

Results of Proficiency Test
Hydraulic Fluid (fresh)
November 2014

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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Report no. iis14L20

February 2015

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1 INTRODUCTION

Since 2003, the Institute for Interlaboratory Studies organized a proficiency test for the analysis of used Hydraulic Fluid every year. From the responses to a questionnaire that was sent to all participants, it was decided that besides the proficiency test for used Hydraulic Fluid also to organize a proficiency test for fresh Hydraulic Fluid during the annual program 2014/2015. In this first interlaboratory study on fresh Hydraulic Fluid, 29 laboratories from 20 different countries have participated. See appendix 2 for the number of participants per country. In this report, the test results of the 2014 interlaboratory study on fresh Hydraulic Fluid are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, The Netherlands, was the organiser of this proficiency test. Analysis for fit-for-use and homogeneity testing were subcontracted. It was decided to send one sample of one litre with fresh oil (Tellus S46), labelled #14219. Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system on IEC/ISO17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organisation was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol can be downloaded from 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 trader. The approximately 180 litre of the bulk material was homogenised. After homogenisation, 60 amber glass bottles of 1 litre were filled and labelled #14219. The homogeneity of the subsamples #14219 was checked by determination of Density in accordance with ASTM D4052 and Viscosity at 40°C according to ASTM D445 on 8 stratified randomly selected samples.

	<i>Density @15 °C in kg/L</i>	<i>Viscosity @40°C in cSt</i>
Sample #14219-1	0.87045	45.37
Sample #14219-2	0.87046	45.37
Sample #14219-3	0.87046	45.36
Sample #14219-4	0.87046	45.37
Sample #14219-5	0.87046	45.36
Sample #14219-6	0.87046	45.37
Sample #14219-7	0.87045	45.37
Sample #14219-8	0.87045	45.34

Table 1: homogeneity test results of subsamples #14219

From the test results of table 1, the repeatabilities were calculated and compared with 0.3 times the corresponding target reproducibility in agreement with the procedure of ISO 13528, Annex B2 in the next table:

	<i>Density @15 °C in kg/L</i>	<i>Viscosity @40°C in cSt</i>
r (Observed)	0.00001	0.03
reference method	ISO12185:96	D445:14e2
0.3 * R (ref. method)	0.00015	0.10

Table 2: repeatabilities of subsamples #14219

Each calculated repeatability was equal or less than 0.3 times the corresponding reproducibility of the respective test method. Therefore, homogeneity of the subsamples #14219 was assumed.

To each of the participating laboratories was dispatched: One 1 litre amber glass bottle, labelled #14219 on October 29, 2014.

2.5 STABILITY OF THE SAMPLES

The stability of Hydraulic Fluid, packed in the brown glass bottles was checked. The material was found sufficiently stable for the period of the proficiency test.

2.6 ANALYSES

The participants were asked to determine Total Acid Number, Copper Corrosion, Density @15°C, Flash Point PMcc, Foam Characteristics (Foam Tendency, Foam Stability), Kinematic Viscosity @40°C and @100°C, Viscosity Stabinger @40°C and @100°C, Viscosity Index,

Pour Point (manual and automated), Sulphur, Water, Water Separability @ 54°C and Calcium, Phosphorus and Zinc on sample #14219.

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

A SDS and a form to confirm receipt of the samples were added to the sample package.

3 RESULTS

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

Directly after the deadline, a reminder fax was sent to the 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.

Results that came in after deadline were not taken into account in the screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

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

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.

In accordance to ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon, Grubbs and Rosner outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test and by R(0.01) for the Rosner General ESD test (see appendix 3, no.15). Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of the averages and the standard deviations.

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

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. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 3; nos.15 and 16). Also a normal Gauss curve was projected over the Kernel Density Graph.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the spread of this interlaboratory study. The target standard deviation was calculated from the literature reproducibility by division with 2.8.

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

The z-scores were calculated in accordance with:

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

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 maybe as follows:

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

4 EVALUATION

In this proficiency test, no serious problems were encountered during dispatch and execution. Only two laboratories (Algeria and Brazil) received the samples late. Only three laboratories reported the results after the final reporting date.

The 29 reporting participants sent in 346 numerical results. Observed were 19 outlying results, which is 5.5% 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 Gaussian distribution. These are referred to as "not OK" or "suspect". The statistical evaluation of these data sets should be used with due care.

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

4.1 EVALUATION PER TEST

In this section, the results are discussed per test. The methods that 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 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 D664:11a.

Copper Corrosion: This determination was not problematic. All participants agreed on a result of 1 (or 1A).

Density @ 15°C: This determination was problematic for a number of laboratories. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ISO12185:96.

Flash Point PMcc: This determination was problematic. No statistical outliers were observed. One result was withdrawn as the used test method is not comparable with ASTM D93. The calculated reproducibility is not in agreement with the requirements of ASTM D93:13e1 method A.

Foaming Characteristics: This determination was very problematic. In total four statistical outliers were observed. The calculated reproducibility for each of the three sequences after rejection of the statistical outliers is not at all in agreement with the requirements of ASTM D892:13.

Foaming Tendency: None of the reporting laboratories reported a positive result for the settling period after 10 min. Therefore all reporting participants agreed on a result of 0 (Nil)

Viscosity Index 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 D2270:10e1.

Kin.Visco.@ 40°C: This determination was not problematic. Only one statistical outlier was observed and the calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of ASTM D445:14e2.

Kin.Visco.@ 100°C: This determination was very problematic. Only one statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of ASTM D445:14e2.

Viscosity Stabinger.@ 40°C: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D7042:14.

Viscosity Stabinger.@ 100°C: This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D7042:14.

Pour Point (manual): This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in full agreement with the requirements of ASTM D97:12.

Pour Point (automated): This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in full agreement with the requirements of ASTM D5950:14.

Sulphur: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ASTM D4294:10.

Water: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility, after rejection of the statistical outlier is in good agreement with the requirements of ASTM D6304:07.

Water Separability @54°C: This determination may not be problematic. No statistical outliers were observed and the calculated reproducibility for “time to reach <3ml emulsion” and “time to reach 37ml water” are both in good agreement with the requirements of ASTM D1401:12. However, four participants reported to have aborted the test and two other participants did not abort the test.

Calcium: All reported results are below the application range (40 – 9000 mg/kg) of ASTM D5185:13e1. Therefore no significant conclusions were drawn.

Phosphorus: This determination was problematic for a number of laboratories. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D5185:13e1.

Zinc: All reported results are below the application range (60 – 1600 mg/kg) of ASTM D5185:13e1. Therefore no significant conclusions were drawn.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and these parameters as found for the group of participating laboratories. The average results and the calculated reproducibilities are compared in the next tables with the reproducibilities, derived from literature standards (in casu the ASTM, IP, ISO and EN standards, see tables in appendix 1).

<i>Parameter</i>	<i>unit</i>	<i>n</i>	<i>average</i>	<i>2.8 * sd</i>	<i>R(lit)</i>
Total Acid Number	mg KOH/g	16	0.14	0.03	0.16
Copper Corrosion		13	1A	n.a.	n.a.
Density @ 15°C	kg/L	25	0.8704	0.0005	0.0005
Flash Point PMcc	°C	25	215.5	18.8	15.3
Foam Charac. Seq I	ml	8	244	245	121
Foam Charac. Seq II	ml	8	14.4	27.4	20.2
Foam Charac. Seq II	ml	8	220	165	111
Viscosity Index		21	104.9	3.4	2.0
Kinematic viscosity @ 40°C	mm/s ²	25	45.237	0.366	0.344
Kinematic viscosity @ 100°C	mm/s ²	22	6.817	0.123	0.052
Viscosity Stabinger @ 40°C	mm/s ²	10	45.355	0.388	0.608
Viscosity Stabinger @ 100°C	mm/s ²	10	6.838	0.078	0.093
Pour Point (manual)	°C	17	-28.3	8.4	9.0
Pour Point (automated)	°C	10	-31.1	3.1	4.5
Sulphur	mg/kg	12	2304	264	282
Water	mg/kg	16	31	28	131
Water Separability ≤3ml emul.	min	7	14.0	2.8	20.0
Water Separability 37ml water	min	7	14.3	2.7	20.0
Calcium as Ca	mg/kg	20	<40	n.a.	n.a.
Phosphorus as P	mg/kg	18	177.8	38.0	57.3
Zinc as Zn	mg/kg	20	<60	n.a.	n.a.

Table 3: reproducibilities of results of sample #14219

results between brackets to be used with care, result was lower (or above) than application range of reference method

Without further statistical calculations, it could be concluded that for many tests there is a good compliance of the group of participating laboratories with the relevant standards. The problematic tests have been discussed in paragraph 4.1.

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2014 WITH THE PREVIOUS PTs.

	<i>November 2014</i>
Number of reporting labs	29
Number of results reported	346
Statistical outliers	19
Percentage outliers	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	<i>November 2014</i>
Total Acid Number	++
Density @ 15°C	+/-
Flash Point PMcc	-
Foam Charac. Seq I	--
Foam Charac. Seq II	--
Foam Charac. Seq II	--
Viscosity Index	--
Kinematic viscosity @ 40°C	+/-
Kinematic viscosity @ 100°C	--
Viscosity Stabinger @ 40°C	++
Viscosity Stabinger @ 100°C	+
Pour Point (manual)	+
Pour Point (automated)	+
Sulphur	+
Water	++
Water Separability ≤3ml emul.	++
Water Separability 37ml water	++
Calcium as Ca	n.e.
Phosphorus as P	++
Zinc as Zn	n.e.

Table 5: comparison determinations against the standard results between brackets to be used with care, result was lower (or above) than application range of reference method.

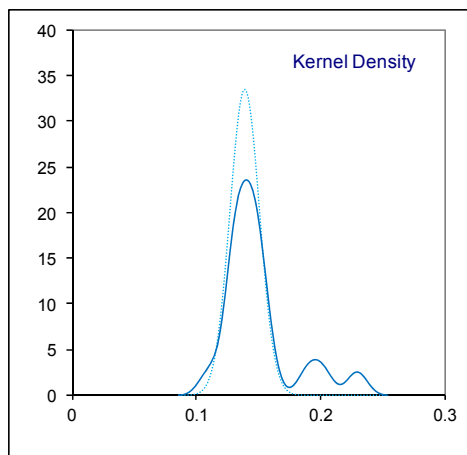
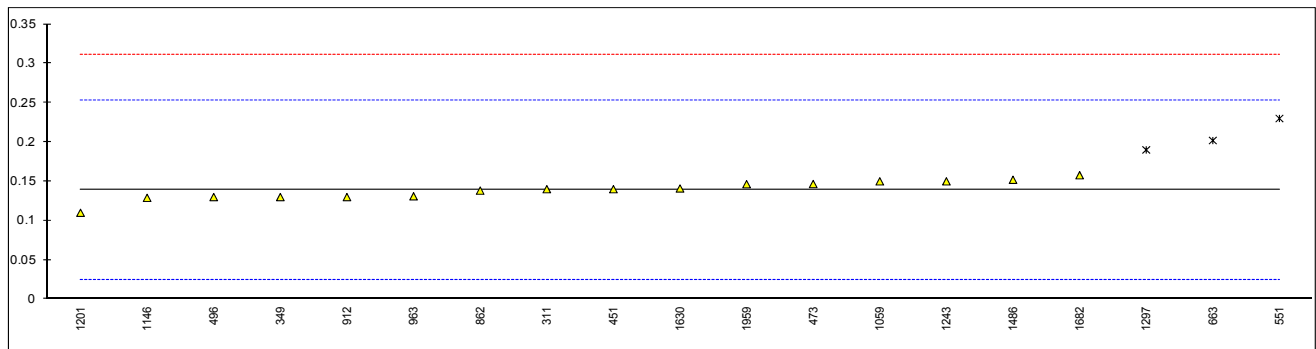
- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- : group performed worse than the standard
- : group performed much worse than the standard

APPENDIX 1

Determination of Acid Number (Total) on sample #14219; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
173		----		----	
255		----		----	
311	D664	0.14		0.02	
349	D664	0.13		-0.15	
432		----		----	
451	D664	0.14		0.02	
473	D664	0.1466		0.13	
496	D664	0.130		-0.15	
541		----		----	
551	D664	0.23	G(0.01)	1.59	
614		----		----	
663	D664	0.202	DG(0.01)	1.10	
862	D664	0.1382		-0.01	
912	D664	0.13		-0.15	
963	D974	0.131		-0.14	
1026	D664	<0.05		<-1.55	
1059	ISO6619	0.15		0.19	
1146	D664	0.129		-0.17	
1201	D664	0.11		-0.50	
1243	D664	0.15		0.19	
1297	D664	0.19	DG(0.01)	0.89	
1328		----		----	
1435		----		----	
1486	ISO6618	0.152		0.23	
1630	D974	0.14095		0.04	
1682	D664	0.1578		0.33	
1842		----		----	
1877		----		----	
1959	GB/T4945	0.1464		0.13	

normality OK
n 16
outliers 3
mean (n) 0.139
st.dev. (n) 0.0119
R(calc.) 0.033
R(D664:11a) 0.161



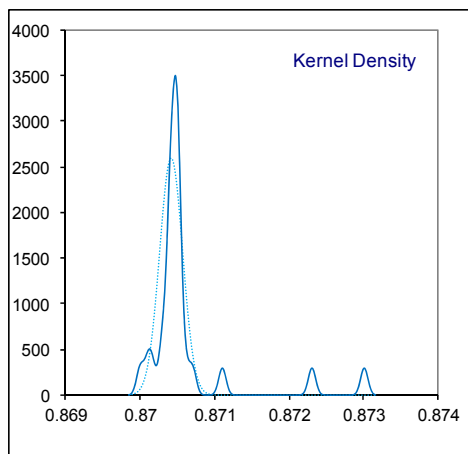
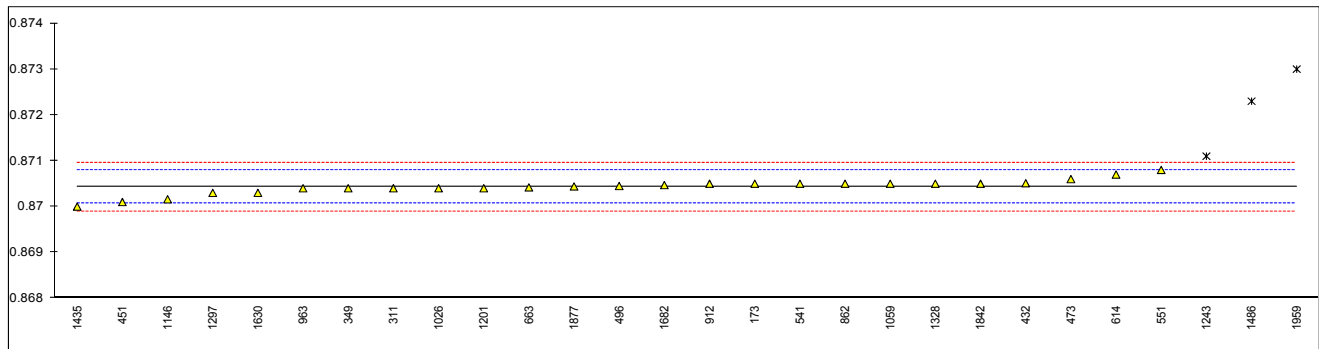
Determination of Copper Corrosion on sample #14219; results in rating

lab	method	value	mark	z(targ)	remarks
173		----		----	
255		----		----	
311		----		----	
349		----		----	
432		----		----	
451		----		----	
473		----		----	
496	ISO2160	1A		----	
541		----		----	
551	D130	1A		----	
614	D130	1A		----	
663	D130	1A		----	
862	D130	1A		----	
912	D130	1A		----	
963		----		----	
1026	D130	1A		----	
1059		----		----	
1146		----		----	
1201	D130	1A		----	
1243	ISO2160	1A		----	
1297		1A		----	
1328		----		----	
1435		----		----	
1486		----		----	
1630	D130	1A		----	
1682	D130	1A		----	
1842	IP154	1B		----	
1877		----		----	
1959		----		----	
	normality	n.a.			
	n	13			
	outliers	0			
	mean (n)	1			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D130:12)	n.a.			

Determination of Density @ 15°C on sample #14219; results in kg/L

lab	method	value	mark	z(targ)	remarks
173	D4052	0.8705		0.39	
255		-----		-----	
311	D4052	0.8704		-0.17	
349	D4052	0.8704		-0.17	
432	D4052	0.8705		0.45	
451	D4052	0.8701	C	-1.85	First reported 870.1 (unit error)
473	D4052	0.8706		0.95	
496	D4052	0.87045		0.11	
541	D4052	0.8705		0.39	
551	D4052	0.8708		2.07	
614	D4052	0.8707		1.51	
663	D4052	0.87042		-0.06	
862	D4052	0.8705		0.39	
912	D4052	0.8705		0.39	
963	D4052	0.8704		-0.17	
1026	D4052	0.8704		-0.17	
1059	ISO12185	0.8705		0.39	
1146	D4052	0.87016		-1.51	
1201	ISO12185	0.8704		-0.17	
1243	ISO12185	0.8711	R(0.01)	3.75	
1297	D4052	0.8703		-0.73	
1328	SH/T0604	0.87050		0.39	
1435	D4052	0.870		-2.41	
1486	ISO12185	0.8723	R(0.01)	10.47	
1630	D1298	0.8703		-0.73	
1682	D4052	0.87047		0.22	
1842	D4052	0.8705		0.39	
1877	D4052	0.87044		0.06	
1959	GB/T1884	0.8730	C,R(0.01)	14.39	First reported 0.8728

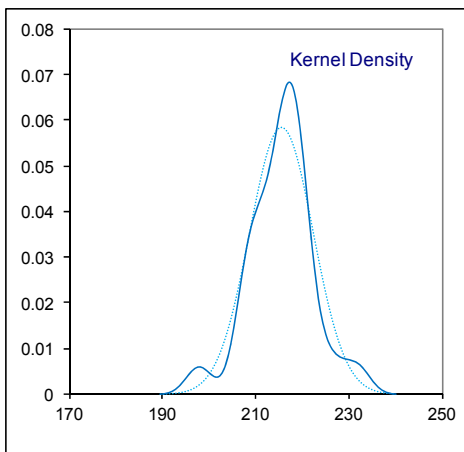
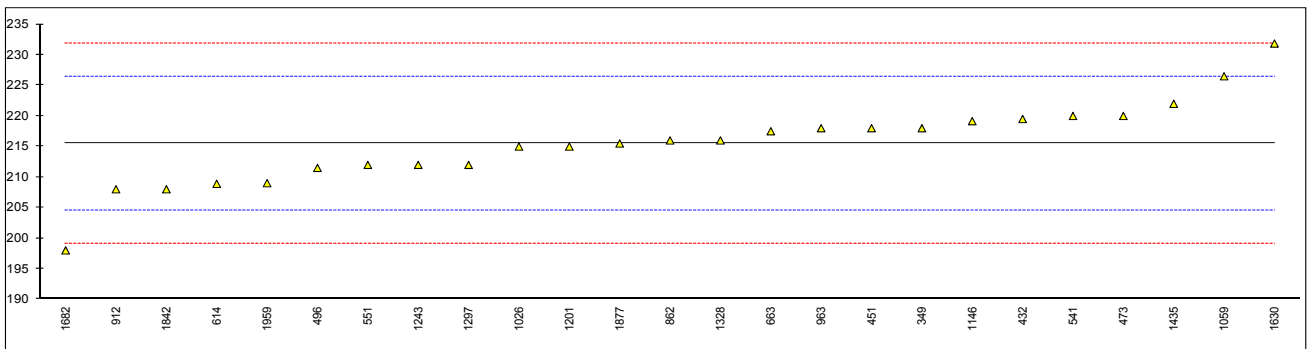
normality suspect
n 25
outliers 3
mean (n) 0.87043
st.dev. (n) 0.000169
R(calc.) 0.00047
R(ISO12185:96) 0.00050



Determination of Flash Point PMcc on sample #14219; results in °C

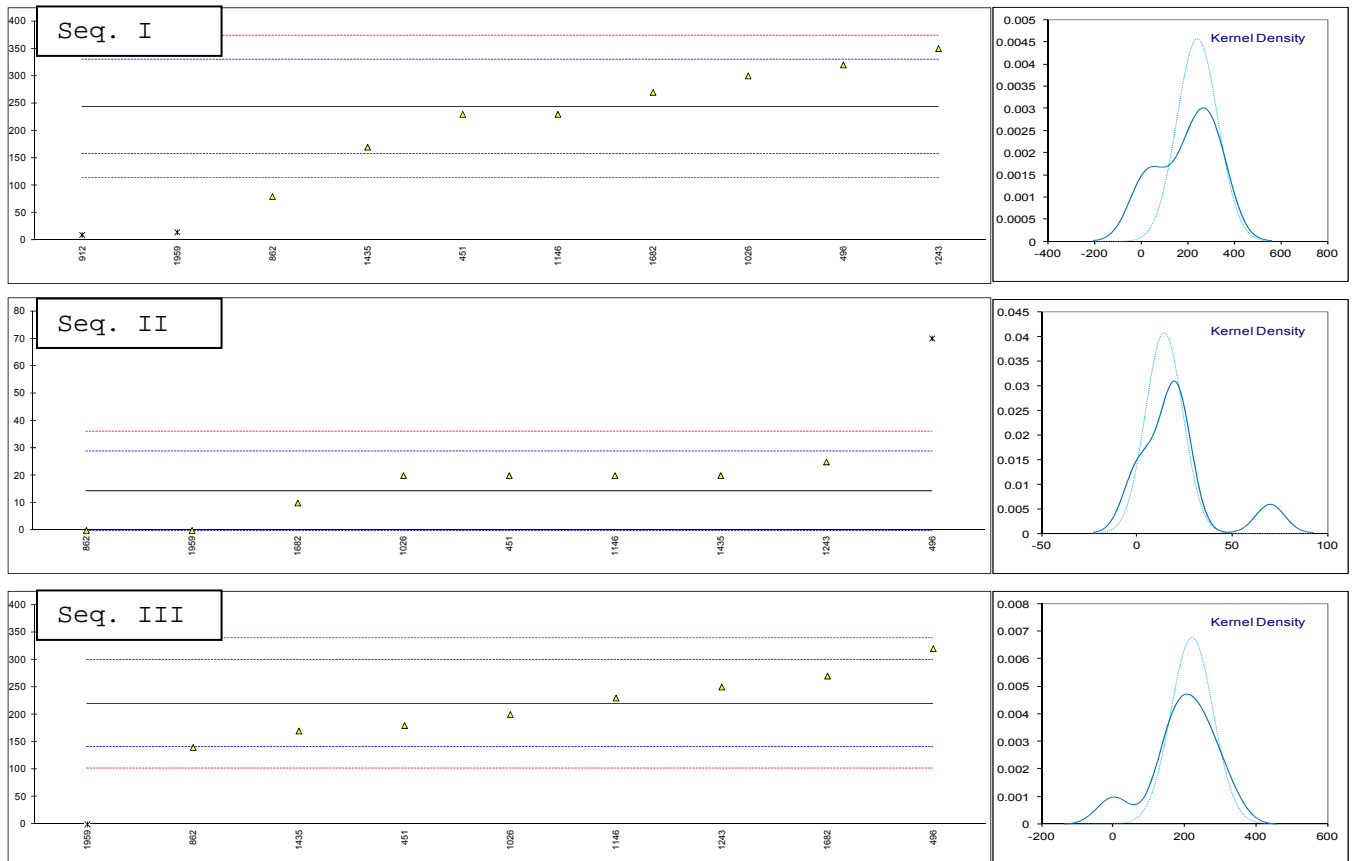
lab	method	value	mark	z(targ)	remarks
173		----		----	
255		----		----	
311		----		----	
349	D93-A	218		0.46	
432	D93-A	219.5		0.73	
451	D93-A	218		0.46	
473	D93-A	220.0		0.82	
496	D93-A	211.5		-0.73	
541	D93-A	220.0		0.82	
551	D93	212		-0.64	
614	D93-A	208.9		-1.21	
663	D93-A	217.5		0.37	
862	D93-A	216.0		0.09	
912	D93-A	208.0		-1.37	
963	D93-B	218		0.46	
1026	D93	215.0		-0.09	
1059	ISO2719	226.5		2.01	
1146	INH-93A	219.15		0.67	
1201	D93-A	215.0		-0.09	
1243	D93	212		-0.64	
1297	D93-B	212		-0.64	
1328	GB/T261	216.0		0.09	
1435	D93-A	222		1.19	
1486		----	W	----	Result withdrawn, test method reported was flashpoint COC (236.66)
1630	D93-A	231.86		2.99	
1682	D93-A	198		-3.20	
1842	D93	208		-1.37	
1877	D93-A	215.5		0.00	
1959	GB/T261	209.0	C	-1.19	First reported 200.0

normality suspect
n 25
outliers 0
mean (n) 215.50
st.dev. (n) 6.714
R(calc.) 18.80
R(D93-A:13e1) 15.30



Determination of Foaming Characteristics, Foaming Tendency (at end of 5 min blowing period) on sample #14219; results in ml

lab	method	Seq. I, Foam	mark	z(targ)	Seq. II, Foam	mark	z(targ)	Seq. III, Foam	mark	z(targ)
173		----		----	----		----	----		----
255		----		----	----		----	----		----
311		----		----	----		----	----		----
349		----		----	----		----	----		----
432		----		----	----		----	----		----
451	D892	230		-0.32	20		0.78	180		-1.01
473		----		----	----		----	----		----
496	D892	320		1.76	70	G(0.01)	7.70	320		2.53
541		----		----	----		----	----		----
551		----		----	----		----	----		----
614		----		----	----		----	----		----
663		----		----	----		----	----		----
862	D892	80		-3.78	0		-1.99	140		-2.02
912	D892	10	DG(0.05)	-5.40	NIL		----	NIL	false -	----
963		----		----	----		----	----		----
1026	D892	300		1.30	20		0.78	200		-0.51
1059		----		----	----		----	----		----
1146	D892	230		-0.32	20		0.78	230		0.25
1201		----		----	----		----	----		----
1243	DIN51566	350		2.46	25		1.47	250		0.76
1297		----		----	----		----	----		----
1328		----		----	----		----	----		----
1435		170		-1.70	20		0.78	170		-1.26
1486		----		----	----		----	----		----
1630		----		----	----		----	----		----
1682		270		0.61	10		-0.61	270		1.26
1842		----		----	----		----	----		----
1877		----		----	----		----	----		----
1959	GB/T12579	15	DG(0.05)	-5.29	0		-1.99	0	G(0.01)	-5.56
normality		OK			OK			OK		
n		8			8			8		
outliers		2			1			1		
mean (n)		243.8			14.4			220.0		
st.dev. (n)		87.49			9.80			59.04		
R(calc.)		245.0			27.4			165.3		
R(D892:13)		121.1			20.2			110.7		



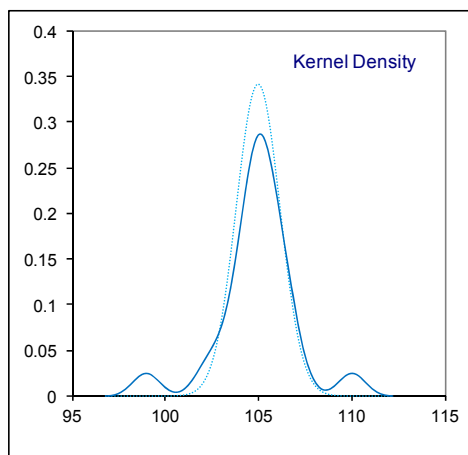
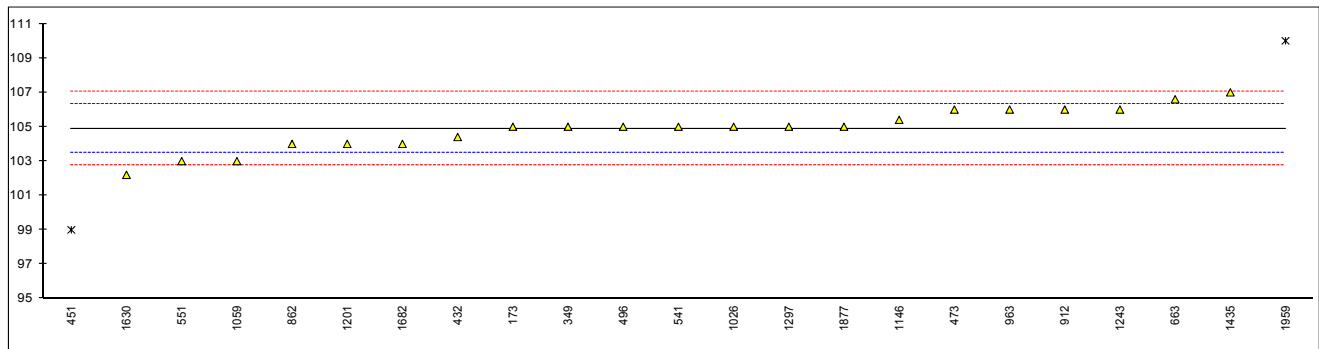
Determination of Foaming Characteristics, Foaming Stability (at end of 10 min settling period) on sample #14219; results in ml

lab	method	Seq. I, Foam	mark	z(targ)	Seq. II, Foam	mark	z(targ)	Seq. III, Foam	mark	z(targ)
173		----		----	----		----	----		----
255		----		----	----		----	----		----
311		----		----	----		----	----		----
349		----		----	----		----	----		----
432		----		----	----		----	----		----
451	D892	NIL		----	NIL		----	NIL		----
473		----		----	----		----	----		----
496	D892	0		----	0		----	0		----
541		----		----	----		----	----		----
551		----		----	----		----	----		----
614		----		----	----		----	----		----
663		----		----	----		----	----		----
862	D892	0		----	0		----	0		----
912	D892	NIL		----	NIL		----	NIL		----
963		----		----	----		----	----		----
1026	D892	0		----	0		----	0		----
1059		----		----	----		----	----		----
1146	D892	0		----	0		----	0		----
1201		----		----	----		----	----		----
1243	DIN51566	0		----	0		----	0		----
1297		----		----	----		----	----		----
1328		----		----	----		----	----		----
1435		----		----	----		----	----		----
1486		----		----	----		----	----		----
1630		----		----	----		----	----		----
1682		----		----	----		----	----		----
1842		----		----	----		----	----		----
1877		----		----	----		----	----		----
1959	GB/T12579	0		----	0		----	0		----
	normality	n.a.			n.a.			n.a.		
	n	8			8			8		
	outliers	n.a.			n.a.			n.a.		
	mean (n)	0			0			0		
	st.dev. (n)	n.a.			n.a.			n.a.		
	R(calc.)	n.a.			n.a.			n.a.		
	R(lit)	n.a.			n.a.			n.a.		

Determination of Viscosity Index on sample #14219; unit less results

lab	method	value	mark	z(targ)	remarks
173	D2270	105		0.16	
255		----		----	
311		----		----	
349	D2270	105		0.16	
432	ISO2909	104.4		-0.68	
451	D2270	99	R(0.05)	-8.24	
473	D2270	106.0		1.56	
496	D2270	105		0.16	
541	D2270	105		0.16	
551	D2270	103		-2.64	
614		----		----	
663	D2270	106.6		2.40	
862	D2270	104		-1.24	
912	D2270	106		1.56	
963	D2270	106		1.56	
1026	D2270	105		0.16	
1059	ISO2909	103		-2.64	
1146	D2270	105.4		0.72	
1201	D2270	104		-1.24	
1243	D2270	106		1.56	
1297	D2270	105		0.16	
1328		----		----	
1435	D2270	107		2.96	
1486		----		----	
1630	D2270	102.2		-3.76	
1682	D2270	104		-1.24	
1842		----		----	
1877	D2270	105		0.16	
1959	GB/T1995	110	C,R(0.05)	7.16	First reported 109.4

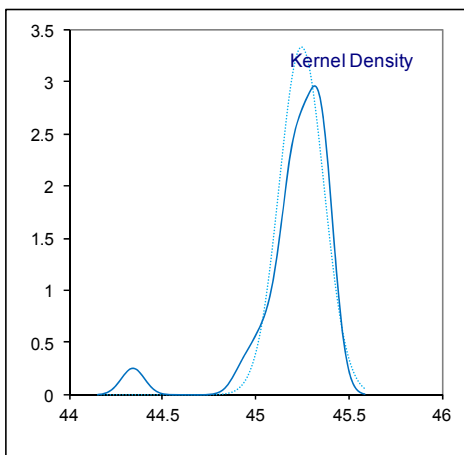
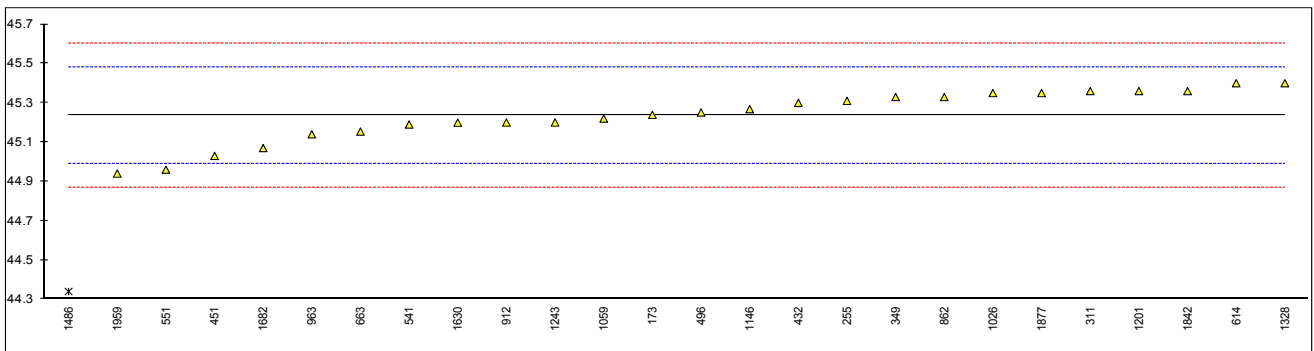
normality OK
n 21
outliers 2
mean (n) 104.89
st.dev. (n) 1.217
R(calc.) 3.41
R(D2270:10e1) 2.00



Determination of Kinematic Viscosity @ 40°C on sample #14219; results in mm/s²

lab	method	value	mark	z(targ)	remarks
173	D445	45.239		0.02	
255	D7279	45.31	C	0.60	First reported 44.21
311	D445	45.36		1.01	
349	D445	45.33		0.76	
432	D445	45.30		0.52	
451	D7279	45.03		-1.68	
473		----		----	
496	D445	45.251		0.12	
541	D445	45.19		-0.38	
551	D7279	44.96		-2.25	
614	D445	45.4		1.33	
663	D445	45.154		-0.67	
862	D445	45.33		0.76	
912	D445	45.20		-0.30	
963	D445	45.14		-0.79	
1026	D445	45.35		0.92	
1059	ISO3104	45.22		-0.13	
1146	D445	45.268		0.26	
1201	D445	45.36		1.01	
1243	D445	45.20		-0.30	
1297		----		----	
1328	GB/T265	45.40		1.33	
1435		----		----	
1486	ISO3104	44.3394	G(0.01)	-7.31	
1630	D445	45.1992		-0.30	
1682	D445	45.07		-1.36	
1842	IP71	45.36		1.01	
1877	D445	45.35		0.92	
1959	GB/T265	44.94		-2.41	

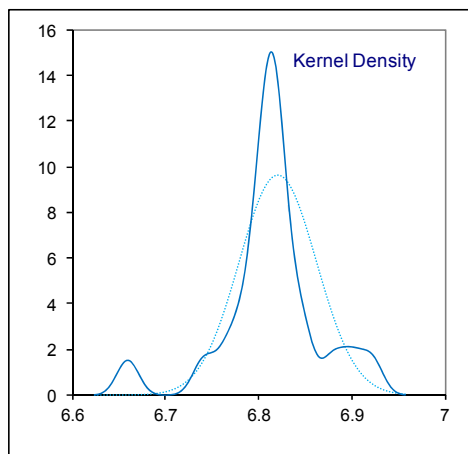
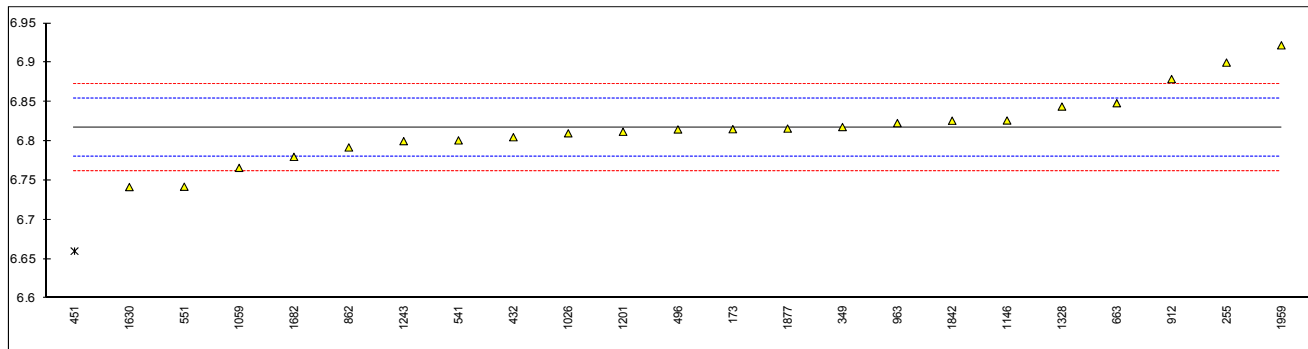
normality OK
n 25
outliers 1
mean (n) 45.2365
st.dev. (n) 0.13078
R(calc.) 0.3662
R(D445:14e2) 0.3439



Determination of Kinematic Viscosity @ 100°C on sample #14219; results in mm/s²

lab	method	value	mark	z(targ)	remarks
173	D445	6.8152		-0.12	
255	D7279	6.90	C	4.47	First reported 6.07
311		-----		-----	
349	D445	6.818		0.03	
432	D445	6.805		-0.67	
451	D7279	6.660	R(0.05)	-8.50	
473		-----		-----	
496	D445	6.8149		-0.13	
541	D445	6.801		-0.89	
551	D7279	6.742		-4.07	
614		-----		-----	
663	D445	6.8483		1.67	
862	D445	6.792		-1.37	
912	D445	6.879		3.33	
963	D445	6.823		0.30	
1026	D445	6.81		-0.40	
1059	ISO3104	6.766		-2.78	
1146	D445	6.8263		0.48	
1201	D445	6.812		-0.29	
1243	D445	6.8		-0.94	
1297		-----		-----	
1328	GB/T265	6.844		1.44	
1435		-----		-----	
1486		-----		-----	
1630	D445	6.7416		-4.10	
1682	D445	6.780		-2.02	
1842	IP71	6.826		0.47	
1877	D445	6.816		-0.07	
1959	GB/T265	6.922	C	5.65	First reported 6.908

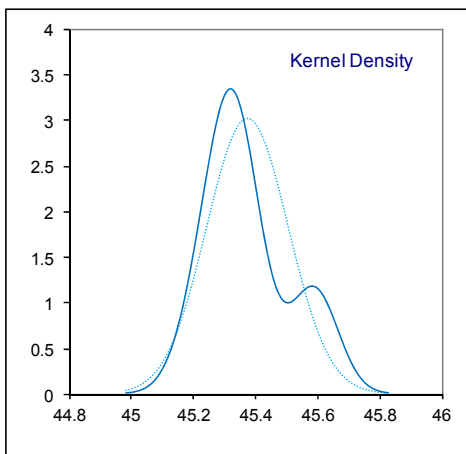
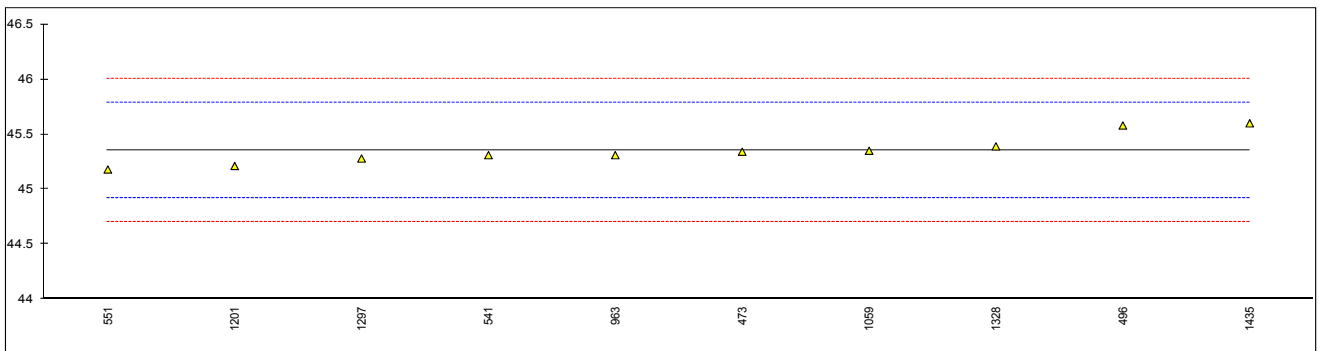
normality suspect
n 22
outliers 1
mean (n) 6.8174
st.dev. (n) 0.04383
R(calc.) 0.1227
R(D445:14e2) 0.0518



Determination of Viscosity Stabinger @ 40°C on sample #14219; results in mm/s²

lab	method	value	mark	z(targ)	remarks
173		----		----	
255		----		----	
311		----		----	
349		----		----	
432		----		----	
451		----		----	
473	D7042	45.3415		-0.06	
496	D7042	45.580		1.03	
541	D7042	45.31		-0.21	
551	D7042	45.18		-0.81	
614		----		----	
663		----		----	
862		----		----	
912		----		----	
963	D7042	45.31		-0.21	
1026		----		----	
1059	D7042	45.35		-0.02	
1146		----		----	
1201	D7042	45.212		-0.66	
1243		----		----	
1297	D7042	45.28		-0.35	
1328	SH/T0870	45.39		0.16	
1435	D7042	45.6		1.13	
1486		----		----	
1630		----		----	
1682		----		----	
1842		----		----	
1877		----		----	
1959		----		----	

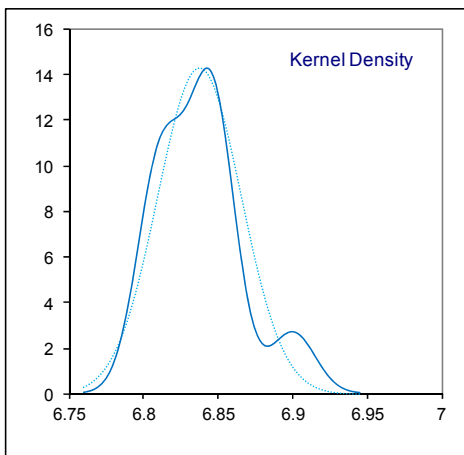
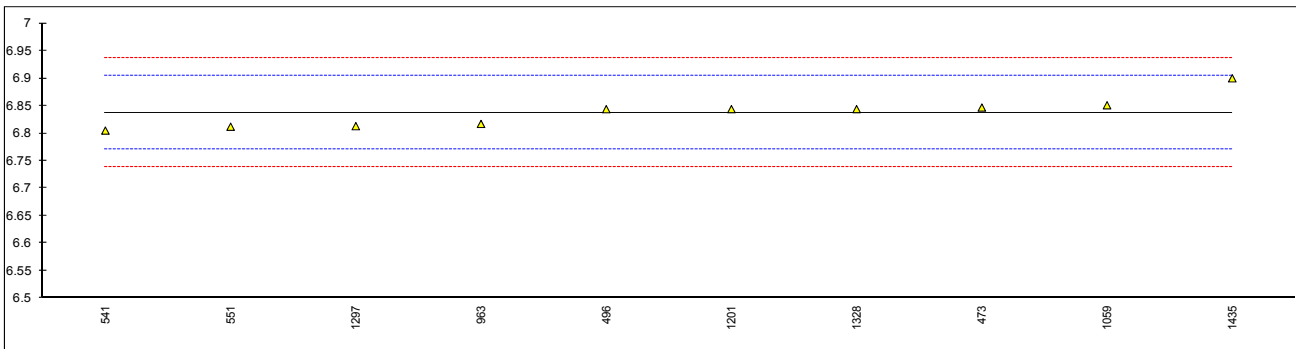
normality suspect
n 10
outliers 0
mean (n) 45.3554
st.dev. (n) 0.13858
R(calc.) 0.3880
R(D7042:14) 0.6082



Determination of Viscosity Stabinger @ 100°C on sample #14219; results in mm/s²

lab	method	value	mark	z(targ)	remarks
173		----		----	
255		----		----	
311		----		----	
349		----		----	
432		----		----	
451		----		----	
473	D7042	6.8469		0.28	
496	D7042	6.8438		0.19	
541	D7042	6.805		-0.99	
551		----		-0.78	
614		----		----	
663		----		----	
862		----		----	
912		----		----	
963	D7042	6.817		-0.62	
1026		----		----	
1059	D7042	6.851		0.40	
1146		----		----	
1201	D7042	6.844		0.19	
1243		----		----	
1297	D7042	6.813		-0.75	
1328	SH/T0870	6.844		0.19	
1435	D7042	6.9		1.88	
1486		----		----	
1630		----		----	
1682		----		----	
1842		----		----	
1877		----		----	
1959		----		----	

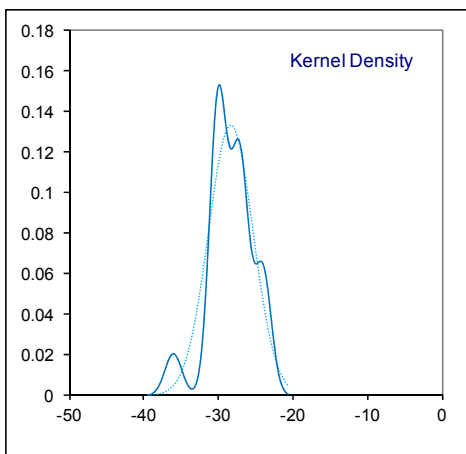
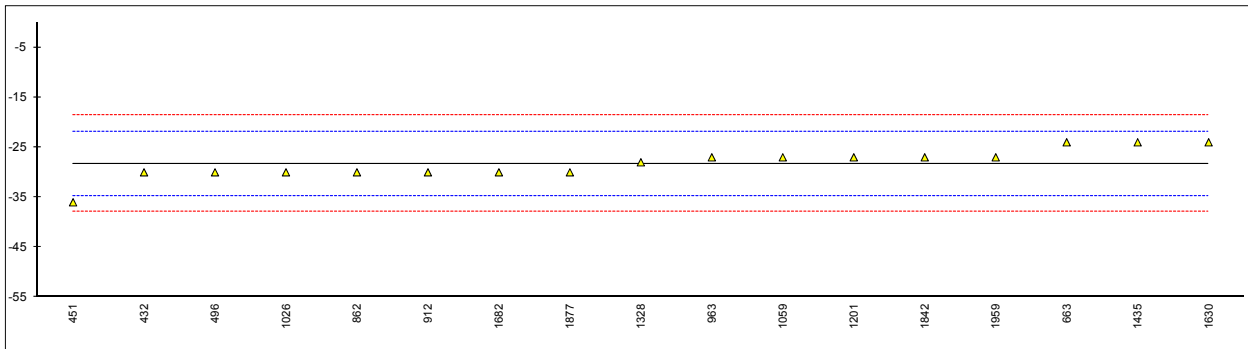
normality Not OK
n 10
outliers 0
mean (n) 6.8377
st.dev. (n) 0.02794
R(calc.) 0.0782
R(D7042:14) 0.0927



Determination of Pour Point, manual on sample #14219; results in °C

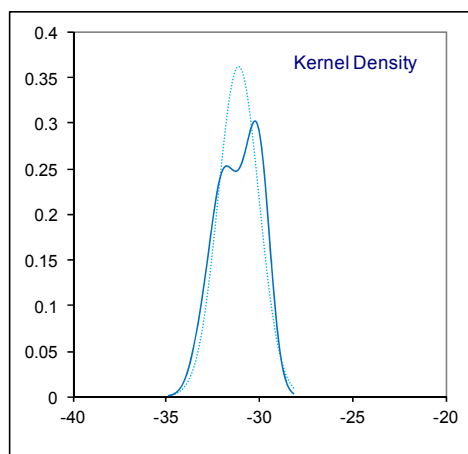
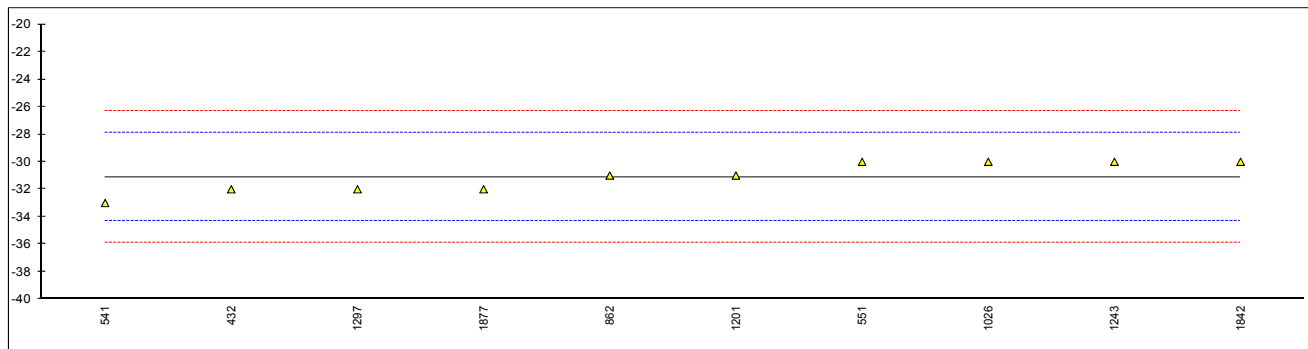
lab	method	value	mark	z(targ)	remarks
173		----		----	
255		----		----	
311		----		----	
349		----		----	
432	D97	-30		-0.53	
451	D5949	-36		-2.40	
473		----		----	
496	D97	-30		-0.53	
541		----		----	
551		----		----	
614		----		----	
663	D97	-24		1.34	
862	D97	-30		-0.53	
912	D97	-30		-0.53	
963	D97	-27		0.40	
1026	D97	-30		-0.53	
1059	ISO3016	-27		0.40	
1146		----		----	
1201	D97	-27		0.40	
1243		----	C	----	Reported first -30 (should be reported as PP automated)
1297		----		----	
1328	GB/T3535	-28		0.09	
1435	D97	-24		1.34	
1486		----		----	
1630	D97	-24		1.34	
1682	D97	-30		-0.53	
1842	D97	-27		0.40	
1877	D97	-30		-0.53	
1959	GB/T3535	-27		0.40	

normality suspect
n 17
outliers 0
mean (n) -28.29
st.dev. (n) 2.995
R(calc.) 8.39
R(D97:12) 9.00



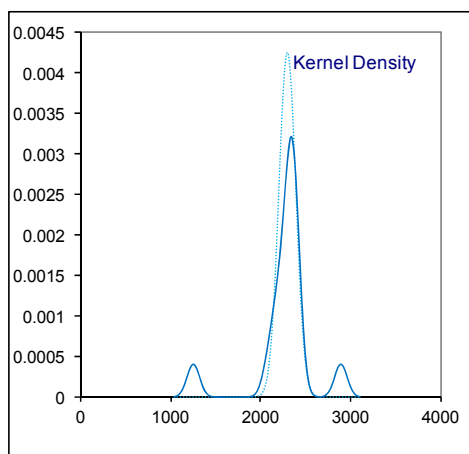
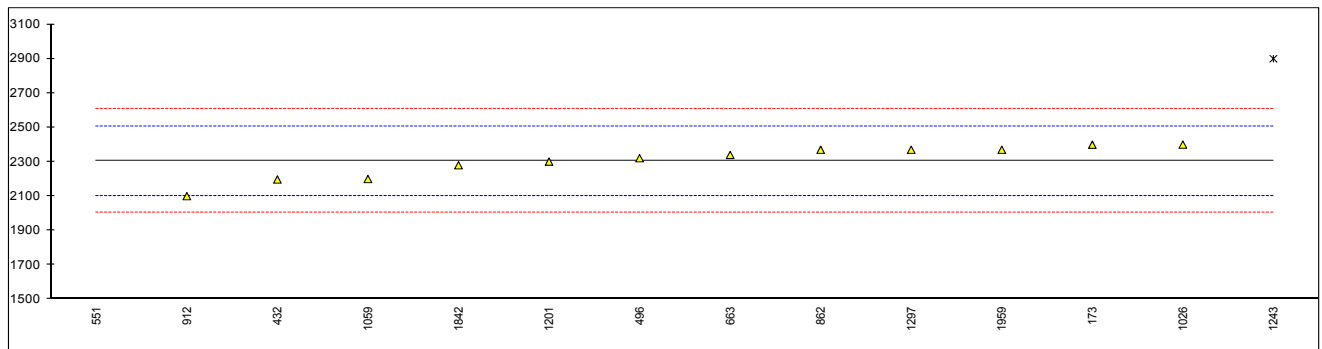
Determination of Pour Point, automated, 1°C interval on sample #14219; results in °C

lab	method	value	mark	z(targ)	remarks
173		----		----	
255		----		----	
311		----		----	
349		----		----	
432	D5950	-32		-0.56	
451		----		----	
473		----		----	
496		----		----	
541	D5950	-33		-1.18	
551	D5950	-30		0.68	
614		----		----	
663		----		----	
862	D5950	-31		0.06	
912		----		----	
963		----		----	
1026	D5950	-30		0.68	
1059		----		----	
1146		----		----	
1201	D5950	-31		0.06	
1243	D5950	-30	C	0.68	Reported first 2900 (should be sulphur, typo error)
1297	D5950	-32		-0.56	
1328		----		----	
1435		----		----	
1486		----		----	
1630		----		----	
1682		----		----	
1842	D5950	-30		0.68	
1877	D5950	-32		-0.56	
1959		----		----	
normality		OK			
n		10			
outliers		0			
mean (n)		-31.10			
st.dev. (n)		1.101			
R(calc.)		3.08			
R(D5950:14)		4.50			



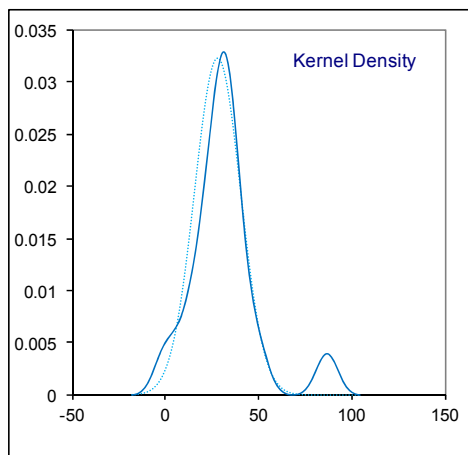
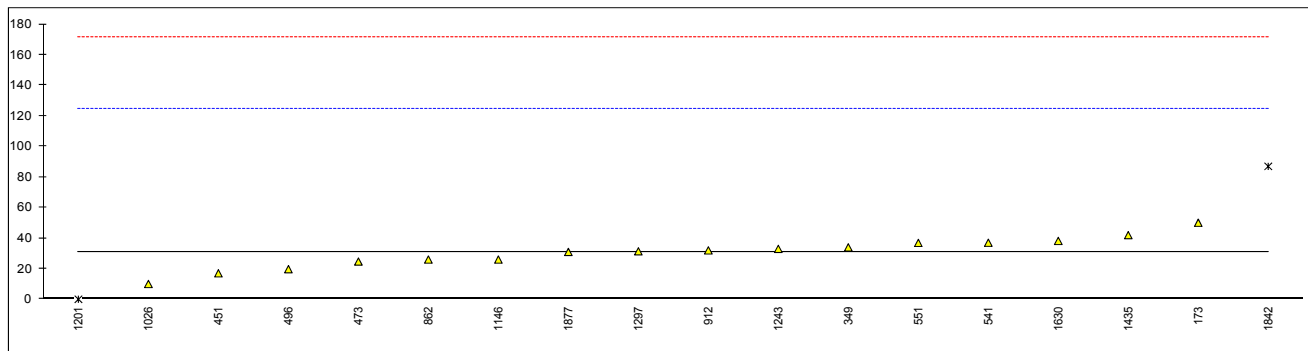
Determination of Sulphur on sample #14219; results in mg/kg

lab	method	value	mark	z(targ)	remarks
173	D4294	2399.6		0.95	
255		----		----	
311		----		----	
349		----		----	
432	D4951	2197		-1.06	
451		----		----	
473		----		----	
496	D2622	2321		0.17	
541		----		----	
551	D5185	1257	G(0.01)	-10.39	
614		----		----	
663	D4294	2340		0.36	
862	D2622	2370		0.65	
912	D5185	2100		-2.03	
963		----		----	
1026	D2622	2400		0.95	
1059	ISO14597	2200		-1.03	
1146		----		----	
1201	ISO8754	2301	C	-0.03	First reported 0.2301 (unit error)
1243		2900	G(0.01)	5.92	
1297	D4294	2370		0.65	
1328		----		----	
1435		----		----	
1486		----		----	
1630		----		----	
1682		----		----	
1842	INH-05	2280		-0.24	
1877		----		----	
1959	GB/T17040	2370		0.65	
normality		OK			
n		12			
outliers		2			
mean (n)		2304.05			
st.dev. (n)		94.135			
R(calc.)		263.58			
R(D4294:10)		282.07			



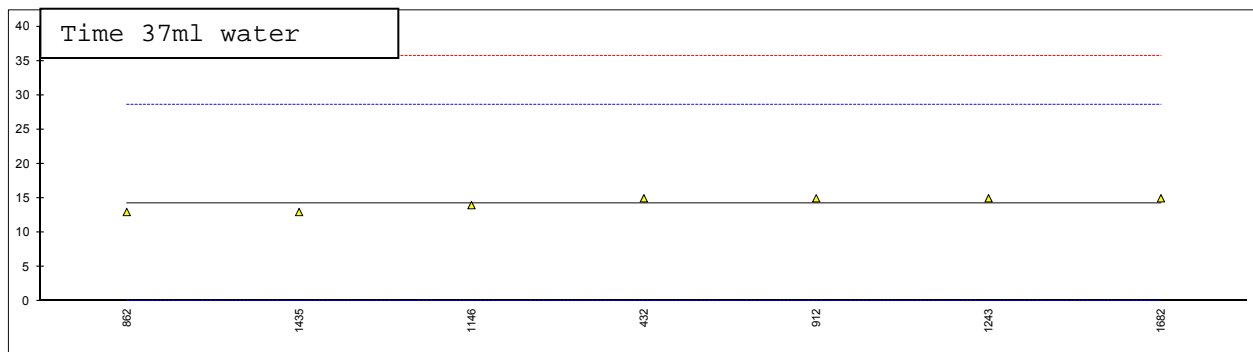
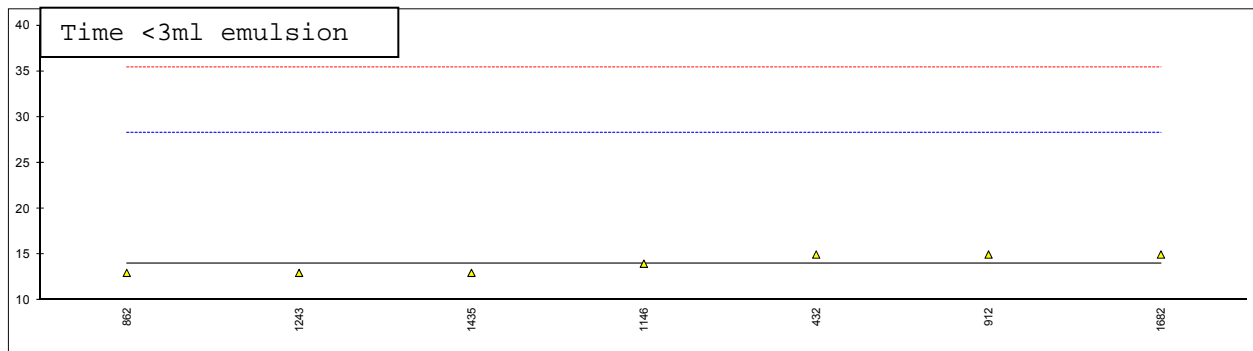
Determination of Water on sample #14219; results in mg/kg

lab	method	value	mark	z(targ)	remarks
173	D6304-A	50.1		0.42	
255		----		----	
311		----		----	
349	D6304-A	34		0.07	
432		----		----	
451	D6304-C	17		-0.29	
473	D6304-C	24.7		-0.12	
496	D6304-C	19.7		-0.23	
541	D6304-A	37		0.14	
551	D6304	36.9		0.14	
614		----		----	
663		----		----	
862	D6304-C	26		-0.10	
912	D6304-C	32		0.03	
963		----		----	
1026	D6304-C	10		-0.44	
1059	ISO12937	<30		----	
1146	D6304-C	26		-0.10	
1201	D6304-C	0	ex	-0.65	Result excluded, zero not a real result
1243	ISO12937	33		0.05	
1297	D6304-A	31.4		0.02	
1328		----		----	
1435	D6304	42		0.24	
1486		----		----	
1630	D6304-A	38.19		0.16	
1682		----		----	
1842	D6304-A	87	G(0.01)	1.20	
1877	D6304-C	31		0.01	
1959		----		----	
normality		OK			
n		16			
outliers		1	(+1 excl)		
mean (n)		30.56			
st.dev. (n)		9.912			
R(calc.)		27.75			
R(D6304:07)		131.46			



Determination of Water Separability at 54 °C on sample #14219; results in min.

lab	method	time to reach			time to reach			time to reach			Test aborted
		≤3 ml emul.	mark	z(targ)	reach 37 ml water	mark	z(targ)	complete break	mark	z(targ)	
173		----		----	----		----	----		----	----
255		----		----	----		----	----		----	----
311		----		----	----		----	----		----	----
349		----		----	----		----	----		----	----
432	D1401	15		0.14	15		0.10	>30		----	Yes
451		----		----	----		----	----		----	----
473		----		----	----		----	----		----	----
496		----		----	----		----	----		----	----
541		----		----	----		----	----		----	----
551		----		----	----		----	----		----	----
614		----		----	----		----	----		----	----
663		----		----	----		----	----		----	----
862	D1401	13		-0.14	13		-0.18	----		----	Yes
912	D1401	15		0.14	15		0.10	20		----	----
963		----		----	----		----	----		----	----
1026		----		----	----		----	----		----	----
1059		----		----	----		----	----		----	----
1146	D1401	14		0.00	14		-0.04	----		----	----
1201		----		----	----		----	----		----	Yes
1243	DIN51396	13		-0.14	15		0.10	----		----	Yes
1297		----		----	----		----	----		----	----
1328		----		----	----		----	----		----	----
1435	D1401	13		-0.14	13		-0.18	----		----	No
1486		----		----	----		----	----		----	----
1630		----		----	----		----	----		----	----
1682	D1401	15		0.14	15		0.10	----		----	No
1842		----		----	----		----	----		----	----
1877		----		----	----		----	----		----	----
1959		----		----	----		----	----		----	----
	normality	n.a.			n.a.			n.a.			
	n	7			7			2			
	outliers	0			0			n.a.			
	mean (n)	14.0			14.29			n.a.			
	st.dev. (n)	1.00			0.95			n.a.			
	R(calc.)	2.8			2.66			n.a.			
	R(D1401:12)	20.0			20.0.			n.a.			



Determination of Water Separability at 54 °C on sample #14219; results in ml.

--- Continued ---

lab	volume oil phase	mark	volume water phase	mark	volume emul. phase	mark
173	----		----		----	
255	----		----		----	
311	----		----		----	
349	----		----		----	
432	42		38		0	
451	----		----		----	
473	----		----		----	
496	----		----		----	
541	----		----		----	
551	----		----		----	
614	----		----		----	
663	----		----		----	
862	41		39		0	
912	----		----		----	
963	----		----		----	
1026	40		40		0	
1059	----		----		----	
1146	40		37		3	
1201	----		----		----	
1243	41		39		0	
1297	----		----		----	
1328	----		----		----	
1435	----		----		----	
1486	----		----		----	
1630	----		----		----	
1682	----		----		----	
1842	----		----		----	
1877	----		----		----	
1959	----		----		----	

Determination of Calcium (Ca) on sample #14219; results in mg/kg.

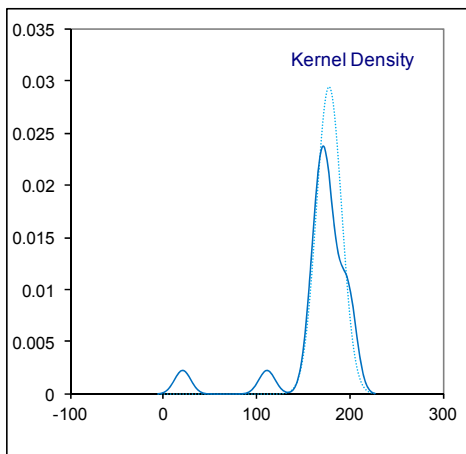
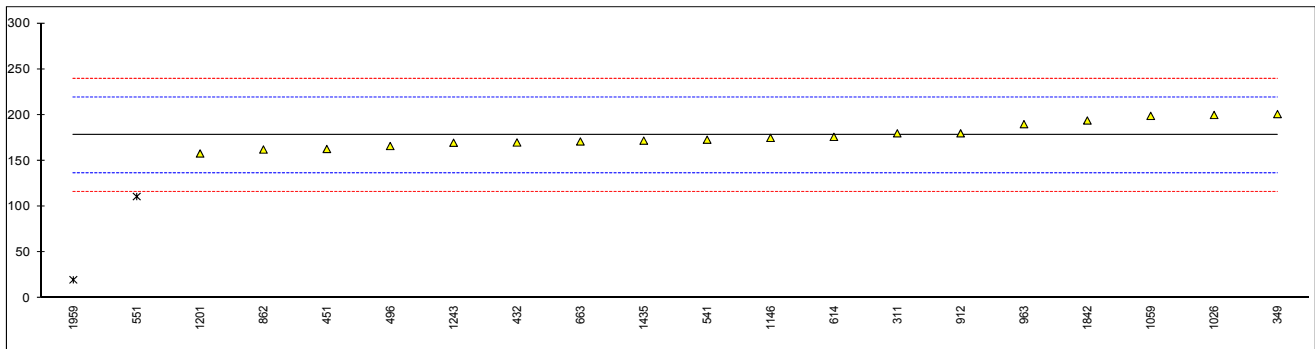
lab	method	value	mark	z(targ)	remarks
173		----		----	
255	INH-02	2.794		----	
311	D5185	<10		----	
349	D5185	0		----	
432	D4951	<1		----	
451	D5185	1.5		----	
473	D5185	0.593		----	
496	DIN51399	0.0		----	
541	D5185	<40		----	
551	D5185	<40		----	
614		----		----	
663	D5185	0.045		----	
862	D5185	<1		----	
912	D5185	1.00		----	
963		----		----	
1026	D5185	< 5		----	
1059	in house	<6		----	
1146	in house	0.0		----	
1201	D5185	0		----	
1243	D5185	0.41		----	
1297		----		----	
1328		----		----	
1435	D5185	<1		----	
1486		----		----	
1630		----		----	
1682		----		----	
1842	INH-01	<1		----	
1877		----		----	
1959	SH/T0749	27.940	C	----	First reported 61.570
	normality	n.a.			
	n	20			
	outliers	0			
	mean (n)	<40			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D5185:13e1)	n.a.			Application range: 40 – 9000 mg/kg

Determination of Phosphorus (P) on sample #14219; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
173		----		----	
255		----		----	
311	D5185	180		0.11	
349	D5185	201		1.13	
432	D4951	170		-0.38	
451	D5185	162.9		-0.73	
473		----		----	
496	DIN51399	166.1		-0.57	
541	D5185	173		-0.23	
551	D5185	111	G(0.01)	-3.26	
614	D5185	176.1		-0.08	
663	D5185	171.1		-0.33	
862	D5185	162.3		-0.76	
912	D5185	180		0.11	
963	D5185	190		0.60	
1026	D5185	200		1.08	
1059	in house	199		1.04	
1146	in house	175		-0.14	
1201	D5185	158		-0.97	
1243	D5185	169.7		-0.40	
1297		----		----	
1328		----		----	
1435	D5185	172		-0.28	
1486		----		----	
1630		----		----	
1682		----		----	
1842	INH-01	194		0.79	
1877		----		----	
1959	SH/T0749	20.124	C,G(0.01)	-7.70	First reported 25.309

normality OK
n 18
outliers 2
mean (n) 177.79
st.dev. (n) 13.572
R(calc.) 38.00
R(D5185:13e1) 57.34

Application range: 10 – 1000 mg/kg



Determination of Zinc (Zn) on sample #14219; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
173		----		----	
255	INH-02	0.38		----	First reported 422.06
311	D5185	<10		----	
349	D5185	1		----	
432	D4951	<1		----	
451	D5185	1.2		----	
473	D5185	0.732		----	
496	DIN51399	0.0		----	
541	D5185	<5		----	
551	D5185	1.15		----	
614		----		----	
663	D5185	2.822		----	
862	D5185	<1		----	
912	D5185	1.00		----	
963		----		----	
1026	D5185	< 5		----	
1059	in house	<3		----	
1146	in house	0.9		----	
1201	D5185	0		----	
1243	D5185	<0.01		----	
1297		----		----	
1328		----		----	
1435	D5185	<1		----	
1486		----		----	
1630		----		----	
1682		----		----	
1842	INH-01	<1		----	
1877		----		----	
1959	SH/T0749	10.697	C	----	First reported 14.264
	normality	n.a.			
	n	20			
	outliers	0			
	mean (n)	<60			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(D5185:13e1)	n.a.			Application range: 60 – 1600 mg/kg

APPENDIX 2

Number of participants per country

1 laboratory in ALGERIA
1 laboratory in ARGENTINA
1 laboratory in AUSTRALIA
1 laboratory in AUSTRIA
2 laboratories in BELGIUM
1 laboratory in BRAZIL
1 laboratory in CHINA, people's Republic
2 laboratories in GERMANY
1 laboratory in GREECE
1 laboratory in HUNGARY
2 laboratories in INDIA
4 laboratories in NETHERLANDS
1 laboratory in NORWAY
1 laboratory in SAUDI ARABIA
1 laboratory in SLOVENIA
1 laboratory in SPAIN
1 laboratory in TANZANIA
1 laboratory in THAILAND
2 laboratories in UNITED KINGDOM
1 laboratory in UNITED STATES OF AMERICA

APPENDIX 3**Abbreviations:**

C	= final result after checking of first reported suspect result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner outlier test
R(0.05)	= straggler in Rosner outlier test
ex	= excluded from calculations
n.a.	= not applicable
E	= error in calculations
U	= reported wrong unit
W	= withdrawn result on request of participant
SDS	= Safety Data Sheet

Literature:

- 1 iis Interlaboratory Studies, Protocol for the Organization, Statistics and Evaluation, April 2014
- 2 ASTM E178-89
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