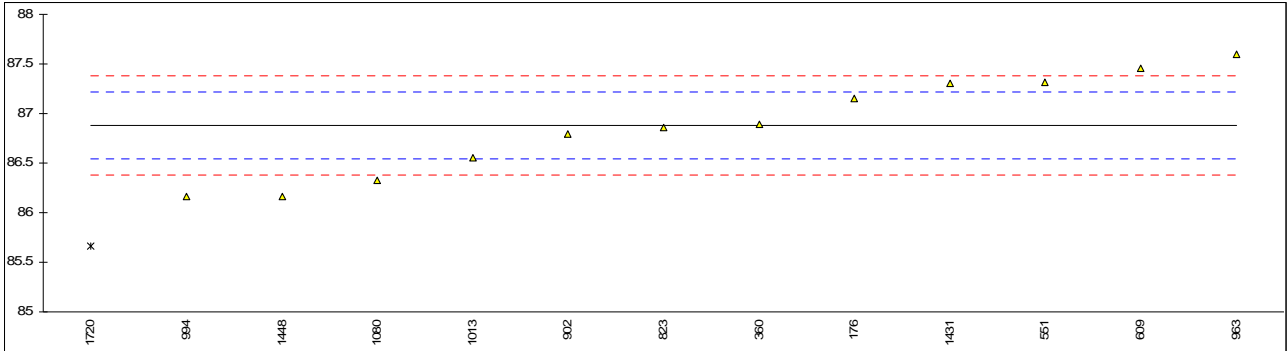


Determination of Viscosity Stabinger @ 40 °C on sample #11041; results in mm²/s

lab	method	value	mark	z(targ)	remarks
128		----		----	
176	D7042	87.15		1.61	
233		----		----	
237		----		----	
252		----		----	
254		----		----	
255		----		----	
311		----		----	
315		----		----	
318		----		----	
331		----		----	
333		----		----	
340		----		----	
343		----		----	
353		----		----	
357		----		----	
360	D7042	86.892		0.07	
396		----		----	
432		----		----	
445		----		----	
446		----		----	
450		----		----	
451		----		----	
473		----		----	
496		----		----	
551	D7042	87.31		2.56	
593		----		----	
608		----		----	
609	D7042	87.460		3.46	
614		----		----	
657		----		----	
663		----		----	
704		----		----	
823	D7042	86.859		-0.13	
840		----		----	
862		----		----	
875		----		----	
886		----		----	
902	D7042	86.79		-0.54	
912		----		----	
963	D7042	87.60		4.30	
994	D7042	86.16		-4.30	
1013	D7042	86.55		-1.97	
1017		----		----	
1023		----		----	
1059		----		----	
1080	D7042	86.33		-3.28	
1094		----		----	
1106		----		----	
1146		----		----	
1161		----		----	
1162		----		----	
1173		----		----	
1203		----		----	
1213		----		----	
1231		----		----	
1235		----		----	
1287		----		----	
1293		----		----	
1316		----		----	
1349		----		----	
1402		----		----	
1406		----		----	
1428		----		----	
1431	D7042	87.30		2.51	
1448	D7042	86.162		-4.29	
1526		----		----	
1613		----		----	
1620		----		----	
1622		----		----	
1650		----		----	
1660		----		----	
1720	D7042	85.663	C,G(0.05)	-7.26	First reported 84.49
1738		----		----	

1800	----	----
1827	----	----
1833	----	----
1850	----	----
1941	----	----
2122	----	----

normality	OK
n	12
outliers	1
mean (n)	86.880
st.dev. (n)	0.4994
R(calc.)	1.398
R(D7042:11)	0.469

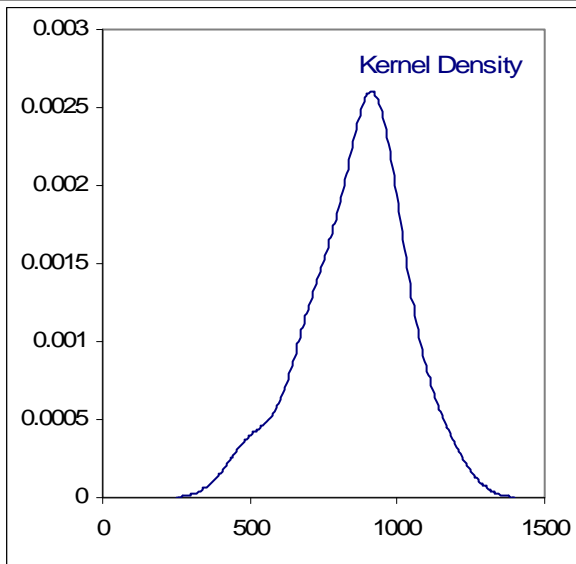
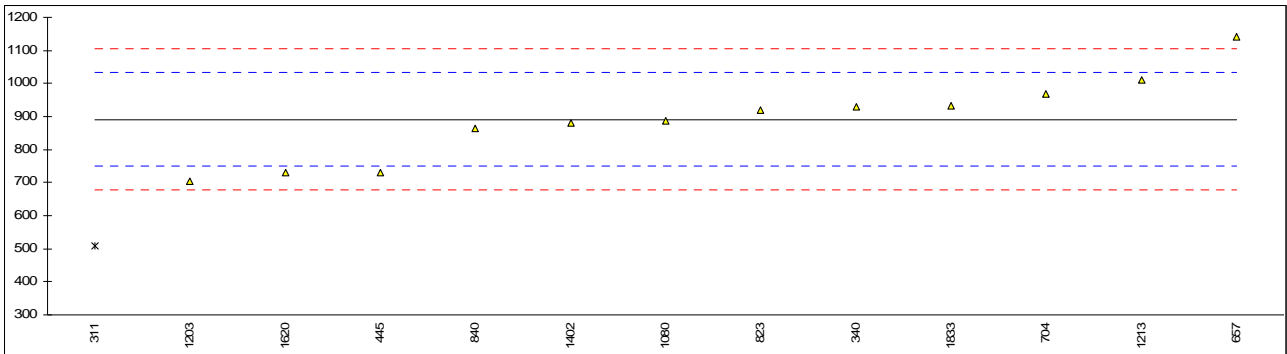


Determination of Nitrogen on sample #11041; results in mg/kg

lab	method	value	mark	z(targ)	remarks
128		----		----	
176		----		----	
233		----		----	
237		----		----	
252		----		----	
254		----		----	
255		----		----	
311	D4629	510	G(0.05)	-5.34	
315		----		----	
318		----		----	
331		----		----	
333		----		----	
340	D3228	930	C	0.54	First reported 0.093
343		----		----	
353		----		----	
357		----		----	
360		----		----	
396		----		----	
432		----		----	
445	D5762	731		-2.24	
446		----		----	
450		----		----	
451		----		----	
473		----		----	
496		----		----	
551		----		----	
593		----		----	
608		----		----	
609		----		----	
614		----		----	
657	D3228	1140		3.48	
663		----		----	
704	D3228	969		1.09	
823	D5762	919.5		0.40	
840	D3228	864.0		-0.38	
862		----		----	
875		----		----	
886		----		----	
902		----		----	
912		----		----	
963		----		----	
994		----		----	
1013		----		----	
1017		----		----	
1023		----		----	
1059		----		----	
1080	D4629	886		-0.07	
1094		----		----	
1106		----		----	
1146		----		----	
1161		----		----	
1162		----		----	
1173		----		----	
1203	D3228	704		-2.62	
1213	D3228	1010		1.66	
1231		----		----	
1235		----		----	
1287		----		----	
1293		----		----	
1316		----		----	
1349		----		----	
1402	D3228	880		-0.16	
1406		----		----	
1428		----		----	
1431		----		----	
1448		----		----	
1526		----		----	
1613		----		----	
1620	D4629	729		-2.27	
1622		----		----	
1650		----		----	
1660		----		----	
1720		----		----	
1738		----		----	

1800	----	----	
1827	----	----	
1833	D3228	932.5	0.58
1850	----	----	
1941	----	----	
2122	----	----	

normality	OK
n	12
outliers	1
mean (n)	891.3
st.dev. (n)	125.77
R(calc.)	352.1
R(D3228:08)	200.0

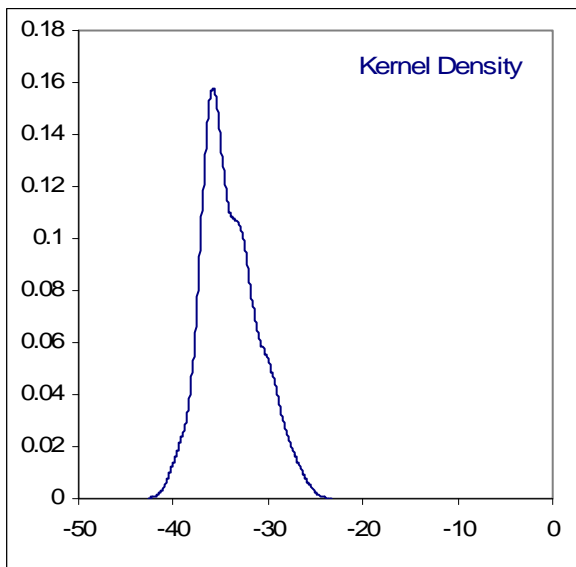
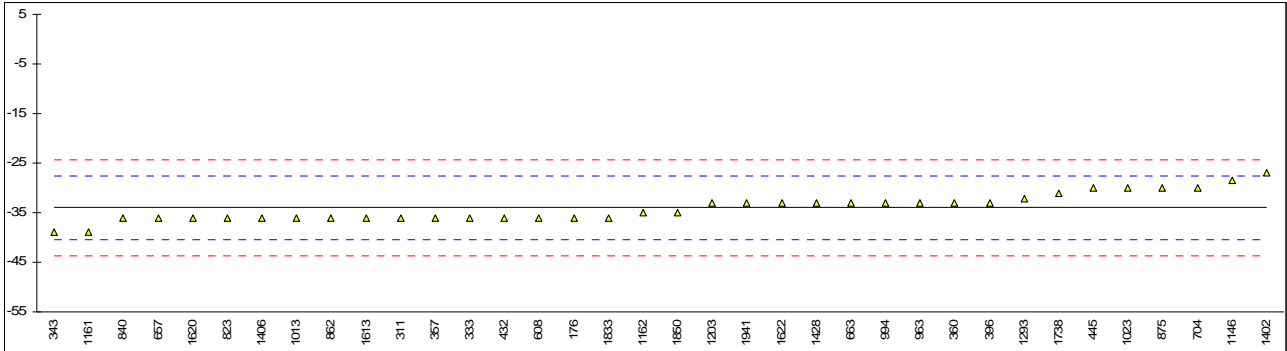


Determination of Pour Point (Manual) on sample #11041; results in °C

lab	method	value	mark	z(targ)	remarks
128		----		----	
176	D97	-36		-0.62	
233		----		----	
237	D97	<-24		----	
252		----		----	
254		----		----	
255		----		----	
311	D5950	-36		-0.62	
315		----		----	
318		----		----	
331		----		----	
333	D97	-36		-0.62	
340		----		----	
343	D97	-39		-1.56	
353		----		----	
357	D97	-36		-0.62	
360	D97	-33		0.31	
396	D97	-33		0.31	
432	D97	-36		-0.62	
445	D97	-30		1.24	
446		----		----	
450		----		----	
451		----		----	
473		----		----	
496		----		----	
551		----		----	
593		----		----	
608	D97	-36		-0.62	
609		----		----	
614		----		----	
657	D97	-36		-0.62	
663	D97	-33		0.31	
704	D97	-30		1.24	
823	D97	-36		-0.62	
840	D97	-36		-0.62	
862	D97	-36		-0.62	
875	D97	-30		1.24	
886		----		----	
902		----		----	
912		----		----	
963	D97	-33		0.31	
994	D97	-33		0.31	
1013	D97	-36		-0.62	
1017		----		----	
1023	D97	-30		1.24	
1059		----		----	
1080		----		----	
1094		----		----	
1106		----		----	
1146	D97	-28.5		1.71	
1161	D97	-39		-1.56	
1162	D97	-35.0		-0.31	
1173		----		----	
1203	ISO3016	-33		0.31	
1213	D97	<-27		----	
1231		----		----	
1235		----		----	
1287		----		----	
1293	D97	-32.2		0.56	
1316		----		----	
1349		----		----	
1402	D97	-27		2.18	
1406	D97	-36		-0.62	
1428	ISO3016	-33		0.31	
1431		----		----	
1448		----		----	
1526		----		----	
1613	D97	-36		-0.62	
1620	D97	-36		-0.62	
1622	D97	-33		0.31	
1650		----		----	
1660		----		----	
1720	D97	<-32.0		----	
1738	D97	-31		0.93	

1800		----	----
1827		----	----
1833	D97	-36	-0.62
1850	ISO3016	-35	-0.31
1941	ISO3016	-33	0.31
2122		----	----

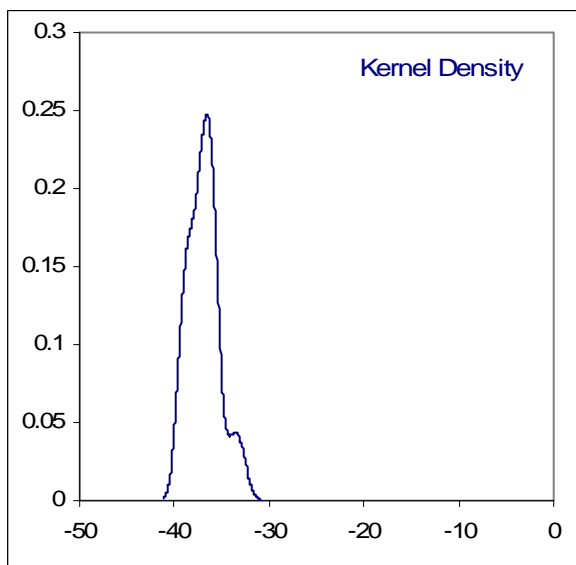
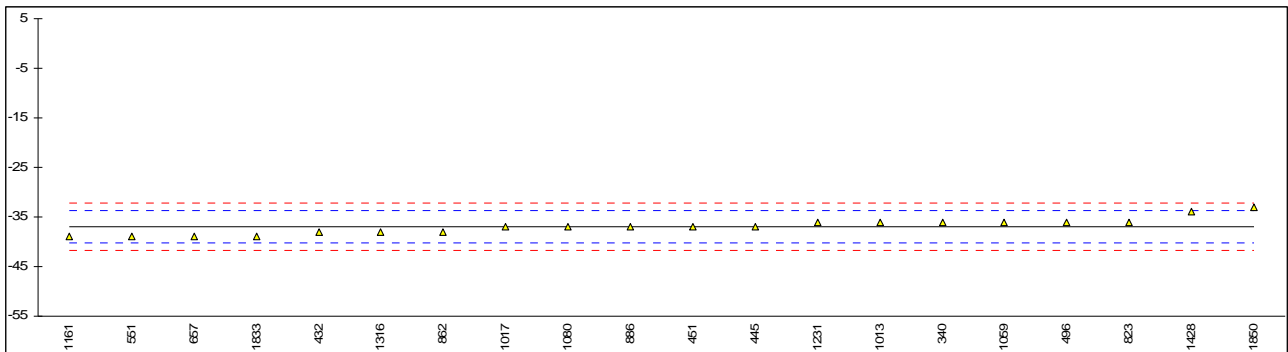
normality	not OK
n	36
outliers	0
mean (n)	-33.99
st.dev. (n)	2.811
R(calc.)	7.87
R(D97:09)	9.00



Determination of Pour Point (Automated, 1°C interval) on sample #11041; results in °C

lab	method	value	mark	z(targ)	remarks
128		----		----	
176		----		----	
233		----		----	
237		----		----	
252		----		----	
254		----		----	
255		----		----	
311		----		----	
315		----		----	
318		----		----	
331		----		----	
333		----		----	
340	D5950	-36		0.56	
343		----		----	
353		----		----	
357		----		----	
360		----		----	
396		----		----	
432	D5950	-38		-0.68	
445	D5950	-37		-0.06	
446		----		----	
450		----		----	
451	D5950	-37		-0.06	
473		----		----	
496	D5950	-36.0		0.56	
551	D5950	-39.0		-1.31	
593		----		----	
608		----		----	
609		----		----	
614		----		----	
657	D5950	-39		-1.31	
663		----		----	
704		----		----	
823	D5950	-36		0.56	
840		----		----	
862	D5950	-38		-0.68	
875		----		----	
886	D5950	-37		-0.06	
902		----		----	
912		----		----	
963		----		----	
994		----		----	
1013	D6892	-36		0.56	
1017	D5950	-37		-0.06	
1023		----		----	
1059	ISO3016	-36		0.56	
1080	D5950	-37		-0.06	
1094		----		----	
1106		----		----	
1146		----		----	
1161	D6749	-39		-1.31	
1162		----		----	
1173		----		----	
1203		----		----	
1213		----		----	
1231	D5950	-36		0.56	
1235		----		----	
1287		----		----	
1293		----		----	
1316	D5950	-38		-0.68	
1349		----		----	
1402		----		----	
1406		----		----	
1428	D6749	-34		1.80	
1431		----		----	
1448		----		----	
1526		----		----	
1613		----		----	
1620		----		----	
1622		----		----	
1650	D5950	<-30		----	
1660		----		----	
1720		----		----	
1738		----		----	

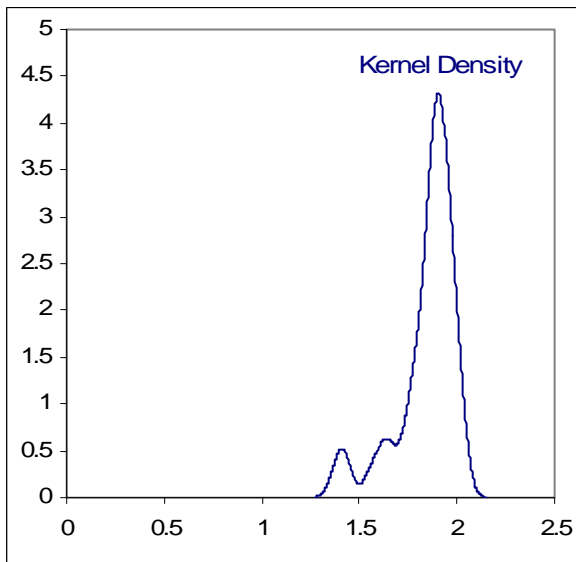
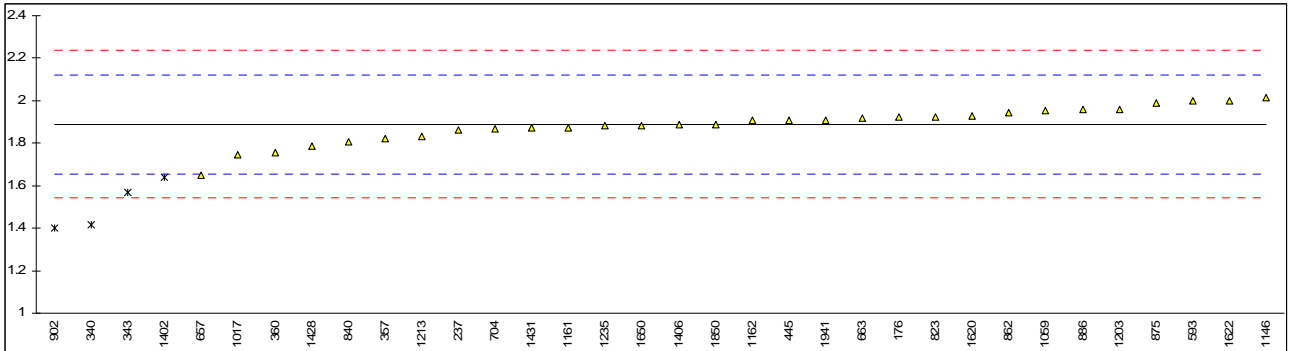
1800		----	----
1827		----	----
1833	D5950	-39	-1.31
1850	ISO3016	-33	2.43
1941		----	----
2122		----	----
normality		OK	
n		20	
outliers		0	
mean (n)		-36.90	
st.dev. (n)		1.619	
R(calc.)		4.53	
R(D5950:07)		4.50	



Determination of Sulphated Ash on sample #11041; results in %M/M

lab	method	value	mark	z(targ)	remarks
128		----		----	
176	D874	1.922		0.29	
233		----		----	
237	D874	1.863		-0.22	
252		----		----	
254		----		----	
255		----		----	
311		----		----	
315		----		----	
318		----		----	
331		----		----	
333		----		----	
340	D874	1.418	DG(0.01)	-4.06	
343	D874	1.57	C,DG(0.05)	-2.75	First reported 1.39
353		----		----	
357	D874	1.82		-0.59	
360	D874	1.757		-1.14	
396		----		----	
432		----		----	
445	D874	1.91		0.19	
446		----		----	
450		----		----	
451		----		----	
473		----		----	
496		----		----	
551		----		----	
593	D874	1.997		0.94	
608		----		----	
609		----		----	
614		----		----	
657	D874	1.65		-2.06	
663	D874	1.916		0.24	
704	D874	1.865		-0.20	
823	D874	1.924		0.31	
840	D874	1.806		-0.71	
862	D874	1.944		0.48	
875	D874	1.99		0.88	
886	D874	1.957		0.59	
902	D874	1.40	DG(0.01)	-4.22	
912		----		----	
963		----		----	
994		----		----	
1013		----		----	
1017	D874	1.746		-1.23	
1023		----		----	
1059	ISO3987	1.955		0.57	
1080		----		----	
1094		----		----	
1106		----		----	
1146	D874	2.017		1.11	
1161	D874	1.875		-0.12	
1162	D874	1.910		0.19	
1173		----		----	
1203	D874	1.96		0.62	
1213	D874	1.83		-0.51	
1231		----		----	
1235	ISO3987	1.884		-0.04	
1287		----		----	
1293		----		----	
1316		----		----	
1349		----		----	
1402	D874	1.64	DG(0.05)	-2.14	
1406	D874	1.888		0.00	
1428	D874	1.786		-0.88	
1431	D874	1.87		-0.16	
1448		----		----	
1526		----		----	
1613		----		----	
1620	D874	1.93		0.36	
1622	D874	2.00		0.96	
1650	D874	1.884		-0.04	
1660		----		----	
1720		----		----	
1738		----		----	

1800		----	----
1827		----	----
1833		----	----
1850	ISO3987	1.89	0.01
1941	ISO3987	1.91	0.19
2122		----	----
normality		OK	
n		30	
outliers		4	
mean (n)		1.889	
st.dev. (n)		0.0823	
R(calc.)		0.230	
R(D874:07)		0.324	

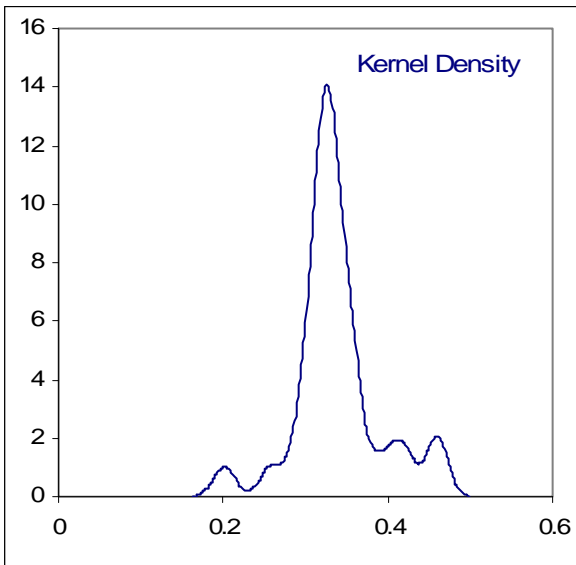
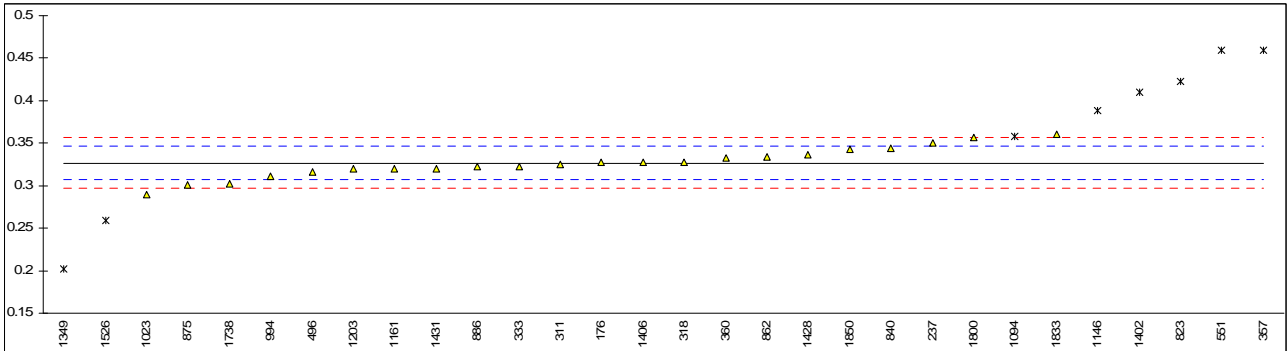


Determination of Sulphur on sample #11041; results in %M/M

lab	method	value	mark	z(targ)	remarks
128		----		----	
176	D2622	0.3271		0.02	
233		----		----	
237	D4294	0.350		2.31	
252		----		----	
254		----		----	
255		----		----	
311	D2622	0.325		-0.18	
315		----		----	
318	INH-I	0.328		0.11	
331		----		----	
333	D2622	0.323		-0.38	
340		----		----	
343		----		----	
353		----		----	
357	D4294	0.46	DG(0.05)	13.31	
360	D2622	0.333		0.61	
396		----		----	
432		----		----	
445		----		----	
446		----		----	
450		----		----	
451		----		----	
473		----		----	
496	D2622	0.3155		-1.13	
551	D2622	0.46	DG(0.05)	13.31	
593		----		----	
608		----		----	
609		----		----	
614		----		----	
657		----		----	
663		----		----	
704		----		----	
823	D4294	0.423	DG(0.05)	9.61	
840	D4294	0.3439		1.70	
862	D2622	0.3340		0.71	
875	D5453	0.3014	C	-2.54	First reported 0.523
886	D2622	0.323		-0.38	
902		----		----	
912		----		----	
963		----		----	
994	D5453	0.3108		-1.60	
1013		----		----	
1017		----		----	
1023	D2622	0.29		-3.68	
1059		----		----	
1080		----	W	----	Result withdrawn
1094	D5185	0.3578	ex	3.09	Result excluded, as test method is not comparable with ASTM D2622
1106		----		----	
1146	D2622	0.389	G(0.05)	6.21	
1161	D2622	0.32		-0.68	
1162		----		----	
1173		----		----	
1203	D2622	0.32		-0.68	
1213		----		----	
1231		----		----	
1235		----		----	
1287		----		----	
1293		----		----	
1316		----		----	
1349	IP336	0.2017	G(0.05)	-12.51	
1402	IP336	0.41	DG(0.05)	8.31	
1406	D2622	0.3280		0.11	
1428	ISO8754	0.336		0.91	
1431	D4294	0.32		-0.68	
1448		----		----	
1526	D5185	0.2585	ex	-6.83	Result excluded, as test method is not comparable with ASTM D2622
1613		----		----	
1620		----		----	
1622		----		----	
1650		----		----	
1660		----		----	
1720		----		----	
1738	D2622	0.302		-2.48	

1800	D2622	0.357		3.01	
1827		-----		-----	
1833	IP336	0.36		3.31	
1850	ISO8754	0.343		1.61	
1941	ISO8754	<0.03	C	-----	First reported 0.42, false negative?
2122		-----		-----	

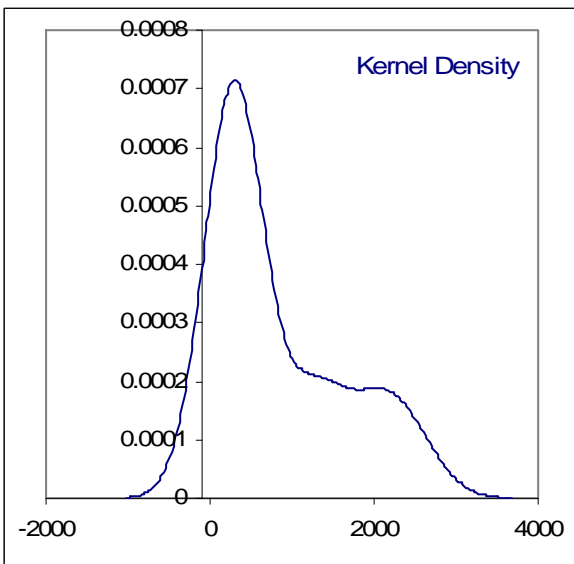
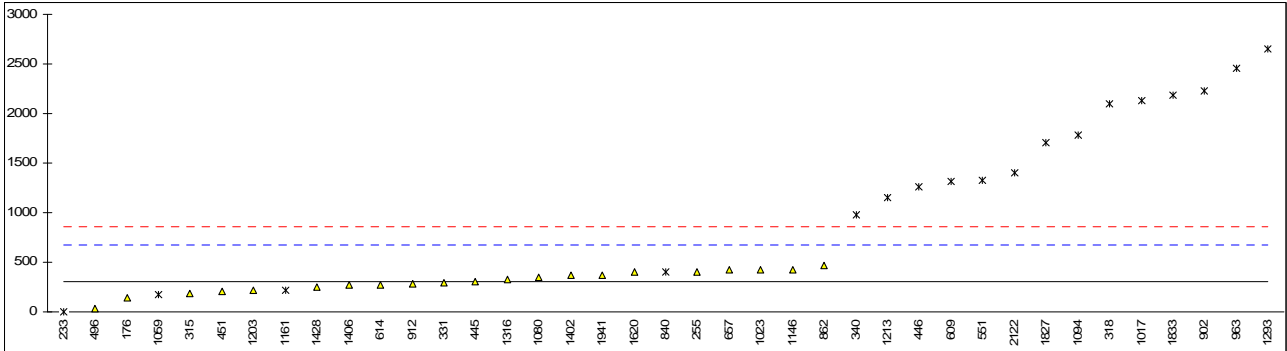
normality	OK	<u>Only ASTM D2622</u>
n	22	OK
outliers	6	13
mean (n)	0.3269	2
st.dev. (n)	0.01753	0.3229
R(calc.)	0.0491	0.01588
R(D2622:10)	0.0280	0.0445
		0.0278



Determination of Water on sample #11041; results in mg/kg

lab	method	value	mark	z(targ)	remarks
128		----		----	
176	D6304-A	139.00	ex	-0.92	Result excluded, see §4.1
233	D7358	0.00	ex	-1.65	Result excluded, see §4.1
237	D95	<500		----	
252		----		----	
254	D95	<500		----	
255	D95	400	ex	0.46	Result excluded, see §4.1
311		----		----	
315	D6304-C	180.4		-0.70	
318	INH-III/E	2100	ex	9.44	Result excluded, see §4.1
331	D6304-C	290		-0.12	
333		----		----	
340	D6304	981	ex	3.53	Result excluded, see §4.1
343		----		----	
353		----		----	
357		----		----	
360		----		----	
396		----		----	
432		----		----	
445	D6304-C	299		-0.07	
446	D6304-A	1257	ex	4.99	Result excluded, see §4.1
450		----		----	
451	D6304-C	210		-0.54	
473		----		----	
496	D6304-C	35.37		-1.46	
551	E203	1329	ex	5.37	Result excluded, see §4.1
593		----		----	
608		----		----	
609	D6304-A	1310.8	ex	5.27	Result excluded, see §4.1
614	D6304-	273	ex	-0.21	Result excluded, see §4.1
657	D6304-C	420		0.57	
663		----		----	
704		----		----	
823		----		----	
840	D95	400	ex	0.46	Result excluded, see §4.1
862	D6304-C	470.5		0.84	
875		----		----	
886		----		----	
902	D6304-A	2225.66	C,ex	10.11	First reported 2327.9. Result excluded, see §4.1
912	D6304-C	278.9		-0.18	
963	D6304-A	2460	C,ex	11.34	First reported 2570. Result excluded, see §4.1
994		----		----	
1013		----		----	
1017	D6304-A	2131	ex	9.61	Result excluded, see §4.1
1023	D6304-	421	ex	0.57	Result excluded, see §4.1
1059	In house	170	ex	-0.75	Result excluded, see §4.1
1080	D6304-C	350		0.20	
1094	D6304-A	1779	ex	7.75	Result excluded, see §4.1
1106		----		----	
1146	D6304-C	429		0.62	
1161	D6304-A	220.3	C,ex	-0.49	First reported 2203. Result excluded, see §4.11
1162		----		----	
1173		----		----	
1203	D6304-	215	ex	-0.51	Result excluded, see §4.1
1213	D6304-	1150	ex	4.42	Result excluded, see §4.1
1231		----		----	
1235		----		----	
1287		----		----	
1293	ISO12937	2650.1	C,ex	12.35	First reported 2284.5. Result excluded, see §4.1
1316	D6304-C	330		0.09	
1349		----		----	
1402	D6304-	368	ex	0.29	Result excluded, see §4.1
1406	D1744	270	ex	-0.22	Result excluded, see §4.1
1428	ISO12937	250	C,ex	-0.33	First reported 2252, Result excluded, see §4.1
1431		----		----	
1448		----		----	
1526	D4377	<5000	ex	----	Result excluded, see §4.1
1613		----		----	
1620	D6304-C	397		0.45	
1622		----		----	
1650		----		----	
1660		----		----	
1720		----		----	
1738		----		----	

1800		-----		-----	
1827	D6304-	1706.3	ex	7.36	Result excluded, see §4.1
1833	D6304-A	2184	C,ex	9.89	First reported 2700, Result excluded, see §4.1
1850		-----		-----	
1941	D6304-C	369		0.30	
2122	IP396	1400	ex	5.75	Result excluded, see §4.1
normality		OK			
n		13			
outliers		0			
mean (n)		312.24			
st.dev. (n)		118.572			
R(calc.)		332.00			
R(D6304:07)		530.11			

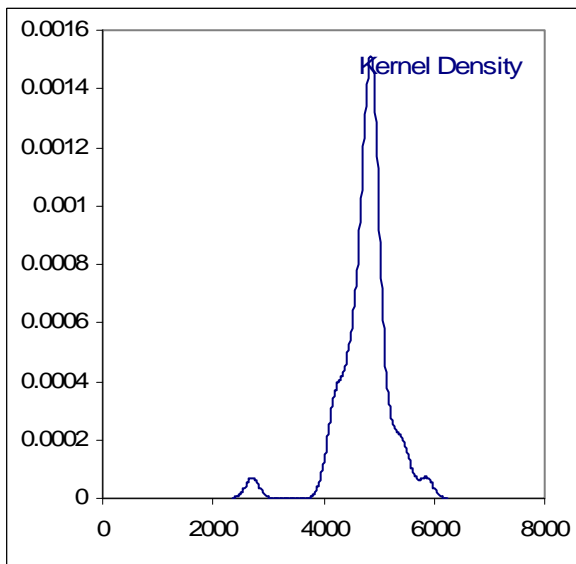
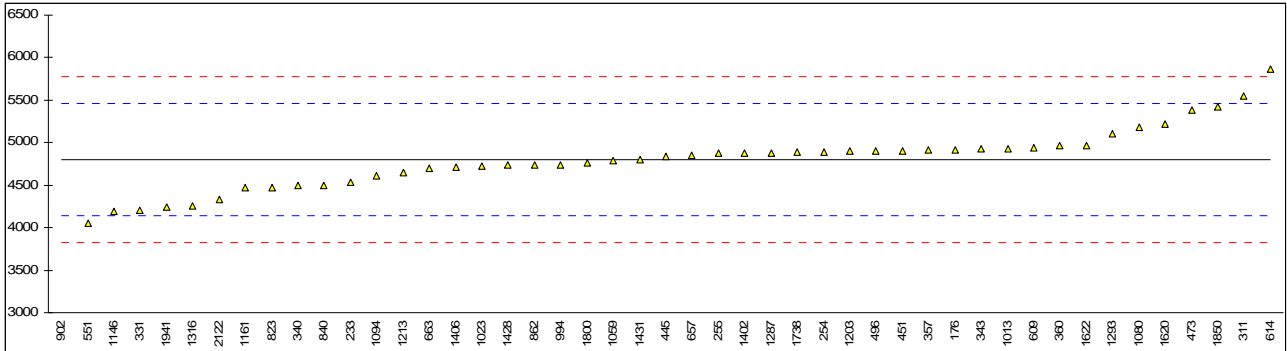


Determination of Calcium (Ca) on sample #11041; results in mg/kg

lab	method	value	mark	z(targ)	remarks
128		----		----	
176	D5185	4920		0.37	
233	D6595	4531.1		-0.82	
237		----		----	
252		----		----	
254	IP308	4894.084		0.29	
255	AAS	4879.3	C	0.24	First reported 3899.1
311	D5185	5545		2.28	
315		----		----	
318		----		----	
331	D5185mod	4205		-1.82	
333		----		----	
340	D5185	4500		-0.92	
343	D5185	4922		0.37	
353		----		----	
357	IP501	4910		0.34	
360	D5185	4960		0.49	
396		----		----	
432		----		----	
445	D5185	4835		0.11	
446		----		----	
450		----		----	
451	D5185mod	4908		0.33	
473	D5185	5377.780		1.77	
496	D5185	4904		0.32	
551	D5185	4052		-2.29	
593		----		----	
608		----		----	
609	D5185	4945		0.44	
614	D5185	5866		3.26	
657	D5185	4850		0.15	
663	D5185	4696.16		-0.32	
704		----		----	
823	D5185	4471.8		-1.00	
840	UOP389	4501.0		-0.92	
862	D5185	4736		-0.20	
875		----		----	
886		----		----	
902	D5185	2710	C,G(0.01)	-6.39	First reported 2730
912		----		----	
963		----		----	
994	D5185	4741		-0.18	
1013	D5185	4930		0.40	
1017		----		----	
1023	D5185	4727		-0.22	
1059	In house	4790		-0.03	
1080	D5185	5181		1.16	
1094	D5185	4615.8		-0.56	
1106		----		----	
1146	D5185	4190		-1.87	
1161	D5185	4471.44		-1.01	
1162		----		----	
1173		----		----	
1203	D5185	4900		0.30	
1213	D4628	4650		-0.46	
1231		----		----	
1235		----		----	
1287	D5185	4883		0.25	
1293	D6595	5105.2		0.93	
1316	D5185	4260		-1.65	
1349		----		----	
1402	D5185	4880		0.24	
1406	D4628	4715		-0.26	
1428	D5185	4733		-0.21	
1431	In house	4807		0.02	
1448		----		----	
1526		----		----	
1613		----		----	
1620	D5185	5217		1.27	
1622	D5185	4970.19		0.52	
1650		----		----	
1660		----		----	
1720		----		----	
1738	D4927	4885		0.26	

1800	In house	4763	-0.11
1827		-----	-----
1833		-----	-----
1850	In house	5416	1.88
1941	D6595	4245	-1.70
2122	D5185	4334	-1.43

normality not OK
 n 46
 outliers 1
 mean (n) 4800.4
 st.dev. (n) 355.46
 R(calc.) 995.3
 R(D5185:09) 915.7

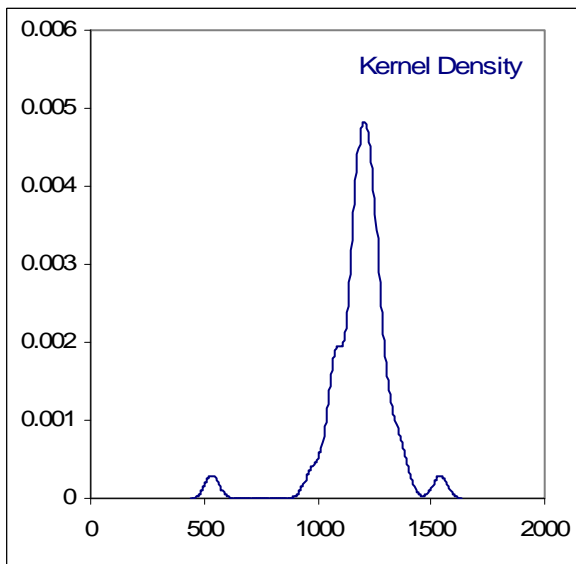
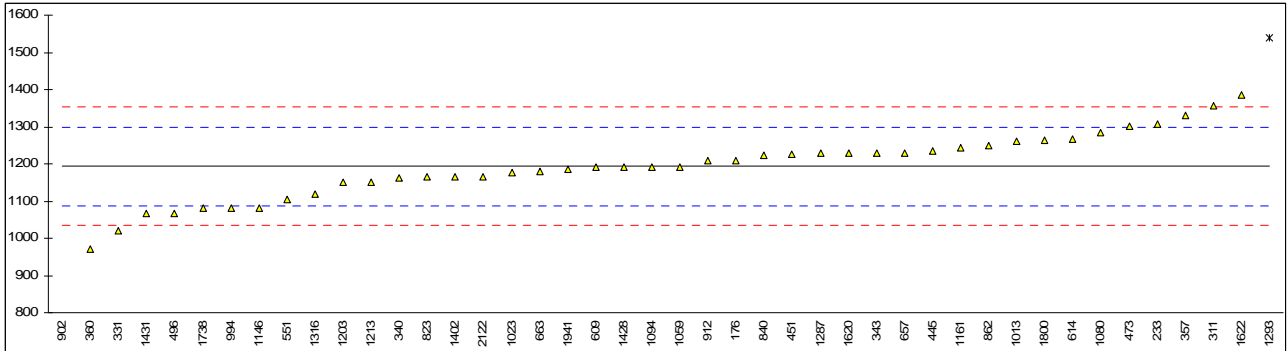


Determination of Phosphorus (P) on sample #11041; results in mg/kg

lab	method	value	mark	z(targ)	remarks
128		----		----	
176	D5185	1210		0.31	
233	D6595	1307.9		2.15	
237		----		----	
252		----		----	
254		----		----	
255		----		----	
311	D5185	1357		3.08	
315		----		----	
318		----		----	
331	D5185mod	1020		-3.27	
333		----		----	
340	D5185	1163		-0.58	
343	D5185	1230		0.69	
353		----		----	
357	IP501	1330		2.57	
360	D5185	970		-4.21	
396		----		----	
432		----		----	
445	D5185	1235		0.78	
446		----		----	
450		----		----	
451	D5185mod	1226		0.61	
473	D5185	1301.90		2.04	
496	D5185	1068		-2.37	
551	D5185	1103		-1.71	
593		----		----	
608		----		----	
609	D5185	1190	C	-0.07	First reported 1449
614	D5185	1266		1.36	
657	D5185	1230		0.69	
663	D5185	1179.23		-0.27	
704		----		----	
823	D5185	1164.7		-0.54	
840	UOP389	1223.9		0.57	
862	D5185	1249		1.04	
875		----		----	
886		----		----	
902	D5185	536.55	C,G(0.01)	-12.38	First reported 570
912	D5185	1210		0.31	
963		----		----	
994	D5185	1082		-2.10	
1013	D5185	1260		1.25	
1017		----		----	
1023	D5185	1177		-0.31	
1059	In house	1192		-0.03	
1080	D5185	1283		1.69	
1094	D5185	1191.4		-0.04	
1106		----		----	
1146	D5185	1082		-2.10	
1161	D5185	1244.64		0.96	
1162		----		----	
1173		----		----	
1203	D5185	1150		-0.82	
1213	D4951	1150		-0.82	
1231		----		----	
1235		----		----	
1287	D5185	1230		0.69	
1293	D6595	1540	C,G(0.05)	6.53	First reported 1427.9
1316	D5185	1120		-1.39	
1349		----		----	
1402	D5185	1165		-0.54	
1406		----		----	
1428	D5185	1191		-0.05	
1431	In house	1067		-2.39	
1448		----		----	
1526		----		----	
1613		----		----	
1620	D5185	1230		0.69	
1622	D5185	1386.19		3.63	
1650		----		----	
1660		----		----	
1720		----		----	
1738	D4927	1080	C	-2.14	First reported 113

1800	In house	1263	1.31
1827		-----	-----
1833		-----	-----
1850		-----	-----
1941	D6595	1186	-0.14
2122	D5185	1166	-0.52

normality	OK
n	42
outliers	2
mean (n)	1193.6
st.dev. (n)	87.78
R(calc.)	245.8
R(D5185:09)	148.6

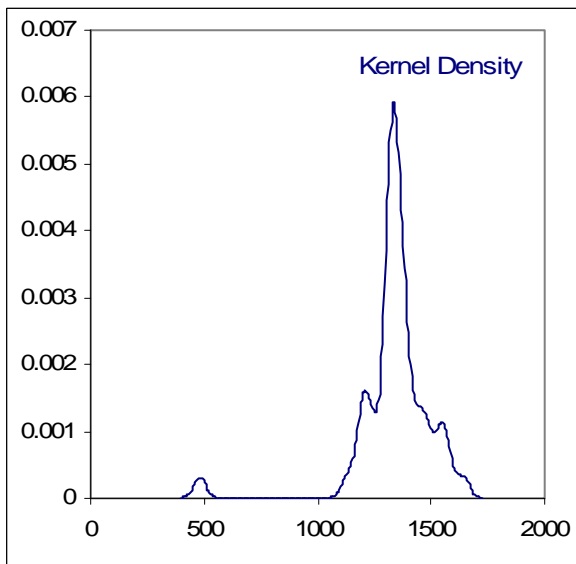
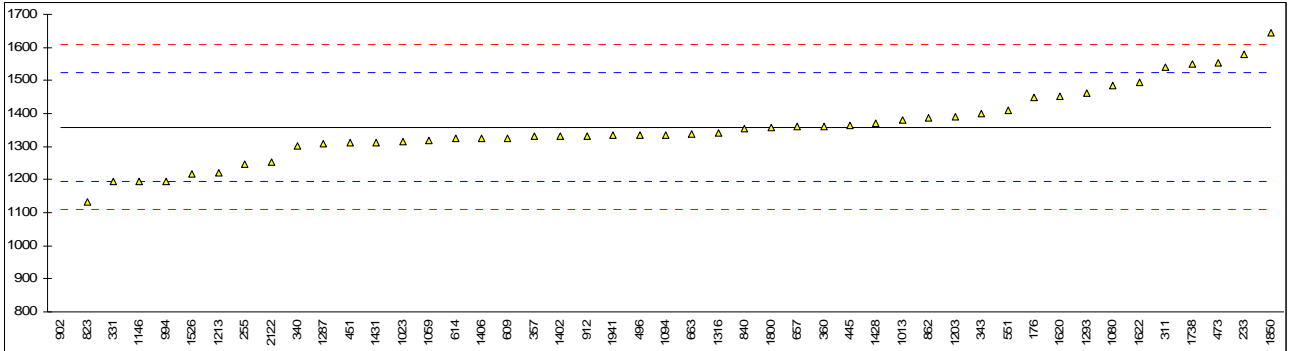


Determination of Zinc (Zn) on sample #11041; results in mg/kg

lab	method	value	mark	z(targ)	remarks
128		----		----	
176	D5185	1450		1.10	
233	D6595	1580.6		2.67	
237		----		----	
252		----		----	
254		----		----	
255	AAS	1245.7	C	-1.37	First reported 597.8
311	D5185	1540		2.18	
315		----		----	
318		----		----	
331	D5185mod	1193		-2.00	
333		----		----	
340	D5185	1301		-0.70	
343	D5185	1400		0.49	
353		----		----	
357	IP501	1330		-0.35	
360	D5185	1360		0.01	
396		----		----	
432		----		----	
445	D5185	1365		0.07	
446		----		----	
450		----		----	
451	D5185mod	1311		-0.58	
473	D5185	1553.060		2.34	
496	D5185	1336		-0.28	
551	D5185	1411		0.63	
593		----		----	
608		----		----	
609	D5185	1325		-0.41	
614	D5185	1324		-0.42	
657	D5185	1360		0.01	
663	D5185	1338.65		-0.25	
704		----		----	
823	D5185	1132.7		-2.73	
840	UOP389	1355.0		-0.05	
862	D5185	1386		0.33	
875		----		----	
886		----		----	
902	D5185	482	C,G(0.01)	-10.58	First reported 526
912	D5185	1331		-0.34	
963		----		----	
994	D5185	1196		-1.97	
1013	D5185	1380		0.25	
1017		----		----	
1023	D5185	1315		-0.53	
1059	In house	1320		-0.47	
1080	D5185	1486		1.53	
1094	D5185	1336.4		-0.27	
1106		----		----	
1146	D5185	1194		-1.99	
1161		----		----	
1162		----		----	
1173		----		----	
1203	D5185	1390		0.37	
1213	D4628	1220		-1.68	
1231		----		----	
1235		----		----	
1287	D5185	1310		-0.59	
1293	D6595	1460.6		1.23	
1316	D5185	1340		-0.23	
1349		----		----	
1402	D5185	1330		-0.35	
1406	D4628	1325		-0.41	
1428	D5185	1372		0.16	
1431	In house	1312		-0.57	
1448		----		----	
1526	D5185	1218.5		-1.70	
1613		----		----	
1620	D5185	1451		1.11	
1622	D5185	1494.24		1.63	
1650		----		----	
1660		----		----	
1720		----		----	
1738	D4927	1549		2.29	

1800	In house	1358	-0.01
1827		-----	-----
1833		-----	-----
1850	INH	1644	3.44
1941	D6595	1334	-0.30
2122	D5185	1252	-1.29

normality OK
 n 46
 outliers 1
 mean (n) 1359.1
 st.dev. (n) 107.82
 R(calc.) 301.9
 R(D5185:09) 232.1



APPENDIX 2

Number of participants per country

1 laboratory in AUSTRALIA
1 laboratory in AUSTRIA
1 laboratory in AZERBAIJAN
1 laboratory in BELGIUM
1 laboratory in BRAZIL
1 laboratory in BULGARIA
1 laboratory in CROATIA
1 laboratory in ECUADOR
1 laboratory in FINLAND
3 laboratories in FRANCE
2 laboratories in GERMANY
1 laboratory in GHANA
2 laboratories in GREECE
2 laboratories in HUNGARY
1 laboratory in INDIA
1 laboratory in INDONESIA
1 laboratory in IRELAND
3 laboratories in ITALY
1 laboratory in JORDAN
2 laboratories in KENYA
1 laboratory in KOREA
4 laboratories in MALAYSIA
1 laboratory in NEGARA BRUNEI DARUSSALAM
2 laboratories in NIGERIA
2 laboratories in NORWAY
3 laboratories in P.R. of CHINA
1 laboratory in POLAND
1 laboratory in PORTUGAL
1 laboratory in REPUBLIC OF MACEDONIA
1 laboratory in RUSSIA
1 laboratory in SAUDI ARABIA
1 laboratory in SERBIA
1 laboratory in SINGAPORE
1 laboratory in SLOVENIA
2 laboratories in SPAIN
1 laboratory in SUDAN
1 laboratory in SWEDEN
1 laboratory in TAIWAN R.O.C.
1 laboratory in TANZANIA
2 laboratories in THAILAND
4 laboratories in THE NETHERLANDS
5 laboratories in TURKEY
2 laboratories in U.S.A.
1 laboratory in UKRAINE
9 laboratories in UNITED KINGDOM
2 laboratories 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
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
SDS	= Material Safety Data Sheet

Literature:

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- 12 J.N. Miller, Analyst, 118, 455, (1993)
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