# Results of Proficiency Test Hydraulic Fluid (fresh) November 2016

Organised by: Institute for Interlaboratory Studies

Spijkenisse, the Netherlands

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### 1 Introduction

Since 2003, the Institute for Interlaboratory Studies organizes a proficiency test for the analyses on <u>used</u> Hydraulic Fluid every year. In 2014 it was decided to organize a proficiency test for the analyses on <u>fresh</u> Hydraulic Fluid next to used Hydralic Fluid. This decision was based on the outcome of a questionnaire that was sent to all participants in 2014. During the annual program 2016/2017, it was decided to continue the round robin for the analyses on fresh Hydraulic Fluid. In this third interlaboratory study on fresh Hydraulic Fluid, 47 laboratories from 35 different countries did register for participation. See appendix 2 for the number of participants per country. In this report, the test results of the 2016 interlaboratory study on fresh Hydraulic Fluid are presented and discussed. This report can also be downloaded from the iis website <u>www.iisnl.com</u>.

### 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 #16240. Participants were requested to report rounded and unrounded test results. The unrounded test 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 based on IEC/ISO17043:2010 (R007). 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, 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 approximately 150 litre of the bulk material was homogenised. After homogenisation, 73 amber glass bottles of 1 litre were filled and labelled #16240. The homogeneity of the subsamples #16240 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.

	Density at 15 °C in kg/L	Viscosity at 40 °C in mm²/s
Sample #16240-1	0.87238	31.75
Sample #16240-2	0.87238	31.75
Sample #16240-3	0.87237	31.76
Sample #16240-4	0.87238	31.79
Sample #16240-5	0.87238	31.75
Sample #16240-6	0.87238	31.78
Sample #16240-7	0.87238	31.77
Sample #16240-8	0.87238	31.77

Table 1: homogeneity test results of subsamples #16240

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

	Density at 15 °C in kg/L	Viscosity at 40 °C in mm <sup>2</sup> /s
r (observed)	0.00001	0.04
reference test method	D4052:15	D445:15a
0.3 x R (ref. test method)	0.00015	0.12

Table 2: repeatabilities of subsamples #16240

Each calculated repeatability was less than 0.3 times the corresponding reproducibility of the reference test methods. Therefore, homogeneity of the subsamples #16240 was assumed.

To each of the participating laboratories one sample of 1 litre amber glass bottle, labelled #16240, was sent on October 26, 2016.

### 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 #16240; 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.

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 results more, 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 results cannot be used for meaningful statistical calculations.

To get comparable test results a detailed report form, on which the units were prescribed as well as the required reference test methods and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The laboratories were also requested to confirm the sample receipt on the same data entry portal. A SDS was added to the sample.

### 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/. 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 April 2014 (iis-protocol, version 3.3).

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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the test results 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 visualise 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)}} = (test result - average of PT) / target standard deviation
```

The  $z_{(target)}$  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, problems were encountered with the dispatch of the samples. Participants in Saudi Arabia received the samples late due to problems with clearance of the sample at customs. One participant reported the test results after the final reporting date and four participants did not report any test results at all. Not all laboratories were able to report all analyses requested.

The 43 reporting participants sent in 597 numerical test results. Observed were 30 outlying test results, which is 5.0% of the numerical test 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 different 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 3.

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.

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. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D664-A:11ae1.
- <u>Copper Corrosion</u>: This determination was not problematic. All participants agreed on a test result of 1 (1A/1B).
- <u>Density at 15°C:</u> This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D4052:15.
- <u>Flash Point PMcc:</u> This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D93-A:16a.
- Foaming Characteristics (Tendency and Stability): This determination was very problematic. In total five statistical outliers were observed over six parameters. The calculated reproducibilities in the Foam Tendency determination for sequences I and III are not at all in agreement with the requirements of ASTM D892:13e1. Therefore no z-scores were calculated for sequences I and III. However, the calculated reproducibility of sequence II after rejection of the statistical outliers is in agreement with the requirements of ASTM D892:13e1. For Foam stability also no z-scores were calculated. Almost all participants reported 0 ml.

This determination is very sensitive in maintenance and execution. Therefore extra information was asked (sample pre-treatment and type of diffuser), but no conclusion could be drawn based on this information. 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 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 test method the foam layer should completely cover the (oil) surface. It is assumed that the way that the foam height is read will be one of the major sources that contributes to the overall variation.

- <u>Kinematic Viscosity at 40°C:</u> 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 D445:15a.
- <u>Kinematic Viscosity at 100°C:</u> 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.

<u>Viscosity Index</u> This determination was very problematic. One statistical outlier was observed and one another test result was excluded due to a calculation error. The calculated reproducibility after rejection of the suspect data is not at all in agreement with the requirements of ASTM D2270:10(2016).

- <u>Viscosity Stabinger at 40°C:</u> This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of ASTM D7042:16e3.
- <u>Viscosity Stabinger at 100°C:</u> This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in full 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 in full agreement with the requirements of ASTM D97:16.
- Pour Point (automated): This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D5950:14.
- Sulphur: 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 D4294:16e1.
- Water content: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D6304:16e1.
- Water Separability at 54°C: This determination was not problematic. In total one statistical outlier was observed over six parameters. The calculated reproducibilities for "time to reach ≤ 3ml emulsion" and "time to reach 37ml water" are both in good agreement with the requirements of ASTM D1401:12e1.
- Calcium: Almost all reported test 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. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D5185:13e1.
- Zinc: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier 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	32	0.59	0.15	0.22
Copper Corrosion, 3 hrs at 50°C	rating	19	1	n.a.	n.a.
Density at 15°C	kg/L	33	0.8724	0.0004	0.0005
Flash Point PMcc	°C	27	177.8	9.2	12.6
Foam Tendency Seq. I	ml	16	58	200	(33)
Foam Tendency Seq. II	ml	15	18.0	11.6	16.1
Foam Tendency Seq. III	ml	16	1.9	11.3	(0.8)
Foam Stability Seq. I	ml	17	0	0	(0)
Foam Stability Seq. II	ml	17	0	0	(0)
Foam Stability Seq. III	ml	17	0	0	(0)
Kinematic viscosity at 40°C	mm²/s	31	31.872	0.247	0.389
Kinematic viscosity at 100°C	mm²/s	30	6.128	0.084	0.085
Viscosity Index		32	143.5	4.3	2.0
Viscosity Stabinger at 40°C	mm²/s	17	31.845	0.303	0.461
Viscosity Stabinger at 100°C	mm²/s	17	6.128	0.087	0.088
Pour Point (manual)	°C	17	-40.5	9.5	9.0
Pour Point (automated), 1°C int.	°C	10	-44.6	6.1	4.5
Sulphur	mg/kg	20	2957	396	331
Water content (by KF)	mg/kg	31	64.1	92.3	205.1
Water Separability at 54°C, distille	ed water				
- Time ≤ 3 ml emulsion	min	17	14.3	14.1	20.0
- Time 37 ml water	min	15	14.3	8.8	20.0
- Time to reach complete break	min	14	16.4	8.3	n.a.
- Volume Oil phase	ml	15	40.1	1.3	n.a.
- Volume Water phase	ml	16	39.3	3.2	n.a.
- Volume Emulsion phase	ml	16	0.6	3.4	n.a.
Calcium as Ca	mg/kg	22	10.5	4.2	(0.3)
Phosphorus as P	mg/kg	27	308	55	75
Zinc as Zn	mg/kg	29	288	55	42
Table 2: reproducibilities of tests on sample	"10010			•	•

Table 3: reproducibilities of tests on sample #16240

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 and the figures reported between brackets in table 3 have been discussed in paragraph 4.1.

### 4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2016 WITH THE PREVIOUS PTs.

	November 2016	November 2015	November 2014
Number of reporting labs	43	45	29
Number of test results reported	597	569	346
Statistical outliers	30	26	19
Percentage outliers	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	November 2016	November 2015	November 2014
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.		
Foam Stability Seq. I	n.e.	n.e.	n.e.
Foam Stability Seq. II	n.e.	n.e.	n.e.
Foam Stability Seq. III	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 method

++: group performed much better than the reference test method

+ : group performed better than the reference test method

+/-: group performance equals the reference test method

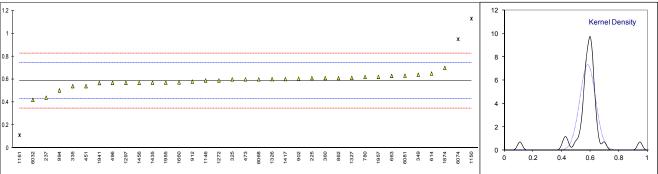
- : group performed worse than the reference test method

-- : group performed much worse than the reference test method

### **APPENDIX 1**

Determination of Acid Number (Total) on sample #16240; results in mg KOH/g

lab	method	value	mark	z(targ)	remarks
173	metriou		mark	<u> </u>	Temarks
225	D974	0.61		0.31	
237	D664-A	0.44		-1.82	
255					
325	D664-A	0.60		0.18	
335	ISO6618	0.54		-0.57	
349	D664-A	0.64		0.68	
360	D974	0.610		0.31	
432					
445					
451		0.54		-0.57	
473	D664-A	0.6		0.18	
496	D664-A	0.57		-0.20	
614	D664-A	0.65		0.81	
663	D664-A	0.63		0.56	
780 862	D664-A D664-A	0.62 0.61		0.43 0.31	
902	D664-A	0.605		0.31	
912	D664-A	0.58		-0.07	
962	D007-74			-0.07	
963					
994	D664-A	0.503		-1.03	
1011	200.71				
1146	D664-A	0.590		0.06	
1150	INH-1752	1.129	C,R(0.01)	6.81	first reported: 1.219
1161	D664-A	0.112	C,R(0.01)	-5.93	first reported: 0.152
1272	ISO6618	0.590		0.06	
1297	D664-A	0.57		-0.20	
1326	D664-A	0.6015		0.20	
1327	D664-A	0.613		0.34	
1417	IP177	0.602		0.21	
1435	D664-A	0.571		-0.18	
1456	D974	0.57		-0.20	
1660 1682	IEC62021-1	0.573 		-0.16 	
1748					
1748					
1874	E2412	0.7		1.43	
1941	ISO6619	0.567		-0.23	
1955	D664-A	0.571		-0.18	
1957	D664-A	0.621		0.44	
1981					
6016					
6032	D664-A	0.42		-2.07	
6068	ISO6618	0.6		0.18	
6074	D664-A	0.95	C,R(0.01)	4.56	first reported: 0.94
6081	D664-A	0.631		0.57	
	normality	not OK			
	n	32			
	outliers	3			
	mean (n) st.dev. (n)	0.5856 0.05449			
	R(calc.)	0.05449			
	R(D664:11ae1)	0.1326			
	(500 1.11001)	0.2200			
1.2 -					12 T

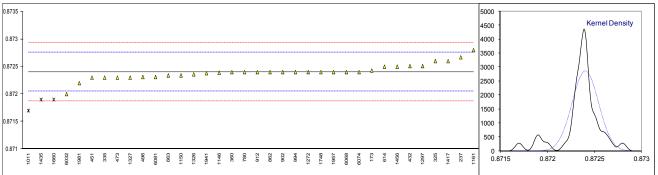


# Determination of Copper Corrosion 3hrs/50°C on sample #16240; results in rating

lab	method	value	mark	z(targ)	remarks
173					
225					
237					
255 325	D130	1.0			
335	D 130	1A 			
349					
360	D130	1A			
432					
445					
451					
473	1000400				
496 614	ISO2160	1A 			
663					
780	D130	1a			
862	D130	1a			
902					
912	D130	1a			
962					
963	D400	4 -			
994	D130	1a			
1011 1146	D130	1a 			
1150	ISO2160	1a			
1161	1002100				
1272					
1297	D130	1A			
1326					
1327	D130	1b			
1417	IP154	1A			
1435 1456	D130	1A			
1660	D100				
1682					
1748	D130	1a			
1797	ISO2160	1a			
1874					
1941	ISO2160	1a			
1955					
1957 1981	D130	 1a			
6016	D130	1a 			
6032					
6068	ISO2160	1a			
6074	D130	1a			
6081					
	normality	n.a.			
	n outliers	19 0			
	mean (n)	1 (1A/1B)			
	st.dev. (n)	n.a.			
	R(calc.)	n.a.			
	R(target)	n.a.			

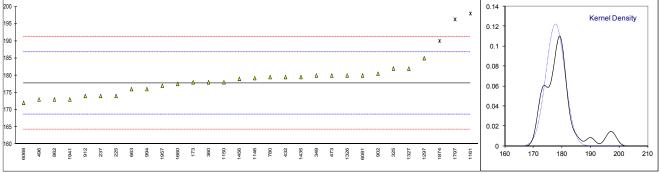
# Determination of Density at 15°C on sample #16240; results in kg/L

lab	method	value	mark	z(targ)	remarks
173	D4052	0.87243	mant	0.14	Tollano
225	D 1002				
237	D4052	0.87267		1.49	
255					
325	D4052	0.8726		1.09	
335	D4052	0.8723		-0.59	
349					
360	D4052	0.8724		-0.03	
432	ISO12185	0.87251		0.59	
445					
451	D4052	0.8723	С	-0.59	first reported: 872.3 kg/L
473	D4052	0.8723	С	-0.59	first reported: 872.3 kg/L
496	D4052	0.87231		-0.53	
614	D4052	0.8725		0.53	
663	D4052	0.87234		-0.36	
780	D4052	0.8724	С	-0.03	reported: 872.4 kg/L
862	D4052	0.8724		-0.03	
902	D4052	0.8724		-0.03	
912 962	D4052	0.8724		-0.03 	
962					
994	D4052	0.8724		-0.03	
1011	D4052	0.8717	R(0.05)	-3.95	
1146	D4052	0.87239	11(0.00)	-0.08	
1150	ISO12185	0.87234		-0.36	
1161	ISO3675	0.8728	С	2.21	first reported: 872.2 kg/L
1272	ISO12185	0.8724	Č	-0.03	first reported: 872.40 kg/L
1297	D4052	0.87251	C	0.59	first reported: 8725.1 kg/L
1326	D4052	0.87236		-0.25	·
1327	D4052	0.8723		-0.59	
1417	IP365	0.8726	С	1.09	first reported: 872.6 kg/L
1435	D4052	0.8719	R(0.05)	-2.83	
1456	D4052	0.8725		0.53	
1660	D7042	0.8719	R(0.05)	-2.83	
1682			_		
1748	D4052	0.8724	С	-0.03	first reported: 872.4 kg/L
1797					
1874	D 4050	0.07000		0.44	
1941	D4052	0.87238	14/	-0.14	first year outside 0.0700
1955 1957	D4052	0.8724	W	-0.03	first reported: 0.8730
1981	D4052 D4052	0.8722	С	-1.15	first reported: 872.2 kg/L
6016	D4032	0.0722	C	-1.15	ilist reported. 672.2 kg/L
6032	D4052	0.8720		-2.27	
6068	ISO12185	0.8724	С	-0.03	first reported: 872.4 kg/L
6074	D4052	0.87240	Ü	-0.03	mot roportou. or 2.1 rig/2
6081	D4052	0.87231	С	-0.53	first reported: 872.31 kg/L
	normality	not OK			
	n	33			
	outliers	3			
	mean (n)	0.872405			
	st.dev. (n)	0.0001395			
	R(calc.)	0.000391			
	R(D4052:15)	0.000500			
0.8735 -	·				5000 т



# Determination of Flash Point PMcc on sample #16240; results in °C

lab	method	value	mark	z(targ)	remarks
173	D93-A	178.0		0.05	
225	D93-A	174.0		-0.84	
237	D93-A	174.0		-0.84	
255					
325	D93-A	182		0.94	
335					
349	D93-A	180		0.50	
360	D93-A	178.0		0.05	
432	D93-A	179.5		0.38	
445	20071				
451					
473	D93-A	180.0		0.50	
496	D93-A	173.0		-1.06	
614	D33-A			-1.00	
663	D93-A	176.0		-0.39	
780	D93-A	179.5		0.38	
862	D93-A	173.0		-1.06	
902	D93-A D93-A	180.5		0.61	
912		174			
962	D93-A	174		-0.84 	
963	D03 A	 176 O		0.20	
994	D93-A	176.0		-0.39	
1011	D02 A	470.0			
1146	D93-A	179.2		0.32	
1150	ISO2719-A	178	C D(0.04)	0.05	first remarked, 202.0
1161	ISO2592	198	C,R(0.01)	4.49	first reported: 203.0
1272	D00 D	405		4.00	
1297	D93-B	185		1.60	
1326	D93-A	180.0		0.50	
1327	D93-A	182.0		0.94	
1417	D00 4	470.5			
1435	D93-A	179.5		0.38	
1456	D93-A	179		0.27	
1660	D93-A	177.5		-0.06	
1682					
1748	1000=00		0.000		
1797	ISO2592	196.3	C,R(0.01)	4.11	first reported: 198.3
1874	D92	190	R(0.05)	2.71	
1941	ISO2719-A	173		-1.06	
1955	Dag 4				
1957	D93-A	177		-0.17	
1981					
6016					
6032					
6068	ISO2719-A	172.0		-1.28	
6074	D92		W		first reported: 200
6081	D93-A	180.0		0.50	
	normality	OK			
	n	27			
	outliers	3			
	mean (n)	177.77			
	st.dev. (n)	3.277			
	R(calc.)	9.18			
	R(D93-A:16a)	12.62			
200 =					0.44

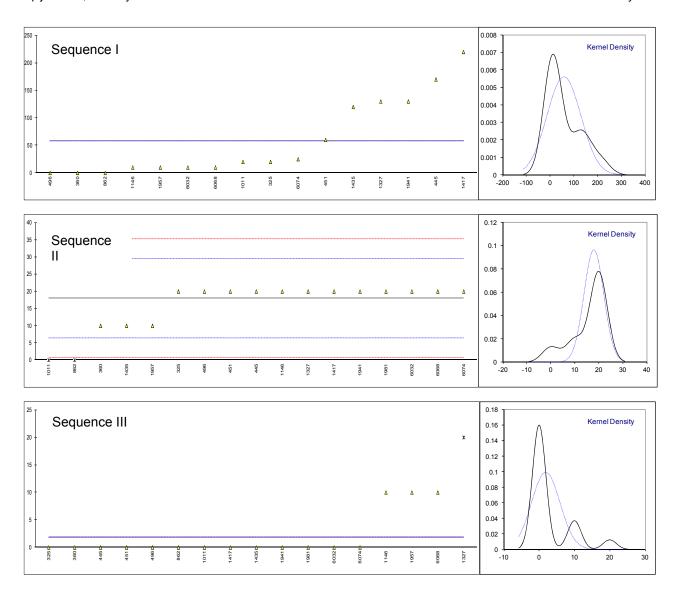


Determination of Foaming Characteristics, Foaming Tendency (at end of 5 min blowing period) on sample #16240: results in ml

173			0; results					- ·-					
225	lab	method	Sample	Diffuser	Seq. I	mark	z(targ)	Seq. II	mark		Seq. III	mark	z(targ)
237													
255   325   D892   As received   Metal/SS   20													
325   D892   As received   Metal/SS   As received   Non-metal   O													
335   349   360   ISO6247   As received   Non-metal   0		D892	As received	Metal/SS							0		
349   360   ISO6247   As received   Non-metal   0		D00 <b>2</b>	710 10001100	Wicken CC									
432													
445 D892         Agliated Metal/SS         Non-metal Refails         60         20         C         0.35 0         0           473 496         D892         As received Metal/SS         0         20         C         0.35 0         0           614 663         780         0         20         0.35 0         0         0           780         D892         As received Metal/SS         0         0         DG(0.01)         -3.13 0         0           902         912         0         0         DG(0.01)         -3.13 0         0         0           962 963         994         Agliated Metal/SS         0         0         DG(0.01)         -3.13 0         0         0           1011 B92         Agitated Metal/SS         20         0         DG(0.01)         -3.13 0         0         0           1150         18506247         As received Metal/SS         10         20         0         0         DG(0.01)         -3.13 0         0         0           1161         1277         0         0         DG(0.01)         -3.13 0         0         0         0         0         0         0         0         0         0         0         0	360	ISO6247	As received	Non-metal	0			10		-1.39	0		
451 D892 As received Metal/SS													
## 1													
A96   B892   As received   Metal/SS   O		D892	As received	Non-metal	60				C		0		
Control   Cont		D803	As received	Motal/SS	0						0		
Fig. 2   F		D092	As received	Wetai/33									
Table   Tabl													
902   912   982   983   994   1011   D892   Agitated   Metal/SS   20   0   0   DG(0.01)   -3.13   0													
962   963   994   1011   D892   Agitated   Metal/SS   20	862	D892	As received	Metal/SS	0			0	DG(0.01)	-3.13	0		
962 963 994 1011 D892	902												
993 994 1011 D892													
994													
1011   D892   Agitated   Metal/SS   10													
1146   ISO6247   As received   Metal/SS   10		D803	Agitatod	Motal/SS					DG(0.01)				
1150 1161 1272 1297 1326									DG(0.01)				
1161   1272		1000247	A3 ICCCIVCU	Wictal/OO									
1272   1297													
1326   1327   1328   1327   1328   1328   1328   1329   1320													
1327   D892   As received   Non-metal   130   20   0.35   20   G(0.01)													
1417   D892   As received   Metal/SS   220													
1435     ISO6247     Agitated     120     —     10     -1.39     0     —       1456     —     —     —     —     —     —     —       1680     —     —     —     —     —     —       1748     —     —     —     —     —     —       1797     —     —     —     —     —     —       1874     —     —     —     —     —     —       1941     ISO6247     As received     Metal/SS     130     —     20     0.35     0     —       1955     D892     As received     Non-metal     10     —     10     -1.39     10     —       1981     D892     Metal/SS     W     —     20     0.35     0     —       6016     —     —     —     —     —     —     —     —       6032     D892     Agitated     Metal/SS     10     —     20     0.35     0     —       6074     D892     Agitated     Non-metal     O     —     20     0.35     0     —       normality     n     16     15     16     16       outliers											_	G(0.01)	
1456 1660 1682 1748 1797 1874 1941 ISO6247 As received Metal/SS 1955 1955 1957 D892 As received Non-metal 10 10 10 1.39 10 10 1981 D892 Metal/SS 10 20 0.35 0 10 10 10 10 10 10 10 10 10 10 10 10 1				Metal/SS									
1660		1300247	Agitateu								U		
1682													
1797													
1874	1748												
1941       ISO6247       As received       Metal/SS       130        20       0.35       0         1957       D892       As received       Non-metal       10        10       -1.39       10	1797												
1955													
1957 D892 As received Non-metal         10         10         -1.39         10         139         10		ISO6247	As received	Metal/SS						0.35	0		
1981 D892       Metal/SS       W       20       0.35       0		D803	As received	Non motal						1 30	10		
6016 6032 D892 6068 ISO6247 Agitated Metal/SS 6074 D892 Agitated Non-metal 6081   normality n outliers mean (n) st.dev. (n) R(calc.)  6032 D892 100 200 0.35 0 20 0.35 10 20 0.35 0 20 0.35 0 20 0.35 0 20 0.35 0 20 0.35 0 30 0.35 0 40 0.35 0 30 0.35 0 40 0.35 0 30 0.35 0 40 0.35 0			As received			\\/							
6032       D892       10       20       0.35       0       20       0.35       10       20       0.35       10       20       0.35       10       20       0.35       10       20       0.35       0       20       0.35       0       20       0.35       0       20       0.35       0       20       0.35       0       20       0.35       0       20       0.35       0       20       0.35       0       20       0.35       0       20       0.35       0       20       0.35       0       20       0.35       0       20       0.35       0       20       0.35       0       20       0.35       0       20       0.35       0       20       0.35       0       20       0.35       0       20       0.35       0       0       20       0.35       0       0       0       0       0       0       0       0       0       0       0       0       0       1       0       0       0       0       0       0       0       0       0       0       0       0 </td <td></td> <td>D002</td> <td></td> <td>Wictairee</td> <td></td> <td>**</td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td> <td></td>		D002		Wictairee		**							
6074 D892 Agitated Non-metal 25 C 20 0.35 0		D892			10			20		0.35	0		
6081	6068	ISO6247	Agitated	Metal/SS	10					0.35	10		
normality     suspect     OK     not OK       n     16     15     16       outliers     0     2     1       mean (n)     58.438     18.000     1.875       st.dev. (n)     71.3143     4.1404     4.0311       R(calc.)     199.680     11.593     11.287		D892	Agitated	Non-metal	25	С		20		0.35	0		
n     16     15     16       outliers     0     2     1       mean (n)     58.438     18.000     1.875       st.dev. (n)     71.3143     4.1404     4.0311       R(calc.)     199.680     11.593     11.287	6081												
n     16     15     16       outliers     0     2     1       mean (n)     58.438     18.000     1.875       st.dev. (n)     71.3143     4.1404     4.0311       R(calc.)     199.680     11.593     11.287								OK			4 01/		
outliers     0     2     1       mean (n)     58.438     18.000     1.875       st.dev. (n)     71.3143     4.1404     4.0311       R(calc.)     199.680     11.593     11.287		•											
mean (n)     58.438     18.000     1.875       st.dev. (n)     71.3143     4.1404     4.0311       R(calc.)     199.680     11.593     11.287					-								
st.dev. (n) 71.3143 4.1404 4.0311 R(calc.) 199.680 11.593 11.287													
R(calc.) 199.680 11.593 11.287													
R(D892:13e1)   (32.897)   16.120   (0.825)		R(calc.)									11.287		
		R(D892:13	Be1)		(32.897)			16.120			(0.825)		

Lab 451 first reported: 60 Lab 1981 first reported: 250 Lab 6074 first reported: 250

Lab 1327 possibly a false positive test result for sequence III?



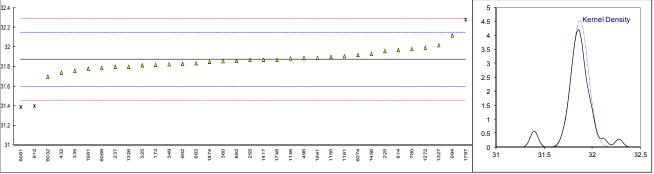
Determination of Foaming Characteristics, Foaming Stability (at end of 10 min settling period) on sample #16240; results in ml

lab	method	Sample	Diffuser	Seq. I	mark	z(targ)	Seq. II	mark	z(targ)	Seq. III	mark	z(targ)
173	memou	Sample	Dillusei		IIIai K	Z(tary)	3eq. II	IIIain	2(tary)	Jeq. III	IIIain	Z(tary)
225												
237												
255												
325	D892	As received	Metal/SS	0			0			0		
335												
349												
360	ISO6247	As received	Non-metal	0			0			0		
432												
445	D892	Agitated	Metal/SS	0			0			0		
451	D892	As received	Non-metal	0			0			0		
473	D000	As resolved	Metal/SS	0			0					
496 614	D892	As received	wetai/55	0			0			0		
663												
780												
862	D892	As received	Metal/SS	0			0			0		
902	2002	, 10 10001100										
912												
962												
963												
994												
1011		Agitated	Metal/SS	0			20	G(0.01)		20	G(0.01)	
1146	ISO6247	As received	Metal/SS	0			0			0		
1150												
1161				0			0			0		
1272 1297												
1326												
1327	D892	As received	Non-metal	0			0			0		
1417	D892	As received	Metal/SS	0			Ö			0		
1435	ISO6247	Agitated		0			Ô			0		
1456		3										
1660												
1682												
1748												
1797												
1874												
1941	ISO6247	As received	Metal/SS	0			0			0		
1955	D000	As received	Non motal									
1957 1981	D892 D892	As received	Non-metal Metal/SS	0	W		0			0		
6016	D092		Wetal/33		VV		0			0		<b></b>
	D892			0			0			0		
6068	ISO6247	Agitated	Metal/SS	0			Ö			0		
6074	D892	Agitated	Non-metal	0			Ô			0		
6081	2002	, igitato a										
	normality			n.a.			n.a.				n.a.	
	n			17			17				17	
	outliers			0			1				1	
	mean (n)			0			0				0	
	st.dev. (n)			0			0				0	
	R(calc.)	201)		0			0				0	
	R(D892:13	oe I)		(0)			(0)			I	(0)	

Lab 1011 possibly a false positive test result for sequences II and III? Lab 1981 first reported: 0  $\,$ 

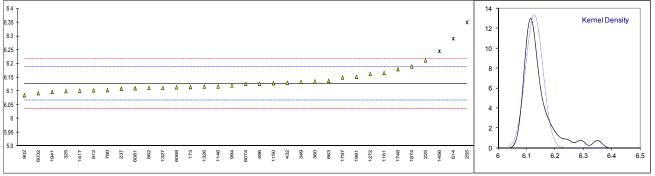
# Determination of Kinematic Viscosity at 40°C on sample #16240; results in mm<sup>2</sup>/s

lab	method	value	mark	z(targ)	remarks			
173	D445	31.8186		-0.39				
225	D445	31.96		0.63				
237	D445	31.80		-0.52				
255	D7279corr	31.87		-0.02				
325	D445	31.815		-0.41				
335	D445	31.761		-0.80				
349	D445	31.825		-0.34				
360	ISO3104	31.857		-0.11				
432	D445	31.74		-0.95				
445								
451								
473								
496	D445	31.888		0.11				
614	D445	31.97		0.70				
663	D445	31.836		-0.26				
780	D445	31.98		0.78				
862	D445	31.86		-0.09				
902	D445	31.83		-0.30				
912	D445	31.40	R(0.01)	-3.40				
962			(,					
963								
994	D445	32.12		1.78				
1011	2							
1146	D445	31.885		0.09				
1150	ISO3104	31.8990		0.19				
1161	ISO3104	31.908		0.26				
1272	ISO3104	31.995		0.88				
1297	1000104							
1326	D445	31.80		-0.52				
1327	D445	32.02		1.06				
1417	D445	31.87		-0.02				
1435	D-1-10							
1456	D445	31.93		0.42				
1660	5110							
1682								
1748	D445	31.87		-0.02				
1797	ISO3104	32.28	R(0.01)	2.94				
1874	D445	31.85	11(0.01)	-0.16				
1941	ISO3104	31.89		0.13				
1955	1000104							
1957								
1981	D445	31.78		-0.66				
6016	D440							
6032	D7279	31.70		-1.24				
6068	ISO3104	31.79		-0.59				
6074	D445	31.92		0.34				
6081	D445	31.39	C,R(0.01)	-3.47	first reported: 31.384696			
0001	D440	01.00	0,11(0.01)	-0.47	mat reported. 31.304030			
	normality	OK						
	n	31						
	outliers	3						
	mean (n)	ა 31.8722						
		0.08815						
	st.dev. (n)							
	R(calc.) R(D445:15a)	0.2468 0.3888						
	K(D445.158)	0.3000						
32.4 T						5 T	<u> </u>	
								Kernel Density



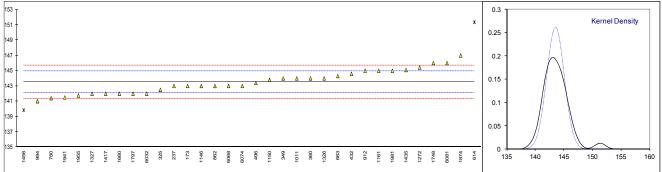
# Determination of Kinematic Viscosity at 100°C on sample #16240; results in mm<sup>2</sup>/s

lab	method	value	mark	z(targ)	remarks
173	D445	6.1147	IIIQIK	-0.44	Tentaino
225	D445	6.212		2.78	
237	D445	6.109		-0.63	
255	D7279corr	6.35	R(0.01)	7.35	
325	D445	6.100	K(0.01)		
335	D443			-0.93 	
	DAAE	 6 1225			
349	D445	6.1335		0.18	
360	ISO3104	6.1356		0.25	
432	D445	6.131		0.10	
445					
451					
473	D445	C 4070		0.04	
496	D445	6.1270	D(0.01)	-0.04	
614	D445	6.291	R(0.01)	5.39	
663	D445	6.1374		0.31	
780	D445	6.104		-0.80	
862	D445	6.112		-0.53	
902	D445	6.085		-1.43	
912	D445	6.103		-0.83	
962					
963	D445			0.24	
994	D443	6.121		-0.24	
1011	DAAE	 6 1170		0.26	
1146	D445	6.1173		-0.36	
1150	ISO3104	6.1287	0	0.02	first reported 6 007
1161	ISO3104	6.166	С	1.25	first reported: 6.237
1272	ISO3104	6.1627		1.15 	
1297	DAAE	 C 117			
1326 1327	D445	6.117		-0.37	
	D445	6.112		-0.53	
1417 1435	D445	6.102		-0.86 	
1456	D445	6.245	R(0.05)	3.87	
1660	D443	0.243	11(0.00)		
1682					
1748	D445	6.18	С	1.72	first reported: 6.222
1797	ISO3104	6.150	O	0.73	ilist reported. 0.222
1874	D445	6.19		2.05	
1941	ISO3104	6.097		-1.03	
1955	1000104			-1.00	
1957					
1981	D445	6.152		0.79	
6016	D443	0.132			
6032	D7279	6.0925		-1.18	
6068	ISO3104	6.114		-0.47	
6074	D445	6.126		-0.07	
6081	D445	6.11067		-0.58	
0001	D-1-10	0.11007		0.00	
	normality	not OK			
	n	30			
	outliers	3			
	mean (n)	6.1281			
	st.dev. (n)	0.02998			
	R(calc.)	0.0839			
	R(D445:15a)	0.0846			
	, ,				



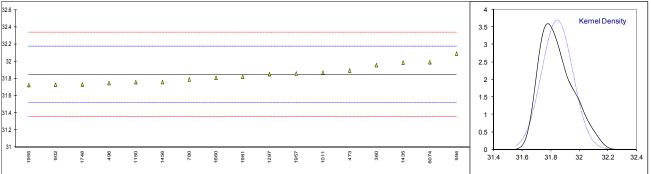
# Determination of Viscosity Index on sample #16240; unit less results

lab         method         value         mark         z(targ)         remarks           173         D2270         143         -0.73           225             237         D2270         143         -0.73           255             325         D2270         142.5         -1.43           335             349         D2270         144         0.67           360         ISO2909         144.0         0.67           432         D2270         144.6         1.51           445             473             496         D2270         143.4         -0.17           614         D2270         151.352         R(0.01)         10.96           663         D2270         144.3         1.09	
225        237     D2270     143     -0.73       255         325     D2270     142.5     -1.43       335         349     D2270     144     0.67       360     ISO2909     144.0     0.67       432     D2270     144.6     1.51       445         451         473         496     D2270     143.4     -0.17       614     D2270     151.352     R(0.01)     10.96	
237 D2270 143 -0.73 255 325 D2270 142.5 -1.43 335 349 D2270 144 0.67 360 ISO2909 144.0 0.67 432 D2270 144.6 1.51 445 451 473 496 D2270 143.4 -0.17 614 D2270 151.352 R(0.01) 10.96	
255	
325 D2270 142.5 -1.43 335 349 D2270 144 0.67 360 ISO2909 144.0 0.67 432 D2270 144.6 1.51 445 451 473 496 D2270 143.4 -0.17 614 D2270 151.352 R(0.01) 10.96	
335	
349     D2270     144     0.67       360     ISO2909     144.0     0.67       432     D2270     144.6     1.51       445         451         473         496     D2270     143.4     -0.17       614     D2270     151.352     R(0.01)     10.96	
360     ISO2909     144.0     0.67       432     D2270     144.6     1.51       445         451         473         496     D2270     143.4     -0.17       614     D2270     151.352     R(0.01)     10.96	
432     D2270     144.6     1.51       445         451         473         496     D2270     143.4     -0.17       614     D2270     151.352     R(0.01)     10.96	
445        451        473        496     D2270     143.4       614     D2270     151.352     R(0.01)       10.96	
451 473 496 D2270 143.4 -0.17 614 D2270 151.352 R(0.01) 10.96	
473 496 D2270 143.4 -0.17 614 D2270 151.352 R(0.01) 10.96	
496 D2270 143.4 -0.17 614 D2270 151.352 R(0.01) 10.96	
614 D2270 151.352 R(0.01) 10.96	
663 D2270 144.3 1.09	
780 D2270 141.4 -2.97	
862 D2270 143 -0.73	
902	
912 D2270	
962	
963	
994 D2270 141 -3.53	
1011 D2270 144 0.67	
1146 D2270 143 -0.73	
1150 ISO2909 143.8 0.39	
1161 D2270 145 C 2.07 first reported: 149	
1272 ISO2909 145.41 2.64	
1297	
1326 D2270 144 0.67	
1327 D2270 142 -2.13	
1417 D2270 142 -2.13	
1435 D2270 145.09 2.19	
1456 D2270 139.8344066 ex, E -5.16 iis calculated: 150	
1660 D2270 142 -2.13	
1682	
1748 D2270 146 C 3.47 first reported: 148.48	
1797 ISO2909 142 -2.13	
1874 D2270 147 4.87	
1941 ISO2909 141.5 -2.83	
1955 D7042 141.75 -2.48	
1957	
1981 D2270 145 2.07	
6016	
6032 D2270 142 -2.13	
6068 ISO2909 143 -0.73	
6074 D2270 143 -0.73	
6081 D2270 146 3.47	
normality OK	
n 32	
outliers 1+1ex	
mean (n) 143.52	
R(calc.) 4.27	
R(D2270:10) 2.00	



# Determination of Viscosity Stabinger at 40°C on sample #16240; results in mm<sup>2</sup>/s

lab	method	value	mark z(tar	g) remarks
173				
225				
237				<del></del>
255				<del></del>
325				<del></del>
335				
349				
360	D7042	31.956	0.6	
432	2. v. <u>-</u>			
445				
451				
473	D7042	31.894	0.3	
496	D7042	31.7465	-0.6	
614	D1042			
663				
780	D7042	31.79	-0.3	
862	D7042		-0.0	
902	D7042	31.73	-0.7	
912	D1072	31.73	-0.7	
962				
963				
903	D7042	32.09	1.4	
1011	D7042	31.87	0.1 	
1146	D7040	 04 7504		
1150	D7042	31.7581	-0.5	
1161				
1272	D7040			
1297	D7042	31.851	0.0	
1326				
1327				
1417	D 70.40			
1435	D7042	31.987	0.8	
1456	D7042	31.7585	-0.5	
1660	D7042	31.81	-0.2	
1682	5-0.0			
1748	D7042	31.732	-0.6	
1797				
1874				
1941				
1955	D7042	31.7235	-0.7	
1957	D7042	31.859	0.0	
1981	D7042	31.82	-0.1	
6016				
6032				<del></del>
6068				
6074	D7042	31.99	3.0	38
6081				<del></del>
	normality	OK		
	n	17		
	outliers	0		
	mean (n)	31.8450		
	st.dev. (n)	0.10816		
	R(calc.)	0.3028		
	R(D7042:16e3)	0.4614		
32.6 T				4
32.0				4 Kernel Density
32.4 -				3.5 1

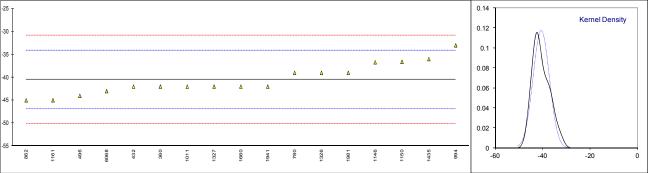


# Determination of Viscosity Stabinger at 100°C on sample #16240; results in mm<sup>2</sup>/s

173	lab	method	value	mark z(targ	)	remar	ks						
237 255 325 325 325 326 338 389 389 389 389 389 389 389 389 389	173												
255					-								
325	237				-								
336					-								
349 D7042 6.1580 0.95 432	325 335				-								
360 D7042 6.1580 0.95 432 445 451 D7042 6.1333 0.17 438 D7042 6.1269 -0.04 68  780 D7042 6.101 -0.86 862  962 D7042 6.073 -1.75 912 962 963  964 D7042 6.140 0.38 1186 D7042 6.140 0.38 1186 D7042 6.117 -0.52 1181  1227 D7042 6.125 -0.08 1327 1327 D7042 6.1648 1.17 1435 D7042 6.1648 1.17 1455 D7042 6.1648 1.17 1680 D7042 6.174 1.46 1680 D7042 6.174 1.55 1787 1787 D7042 6.174 1.55 1789 D7042 6.177 1.55 1789 D7042 6.126 -0.11 1891 D7042 6.140 0.01 1895 D7042 6.1648 1.17 1895 D7042 6.1648 1.17 1895 D7042 6.1648 1.17 1895 D7042 6.141 0.41 1896 D7042 6.126 0.01 1898 D7042 0.08890					_								
432 445 451 461 473 D7042 6.1333 0.17 486 D7042 6.1269 0.04 683 D7042 6.101 0.88 682 D7042 6.101 0.88 682 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.0 0.		D7042	6 1580										
445 473 D7042 6.1333 0.17 486 D7042 6.1269 0.004 614	432	D1042											
451	445												
473 D7042 6.1333 0.17 486 D7042 6.1269 -0.04 614					-								
614 683 780 D7042 6.101 -0.86 862 902 D7042 6.073 -1.75 912	473	D7042	6.1333	0.1	7								
683 780 D7042 6.101 -0.88 862		D7042	6.1269	-0.04	4								
780 D7042 6.101 -0.86 862													
862		D7040											
902 D7042 6.073 -1.75 912	780	D7042											
962	002	D7042											
962 963 994 D7042 6.125 -0.10 1011 D7042 6.140 0.38 1146		D7042											
963 994 D7042 6.125 -0.10 1011 D7042 6.140 0.38 1146	962												
994 D7042 6.125 -0.10 1011 D7042 6.140 0.38 1146													
1011 D7042 6.140 0.38 1146	994	D7042											
1146													
1150 D7042 6.1117 -0.52 1161					-								
1272	1150	D7042		-0.5	2								
1297 D7042 6.1257 -0.08 1326	1161				-								
1326 1327 1417	1272												
1327	1297	D7042											
1417					-								
1435 D7042 6.174 1.46 1456 D7042 6.1648 1.17 1660 D7042 6.09 -1.21 1682	1327				-								
1456 D7042 6.1648 1.17 1660 D7042 6.09 -1.21 1682 1748 D7042 6.177 1.55 1797 1874 1955 D7042 6.0754 -1.67 1957 D7042 6.1246 -0.11 1981 6.141 0.41 6016 6032 6068 6074 D7042 6.136 0.25 6074 D7042 6.136 0.25 6081  normality OK n		D7042											
1660 D7042 6.09 -1.21 1882 1748 D7042 6.177 1.55 1797 1874 1941 1955 D7042 6.0754 -1.67 1957 D7042 6.1246 -0.11 1981 6.141 0.41 6016 6032 6032 6074 D7042 6.136 0.25 6068 6074 D7042 6.136 0.25 6081  normality OK n 17 outliers 0 mean (n) 6.12808 st.dev. (n) 0.031058 R(calc.) 0.08696 R(D7042:16e3) 0.08820	1456												
1682	1660												
1748 D7042 6.177 1.55 1797	1682												
1797 1874 1941 1955 D7042 6.0754 -1.67 1957 D7042 6.1246 -0.11 1961 6.141 0.41 6016 6032 6068 6074 D7042 6.136 0.25 6081   normality OK n		D7042	6.177	1.5	5								
1941	1797												
1955 D7042 6.0754 -1.67 1957 D7042 6.1246 -0.11 1981 6.141 0.41 6016 6032 6088 6074 D7042 6.136 0.25 6081  normality OK n 17 outliers 0 mean (n) 6.12808 st.dev. (n) 0.031058 R(calc.) 0.08696 R(D7042:16e3) 0.08820													
1957 D7042 6.1246 -0.11 1981 6.141 0.41 6016 6032 6074 D7042 6.136 0.25 6081  normality OK n 17 outliers 0 mean (n) 6.12808 st.dev. (n) 0.031058 R(calc.) 0.08696 R(D7042:16e3) 0.08820	1941												
1981 6.141 0.41 6016 6032 6068 6074 D7042 6.136 0.25 6081  normality OK n 17 outliers 0 mean (n) 6.12808 st.dev. (n) 0.031058 R(calc.) 0.08696 R(D7042:16e3) 0.08820			6.0754										
6016 6032 6074 D7042 6.136 0.25 6081   normality OK n 17 outliers 0 mean (n) 6.12808 st.dev. (n) 0.031058 R(calc.) 0.08696 R(D7042:16e3) 0.08820	1957	D7042											
6032	6016												
6068 6074 D7042 6.136 0.25 6081													
6074 D7042 6.136 0.25 6081 OK n 17 outliers 0 mean (n) 6.12808 st.dev. (n) 0.031058 R(calc.) 0.08696 R(D7042:16e3) 0.08820	6068												
normality OK n 17 outliers 0 mean (n) 6.12808 st.dev. (n) 0.031058 R(calc.) 0.08696 R(D7042:16e3) 0.08820		D7042											
normality OK n 17 outliers 0 mean (n) 6.12808 st.dev. (n) 0.031058 R(calc.) 0.08696 R(D7042:16e3) 0.08820													
n 17 outliers 0 mean (n) 6.12808 st.dev. (n) 0.031058 R(calc.) 0.08696 R(D7042:16e3) 0.08820													
outliers 0 mean (n) 6.12808 st.dev. (n) 0.031058 R(calc.) 0.08696 R(D7042:16e3) 0.08820		normality											
mean (n) 6.12808 st.dev. (n) 0.031058 R(calc.) 0.08696 R(D7042:16e3) 0.08820													
st.dev. (n) 0.031058 R(calc.) 0.08696 R(D7042:16e3) 0.08820													
R(calc.) 0.08696 R(D7042:16e3) 0.08820		mean (n)											
R(D7042:16e3) 0.08820													
6.25 6.2 6.15 6.1 6.1 6.1 6.1 6.1 6.1 6.1 6.1 6.1 6.1		R(Calc.)											
62 12 10 . 6.15		. (01072.1063)	0.00020										
6.15 6.15 6.17 6.19 6.19 6.19 6.19 6.19 6.19 6.19 6.19	6.25 T											14	
6.15 - 6.15 - 6.16 - 6.												12	Kernel Density
6.15 6.1	6.2 -											12	// \
8.15 6.1									Α.	Δ	Δ	10 -	// \
	6.15 -					A	Δ	Δ	_			۾ ا	// \
			Δ Δ	Δ Δ Δ	Δ						_		// \
	6.1 -		Δ									6 -	/ \
8.05												4 -	/ \
	6.05 -												// \\
6 8 9 9 9 9 9 1 1 1 1 1 1 1 1 1 1 1 1 1 1												2 :	// \
X X X X X X X X X X X X X X X X X X X		iδ δ ο	8 7 4	2 9 %	.4	-	E	9	9	ίδ	go.	0	
	8	166 166	116 99 99	125 4 4 74	607	101	198	88	4	143	174	6	6 6.05 6.1 6.15 6.2 6.25

# Determination of Pour Point, manual on sample #16240; results in °C

lab	method	value	mark	z(targ)	remarks
173					
225					
237	D97	<-24			
255					
325					
335					
349					
360	D97	-42		-0.47	
432	D97	-42		-0.47	
445					
451					
473					
496	D97	-44		-1.09	
614					
663	D97	<-39			
780	D97	-39		0.46	
862	D97	-45		-1.40	
902					
912					
962					
963	D07			2.22	
994	D97	-33		2.33	
1011	D97 ISO3016	-42 -36.7		-0.47 1.18	
1146 1150	ISO3016	-36.6		1.16	
1161	D97	-30.0 -45		-1.40	
1272	Dai	-45		-1.40	
1297					
1326	D97	-39		0.46	
1327	D97	-42		-0.47	
1417					
1435	ISO3016	-36		1.40	
1456					
1660	D97	-42		-0.47	
1682					
1748					
1797					
1874	1000010				
1941	ISO3016	-42		-0.47	
1955					
1957	D07			0.40	
1981 6016	D97	-39 		0.46	
6032					
6068	ISO3016	-43		-0.78	
6074	1303010			-0.76	
6081					
0001					
	normality	OK			
	n	17			
	outliers	0			
	mean (n)	-40.49			
	st.dev. (n)	3.404			
	R(calc.)	9.53			
	R(D97:16)	9.00			
-25 <sub>T</sub>					0.14
-23 T					V. 14 Kernel Density

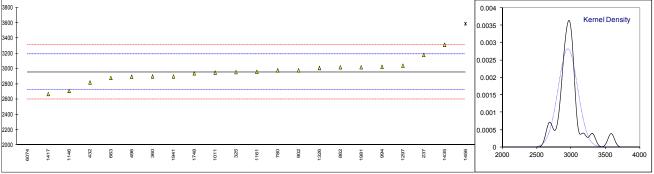


# Determination of Pour Point, automated, 1°C interval on sample #16240; results in °C

lab	method	value	mark	z(targ)	remarks		
173			HIMIN		. 511101110		
225							
237							
255							
325	D5950	-44		0.34			
335 349							
349 360							
432	D5950	-44		0.34			
445	20000						
451							
473							
496		-44.5		0.03			
614							
663 780							
862	D5950	-48		-2.15			
902	20000						
912							
962							
963							
994	D6892	 -42		1.59			
1011 1146	D0092	-4Z 		1.59			
1150							
1161	D6749	-44		0.34			
1272							
1297	D5950	-43.0		0.96			
1326							
1327 1417	D5950	 -42		1.59			
1435	D3930	-42		1.59			
1456							
1660							
1682							
1748	D5950	-48		-2.15			
1797							
1874 1941							
1955							
1957							
1981							
6016							
6032							
6068 6074	D5949	 -46		-0.90			
6081	D3949	-40		-0.90			
0001							
	normality	OK					
	n	10					
	outliers	0					
	mean (n)	-44.55 2.166					
	st.dev. (n) R(calc.)	6.06					
	R(D5950:14)	4.50					
-30 T	, /	•					0.2
00							0.18 - /h Kernel Density
-35 +							0.16 -
[ ]							0.14 -
-40							0.12
1 -						Δ Δ	0.1 -
-45		Δ	Δ	Δ	Δ		0.08 -
		Δ					0.06 -
-50 -	Δ						0.04 -
							0.02 -
-55		* **	10	N			
862	1748	496	325	432	1161	1101	-60 -40 -20 0
1							

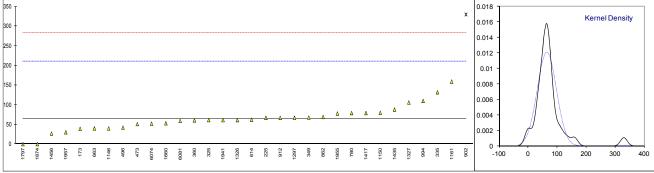
# Determination of Sulphur on sample #16240; results in mg/kg

lab	method	value	mark	z(targ)	remarks	
173						
225						
237	D4294	3180		1.88		
255	DE405			0.04		
325 335	D5185corr.	2956 		-0.01 		
349						
360	ISO8754	2900		-0.49		
432	D4951	2820		-1.16		
445	D 1001					
451						
473						
496	D2622	2896		-0.52		
614						
663	D5453	2880		-0.65		
780	D4294	2980		0.19		
862	D2622	3020		0.53		
902	D4294	2980		0.19		
912 962						
962						
994	D4294	3024		0.56		
1011	D6481	2950		-0.06		
1146	D4294	2710		-2.09		
1150						
1161	ISO8754	2960		0.02		
1272						
1297	D4294	3040		0.70		
1326	D4294	3010		0.44		
1327						
1417	IP336	2669.5		-2.43		
1435	D5185	3314	D(0.01)	3.01		
1456 1660	D5185	3589.5	R(0.01)	5.34		
1682						
1748	D6481	2940		-0.15		
1797	20101					
1874						
1941	ISO8754	2900	С	-0.49	first reported: 2465	
1955						
1957						
1981	D4294	3020		0.53		
6016						
6032						
6068	Dacaa	4000	C D(0.04)	10.54	first remarks do 0.4 mar/les	
6074	D2622	1000	C,R(0.01)	-16.54 	first reported: 0.1 mg/kg	
6081						
	normality	suspect				
	n	20				
	outliers	2				
	mean (n)	_ 2957.48				
	st.dev. (n)	141.289				
	R(calc.)	395.61				
	R(D4294:16e1)	331.32				
3800 T						0.004



# Determination of Water Content by KF on sample #16240; results in mg/kg

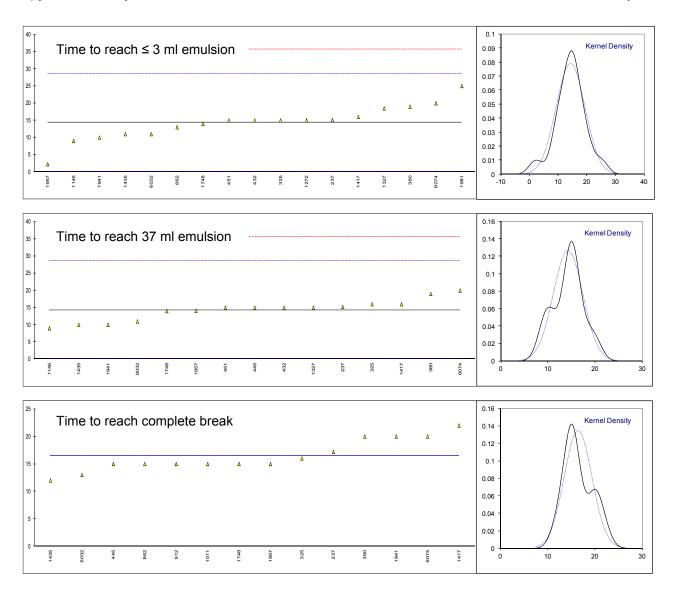
lab	method	value	mark	z(targ)	remarks
173	D6304-C	39		-0.34	
225	D6304-A	66.84		0.04	
237					
255					
325	D6304-C	61		-0.04	
335	D6304-A	132		0.93	
349	D6304-A	67.5		0.05	
360	D6304-A	60.5		-0.05	
432					
445					
451					
473	D6304-C	51		-0.18	
496	D6304-C	42		-0.30	
614	D6304-C	62.3		-0.02	
663	D6304-C	40		-0.33	
780	D6304-A	79		0.20	
862	D6304-C	69		0.07	
902	D6304-A	330.0	C,R(0.01)	3.63	first reported: 360.91
912	D6304-C	67		0.04	
962					
963	D0004 4				
994	D6304-A	110		0.63	
1011	D0004.0				
1146	D6304-C	40		-0.33	
1150	ISO12937	80		0.22	
1161	D6304-A	159.334		1.30	
1272	D6204 A	 67.1		0.04	
1297	D6304-A	67.1		0.04	
1326 1327	D6304-A D6304-C	61.1		-0.04 0.57	
	D6304-C D6304-A	106		0.57 0.20	
1417 1435	D0304-A D1744	79 88		0.20	
1456	D6304-A	26.74		-0.51	
1660	IEC60814	53		-0.15	
1682	ILC00014			-0.13	
1748					
1797	D95	0		-0.88	
1874	E2412	Ö		-0.88	
1941	D6304-C	61		-0.04	
1955	D6304-A	78		0.19	
1957	D6304-C	30		-0.47	
1981	2000.0				
6016					
6032					
6068					
6074	D6304-A	52		-0.17	
6081	D6304-A	59.4800		-0.06	
	normality	suspect			
	n	31			
	outliers	1			
	mean (n)	64.126			
	st.dev. (n)	32.9784			
	R(calc.)	92.340			
	R(D6304:16e1)	205.065			
350 -					0.018



Determination of Water Separability at 54 °C, distilled water on sample #16240; results in min.

lab	method	≤3 ml emul.	mark z(targ)	37 ml water	mark z(targ)	compl. break mark	z(targ)	aborted
173								
225								
237		15.10	0.11	15.23	0.13	17.19		NO
255								
325	D1401	15	0.09	16	0.24	16		NO
335								
349								
360	ISO6614	19	0.65	19	0.66	20		NO
432	D1401	15	0.09		0.10	>30		YES
445	D1401			15	0.10	15		
451		15	0.09	15	0.10			NO
473								
496								
614								
663								
780	D4404					45		\/E0 +\
	D1401	13	-0.19			15		YES *)
902						45		NO
912						15		NO
962								
963								
994	D1401					 4 <i>E</i>		
1011	D1401	0	0.75		0.74	15		NO
1146		9	-0.75 	9	-0.74			NO
1150 1161								
1272	ISO6614	15	0.09					NO
1297	1300014		0.09					140
1326								
	D1401	18.5	0.58	15.0	0.10			YES
	D1401	16	0.23		0.24	22		NO
	ISO6614	11	-0.47	9.97	-0.60	12.00		NO
1456								
1660								
1682								
1748	D1401	14	-0.05	14	-0.04	15		YES *)
1797								-
1874								
1941	D1401	10	-0.61	10	-0.60	20		NO
1955								
	D1401	2.25	-1.69	14.13	-0.02	15		NO
1981		25	1.49					
6016	D4404							
	D1401	11	-0.47	11	-0.46	13		NO
6068	D4404		0.70					NO
	D1401	20	0.79		0.80	20		NO
6081								
normal	lity.	auanaat		ок		OK		
n	iity	suspect 17		15		14		
outliers	2	0		0		0		
mean (		14.34		14.29		16.44		
st.dev.		5.047		3.157		2.957		
R(calc.		14.13		8.84		8.28		
	.) 01:12e1)	20.00		20.00		n.a.		
, 5 1-1	,	_0.00		1 - 2.00		· · · <del>- · ·</del>		1

<sup>\*)</sup> Lab 862 and 1748 reported both that the test had been aborted but also a complete break before 30 min.

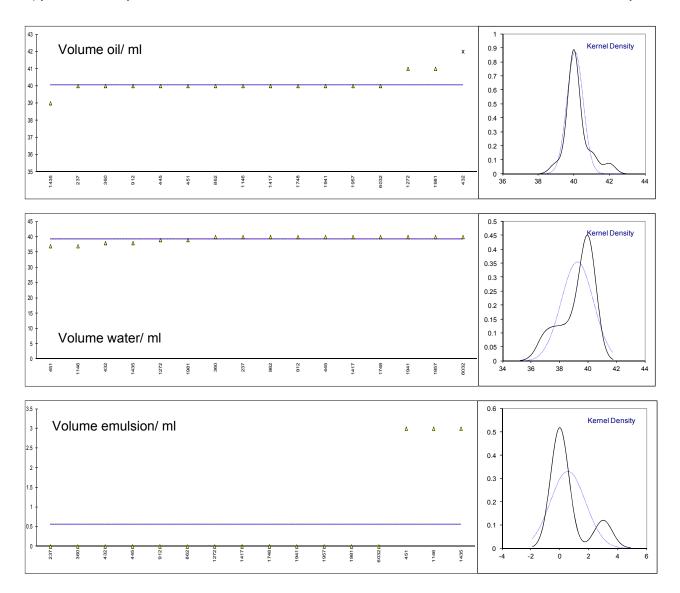


# Determination of Water Separability at 54 °C on sample #16240; results in ml.

### --- Continued ----

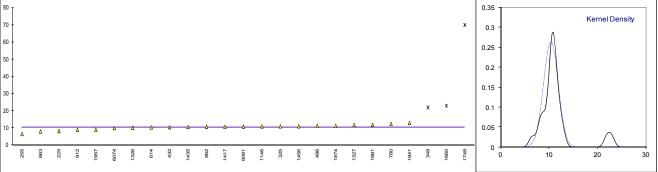
lab	method	oil	mark	z(targ)	water	mark z(targ	g) emulsion	mark z(targ)	aborted
173									
225									
237		40			40		0		NO
255									
325	D1401								NO
335									
349									
360	ISO6614	40			40		0		NO
432	D1401	42	G(0.05)		38		0		YES
445	D1401	40			40		0		
451		40			37		3		NO
473									
496									
614									
663									
780									
862	D1401	40			40		0		YES
902									NO
912		40			40		0		NO
962									
963									
994	D4404								
1011	D1401	40			27				NO
1146		40			37		3		NO
1150 1161									
	ISO6614	41.0			39.0		0.0		NO
1272	1300014	41.0			39.0		0.0		NO
1326									
	D1401								YES
1417		40			40		_		NO
1435	ISO6614	39			38		3		NO
1456	1000011								110
1660									
1682									
1748	D1401	40			40		0		YES
1797									
1874									
1941	D1401	40			40		0		NO
1955									
1957	D1401	40			40		0		NO
1981		41			39		0		
6016									
	D1401	40			40		0		NO
6068									
6074	D1401								NO
6081									
n.c	li4.	not Old			OK		not Old		
norma	шу	not OK			OK		not OK		
n	•	15 1			16		16		
outlier		1			0		0		
mean st.dev	(II) (D)	40.07 0.458			39.25 1.125		0.563 1.2093		
R(calc		1.28			3.15		3.386		
R(targ		n.a.			n.a.		n.a.		
ritary	Ci)	n.a.			11.a.		ı ıı.a.		I

Lab 432 possibly a false positive test result for oil phase?



# Determination of Calcium (Ca) on sample #16240; results in mg/kg.

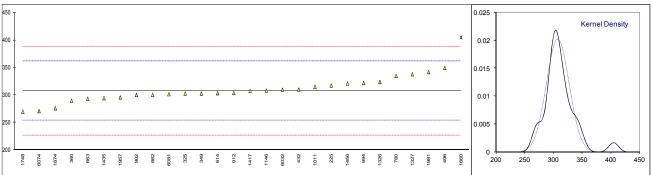
lab	method	value	mark	z(targ)	remarks
173					
225	D6595	8.282			
237					
255	INH-01	6.54			
325	D5185	11			
335					
349	D5185	22	C,R(0.01)		first reported: 35; possibly a false positive test result?
360	D5185	< 40	0,11(0.01)		mot reported. So, possibly a false positive test result.
432	D4951	10.52			
445	D-1001				
451					
473					
	D5185	11 20			
496		11.39			
614	D5185	10.4			
663	D5185	8.06			
780	D5185	12.4			
862	D5185	10.8			
902	D5185	<40			
912	D5185	9			
962					
963					
994	D5185	<40			
1011					
1146	In house	10.99			
1150					
1161					
1272					
1297					
1326		10.2			
1327	D5185	11.8			
1417	D5185	10.8			
1435	D5185	10.66			
1456	D5185	11.0			
1660	D4951	23	R(0.01)		possibly a false positive test result?
1682					
1748	D6481	70	C,R(0.01)		first reported: 40; possibly a false positive test result?
1797					
1874	D6595	11.40			
1941	D4628	13	С		first reported: 16
1955					
1957	D5185	9			
1981	D4951	12			
6016					
6032					
6068					
6074	D6595	10			
6081		10.85			
	normality	suspect			Application range: 40-9000 mg/kg
	n	22			
	outliers	3			
	mean (n)	10.459			
	st.dev. (n)	1.5093			
	R(calc.)	4.226			
	R(D5185:13e1)	(0.317)			
00	· · · · · · · · · · · · · · · · · · ·				
80 T					0.35



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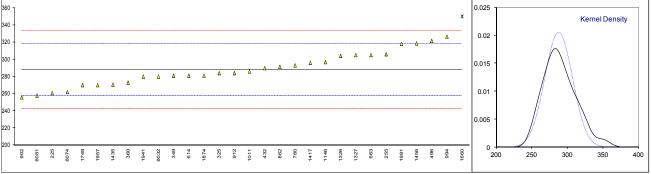
# Determination of Phosphorus (P) on sample #16240; results in mg/kg.

lab	method	value	mark	z(targ)	remarks		
173							
225	D6595	317.70		0.37			
237							
255	D=10=						
325	D5185	303		-0.18			
335	DE10E	202		0.10			
349 360	D5185 D5185	303 290		-0.18 -0.66			
432	D4951	310.1		0.09			
445	D 1001						
451							
473							
496	D5185	349.55		1.55			
614	D5185	303.6		-0.15			
663	D5185	293.67		-0.52			
780	D5185	335		1.01			
862	D5185	300.5		-0.27			
902 912	D5185 D5185	300.31 304		-0.28			
962	D3163	30 <del>4</del> 		-0.14 			
963							
994	D5185	322.0		0.53			
1011	D5185	315		0.27			
1146	In house	308.3		0.02			
1150							
1161							
1272							
1297		204		0.00			
1326 1327	D5185	324 338		0.60 1.12			
1417	D5185	308		0.01			
1435	D5185	294.6		-0.49			
1456	D5185	321.25		0.50			
1660	D4951	405	R(0.01)	3.61			
1682							
1748	D6481	270		-1.40			
1797	D						
1874	D6595	276.10		-1.17			
1941 1955							
1955	D5185	296		-0.44			
1981	D4951	342		1.27			
6016	D 1001						
6032	D6595	310		0.08			
6068							
6074	D6595	271		-1.36			
6081		301.8		-0.22			
		OK					
	normality	OK					
	n outliers	27 1					
	mean (n)	307.721					
	st.dev. (n)	19.8176					
	R(calc.)	55.489					
	R(D5185:13e1)	75.431					
450 T	<u> </u>					0.025 1	



# Determination of Zinc (Zn) on sample #16240; results in mg/kg.

lab	method	value	mark	z(targ)	remarks
173					
225	D5185	260.72		-1.81	
237					
255	INH-01	305.97		1.20	
325	D5185	284		-0.26	
335	DE10E	 281		-0.46	
349 360	D5185 D5185	273		-0.46	
432	D4951	289.8		0.13	
445	D 1001				
451					
473					
496	D5185	321.60		2.24	
614	D5185	281.1		-0.45	
663	D5185	305.04		1.14	
780	D5185	293		0.34	
862	D5185	291.4		0.23	
902	D5185	255.8		-2.13	
912 962	D5185	284		-0.26 	
963					
994	D5185	326.4		2.56	
1011	D5185	286		-0.13	
1146	In house	296.8		0.59	
1150					
1161					
1272					
1297				4.07	
1326 1327	DE10E	304		1.07 1.14	
1417	D5185 D5185	305 296		0.54	
1435	D5185	270.6		-1.15	
1456	D5185	318.625		2.04	
1660	D4951	350	R(0.05)	4.13	
1682			` ,		
1748 1797	D6481	270		-1.19 	
1874	D6595	281.10		-0.45	
1941	D4828	280	С	-0.52	first reported: 220
1955					
1957	D5185	270		-1.19	
1981	D4951	318		2.00	
6016					
6032	D6595	280		-0.52	
6068	D6595	262		 -1.72	
6074 6081	D0090	262 257.8		-2.00	
0001		207.0		-2.00	
	normality	OK			
	n	29			
	outliers	1			
	mean (n)	287.888			
	st.dev. (n)	19.4878			
	R(calc.)	54.566			
	R(D5185:13e1)	42.094			
					0.025



### **APPENDIX 2**

### Number of participants per country

- 1 lab in ALGERIA
- 2 labs in AUSTRALIA
- 1 lab in AUSTRIA
- 1 lab in AZERBAIJAN
- 2 labs in BELGIUM
- 2 labs in BULGARIA
- 3 labs in CHINA, People's Republic
- 1 lab in COTE D'IVOIRE
- 1 lab in FRANCE
- 1 lab in GERMANY
- 1 lab in GREECE
- 1 lab in INDIA
- 1 lab in ISRAEL
- 1 lab in ITALY
- 1 lab in JORDAN
- 1 lab in KAZAKHSTAN
- 2 labs in MALAYSIA
- 1 lab in MEXICO
- 1 lab in NETHERLANDS
- 1 lab in NIGERIA
- 1 lab in NORWAY
- 1 lab in PERU
- 1 lab in POLAND
- 1 lab in PORTUGAL
- 1 lab in ROMANIA
- 1 lab in RUSSIAN FEDERATION
- 2 labs in SAUDI ARABIA
- 2 labs in SERBIA
- 1 lab in SOUTH KOREA
- 1 lab in SPAIN
- 1 lab in TANZANIA
- 1 lab in THAILAND
- 3 labs in TURKEY
- 3 labs in UNITED KINGDOM
- 1 lab in UNITED STATES OF AMERICA

#### **APPENDIX 3**

### Abbreviations:

DG(0.05)

C = final test result after checking of first reported suspect test result

 $\begin{array}{ll} D(0.01) & = \text{outlier in Dixon's outlier test} \\ D(0.05) & = \text{straggler in Dixon's outlier test} \\ G(0.01) & = \text{outlier in Grubbs' outlier test} \\ G(0.05) & = \text{straggler in Grubbs' outlier test} \\ DG(0.01) & = \text{outlier in Double Grubbs' outlier test} \\ \end{array}$ 

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

= straggler in Double Grubbs' outlier test

n.a. = not applicable
n.e. = not evaluated
n.d. = not detected
fr. = first reported

SDS = Safety Data Sheet

### Literature:

- iis Interlaboratory Studies, Protocol for the Organization, Statistics and Evaluation, April 2014
- 2 ASTM E178:16
- 3 ASTM E1301:95(2003)
- 4 ISO 5725:86
- 5 ISO 5725, parts 1-6, 1994
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- 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, <u>331</u>, 513, (1988)
- 12 J.N. Miller, Analyst, <u>118</u>, 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, <u>127</u>, 1359-1364 (2002)
- Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, <u>25(2)</u>, 165-172, (1983)