

Results of Proficiency Test  
Hydraulic Fluid (fresh)  
November 2017

Organised by: Institute for Interlaboratory Studies  
Spijkenisse, the Netherlands

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Report no. iis17L07

February 2018

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## 1 INTRODUCTION

Since 2003, the Institute for Interlaboratory Studies (iis) organizes a proficiency test (PT) for the analysis on used Hydraulic Fluid every year. In 2014 it was decided to organize a proficiency test for the analyses on 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 2017/2018, it was decided to continue the round robin for the analysis on fresh Hydraulic Fluid. In this interlaboratory study, 46 laboratories from 33 different countries did register for participation. See appendix 3 for the number of participants per country. In this report, the test results of the 2017 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 (PT). Sample analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC 17025 accredited laboratory. It was decided to send one sample of one litre of fresh oil, labelled #17225. The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

### 2.1 ACCREDITATION

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

### 2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organization, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4). This protocol is electronically available through 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 supplier. The bulk material was homogenised. After homogenisation, 68 amber glass bottles of 1 litre were filled and labelled #17225. The homogeneity of the subsamples #17225 was checked by determination of Density at 15°C in accordance with ASTM D4052 and Kinematic Viscosity at 40°C according to ASTM D445 on 10 stratified randomly selected samples.

	<i>Density at 15 °C in kg/L</i>	<i>Kinematic Viscosity at 40 °C in mm<sup>2</sup>/s</i>
Sample #17225-1	0.87460	97.91
Sample #17225-2	0.87460	97.92
Sample #17225-3	0.87460	97.91
Sample #17225-4	0.87461	97.89
Sample #17225-5	0.87460	97.92
Sample #17225-6	0.87460	97.93
Sample #17225-7	0.87460	97.90
Sample #17225-8	0.87461	97.93
Sample #17225-9	0.87461	97.88
Sample #17225-10	0.87461	97.92

Table 1: homogeneity test results of subsamples #17225

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

	<i>Density at 15 °C in kg/L</i>	<i>Kinematic Viscosity at 40 °C in mm<sup>2</sup>/s</i>
r (observed)	0.00001	0.05
reference test method	D4052:16	D445:17a
0.3 x R (ref. test method)	0.00015	0.36

Table 2: evaluation of the repeatabilities of subsamples #17225

The calculated repeatabilities in table 2 were both less than 0.3 times the corresponding reproducibilities of the reference test methods. Therefore, homogeneity of the subsamples #17225 was assumed.

To each of the participating laboratories one sample of 1 litre amber glass bottle, labelled #17225, was sent on October 25, 2017. An SDS was added to the sample package.

## 2.5 STABILITY OF THE SAMPLES

The stability of fresh Hydraulic Fluid, packed in amber 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 on sample #17225; 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 82°C and Calcium, Phosphorus and Zinc. Also additional questions were asked about the foam determination.

It was explicitly requested to treat the samples as if they were routine samples and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report 'less than' results, which are above the detection limit, because such test results cannot be used for meaningful statistical calculations.

To get comparable test results, a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal [www.kpmd.co.uk/sgs-iis/](http://www.kpmd.co.uk/sgs-iis/). The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website [www.iisnl.com](http://www.iisnl.com).

## 3 RESULTS

During five weeks after sample dispatch, the test 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 reported test 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 test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test 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 reported test results (no reanalysis). Additional or corrected test results are used for data analysis and original test results are placed under 'Remarks' in the test result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

### 3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of March 2017 (iis-protocol, version 3.4).

For the statistical evaluation, the *unrounded* (when available) figures were used instead of the rounded test results. Test 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. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

According to ISO 5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

Finally, the reproducibilities were calculated from the standard deviations by multiplying 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 test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. 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, EN or ISO 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 according to:

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

The  $Z_{(\text{target})}$  scores are listed in the test result tables in appendix 1.

Absolute values for  $z < 2$  are very common and absolute values for  $z > 3$  are very rare.

The usual interpretation of z-scores is as follows:

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

## 4 EVALUATION

In this proficiency test no severe problems were encountered with the dispatch of the samples. Not all laboratories were able to report all analyses requested.

In total 45 participants reported 610 numerical test results. Observed were 28 outlying test results, which is 4.6% of the numerical test 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.

### 4.1 EVALUATION PER TEST

In this section, the test results are discussed per test. The test methods that are reported by the laboratories are taken into account for explaining the observed differences when possible and applicable. These test 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 4.

Unfortunately, a suitable standard test method, providing the precision data, is not available for all determinations. For the tests, that have no available precision data, the calculated reproducibility was compared against the reproducibility from the Horwitz equation.

In the iis PT reports, test methods are referred to with a number (e.g. D2270) and an added designation for the year that the test method was adopted or revised (e.g. D2270:10). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D2270:10(2016)). In the tables of appendix 1 only the test method number and year of adoption or revision will be used.



Acid Number (total): This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D664-A:11ae1.

Copper Corrosion: This determination was not problematic. Almost all participants agreed on a test result of 1 (1a), and one participant reported "nil".

Density at 15°C: This determination was very problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D4052:16.

Flash Point PMcc: This determination was not problematic. Three statistical outliers were observed and two test results were excluded. However, the calculated reproducibility after rejection of the suspect data is in full agreement with the requirements of ASTM D93-A:16a.

Foaming Characteristics (Tendency and Stability): This determination was problematic. In total four statistical outliers were observed and three test results were excluded over 6 parameters.

The calculated reproducibility in the Foam Tendency determination for sequence I is after rejection of the suspect data almost in agreement with the requirements of ASTM D892:13e1. However, the Foam Tendency determination for sequence II is after rejection of the suspect data not in agreement with the requirements of ASTM D892:13e1. And the Foam Tendency determination for sequence III is after rejection of the suspect data not at all in agreement with the requirements of ASTM D892:13e1. Therefore no z-scores were calculated for sequence III.

For Foam stability also no z-scores were calculated. Almost all participants reported 0 ml.

This determination is very sensitive in maintenance and execution.

In ASTM D892:13e1 many tips and tricks are given in the test method part X1. Possible sources for the large variation are the cleaning and checking of the air diffuser, air tubes and test cylinders, the air flow rate used during the blowing period. Therefore extra information was asked (see appendix 2), but no clear conclusion could be drawn based on this information.

Kinematic Viscosity at 40°C: 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 D445:17a.

Kinematic Viscosity at 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 D445:17a.

Viscosity Index This determination was not problematic. No statistical outliers were observed, but three test results were excluded. However, the calculated

reproducibility after rejection of the suspect data is in full agreement with the requirements of ASTM D2270:10(2016).

Viscosity Stabinger at 40°C: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D7042:16e3.

Viscosity Stabinger at 100°C: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D7042:16e3.

Pour Point (manual): This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is almost in agreement with the requirements of ASTM D97:17a.

Pour Point (automated): This determination was problematic. No statistical outliers were observed, but one test result was excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of ASTM D5950:14.

Sulphur: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of ASTM D4294:16e1.

Water content: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D6304:16e1.

Water Separability at 82°C: This determination was not problematic. In total six statistical outliers were observed over six parameters. However, the calculated reproducibilities for “time to reach  $\leq 3$ ml emulsion” and “time to reach 37ml water” are both in good agreement with the requirements of ASTM D1401:12e1.

Calcium: This determination may not be problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated reproducibility using the Horwitz equation, but not at all in agreement with the strict requirements of ASTM D5185:13e1 (mean value of Calcium is identical to the lower limit of the application range).

Phosphorus: 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 D5185:13e1.

Zinc: This determination was problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D5185:13e1.

#### 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant reference test method and the reproducibility as found for the group of participating laboratories. The target reproducibilities derived from literature reference test methods (R (lit)) and the calculated reproducibilities ( $2.8 * sd$ ) are compared in the next table:

Parameter	unit	n	mean	2.8 * sd	R (lit)
Acid Number (total)	mg KOH/g	37	0.37	0.13	0.19
Copper Corrosion, 3 hrs at 50°C	rating	24	1	n.a.	n.a.
Density at 15°C	kg/L	39	0.8747	0.0012	0.0005
Flash Point PMcc	°C	30	237.0	16.3	16.8
Foam Tendency Seq. I	ml	13	11.5	21.7	19.3
Foam Tendency Seq. II	ml	15	18.3	23.5	16.2
Foam Tendency Seq. III	ml	14	7.9	26.7	(3.5)
Foam Stability Seq. I	ml	14	0	0	(0)
Foam Stability Seq. II	ml	16	0	0	(0)
Foam Stability Seq. III	ml	15	0	0	(0)
Kinematic viscosity at 40°C	mm <sup>2</sup> /s	32	97.843	0.811	1.194
Kinematic viscosity at 100°C	mm <sup>2</sup> /s	34	11.176	0.120	0.154
Viscosity Index		34	99.4	1.8	2
Viscosity Stabinger at 40°C	mm <sup>2</sup> /s	18	97.966	1.456	1.180
Viscosity Stabinger at 100°C	mm <sup>2</sup> /s	18	11.179	0.168	0.120
Pour Point (manual)	°C	23	-16.6	9.9	9
Pour Point (automated), 1°C int.	°C	11	-17.0	6.6	4.5
Sulphur	mg/kg	19	543	113	111
Water content (by KF)	mg/kg	30	51.9	76.6	180.6
<b>Water Separability at 82°C, distilled water</b>					
- Time ≤ 3 ml emulsion	min	14	12.3	17.1	25
- Time 37 ml water	min	14	12.4	16.9	25
- Volume Oil phase	ml	12	40.7	3.2	n.a.
- Volume Water phase	ml	13	38.5	4.7	n.a.
- Volume Emulsion phase	ml	12	0.6	3.3	n.a.
Calcium as Ca	mg/kg	27	39.7	7.4	10.2 *)
Phosphorus as P	mg/kg	27	259	46	69
Zinc as Zn	mg/kg	29	271	43	39

Table 3: reproducibilities of tests on sample #17225

\*) reproducibility via the Horwitz equation

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 reference test methods. The problematic tests have been discussed in paragraph 4.1.

#### 4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2017 WITH THE PREVIOUS PTs.

	<i>November 2017</i>	<i>November 2016</i>	<i>November 2015</i>	<i>November 2014</i>
Number of reporting labs	45	43	45	29
Number of test results reported	610	597	569	346
Statistical outliers	28	30	26	19
Percentage outliers	4.6%	5.0%	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 reference test methods. The conclusions are given the following table:

Determination	<i>November 2017</i>	<i>November 2016</i>	<i>November 2015</i>	<i>November 2014</i>
Acid Number (total)	+	+	++	++
Density at 15°C	--	+	-	+/-
Flash Point PMcc	+/-	+	+	-
Foam Tendency Seq. I	+/-	n.e.	--	--
Foam Tendency Seq. II	-	+	+/-	--
Foam Tendency Seq. III	n.e.	n.e.	--	--
Foam Stability Seq. I	n.e.	n.e.	n.e.	n.e.
Foam Stability Seq. II	n.e.	n.e.	n.e.	n.e.
Foam Stability Seq. III	n.e.	n.e.	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.	n.e.
Phosphorus as P	+	+	+	++
Zinc as Zn	-	-	n.e.	n.e.

Table 5: comparison determinations against the reference test methods

\*) against the Horwitz equation

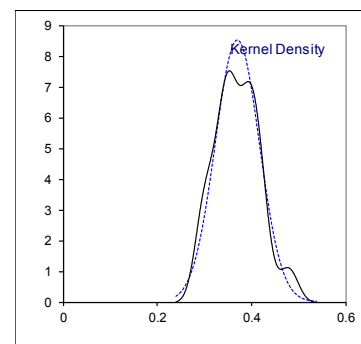
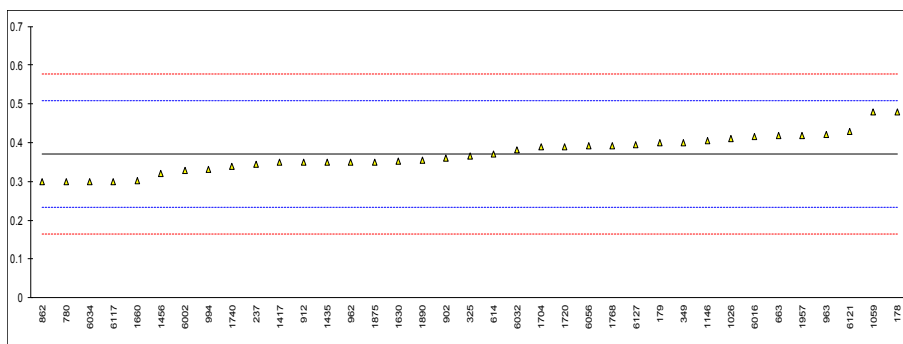
- ++: group performed much better than the reference test method
- + : group performed better than the reference test method
- +/-: group performance equals the reference test method
- : group performed worse than the reference test method
- : group performed much worse than the reference test method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

**APPENDIX 1**

Determination of Acid Number (Total) on sample #17225; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
173		----		----	
178	INH-1118	0.48		1.58	
179	D664-B	0.40		0.42	
237	D664-A	0.3433		-0.40	
255		----		----	
325	D664-A	0.365		-0.09	
349	D664-A	0.40		0.42	
432		----		----	
614	D664-A	0.37		-0.02	
621		----		----	
663	D664-A	0.418		0.68	
780	D664-A	0.30		-1.03	
862	D664-A	0.3		-1.03	
902	D664-A	0.36		-0.16	
912	D664-A	0.35		-0.31	
962	D974	0.35		-0.31	
963	D664-A	0.42		0.71	
994	D664-A	0.3305		-0.59	
1011		----		----	
1026	D664-A	0.41		0.56	
1059	ISO6619	0.48		1.58	
1146	D664-A	0.404		0.48	
1417	D664-A	0.349		-0.32	
1435	D664-A	0.350		-0.31	
1456	D974	0.32		-0.74	
1630	D974	0.3533		-0.26	
1660		0.301		-1.01	
1682		----		----	
1704	D664-A	0.388		0.25	
1720	D664-A	0.39		0.27	
1740	D664-A	0.34		-0.45	
1748		----		----	
1768	ISO6618	0.3927		0.31	
1875	ISO6618	0.35		-0.31	
1890	ISO6619	0.355		-0.23	
1957	D664-A	0.418		0.68	
4043		----		----	
6002	D664-A	0.328		-0.62	
6016	D664-A	0.417		0.67	
6032	D664-A	0.3821		0.16	
6034	D664-A	0.30		-1.03	
6056	INH-5088	0.391		0.29	
6117	GB/T4945	0.30		-1.03	
6121	DIN51558	0.43		0.85	
6127	D664-A	0.3940		0.33	
7003		----		----	

normality OK  
n 37  
outliers 0  
mean (n) 0.3711  
st.dev. (n) 0.04670  
R(calc.) 0.1308  
st.dev.(D664:11ae1) 0.06904  
R(D664:11ae1) 0.1933

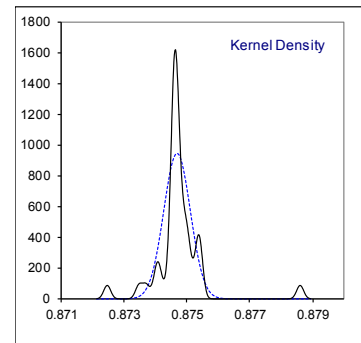
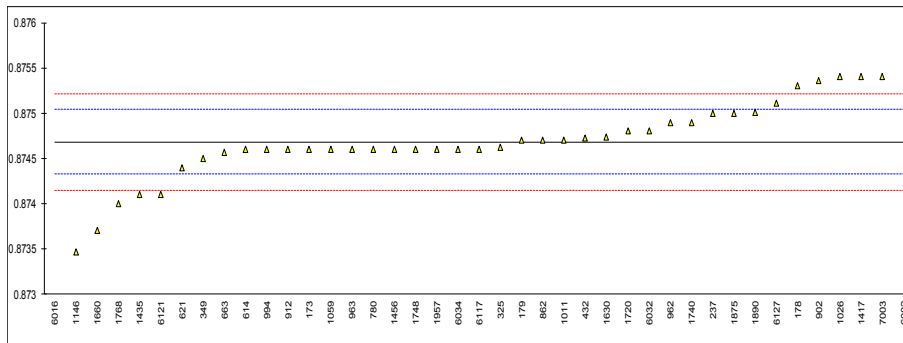


## Determination of Copper Corrosion 3hrs at 50°C on sample #17225; results in rating

lab	method	value	mark	z(targ)	remarks
173		----		----	
178		----		----	
179		----		----	
237	D130	1		----	
255		----		----	
325	D130	1A		----	
349	D130	1a		----	
432		----		----	
614	D130	1A		----	
621	D130	1A		----	
663	D130	1a		----	
780	D130	1a		----	
862	D130	1a		----	
902		----		----	
912	D130	1a		----	
962		----		----	
963	D130	1a		----	
994	D130	1a		----	
1011	D130	1a		----	
1026	D130	1A		----	
1059	ISO2160	1a		----	
1146		----		----	
1417	IP154	1A		----	
1435		----		----	
1456	D130	1A		----	
1630	D130	NIL		----	
1660		----		----	
1682		----		----	
1704	D130	1a		----	light orange
1720		----		----	
1740	D130	1A		----	
1748	D130	1a		----	
1768		----		----	
1875		----		----	
1890		----		----	
1957		----		----	
4043		----		----	
6002	ISO2160	1a		----	
6016		----		----	
6032		----		----	
6034	D130	1a		----	
6056		----		----	
6117	GB/T5096	1a		----	
6121	ISO2160	1a		----	
6127	D130	1a		----	
7003		----		----	
n		24			
mean (n)		1 (1a)			

Determination of Density at 15°C on sample #17225; results in kg/L

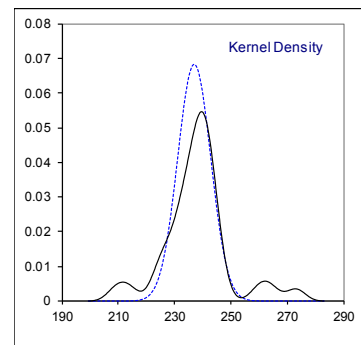
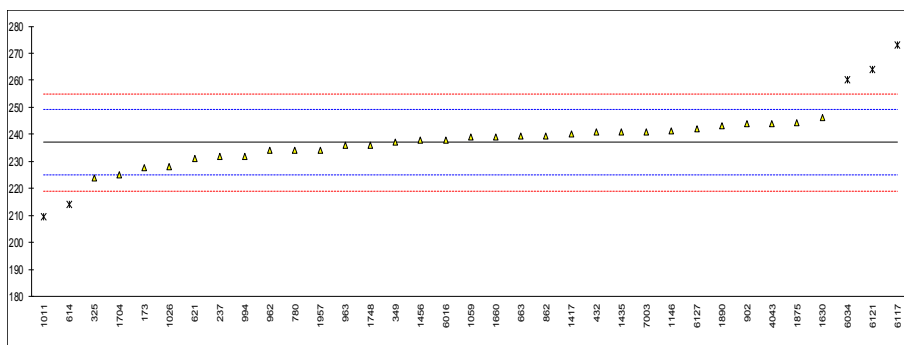
lab	method	value	mark	z(targ)	remarks
173	D4052	0.8746		-0.46	
178	D4052	0.8753	C	3.46	first reported 0.8753 kg/m <sup>3</sup>
179	D4052	0.8747	C	0.10	first reported 0.8747 kg/m <sup>3</sup>
237	D4052	0.8750		1.78	
255		----		----	
325	D4052	0.87462		-0.35	
349	D4052	0.8745		-1.02	
432	D4052	0.87472		0.21	
614	D4052	0.8746		-0.46	
621	D4052	0.8744		-1.58	
663	D4052	0.87457		-0.63	
780	D4052	0.8746		-0.46	
862	D4052	0.8747		0.10	
902	D4052	0.87536		3.80	
912	D4052	0.8746		-0.46	
962	D4052	0.8749		1.22	
963	D4052	0.8746		-0.46	
994	D4052	0.8746		-0.46	
1011	D4052	0.8747		0.10	
1026	D4052	0.8754		4.02	
1059	D4052	0.8746		-0.46	
1146	D4052	0.87347		-6.79	
1417	IP365	0.8754		4.02	
1435	D4052	0.8741		-3.26	
1456	D4052	0.8746		-0.46	
1630	D4052	0.87474	C	0.32	first reported 0.87142 kg/m <sup>3</sup>
1660	D7042	0.8737		-5.50	
1682		----		----	
1704		----		----	
1720	D4052	0.8748		0.66	
1740	D4052	0.8749		1.22	
1748	D4052	0.8746		-0.46	
1768	ISO3675	0.8740		-3.82	
1875	D7042	0.8750		1.78	
1890	ISO12185	0.87501		1.84	
1957	D4052	0.8746		-0.46	
4043		----		----	
6002	ISO3675	0.8786	C,R(0.01)	21.94	first reported 876.8 kg/m <sup>3</sup>
6016	D4052	0.87246	R(0.01)	-12.44	
6032	D4052	0.8748		0.66	
6034	D4052	0.8746	U	-0.46	reported 0.8746 kg/m <sup>3</sup>
6056		----		----	
6117	SH/T0604	0.8746		-0.46	
6121	ISO12185	0.8741		-3.26	
6127	D4052	0.87511		2.40	
7003	D4052	0.8754		4.02	
normality		suspect			
n		39			
outliers		2			
mean (n)		0.87468			
st.dev. (n)		0.000423			
R(calc.)		0.00118			
st.dev.(D4052:16)		0.000179			
R(D4052:16)		0.00050			





Determination of Flash Point PMcc on sample #17225; results in °C

lab	method	value	mark	z(targ)	remarks
173	D93-A	227.5		-1.59	
178		----		----	
179		----		----	
237	D93-B	232.0		-0.84	
255		----		----	
325	D93-A	224		-2.17	
349	D93-A	237		0.00	
432	D93-A	241.0		0.66	
614	D93-A	214	R(0.05)	-3.83	
621	D93-A	231	C	-1.00	first reported 213.0
663	D93-A	239.4		0.39	
780	D93-A	234.0		-0.50	
862	D93-A	239.5		0.41	
902	D93-A	244.0		1.16	
912		----		----	
962	D93-A	234		-0.50	
963	D93-A	236.0		-0.17	
994	D93-A	232.0		-0.84	
1011	D93-A	209.5	C,R(0.05)	-4.58	first reported 197.5
1026	D93-A	228.0		-1.50	
1059	ISO2719-A	239.0		0.33	
1146	D93-A	241.1		0.68	
1417	D93-A	240		0.49	
1435	D93-A	241.0		0.66	
1456	D93-A	238.0		0.16	
1630	D93-A	246		1.49	
1660	D93-A	239		0.33	
1682		----		----	
1704	D93-A	225	C	-2.00	first reported 213
1720		----		----	
1740		----	W	----	first reported 217
1748	D93-A	236		-0.17	
1768		----		----	
1875	ISO2719-A	244.3		1.21	
1890	ISO2719-B	243		0.99	
1957	D93-A	234.0		-0.50	
4043	ISO15267	244.0000		1.16	
6002		----		----	
6016	D93-A	238		0.16	
6032		----		----	
6034	D93-A	260	R(0.05)	3.82	
6056		----		----	
6117	GB/T3536	273	ex	5.99	excluded: open cup method is not equivalent to Flash Point PMcc
6121	ISO2592	264	ex	4.49	excluded: open cup method is not equivalent to Flash Point PMcc
6127	D93-A	242		0.83	
7003	D93-B	241.0		0.66	
normality		OK			
n		30			
outliers		3 (+2 ex)			
mean (n)		237.03			
st.dev. (n)		5.838			
R(calc.)		16.35			
st.dev.(D93-A:16a)		6.010			
R(D93-A:16a)		16.83			

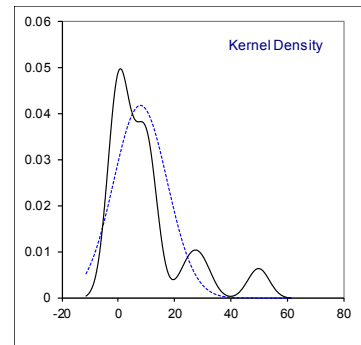
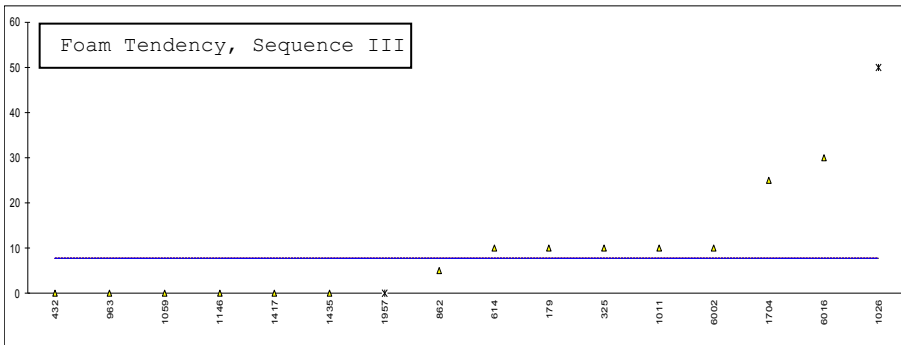
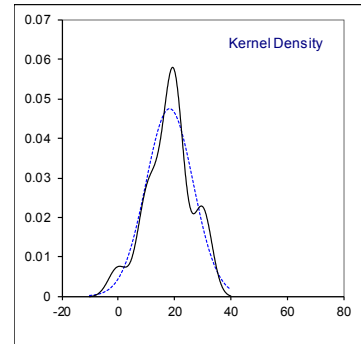
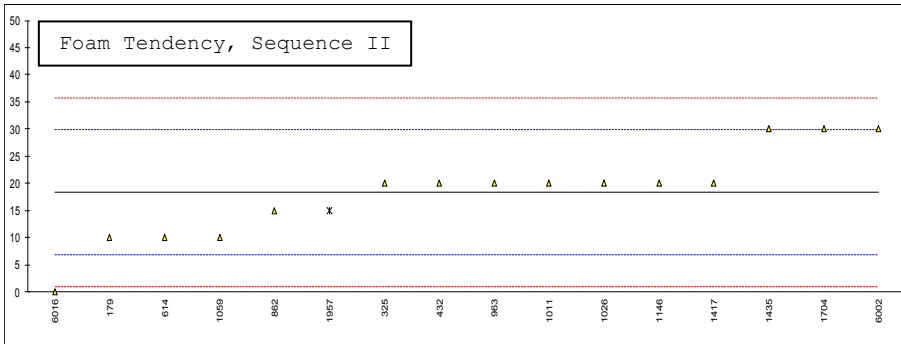
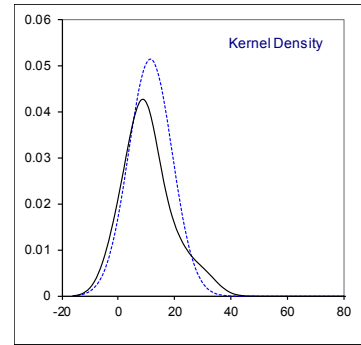
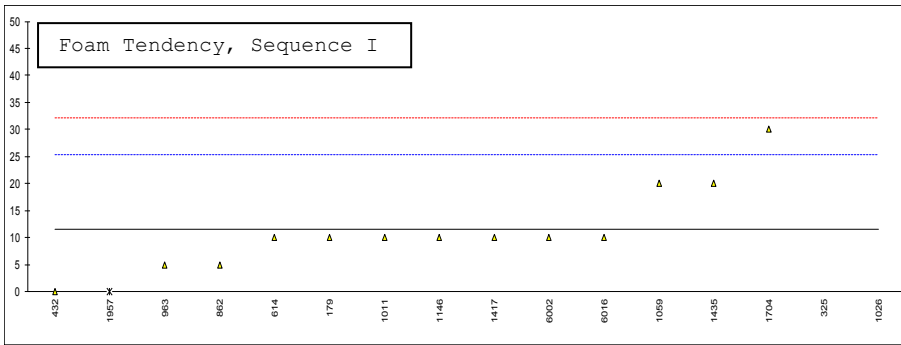


Determination of Foaming Characteristics, Foaming Tendency (at end of 5 min blowing period) on sample #17225; results in ml

lab	method	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
173		----		----	----		----	----		----
178		----		----	----		----	----		----
179	D892	10		-0.22	10		-1.44	10		----
237		----		----	----		----	----		----
255		----		----	----		----	----		----
325	D892	200 *)	C,G(0.01)	27.35	20		0.29	10 *)	C	----
349		----		----	----		----	----		----
432	D892	0		-1.67	20		0.29	0		----
614	D892	10		-0.22	10		-1.44	10		----
621		----		----	----		----	----		----
663		----		----	----		----	----		----
780		----		----	----		----	----		----
862	D892	5		-0.95	15		-0.58	5		----
902		----		----	----		----	----		----
912		----		----	----		----	----		----
962		----		----	----		----	----		----
963	D892	5		-0.95	20		0.29	0		----
994		----		----	----		----	----		----
1011	D892	10		-0.22	20		0.29	10		----
1026	D892	350	G(0.01)	49.11	20		0.29	50	G(0.05)	----
1059	D892	20		1.23	10		-1.44	0		----
1146	D892	10		-0.22	20		0.29	0		----
1417	D892	10		-0.22	20		0.29	0		----
1435	ISO6247	20		1.23	30		2.02	0		----
1456		----		----	----		----	----		----
1630		----		----	----		----	----		----
1660		----		----	----		----	----		----
1682		----		----	----		----	----		----
1704	D892	30		2.68	30 *)	C	2.02	25		----
1720		----		----	----		----	----		----
1740		----		----	----		----	----		----
1748	D892	----		----	----		----	----		----
1768		----		----	----		----	----		----
1875		----		----	----		----	----		----
1890		----		----	----		----	----		----
1957		0	ex **)	-1.67	15	ex **)	-0.58	0	ex **)	----
4043		----		----	----		----	----		----
6002	ISO6247	10		-0.22	30		2.02	10		----
6016	D892	10		-0.22	0		-3.17	30		----
6032		----		----	----		----	----		----
6034		----		----	----		----	----		----
6056		----		----	----		----	----		----
6117		----		----	----		----	----		----
6121		----		----	----		----	----		----
6127		----		----	----		----	----		----
7003		----		----	----		----	----		----
normality		not OK			OK			not OK		
n		13			15			14		
outliers		2 (+1 ex)			0 (+1 ex)			1 (+1 ex)		
mean (n)		11.54			18.33			7.86		
st.dev. (n)		7.7418			8.3808			9.550		
R(calc.)		21.68			23.47			26.74		
st.dev.(D892:13e1)		6.891			5.788			(1.235)		
R(D892:13e1)		19.30			16.21			(3.46)		

\*)  
 Lab 325 first reported 460,320  
 Lab 1704 first reported 60

\*\*)  
 Lab 1957 excluded: Gas diffuser was not cleansed (see appendix 2)



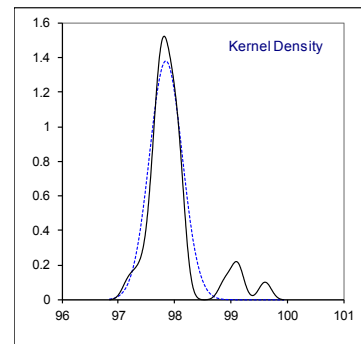
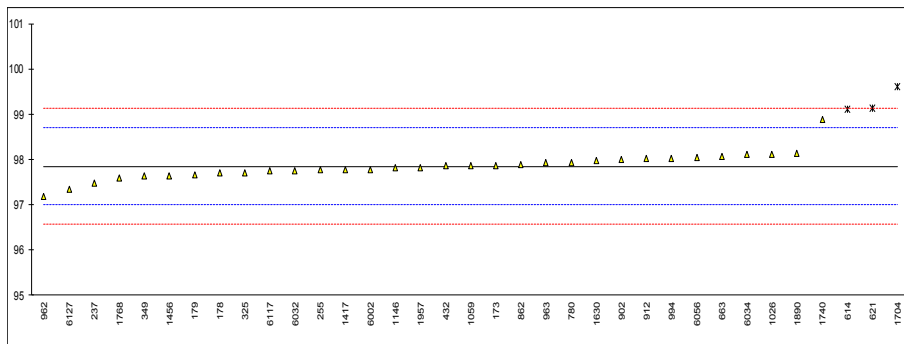
Determination of Foaming Characteristics, Foaming Stability (at end of 10 min settling period) on sample #17225; results in ml

lab	method	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
173		----		----			----			----
178		----		----			----			----
179	D892	Nil		----	0		----	Nil		----
237		----		----			----			----
255		----		----			----			----
325	D892	0		----	0		----	0		----
349		----		----			----			----
432	D892	0		----	0		----	0		----
614	D892	0		----	0		----	0		----
621		----		----			----			----
663		----		----			----			----
780		----		----			----			----
862	D892	0		----	0		----	0		----
902		----		----			----			----
912		----		----			----			----
962		----		----			----			----
963	D892	0		----	0		----	0		----
994		----		----			----			----
1011	D892	0		----	0		----	0		----
1026	D892	20	G(0.01), false positive?	----	0		----	0		----
1059	D892	0		----	0		----	0		----
1146	D892	0		----	0		----	0		----
1417	D892	0		----	0		----	0		----
1435	ISO6247	0		----	0		----	0		----
1456		----		----			----			----
1630		----		----			----			----
1660		----		----			----			----
1682		----		----			----			----
1704	D892	0		----	0		----	0		----
1720		----		----			----			----
1740		----		----			----			----
1748		----		----			----			----
1768		----		----			----			----
1875		----		----			----			----
1890		----		----			----			----
1957		0		----	0		----	0		----
4043		----		----			----			----
6002	ISO6247	0		----	0		----	0		----
6016	D892	0		----	0		----	0		----
6032		----		----			----			----
6034		----		----			----			----
6056		----		----			----			----
6117		----		----			----			----
6121		----		----			----			----
6127		----		----			----			----
7003		----		----			----			----
normality		n.a.			n.a.			n.a.		
n		14			16			15		
outliers		1			0			0		
mean (n)		0			0			0		
st.dev. (n)		0			0			0		
R(calc.)		0			0			0		
st.dev.(D892:13e1)		(0)			(0)			(0)		
R(D892:13e1)		(0)			(0)			(0)		

Determination of Kinematic Viscosity at 40°C on sample #17225; results in mm<sup>2</sup>/s

lab	method	value	mark	z(targ)	remarks
173	D445	97.86		0.04	
178	D445	97.7		-0.34	
179	D445	97.66		-0.43	
237	D445	97.48		-0.85	
255		97.76		-0.19	method used: D7279 corrected to D445
325	D445	97.71		-0.31	
349	D445	97.63		-0.50	
432	D445	97.85		0.02	
614	D445	99.10	R(0.01)	2.95	
621	D445	99.12	C,R(0.01)	3.00	first reported 96.41
663	D445	98.062		0.51	
780	D445	97.92		0.18	
862	D445	97.89		0.11	
902	D445	98.00		0.37	
912	D445	98.01		0.39	
962	D445	97.18		-1.56	
963	D445	97.92		0.18	
994	D445	98.01		0.39	
1011		----		----	
1026	D445	98.12	C	0.65	first reported 33.98
1059	ISO3104	97.85		0.02	
1146	D445	97.807		-0.08	
1417	D445	97.76		-0.19	
1435		----		----	
1456	D445	97.63		-0.50	
1630	D445	97.9723		0.30	
1660		----		----	
1682		----		----	
1704	D445	99.60	C,R(0.01)	4.12	first reported 96.302
1720		----		----	
1740	D445	98.89		2.46	
1748		----		----	
1768	ISO3104	97.59		-0.59	
1875		----		----	
1890	ISO3104	98.131		0.68	
1957	D445	97.82		-0.05	
4043		----		----	
6002	ISO3104	97.77		-0.17	
6016		----		----	
6032		97.755		-0.21	method used: D7279 corrected to D445
6034	D445	98.10		0.60	
6056	D445	98.047		0.48	
6117	GB/T265	97.745		-0.23	
6121		----		----	
6127	D445	97.35		-1.16	
7003		----		----	

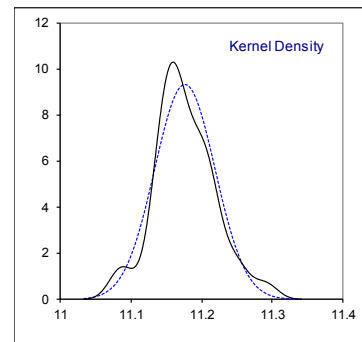
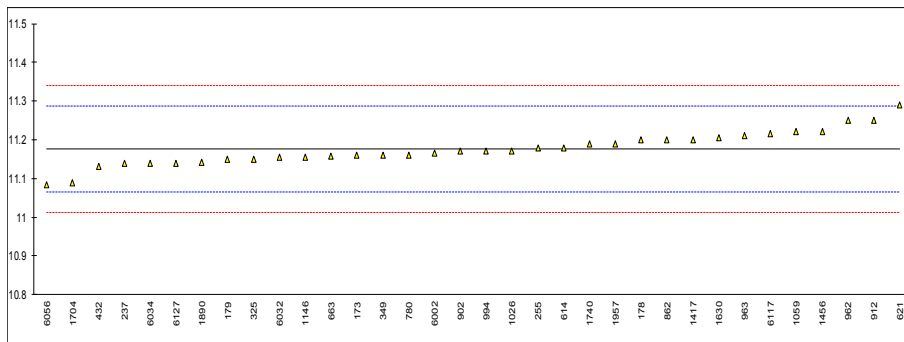
normality not OK  
n 32  
outliers 3  
mean (n) 97.8431  
st.dev. (n) 0.28972  
R(calc.) 0.8112  
st.dev.(D445:17a) 0.42632  
R(D445:17a) 1.1937



Determination of Kinematic Viscosity at 100°C on sample #17225; results in mm<sup>2</sup>/s

lab	method	value	mark	z(targ)	remarks
173	D445	11.16		-0.29	
178	D445	11.20		0.44	
179	D445	11.15		-0.47	
237	D445	11.14		-0.65	
255		11.18		0.07	method used: D7279 corrected to D445
325	D445	11.15		-0.47	
349	D445	11.16		-0.29	
432	D445	11.13		-0.84	
614	D445	11.18		0.07	
621	D445	11.29		2.07	
663	D445	11.158		-0.33	
780	D445	11.16		-0.29	
862	D445	11.20		0.44	
902	D445	11.17		-0.11	
912	D445	11.25		1.34	
962	D445	11.25		1.34	
963	D445	11.21		0.62	
994	D445	11.17		-0.11	
1011		----		----	
1026	D445	11.17	C	-0.11	first reported 9.18
1059	ISO3104	11.22		0.80	
1146	D445	11.154		-0.40	
1417	D445	11.20		0.44	
1435		----		----	
1456	D445	11.22		0.80	
1630	D445	11.2052		0.53	
1660		----		----	
1682		----		----	
1704	D445	11.089		-1.58	
1720		----		----	
1740	D445	11.19		0.25	
1748		----		----	
1768		----		----	
1875		----		----	
1890	ISO3104	11.141		-0.64	
1957	D445	11.19		0.25	
4043		----		----	
6002	ISO3104	11.165		-0.20	
6016		----		----	
6032		11.1538		-0.40	method used: D7279 corrected to D445
6034	D445	11.14		-0.65	
6056	D445	11.084		-1.67	
6117	GB/T265	11.215		0.71	
6121		----		----	
6127	D445	11.14		-0.65	
7003		----		----	

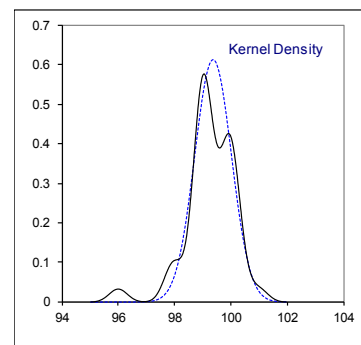
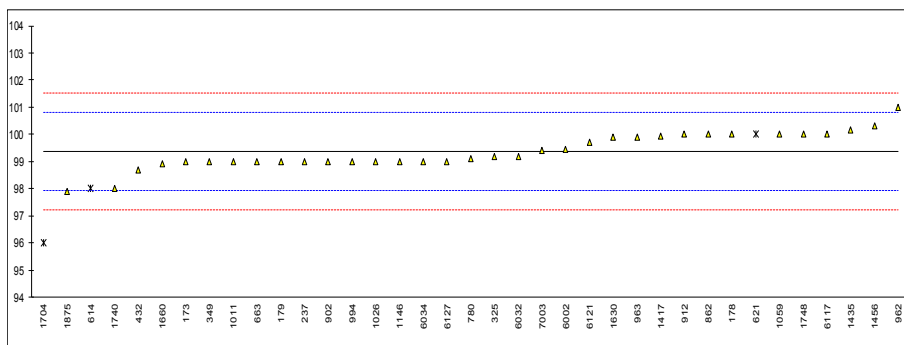
normality OK  
n 34  
outliers 0  
mean (n) 11.1760  
st.dev. (n) 0.04279  
R(calc.) 0.1198  
st.dev.(D445:17a) 0.05508  
R(D445:17a) 0.1542



Determination of Viscosity Index on sample #17225; unit less results

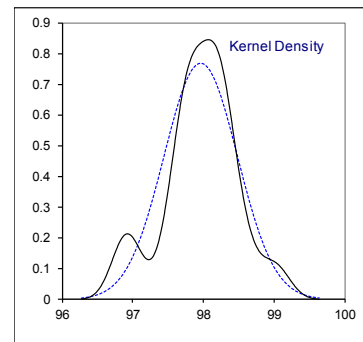
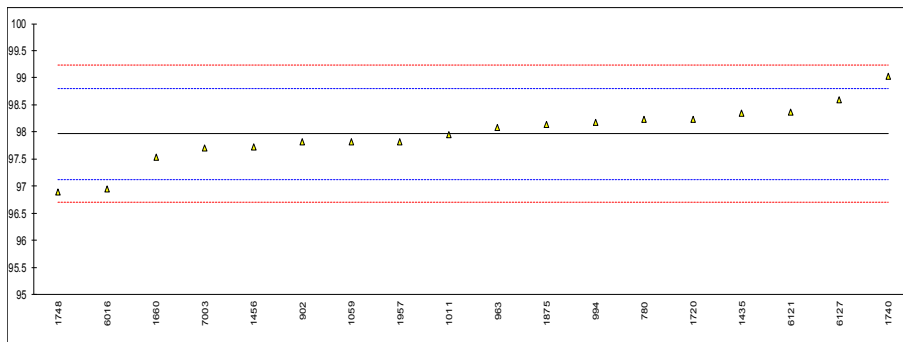
lab	method	value	mark	z(targ)	iis calc.	mark	remarks
173	D2270	99		-0.53	99.19		
178	D2270	100		0.87	100.01		
179	D2270	99		-0.53	99.28		
237	D2270	99		-0.53	99.35		
255		----		----	99.63		
325	D2270	99.2		-0.25	99.22		
349	D2270	99		-0.53	99.48		
432	D2270	98.7		-0.95	98.73		
614	D2270	98	ex	-1.93	97.98	ex	outlier in viscosity 40 °C
621	D2270	100	C,ex	0.87	99.56	ex	first reported 103, outlier in viscosity 40 °C
663	D2270	99		-0.53	98.91		
780	D2270	99.1		-0.39	99.12		
862	D2270	100		0.87	99.78		
902	D2270	99		-0.53	99.18		
912	D2270	100		0.87	100.38		
962	D2270	101		2.27	101.52		
963	D2270	99.9		0.73	99.89		
994	D2270	99		-0.53	99.17		
1011	D2270	99		-0.53	----		
1026	D2270	99	C	-0.53	99.03		first reported 270
1059	ISO2909	100		0.87	100.13		
1146	D2270	99		-0.53	99.16		
1417	D2270	99.94		0.79	99.94		
1435	D2270	100.17		1.11	----		
1456	D2270	100.316		1.32	100.43		
1630	D2270	99.8852		0.71	99.75		
1660	D2270	98.9		-0.67	----		
1682		----		----	----		
1704	D2270	96	C,ex	-4.73	95.89	ex	first reported 100.16, outlier in viscosity 40 °C
1720		----		----	----		
1740	D2270	98		-1.93	98.40		
1748	D2270	100		0.87	99.77		VI based on Stabinger results
1768		----		----	----		
1875	ISO2909	97.9		-2.07	97.55		VI based on Stabinger results
1890		----		----	98.56		
1957		----		----	99.71		
4043		----		----	----		
6002	ISO2909	99.46		0.12	99.38		
6016		----		----	----		
6032	D2270	99.2		-0.25	99.23		
6034	D2270	99		-0.53	98.58		
6056		----		----	97.75		
6117	GB/T1995	100		0.87	100.19		
6121	ISO2909	99.7		0.45	99.49		
6127	D2270	99		-0.53	99.51		
7003	D7042	99.4		0.03	99.48		VI based on Stabinger results

normality	OK	iis calculated	suspect
n	34		35
outliers	0 (+3 ex)		0 (+3 ex)
mean (n)	99.38		99.40
st.dev. (n)	0.652		0.745
R(calc.)	1.83		2.09
st.dev.(D2270:10)	0.714		0.714
R(D2270:10)	2		2



Determination of Viscosity Stabinger at 40°C on sample #17225; results in mm<sup>2</sup>/s

lab	method	value	mark	z(targ)	remarks
173		----		----	
178		----		----	
179		----		----	
237		----		----	
255		----		----	
325		----		----	
349		----		----	
432		----		----	
614		----		----	
621		----		----	
663		----		----	
780	D7042	98.23		0.63	
862		----		----	
902	D7042	97.82		-0.35	
912		----		----	
962		----		----	
963	D7042	98.08		0.27	
994	D7042	98.17		0.48	
1011	D7042	97.95		-0.04	
1026		----		----	
1059	D7042	97.82		-0.35	
1146		----		----	
1417		----		----	
1435	D7042	98.34		0.89	
1456	D7042	97.72		-0.58	
1630		----		----	
1660	D7042	97.531		-1.03	
1682		----		----	
1704		----		----	
1720	D7042	98.24	C	0.65	first reported 99.52
1740	D7042	99.02		2.50	
1748	D7042	96.90		-2.53	
1768		----		----	
1875	D7042	98.133		0.40	
1890		----		----	
1957	D7042	97.82		-0.35	
4043		----		----	
6002		----		----	
6016	D7042	96.947		-2.42	
6032		----		----	
6034		----		----	
6056		----		----	
6117		----		----	
6121	DIN51659-2	98.36		0.94	
6127	D7042	98.60		1.51	
7003	D7042	97.7		-0.63	
normality		OK			
n		18			
outliers		0			
mean (n)		97.9656			
st.dev. (n)		0.51988			
R(calc.)		1.4557			
st.dev.(D7042:16e3)		0.42147			
R(D7042:16e3)		1.1801			

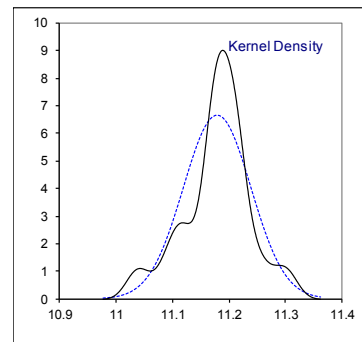
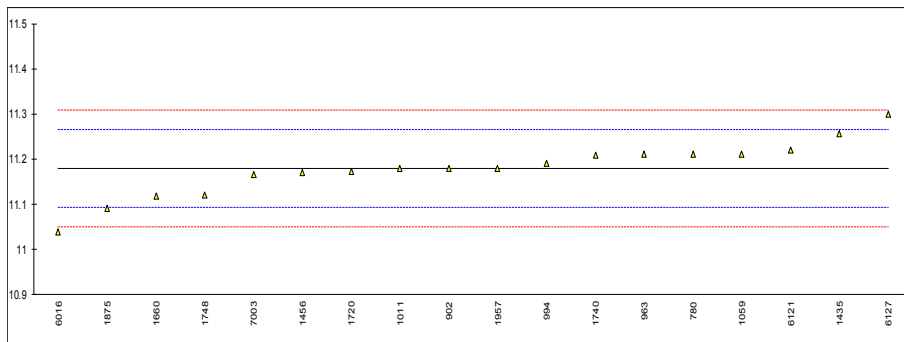




Determination of Viscosity Stabinger at 100°C on sample #17225; results in mm<sup>2</sup>/s

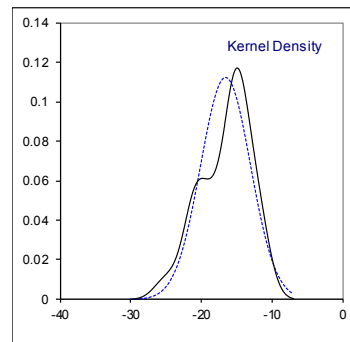
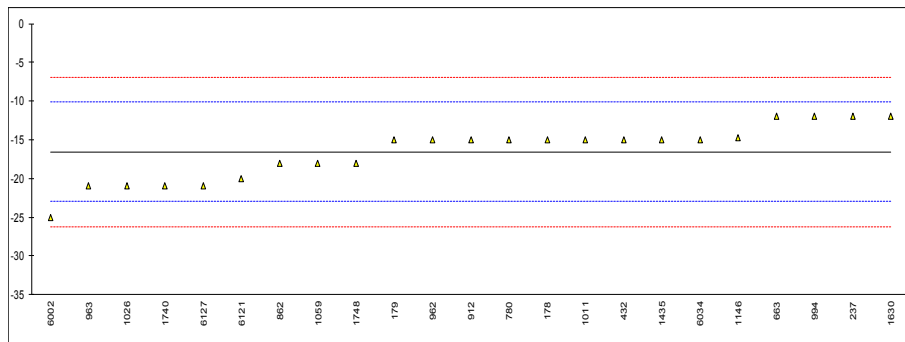
lab	method	value	mark	z(targ)	remarks
173		----		----	
178		----		----	
179		----		----	
237		----		----	
255		----		----	
325		----		----	
349		----		----	
432		----		----	
614		----		----	
621		----		----	
663		----		----	
780	D7042	11.21		0.72	
862		----		----	
902	D7042	11.18		0.02	
912		----		----	
962		----		----	
963	D7042	11.21		0.72	
994	D7042	11.19		0.26	
1011	D7042	11.18		0.02	
1026		----		----	
1059	D7042	11.21		0.72	
1146		----		----	
1417		----		----	
1435	D7042	11.256		1.79	
1456	D7042	11.17		-0.21	
1630		----		----	
1660	D7042	11.117		-1.44	
1682		----		----	
1704		----		----	
1720	D7042	11.173	C	-0.14	first reported 11.62
1740	D7042	11.209		0.70	
1748	D7042	11.121		-1.35	
1768		----		----	
1875	D7042	11.091		-2.05	
1890		----		----	
1957	D7042	11.18		0.02	
4043		----		----	
6002		----		----	
6016	D7042	11.039		-3.26	
6032		----		----	
6034		----		----	
6056		----		----	
6117		----		----	
6121	DIN51659-2	11.22		0.95	
6127	D7042	11.30		2.82	
7003	D7042	11.166		-0.30	

normality suspect  
n 18  
outliers 0  
mean (n) 11.1790  
st.dev. (n) 0.05990  
R(calc.) 0.1677  
st.dev.(D7042:16e3) 0.04295  
R(D7042:16e3) 0.1203



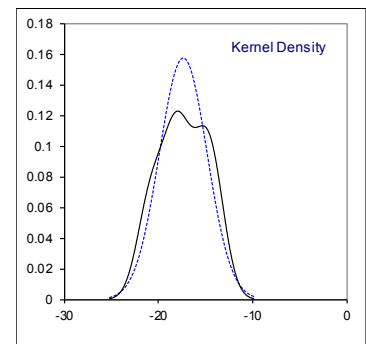
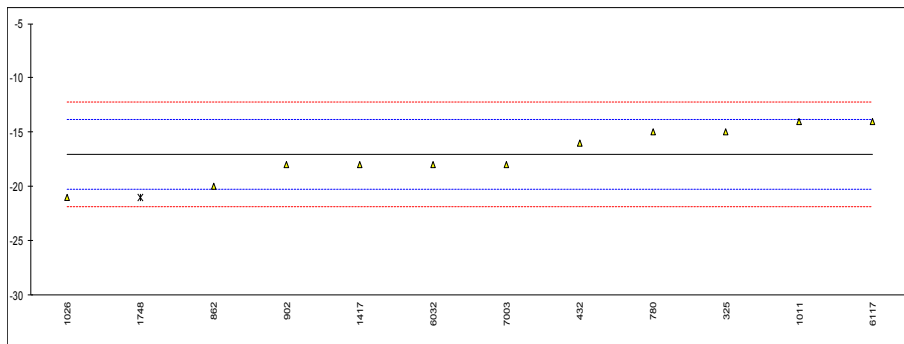
Determination of Pour Point, manual on sample #17225; results in °C

lab	method	value	mark	z(targ)	remarks
173		----		----	
178	D97	-15		0.48	
179	D97	-15		0.48	
237	D97	-12		1.42	
255		----		----	
325		----		----	
349		----		----	
432	D97	-15		0.48	
614		----		----	
621		----		----	
663	D97	-12		1.42	
780	D97	-15		0.48	
862	D97	-18		-0.45	
902		----		----	
912	D97	-15		0.48	
962	D97	-15		0.48	
963	D97	-21		-1.38	
994	D97	-12		1.42	
1011	D97	-15		0.48	
1026	D97	-21		-1.38	
1059	ISO3016	-18		-0.45	
1146	D97	-14.8		0.55	
1417		----		----	
1435	ISO3016	-15		0.48	
1456		----		----	
1630	D97	-12		1.42	
1660		----		----	
1682		----		----	
1704		----		----	
1720		----		----	
1740	D97	-21		-1.38	
1748	D97	-18		-0.45	
1768		----		----	
1875		----		----	
1890		----		----	
1957		----		----	
4043		----		----	
6002	ISO3016	-25		-2.63	
6016		----		----	
6032		----		----	
6034	D97	-15		0.48	
6056		----		----	
6117		----		----	
6121	In house	-20		-1.07	
6127	D97	-21		-1.38	
7003		----		----	
normality		OK			
n		23			
outliers		0			
mean (n)		-16.56			
st.dev. (n)		3.544			
R(calc.)		9.92			
st.dev.(D97:17a)		3.214			
R(D97:17a)		9			



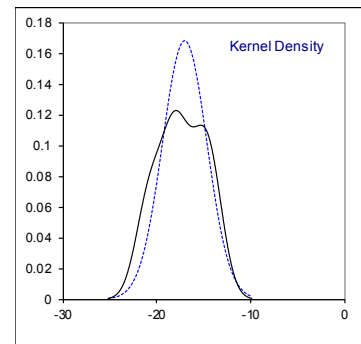
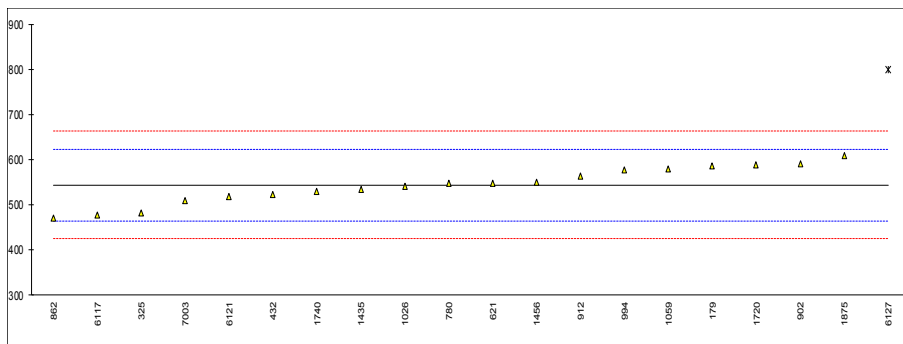
Determination of Pour Point, automated, 1°C interval on sample #17225; results in °C

lab	method	value	mark	z(targ)	remarks
173		----		----	
178		----		----	
179		----		----	
237		----		----	
255		----		----	
325	D5950	-15		1.24	
349		----		----	
432	D5950	-16		0.62	
614		----		----	
621		----		----	
663		----		----	
780	D5950	-15		1.24	
862	D5950	-20		-1.87	
902	D5950	-18		-0.62	
912		----		----	
962		----		----	
963		----		----	
994		----		----	
1011	D6892	-14		1.87	
1026	D5950	-21		-2.49	
1059		----		----	
1146		----		----	
1417	D5950	-18		-0.62	
1435		----		----	
1456		----		----	
1630		----		----	
1660		----		----	
1682		----		----	
1704		----		----	
1720		----		----	
1740		----		----	
1748	D7346	-21	ex	-2.49	excluded: 3 °C interval
1768		----		----	
1875		----		----	
1890		----		----	
1957		----		----	
4043		----		----	
6002		----		----	
6016		----		----	
6032	D97	-18		-0.62	
6034		----		----	
6056		----		----	
6117	NB/SH/T0886	-14		1.87	
6121		----		----	
6127		----		----	
7003	D5950	-18		-0.62	
normality		OK			
n		11			
outliers		0 (+1 ex)			
mean (n)		-17.00			
st.dev. (n)		2.366			
R(calc.)		6.63			
st.dev.(D5950:14)		1.607			
R(D5950:14)		4.5			



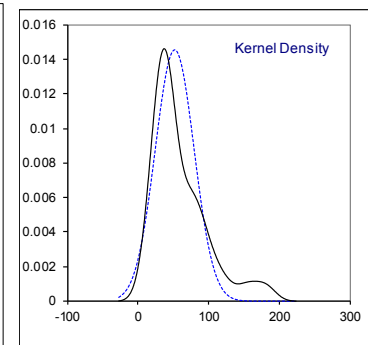
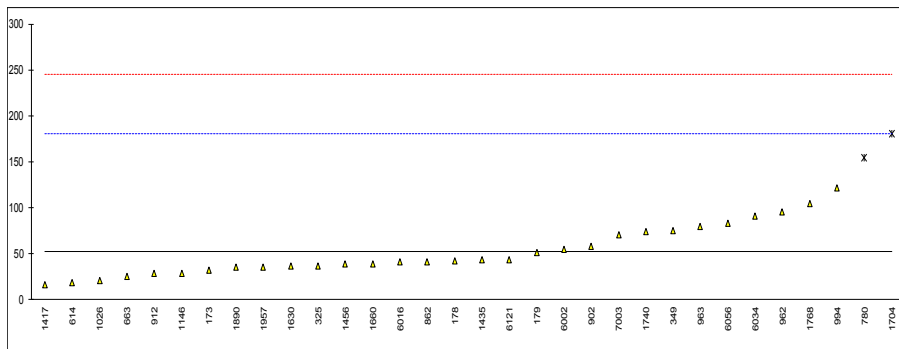
Determination of Sulphur on sample #17225; results in mg/kg

lab	method	value	mark	z(targ)	remarks
173		----		----	
178		----		----	
179	D4294	586		1.08	
237		----		----	
255		----		----	
325	D5185	482		-1.54	
349		----		----	
432	D5185	523.7		-0.49	
614		----		----	
621	D4294	548		0.12	
663		----		----	
780	D4294	547		0.09	
862	D2622	471		-1.82	
902	D4294	590		1.18	
912	D4294	564		0.52	
962		----		----	
963		----		----	
994	D4294	578		0.87	
1011		----		----	
1026	D2622	540		-0.08	
1059	ISO14596Mod.	580		0.92	
1146		----		----	
1417		----		----	
1435	D5185	533		-0.26	
1456	D5185	549		0.14	
1630		----		----	
1660	D4294	<50		<-12.43	possibly a false negative test result?
1682		----		----	
1704		----		----	
1720	D4294	588		1.13	
1740	D4294	530		-0.33	
1748		----		----	
1768		----		----	
1875	DIN51724	609		1.66	
1890		----		----	
1957		----		----	
4043		----		----	
6002		----		----	
6016		----		----	
6032		----		----	
6034		----		----	
6056		----		----	
6117	SH/T0689	477		-1.67	
6121	In house	519		-0.61	
6127	D5185	800	R(0.01)	6.47	
7003	D5453	507.9		-0.89	
normality		OK			
n		19			
outliers		1			
mean (n)		543.29			
st.dev. (n)		40.324			
R(calc.)		112.91			
st.dev.(D4294:16e1)		39.695			
R(D4294:16e1)		111.15			



Determination of Water Content by KF on sample #17225; results in mg/kg

lab	method	value	mark	z(targ)	remarks
173	D6304-C	32		-0.31	
178	D6304-C	42		-0.15	
179	D6304-C	51		-0.01	
237		----		----	
255		----		----	
325	D6304-C	36.5		-0.24	
349	D6304-A	75		0.36	
432		----		----	
614	D6304-A	18		-0.53	
621		----		----	
663	D6304-C	25.4		-0.41	
780	D6304-A	154	R(0.05)	1.58	
862	D6304-C	41		-0.17	
902	D6304-A	57.9		0.09	
912	D6304-C	28		-0.37	
962	D6304-A	95		0.67	
963	D6304-A	79		0.42	
994	D6304-A	122		1.09	
1011		----		----	
1026	D6304-C	21		-0.48	
1059		----		----	
1146	D6304-C	28		-0.37	
1417	D6304-A	16		-0.56	
1435	D6304-A	43		-0.14	
1456	D6304-A	39	C	-0.20	first reported <10
1630	ISO13567	36.28		-0.24	
1660	IEC60814	39		-0.20	
1682		----		----	
1704	D6304-A	180	R(0.05)	1.99	
1720		----		----	
1740	D6304-C	74		0.34	
1748		----		----	
1768	ISO3733	105		0.82	
1875		----		----	
1890	EN60814	35.0		-0.26	
1957	D6304-A	35.2		-0.26	
4043		----		----	
6002	D6304-C	55.02		0.05	
6016	D6304-A	40.7		-0.17	
6032		----		----	
6034	D6304-A	90.57		0.60	
6056	ISO12937	83		0.48	
6117	GB/T260	----		----	reported: trace %M/M
6121	DIN51777	43		-0.14	
6127		----		----	
7003	E1064	70		0.28	
normality		OK			
n		30			
outliers		2			
mean (n)		51.886			
st.dev. (n)		27.3528			
R(calc.)		76.588			
st.dev.(D6304:16e1)		64.4976			
R(D6304:16e1)		180.593			

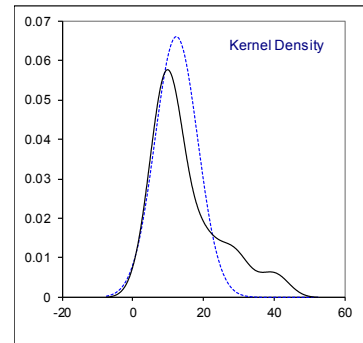
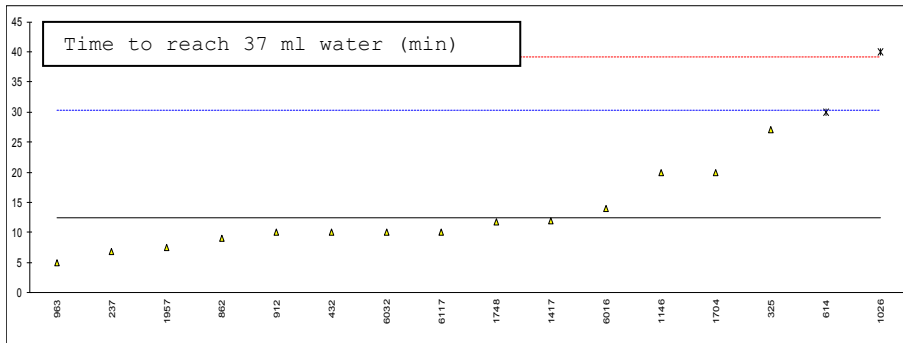
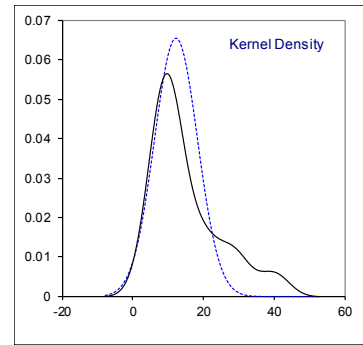
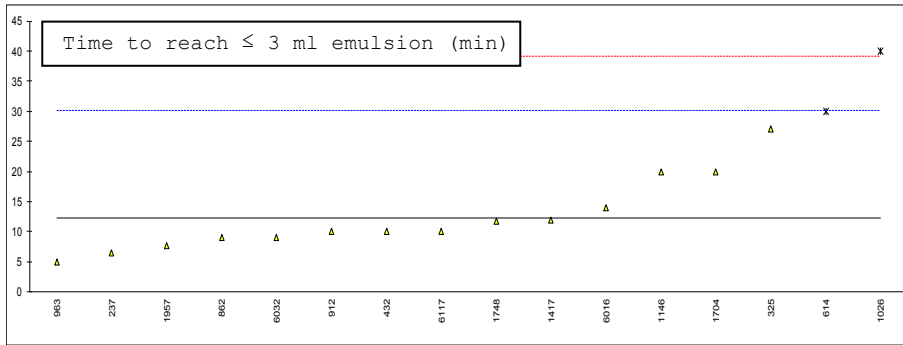


Determination of Water Separability at 82 °C, distilled water on sample #17225; results in min.

lab	method	≤3 ml emul.	mark	z(targ)	37 ml water	mark	z(targ)	compl. break	mark	z(targ)	aborted	time aborted
173		----		----	----		----	----		----	----	----
178		----		----	----		----	----		----	----	----
179		----		----	----		----	10		----	NO	----
237	D1401	6.498		-0.65	6.78		-0.63	10.282		----	NO	----
255		----		----	----		----	----		----	----	----
325	D1401	27		1.65	27		1.64	----		----	YES	27
349		----		----	----		----	----		----	----	----
432	D1401	10		-0.26	10		-0.26	>60		----	YES	60
614	D1401	30	DG(0.05)	1.98	30	DG(0.05)	1.98	----		----	YES	----
621		----		----	----		----	----		----	----	----
663		----		----	----		----	----		----	----	----
780		----		----	----		----	----		----	----	----
862	D1401	9		-0.37	9		-0.38	10		----	NO	----
902		----		----	----		----	----		----	----	----
912	D1401	10		-0.26	10		-0.26	----		----	NO	----
962		----		----	----		----	----		----	----	----
963	D1401	5		-0.82	5		-0.82	6		----	NO	----
994		----		----	----		----	----		----	----	----
1011		----		----	----		----	20		----	NO	20
1026	D1401	40 *)	C,DG(0.05)	3.10	40 *)	C,DG(0.05)	3.10	45		----	NO	----
1059		----		----	----		----	----		----	----	----
1146	D1401	20		0.86	20		0.86	----		----	----	----
1417	D1401	12		-0.03	12		-0.04	12		----	NO	----
1435		----		----	----		----	----		----	----	----
1456		----		----	----		----	----		----	----	----
1630		----		----	----		----	----		----	----	----
1660		----		----	----		----	----		----	----	----
1682		----		----	----		----	----		----	----	----
1704		20		0.86	20		0.86	20		----	NO	----
1720		----		----	----		----	----		----	----	----
1740		----		----	----		----	----		----	----	----
1748		11.75		-0.06	5		-0.07	16		----	NO	----
1768		----		----	----		----	----		----	----	----
1875		----		----	----		----	----		----	----	----
1890		----		----	----		----	----		----	----	----
1957	D1401	7.67		-0.52	7.59		-0.53	15.34		----	----	----
4043		----		----	----		----	----		----	----	----
6002	ISO6614	>60	false positive?	>5.34	>60	false positive?	>5.34	>60		----	YES	60
6016	D1401	14		0.19	14		0.18	>30		----	YES	30 **)
6032	D1401	9		-0.37	10		-0.26	10		----	NO	----
6034		----		----	----		----	----		----	----	----
6056		----		----	----		----	----		----	----	----
6117	GB/T7305	10		-0.26	10		-0.26	10		----	----	----
6121		----		----	----		----	----		----	----	----
6127		----		----	----		----	----		----	----	----
7003		----		----	----		----	----		----	----	----
normality		not OK			not OK							
n		14			14							
outliers		2			2							
mean (n)		12.28			12.37							
st.dev. (n)		6.102			6.050							
R(calc.)		17.08			16.94							
st.dev.(D1401:12e1)		8.929			8.929							
R(D1401:12e1)		25			25							

\*)  
Lab 1026 first reported 45, 45

\*\*)  
Lab 6016 reported that the test has been aborted at 30 min. ASTM method D1401 describes that the test may be aborted after 60 min when testing at 82 °C



Determination of Water Separability at 82 °C, distilled water on sample #17225; results in ml.

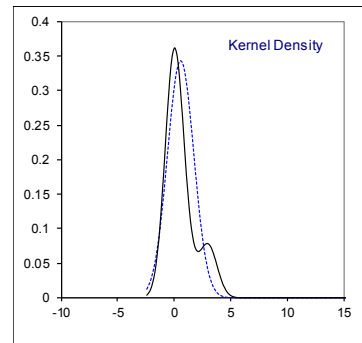
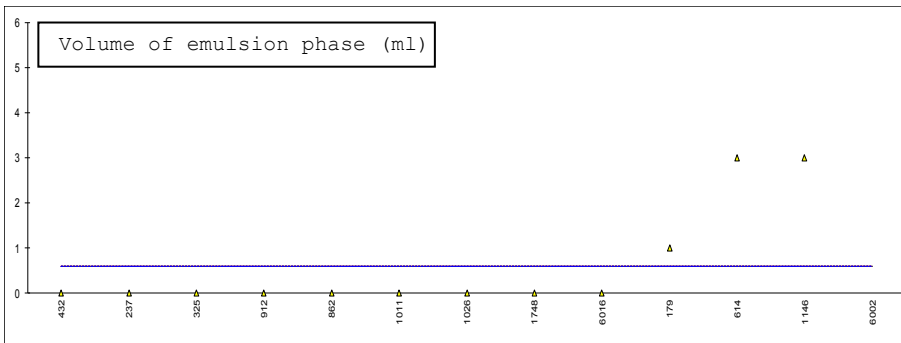
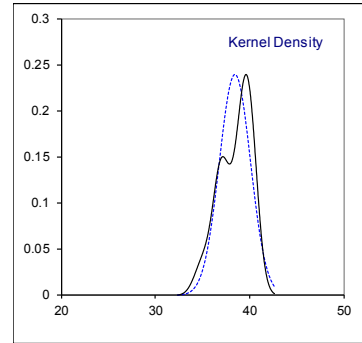
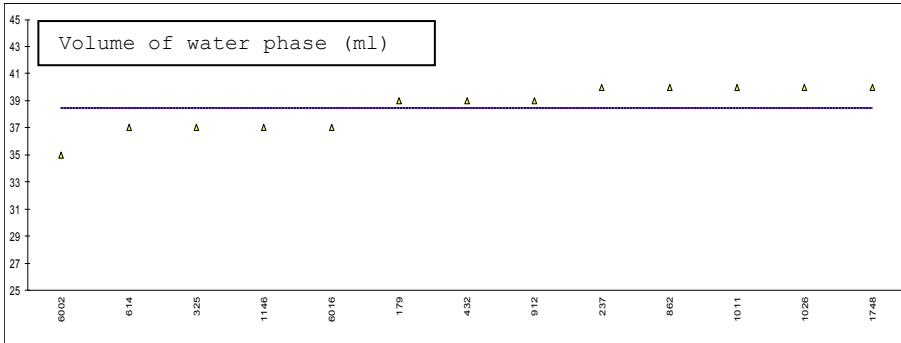
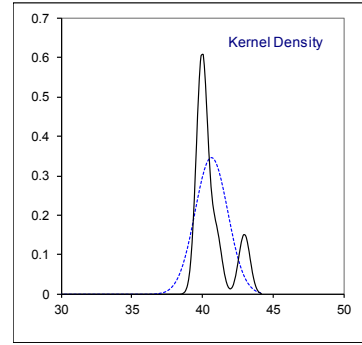
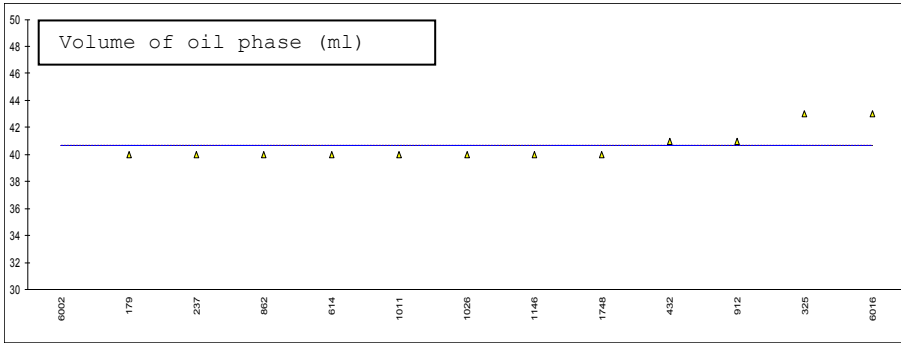
--- Continued ---

lab	method	oil	mark	z(targ)	water	mark	z(targ)	emulsion	mark	z(targ)	aborted	time aborted
173		----		----	----		----	----		----	----	----
178		----		----	----		----	----		----	----	----
179		40		----	39		----	1		----	NO	----
237	D1401	40		----	40		----	0		----	NO	----
255		----		----	----		----	----		----	----	----
325	D1401	43		----	37		----	0		----	YES	27
349		----		----	----		----	----		----	----	----
432	D1401	41		----	39		----	0		----	YES	60
614	D1401	40		----	37		----	3		----	YES	----
621		----		----	----		----	----		----	----	----
663		----		----	----		----	----		----	----	----
780		----		----	----		----	----		----	----	----
862	D1401	40		----	40		----	0		----	NO	----
902		----		----	----		----	----		----	----	----
912	D1401	41		----	39		----	0		----	NO	----
962		----		----	----		----	----		----	----	----
963	D1401	----		----	----		----	----		----	NO	----
994		----		----	----		----	----		----	----	----
1011		40		----	40		----	0		----	NO	20
1026	D1401	40		----	40		----	0		----	NO	----
1059		----		----	----		----	----		----	----	----
1146	D1401	40		----	37		----	3		----	----	----
1417	D1401	----		----	----		----	----		----	NO	----
1435		----		----	----		----	----		----	----	----
1456		----		----	----		----	----		----	----	----
1630		----		----	----		----	----		----	----	----
1660		----		----	----		----	----		----	----	----
1682		----		----	----		----	----		----	----	----
1704		----		----	----		----	----		----	NO	----
1720		----		----	----		----	----		----	----	----
1740		----		----	----		----	----		----	----	----
1748		40		----	40		----	0		----	NO	----
1768		----		----	----		----	----		----	----	----
1875		----		----	----		----	----		----	----	----
1890		----		----	----		----	----		----	----	----
1957	D1401	----		----	----		----	----		----	----	----
4043		----		----	----		----	----		----	----	----
6002	ISO6614	5 *)	G(0.01)	----	35		----	40 *)	G(0.01)	----	YES	60
6016	D1401	43		----	37		----	0		----	YES	30
6032	D1401	----		----	----		----	----		----	NO	----
6034		----		----	----		----	----		----	----	----
6056		----		----	----		----	----		----	----	----
6117	GB/T7305	----		----	----		----	----		----	----	----
6121		----		----	----		----	----		----	----	----
6127		----		----	----		----	----		----	----	----
7003		----		----	----		----	----		----	----	----
normality		not OK			OK			not OK				
n		12			13			12				
outliers		1			0			1				
mean (n)		40.67			38.46			0.58				
st.dev. (n)		1.155			1.664			1.165				
R(calc.)		3.23			4.66			3.26				
st.dev.(target)		n.a.			n.a.			n.a.				
R(target)		n.a.			n.a.			n.a.				

\*)

Lab 6002 probably mixed up the test results for volume of oil phase and the test results of volume of emulsion phase



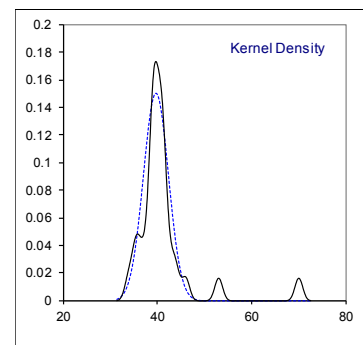
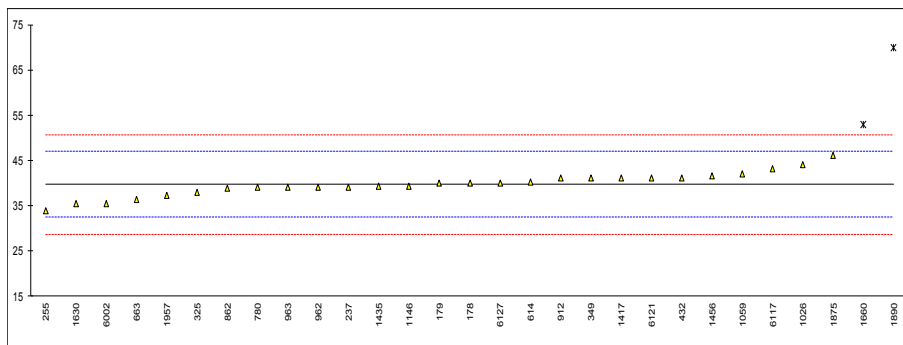


Determination of Calcium (Ca) on sample #17225; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
173		----		----	
178	D5185	40		0.08	
179	D5185	40		0.08	
237		39.12	C	-0.16	first reported 33.80
255	INH-OL1	33.91	C	-1.59	first reported 31.4
325	D5185	38		-0.47	
349		41		0.36	
432	D4951	41.1		0.38	
614	D5185	40.2		0.14	
621		----		----	
663	D5185	36.35		-0.92	
780	D5185	39		-0.19	
862	D5185	38.8		-0.25	
902		----		----	
912	D5185	41		0.36	
962		39		-0.19	
963	D5185	39.0		-0.19	
994		----		----	
1011		----		----	
1026	D5185	44		1.18	
1059	In house	42		0.63	
1146	In house	39.31		-0.11	
1417	D5185	41		0.36	
1435		39.26		-0.12	
1456	D5185	41.6		0.52	
1630	D5185	35.38	C	-1.18	first reported 25.68
1660	D5185	53	R(0.01)	3.64	
1682		----		----	
1704		----		----	
1720		----		----	
1740		----		----	
1748		----		----	
1768		----		----	
1875	DIN51460-1	46		1.73	
1890	In house	70	R(0.01)	8.30	
1957	D5185	37.20		-0.68	
4043		----		----	
6002	D6595	35.42		-1.17	
6016		----		----	
6032		----		----	
6034		----		----	
6056		----		----	
6117	GB/T17476	43.19		0.96	
6121	DIN51777	41		0.36	
6127	D5185	40		0.08	
7003		----		----	

normality OK  
n 27  
outliers 2  
mean (n) 39.698  
st.dev. (n) 2.6481  
R(calc.) 7.415  
st.dev.(Horwitz) 3.6496  
R(Horwitz) 10.219

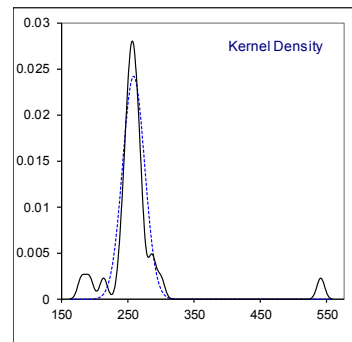
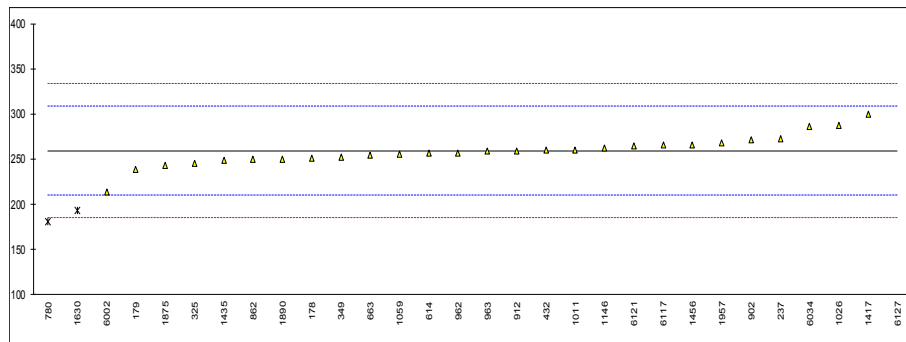
compare R(D5185:13e1) = 1.808 (application range 40-9000 mg/kg)



Determination of Phosphorus (P) on sample #17225; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
173		----		----	
178	D5185	251		-0.33	
179	D5185	239		-0.82	
237		272.3	C	0.53	first reported 29.31
255		----		----	
325	D5185	245		-0.57	
349		252		-0.29	
432	D4951	259.6		0.02	
614	D5185	256.2		-0.12	
621		----		----	
663	D5185	254.8		-0.18	
780	D5185	181	R(0.05)	-3.16	
862	D5185	250		-0.37	
902	D5185	271.0		0.48	
912	D5185	259		-0.01	
962		257		-0.09	
963	D5185	258.5		-0.03	
994		----		----	
1011	D5185	260		0.03	
1026	D5185	287		1.13	
1059	In house	256		-0.13	
1146	In house	262.3		0.13	
1417	D5185	300		1.65	
1435		248.2		-0.44	
1456	D5185	266.2		0.29	
1630	D5185	192.93	R(0.05)	-2.68	
1660		----		----	
1682		----		----	
1704		----		----	
1720		----		----	
1740		----		----	
1748		----		----	
1768		----		----	
1875	DIN51460-1	243		-0.65	
1890	In house	250		-0.37	
1957	D5185	267.9		0.35	
4043		----		----	
6002	D6595	214.03		-1.83	
6016		----		----	
6032		----		----	
6034	D5185	286.29		1.10	
6056		----		----	
6117	GB/T17476	265.8		0.27	
6121	DIN51777	265		0.24	
6127	D5185	540	R(0.01)	11.36	
7003		----		----	

normality not OK  
n 27  
outliers 3  
mean (n) 259.153  
st.dev. (n) 16.4787  
R(calc.) 46.140  
st.dev.(D5185:13e1) 24.7223  
R(D5185:13e1) 69.222

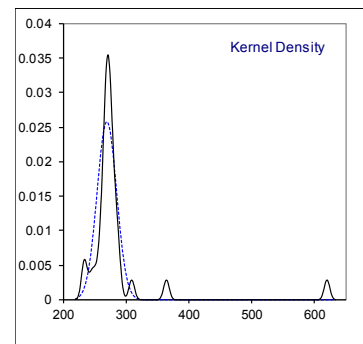
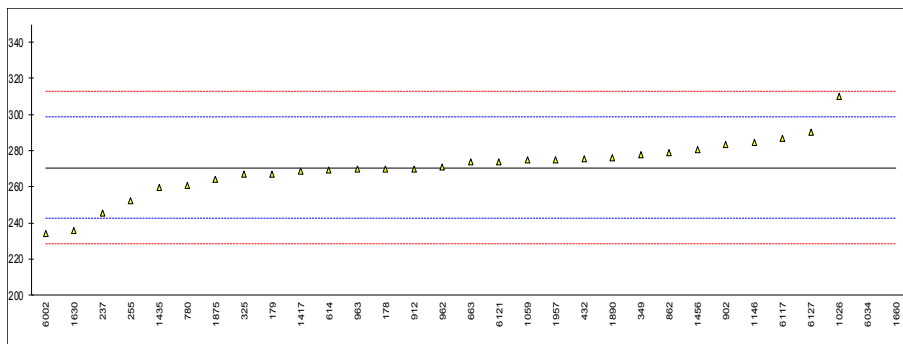


Determination of Zinc (Zn) on sample #17225; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
173		----		----	
178	D5185	270		-0.04	
179	D5185	267		-0.25	
237		245.5		-1.78	
255	INH-OL1	252		-1.32	
325	D5185	267		-0.25	
349		278		0.53	
432	D4951	275.6		0.36	
614	D5185	269.2		-0.10	
621		----		----	
663	D5185	273.7		0.22	
780	D5185	261		-0.68	
862	D5185	279		0.60	
902	D5185	283.5		0.92	
912	D5185	270		-0.04	
962		271		0.03	
963	D5185	269.7		-0.06	
994		----		----	
1011		----		----	
1026	D5185	310		2.81	
1059	In house	275		0.32	
1146	In house	284.8		1.01	
1417	D5185	268.5		-0.15	
1435		259.8		-0.77	
1456	D5185	280.3		0.69	
1630	D5185	235.6	C	-2.49	first reported 202.36
1660	D5185	620	R(0.01)	24.89	
1682		----		----	
1704		----		----	
1720		----		----	
1740		----		----	
1748		----		----	
1768		----		----	
1875	DIN51460-1	264		-0.47	
1890	In house	276		0.39	
1957	D5185	275.0		0.32	
4043		----		----	
6002	D6595	234.13		-2.59	
6016		----		----	
6032		----		----	
6034	D5185	365.12	R(0.01)	6.73	
6056		----		----	
6117	GB/T17476	287.0		1.17	
6121	DIN51777	274		0.24	
6127	D5185	290		1.38	
7003		----		----	

normality  
n  
outliers  
mean (n)  
st.dev. (n)  
R(calc.)  
st.dev.(D5185:13e1)  
R(D5185:13e1)

suspect  
29  
2  
270.563  
15.4879  
43.366  
14.0416  
39.316



**APPENDIX 2**

## Reported details: Foam determination

lab	method	Sample used	Diffuser type	Cylinder cleansed	Gas diffuser cleansed	Air tube cleansed	Air flow rate constant
173		----	----	----	----	----	----
178		----	----	----	----	----	----
179	D892	As received	Metal (stainless steel)	YES	YES	Unknown	Unknown
237		----	----	----	----	----	----
255		----	----	----	----	----	----
325	D892	As received	Metal (stainless steel)	YES	YES	YES	Yes: without readjustment
349		----	----	----	----	----	----
432	D892	As received	Stone (non-metallic)	YES	YES	YES	Yes: without readjustment
614	D892	As received	Metal (stainless steel)	YES	YES	NO	Yes: without readjustment
621		----	----	----	----	----	----
663		----	----	----	----	----	----
780		----	----	----	----	----	----
862	D892	As received	Stone (non-metallic)	YES	YES	YES	Yes: without readjustment
902		----	----	----	----	----	----
912		----	----	----	----	----	----
962		----	----	----	----	----	----
963	D892	As received	Metal (stainless steel)	YES	YES	YES	Yes: without readjustment
994		----	----	----	----	----	----
1011	D892	After agitation, option A	Metal (stainless steel)	YES	YES	YES	Yes: without readjustment
1026	D892	As received	Metal (stainless steel)	YES	YES	YES	Yes: without readjustment
1059	D892	As received	Metal (stainless steel)	YES	YES	YES	Yes: readjustment needed
1146	D892	As received	Metal (stainless steel)	YES	YES	YES	Yes: without readjustment
1417	D892	As received	Metal (stainless steel)	YES	YES	YES	Yes: readjustment needed
1435	ISO6247	----	Stone (non-metallic)	YES	YES	NO	Yes: without readjustment
1456		----	----	----	----	----	----
1630		----	----	----	----	----	----
1660		----	----	----	----	----	----
1682		----	----	----	----	----	----
1704	D892	As received	Metal (stainless steel)	YES	YES	YES	Yes: without readjustment
1720		----	----	----	----	----	----
1740		----	----	----	----	----	----
1748	D892	----	----	----	----	----	----
1768		----	----	----	----	----	----
1875		----	----	----	----	----	----
1890		----	----	----	----	----	----
1957		As received	Metal (stainless steel)	YES	NO	NO	----
4043		----	----	----	----	----	----
6002	ISO6247	----	Metal (stainless steel)	YES	YES	YES	Yes: without readjustment
6016	D892	As received	Stone (non-metallic)	YES	YES	YES	Yes: without readjustment
6032		----	----	----	----	----	----
6034		----	----	----	----	----	----
6056		----	----	----	----	----	----
6117		----	----	----	----	----	----
6121		----	----	----	----	----	----
6127		----	----	----	----	----	----
7003		----	----	----	----	----	----

## Reported details: Foam determination – continued

lab	method	cleansing cylinder	cleansing gas diffuser	cleansing air tube
173				
178				
179	D892		Per D892 Standard	Per D892 Standard
237				
255				
325	D892	with solvent in Seta cleaning device	with detergent and solvent	with solvent in Seta cleaning device
349				
432	D892			
614	D892	air is constantly on, no cleaning required	washed with solvent 13	flushed with solvent 13 and air
621				
663				
780				
862	D892	n-heptane	petroleum ether 60-90Å, absolute ethyl alcohol, deionized water, absolute ethyl alcohol	n-heptane
902				
912				
962				
963	D892	Cleaned the gas tube with heptane & toluene, wipe with moistened heptane & dry cloth.	Cleaned the cylinder with heptane, Washed with distilled water, then acetone and dry in oven	Cleaned the gas diffuser with heptane & toluene, repeated washing 5 times & dried in clean air.
994				
1011	D892	heptane	heptane	heptane
1026	D892			
1059	D892	According 9.1.2. (see ASTM D 892).	Rinsing with solvent (heptane or similar); after that washing and drying in washing machine.	According 9.1.2. (see ASTM D 892).
1146	D892	Petroleum ether & dried with dry air	Petroleum ether & dried with dry air	Petroleum ether & dried with dry air
1417	D892	Toluene & Petroleum Spirit	Detergent, Petroleum Spirit, Toluene, Distilled water & acetone with heptane and ether	Toluene & Petroleum Spirit
1435	ISO6247			placed in heptane, blow air through it, repeat with ether and blow dry with air
1456				
1630				
1660				
1682				
1704	D892	by Heptane	by Toluene and heptane	by Toluene and heptane
1720				
1740				
1748	D892			
1768				
1875				
1890				
1957		Not applicable	Used toluene, wash with tap water, wash with acetone and dry in oven	Not applicable
4043				
6002	ISO6247	the same like a diffuser	we washed cylinder with toluene, 2-propanol next detergent, rinsed with distilled water and acetone.	Diffuser cleansed with toluene and heptane, next dried with air
6016	D892	air, wipe outside of air inlet tube with cloth moistened with heptane, then dry cloth	Heptane, detergent, DI water, acetone, drying oven	Toluene, air, heptane, air
6032				
6034				
6056				
6117				
6121				
6127				
7003				

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

1 lab in ALGERIA  
2 labs in AUSTRALIA  
1 lab in AUSTRIA  
1 lab in AZERBAIJAN  
3 labs in BELGIUM  
2 labs in CHINA, People's Republic  
1 lab in FRANCE  
2 labs in GERMANY  
1 lab in GREECE  
3 labs in INDIA  
1 lab in INDONESIA  
1 lab in IRAN, Islamic Republic of  
1 lab in ITALY  
1 lab in JORDAN  
1 lab in KAZAKHSTAN  
1 lab in MALAYSIA  
2 labs in NETHERLANDS  
1 lab in NIGERIA  
1 lab in PERU  
1 lab in POLAND  
1 lab in PORTUGAL  
1 lab in RUSSIAN FEDERATION  
2 labs in SAUDI ARABIA  
1 lab in SERBIA  
1 lab in SLOVENIA  
2 labs in SPAIN  
1 lab in SUDAN  
1 lab in TAIWAN (R.O.C.)  
1 lab in TANZANIA  
1 lab in THAILAND  
2 labs in TURKEY  
1 lab in UNITED KINGDOM  
3 labs in UNITED STATES OF AMERICA

**APPENDIX 4****Abbreviations:**

C	= final test result after checking of first reported suspect test 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's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= probably an error in calculations
U	= test result probably reported in a different unit
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
SDS	= Safety Data Sheet

**Literature:**

- 1 iis Interlaboratory Studies, Protocol for the Organization, Statistics and Evaluation, March 2017
- 2 ASTM E178:16a
- 3 ASTM E1301:95(2003)
- 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, Fr. Z. Anal. Chem, 331, 513, (1988)
- 12 J.N. Miller, Analyst, 118, 455, (1993)
- 13 Analytical Methods Committee Technical Brief, No 4 January 2001
- 14 P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364 (2002)
- 15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)
- 16 Horwitz, R. Albert, J. AOAC Int, 79, 3, 589, (1996)