Institute for
Interlaboratory Studies

# Results of Proficiency Test
# Grease
# May 2022


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

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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.

Since 2017 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Grease based on the latest version of ASTM D4950 every year. During the annual proficiency testing program 2021/2022 it was decided to continue the round robin for the analysis of Grease.

In this interlaboratory study 14 laboratories in 10 countries registered for participation, see appendix 3 for the number of participants per country. In this report the results of the Grease 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 Grease sample of 5 kilograms labelled #22078.
The 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/IEC17043:2010. 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 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

A batch of 24 Grease containers was obtained from a local supplier and labelled #22078. The homogeneity of the subsamples was checked by determination of Cone Penetration-worked in accordance with ASTM D217 on 4 stratified randomly selected subsamples.

| | Cone Penetration-worked in 0.1 mm |
|---|---|
| sample #22078-1 | 252 |
| sample #22078-2 | 250 |
| sample #22078-3 | 254 |
| sample #22078-4 | 253 |

Table 1: homogeneity test results of subsamples #22078

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility of the reference test method in agreement with the procedure of ISO13528, Annex B2 in the next table.

| | Cone Penetration-worked in 0.1 mm |
|---|---|
| r (observed) | 4.8 |
| reference test method | ASTM D217:21 |
| 0.3 x R (reference test method) | 6.9 |

Table 2: evaluation of the repeatability of subsamples #22078

The calculated repeatability is in agreement with 0.3 times the reproducibility of the reference test method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one container of 5 kg Grease labelled #22078 was sent on April 13, 2022. An SDS was added to the sample package.

## 2.5 STABILITY OF THE SAMPLES

The stability of Grease in 5 kg plastic cans was checked. The material was found sufficiently stable for the period of the proficiency test.

### 2.6 ANALYZES

The participants were requested to determine: Cone Penetration (unworked, worked and prolonged work), Copper Corrosion 24 hrs at 100 °C, Dropping Point, Extreme Pressure Properties (Four-ball method), Oil Separation-Conical Sieve, Roll Stability-Penetration change (¼ and ½ Scale Penetrometer), Water, Wear Preventive Characteristics, Elemental Analyzes: 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 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 reference test methods (when applicable) 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 appendices 1 and 2 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 analysis and original test results are placed under 'Remarks' in the 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.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner’s outlier test can be used. 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 visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

### 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 (derived from e.g. ISO or ASTM test methods), 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, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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. Therefore, 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

Some problems were encountered with the dispatch of the samples due to COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with another week. One participant reported test results after the extended reporting date and three other participants did not report any test results. Not all participants were able to report all tests requested.
In total 11 participants reported 78 numerical test results. Observed were 2 outlying test results, which is 2.6%. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

Not all data sets proved to have a normal Gaussian distribution. These are referred to as “not OK”, “suspect” or “unknown”. 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 original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 4.

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). When a method has been reapproved an “R” will be added and the year of approval (e.g. D2266:01R15).

Cone Penetration - unworked: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D217:21a.

Cone Penetration - worked: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D217:21a.

Cone Penetration - prolonged: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D217:21a.

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

Dropping Point: This determination was very problematic. The consensus value could not be determined correctly. Therefore, no z-scores we calculated.

Extreme-Pressure Properties (Four Ball method) – Weld Point: This determination was very problematic. The calculated reproducibility is very large in comparison with the requirements of ASTM D2596:20. Therefore, no z-scores are calculated.

Extreme-Pressure Properties (Four Ball method) – Load Wear Index: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D2596:20.

Extreme-Pressure Properties (Four Ball method) – Last Non-Seizure Load: Only three participants reported test results for this determination. Therefore, no z-scores are calculated.

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

Roll Stability – Penetration change ¼ and ½ Scale Penetrometer: Respectively only one and three participants reported test results for this determination. Therefore, no z-scores are calculated.

Water: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in full agreement with the requirements of ASTM D6304-B:20, but is not in agreement with the requirements of ASTM D6304-C:20.

Wear Preventive Characteristics: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D2266:01R15.

Calcium: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D7303:17.

Lithium: Only three participants reported test results for this determination. Therefore, no z-scores are calculated.

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

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

Zinc: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D7303:17.

For all other elements the majority of the participants agreed on concentrations near or below the limit of detections, see appendix 2. Therefore, no z-scores are calculated for these determinations. The reported test results are given in appendix 2.

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

| Parameter | unit | n | average | 2.8 * sd | R(lit) |
|---|---|---|---|---|---|
| Cone Penetration-unworked | 0.1 mm | 8 | 260 | 34 | 22 |
| Cone Penetration-worked | 0.1 mm | 9 | 267 | 23 | 23 |
| Cone Penetration-prolonged work | 0.1 mm | 6 | 293 | 19 | 29 |
| Copper Corr. 24 hrs at 100 °C | | 8 | 1 (1a/1b) | n.a. | n.a. |
| Dropping Point | °C | 8 | n.e. | n.e. | n.e. |
| Weld Point | kgf | 5 | 286 | 80 | (20) |
| Load Wear Index | kgf | 4 | 43 | 13 | 19 |
| Last Non-Seizure Load | kgf | 3 | 93 | 32 | (73) |
| Oil Separation-Conical Sieve | %M/M | 4 | 1.6 | 3.8 | 1.9 |
| ¼ Scale Penetrometer | 0.1 mm | 1 | n.e. | n.e. | n.e. |
| ½ Scale Penetrometer | 0.1 mm | 3 | n.e. | n.e. | n.e. |
| Water | mg/kg | 4 | 462 | 277 | 292 |
| Wear Preventive Characteristics | mm | 4 | 0.71 | 0.51 | 0.37 |
| Calcium as Ca | mg/kg | 5 | 840 | 203 | 356 |
| Lithium as Li | mg/kg | 3 | 4720 | 3939 | (938) |
| Phosphorus as P | mg/kg | 6 | 1790 | 583 | 635 |
| Sulfur as S | mg/kg | 3 | 5921 | 2309 | 2734 |
| Zinc as Zn | mg/kg | 4 | 2530 | 349 | 502 |

Table 3: reproducibilities of tests on sample #22078

For results between brackets no -z-scores are calculated

Without further statistical calculations it can be concluded that for a number of tests there is a good compliance of the group of participants with the reference test methods. The problematic tests have been discussed in paragraph 4.1.

### 4.3 COMPARISON OF THE PROFICIENCY TEST OF MAY 2022 WITH PREVIOUS PTS

| | May 2022 | May 2021 | September 2019 | September 2018 | September 2017 |
|---|---|---|---|---|---|
| Number of reporting laboratories | 11 | 16 | 13 | 17 | 14 |
| Number of test results | 78 | 105 | 113 | 124 | 92 |
| Number of statistical outliers | 2 | 1 | 4 | 1 | 4 |
| Percentage of statistical outliers | 2.6% | 1.0% | 3.5% | 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 test was compared to the requirements of the reference test methods. The conclusions are given the following table.

| Parameter | May 2022 | May 2021 | September 2019 | 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 | (++) | (++) | -- | ++ | + |
| Oil Separation-Conical Sieve | (--) | (-) | - | +/- | + |
| ¼ Scale Penetrometer | n.e. | n.e. | ++ | n.e. | n.e. |
| ½ Scale Penetrometer | n.e. | n.e. | + | n.e. | n.e. |
| Water | +/- | - | - | +/- | +/- |
| Wear Preventive Characteristics | - | n.e. | +/- | ++ | ++ |
| Calcium as Ca | + | + | - | + | - |
| Lithium as Li | (--) | -- | (-) | - | - |
| Molybdenum as Mo | n.e. | - | n.a. | n.a. | n.a. |
| Phosphorus as P | + | - | n.e. | + | n.e. |
| Sodium as Na | n.e. | n.e. | ++ | n.e. | n.a. |
| Sulfur as S | + | + | - | - | n.e. |
| Zinc as Zn | + | - | n.e. | -- | n.e. |

Table 5: comparison of determinations to the reference test methods

For results between brackets no z-scores are calculated

The following performance categories were used:

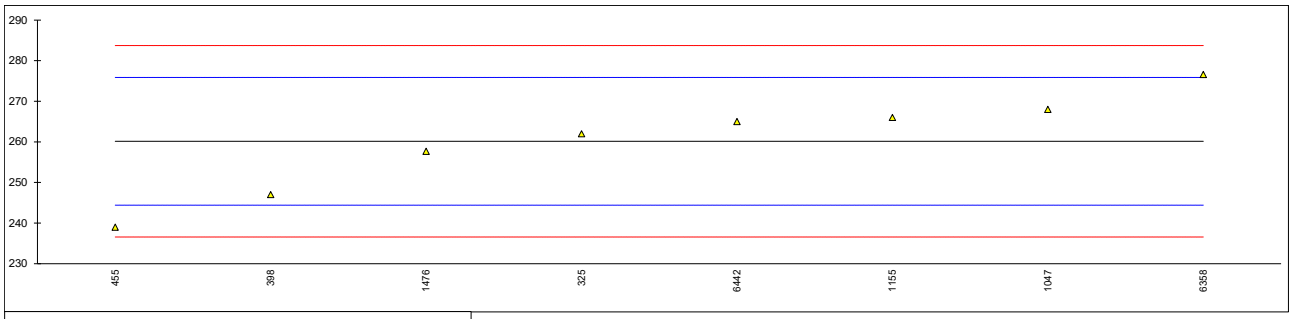
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test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

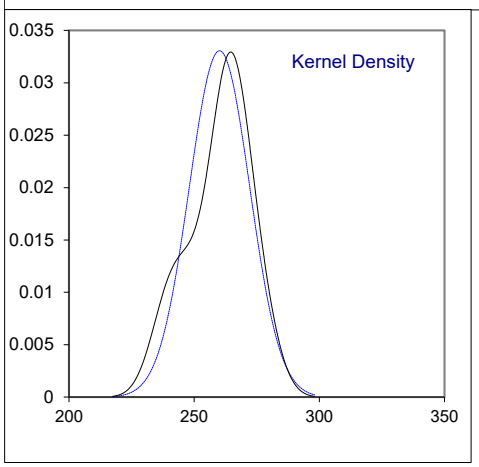
**APPENDIX 1**

Determination of Cone Penetration - unworked on sample #22078; results in 0.1 mm

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 179 | | ----- | | ----- | |
| 325 | D217 | 262 | | 0.23 | |
| 349 | | ----- | | ----- | |
| 398 | D217 | 247 | | -1.67 | |
| 455 | D217 | 239 | | -2.69 | |
| 862 | | ----- | | ----- | |
| 1047 | ISO2137 | 268 | | 1.00 | |
| 1155 | D217 | 266 | | 0.74 | |
| 1328 | | ----- | | ----- | |
| 1476 | ISO2137 | 257.66 | | -0.32 | |
| 6068 | | ----- | | ----- | |
| 6253 | | ----- | | ----- | |
| 6358 | D217 | 276.6 | | 2.09 | |
| 6442 | D217 | 265 | | 0.62 | |

| | |
|---|---|
| normality | unknown |
| n | 8 |
| outliers | 0 |
| mean (n) | 260.16 |
| st.dev. (n) | 12.070 |
| R(calc.) | 33.80 |
| st.dev.(D217:21a) | 7.857 |
| R(D217:21a) | 22 |

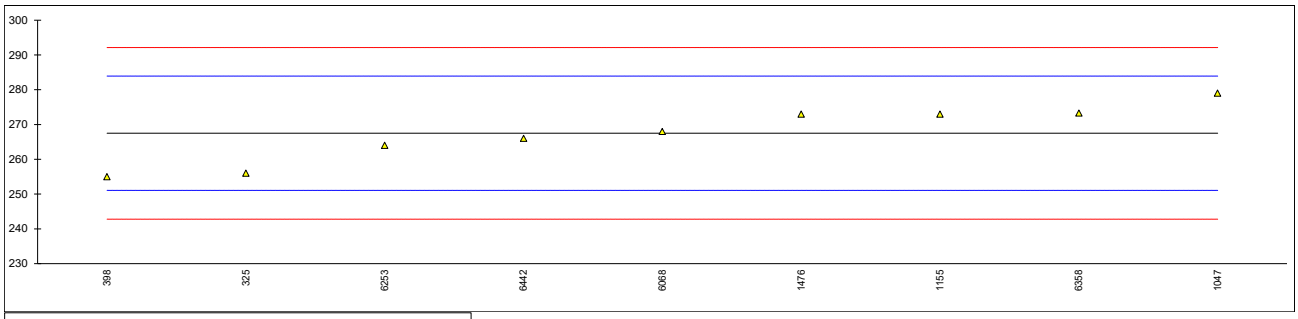


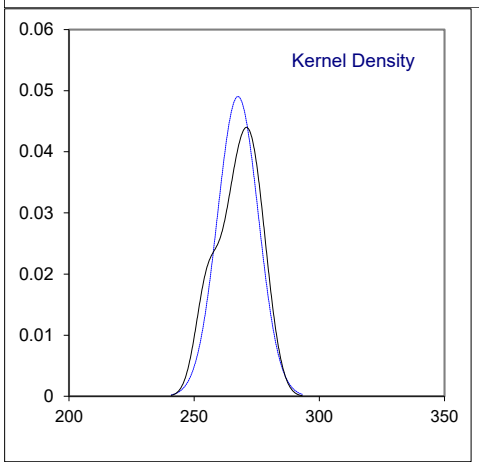



## Determination of Cone Penetration - worked on sample #22078; results in 0.1 mm

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 179 | | ----- | | ----- | |
| 325 | D217 | 256 | | -1.40 | |
| 349 | | ----- | | ----- | |
| 398 | D217 | 255 | | -1.52 | |
| 455 | | ----- | | ----- | |
| 862 | | ----- | | ----- | |
| 1047 | ISO2137 | 279 | | 1.40 | |
| 1155 | D217 | 273 | | 0.67 | |
| 1328 | | ----- | | ----- | |
| 1476 | ISO2137 | 273 | | 0.67 | |
| 6068 | ISO2137 | 268 | | 0.06 | |
| 6253 | ISO2137 | 264 | | -0.42 | |
| 6358 | D217 | 273.3 | | 0.71 | |
| 6442 | D217 | 266 | | -0.18 | |

| | |
|---|---|
| normality | OK |
| n | 9 |
| outliers | 0 |
| mean (n) | 267.48 |
| st.dev. (n) | 8.137 |
| R(calc.) | 22.78 |
| st.dev.(D217:21a) | 8.214 |
| R(D217:21a) | 23 |

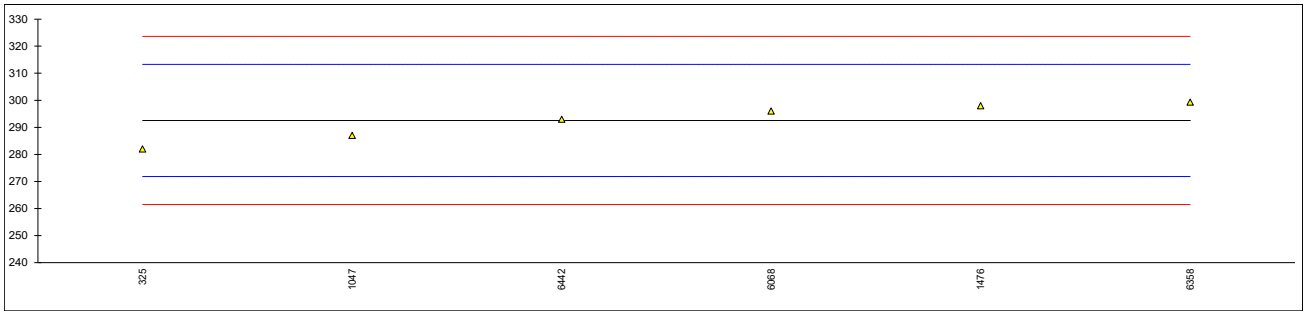


Determination of Cone Penetration – prolonged work on sample #22078; results in 0.1 mm

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 179 | | ----- | | ----- | |
| 325 | D217 | 282 | | -1.02 | |
| 349 | | ----- | | ----- | |
| 398 | | ----- | | ----- | |
| 455 | | ----- | | ----- | |
| 862 | | ----- | | ----- | |
| 1047 | ISO2137 | 287 | | -0.54 | |
| 1155 | | ----- | | ----- | |
| 1328 | | ----- | | ----- | |
| 1476 | ISO2137 | 298 | | 0.53 | |
| 6068 | D217 | 296 | | 0.33 | |
| 6253 | | ----- | | ----- | |
| 6358 | D217 | 299.3 | | 0.65 | |
| 6442 | D217 | 293 | | 0.04 | |

| | |
|---|---|
| normality | unknown |
| n | 6 |
| outliers | 0 |
| mean (n) | 292.55 |
| st.dev. (n) | 6.775 |
| R(calc.) | 18.97 |
| st.dev.(D217:21a) | 10.357 |
| R(D217:21a) | 29 |



## Determination of Copper Corrosion 24 hrs at 100 °C on sample #22078

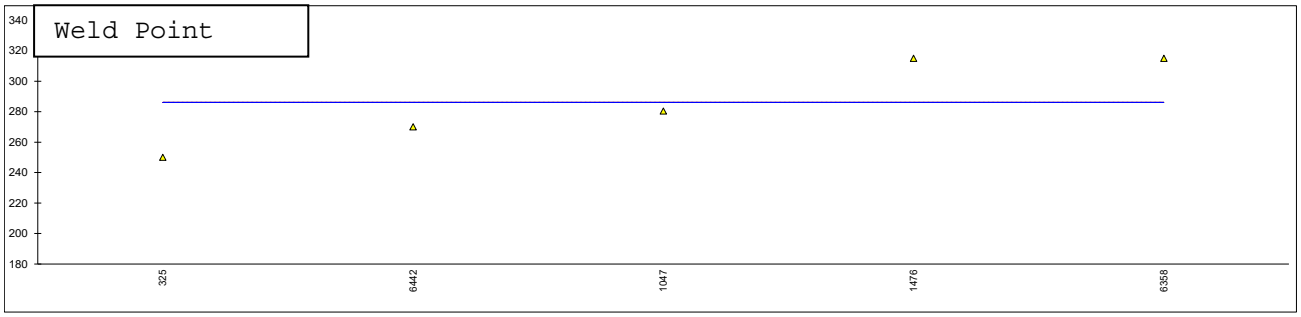
| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 179 | | ----- | | ----- | |
| 325 | DIN51811 | 1B-slight | | ----- | |
| 349 | | ----- | | ----- | |
| 398 | D4048 | 1a | | ----- | |
| 455 | | ----- | | ----- | |
| 862 | | ----- | | ----- | |
| 1047 | D4048 | 1A | | ----- | |
| 1155 | D4048 | 1a | | ----- | |
| 1328 | | ----- | | ----- | |
| 1476 | D4048 | 1a | | ----- | |
| 6068 | D4048 | 1a | | ----- | |
| 6253 | | ----- | | ----- | |
| 6358 | D4048 | 1a | | ----- | |
| 6442 | D4048 | 1b | | ----- | |
| | n | 8 | | | |
| | mean (n) | 1 (1a/1b) | | | |

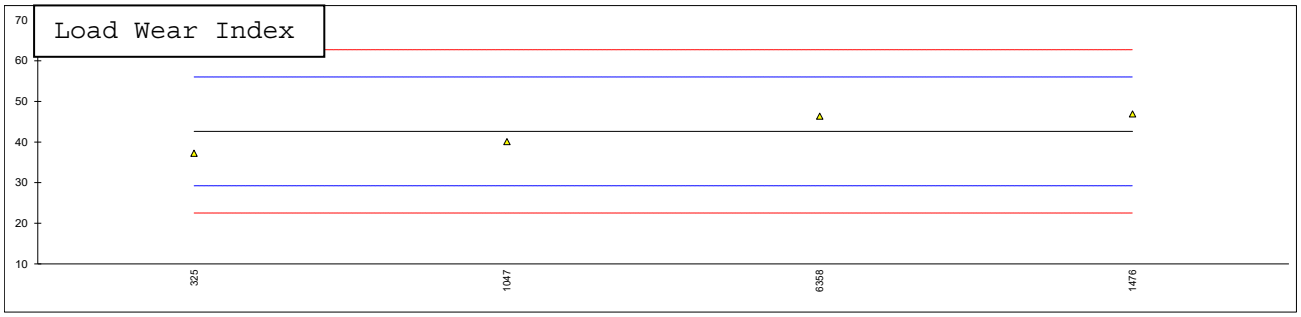
Determination of Dropping Point on sample #22078; results in °C

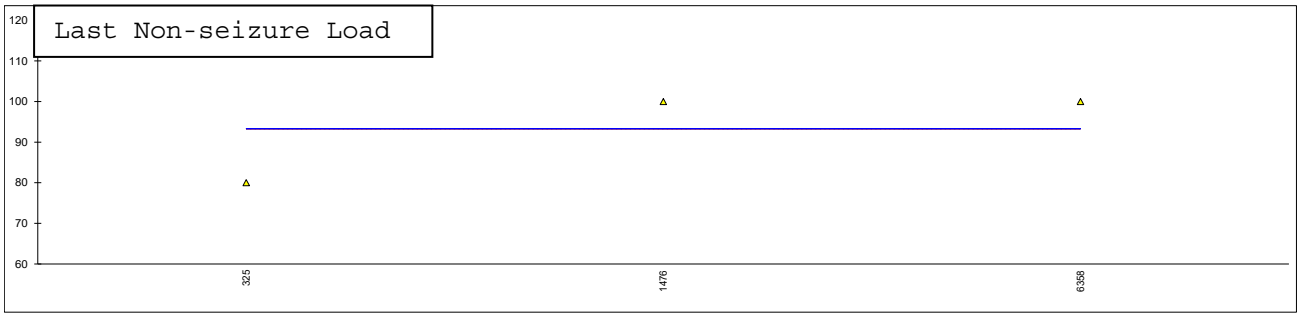
| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 179 | | ----- | | ----- | |
| 325 | In house | >300 | | ----- | |
| 349 | | ----- | | ----- | |
| 398 | D2265 | 279 | | ----- | |
| 455 | | ----- | | ----- | |
| 862 | | ----- | | ----- | |
| 1047 | ISO2176 | 267 | | ----- | |
| 1155 | D2265 | 301.7 | | ----- | |
| 1328 | | ----- | | ----- | |
| 1476 | ISO6299 | >304 | | ----- | |
| 6068 | | ----- | | ----- | |
| 6253 | ISO6299 | 270 | | ----- | |
| 6358 | D2265 | >304 | | ----- | |
| 6442 | D2265 | > 308 | | ----- | |
| | n | 8 | | | |
| | mean (n) | n.e. | | | |

Determination of Extreme-Pressure Properties (Four-ball method) on sample #22078; 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 N-S Load | mark | z(targ) |
|---|---|---|---|---|---|---|---|---|---|---|
| 179 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 325 | D2596 | 250 | | ----- | 37.22 | | -0.81 | 80 | | ----- |
| 349 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 398 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 455 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 862 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1047 | ISO20623 | 280.4 | | ----- | 40.08 | | -0.38 | ----- | | ----- |
| 1155 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1328 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1476 | D2596 | 315 | | ----- | 46.898 | | 0.63 | 100 | | ----- |
| 6068 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 6253 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 6358 | PN-C-04147 | 315 | | ----- | 46.38 | | 0.56 | 100 | | ----- |
| 6442 | D2596 | 270 | | ----- | ----- | | ----- | ----- | | ----- |
| | normality | unknown | | | unknown | | | unknown | | |
| | n | 5 | | | 4 | | | 3 | | |
| | outliers | 0 | | | 0 | | | 0 | | |
| | mean (n) | 286.08 | | | 42.645 | | | 93.33 | | |
| | st.dev. (n) | 28.572 | | | 4.7626 | | | 11.547 | | |
| | R(calc.) | 80.00 | | | 13.335 | | | 32.33 | | |
| | st.dev (D2596:20) | (7.143) | | | 6.7013 | | | (26.000) | | |
| | R(D2596:20) | (20) | | | 18.764 | | | (72.80) | | |



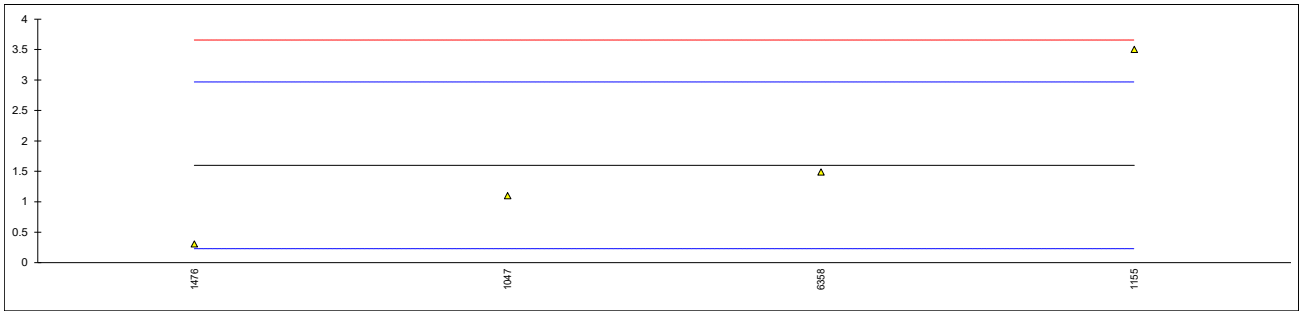



## Determination of Oil Separation – Conical Sieve on sample #22078; results in %M/M

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 179 | | ----- | | ----- | |
| 325 | | ----- | | ----- | |
| 349 | | ----- | | ----- | |
| 398 | | ----- | | ----- | |
| 455 | | ----- | | ----- | |
| 862 | | ----- | | ----- | |
| 1047 | PN-V-04047 | 1.1 | | -0.73 | |
| 1155 | BS2000-121 | 3.5 | | 2.77 | |
| 1328 | | ----- | | ----- | |
| 1476 | PN-V-04047 | 0.306 | | -1.89 | |
| 6068 | | ----- | | ----- | |
| 6253 | | ----- | | ----- | |
| 6358 | D6184 | 1.49 | | -0.16 | |
| 6442 | | ----- | | ----- | |

| | |
|---|---|
| normality | unknown |
| n | 4 |
| outliers | 0 |
| mean (n) | 1.599 |
| st.dev. (n) | 1.3597 |
| R(calc.) | 3.807 |
| st.dev.(D6184:17) | 0.6851 |
| R(D6184:17) | 1.918 |



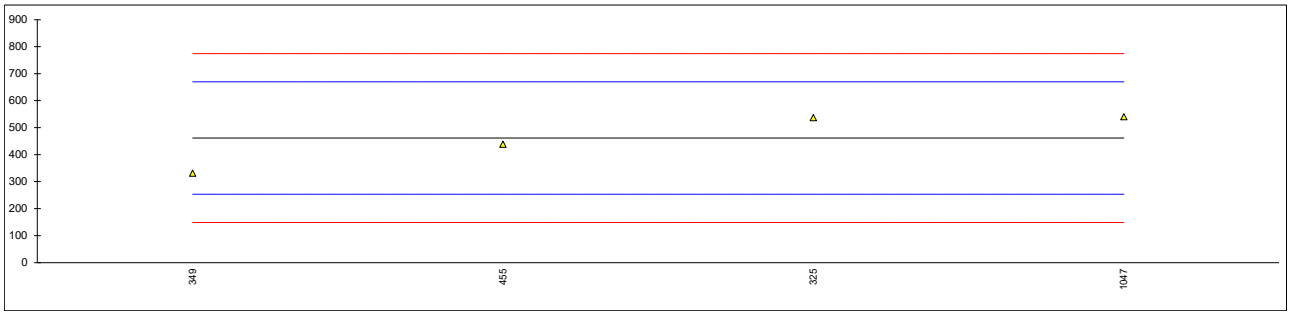
Determination of Roll Stability - Penetration change on sample #22078; results in 0.1 mm

| lab | method | ¼ Scale Pen. | mark | z(targ) | ½ Scale Pen. | mark | z(targ) | remarks |
|---|---|---|---|---|---|---|---|---|
| 179 | | ----- | | ----- | ----- | | ----- | |
| 325 | D1831 | ----- | | ----- | 128 | | ----- | |
| 349 | | ----- | | ----- | ----- | | ----- | |
| 398 | | ----- | | ----- | ----- | | ----- | |
| 455 | | ----- | | ----- | ----- | | ----- | |
| 862 | | ----- | | ----- | ----- | | ----- | |
| 1047 | D1831 | ----- | | ----- | 12 | | ----- | |
| 1155 | | ----- | | ----- | ----- | | ----- | |
| 1328 | | ----- | | ----- | ----- | | ----- | |
| 1476 | | ----- | | ----- | ----- | | ----- | |
| 6068 | | ----- | | ----- | ----- | | ----- | |
| 6253 | | ----- | | ----- | ----- | | ----- | |
| 6358 | D1831 | ----- | | ----- | -2 | | ----- | |
| 6442 | D1831 | 19 | | ----- | ----- | | ----- | |
| | n | 1 | | | 3 | | | |

## Determination of Water on sample #22078; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 179 | | ----- | | ----- | |
| 325 | D6304-C:20 | 537 | | 0.72 | |
| 349 | D6304-C:20 | 331 | | -1.25 | |
| 398 | | ----- | | ----- | |
| 455 | D6304-B:20 | 438.4 | | -0.22 | |
| 862 | | ----- | | ----- | |
| 1047 | D6304-C:16e1 | 540 | | 0.75 | |
| 1155 | | ----- | | ----- | |
| 1328 | | ----- | | ----- | |
| 1476 | | ----- | | ----- | |
| 6068 | | ----- | | ----- | |
| 6253 | | ----- | | ----- | |
| 6358 | | ----- | | ----- | |
| 6442 | | ----- | | ----- | |

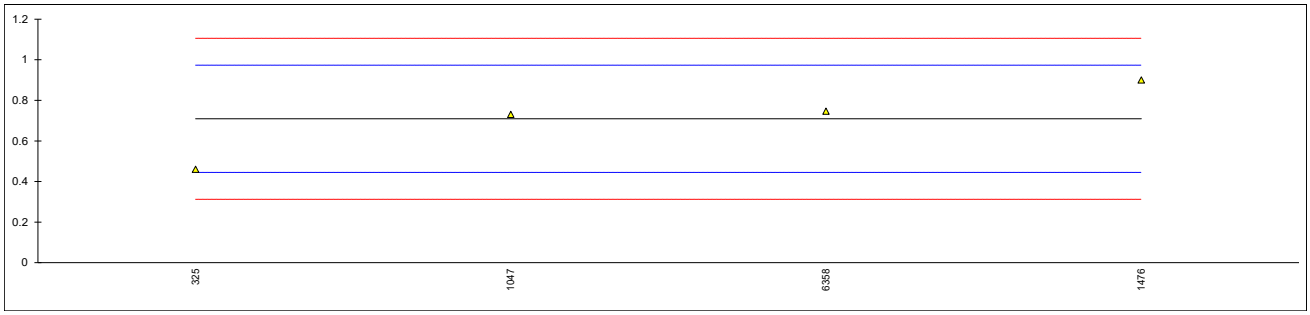
| | | |
|---|---|---|
| | normality | unknown |
| | n | 4 |
| | outliers | 0 |
| | mean (n) | 461.60 |
| | st.dev. (n) | 99.039 |
| | R(calc.) | 277.31 |
| | st.dev.(D6304-B:20) | 104.124 |
| | R(D6304-B:20) | 291.55 |
| Compare | | |
| | R(D6304-C:20) | 153.11 |



Determination of Wear Preventive Characteristics on sample #22078; results in mm

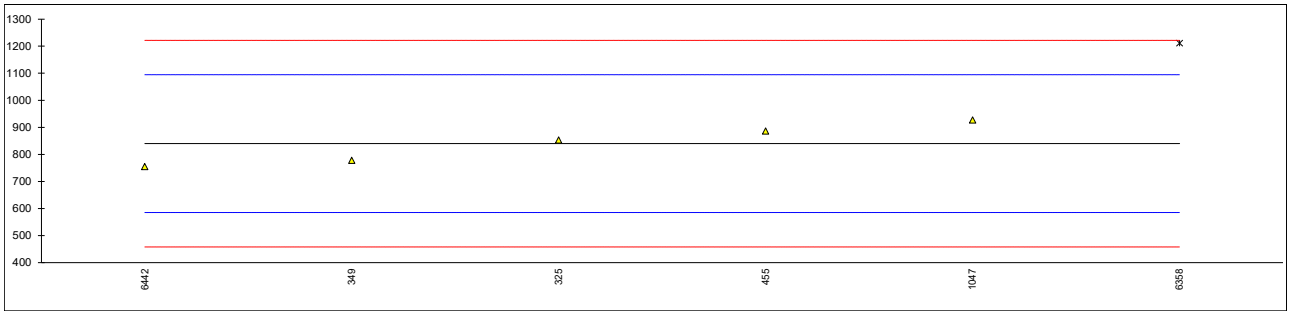
| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 179 | | ----- | | ----- | |
| 325 | D2266 | 0.46 | | -1.88 | |
| 349 | | ----- | | ----- | |
| 398 | | ----- | | ----- | |
| 455 | | ----- | | ----- | |
| 862 | | ----- | | ----- | |
| 1047 | ISO20623 | 0.73 | | 0.16 | |
| 1155 | | ----- | | ----- | |
| 1328 | | ----- | | ----- | |
| 1476 | D2266 | 0.90 | | 1.45 | |
| 6068 | | ----- | | ----- | |
| 6253 | | ----- | | ----- | |
| 6358 | In house | 0.746 | | 0.28 | |
| 6442 | | ----- | | ----- | |

| | |
|---|---|
| normality | unknown |
| n | 4 |
| outliers | 0 |
| mean (n) | 0.709 |
| st.dev. (n) | 0.1828 |
| R(calc.) | 0.512 |
| st.dev.(D2266:01R15) | 0.1321 |
| R(D2266:01R15) | 0.37 |



## Determination of Calcium as Ca on sample #22078; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 179 | | ----- | | ----- | |
| 325 | INH-X-ray | 853 | | 0.10 | |
| 349 | D7303 | 778 | | -0.49 | |
| 398 | | ----- | | ----- | |
| 455 | D7303 | 886.25 | | 0.36 | |
| 862 | | ----- | | ----- | |
| 1047 | ISO20623 | 927 | | 0.68 | |
| 1155 | | ----- | | ----- | |
| 1328 | | ----- | | ----- | |
| 1476 | | ----- | | ----- | |
| 6068 | | ----- | | ----- | |
| 6253 | | ----- | | ----- | |
| 6358 | D7303 | 1211 | G(0.05) | 2.92 | |
| 6442 | D7303 | 755 | | -0.67 | |
| | | | | | |
| | normality | unknown | | | |
| | n | 5 | | | |
| | outliers | 1 | | | |
| | mean (n) | 839.8 | | | |
| | st.dev. (n) | 72.36 | | | |
| | R(calc.) | 202.6 | | | |
| | st.dev.(D7303:17) | 127.26 | | | |
| | R(D7303:17) | 356.3 | | | Application range: 20-50000 |

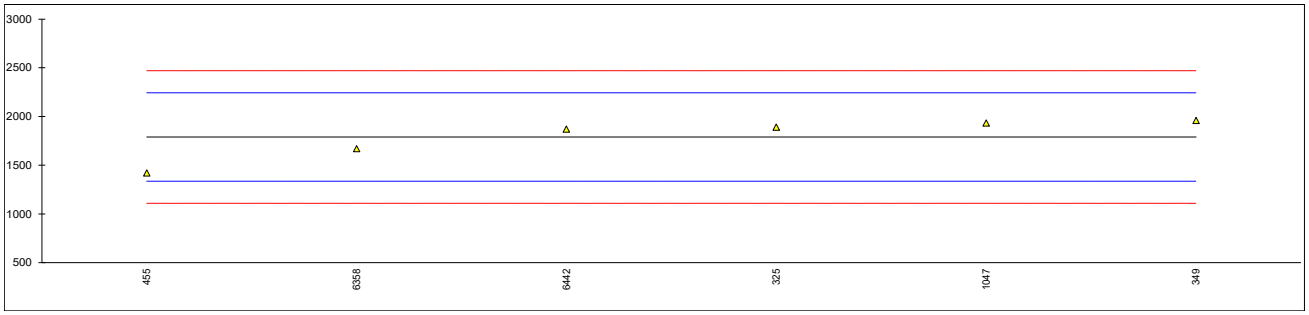


Determination of Lithium as Li on sample #22078; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 179 | | ----- | | ----- | |
| 325 | | ----- | | ----- | |
| 349 | | ----- | | ----- | |
| 398 | | ----- | | ----- | |
| 455 | D7303 | 5357.6 | | ----- | |
| 862 | | ----- | | ----- | |
| 1047 | ISO20623 | 5695 | | ----- | |
| 1155 | | ----- | | ----- | |
| 1328 | | ----- | | ----- | |
| 1476 | | ----- | | ----- | |
| 6068 | | ----- | | ----- | |
| 6253 | | ----- | | ----- | |
| 6358 | D7303 | 3107 | | ----- | |
| 6442 | | ----- | | ----- | |
| | normality | unknown | | | |
| | n | 3 | | | |
| | outliers | 0 | | | |
| | mean (n) | 4719.9 | | | |
| | st.dev. (n) | 1406.93 | | | |
| | R(calc.) | 3939.4 | | | |
| | st.dev.(D7303:17) | (335.10) | | | |
| | R(D7303:17) | (938.3) | | | Application range: 300-3200 |

## Determination of Phosphorus as P on sample #22078; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 179 | | ----- | | ----- | |
| 325 | INH-X-ray | 1890 | | 0.44 | |
| 349 | D7303 | 1959 | | 0.75 | |
| 398 | | ----- | | ----- | |
| 455 | D7303 | 1419.6 | | -1.63 | |
| 862 | | ----- | | ----- | |
| 1047 | ISO20623 | 1932 | | 0.63 | |
| 1155 | | ----- | | ----- | |
| 1328 | | ----- | | ----- | |
| 1476 | | ----- | | ----- | |
| 6068 | | ----- | | ----- | |
| 6253 | | ----- | | ----- | |
| 6358 | D7303 | 1669.5 | | -0.53 | |
| 6442 | D7303 | 1870 | | 0.35 | |
| | | | | | |
| | normality | unknown | | | |
| | n | 6 | | | |
| | outliers | 0 | | | |
| | mean (n) | 1790.0 | | | |
| | st.dev. (n) | 208.25 | | | |
| | R(calc.) | 583.1 | | | |
| | st.dev.(D7303:17) | 226.72 | | | |
| | R(D7303:17) | 634.8 | | | Application range: 50-2000 |

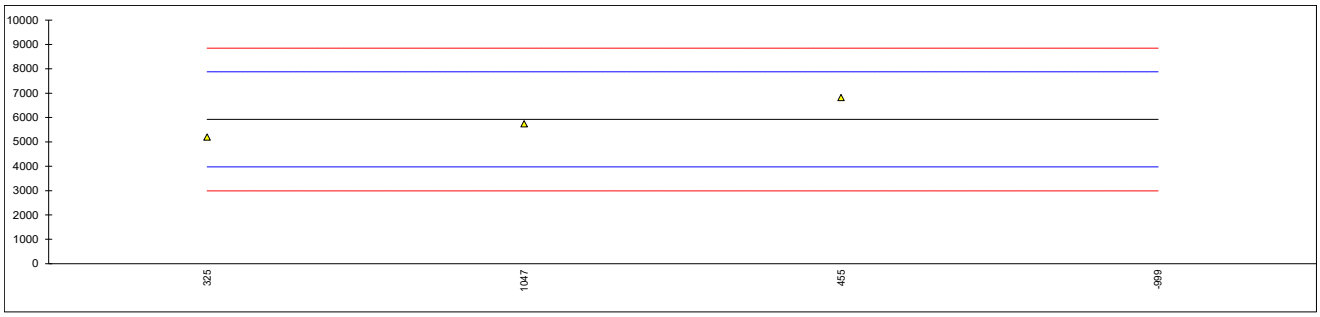


## Determination of Sulfur as S on sample #22078; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 179 | | ----- | | ----- | |
| 325 | INH-X-ray | 5200 | | -0.74 | |
| 349 | | ----- | | ----- | |
| 398 | | ----- | | ----- | |
| 455 | D7303 | 6820.1 | | 0.92 | |
| 862 | | ----- | | ----- | |
| 1047 | ISO20623 | 5744 | | -0.18 | |
| 1155 | | ----- | | ----- | |
| 1328 | | ----- | | ----- | |
| 1476 | | ----- | | ----- | |
| 6068 | | ----- | | ----- | |
| 6253 | | ----- | | ----- | |
| 6358 | | ----- | | ----- | |
| 6442 | | ----- | | ----- | |

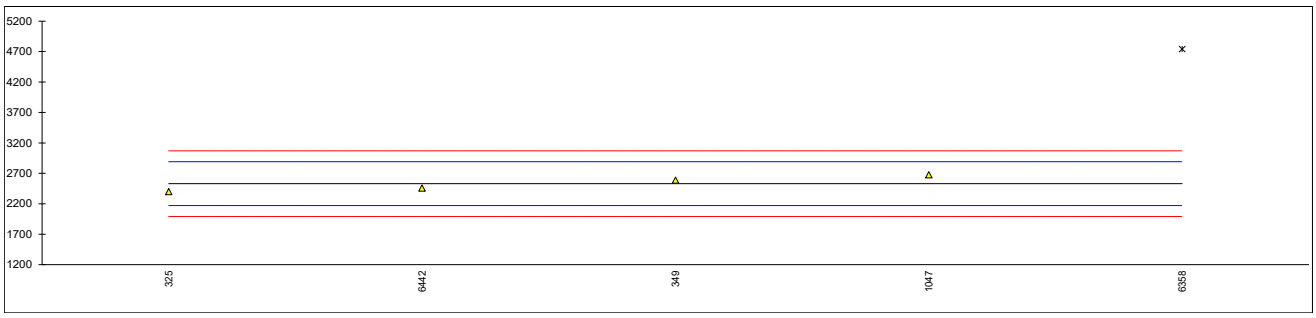
| | | |
|---|---|---|
| | normality | unknown |
| | n | 3 |
| | outliers | 0 |
| | mean (n) | 5921.4 |
| | st.dev. (n) | 824.48 |
| | R(calc.) | 2308.6 |
| | st.dev.(D7303:17) | 976.53 |
| | R(D7303:17) | 2734.3 |

Application range: 1600-28000



## Determination of Zinc as Zn on sample #22078; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 179 | | ----- | | ----- | |
| 325 | INH-X-ray | 2400 | | -0.73 | |
| 349 | D7303 | 2588 | | 0.32 | |
| 398 | | ----- | | ----- | |
| 455 | | ----- | | ----- | |
| 862 | | ----- | | ----- | |
| 1047 | ISO20623 | 2675 | | 0.81 | |
| 1155 | | ----- | | ----- | |
| 1328 | | ----- | | ----- | |
| 1476 | | ----- | | ----- | |
| 6068 | | ----- | | ----- | |
| 6253 | | ----- | | ----- | |
| 6358 | D7303 | 4740 | G(0.01) | 12.32 | |
| 6442 | D7303 | 2458 | | -0.40 | |
| | | | | | |
| | normality | unknown | | | |
| | n | 4 | | | |
| | outliers | 1 | | | |
| | mean (n) | 2530.3 | | | |
| | st.dev. (n) | 124.46 | | | |
| | R(calc.) | 348.5 | | | |
| | st.dev.(D7303:17) | 179.34 | | | |
| | R(D7303:17) | 502.2 | | | Application range: 300-2200 |



**APPENDIX 2**

Other reported test results on sample #22078; results in mg/kg

| Lab | Al | Sb | Ba | Fe | Mg | Mo | Si | Na |
|---|---|---|---|---|---|---|---|---|
| 179 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 325 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 349 | <1 | <1 | 1 | <1 | 5 | 2 | 5 | 11 |
| 398 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 455 | 2.7 | ----- | 0.0 | 0.7 | 9.0 | 0.0 | 7.7 | 10.1 |
| 862 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 1047 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 1155 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 1328 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 1476 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 6068 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 6253 | ----- | ----- | ----- | ----- | ----- | ----- | ----- | ----- |
| 6358 | <0,1 | ----- | <0,1 | 29.02 | 18.16 | <0,1 | 27.54 | 190.4 |
| 6442 | < 10 | ----- | < 10 | < 10 | < 10 | < 10 | < 10 | 12 |
| Range * | 10-600 | 10-2300 | 50-800 | 10-360 | 30-10000 | 50-22000 | 10-15000 | 30-1500 |

*) of ASTM D7303 for the different elements in mg/kg

## APPENDIX 3

### Number of participants per country

1 lab in BELGIUM
2 labs in CHINA, People's Republic
1 lab in ITALY
1 lab in MOROCCO
4 labs in POLAND
1 lab in PORTUGAL
1 lab in SERBIA
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 | = calculation difference between reported test result and result calculated by iis |
| W | = test result withdrawn on request of participant |
| ex | = test result excluded from statistical evaluation |
| n.a. | = not applicable |
| n.e. | = not evaluated |
| n.d. | = not detected |
| fr. | = first reported |
| SDS | = Safety Data Sheet |

### Literature


1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2 ISO5725:86
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7 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
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11 W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)
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