# Results of Proficiency Test
# Grease
# September 2018

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

Author: ing. M. Meijer
Corrector: ing. A.S. Noordman-de Neef & ing. R.J. Starink
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## 1 INTRODUCTION

Grease is a solid to semifluid product. It is a mixture of an oil (often mineral), a thickener (usually a metal soap) and an additive package. This formulation provides a low viscosity at application, will thin when shear is applied and will become semisolid again when the machine stops. Grease is used in machinery that cannot be lubricated by oil, because oil would drip out, water resistance while lubricating is required or when conditions are extreme in high temperature, pressure or variation of loads. Greases can also provide water resistance, for this the formation of an emulsion by the combination of oil and soap is important.

In 2017 the Institute for Interlaboratory Studies (iis) organized a proficiency scheme for Grease for the first time. During the annual proficiency testing program 2018/2019, it was decided to continue the round robin for the analysis of Grease. In this interlaboratory study 18 laboratories in 14 different countries registered for participation. See appendix 3 for the number of participants per country. In this report, the results of the 2018 interlaboratory study on Grease 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 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 five kilograms of Grease in a plastic container, labelled #18177. 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 ISO/IEC 17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant’s data. Feedback from the participants on the reported data is encouraged and customer’s satisfaction is measured on regular basis by sending out questionnaires.

### 2.2 PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report ‘iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation’ of June 2018 (iis-protocol, version 3.5). 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 of grease was purchased from a local retailer. Twenty plastic containers from one batch of multipurpose lithium grease were labelled #18177. The homogeneity of the subsamples #18177 was checked by determination of Cone Penetration-worked in accordance with ASTM D217 on 3 stratified randomly selected samples:

| | Cone Penetration-worked in 0.1mm |
|---|---|
| Sample #18177-1 | 278 |
| Sample #18177-2 | 280 |
| Sample #18177-3 | 276 |

Table 1: homogeneity test results of subsamples #18177

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

| | Cone Penetration-worked in 0.1mm |
|---|---|
| r (observed) | 5.6 |
| reference test method | ASTM D217:17 |
| 0.3 * R (ref. test method) | 6.9 |

Table 2: evaluation of the repeatability of subsamples #18177

The calculated repeatability was less than 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneity of the subsamples #18177 was assumed.

To each of the participating laboratories one container of 5 kg, labelled #18177, was sent on September 5, 2018. An SDS was added to the sample package.

## 2.5 ANALYSES

The participants were asked to determine on sample #18177; Cone Penetration (unworked, worked and prolonged), Copper Corrosion 24 hrs at 100°C, Dropping Point, Extreme Pressure Properties (four-ball method), Leakage amount, Oil Separation (conical sieve), Oxidation Stability (100 hr) Pressure drop, Roll Stability Penetration Change (¼ and ½ scale penetrometer), Water by KF, Water Spray-Off, Water Washout at 79°C, Wear Preventative Characteristics, Elements as Aluminum, Antimony, Barium, Calcium, Iron, Lithium, Magnesium, Molybdenum, Phosphorus, Silicon, Sodium, Sulfur and Zinc.

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 results, but report as much significant figures as possible. It was also requested not to report ‘less than’ results, which are above the detection limit, because such 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 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 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 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 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. 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 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 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. 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:

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

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 problems were encountered with the dispatch of the samples. Three participants reported the test results after the final reporting date and one participant did not report any test result at all. Not all laboratories were able to report all analyses requested.
The 17 reporting participants sent in 124 numerical test results. Observed was 1 outlying test result, which is 0.8% 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 test methods, which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the reported test results. The abbreviations, used in these tables, are listed in appendix 3.

In the iis PT reports, ASTM methods are referred to with a number (e.g. D2266) and an added designation for the year that the method was adopted or revised (e.g. D2266:01). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D2266:01(2015)). In the results tables of Appendix 1 only the method number and year of adoption or revision e.g. D2266:01 is used.

The original data sets proved to have a normal Gaussian distribution. Or it was unknown due to the limited number of test results. The statistical evaluation of these data sets should be used with due care.

Cone Penetration unworked: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility was in agreement with the requirements of ASTM D217:17.

Cone Penetration worked: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility was in agreement with the requirements of ASTM D217:17.

Cone Penetration prolonged: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility was in agreement with the requirements of ASTM D217:17.

Copper Corrosion: This determination was not problematic. Eight participants reported a test result. Five participants agreed on a test result of 1 or 1a. Two participants reported a "Pass" and one participant reported a test result of 3A which is deviating from the other participants.

Dropping Point: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements of ASTM D2265:15e1. Therefore, it was decided not to calculate z-scores.

Extreme Pressure Properties – Weld Point: This determination was not problematic. The test results of the three reporting participants fall within one increment step. The reproducibility is in agreement with the requirements of ASTM D2596:15.

Extreme Pressure Properties – Load Wear Index: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D2596:15.

Extreme Pressure Properties – Last Non-Seizure Load: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D2596:15.

Oil Separation-Conical Sieve: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D6184:17.

Water by KF: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D6304:16e1.

Water washout: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D1264:18.

Wear Preventative Characteristics: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D2266:01(2015).

There were only one or two test results reported for the determination of Leakage amount, Oxidation stability, Roll Stability – Penetration change ¼ scale penetrometer and ½ scale penetrometer and Water Spray-off. These tests were not evaluated. The reported test results are summarized in appendix 2.

With respect to elemental analyses; Test Method ASTM D7303:17 states in section 5.2: “Although widely used in other sectors of the oil industry for metal analysis, ICP-AES based Test Methods D4951 or D5185 cannot be used for analyzing greases because of their insolubility in organic solvents used in these test methods. Hence, grease samples need to be brought into aqueous solution by acid decomposition before ICP-AES measurements.” Therefore, it was decided to exclude test results from test method ASTM D5185.

Calcium: This determination was not problematic. One statistical outlier was observed and three other test results were excluded. The calculated reproducibility after rejection of the suspect data is in agreement with the requirements of ASTM D7303:17.

Lithium: This determination may be problematic. No statistical outliers were observed. Two test results were excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of ASTM D7303:17. It was decided not to calculate z-scores due to the low number of test results.

Phosphorus: This determination was not problematic. No statistical outliers were observed. Two test results were excluded. The calculated reproducibility is in agreement with the requirements of ASTM D7303:17.

Sulfur: This determination was problematic. No statistical outliers were observed. Two test results were excluded. The calculated reproducibility is not in agreement with the requirements of ASTM D7303:17.

Zinc: This determination was problematic. No statistical outliers were observed. Three test results were excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of ASTM D7303:17.

For Aluminum, Antimony, Barium, Iron, Magnesium, Molybdenum, Silicon and Sodium the majority of the participants agreed on a concentration near or below the limit of detection. Therefore, it was decided not to calculate z-scores for these determinations.

## 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method and the reproducibility as found for the group of participating laboratories that participated. The average results, calculated reproducibilities and reproducibilities derived from reference test methods are compared in the next table:

| Parameter | unit | n | average | 2.8 * sd | R (lit) |
|---|---|---|---|---|---|
| Cone Penetration - unworked | 0.1 mm | 11 | 275 | 17 | 22 |
| Cone Penetration - worked | 0.1 mm | 12 | 274 | 17 | 23 |
| Cone Penetration – prolonged | 0.1 mm | 7 | 287 | 19 | 29 |
| Copper Corrosion 24 hrs at 100°C | | 7 | 1a | n.a. | n.a. |

| Parameter | unit | n | average | 2.8 * sd | R (lit) |
|---|---|---|---|---|---|
| Dropping Point | °C | 9 | 203 | 29 | 12 |
| **Extreme-Pressure Properties (four-ball method)** | | | | | |
| - Weld Point | kgf | 3 | 200-250 | n.a. | 1 increment |
| - Load Wear Index | kgf | 3 | 42.7 | 11.4 | 18.8 |
| - Last Non-Seizure Load | kgf | 3 | 93.3 | 32.3 | 72.8 |
| Leakage amount | g | 1 | n.a. | n.a. | n.a. |
| Oil separation – Conical Sieve | %M/M | 5 | 2.5 | 2.4 | 2.4 |
| Oxidation stability - pressure drop | kPa | 1 | n.a. | n.a. | n.a. |
| **Roll Stability – Penetration change** | | | | | |
| - ¼ scale penetrometer | 0.1 mm | 2 | n.a. | n.a. | n.a. |
| - ½ scale penetrometer | 0.1 mm | 1 | n.a. | n.a. | n.a. |
| Water by KF | mg/kg | 6 | 1055 | 1058 | 1100 |
| Water Spray-off | %M/M | 1 | n.a. | n.a. | n.a. |
| Water Washout at 79°C | %M/M | 3 | 2.16 | 1.03 | 7.44 |
| Wear Preventative Characteristics | mm | 3 | 0.43 | 0.05 | 0.37 |
| Calcium as Ca | mg/kg | 3 | 718 | 251 | 319 |
| Lithium as Li | mg/kg | 2 | (2673) | (916) | (521) |
| Phosphorus as P | mg/kg | 4 | 850 | 298 | 383 |
| Sulfur as S | mg/kg | 3 | 11464 | 7925 | 4822 |
| Zinc as Zn | mg/kg | 3 | 1714 | 800 | 359 |

Table 3: reproducibilities of tests on sample #18177
Results between brackets is based on a low number of data.

Without further statistical calculations it can be concluded that for many tests there is a good compliance of the group of participating laboratories with the reference test methods. The problematic tests have been discussed in paragraph 4.1. Unfortunately, not all laboratories performed all tests resulting in a low number of results for several tests.

## 4.3 COMPARISON OF THE PROFICIENCY TEST OF SEPTEMBER 2018 WITH PREVIOUS PT

| | September 2018 | September 2017 |
|---|---|---|
| Number of reporting labs | 17 | 14 |
| Number of test results | 124 | 92 |
| Statistical outliers | 1 | 4 |
| Percentage outliers | 0.8% | 4.3% |

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 | September 2018 | September 2017 |
|---|---|---|
| Cone Penetration - unworked | + | +/- |
| Cone Penetration - worked | + | +/- |
| Cone Penetration – prolonged work | + | + |
| Dropping Point | -- | - |
| Weld Point | +/- | + |
| Load Wear Index | + | n.e. |
| Last Non-Seizure Load | ++ | + |
| Leakage amount | n.e. | n.e. |
| Oil separation – Conical Sieve | +/- | + |
| Oxidation stability - pressure drop | n.e. | n.e. |
| ¼ scale penetrometer | n.e. | n.e. |
| ½ scale penetrometer | n.e. | n.e. |
| Water by KF | +/- | +/- |
| Water Spray-off | n.e. | n.e. |
| Water Washout at 79°C | ++ | ++ |
| Wear Preventative Characteristics | ++ | ++ |
| Calcium as Ca | + | - |
| Lithium as Li | - | - |
| Phosphorus as P | + | n.e. |
| Sulfur as S | - | n.e. |
| Zinc as Zn | -- | n.e. |

Table 5: comparison determinations against the reference test method

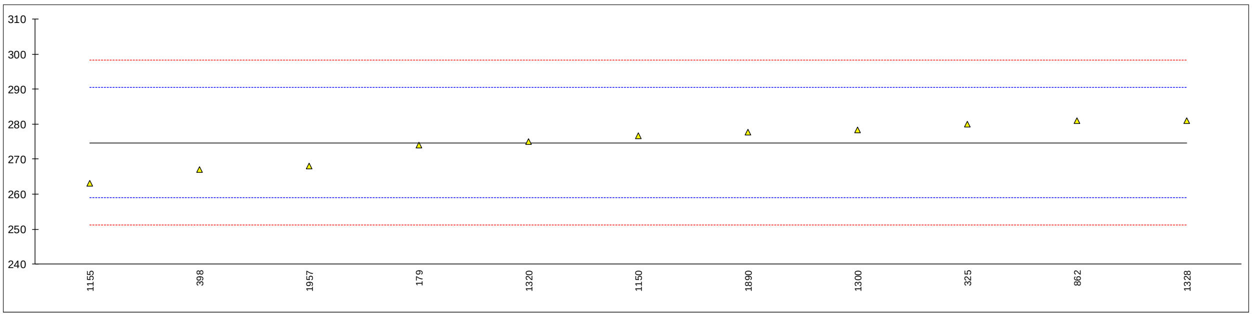
The performance of the determinations against the requirements of the respective reference test methods is listed in the above table. The following performance categories were used:

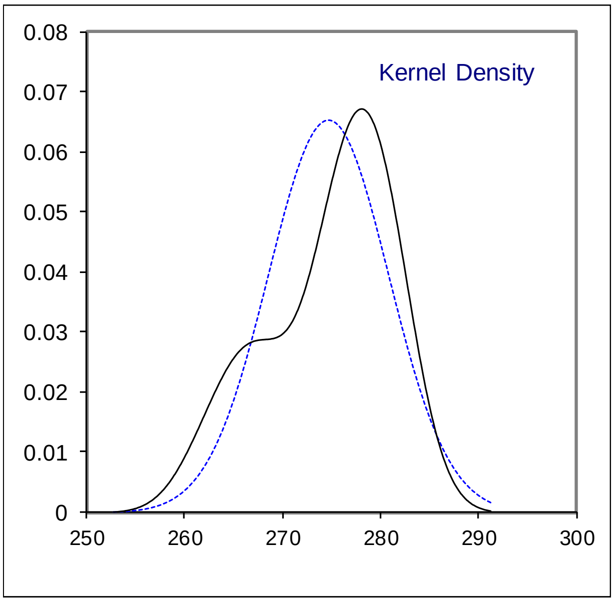
++: group performed much better than the reference test method
+ : group performed better than the reference test method
+/-: group performance equals the reference test method
- : group performed worse than the reference test method
-- : group performed much worse than the reference test method
n.e.: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

## APPENDIX 1

Determination of Cone Penetration - unworked on sample #18177; results in 0.1 mm

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 179 | D217 | 274 | | -0.09 | |
| 325 | D217 | 280 | | 0.68 | |
| 349 | | ----- | | ----- | |
| 398 | D217 | 267 | | -0.98 | |
| 603 | | ----- | | ----- | |
| 862 | D217 | 281 | | 0.80 | |
| 962 | | ----- | | ----- | |
| 1150 | ISO2137 | 276.6 | | 0.24 | |
| 1155 | D217 | 263 | | -1.49 | |
| 1300 | D217 | 278.3 | | 0.46 | |
| 1320 | ISO2137 | 275 | | 0.04 | |
| 1328 | GB/T269 | 281 | | 0.80 | |
| 1330 | | ----- | | ----- | |
| 1367 | | ----- | | ----- | |
| 1890 | ISO2137 | 277.7 | | 0.38 | |
| 1957 | D217 | 268.0 | | -0.85 | |
| 6068 | | ----- | | ----- | |
| 6206 | | ----- | | ----- | |
| | normality | OK | | | |
| | n | 11 | | | |
| | outliers | 0 | | | |
| | mean (n) | 274.69 | | | |
| | st.dev. (n) | 6.121 | | | |
| | R(calc.) | 17.14 | | | |
| | st.dev.(D217:17) | 7.857 | | | |
| | R(D217:17) | 22 | | | |

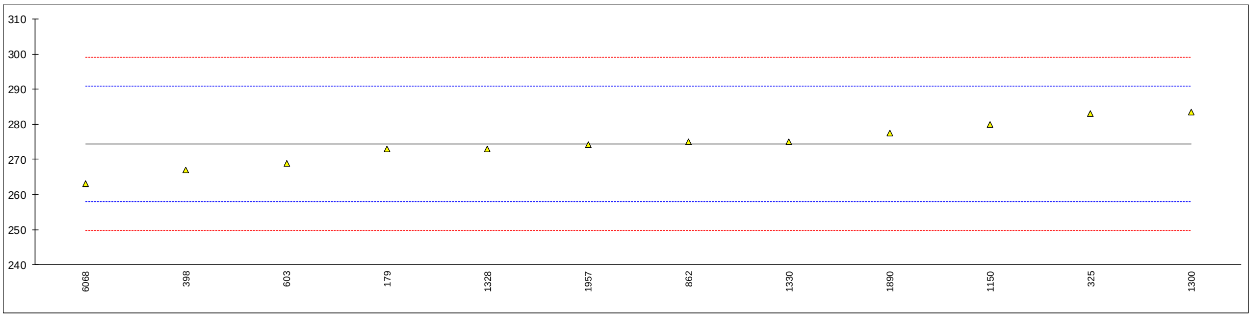


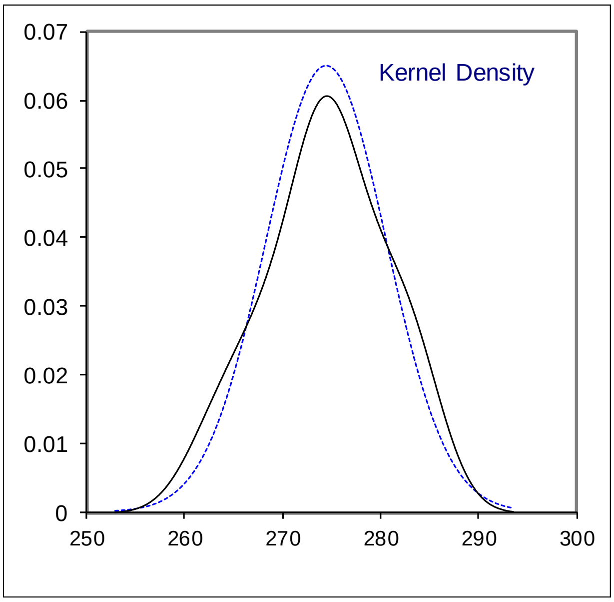



## Determination of Cone Penetration - worked on sample #18177; results in 0.1 mm

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 179 | D217 | 273 | | -0.17 | |
| 325 | D217 | 283 | | 1.04 | |
| 349 | | ----- | | ----- | |
| 398 | D217 | 267 | | -0.90 | |
| 603 | D217 | 268.9 | | -0.67 | |
| 862 | D217 | 275 | | 0.07 | |
| 962 | | ----- | | ----- | |
| 1150 | ISO2137 | 280 | | 0.68 | |
| 1155 | | ----- | | ----- | |
| 1300 | D217 | 283.4 | | 1.09 | |
| 1320 | | ----- | | ----- | |
| 1328 | GB/T269 | 273 | | -0.17 | |
| 1330 | D217 | 275 | | 0.07 | |
| 1367 | | ----- | | ----- | |
| 1890 | ISO2137 | 277.5 | | 0.38 | |
| 1957 | D217 | 274.2 | | -0.03 | |
| 6068 | ISO2137 | 263 | | -1.39 | |
| 6206 | | ----- | | ----- | |

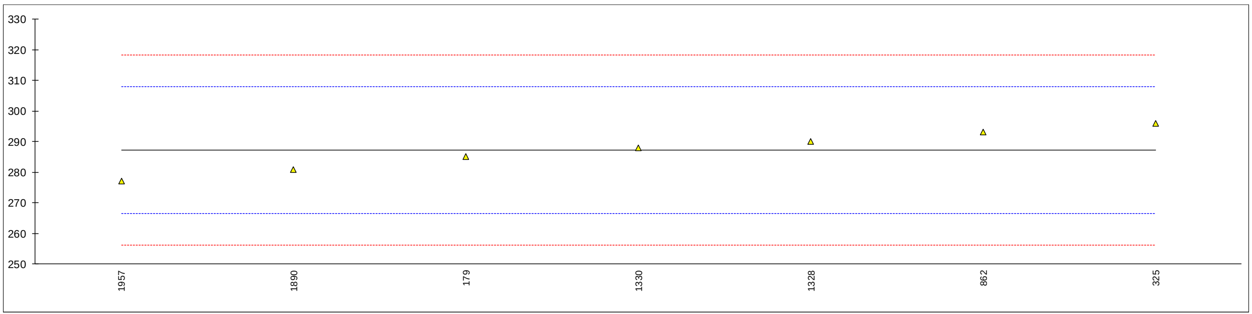
| | |
|---|---|
| normality | OK |
| n | 12 |
| outliers | 0 |
| mean (n) | 274.42 |
| st.dev. (n) | 6.147 |
| R(calc.) | 17.21 |
| st.dev.(D217:17) | 8.214 |
| R(D217:17) | 23 |

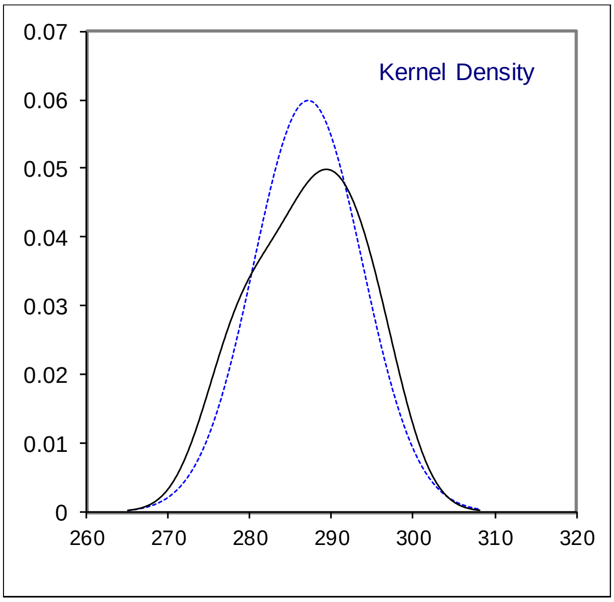


Determination of Cone Penetration – prolonged work on sample #18177; results in 0.1 mm

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 179 | D217 | 285 | | -0.21 | |
| 325 | D217 | 296 | | 0.86 | |
| 349 | | ----- | | ----- | |
| 398 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 862 | D217 | 293 | | 0.57 | |
| 962 | | ----- | | ----- | |
| 1150 | | ----- | | ----- | |
| 1155 | | ----- | | ----- | |
| 1300 | | ----- | | ----- | |
| 1320 | | ----- | | ----- | |
| 1328 | GB/T269 | 290 | | 0.28 | |
| 1330 | D217 | 288 | | 0.08 | |
| 1367 | | ----- | | ----- | |
| 1890 | ISO2137 | 280.8 | | -0.61 | |
| 1957 | D217 | 277.2 | | -0.96 | |
| 6068 | | ----- | | ----- | |
| 6206 | | ----- | | ----- | |
| | normality | unknown | | | |
| | n | 7 | | | |
| | outliers | 0 | | | |
| | mean (n) | 287.14 | | | |
| | st.dev. (n) | 6.650 | | | |
| | R(calc.) | 18.62 | | | |
| | st.dev.(D217:17) | 10.357 | | | |
| | R(D217:17) | 29 | | | |



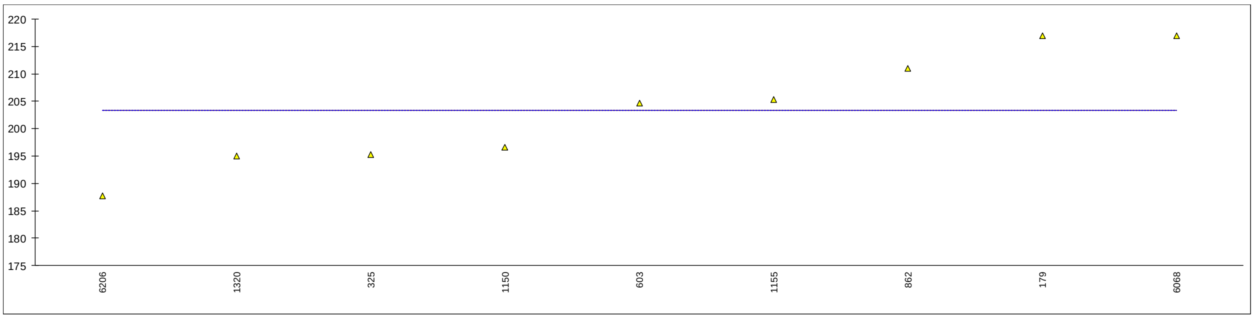



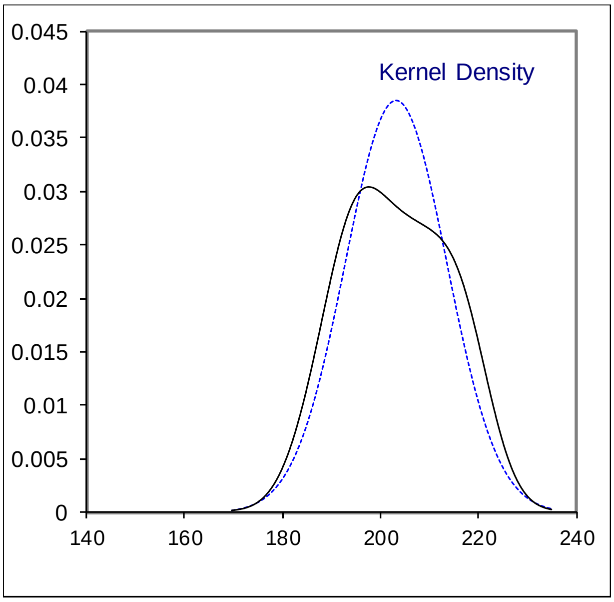
# Determination of Copper Corrosion 24 hrs at 100°C on sample #18177

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 179 | D4048 | 1A | | ----- | |
| 325 | DIN51811 | 3A | | ----- | outlier: test result deviating from the other participants |
| 349 | | ----- | | ----- | |
| 398 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 862 | D4048 | 1a | | ----- | |
| 962 | | ----- | | ----- | |
| 1150 | | ----- | | ----- | |
| 1155 | D4048 | 1a | | ----- | |
| 1300 | D130 | 1 | | ----- | |
| 1320 | | ----- | | ----- | |
| 1328 | | PASS | | ----- | |
| 1330 | D4048 | pass? | | ----- | reported: 合格 |
| 1367 | | ----- | | ----- | |
| 1890 | | ----- | | ----- | |
| 1957 | D4048 | 1a | | ----- | |
| 6068 | | ----- | | ----- | |
| 6206 | | ----- | | ----- | |
| | | | | | |
| | n | 7 | | | |
| | outliers | 1 | | | |
| | mean (n) | 1a/ pass | | | |

## Determination of Dropping Point on sample #18177; results in °C

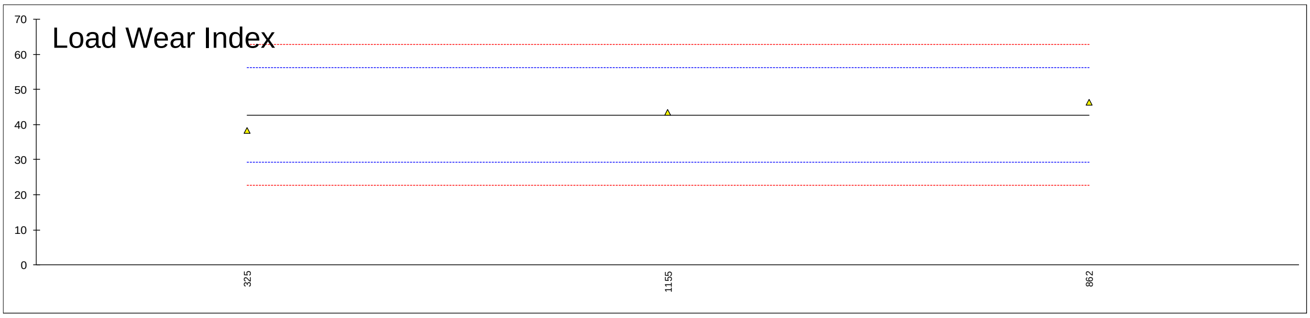
| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 179 | D2265 | 217.0 | | ----- | |
| 325 | INH-396/566 | 195.3 | | ----- | |
| 349 | | ----- | | ----- | |
| 398 | | ----- | | ----- | |
| 603 | D2265 | 204.7 | | ----- | |
| 862 | D2265 | 211 | | ----- | |
| 962 | | ----- | | ----- | |
| 1150 | ISO2176 | 196.6 | | ----- | |
| 1155 | D2265 | 205.3 | | ----- | |
| 1300 | | ----- | | ----- | |
| 1320 | D2265 | 195 | | ----- | |
| 1328 | | ----- | | ----- | |
| 1330 | | ----- | | ----- | |
| 1367 | | ----- | | ----- | |
| 1890 | | ----- | | ----- | |
| 1957 | | ----- | W | ----- | test result withdrawn, reported 186.7 |
| 6068 | ISO6299 | 217 | | ----- | |
| 6206 | D3954 | 187.8 | | ----- | |
| | | | | | |
| | normality | OK | | | |
| | n | 9 | | | |
| | outliers | 0 | | | |
| | mean (n) | 203.30 | | | |
| | st.dev. (n) | 10.360 | | | |
| | R(calc.) | 29.01 | | | |
| | st.dev.(D2265:15e1) | (4.286) | | | |
| | R(D2265:15e1) | (12) | | | |

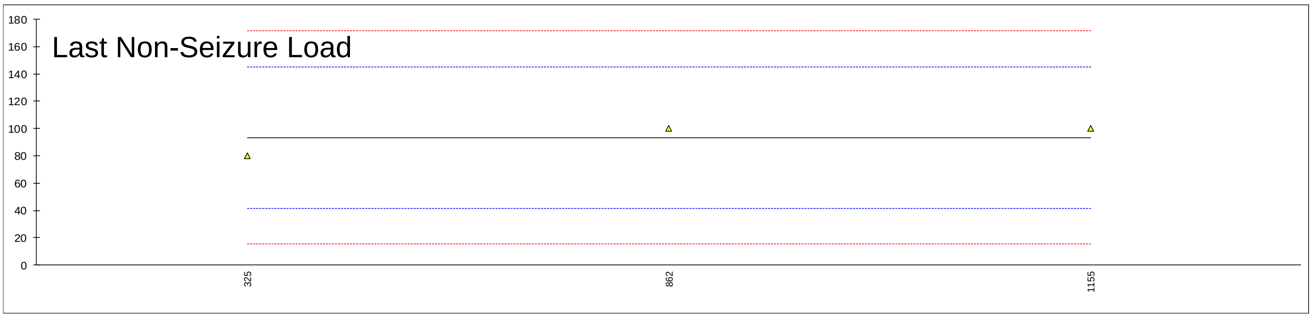


Determination of Extreme-Pressure Properties (four-ball method) on sample #18177; Weld Point, Load Wear Index (LWI) and Last Non-Seizure Load results in kgf

| lab | method | Weld Point | mark | z(targ) | LWI | mark | z(targ) | Last Non-Seizure Load | mark | z(targ) |
|---|---|---|---|---|---|---|---|---|---|---|
| 179 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 325 | D2596 | 250 | | ----- | 38.35 | | -0.65 | 80 | | -0.51 |
| 349 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 398 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 603 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 862 | D2596 | 250 | | ----- | 46.4 | | 0.54 | 100 | | 0.26 |
| 962 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1150 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1155 | D2596 | 200 | | ----- | 43.49 | | 0.11 | 100 | | 0.26 |
| 1300 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1320 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1328 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1330 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1367 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1890 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1957 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 6068 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 6206 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| | normality | | | | unknown | | | unknown | | |
| | n | | | | 3 | | | 3 | | |
| | outliers | | | | 0 | | | 0 | | |
| | mean (n) | | | | 42.7 | | | 93.3 | | |
| | st.dev. (n) | | | | 4.08 | | | 11.55 | | |
| | R(calc.) | | | | 11.4 | | | 32.3 | | |
| | st.dev.(D2596:15) | | | | 6.72 | | | 26.00 | | |
| | R(D2596:15) | | | | 18.8 | | | 72.8 | | |

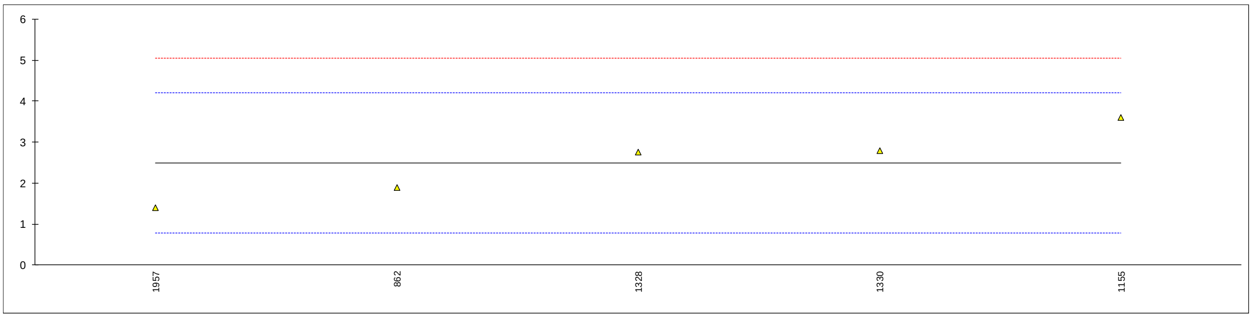


# Determination of Oil separation – Conical Sieve on sample #18177; results in %M/M

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 179 | | ----- | | ----- | |
| 325 | | ----- | | ----- | |
| 349 | | ----- | | ----- | |
| 398 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 862 | D6184 | 1.9 | | -0.69 | |
| 962 | | ----- | | ----- | |
| 1150 | | ----- | | ----- | |
| 1155 | BS200-121 | 3.6 | | 1.30 | |
| 1300 | | ----- | | ----- | |
| 1320 | | ----- | | ----- | |
| 1328 | NB/SH/T0324 | 2.75 | | 0.30 | |
| 1330 | D6184 | 2.8 | | 0.36 | |
| 1367 | | ----- | | ----- | |
| 1890 | | ----- | | ----- | |
| 1957 | D6184 | 1.40 | | -1.27 | |
| 6068 | | ----- | | ----- | |
| 6206 | | ----- | | ----- | |

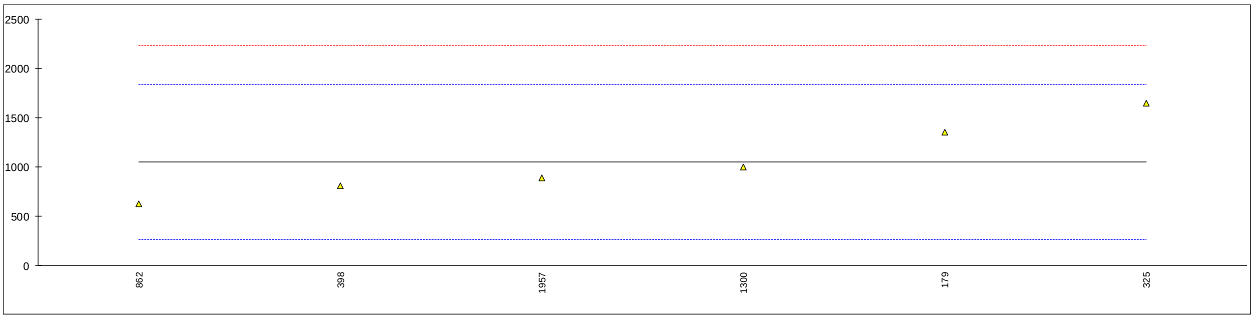
| | |
|---|---|
| normality | unknown |
| n | 5 |
| outliers | 0 |
| mean (n) | 2.49 |
| st.dev. (n) | 0.856 |
| R(calc.) | 2.40 |
| st.dev.(D6184:17) | 0.855 |
| R(D6184:17) | 2.39 |



# Determination of Water by KF on sample #18177; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 179 | D6304-C | 1356 | | 0.77 | |
| 325 | D6304-C | 1647 | | 1.51 | |
| 349 | | ----- | | ----- | |
| 398 | D6304-C | 810 | | -0.62 | |
| 603 | | ----- | | ----- | |
| 862 | D6304-C | 627 | | -1.09 | |
| 962 | | ----- | | ----- | |
| 1150 | | ----- | | ----- | |
| 1155 | | ----- | | ----- | |
| 1300 | D6304-A | 998 | | -0.14 | |
| 1320 | | ----- | | ----- | |
| 1328 | | ----- | | ----- | |
| 1330 | | ----- | | ----- | |
| 1367 | | ----- | | ----- | |
| 1890 | | ----- | | ----- | |
| 1957 | D6304-C | 891.4 | | -0.42 | |
| 6068 | | ----- | | ----- | |
| 6206 | | ----- | | ----- | |

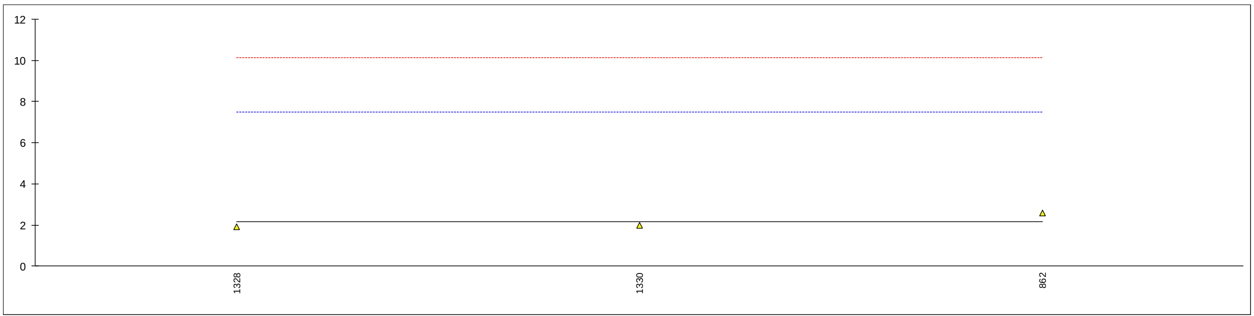
| | |
|---|---|
| normality | unknown |
| n | 6 |
| outliers | 0 |
| mean (n) | 1054.9 |
| st.dev. (n) | 377.97 |
| R(calc.) | 1058.3 |
| st.dev.(D6304:16e1) | 393.04 |
| R(D6304:16e1) | 1100.5 |



# Determination of Water Washout at 79°C on sample #18177; results in %M/M

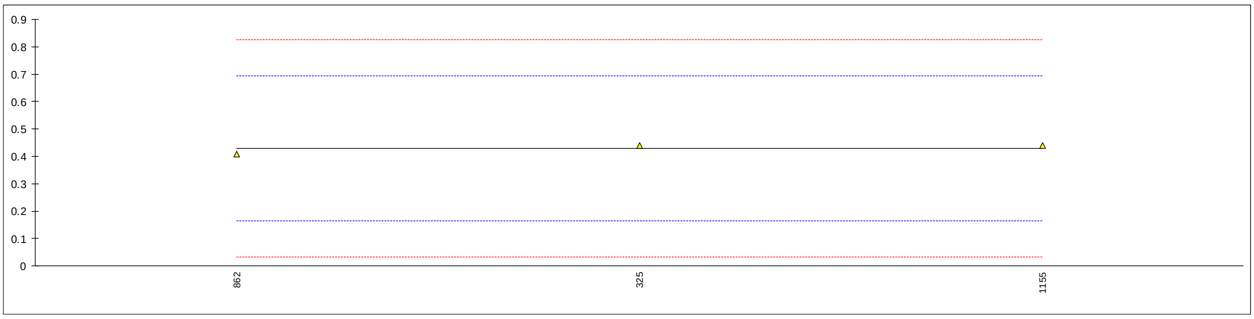
| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 179 | | ----- | | ----- | |
| 325 | | ----- | | ----- | |
| 349 | | ----- | | ----- | |
| 398 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 862 | D1264 | 2.58 | | 0.16 | |
| 962 | | ----- | | ----- | |
| 1150 | | ----- | | ----- | |
| 1155 | | ----- | | ----- | |
| 1300 | | ----- | | ----- | |
| 1320 | | ----- | | ----- | |
| 1328 | SH/T0109 | 1.9 | | -0.10 | |
| 1330 | D1264 | 2 | | -0.06 | |
| 1367 | | ----- | | ----- | |
| 1890 | | ----- | | ----- | |
| 1957 | | ----- | | ----- | |
| 6068 | | ----- | | ----- | |
| 6206 | | ----- | | ----- | |

| | |
|---|---|
| normality | unknown |
| n | 3 |
| outliers | 0 |
| mean (n) | 2.160 |
| st.dev. (n) | 0.3672 |
| R(calc.) | 1.028 |
| st.dev.(D1264:18) | 2.6557 |
| R(D1264:18) | 7.436 |



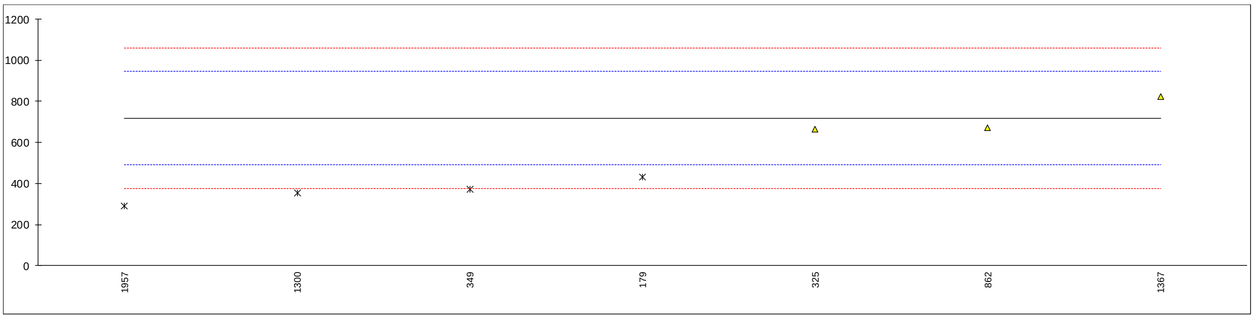
Determination of Wear Preventative Characteristics on sample #18177; results in mm

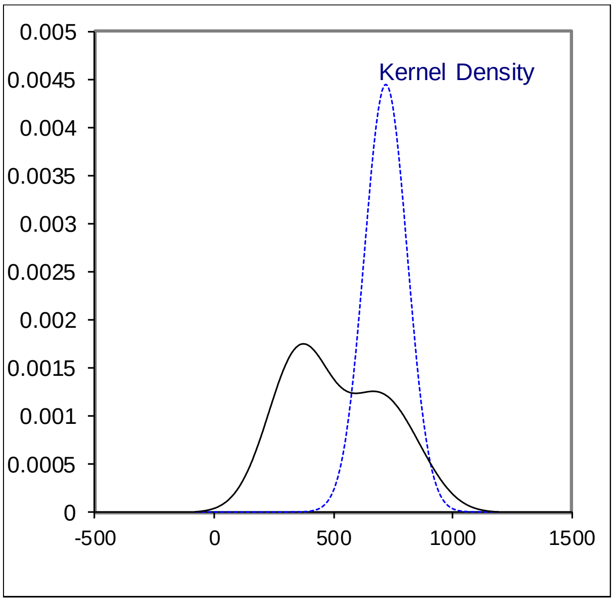
| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 179 | | ----- | | ----- | |
| 325 | D2266 | 0.44 | | 0.08 | |
| 349 | | ----- | | ----- | |
| 398 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 862 | D2266 | 0.407 | | -0.17 | |
| 962 | | ----- | | ----- | |
| 1150 | | ----- | | ----- | |
| 1155 | D2266 | 0.44 | | 0.08 | |
| 1300 | | ----- | | ----- | |
| 1320 | | ----- | | ----- | |
| 1328 | | ----- | | ----- | |
| 1330 | | ----- | | ----- | |
| 1367 | | ----- | | ----- | |
| 1890 | | ----- | | ----- | |
| 1957 | | ----- | | ----- | |
| 6068 | | ----- | | ----- | |
| 6206 | | ----- | | ----- | |
| | | | | | |
| | normality | unknown | | | |
| | n | 3 | | | |
| | outliers | 0 | | | |
| | mean (n) | 0.429 | | | |
| | st.dev. (n) | 0.0191 | | | |
| | R(calc.) | 0.053 | | | |
| | st.dev.(D2266:01) | 0.1321 | | | |
| | R(D2266:01) | 0.370 | | | |



## Determination of Calcium as Ca on sample #18177; results in mg/kg

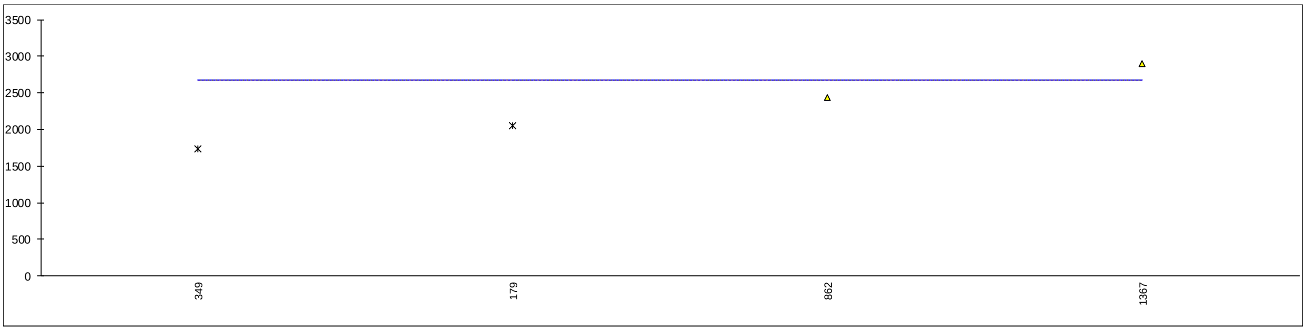
| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 179 | D5185 | 433 | ex | -2.50 | see paragraph 4.1 |
| 325 | In house | 663 | | -0.48 | |
| 349 | D5185 | 370 | ex | -3.06 | see paragraph 4.1 |
| 398 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 862 | D7303 | 670 | | -0.42 | |
| 962 | | ----- | | ----- | |
| 1150 | | ----- | | ----- | |
| 1155 | | ----- | | ----- | |
| 1300 | D5185 | 355.1 | ex | -3.19 | see paragraph 4.1 |
| 1320 | | ----- | | ----- | |
| 1328 | | ----- | | ----- | |
| 1330 | | ----- | | ----- | |
| 1367 | D7303 | 821.64 | | 0.91 | |
| 1890 | | ----- | | ----- | |
| 1957 | | 289 | D(0.05) | -3.77 | |
| 6068 | | ----- | | ----- | |
| 6206 | | ----- | | ----- | |
| | normality | unknown | | | |
| | n | 3 | | | |
| | outliers | 1 (+3ex) | | | |
| | mean (n) | 718.2 | | | |
| | st.dev. (n) | 89.64 | | | |
| | R(calc.) | 251.0 | | | |
| | st.dev.(D7303:17) | 113.94 | | | |
| | R(D7303:17) | 319.0 | | | application range D7303: 20-50000 mg/kg |



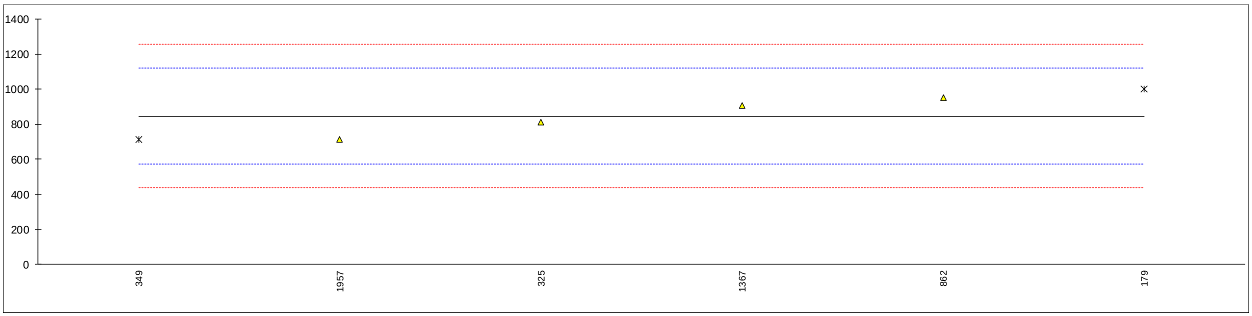
## Determination of Lithium as Li on sample #18177; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 179 | D5185 | 2048 | ex | ----- | see paragraph 4.1 |
| 325 | | ----- | | ----- | |
| 349 | D5185 | 1741 | ex | ----- | see paragraph 4.1 |
| 398 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 862 | D7303 | 2442 | | ----- | |
| 962 | | ----- | | ----- | |
| 1150 | | ----- | | ----- | |
| 1155 | | ----- | | ----- | |
| 1300 | | ----- | | ----- | |
| 1320 | | ----- | | ----- | |
| 1328 | | ----- | | ----- | |
| 1330 | | ----- | | ----- | |
| 1367 | D7303 | 2904.46 | | ----- | |
| 1890 | | ----- | | ----- | |
| 1957 | | ----- | | ----- | |
| 6068 | | ----- | | ----- | |
| 6206 | | ----- | | ----- | |
| | | | | | |
| | normality | unknown | | | |
| | n | 2 | | | |
| | outliers | 0 (+2ex) | | | |
| | mean (n) | 2673.2 | | | |
| | st.dev. (n) | 327.01 | | | |
| | R(calc.) | 915.62 | | | |
| | st.dev.(D7303:17) | (186.04) | | | |
| | R(D7303:17) | (520.9) | | | application range D7303: 300-3200 mg/kg |



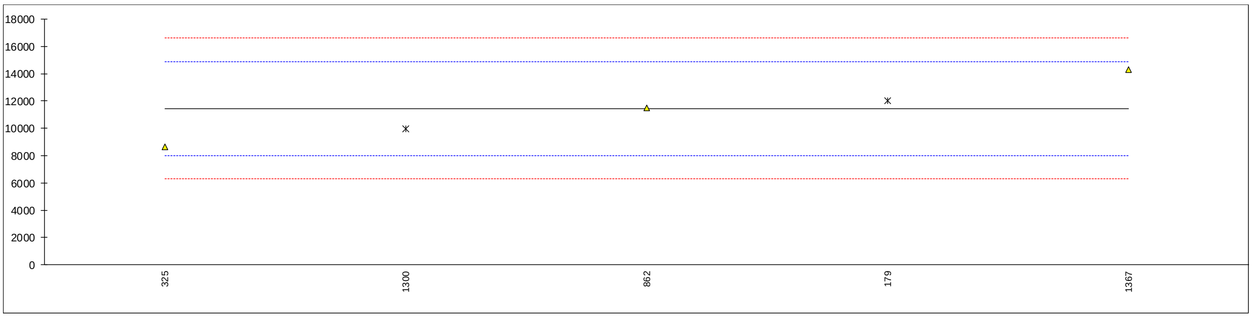
# Determination of Phosphorus as P on sample #18177; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 179 | D5185 | 1001 | ex | 1.13 | see paragraph 4.1 |
| 325 | In house | 813 | | -0.24 | |
| 349 | D5185 | 712 | ex | -0.98 | see paragraph 4.1 |
| 398 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 862 | D7303 | 952 | | 0.77 | |
| 962 | | ----- | | ----- | |
| 1150 | | ----- | | ----- | |
| 1155 | | ----- | | ----- | |
| 1300 | D5185 | <0.1 | | <-6.18 | possibly a false negative test result? |
| 1320 | | ----- | | ----- | |
| 1328 | | ----- | | ----- | |
| 1330 | | ----- | | ----- | |
| 1367 | D7303 | 906.78 | | 0.44 | |
| 1890 | | ----- | | ----- | |
| 1957 | | 712 | | -0.98 | |
| 6068 | | ----- | | ----- | |
| 6206 | | ----- | | ----- | |
| | | | | | |
| | normality | unknown | | | |
| | n | 4 | | | |
| | outliers | 0 (+2ex) | | | |
| | mean (n) | 845.9 | | | |
| | st.dev. (n) | 106.42 | | | |
| | R(calc.) | 298.0 | | | |
| | st.dev.(D7303:17) | 136.80 | | | |
| | R(D7303:17) | 383.0 | | | application range D7303: 50-2000 mg/kg |



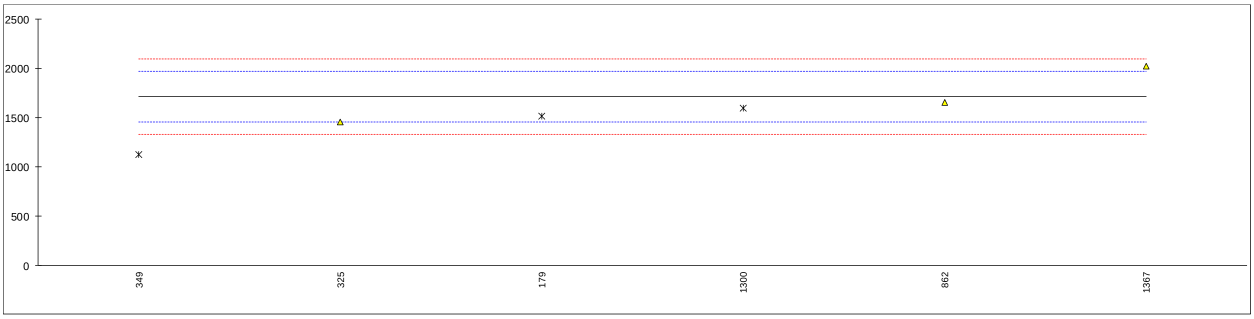
## Determination of Sulfur as S on sample #18177; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 179 | D5185 | 12012 | ex | 0.32 | see paragraph 4.1 |
| 325 | In house | 8630 | | -1.65 | |
| 349 | | ----- | | ----- | |
| 398 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 862 | D7303 | 11470 | | 0.00 | |
| 962 | | ----- | | ----- | |
| 1150 | | ----- | | ----- | |
| 1155 | | ----- | | ----- | |
| 1300 | D5185 | 9975.9 | ex | -0.86 | see paragraph 4.1 |
| 1320 | | ----- | | ----- | |
| 1328 | | ----- | | ----- | |
| 1330 | | ----- | | ----- | |
| 1367 | D7303 | 14290.88 | | 1.64 | |
| 1890 | | ----- | | ----- | |
| 1957 | | ----- | | ----- | |
| 6068 | | ----- | | ----- | |
| 6206 | | ----- | | ----- | |
| | | | | | |
| | normality | unknown | | | |
| | n | 3 | | | |
| | outliers | 0 (+2ex) | | | |
| | mean (n) | 11463.6 | | | |
| | st.dev. (n) | 2830.45 | | | |
| | R(calc.) | 7925.2 | | | |
| | st.dev.(D7303:17) | 1722.17 | | | |
| | R(D7303:17) | 4822.1 | | | application range D7303: 1600-28000 mg/kg |



## Determination of Zinc as Zn on sample #18177; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 179 | D5185 | 1516 | ex | -1.54 | see paragraph 4.1 |
| 325 | In house | 1460 | | -1.98 | |
| 349 | D5185 | 1130 | ex | -4.55 | see paragraph 4.1 |
| 398 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 862 | D7303 | 1658 | | -0.43 | |
| 962 | | ----- | | ----- | |
| 1150 | | ----- | | ----- | |
| 1155 | | ----- | | ----- | |
| 1300 | D5185 | 1593.6 | ex | -0.94 | see paragraph 4.1 |
| 1320 | | ----- | | ----- | |
| 1328 | | ----- | | ----- | |
| 1330 | | ----- | | ----- | |
| 1367 | D7303 | 2023.18 | | 2.41 | |
| 1890 | | ----- | | ----- | |
| 1957 | | ----- | W | ----- | test result withdrawn, reported 1006 |
| 6068 | | ----- | | ----- | |
| 6206 | | ----- | | ----- | |
| | normality | unknown | | | |
| | n | 3 | | | |
| | outliers | 0 (+3ex) | | | |
| | mean (n) | 1713.7 | | | |
| | st.dev. (n) | 285.70 | | | |
| | R(calc.) | 799.9 | | | |
| | st.dev.(D7303:17) | 128.24 | | | |
| | R(D7303:17) | 359.1 | | | application range D7303: 300-2200 mg/kg |



## Appendix 2: Other reported test results

Determination of Leakage amount (g), Oxidation stability (100 hr), Roll Stability – Penetration change ¼ scale penetrometer and ½ scale penetrometer (0.1 mm) and Water Spray-off (%M/M) on sample #18177

| lab | Leakage amount | Oxidation Stab. | Roll stability ¼ | Roll stability ½ | Water Spray-off |
|---|---|---|---|---|---|
| 179 | ---- | ---- | ---- | ---- | ---- |
| 325 | ---- | ---- | ---- | 131.6 | ---- |
| 349 | ---- | ---- | ---- | ---- | ---- |
| 398 | ---- | ---- | ---- | ---- | ---- |
| 603 | ---- | ---- | ---- | ---- | ---- |
| 862 | ---- | 11.1 | 5.6 | ---- | ---- |
| 962 | ---- | ---- | ---- | ---- | ---- |
| 1150 | ---- | ---- | ---- | ---- | ---- |
| 1155 | ---- | ---- | ---- | ---- | ---- |
| 1300 | ---- | ---- | ---- | ---- | ---- |
| 1320 | ---- | ---- | ---- | ---- | ---- |
| 1328 | 0.8 | ---- | 1 | ---- | 30.86 |
| 1330 | ---- | ---- | ---- | ---- | ---- |
| 1367 | ---- | ---- | ---- | ---- | ---- |
| 1890 | ---- | ---- | ---- | ---- | ---- |
| 1957 | ---- | ---- | ---- | ---- | ---- |
| 6068 | ---- | ---- | ---- | ---- | ---- |
| 6206 | ---- | ---- | ---- | ---- | ---- |

All reported test results of Aluminum, Antimony, Barium, Iron, Magnesium, Molybdenum, Silicon and Sodium on sample #18177; results in mg/kg

| Lab | Al | Sb | Ba | Fe | Mg | Mo | Si | Na |
|---|---|---|---|---|---|---|---|---|
| 179 | <1 | <1 | <1 | <1 | 1 | 3 | 1 | 4 |
| 325 | <20 | <20 | <20 | <20 | <20 | <20 | <20 | <20 |
| 349 | 7 | 0 | 5 | 3 | 5 | 15 | 6 | 0 |
| 398 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 603 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 862 | <10 | <10 | <50 | <10 | <30 | <50 | <10 | <30 |
| 962 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 1150 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 1155 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 1300 | 0.44 | 0.15 | 0.05 | 0.65 | 1.49 | 1.84 | 263.8 C | <0.1 |
| 1320 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 1328 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 1330 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 1367 | 1.89 | ----- | 0.02 | 2.60 | 5.93 | ----- | 2.70 | 18.37 |
| 1890 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 1957 | <1 | ----- | <1 | <1 | <1 | <1 | <1 | <1 |
| 6068 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 6206 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |

Lab 1300 first reported 172.1; possibly a false positive test result?

## APPENDIX 3

### Number of participants per country

2 labs in BELGIUM
1 lab in BULGARIA
3 labs in CHINA, People's Republic
1 lab in ESTONIA
1 lab in GERMANY
1 lab in ITALY
2 labs in MALAYSIA
1 lab in POLAND
1 lab in SAUDI ARABIA
1 lab in SERBIA
1 lab in SLOVAKIA
1 lab in SPAIN
1 lab in UNITED KINGDOM
1 lab in UNITED STATES OF AMERICA

**APPENDIX 4**

**Abbreviations**:

| | |
|---|---|
| C | = final test result after checking of first reported suspect test result |
| D(0.01) | = outlier in Dixon's outlier test |
| D(0.05) | = straggler in Dixon's outlier test |
| G(0.01) | = outlier in Grubbs' outlier test |
| G(0.05) | = straggler in Grubbs' outlier test |
| DG(0.01) | = outlier in Double Grubbs' outlier test |
| DG(0.05) | = straggler in Double Grubbs' outlier test |
| R(0.01) | = outlier in Rosner's outlier test |
| R(0.05) | = straggler in Rosner's outlier test |
| E | = probably an error in calculations |
| U | = test result probably reported in a different unit |
| W | = test result withdrawn on request of participant |
| ex | = test result excluded from statistical evaluation |
| n.a. | = not applicable |
| n.e. | = not evaluated |
| n.d. | = not detected |
| fr. | = first reported |
| SDS | = Safety Data Sheet |

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