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

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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# 1 INTRODUCTION

Since 2003 the Institute for Interlaboratory Studies (iis) organizes a proficiency test for the analyzes of used Hydraulic Oil every year. During the annual program 2019/2020 it was decided to continue the round robin for the analyzes of used Hydraulic Oil. This interlaboratory study contains also a PT for the determination of Metals in used Hydraulic Oil.

In the PT for the regular analyzes 62 laboratories in 41 different countries registered for participation. In the PT on Metals 50 laboratories in 35 different countries registered for participation. In total 64 laboratories in 43 different countries registered for at least one of the two PTs. See appendix 2 for the number of participants per country.
In this report, the results of the 2019 used Hydraulic Oil proficiency test are presented and discussed. This report is also available as PDF file from 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. In this proficiency test the participants received depending on the registration: one liter glass bottle with used Hydraulic Oil labelled #19236 for the round with regular analyzes and/or a 50 mL PE bottle with used Hydraulic Oil labelled #19237 for the wear Metals round. 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

Preparation of samples for the PT on regular analyzes in used Hydraulic Oil, iis19L10

Approximately 90 liter of used Hydraulic Oil was obtained from a local supplier. After homogenisation 84 amber glass bottles of one liter were filled and labelled #19236. The homogeneity of the subsamples #19236 was checked by determination of Density at 15°C in accordance with ASTM D4052 and Kinematic Viscosity at 40°C according to ASTM D445 on 8 stratified randomly selected samples.

| | Density at 15°C in kg/L | Kinematic Viscosity at 40°C in $mm^2/s$ |
|---|---|---|
| Sample #19236-1 | 0.86921 | 68.05 |
| Sample #19236-2 | 0.86921 | 68.14 |
| Sample #19236-3 | 0.86922 | 68.11 |
| Sample #19236-4 | 0.86920 | 68.09 |
| Sample #19236-5 | 0.86921 | 68.09 |
| Sample #19236-6 | 0.86920 | 68.03 |
| Sample #19236-7 | 0.86921 | 68.12 |
| Sample #19236-8 | 0.86920 | 68.10 |

Table 1: homogeneity test results of subsamples #19236

From the above test results the repeatabilities were calculated and compared with 0.3 times the reproducibilities 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 | 0.101 |
| reference test method | ISO12185:96 | iis memo 1401 |
| 0.3 x R (ref. test method) | 0.00015 | 0.368 |

Table 2: evaluation of the repeatabilities of subsamples #19236

The calculated repeatabilities were lower than 0.3 times the reproducibilities of the reference test methods. Therefore, homogeneity of the subsamples was assumed.

Preparation of samples for PT on Metals in used Hydraulic Oil, iis19L10M

Approximately 10 liter of used Hydraulic Oil was obtained from a local supplier who also did also add different elements. After homogenisation 70 PE bottles of 50 mL were filled and labelled #19237. The homogeneity of the subsamples #19237 was checked by the determination of Copper and Nickel in accordance with ASTM D5185 on 8 stratified randomly selected samples.

| | Copper as Cu in mg/kg | Nickel as Ni in mg/kg |
|---|---|---|
| Sample #19237-1 | 15 | 12 |
| Sample #19237-2 | 15 | 12 |
| Sample #19237-3 | 15 | 12 |
| Sample #19237-4 | 15 | 12 |
| Sample #19237-5 | 15 | 12 |
| Sample #19237-6 | 15 | 13 |
| Sample #19237-7 | 15 | 12 |
| Sample #19237-8 | 16 | 13 |

Table 3: homogeneity test results of subsamples #19237

From the test results of table 3 the repeatabilities were calculated and compared with 0.3 times the reproducibilities of the reference test method in agreement with the procedure of ISO13528, Annex B2 in the next table.

| | Copper as Cu in mg/kg | Nickel as Ni in mg/kg |
|---|---|---|
| r (observed) | 0.99 | 1.30 |
| reference test method | D5185:18 | D5185:18 |
| 0.3 x R (ref. test method) | 1.09 | 1.58 |

Table 4: evaluation of the repeatabilities of subsamples #19237

The calculated repeatabilities were in agreement with 0.3 times the corresponding reproducibilities of the reference test method. Therefore, homogeneity of the subsamples was assumed.

Depending on the registration to each of the participating laboratories one 1 liter bottle labelled #19236 and/or one 50 mL PE bottle labelled #19237 was dispatched on October 16, 2019. An SDS was added to the sample package.

## 2.5 STABILITY OF THE SAMPLES

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

## 2.6 ANALYZES

The participants were requested to determine on sample #19236: Acid Number (Total), Density at 15°C, Flash Point PMcc, Kinematic Viscosity at 40 and 100°C, Viscosity Stabinger at 40 and 100°C, Sulfur, Water and Level of Contamination (counts/mL and scale number). And on sample #19237 23 elements, wear metals and additives: Al, Ba, B, Cd, Cr, Cu, Fe, Pb, Li, Mg, Mn, Mo, Ni, K, Si, Ag, Na, Sn, Ti, V, Ca, P and Zn.
Also some additional questions were asked about Acid Number (Total) and Level of Contamination.

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

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 analyzis 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 them with a factor of 2.8.

### 3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also a normal Gauss curve was projected over the Kernel Density Graph for reference.

### 3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM 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:

$z_{(target)}$ = (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 sample dispatch. For the round with the regular analyzes, one participant reported the test results after the final reporting date and one other participant did not report any test results at all.
For the Metals round one participant reported the test results after the final reporting date and five other participants did not report any test results at all. Not all participants were able to report all analyzes requested.
In total, 63 participants reported in total 1402 numerical test results. Observed were 87 outlying test results, which is 6.2%. 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 SAMPLE AND PER TEST

In this section the results are discussed per sample and 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. The abbreviations, used in these tables, are explained in appendix 3.

In the iis PT reports the ASTM test methods are referred to with a number e.g. ASTM D7647 and an added designation for the year that the test method was adopted or revised e.g. ASTM D7647:10. If applicable a designation in parentheses is added to designate the year of reapproval e.g. ASTM D7647:10(2018). 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.

**Sample #19236**

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 full agreement with the requirements of ASTM D664-A:18e2 using Buffer End Point 60 mL. However, the calculated reproducibility is not in agreement with the precision data of the procedures Buffer End Point 125 mL and Inflection Point 60 and 125 mL.

Density at 15°C: This determination was problematic for some laboratories. Eight 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 may not be problematic depending on the test method used. One statistical outlier was observed and one other test result was excluded. The calculated reproducibility after rejection of the suspect data is in full agreement with the requirements of ASTM D93:19 procedure A, but not with procedure B. When the test results of procedures A and B are evaluated separately both calculated reproducibilities are in full agreement with the requirements of ASTM D93:19 procedures A or B respectively.

Kin.Viscosity at 40°C: The precision statement given in ASTM D445 for used (in-service) formulated oils appears to be very strict, see previous iis reports iis17L08 and iis18L10. Therefore, the target reproducibility being used is calculated from the reproducibilities found in previous iis PTs on used oils (see lit. 17). 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 average reproducibility found for used oils in previous iis PTs (iis memo 1401), and in this case also in (full) agreement with the requirements of ASTM D445:19.

Kin.Viscosity at 100°C: See the explanation about selection of the target reproducibility at Kinematic Viscosity at 40°C.
This determination was not problematic. Five statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the average reproducibility found for used oils in previous iis PTs (iis memo 1401), but not in agreement with the requirements of ASTM D445:19.

Viscosity Stabinger at 40°C: This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D7042:16e3.

Viscosity Stabinger at 100°C: This determination was not problematic. No statistical outliers were observed but three test results were excluded. The calculated reproducibility after rejection of the suspect data is in agreement with the requirements of ASTM D7042:16e3.

Sulfur: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D4294:16e1.

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

Level of Contamination: When the data of the last four round robins are evaluated no significant differences between the test results from ISO11500 and the test results of ASTM D7647 could be found. The reproducibility of ASTM D7647 is used for the calculation of the z-scores because ISO11500 does not mention a reproducibility but only ’ Maximum allowable difference’ in Annex A. For calibration of equipment all participants that responded used ISO11171.
This determination was problematic. In total nineteen statistical outliers were observed over six parameters (15 for counts/mL and 4 for scale number) and another twenty-six test results were excluded. The calculated reproducibilities after rejection of the suspect data are not in agreement with the requirements of ASTM D7647:10(2018).

The reference method for the analyzes of wear metals is test method ASTM D5185:18. For some metals i.e. Ba and K the consensus value of the group is either above or below the respective application ranges on which the requirements of ASTM D5185:18 are based. However, it was decided to use the reproducibility from ASTM D5185:18 as the calculated reproducibility (after the rejection of the statistical outliers) is in general in good agreement with the requirements of ASTM D5185:18. More discussion per metal is given below.

**Sample #19237**

Aluminum: 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 D5185:18.

Barium: This determination was not problematic. Two statistical outliers were observed and one other test result was excluded. However, the calculated reproducibility after rejection of the suspect data is in agreement with the requirements of ASTM D5185:18.

Boron: This determination was not problematic. Three statistical outliers were observed and one other test result was excluded. However, the calculated reproducibility after rejection of the suspect data is in agreement with the requirements of ASTM D5185:18.

Cadmium: This determination may be problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the estimated reproducibility using the Horwitz equation.

Chromium: This determination was not problematic. One statistical outlier was observed and one other test result was excluded. However, the calculated reproducibility after rejection of the suspect data is in full agreement with the requirements of ASTM D5185:18.

Copper: This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D5185:18.

Iron: This determination was not problematic. Two statistical outliers were observed and one other test result was excluded. However, the calculated reproducibility after rejection of the suspect data is in agreement with the requirements of ASTM D5185:18.

Lead: 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 D5185:18.

Lithium: 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.

Magnesium: This determination was not problematic. Two statistical outliers were observed and one other test result was excluded. However, the calculated reproducibility after rejection of the suspect data is in agreement with the requirements of ASTM D5185:18.

Manganese: 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 D5185:18.

Molybdenum: This determination was not problematic. No statistical outliers were observed but one test result was excluded. The calculated reproducibility after rejection of the suspect data is in agreement with the requirements of ASTM D5185:18.

Nickel: 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 D5185:18.

Potassium: Twenty-four participants agreed on a level of <1 mg/kg. Therefore, no z-scores were calculated.

Silicon: This determination was not problematic. Four statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D5185:18.

Silver: This determination was not problematic. Three statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D5185:18.

Sodium: This determination was not problematic. Three statistical outliers were observed and one other test result was excluded. However, the calculated reproducibility after rejection of the suspect data is in full agreement with the requirements of ASTM D5185:18.

Tin: 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 D5185:18.

Titanium: This determination was not problematic. Two statistical outliers were observed and one other test result was excluded. However, the calculated reproducibility after rejection of the suspect data is in agreement with the requirements of ASTM D5185:18.

Vanadium: 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 D5185:18.

Calcium: This determination was not problematic. Two statistical outliers were observed and one other test result was excluded. However, the calculated reproducibility after rejection of the suspect data is in agreement with the estimated reproducibility using the Horwitz equation, but not with the strict requirements of ASTM D5185:18.

Phosphorus: This determination was not problematic. One statistical outlier was observed and one other test result was excluded. However, the calculated reproducibility after rejection of the suspect data is in agreement with the requirements of ASTM D5185:18.

Zinc: This determination was not problematic. One statistical outlier was observed and one other test result was excluded. However, the calculated reproducibility after rejection of the suspect data is in full 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 reproducibility derived from literature reference test methods (in casu ASTM and ISO reference test methods) are presented in the next tables.

| Parameter | unit | n | average | 2.8 * sd | R(lit) |
|---|---|---|---|---|---|
| Acid Number (Total) | mg KOH/g | 44 | 0.38 | 0.20 | 0.22 |
| Density at 15°C | kg/L | 41 | 0.8692 | 0.0003 | 0.0005 |
| Flash Point PMcc | °C | 37 | 213.7 | 14.5 | 15.2 |
| Kinematic Viscosity at 40°C | $mm^2/s$ | 44 | 68.314 | 0.819 | 1.230 |
| Kinematic Viscosity at 100°C | $mm^2/s$ | 39 | 10.478 | 0.142 | 0.231 |
| Viscosity Stabinger at 40°C | $mm^2/s$ | 15 | 68.376 | 0.480 | 0.859 |
| Viscosity Stabinger at 100°C | $mm^2/s$ | 16 | 10.478 | 0.095 | 0.116 |
| Sulfur | mg/kg | 25 | 695 | 179 | 130 |
| Water | mg/kg | 47 | 64.8 | 73.6 | 206.4 |
| L. of Contamination ≥ 4 µm (c) | counts/mL | 23 | 221 | 392 | 252 |

| Parameter | unit | n | average | 2.8 * sd | R(lit) |
|---|---|---|---|---|---|
| L. of Contamination ≥ 6 µm (c) | counts/mL | 23 | 61.4 | 150.9 | 47.1 |
| L. of Contamination ≥ 14 µm (c) | counts/mL | 23 | 6.8 | 17.0 | 9.3 |
| L. of Contamination ≥ 4 µm (c) | scale no. | 22 | 14.6 | 2.7 | 1.7 |
| L. of Contamination ≥ 6 µm (c) | scale no. | 22 | 12.5 | 3.0 | 1.2 |
| L. of Contamination ≥ 14 µm (c) | scale no. | 22 | 9.2 | 3.7 | 2.0 |

Table 5: performance evaluation sample #19236

| Element | unit | n | average | 2.8 * sd | R(lit) |
|---|---|---|---|---|---|
| Aluminum as Al | mg/kg | 41 | 12.3 | 3.8 | 7.3 |
| Barium as Ba | mg/kg | 36 | 25.2 | 4.0 | 11.5 |
| Boron as B | mg/kg | 31 | 11.8 | 5.4 | 13.3 |
| Cadmium as Cd | mg/kg | 28 | 11.3 | 4.1 | 3.5 |
| Chromium as Cr | mg/kg | 42 | 12.0 | 3.6 | 3.7 |
| Copper as Cu | mg/kg | 40 | 14.0 | 2.6 | 3.4 |
| Iron as Fe | mg/kg | 41 | 15.0 | 3.2 | 4.5 |
| Lead as Pb | mg/kg | 37 | 12.1 | 3.5 | 7.4 |
| Lithium as Li | mg/kg | 12 | 54.3 | 4.5 | 13.3 |
| Magnesium as Mg | mg/kg | 39 | 23.3 | 5.6 | 8.1 |
| Manganese as Mn | mg/kg | 39 | 11.3 | 2.6 | 2.4 |
| Molybdenum as Mo | mg/kg | 38 | 11.6 | 2.7 | 3.6 |
| Nickel as Ni | mg/kg | 43 | 11.7 | 2.8 | 5.1 |
| Potassium as K | mg/kg | 21 | 0.6 | 2.1 | (5.7) |
| Silicon as Si | mg/kg | 38 | 11.8 | 2.5 | 7.6 |
| Silver as Ag | mg/kg | 35 | 11.7 | 3.2 | 4.1 |
| Sodium as Na | mg/kg | 36 | 12.1 | 6.1 | 6.5 |
| Tin as Sn | mg/kg | 37 | 10.5 | 4.0 | 9.0 |
| Titanium as Ti | mg/kg | 40 | 11.8 | 3.2 | 8.0 |
| Vanadium as V | mg/kg | 42 | 11.6 | 2.8 | 4.2 |
| Calcium as Ca | mg/kg | 36 | 77.5 | 14.2 | 18.0 |
| Phosphorus as P | mg/kg | 40 | 266 | 48 | 70 |
| Zinc as Zn | mg/kg | 39 | 309 | 48 | 45 |

Table 6: : performance evaluation sample #19237

Results between brackets should be used with due care because of low concentration of element

Without further statistical calculations, it can be concluded that for many tests there is a good compliance of the group of participants 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 PREVIOUS PTS

| | November 2019 | November 2018 | November 2017 | November 2016 | November 2015 |
|---|---|---|---|---|---|
| Number of reporting laboratories | 63 | 52 | 57 | 51 | 55 |
| Number of test results | 1402 | 1053 | 1202 | 1168 | 1126 |
| Number of statistical outliers | 87 | 49 | 89 | 29 | 43 |
| Percentage outliers | 6.2% | 4.7% | 7.4% | 2.5% | 3.8% |

Table 7: 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 reference test methods. The conclusions are given in the following table.

| Determination | November 2019 | November 2018 | November 2017 | November 2016 | November 2015 |
|---|---|---|---|---|---|
| Acid Number (Total) | +/- | +/- | + | + | + |
| Density at 15°C | + | +/- | +/- | +/- | +/- |
| Flash Point PMcc | +/- | +/- | +/- | - | + |
| Kinematic Viscosity at 40°C | + | + | + | + | ++ |
| Kinematic Viscosity at 100°C | + | + | + | +/- | +/- |
| Viscosity Stabinger at 40°C | + | +/- | + | +/- | ++ |
| Viscosity Stabinger at 100°C | + | - | - | +/- | + |
| Sulfur | - | -- | - | - | -- |
| Water | ++ | ++ | ++ | ++ | ++ |
| L. of Contamination – counts/mL | - | - | -- | - | -- |
| L. of Contamination – scale no. | - | - | - | - | n.e. |
| Aluminum as Al | + | + | ++ | ++ | ++ |
| Barium as Ba | ++ | ++ | + | ++ | + |
| Boron as B | ++ | ++ | ++ | n.e. | n.e. |
| Cadmium as Cd | - | n.e. | + | n.e. | n.e. |
| Chromium as Cr | +/- | n.e. | +/- | + | + |
| Copper as Cu | + | + | + | +/- | + |
| Iron as Fe | + | +/- | + | + | ++ |
| Lead as Pb | ++ | ++ | ++ | ++ | ++ |
| Lithium as Li | ++ | n.e. | + | (--) | - |
| Magnesium as Mg | + | + | + | + | +/- |
| Manganese as Mn | +/- | +/- | -- | +/- | +/- |
| Molybdenum as Mo | + | ++ | +/- | + | +/- |
| Nickel as Ni | + | +/- | ++ | ++ | ++ |
| Potassium as K | (+) | ++ | + | n.e. | n.e. |
| Silicon as Si | ++ | + | + | ++ | ++ |

| Determination | November 2019 | November 2018 | November 2017 | November 2016 | November 2015 |
|---|---|---|---|---|---|
| Silver as Ag | + | n.e. | + | +/- | + |
| Sodium as Na | +/- | +/- | - | +/- | +/- |
| Tin as Sn | ++ | + | ++ | ++ | ++ |
| Titanium as Ti | ++ | n.e. | ++ | ++ | ++ |
| Vanadium as V | + | n.e. | + | + | ++ |
| Calcium as Ca *) | + | -- | -- | - | - |
| Phosphorus as P | + | - | - | + | +/- |
| Zinc as Zn | +/- | - | +/- | - | -- |

Table 8: comparison determinations against the reference test methods

*) Based on Horwitz for 2019 and ASTM D5185 for earlier PTs

In the table above the following performance categories were used:

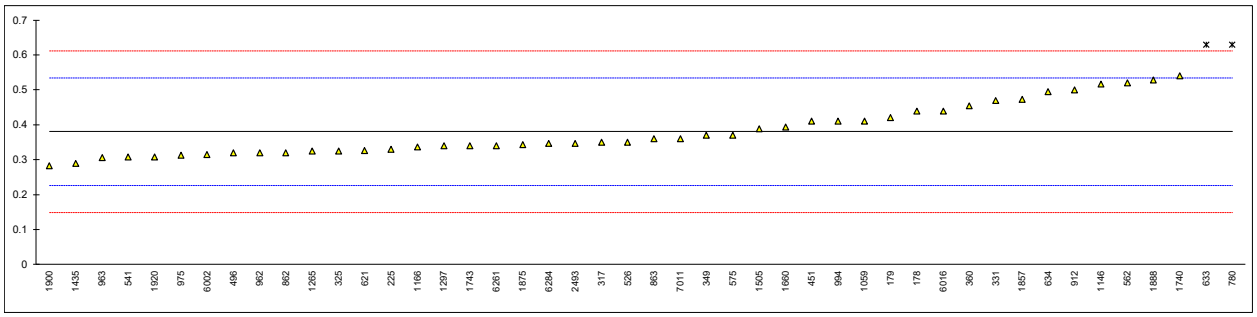
++: group performed much better than the reference test method
+ : group performed better than the reference test method
+/-: group performance equals the reference test method
- : group performed worse than the reference test method
-- : group performed much worse than the reference test method
n.e.: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

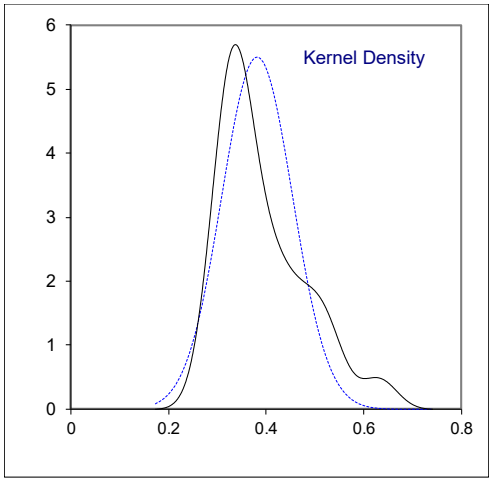
**APPENDIX 1**

Determination of Acid Number (Total) on sample #19236; results in mg KOH/g

| lab | method | value | mark | z(targ) | end point | volume (mL) | remarks |
|---|---|---|---|---|---|---|---|
| 178 | D664-A | 0.44 | | 0.77 | ----- | 60 | |
| 179 | D664-A | 0.42 | | 0.51 | IP | 125 | |
| 225 | D974 | 0.33 | | -0.66 | ----- | ----- | |
| 237 | | ----- | | ----- | ----- | ----- | |
| 255 | | ----- | | ----- | ----- | ----- | |
| 257 | | ----- | | ----- | ----- | ----- | |
| 311 | | ----- | | ----- | ----- | ----- | |
| 317 | D974 | 0.35 | | -0.40 | IP | ----- | |
| 325 | D664-A | 0.325 | | -0.72 | BEP (pH 10) | 125 | |
| 331 | D664Mod. | 0.47 | | 1.16 | IP | 60 | |
| 335 | | ----- | | ----- | ----- | ----- | |
| 339 | | ----- | | ----- | ----- | ----- | |
| 349 | D664-A | 0.37 | | -0.14 | IP | 125 | |
| 360 | D664-A | 0.455 | | 0.96 | IP | 60 | |
| 398 | | ----- | | ----- | ----- | ----- | |
| 432 | | ----- | | ----- | ----- | ----- | |
| 442 | | ----- | | ----- | ----- | ----- | |
| 451 | D664-A | 0.41 | | 0.38 | BEP (pH 10) | 60 | |
| 496 | D664-A | 0.32 | | -0.79 | BEP (pH 10) | 60 | |
| 526 | D974 | 0.35 | | -0.40 | ----- | ----- | |
| 541 | D974 | 0.308 | | -0.94 | ----- | ----- | |
| 562 | D664 | 0.52 | | 1.81 | ----- | ----- | |
| 575 | D664-A | 0.37 | | -0.14 | BEP (pH 10) | 60 | |
| 603 | | ----- | | ----- | ----- | ----- | |
| 614 | | ----- | | ----- | ----- | ----- | |
| 621 | D664-A | 0.326 | | -0.71 | IP | 60 | |
| 633 | D664-A | 0.63 | DG(0.05) | 3.23 | IP | 125 | |
| 634 | D664-A | 0.495 | | 1.48 | ----- | ----- | |
| 780 | D664-A | 0.63 | DG(0.05) | 3.23 | IP | 60 | |
| 862 | D664-A | 0.32 | | -0.79 | ----- | ----- | |
| 863 | D664-A | 0.36 | | -0.27 | ----- | ----- | |
| 912 | D664-A | 0.5 | | 1.55 | ----- | ----- | |
| 962 | D974 | 0.32 | | -0.79 | ----- | ----- | |
| 963 | D664-A | 0.306 | | -0.97 | BEP (pH 11) | 60 | |
| 975 | D664-A | 0.313 | | -0.88 | IP | 60 | |
| 994 | D664-A | 0.41 | C | 0.38 | IP | 125 | fr. 0.61 |
| 1059 | ISO6619 | 0.41 | | 0.38 | BEP (pH 11) | 60 | |
| 1146 | D664-A | 0.517 | | 1.77 | BEP (pH 11) | 125 | |
| 1166 | D664-A | 0.3358 | | -0.58 | IP | 60 | |
| 1265 | D664-A | 0.3247 | | -0.73 | IP | 60 | |
| 1297 | D664-A | 0.34 | | -0.53 | IP | 125 | |
| 1397 | | ----- | | ----- | ----- | ----- | |
| 1435 | D664-A | 0.29 | | -1.18 | BEP (pH 10) | 100 | |
| 1505 | D974 | 0.3895 | | 0.11 | ----- | ----- | |
| 1531 | | ----- | | ----- | ----- | ----- | |
| 1660 | D664-A | 0.393 | | 0.16 | BEP (pH 11) | 60 | |
| 1720 | | ----- | | ----- | ----- | ----- | |
| 1740 | D664-A | 0.54 | | 2.07 | IP | 60 | |
| 1743 | D664-A | 0.34 | | -0.53 | BEP (pH 11) | 60 | |
| 1788 | | ----- | | ----- | ----- | ----- | |
| 1857 | D664-A | 0.473 | | 1.20 | IP | 125 | |
| 1875 | ISO6618 | 0.343 | | -0.49 | ----- | ----- | |
| 1888 | D664-A | 0.528 | | 1.91 | BEP (pH 11) | 60 | |
| 1900 | D664-A | 0.282 | | -1.28 | ----- | 60 | |
| 1920 | D664-A | 0.308 | | -0.94 | IP | 125 | |
| 2493 | ISO6618 | 0.347 | | -0.44 | IP | 125 | |
| 6002 | D664-A | 0.315 | | -0.85 | BEP (pH 10) | 60 | |
| 6016 | D664-A | 0.440 | | 0.77 | ----- | ----- | |
| 6257 | | ----- | | ----- | ----- | ----- | |
| 6261 | D664-A | 0.34 | | -0.53 | ----- | ----- | |
| 6284 | D974 | 0.346 | | -0.45 | ----- | ----- | |
| 7011 | D664-A | 0.36 | | -0.27 | ----- | ----- | |

| | | IP only | BEP only |
|---|---|---|---|
| normality | OK | suspect | suspect |
| n | 44 | 17 | 12 |
| outliers | 2 | 0 | 0 |
| mean (n) | 0.3807 | 0.4143 | 0.3770 |
| st.dev. (n) | 0.07239 | 0.10512 | 0.07901 |
| R(calc.) | 0.2027 | 0.2943 | 0.2212 |
| st.dev.(D664-A:18e2, BEP 60mL) | 0.07713 | ----- | 0.07640 |
| R(D664-A:18e2, BEP 60mL) | 0.2160 | ----- | 0.2139 |
| Compare | | | |
| R(D664-A:18e2, IP 60mL) | 0.1822 | 0.1953 | ----- |
| R(D664-A:18e2, IP 125mL) | 0.0799 | 0.0873 | ----- |
| R(D664-A:18e2, BEP 125mL) | 0.1145 | ----- | 0.1133 |

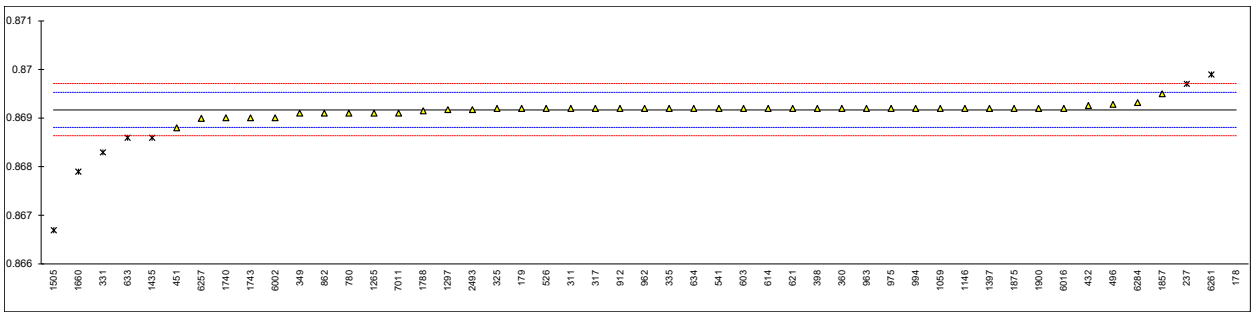


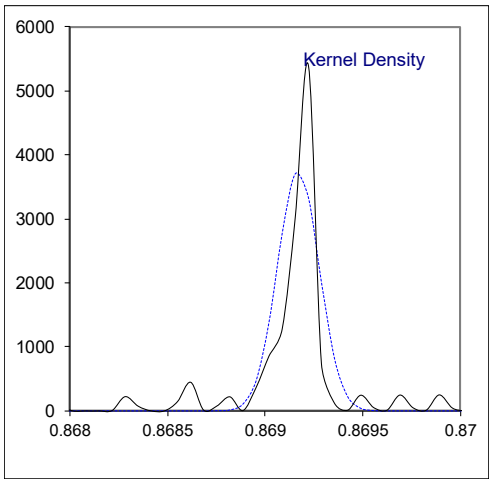



## Determination of Density at 15°C on sample #19236; results in kg/L

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D4052 | 0.8836 | R(0.01) | 80.81 | |
| 179 | D4052 | 0.8692 | | 0.17 | |
| 225 | | ----- | | ----- | |
| 237 | D4052 | 0.8697 | R(0.01) | 2.97 | |
| 255 | | ----- | | ----- | |
| 257 | | ----- | | ----- | |
| 311 | D4052 | 0.8692 | | 0.17 | |
| 317 | D4052 | 0.8692 | | 0.17 | |
| 325 | D4052 | 0.8692 | | 0.17 | |
| 331 | ISO12185 | 0.8683 | R(0.01) | -4.87 | |
| 335 | D1298 | 0.8692 | | 0.17 | |
| 339 | | ----- | | ----- | |
| 349 | D4052 | 0.8691 | | -0.39 | |
| 360 | D4052 | 0.8692 | | 0.17 | |
| 398 | ISO12185 | 0.8692 | C | 0.17 | reported 869.2 kg/L |
| 432 | D4052 | 0.86926 | | 0.51 | |
| 442 | | ----- | | ----- | |
| 451 | D4052 | 0.8688 | | -2.07 | |
| 496 | ISO12185 | 0.86928 | | 0.62 | |
| 526 | D4052 | 0.8692 | | 0.17 | |
| 541 | D4052 | 0.86920 | | 0.17 | |
| 562 | | ----- | | ----- | |
| 575 | | ----- | | ----- | |
| 603 | D4052 | 0.8692 | | 0.17 | |
| 614 | D4052 | 0.8692 | | 0.17 | |
| 621 | D4052 | 0.8692 | | 0.17 | |
| 633 | D4052 | 0.8686 | R(0.01) | -3.19 | |
| 634 | D4052 | 0.8692 | | 0.17 | |
| 780 | ISO12185 | 0.8691 | | -0.39 | |
| 862 | D4052 | 0.8691 | | -0.39 | |
| 863 | | ----- | | ----- | |
| 912 | ISO12185 | 0.8692 | | 0.17 | |
| 962 | D4052 | 0.8692 | | 0.17 | |
| 963 | D4052 | 0.8692 | | 0.17 | |
| 975 | D4052 | 0.8692 | | 0.17 | |
| 994 | ISO12185 | 0.8692 | | 0.17 | |
| 1059 | ISO12185 | 0.8692 | | 0.17 | |
| 1146 | D4052 | 0.8692 | | 0.17 | |
| 1166 | | ----- | | ----- | |
| 1265 | D4052 | 0.8691 | | -0.39 | |
| 1297 | D4052 | 0.86917 | | 0.01 | |
| 1397 | ISO12185 | 0.8692 | | 0.17 | |
| 1435 | D7042 | 0.8686 | R(0.01) | -3.19 | |
| 1505 | D7042 | 0.8667 | R(0.01) | -13.83 | |
| 1531 | | ----- | | ----- | |
| 1660 | D7042 | 0.8679 | R(0.01) | -7.11 | |
| 1720 | | ----- | | ----- | |
| 1740 | D7042 | 0.8690 | | -0.95 | |
| 1743 | ISO12185 | 0.8690 | | -0.95 | |
| 1788 | D4052 | 0.869145 | C | -0.13 | first reported 0.869145 kg/m$^3$ |
| 1857 | ISO12185 | 0.8695 | | 1.85 | |
| 1875 | D7042 | 0.8692 | | 0.17 | |
| 1888 | | ----- | | ----- | |
| 1900 | D4052 | 0.8692 | C | 0.17 | first reported 0.8692 kg/m$^3$ |
| 1920 | | ----- | | ----- | |
| 2493 | ISO12185 | 0.86917 | | 0.01 | |
| 6002 | ISO3675 | 0.8690 | | -0.95 | |
| 6016 | D4052 | 0.8692 | | 0.17 | |
| 6257 | ISO12185 | 0.86899 | | -1.00 | |
| 6261 | D4052 | 0.8699 | R(0.01) | 4.09 | |
| 6284 | D4052 | 0.869312 | | 0.80 | |
| 7011 | D1298 | 0.8691 | | -0.39 | |

| | |
|---|---|
| normality | not OK |
| n | 41 |
| outliers | 8 |
| mean (n) | 0.86917 |
| st.dev. (n) | 0.000106 |
| R(calc.) | 0.00030 |
| st.dev.(ISO12185:96) | 0.000179 |
| R(ISO12185:96) | 0.00050 |

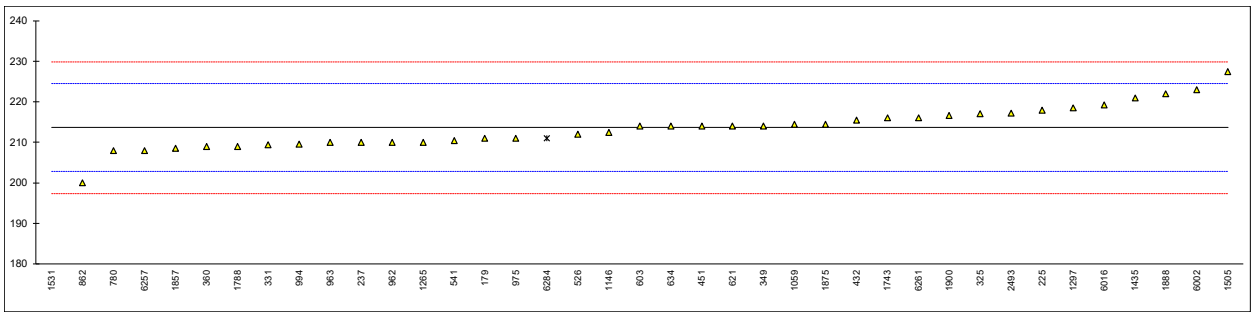

0.871
0.87
0.869
0.868
0.867
0.866
1505 1660 331 633 1435 451 6257 1740 1743 6002 349 862 780 1265 7011 1788 1297 2493 325 179 526 311 317 912 962 335 634 541 603 614 621 398 360 963 975 994 1059 1146 1397 1875 1900 6016 432 496 6284 1857 237 6261 178

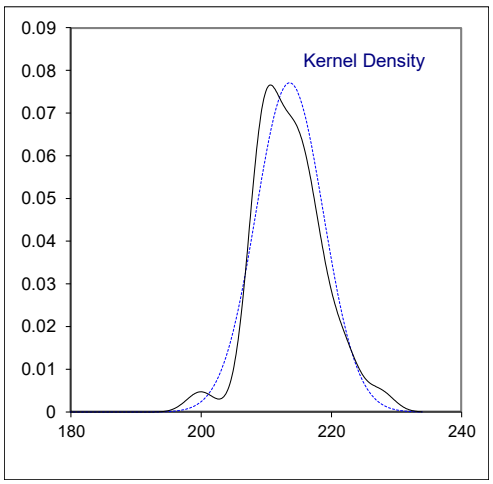

Kernel Density
6000
5000
4000
3000
2000
1000
0
0.868
0.8685
0.869
0.8695
0.87

## Determination of Flash Point PMcc on sample #19236; results in °C

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | | ----- | | ----- | |
| 179 | D93-A | 211 | | -0.49 | |
| 225 | D93-B | 218.0 | | 0.80 | |
| 237 | D93-B | 210 | | -0.67 | |
| 255 | | ----- | | ----- | |
| 257 | | ----- | | ----- | |
| 311 | | ----- | | ----- | |
| 317 | | ----- | | ----- | |
| 325 | D93-A | 217.0 | | 0.62 | |
| 331 | D93-A | 209.4 | | -0.78 | |
| 335 | | ----- | | ----- | |
| 339 | D93-B | >190 | | ----- | |
| 349 | D93-A | 214 | | 0.06 | |
| 360 | D93-A | 209.0 | | -0.86 | |
| 398 | | ----- | | ----- | |
| 432 | D93-A | 215.5 | | 0.34 | |
| 442 | | ----- | | ----- | |
| 451 | D93-A | 214 | | 0.06 | |
| 496 | | ----- | | ----- | |
| 526 | D93-B | 212 | | -0.30 | |
| 541 | D93-A | 210.50 | | -0.58 | |
| 562 | | ----- | | ----- | |
| 575 | | ----- | | ----- | |
| 603 | D93-A | 214 | | 0.06 | |
| 614 | | ----- | | ----- | |
| 621 | D93-A | 214.0 | | 0.06 | |
| 633 | | ----- | | ----- | |
| 634 | D93-B | 214.0 | | 0.06 | |
| 780 | D93-A | 208.0 | | -1.04 | |
| 862 | D93-A | 200 | | -2.52 | |
| 863 | | ----- | | ----- | |
| 912 | | ----- | | ----- | |
| 962 | D93-A | 210 | | -0.67 | |
| 963 | D93-A | 210.0 | | -0.67 | |
| 975 | D93-A | 211.0 | | -0.49 | |
| 994 | D93-A | 209.5 | | -0.77 | |
| 1059 | ISO2719-A | 214.5 | | 0.16 | |
| 1146 | D93-A | 212.5 | | -0.21 | |
| 1166 | | ----- | | ----- | |
| 1265 | D93 | 210 | | -0.67 | |
| 1297 | D93-B | 218.5 | | 0.90 | |
| 1397 | | ----- | | ----- | |
| 1435 | D93-A | 221.0 | | 1.36 | |
| 1505 | D93-A | 227.5 | | 2.56 | |
| 1531 | D93-B | 160 | R(0.01) | -9.90 | |
| 1660 | | ----- | | ----- | |
| 1720 | | ----- | | ----- | |
| 1740 | | ----- | | ----- | |
| 1743 | ISO2719-A | 216 | | 0.43 | |
| 1788 | D93-B | 209.0 | | -0.86 | |
| 1857 | D93-B | 208.5 | | -0.95 | |
| 1875 | ISO2719-B | 214.5 | | 0.16 | |
| 1888 | ISO2719-A | 222 | | 1.54 | |
| 1900 | D7094 | 216.7 | | 0.56 | |
| 1920 | | ----- | | ----- | |
| 2493 | D93-A | 217.2 | | 0.66 | |
| 6002 | ISO2719-A | 223.0 | | 1.73 | |
| 6016 | D93-A | 219.2 | | 1.02 | |
| 6257 | ISO2719-A | 208.0 | | -1.04 | |
| 6261 | D93-A | 216.0 | | 0.43 | |
| 6284 | D92 | 211 | ex,C | -0.49 | first reported 231, D93-A |
| 7011 | | ----- | | ----- | |

| | | | D93-A & ISO 2719-A only | D93-B & ISO 2719-B only |
|---|---|---|---|---|
| | normality | suspect | OK | OK |
| | n | 37 | 27 | 8 |
| | outliers | 1 +1ex | 0 | 1 |
| | mean (n) | 213.65 | 213.84 | 213.06 |
| | st.dev. (n) | 5.170 | 5.654 | 3.868 |
| | R(calc.) | 14.48 | 15.83 | 10.83 |
| | st.dev.(D93-A:19) | 5.418 | 5.421 | ----- |
| | R(D93-A:19) | 15.17 | 15.18 | ----- |
| Compare | | | | |
| | R(D93-B:19) | 10 | ----- | 10 |

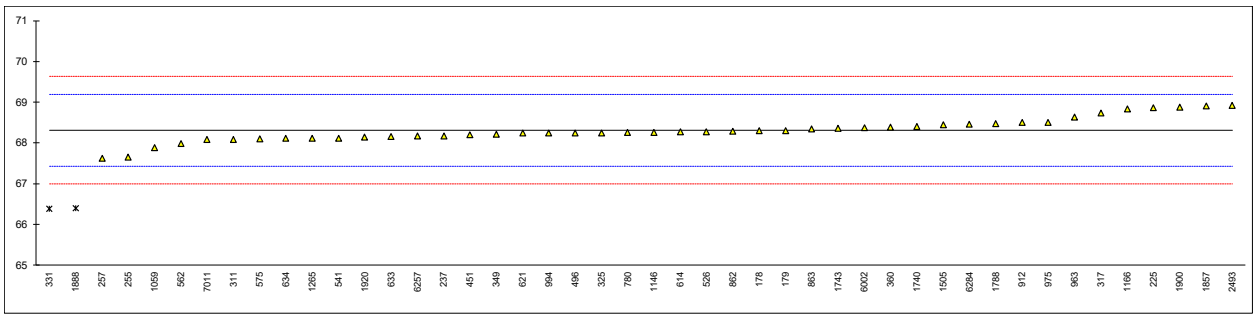

240
230
220
210
200
190
180
1531
862
780
6257
1857
360
1788
331
994
963
237
962
1265
541
179
975
6284
526
1146
603
634
451
621
349
1059
1875
432
1743
6261
1900
325
2493
225
1297
6016
1435
1888
6002
1505

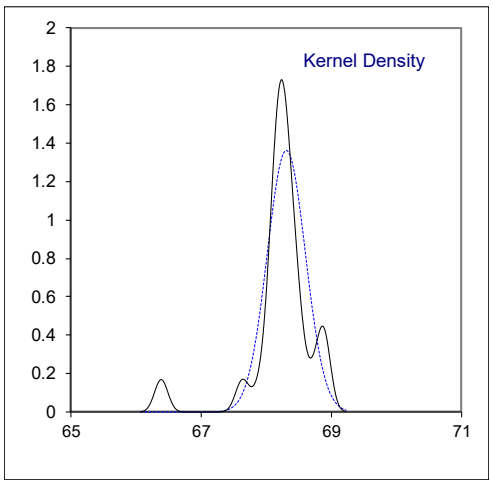

Kernel Density
0.09
0.08
0.07
0.06
0.05
0.04
0.03
0.02
0.01
0
180
200
220
240

## Determination of Kinematic Viscosity at 40°C on sample #19236; results in $mm^2/s$

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D445 | 68.3 | | -0.03 | |
| 179 | D445 | 68.31 | | -0.01 | |
| 225 | D445 | 68.87 | | 1.27 | |
| 237 | D445 | 68.18 | | -0.30 | |
| 255 | D7279 corrected to D445 | 67.66 | | -1.49 | |
| 257 | D7279 corrected to D445 | 67.62 | | -1.58 | |
| 311 | D445 | 68.09 | | -0.51 | |
| 317 | D445 | 68.74 | | 0.97 | |
| 325 | D445 | 68.245 | | -0.16 | |
| 331 | D7279Mod. | 66.39 | R(0.01) | -4.38 | |
| 335 | | ----- | | ----- | |
| 339 | | ----- | | ----- | |
| 349 | D445 | 68.22 | | -0.21 | |
| 360 | D445 | 68.383 | | 0.16 | |
| 398 | | ----- | | ----- | |
| 432 | | ----- | | ----- | |
| 442 | | ----- | | ----- | |
| 451 | D7279 corrected to D445 | 68.2 | C | -0.26 | first reported as Kinematic Viscosity at 100°C |
| 496 | D445 | 68.244 | | -0.16 | |
| 526 | D445 | 68.27 | | -0.10 | |
| 541 | D445 | 68.119 | | -0.44 | |
| 562 | D7279 | 67.9825 | | -0.75 | |
| 575 | D7279 corrected to D445 | 68.1 | | -0.49 | |
| 603 | | ----- | | ----- | |
| 614 | D445 | 68.27 | | -0.10 | |
| 621 | D445 | 68.24 | | -0.17 | |
| 633 | D7279 corrected to D445 | 68.158 | | -0.35 | |
| 634 | D7279 corrected to D445 | 68.11 | C | -0.46 | first reported 66.495 |
| 780 | D445 | 68.26 | | -0.12 | |
| 862 | D445 | 68.291 | | -0.05 | |
| 863 | D7279 | 68.34 | | 0.06 | |
| 912 | D445 | 68.50 | | 0.42 | |
| 962 | | ----- | | ----- | |
| 963 | D445 | 68.63 | | 0.72 | |
| 975 | D445 | 68.51 | | 0.45 | |
| 994 | D445 | 68.24 | | -0.17 | |
| 1059 | ISO3104 | 67.89 | | -0.96 | |
| 1146 | D445 | 68.263 | | -0.12 | |
| 1166 | ISO3104 | 68.8436 | | 1.21 | |
| 1265 | D7279 corrected to D445 | 68.11 | | -0.46 | |
| 1297 | | ----- | | ----- | |
| 1397 | | ----- | | ----- | |
| 1435 | | ----- | | ----- | |
| 1505 | D445 | 68.441 | | 0.29 | |
| 1531 | | ----- | | ----- | |
| 1660 | | ----- | | ----- | |
| 1720 | | ----- | | ----- | |
| 1740 | D445 | 68.40 | | 0.20 | |
| 1743 | D7279 corrected to D445 | 68.36 | | 0.11 | |
| 1788 | D445 | 68.4707 | | 0.36 | |
| 1857 | D445 | 68.912 | | 1.36 | |
| 1875 | | ----- | | ----- | |
| 1888 | D445 | 66.40 | C,R(0.01) | -4.36 | first reported 70.26 |
| 1900 | D445 | 68.876 | | 1.28 | |
| 1920 | D445 | 68.149 | | -0.37 | |
| 2493 | D445 | 68.92 | | 1.38 | |
| 6002 | ISO3104 | 68.37 | | 0.13 | |
| 6016 | | ----- | | ----- | |
| 6257 | ISO3104 | 68.1711 | | -0.32 | |
| 6261 | | ----- | W | ----- | test result withdrawn, reported 66.44 |
| 6284 | D445 | 68.46 | C | 0.33 | first reported 63.859887 |
| 7011 | D445 | 68.080 | | -0.53 | |

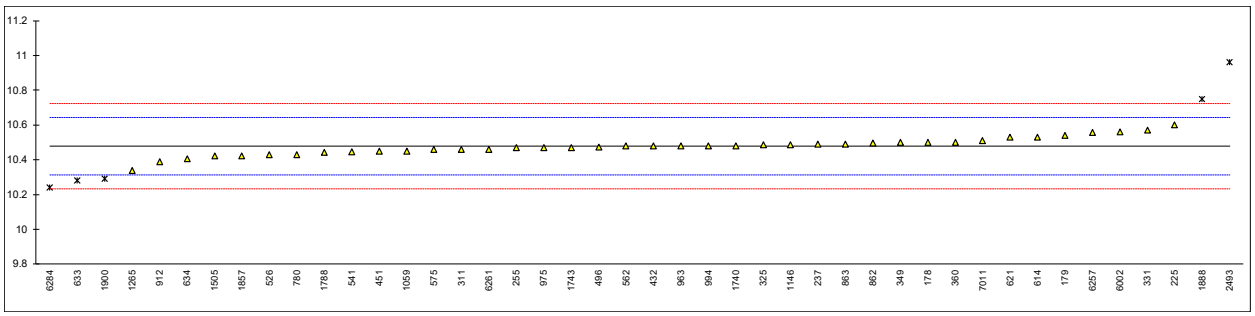
| | | | |
|---|---|---|---|
| | normality | OK | |
| | n | 44 | |
| | outliers | 2 | |
| | mean (n) | 68.3136 | |
| | st.dev. (n) | 0.29247 | |
| | R(calc.) | 0.8189 | |
| | st.dev.(iis memo 1401) | 0.43916 | |
| | R(iis memo 1401) | 1.2296 | see appendix 3, ref. 17 |
| Compare | | | |
| | R(D445:19) | 0.8567 | |

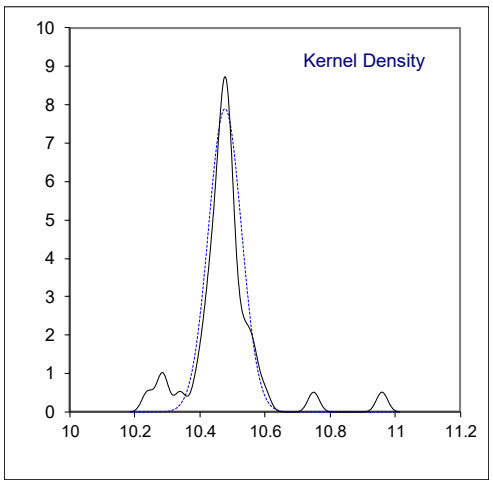

71
70
69
68
67
66
65
331 1888 257 255 1059 562 7011 311 575 634 1285 541 1920 633 6257 237 451 349 621 994 496 325 780 1146 614 526 862 178 179 863 1743 6002 360 1740 1505 6284 1788 912 975 963 317 1166 225 1900 1857 2493


Kernel Density
2
1.8
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
65
67
69
71

## Determination of Kinematic Viscosity at 100°C on sample #19236; results in $mm^2/s$

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D445 | 10.50 | | 0.27 | |
| 179 | D445 | 10.54 | | 0.75 | |
| 225 | D445 | 10.60 | | 1.48 | |
| 237 | D445 | 10.49 | | 0.15 | |
| 255 | D7279 corrected to D445 | 10.47 | | -0.10 | |
| 257 | | ----- | | ----- | |
| 311 | D445 | 10.46 | | -0.22 | |
| 317 | | ----- | | ----- | |
| 325 | D445 | 10.485 | | 0.09 | |
| 331 | D7279Mod. | 10.57 | | 1.12 | |
| 335 | | ----- | | ----- | |
| 339 | | ----- | | ----- | |
| 349 | D445 | 10.50 | | 0.27 | |
| 360 | D445 | 10.501 | | 0.28 | |
| 398 | | ----- | | ----- | |
| 432 | D445 | 10.48 | C | 0.03 | first reported as Kinematic Viscosity at 40°C |
| 442 | | ----- | | ----- | |
| 451 | D7279 corrected to D445 | 10.45 | C | -0.34 | first reported as Kinematic Viscosity at 40°C |
| 496 | D445 | 10.472 | | -0.07 | |
| 526 | D445 | 10.43 | | -0.58 | |
| 541 | D445 | 10.447 | | -0.38 | |
| 562 | D7279 | 10.48 | | 0.03 | |
| 575 | D7279 corrected to D445 | 10.46 | | -0.22 | |
| 603 | | ----- | | ----- | |
| 614 | D445 | 10.53 | | 0.63 | |
| 621 | D445 | 10.53 | | 0.63 | |
| 633 | D7279 corrected to D445 | 10.282 | R(0.05) | -2.38 | |
| 634 | D7279 corrected to D445 | 10.404 | | -0.90 | |
| 780 | D445 | 10.43 | | -0.58 | |
| 862 | D445 | 10.496 | | 0.22 | |
| 863 | D7279 | 10.49 | | 0.15 | |
| 912 | D445 | 10.39 | | -1.07 | |
| 962 | | ----- | | ----- | |
| 963 | D445 | 10.48 | | 0.03 | |
| 975 | D445 | 10.47 | | -0.10 | |
| 994 | D445 | 10.48 | | 0.03 | |
| 1059 | ISO3104 | 10.45 | | -0.34 | |
| 1146 | D445 | 10.486 | | 0.10 | |
| 1166 | | ----- | | ----- | |
| 1265 | D7279 corrected to D445 | 10.34 | | -1.67 | |
| 1297 | | ----- | | ----- | |
| 1397 | | ----- | | ----- | |
| 1435 | | ----- | | ----- | |
| 1505 | D445 | 10.422 | | -0.68 | |
| 1531 | | ----- | | ----- | |
| 1660 | | ----- | | ----- | |
| 1720 | | ----- | | ----- | |
| 1740 | D445 | 10.48 | | 0.03 | |
| 1743 | D7279 corrected to D445 | 10.47 | | -0.10 | |
| 1788 | D445 | 10.4444 | | -0.41 | |
| 1857 | D445 | 10.422 | | -0.68 | |
| 1875 | | ----- | | ----- | |
| 1888 | D445 | 10.75 | C,R(0.05) | 3.31 | first reported 11.18 |
| 1900 | D445 | 10.29 | R(0.05) | -2.28 | |
| 1920 | | ----- | | ----- | |
| 2493 | D445 | 10.96 | C,R(0.01) | 5.86 | first reported 11.02 |
| 6002 | ISO3104 | 10.560 | | 1.00 | |
| 6016 | | ----- | | ----- | |
| 6257 | ISO3104 | 10.5584 | | 0.98 | |
| 6261 | D445 | 10.46 | C | -0.22 | first reported 10.21 |
| 6284 | D445 | 10.24 | C,R(0.05) | -2.89 | first reported 9.9740256 |
| 7011 | D445 | 10.510 | | 0.39 | |
| | normality | OK | | | |
| | n | 39 | | | |
| | outliers | 5 | | | |
| | mean (n) | 10.4779 | | | |
| | st.dev. (n) | 0.05054 | | | |
| | R(calc.) | 0.1415 | | | |
| | st.dev.(iis memo 1401) | 0.08233 | | | |
| | R(iis memo 1401) | 0.2305 | | see appendix 3, ref. 17 | |
| Compare | | | | | |
| | R(D445:19) | 0.1046 | | | |

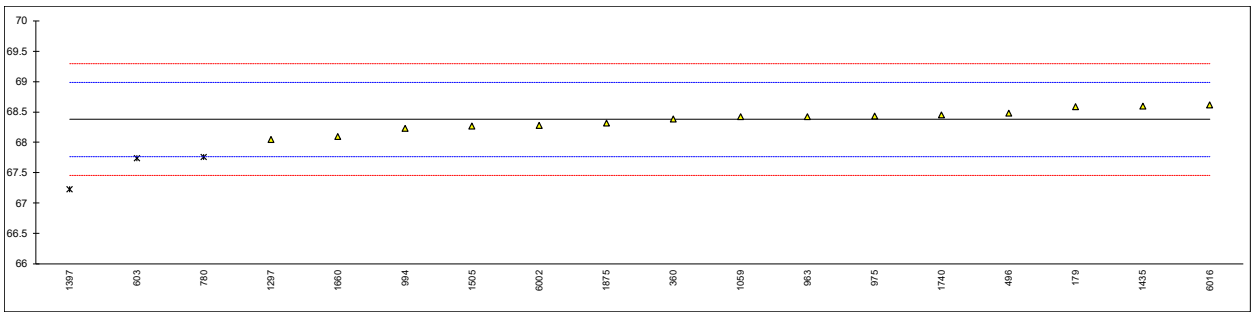

11.2
11
10.8
10.6
10.4
10.2
10
9.8
6284
633
1900
1265
912
634
1505
1857
526
780
1788
541
451
1059
575
311
6281
255
975
1743
496
562
432
963
994
1740
325
1146
237
863
862
349
178
360
7011
621
614
179
6257
6002
331
225
1888
2493

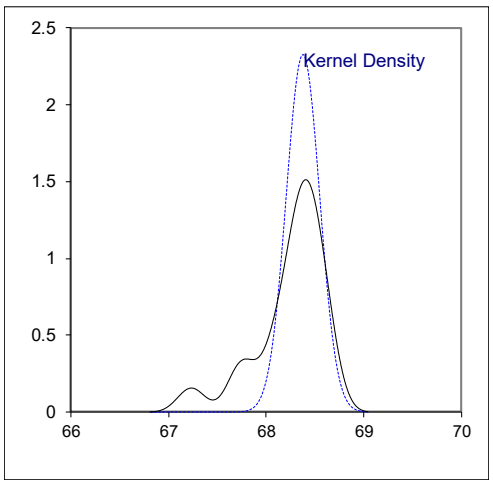

Kernel Density
10
9
8
7
6
5
4
3
2
1
0
10
10.2
10.4
10.6
10.8
11
11.2

## Determination of Viscosity Stabinger at 40°C on sample #19236; results in $mm^2/s$

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | | ----- | | ----- | |
| 179 | D7042 | 68.59 | | 0.70 | |
| 225 | | ----- | | ----- | |
| 237 | | ----- | | ----- | |
| 255 | | ----- | | ----- | |
| 257 | | ----- | | ----- | |
| 311 | | ----- | | ----- | |
| 317 | | ----- | | ----- | |
| 325 | | ----- | | ----- | |
| 331 | | ----- | | ----- | |
| 335 | | ----- | | ----- | |
| 339 | | ----- | | ----- | |
| 349 | | ----- | | ----- | |
| 360 | D7042 | 68.387 | | 0.04 | |
| 398 | | ----- | | ----- | |
| 432 | | ----- | | ----- | |
| 442 | | ----- | | ----- | |
| 451 | | ----- | | ----- | |
| 496 | D7042 | 68.480 | | 0.34 | |
| 526 | | ----- | | ----- | |
| 541 | | ----- | | ----- | |
| 562 | | ----- | | ----- | |
| 575 | | ----- | | ----- | |
| 603 | D7042 | 67.736 | DG(0.05) | -2.09 | |
| 614 | | ----- | | ----- | |
| 621 | | ----- | | ----- | |
| 633 | | ----- | | ----- | |
| 634 | | ----- | | ----- | |
| 780 | D7042 | 67.76 | DG(0.05) | -2.01 | |
| 862 | | ----- | | ----- | |
| 863 | | ----- | | ----- | |
| 912 | | ----- | | ----- | |
| 962 | | ----- | | ----- | |
| 963 | D7042 | 68.42 | | 0.14 | |
| 975 | D7042 | 68.43 | | 0.18 | |
| 994 | D7042 | 68.23 | | -0.48 | |
| 1059 | D7042 | 68.42 | | 0.14 | |
| 1146 | | ----- | | ----- | |
| 1166 | | ----- | | ----- | |
| 1265 | | ----- | | ----- | |
| 1297 | D7042 | 68.047 | | -1.07 | |
| 1397 | D7042 | 67.23 | C,G(0.05) | -3.74 | first reported 67.33 |
| 1435 | D7042 | 68.601 | | 0.73 | |
| 1505 | D7042 | 68.269 | | -0.35 | |
| 1531 | | ----- | | ----- | |
| 1660 | D7042 | 68.096 | | -0.91 | |
| 1720 | | ----- | | ----- | |
| 1740 | D7042 | 68.45 | | 0.24 | |
| 1743 | | ----- | | ----- | |
| 1788 | | ----- | | ----- | |
| 1857 | | ----- | | ----- | |
| 1875 | D7042 | 68.319 | | -0.19 | |
| 1888 | | ----- | | ----- | |
| 1900 | | ----- | | ----- | |
| 1920 | | ----- | | ----- | |
| 2493 | | ----- | | ----- | |
| 6002 | | 68.28 | | -0.31 | |
| 6016 | D7042 | 68.620 | | 0.80 | |
| 6257 | | ----- | | ----- | |
| 6261 | | ----- | W | ----- | test result withdrawn, reported 66.38 |
| 6284 | | ----- | | ----- | |
| 7011 | | ----- | | ----- | |

| | |
|---|---|
| normality | OK |
| n | 15 |
| outliers | 3 |
| mean (n) | 68.3759 |
| st.dev. (n) | 0.17135 |
| R(calc.) | 0.4798 |
| st.dev.(D7042:16e3) | 0.30660 |
| R(D7042:16e3) | 0.8585 |

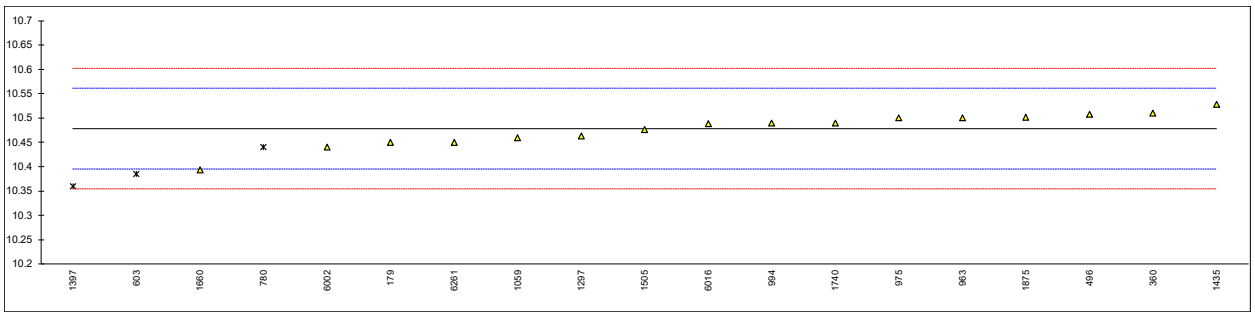

70
69.5
69
68.5
68
67.5
67
66.5
66
1397
603
780
1297
1660
994
1505
6002
1875
360
1059
963
975
1740
496
179
1435
6016

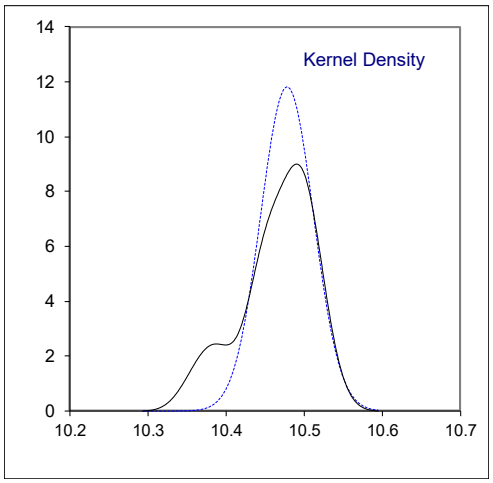

Kernel Density
2.5
2
1.5
1
0.5
0
66
67
68
69
70

## Determination of Viscosity Stabinger at 100°C on sample #19236; results in $mm^2/s$

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | | ----- | | ----- | |
| 179 | D7042 | 10.45 | | -0.68 | |
| 225 | | ----- | | ----- | |
| 237 | | ----- | | ----- | |
| 255 | | ----- | | ----- | |
| 257 | | ----- | | ----- | |
| 311 | | ----- | | ----- | |
| 317 | | ----- | | ----- | |
| 325 | | ----- | | ----- | |
| 331 | | ----- | | ----- | |
| 335 | | ----- | | ----- | |
| 339 | | ----- | | ----- | |
| 349 | | ----- | | ----- | |
| 360 | D7042 | 10.510 | | 0.77 | |
| 398 | | ----- | | ----- | |
| 432 | | ----- | | ----- | |
| 442 | | ----- | | ----- | |
| 451 | | ----- | | ----- | |
| 496 | D7042 | 10.508 | | 0.73 | |
| 526 | | ----- | | ----- | |
| 541 | | ----- | | ----- | |
| 562 | | ----- | | ----- | |
| 575 | | ----- | | ----- | |
| 603 | D7042 | 10.385 | ex | -2.25 | excluded as outlier at Viscosity Stabinger at 40°C |
| 614 | | ----- | | ----- | |
| 621 | | ----- | | ----- | |
| 633 | | ----- | | ----- | |
| 634 | | ----- | | ----- | |
| 780 | D7042 | 10.44 | ex | -0.92 | excluded as outlier at Viscosity Stabinger at 40°C |
| 862 | | ----- | | ----- | |
| 863 | | ----- | | ----- | |
| 912 | | ----- | | ----- | |
| 962 | | ----- | | ----- | |
| 963 | D7042 | 10.50 | | 0.53 | |
| 975 | D7042 | 10.50 | | 0.53 | |
| 994 | D7042 | 10.49 | | 0.29 | |
| 1059 | D7042 | 10.46 | | -0.44 | |
| 1146 | | ----- | | ----- | |
| 1166 | | ----- | | ----- | |
| 1265 | | ----- | | ----- | |
| 1297 | D7042 | 10.463 | | -0.36 | |
| 1397 | D7042 | 10.36 | ex | -2.85 | excluded as outlier at Viscosity Stabinger at 40°C |
| 1435 | D7042 | 10.528 | | 1.21 | |
| 1505 | D7042 | 10.476 | | -0.05 | |
| 1531 | | ----- | | ----- | |
| 1660 | D7042 | 10.393 | | -2.06 | |
| 1720 | | ----- | | ----- | |
| 1740 | D7042 | 10.49 | | 0.29 | |
| 1743 | | ----- | | ----- | |
| 1788 | | ----- | | ----- | |
| 1857 | | ----- | | ----- | |
| 1875 | D7042 | 10.502 | | 0.58 | |
| 1888 | | ----- | | ----- | |
| 1900 | | ----- | | ----- | |
| 1920 | | ----- | | ----- | |
| 2493 | | ----- | | ----- | |
| 6002 | | 10.440 | | -0.92 | |
| 6016 | D7042 | 10.488 | | 0.24 | |
| 6257 | | ----- | | ----- | |
| 6261 | D7042 | 10.45 | | -0.68 | |
| 6284 | | ----- | | ----- | |
| 7011 | | ----- | | ----- | |

| | |
|---|---|
| normality | suspect |
| n | 16 |
| outliers | 0 +3ex |
| mean (n) | 10.4780 |
| st.dev. (n) | 0.03381 |
| R(calc.) | 0.0947 |
| st.dev.(D7042:16e3) | 0.04136 |
| R(D7042:16e3) | 0.1158 |

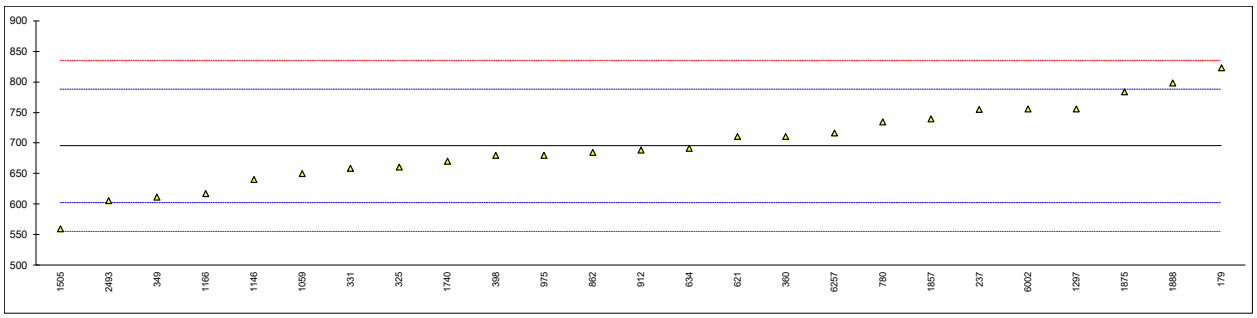

10.7
10.65
10.6
10.55
10.5
10.45
10.4
10.35
10.3
10.25
10.2
1397
603
1660
780
6002
179
6261
1059
1297
1505
6016
994
1740
975
963
1875
496
360
1435

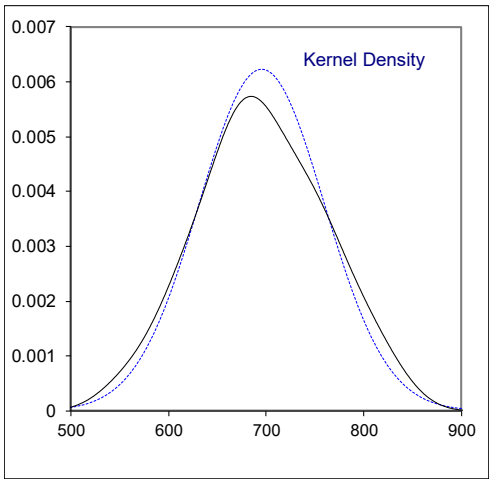

Kernel Density
14
12
10
8
6
4
2
0
10.2
10.3
10.4
10.5
10.6
10.7

## Determination of Sulfur on sample #19236; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | | ----- | | ----- | |
| 179 | D4294 | 823 | | 2.74 | |
| 225 | | ----- | | ----- | |
| 237 | D4294 | 755 | | 1.28 | |
| 255 | | ----- | | ----- | |
| 257 | | ----- | | ----- | |
| 311 | | ----- | | ----- | |
| 317 | | ----- | | ----- | |
| 325 | INH-6443 | 661 | C | -0.74 | first reported 0.0661mg/kg |
| 331 | ISO8754 | 658.25 | | -0.80 | |
| 335 | | ----- | | ----- | |
| 339 | INH-050 | <1000 | | ----- | |
| 349 | D2622 | 611 | | -1.81 | |
| 360 | ISO8754 | 711 | | 0.34 | |
| 398 | D4294 | 680 | | -0.33 | |
| 432 | | ----- | | ----- | |
| 442 | | ----- | | ----- | |
| 451 | | ----- | | ----- | |
| 496 | | ----- | | ----- | |
| 526 | | ----- | | ----- | |
| 541 | | ----- | | ----- | |
| 562 | | ----- | | ----- | |
| 575 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 614 | | ----- | | ----- | |
| 621 | D4294 | 711 | | 0.34 | |
| 633 | | ----- | | ----- | |
| 634 | D4294 | 691.5 | | -0.08 | |
| 780 | D4294 | 735 | | 0.85 | |
| 862 | D2622 | 684.9 | | -0.22 | |
| 863 | | ----- | | ----- | |
| 912 | D4294 | 688 | | -0.16 | |
| 962 | | ----- | | ----- | |
| 963 | | ----- | | ----- | |
| 975 | D4294 | 680 | | -0.33 | |
| 994 | | ----- | | ----- | |
| 1059 | ISO14596Mod. | 650 | | -0.97 | |
| 1146 | D4294 | 640 | | -1.19 | |
| 1166 | In house | 617.0 | | -1.68 | |
| 1265 | | ----- | | ----- | |
| 1297 | D4294 | 756.15 | | 1.31 | |
| 1397 | | ----- | | ----- | |
| 1435 | | ----- | | ----- | |
| 1505 | D5185 | 559.9 | | -2.91 | |
| 1531 | | ----- | | ----- | |
| 1660 | | ----- | | ----- | |
| 1720 | | ----- | | ----- | |
| 1740 | D4294 | 670 | | -0.54 | |
| 1743 | | ----- | | ----- | |
| 1788 | | ----- | | ----- | |
| 1857 | D4294 | 739.8 | | 0.96 | |
| 1875 | | 783.7 | | 1.90 | |
| 1888 | D5185 | 797.7 | C | 2.20 | first reported 1309.7 |
| 1900 | | ----- | | ----- | |
| 1920 | | ----- | | ----- | |
| 2493 | ISO10304-1 | 605.8 | | -1.92 | |
| 6002 | D5185 | 756 | | 1.31 | |
| 6016 | | ----- | | ----- | |
| 6257 | ISO8754 | 716 | C | 0.45 | first reported 160.55 |
| 6261 | | ----- | W | ----- | test result withdrawn, reported 0.27 |
| 6284 | | ----- | | ----- | |
| 7011 | | ----- | | ----- | |

| | |
|---|---|
| normality | OK |
| n | 25 |
| outliers | 0 |
| mean (n) | 695.3 |
| st.dev. (n) | 64.08 |
| R(calc.) | 179.4 |
| st.dev.(D4294:16e1) | 46.54 |
| R(D4294:16e1) | 130.3 |

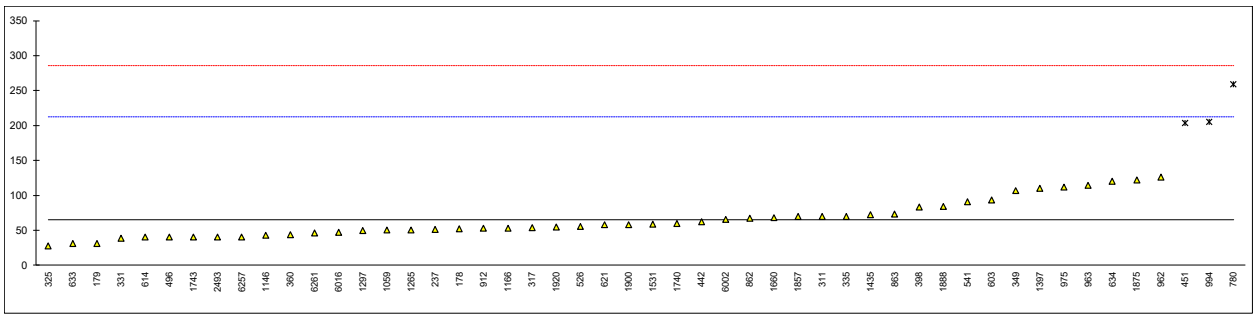

900
850
800
750
700
650
600
550
500
1505
2483
349
1166
1146
1059
331
325
1740
398
975
862
912
634
621
360
6257
780
1857
237
6002
1297
1875
1888
179

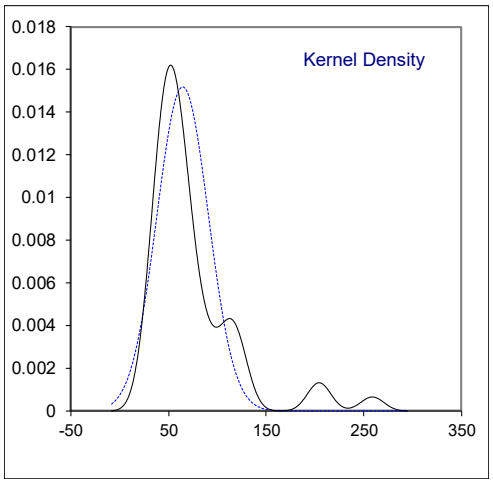

Kernel Density
0.007
0.006
0.005
0.004
0.003
0.002
0.001
0
500
600
700
800
900

## Determination of Water on sample #19236; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D6304-C | 52 | | -0.17 | |
| 179 | D6304-C | 31 | | -0.46 | |
| 225 | | ----- | | ----- | |
| 237 | D6304-C | 51.2 | | -0.18 | |
| 255 | | ----- | | ----- | |
| 257 | | ----- | | ----- | |
| 311 | D6304-A | 70 | | 0.07 | |
| 317 | D6304-A | 54 | | -0.15 | |
| 325 | D6304-C | 28 | | -0.50 | |
| 331 | D6304Mod. | 38.33 | | -0.36 | |
| 335 | D6304-A | 70 | | 0.07 | |
| 339 | | ----- | | ----- | |
| 349 | D6304-A | 107 | | 0.57 | |
| 360 | D6304-A | 43.4 | | -0.29 | |
| 398 | D6304-C | 83.4 | | 0.25 | |
| 432 | | ----- | | ----- | |
| 442 | IP438 | 62.0 | | -0.04 | |
| 451 | D6304-C | 204 | R(0.01) | 1.89 | |
| 496 | D6304-C | 40 | | -0.34 | |
| 526 | D6304-A | 55.84 | | -0.12 | |
| 541 | D6304-A | 90.5 | | 0.35 | |
| 562 | | ----- | | ----- | |
| 575 | | ----- | | ----- | |
| 603 | D6304-C | 93.0 | | 0.38 | |
| 614 | D6304-C | 40 | | -0.34 | |
| 621 | D6304-A | 58 | | -0.09 | |
| 633 | D6304-C | 30.7 | | -0.46 | |
| 634 | D6304-A | 120.5 | | 0.76 | |
| 780 | D6304-C | 259 | R(0.01) | 2.63 | |
| 862 | D6304-C | 67 | | 0.03 | |
| 863 | D6304-C | 73 | | 0.11 | |
| 912 | D6304-C | 53 | | -0.16 | |
| 962 | D6304-A | 126 | | 0.83 | |
| 963 | D6304-C | 114 | | 0.67 | |
| 975 | D6304-A | 112 | | 0.64 | |
| 994 | IP439 | 205 | R(0.01) | 1.90 | |
| 1059 | D6304-C | 50 | | -0.20 | |
| 1146 | D6304-C | 43 | | -0.30 | |
| 1166 | D6304-A | 53 | | -0.16 | |
| 1265 | D6304-C | 50.61 | | -0.19 | |
| 1297 | D6304-A | 49.2 | | -0.21 | |
| 1397 | ISO12937 | 110 | | 0.61 | |
| 1435 | D6304-A | 72.0 | | 0.10 | |
| 1505 | D6304-A | <10 | | ----- | |
| 1531 | D6304-A | 59.0 | | -0.08 | |
| 1660 | EN60814 | 68 | | 0.04 | |
| 1720 | | ----- | | ----- | |
| 1740 | D6304-C | 60 | | -0.07 | |
| 1743 | ISO12937 | 40 | | -0.34 | |
| 1788 | | ----- | | ----- | |
| 1857 | D6304-A | 69.7 | | 0.07 | |
| 1875 | ISO12937 | 122 | | 0.78 | |
| 1888 | EN60814 | 84.23 | | 0.26 | |
| 1900 | D6304-C | 58 | | -0.09 | |
| 1920 | D6304-C | 55 | | -0.13 | |
| 2493 | ISO12937 | 40 | | -0.34 | |
| 6002 | In house | 65.15 | | 0.00 | |
| 6016 | D6304-A | 47 | | -0.24 | |
| 6257 | | 40.2 | | -0.33 | |
| 6261 | D6304-A | 46.0 | | -0.26 | |
| 6284 | | ----- | | ----- | |
| 7011 | | ----- | | ----- | |

| | |
|---|---|
| normality | OK |
| n | 47 |
| outliers | 3 |
| mean (n) | 64.808 |
| st.dev. (n) | 26.2735 |
| R(calc.) | 73.566 |
| st.dev.(D6304:16e1) | 73.7040 |
| R(D6304:16e1) | 206.371 |

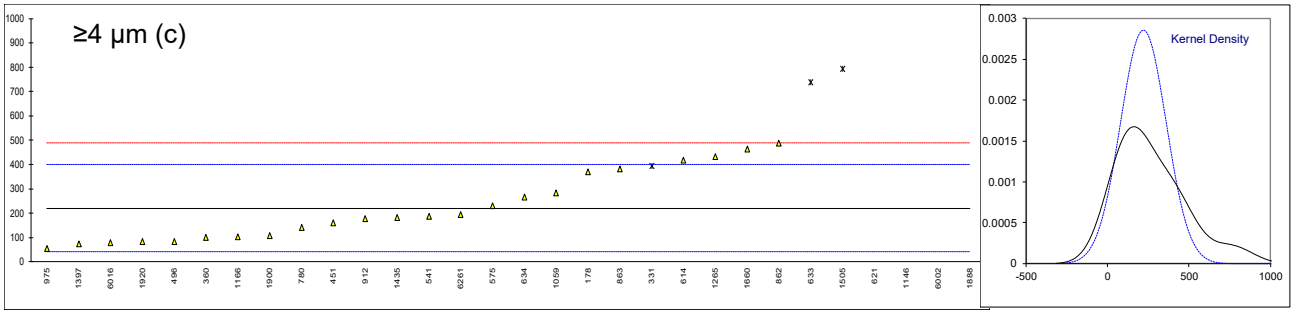

350
300
250
200
150
100
50
0
325 633 179 331 614 496 1743 2493 6257 1146 360 6261 6016 1297 1059 1265 237 178 912 1166 317 1920 526 621 1900 1531 1740 442 6002 862 1660 1857 311 335 1435 863 398 1888 541 603 349 1397 975 963 634 1875 962 451 994 780

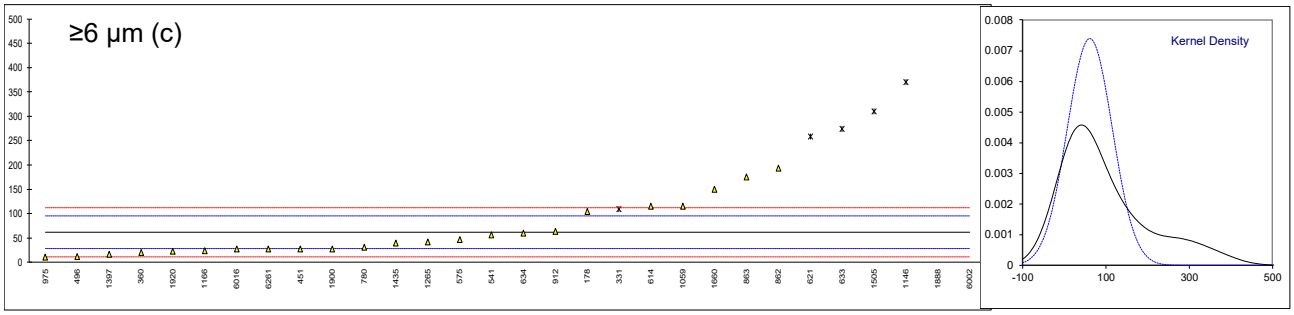

Kernel Density
0.018
0.016
0.014
0.012
0.01
0.008
0.006
0.004
0.002
0
-50
50
150
250
350

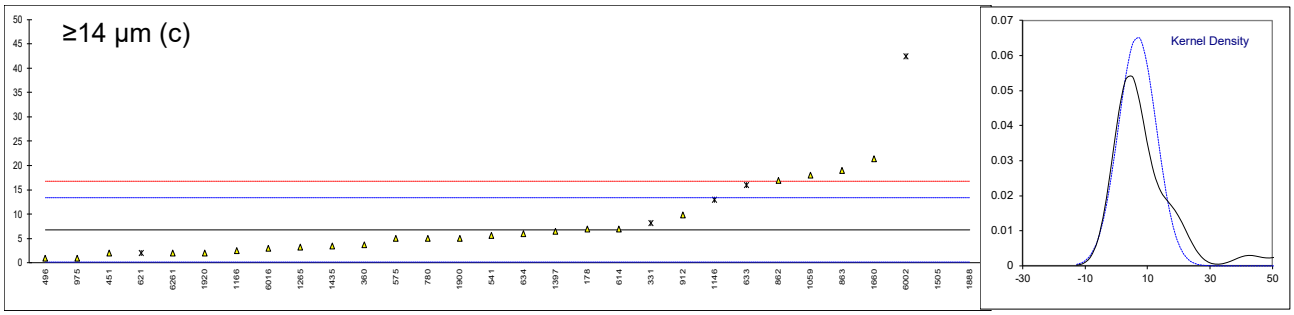
## Determination of Level of Contamination on sample #19236; results in counts/mL

| lab | method | ≥ 4 µm (c) | mark | z(targ) | ≥ 6 µm (c) | mark | z(targ) | ≥ 14 µm (c) | mark | z(targ) |
|---|---|---|---|---|---|---|---|---|---|---|
| 178 | ISO4406 | 369 | | 1.65 | 104 | | 2.53 | 7 | | 0.06 |
| 179 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 225 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 237 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 255 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 257 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 311 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 317 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 325 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 331 | ISO4406Mod. | 393.40 | ex | 1.92 | 108.8 | ex | 2.82 | 8.2 | ex | 0.42 |
| 335 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 339 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 349 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 360 | ISO4406 | 101.8 | | -1.32 | 20.6 | | -2.42 | 3.7 | | -0.94 |
| 398 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 432 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 442 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 451 | D7647 | 161 | | -0.66 | 28 | | -1.98 | 2 | | -1.45 |
| 496 | D7647 | 85 | | -1.51 | 12 | | -2.93 | 1 | | -1.75 |
| 526 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 541 | ISO11500 | 187.9 | | -0.36 | 55.9 | | -0.32 | 5.63 | | -0.35 |
| 562 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 575 | D7647 | 230 | | 0.11 | 47 | | -0.85 | 5 | | -0.54 |
| 603 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 614 | ISO4406 | 417 | C | 2.18 | 115 | C | 3.19 | 7 | C | 0.06 |
| 621 | ISO4407 | 1660 | R(0.01) | 16.00 | 258 | R(0.05) | 11.68 | 2 | ex | -1.45 |
| 633 | D7647 | 737.4 | R(0.05) | 5.75 | 274.0 | R(0.05) | 12.63 | 16.0 | ex | 2.78 |
| 634 | ISO11500 | 266 | | 0.51 | 60 | | -0.08 | 6 | | -0.24 |
| 780 | ISO11500 | 142 | | -0.87 | 31 | | -1.80 | 5 | | -0.54 |
| 862 | ISO11500 | 488 | | 2.97 | 194 | | 7.88 | 17 | | 3.08 |
| 863 | ISO11500 | 381 | | 1.78 | 175 | | 6.75 | 19 | | 3.68 |
| 912 | D7647 | 178 | | -0.47 | 63.5 | | 0.13 | 9.9 | | 0.94 |
| 962 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 963 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 975 | D7647 | 55 | | -1.84 | 11 | | -2.99 | 1 | | -1.75 |
| 994 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1059 | D7647 | 284 | | 0.71 | 115 | | 3.19 | 18 | | 3.38 |
| 1146 | ISO11500 | 1800 | R(0.01) | 17.56 | 370 | R(0.05) | 18.33 | 13 | ex | 1.87 |
| 1166 | ISO4407 | 102 | C | -1.32 | 24.40 | C | -2.20 | 2.50 | C | -1.30 |
| 1265 | ISO11500 | 433.6 | | 2.37 | 42.5 | | -1.12 | 3.2 | | -1.09 |
| 1297 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1397 | D7647 | 74.8 | | -1.62 | 17.2 | | -2.62 | 6.5 | | -0.09 |
| 1435 | D7647 | 183.40 | | -0.41 | 39.67 | | -1.29 | 3.53 | | -0.99 |
| 1505 | D7647 | 794 | R(0.05) | 6.38 | 310 | R(0.05) | 14.77 | 55 | R(0.01) | 14.54 |
| 1531 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1660 | | 465 | | 2.72 | 150 | | 5.27 | 21.4 | | 4.41 |
| 1720 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1740 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1743 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1788 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1857 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1875 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1888 | | 3332.17 | R(0.01) | 34.59 | 1497.43 | R(0.01) | 85.30 | 546.50 | R(0.01) | 162.85 |
| 1900 | D7596 | 109 | | -1.24 | 28 | | -1.98 | 5 | | -0.54 |
| 1920 | D7596 | 83.740 | | -1.52 | 22.806 | | -2.29 | 2.045 | | -1.43 |
| 2493 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 6002 | D7596 | 3088.6 | C,R(0.01) | 31.88 | 1627.8 | C,R(0.01) | 93.05 | 42.4 | C,R(0.01) | 10.74 |
| 6016 | ISO4406 | 79.5 | | -1.57 | 27.3 | | -2.02 | 3 | | -1.15 |
| 6257 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 6261 | ISO4407 | 194.8 | | -0.29 | 27.4 | | -2.02 | 2.0 | | -1.45 |
| 6284 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 7011 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| | normality | OK | | | suspect | | | suspect | | |
| | n | 23 | | | 23 | | | 23 | | |
| | outliers | 6 +1ex | | | 6 +1ex | | | 3 +4ex | | |
| | mean (n) | 220.502 | | | 61.360 | | | 6.800 | | |
| | st.dev. (n) | 139.8215 | | | 53.8951 | | | 6.0871 | | |
| | R(calc.) | 391.500 | | | 150.906 | | | 17.044 | | |
| | st.dev.(D7647:10) | 89.9520 | | | 16.8352 | | | 3.3142 | | |
| | R(D7647:10) | 251.866 | | | 47.139 | | | 9.280 | | |

Lab 331 test results excluded as statistical outliers in scale number
Lab 614 first reported as ISO 4406 scale number
Lab 621 test result excluded as two other related test results are statistical outliers
Lab 633 test result excluded as two other related test results are statistical outliers
Lab 1146 test result excluded as two other related test results are statistical outliers
Lab 1166 first reported 10200 at ≥4µm, 2440 at ≥6µm, 250 at ≥14µm
Lab 6002 first reported 4088.6 at ≥4µm, 1267.8 at ≥6µm, 52.4 at ≥14µm



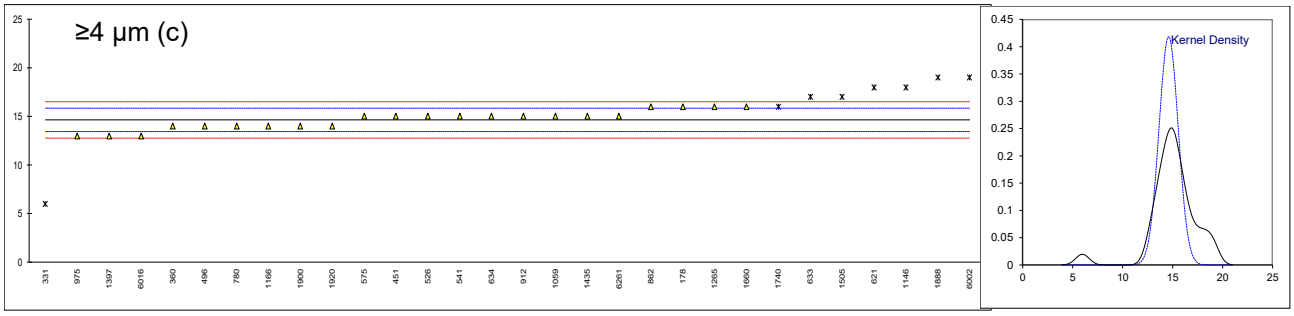



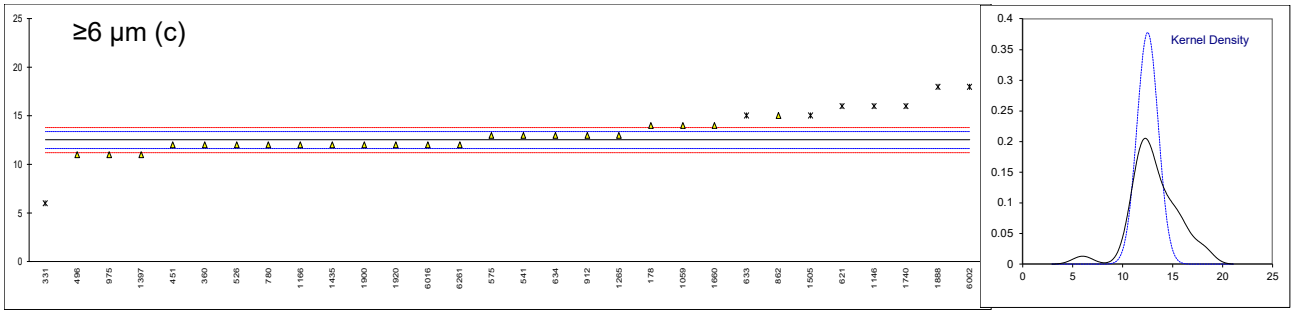



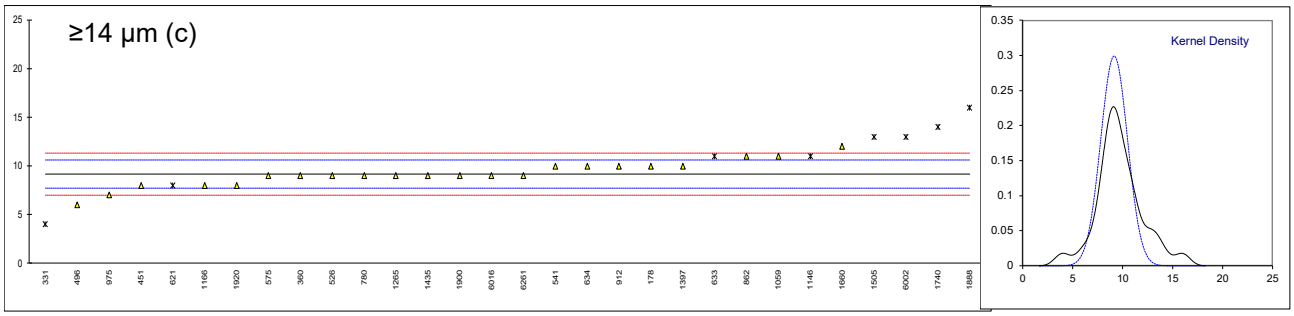
Determination of Level of Contamination acc. to ISO4406 scale on sample #19236;
results in scale number

| lab | method | ≥ 4 µm (c) | mark | z(targ) | ≥ 6 µm (c) | mark | z(targ) | ≥ 14 µm (c) | mark | z(targ) |
|---|---|---|---|---|---|---|---|---|---|---|
| 178 | ISO4406 | 16 | | 2.22 | 14 | | 3.46 | 10 | | 1.13 |
| 179 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 225 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 237 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 255 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 257 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 311 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 317 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 325 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 331 | ISO4406Mod. | 6 | E,R(0.01) | -14.07 | 6 | E,R(0.01) | -15.00 | 4 | E,R(0.05 | -7.18 |
| 335 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 339 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 349 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 360 | ISO4406 | 14 | | -1.04 | 12 | | -1.15 | 9 | | -0.25 |
| 398 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 432 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 442 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 451 | ISO4406 | 15 | | 0.59 | 12 | | -1.15 | 8 | | -1.64 |
| 496 | ISO4406 | 14 | | -1.04 | 11 | | -3.46 | 6 | E | -4.41 |
| 526 | ISO4406 | 15 | | 0.59 | 12 | | -1.15 | 9 | | -0.25 |
| 541 | ISO4406 | 15 | | 0.59 | 13 | | 1.15 | 10 | | 1.13 |
| 562 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 575 | ISO4406 | 15 | | 0.59 | 13 | | 1.15 | 9 | | -0.25 |
| 603 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 614 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 621 | ISO4406 | 18 | ex | 5.48 | 16 | E,ex | 8.08 | 8 | ex | -1.64 |
| 633 | ISO4406 | 17 | ex | 3.85 | 15 | ex | 5.77 | 11 | ex | 2.52 |
| 634 | ISO4406 | 15 | | 0.59 | 13 | | 1.15 | 10 | | 1.13 |
| 780 | ISO11500 | 14 | | -1.04 | 12 | | -1.15 | 9 | | -0.25 |
| 862 | ISO4406 | 16 | | 2.22 | 15 | | 5.77 | 11 | | 2.52 |
| 863 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 912 | D7647 | 15 | | 0.59 | 13 | | 1.15 | 10 | | 1.13 |
| 962 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 963 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 975 | ISO4406 | 13 | | -2.67 | 11 | | -3.46 | 7 | | -3.02 |
| 994 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1059 | ISO4406 | 15 | | 0.59 | 14 | | 3.46 | 11 | | 2.52 |
| 1146 | ISO4406 | 18 | ex | 5.48 | 16 | ex | 8.08 | 11 | ex | 2.52 |
| 1166 | ISO4406 | 14 | | -1.04 | 12 | | -1.15 | 8 | | -1.64 |
| 1265 | ISO4406 | 16 | | 2.22 | 13 | | 1.15 | 9 | | -0.25 |
| 1297 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1397 | D7647 | 13 | | -2.67 | 11 | | -3.46 | 10 | | 1.13 |
| 1435 | | 15 | | 0.59 | 12 | | -1.15 | 9 | | -0.25 |
| 1505 | ISO4406 | 17 | ex | 3.85 | 15 | ex | 5.77 | 13 | ex | 5.29 |
| 1531 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1660 | ISO4406 | 16 | | 2.22 | 14 | | 3.46 | 12 | | 3.90 |
| 1720 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1740 | ISO4406 | 16 | ex,C | 2.22 | 16 | ex,C | 8.08 | 14 | R(0.05) | 6.67 |
| 1743 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1788 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1857 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1875 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 1888 | ISO4406 | 19 | ex | 7.11 | 18 | ex | 12.70 | 16 | ex | 9.44 |
| 1900 | D7647 | 14 | | -1.04 | 12 | | -1.15 | 9 | | -0.25 |
| 1920 | ISO4406 | 14 | | -1.04 | 12 | | -1.15 | 8 | | -1.64 |
| 2493 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 6002 | ISO4406 | 19 | ex | 7.11 | 18 | ex,C | 12.70 | 13 | ex | 5.29 |
| 6016 | ISO4406 | 13 | | -2.67 | 12 | | -1.15 | 9 | | -0.25 |
| 6257 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 6261 | ISO4406 | 15 | | 0.59 | 12 | | -1.15 | 9 | E | -0.25 |
| 6284 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 7011 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| | normality | OK | | | OK | | | OK | | |
| | n | 22 | | | 22 | | | 22 | | |
| | outliers | 1 +7ex | | | 1 +7ex | | | 2 +6ex | | |
| | mean (n) | 14.64 | | | 12.50 | | | 9.18 | | |
| | st.dev. (n) | 0.953 | | | 1.058 | | | 1.332 | | |
| | R(calc.) | 2.67 | | | 2.96 | | | 3.73 | | |
| | st.dev.(D7647:10) | 0.614 | | | 0.433 | | | 0.722 | | |
| | R(D7647:10) | 1.72 | | | 1.21 | | | 2.02 | | |

Lab 331 no correct conversion from counts/mL to ISO4406 scale number respectively 16, 14, 10
Lab 496 no correct conversion from counts/mL to ISO4406 scale number ≥ 14 µm (c) =7
Lab 621 test results excluded as statistical outlier in counts/mL
Lab 621 no correct conversion from counts/mL to ISO4406 scale number ≥ 6 µm (c) =15
Lab 633 test results excluded as statistical outlier in counts/mL
Lab 1146 test results excluded as statistical outlier in counts/mL
Lab 1505 test results excluded as statistical outlier in counts/mL
Lab 1740 test results excluded as statistical outlier at related scale number ≥ 14 µm (c)
Lab 1888 test results excluded as statistical outlier in counts/mL
Lab 6002 test results excluded as statistical outlier in counts/mL
Lab 6261 no correct conversion from counts/mL to ISO4406 scale number ≥ 14 µm (c) =8

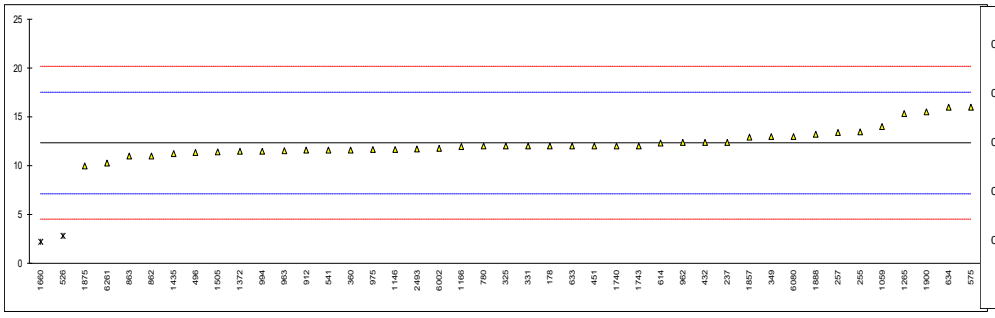


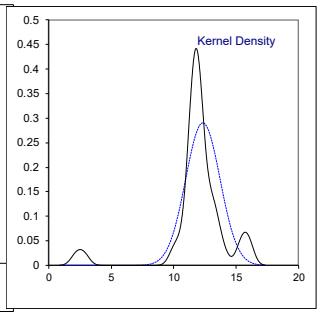



## Determination of Aluminum as Al on sample #19237; results in mg/kg

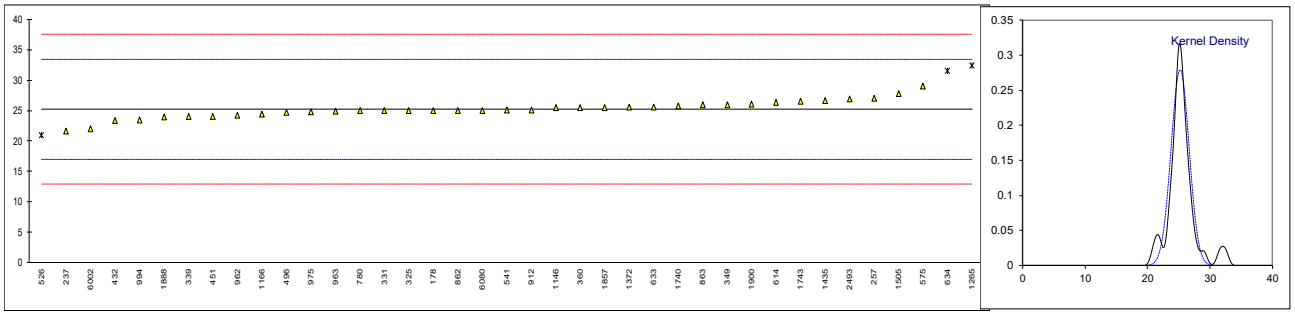
| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D5185 | 12 | | -0.13 | |
| 179 | | ----- | | ----- | |
| 237 | D5185 | 12.40 | | 0.03 | |
| 255 | D6595 | 13.45 | | 0.43 | |
| 257 | D6595 | 13.43 | | 0.42 | |
| 311 | | ----- | | ----- | |
| 325 | D5185 | 12 | | -0.13 | |
| 331 | D5185Mod. | 12 | | -0.13 | |
| 339 | | ----- | | ----- | |
| 349 | D5185 | 13 | | 0.26 | |
| 360 | D5185 | 11.6 | | -0.28 | |
| 398 | | ----- | | ----- | |
| 432 | D5185 | 12.4 | | 0.03 | |
| 451 | D5185 | 12 | | -0.13 | |
| 496 | D5185 | 11.35 | | -0.38 | |
| 526 | D5185 | 2.843 | C,R(0.01) | -3.64 | first reported 3 |
| 541 | D5185 | 11.6 | | -0.28 | |
| 575 | D6595 | 16 | | 1.41 | |
| 603 | | ----- | | ----- | |
| 614 | D5185 | 12.3 | | -0.01 | |
| 633 | D6595 | 12.0 | | -0.13 | |
| 634 | D6595 | 15.98 | | 1.40 | |
| 780 | D5185 | 12 | | -0.13 | |
| 862 | D5185 | 11 | | -0.51 | |
| 863 | D5185 | 11 | | -0.51 | |
| 912 | D5185 | 11.6 | | -0.28 | |
| 962 | D5185 | 12.37 | | 0.02 | |
| 963 | D5185 | 11.52 | | -0.31 | |
| 975 | D5185 | 11.64 | | -0.26 | |
| 994 | D5185 | 11.5 | | -0.32 | |
| 1059 | In house | 14 | | 0.64 | |
| 1146 | D5185 | 11.66 | | -0.26 | |
| 1166 | In house | 11.93 | | -0.15 | |
| 1265 | D6595 | 15.322 | | 1.15 | |
| 1297 | | ----- | | ----- | |
| 1372 | D5185 | 11.46 | | -0.33 | |
| 1435 | D5185 | 11.25 | | -0.41 | |
| 1505 | D5185 | 11.39 | | -0.36 | |
| 1660 | D5185 | 2.2 | R(0.01) | -3.88 | |
| 1740 | D5185 | 12 | | -0.13 | |
| 1743 | D5185 | 12 | | -0.13 | |
| 1857 | D5185 | 12.9 | | 0.22 | |
| 1875 | | 10 | | -0.89 | |
| 1888 | D5185 | 13.2 | | 0.33 | |
| 1900 | D5185 | 15.51 | | 1.22 | |
| 2493 | In house | 11.7 | | -0.24 | |
| 6002 | D5185 | 11.8 | | -0.20 | |
| 6016 | | ----- | | ----- | |
| 6080 | D5185 | 13 | | 0.26 | |
| 6261 | D5185 | 10.301 | | -0.78 | |
| | normality | not OK | | | |
| | n | 41 | | | |
| | outliers | 2 | | | |
| | mean (n) | 12.331 | | | |
| | st.dev. (n) | 1.3741 | | | |
| | R(calc.) | 3.848 | | | |
| | st.dev.(D5185:18) | 2.6079 | | | |
| | R(D5185:18) | 7.302 | | | application range 6 – 40 mg/kg |





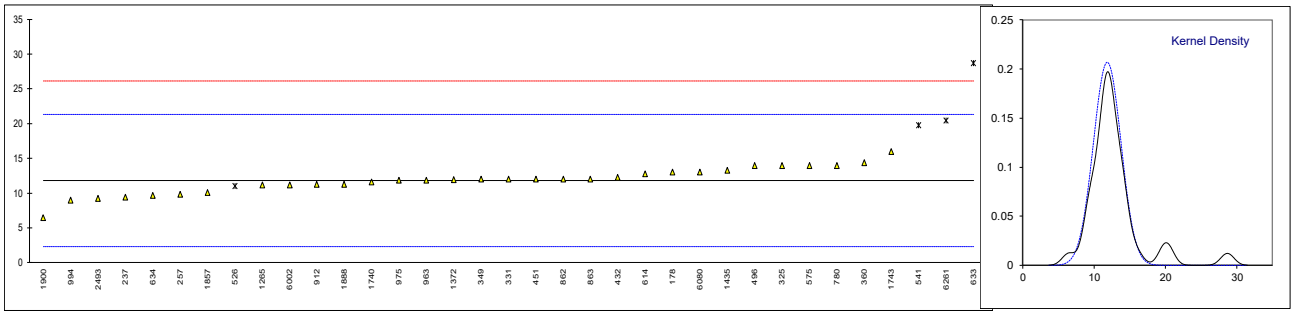
## Determination of Barium as Ba on sample #19237; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D5185 | 25 | | -0.05 | |
| 179 | | ----- | | ----- | |
| 237 | D5185 | 21.63 | C | -0.87 | first reported 14.59 |
| 255 | | ----- | | ----- | |
| 257 | D6595 | 27.06 | | 0.45 | |
| 311 | | ----- | | ----- | |
| 325 | D5185 | 25 | | -0.05 | |
| 331 | D5185Mod. | 25 | | -0.05 | |
| 339 | INH-165 | 24 | | -0.29 | |
| 349 | D5185 | 26 | | 0.19 | |
| 360 | D5185 | 25.5 | | 0.07 | |
| 398 | | ----- | | ----- | |
| 432 | D5185 | 23.4 | | -0.44 | |
| 451 | D5185 | 24 | | -0.29 | |
| 496 | D5185 | 24.67 | | -0.13 | |
| 526 | D5185 | 21 | ex | -1.03 | excluded as many statistical outliers in other related metal analyzes |
| 541 | D5185 | 25.07 | | -0.03 | |
| 575 | D6595 | 29 | | 0.92 | |
| 603 | | ----- | | ----- | |
| 614 | D5185 | 26.33 | | 0.27 | |
| 633 | D6595 | 25.6 | | 0.10 | |
| 634 | D6595 | 31.57 | R(0.01) | 1.55 | |
| 780 | D5185 | 25 | | -0.05 | |
| 862 | D5185 | 25 | | -0.05 | |
| 863 | D5185 | 26 | | 0.19 | |
| 912 | D5185 | 25.1 | | -0.03 | |
| 962 | D5185 | 24.28 | | -0.23 | |
| 963 | D5185 | 24.86 | | -0.08 | |
| 975 | D5185 | 24.84 | | -0.09 | |
| 994 | D5185 | 23.5 | | -0.42 | |
| 1059 | | ----- | | ----- | |
| 1146 | D5185 | 25.49 | | 0.07 | |
| 1166 | In house | 24.46 | | -0.18 | |
| 1265 | D6595 | 32.445 | R(0.01) | 1.76 | |
| 1297 | | ----- | | ----- | |
| 1372 | D5185 | 25.55 | | 0.08 | |
| 1435 | D5185 | 26.68 | | 0.36 | |
| 1505 | D5185 | 27.79 | | 0.63 | |
| 1660 | | ----- | | ----- | |
| 1740 | D5185 | 25.8 | | 0.14 | |
| 1743 | D5185 | 26.5 | | 0.31 | |
| 1857 | D5185 | 25.52 | | 0.08 | |
| 1875 | | ----- | | ----- | |
| 1888 | D5185 | 23.9 | | -0.32 | |
| 1900 | D5185 | 26.067 | | 0.21 | |
| 2493 | In house | 26.9 | | 0.41 | |
| 6002 | D5185 | 22.0 | | -0.78 | |
| 6016 | | ----- | | ----- | |
| 6080 | D5185 | 25 | | -0.05 | |
| 6261 | | ----- | | ----- | |
| | normality | suspect | | | |
| | n | 36 | | | |
| | outliers | 2 +1ex | | | |
| | mean (n) | 25.208 | | | |
| | st.dev. (n) | 1.4290 | | | |
| | R(calc.) | 4.001 | | | |
| | st.dev.(D5185:18) | 4.1031 | | | |
| | R(D5185:18) | 11.489 | | | application range 0.5 – 4 mg/kg |



## Determination of Boron as B on sample #19237; results in mg/kg

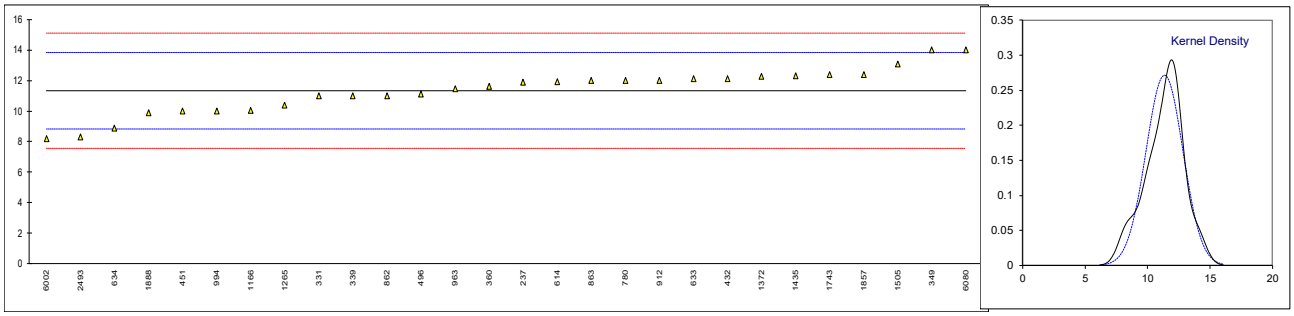
| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D5185 | 13 | | 0.25 | |
| 179 | | ----- | | ----- | |
| 237 | D5185 | 9.42 | | -0.51 | |
| 255 | | ----- | | ----- | |
| 257 | D6595 | 9.85 | | -0.42 | |
| 311 | | ----- | | ----- | |
| 325 | D5185 | 14 | | 0.46 | |
| 331 | D5185Mod. | 12 | | 0.04 | |
| 339 | INH-165 | <50 | | ----- | |
| 349 | D5185 | 12 | | 0.04 | |
| 360 | D5185 | 14.4 | | 0.54 | |
| 398 | | ----- | | ----- | |
| 432 | D5185 | 12.3 | | 0.10 | |
| 451 | D5185 | 12 | | 0.04 | |
| 496 | D5185 | 13.99 | | 0.45 | |
| 526 | D5185 | 11 | ex | -0.17 | excluded as many statistical outliers in other related metal analyzes |
| 541 | D5185 | 19.8 | R(0.05) | 1.67 | |
| 575 | D6595 | 14 | | 0.46 | |
| 603 | | ----- | | ----- | |
| 614 | D5185 | 12.8 | | 0.20 | |
| 633 | D6595 | 28.7 | R(0.01) | 3.55 | |
| 634 | D6595 | 9.68 | | -0.45 | |
| 780 | D5185 | 14 | | 0.46 | |
| 862 | D5185 | 12 | | 0.04 | |
| 863 | D5185 | 12 | | 0.04 | |
| 912 | D5185 | 11.3 | | -0.11 | |
| 962 | | ----- | | ----- | |
| 963 | D5185 | 11.85 | | 0.00 | |
| 975 | D5185 | 11.84 | | 0.00 | |
| 994 | D5185 | 9.0 | | -0.59 | |
| 1059 | | ----- | | ----- | |
| 1146 | | ----- | | ----- | |
| 1166 | | ----- | | ----- | |
| 1265 | D6595 | 11.148 | | -0.14 | |
| 1297 | | ----- | | ----- | |
| 1372 | D5185 | 11.97 | | 0.03 | |
| 1435 | D5185 | 13.30 | | 0.31 | |
| 1505 | | ----- | | ----- | |
| 1660 | | ----- | | ----- | |
| 1740 | D5185 | 11.6 | | -0.05 | |
| 1743 | D5185 | 16 | | 0.88 | |
| 1857 | D5185 | 10.1 | | -0.36 | |
| 1875 | | ----- | | ----- | |
| 1888 | D5185 | 11.3 | | -0.11 | |
| 1900 | D5185 | 6.437 | | -1.13 | |
| 2493 | In house | 9.21 | | -0.55 | |
| 6002 | D5185 | 11.2 | | -0.13 | |
| 6016 | | ----- | | ----- | |
| 6080 | D5185 | 13 | | 0.25 | |
| 6261 | D5185 | 20.413 | R(0.05) | 1.80 | |
| | normality | suspect | | | |
| | n | 31 | | | |
| | outliers | 3 +1ex | | | |
| | mean (n) | 11.829 | | | |
| | st.dev. (n) | 1.9304 | | | |
| | R(calc.) | 5.405 | | | |
| | st.dev.(D5185:18) | 4.7590 | | | |
| | R(D5185:18) | 13.325 | | | application range 4 – 30 mg/kg |



## Determination of Cadmium as Cd on sample #19237; results in mg/kg

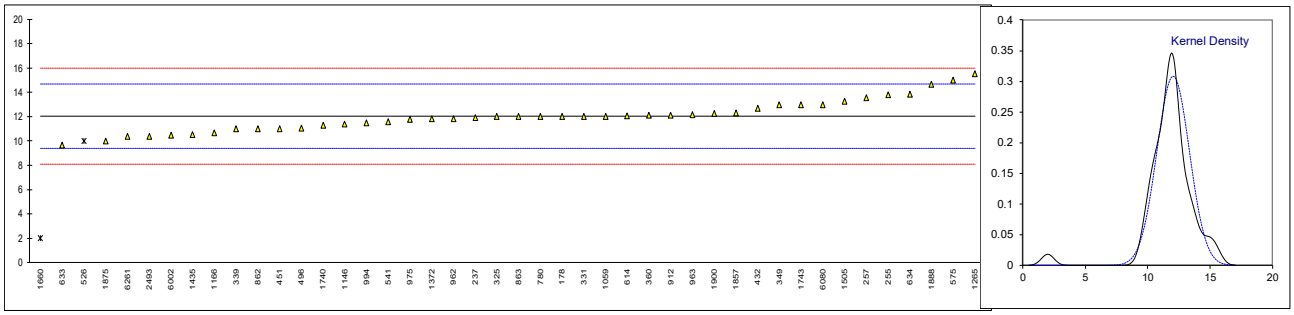
| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | | ----- | | ----- | |
| 179 | | ----- | | ----- | |
| 237 | D5185 | 11.87 | | 0.43 | |
| 255 | | ----- | | ----- | |
| 257 | | ----- | | ----- | |
| 311 | | ----- | | ----- | |
| 325 | | ----- | | ----- | |
| 331 | D5185Mod. | 11 | | -0.26 | |
| 339 | INH-165 | 11 | | -0.26 | |
| 349 | D5185 | 14 | | 2.12 | |
| 360 | D5185 | 11.6 | | 0.21 | |
| 398 | | ----- | | ----- | |
| 432 | D5185 | 12.1 | | 0.61 | |
| 451 | D5185 | 10 | | -1.06 | |
| 496 | D5185 | 11.11 | | -0.18 | |
| 526 | | ----- | | ----- | |
| 541 | | ----- | | ----- | |
| 575 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 614 | D5185 | 11.94 | | 0.48 | |
| 633 | D6595 | 12.1 | | 0.61 | |
| 634 | D6595 | 8.87 | | -1.96 | |
| 780 | D5185 | 12 | | 0.53 | |
| 862 | D5185 | 11 | | -0.26 | |
| 863 | D5185 | 12 | | 0.53 | |
| 912 | D5185 | 12.0 | | 0.53 | |
| 962 | | ----- | | ----- | |
| 963 | D5185 | 11.45 | | 0.09 | |
| 975 | | ----- | | ----- | |
| 994 | D5185 | 10.0 | | -1.06 | |
| 1059 | | ----- | | ----- | |
| 1146 | | ----- | | ----- | |
| 1166 | In house | 10.04 | | -1.03 | |
| 1265 | D6595 | 10.368 | | -0.77 | |
| 1297 | | ----- | | ----- | |
| 1372 | D5185 | 12.26 | | 0.74 | |
| 1435 | D5185 | 12.31 | | 0.78 | |
| 1505 | D5185 | 13.08 | | 1.39 | |
| 1660 | | ----- | | ----- | |
| 1740 | | ----- | | ----- | |
| 1743 | D5185 | 12.4 | | 0.85 | |
| 1857 | D5185 | 12.4 | | 0.85 | |
| 1875 | | ----- | | ----- | |
| 1888 | D5185 | 9.9 | | -1.14 | |
| 1900 | | ----- | | ----- | |
| 2493 | In house | 8.29 | | -2.42 | |
| 6002 | D5185 | 8.21 | C | -2.48 | first reported 6.21 |
| 6016 | | ----- | | ----- | |
| 6080 | D5185 | 14 | | 2.12 | |
| 6261 | | ----- | | ----- | |

| | |
|---|---|
| normality | OK |
| n | 28 |
| outliers | 0 |
| mean (n) | 11.332 |
| st.dev. (n) | 1.4694 |
| R(calc.) | 4.114 |
| st.dev.(Horwitz) | 1.2582 |
| R(Horwitz) | 3.523 |



## Determination of Chromium as Cr on sample #19237; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D5185 | 12 | | -0.03 | |
| 179 | | ----- | | ----- | |
| 237 | D5185 | 11.91 | | -0.10 | |
| 255 | D6595 | 13.82 | | 1.35 | |
| 257 | D6595 | 13.57 | | 1.16 | |
| 311 | | ----- | | ----- | |
| 325 | D5185 | 12 | | -0.03 | |
| 331 | D5185Mod. | 12 | | -0.03 | |
| 339 | INH-165 | 11 | | -0.79 | |
| 349 | D5185 | 13 | | 0.73 | |
| 360 | D5185 | 12.1 | | 0.04 | |
| 398 | | ----- | | ----- | |
| 432 | D5185 | 12.7 | | 0.50 | |
| 451 | D5185 | 11 | | -0.79 | |
| 496 | D5185 | 11.067 | | -0.74 | |
| 526 | D5185 | 10 | ex | -1.55 | excluded as many statistical outliers in other related metal analyzes |
| 541 | D5185 | 11.6 | | -0.34 | |
| 575 | D6595 | 15 | | 2.24 | |
| 603 | | ----- | | ----- | |
| 614 | D5185 | 12.09 | | 0.04 | |
| 633 | D6595 | 9.68 | | -1.79 | |
| 634 | D6595 | 13.83 | | 1.35 | |
| 780 | D5185 | 12 | | -0.03 | |
| 862 | D5185 | 11 | | -0.79 | |
| 863 | D5185 | 12 | | -0.03 | |
| 912 | D5185 | 12.1 | | 0.04 | |
| 962 | D5185 | 11.85 | | -0.15 | |
| 963 | D5185 | 12.18 | | 0.10 | |
| 975 | D5185 | 11.8 | | -0.18 | |
| 994 | D5185 | 11.5 | | -0.41 | |
| 1059 | In house | 12 | | -0.03 | |
| 1146 | D5185 | 11.41 | | -0.48 | |
| 1166 | In house | 10.69 | | -1.02 | |
| 1265 | D6595 | 15.510 | | 2.63 | |
| 1297 | | ----- | | ----- | |
| 1372 | D5185 | 11.81 | | -0.18 | |
| 1435 | D5185 | 10.52 | | -1.15 | |
| 1505 | D5185 | 13.26 | | 0.92 | |
| 1660 | D5185 | 2.0 | R(0.01) | -7.61 | |
| 1740 | D5185 | 11.3 | | -0.56 | |
| 1743 | D5185 | 13 | | 0.73 | |
| 1857 | D5185 | 12.3 | | 0.19 | |
| 1875 | | 10 | | -1.55 | |
| 1888 | D5185 | 14.67 | C | 1.99 | first reported 20.6 |
| 1900 | D5185 | 12.27 | | 0.17 | |
| 2493 | In house | 10.4 | | -1.24 | |
| 6002 | D5185 | 10.5 | | -1.17 | |
| 6016 | | ----- | | ----- | |
| 6080 | D5185 | 13 | | 0.73 | |
| 6261 | D5185 | 10.363 | | -1.27 | |
| | | | | | |
| | normality | OK | | | |
| | n | 42 | | | |
| | outliers | 1 +1ex | | | |
| | mean (n) | 12.043 | | | |
| | st.dev. (n) | 1.2954 | | | |
| | R(calc.) | 3.627 | | | |
| | st.dev.(D5185:18) | 1.3200 | | | |
| | R(D5185:18) | 3.696 | | | application range 1 – 40 mg/kg |

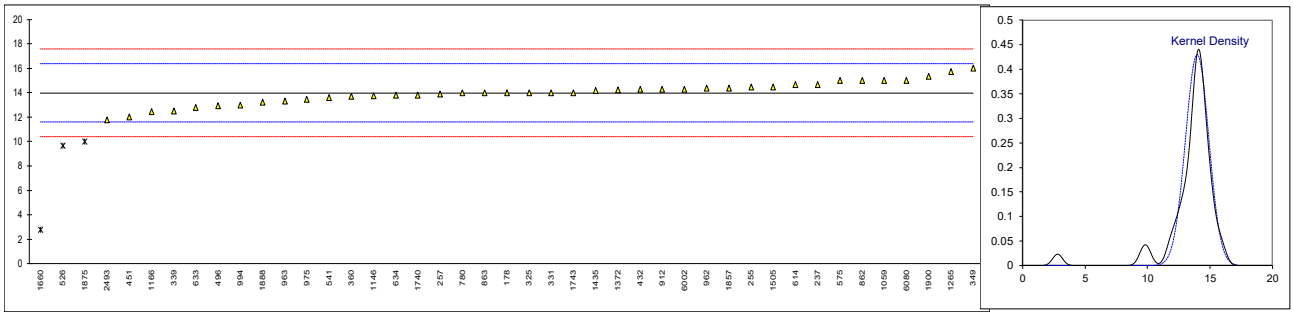


## Determination of Copper as Cu on sample #19237; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D5185 | 14 | | 0.01 | |
| 179 | | ----- | | ----- | |
| 237 | D5185 | 14.69 | | 0.59 | |
| 255 | D6595 | 14.46 | | 0.40 | |
| 257 | D6595 | 13.89 | | -0.08 | |
| 311 | | ----- | | ----- | |
| 325 | D5185 | 14 | | 0.01 | |
| 331 | D5185Mod. | 14 | | 0.01 | |
| 339 | INH-165 | 12.5 | | -1.24 | |
| 349 | D5185 | 16 | | 1.68 | |
| 360 | D5185 | 13.7 | | -0.24 | |
| 398 | | ----- | | ----- | |
| 432 | D5185 | 14.3 | | 0.26 | |
| 451 | D5185 | 12 | | -1.66 | |
| 496 | D5185 | 12.93 | | -0.88 | |
| 526 | D5185 | 9.660 | C,R(0.01) | -3.61 | first reported 10 |
| 541 | D5185 | 13.6 | | -0.32 | |
| 575 | D6595 | 15 | | 0.85 | |
| 603 | | ----- | | ----- | |
| 614 | D5185 | 14.65 | | 0.56 | |
| 633 | D6595 | 12.8 | | -0.99 | |
| 634 | D6595 | 13.78 | | -0.17 | |
| 780 | D5185 | 14 | | 0.01 | |
| 862 | D5185 | 15 | | 0.85 | |
| 863 | D5185 | 14 | | 0.01 | |
| 912 | D5185 | 14.3 | | 0.26 | |
| 962 | D5185 | 14.38 | | 0.33 | |
| 963 | D5185 | 13.31 | | -0.56 | |
| 975 | D5185 | 13.48 | | -0.42 | |
| 994 | D5185 | 13.0 | | -0.82 | |
| 1059 | In house | 15 | | 0.85 | |
| 1146 | D5185 | 13.76 | | -0.19 | |
| 1166 | In house | 12.44 | | -1.29 | |
| 1265 | D6595 | 15.741 | | 1.47 | |
| 1297 | | ----- | | ----- | |
| 1372 | D5185 | 14.21 | | 0.19 | |
| 1435 | D5185 | 14.19 | | 0.17 | |
| 1505 | D5185 | 14.46 | | 0.40 | |
| 1660 | D5185 | 2.8 | R(0.01) | -9.33 | |
| 1740 | D5185 | 13.8 | | -0.15 | |
| 1743 | D5185 | 14 | | 0.01 | |
| 1857 | D5185 | 14.4 | | 0.35 | |
| 1875 | | 10 | R(0.01) | -3.32 | |
| 1888 | D5185 | 13.2 | | -0.65 | |
| 1900 | D5185 | 15.318 | | 1.11 | |
| 2493 | In house | 11.8 | | -1.82 | |
| 6002 | D5185 | 14.3 | | 0.26 | |
| 6016 | | ----- | | ----- | |
| 6080 | D5185 | 15 | | 0.85 | |
| 6261 | | ----- | W | ----- | test result withdrawn, reported 9.427 |

| | | |
|---|---|---|
| | normality | OK |
| | n | 40 |
| | outliers | 3 |
| | mean (n) | 13.985 |
| | st.dev. (n) | 0.9287 |
| | R(calc.) | 2.600 |
| | st.dev.(D5185:18) | 1.1987 |
| | R(D5185:18) | 3.356 |

application range 2 – 160 mg/kg

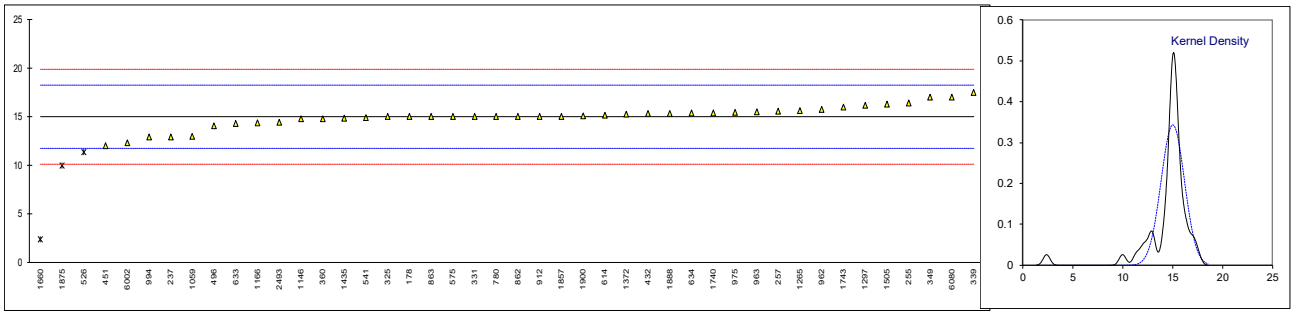


## Determination of Iron as Fe on sample #19237; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D5185 | 15 | | -0.01 | |
| 179 | | ----- | | ----- | |
| 237 | D5185 | 12.94 | | -1.28 | |
| 255 | D6595 | 16.43 | | 0.87 | |
| 257 | D6595 | 15.58 | | 0.35 | |
| 311 | | ----- | | ----- | |
| 325 | D5185 | 15 | | -0.01 | |
| 331 | D5185Mod. | 15 | | -0.01 | |
| 339 | INH-165 | 17.5 | | 1.53 | |
| 349 | D5185 | 17 | | 1.22 | |
| 360 | D5185 | 14.8 | | -0.13 | |
| 398 | | ----- | | ----- | |
| 432 | D5185 | 15.3 | | 0.17 | |
| 451 | D5185 | 12 | | -1.86 | |
| 496 | D5185 | 14.05 | | -0.60 | |
| 526 | D5185 | 11.371 | ex,C | -2.25 | fr. 11, excluded as many statistical outliers in other related metal analyzes |
| 541 | D5185 | 14.9 | | -0.07 | |
| 575 | D6595 | 15 | | -0.01 | |
| 603 | | ----- | | ----- | |
| 614 | D5185 | 15.14 | | 0.08 | |
| 633 | D6595 | 14.3 | | -0.44 | |
| 634 | D6595 | 15.36 | | 0.21 | |
| 780 | D5185 | 15 | | -0.01 | |
| 862 | D5185 | 15 | | -0.01 | |
| 863 | D5185 | 15 | | -0.01 | |
| 912 | D5185 | 15.0 | | -0.01 | |
| 962 | D5185 | 15.73 | | 0.44 | |
| 963 | D5185 | 15.50 | | 0.30 | |
| 975 | D5185 | 15.45 | | 0.27 | |
| 994 | D5185 | 12.9 | | -1.31 | |
| 1059 | In house | 13 | | -1.24 | |
| 1146 | D5185 | 14.76 | | -0.16 | |
| 1166 | In house | 14.38 | | -0.39 | |
| 1265 | D6595 | 15.643 | | 0.39 | |
| 1297 | In house | 16.189 | | 0.72 | |
| 1372 | D5185 | 15.25 | | 0.14 | |
| 1435 | D5185 | 14.85 | | -0.10 | |
| 1505 | D5185 | 16.28 | | 0.78 | |
| 1660 | D5185 | 2.4 | R(0.01) | -7.78 | |
| 1740 | D5185 | 15.4 | | 0.24 | |
| 1743 | D5185 | 16 | | 0.61 | |
| 1857 | D5185 | 15.0 | | -0.01 | |
| 1875 | | 10 | R(0.01) | -3.09 | |
| 1888 | D5185 | 15.3 | | 0.17 | |
| 1900 | D5185 | 15.110 | | 0.06 | |
| 2493 | In house | 14.4 | | -0.38 | |
| 6002 | D5185 | 12.3 | | -1.68 | |
| 6016 | | ----- | | ----- | |
| 6080 | D5185 | 17 | | 1.22 | |
| 6261 | | ----- | W | ----- | test result withdrawn, reported 9.938 |

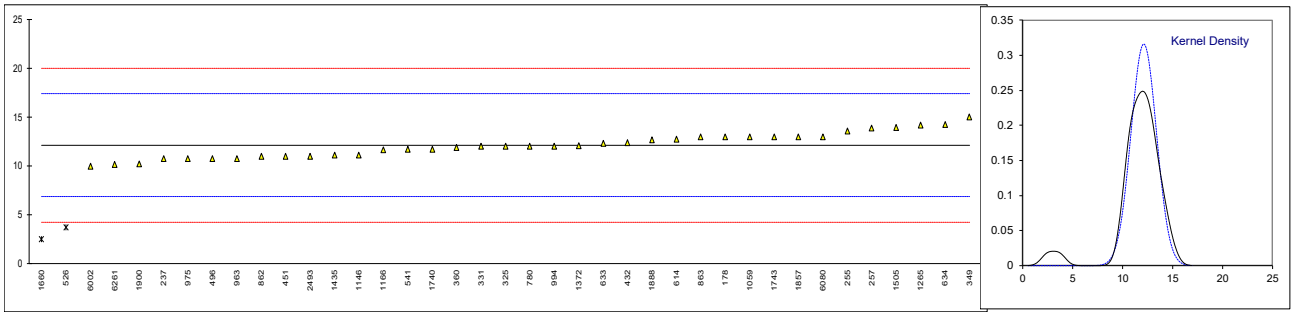
| | | |
|---|---|---|
| | normality | suspect |
| | n | 41 |
| | outliers | 2 +1ex |
| | mean (n) | 15.018 |
| | st.dev. (n) | 1.1602 |
| | R(calc.) | 3.249 |
| | st.dev.(D5185:18) | 1.6223 |
| | R(D5185:18) | 4.543 |

application range 2 – 140 mg/kg



## Determination of Lead as Pb on sample #19237; results in mg/kg

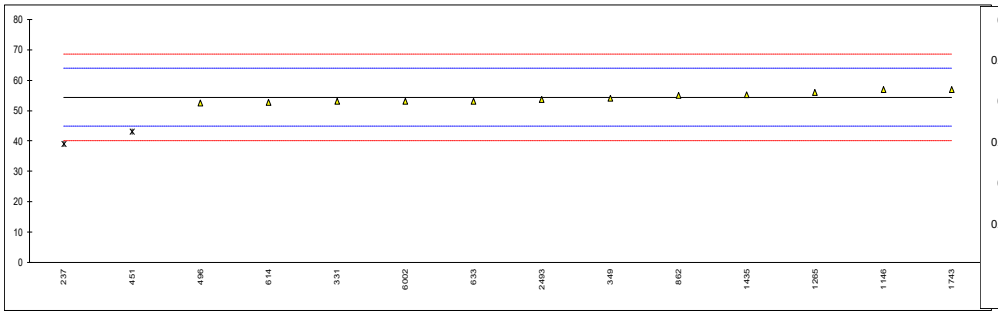
| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D5185 | 13 | | 0.33 | |
| 179 | | ----- | | ----- | |
| 237 | D5185 | 10.73 | | -0.53 | |
| 255 | D6595 | 13.58 | | 0.55 | |
| 257 | D6595 | 13.87 | | 0.66 | |
| 311 | | ----- | | ----- | |
| 325 | D5185 | 12 | | -0.05 | |
| 331 | D5185Mod. | 12 | | -0.05 | |
| 339 | | ----- | | ----- | |
| 349 | D5185 | 15 | | 1.09 | |
| 360 | D5185 | 11.9 | | -0.09 | |
| 398 | | ----- | | ----- | |
| 432 | D5185 | 12.4 | | 0.11 | |
| 451 | D5185 | 11 | | -0.43 | |
| 496 | D5185 | 10.755 | | -0.52 | |
| 526 | D5185 | 3.702 | C,R(0.01) | -3.20 | first reported 4 |
| 541 | D5185 | 11.7 | | -0.16 | |
| 575 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 614 | D5185 | 12.72 | | 0.23 | |
| 633 | D6595 | 12.3 | | 0.07 | |
| 634 | D6595 | 14.26 | | 0.81 | |
| 780 | D5185 | 12 | | -0.05 | |
| 862 | D5185 | 11 | | -0.43 | |
| 863 | D5185 | 13 | | 0.33 | |
| 912 | | ----- | | ----- | |
| 962 | | ----- | | ----- | |
| 963 | D5185 | 10.76 | | -0.52 | |
| 975 | D5185 | 10.74 | | -0.53 | |
| 994 | D5185 | 12.0 | | -0.05 | |
| 1059 | In house | 13 | | 0.33 | |
| 1146 | D5185 | 11.13 | | -0.38 | |
| 1166 | In house | 11.68 | | -0.17 | |
| 1265 | D6595 | 14.183 | | 0.78 | |
| 1297 | | ----- | | ----- | |
| 1372 | D5185 | 12.07 | | -0.02 | |
| 1435 | D5185 | 11.09 | | -0.39 | |
| 1505 | D5185 | 13.96 | | 0.70 | |
| 1660 | D5185 | 2.5 | R(0.01) | -3.66 | |
| 1740 | D5185 | 11.7 | | -0.16 | |
| 1743 | D5185 | 13 | | 0.33 | |
| 1857 | D5185 | 13.0 | | 0.33 | |
| 1875 | | ----- | | ----- | |
| 1888 | D5185 | 12.7 | | 0.22 | |
| 1900 | D5185 | 10.220 | | -0.72 | |
| 2493 | In house | 11.0 | | -0.43 | |
| 6002 | D5185 | 9.97 | | -0.82 | |
| 6016 | | ----- | | ----- | |
| 6080 | D5185 | 13 | | 0.33 | |
| 6261 | D5185 | 10.159 | | -0.75 | |
| | | | | | |
| | normality | OK | | | |
| | n | 37 | | | |
| | outliers | 2 | | | |
| | mean (n) | 12.124 | | | |
| | st.dev. (n) | 1.2611 | | | |
| | R(calc.) | 3.531 | | | |
| | st.dev.(D5185:18) | 2.6307 | | | |
| | R(D5185:18) | 7.366 | | | application range 10 – 160 mg/kg |

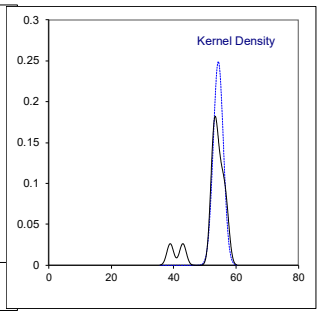


## Determination of Lithium as Li on sample #19237; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | | ----- | | ----- | |
| 179 | | ----- | | ----- | |
| 237 | D5185 | 38.95 | C,G(0.01) | -3.23 | first reported 63.45 |
| 255 | | ----- | | ----- | |
| 257 | | ----- | | ----- | |
| 311 | | ----- | | ----- | |
| 325 | | ----- | | ----- | |
| 331 | D5185Mod. | 53 | | -0.28 | |
| 339 | | ----- | | ----- | |
| 349 | D5185 | 54 | | -0.07 | |
| 360 | | ----- | | ----- | |
| 398 | | ----- | | ----- | |
| 432 | | ----- | | ----- | |
| 451 | D5185 | 43 | G(0.01) | -2.38 | |
| 496 | D5185 | 52.59 | | -0.37 | |
| 526 | | ----- | | ----- | |
| 541 | | ----- | | ----- | |
| 575 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 614 | D5185 | 52.7 | | -0.35 | |
| 633 | D6595 | 53.1 | | -0.26 | |
| 634 | | ----- | | ----- | |
| 780 | | ----- | | ----- | |
| 862 | D5185 | 55 | | 0.14 | |
| 863 | | ----- | | ----- | |
| 912 | | ----- | | ----- | |
| 962 | | ----- | | ----- | |
| 963 | | ----- | | ----- | |
| 975 | | ----- | | ----- | |
| 994 | | ----- | | ----- | |
| 1059 | | ----- | | ----- | |
| 1146 | D5185 | 56.85 | | 0.52 | |
| 1166 | | ----- | | ----- | |
| 1265 | D6595 | 55.928 | | 0.33 | |
| 1297 | | ----- | | ----- | |
| 1372 | | ----- | | ----- | |
| 1435 | D5185 | 55.22 | | 0.18 | |
| 1505 | | ----- | | ----- | |
| 1660 | | ----- | | ----- | |
| 1740 | | ----- | | ----- | |
| 1743 | D5185 | 57 | | 0.56 | |
| 1857 | | ----- | | ----- | |
| 1875 | | ----- | | ----- | |
| 1888 | | ----- | | ----- | |
| 1900 | | ----- | | ----- | |
| 2493 | In house | 53.7 | | -0.14 | |
| 6002 | D6595 | 53.09 | | -0.26 | |
| 6016 | | ----- | | ----- | |
| 6080 | | ----- | | ----- | |
| 6261 | | ----- | W | ----- | test result withdrawn, reported <1 |

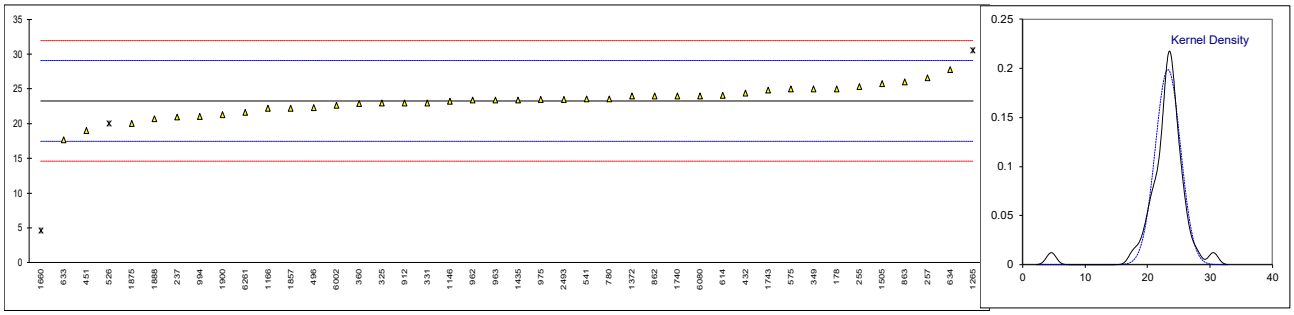
| | |
|---|---|
| normality | OK |
| n | 12 |
| outliers | 2 |
| mean (n) | 54.348 |
| st.dev. (n) | 1.6034 |
| R(calc.) | 4.490 |
| st.dev.(Horwitz) | 4.7658 |
| R(Horwitz) | 13.344 |



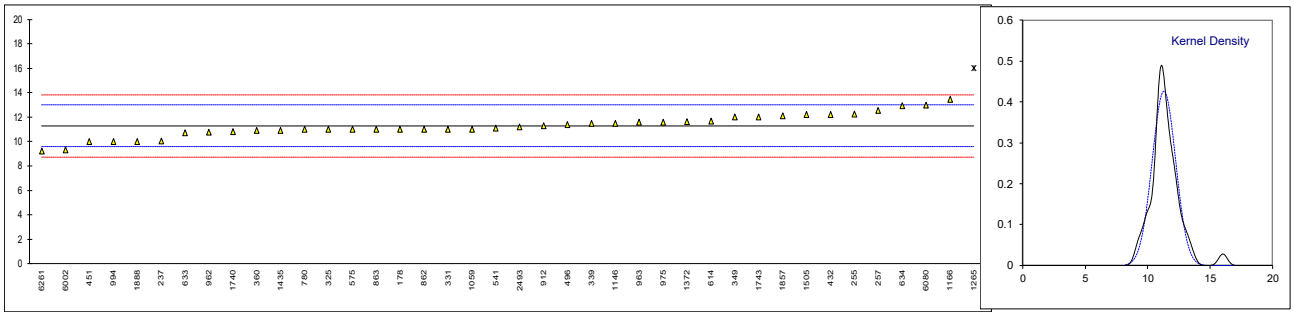
## Determination of Magnesium as Mg on sample #19237; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D5185 | 25 | | 0.60 | |
| 179 | | ----- | | ----- | |
| 237 | D5185 | 20.98 | | -0.78 | |
| 255 | D6595 | 25.37 | | 0.73 | |
| 257 | D6595 | 26.57 | | 1.14 | |
| 311 | | ----- | | ----- | |
| 325 | D5185 | 23 | | -0.09 | |
| 331 | D5185Mod. | 23 | | -0.09 | |
| 339 | INH-165 | <50 | | ----- | |
| 349 | D5185 | 25 | | 0.60 | |
| 360 | D5185 | 22.92 | | -0.11 | |
| 398 | | ----- | | ----- | |
| 432 | D5185 | 24.4 | | 0.40 | |
| 451 | D5185 | 19 | | -1.47 | |
| 496 | D5185 | 22.28 | | -0.34 | |
| 526 | D5185 | 20 | ex | -1.12 | excluded as many statistical outliers in other related metal analyzes |
| 541 | D5185 | 23.58 | | 0.11 | |
| 575 | D6595 | 25 | | 0.60 | |
| 603 | | ----- | | ----- | |
| 614 | D5185 | 24.1 | | 0.29 | |
| 633 | D6595 | 17.7 | | -1.91 | |
| 634 | D6595 | 27.80 | | 1.57 | |
| 780 | D5185 | 23.6 | | 0.12 | |
| 862 | D5185 | 24 | | 0.26 | |
| 863 | D5185 | 26 | | 0.95 | |
| 912 | D5185 | 23.0 | | -0.09 | |
| 962 | D5185 | 23.39 | | 0.05 | |
| 963 | D5185 | 23.43 | | 0.06 | |
| 975 | D5185 | 23.46 | | 0.07 | |
| 994 | D5185 | 21.0 | | -0.78 | |
| 1059 | | ----- | | ----- | |
| 1146 | D5185 | 23.21 | | -0.01 | |
| 1166 | In house | 22.21 | | -0.36 | |
| 1265 | D6595 | 30.507 | R(0.05) | 2.50 | |
| 1297 | | ----- | | ----- | |
| 1372 | D5185 | 23.98 | | 0.25 | |
| 1435 | D5185 | 23.43 | | 0.06 | |
| 1505 | D5185 | 25.75 | | 0.86 | |
| 1660 | D5185 | 4.6 | R(0.01) | -6.43 | |
| 1740 | D5185 | 24 | | 0.26 | |
| 1743 | D5185 | 24.8 | | 0.53 | |
| 1857 | D5185 | 22.24 | | -0.35 | |
| 1875 | | 20 | | -1.12 | |
| 1888 | D5185 | 20.7 | | -0.88 | |
| 1900 | D5185 | 21.272 | | -0.68 | |
| 2493 | In house | 23.5 | | 0.09 | |
| 6002 | D5185 | 22.6 | | -0.23 | |
| 6016 | | ----- | | ----- | |
| 6080 | D5185 | 24 | | 0.26 | |
| 6261 | D5185 | 21.583 | | -0.58 | |
| | | | | | |
| | normality | OK | | | |
| | n | 39 | | | |
| | outliers | 2 +1ex | | | |
| | mean (n) | 23.253 | | | |
| | st.dev. (n) | 2.0060 | | | |
| | R(calc.) | 5.617 | | | |
| | st.dev.(D5185:18) | 2.8997 | | | |
| | R(D5185:18) | 8.119 | | | application range 5 – 1700 mg/kg |



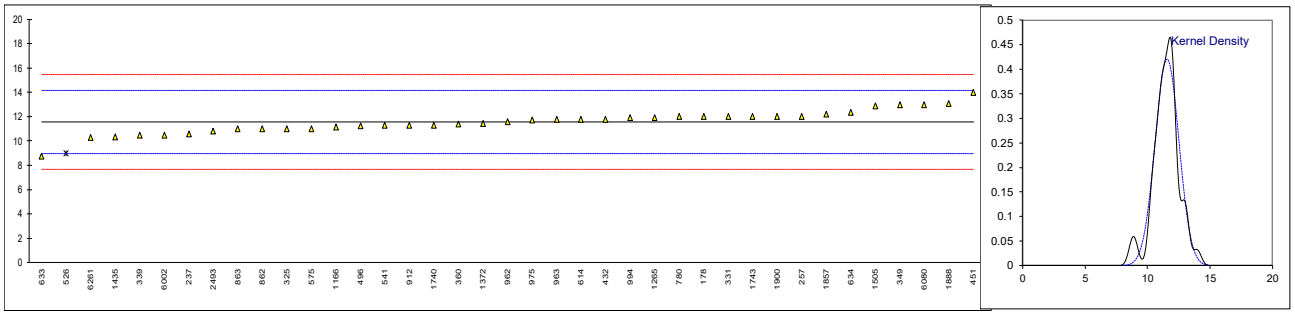
## Determination of Manganese as Mn on sample #19237; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D5185 | 11 | | -0.33 | |
| 179 | | ----- | | ----- | |
| 237 | D5185 | 10.06 | | -1.44 | |
| 255 | D6595 | 12.28 | | 1.17 | |
| 257 | D6595 | 12.56 | | 1.50 | |
| 311 | | ----- | | ----- | |
| 325 | D5185 | 11 | | -0.33 | |
| 331 | D5185Mod. | 11 | | -0.33 | |
| 339 | INH-165 | 11.5 | | 0.26 | |
| 349 | D5185 | 12 | | 0.84 | |
| 360 | D5185 | 10.9 | | -0.45 | |
| 398 | | ----- | | ----- | |
| 432 | D5185 | 12.2 | | 1.08 | |
| 451 | D5185 | 10 | | -1.51 | |
| 496 | D5185 | 11.39 | | 0.13 | |
| 526 | | ----- | | ----- | |
| 541 | D5185 | 11.1 | | -0.21 | |
| 575 | D6595 | 11 | C | -0.33 | first reported 14 |
| 603 | | ----- | | ----- | |
| 614 | D5185 | 11.7 | | 0.49 | |
| 633 | D6595 | 10.7 | | -0.68 | |
| 634 | D6595 | 12.95 | | 1.96 | |
| 780 | D5185 | 11 | | -0.33 | |
| 862 | D5185 | 11 | | -0.33 | |
| 863 | D5185 | 11 | | -0.33 | |
| 912 | D5185 | 11.3 | | 0.02 | |
| 962 | D5185 | 10.77 | | -0.60 | |
| 963 | D5185 | 11.59 | | 0.36 | |
| 975 | D5185 | 11.60 | | 0.37 | |
| 994 | D5185 | 10.0 | | -1.51 | |
| 1059 | In house | 11 | | -0.33 | |
| 1146 | D5185 | 11.51 | | 0.27 | |
| 1166 | In house | 13.47 | | 2.57 | |
| 1265 | D6595 | 16.010 | R(0.01) | 5.56 | |
| 1297 | | ----- | | ----- | |
| 1372 | D5185 | 11.62 | | 0.40 | |
| 1435 | D5185 | 10.92 | | -0.43 | |
| 1505 | D5185 | 12.19 | | 1.07 | |
| 1660 | | ----- | | ----- | |
| 1740 | D5185 | 10.8 | | -0.57 | |
| 1743 | D5185 | 12 | | 0.84 | |
| 1857 | D5185 | 12.1 | | 0.96 | |
| 1875 | | ----- | | ----- | |
| 1888 | D5185 | 10 | | -1.51 | |
| 1900 | | ----- | | ----- | |
| 2493 | In house | 11.2 | | -0.10 | |
| 6002 | D5185 | 9.35 | | -2.27 | |
| 6016 | | ----- | | ----- | |
| 6080 | D5185 | 13 | | 2.02 | |
| 6261 | D5185 | 9.253 | | -2.39 | |
| | | | | | |
| | normality | OK | | | |
| | n | 39 | | | |
| | outliers | 1 | | | |
| | mean (n) | 11.282 | | | |
| | st.dev. (n) | 0.9343 | | | |
| | R(calc.) | 2.616 | | | |
| | st.dev.(D5185:18) | 0.8505 | | | |
| | R(D5185:18) | 2.381 | | | application range 5 – 700 mg/kg |



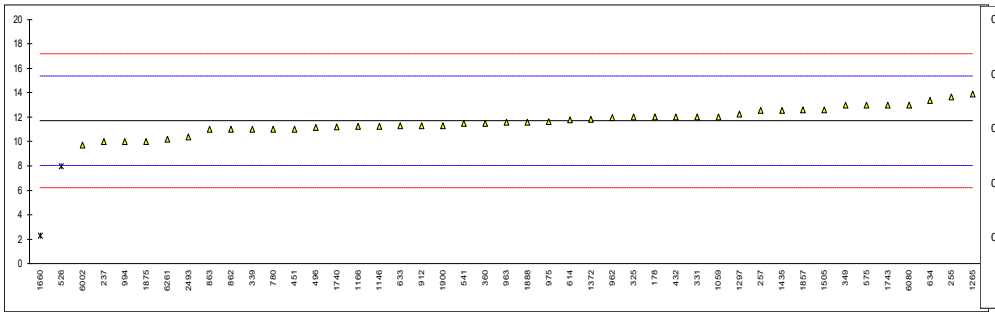
## Determination of Molybdenum as Mo on sample #19237; results in mg/kg

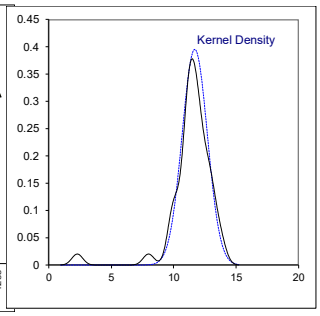
| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D5185 | 12 | | 0.32 | |
| 179 | | ----- | | ----- | |
| 237 | D5185 | 10.57 | | -0.78 | |
| 255 | | ----- | | ----- | |
| 257 | D6595 | 12.04 | | 0.35 | |
| 311 | | ----- | | ----- | |
| 325 | D5185 | 11 | | -0.45 | |
| 331 | D5185Mod. | 12 | | 0.32 | |
| 339 | INH-165 | 10.5 | | -0.83 | |
| 349 | D5185 | 13 | | 1.09 | |
| 360 | D5185 | 11.4 | | -0.14 | |
| 398 | | ----- | | ----- | |
| 432 | D5185 | 11.8 | | 0.17 | |
| 451 | D5185 | 14 | | 1.86 | |
| 496 | D5185 | 11.24 | | -0.26 | |
| 526 | D5185 | 9 | ex | -1.98 | excluded as many statistical outliers in other related metal analyzes |
| 541 | D5185 | 11.3 | | -0.22 | |
| 575 | D6595 | 11 | | -0.45 | |
| 603 | | ----- | | ----- | |
| 614 | D5185 | 11.8 | | 0.17 | |
| 633 | D6595 | 8.77 | | -2.16 | |
| 634 | D6595 | 12.35 | | 0.59 | |
| 780 | D5185 | 12 | | 0.32 | |
| 862 | D5185 | 11 | | -0.45 | |
| 863 | D5185 | 11 | | -0.45 | |
| 912 | D5185 | 11.3 | | -0.22 | |
| 962 | D5185 | 11.60 | | 0.01 | |
| 963 | D5185 | 11.77 | | 0.15 | |
| 975 | D5185 | 11.73 | | 0.11 | |
| 994 | D5185 | 11.9 | | 0.25 | |
| 1059 | | ----- | | ----- | |
| 1146 | | ----- | | ----- | |
| 1166 | In house | 11.16 | | -0.32 | |
| 1265 | D6595 | 11.936 | | 0.27 | |
| 1297 | | ----- | | ----- | |
| 1372 | D5185 | 11.42 | | -0.12 | |
| 1435 | D5185 | 10.35 | | -0.95 | |
| 1505 | D5185 | 12.91 | | 1.02 | |
| 1660 | | ----- | | ----- | |
| 1740 | D5185 | 11.3 | | -0.22 | |
| 1743 | D5185 | 12 | | 0.32 | |
| 1857 | D5185 | 12.2 | | 0.48 | |
| 1875 | | ----- | | ----- | |
| 1888 | D5185 | 13.1 | | 1.17 | |
| 1900 | D5185 | 12.013 | | 0.33 | |
| 2493 | In house | 10.8 | | -0.60 | |
| 6002 | D5185 | 10.5 | | -0.83 | |
| 6016 | | ----- | | ----- | |
| 6080 | D5185 | 13 | | 1.09 | |
| 6261 | D5185 | 10.305 | | -0.98 | |
| | | | | | |
| | normality | suspect | | | |
| | n | 38 | | | |
| | outliers | 0 +1ex | | | |
| | mean (n) | 11.581 | | | |
| | st.dev. (n) | 0.9496 | | | |
| | R(calc.) | 2.659 | | | |
| | st.dev.(D5185:18) | 1.3010 | | | |
| | R(D5185:18) | 3.643 | | | application range 5 – 200 mg/kg |



## Determination of Nickel as Ni on sample #19237; results in mg/kg

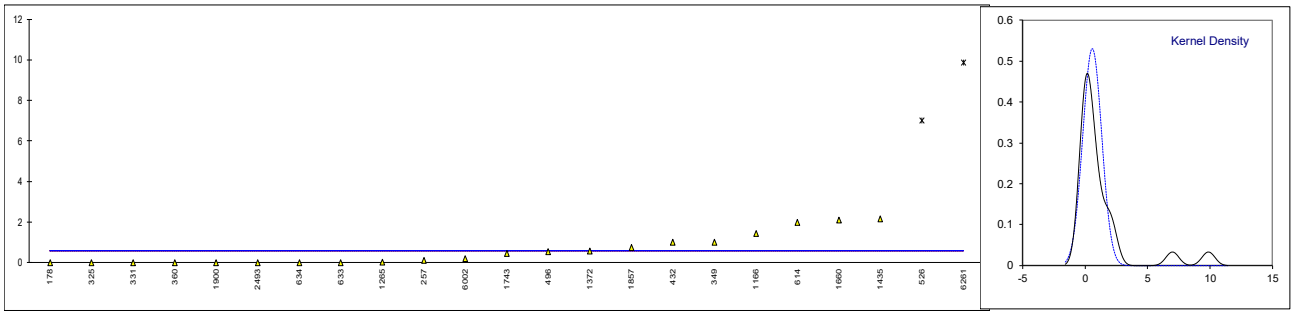
| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D5185 | 12 | | 0.16 | |
| 179 | | ----- | | ----- | |
| 237 | D5185 | 9.99 | | -0.93 | |
| 255 | D6595 | 13.66 | | 1.07 | |
| 257 | D6595 | 12.56 | | 0.47 | |
| 311 | | ----- | | ----- | |
| 325 | D5185 | 12 | | 0.16 | |
| 331 | D5185Mod. | 12 | | 0.16 | |
| 339 | INH-165 | 11 | | -0.38 | |
| 349 | D5185 | 13 | | 0.71 | |
| 360 | D5185 | 11.5 | | -0.11 | |
| 398 | | ----- | | ----- | |
| 432 | D5185 | 12.0 | | 0.16 | |
| 451 | D5185 | 11 | | -0.38 | |
| 496 | D5185 | 11.175 | | -0.29 | |
| 526 | D5185 | 8 | R(0.05) | -2.02 | |
| 541 | D5185 | 11.5 | | -0.11 | |
| 575 | D6595 | 13 | | 0.71 | |
| 603 | | ----- | | ----- | |
| 614 | D5185 | 11.8 | | 0.06 | |
| 633 | D6595 | 11.3 | | -0.22 | |
| 634 | D6595 | 13.36 | | 0.91 | |
| 780 | D5185 | 11 | | -0.38 | |
| 862 | D5185 | 11 | | -0.38 | |
| 863 | D5185 | 11 | | -0.38 | |
| 912 | D5185 | 11.3 | | -0.22 | |
| 962 | D5185 | 11.96 | | 0.14 | |
| 963 | D5185 | 11.59 | | -0.06 | |
| 975 | D5185 | 11.63 | | -0.04 | |
| 994 | D5185 | 10.0 | | -0.93 | |
| 1059 | In house | 12 | | 0.16 | |
| 1146 | D5185 | 11.27 | | -0.23 | |
| 1166 | In house | 11.23 | | -0.26 | |
| 1265 | D6595 | 13.897 | | 1.20 | |
| 1297 | In house | 12.243 | | 0.30 | |
| 1372 | D5185 | 11.83 | | 0.07 | |
| 1435 | D5185 | 12.57 | | 0.48 | |
| 1505 | D5185 | 12.62 | | 0.50 | |
| 1660 | D5185 | 2.3 | R(0.01) | -5.13 | |
| 1740 | D5185 | 11.2 | | -0.27 | |
| 1743 | D5185 | 13 | | 0.71 | |
| 1857 | D5185 | 12.6 | | 0.49 | |
| 1875 | | 10 | | -0.93 | |
| 1888 | D5185 | 11.6 | | -0.05 | |
| 1900 | D5185 | 11.310 | | -0.21 | |
| 2493 | In house | 10.4 | | -0.71 | |
| 6002 | D5185 | 9.73 | | -1.07 | |
| 6016 | | ----- | | ----- | |
| 6080 | D5185 | 13 | | 0.71 | |
| 6261 | D5185 | 10.182 | | -0.83 | |
| | | | | | |
| | normality | OK | | | |
| | n | 43 | | | |
| | outliers | 2 | | | |
| | mean (n) | 11.698 | | | |
| | st.dev. (n) | 1.0085 | | | |
| | R(calc.) | 2.824 | | | |
| | st.dev.(D5185:18) | 1.8323 | | | |
| | R(D5185:18) | 5.130 | | | application range 5 – 40 mg/kg |





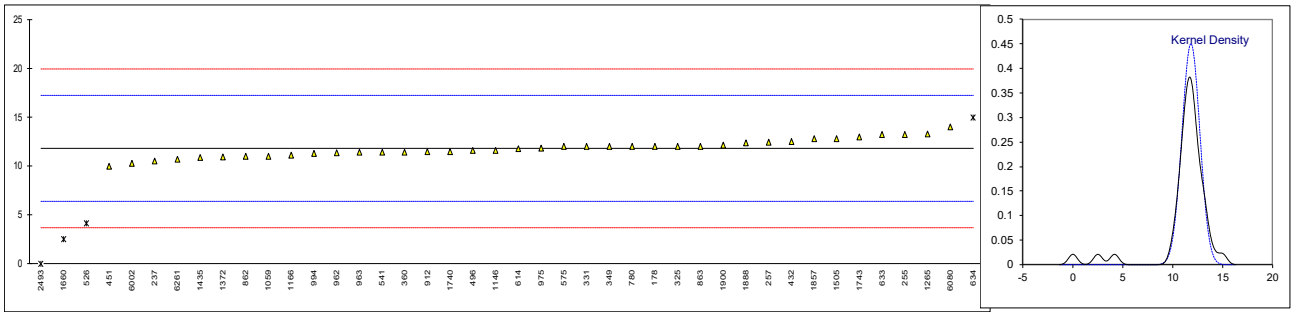
## Determination of Potassium as K on sample #19237; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D5185 | 0 | | ----- | |
| 179 | | ----- | | ----- | |
| 237 | D5185 | <1 | | ----- | |
| 255 | | ----- | | ----- | |
| 257 | D6595 | 0.12 | | ----- | |
| 311 | | ----- | | ----- | |
| 325 | D5185 | 0 | | ----- | |
| 331 | D5185Mod. | 0 | | ----- | |
| 339 | INH-165 | <50 | | ----- | |
| 349 | D5185 | 1 | | ----- | |
| 360 | D5185 | 0 | | ----- | |
| 398 | | ----- | | ----- | |
| 432 | D5185 | 1 | | ----- | |
| 451 | D5185 | < 1 | | ----- | |
| 496 | D5185 | 0.53 | | ----- | |
| 526 | D5185 | 7 | R(0.01) | ----- | |
| 541 | D5185 | <40 | | ----- | |
| 575 | | ----- | | ----- | |
| 603 | | ----- | | ----- | |
| 614 | D5185 | 2.0 | | ----- | |
| 633 | D6595 | 0.01 | | ----- | |
| 634 | D6595 | 0.008 | | ----- | |
| 780 | D5185 | <1 | | ----- | |
| 862 | D5185 | <1 | | ----- | |
| 863 | D5185 | <1 | | ----- | |
| 912 | | ----- | | ----- | |
| 962 | | ----- | | ----- | |
| 963 | | ----- | | ----- | |
| 975 | | ----- | | ----- | |
| 994 | | ----- | | ----- | |
| 1059 | | ----- | | ----- | |
| 1146 | | ----- | | ----- | |
| 1166 | In house | 1.45 | | ----- | |
| 1265 | D6595 | 0.025 | | ----- | |
| 1297 | | ----- | | ----- | |
| 1372 | D5185 | 0.57 | | ----- | |
| 1435 | D5185 | 2.155 | | ----- | |
| 1505 | | ----- | | ----- | |
| 1660 | D5185 | 2.1 | | ----- | |
| 1740 | D5185 | <0.1 | | ----- | |
| 1743 | D5185 | 0.47 | | ----- | |
| 1857 | D5185 | 0.75 | | ----- | |
| 1875 | | ----- | | ----- | |
| 1888 | | ----- | | ----- | |
| 1900 | D5185 | 0 | | ----- | |
| 2493 | In house | 0.00 | | ----- | |
| 6002 | D5185 | 0.207 | | ----- | |
| 6016 | | ----- | | ----- | |
| 6080 | D5185 | <1 | | ----- | |
| 6261 | D5185 | 9.880 | R(0.01) | ----- | |
| | | | | | |
| | normality | suspect | | | |
| | n | 21 | | | |
| | outliers | 2 | | | |
| | mean (n) | 0.590 | | | |
| | st.dev. (n) | 0.7513 | | | |
| | R(calc.) | 2.104 | | | |
| | st.dev.(D5185:18) | (2.0229) | | | |
| | R(D5185:18) | (5.664) | | | application range 40 – 1200 mg/kg |



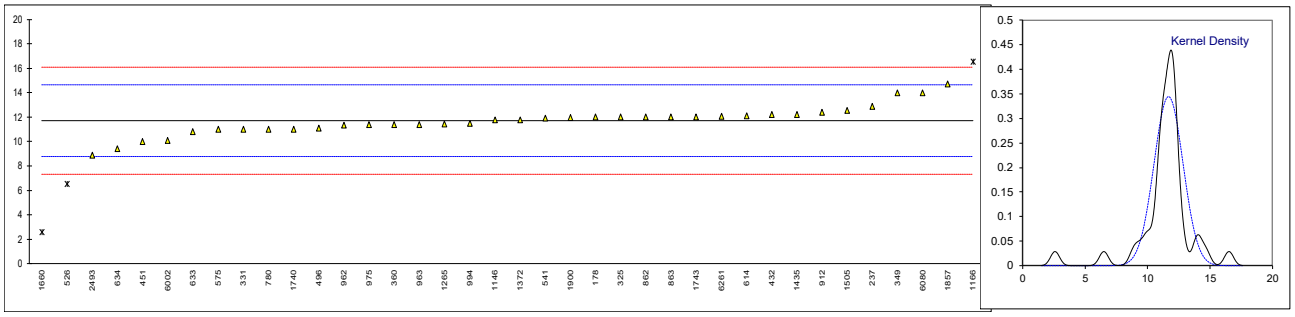
## Determination of Silicon as Si on sample #19237; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D5185 | 12 | | 0.07 | |
| 179 | | ----- | | ----- | |
| 237 | D5185 | 10.53 | | -0.47 | |
| 255 | D6595 | 13.21 | | 0.52 | |
| 257 | D6595 | 12.43 | | 0.23 | |
| 311 | | ----- | | ----- | |
| 325 | D5185 | 12 | | 0.07 | |
| 331 | D5185Mod. | 12 | | 0.07 | |
| 339 | | ----- | | ----- | |
| 349 | D5185 | 12 | | 0.07 | |
| 360 | D5185 | 11.4 | | -0.15 | |
| 398 | | ----- | | ----- | |
| 432 | D5185 | 12.5 | | 0.25 | |
| 451 | D5185 | 10 | | -0.67 | |
| 496 | D5185 | 11.57 | | -0.09 | |
| 526 | D5185 | 4.163 | C,R(0.01) | -2.82 | first reported 4 |
| 541 | D5185 | 11.4 | | -0.15 | |
| 575 | D6595 | 12 | | 0.07 | |
| 603 | | ----- | | ----- | |
| 614 | D5185 | 11.8 | | 0.00 | |
| 633 | D6595 | 13.2 | | 0.51 | |
| 634 | D6595 | 14.98 | C,R(0.05) | 1.17 | first reported 18.14 |
| 780 | D5185 | 12 | | 0.07 | |
| 862 | D5185 | 11 | | -0.30 | |
| 863 | D5185 | 12 | | 0.07 | |
| 912 | D5185 | 11.5 | | -0.11 | |
| 962 | D5185 | 11.36 | | -0.17 | |
| 963 | D5185 | 11.39 | | -0.15 | |
| 975 | D5185 | 11.81 | | 0.00 | |
| 994 | D5185 | 11.3 | | -0.19 | |
| 1059 | In house | 11 | | -0.30 | |
| 1146 | D5185 | 11.61 | | -0.07 | |
| 1166 | In house | 11.09 | | -0.27 | |
| 1265 | D6595 | 13.269 | | 0.54 | |
| 1297 | | ----- | | ----- | |
| 1372 | D5185 | 10.93 | | -0.32 | |
| 1435 | D5185 | 10.85 | | -0.35 | |
| 1505 | D5185 | 12.81 | | 0.37 | |
| 1660 | D5185 | 2.5 | R(0.01) | -3.43 | |
| 1740 | D5185 | 11.5 | | -0.11 | |
| 1743 | D5185 | 13 | | 0.44 | |
| 1857 | D5185 | 12.8 | | 0.36 | |
| 1875 | | ----- | | ----- | |
| 1888 | D5185 | 12.4 | | 0.22 | |
| 1900 | D5185 | 12.158 | | 0.13 | |
| 2493 | In house | 0.00 | R(0.01) | -4.35 | |
| 6002 | D5185 | 10.3 | | -0.56 | |
| 6016 | | ----- | | ----- | |
| 6080 | D5185 | 14 | | 0.81 | |
| 6261 | D5185 | 10.673 | | -0.42 | |
| | | | | | |
| | normality | OK | | | |
| | n | 38 | | | |
| | outliers | 4 | | | |
| | mean (n) | 11.810 | | | |
| | st.dev. (n) | 0.8865 | | | |
| | R(calc.) | 2.482 | | | |
| | st.dev.(D5185:18) | 2.7128 | | | |
| | R(D5185:18) | 7.596 | | | application range 8 – 50 mg/kg |



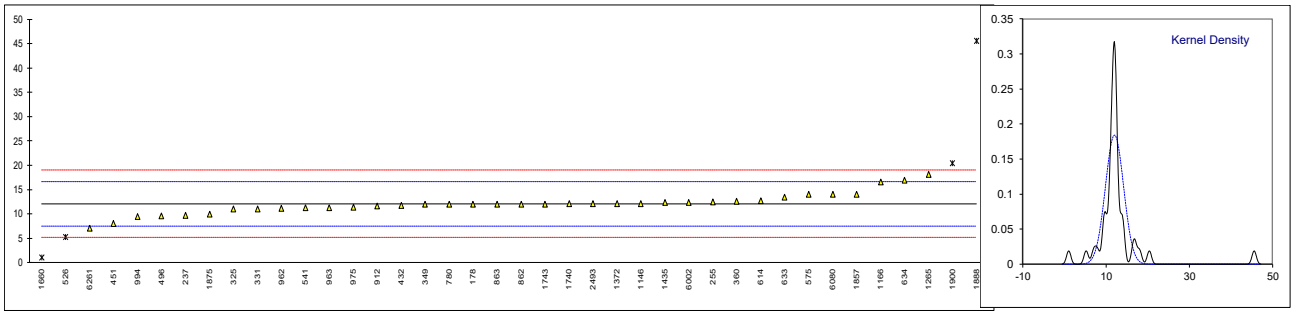
## Determination of Silver as Ag on sample #19237; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D5185 | 12 | | 0.21 | |
| 179 | | ----- | | ----- | |
| 237 | D5185 | 12.87 | | 0.80 | |
| 255 | | ----- | | ----- | |
| 257 | | ----- | | ----- | |
| 311 | | ----- | | ----- | |
| 325 | D5185 | 12 | | 0.21 | |
| 331 | D5185Mod. | 11 | | -0.48 | |
| 339 | | ----- | | ----- | |
| 349 | D5185 | 14 | | 1.58 | |
| 360 | D5185 | 11.4 | | -0.20 | |
| 398 | | ----- | | ----- | |
| 432 | D5185 | 12.2 | | 0.34 | |
| 451 | D5185 | 10 | | -1.16 | |
| 496 | D5185 | 11.11 | | -0.40 | |
| 526 | D5185 | 6.512 | C,R(0.01) | -3.55 | first reported 7 |
| 541 | D5185 | 11.9 | | 0.14 | |
| 575 | D6595 | 11 | | -0.48 | |
| 603 | | ----- | | ----- | |
| 614 | D5185 | 12.1 | | 0.28 | |
| 633 | D6595 | 10.8 | | -0.61 | |
| 634 | D6595 | 9.42 | | -1.56 | |
| 780 | D5185 | 11 | | -0.48 | |
| 862 | D5185 | 12 | | 0.21 | |
| 863 | D5185 | 12 | | 0.21 | |
| 912 | D5185 | 12.4 | | 0.48 | |
| 962 | D5185 | 11.36 | | -0.23 | |
| 963 | D5185 | 11.40 | | -0.20 | |
| 975 | D5185 | 11.39 | | -0.21 | |
| 994 | D5185 | 11.5 | | -0.13 | |
| 1059 | | ----- | | ----- | |
| 1146 | D5185 | 11.79 | | 0.06 | |
| 1166 | In house | 16.54 | R(0.01) | 3.31 | |
| 1265 | D6595 | 11.450 | | -0.17 | |
| 1297 | | ----- | | ----- | |
| 1372 | D5185 | 11.80 | | 0.07 | |
| 1435 | D5185 | 12.22 | | 0.36 | |
| 1505 | D5185 | 12.57 | | 0.60 | |
| 1660 | D5185 | 2.6 | R(0.01) | -6.22 | |
| 1740 | D5185 | 11 | | -0.48 | |
| 1743 | D5185 | 12 | | 0.21 | |
| 1857 | D5185 | 14.7 | | 2.05 | |
| 1875 | | ----- | | ----- | |
| 1888 | | ----- | | ----- | |
| 1900 | D5185 | 11.950 | | 0.17 | |
| 2493 | In house | 8.90 | | -1.91 | |
| 6002 | D5185 | 10.1 | | -1.09 | |
| 6016 | | ----- | | ----- | |
| 6080 | D5185 | 14 | | 1.58 | |
| 6261 | D5185 | 12.069 | | 0.25 | |
| | | | | | |
| | normality | suspect | | | |
| | n | 35 | | | |
| | outliers | 3 | | | |
| | mean (n) | 11.697 | | | |
| | st.dev. (n) | 1.1591 | | | |
| | R(calc.) | 3.246 | | | |
| | st.dev.(D5185:18) | 1.4621 | | | |
| | R(D5185:18) | 4.094 | | | application range 0.5 – 50 mg/kg |



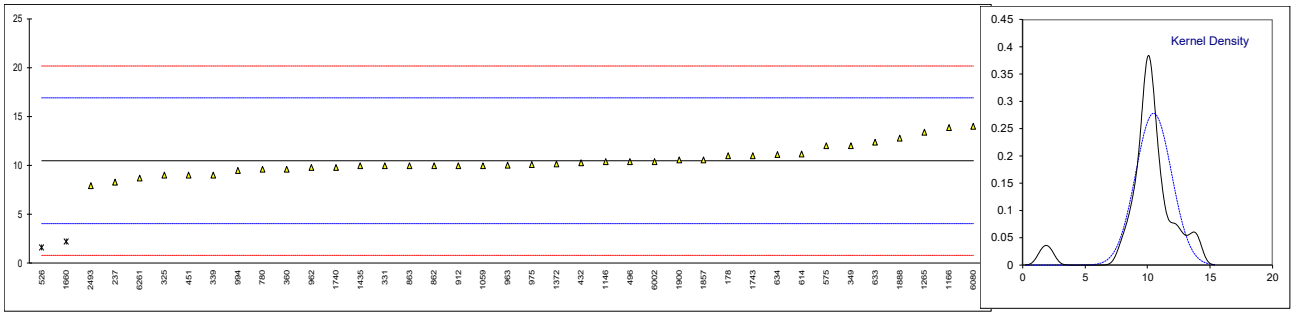
## Determination of Sodium as Na on sample #19237; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D5185 | 12 | | -0.04 | |
| 179 | | ----- | | ----- | |
| 237 | D5185 | 9.76 | | -1.01 | |
| 255 | D6595 | 12.45 | | 0.16 | |
| 257 | | ----- | | ----- | |
| 311 | | ----- | | ----- | |
| 325 | D5185 | 11 | | -0.47 | |
| 331 | D5185Mod. | 11 | | -0.47 | |
| 339 | INH-165 | <50 | | ----- | |
| 349 | D5185 | 12 | | -0.04 | |
| 360 | D5185 | 12.6 | | 0.23 | |
| 398 | | ----- | | ----- | |
| 432 | D5185 | 11.8 | | -0.12 | |
| 451 | D5185 | 8 | | -1.77 | |
| 496 | D5185 | 9.64 | | -1.06 | |
| 526 | D5185 | 5.276 | ex,C | -2.95 | fr. 5, excluded as many statistical outliers in other related metal analyzes |
| 541 | D5185 | 11.3 | | -0.34 | |
| 575 | D6595 | 14 | C | 0.83 | first reported 19 |
| 603 | | ----- | | ----- | |
| 614 | D5185 | 12.7 | | 0.27 | |
| 633 | D6595 | 13.4 | | 0.57 | |
| 634 | D6595 | 16.99 | | 2.13 | |
| 780 | D5185 | 12 | | -0.04 | |
| 862 | D5185 | 12 | | -0.04 | |
| 863 | D5185 | 12 | | -0.04 | |
| 912 | D5185 | 11.7 | | -0.17 | |
| 962 | D5185 | 11.17 | | -0.40 | |
| 963 | D5185 | 11.31 | | -0.33 | |
| 975 | D5185 | 11.36 | | -0.31 | |
| 994 | D5185 | 9.5 | | -1.12 | |
| 1059 | | ----- | | ----- | |
| 1146 | D5185 | 12.19 | | 0.05 | |
| 1166 | In house | 16.60 | | 1.96 | |
| 1265 | D6595 | 18.105 | | 2.61 | |
| 1297 | | ----- | | ----- | |
| 1372 | D5185 | 12.16 | | 0.03 | |
| 1435 | D5185 | 12.39 | | 0.13 | |
| 1505 | | ----- | | ----- | |
| 1660 | D5185 | 1.1 | R(0.01) | -4.77 | |
| 1740 | D5185 | 12.1 | | 0.01 | |
| 1743 | D5185 | 12 | | -0.04 | |
| 1857 | D5185 | 14.1 | | 0.88 | |
| 1875 | | 10 | | -0.90 | |
| 1888 | D5185 | 45.56 | C,R(0.01) | 14.53 | first reported 33.2 |
| 1900 | D5185 | 20.428 | R(0.05) | 3.62 | |
| 2493 | In house | 12.1 | | 0.01 | |
| 6002 | D5185 | 12.4 | | 0.14 | |
| 6016 | | ----- | | ----- | |
| 6080 | D5185 | 14 | | 0.83 | |
| 6261 | D5185 | 7.088 | | -2.17 | |
| | | | | | |
| | normality | suspect | | | |
| | n | 36 | | | |
| | outliers | 3 +1ex | | | |
| | mean (n) | 12.081 | | | |
| | st.dev. (n) | 2.1651 | | | |
| | R(calc.) | 6.062 | | | |
| | st.dev.(D5185:18) | 2.3042 | | | |
| | R(D5185:18) | 6.452 | | | application range 7 – 70 mg/kg |



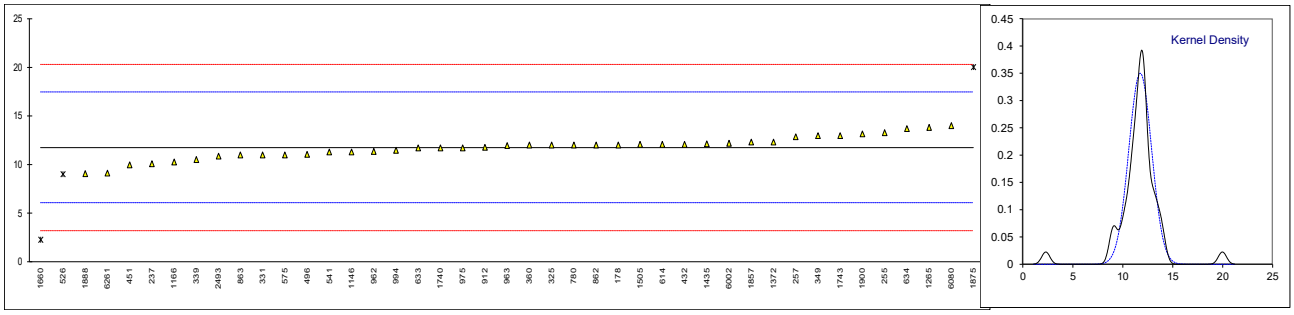
## Determination of Tin as Sn on sample #19237; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D5185 | 11 | | 0.16 | |
| 179 | | ----- | | ----- | |
| 237 | D5185 | 8.27 | | -0.69 | |
| 255 | | ----- | | ----- | |
| 257 | | ----- | | ----- | |
| 311 | | ----- | | ----- | |
| 325 | D5185 | 9 | | -0.46 | |
| 331 | D5185Mod. | 10 | | -0.15 | |
| 339 | INH-165 | 9 | | -0.46 | |
| 349 | D5185 | 12 | | 0.47 | |
| 360 | D5185 | 9.6 | | -0.27 | |
| 398 | | ----- | | ----- | |
| 432 | D5185 | 10.3 | | -0.06 | |
| 451 | D5185 | 9 | | -0.46 | |
| 496 | D5185 | 10.40 | | -0.03 | |
| 526 | D5185 | 1.591 | C,R(0.01) | -2.76 | first reported 1 |
| 541 | D5185 | <10 | | ----- | |
| 575 | D6595 | 12 | | 0.47 | |
| 603 | | ----- | | ----- | |
| 614 | D5185 | 11.16 | | 0.21 | |
| 633 | D6595 | 12.4 | | 0.60 | |
| 634 | D6595 | 11.13 | | 0.20 | |
| 780 | D5185 | 9.6 | | -0.27 | |
| 862 | D5185 | 10 | | -0.15 | |
| 863 | D5185 | 10 | | -0.15 | |
| 912 | D5185 | 10 | | -0.15 | |
| 962 | D5185 | 9.80 | | -0.21 | |
| 963 | D5185 | 10.06 | | -0.13 | |
| 975 | D5185 | 10.11 | | -0.12 | |
| 994 | D5185 | 9.5 | | -0.31 | |
| 1059 | In house | 10 | | -0.15 | |
| 1146 | D5185 | 10.39 | | -0.03 | |
| 1166 | In house | 13.86 | | 1.05 | |
| 1265 | D6595 | 13.384 | | 0.90 | |
| 1297 | | ----- | | ----- | |
| 1372 | D5185 | 10.14 | | -0.11 | |
| 1435 | D5185 | 9.976 | | -0.16 | |
| 1505 | | ----- | | ----- | |
| 1660 | D5185 | 2.2 | R(0.01) | -2.57 | |
| 1740 | D5185 | 9.8 | | -0.21 | |
| 1743 | D5185 | 11 | | 0.16 | |
| 1857 | D5185 | 10.6 | | 0.04 | |
| 1875 | | ----- | | ----- | |
| 1888 | D5185 | 12.8 | | 0.72 | |
| 1900 | D5185 | 10.552 | | 0.02 | |
| 2493 | In house | 7.92 | | -0.80 | |
| 6002 | D5185 | 10.4 | | -0.03 | |
| 6016 | | ----- | | ----- | |
| 6080 | D5185 | 14 | | 1.09 | |
| 6261 | D5185 | 8.727 | | -0.55 | |
| | | | | | |
| | normality | OK | | | |
| | n | 37 | | | |
| | outliers | 2 | | | |
| | mean (n) | 10.483 | | | |
| | st.dev. (n) | 1.4317 | | | |
| | R(calc.) | 4.009 | | | |
| | st.dev.(D5185:18) | 3.2193 | | | |
| | R(D5185:18) | 9.014 | | | application range 10 – 40 mg/kg |



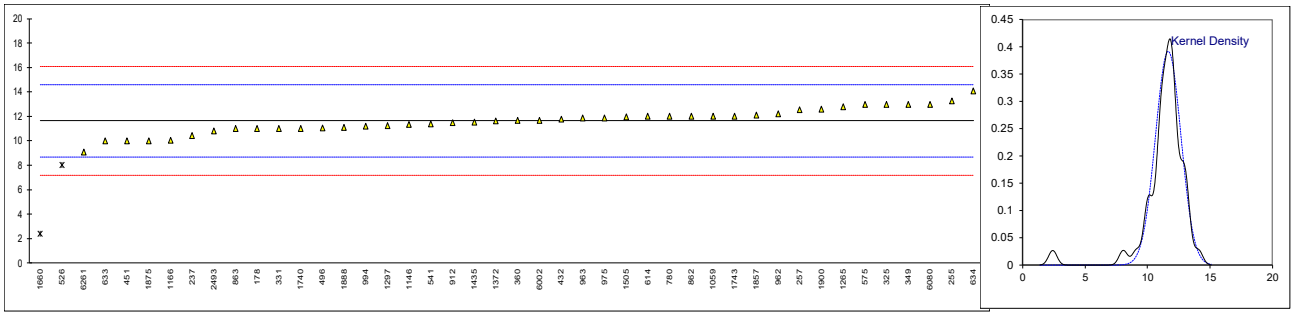
## Determination of Titanium as Ti on sample #19237; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D5185 | 12 | | 0.08 | |
| 179 | | ----- | | ----- | |
| 237 | D5185 | 10.11 | | -0.58 | |
| 255 | D6595 | 13.29 | | 0.54 | |
| 257 | D6595 | 12.87 | | 0.39 | |
| 311 | | ----- | | ----- | |
| 325 | D5185 | 12 | | 0.08 | |
| 331 | D5185Mod. | 11 | | -0.27 | |
| 339 | INH-165 | 10.5 | | -0.44 | |
| 349 | D5185 | 13 | | 0.43 | |
| 360 | D5185 | 12.0 | | 0.08 | |
| 398 | | ----- | | ----- | |
| 432 | D5185 | 12.1 | | 0.12 | |
| 451 | D5185 | 10 | | -0.62 | |
| 496 | D5185 | 11.04 | | -0.25 | |
| 526 | D5185 | 9 | ex | -0.97 | excluded as many statistical outliers in other related metal analyzes |
| 541 | D5185 | 11.3 | | -0.16 | |
| 575 | D6595 | 11 | | -0.27 | |
| 603 | | ----- | | ----- | |
| 614 | D5185 | 12.1 | | 0.12 | |
| 633 | D6595 | 11.7 | | -0.02 | |
| 634 | D6595 | 13.71 | | 0.68 | |
| 780 | D5185 | 12 | | 0.08 | |
| 862 | D5185 | 12 | | 0.08 | |
| 863 | D5185 | 11 | | -0.27 | |
| 912 | D5185 | 11.8 | | 0.01 | |
| 962 | D5185 | 11.38 | | -0.14 | |
| 963 | D5185 | 11.95 | | 0.07 | |
| 975 | D5185 | 11.74 | | -0.01 | |
| 994 | D5185 | 11.5 | | -0.09 | |
| 1059 | | ----- | | ----- | |
| 1146 | D5185 | 11.32 | | -0.16 | |
| 1166 | In house | 10.30 | | -0.52 | |
| 1265 | D6595 | 13.848 | | 0.73 | |
| 1297 | | ----- | | ----- | |
| 1372 | D5185 | 12.33 | | 0.20 | |
| 1435 | D5185 | 12.11 | | 0.12 | |
| 1505 | D5185 | 12.07 | | 0.11 | |
| 1660 | D5185 | 2.3 | R(0.01) | -3.33 | |
| 1740 | D5185 | 11.7 | | -0.02 | |
| 1743 | D5185 | 13 | | 0.43 | |
| 1857 | D5185 | 12.3 | | 0.19 | |
| 1875 | | 20 | R(0.01) | 2.90 | |
| 1888 | D5185 | 9.1 | | -0.94 | |
| 1900 | D5185 | 13.171 | | 0.49 | |
| 2493 | In house | 10.9 | | -0.30 | |
| 6002 | D5185 | 12.2 | | 0.15 | |
| 6016 | | ----- | | ----- | |
| 6080 | D5185 | 14 | | 0.79 | |
| 6261 | D5185 | 9.152 | | -0.92 | |
| | normality | OK | | | |
| | n | 40 | | | |
| | outliers | 2 +1ex | | | |
| | mean (n) | 11.765 | | | |
| | st.dev. (n) | 1.1372 | | | |
| | R(calc.) | 3.184 | | | |
| | st.dev.(D5185:18) | 2.8442 | | | |
| | R(D5185:18) | 7.964 | | | application range 5 – 40 mg/kg |



## Determination of Vanadium as V on sample #19237; results in mg/kg

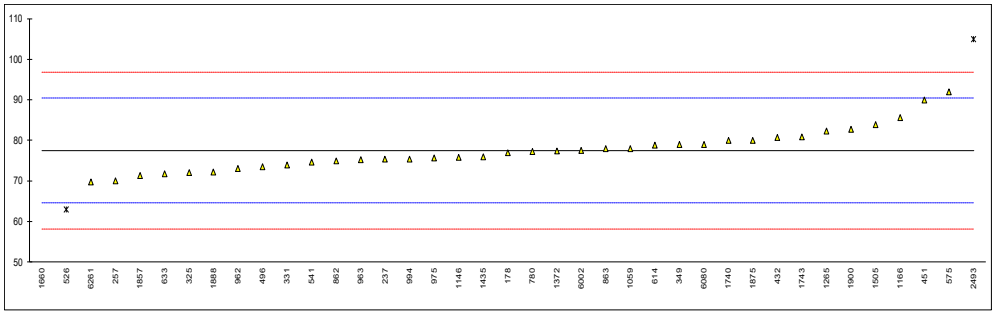
| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D5185 | 11 | | -0.43 | |
| 179 | | ----- | | ----- | |
| 237 | D5185 | 10.42 | | -0.82 | |
| 255 | D6595 | 13.26 | | 1.09 | |
| 257 | D6595 | 12.56 | | 0.62 | |
| 311 | | ----- | | ----- | |
| 325 | D5185 | 13 | | 0.91 | |
| 331 | D5185Mod. | 11 | | -0.43 | |
| 339 | | ----- | | ----- | |
| 349 | D5185 | 13 | | 0.91 | |
| 360 | D5185 | 11.7 | | 0.04 | |
| 398 | | ----- | | ----- | |
| 432 | D5185 | 11.8 | | 0.11 | |
| 451 | D5185 | 10 | | -1.10 | |
| 496 | D5185 | 11.05 | | -0.40 | |
| 526 | D5185 | 8.045 | C,R(0.05) | -2.42 | first reported 8 |
| 541 | D5185 | 11.4 | | -0.16 | |
| 575 | D6595 | 13 | | 0.91 | |
| 603 | | ----- | | ----- | |
| 614 | D5185 | 12.0 | | 0.24 | |
| 633 | D6595 | 10.0 | | -1.10 | |
| 634 | D6595 | 14.07 | | 1.63 | |
| 780 | D5185 | 12 | | 0.24 | |
| 862 | D5185 | 12 | | 0.24 | |
| 863 | D5185 | 11 | | -0.43 | |
| 912 | D5185 | 11.5 | | -0.10 | |
| 962 | D5185 | 12.23 | | 0.40 | |
| 963 | D5185 | 11.87 | | 0.15 | |
| 975 | D5185 | 11.87 | | 0.15 | |
| 994 | D5185 | 11.2 | | -0.30 | |
| 1059 | In house | 12 | | 0.24 | |
| 1146 | D5185 | 11.36 | | -0.19 | |
| 1166 | In house | 10.06 | | -1.06 | |
| 1265 | D6595 | 12.779 | | 0.76 | |
| 1297 | In house | 11.270 | | -0.25 | |
| 1372 | D5185 | 11.64 | | 0.00 | |
| 1435 | D5185 | 11.55 | | -0.06 | |
| 1505 | D5185 | 11.95 | | 0.21 | |
| 1660 | D5185 | 2.4 | R(0.01) | -6.21 | |
| 1740 | D5185 | 11 | | -0.43 | |
| 1743 | D5185 | 12 | | 0.24 | |
| 1857 | D5185 | 12.1 | | 0.31 | |
| 1875 | | 10 | | -1.10 | |
| 1888 | D5185 | 11.12 | C | -0.35 | first reported 16.2 |
| 1900 | D5185 | 12.587 | | 0.64 | |
| 2493 | In house | 10.8 | | -0.57 | |
| 6002 | D5185 | 11.7 | | 0.04 | |
| 6016 | | ----- | | ----- | |
| 6080 | D5185 | 13 | | 0.91 | |
| 6261 | D5185 | 9.094 | | -1.71 | |
| | | | | | |
| | normality | OK | | | |
| | n | 42 | | | |
| | outliers | 2 | | | |
| | mean (n) | 11.641 | | | |
| | st.dev. (n) | 1.0170 | | | |
| | R(calc.) | 2.848 | | | |
| | st.dev.(D5185:18) | 1.4880 | | | |
| | R(D5185:18) | 4.166 | | | application range 1 – 50 mg/kg |

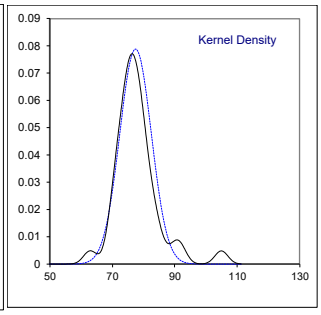


## Determination of Calcium as Ca on sample #19237; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D5185 | 77 | | -0.08 | |
| 179 | | ----- | | ----- | |
| 237 | D5185 | 75.32 | | -0.34 | |
| 255 | | ----- | | ----- | |
| 257 | D6595 | 69.97 | | -1.17 | |
| 311 | | ----- | | ----- | |
| 325 | D5185 | 72 | | -0.86 | |
| 331 | D5185Mod. | 74 | | -0.55 | |
| 339 | | ----- | | ----- | |
| 349 | D5185 | 79 | | 0.23 | |
| 360 | | ----- | | ----- | |
| 398 | | ----- | | ----- | |
| 432 | D5185 | 80.7 | | 0.49 | |
| 451 | D5185 | 90 | | 1.93 | |
| 496 | D5185 | 73.45 | | -0.63 | |
| 526 | D5185 | 63 | ex | -2.26 | excluded as many statistical outliers in other related metal analyzes |
| 541 | D5185 | 74.72 | | -0.44 | |
| 575 | D6595 | 92 | | 2.24 | |
| 603 | | ----- | | ----- | |
| 614 | D5185 | 78.9 | | 0.21 | |
| 633 | D6595 | 71.8 | | -0.89 | |
| 634 | | ----- | | ----- | |
| 780 | D5185 | 77.3 | | -0.04 | |
| 862 | D5185 | 75 | | -0.39 | |
| 863 | D5185 | 78 | | 0.07 | |
| 912 | | ----- | | ----- | |
| 962 | D5185 | 73.12 | | -0.68 | |
| 963 | D5185 | 75.17 | | -0.37 | |
| 975 | D5185 | 75.73 | | -0.28 | |
| 994 | D5185 | 75.4 | | -0.33 | |
| 1059 | In house | 78 | | 0.07 | |
| 1146 | D5185 | 75.78 | | -0.27 | |
| 1166 | In house | 85.57 | | 1.25 | |
| 1265 | D6595 | 82.352 | | 0.75 | |
| 1297 | | ----- | | ----- | |
| 1372 | D5185 | 77.34 | | -0.03 | |
| 1435 | D5185 | 76.00 | | -0.24 | |
| 1505 | D5185 | 83.9 | | 0.99 | |
| 1660 | D5185 | 14 | R(0.01) | -9.86 | |
| 1740 | D5185 | 80 | | 0.38 | |
| 1743 | D5185 | 80.9 | | 0.52 | |
| 1857 | D5185 | 71.41 | | -0.95 | |
| 1875 | | 80 | | 0.38 | |
| 1888 | D5185 | 72.2 | | -0.83 | |
| 1900 | D5185 | 82.789 | | 0.82 | |
| 2493 | In house | 105 | C,R(0.01) | 4.26 | first reported 138 |
| 6002 | D6595 | 77.6 | | 0.01 | |
| 6016 | | ----- | | ----- | |
| 6080 | D5185 | 79 | | 0.23 | |
| 6261 | D5185 | 69.795 | | -1.20 | |

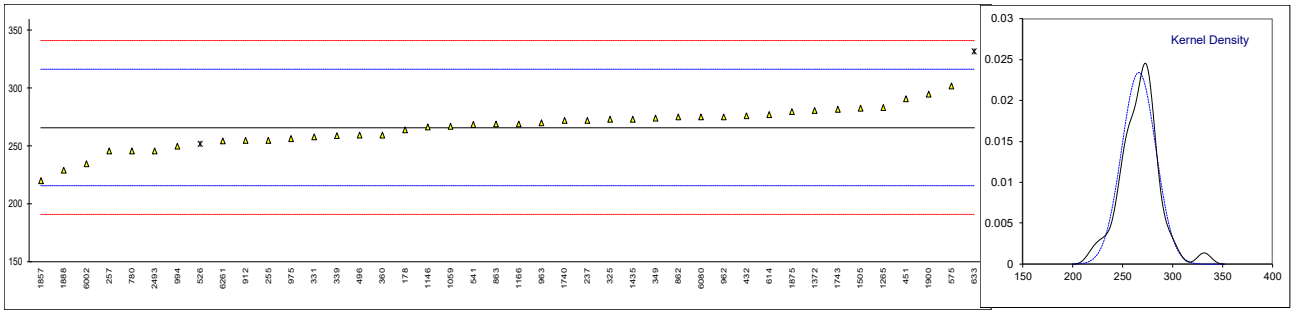
| | | |
|---|---|---|
| | normality | suspect |
| | n | 36 |
| | outliers | 2 +1ex |
| | mean (n) | 77.534 |
| | st.dev. (n) | 5.0551 |
| | R(calc.) | 14.154 |
| | st.dev.(Horwitz) | 6.4449 |
| | R(Horwitz) | 18.046 |
| Compare | | |
| | R(D5185:18) | 4.290 |



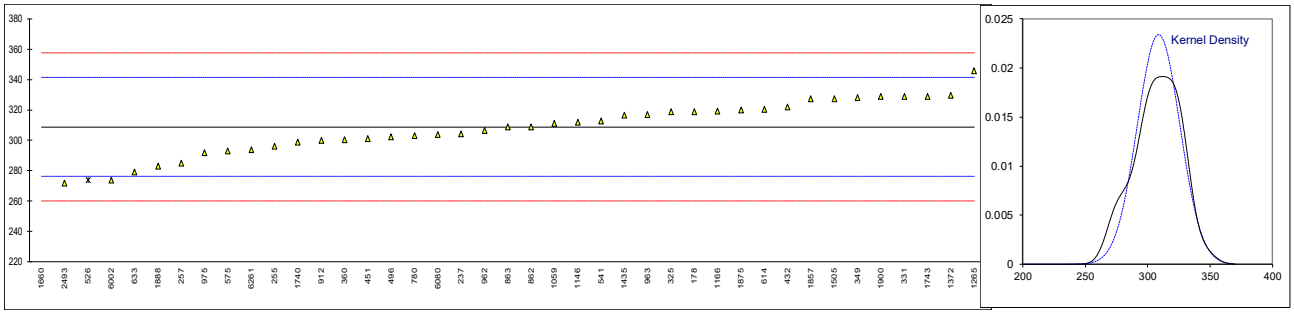
## Determination of Phosphorus as P on sample #19237; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D5185 | 264 | | -0.08 | |
| 179 | | ----- | | ----- | |
| 237 | D5185 | 272.1 | | 0.24 | |
| 255 | D6595 | 255 | | -0.44 | |
| 257 | D6595 | 245.68 | | -0.81 | |
| 311 | | ----- | | ----- | |
| 325 | D5185 | 273 | | 0.28 | |
| 331 | D5185Mod. | 258 | | -0.32 | |
| 339 | INH-165 | 259 | | -0.28 | |
| 349 | D5185 | 274 | | 0.32 | |
| 360 | D5185 | 259.4 | | -0.27 | |
| 398 | | ----- | | ----- | |
| 432 | D5185 | 276.1 | | 0.40 | |
| 451 | D5185 | 291 | | 0.99 | |
| 496 | D5185 | 259.347 | | -0.27 | |
| 526 | D5185 | 252 | ex | -0.56 | excluded as many statistical outliers in other related metal analyzes |
| 541 | D5185 | 268.8 | | 0.11 | |
| 575 | D6595 | 302 | | 1.43 | |
| 603 | | ----- | | ----- | |
| 614 | D5185 | 277.4 | | 0.45 | |
| 633 | D6595 | 331.6 | R(0.05) | 2.62 | |
| 634 | | ----- | | ----- | |
| 780 | D5185 | 246 | | -0.80 | |
| 862 | D5185 | 275 | | 0.36 | |
| 863 | D5185 | 269 | | 0.12 | |
| 912 | D5185 | 255 | | -0.44 | |
| 962 | D5185 | 275.16 | | 0.36 | |
| 963 | D5185 | 270.1 | | 0.16 | |
| 975 | D5185 | 256.3 | | -0.39 | |
| 994 | D5185 | 250 | | -0.64 | |
| 1059 | In house | 267 | | 0.04 | |
| 1146 | D5185 | 266.8 | | 0.03 | |
| 1166 | In house | 269.01 | | 0.12 | |
| 1265 | D6595 | 283.24 | | 0.68 | |
| 1297 | | ----- | | ----- | |
| 1372 | D5185 | 281.00 | | 0.60 | |
| 1435 | D5185 | 273.2 | | 0.28 | |
| 1505 | D5185 | 282.6 | | 0.66 | |
| 1660 | | ----- | | ----- | |
| 1740 | D5185 | 272 | | 0.24 | |
| 1743 | D5185 | 282 | | 0.64 | |
| 1857 | D5185 | 220.3 | | -1.83 | |
| 1875 | | 280 | | 0.56 | |
| 1888 | D5185 | 229 | | -1.48 | |
| 1900 | D5185 | 295.09 | | 1.16 | |
| 2493 | In house | 246 | C | -0.80 | first reported 178 |
| 6002 | D5185 | 235 | | -1.24 | |
| 6016 | | ----- | | ----- | |
| 6080 | D5185 | 275 | | 0.36 | |
| 6261 | D5185 | 254.711 | | -0.45 | |
| | | | | | |
| | normality | OK | | | |
| | n | 40 | | | |
| | outliers | 1 +1ex | | | |
| | mean (n) | 266.083 | | | |
| | st.dev. (n) | 17.0465 | | | |
| | R(calc.) | 47.730 | | | |
| | st.dev.(D5185:18) | 25.0507 | | | |
| | R(D5185:18) | 70.142 | | | application range 10 – 1000 mg/kg |



## Determination of Zinc as Zn on sample #19237; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 178 | D5185 | 319 | | 0.63 | |
| 179 | | ----- | | ----- | |
| 237 | D5185 | 304.3 | | -0.27 | |
| 255 | D6595 | 296 | | -0.78 | |
| 257 | D6595 | 284.88 | | -1.47 | |
| 311 | | ----- | | ----- | |
| 325 | D5185 | 319 | | 0.63 | |
| 331 | D5185Mod. | 329 | | 1.25 | |
| 339 | | ----- | | ----- | |
| 349 | D5185 | 328 | | 1.19 | |
| 360 | D5185 | 300.2 | | -0.53 | |
| 398 | | ----- | | ----- | |
| 432 | D5185 | 322.0 | | 0.82 | |
| 451 | D5185 | 301 | | -0.48 | |
| 496 | D5185 | 302.3 | | -0.40 | |
| 526 | D5185 | 274 | ex | -2.14 | excluded as many statistical outliers in other related metal analyzes |
| 541 | D5185 | 312.6 | | 0.24 | |
| 575 | D6595 | 293 | | -0.97 | |
| 603 | | ----- | | ----- | |
| 614 | D5185 | 320.4 | | 0.72 | |
| 633 | D6595 | 279.3 | | -1.81 | |
| 634 | | ----- | | ----- | |
| 780 | D5185 | 303 | | -0.35 | |
| 862 | D5185 | 309 | | 0.02 | |
| 863 | D5185 | 309 | | 0.02 | |
| 912 | D5185 | 300 | | -0.54 | |
| 962 | D5185 | 306.71 | | -0.12 | |
| 963 | D5185 | 317.0 | | 0.51 | |
| 975 | D5185 | 291.8 | | -1.04 | |
| 994 | | ----- | | ----- | |
| 1059 | In house | 311 | | 0.14 | |
| 1146 | D5185 | 311.9 | | 0.20 | |
| 1166 | In house | 319.17 | | 0.64 | |
| 1265 | D6595 | 345.92 | | 2.29 | |
| 1297 | | ----- | | ----- | |
| 1372 | D5185 | 329.60 | | 1.29 | |
| 1435 | D5185 | 316.6 | | 0.49 | |
| 1505 | D5185 | 327.5 | | 1.16 | |
| 1660 | D5185 | 75 | R(0.01) | -14.40 | |
| 1740 | D5185 | 299 | | -0.60 | |
| 1743 | D5185 | 329 | | 1.25 | |
| 1857 | D5185 | 327.3 | | 1.14 | |
| 1875 | | 320 | | 0.69 | |
| 1888 | D5185 | 283 | | -1.58 | |
| 1900 | D5185 | 328.92 | | 1.24 | |
| 2493 | In house | 272 | | -2.26 | |
| 6002 | D5185 | 274 | | -2.14 | |
| 6016 | | ----- | | ----- | |
| 6080 | D5185 | 304 | | -0.29 | |
| 6261 | D5185 | 293.864 | | -0.92 | |
| | | | | | |
| | normality | OK | | | |
| | n | 39 | | | |
| | outliers | 1 +1ex | | | |
| | mean (n) | 308.725 | | | |
| | st.dev. (n) | 17.0367 | | | |
| | R(calc.) | 47.703 | | | |
| | st.dev.(D5185:18) | 16.2349 | | | |
| | R(D5185:18) | 45.458 | | | application range 60 – 1600 mg/kg |



## APPENDIX 2

### Number of participants per country

| | |
|---|---|
| 1 lab in | ARGENTINA |
| 1 lab in | AUSTRALIA |
| 1 lab in | AUSTRIA |
| 1 lab in | AZERBAIJAN |
| 2 labs in | BELGIUM |
| 1 lab in | BULGARIA |
| 1 lab in | CHILE |
| 2 labs in | CHINA, People's Republic |
| 1 lab in | COLOMBIA |
| 1 lab in | COTE D'IVOIRE |
| 1 lab in | CROATIA |
| 1 lab in | EGYPT |
| 4 labs in | FRANCE |
| 2 labs in | GERMANY |
| 4 labs in | GREECE |
| 1 lab in | HUNGARY |
| 1 lab in | INDIA |
| 1 lab in | INDONESIA |
| 1 lab in | IRAN, Islamic Republic of |
| 2 labs in | ITALY |
| 1 lab in | KAZAKHSTAN |
| 2 labs in | MALAYSIA |
| 1 lab in | MEXICO |
| 1 lab in | MOROCCO |
| 3 labs in | NETHERLANDS |
| 1 lab in | NIGERIA |
| 1 lab in | NORWAY |
| 1 lab in | OMAN |
| 2 labs in | PHILIPPINES |
| 2 labs in | POLAND |
| 1 lab in | PORTUGAL |
| 1 lab in | QATAR |
| 2 labs in | RUSSIAN FEDERATION |
| 2 labs in | SAUDI ARABIA |
| 1 lab in | SINGAPORE |
| 1 lab in | SLOVENIA |
| 1 lab in | SPAIN |
| 1 lab in | SUDAN |
| 2 labs in | SWEDEN |
| 2 labs in | TANZANIA |
| 1 lab in | UNITED ARAB EMIRATES |
| 2 labs in | UNITED KINGDOM |
| 2 labs in | UNITED STATES OF AMERICA |

**APPENDIX 3**

**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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