# Results of Proficiency Test
# Hydraulic Oil (fresh)
# November 2019


Organised by: Institute for Interlaboratory Studies
Spijkenisse, The Netherlands

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# 1 INTRODUCTION

Since 2014 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for Hydraulic Oil (fresh) every year. During the annual proficiency test program of 2019/2020, it was decided to continue the round robin for the analyzes on Hydraulic Oil.

In this interlaboratory study 36 laboratories in 28 different countries registered for participation. See appendix 3 for the number of participants per country. In this report, the results of the 2019 Hydraulic Oil (fresh) proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

# 2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send one sample of 1L labelled #19235.
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/IEC17043:2010 (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 Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through 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

Approximately 70 liters of fresh Hydraulic Oil was obtained from a local supplier. After homogenization 60 amber glass bottles of 1L were filled and labelled #19235. The homogeneity of the subsamples #19235 was checked by determination of Density in accordance with ASTM D4052 and Kinematic Viscosity at 40°C in accordance with ASTM D445 on 8 stratified randomly selected samples.

| | Density at 15°C in kg/L | Kinematic Viscosity at 40°C in $mm^2/s$ |
|---|---|---|
| Sample #19235-1 | 0.85502 | 32.18 |
| Sample #19235-2 | 0.85502 | 32.21 |
| Sample #19235-3 | 0.85502 | 32.21 |
| Sample #19235-4 | 0.85503 | 32.15 |
| Sample #19235-5 | 0.85504 | 32.19 |
| Sample #19235-6 | 0.85504 | 32.17 |
| Sample #19235-7 | 0.85503 | 32.17 |
| Sample #19235-8 | 0.85503 | 32.19 |

Table 1: homogeneity test results of subsamples #19235

From the above test results the repeatabilities were calculated and compared with 0.3 times the corresponding reproducibility of the reference test methods in agreement with the procedure of ISO13528, Annex B2 in the next table.

| | Density at 15°C in kg/L | Kinematic Viscosity at 40°C in $mm^2/s$ |
|---|---|---|
| r (observed) | 0.00002 | 0.06 |
| reference test method | ISO12185:96 | D445:19 |
| 0.3 x R (ref. test method) | 0.00015 | 0.12 |

Table 2: evaluation of the repeatabilities of subsamples #19235

The calculated repeatabilities are in agreement with 0.3 times the corresponding reproducibility of the reference test methods. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one 1L bottle labelled #19235 was sent on October 16, 2019. An SDS was added to the sample package.

## 2.5 STABILITY OF THE SAMPLES

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

## 2.6 ANALYZES

The participants were asked to determine on sample #19235: Acid Number (Total), Copper Corrosion (3hrs at 50°C), Density at 15°C, Flash Point PMcc, Foaming 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), Sulfur, Water, Water Separability at 54°C and Calcium, Phosphorus and Zinc. Also, some additional questions were asked about Acid Number (Total) and Foaming Characteristics.

It was explicitly requested to treat the sample as if it was a routine sample 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’ test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

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 appropriate 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/. 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.

# 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 reanalyzes). Additional or corrected test results are used for data analyzes and the 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 June 2018 (iis-protocol, version 3.5).
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 ISO5725 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. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

Finally, the reproducibilities were calculated from the standard deviations by multiplying these 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. The Kernel Density Graph 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 or ISO reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency tests could be used.
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:

$z_{(target)}$ = (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 no major problems were encountered with the dispatch of the samples. Two participants reported the test results after the final reporting date and one other participant did not report any test results. Not all laboratories were able to report all analyzes requested.
In total 35 participants did report 504 numerical test results. Observed were 23 outlying test results, which is 4.6%. 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, see also paragraph 3.1.

### 4.1 EVALUATION PER TEST

In this section, the reported test results are discussed per test. The test methods, which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the reported test results. The abbreviations, used in these tables, are explained in appendix 4.

In 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.

Unfortunately, a suitable reference 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 estimated from the Horwitz equation.

Acid Number (Total): 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 inflection point at titration volume 60 mL and Buffer End Point at titration volume 60 mL from ASTM D664-A:18e2. However, the calculated reproducibility is not in agreement with the 125 mL requirements.
When evaluated separately for the type of endpoint the calculated reproducibility of the group using Inflection Point (IP) was in agreement with the requirements of inflection point at titration volume 60 mL, but not for 125 mL. The calculated reproducibility of the group using BEP (pH 10 and 11) is in agreement with the requirements of BEP (pH 10) from test method D664-A:18e2 for 60 mL, but not for 125 mL.
It is observed that five participants reported to have used BEP (pH 11) as determination end point and three reported to have used BEP (pH 10). In method ASTM D664-A version 2018e2 the Buffer End Point has been changed to pH 10.

Copper Corrosion: This determination was not problematic. All reporting participants agreed on a test result of 1 (1a, 1b).

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

Flash Point PMcc: This determination was not problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D93-A:19.

Foaming Characteristics (Tendency and Stability): This determination was problematic. In total three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers in the Foam Tendency determination for sequence I is in agreement with the requirements of ASTM D892:18. The calculated reproducibilities for sequence II and III are not in agreement with the requirements of ASTM D892:18. The variation in the test results for sequence III is very large. Therefore, it was decided not to calculate z-scores.
All reporting participants reported 0 mL for Foam Stability.
This determination is very sensitive in maintenance and execution. In ASTM D892:18 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). Almost all participants have given the same answers or did not report this information. Therefore, no conclusions could be drawn.

Kinematic Viscosity at 40°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D445:19.

Kinematic Viscosity at 100°C: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of ASTM D445:19.

Viscosity Index: This determination was problematic. Two statistical outliers were observed and one test result was excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of ASTM D2270:10(2016). Three participants made possibly a calculation error? It was noticed in another proficiency test iis19L03 of Base Oil that a relatively small bias in the K.V. measurements can give a large bias in the V.I. calculations.

Viscosity Stabinger at 40°C: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good 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. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D97:17b.

Pour Point, Automated, 1°C interval: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D5950:14.

Sulfur: 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 D4294:16e1.

Water: 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 54°C: This determination was not problematic. No statistical outliers were observed over six parameters. The calculated reproducibilities for “time to reach ≤ 3mL emulsion” and “time to reach 37mL water” are both in agreement with the requirements of ASTM D1401:18a.

Calcium: 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 estimated reproducibility using the Horwitz equation, but not at all with the strict requirements of ASTM D5185:18.

Phosphorus: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D5185:18.

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 D5185:18.

### 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 number of significant test results, the average result, the calculated reproducibility (2.8 * standard deviation) and the target reproducibilities derived from reference test methods (in casu ASTM test methods) are presented in the next table.

| Parameter | unit | n | average | 2.8 * sd | R(lit) |
|---|---|---|---|---|---|
| Acid Number (Total) | mg KOH/g | 25 | 0.49 | 0.21 | 0.22 |
| Copper Corrosion, 3 hrs at 50°C | | 17 | 1 (1a/1b) | n.a. | n.a. |
| Density at 15°C | kg/L | 28 | 0.8550 | 0.0003 | 0.0005 |
| Flash Point PMcc | °C | 24 | 201.3 | 7.3 | 14.3 |
| Foam Tendency Seq. I | mL | 13 | 15.4 | 14.5 | 20.4 |

| Parameter | unit | n | average | 2.8 * sd | R(lit) |
|---|---|---|---|---|---|
| Foam Tendency Seq. II | mL | 15 | 25.3 | 23.4 | 18.0 |
| Foam Tendency Seq. III | mL | 14 | 17.1 | 25.6 | (7.5) |
| Foam Stability Seq. I | mL | 15 | 0 | n.a. | n.a. |
| Foam Stability Seq. II | mL | 15 | 0 | n.a. | n.a. |
| Foam Stability Seq. III | mL | 15 | 0 | n.a. | n.a. |
| Kinematic Viscosity at 40°C | $mm^2/s$ | 27 | 32.182 | 0.364 | 0.393 |
| Kinematic Viscosity at 100°C | $mm^2/s$ | 23 | 6.340 | 0.068 | 0.088 |
| Viscosity Index | | 24 | 152.6 | 3.6 | 2 |
| Viscosity Stabinger at 40°C | $mm^2/s$ | 13 | 32.199 | 0.291 | 0.465 |
| Viscosity Stabinger at 100°C | $mm^2/s$ | 15 | 6.348 | 0.119 | 0.090 |
| Pour Point, Manual | °C | 13 | -42.5 | 7.0 | 9 |
| Pour Point, Automated, 1°C int. | °C | 11 | -44.7 | 6.0 | 4.5 |
| Sulfur | mg/kg | 19 | 788 | 187 | 141 |
| Water | mg/kg | 22 | 46.9 | 79.8 | 169.9 |
| Water Separability at 54°C, distilled water | | | | | |
| - Time ≤ 3 mL emulsion | minutes | 12 | 6.3 | 9.5 | 20 |
| - Time 37 mL water | minutes | 12 | 6.3 | 9.6 | 20 |
| - Complete Break (40-40-0) | minutes | 15 | 7.4 | 9.2 | n.a. |
| - Volume Oil phase | mL | 9 | 40.1 | 0.9 | n.a. |
| - Volume Water phase | mL | 9 | 39.6 | 2.8 | n.a. |
| - Volume Emulsion phase | mL | 9 | 0.3 | 2.8 | n.a. |
| Calcium as Ca | mg/kg | 26 | 49.6 | 11.9 | 12.3 |
| Phosphorus as P | mg/kg | 29 | 342 | 80 | 80 |
| Zinc as Zn | mg/kg | 29 | 449 | 78 | 69 |

Table 3: performance evaluation sample #19235

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 2019 WITH THE PREVIOUS PTS.

| | November 2019 | November 2018 | November 2017 | November 2016 | November 2015 |
|---|---|---|---|---|---|
| Number of reporting laboratories | 35 | 35 | 45 | 43 | 45 |
| Number of test results reported | 504 | 465 | 610 | 597 | 569 |
| Number of statistical outliers | 23 | 18 | 28 | 30 | 26 |
| Percentage outliers | 4.6% | 3.9% | 4.6% | 5.0% | 4.6% |

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.

| | November 2019 | November 2018 | November 2017 | November 2016 | November 2015 |
|---|---|---|---|---|---|
| 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+II+III | n.e. | 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. | - | + | - | - | + |
| Sulfur | - | + | +/- | - | + |
| Water | ++ | ++ | ++ | ++ | ++ |
| Water Separability ≤ 3mL emul. | ++ | + | + | + | ++ |
| Water Separability 37mL water | ++ | + | + | ++ | ++ |
| Calcium as Ca | +/- | - | + | n.e. | n.e. |
| Phosphorus as P | +/- | + | + | + | + |
| Zinc as Zn | - | n.e. | - | - | n.e. |

Table 5: comparison determinations against the reference test methods

The following performance categories were used:

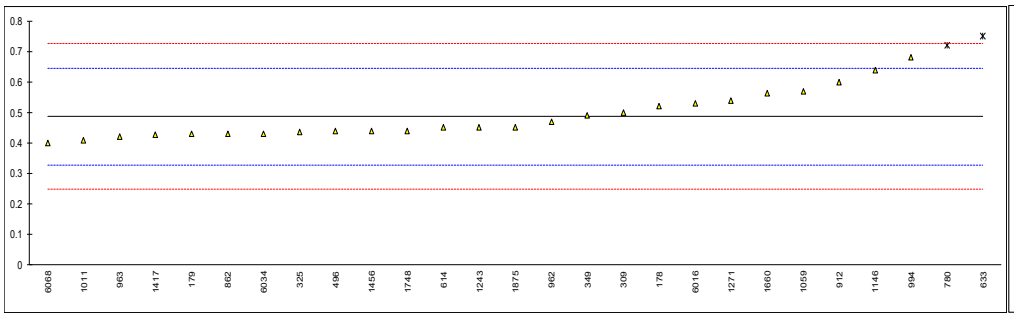
++: group performed much better than the reference test method
+ : group performed better than the reference test method
+/-: group performance equals the reference test method
- : group performed worse than the reference test method
-- : group performed much worse than the reference test method
n.e.: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

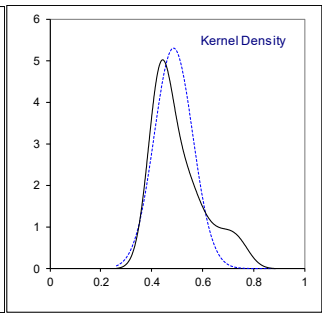
## APPENDIX 1

Determination of Acid Number (Total) on sample #19235; results in mg KOH/g

| lab | method | value | mark | z(targ) | remarks | end point | Volume of titration solvent |
|---|---|---|---|---|---|---|---|
| 178 | D664-A | 0.52 | | 0.43 | | --- | 60 mL |
| 179 | D664-A | 0.43 | | -0.71 | | Inflection Point | 60 mL |
| 219 | | ----- | | ----- | | --- | --- |
| 237 | | ----- | | ----- | | --- | --- |
| 255 | | ----- | | ----- | | --- | --- |
| 257 | | ----- | | ----- | | --- | --- |
| 309 | D664-A | 0.50 | | 0.17 | | Buffer End Point (pH 10) | 60 mL |
| 325 | D664-A | 0.435 | | -0.64 | | Buffer End Point (pH 10) | 125 mL |
| 349 | D664-A | 0.49 | | 0.05 | | Inflection Point | 125 mL |
| 432 | | ----- | | ----- | | --- | --- |
| 496 | D664-A | 0.44 | | -0.58 | | Buffer End Point (pH 10) | 60 mL |
| 614 | D664-A | 0.45 | | -0.45 | | --- | 60 mL |
| 633 | D664-A | 0.75 | G(0.05) | 3.32 | | Inflection Point | 125 mL |
| 780 | D664-A | 0.72 | G(0.05) | 2.94 | | Inflection Point | 60 mL |
| 862 | D664-A | 0.43 | | -0.71 | | Inflection Point | 60 mL |
| 912 | D664-A | 0.6 | | 1.43 | | --- | --- |
| 962 | D974 | 0.47 | | -0.20 | | --- | --- |
| 963 | D664-A | 0.421 | | -0.82 | | Buffer End Point (pH 11) | 60 mL |
| 994 | D664-A | 0.68 | | 2.44 | | Inflection Point | 125 mL |
| 1011 | D664-A | 0.41 | | -0.96 | | --- | --- |
| 1059 | ISO6619 | 0.57 | | 1.05 | | Buffer End Point (pH 11) | 60 mL |
| 1146 | D664-A | 0.639 | | 1.92 | | Buffer End Point (pH 11) | 125 mL |
| 1182 | | ----- | | ----- | | --- | --- |
| 1243 | ISO6618 | 0.45 | | -0.45 | | Inflection Point | 60 mL |
| 1271 | ISO6618 | 0.54 | | 0.68 | | --- | 125 mL |
| 1417 | D664-A | 0.427 | | -0.74 | | Inflection Point | 60 mL |
| 1456 | D974 | 0.44 | | -0.58 | | Buffer End Point (pH 11) | 125 mL |
| 1660 | D664-A | 0.563 | | 0.97 | | Buffer End Point (pH 11) | 60 mL |
| 1720 | | ----- | | ----- | | --- | --- |
| 1748 | D664-A | 0.44 | | -0.58 | | Inflection Point | 125 mL |
| 1875 | ISO6618 | 0.4501 | | -0.45 | | Inflection Point | 60 mL |
| 6016 | D664-A | 0.528 | | 0.53 | | --- | --- |
| 6034 | D664-A | 0.43 | | -0.71 | | --- | --- |
| 6068 | ISO6618 | 0.4 | | -1.08 | | --- | --- |
| 6257 | | ----- | | ----- | | --- | --- |
| 6277 | | ----- | | ----- | | --- | --- |

| | | | BEP (pH 10+pH11) (60 + 125 mL) | Inflection point (60 + 125 mL) |
|---|---|---|---|---|
| | normality | suspect | OK | not OK |
| | n | 25 | 8 | 8 |
| | outliers | 2 | 0 | 2 |
| | mean (n) | 0.4861 | 0.5010 | 0.4746 |
| | st.dev. (n) | 0.07540 | 0.08093 | 0.08542 |
| | R(calc.) | 0.2111 | 0.2266 | 0.2392 |
| | st.dev.(D664-A:18e2, IP 60mL) | 0.07951 | ---- | 0.07797 |
| | R(D664-A:18e2, IP 60mL) | 0.2226 | ---- | 0.2183 |
| Compare | | | | |
| | R(D664-A:18e2, BEP - 60mL) | 0.2742 | 0.2823 | ---- |
| | R(D664-A:18e2, BEP - 125 mL) | 0.1480 | 0.1528 | ---- |
| | R(D664-A:18e2, IP 125mL) | 0.1031 | ---- | 0.1006 |



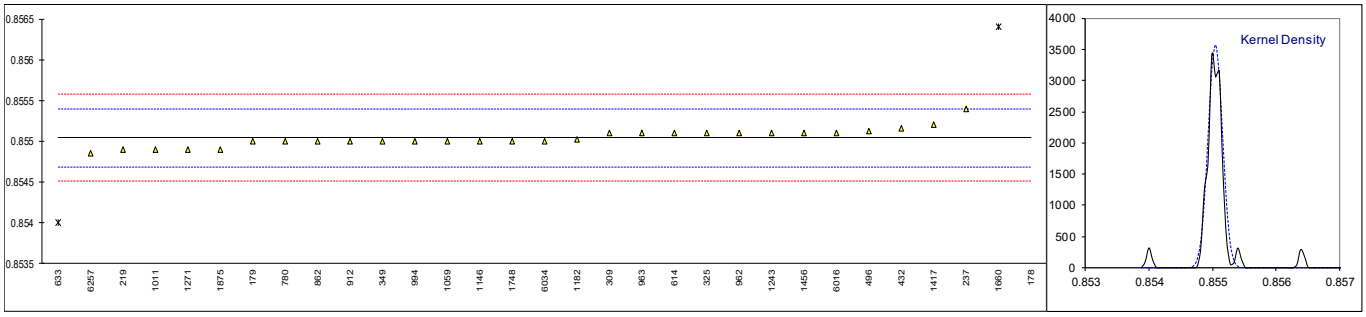
Determination of Copper Corrosion 3hrs at 50°C on sample #19235;

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | | ----- | | ----- | |
| 179 | D130 | 1A | | ----- | |
| 219 | | ----- | | ----- | |
| 237 | D130 | 1A | | ----- | |
| 255 | | ----- | | ----- | |
| 257 | | ----- | | ----- | |
| 309 | | ----- | | ----- | |
| 325 | D130 | 1A | | ----- | |
| 349 | | ----- | | ----- | |
| 432 | | ----- | | ----- | |
| 496 | | ----- | | ----- | |
| 614 | D130 | 1a | | ----- | |
| 633 | D130 | 1a | | ----- | |
| 780 | D130 | 1a | | ----- | |
| 862 | D130 | 1a | | ----- | |
| 912 | | ----- | | ----- | |
| 962 | | ----- | | ----- | |
| 963 | | ----- | | ----- | |
| 994 | D130 | 1a | | ----- | |
| 1011 | D130 | 1a | | ----- | |
| 1059 | ISO2160 | 1a | | ----- | |
| 1146 | | ----- | | ----- | |
| 1182 | D130 | 1 | | ----- | |
| 1243 | ISO2160 | 1a | | ----- | |
| 1271 | D130 | 1a | | ----- | |
| 1417 | IP154 | 1B | | ----- | |
| 1456 | D130 | 1A | | ----- | |
| 1660 | | ----- | | ----- | |
| 1720 | | ----- | | ----- | |
| 1748 | D130 | 1a | | ----- | |
| 1875 | | ----- | | ----- | |
| 6016 | | ----- | | ----- | |
| 6034 | D130 | 1a | | ----- | |
| 6068 | | ----- | | ----- | |
| 6257 | | ----- | | ----- | |
| 6277 | | ----- | | ----- | |
| | n | 17 | | | |
| | mean (n) | 1A / 1B | | | |

## Determination of Density at 15°C on sample #19235; results in kg/L

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D4052 | 0.8686 | R(0.01) | 75.93 | |
| 179 | D4052 | 0.8550 | | -0.23 | |
| 219 | D1298 | 0.8549 | | -0.79 | |
| 237 | D4052 | 0.8554 | C | 2.01 | First reported 0.8697 |
| 255 | | ----- | | ----- | |
| 257 | | ----- | | ----- | |
| 309 | D4052 | 0.85510 | | 0.33 | |
| 325 | D4052 | 0.8551 | | 0.33 | |
| 349 | D4052 | 0.8550 | | -0.23 | |
| 432 | D4052 | 0.85516 | | 0.67 | |
| 496 | ISO12185 | 0.85512 | | 0.44 | |
| 614 | D4052 | 0.8551 | | 0.33 | |
| 633 | D4052 | 0.8540 | R(0.01) | -5.83 | |
| 780 | ISO12185 | 0.855 | | -0.23 | |
| 862 | D4052 | 0.8550 | | -0.23 | |
| 912 | ISO12185 | 0.855 | | -0.23 | |
| 962 | D4052 | 0.8551 | | 0.33 | |
| 963 | D4052 | 0.8551 | | 0.33 | |
| 994 | ISO12185 | 0.8550 | | -0.23 | |
| 1011 | D4052 | 0.8549 | | -0.79 | |
| 1059 | ISO12185 | 0.855 | | -0.23 | |
| 1146 | D4052 | 0.8550 | | -0.23 | |
| 1182 | ISO12185 | 0.855021 | | -0.11 | |
| 1243 | ISO12185 | 0.8551 | | 0.33 | |
| 1271 | D4052 | 0.8549 | | -0.79 | |
| 1417 | IP365 | 0.8552 | | 0.89 | |
| 1456 | D4052 | 0.8551 | | 0.33 | |
| 1660 | D7042 | 0.8564 | R(0.01) | 7.61 | |
| 1720 | | ----- | | ----- | |
| 1748 | D4052 | 0.855 | | -0.23 | |
| 1875 | D7042 | 0.8549 | | -0.79 | |
| 6016 | D4052 | 0.8551 | | 0.33 | |
| 6034 | D4052 | 0.8550 | | -0.23 | |
| 6068 | | ----- | | ----- | |
| 6257 | ISO12185 | 0.85485 | | -1.07 | |
| 6277 | | ----- | | ----- | |

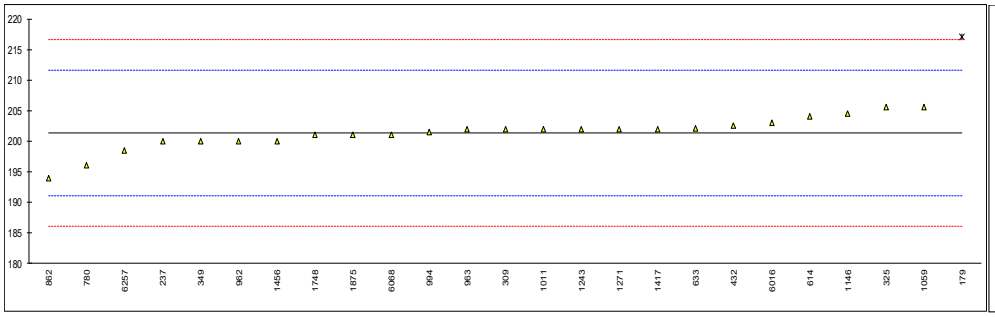
| | |
|---|---|
| normality | not OK |
| n | 28 |
| outliers | 3 |
| mean (n) | 0.85504 |
| st.dev. (n) | 0.000111 |
| R(calc.) | 0.00031 |
| st.dev.(ISO12185:96) | 0.000179 |
| R(ISO12185:96) | 0.0005 |

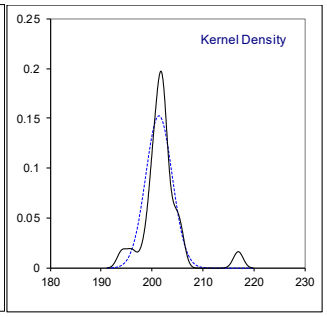


## Determination of Flash Point PMcc on sample #19235; results in °C

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | | ----- | | ----- | |
| 179 | D93-A | 217.0 | R(0.01) | 3.07 | |
| 219 | | ----- | | ----- | |
| 237 | D93-A | 200 | | -0.26 | |
| 255 | | ----- | | ----- | |
| 257 | | ----- | | ----- | |
| 309 | D93-A | 202.0 | | 0.13 | |
| 325 | D93-A | 205.5 | | 0.82 | |
| 349 | D93-A | 200 | | -0.26 | |
| 432 | D93-A | 202.5 | | 0.23 | |
| 496 | | ----- | | ----- | |
| 614 | D93-A | 204 | | 0.52 | |
| 633 | D93-C | 202.1 | | 0.15 | |
| 780 | D93-A | 196.0 | | -1.04 | |
| 862 | D93-A | 194 | | -1.44 | |
| 912 | | ----- | | ----- | |
| 962 | D93-A | 200 | | -0.26 | |
| 963 | D93-A | 202.0 | | 0.13 | |
| 994 | D93-A | 201.5 | | 0.03 | |
| 1011 | D93-A | 202.0 | | 0.13 | |
| 1059 | ISO2719-A | 205.5 | | 0.82 | |
| 1146 | D93-A | 204.5 | | 0.62 | |
| 1182 | | ----- | W | ----- | Test result withdrawn, reported 190.1 |
| 1243 | ISO2719-A | 202 | | 0.13 | |
| 1271 | ISO2719-A | 202 | | 0.13 | |
| 1417 | D93-A | 202 | | 0.13 | |
| 1456 | D93-A | 200.0 | | -0.26 | |
| 1660 | | ----- | | ----- | |
| 1720 | | ----- | | ----- | |
| 1748 | D93-A | 201 | | -0.07 | |
| 1875 | ISO2719-A | 201.0 | | -0.07 | |
| 6016 | D93-A | 203 | | 0.33 | |
| 6034 | | ----- | | ----- | |
| 6068 | ISO2719-A | 201.0 | | -0.07 | |
| 6257 | ISO2719-A | 198.4 | | -0.57 | |
| 6277 | | ----- | | ----- | |

| | |
|---|---|
| normality | not OK |
| n | 24 |
| outliers | 1 |
| mean (n) | 201.33 |
| st.dev. (n) | 2.617 |
| R(calc.) | 7.33 |
| st.dev.(D93-A:19) | 5.105 |
| R(D93-A:19) | 14.29 |



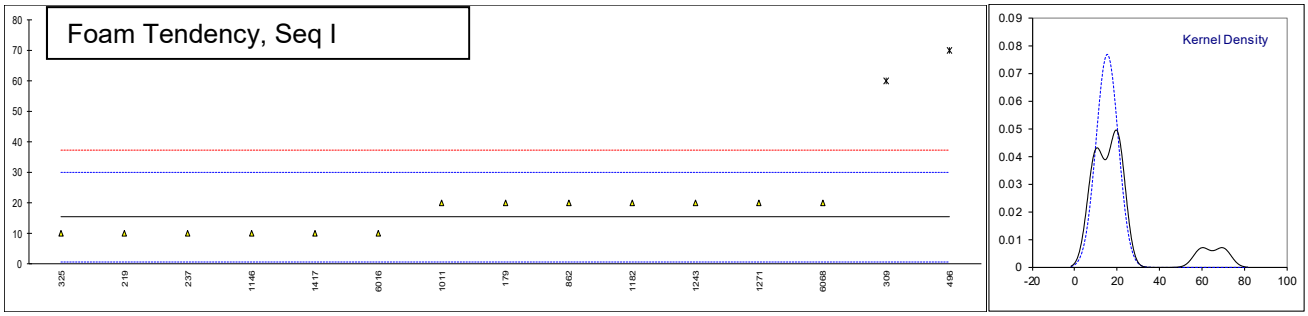



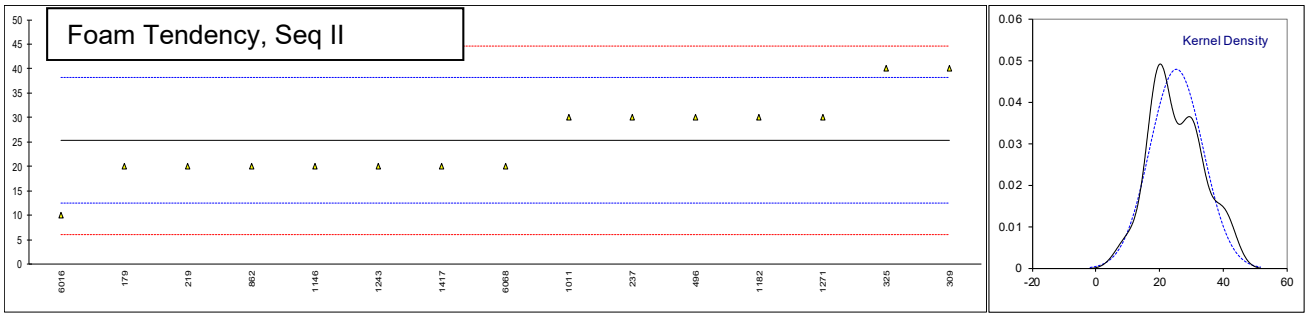
Determination of Foaming Characteristics, Foaming Tendency (at end of 5 min blowing period) on sample #19235; results in mL

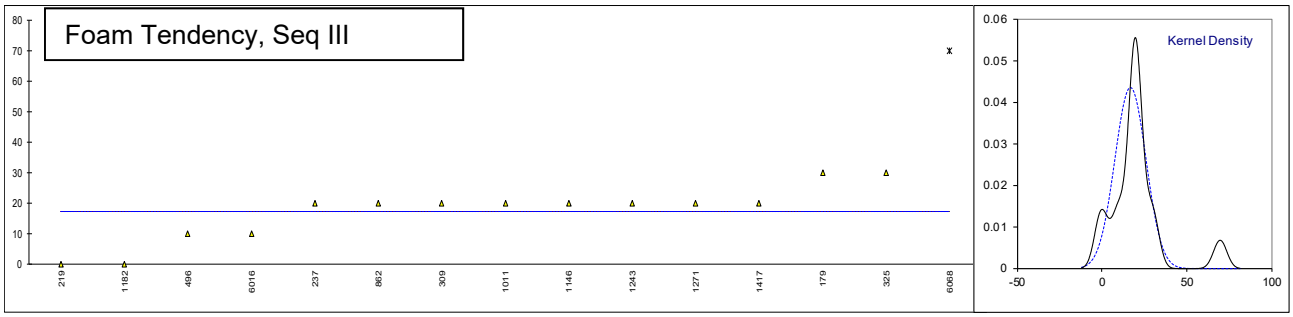
| lab | method | Seq. I | mark | z(targ) | Seq. II | mark | z(targ) | Seq. III | mark | z(targ) |
|---|---|---|---|---|---|---|---|---|---|---|
| 178 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 179 | D892 | 20 | | 0.63 | 20 | | -0.83 | 30 | | ----- |
| 219 | D892 | 10 | | -0.74 | 20 | | -0.83 | 0 | | ----- |
| 237 | D892 | 10 | | -0.74 | 30 | | 0.72 | 20 | | ----- |
| 255 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 257 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 309 | D892 | 60 | G(0.01) | 6.12 | 40 | | 2.28 | 20 | | ----- |
| 325 | D892 | 10 | | -0.74 | 40 | | 2.28 | 30 | | ----- |
| 349 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 432 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 496 | D892 | 70 | G(0.05) | 7.49 | 30 | | 0.72 | 10 | | ----- |
| 614 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 633 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 780 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 862 | D892 | 20 | | 0.63 | 20 | | -0.83 | 20 | | ----- |
| 912 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 962 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 963 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 994 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1011 | D892 | 20 | | 0.63 | 30 | | 0.72 | 20 | | ----- |
| 1059 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1146 | ISO6247 | 10 | | -0.74 | 20 | | -0.83 | 20 | | ----- |
| 1182 | D892 | 20 | | 0.63 | 30 | | 0.72 | 0 | | ----- |
| 1243 | D892 | 20 | | 0.63 | 20 | | -0.83 | 20 | | ----- |
| 1271 | ISO6247 | 20 | | 0.63 | 30 | | 0.72 | 20 | C | ----- |
| 1417 | D892 | 10 | | -0.74 | 20 | | -0.83 | 20 | | ----- |
| 1456 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1660 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1720 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1748 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1875 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 6016 | D892 | 10 | | -0.74 | 10 | | -2.38 | 10 | | ----- |
| 6034 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 6068 | ISO6247 | 20 | | 0.63 | 20 | | -0.83 | 70 | G(0.01) | ----- |
| 6257 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 6277 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |

| | | | |
|---|---|---|---|
| normality | OK | OK | OK |
| n | 13 | 15 | 14 |
| outliers | 2 | 0 | 1 |
| mean (n) | 15.38 | 25.33 | 17.14 |
| st.dev. (n) | 5.189 | 8.338 | 9.139 |
| R(calc.) | 14.53 | 23.35 | 25.59 |
| st.dev.(D892:18) | 7.290 | 6.438 | (2.694) |
| R(D892:18) | 20.41 | 18.03 | (7.54) |

Lab 1271: First reported 100


Foam Tendency, Seq I
Kernel Density


Foam Tendency, Seq II
Kernel Density


Foam Tendency, Seq III
Kernel Density

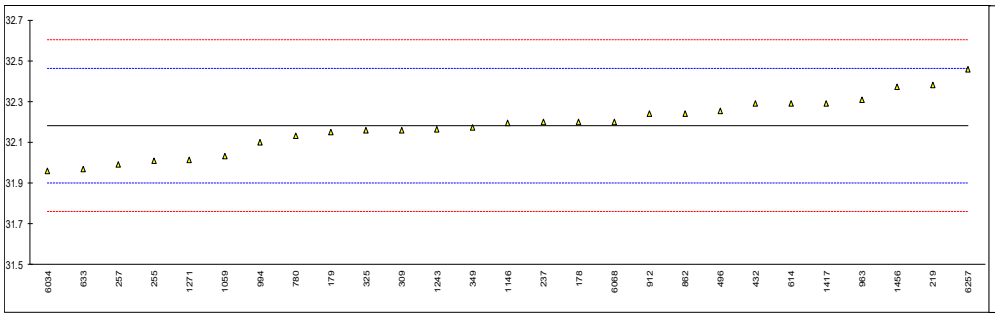
Determination of Foaming Characteristics, Foaming Stability (at end of 10 min settling period) on sample #19235; results in mL

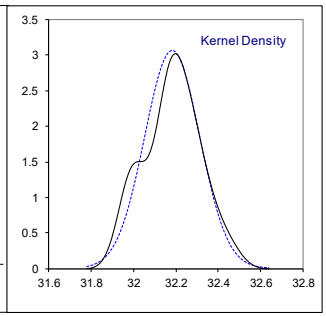
| lab | method | Seq. I | mark | z(targ) | Seq. II | mark | z(targ) | Seq. III | mark | z(targ) |
|---|---|---|---|---|---|---|---|---|---|---|
| 178 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 179 | D892 | 0 | | ----- | 0 | | ----- | 0 | | ----- |
| 219 | D892 | 0 | | ----- | 0 | | ----- | 0 | | ----- |
| 237 | D892 | 0 | | ----- | 0 | | ----- | 0 | | ----- |
| 255 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 257 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 309 | D892 | 0 | | ----- | 0 | | ----- | 0 | | ----- |
| 325 | D892 | 0 | | ----- | 0 | | ----- | 0 | | ----- |
| 349 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 432 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 496 | D892 | 0 | | ----- | 0 | | ----- | 0 | | ----- |
| 614 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 633 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 780 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 862 | D892 | 0 | | ----- | 0 | | ----- | 0 | | ----- |
| 912 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 962 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 963 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 994 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1011 | D892 | 0 | | ----- | 0 | | ----- | 0 | | ----- |
| 1059 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1146 | ISO6247 | 0 | | ----- | 0 | | ----- | 0 | | ----- |
| 1182 | D892 | 0 | | ----- | 0 | | ----- | 0 | | ----- |
| 1243 | D892 | 0 | | ----- | 0 | | ----- | 0 | | ----- |
| 1271 | ISO6247 | 0 | | ----- | 0 | | ----- | 0 | | ----- |
| 1417 | D892 | 0 | | ----- | 0 | | ----- | 0 | | ----- |
| 1456 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1660 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1720 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1748 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1875 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 6016 | D892 | 0 | | ----- | 0 | | ----- | 0 | | ----- |
| 6034 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 6068 | ISO6247 | 0 | | ----- | 0 | | ----- | 0 | | ----- |
| 6257 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 6277 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| | n | 15 | | | 15 | | | 15 | | |
| | mean (n) | 0 | | | 0 | | | 0 | | |

Determination of Kinematic Viscosity at 40°C on sample #19235; results in mm$^2$/s

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D445 | 32.2 | | 0.13 | |
| 179 | D445 | 32.15 | | -0.23 | |
| 219 | D7279 corrected to D445 | 32.379 | | 1.40 | |
| 237 | D445 | 32.20 | | 0.13 | |
| 255 | D7279 corrected to D445 | 32.01 | | -1.23 | |
| 257 | D7279 corrected to D445 | 31.99 | | -1.37 | |
| 309 | D445 | 32.16 | | -0.16 | |
| 325 | D445 | 32.16 | | -0.16 | |
| 349 | D445 | 32.17 | | -0.09 | |
| 432 | D445 | 32.29 | | 0.77 | |
| 496 | D445 | 32.253 | | 0.50 | |
| 614 | D445 | 32.29 | | 0.77 | |
| 633 | D7279 corrected to D445 | 31.968 | | -1.53 | |
| 780 | D445 | 32.13 | | -0.37 | |
| 862 | D445 | 32.241 | | 0.42 | |
| 912 | D445 | 32.24 | | 0.41 | |
| 962 | | ----- | | ----- | |
| 963 | D445 | 32.31 | | 0.91 | |
| 994 | D445 | 32.10 | | -0.59 | |
| 1011 | | ----- | | ----- | |
| 1059 | ISO3104 | 32.03 | | -1.09 | |
| 1146 | D445 | 32.193 | | 0.08 | |
| 1182 | | ----- | | ----- | |
| 1243 | D7279 corrected to D445 | 32.165 | | -0.12 | |
| 1271 | ISO3104 | 32.012 | | -1.21 | |
| 1417 | D445 | 32.29 | | 0.77 | |
| 1456 | D445 | 32.37 | | 1.34 | |
| 1660 | | ----- | | ----- | |
| 1720 | | ----- | | ----- | |
| 1748 | | ----- | | ----- | |
| 1875 | | ----- | | ----- | |
| 6016 | | ----- | | ----- | |
| 6034 | D445 | 31.96 | | -1.58 | |
| 6068 | ISO3104 | 32.20 | | 0.13 | |
| 6257 | ISO3104 | 32.4583 | | 1.97 | |
| 6277 | | ----- | | ----- | |

| | |
|---|---|
| normality | OK |
| n | 27 |
| outliers | 0 |
| mean (n) | 32.1822 |
| st.dev. (n) | 0.13012 |
| R(calc.) | 0.3643 |
| st.dev.(D445:19) | 0.14022 |
| R(D445:19) | 0.3926 |

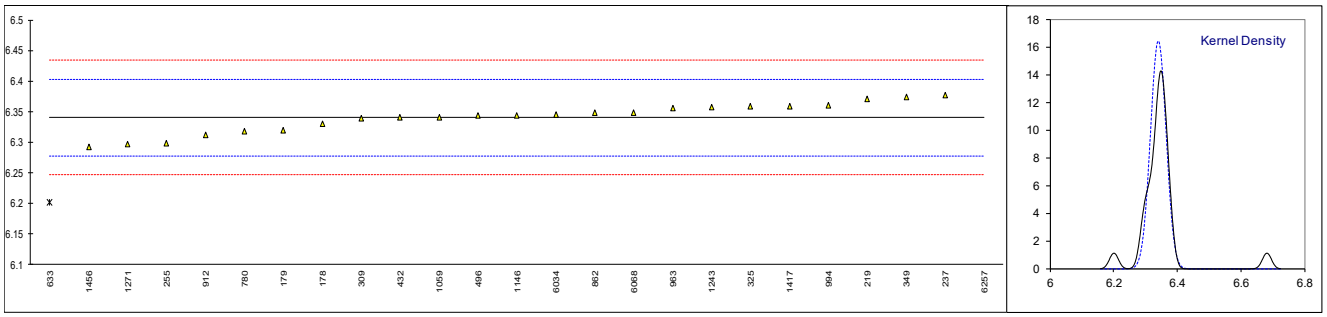


Determination of Kinematic Viscosity at 100°C on sample #19235; results in $mm^2/s$

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D445 | 6.33 | | -0.33 | |
| 179 | D445 | 6.32 | | -0.65 | |
| 219 | D7279 corrected to D445 | 6.371 | | 0.98 | |
| 237 | D445 | 6.377 | | 1.17 | |
| 255 | D7279 corrected to D445 | 6.299 | | -1.32 | |
| 257 | | ----- | | ----- | |
| 309 | D445 | 6.339 | | -0.04 | |
| 325 | D445 | 6.359 | | 0.60 | |
| 349 | D445 | 6.374 | | 1.08 | |
| 432 | D445 | 6.340 | | -0.01 | |
| 496 | D445 | 6.3432 | | 0.09 | |
| 614 | | ----- | | ----- | |
| 633 | D7279 corrected to D445 | 6.2008 | R(0.01) | -4.47 | |
| 780 | D445 | 6.318 | | -0.72 | |
| 862 | D445 | 6.3476 | | 0.23 | |
| 912 | D445 | 6.312 | | -0.91 | |
| 962 | | ----- | | ----- | |
| 963 | D445 | 6.355 | | 0.47 | |
| 994 | D445 | 6.360 | | 0.63 | |
| 1011 | | ----- | | ----- | |
| 1059 | ISO3104 | 6.341 | | 0.02 | |
| 1146 | D445 | 6.3442 | | 0.12 | |
| 1182 | | ----- | | ----- | |
| 1243 | D7279 corrected to D445 | 6.357 | | 0.53 | |
| 1271 | ISO3104 | 6.297 | | -1.39 | |
| 1417 | D445 | 6.359 | | 0.60 | |
| 1456 | D445 | 6.292 | | -1.55 | |
| 1660 | | ----- | | ----- | |
| 1720 | | ----- | | ----- | |
| 1748 | | ----- | | ----- | |
| 1875 | | ----- | | ----- | |
| 6016 | | ----- | | ----- | |
| 6034 | D445 | 6.345 | | 0.15 | |
| 6068 | ISO3104 | 6.348 | | 0.24 | |
| 6257 | ISO3104 | 6.682 | C,R(0.01) | 10.93 | First reported 6.4626 |
| 6277 | | ----- | | ----- | |

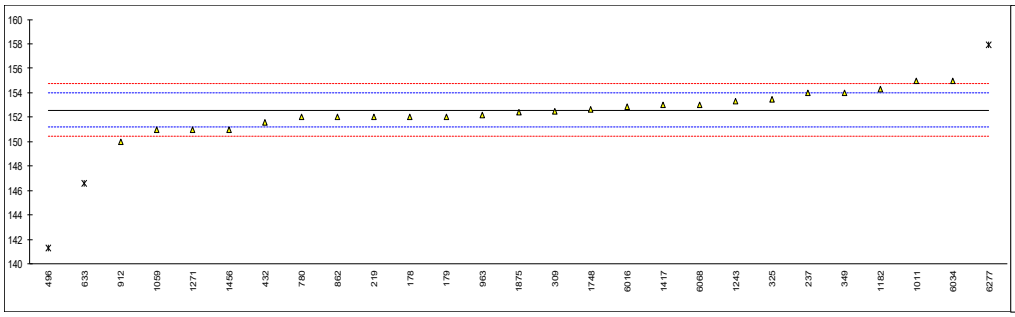
| | |
|---|---|
| normality | OK |
| n | 23 |
| outliers | 2 |
| mean (n) | 6.3403 |
| st.dev. (n) | 0.02421 |
| R(calc.) | 0.0678 |
| st.dev.(D445:19) | 0.03125 |
| R(D445:19) | 0.0875 |

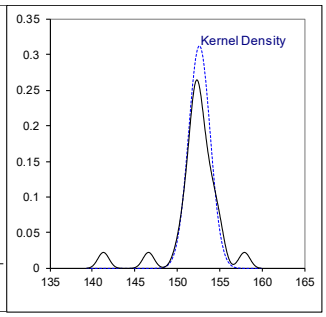


## Determination of Viscosity Index on sample #19235;

| lab | method | value | mark | z(targ) | iis calc | | remarks |
|---|---|---|---|---|---|---|---|
| 178 | D2270 | 152 | | -0.83 | 152 | | |
| 179 | D2270 | 152 | | -0.83 | 152 | | |
| 219 | D2270 | 152 | | -0.83 | 153 | | |
| 237 | D2270 | 154 | | 1.97 | 154 | | |
| 255 | | ----- | | ----- | 151 | | |
| 257 | | ----- | | ----- | ----- | | |
| 309 | D2270 | 152.481 | | -0.16 | 152 | | |
| 325 | D2270 | 153.5 | | 1.27 | 154 | | |
| 349 | D2270 | 154 | | 1.97 | 154 | | |
| 432 | D2270 | 151.6 | | -1.39 | 152 | | |
| 496 | D2270 | 141.29 | E,R(0.01) | -15.82 | 152 | | Calculation error? |
| 614 | | ----- | | ----- | ----- | | |
| 633 | D2270 | 146.6 | ex | -8.39 | 147 | ex | Test result excluded, outlier in KV 100°C |
| 780 | D2270 | 152 | | -0.83 | 152 | | |
| 862 | D2270 | 152 | | -0.83 | 152 | | |
| 912 | D2270 | 150 | | -3.63 | 150 | | |
| 962 | | ----- | | ----- | ----- | | |
| 963 | D2270 | 152.2 | | -0.55 | 152 | | |
| 994 | | ----- | | ----- | 154 | | |
| 1011 | D2270 | 155 | | 3.37 | 155 | | |
| 1059 | ISO2909 | 151 | E | -2.23 | 154 | | Calculation error? |
| 1146 | | ----- | | ----- | 153 | | |
| 1182 | D2270 | 154.3 | | 2.39 | 152 | | |
| 1243 | ISO2909 | 153.3 | | 0.99 | 153 | | |
| 1271 | ISO2909 | 151 | | -2.23 | 151 | | |
| 1417 | D2270 | 153 | | 0.57 | 153 | | |
| 1456 | D2270 | 151 | E | -2.23 | 148 | R(0.01) | Calculation error? |
| 1660 | | ----- | | ----- | 153 | | |
| 1720 | | ----- | | ----- | 153 | | |
| 1748 | D2270 | 152.6 | | 0.01 | 154 | | |
| 1875 | ISO2909 | 152.4 | | -0.27 | 153 | | |
| 6016 | D2270 | 152.836 | C | 0.34 | ----- | | First reported 142.787 |
| 6034 | D2270 | 155 | | 3.37 | 154 | | |
| 6068 | ISO2909 | 153 | | 0.57 | 153 | | |
| 6257 | | ----- | | ----- | 168 | R(0.01) | |
| 6277 | D2270 | 157.9 | R(0.05) | 7.43 | 158 | R(0.01) | |

| | | |
|---|---|---|
| normality | OK | OK |
| n | 24 | 28 |
| outliers | 2 (+1ex) | 3 (+1ex) |
| mean (n) | 152.59 | 152.75 |
| st.dev. (n) | 1.276 | 1.143 |
| R(calc.) | 3.57 | 3.20 |
| st.dev.(D2270:10) | 0.714 | 0.714 |
| R(D2270:10) | 2 | 2 |

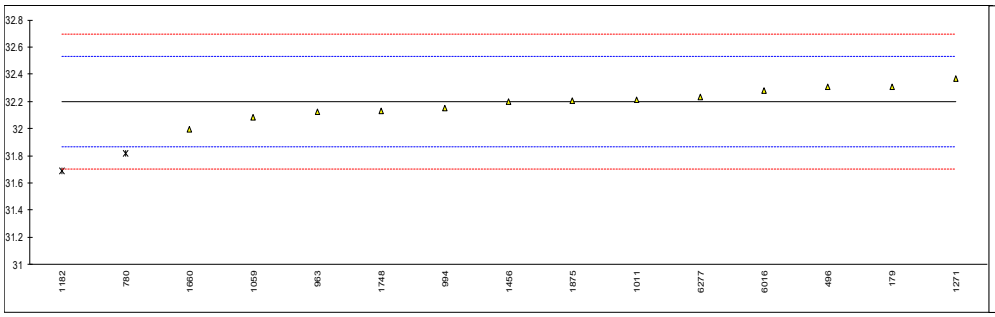


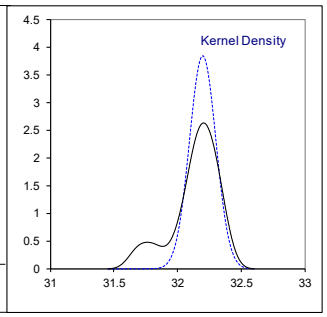



Determination of Viscosity Stabinger at 40°C on sample #19235; results in $mm^2/s$

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | | ----- | | ----- | |
| 179 | D7042 | 32.31 | | 0.67 | |
| 219 | | ----- | | ----- | |
| 237 | | ----- | | ----- | |
| 255 | | ----- | | ----- | |
| 257 | | ----- | | ----- | |
| 309 | | ----- | | ----- | |
| 325 | | ----- | | ----- | |
| 349 | | ----- | | ----- | |
| 432 | | ----- | | ----- | |
| 496 | D7042 | 32.306 | | 0.64 | |
| 614 | | ----- | | ----- | |
| 633 | | ----- | | ----- | |
| 780 | D7042 | 31.82 | DG(0.05) | -2.28 | |
| 862 | | ----- | | ----- | |
| 912 | | ----- | | ----- | |
| 962 | | ----- | | ----- | |
| 963 | D7042 | 32.12 | | -0.48 | |
| 994 | D7042 | 32.15 | | -0.30 | |
| 1011 | D7042 | 32.21 | | 0.06 | |
| 1059 | D7042 | 32.08 | | -0.72 | |
| 1146 | | ----- | | ----- | |
| 1182 | D7042 | 31.69 | C,DG(0.05) | -3.07 | First reported 31.39 |
| 1243 | | ----- | | ----- | |
| 1271 | D7042 | 32.367 | | 1.01 | |
| 1417 | | ----- | | ----- | |
| 1456 | D7042 | 32.20 | | 0.00 | |
| 1660 | D7042 | 31.995 | | -1.23 | |
| 1720 | | ----- | | ----- | |
| 1748 | D7042 | 32.131 | | -0.41 | |
| 1875 | D7042 | 32.207 | | 0.05 | |
| 6016 | D7042 | 32.283 | | 0.50 | |
| 6034 | | ----- | | ----- | |
| 6068 | | ----- | | ----- | |
| 6257 | | ----- | | ----- | |
| 6277 | D7042 | 32.232 | | 0.20 | |

| | |
|---|---|
| normality | OK |
| n | 13 |
| outliers | 2 |
| mean (n) | 32.1993 |
| st.dev. (n) | 0.10383 |
| R(calc.) | 0.2907 |
| st.dev.(D7042:16e3) | 0.16615 |
| R(D7042:16e3) | 0.4652 |

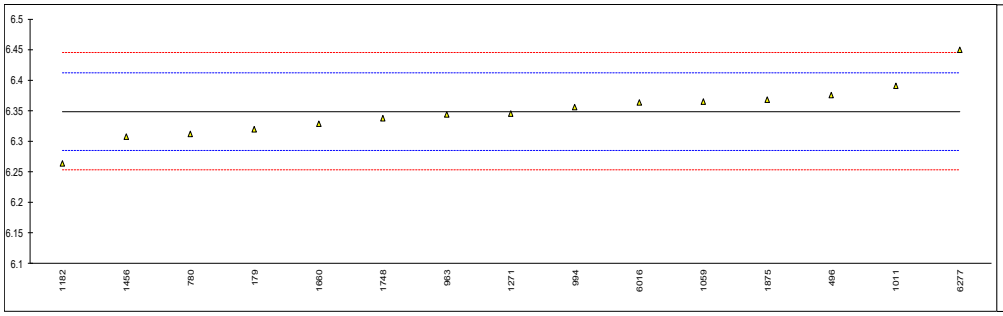


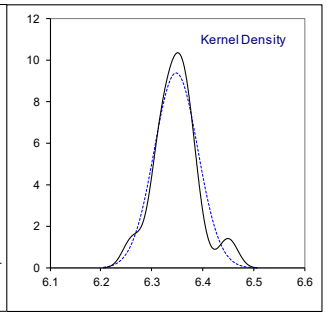



Determination of Viscosity Stabinger at 100°C on sample #19235; results in $mm^2/s$

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | | ----- | | ----- | |
| 179 | D7042 | 6.32 | | -0.88 | |
| 219 | | ----- | | ----- | |
| 237 | | ----- | | ----- | |
| 255 | | ----- | | ----- | |
| 257 | | ----- | | ----- | |
| 309 | | ----- | | ----- | |
| 325 | | ----- | | ----- | |
| 349 | | ----- | | ----- | |
| 432 | | ----- | | ----- | |
| 496 | D7042 | 6.3759 | | 0.86 | |
| 614 | | ----- | | ----- | |
| 633 | | ----- | | ----- | |
| 780 | D7042 | 6.312 | C | -1.13 | First reported 6.212 |
| 862 | | ----- | | ----- | |
| 912 | | ----- | | ----- | |
| 962 | | ----- | | ----- | |
| 963 | D7042 | 6.343 | | -0.16 | |
| 994 | D7042 | 6.355 | | 0.21 | |
| 1011 | D7042 | 6.390 | | 1.31 | |
| 1059 | D7042 | 6.365 | | 0.52 | |
| 1146 | | ----- | | ----- | |
| 1182 | D7042 | 6.263 | | -2.66 | |
| 1243 | | ----- | | ----- | |
| 1271 | D7042 | 6.3451 | | -0.10 | |
| 1417 | | ----- | | ----- | |
| 1456 | D7042 | 6.307 | | -1.29 | |
| 1660 | D7042 | 6.3285 | | -0.62 | |
| 1720 | | ----- | | ----- | |
| 1748 | D7042 | 6.3378 | | -0.33 | |
| 1875 | D7042 | 6.3681 | | 0.62 | |
| 6016 | D7042 | 6.363 | | 0.46 | |
| 6034 | | ----- | | ----- | |
| 6068 | | ----- | | ----- | |
| 6257 | | ----- | | ----- | |
| 6277 | D7042 | 6.45 | | 3.18 | |

| | |
|---|---|
| normality | suspect |
| n | 15 |
| outliers | 0 |
| mean (n) | 6.3482 |
| st.dev. (n) | 0.04261 |
| R(calc.) | 0.1193 |
| st.dev.(D7042:16e3) | 0.03200 |
| R(D7042:16e3) | 0.0896 |

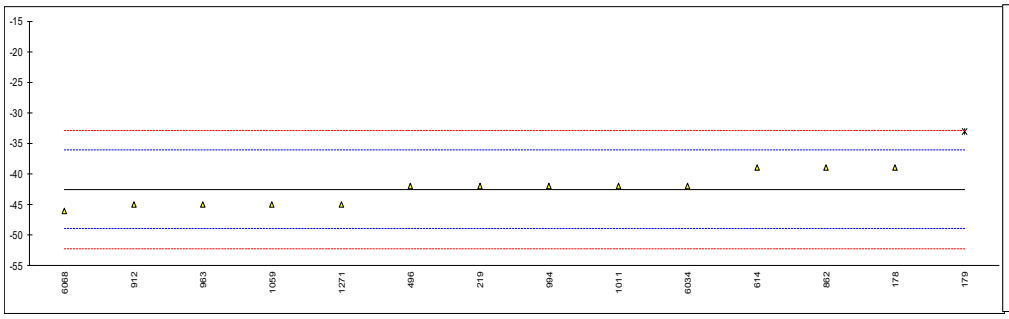


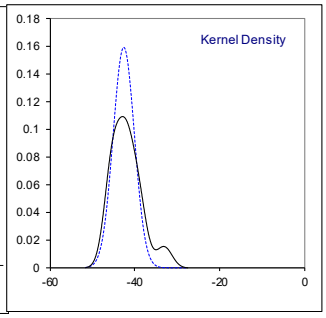



## Determination of Pour Point, Manual on sample #19235; results in °C

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D97 | -39 | | 1.10 | |
| 179 | D97 | -33 | G(0.05) | 2.97 | |
| 219 | D97 | -42 | | 0.17 | |
| 237 | D97 | <-24 | | ----- | |
| 255 | | ----- | | ----- | |
| 257 | | ----- | | ----- | |
| 309 | | ----- | | ----- | |
| 325 | | ----- | | ----- | |
| 349 | | ----- | | ----- | |
| 432 | | ----- | | ----- | |
| 496 | D97 | -42 | | 0.17 | |
| 614 | D97 | -39 | | 1.10 | |
| 633 | | ----- | | ----- | |
| 780 | D97 | ≤-42 | | ----- | |
| 862 | D97 | -39 | | 1.10 | |
| 912 | D97 | -45 | | -0.77 | |
| 962 | | ----- | | ----- | |
| 963 | D97 | -45 | | -0.77 | |
| 994 | D97 | -42 | | 0.17 | |
| 1011 | D97 | -42 | | 0.17 | |
| 1059 | ISO3016 | -45 | | -0.77 | |
| 1146 | | ----- | | ----- | |
| 1182 | | ----- | | ----- | |
| 1243 | | ----- | | ----- | |
| 1271 | ISO3016 | -45 | | -0.77 | |
| 1417 | | ----- | | ----- | |
| 1456 | | ----- | | ----- | |
| 1660 | | ----- | | ----- | |
| 1720 | | ----- | | ----- | |
| 1748 | | ----- | | ----- | |
| 1875 | | ----- | | ----- | |
| 6016 | | ----- | | ----- | |
| 6034 | D97 | -42 | | 0.17 | |
| 6068 | ISO3016 | -46 | | -1.08 | |
| 6257 | | ----- | | ----- | |
| 6277 | | ----- | | ----- | |

| | |
|---|---|
| normality | OK |
| n | 13 |
| outliers | 1 |
| mean (n) | -42.54 |
| st.dev. (n) | 2.504 |
| R(calc.) | 7.01 |
| st.dev.(D97:17b) | 3.214 |
| R(D97:17b) | 9 |

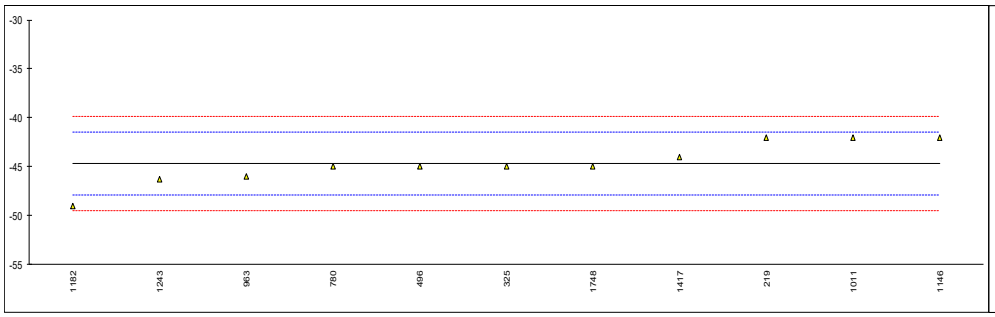


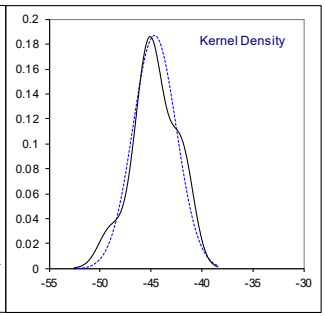



Determination of Pour Point, Automated, 1°C interval on sample #19235; results in °C

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | | ----- | | ----- | |
| 179 | | ----- | | ----- | |
| 219 | D5950 | -42 | | 1.66 | |
| 237 | | ----- | | ----- | |
| 255 | | ----- | | ----- | |
| 257 | | ----- | | ----- | |
| 309 | | ----- | | ----- | |
| 325 | D5950 | -45 | | -0.21 | |
| 349 | | ----- | | ----- | |
| 432 | | ----- | | ----- | |
| 496 | D5950 | -45 | | -0.21 | |
| 614 | | ----- | | ----- | |
| 633 | | ----- | | ----- | |
| 780 | D5950 | -45 | | -0.21 | |
| 862 | | ----- | | ----- | |
| 912 | | ----- | | ----- | |
| 962 | | ----- | | ----- | |
| 963 | D5950 | -46 | | -0.83 | |
| 994 | | ----- | | ----- | |
| 1011 | | -42 | | 1.66 | |
| 1059 | | ----- | | ----- | |
| 1146 | D6893 | -42.0 | | 1.66 | |
| 1182 | D5949 | -49 | | -2.70 | |
| 1243 | D7346 | -46.3 | | -1.02 | |
| 1271 | | ----- | | ----- | |
| 1417 | D5950 | -44 | | 0.41 | |
| 1456 | | ----- | | ----- | |
| 1660 | | ----- | | ----- | |
| 1720 | | ----- | | ----- | |
| 1748 | D7346 | -45 | | -0.21 | |
| 1875 | | ----- | | ----- | |
| 6016 | | ----- | | ----- | |
| 6034 | | ----- | | ----- | |
| 6068 | | ----- | | ----- | |
| 6257 | | ----- | | ----- | |
| 6277 | | ----- | | ----- | |

| | |
|---|---|
| normality | OK |
| n | 11 |
| outliers | 0 |
| mean (n) | -44.66 |
| st.dev. (n) | 2.132 |
| R(calc.) | 5.97 |
| st.dev.(D5950:14) | 1.607 |
| R(D5950:14) | 4.5 |

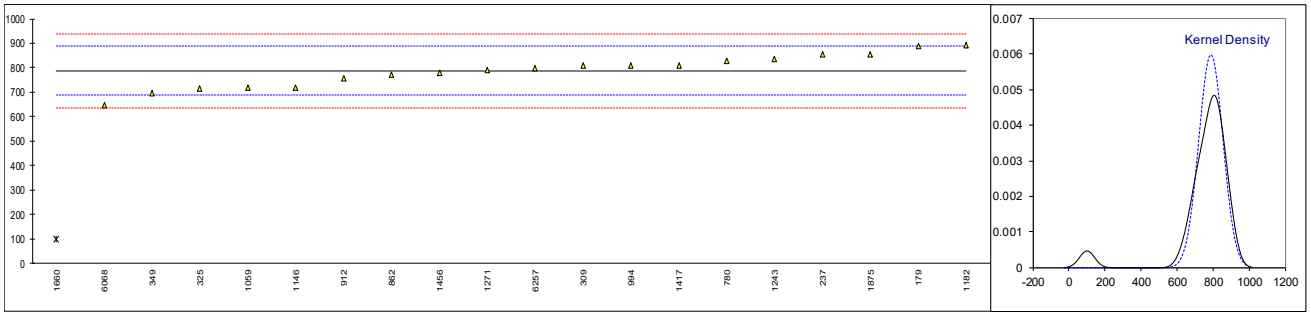


## Determination of Sulfur on sample #19235; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | | ----- | | ----- | |
| 179 | D4294 | 888 | | 1.98 | |
| 219 | | ----- | | ----- | |
| 237 | D4294 | 853 | | 1.29 | |
| 255 | | ----- | | ----- | |
| 257 | | ----- | | ----- | |
| 309 | D4294 | 810 | C | 0.44 | First reported 0.081 mg/kg |
| 325 | D5185 | 713 | | -1.49 | |
| 349 | D2622 | 696 | | -1.82 | |
| 432 | | ----- | | ----- | |
| 496 | | ----- | | ----- | |
| 614 | | ----- | | ----- | |
| 633 | | ----- | | ----- | |
| 780 | D4294 | 827 | | 0.77 | |
| 862 | D4294 | 772 | | -0.32 | |
| 912 | D4294 | 755 | | -0.65 | |
| 962 | | ----- | | ----- | |
| 963 | | ----- | | ----- | |
| 994 | D4294 | 810 | | 0.44 | |
| 1011 | | ----- | | ----- | |
| 1059 | ISO14596Mod. | 720 | | -1.35 | |
| 1146 | D4294 | 720 | | -1.35 | |
| 1182 | D4294 | 893 | | 2.08 | |
| 1243 | ISO8754 | 837 | | 0.97 | |
| 1271 | D4294 | 790 | C | 0.04 | First reported 1245 |
| 1417 | IP336 | 810 | | 0.44 | |
| 1456 | D5185 | 778 | | -0.20 | |
| 1660 | | 100 | R(0.01) | -13.64 | |
| 1720 | | ----- | | ----- | |
| 1748 | | ----- | | ----- | |
| 1875 | | 854.3 | | 1.31 | |
| 6016 | | ----- | | ----- | |
| 6034 | | ----- | | ----- | |
| 6068 | ISO20884 | 646.8 | | -2.80 | |
| 6257 | ISO8754 | 799 | C | 0.22 | First reported 252.7 |
| 6277 | | ----- | | ----- | |

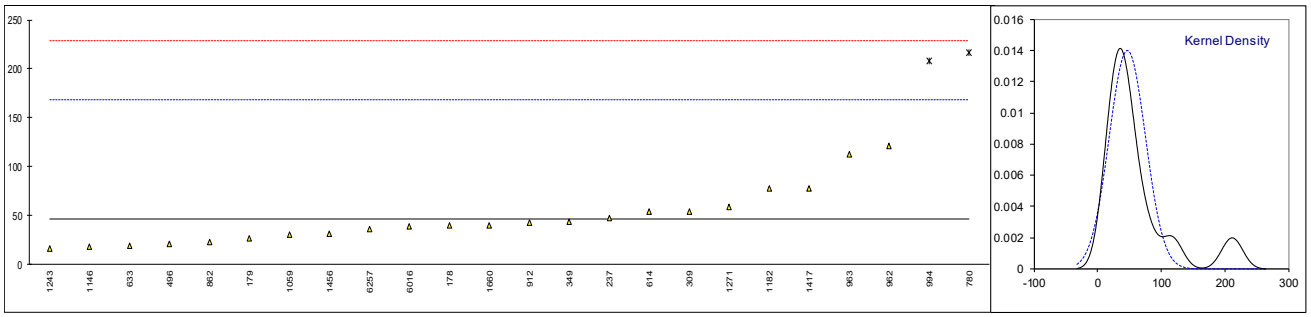
| | |
|---|---|
| normality | OK |
| n | 19 |
| outliers | 1 |
| mean (n) | 788.01 |
| st.dev. (n) | 66.610 |
| R(calc.) | 186.51 |
| st.dev.(D4294:16e1) | 50.447 |
| R(D4294:16e1) | 141.25 |



## Determination of Water on sample #19235; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D6304-C | 40 | | -0.11 | |
| 179 | D6304-C | 27 | | -0.33 | |
| 219 | | ----- | | ----- | |
| 237 | D6304-C | 47.5 | | 0.01 | |
| 255 | | ----- | | ----- | |
| 257 | | ----- | | ----- | |
| 309 | D6304-A | 54 | | 0.12 | |
| 325 | | ----- | | ----- | |
| 349 | D6304-C | 44 | | -0.05 | |
| 432 | | ----- | | ----- | |
| 496 | D6304-C | 21 | | -0.43 | |
| 614 | D6304-C | 54 | | 0.12 | |
| 633 | D6304-C | 19.35 | | -0.45 | |
| 780 | D6304-C | 216 | R(0.01) | 2.79 | |
| 862 | D6304-c | 23 | | -0.39 | |
| 912 | D6304-C | 43 | | -0.06 | |
| 962 | D6304-A | 121 | | 1.22 | |
| 963 | D6304-C | 113 | | 1.09 | |
| 994 | IP439 | 208 | R(0.01) | 2.65 | |
| 1011 | | ----- | | ----- | |
| 1059 | D6304-C | 30 | | -0.28 | |
| 1146 | D6304-C | 18 | | -0.48 | |
| 1182 | ISO12937 | 77.85 | | 0.51 | |
| 1243 | ISO12937 | 16 | | -0.51 | |
| 1271 | D6304-A | 59 | | 0.20 | |
| 1417 | D6304-A | 78 | | 0.51 | |
| 1456 | D6304-A | 31.4 | | -0.26 | |
| 1660 | EN60814 | 40 | | -0.11 | |
| 1720 | | ----- | | ----- | |
| 1748 | | ----- | | ----- | |
| 1875 | | ----- | | ----- | |
| 6016 | D6304-A | 38.5 | | -0.14 | |
| 6034 | | ----- | | ----- | |
| 6068 | | ----- | | ----- | |
| 6257 | ISO12937 | 35.9 | | -0.18 | |
| 6277 | | ----- | | ----- | |

| | |
|---|---|
| normality | not OK |
| n | 22 |
| outliers | 2 |
| mean (n) | 46.886 |
| st.dev. (n) | 28.4990 |
| R(calc.) | 79.797 |
| st.dev.(D6304:16e1) | 60.6936 |
| R(D6304:16e1) | 169.942 |

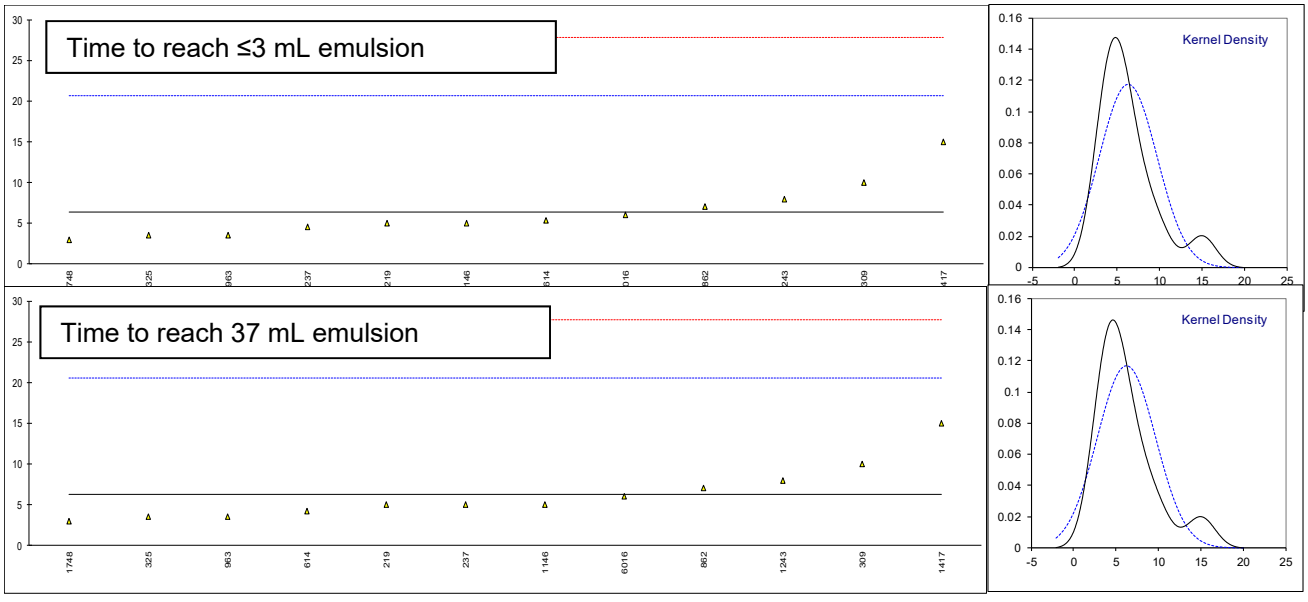


Determination of Water Separability at 54°C, distilled water on sample #19235; results in minutes

| lab | method | ≤3 mL emulsion | m. | z(targ) | 37 mL water | m. | z(targ) | compl. break (40-40-0) | m. | z(targ) | test aborted | time aborted |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 178 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | ----- |
| 179 | D1401 | ----- | | ----- | ----- | | ----- | 11 | | ----- | NO | ----- |
| 219 | D1401 | 5 | | -0.19 | 5 | | -0.18 | 5 | | ----- | NO | ----- |
| 237 | D1401 | 4.6 | | -0.24 | 5.0 | | -0.18 | 7.0 | | ----- | NO | ----- |
| 255 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | ----- |
| 257 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | ----- |
| 309 | D1401 | 10 | | 0.51 | 10 | | 0.52 | 10 | | ----- | YES | 10 |
| 325 | D1401 | 3.5 | | -0.40 | 3.5 | | -0.39 | 3.5 | | ----- | NO | ----- |
| 349 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | ----- |
| 432 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | ----- |
| 496 | | ----- | | ----- | ----- | | ----- | 5 | | ----- | NO | ----- |
| 614 | D1401 | 5.38 | | -0.13 | 4.2 | | -0.29 | 6.3 | | ----- | NO | ----- |
| 633 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | ----- |
| 780 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | ----- |
| 862 | D1401 | 7 | | 0.09 | 7 | | 0.10 | 10 | | ----- | ----- | ----- |
| 912 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | ----- |
| 962 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | ----- |
| 963 | D1401 | 3.5 | | -0.40 | 3.5 | | -0.39 | 4.2 | | ----- | NO | ----- |
| 994 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | ----- |
| 1011 | | ----- | | ----- | ----- | | ----- | 5 | | ----- | NO | ----- |
| 1059 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | ----- |
| 1146 | D1401 | 5 | | -0.19 | 5 | | -0.18 | ----- | | ----- | ----- | ----- |
| 1182 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | ----- |
| 1243 | ISO6614 | 8 | | 0.23 | 8 | | 0.24 | 10 | | ----- | NO | ----- |
| 1271 | | ----- | | ----- | ----- | | ----- | 9 | | ----- | ----- | ----- |
| 1417 | D1401 | 15 | | 1.21 | 15 | | 1.22 | 15 | | ----- | NO | ----- |
| 1456 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | ----- |
| 1660 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | ----- |
| 1720 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | ----- |
| 1748 | | 3 | | -0.47 | 3 | | -0.46 | 4 | | ----- | YES | 4 |
| 1875 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | ----- |
| 6016 | D1401 | 6.02 | | -0.04 | 6.02 | | -0.03 | 6.57 | | ----- | ----- | ----- |
| 6034 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | ----- |
| 6068 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | ----- |
| 6257 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | ----- |
| 6277 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | ----- |

| | | | |
|---|---|---|---|
| normality | not OK | not OK | OK |
| n | 12 | 12 | 15 |
| outliers | 0 | 0 | 0 |
| mean (n) | 6.33 | 6.27 | 7.44 |
| st.dev. (n) | 3.391 | 3.423 | 3.272 |
| R(calc.) | 9.50 | 9.58 | 9.16 |
| st.dev.(D1401:18a) | 7.143 | 7.143 | n.a. |
| R(D1401:18a) | 20 | 20 | n.a. |

m=mark



Determination of Water Separability at 54°C, distilled water on sample #19235; results in mL.
--- Continued ----

| lab | method | oil | mark | z(targ) | water | mark | z(targ) | emulsion | mark | z(targ) |
|---|---|---|---|---|---|---|---|---|---|---|
| 178 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 179 | D1401 | 40 | | ----- | 40 | | ----- | 0 | | ----- |
| 219 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 237 | D1401 | 40 | | ----- | 40 | | ----- | 0 | | ----- |
| 255 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 257 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 309 | D1401 | 40 | | ----- | 40 | | ----- | 0 | | ----- |
| 325 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 349 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 432 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 496 | D1401 | 41 | | ----- | 39 | | ----- | 0 | | ----- |
| 614 | D1401 | 40 | | ----- | 40 | | ----- | 0 | | ----- |
| 633 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 780 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 862 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 912 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 962 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 963 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 994 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1011 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1059 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1146 | D1401 | 40 | | ----- | 37 | | ----- | 3 | | ----- |
| 1182 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1243 | ISO6614 | 40 | | ----- | 40 | | ----- | 0 | | ----- |
| 1271 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1417 | D1401 | 40 | | ----- | 40 | | ----- | 0 | | ----- |
| 1456 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1660 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1720 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1748 | | 40 | | ----- | 40 | | ----- | 0 | | ----- |
| 1875 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 6016 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 6034 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 6068 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 6257 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 6277 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |

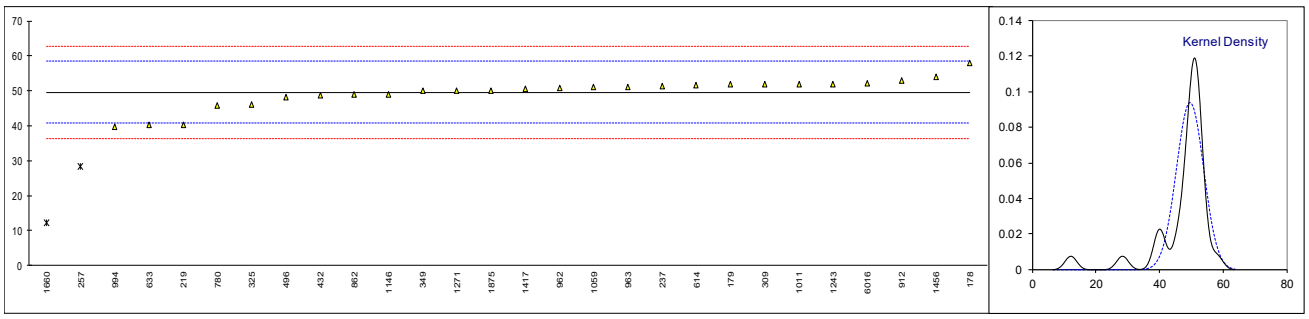
| | | | |
|---|---|---|---|
| normality | not OK | not OK | not OK |
| n | 9 | 9 | 9 |
| outliers | 0 | 0 | 0 |
| mean (n) | 40.11 | 39.56 | 0.33 |
| st.dev. (n) | 0.333 | 1.014 | 1.000 |
| R(calc.) | 0.93 | 2.84 | 2.80 |
| st.dev.(target) | n.a. | n.a. | n.a. |
| R(target) | n.a. | n.a. | n.a. |

Determination of Calcium as Ca on sample #19235; results in mg/kg.

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | | 58 | | 1.91 | |
| 179 | D5185 | 52 | | 0.55 | |
| 219 | D4951 | 40.21 | C | -2.12 | First reported 62.0 |
| 237 | D5185 | 51.39 | | 0.41 | |
| 255 | | ----- | | ----- | |
| 257 | D6595 | 28.45 | R(0.01) | -4.79 | |
| 309 | D5185 | 52 | | 0.55 | |
| 325 | D5185 | 46 | | -0.81 | |
| 349 | D5185 | 50 | | 0.10 | |
| 432 | D5185 | 48.7 | | -0.20 | |
| 496 | D5185 | 48.26 | | -0.30 | |
| 614 | D5185 | 51.7 | | 0.48 | |
| 633 | D6595 | 40.2 | | -2.13 | |
| 780 | D5185 | 45.8 | | -0.86 | |
| 862 | D5185 | 49 | | -0.13 | |
| 912 | D5185 | 53 | | 0.78 | |
| 962 | D5185 | 50.93 | | 0.31 | |
| 963 | D5185 | 51.09 | | 0.34 | |
| 994 | D5185 | 39.77 | | -2.22 | |
| 1011 | D5185 | 52 | | 0.55 | |
| 1059 | In house | 51 | | 0.32 | |
| 1146 | D5185 | 49.07 | | -0.11 | |
| 1182 | | ----- | | ----- | |
| 1243 | DIN51399 | 52 | | 0.55 | |
| 1271 | | 50 | C | 0.10 | First reported 37 |
| 1417 | In house | 50.6 | | 0.23 | |
| 1456 | D5185 | 54.1 | C | 1.03 | First reported 65.3 |
| 1660 | D4951 | 12.3 | R(0.01) | -8.46 | |
| 1720 | | ----- | | ----- | |
| 1748 | | ----- | | ----- | |
| 1875 | | 50 | | 0.10 | |
| 6016 | D5185 | 52.14 | | 0.58 | |
| 6034 | | ----- | | ----- | |
| 6068 | | ----- | | ----- | |
| 6257 | | ----- | | ----- | |
| 6277 | | ----- | | ----- | |

| | | |
|---|---|---|
| | normality | suspect |
| | n | 26 |
| | outliers | 2 |
| | mean (n) | 49.575 |
| | st.dev. (n) | 4.2459 |
| | R(calc.) | 11.889 |
| | st.dev.(Horwitz) | 4.4078 |
| | R(Horwitz) | 12.342 |
| Compare | | |
| | R(D5185:18) | 2.398 |

Application range D5185:18 = 40 – 9000 mg/kg

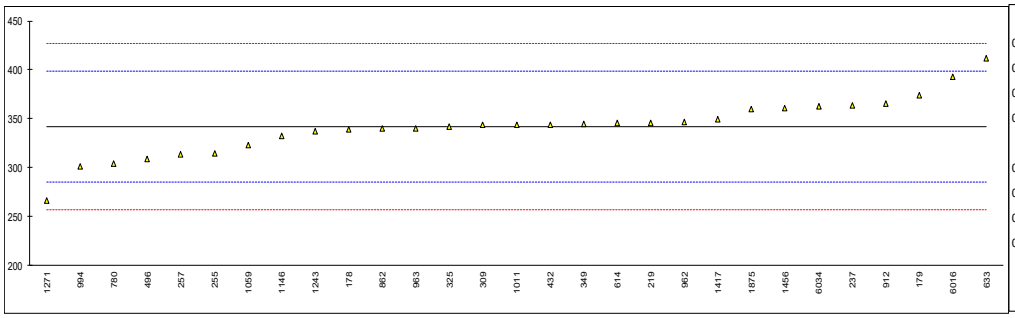


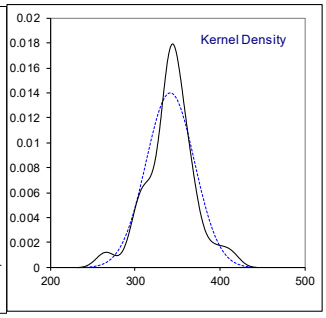
Determination of Phosphorus as P on sample #19235; results in mg/kg.

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | | 339 | | -0.10 | |
| 179 | D5185 | 374 | | 1.13 | |
| 219 | D4951 | 346.0 | | 0.14 | |
| 237 | D5185 | 363.15 | | 0.75 | |
| 255 | D6595 | 314 | | -0.98 | |
| 257 | D6595 | 313.41 | | -1.00 | |
| 309 | D5185 | 344 | | 0.07 | |
| 325 | D5185 | 342 | | 0.00 | |
| 349 | D5185 | 345 | | 0.11 | |
| 432 | D5185 | 344.1 | | 0.08 | |
| 496 | D5185 | 309.19 | | -1.15 | |
| 614 | D5185 | 346 | | 0.14 | |
| 633 | D6595 | 411.7 | | 2.46 | |
| 780 | D5185 | 304 | | -1.34 | |
| 862 | D5185 | 340 | | -0.07 | |
| 912 | D5185 | 365 | | 0.81 | |
| 962 | D5185 | 346.44 | | 0.16 | |
| 963 | D5185 | 340.3 | | -0.06 | |
| 994 | D5185 | 301 | | -1.44 | |
| 1011 | D5185 | 344 | | 0.07 | |
| 1059 | In house | 323 | | -0.67 | |
| 1146 | D5185 | 332.5 | | -0.33 | |
| 1182 | | ----- | | ----- | |
| 1243 | DIN51399 | 337 | | -0.17 | |
| 1271 | | 266.2 | | -2.67 | |
| 1417 | In house | 349.2 | | 0.26 | |
| 1456 | D5185 | 361 | | 0.67 | |
| 1660 | | ----- | | ----- | |
| 1720 | | ----- | | ----- | |
| 1748 | | ----- | | ----- | |
| 1875 | | 360 | | 0.64 | |
| 6016 | D5185 | 392.5 | | 1.78 | |
| 6034 | D5185 | 362.5 | | 0.72 | |
| 6068 | | ----- | | ----- | |
| 6257 | | ----- | | ----- | |
| 6277 | | ----- | | ----- | |

| | |
|---|---|
| normality | suspect |
| n | 29 |
| outliers | 0 |
| mean (n) | 341.938 |
| st.dev. (n) | 28.4601 |
| R(calc.) | 79.688 |
| st.dev.(D5185:18) | 28.3977 |
| R(D5185:18) | 79.514 |

Application range D5185:18 = 10 – 1000 mg/kg



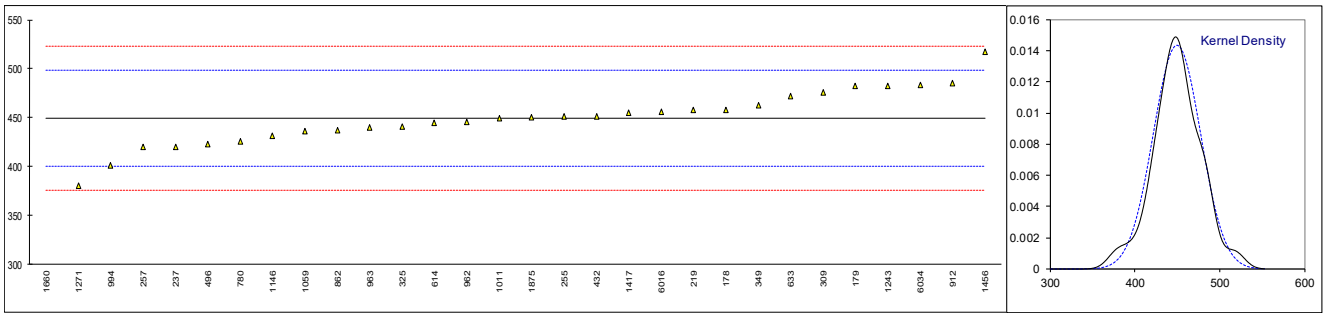



Determination of Zinc as Zn on sample #19235; results in mg/kg.

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | | 458 | | 0.35 | |
| 179 | D5185 | 482 | | 1.32 | |
| 219 | D4951 | 458.0 | | 0.35 | |
| 237 | D5185 | 420.4 | | -1.19 | |
| 255 | D6595 | 451 | | 0.06 | |
| 257 | D6595 | 420.39 | | -1.19 | |
| 309 | D5185 | 476 | | 1.08 | |
| 325 | D5185 | 441 | | -0.35 | |
| 349 | D5185 | 463 | | 0.55 | |
| 432 | D5185 | 451.6 | | 0.09 | |
| 496 | D5185 | 423.01 | | -1.08 | |
| 614 | D5185 | 445 | | -0.18 | |
| 633 | D6595 | 472.3 | | 0.93 | |
| 780 | D5185 | 426 | | -0.96 | |
| 862 | D5185 | 437 | | -0.51 | |
| 912 | D5185 | 485 | | 1.45 | |
| 962 | D5185 | 445.44 | | -0.17 | |
| 963 | D5185 | 439.7 | | -0.40 | |
| 994 | D5185 | 401 | | -1.98 | |
| 1011 | D5185 | 449 | | -0.02 | |
| 1059 | In house | 436 | | -0.55 | |
| 1146 | D5185 | 431.9 | | -0.72 | |
| 1182 | | ----- | | ----- | |
| 1243 | DIN51399 | 482 | | 1.32 | |
| 1271 | | 380 | | -2.83 | |
| 1417 | In house | 454.9 | | 0.22 | |
| 1456 | D5185 | 517 | | 2.75 | |
| 1660 | D4951 | 110 | R(0.01) | -13.83 | |
| 1720 | | ----- | | ----- | |
| 1748 | | ----- | | ----- | |
| 1875 | | 450 | | 0.02 | |
| 6016 | D5185 | 455.6 | | 0.25 | |
| 6034 | D5185 | 483.2 | | 1.37 | |
| 6068 | | ----- | | ----- | |
| 6257 | | ----- | | ----- | |
| 6277 | | ----- | | ----- | |

| | |
|---|---|
| normality | OK |
| n | 29 |
| outliers | 1 |
| mean (n) | 449.498 |
| st.dev. (n) | 27.8202 |
| R(calc.) | 77.897 |
| st.dev.(D5185:18) | 24.5426 |
| R(D5185:18) | 68.719 |

Application range D5185;18 = 60 – 1600 mg/kg



## APPENDIX 2

### Analytical details: Foam determination

| lab | Sample used | Diffuser type | Cylinder cleansed | Gas diffuser cleansed | Air tube cleansed | Air flow rate constant |
|---|---|---|---|---|---|---|
| 178 | --- | --- | --- | --- | --- | --- |
| 179 | As received | Metal (Stainless Steel) | Yes | Yes | Yes | re-adjustment NOT needed |
| 219 | --- | --- | --- | --- | --- | --- |
| 237 | As received | Metal (Stainless Steel) | Yes | Yes | Yes | re-adjustment NOT needed |
| 255 | --- | --- | --- | --- | --- | --- |
| 257 | --- | --- | --- | --- | --- | --- |
| 309 | After agitation (option A) | Metal (Stainless Steel) | Yes | ? | ? | re-adjustment NOT needed |
| 325 | As received | Metal (Stainless Steel) | Yes | Yes | Yes | re-adjustment NOT needed |
| 349 | --- | --- | --- | --- | --- | --- |
| 432 | --- | --- | --- | --- | --- | --- |
| 496 | --- | --- | --- | --- | --- | --- |
| 614 | --- | --- | --- | --- | --- | --- |
| 633 | --- | --- | --- | --- | --- | --- |
| 780 | --- | --- | --- | --- | --- | --- |
| 862 | As received | Metal (Stainless Steel) | Yes | Yes | Yes | re-adjustment NOT needed |
| 912 | --- | --- | --- | --- | --- | --- |
| 962 | --- | --- | --- | --- | --- | --- |
| 963 | --- | --- | --- | --- | --- | --- |
| 994 | --- | --- | --- | --- | --- | --- |
| 1011 | --- | --- | --- | --- | --- | --- |
| 1059 | --- | --- | --- | --- | --- | --- |
| 1146 | As received | Metal (Stainless Steel) | ? | Yes | ? | re-adjustment NOT needed |
| 1182 | As received | Metal (Stainless Steel) | Yes | Yes | Yes | re-adjustment NOT needed |
| 1243 | As received | Stone (Non-Metallic) | No | No | No | re-adjustment NOT needed |
| 1271 | After agitation (option A) | Stone (Non-Metallic) | Yes | Yes | Yes | re-adjustment NOT needed |
| 1417 | As received | Metal (Stainless Steel) | Yes | Yes | Yes | re-adjustment was needed |
| 1456 | --- | --- | --- | --- | --- | --- |
| 1660 | --- | --- | --- | --- | --- | --- |
| 1720 | --- | --- | --- | --- | --- | --- |
| 1748 | --- | --- | --- | --- | --- | --- |
| 1875 | --- | --- | --- | --- | --- | --- |
| 6016 | --- | --- | --- | --- | --- | --- |
| 6034 | --- | --- | --- | --- | --- | --- |
| 6068 | --- | --- | --- | --- | --- | --- |
| 6257 | --- | --- | --- | --- | --- | --- |
| 6277 | --- | --- | --- | --- | --- | --- |

? = I do not know

## APPENDIX 3

### Number of participants per country

2 labs in AUSTRALIA
1 lab in AUSTRIA
1 lab in AZERBAIJAN
1 lab in BELGIUM
1 lab in BOSNIA and HERZEGOVINA
1 lab in CHINA, People's Republic
1 lab in EGYPT
3 labs in GERMANY
1 lab in GREECE
1 lab in INDIA
1 lab in ITALY
1 lab in JORDAN
1 lab in KAZAKHSTAN
2 labs in NETHERLANDS
1 lab in NIGERIA
1 lab in PHILIPPINES
2 labs in POLAND
1 lab in PORTUGAL
1 lab in RUSSIAN FEDERATION
2 labs in SAUDI ARABIA
1 lab in SLOVENIA
1 lab in SPAIN
1 lab in SUDAN
2 labs in TANZANIA
1 lab in TUNISIA
1 lab in TURKEY
1 lab in UNITED KINGDOM
2 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 | = possibly an error in calculations |
| 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**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