

**Results of Proficiency Test  
Hydraulic Fluid (fresh)  
November 2015**

**Organised by:** Institute for Interlaboratory Studies  
Spijkenisse, the Netherlands

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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. In the annual program 2014/2015 it was decided to organize a proficiency test for the analysis of fresh Hydraulic Fluid next to used Hydraulic Fluid. This decision was based on the outcome of a questionnaire that was sent to all participants in 2014. During the annual program 2015/2016, it was decided to continue the round robin for the analysis of fresh Hydraulic Fluid. In this second interlaboratory study on fresh Hydraulic Fluid, 47 laboratories from 34 different countries have participated. See appendix 2 for the number of participants per country. In this report, the test results of the 2015 interlaboratory study on fresh Hydraulic Fluid are presented and discussed. This report can also be downloaded from the iis website [www.iisnl.com](http://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 to an accredited laboratory. It was decided to send one sample of one litre of fresh oil (Tellus S46), labelled #15219. 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 of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol can be downloaded from the iis website [www.iisnl.com](http://www.iisnl.com), from the FAQ page.

### 2.3 CONFIDENTIALITY STATEMENT

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

## 2.4 SAMPLES

The necessary bulk material was obtained from a local trader. The approximately 110 litre of the bulk material was homogenised. After homogenisation, 108 amber glass bottles of 1 litre were filled and labelled #15219. The homogeneity of the subsamples #15219 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 at 15 °C in kg/L</i>	<i>Viscosity at 40 °C in mm<sup>2</sup>/s</i>
Sample #15219-1	0.87051	45.25
Sample #15219-2	0.87051	45.25
Sample #15219-3	0.87052	45.26
Sample #15219-4	0.87052	45.26
Sample #15219-5	0.87051	45.23
Sample #15219-6	0.87051	45.23
Sample #15219-7	0.87051	45.23
Sample #15219-8	0.87051	45.24

Table 1: homogeneity test results of subsamples #15219

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

	<i>Density at 15 °C in kg/L</i>	<i>Viscosity at 40 °C in mm<sup>2</sup>/s</i>
r (observed)	0.00001	0.03
reference method	D4052:15	D445:15a
0.3 x R (ref. method)	0.00016	0.17

Table 2: repeatabilities of subsamples #15219

Each calculated repeatability was less than 0.3 times the corresponding reproducibility of the respective test methods. Therefore, homogeneity of the subsamples #15219 was assumed.

To each of the participating laboratories one sample of 1 litre amber glass bottle, labelled #15219, was sent on October 28, 2015.

## 2.5 STABILITY OF THE SAMPLES

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

## 2.6 ANALYSES

The participants were asked to determine; Acid Number (total), Copper Corrosion (3hrs at 50°C), Density at 15°C, Flash Point PMcc, Foam Characteristics (Foam Tendency, Foam Stability), Kinematic Viscosity at 40°C and at 100°C, Viscosity Index, Viscosity Stabinger at 40°C and at 100°C, Pour Point (manual and automated), Sulphur, Water content (by KF), Water Separability at 54°C and Calcium, Phosphorus and Zinc on sample #15219.

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/](http://www.kpmd.co.uk/sgs-iis/).

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 via the data entry portal [www.kpmd.co.uk/sgs-iis/](http://www.kpmd.co.uk/sgs-iis/). The original results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder 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.

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.

According to ISO 5725 the original results per determination were submitted to Dixon's and/or Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) or D(1) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) or D(5) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of 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. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

### 3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation 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, problems were encountered with the dispatch of the samples. Participants in Saudi Arabia and Kazakhstan received the samples late due to customs. Five participants reported the results after the final reporting date and two participants did not report any result at all.

The 45 reporting participants sent in 569 numerical results. Observed were 26 outlying results, which is 4.6% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

### 4.1 EVALUATION PER TEST

In this section, the results are discussed per 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 mentioned in the tables in appendix 1 together with the original data. The abbreviations, used in these tables, are listed in appendix 3. In the iis PT reports, ASTM methods are referred to with a number (e.g. D4052) and an added designation for the year that the method was adopted or revised (e.g. D4052:15).

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.

Acid Number (total): 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 D664:11a.

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

Density at 15°C: This determination was problematic. Four statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of D4052:15.

Flash Point PMcc: This determination was not problematic. No statistical outliers were observed. One result was excluded as the used test method is not equivalent to ASTM D93 (determined in an open cup). The calculated reproducibility is in agreement with the requirements of ASTM D93:15a method A.

Foaming Characteristics (Tendency and Stability): This determination was very problematic. In total two statistical outliers were observed in the Foam Stability determination. The calculated reproducibilities in the Foam Tendency determination for sequences I and III are not at all in agreement with the requirements of ASTM D892:13.

This method is very sensitive in maintenance and execution. Several tips and tricks are given in the method part X1. Possible sources for the large variation are the cleaning and checking of the air diffuser, air tubes and test cylinders, the amount of air used during the test, the way an aliquot of the sample is taken (pre-warmed to remove any thermal history and gently homogenisation of the sample) and the way that the foam height is read in the test cylinder. According to the method the foam layer should completely cover the (oil) surface.

Kinematic Viscosity at 40°C: This determination was problematic for a number of participants.

Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D445:15a.

Kinematic Viscosity at 100°C: This determination was problematic for a number of participants. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in full agreement with the requirements of ASTM D445:15a.

Viscosity Index This determination was problematic for a number of participants. Five statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D2270:10e1.

Viscosity Stabinger at 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 at 100°C: 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 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 D97:15.

Pour Point (automated): This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good 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 content: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility, after rejection of the statistical outliers is in good agreement with the requirements of ASTM D6304:07.
- Water Separability at 54°C: This determination may not be problematic. Four statistical outliers were observed in total over six parameters. The calculated reproducibilities for “time to reach  $\leq$  3ml emulsion” and “time to reach 37ml water” are both in good agreement with the requirements of ASTM D1401:12e1.
- 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 not problematic. No statistical outliers were observed. The calculated reproducibility 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 the reproducibility as found for the group of participating laboratories. The target reproducibilities derived from literature standards and the calculated reproducibilities are compared in the next table.

Parameter	unit	n	mean	2.8 * sd	R (lit)
Acid Number (total)	mg KOH/g	34	0.13	0.07	0.16
Copper Corrosion, 3 hrs at 50°C	rating	26	1	n.a.	n.a.
Density at 15°C	kg/L	36	0.8705	0.0006	0.0005
Flash Point PMcc	°C	34	216.0	11.4	15.3
Foam Tendency Seq. I	ml	14	178	305	67
Foam Tendency Seq. II	ml	14	23.2	17.0	17.5
Foam Tendency Seq. III	ml	13	173	221	76
Foam Stability Seq. I	ml	14	0	n.a.	n.a.
Foam Stability Seq. II	ml	15	0	n.a.	n.a.
Foam Stability Seq. III	ml	13	0	n.a.	n.a.
Kinematic viscosity at 40°C	mm/s <sup>2</sup>	35	45.320	0.539	0.553
Kinematic viscosity at 100°C	mm/s <sup>2</sup>	32	6.808	0.081	0.094
Viscosity Index		29	104.7	1.7	2.0
Viscosity Stabinger at 40°C	mm/s <sup>2</sup>	16	45.333	0.408	0.608
Viscosity Stabinger at 100°C	mm/s <sup>2</sup>	15	6.828	0.078	0.093
Pour Point (manual)	°C	26	-28.2	8.9	9.0
Pour Point (automated), 1°C int.	°C	16	-31.0	3.5	4.5
Sulphur	mg/kg	22	2352	254	286
Water content (by KF)	mg/kg	32	35	31	143
<b>Water Separability at 54°C, distilled water</b>					
Time ≤ 3 ml emulsion	min	14	12.4	8.2	20.0
Time 37 ml water	min	12	13.2	10.5	20.0
Time to reach complete break	min	8	16.4	17.5	n.a.
Volume Oil phase	ml	11	40.0	5.6	n.a.
Volume Water phase	ml	11	38.6	4.0	n.a.
Volume Emulsion phase	ml	11	1.4	7.6	n.a.
Calcium as Ca	mg/kg	28	<40	n.a.	n.a.
Phosphorus as P	mg/kg	29	176	50	57
Zinc as Zn	mg/kg	29	<60	n.a.	n.a.

Table 3: reproducibilities of results of sample #15219

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 2015 WITH THE PREVIOUS PTs.

	<i>November 2015</i>	<i>November 2014</i>
Number of reporting labs	45	29
Number of results reported	569	346
Statistical outliers	26	19
Percentage outliers	4.6%	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 2015</i>	<i>November 2014</i>
Acid Number (total)	++	++
Density at 15°C	-	+/-
Flash Point PMcc	+	-
Foam Tendency Seq. I	--	--
Foam Tendency Seq. II	+/-	--
Foam Tendency Seq. III	--	--
Foam Stability Seq. I	n.e.	n.e.
Foam Stability Seq. II	n.e.	n.e.
Foam Stability Seq. III	n.e.	n.e.
Kinematic viscosity at 40°C	+/-	+/-
Kinematic viscosity at 100°C	+	--
Viscosity Index	+	--
Viscosity Stabinger at 40°C	+	++
Viscosity Stabinger at 100°C	+	+
Pour Point (manual)	+/-	+
Pour Point (automated), 1°C int.	+	+
Sulphur	+	+
Water content (by KF)	++	++
Water Separability ≤ 3ml	++	++
Water Separability 37ml water	++	++
Calcium as Ca	n.e.	n.e.
Phosphorus as P	+	++
Zinc as Zn	n.e.	n.e.

Table 5: comparison determinations against the standard

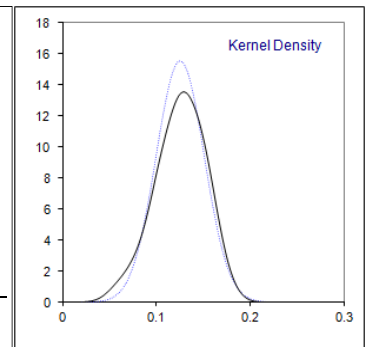
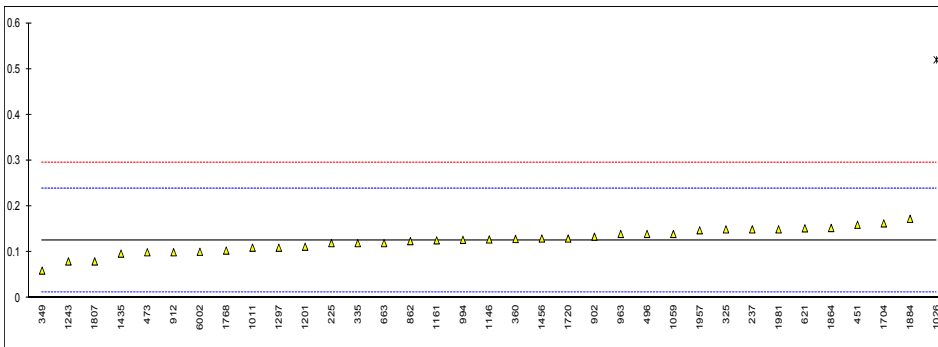
- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- : group performed worse than the standard
- : group performed much worse than the standard

**APPENDIX 1**

**Determination of Acid Number (Total) on sample #15219; results in mg KOH/g**

lab	method	value	mark	z(targ)	remarks
225	D664	0.12		-0.09	
237	D664	0.15		0.44	
255		----		----	
325	D664	0.15		0.44	
335	ISO6618	0.12		-0.09	
349	D664	0.06		-1.15	
360	ISO6618	0.129		0.06	
432		----		----	
451	D664	0.16		0.61	
473	D664	0.1		-0.45	
496	D664	0.14		0.26	
541		----		----	
614		----		----	
621	D664	0.1523		0.48	
663	D664	0.12		-0.09	
862	D664	0.1243		-0.02	
902	D664	0.134		0.15	
912	D664	0.1		-0.45	
963	D974	0.140		0.26	
994	D664	0.127		0.03	
1011	D664	0.11		-0.27	Also reported D974: 0.15 mg KOH/g
1026	D664	0.52	R(0.01)	6.96	
1059	ISO6619	0.14		0.26	
1146	D664	0.128		0.05	
1161	D664	0.126		0.01	
1184		----		----	
1201	D664	0.112		-0.24	
1243	D664	0.08		-0.80	
1297	D664	0.11		-0.27	
1435	D664-A	0.097		-0.50	
1456	D664	0.13		0.08	
1461		----		----	
1660		----		----	
1704	D664	0.163		0.66	
1720	D664	0.13		0.08	
1748		----		----	
1768	ISO6618	0.1037		-0.38	
1799		----		----	
1807	D664	0.08	C	-0.80	first reported: 0.05
1842		----		----	
1864	D664	0.153		0.49	
1877		----		----	
1884	D664	0.173		0.84	
1957	D664	0.148		0.40	
1981	D974	0.15		0.44	
6002	D664	0.101		-0.43	
6016		----		----	

normality OK  
n 34  
outliers 1  
mean (n) 0.1253  
st.dev. (n) 0.02568  
R(calc.) 0.0719  
R(D664:11a) 0.1587

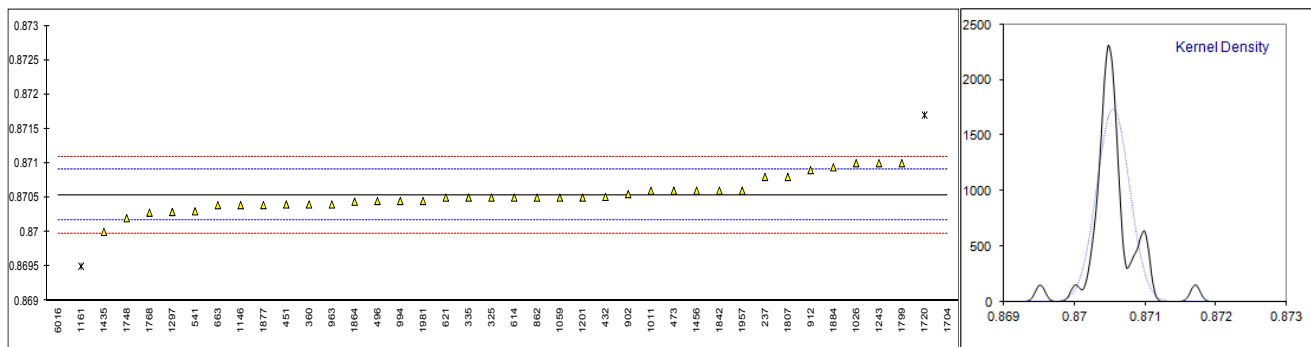


## Determination of Copper Corrosion 3hrs/50°C on sample #15219; results in rating

lab	method	value	mark	z(targ)	remarks
225		----		----	
237		----		----	
255		----		----	
325	D130	1A		----	
335		----		----	
349		----		----	
360	D130	1A		----	
432		----		----	
451		----		----	
473		----		----	
496	D130	1A		----	
541	D130	1A		----	
614	D130	1A		----	
621	D130	1A		----	
663	D130	1A		----	
862	D130	1A		----	
902		----		----	
912	D130	1A		----	
963		----		----	
994	D130	1A		----	
1011	D130	1B		----	
1026	D130	1A		----	
1059	ISO2160	1A		----	
1146		----		----	
1161	ISO2160	1A		----	
1184		----		----	
1201	D130	1A		----	
1243	ISO2160	1B		----	
1297	D130	1A		----	
1435		----		----	
1456	D130	1A		----	
1461		----		----	
1660		----		----	
1704		----		----	
1720		----		----	
1748	D130	1A		----	
1768		----		----	
1799	D130	1B		----	reported: 3hrs at 100°C
1807	D130	1A		----	
1842	IP154	1B		----	
1864	D130	1A		----	
1877		----		----	
1884	D130	1A		----	
1957		----		----	
1981	D130	1A		----	
6002	ISO2160	1A		----	
6016		----		----	
	n	26			
	mean (n)	1			

Determination of Density at 15°C on sample #15219; results in kg/L

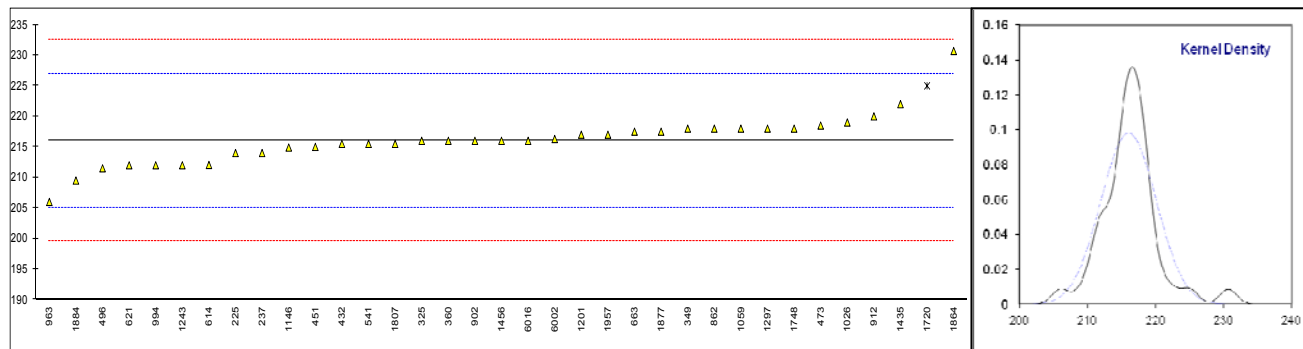
lab	method	value	mark	z(targ)	remarks
225		----		----	
237	D4052	0.8708		1.43	
255		----		----	
325	D4052	0.8705		-0.18	
335	D4052	0.8705		-0.18	
349		----		----	
360	D4052	0.8704		-0.72	
432	D4052	0.87051		-0.13	
451	D4052	0.8704		-0.72	
473	D4052	0.8706		0.35	
496	D4052	0.87045		-0.45	
541	D4052	0.8703	C	-1.26	first reported: 0.8700
614	D4052	0.8705		-0.18	
621	D4052	0.8705		-0.18	
663	D4052	0.87039		-0.78	
862	D4052	0.8705		-0.18	
902	D4052	0.87055		0.09	
912	D4052	0.8709		1.97	
963	D4052	0.8704		-0.72	
994	D4052	0.87045		-0.45	
1011	D4052	0.8706		0.35	
1026	D4052	0.8710		2.51	
1059	ISO12185	0.8705		-0.18	
1146	ISO12185	0.87039		-0.78	
1161	ISO3675	0.8695	R(0.01)	-5.57	
1184		----		----	
1201	D4052	0.8705		-0.18	
1243	ISO12185	0.8710		2.51	
1297	D4052	0.87029		-1.31	
1435	D4052	0.870		-2.88	
1456	D4052	0.8706		0.35	
1461		----		----	
1660		----		----	
1704	D1298	0.8813	C,R(0.01)	57.97	first reported: 0.868
1720	D4052	0.8717	C,R(0.01)	6.28	first reported: 871.7 kg/L
1748	D4052	0.8702		-1.80	
1768	EN ISO3675	0.87028		-1.37	
1799	D4052	0.8710		2.51	
1807	D4052	0.8708		1.43	
1842	D4052	0.8706		0.35	
1864	D4052	0.87044		-0.51	
1877	D4052	0.87039		-0.78	
1884	D4052	0.87094		2.19	
1957	D4052	0.8706		0.35	
1981	D4052	0.87045		-0.45	
6002		----		----	
6016	D4052	0.86849	R(0.01)	-11.01	
normality	OK				
n	36				
outliers	4				
mean (n)	0.870534				
st.dev. (n)	0.0002295				
R(calc.)	0.00064				
R(D4052:15)	0.00052				



Determination of Flash Point PMcc on sample #15219; results in °C

lab	method	value	mark	z(targ)	remarks
225	D93-A	214		-0.37	
237	D93-A	214		-0.37	
255		----		----	
325	D93-A	216.0		-0.01	
335		----		----	
349	D93-A	218		0.36	
360	D93-A	216.0		-0.01	
432	D93-A	215.5		-0.10	
451	D93-A	215		-0.19	
473	D93-A	218.5		0.45	
496	D93-A	211.5		-0.83	
541	D93-A	215.5		-0.10	
614	D93-A	212.05		-0.73	
621	D93-A	212.0		-0.74	
663	D93-A	217.5		0.27	
862	D93-A	218.0		0.36	
902	D93-A	216.0		-0.01	
912	D93-A	220		0.73	
963	D93-A	206		-1.83	
994	D93-A	212.0		-0.74	
1011		----		----	
1026	D93-A	219		0.54	
1059	ISO2719-A	218.0		0.36	
1146	INH-93A	214.9		-0.21	
1161		----		----	
1184		----		----	
1201	D93-A	217.0		0.18	
1243	D93-A	212		-0.74	
1297	D93-B	218.0		0.36	
1435	D93-A	222		1.09	
1456	D93-A	216.0		-0.01	
1461		----		----	
1660		----		----	
1704		----		----	
1720	D92	225.0	ex	1.64	excluded; open cup method is not equivalent to Flash Point PMcc
1748	D93-A	218		0.36	
1768		----		----	
1799		----		----	
1807	D93-A	215.5	C	-0.10	first reported: 175.5
1842		----		----	
1864		230.7		2.68	
1877	D93-A	217.5		0.27	
1884	D93-A	209.5		-1.19	
1957	D93-A	217.0		0.18	
1981		----		----	
6002	ISO2719-A	216.3		0.05	
6016	D93-B	216		-0.01	

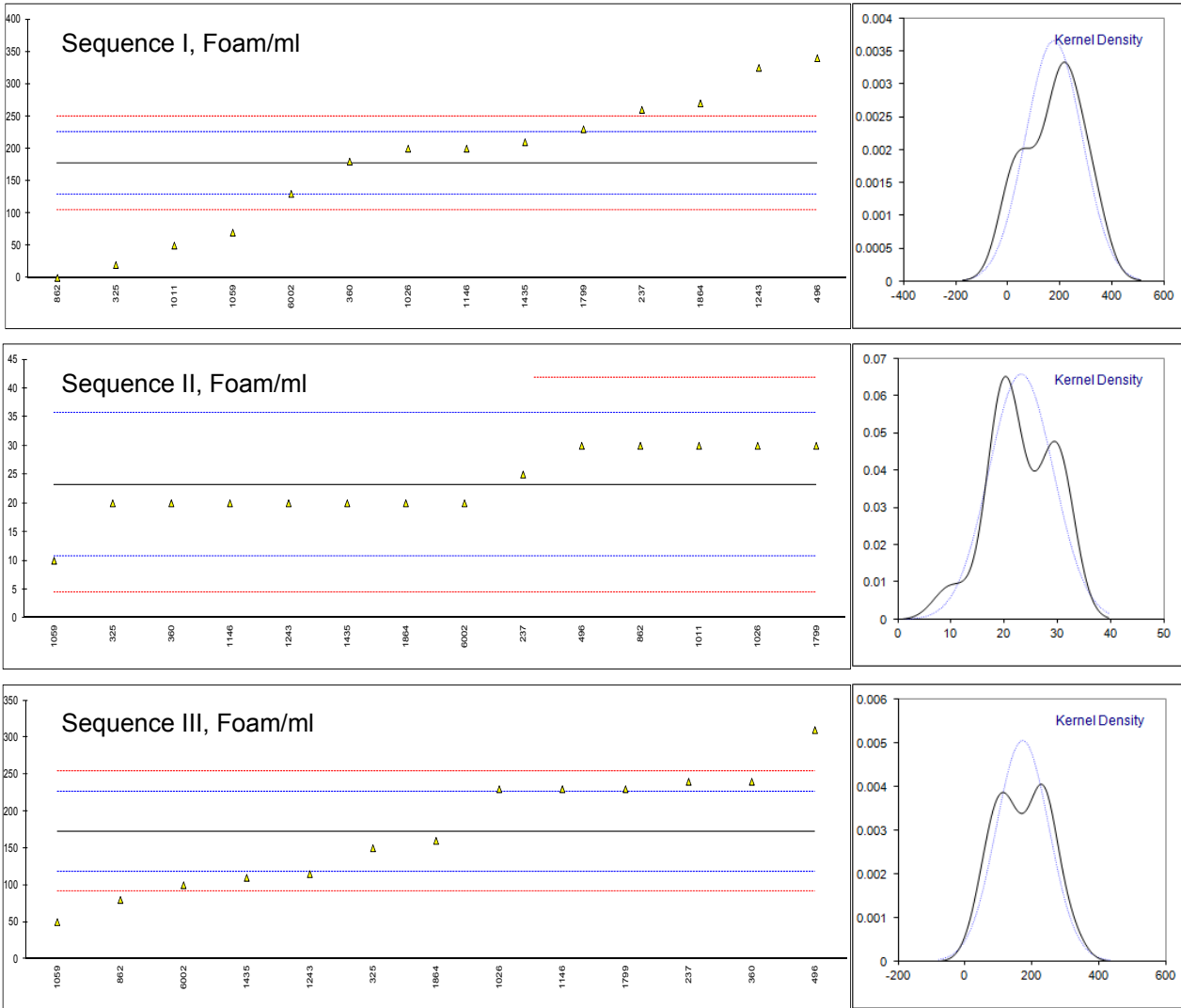
normality not OK  
n 34  
outliers 0+1ex  
mean (n) 216.03  
st.dev. (n) 4.071  
R(calc.) 11.40  
R(D93-A:15a) 15.34



Determination of Foaming Characteristics, Foaming Tendency (at end of 5 min blowing period) on sample #15219; results in ml

lab	method	Seq. I, Foam mark	z(targ)	Seq. II, Foam mark	z(targ)	Seq. III, Foam mark	z(targ)
225		----	----	----	----	----	----
237	D892	260	3.43	25	0.29	240	2.48
255		----	----	----	----	----	----
325	D892	20	-6.54	20	-0.52	150	-0.84
335		----	----	----	----	----	----
349		----	----	----	----	----	----
360	D892	180	0.10	20	-0.52	240	2.48
432		----	----	----	----	----	----
451		----	----	----	----	----	----
473		----	----	----	----	----	----
496	D892	340	6.75	30	1.09	310	5.06
541		----	----	----	----	----	----
614		----	----	----	----	----	----
621		----	----	----	----	----	----
663		----	----	----	----	----	----
862	D892	0	-7.37	30	1.09	80	-3.42
902		----	----	----	----	----	----
912		----	----	----	----	----	----
963		----	----	----	----	----	----
994		----	----	----	----	----	----
1011	D892	50	-5.29	30	1.09	----	----
1026	D892	200	0.93	30	1.09	230	2.11
1059	D892	70	-4.46	10	-2.12	50	-4.52
1146	ISO6247	200	0.93	20	-0.52	230	2.11
1161		----	----	----	----	----	----
1184		----	----	----	----	----	----
1201		----	----	----	----	----	----
1243	D892	325	6.13	20	-0.52	115	-2.13
1297		----	----	----	----	----	----
1435	ISO6247	210	1.35	20	-0.52	110	-2.31
1456		----	----	----	----	----	----
1461		----	----	----	----	----	----
1660		----	----	----	----	----	----
1704		----	----	----	----	----	----
1720		----	----	----	----	----	----
1748		----	----	----	----	----	----
1768		----	----	----	----	----	----
1799	D892	230	2.18	30	1.09	230	2.11
1807		----	----	----	----	----	----
1842		----	----	----	----	----	----
1864	D892	270	3.84	20	-0.52	160	-0.47
1877		----	----	----	----	----	----
1884		----	----	----	----	----	----
1957		----	----	----	----	----	----
1981		----	----	----	----	----	----
6002	ISO6247	130	-1.97	20	-0.52	100	-2.68
6016		----	----	----	----	----	----
	normality	OK		OK		OK	
	n	14		14		13	
	outliers	0		0		0	
	mean (n)	177.50		23.214		172.692	
	st.dev. (n)	109.100		6.0787		78.8621	
	R(calc.)	305.48		17.020		220.814	
	R(D892:13)	67.42		17.476		75.985	





Determination of Foaming Characteristics, Foaming Stability (at end of 10 min settling period) on sample #15219; results in ml

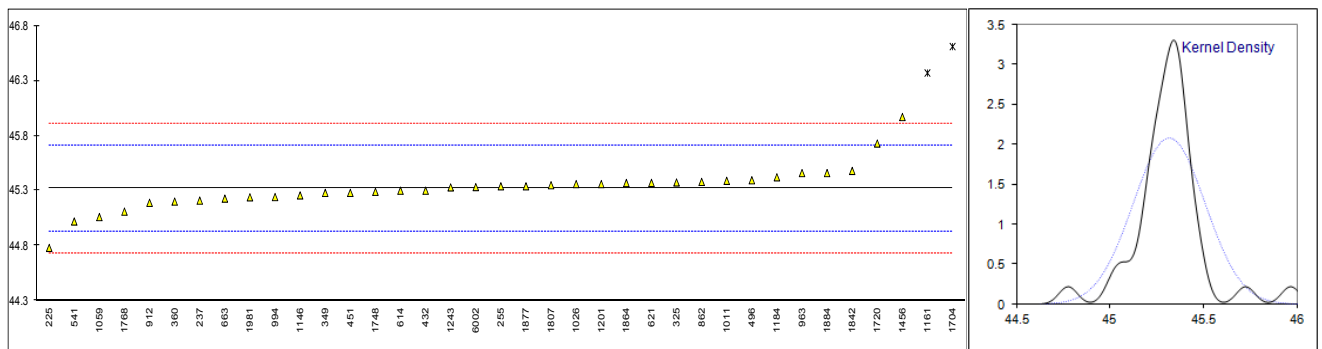
lab	method	Seq. I, Foam	mark	z(targ)	Seq. II, Foam	mark	z(targ)	Seq. III, Foam	mark	z(targ)
225		----		----	----		----	----		----
237	D892	0		----	0		----	0		----
255		----		----	----		----	----		----
325	D892	0		----	0		----	0		----
335		----		----	----		----	----		----
349		----		----	----		----	----		----
360	D892	0		----	0		----	0		----
432		----		----	----		----	----		----
451		----		----	----		----	----		----
473		----		----	----		----	----		----
496	D892	0		----	0		----	0		----
541		----		----	----		----	----		----
614		----		----	----		----	----		----
621		----		----	----		----	----		----
663		----		----	----		----	----		----
862	D892	0		----	0		----	0		----
902		----		----	----		----	----		----
912		----		----	----		----	----		----
963		----		----	----		----	----		----
994		----		----	----		----	----		----
1011	D892	0		----	0		----	0		----
1026	D892	50	G(1), f+	----	0		----	50	G(1), f+	----
1059	D892	0		----	0		----	0		----
1146	ISO6247	0		----	0		----	0		----
1161		----		----	----		----	----		----
1184		----		----	----		----	----		----
1201		----		----	----		----	----		----
1243	D892	0		----	0		----	0		----
1297		----		----	----		----	----		----
1435	ISO6247	0		----	0		----	0		----
1456		----		----	----		----	----		----
1461		----		----	----		----	----		----
1660		----		----	----		----	----		----
1704		----		----	----		----	----		----
1720		----		----	----		----	----		----
1748		----		----	----		----	----		----
1768		----		----	----		----	----		----
1799	D892	0		----	0		----	0		----
1807		----		----	----		----	----		----
1842		----		----	----		----	----		----
1864	D892	0		----	0		----	0		----
1877		----		----	----		----	----		----
1884	D892	0		----	0		----	0		----
1957		----		----	----		----	----		----
1981		----		----	----		----	----		----
6002	ISO6247	0		----	0		----	0		----
6016		----		----	----		----	----		----
n		14			15			13		
outliers		1			0			1		
mean (n)		0			0			0		

f+ = possible false positive test result

Determination of Kinematic Viscosity at 40°C on sample #15219; results in mm/s<sup>2</sup>

lab	method	value	mark	z(targ)	remarks
225	D445	44.78		-2.73	
237	D445	45.21		-0.56	
255	D7279	45.34		0.10	
325	D445	45.375		0.28	
335		----		----	
349	D445	45.28		-0.20	
360	D445	45.201		-0.60	
432	D445	45.30		-0.10	
451	D7279	45.28		-0.20	
473		----		----	
496	D445	45.395		0.38	
541	D445	45.02		-1.52	
614	D445	45.3		-0.10	
621	D445	45.371		0.26	
663	D445	45.228		-0.47	
862	D445	45.38		0.30	
902		----		----	
912	D445	45.19		-0.66	
963	D445	45.46		0.71	
994	D445	45.242		-0.40	
1011	D7042	45.39		0.35	
1026	D445	45.36		0.20	
1059	ISO3104	45.06		-1.32	
1146	D445	45.257		-0.32	
1161	ISO3104	46.37	R(0.01)	5.32	
1184	D445	45.422		0.52	
1201	D445	45.36		0.20	
1243	D7279	45.33		0.05	
1297		----		----	
1435		----		----	
1456	D445	45.97		3.29	
1461		----		----	
1660		----		----	
1704	D445	46.61	C,R(0.01)	6.53	first reported: 45.096
1720	D7042	45.729		2.07	
1748	D445	45.29		-0.15	
1768	ISO3104	45.1096		-1.07	
1799		----		----	
1807	D445	45.35		0.15	
1842	IP71	45.48		0.81	
1864	D445	45.37		0.25	
1877	D445	45.34		0.10	
1884	D445	45.46		0.71	
1957		----		----	
1981	D445	45.24		-0.41	
6002	ISO3104	45.333		0.07	
6016		----		----	

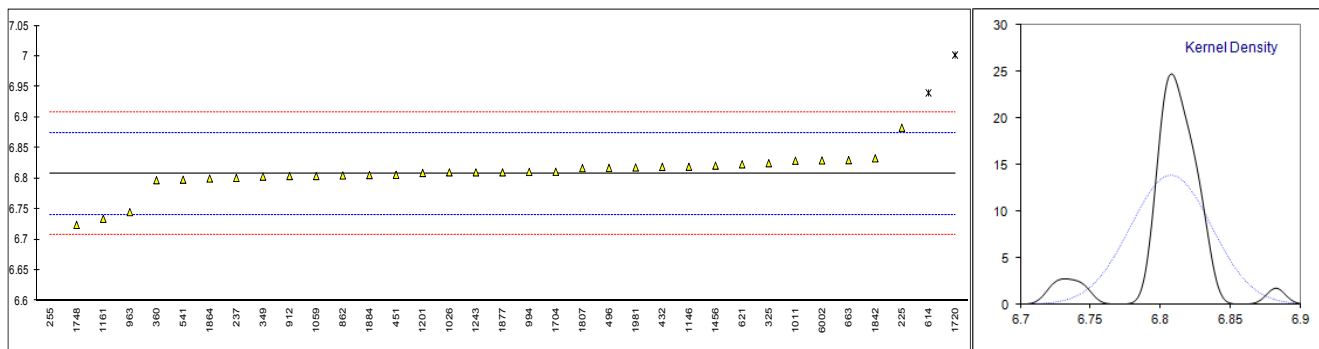
normality not OK  
n 35  
outliers 2  
mean (n) 45.3201  
st.dev. (n) 0.19242  
R(calc.) 0.5388  
R(D445:15a) 0.5529



Determination of Kinematic Viscosity at 100°C on sample #15219; results in mm/s<sup>2</sup>

lab	method	value	mark	z(targ)	remarks
225	D445	6.883		2.24	
237	D445	6.801		-0.20	
255	D7279	6.345	C,R(0.01)	-13.79	first reported: 6.94
325	D445	6.825		0.51	
335		----		----	
349	D445	6.803	C	-0.14	first reported: 6.531
360	D445	6.7972		-0.32	
432	D445	6.819		0.33	
451	D7279	6.806		-0.06	
473		----		----	
496	D445	6.8173		0.28	
541	D445	6.798		-0.29	
614	D445	6.94	R(0.01)	3.94	
621	D445	6.8232		0.46	
663	D445	6.8300		0.66	
862	D445	6.805		-0.09	
902		----		----	
912	D445	6.804		-0.12	
963	D445	6.745		-1.87	
994	D445	6.8107		0.08	
1011	D7042	6.829		0.63	
1026	D445	6.81		0.06	
1059	ISO3104	6.804		-0.12	
1146	D445	6.8192		0.34	
1161	ISO3104	6.734		-2.20	
1184		----		----	
1201	D445	6.809		0.03	
1243	D7279	6.81		0.06	
1297		----		----	
1435		----		----	
1456	D445	6.821		0.39	
1461		----		----	
1660		----		----	
1704	D445	6.811	C	0.09	first reported: 6.893
1720	D7042	7.002	C,R(0.01)	5.79	first reported: 6.946
1748	D445	6.724		-2.50	
1768		----		----	
1799		----		----	
1807	D445	6.817		0.27	
1842	IP71	6.833		0.75	
1864	D445	6.7999		-0.24	
1877	D445	6.810		0.06	
1884	D445	6.8056		-0.07	
1957		----		----	
1981	D445	6.818		0.30	
6002	ISO3104	6.8295		0.64	
6016		----		----	

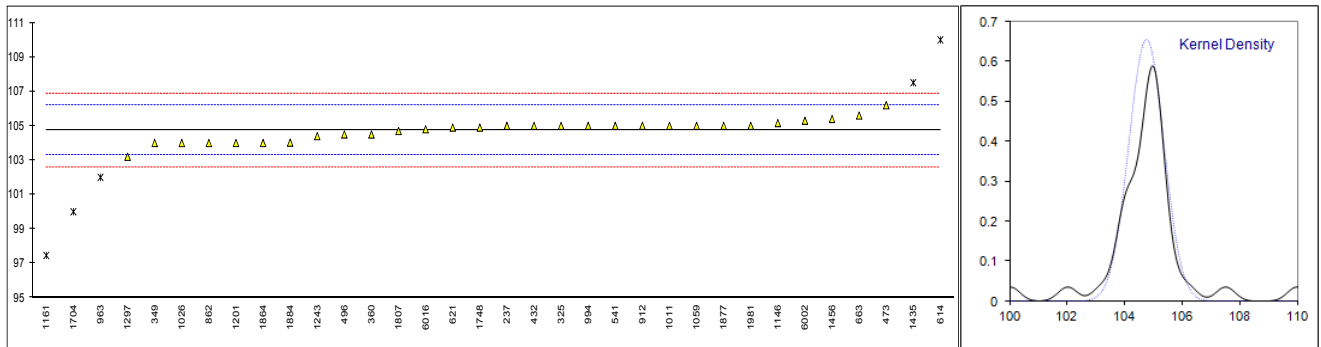
normality not OK  
n 32  
outliers 3  
mean (n) 6.8079  
st.dev. (n) 0.02884  
R(calc.) 0.0807  
R(D445:15a) 0.0939



Determination of Viscosity Index on sample #15219; unit less results

lab	method	value	mark	z(targ)	remarks
225		----		----	
237	D2270	105		0.36	
255		----		----	
325	D2270	105		0.36	
335		----		----	
349	D2270	104	C	-1.04	first reported: 93
360	ISO2909	104.5		-0.34	
432	D2270	105.0		0.36	
451		----		----	
473	D2270	106.2		2.04	
496	D2270	104.5		-0.34	
541	D2270	105		0.36	
614	D2270	110	R(0.01)	7.36	
621	D2270	104.9		0.22	
663	D2270	105.6		1.20	
862	D2270	104		-1.04	
902		----		----	
912	D2270	105		0.36	
963	D2270	102	R(0.01)	-3.84	
994	D2270	105		0.36	
1011	D2270	105		0.36	
1026	D2270	104		-1.04	
1059	ISO2909	105		0.36	
1146	D2270	105.162		0.58	
1161	D2270	97.46	R(0.01)	-10.20	
1184		----		----	
1201	D2270	104		-1.04	
1243	D2270	104.4		-0.48	
1297	D2270	103.2		-2.16	
1435	D2270	107.51	R(0.01)	3.87	
1456	D2270	105.4		0.92	
1461		----		----	
1660		----		----	
1704	D2270	100	C,R(0.01)	-6.64	first reported: 108.67
1720		----		----	
1748	D2270	104.9		0.22	
1768		----		----	
1799		----		----	
1807	D2270	104.7		-0.06	
1842		----		----	
1864	D2270	104		-1.04	
1877	D2270	105		0.36	
1884	D2270	104.0248		-1.01	
1957		----		----	
1981	D2270	105		0.36	
6002	ISO2909	105.3		0.78	
6016	D2270	104.8		0.08	

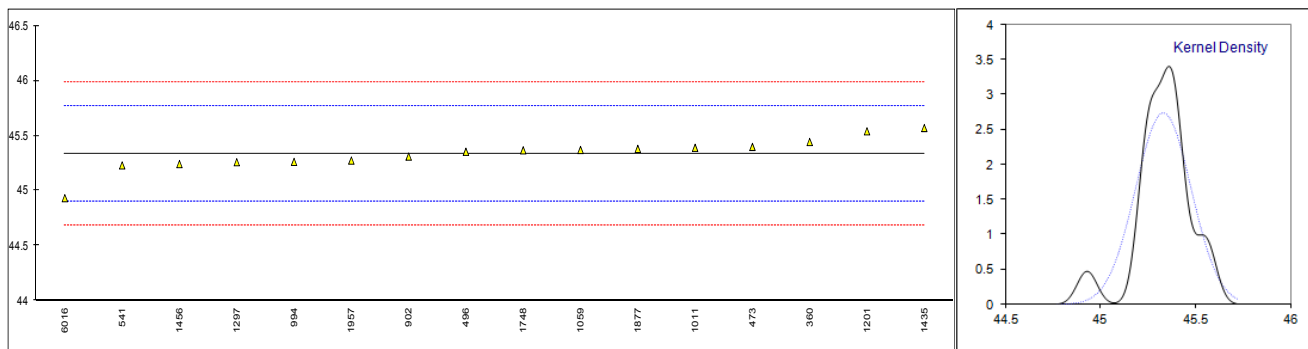
normality OK  
n 29  
outliers 5  
mean (n) 104.74  
st.dev. (n) 0.609  
R(calc.) 1.71  
R(D2270:10e1) 2.00



Determination of Viscosity Stabinger at 40°C on sample #15219; results in mm/s<sup>2</sup>

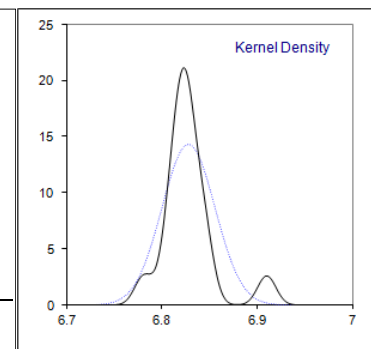
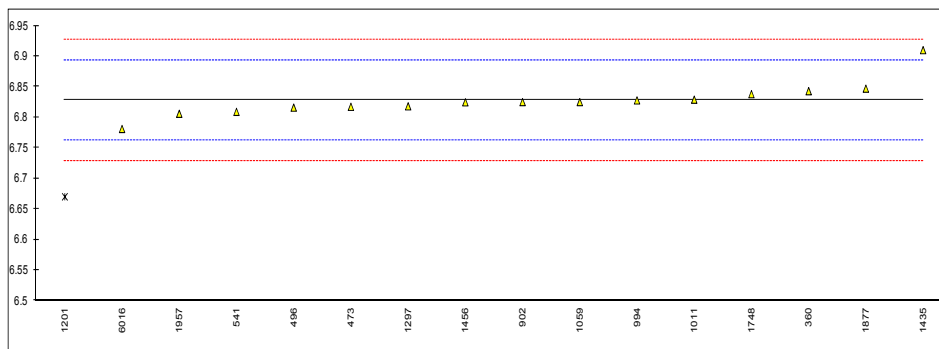
lab	method	value	mark	z(target)	remarks
225		----		----	
237		----		----	
255		----		----	
325		----		----	
335		----		----	
349		----		----	
360	D7042	45.443		0.51	
432		----		----	
451		----		----	
473	D7042	45.398		0.30	
496	D7042	45.354		0.10	
541	D7042	45.23		-0.47	
614		----		----	
621		----		----	
663		----		----	
862		----		----	
902	D7042	45.31		-0.10	
912		----		----	
963		----		----	
994	D7042	45.262		-0.32	
1011	D7042	45.39		0.26	
1026		----		----	
1059	D7042	45.37		0.17	
1146		----		----	
1161		----		----	
1184		----		----	
1201	D7042	45.54		0.96	
1243		----		----	
1297	D7042	45.259		-0.34	
1435	D7042	45.57		1.09	
1456	D7042	45.242		-0.42	
1461		----		----	
1660		----		----	
1704		----		----	
1720		----		----	
1748	D7042	45.367		0.16	
1768		----		----	
1799		----		----	
1807		----		----	
1842		----		----	
1864		----		----	
1877	D7042	45.38		0.22	
1884		----		----	
1957	D7042	45.274		-0.27	
1981		----		----	
6002		----		----	
6016	D7042	44.932		-1.84	

normality not OK  
n 16  
outliers 0  
mean (n) 45.3326  
st.dev. (n) 0.14560  
R(calc.) 0.407675  
R(D7042:14) 0.60799



Determination of Viscosity Stabinger at 100°C on sample #15219; results in mm/s<sup>2</sup>

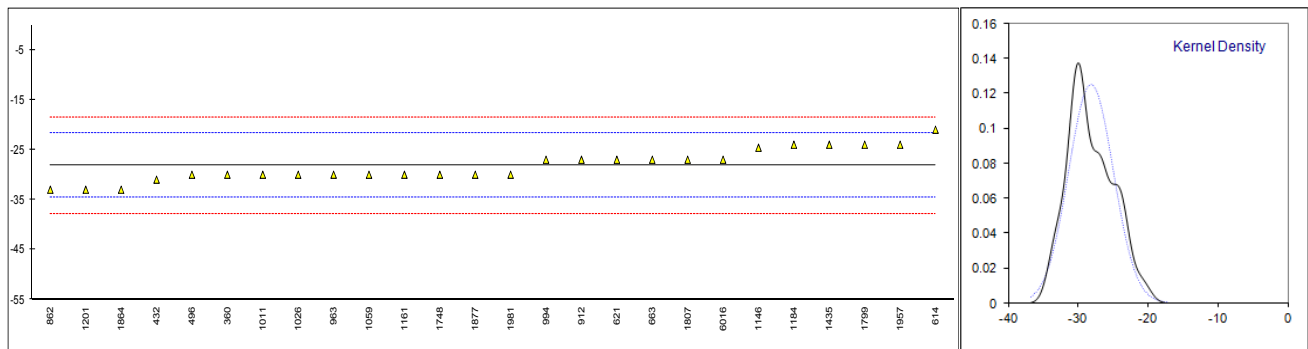
lab	method	value	mark	z(targ)	remarks
225		----		----	
237		----		----	
255		----		----	
325		----		----	
335		----		----	
349		----		----	
360	D7042	6.8429		0.46	
432		----		----	
451		----		----	
473	D7042	6.8173		-0.32	
496	D7042	6.81596		-0.36	
541	D7042	6.809		-0.57	
614		----		----	
621		----		----	
663		----		----	
862		----		----	
902	D7042	6.825		-0.08	
912		----		----	
963		----		----	
994	D7042	6.8278		0.00	
1011	D7042	6.829		0.04	
1026		----		----	
1059	D7042	6.825		-0.08	
1146		----		----	
1161		----		----	
1184		----		----	
1201	D7042	6.670	D(0.01)	-4.77	
1243		----		----	
1297	D7042	6.8181		-0.29	
1435	D7042	6.91		2.49	
1456	D7042	6.8247		-0.09	
1461		----		----	
1660		----		----	
1704		----		----	
1720		----		----	
1748	D7042	6.838		0.31	
1768		----		----	
1799		----		----	
1807		----		----	
1842		----		----	
1864		----		----	
1877	D7042	6.847		0.58	
1884		----		----	
1957	D7042	6.8059		-0.66	
1981		----		----	
6002		----		----	
6016	D7042	6.781		-1.41	
normality		not OK			
n		15			
outliers		1			
mean (n)		6.82778			
st.dev. (n)		0.027841			
R(calc.)		0.07796			
R(D7042:14)		0.09264			



Determination of Pour Point, manual on sample #15219; results in °C

lab	method	value	mark	z(targ)	remarks
225		----		----	
237	D97	<-24		----	
255		----		----	
325		----		----	
335		----		----	
349		----		----	
360	D97	-30		-0.56	
432	D97	-31		-0.87	
451		----		----	
473		----		----	
496	D97	-30.0		-0.56	
541	D97	<-24		----	
614	D97	-21		2.24	
621	D97	-27.0		0.38	
663	D97	-27		0.38	
862	D97	-33		-1.49	
902		----		----	
912	D97	-27		0.38	
963	D97	-30		-0.56	
994	D97	-27		0.38	
1011	D97	-30		-0.56	
1026	D97	-30		-0.56	
1059	ISO3016	-30		-0.56	
1146	D97	-24.6		1.12	
1161	D97	-30		-0.56	
1184	D97	-24		1.31	
1201	D97	-33		-1.49	
1243		----		----	
1297		----		----	
1435	ISO3016	-24		1.31	
1456		----		----	
1461		----		----	
1660		----		----	
1704		----		----	
1720		----		----	
1748	D97	-30		-0.56	
1768		----		----	
1799	D97	-24		1.31	
1807	D97	-27		0.38	
1842		----		----	
1864	D97	-33		-1.49	
1877	D97	-30		-0.56	
1884		----		----	
1957	D97	-24		1.31	
1981	D97	-30		-0.56	
6002		----		----	
6016	D97	-27		0.38	

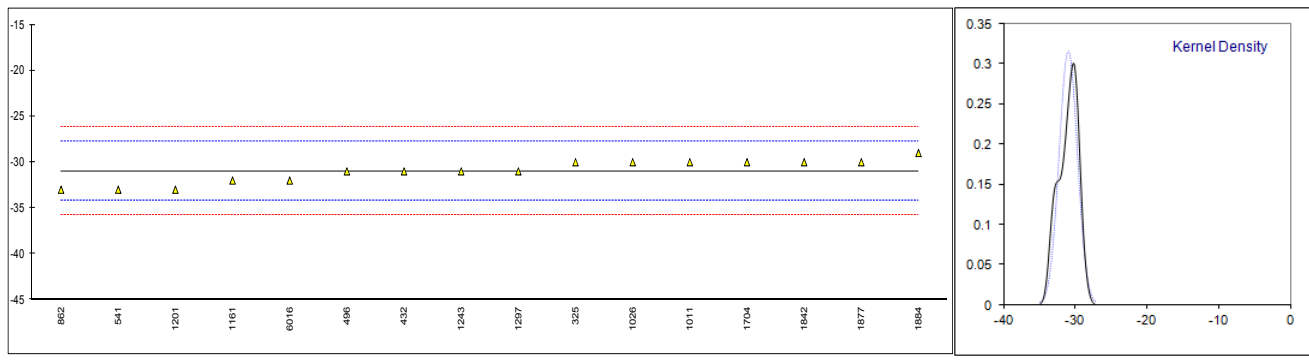
normality OK  
n 26  
outliers 0  
mean (n) -28.22  
st.dev. (n) 3.183  
R(calc.) 8.91  
R(D97:15) 9.00





Determination of Pour Point, automated, 1°C interval on sample #15219; results in °C

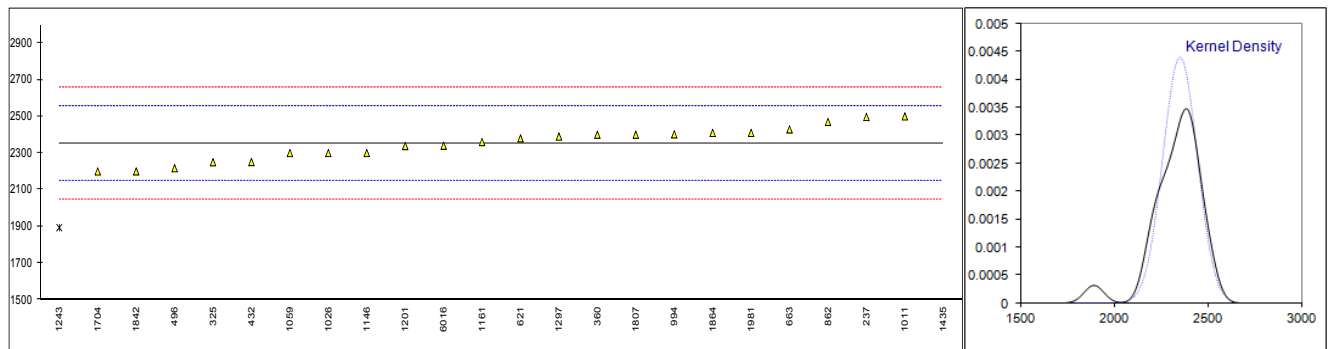
lab	method	value	mark	z(targ)	remarks
225		----		----	
237		----		----	
255		----		----	
325	D5950	-30		0.62	
335		----		----	
349		----		----	
360		----		----	
432	D5950	-31		0.00	
451		----		----	
473		----		----	
496	D5950	-31.0		0.00	
541	D5950	-33		-1.24	
614		----		----	
621		----		----	
663		----		----	
862	D5950	-33		-1.24	
902		----		----	
912		----		----	
963		----		----	
994		----		----	
1011	D6892	-30		0.62	
1026	D5950	-30		0.62	
1059		----		----	
1146		----		----	
1161	D6749	-32		-0.62	
1184		----		----	
1201	D5950	-33		-1.24	
1243	D5950	-31		0.00	
1297	D5950	-31.0		0.00	
1435		----		----	
1456		----		----	
1461		----		----	
1660		----		----	
1704	D5950	-30		0.62	
1720		----		----	
1748		----		----	
1768		----		----	
1799		----		----	
1807		----		----	
1842	D5950	-30		0.62	
1864		----		----	
1877	D5950	-30		0.62	
1884	D5950	-29		1.24	
1957		----		----	
1981		----		----	
6002		----		----	
6016	D5950	-32		-0.62	
	normality	OK			
	n	16			
	outliers	0			
	mean (n)	-31.00			
	st.dev. (n)	1.265			
	R(calc.)	3.54			
	R(D5950:14)	4.50			



Determination of Sulphur on sample #15219; results in mg/kg

lab	method	value	mark	z(targ)	remarks
225		----		----	
237	D4294	2496.8		1.42	
255		----		----	
325	D6443Mod.	2250		-1.00	
335		----		----	
349		----		----	
360	D4294	2400		0.47	
432	D4951	2251		-0.99	
451		----		----	
473		----		----	
496	D2622	2217		-1.32	
541		----		----	
614		----		----	
621	D4294	2380		0.27	
663	D4294	2429		0.75	
862	D2622	2470		1.16	
902		----		----	
912		----		----	
963		----		----	
994	D4294	2402		0.49	
1011	D6481	2500		1.45	
1026	D2622	2300		-0.51	
1059	ISO14596	2300		-0.51	
1146	ISO8754	2300	C	-0.51	first reported: 0.23 mg/kg (unit error)
1161	ISO8754	2360		0.08	
1184		----		----	
1201	D4294	2338	C	-0.14	reported: 0.2338 mg/kg (unit error)
1243	ISO8754	1893	R(0.01)	-4.50	
1297	D4294	2390		0.37	
1435		4839	R(0.01)	24.36	
1456		----		----	
1461		----		----	
1660		----		----	
1704	D2622	2199.817		-1.49	
1720		----		----	
1748		----		----	
1768		----		----	
1799		----		----	
1807	D4294	2400	C	0.47	first reported: 0.24 mg/kg (unit error)
1842	INH-05	2200		-1.49	
1864	D4294	2410		0.57	
1877		----		----	
1884		----		----	
1957		----		----	
1981	D4294	2410		0.57	
6002		----		----	
6016	D4294	2340		-0.12	

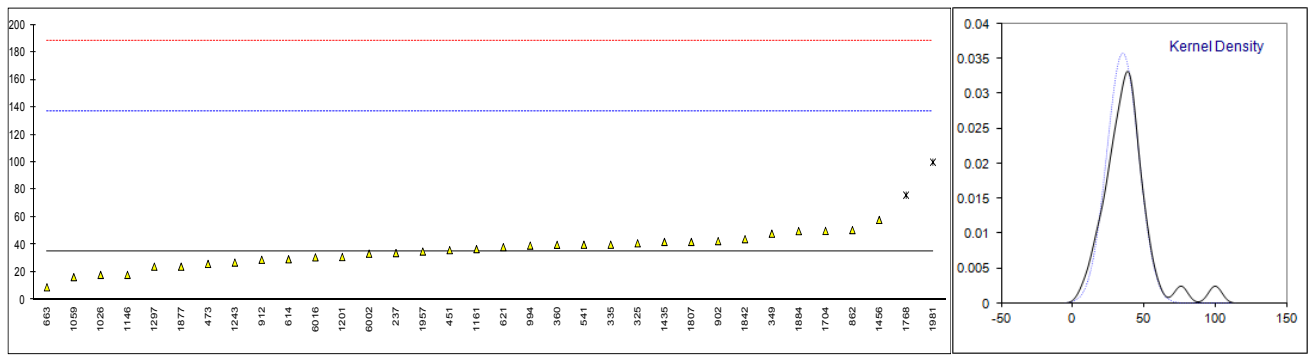
normality OK  
n 22  
outliers 2  
mean (n) 2351.98  
st.dev. (n) 90.606  
R(calc.) 253.70  
R(D4294:10) 285.84



Determination of Water Content by KF on sample #15219; results in mg/kg

lab	method	value	mark	z(targ)	remarks
225		----		----	
237	D6304-C	33.88		-0.02	
255		----		----	
325	D6304-C	41		0.12	
335	ISO12937	40		0.10	
349	D6304-A	48		0.25	
360	D6304-A	39.9		0.09	
432		----		----	
451	D6304-C	36		0.02	
473	D6304-C	26		-0.18	
496		----		----	
541	D6304-A	40		0.10	
614	D6304-C	29.4		-0.11	
621	D6304-B	38.2		0.06	
663	D6304-C	9.0		-0.51	
862	D6304-C	50.6		0.30	
902	D6304-A	42.60		0.15	
912	D6304-C	29		-0.12	
963		----		----	
994	D6304-C	39.35		0.08	
1011		----		----	
1026	D6304-C	18		-0.34	
1059	D6304-Cmod.	16.4		-0.37	
1146	D6304-C	18		-0.34	
1161	ISO12937	36.745		0.03	
1184		----		----	
1201	D6304-A	31		-0.08	
1243	D6304-A	27		-0.16	
1297	D6304-A	24.0		-0.22	
1435	D1744	42.0		0.14	
1456	D6304-A	58		0.45	
1461		----		----	
1660		----		----	
1704	D6304-A	50		0.29	
1720		----		----	
1748		----		----	
1768	ISO3733	76	R(0.05)	0.80	
1799		----		----	
1807	D6304-A	42		0.14	
1842	D6304-A	44		0.17	
1864		----		----	
1877	D6304-C	24		-0.22	
1884	D6304-A	49.89		0.29	
1957	D6304-A	35.0		0.00	
1981	D6304-C	100	R(0.01)	1.27	
6002	ISO12937	33.448		-0.03	
6016	D6304-B	30.8		-0.08	

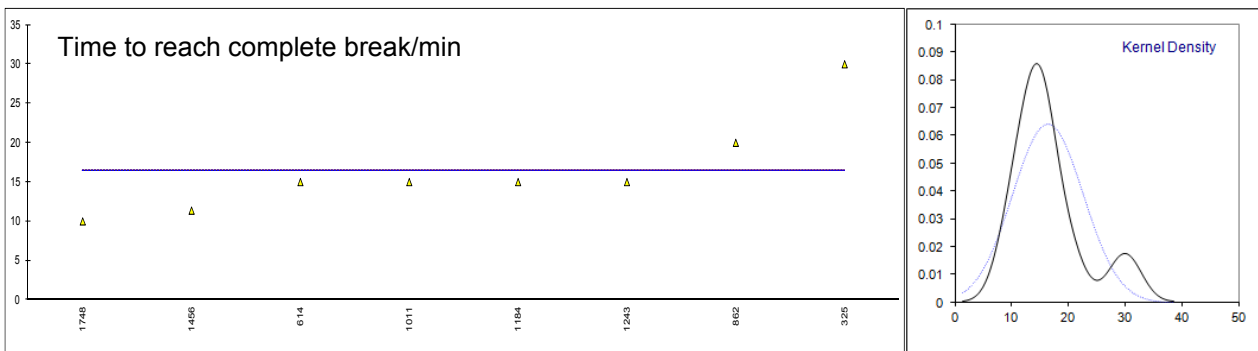
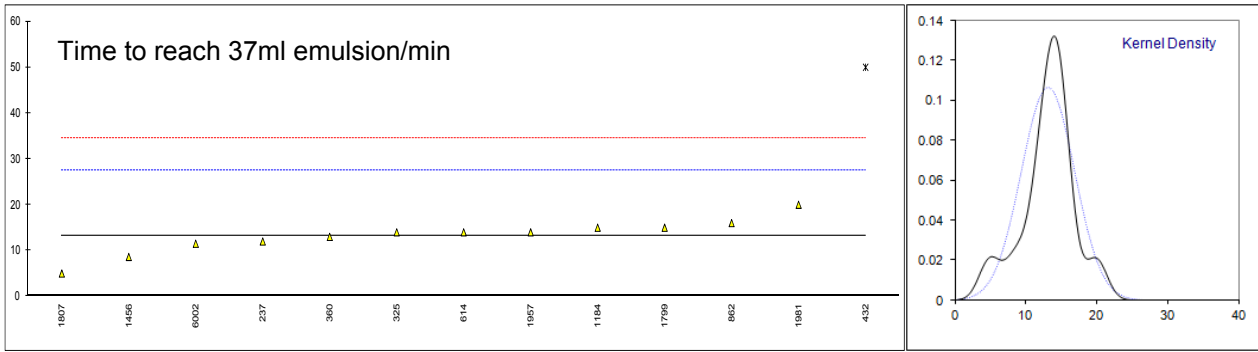
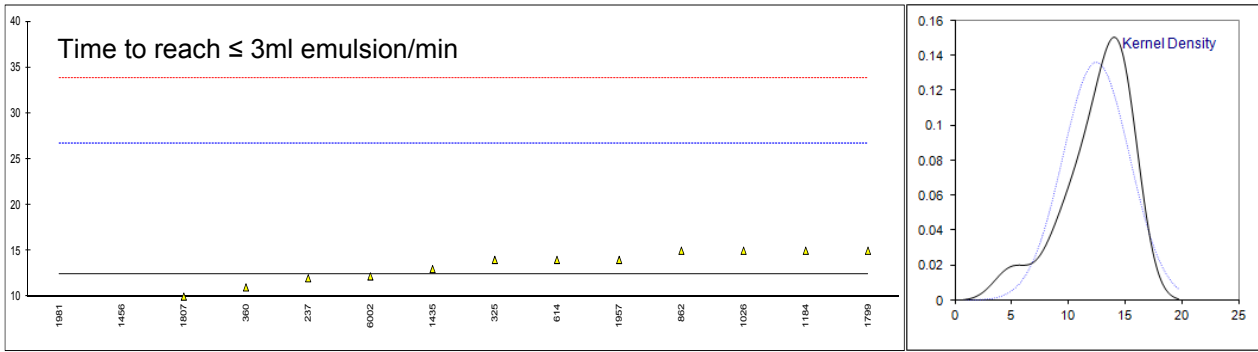
normality OK  
n 32  
outliers 2  
mean (n) 35.10  
st.dev. (n) 11.158  
R(calc.) 31.24  
R(D6304:07) 142.84



Determination of Water Separability at 54 °C on sample #15219; results in min.

lab	method	time to reach ≤3 ml emul.			time to reach 37 ml water			time to reach complete break			Test aborted
		mark	z(targ)		mark	z(targ)		mark	z(targ)		
225											
237	D1401	12	-0.06		12	-0.16		>30			yes
255											
325	D1401	14	0.22		14	0.12		30			
335											
349											
360	D1401	11	-0.20		13	-0.02					
432	D1401	>50			50	D(1) 5.16		>30			yes
451											
473											
496											
541											
614	D1401	14	0.22		14	0.12		15			no
621											
663											
862	D1401	15	0.36		16	0.40		20			no
902											
912											
963											
994											
1011								15			no
1026	D1401	15	0.36								
1059											
1146	D1401	n.a.			n.a.			n.a.			yes
1161											
1184	D1401	15	0.36		15	0.26		15			no
1201											
1243								15			
1297											
1435	ISO6614	13	0.08								
1456	D1401	8.62	-0.53		8.62	-0.64		11.37			no
1461											
1660											
1704											
1720											
1748	D1401							10			
1768											
1799	D1401	15	0.36		15	0.26					yes
1807	D1401	10	-0.34		5	-1.14					no
1842											
1864											
1877											
1884											
1957	D1401	14	0.22		14	0.12		>30			yes ?
1981	D1401	5	-1.04		20	0.96					
6002	ISO6614	12.2	-0.03		11.5	-0.23		>30			yes
6016											
	normality	suspect			suspect			unknown			
	n	14			12			8			
	outliers	0			1			0			
	mean (n)	12.42			13.18			16.42			
	st.dev. (n)	2.936			3.755			6.234			
	R(calc.)	8.22			10.51			17.45			
	R(D1401:12e1)	20.00			20.00			n.a.			

Lab 1957 reported that the test was aborted but reported also 0 ml for emulsion



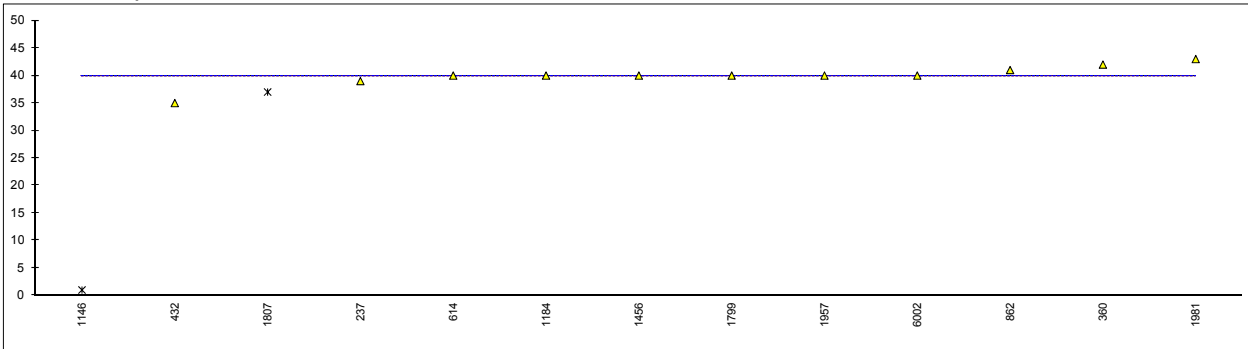
Determination of Water Separability at 54 °C on sample #15219; results in ml.

--- Continued ---

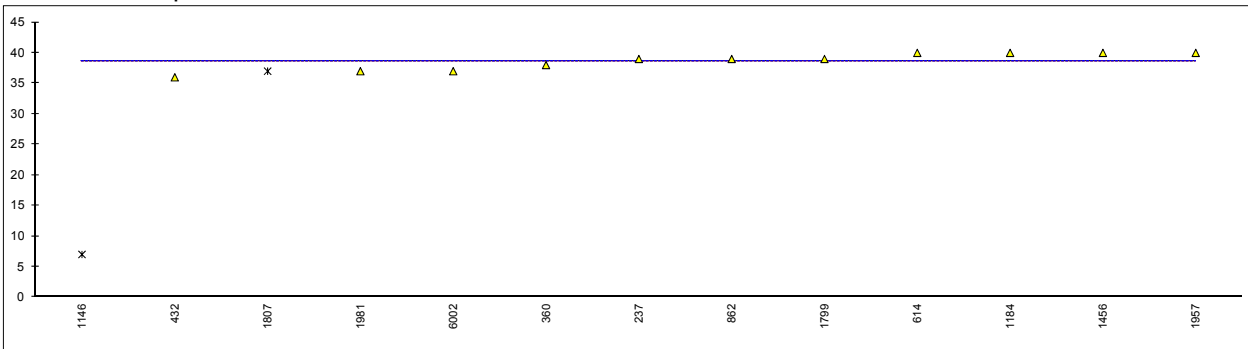
lab	method	volume oil phase	mark	volume water phase	mark	volume emul. phase	mark
225		----		----		----	
237	D1401	39		39		2	
255		----		----		----	
325		----		----		----	
335		----		----		----	
349		----		----		----	
360	D1401	42		38		0	
432	D1401	35		36		9	
451		----		----		----	
473		----		----		----	
496		----		----		----	
541		----		----		----	
614	D1401	40		40		0	
621		----		----		----	
663		----		----		----	
862	D1401	41		39		0	
902		----		----		----	
912		----		----		----	
963		----		----		----	
994		----		----		----	
1011		----		----		----	
1026		----		----		----	
1059		----		----		----	
1146	D1401	1	D(0.01)	7	D(0.01)	72	D(0.01)
1161		----		----		----	
1184	D1401	40		40		0	
1201		----		----		----	
1243		----		----		----	
1297		----		----		----	
1435		----		----		----	
1456	D1401	40		40		0	
1461		----		----		----	
1660		----		----		----	
1704		----		----		----	
1720		----		----		----	
1748		----		----		----	
1768		----		----		----	
1799	D1401	40		39		1	
1807	D1401	37	E	37	E	3	E
1842		----		----		----	
1864		----		----		----	
1877		----		----		----	
1884		----		----		----	
1957	D1401	40		40		0	
1981	D1401	43		37		0	
6002	ISO6614	40		37		3	
6016		----		----		----	
	normality	not OK		OK		not OK	
	n	11		11		11	
	outliers	1+1ex		1+1ex		1+1ex	
	mean (n)	40.0		38.6		1.36	
	st.dev. (n)	2.00		1.43		2.730	
	R(calc.)	5.6		4.0		7.64	
	R(target)	n.a.		n.a.		n.a.	

Lab 1807 made a calculation error, total volume of oil + water + emulsion should be 80ml

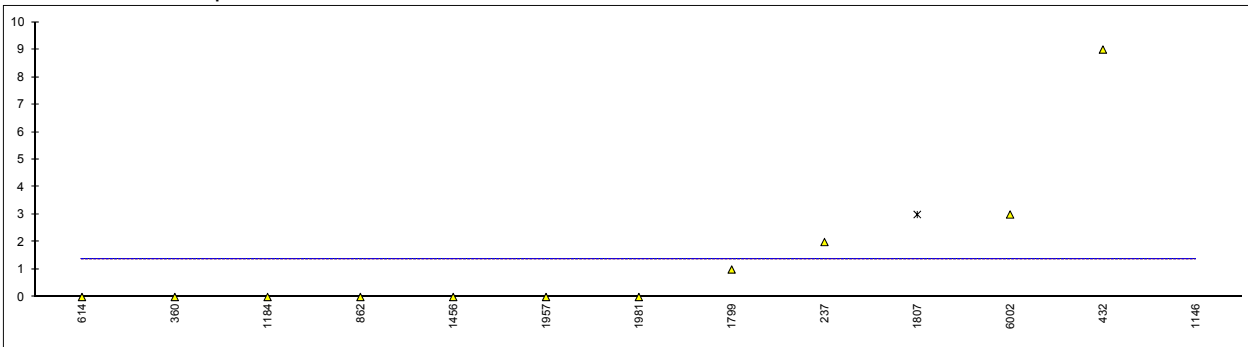
Volume oil phase/ml



Volume water phase/ml



Volume emulsion phase/ml

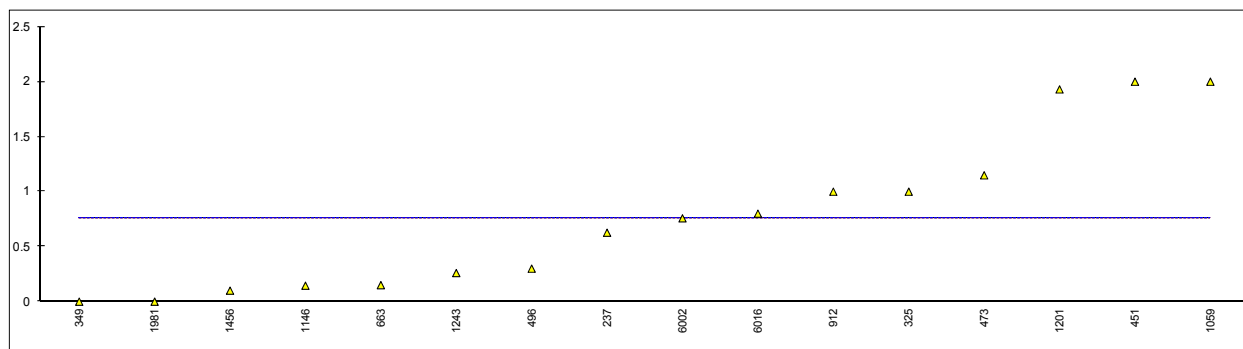


Determination of Calcium (Ca) on sample #15219; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
225		----		----	
237	D5185	0.627		----	
255	INH-1	<1		----	
325	D5185	1		----	
335		----		----	
349	D5185	0		----	
360	D5185	<1.0		----	
432	D4951	<1		----	
451	INH-116	2		----	
473	D5185	1.149		----	
496	DIN51399-1	0.3		----	
541	D5185	<40		----	
614		----		----	
621		----		----	
663	D5185	0.15		----	
862	D5185	<1		----	
902		----		----	
912	D5185	1		----	
963	D5185	<0.100		----	
994	D5185	<40		----	
1011	D5185	<10		----	
1026	D5185	<5		----	
1059	in house	2		----	
1146	in house	0.1451		----	
1161		----		----	
1184		----		----	
1201	D5185	1.93		----	
1243	D5185	0.26		----	
1297		----		----	
1435	D5185	<1		----	
1456	D5185	0.1		----	
1461		----		----	
1660		----		----	
1704		----		----	
1720		----		----	
1748		----		----	
1768		----		----	
1799		----		----	
1807	D5185	<2.5		----	
1842		----		----	
1864	D5185	<0.16		----	
1877		----		----	
1884		----		----	
1957		----		----	
1981	D5185	0		----	
6002	D6595	0.758		----	
6016	D5185	0.80		----	

normality n.a.  
n 28  
outliers 0  
mean (n) <40  
st.dev. (n) n.a.  
R(calc.) n.a.  
R(D5185:13e1) n.a.

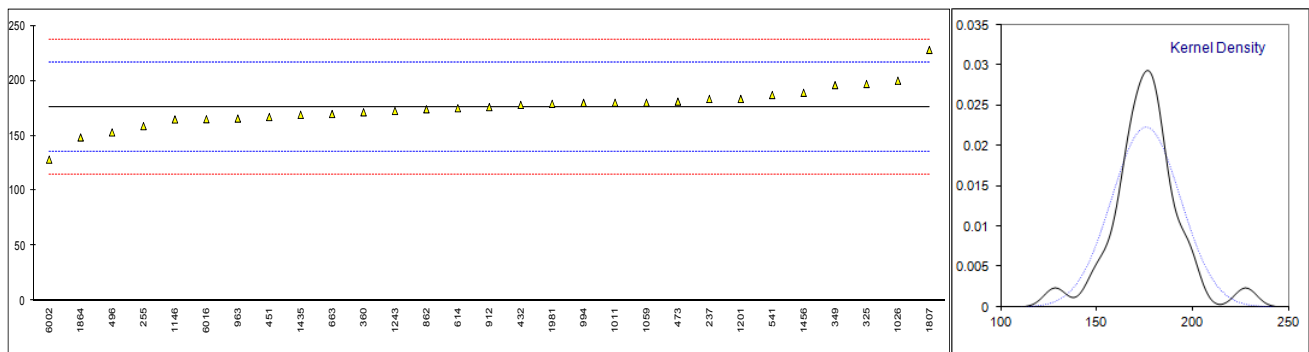
Application range: 40 – 9000 mg/kg





Determination of Phosphorus (P) on sample #15219; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
225		----		----	
237	D5185	183.4		0.37	
255	INH-1	158.70		-0.84	
325	D5185	197		1.04	
335		----		----	
349	D5185	196		0.99	
360	D5185	171.3		-0.22	
432	D4951	178		0.11	
451	INH-116	167		-0.44	
473	D5185	181.0		0.25	
496	DIN51399-1	153		-1.12	
541	D5185	187		0.55	
614	D5185	175		-0.04	
621		----		----	
663	D5185	169.7		-0.30	
862	D5185	174.0		-0.09	
902		----		----	
912	D5185	176		0.01	
963	D5185	165.6		-0.50	
994	D5185	179.9		0.20	
1011	D5185	180		0.20	
1026	D5185	200		1.19	
1059	in house	180		0.20	
1146	in house	164.8		-0.54	
1161		----		----	
1184		----		----	
1201	D5185	183.4		0.37	
1243	D5185	172.5		-0.17	
1297		----		----	
1435	D5185	169		-0.34	
1456	D5185	189.0		0.65	
1461		----		----	
1660		----		----	
1704		----	W	----	first reported: 560
1720		----		----	
1748		----		----	
1768		----		----	
1799		----		----	
1807	D5185	228	C	2.56	first reported: 268
1842		----		----	
1864	D5185	148.383		-1.35	
1877		----		----	
1884		----		----	
1957		----		----	
1981	D5185	179		0.15	
6002	D6595	128.29		-2.34	
6016	D5185	165		-0.53	
normality		not OK			
n		29			
outliers		0			
mean (n)		175.86			
st.dev. (n)		17.964			
R(calc.)		50.30			
R(D5185:13e1)		57.02			

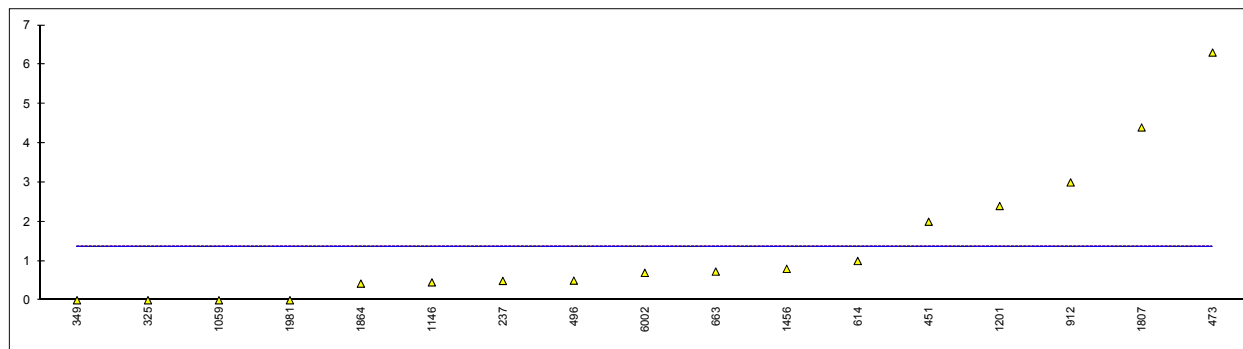


Determination of Zinc (Zn) on sample #15219; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
225		----		----	
237	D5185	0.493		----	
255	INH-1	<1		----	
325	D5185	0		----	
335		----		----	
349	D5185	0		----	
360	D5185	<1.0		----	
432	D4951	<1		----	
451	INH-116	2		----	
473	D5185	6.307		----	
496	DIN51399-1	0.5		----	
541	D5185	<60		----	
614	D5185	1		----	
621		----		----	
663	D5185	0.73		----	
862	D5185	<1		----	
902		----		----	
912	D5185	3		----	
963	D5185	<0.100		----	
994	D5185	<60		----	
1011	D5185	<10		----	
1026	D5185	<5		----	
1059	in house	0		----	
1146	in house	0.4559		----	
1161		----		----	
1184		----		----	
1201	D5185	2.4		----	
1243	D5185	<0.01		----	
1297		----		----	
1435	D5185	<1		----	
1456	D5185	0.8		----	
1461		----		----	
1660		----		----	
1704		----		----	
1720		----		----	
1748		----		----	
1768		----		----	
1799		----		----	
1807	D5185	4.4		----	
1842		----		----	
1864	D5185	0.424		----	
1877		----		----	
1884		----		----	
1957		----		----	
1981	D5185	0		----	
6002	D6595	0.698		----	
6016	D5185	<0.01		----	

normality n.a.  
n 29  
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 lab in ARGENTINA  
2 labs in AUSTRALIA  
1 lab in AUSTRIA  
1 lab in AZERBAIJAN  
3 labs in BELGIUM  
2 labs in BULGARIA  
1 lab in CHINA, People's Republic  
1 lab in COTE D'IVOIRE  
1 lab in FRANCE  
1 lab in GEORGIA  
2 labs in GERMANY  
1 lab in GREECE  
2 labs in INDIA  
1 lab in INDONESIA  
1 lab in ITALY  
1 lab in JORDAN  
1 lab in KAZAKHSTAN  
1 lab in MALAYSIA  
3 labs in NETHERLANDS  
1 lab in NIGERIA  
1 lab in NORWAY  
1 lab in POLAND  
1 lab in PORTUGAL  
1 lab in ROMANIA  
1 lab in SAUDI ARABIA  
1 lab in SERBIA  
1 lab in SLOVENIA  
1 lab in SOUTH KOREA  
2 labs in SPAIN  
1 lab in SUDAN  
1 lab in TANZANIA  
1 lab in THAILAND  
4 labs in TURKEY  
2 labs in UNITED KINGDOM

**APPENDIX 3****Abbreviations:**

C	= final result after checking of first reported suspect result
D(0.01) or D(1)	= outlier in Dixon's outlier test
D(0.05) or D(5)	= 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	= test result excluded from calculations
n.a.	= not applicable
E	= probably error in calculations
U	= reported in a deviating 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
- 3 ASTM E1301-89
- 4 ISO 5725-86
- 5 ISO 5725, parts 1-6, 1994
- 6 ISO 13528-05
- 7 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
- 8 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 9 IP 367/84
- 10 DIN 38402 T41/42
- 11 P.L. Davies, First reported Z. Anal. Chem, 331, 513, (1988)
- 12 J.N. Miller, Analyst, 118, 455, (1993)
- 13 Analytical Methods Committee Technical Brief, No4 January 2001
- 14 The Royal Society of Chemistry 2002, Analyst 2002, 127 page1359-1364, P.J. Lowthian and M. Thompson (see <http://www.rsc.org/suppdata/an/b2/b205600n/>).
- 15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), pp. 165-172, (1983)