

# **Results of Proficiency Test**

## **Vacuum Gasoil**

### **December 2015**

Organised by: Institute for Interlaboratory Studies  
Spijkenisse, the Netherlands

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## 1 INTRODUCTION

In 2014, a proficiency test was organized for Vacuum Gas Oil (VGO) by the Institute for Interlaboratory Studies on request of several participants. During the annual proficiency testing program 2015/2016, it was decided to continue the round robin for the analysis of VGO. In this interlaboratory study 59 laboratories in 27 different countries have participated. See appendix 3 for the number of participants per country. In this report, the results of the 2015 VGO proficiency test are presented and discussed. This report is also available through the iis internet site [www.iisnl.com](http://www.iisnl.com).

## 2 SETUP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test. It was decided to send one bottle of 1L VGO (labelled #15250) that was obtained from a local supplier. The analyses for fit-for-use and homogeneity were subcontracted to an accredited laboratory. Participants were requested to report the analytical results using the indicating units and to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

### 2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010 (R007). This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on a regular basis by sending out questionnaires.

### 2.2 PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of April 2014 (iis-protocol, version 3.3). This protocol can be downloaded from the FAQ page of the iis website [www.iisnl.com](http://www.iisnl.com).

### 2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

## 2.4 SAMPLES

The necessary bulk material was obtained from a local supplier. The 80 litre bulk material was transferred after homogenizing into 73 brown glass bottles of 1 litre (labelled #15250). The homogeneity of the subsamples #15250 was checked by determination of Density at 15°C in accordance with ISO12185 on 8 stratified randomly selected samples.

|                 | Density at 15 °C<br>in kg/m <sup>3</sup> |
|-----------------|------------------------------------------|
| Sample #15250-1 | 915.30                                   |
| Sample #15250-2 | 915.30                                   |
| Sample #15250-3 | 915.30                                   |
| Sample #15250-4 | 915.30                                   |
| Sample #15250-5 | 915.20                                   |
| Sample #15250-6 | 915.20                                   |
| Sample #15250-7 | 915.30                                   |
| Sample #15250-8 | 915.30                                   |

Table 1: homogeneity test results of subsamples #15250

From the above test results, the repeatability was calculated and compared with 0.3 times the corresponding reproducibility in agreement with the procedure of ISO 13528, Annex B2 in the next table:

|                           | Density at 15 °C<br>in kg/m <sup>3</sup> |
|---------------------------|------------------------------------------|
| r (observed)              | 0.05                                     |
| reference method          | ISO12185:96                              |
| 0.3 x R(reference method) | 0.45                                     |

Table 2: evaluation of the repeatability of the subsamples #15250

The calculated repeatability is less than 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneity of the subsamples #15250 was assumed.

To each of the participating laboratories, 1 sample of 1 L brown glass (labelled #15250) was sent on Nov 25, 2015.

## 2.5 STABILITY OF THE SAMPLES

The stability of Vacuum Gas Oil was tested in the past and proved to be sufficiently stable for the period of the proficiency test.

## 2.6 ANALYSES

The participants were requested to determine on sample #15250: Acid Number (Total), Aniline Point, Asphaltenes, Carbon Residue (Micro method), Density at 15°C, Flash Point PMcc, Kinematic Viscosity at 50°C and at 100°C, Nitrogen, Pour Point (manual or automatic), Sulphur (Total), Metals (Arsenic, Calcium, Copper, Iron, Nickel, Silicon, Sodium, Vanadium), Simulated Distillation and Distillation (IBP, 10% rec, 30% rec, 50% rec, 70% rec, 90% rec and FBP).

To get comparable results, a detailed report form on which the units were prescribed as well as the required standards and a letter of instructions were prepared and made available on the data entry portal [www.kpmd.co.uk/sgs-iis/](http://www.kpmd.co.uk/sgs-iis/). A SDS and a form to confirm receipt of the sample were added to the sample package.

## 3 RESULTS

During five weeks after sample dispatch, the results of the individual laboratories were received via the data entry portal [www.kpmd.co.uk/sgs-iis/](http://www.kpmd.co.uk/sgs-iis/). The reported results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after deadline, a reminder was sent to those laboratories that had not reported results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A 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 (raw data of the) reported results. Additional or corrected results have been used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

### 3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' (iis-protocol, April 2014 version 3.3). For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. Results reported as '<...' or '>...' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.

In accordance to ISO 5725 the original results per determination were submitted to Dixon's, and/or 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 the averages and the standard deviations.

For each assigned value, the uncertainty was determined in accordance to ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance to ISO13528. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

### 3.2 GRAPHICS

In order to visualise the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for each determination (see appendix 1). On the Y-axis the reported analysis results are plotted. The corresponding laboratory numbers are under 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 standard. 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. In order to be able to have an objective evaluation of the performance of each participant, it was decided to evaluate this performance against the literature requirements, e.g. ASTM reproducibilities. Therefore, the z-scores were calculated using a target standard deviation. This target standard deviation was calculated from the literature reproducibility (R) by division with 2.8.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated according to:

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

The  $Z_{(\text{target})}$  scores are listed 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 interlaboratory study, some problems with sample dispatch were encountered due to several reasons. Four participants reported the test results after the final reporting date and five participants did not report any test results at all. Not all laboratories were able to report all requested parameters. In total 54 participants reported 897 test results. Observed were 22 outlying results, which is 2.5% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

### 4.1 EVALUATION PER TEST

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

In the iis PT reports, ASTM methods are referred to with a number (e.g. D2086) and an added designation for the year that the method was adopted or revised (e.g. D2086-08). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D2086-08 (2013)). In the results tables of Appendix 1 only the method number and year of adoption or revision will be used.

Although VGO is an important feedstock for cracking installations, there are very few analytical standard methods specifically designed for the analysis of VGO. Most parameters are to be determined by using methods that are intended for residual fuel oil and blending components, where applicable, precision data for Fuel Oil were used.

The IP-PM-CW standard is a specific method for the determination of metallic elements in vacuum gasoil in the concentration range 0.1 mg/kg to 10 mg/kg. Regretfully IP-PM-CW does not mention precision data. It is expected that the precision data of this method will be determined in the near future.

Acid Number (total): This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D664:11a.

Aniline Point: This determination was very problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not at all in agreement with the requirements of ASTM D611:12.

The test results of the aniline point of n-heptane vary over a small range (69.2 – 69.5). When the Aniline Point results of the participants who had reported a value for the aniline point of n-heptane are only evaluated, the reproducibility hardly improves.

- Asphaltenes: No significant conclusions were drawn because the precision data of IP143:04 are applicable to values between 0.50% M/M and 30.0% M/M.
- Carbon Residue: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of ASTM D4530:15.
- Density at 15°C: 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 ISO 12185:96.
- Flash Point PMcc: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with requirements of ASTM D93-B:15a.
- Kin.Visco. at 50°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D445:15a.
- Kin.Visco.at 100°C: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of ASTM D445:15a.
- Nitrogen: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with ASTM D5762:12.
- Pour Point (Manual): This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with ASTM D97:15.
- Pour Point (Automatic): This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with ASTM D5950:14.
- Sulphur (Total): This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D4294:10.
- Arsenic (As): Only two participants reported a numerical result. Therefore no significant conclusions were drawn.

Calcium (Ca):

This determination may be not problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is almost in agreement with the requirements of IP501:05.

Copper (Cu):

All reported test results were near or below the application range of method IP-PM-CW (0.1 – 10 mg/kg). Therefore no statistical conclusions were drawn.

Iron (Fe):

This determination may be not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in full agreement with the requirements of IP501:05.

Nickel (Ni):

This determination may be not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of IP501:05.

Silicon (Si):

This determination may be problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the (strict) requirements of IP501:05, nor with the less strict requirements of IP470. When the IP501 results test results were evaluated separately, the calculated reproducibility is still not in agreement with the requirements of IP501:05.

Sodium (Na):

This determination may be not problematic. No statistical outliers were observed. The calculated reproducibility in full agreement with the requirements of IP501:05.

Vanadium (V):

This determination may be not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in good agreement with the requirements of IP501:05.

Sim. Distillation

The Simulated Distillation according to ASTM D6352:14 was partly problematic. In total two statistical outliers were observed. The calculated reproducibilities were in agreement with the requirements of ASTM D6352:15 for IBP, 30% and 50% recovery. For 10%, 70%, 90% recovery and FBP the calculated reproducibilities were not in agreement with the requirements of ASTM D6352:15.

DistillationAcc. D1160

The distillation according to ASTM D1160 was partly problematic. In total two statistical outliers were observed and one result was excluded as the determination was terminated prior to the reading of FBP. The calculated reproducibilities were in agreement for 30%, 50%, 90% recovery and FBP with the requirements of ASTM D1160:15. For IBP, 10%, 70% recovery the calculated reproducibilities were not in agreement with the requirements of ASTM D1160:15.

## 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and the reproducibility as found for the group of participating laboratories that participated. The average results, calculated reproducibilities and reproducibilities derived from literature standards (in casu ASTM standards), are compared in the next table.

| Parameter                           | unit               | n  | Average | 2.8 * sd | R(lit) |
|-------------------------------------|--------------------|----|---------|----------|--------|
| Acid Number (Total)                 | mg KOH/g           | 39 | 0.87    | 0.18     | 0.26   |
| Aniline Point                       | °C                 | 23 | 81.6    | 1.9      | 1.0    |
| Asphaltenes                         | %M/M               | 26 | 0.13    | 0.31     | (0.03) |
| Carbon Residue, micro method        | %M/M               | 38 | 0.40    | 0.16     | 0.16   |
| Density at 15 °C                    | kg/m <sup>3</sup>  | 52 | 915.6   | 1.6      | 1.5    |
| Flash Point PMcc                    | °C                 | 43 | 168.1   | 6.6      | 10.0   |
| Kinematic Viscosity at 50 °C        | mm <sup>2</sup> /s | 49 | 32.6    | 0.7      | 2.4    |
| Kinematic Viscosity at 100 °C       | mm <sup>2</sup> /s | 42 | 6.7     | 0.2      | 0.6    |
| Nitrogen                            | mg/kg              | 29 | 1396    | 406      | 371    |
| Pour Point, manual                  | °C                 | 38 | 26.1    | 8.1      | 9.0    |
| Pour Point, automated, 3°C interval | °C                 | 11 | 26.7    | 4.6      | 4.5    |
| Sulphur Content (Total)             | %M/M               | 52 | 0.52    | 0.03     | 0.05   |
| Arsenic (As)                        | mg/kg              | 6  | <1      | n.a.     | n.a.   |
| Calcium (Ca)                        | mg/kg              | 20 | 0.66    | 0.56     | 0.49   |
| Copper (Cu)                         | mg/kg              | 25 | <0.1    | n.a.     | n.a.   |
| Iron (Fe)                           | mg/kg              | 32 | 0.53    | 0.66     | 0.66   |
| Nickel (Ni)                         | mg/kg              | 30 | 0.16    | 0.19     | 0.61   |
| Silicon (Si)                        | mg/kg              | 16 | 0.65    | 1.20     | 0.21   |
| Sodium (Na)                         | mg/kg              | 31 | 0.92    | 1.05     | 1.02   |
| Vanadium (V)                        | mg/kg              | 31 | 0.37    | 0.52     | 0.93   |
| Simulated distillation D6352        |                    |    |         |          |        |
| - Initial Boiling Point             | °C                 | 13 | 243.5   | 28.2     | 49.1   |
| - Temp 10% recovered                | °C                 | 13 | 330.9   | 9.1      | 7.1    |
| - Temp 30% recovered                | °C                 | 12 | 390.5   | 5.4      | 5.9    |
| - Temp 50% recovered                | °C                 | 12 | 427.3   | 6.1      | 6.4    |
| - Temp 70% recovered                | °C                 | 13 | 466.7   | 8.7      | 7.2    |
| - Temp 90% recovered                | °C                 | 13 | 519.8   | 12.8     | 10.5   |
| - Final Boiling Point               | °C                 | 12 | 617.4   | 105.1    | 38.1   |

| Parameter               | unit | n  | Average | 2.8 * sd | R(lit) |
|-------------------------|------|----|---------|----------|--------|
| Distillation D1160      |      |    |         |          |        |
| - Initial Boiling Point | °C   | 24 | 265.7   | 56.7     | 49.5   |
| - Temp 10% recovered    | °C   | 24 | 347.6   | 24.3     | 17.1   |
| - Temp 30% recovered    | °C   | 22 | 399.2   | 10.7     | 12.7   |
| - Temp 50% recovered    | °C   | 24 | 433.1   | 11.8     | 11.6   |
| - Temp 70% recovered    | °C   | 24 | 468.0   | 12.3     | 9.8    |
| - Temp 90% recovered    | °C   | 24 | 518.4   | 16.8     | 22.4   |
| - Final Boiling Point   | °C   | 22 | 553.8   | 26.3     | 26.9   |

Table 3: reproducibilities of results of sample #15250

results between brackets should be used with care, because the average was below the application range

Without further statistical calculations it can be concluded that for a number of tests there is a not a good compliance of the group of participants with the relevant standards. The tests that are problematic have been discussed in paragraph 4.1.

#### 4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2015 WITH PREVIOUS PTS

|                            | December 2015 | December 2014 | November 2013 |
|----------------------------|---------------|---------------|---------------|
| Number of reporting labs   | 54            | 51            | 32            |
| Number of results reported | 897           | 785           | 474           |
| Statistical outliers       | 22            | 35            | 12            |
| Percentage outliers        | 2.5%          | 4.5%          | 2.5%          |

Table 4: comparison with previous proficiency tests.

In proficiency tests, outlier percentages of 3% - 7.5% are quite normal.

The performance of the determinations of the proficiency tests was compared against the requirements of the respective standards. The conclusions are given in the following table:

| Determination                 | December 2015 | December 2014 | November 2013 |
|-------------------------------|---------------|---------------|---------------|
| Acid Number (Total)           | +             | +             | ++            |
| Aniline Point                 | --            | --            | --            |
| Asphaltenes                   | (--)          | (--)          | (--)          |
| Carbon Residue, micro method  | +/-           | --            | +             |
| Density at 15 °C              | +/-           | +/-           | +/-           |
| Flash Point PMcc              | +             | +/-           | +             |
| Kinematic Viscosity at 50 °C  | ++            | --            | ++            |
| Kinematic Viscosity at 100 °C | ++            | ++            | ++            |
| Nitrogen                      | +/-           | -             | +             |
| Sulphur Content (Total)       | +             | +/-           | +/-           |
| Arsenic (As)                  | n.e.          | n.e.          | n.e.          |
| Calcium (Ca)                  | +/-           | --            | -             |
| Copper (Cu)                   | n.e.          | n.e.          | n.e.          |
| Nickel (Ni)                   | ++            | +/-           | ++            |
| Iron (Fe)                     | +/-           | +             | ++            |
| Silicon (Si)                  | --            | (--)          | (--)          |
| Sodium (Na)                   | +/-           | +/-           | +/-           |

| Determination                | December 2015 | December 2014 | November 2013 |
|------------------------------|---------------|---------------|---------------|
| Vanadium (V)                 | +             | ++            | ++            |
| Simulated distillation D6352 |               |               |               |
| - Initial Boiling Point      | +             | +/-           | +             |
| - Temp 10% recovered         | -             | -             | +             |
| - Temp 30% recovered         | +/-           | -             | -             |
| - Temp 50% recovered         | +/-           | -             | -             |
| - Temp 70% recovered         | -             | +/-           | -             |
| - Temp 90% recovered         | -             | +/-           | +             |
| - Final Boiling Point        | --            | -             | +             |
| Distillation D1160           |               |               |               |
| - Initial Boiling Point      | -             | --            | --            |
| - Temp 10% recovered         | -             | +             | +             |
| - Temp 30% recovered         | +             | +/-           | +             |
| - Temp 50% recovered         | +/-           | +/-           | +             |
| - Temp 70% recovered         | -             | -             | +             |
| - Temp 90% recovered         | +             | ++            | ++            |
| - Final Boiling Point        | +/-           | -             | -             |

Table 5: comparison determinations against the standard

results between brackets should be used with care, because the average was below the application range

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

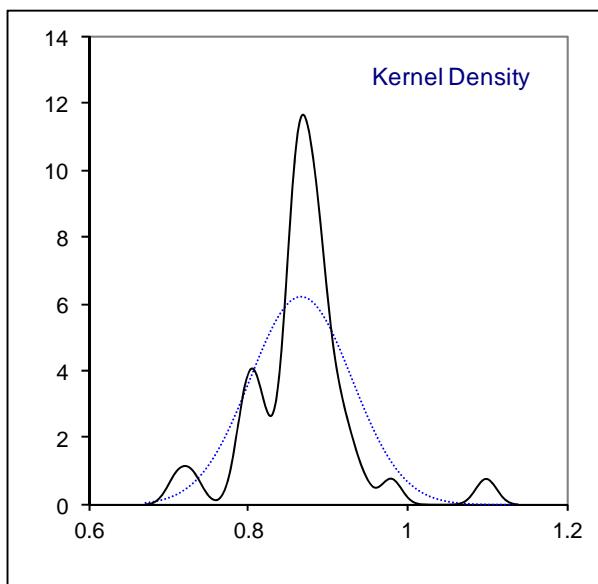
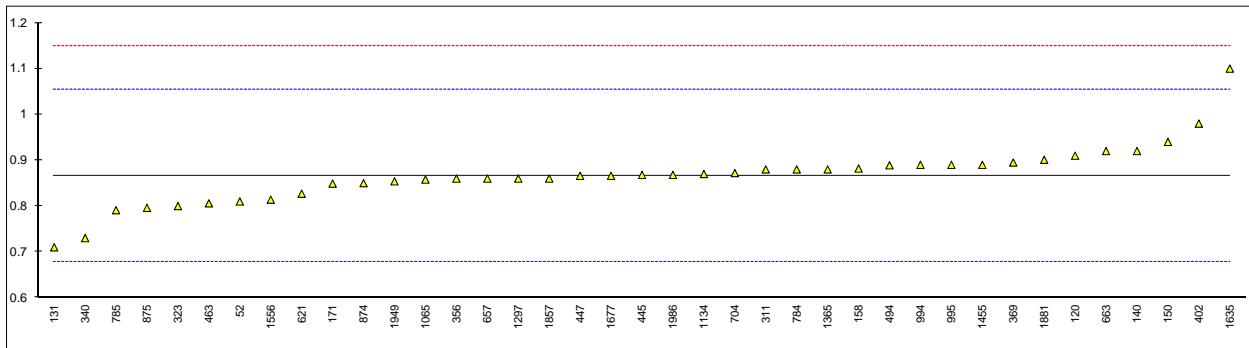
- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- : group performed worse than the standard
- : group performed much worse than the standard
- n.e.: not evaluated

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**APPENDIX 1**

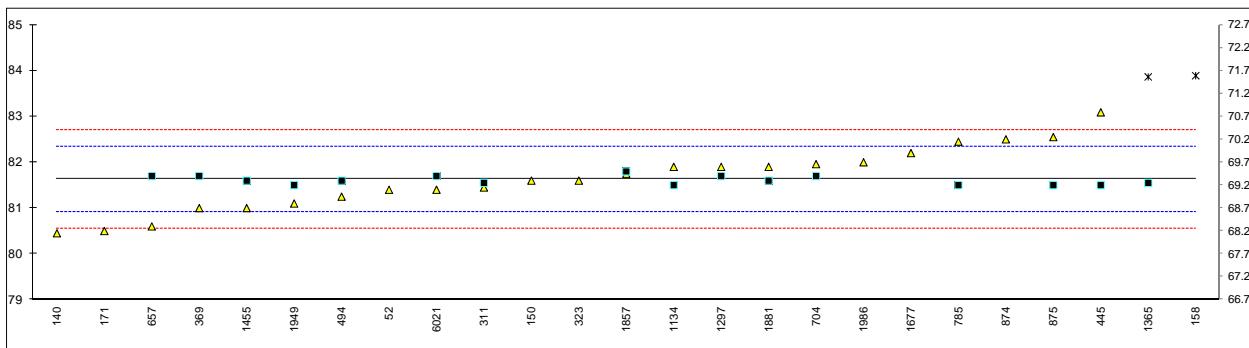
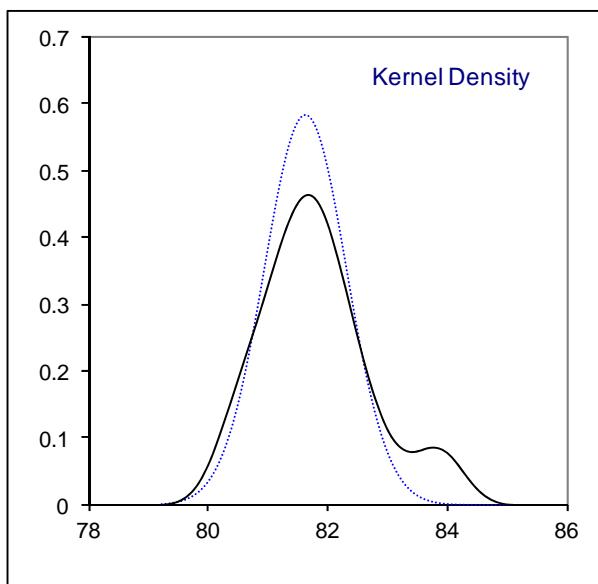
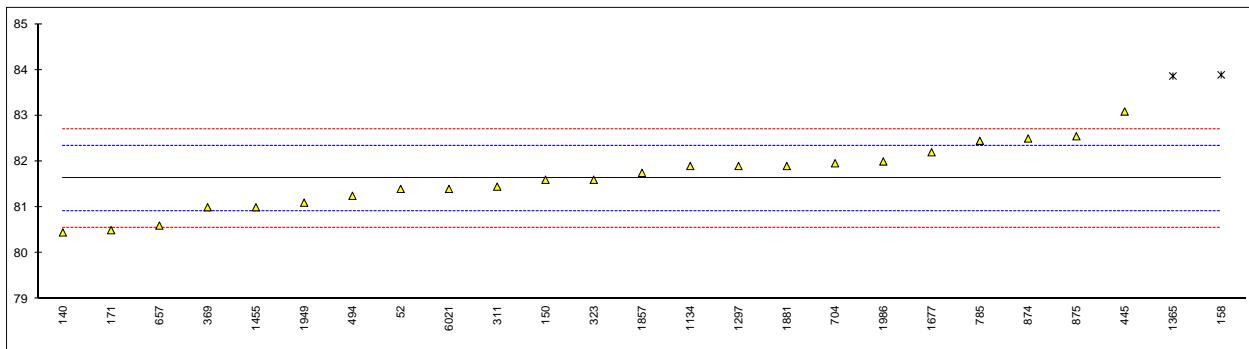
Determination of Acid Number (Total) on sample #15250; results in mg KOH/g

| lab         | method | value   | mark | z(targ) | remarks              |
|-------------|--------|---------|------|---------|----------------------|
| 52          | D664   | 0.81    |      | -0.60   |                      |
| 62          |        | ----    |      | -----   |                      |
| 120         | D664   | 0.91    |      | 0.46    |                      |
| 131         | D664   | 0.71    |      | -1.66   |                      |
| 133         |        | ----    |      | -----   |                      |
| 140         | D664   | 0.920   |      | 0.57    |                      |
| 150         | D664   | 0.94    |      | 0.78    |                      |
| 158         | D664   | 0.8820  |      | 0.17    |                      |
| 171         | D664   | 0.849   |      | -0.19   |                      |
| 228         |        | ----    |      | -----   |                      |
| 311         | D664   | 0.88    |      | 0.14    |                      |
| 313         |        | ----    |      | -----   |                      |
| 317         |        | ----    |      | -----   |                      |
| 323         | D664   | 0.80    |      | -0.71   |                      |
| 333         |        | ----    |      | -----   |                      |
| 334         |        | ----    |      | -----   |                      |
| 336         |        | ----    |      | -----   |                      |
| 337         |        | ----    |      | -----   |                      |
| 340         | D664   | 0.730   |      | -1.45   |                      |
| 356         | D664   | 0.86    | C    | -0.07   | First reported: 0.02 |
| 369         | D664   | 0.895   |      | 0.30    |                      |
| 402         | D664   | 0.98    |      | 1.21    |                      |
| 445         | D664   | 0.868   |      | 0.02    |                      |
| 447         | D664   | 0.866   |      | 0.00    |                      |
| 463         | D664   | 0.806   |      | -0.64   |                      |
| 494         | D664   | 0.889   |      | 0.24    |                      |
| 621         | D664   | 0.827   |      | -0.42   |                      |
| 657         | D664   | 0.86    |      | -0.07   |                      |
| 663         | D664   | 0.92    |      | 0.57    |                      |
| 704         | D664   | 0.872   |      | 0.06    |                      |
| 732         |        | ----    |      | -----   |                      |
| 743         |        | ----    |      | -----   |                      |
| 781         |        | ----    |      | -----   |                      |
| 784         | D664   | 0.88    |      | 0.14    |                      |
| 785         | D664   | 0.791   |      | -0.80   |                      |
| 791         |        | ----    |      | -----   |                      |
| 874         | D664   | 0.85    |      | -0.18   |                      |
| 875         | D664   | 0.796   |      | -0.75   |                      |
| 994         | D664   | 0.89    |      | 0.25    |                      |
| 995         | D664   | 0.89    |      | 0.25    |                      |
| 1065        | D664   | 0.8578  |      | -0.09   |                      |
| 1134        | D664   | 0.87    |      | 0.04    |                      |
| 1297        | D664   | 0.86    |      | -0.07   |                      |
| 1365        | D664   | 0.88    |      | 0.14    |                      |
| 1379        |        | ----    |      | -----   |                      |
| 1397        |        | ----    |      | -----   |                      |
| 1455        | D664   | 0.89    |      | 0.25    |                      |
| 1510        |        | ----    |      | -----   |                      |
| 1543        |        | ----    |      | -----   |                      |
| 1556        | D664   | 0.814   |      | -0.56   |                      |
| 1635        | D664   | 1.10    |      | 2.48    |                      |
| 1677        | D664   | 0.866   |      | 0.00    |                      |
| 1857        | D664   | 0.86    |      | -0.07   |                      |
| 1881        | D664   | 0.901   |      | 0.37    |                      |
| 1949        | D664-A | 0.854   |      | -0.13   |                      |
| 1986        | D664   | 0.868   |      | 0.02    |                      |
| 1995        |        | ----    |      | -----   |                      |
| 6020        |        | ----    |      | -----   |                      |
| 6021        |        | ----    |      | -----   |                      |
|             |        |         |      |         |                      |
| normality   |        | not OK  |      |         |                      |
| n           |        | 39      |      |         |                      |
| outliers    |        | 0       |      |         |                      |
| mean (n)    |        | 0.8665  |      |         |                      |
| st.dev. (n) |        | 0.06403 |      |         |                      |
| R(calc.)    |        | 0.1793  |      |         |                      |
| R(D664:11a) |        | 0.2632  |      |         |                      |



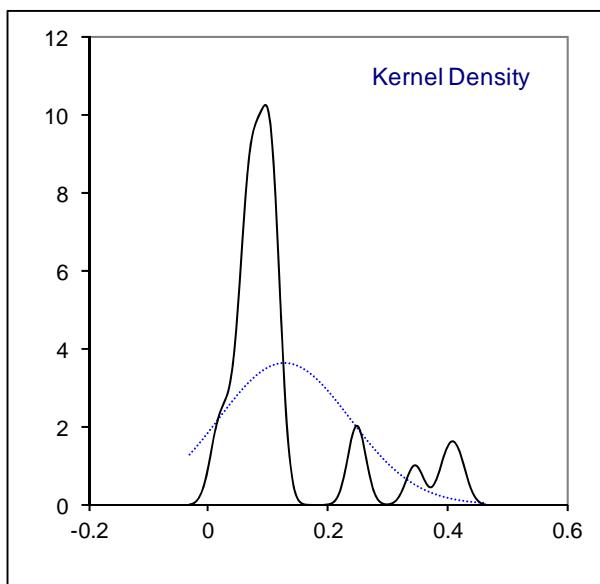
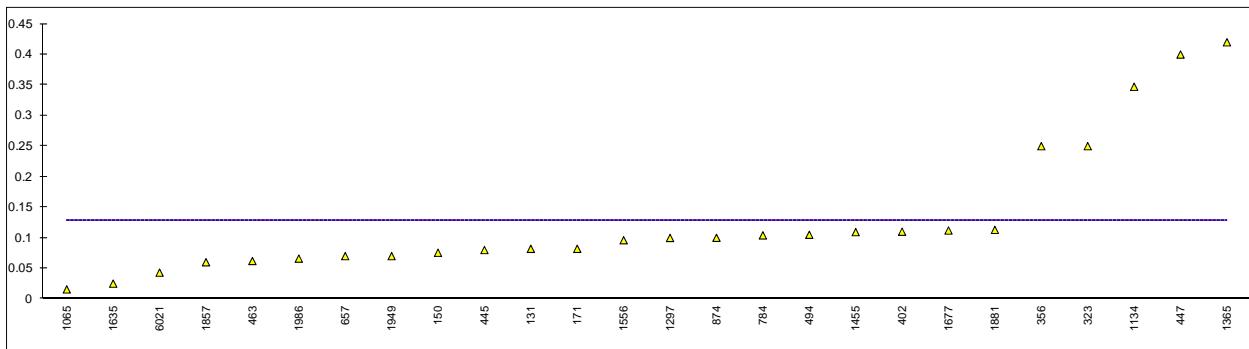
## Determination of Aniline Point on sample #15250; results in °C

| lab  | method      | value  | mark     | z(targ) | remarks                                                | Aniline Point n-Heptane |
|------|-------------|--------|----------|---------|--------------------------------------------------------|-------------------------|
| 52   | D611-E      | 81.4   |          | -0.65   |                                                        | ---                     |
| 62   |             | ----   |          | ----    |                                                        | ---                     |
| 120  |             | ----   |          | ----    |                                                        | ---                     |
| 131  |             | ----   |          | ----    |                                                        | ---                     |
| 133  |             | ----   |          | ----    |                                                        | ---                     |
| 140  | D611-B      | 80.45  |          | -3.31   |                                                        | ---                     |
| 150  | D611-E      | 81.60  |          | -0.09   |                                                        | ---                     |
| 158  | D611-B      | 83.89  | DG(0.05) | 6.32    |                                                        | ---                     |
| 171  | D611-E      | 80.5   | C        | -3.17   | First reported: 84.1                                   | ---                     |
| 228  |             | ----   |          | ----    |                                                        | ---                     |
| 311  | D611-B      | 81.45  |          | -0.51   |                                                        | 69.25                   |
| 313  |             | ----   |          | ----    |                                                        | ---                     |
| 317  |             | ----   |          | ----    |                                                        | ---                     |
| 323  | D611-A      | 81.6   |          | -0.09   |                                                        | ---                     |
| 333  |             | ----   |          | ----    |                                                        | ---                     |
| 334  |             | ----   |          | ----    |                                                        | ---                     |
| 336  |             | ----   |          | ----    |                                                        | ---                     |
| 337  |             | ----   |          | ----    |                                                        | ---                     |
| 340  |             | ----   |          | ----    |                                                        | ---                     |
| 356  |             | ----   |          | ----    |                                                        | ---                     |
| 369  | D611-B      | 81.0   | C        | -1.77   | First reported: 84.3                                   | 69.4                    |
| 402  |             | ----   |          | ----    |                                                        | ---                     |
| 445  | D611-B      | 83.09  |          | 4.08    |                                                        | 69.20                   |
| 447  |             | ----   |          | ----    |                                                        | ---                     |
| 463  |             | ----   |          | ----    |                                                        | ---                     |
| 494  | D611-E      | 81.25  |          | -1.07   |                                                        | 69.3                    |
| 621  |             | ----   |          | ----    |                                                        | ---                     |
| 657  | D611-B      | 80.6   | C        | -2.89   | First reported: 79                                     | 69.40                   |
| 663  |             | ----   |          | ----    |                                                        | ---                     |
| 704  | D611-B      | 81.96  |          | 0.92    |                                                        | 69.40                   |
| 732  |             | ----   |          | ----    |                                                        | ---                     |
| 743  |             | ----   |          | ----    |                                                        | ---                     |
| 781  |             | ----   |          | ----    |                                                        | ---                     |
| 784  |             | ----   |          | ----    |                                                        | ---                     |
| 785  | D611-E      | 82.45  |          | 2.29    |                                                        | 69.2                    |
| 791  |             | ----   |          | ----    |                                                        | ---                     |
| 874  | D611-E      | 82.50  |          | 2.43    |                                                        | ---                     |
| 875  | D611-E      | 82.55  |          | 2.57    |                                                        | 69.20                   |
| 994  |             | ----   |          | ----    |                                                        | ---                     |
| 995  |             | ----   |          | ----    |                                                        | ---                     |
| 1065 |             | ----   |          | ----    |                                                        | ---                     |
| 1134 | D611-B      | 81.9   |          | 0.75    |                                                        | 69.2                    |
| 1297 | D611-E      | 81.9   |          | 0.75    |                                                        | 69.4                    |
| 1365 | D611-E      | 83.863 | DG(0.05) | 6.25    |                                                        | 69.25                   |
| 1379 |             | ----   |          | ----    |                                                        | ---                     |
| 1397 |             | ----   |          | ----    |                                                        | ---                     |
| 1455 | D611-E      | 81.00  |          | -1.77   |                                                        | 69.30                   |
| 1510 |             | ----   |          | ----    |                                                        | ---                     |
| 1543 |             | ----   |          | ----    |                                                        | ---                     |
| 1556 |             | ----   |          | ----    |                                                        | ---                     |
| 1635 |             | ----   |          | ----    |                                                        | ---                     |
| 1677 | D611-A      | 82.2   |          | 1.59    |                                                        | ---                     |
| 1857 | D611-B      | 81.75  |          | 0.33    |                                                        | 69.5                    |
| 1881 | D611-B      | 81.900 |          | 0.75    |                                                        | 69.300                  |
| 1949 | D611-B      | 81.10  |          | -1.49   |                                                        | 69.20                   |
| 1986 | D611-B      | 82.00  |          | 1.03    |                                                        | ---                     |
| 1995 |             | ----   |          | ----    |                                                        | ---                     |
| 6020 |             | ----   |          | ----    | <b>results Aniline Point only</b>                      | ---                     |
| 6021 | D611-B      | 81.40  |          | -0.65   | of which an Aniline Point<br>of n-Heptane was reported | 69.40                   |
|      | normality   | OK     |          |         | OK                                                     |                         |
|      | n           | 23     |          | 15      |                                                        | 16                      |
|      | outliers    | 2      |          | 1       |                                                        | 0                       |
|      | mean (n)    | 81.633 |          | 81.687  |                                                        | 69.306                  |
|      | st.dev. (n) | 0.6842 |          | 0.6713  |                                                        | 0.0981                  |
|      | R(calc.)    | 1.916  |          | 1.880   |                                                        | 0.275                   |
|      | R(D611:12)  | 1.000  |          | 1.000   |                                                        | n.a.                    |



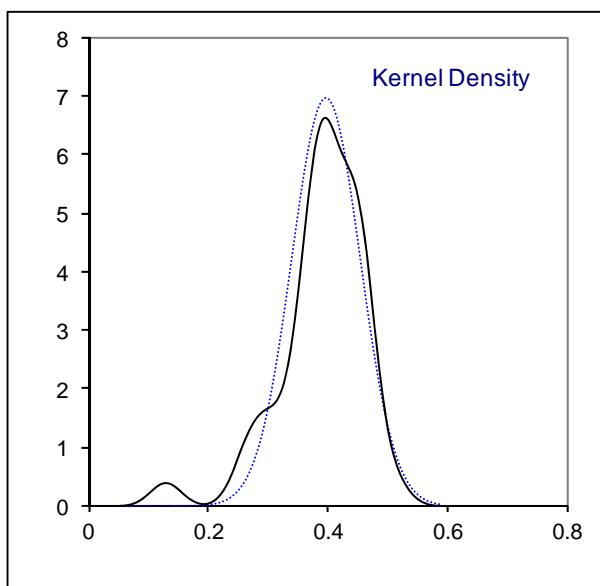
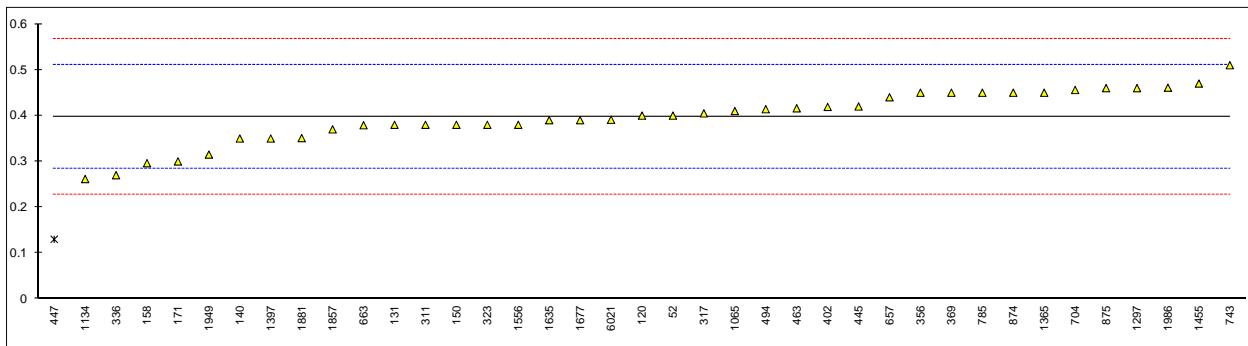
## Determination of Asphaltenes on sample #15250; results in %M/M

| lab         | method  | value    | mark | z(targ) | remarks                                                 |
|-------------|---------|----------|------|---------|---------------------------------------------------------|
| 52          |         | ----     |      |         |                                                         |
| 62          |         | ----     |      |         |                                                         |
| 120         |         | ----     |      |         |                                                         |
| 131         | D6560   | 0.082    |      |         |                                                         |
| 133         |         | ----     |      |         |                                                         |
| 140         |         | ----     |      |         |                                                         |
| 150         | IP143   | 0.0755   |      |         |                                                         |
| 158         |         | ----     |      |         |                                                         |
| 171         | IP143   | 0.082    | C    |         | First reported: 0.648                                   |
| 228         |         | ----     |      |         |                                                         |
| 311         |         | ----     |      |         |                                                         |
| 313         |         | ----     |      |         |                                                         |
| 317         |         | ----     |      |         |                                                         |
| 323         | IP143   | 0.25     |      |         |                                                         |
| 333         |         | ----     |      |         |                                                         |
| 334         |         | ----     |      |         |                                                         |
| 336         |         | ----     |      |         |                                                         |
| 337         |         | ----     |      |         |                                                         |
| 340         |         | ----     |      |         |                                                         |
| 356         | IP143   | 0.25     |      |         |                                                         |
| 369         |         | ----     |      |         |                                                         |
| 402         | D6560   | 0.11     |      |         |                                                         |
| 445         | IP143   | 0.08     |      |         |                                                         |
| 447         | IP143   | 0.40     |      |         |                                                         |
| 463         | IP143   | 0.062    |      |         |                                                         |
| 494         | IP143   | 0.105    |      |         |                                                         |
| 621         |         | ----     |      |         |                                                         |
| 657         | IP143   | 0.07     |      |         |                                                         |
| 663         |         | ----     |      |         |                                                         |
| 704         | IP143   | <0.50    |      |         |                                                         |
| 732         |         | ----     |      |         |                                                         |
| 743         |         | ----     |      |         |                                                         |
| 781         |         | ----     |      |         |                                                         |
| 784         | IP143   | 0.104    |      |         |                                                         |
| 785         |         | ----     |      |         |                                                         |
| 791         |         | ----     |      |         |                                                         |
| 874         | IP143   | 0.10     |      |         |                                                         |
| 875         |         | ----     |      |         |                                                         |
| 994         |         | ----     |      |         |                                                         |
| 995         | IP143   | <0.5     |      |         |                                                         |
| 1065        | IP143   | 0.01551  |      |         |                                                         |
| 1134        | IP143   | 0.3472   |      |         |                                                         |
| 1297        | D6560   | 0.0998   |      |         |                                                         |
| 1365        | IP143   | 0.42     |      |         |                                                         |
| 1379        |         | ----     |      |         |                                                         |
| 1397        | D6560   | <0.5     |      |         |                                                         |
| 1455        | INH-642 | 0.1093   |      |         |                                                         |
| 1510        |         | ----     |      |         |                                                         |
| 1543        |         | ----     |      |         |                                                         |
| 1556        | IP143   | 0.0961   |      |         |                                                         |
| 1635        | D6560   | 0.025    |      |         |                                                         |
| 1677        | INH-642 | 0.112    |      |         |                                                         |
| 1857        | IP143   | 0.06     |      |         |                                                         |
| 1881        | INH-642 | 0.113    |      |         |                                                         |
| 1949        | IP143   | 0.07     | C    |         | First reported: 0.28, also reported for INH-642: 0.0989 |
| 1986        | D6560   | 0.066    |      |         |                                                         |
| 1995        |         | ----     |      |         |                                                         |
| 6020        |         | ----     |      |         |                                                         |
| 6021        | IP143   | 0.043    |      |         |                                                         |
| normality   |         |          |      |         |                                                         |
| n           |         | not OK   |      |         | precision applicable between 0.50 and 30.0 %M/M         |
| outliers    |         | 26       |      |         |                                                         |
| mean (n)    |         | 0        |      |         |                                                         |
| st.dev. (n) |         | 0.1287   |      |         |                                                         |
| R(calc.)    |         | 0.10977  |      |         |                                                         |
| R(IP143:04) |         | 0.3074   |      |         |                                                         |
|             |         | (0.0257) |      |         | Compare R(iis14G06)=0.2978 at a mean conc. of 0.10%M/M  |



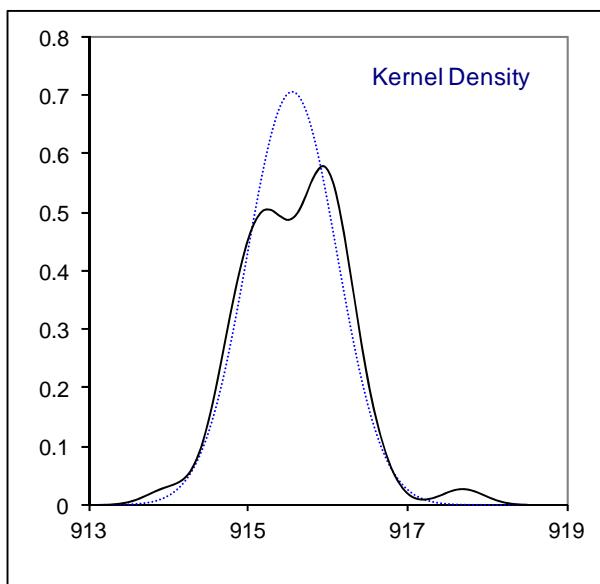
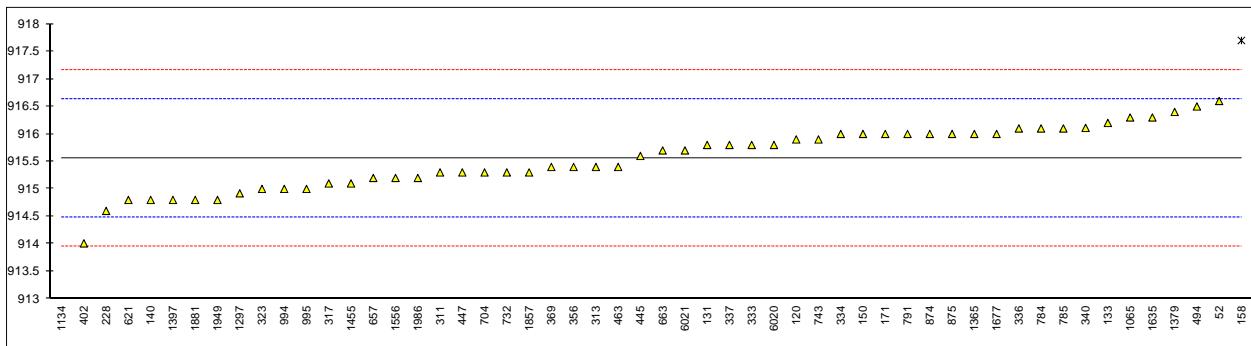
## Determination of Carbon Residue, Micro method on sample #15250; results in %M/M

| lab         | method   | value   | mark    | z(targ) | remarks |
|-------------|----------|---------|---------|---------|---------|
| 52          | D4530    | 0.40    |         | 0.04    |         |
| 62          |          | -----   |         | -----   |         |
| 120         | D4530    | 0.40    |         | 0.04    |         |
| 131         | D4530    | 0.38    |         | -0.31   |         |
| 133         |          | -----   |         | -----   |         |
| 140         | D4530    | 0.35    |         | -0.84   |         |
| 150         | D4530    | 0.38    |         | -0.31   |         |
| 158         | D4530    | 0.2962  |         | -1.78   |         |
| 171         | D4530    | 0.30    |         | -1.72   |         |
| 228         |          | -----   |         | -----   |         |
| 311         | D4530    | 0.38    |         | -0.31   |         |
| 313         |          | -----   |         | -----   |         |
| 317         | D4530    | 0.405   |         | 0.13    |         |
| 323         | D4530    | 0.38    |         | -0.31   |         |
| 333         |          | -----   |         | -----   |         |
| 334         |          | -----   |         | -----   |         |
| 336         | D4530    | 0.27    |         | -2.24   |         |
| 337         |          | -----   |         | -----   |         |
| 340         |          | -----   |         | -----   |         |
| 356         | D4530    | 0.45    |         | 0.92    |         |
| 369         | D4530    | 0.45    |         | 0.92    |         |
| 402         | ISO10370 | 0.419   |         | 0.38    |         |
| 445         | D4530    | 0.42    |         | 0.40    |         |
| 447         | D4530    | 0.13    | R(0.01) | -4.71   |         |
| 463         | D4530    | 0.416   |         | 0.33    |         |
| 494         | D4530    | 0.414   |         | 0.29    |         |
| 621         |          | -----   |         | -----   |         |
| 657         | D4530    | 0.44    |         | 0.75    |         |
| 663         | D4530    | 0.379   |         | -0.33   |         |
| 704         | D4530    | 0.456   |         | 1.03    |         |
| 732         |          | -----   |         | -----   |         |
| 743         | D4530    | 0.51    |         | 1.98    |         |
| 781         |          | -----   |         | -----   |         |
| 784         |          | -----   |         | -----   |         |
| 785         | D4530    | 0.45    |         | 0.92    |         |
| 791         |          | -----   |         | -----   |         |
| 874         | D4530    | 0.45    |         | 0.92    |         |
| 875         | D4530    | 0.46    |         | 1.10    |         |
| 994         |          | -----   |         | -----   |         |
| 995         |          | -----   |         | -----   |         |
| 1065        | D4530    | 0.41    |         | 0.22    |         |
| 1134        | IP398    | 0.2616  |         | -2.39   |         |
| 1297        | D4530    | 0.460   |         | 1.10    |         |
| 1365        | D4530    | 0.45    |         | 0.92    |         |
| 1379        |          | -----   |         | -----   |         |
| 1397        | D4530    | 0.35    |         | -0.84   |         |
| 1455        | D4530    | 0.47    |         | 1.28    |         |
| 1510        |          | -----   |         | -----   |         |
| 1543        |          | -----   |         | -----   |         |
| 1556        | ISO10370 | 0.380   |         | -0.31   |         |
| 1635        | D4530    | 0.39    |         | -0.13   |         |
| 1677        | D4530    | 0.39    |         | -0.13   |         |
| 1857        | D4530    | 0.37    |         | -0.48   |         |
| 1881        | D4530    | 0.351   |         | -0.82   |         |
| 1949        | D4530    | 0.315   |         | -1.45   |         |
| 1986        | ISO10370 | 0.461   |         | 1.12    |         |
| 1995        |          | -----   |         | -----   |         |
| 6020        |          | -----   |         | -----   |         |
| 6021        | D4530    | 0.391   |         | -0.11   |         |
| <br>        |          |         |         |         |         |
| normality   |          |         |         |         |         |
| n           |          | OK      |         |         |         |
| outliers    |          | 38      |         |         |         |
| mean (n)    |          | 1       |         |         |         |
| st.dev. (n) |          | 0.3975  |         |         |         |
| R(calc.)    |          | 0.05727 |         |         |         |
| R(D4530:15) |          | 0.1603  |         |         |         |
|             |          | 0.1590  |         |         |         |



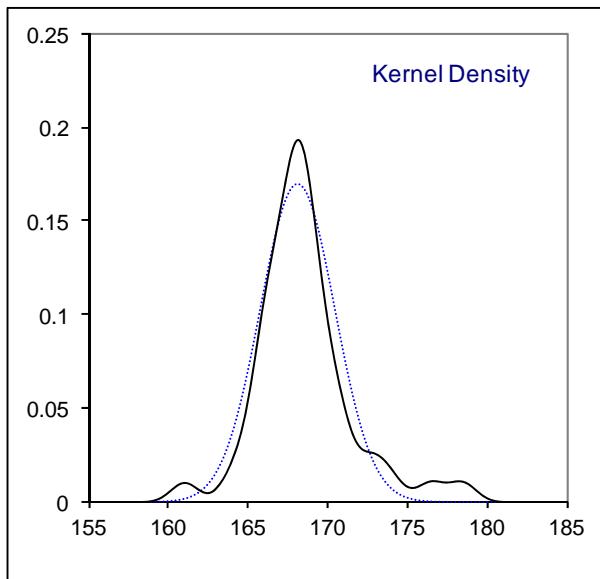
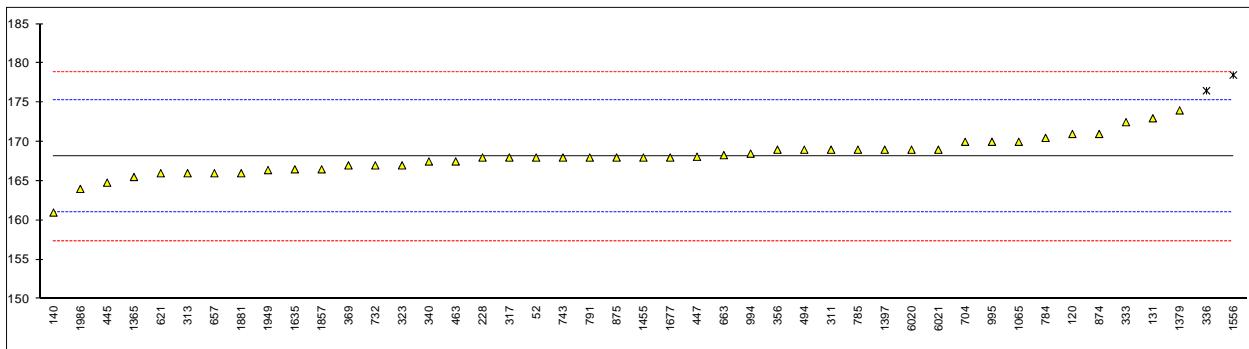
Determination of Density at 15°C on sample #15250; results in kg/m<sup>3</sup>

| lab            | method   | value  | mark    | z(targ) | remarks                                  |
|----------------|----------|--------|---------|---------|------------------------------------------|
| 52             | D4052    | 916.6  |         | 1.94    |                                          |
| 62             |          | -----  |         | -----   |                                          |
| 120            | D4052    | 915.9  |         | 0.63    |                                          |
| 131            | D4052    | 915.8  |         | 0.45    |                                          |
| 133            | D4052    | 916.2  |         | 1.19    |                                          |
| 140            | D4052    | 914.8  |         | -1.42   |                                          |
| 150            | D4052    | 916.0  |         | 0.82    |                                          |
| 158            | D1298    | 917.7  | R(0.05) | 3.99    |                                          |
| 171            | D4052    | 916    |         | 0.82    |                                          |
| 228            | D4052    | 914.6  |         | -1.79   |                                          |
| 311            | ISO12185 | 915.3  |         | -0.49   |                                          |
| 313            | ISO12185 | 915.4  |         | -0.30   |                                          |
| 317            | D4052    | 915.1  |         | -0.86   |                                          |
| 323            | ISO12185 | 915.0  |         | -1.05   |                                          |
| 333            | D4052    | 915.8  |         | 0.45    |                                          |
| 334            | D4052    | 916.0  |         | 0.82    |                                          |
| 336            | ISO12185 | 916.1  |         | 1.01    |                                          |
| 337            | ISO12185 | 915.8  |         | 0.45    |                                          |
| 340            | ISO12185 | 916.11 |         | 1.03    |                                          |
| 356            | D4052    | 915.4  |         | -0.30   |                                          |
| 369            | D4052    | 915.4  |         | -0.30   |                                          |
| 402            | ISO12185 | 914.01 |         | -2.89   |                                          |
| 445            | D4052    | 915.6  |         | 0.07    |                                          |
| 447            | IP365    | 915.3  | C       | -0.49   | Reported: 0.9153 kg/m <sup>3</sup>       |
| 463            | ISO12185 | 915.4  |         | -0.30   |                                          |
| 494            | D4052    | 916.5  |         | 1.75    |                                          |
| 621            | D4052    | 914.8  |         | -1.42   |                                          |
| 657            | D4052    | 915.2  |         | -0.67   |                                          |
| 663            | D4052    | 915.7  |         | 0.26    |                                          |
| 704            | ISO12185 | 915.3  |         | -0.49   |                                          |
| 732            | ISO12185 | 915.3  |         | -0.49   |                                          |
| 743            | D1298    | 915.9  |         | 0.63    |                                          |
| 781            |          | -----  |         | -----   |                                          |
| 784            | D1298    | 916.1  |         | 1.01    |                                          |
| 785            | D1298    | 916.1  |         | 1.01    |                                          |
| 791            | D1298    | 916.0  |         | 0.82    |                                          |
| 874            | ISO12185 | 916.0  | C       | 0.82    | First reported: 0.9160 kg/m <sup>3</sup> |
| 875            | D1298    | 916.0  |         | 0.82    |                                          |
| 994            | ISO12185 | 915.0  |         | -1.05   |                                          |
| 995            | D4052    | 915.0  |         | -1.05   |                                          |
| 1065           | D1298    | 916.3  |         | 1.38    |                                          |
| 1134           | IP365    | 891.0  | R(0.01) | -45.85  |                                          |
| 1297           | D4052    | 914.92 |         | -1.20   |                                          |
| 1365           | D4052    | 916.0  |         | 0.82    |                                          |
| 1379           | D4052    | 916.4  |         | 1.57    |                                          |
| 1397           | ISO12185 | 914.8  |         | -1.42   |                                          |
| 1455           | ISO12185 | 915.1  |         | -0.86   |                                          |
| 1510           |          | -----  |         | -----   |                                          |
| 1543           |          | -----  |         | -----   |                                          |
| 1556           | ISO12185 | 915.2  |         | -0.67   |                                          |
| 1635           | ISO12185 | 916.3  |         | 1.38    |                                          |
| 1677           | D4052    | 916.0  |         | 0.82    |                                          |
| 1857           | ISO12185 | 915.3  |         | -0.49   |                                          |
| 1881           | ISO12185 | 914.8  |         | -1.42   |                                          |
| 1949           | ISO12185 | 914.8  |         | -1.42   |                                          |
| 1986           | ISO3675  | 915.2  |         | -0.67   |                                          |
| 1995           |          | -----  |         | -----   |                                          |
| 6020           | ISO12185 | 915.8  |         | 0.45    |                                          |
| 6021           | ISO12185 | 915.7  |         | 0.26    |                                          |
| <br>           |          |        |         |         |                                          |
| normality      |          |        |         |         |                                          |
| n              |          |        |         |         |                                          |
| outliers       |          |        |         |         |                                          |
| mean (n)       |          |        |         |         |                                          |
| st.dev. (n)    |          |        |         |         |                                          |
| R(calc.)       |          |        |         |         |                                          |
| R(ISO12185:96) |          |        |         |         |                                          |



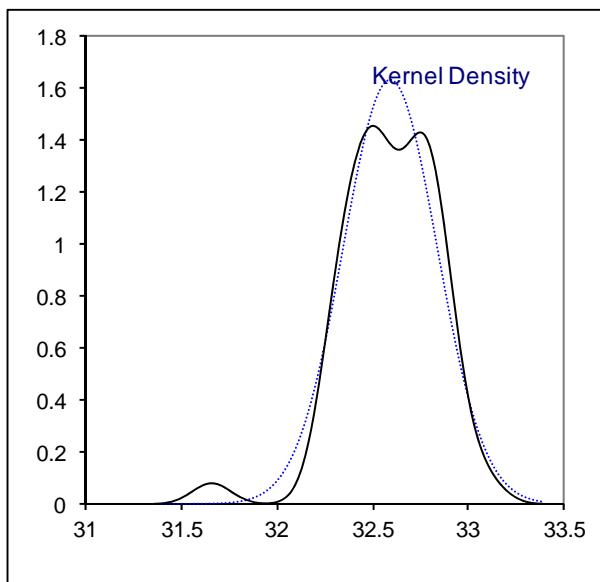
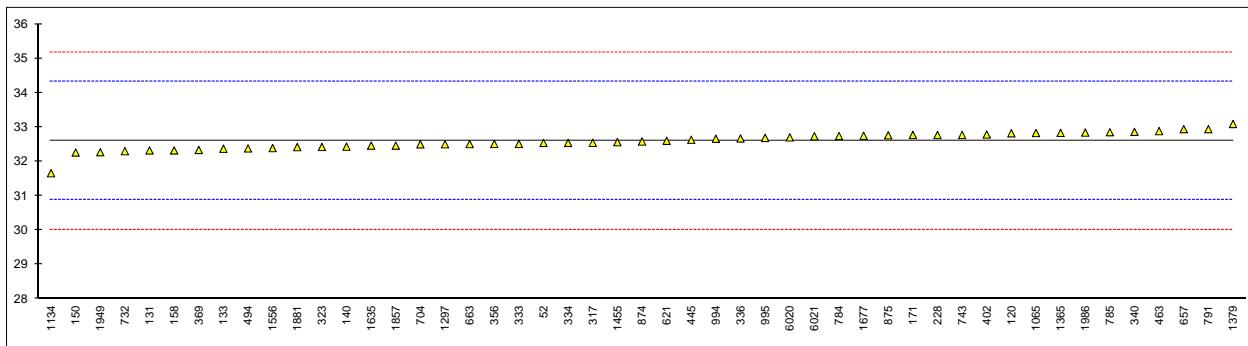
## Determination of Flash Point PMcc on sample #15250; results in °C

| lab          | method  | value   | mark    | z(targ) | remarks               |
|--------------|---------|---------|---------|---------|-----------------------|
| 52           | D93-A   | 168.0   |         | -0.03   |                       |
| 62           |         | -----   |         | -----   |                       |
| 120          | D93-B   | 171     |         | 0.81    |                       |
| 131          | D93-B   | 173     |         | 1.37    |                       |
| 133          |         | -----   |         | -----   |                       |
| 140          | D93-B   | 161.0   |         | -1.99   |                       |
| 150          | D93-B   | >110    |         | -----   |                       |
| 158          | D93-B   | >110    |         | -----   |                       |
| 171          |         | -----   |         | -----   |                       |
| 228          | D93-B   | 168.0   |         | -0.03   |                       |
| 311          | D93-B   | 169.0   |         | 0.25    |                       |
| 313          | D93-B   | 166.0   | C       | -0.59   | First reported: 156.0 |
| 317          | D93-B   | 168.0   |         | -0.03   |                       |
| 323          | D93-B   | 167.0   |         | -0.31   |                       |
| 333          | D93-B   | 172.5   |         | 1.23    |                       |
| 334          |         | -----   |         | -----   |                       |
| 336          | D93-B   | 176.5   | R(0.05) | 2.35    |                       |
| 337          |         | -----   |         | -----   |                       |
| 340          | D93-B   | 167.5   |         | -0.17   |                       |
| 356          | D93-B   | 169.0   |         | 0.25    |                       |
| 369          | D93-B   | 167.0   |         | -0.31   |                       |
| 402          |         | -----   |         | -----   |                       |
| 445          | D93-B   | 164.8   |         | -0.93   |                       |
| 447          | D93-B   | 168.1   |         | 0.00    |                       |
| 463          | D93-B   | 167.5   |         | -0.17   |                       |
| 494          | D93-A   | 169.0   |         | 0.25    |                       |
| 621          | D93-B   | 166.0   |         | -0.59   |                       |
| 657          | D93-B   | 166     |         | -0.59   |                       |
| 663          | D93-B   | 168.3   |         | 0.05    |                       |
| 704          | D93-B   | 170.0   |         | 0.53    |                       |
| 732          | D93-B   | 167.0   |         | -0.31   |                       |
| 743          | D93-B   | 168.0   |         | -0.03   |                       |
| 781          |         | -----   |         | -----   |                       |
| 784          | D93-B   | 170.5   |         | 0.67    |                       |
| 785          | D93-B   | 169.0   |         | 0.25    |                       |
| 791          | D93-B   | 168.0   |         | -0.03   |                       |
| 874          | D93-B   | 171.0   |         | 0.81    |                       |
| 875          | D93-B   | 168.0   |         | -0.03   |                       |
| 994          | D93-B   | 168.5   |         | 0.11    |                       |
| 995          | D93-B   | 170.0   |         | 0.53    |                       |
| 1065         | D93-B   | 170     |         | 0.53    |                       |
| 1134         |         | -----   |         | -----   |                       |
| 1297         |         | -----   |         | -----   |                       |
| 1365         | D93-B   | 165.52  |         | -0.72   |                       |
| 1379         | D93-B   | 174     |         | 1.65    |                       |
| 1397         | D93-A   | 169.0   |         | 0.25    |                       |
| 1455         | D93-A   | 168.0   |         | -0.03   |                       |
| 1510         |         | -----   |         | -----   |                       |
| 1543         |         | -----   |         | -----   |                       |
| 1556         | ISO2719 | 178.5   | R(0.05) | 2.91    |                       |
| 1635         | D93-B   | 166.5   |         | -0.45   |                       |
| 1677         | D93-B   | 168.0   |         | -0.03   |                       |
| 1857         | D93-B   | 166.5   |         | -0.45   |                       |
| 1881         | D93-B   | 166.0   |         | -0.59   |                       |
| 1949         | D93-B   | 166.4   |         | -0.48   |                       |
| 1986         | ISO2719 | 164.0   |         | -1.15   |                       |
| 1995         |         | -----   |         | -----   |                       |
| 6020         | D93-B   | 169.0   |         | 0.25    |                       |
| 6021         | D93-B   | 169.0   |         | 0.25    |                       |
| <br>         |         |         |         |         |                       |
| normality    |         |         |         |         |                       |
| n            |         | suspect |         |         |                       |
| outliers     |         | 43      |         |         |                       |
| mean (n)     |         | 2       |         |         |                       |
| st.dev. (n)  |         | 168.11  |         |         |                       |
| R(calc.)     |         | 2.352   |         |         |                       |
| R(D93-B:15a) |         | 6.59    |         |         |                       |
|              |         | 10.00   |         |         |                       |



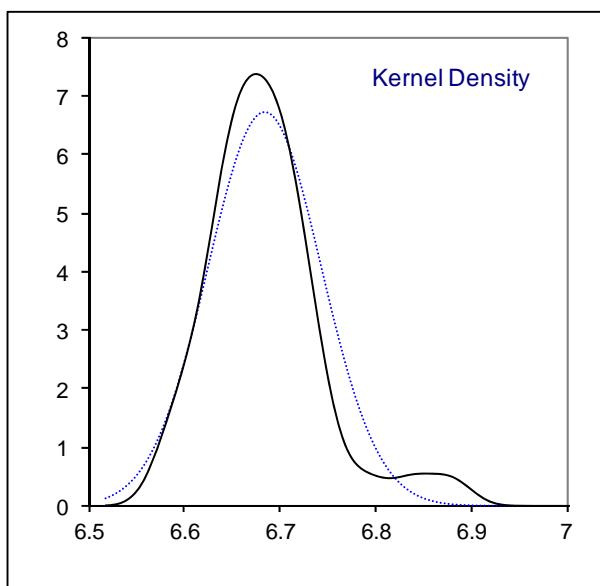
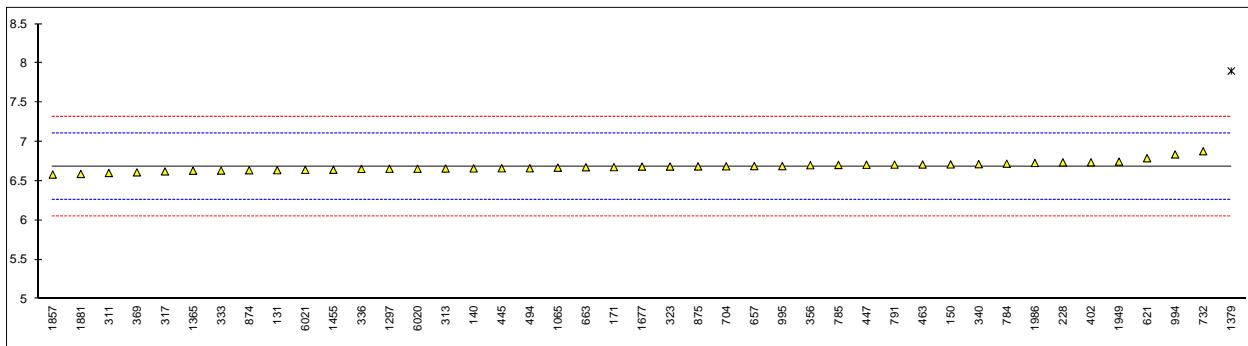
Determination of Kinematic Viscosity at 50°C on sample #15250; results in mm<sup>2</sup>/s

| lab         | method  | value      | mark | z(targ) | remarks |
|-------------|---------|------------|------|---------|---------|
| 52          | D445    | 32.54      |      | -0.06   |         |
| 62          |         | -----      |      | -----   |         |
| 120         | D445    | 32.82      |      | 0.27    |         |
| 131         | D445    | 32.32      |      | -0.31   |         |
| 133         | D445    | 32.37      |      | -0.26   |         |
| 140         | D445    | 32.43      |      | -0.19   |         |
| 150         | D445    | 32.26      |      | -0.38   |         |
| 158         | D445    | 32.3202    |      | -0.31   |         |
| 171         | D445    | 32.77      |      | 0.21    |         |
| 228         | D445    | 32.77      |      | 0.21    |         |
| 311         |         | -----      |      | -----   |         |
| 313         |         | -----      |      | -----   |         |
| 317         | D445    | 32.542     |      | -0.06   |         |
| 323         | D445    | 32.42      |      | -0.20   |         |
| 333         | ISO3104 | 32.51      |      | -0.09   |         |
| 334         | D445    | 32.54      |      | -0.06   |         |
| 336         | D445    | 32.67      |      | 0.09    |         |
| 337         |         | -----      |      | -----   |         |
| 340         | D445    | 32.860     |      | 0.31    |         |
| 356         | D445    | 32.51      |      | -0.09   |         |
| 369         | D445    | 32.334     |      | -0.30   |         |
| 402         |         | 32.785     |      | 0.22    |         |
| 445         | D445    | 32.63      |      | 0.05    |         |
| 447         |         | -----      |      | -----   |         |
| 463         | D7042   | 32.882     |      | 0.34    |         |
| 494         | D445    | 32.378     |      | -0.25   |         |
| 621         | D445    | 32.60      |      | 0.01    |         |
| 657         | D445    | 32.94      |      | 0.40    |         |
| 663         | D445    | 32.506     |      | -0.10   |         |
| 704         | D445    | 32.499     |      | -0.11   |         |
| 732         | D445    | 32.30      |      | -0.34   |         |
| 743         | D445    | 32.77      |      | 0.21    |         |
| 781         |         | -----      |      | -----   |         |
| 784         | D445    | 32.74      |      | 0.17    |         |
| 785         | D445    | 32.851     |      | 0.30    |         |
| 791         | D445    | 32.94      |      | 0.40    |         |
| 874         | D445    | 32.58      |      | -0.01   |         |
| 875         | D445    | 32.76      |      | 0.20    |         |
| 994         | D445    | 32.66      |      | 0.08    |         |
| 995         | D445    | 32.685     |      | 0.11    |         |
| 1065        | D445    | 32.825     |      | 0.27    |         |
| 1134        | D445    | 31.6572332 |      | -1.08   |         |
| 1297        | D7042   | 32.500     |      | -0.11   |         |
| 1365        | D445    | 32.83      |      | 0.28    |         |
| 1379        | D445    | 33.09      |      | 0.58    |         |
| 1397        |         | -----      |      | -----   |         |
| 1455        | D445    | 32.56      |      | -0.04   |         |
| 1510        |         | -----      |      | -----   |         |
| 1543        |         | -----      |      | -----   |         |
| 1556        | ISO3104 | 32.388     |      | -0.24   |         |
| 1635        | ISO3104 | 32.46      |      | -0.15   |         |
| 1677        | D445    | 32.747     |      | 0.18    |         |
| 1857        | D445    | 32.46      |      | -0.15   |         |
| 1881        | D445    | 32.417     |      | -0.20   |         |
| 1949        | D445    | 32.269     |      | -0.37   |         |
| 1986        | D445    | 32.84      |      | 0.29    |         |
| 1995        |         | -----      |      | -----   |         |
| 6020        | D445    | 32.70      |      | 0.13    |         |
| 6021        | D445    | 32.734     |      | 0.17    |         |
| <br>        |         |            |      |         |         |
| normality   |         |            |      |         |         |
| n           |         |            |      |         |         |
| outliers    |         |            |      |         |         |
| mean (n)    |         |            |      |         |         |
| st.dev. (n) |         |            |      |         |         |
| R(calc.)    |         |            |      |         |         |
| R(D445:15a) |         |            |      |         |         |



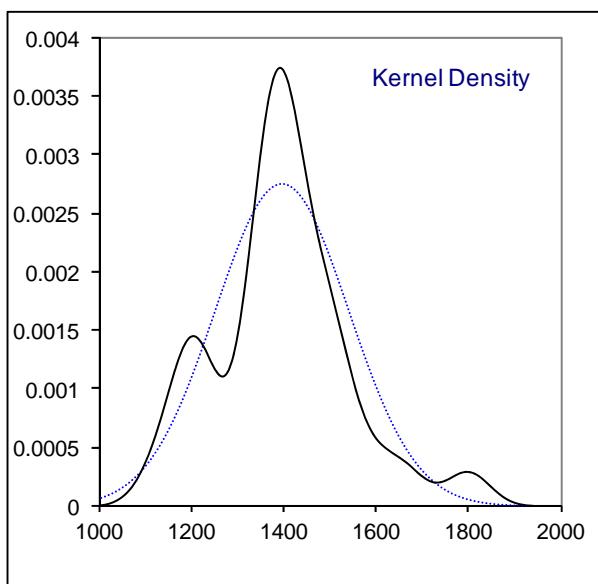
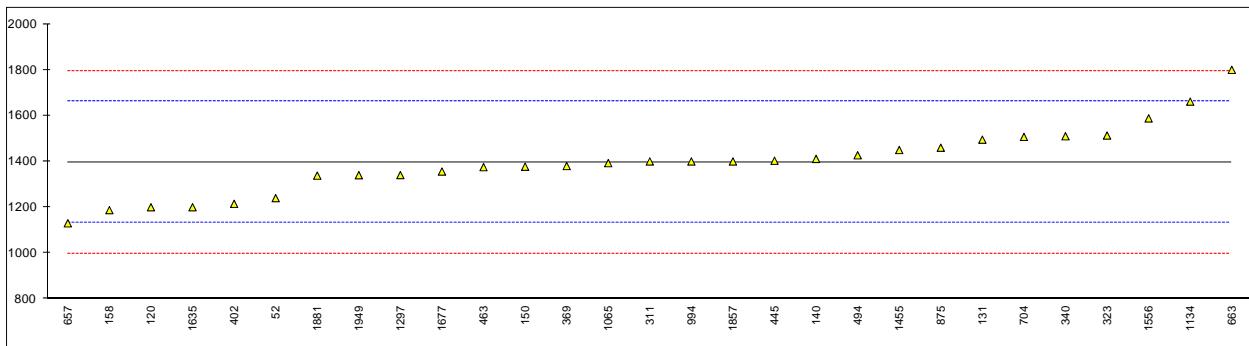
Determination of Kinematic Viscosity at 100°C on sample #15250; results in mm<sup>2</sup>/s

| lab         | method  | value   | mark    | z(targ) | remarks               |
|-------------|---------|---------|---------|---------|-----------------------|
| 52          |         | ----    |         | ----    |                       |
| 62          |         | ----    |         | ----    |                       |
| 120         |         | ----    |         | ----    |                       |
| 131         | D445    | 6.639   |         | -0.22   |                       |
| 133         |         | ----    |         | ----    |                       |
| 140         | D445    | 6.661   |         | -0.11   |                       |
| 150         | D445    | 6.712   |         | 0.13    |                       |
| 158         |         | ----    |         | ----    |                       |
| 171         | D445    | 6.678   |         | -0.03   |                       |
| 228         | D445    | 6.738   |         | 0.25    |                       |
| 311         | D445    | 6.603   |         | -0.39   |                       |
| 313         | D445    | 6.660   |         | -0.12   |                       |
| 317         | D445    | 6.6231  |         | -0.29   |                       |
| 323         | D445    | 6.685   |         | 0.00    |                       |
| 333         | ISO3104 | 6.634   |         | -0.24   |                       |
| 334         |         | ----    |         | ----    |                       |
| 336         | D445    | 6.655   |         | -0.14   |                       |
| 337         |         | ----    |         | ----    |                       |
| 340         | D445    | 6.7156  |         | 0.15    |                       |
| 356         | D445    | 6.702   |         | 0.08    |                       |
| 369         | D445    | 6.611   |         | -0.35   |                       |
| 402         | D7042   | 6.7395  |         | 0.26    |                       |
| 445         | D445    | 6.663   |         | -0.10   |                       |
| 447         | D445    | 6.708   |         | 0.11    |                       |
| 463         | D7042   | 6.7110  |         | 0.13    |                       |
| 494         | D445    | 6.663   |         | -0.10   |                       |
| 621         | D445    | 6.791   |         | 0.51    |                       |
| 657         | D445    | 6.691   |         | 0.03    |                       |
| 663         | D445    | 6.6766  |         | -0.04   |                       |
| 704         | D445    | 6.688   |         | 0.02    |                       |
| 732         | D445    | 6.88    |         | 0.93    |                       |
| 743         |         | ----    |         | ----    |                       |
| 781         |         | ----    |         | ----    |                       |
| 784         | D445    | 6.721   |         | 0.17    |                       |
| 785         | D445    | 6.7053  |         | 0.10    |                       |
| 791         | D445    | 6.71    |         | 0.12    |                       |
| 874         | D445    | 6.638   |         | -0.22   |                       |
| 875         | D445    | 6.687   |         | 0.01    |                       |
| 994         | D445    | 6.839   |         | 0.74    |                       |
| 995         | D445    | 6.691   |         | 0.03    |                       |
| 1065        | D445    | 6.67085 |         | -0.07   |                       |
| 1134        |         | ----    |         | ----    |                       |
| 1297        | D7042   | 6.6568  |         | -0.13   |                       |
| 1365        | D445    | 6.632   |         | -0.25   |                       |
| 1379        | D445    | 7.901   | R(0.01) | 5.80    |                       |
| 1397        |         | ----    |         | ----    |                       |
| 1455        | D445    | 6.644   |         | -0.19   |                       |
| 1510        |         | ----    |         | ----    |                       |
| 1543        |         | ----    |         | ----    |                       |
| 1556        |         | ----    | W       | ----    | First reported: 5.001 |
| 1635        |         | ----    |         | ----    |                       |
| 1677        | D445    | 6.6842  |         | 0.00    |                       |
| 1857        | D445    | 6.583   |         | -0.48   |                       |
| 1881        | D445    | 6.5912  |         | -0.45   |                       |
| 1949        | D445    | 6.7479  |         | 0.30    |                       |
| 1986        | D445    | 6.730   |         | 0.22    |                       |
| 1995        |         | ----    |         | ----    |                       |
| 6020        | D445    | 6.657   |         | -0.13   |                       |
| 6021        | D445    | 6.6432  |         | -0.20   |                       |
| <br>        |         |         |         |         |                       |
| normality   |         |         |         |         |                       |
| n           |         | not OK  |         |         |                       |
| outliers    |         | 42      |         |         |                       |
| mean (n)    |         | 1       |         |         |                       |
| st.dev. (n) |         | 6.685   |         |         |                       |
| R(calc.)    |         | 0.0592  |         |         |                       |
| R(D445:15a) |         | 0.166   |         |         |                       |
|             |         | 0.587   |         |         |                       |



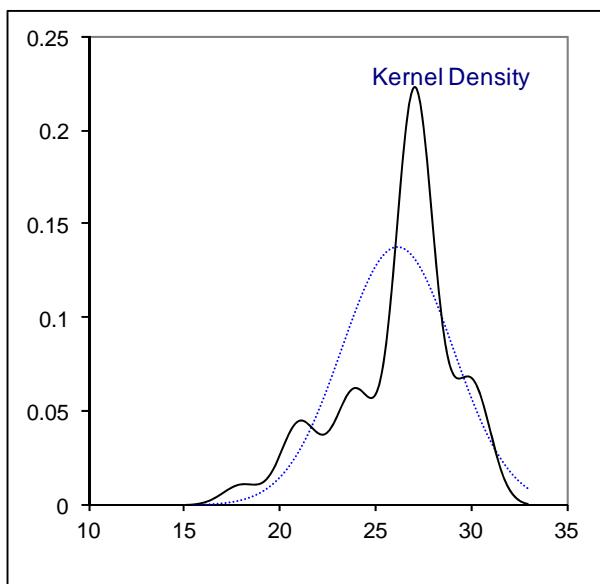
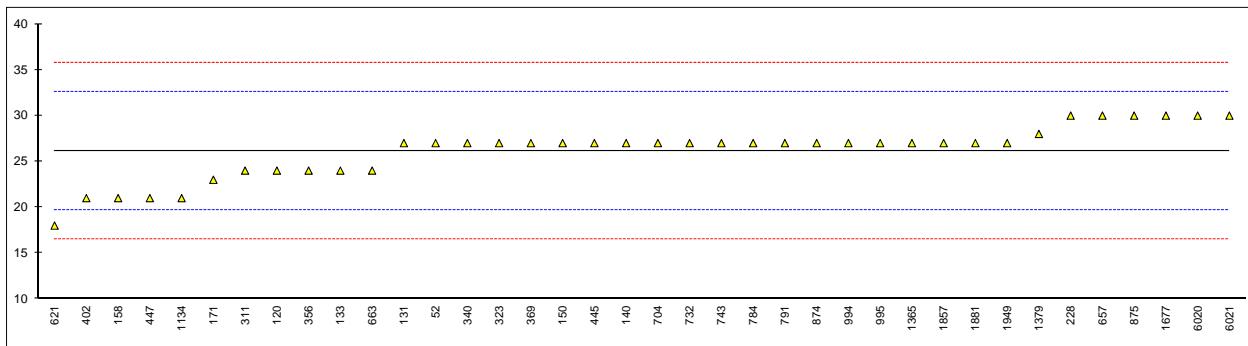
## Determination of Nitrogen on sample #15250; results in mg/kg

| lab         | method            | value    | mark | z(targ) | remarks                 |
|-------------|-------------------|----------|------|---------|-------------------------|
| 52          | D5762             | 1240     |      | -1.18   |                         |
| 62          |                   | ----     |      | ----    |                         |
| 120         | D5762 Gravimetric | 1200     | C    | -1.48   | First reported: 2500    |
| 131         | D5762 Gravimetric | 1495     |      | 0.74    |                         |
| 133         |                   | ----     |      | ----    |                         |
| 140         | D5762 Gravimetric | 1411     |      | 0.11    |                         |
| 150         | D5762 Volumetric  | 1377     |      | -0.15   |                         |
| 158         | D5762 Gravimetric | 1187.5   |      | -1.57   |                         |
| 171         |                   | ----     |      | ----    |                         |
| 228         |                   | ----     |      | ----    |                         |
| 311         | D5762 Volumetric  | 1400     |      | 0.03    |                         |
| 313         |                   | ----     |      | ----    |                         |
| 317         |                   | ----     |      | ----    |                         |
| 323         | D5762 Gravimetric | 1513     |      | 0.88    |                         |
| 333         |                   | ----     |      | ----    |                         |
| 334         |                   | ----     |      | ----    |                         |
| 336         |                   | ----     |      | ----    |                         |
| 337         |                   | ----     |      | ----    |                         |
| 340         | D5762 Gravimetric | 1510     |      | 0.86    |                         |
| 356         |                   | ----     |      | ----    |                         |
| 369         | D3228             | 1380     |      | -0.12   |                         |
| 402         | D5762 Volumetric  | 1215     |      | -1.37   |                         |
| 445         | D5762 Gravimetric | 1403     | C    | 0.05    | Reported: 1403 %M/M     |
| 447         |                   | ----     |      | ----    |                         |
| 463         | D5762 Gravimetric | 1375     |      | -0.16   |                         |
| 494         | D5762 Gravimetric | 1427     |      | 0.23    |                         |
| 621         |                   | ----     |      | ----    |                         |
| 657         | D5762 Gravimetric | 1130     | C    | -2.01   | Reported: 1130 %M/M     |
| 663         | D5762 Gravimetric | 1800     |      | 3.04    |                         |
| 704         | D5762 Volumetric  | 1507     |      | 0.83    |                         |
| 732         |                   | ----     |      | ----    |                         |
| 743         |                   | ----     |      | ----    |                         |
| 781         |                   | ----     |      | ----    |                         |
| 784         |                   | ----     |      | ----    |                         |
| 785         |                   | ----     |      | ----    |                         |
| 791         |                   | ----     |      | ----    |                         |
| 874         |                   | ----     |      | ----    |                         |
| 875         | D5762 Gravimetric | 1460     |      | 0.48    |                         |
| 994         | D5762 Volumetric  | 1400     |      | 0.03    |                         |
| 995         |                   | ----     |      | ----    |                         |
| 1065        |                   | 1392.46  | C    | -0.03   | First reported: 2104.83 |
| 1134        | D5762 Volumetric  | 1661.236 |      | 2.00    |                         |
| 1297        | D4629             | 1340.5   |      | -0.42   |                         |
| 1365        |                   | ----     |      | ----    |                         |
| 1379        |                   | ----     |      | ----    |                         |
| 1397        |                   | ----     |      | ----    |                         |
| 1455        | D5762 Gravimetric | 1450     |      | 0.40    |                         |
| 1510        |                   | ----     |      | ----    |                         |
| 1543        |                   | ----     |      | ----    |                         |
| 1556        | D5762 Gravimetric | 1588     |      | 1.44    |                         |
| 1635        | D5762 Gravimetric | 1200     |      | -1.48   |                         |
| 1677        | D5762 Gravimetric | 1356     |      | -0.30   |                         |
| 1857        | D5762 Volumetric  | 1400     |      | 0.03    |                         |
| 1881        | D5762 Volumetric  | 1338     |      | -0.44   |                         |
| 1949        | D5762             | 1340     |      | -0.43   |                         |
| 1986        |                   | ----     |      | ----    |                         |
| 1995        |                   | ----     |      | ----    |                         |
| 6020        |                   | ----     |      | ----    |                         |
| 6021        |                   | ----     |      | ----    |                         |
| <br>        |                   |          |      |         |                         |
| normality   |                   |          |      |         |                         |
| n           |                   | suspect  |      |         |                         |
|             |                   | 29       |      |         |                         |
| outliers    |                   | 0        |      |         |                         |
| mean (n)    |                   | 1396.44  |      |         |                         |
| st.dev. (n) |                   | 144.906  |      |         |                         |
| R(calc.)    |                   | 405.74   |      |         |                         |
| R(D5762:12) |                   | 371.45   |      |         |                         |



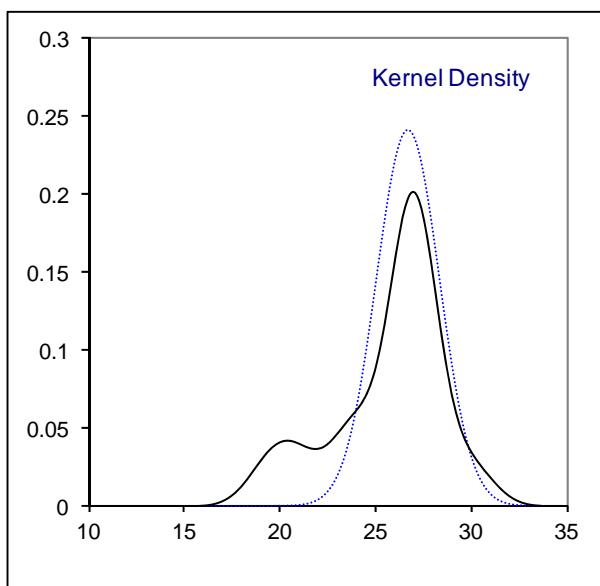
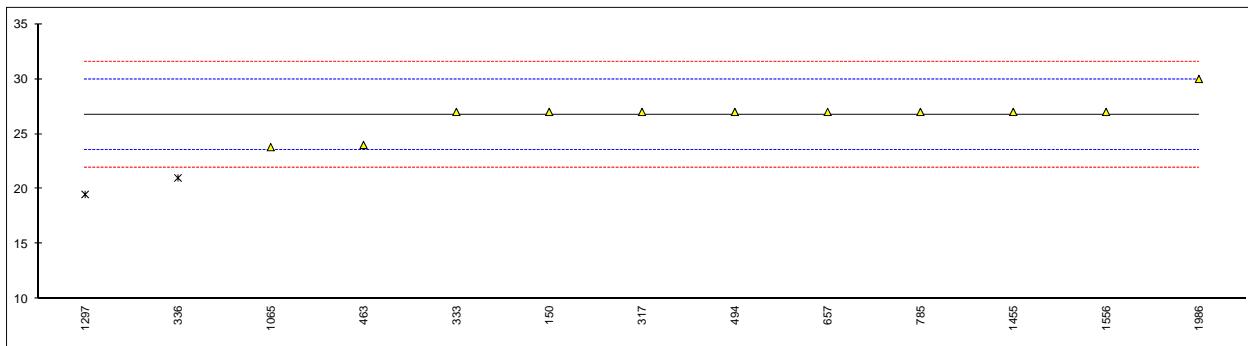
## Determination of Pour Point, manual on sample #15250; results in °C

| lab         | method | value | mark | z(targ) | remarks            |
|-------------|--------|-------|------|---------|--------------------|
| 52          | D97    | 27    |      | 0.27    |                    |
| 62          |        | ----  |      | ----    |                    |
| 120         | D97    | 24    |      | -0.66   |                    |
| 131         | D97    | 27    |      | 0.27    |                    |
| 133         | D97    | 24    |      | -0.66   |                    |
| 140         | D97    | 27    |      | 0.27    |                    |
| 150         | D97    | 27    |      | 0.27    |                    |
| 158         | D97    | 21    |      | -1.60   |                    |
| 171         | D97    | 23    |      | -0.97   |                    |
| 228         | D97    | 30    |      | 1.20    |                    |
| 311         | D97    | 24    |      | -0.66   |                    |
| 313         |        | ----  |      | ----    |                    |
| 317         |        | ----  |      | ----    |                    |
| 323         | D97    | 27    |      | 0.27    |                    |
| 333         |        | ----  |      | ----    |                    |
| 334         |        | ----  |      | ----    |                    |
| 336         |        | ----  |      | ----    |                    |
| 337         |        | ----  |      | ----    |                    |
| 340         | D97    | 27    |      | 0.27    |                    |
| 356         | D97    | 24    |      | -0.66   |                    |
| 369         | D97    | 27    |      | 0.27    |                    |
| 402         | D97    | 21    |      | -1.60   |                    |
| 445         | D97    | 27    |      | 0.27    |                    |
| 447         | D97    | 21    |      | -1.60   |                    |
| 463         |        | ----  |      | ----    |                    |
| 494         |        | ----  |      | ----    |                    |
| 621         | D97    | 18    | C    | -2.53   | First reported: 15 |
| 657         | D97    | 30    |      | 1.20    |                    |
| 663         | D97    | 24    |      | -0.66   |                    |
| 704         | D97    | 27    |      | 0.27    |                    |
| 732         | D97    | 27    |      | 0.27    |                    |
| 743         | D97    | 27    |      | 0.27    |                    |
| 781         |        | ----  |      | ----    |                    |
| 784         | D97    | 27    |      | 0.27    |                    |
| 785         |        | ----  |      | ----    |                    |
| 791         | D97    | 27    |      | 0.27    |                    |
| 874         | D97    | 27    |      | 0.27    |                    |
| 875         | D97    | 30    |      | 1.20    |                    |
| 994         | D97    | 27    |      | 0.27    |                    |
| 995         | D97    | 27    |      | 0.27    |                    |
| 1065        |        | ----  |      | ----    |                    |
| 1134        | IP15   | 21    |      | -1.60   |                    |
| 1297        |        | ----  |      | ----    |                    |
| 1365        | D97    | 27    |      | 0.27    |                    |
| 1379        | D97    | 28    |      | 0.58    |                    |
| 1397        |        | ----  |      | ----    |                    |
| 1455        |        | ----  |      | ----    |                    |
| 1510        |        | ----  |      | ----    |                    |
| 1543        |        | ----  |      | ----    |                    |
| 1556        |        | ----  |      | ----    |                    |
| 1635        |        | ----  |      | ----    |                    |
| 1677        | D97    | 30    |      | 1.20    |                    |
| 1857        | D97    | 27    |      | 0.27    |                    |
| 1881        | D97    | 27    |      | 0.27    |                    |
| 1949        | D97    | 27    |      | 0.27    |                    |
| 1986        |        | ----  |      | ----    |                    |
| 1995        |        | ----  |      | ----    |                    |
| 6020        | D97    | 30    |      | 1.20    |                    |
| 6021        | D97    | 30    |      | 1.20    |                    |
| <br>        |        |       |      |         |                    |
| normality   |        |       |      |         |                    |
| n           |        | OK    |      |         |                    |
| outliers    |        | 38    |      |         |                    |
| mean (n)    |        | 0     |      |         |                    |
| st.dev. (n) |        | 26.13 |      |         |                    |
| R(calc.)    |        | 2.905 |      |         |                    |
| R(D97:15)   |        | 8.14  |      |         |                    |
|             |        | 9.00  |      |         |                    |



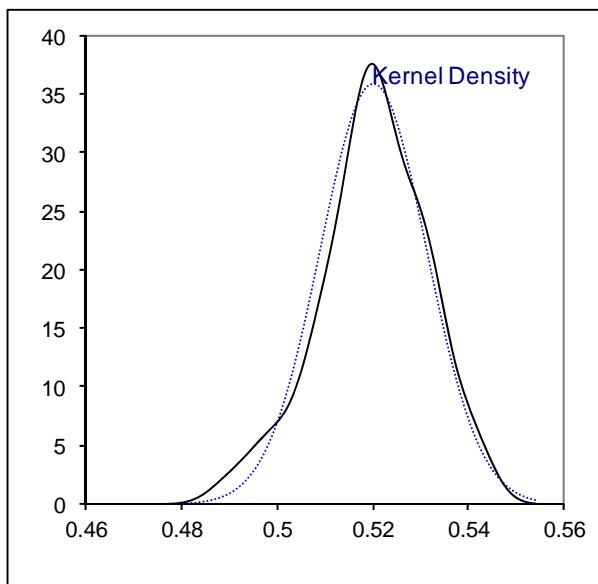
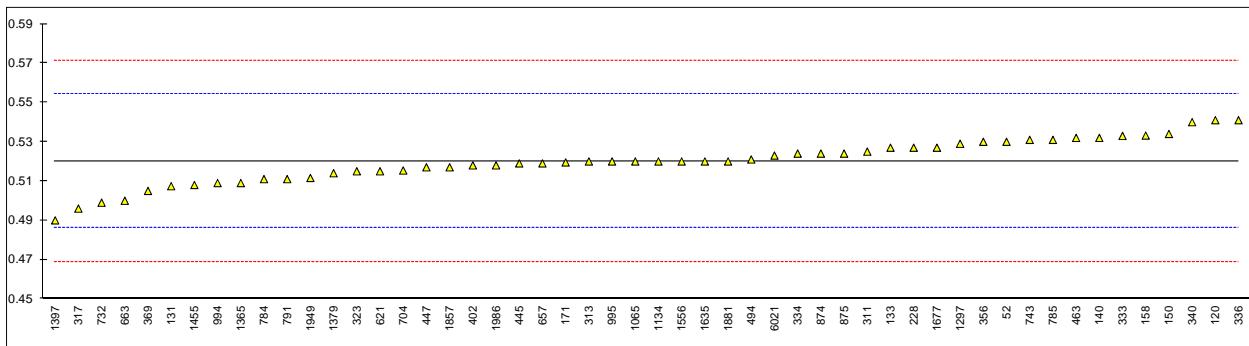
## Determination of Pour Point, automated, 3°C interval on sample #15250; results in °C

| lab         | method  | value   | mark     | z(targ) | remarks |
|-------------|---------|---------|----------|---------|---------|
| 52          |         | ----    |          | ----    |         |
| 62          |         | ----    |          | ----    |         |
| 120         |         | ----    |          | ----    |         |
| 131         |         | ----    |          | ----    |         |
| 133         |         | ----    |          | ----    |         |
| 140         |         | ----    |          | ----    |         |
| 150         | D5950   | 27      |          | 0.18    |         |
| 158         |         | ----    |          | ----    |         |
| 171         |         | ----    |          | ----    |         |
| 228         |         | ----    |          | ----    |         |
| 311         |         | ----    |          | ----    |         |
| 313         |         | ----    |          | ----    |         |
| 317         | D6749   | 27      |          | 0.18    |         |
| 323         |         | ----    |          | ----    |         |
| 333         | D5950   | 27      |          | 0.18    |         |
| 334         |         | ----    |          | ----    |         |
| 336         | D5950   | 21      | DG(0.05) | -3.55   |         |
| 337         |         | ----    |          | ----    |         |
| 340         |         | ----    |          | ----    |         |
| 356         |         | ----    |          | ----    |         |
| 369         |         | ----    |          | ----    |         |
| 402         |         | ----    |          | ----    |         |
| 445         |         | ----    |          | ----    |         |
| 447         |         | ----    |          | ----    |         |
| 463         | D6892   | 24      |          | -1.69   |         |
| 494         | D5950   | 27      |          | 0.18    |         |
| 621         |         | ----    |          | ----    |         |
| 657         | D5950   | 27      |          | 0.18    |         |
| 663         |         | ----    |          | ----    |         |
| 704         |         | ----    |          | ----    |         |
| 732         |         | ----    |          | ----    |         |
| 743         |         | ----    |          | ----    |         |
| 781         |         | ----    |          | ----    |         |
| 784         |         | ----    |          | ----    |         |
| 785         | D6749   | 27      |          | 0.18    |         |
| 791         |         | ----    |          | ----    |         |
| 874         |         | ----    |          | ----    |         |
| 875         |         | ----    |          | ----    |         |
| 994         |         | ----    |          | ----    |         |
| 995         |         | ----    |          | ----    |         |
| 1065        | D5950   | 23.8    |          | -1.81   |         |
| 1134        |         | ----    |          | ----    |         |
| 1297        | D5950   | 19.5    | DG(0.05) | -4.49   |         |
| 1365        |         | ----    |          | ----    |         |
| 1379        |         | ----    |          | ----    |         |
| 1397        |         | ----    |          | ----    |         |
| 1455        | D5950   | 27      |          | 0.18    |         |
| 1510        |         | ----    |          | ----    |         |
| 1543        |         | ----    |          | ----    |         |
| 1556        | ISO3016 | 27      |          | 0.18    |         |
| 1635        |         | ----    |          | ----    |         |
| 1677        |         | ----    |          | ----    |         |
| 1857        |         | ----    |          | ----    |         |
| 1881        |         | ----    |          | ----    |         |
| 1949        |         | ----    |          | ----    |         |
| 1986        | ISO3016 | 30      |          | 2.05    |         |
| 1995        |         | ----    |          | ----    |         |
| 6020        |         | ----    |          | ----    |         |
| 6021        |         | ----    |          | ----    |         |
|             |         |         |          |         |         |
| normality   |         | suspect |          |         |         |
| n           |         | 11      |          |         |         |
| outliers    |         | 2       |          |         |         |
| mean (n)    |         | 26.71   |          |         |         |
| st.dev. (n) |         | 1.653   |          |         |         |
| R(calc.)    |         | 4.63    |          |         |         |
| R(D5950:14) |         | 4.50    |          |         |         |



## Determination of Total Sulphur on sample #15250; results in %M/M

| lab         | method  | value   | mark | z(targ) | remarks |
|-------------|---------|---------|------|---------|---------|
| 52          | D4294   | 0.53    |      | 0.58    |         |
| 62          |         | -----   |      | -----   |         |
| 120         | D4294   | 0.541   |      | 1.23    |         |
| 131         | D4294   | 0.5074  |      | -0.74   |         |
| 133         | D4294   | 0.527   |      | 0.41    |         |
| 140         | D4294   | 0.532   |      | 0.70    |         |
| 150         | D4294   | 0.534   |      | 0.82    |         |
| 158         | D4294   | 0.5332  |      | 0.77    |         |
| 171         | D4294   | 0.5195  |      | -0.03   |         |
| 228         | D4294   | 0.527   |      | 0.41    |         |
| 311         | D4294   | 0.525   |      | 0.29    |         |
| 313         | D4294   | 0.52    |      | 0.00    |         |
| 317         | D2622   | 0.496   |      | -1.41   |         |
| 323         | D4294   | 0.515   |      | -0.30   |         |
| 333         | D4294   | 0.533   |      | 0.76    |         |
| 334         | D4294   | 0.524   |      | 0.23    |         |
| 336         | D4294   | 0.541   |      | 1.23    |         |
| 337         |         | -----   |      | -----   |         |
| 340         | D4294   | 0.540   |      | 1.17    |         |
| 356         | D4294   | 0.530   |      | 0.58    |         |
| 369         | D2622   | 0.505   |      | -0.88   |         |
| 402         | D4294   | 0.518   |      | -0.12   |         |
| 445         | IP336   | 0.519   |      | -0.06   |         |
| 447         | IP336   | 0.517   |      | -0.18   |         |
| 463         | D4294   | 0.532   |      | 0.70    |         |
| 494         | D4294   | 0.521   |      | 0.06    |         |
| 621         | D4294   | 0.515   |      | -0.30   |         |
| 657         | D4294   | 0.519   |      | -0.06   |         |
| 663         | D4294   | 0.500   |      | -1.18   |         |
| 704         | D4294   | 0.5154  |      | -0.27   |         |
| 732         | D4294   | 0.499   |      | -1.24   |         |
| 743         | D4294   | 0.531   |      | 0.64    |         |
| 781         |         | -----   |      | -----   |         |
| 784         | D4294   | 0.511   |      | -0.53   |         |
| 785         | D4294   | 0.531   |      | 0.64    |         |
| 791         | D4294   | 0.511   |      | -0.53   |         |
| 874         | D4294   | 0.524   |      | 0.23    |         |
| 875         | D4294   | 0.524   |      | 0.23    |         |
| 994         | D4294   | 0.509   |      | -0.65   |         |
| 995         | D4294   | 0.520   |      | 0.00    |         |
| 1065        | D4294   | 0.52    |      | 0.00    |         |
| 1134        | IP336   | 0.52    |      | 0.00    |         |
| 1297        | D4294   | 0.5290  |      | 0.53    |         |
| 1365        | D4294   | 0.509   |      | -0.65   |         |
| 1379        | D4294   | 0.514   |      | -0.36   |         |
| 1397        | D2622   | 0.49    |      | -1.77   |         |
| 1455        | D2622   | 0.508   |      | -0.71   |         |
| 1510        |         | -----   |      | -----   |         |
| 1543        |         | -----   |      | -----   |         |
| 1556        | ISO8754 | 0.520   |      | 0.00    |         |
| 1635        | D4294   | 0.52    |      | 0.00    |         |
| 1677        | D4294   | 0.527   |      | 0.41    |         |
| 1857        | D4294   | 0.517   |      | -0.18   |         |
| 1881        | D4294   | 0.520   |      | 0.00    |         |
| 1949        | D4294   | 0.5116  |      | -0.50   |         |
| 1986        | D4294   | 0.518   |      | -0.12   |         |
| 1995        |         | -----   |      | -----   |         |
| 6020        |         | -----   |      | -----   |         |
| 6021        | D4294   | 0.5230  |      | 0.17    |         |
| <br>        |         |         |      |         |         |
| normality   |         |         |      |         |         |
| n           |         | OK      |      |         |         |
| outliers    |         | 52      |      |         |         |
| mean (n)    |         | 0       |      |         |         |
| st.dev. (n) |         | 0.5201  |      |         |         |
| R(calc.)    |         | 0.01113 |      |         |         |
| R(D4294:10) |         | 0.0312  |      |         |         |
|             |         | 0.0477  |      |         |         |



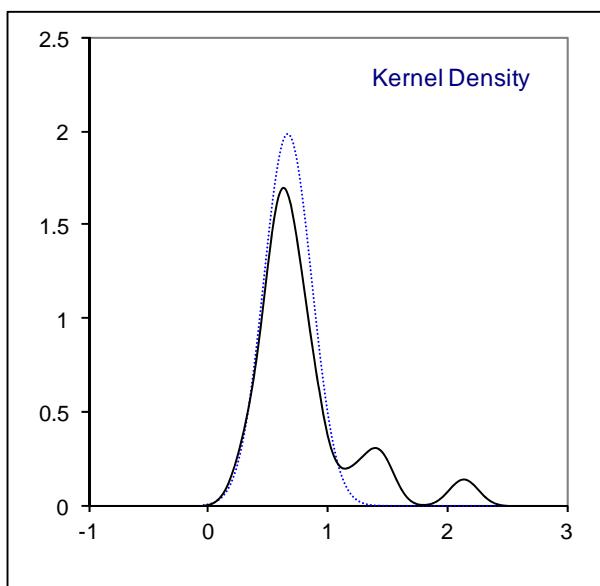
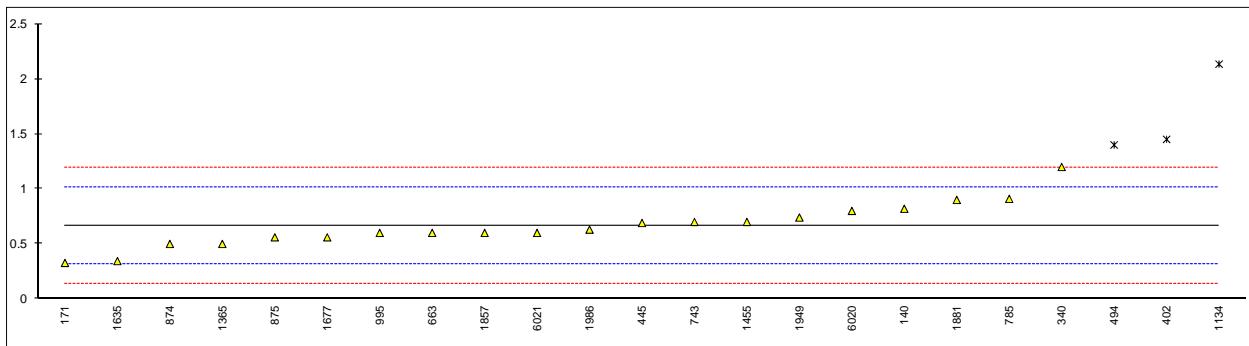
## Determination of Arsenic on sample #15250; results in mg/kg

| lab       | method   | value | mark | z(targ) | remarks |
|-----------|----------|-------|------|---------|---------|
| 52        |          | ----  |      | ----    |         |
| 62        |          | ----  |      | ----    |         |
| 120       | IP501    | <1    |      | ----    |         |
| 131       |          | ----  |      | ----    |         |
| 133       |          | ----  |      | ----    |         |
| 140       |          | ----  |      | ----    |         |
| 150       | D5708    | <1    |      | ----    |         |
| 158       |          | ----  |      | ----    |         |
| 171       |          | ----  |      | ----    |         |
| 228       |          | ----  |      | ----    |         |
| 311       | UOP986   | <1    |      | ----    |         |
| 313       |          | ----  |      | ----    |         |
| 317       |          | ----  |      | ----    |         |
| 323       | in house | <50   |      | ----    |         |
| 333       |          | ----  |      | ----    |         |
| 334       |          | ----  |      | ----    |         |
| 336       |          | ----  |      | ----    |         |
| 337       |          | ----  |      | ----    |         |
| 340       |          | ----  |      | ----    |         |
| 356       |          | ----  |      | ----    |         |
| 369       |          | ----  |      | ----    |         |
| 402       |          | ----  |      | ----    |         |
| 445       |          | ----  |      | ----    |         |
| 447       |          | ----  |      | ----    |         |
| 463       |          | ----  |      | ----    |         |
| 494       |          | ----  |      | ----    |         |
| 621       |          | ----  |      | ----    |         |
| 657       |          | ----  |      | ----    |         |
| 663       |          | ----  |      | ----    |         |
| 704       |          | ----  |      | ----    |         |
| 732       |          | ----  |      | ----    |         |
| 743       |          | ----  |      | ----    |         |
| 781       |          | ----  |      | ----    |         |
| 784       |          | ----  |      | ----    |         |
| 785       |          | ----  |      | ----    |         |
| 791       |          | ----  |      | ----    |         |
| 874       |          | ----  |      | ----    |         |
| 875       |          | ----  |      | ----    |         |
| 994       |          | ----  |      | ----    |         |
| 995       |          | ----  |      | ----    |         |
| 1065      |          | ----  |      | ----    |         |
| 1134      | E885     | 0.009 |      | ----    |         |
| 1297      | in house | 0.027 |      | ----    |         |
| 1365      |          | ----  |      | ----    |         |
| 1379      |          | ----  |      | ----    |         |
| 1397      |          | ----  |      | ----    |         |
| 1455      |          | ----  |      | ----    |         |
| 1510      |          | ----  |      | ----    |         |
| 1543      |          | ----  |      | ----    |         |
| 1556      |          | ----  |      | ----    |         |
| 1635      | D5185    | <2    |      | ----    |         |
| 1677      | IP PM CW | < 0.1 |      | ----    |         |
| 1857      |          | ----  |      | ----    |         |
| 1881      |          | ----  |      | ----    |         |
| 1949      |          | ----  |      | ----    |         |
| 1986      |          | ----  |      | ----    |         |
| 1995      |          | ----  |      | ----    |         |
| 6020      |          | ----  |      | ----    |         |
| 6021      |          | ----  |      | ----    |         |
| normality |          | n.a.  |      |         |         |
| n         |          | 6     |      |         |         |
| outliers  |          | 0     |      |         |         |
| mean (n)  |          | <1    |      |         |         |

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## Determination of Calcium on sample #15250; results in mg/kg

| lab         | method     | value   | mark    | z(targ) | remarks                                     |
|-------------|------------|---------|---------|---------|---------------------------------------------|
| 52          | IP470      | <1      |         | ----    |                                             |
| 62          |            | ----    |         | ----    |                                             |
| 120         |            | ----    |         | ----    |                                             |
| 131         |            | ----    |         | ----    |                                             |
| 133         |            | ----    |         | ----    |                                             |
| 140         | D5708      | 0.82    |         | 0.88    |                                             |
| 150         | D5708      | <1      |         | ----    |                                             |
| 158         |            | ----    |         | ----    |                                             |
| 171         | D5708      | 0.327   |         | -1.91   |                                             |
| 228         |            | ----    |         | ----    |                                             |
| 311         |            | ----    |         | ----    |                                             |
| 313         |            | ----    |         | ----    |                                             |
| 317         | IP501      | <0.5    |         | ----    |                                             |
| 323         | D4752BMod. | <1      |         | ----    |                                             |
| 333         |            | ----    |         | ----    |                                             |
| 334         |            | ----    |         | ----    |                                             |
| 336         |            | ----    |         | ----    |                                             |
| 337         |            | ----    |         | ----    |                                             |
| 340         | IP501      | 1.2     |         | 3.04    |                                             |
| 356         |            | ----    |         | ----    |                                             |
| 369         | IP501      | <3      |         | ----    |                                             |
| 402         | IP501      | 1.451   | R(0.05) | 4.46    |                                             |
| 445         | IP PM CW   | 0.69    |         | 0.15    |                                             |
| 447         |            | ----    |         | ----    |                                             |
| 463         |            | ----    |         | ----    |                                             |
| 494         | IP501      | 1.4     | R(0.05) | 4.18    |                                             |
| 621         |            | ----    |         | ----    |                                             |
| 657         | IP501      | <3      |         | ----    |                                             |
| 663         | IP501      | 0.6     |         | -0.36   |                                             |
| 704         | IP470      | <3      |         | ----    |                                             |
| 732         |            | ----    |         | ----    |                                             |
| 743         | IP470      | 0.7     |         | 0.20    |                                             |
| 781         |            | ----    |         | ----    |                                             |
| 784         |            | ----    |         | ----    |                                             |
| 785         | IP470      | 0.91    |         | 1.40    |                                             |
| 791         |            | ----    |         | ----    |                                             |
| 874         | IP501      | 0.5     |         | -0.93   |                                             |
| 875         | IP501      | 0.56    |         | -0.59   |                                             |
| 994         | IP501      | <3      |         | ----    |                                             |
| 995         | D5863      | 0.6     |         | -0.36   |                                             |
| 1065        |            | ----    |         | ----    |                                             |
| 1134        | IP PM CW   | 2.135   | R(0.01) | 8.35    |                                             |
| 1297        |            | ----    |         | ----    |                                             |
| 1365        | IP470      | 0.5     |         | -0.93   |                                             |
| 1379        |            | ----    |         | ----    |                                             |
| 1397        |            | ----    |         | ----    |                                             |
| 1455        | IP PM CW   | 0.7     |         | 0.20    |                                             |
| 1510        |            | ----    |         | ----    |                                             |
| 1543        |            | ----    |         | ----    |                                             |
| 1556        |            | ----    |         | ----    |                                             |
| 1635        | D5185      | 0.344   |         | -1.82   |                                             |
| 1677        | IP PM CW   | 0.56    |         | -0.59   |                                             |
| 1857        | IP501      | 0.6     |         | -0.36   |                                             |
| 1881        | IP PM CW   | 0.90    |         | 1.34    |                                             |
| 1949        | IP PM CW   | 0.74    |         | 0.43    |                                             |
| 1986        | IP470      | 0.63    |         | -0.19   |                                             |
| 1995        |            | ----    |         | ----    |                                             |
| 6020        | IP470      | 0.8     |         | 0.77    |                                             |
| 6021        | IP501      | 0.6     |         | -0.36   |                                             |
| <br>        |            |         |         |         |                                             |
| normality   |            | suspect |         |         | Application range IP501/IP470: 3-100 mg/kg  |
| n           |            | 20      |         |         | Application range IP PM CW/04: 0.1-10 mg/kg |
| outliers    |            | 3       |         |         |                                             |
| mean (n)    |            | 0.664   |         |         |                                             |
| st.dev. (n) |            | 0.2006  |         |         |                                             |
| R(calc.)    |            | 0.562   |         |         |                                             |
| R(IP501:05) |            | 0.494   |         |         | Compare R(IP470:05) = 3.583                 |



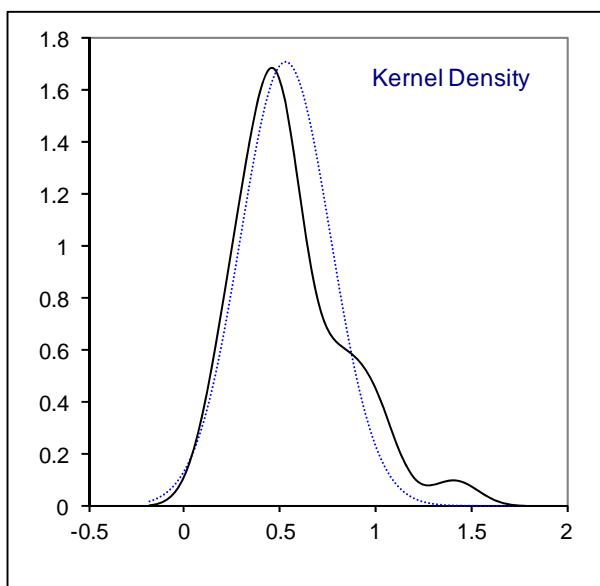
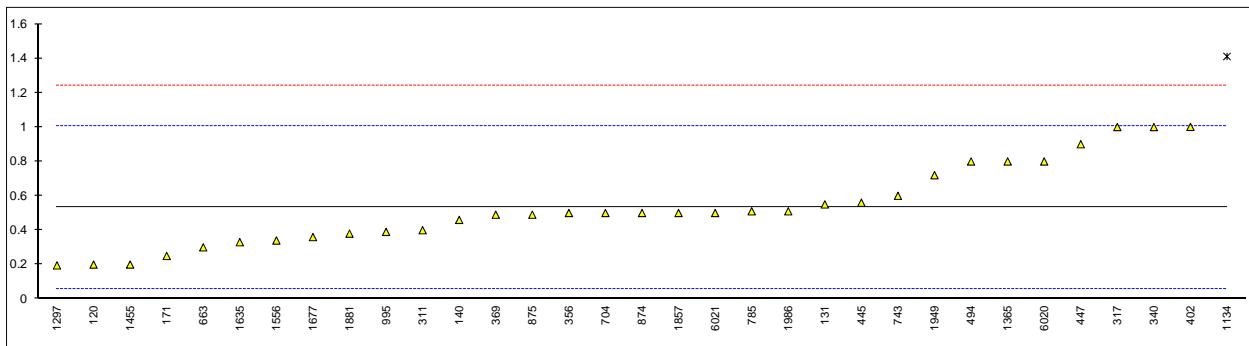
## Determination of Copper on sample #15250; results in mg/kg

| lab                                           | method     | value | mark | z(targ) | remarks |
|-----------------------------------------------|------------|-------|------|---------|---------|
| 52                                            |            | ----  |      | ----    |         |
| 62                                            |            | ----  |      | ----    |         |
| 120                                           | IP501      | <0.1  |      | ----    |         |
| 131                                           |            | ----  |      | ----    |         |
| 133                                           |            | ----  |      | ----    |         |
| 140                                           | D5708      | 0.02  |      | ----    |         |
| 150                                           | D5708      | <1    |      | ----    |         |
| 158                                           |            | ----  |      | ----    |         |
| 171                                           | D5708      | 0.18  |      | ----    |         |
| 228                                           |            | ----  |      | ----    |         |
| 311                                           | IP PM CW   | <0.1  |      | ----    |         |
| 313                                           |            | ----  |      | ----    |         |
| 317                                           | D7111      | <0.5  |      | ----    |         |
| 323                                           | D4752BMod. | <1    |      | ----    |         |
| 333                                           |            | ----  |      | ----    |         |
| 334                                           |            | ----  |      | ----    |         |
| 336                                           |            | ----  |      | ----    |         |
| 337                                           |            | ----  |      | ----    |         |
| 340                                           |            | ----  |      | ----    |         |
| 356                                           | IP PM CW   | <0.1  |      | ----    |         |
| 369                                           | IP PM CW   | <0.1  |      | ----    |         |
| 402                                           | IP501      | 0.01  |      | ----    |         |
| 445                                           | IP PM CW   | <0.1  |      | ----    |         |
| 447                                           | IP PM CW   | 0     |      | ----    |         |
| 463                                           |            | ----  |      | ----    |         |
| 494                                           | D7111      | 0.01  |      | ----    |         |
| 621                                           |            | ----  |      | ----    |         |
| 657                                           | IP501      | <1    |      | ----    |         |
| 663                                           |            | ----  |      | ----    |         |
| 704                                           | IP PM CW   | <0.1  |      | ----    |         |
| 732                                           |            | ----  |      | ----    |         |
| 743                                           | IP470      | 0.004 |      | ----    |         |
| 781                                           |            | ----  |      | ----    |         |
| 784                                           |            | ----  |      | ----    |         |
| 785                                           | IP470      | 0.03  |      | ----    |         |
| 791                                           | IP470      | <1    |      | ----    |         |
| 874                                           | IP501      | 0.0   |      | ----    |         |
| 875                                           | IP501      | <1    |      | ----    |         |
| 994                                           | IP501      | <0.1  |      | ----    |         |
| 995                                           | D5863      | <0.1  |      | ----    |         |
| 1065                                          |            | ----  |      | ----    |         |
| 1134                                          | IP PM CW   | 0.172 |      | ----    |         |
| 1297                                          |            | ----  |      | ----    |         |
| 1365                                          | in house   | 0.03  |      | ----    |         |
| 1379                                          |            | ----  |      | ----    |         |
| 1397                                          |            | ----  |      | ----    |         |
| 1455                                          | IP PM CW   | <0.1  |      | ----    |         |
| 1510                                          |            | ----  |      | ----    |         |
| 1543                                          |            | ----  |      | ----    |         |
| 1556                                          | in house   | 0.005 |      | ----    |         |
| 1635                                          | D5185      | 0.41  |      | ----    |         |
| 1677                                          | IP PM CW   | <0.1  |      | ----    |         |
| 1857                                          | UOP407     | 0.007 |      | ----    |         |
| 1881                                          | IP PM CW   | 0.02  |      | ----    |         |
| 1949                                          | IP PM CW   | 0.04  |      | ----    |         |
| 1986                                          | IP PM CW   | 0.01  |      | ----    |         |
| 1995                                          |            | ----  |      | ----    |         |
| 6020                                          | IP470      | 0.01  |      | ----    |         |
| 6021                                          | IP PM CW   | 0.01  |      | ----    |         |
| normality                                     |            |       |      |         |         |
| n                                             |            |       |      |         |         |
| outliers                                      |            |       |      |         |         |
| mean (n)                                      |            |       |      |         |         |
| st.dev. (n)                                   |            |       |      |         |         |
| R(calc.)                                      |            |       |      |         |         |
| R(lit)                                        |            |       |      |         |         |
| Application range IP PM CW/04: 0.1 – 10 mg/kg |            |       |      |         |         |

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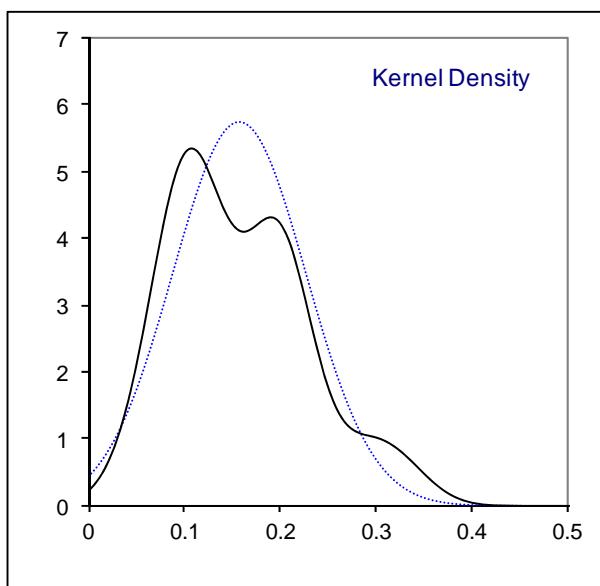
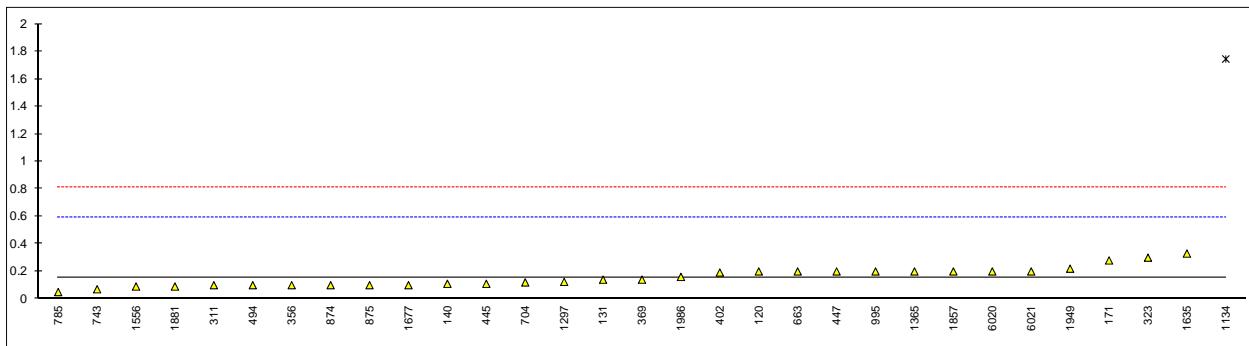
## Determination of Iron on sample #15250; results in mg/kg

| lab                                           | method     | value | mark    | z(targ) | remarks             |
|-----------------------------------------------|------------|-------|---------|---------|---------------------|
| 52                                            | IP470      | <1    |         | ----    |                     |
| 62                                            |            | ----  |         | ----    |                     |
| 120                                           | IP501      | 0.2   | C       | -1.40   | First reported: 1.5 |
| 131                                           | IP501      | 0.55  |         | 0.07    |                     |
| 133                                           |            | ----  |         | ----    |                     |
| 140                                           | D5708      | 0.46  |         | -0.31   |                     |
| 150                                           | D5708      | <1    |         | ----    |                     |
| 158                                           |            | ----  |         | ----    |                     |
| 171                                           | D5708      | 0.25  |         | -1.19   |                     |
| 228                                           |            | ----  |         | ----    |                     |
| 311                                           | IP PM CW   | 0.4   |         | -0.56   |                     |
| 313                                           |            | ----  |         | ----    |                     |
| 317                                           | IP501      | 1     | C       | 1.98    | First reported: 2   |
| 323                                           | D4752BMod. | <1    |         | ----    |                     |
| 333                                           |            | ----  |         | ----    |                     |
| 334                                           |            | ----  |         | ----    |                     |
| 336                                           |            | ----  |         | ----    |                     |
| 337                                           |            | ----  |         | ----    |                     |
| 340                                           | IP501      | 1     |         | 1.98    |                     |
| 356                                           | IP PM CW   | 0.5   |         | -0.14   |                     |
| 369                                           | IP PM CW   | 0.49  |         | -0.18   |                     |
| 402                                           | IP501      | 1.001 |         | 1.98    |                     |
| 445                                           | IP PM CW   | 0.56  |         | 0.12    |                     |
| 447                                           | IP PM CW   | 0.9   |         | 1.55    |                     |
| 463                                           |            | ----  |         | ----    |                     |
| 494                                           | IP501      | 0.8   |         | 1.13    |                     |
| 621                                           |            | ----  |         | ----    |                     |
| 657                                           | IP501      | <2    |         | ----    |                     |
| 663                                           | IP501      | 0.3   |         | -0.98   |                     |
| 704                                           | IP PM CW   | 0.50  |         | -0.14   |                     |
| 732                                           |            | ----  |         | ----    |                     |
| 743                                           | IP470      | 0.6   |         | 0.29    |                     |
| 781                                           |            | ----  |         | ----    |                     |
| 784                                           |            | ----  |         | ----    |                     |
| 785                                           | IP470      | 0.51  |         | -0.09   |                     |
| 791                                           | IP470      | <2    |         | ----    |                     |
| 874                                           | IP501      | 0.5   |         | -0.14   |                     |
| 875                                           | IP501      | 0.49  |         | -0.18   |                     |
| 994                                           | IP501      | <2    |         | ----    |                     |
| 995                                           | D5863      | 0.39  |         | -0.60   |                     |
| 1065                                          |            | ----  |         | ----    |                     |
| 1134                                          | IP PM CW   | 1.411 | R(0.05) | 3.71    |                     |
| 1297                                          | D5708      | 0.195 |         | -1.43   |                     |
| 1365                                          | IP470      | 0.8   |         | 1.13    |                     |
| 1379                                          |            | ----  |         | ----    |                     |
| 1397                                          |            | ----  |         | ----    |                     |
| 1455                                          | IP PM CW   | 0.2   |         | -1.40   |                     |
| 1510                                          |            | ----  |         | ----    |                     |
| 1543                                          |            | ----  |         | ----    |                     |
| 1556                                          | in house   | 0.34  |         | -0.81   |                     |
| 1635                                          | D5185      | 0.33  |         | -0.85   |                     |
| 1677                                          | IP PM CW   | 0.36  |         | -0.73   |                     |
| 1857                                          | IP501      | 0.5   |         | -0.14   |                     |
| 1881                                          | IP PM CW   | 0.38  |         | -0.64   |                     |
| 1949                                          | IP PM CW   | 0.72  |         | 0.79    |                     |
| 1986                                          | IP PM CW   | 0.51  |         | -0.09   |                     |
| 1995                                          |            | ----  |         | ----    |                     |
| 6020                                          | IP470      | 0.8   |         | 1.13    |                     |
| 6021                                          | IP501      | 0.5   |         | -0.14   |                     |
| normality                                     |            |       |         |         |                     |
| n                                             |            |       |         |         |                     |
| outliers                                      |            |       |         |         |                     |
| mean (n)                                      |            |       |         |         |                     |
| st.dev. (n)                                   |            |       |         |         |                     |
| R(calc.)                                      |            |       |         |         |                     |
| R(IP501:05)                                   |            |       |         |         |                     |
| Application range IP501/IP470: 2 – 60 mg/kg   |            |       |         |         |                     |
| Application range IP PM CW/04: 0.1 – 10 mg/kg |            |       |         |         |                     |
| Compare R(IP470:05) = 1.2927                  |            |       |         |         |                     |



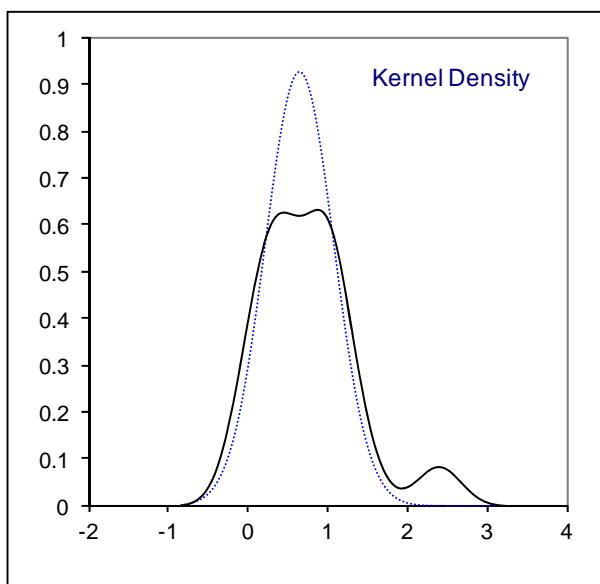
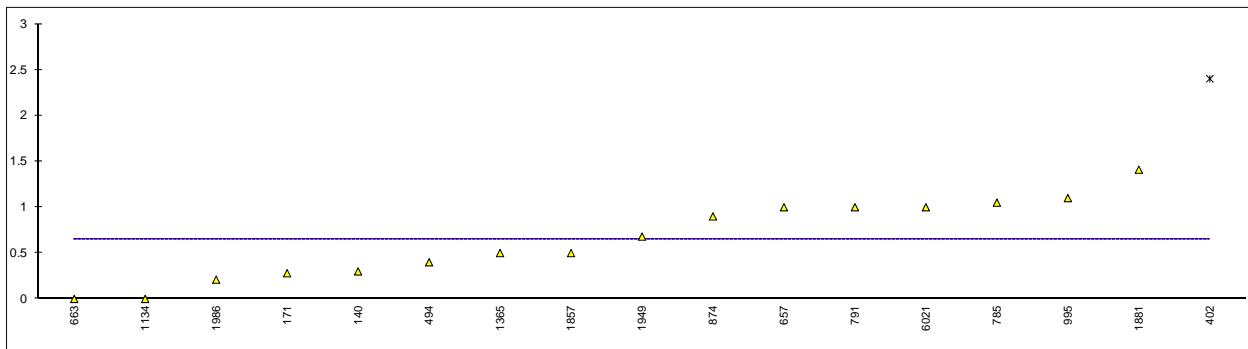
## Determination of Nickel on sample #15250; results in mg/kg

| lab                                           | method   | value | mark    | z(targ) | remarks |
|-----------------------------------------------|----------|-------|---------|---------|---------|
| 52                                            | IP470    | <1    |         | ----    |         |
| 62                                            |          | ----  |         | ----    |         |
| 120                                           | IP501    | 0.2   |         | 0.20    |         |
| 131                                           | IP501    | 0.14  |         | -0.08   |         |
| 133                                           |          | ----  |         | ----    |         |
| 140                                           | D5708    | 0.11  |         | -0.22   |         |
| 150                                           | D5708    | <1    |         | ----    |         |
| 158                                           |          | ----  |         | ----    |         |
| 171                                           | D5708    | 0.28  |         | 0.56    |         |
| 228                                           |          | ----  |         | ----    |         |
| 311                                           | IP PM CW | 0.1   |         | -0.26   |         |
| 313                                           |          | ----  |         | ----    |         |
| 317                                           | IP501    | <1    |         | ----    |         |
| 323                                           | INH-018  | 0.3   |         | 0.66    |         |
| 333                                           |          | ----  |         | ----    |         |
| 334                                           |          | ----  |         | ----    |         |
| 336                                           |          | ----  |         | ----    |         |
| 337                                           |          | ----  |         | ----    |         |
| 340                                           | IP501    | <1    |         | ----    |         |
| 356                                           | IP PM CW | 0.1   |         | -0.26   |         |
| 369                                           | IP PM CW | 0.14  |         | -0.08   |         |
| 402                                           | IP501    | 0.192 |         | 0.16    |         |
| 445                                           | IP PM CW | 0.11  |         | -0.22   |         |
| 447                                           | IP PM CW | 0.2   |         | 0.20    |         |
| 463                                           |          | ----  |         | ----    |         |
| 494                                           | IP501    | 0.1   |         | -0.26   |         |
| 621                                           |          | ----  |         | ----    |         |
| 657                                           | IP501    | <1    |         | ----    |         |
| 663                                           | IP501    | 0.2   |         | 0.20    |         |
| 704                                           | IP PM CW | 0.12  |         | -0.17   |         |
| 732                                           |          | ----  |         | ----    |         |
| 743                                           | IP470    | 0.07  |         | -0.40   |         |
| 781                                           |          | ----  |         | ----    |         |
| 784                                           |          | ----  |         | ----    |         |
| 785                                           | IP470    | 0.05  |         | -0.49   |         |
| 791                                           | IP470    | <1    |         | ----    |         |
| 874                                           | IP501    | 0.1   |         | -0.26   |         |
| 875                                           | IP501    | 0.10  |         | -0.26   |         |
| 994                                           | IP501    | <1    |         | ----    |         |
| 995                                           | D5863    | 0.20  |         | 0.20    |         |
| 1065                                          |          | ----  |         | ----    |         |
| 1134                                          | IP PM CW | 1.745 | R(0.01) | 7.31    |         |
| 1297                                          | D5708    | 0.124 |         | -0.15   |         |
| 1365                                          | IP470    | 0.2   |         | 0.20    |         |
| 1379                                          |          | ----  |         | ----    |         |
| 1397                                          |          | ----  |         | ----    |         |
| 1455                                          | IP PM CW | <0.1  |         | ----    |         |
| 1510                                          |          | ----  |         | ----    |         |
| 1543                                          |          | ----  |         | ----    |         |
| 1556                                          | in house | 0.09  |         | -0.31   |         |
| 1635                                          | D5185    | 0.33  |         | 0.79    |         |
| 1677                                          | IP PM CW | 0.10  |         | -0.26   |         |
| 1857                                          | IP501    | 0.2   |         | 0.20    |         |
| 1881                                          | IP PM CW | 0.09  |         | -0.31   |         |
| 1949                                          | IP PM CW | 0.22  |         | 0.29    |         |
| 1986                                          | IP PM CW | 0.16  |         | 0.01    |         |
| 1995                                          |          | ----  |         | ----    |         |
| 6020                                          | IP470    | 0.2   |         | 0.20    |         |
| 6021                                          | IP501    | 0.2   |         | 0.20    |         |
| normality                                     |          |       |         |         |         |
| n                                             |          |       |         |         |         |
| outliers                                      |          |       |         |         |         |
| mean (n)                                      |          |       |         |         |         |
| st.dev. (n)                                   |          |       |         |         |         |
| R(calc.)                                      |          |       |         |         |         |
| R(IP501:05)                                   |          |       |         |         |         |
| Application range IP501/IP470: 1 – 100 mg/kg  |          |       |         |         |         |
| Application range IP PM CW/04: 0.1 – 10 mg/kg |          |       |         |         |         |
| Compare R(IP470:05) = 0.8442                  |          |       |         |         |         |



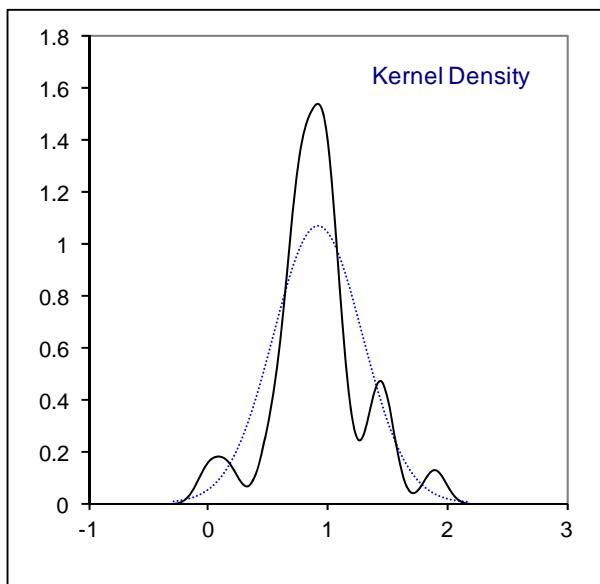
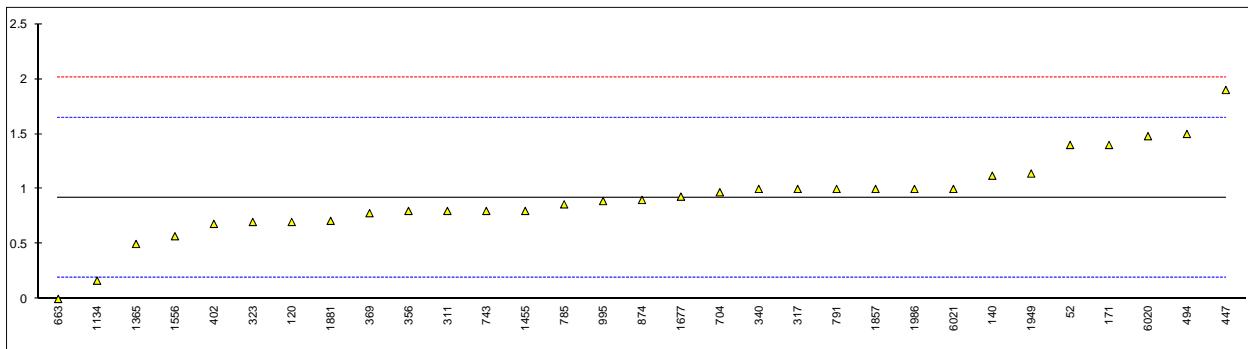
## Determination of Silicon on sample #15250; results in mg/kg

| lab                                 | method   | value   | mark    | z(targ) | remarks                                       |
|-------------------------------------|----------|---------|---------|---------|-----------------------------------------------|
| 52                                  | IP470    | <10     |         | ----    |                                               |
| 62                                  |          | -----   |         | -----   |                                               |
| 120                                 | IP501    | <0.1    |         | -----   |                                               |
| 131                                 |          | -----   |         | -----   |                                               |
| 133                                 |          | -----   |         | -----   |                                               |
| 140                                 | D5708    | 0.30    |         | -----   |                                               |
| 150                                 | D5708    | <1      |         | -----   |                                               |
| 158                                 |          | -----   |         | -----   |                                               |
| 171                                 | D5708    | 0.28    |         | -----   |                                               |
| 228                                 |          | -----   |         | -----   |                                               |
| 311                                 | UOP796   | <1.0    |         | -----   |                                               |
| 313                                 |          | -----   |         | -----   |                                               |
| 317                                 | IP501    | <10     |         | -----   |                                               |
| 323                                 |          | <10     |         | -----   |                                               |
| 333                                 |          | -----   |         | -----   |                                               |
| 334                                 |          | -----   |         | -----   |                                               |
| 336                                 |          | -----   |         | -----   |                                               |
| 337                                 |          | -----   |         | -----   |                                               |
| 340                                 | IP501    | <10     |         | -----   |                                               |
| 356                                 |          | -----   |         | -----   |                                               |
| 369                                 | D5184    | <5      |         | -----   |                                               |
| 402                                 | IP501    | 2.402   | G(0.05) | -----   |                                               |
| 445                                 |          | -----   |         | -----   |                                               |
| 447                                 |          | -----   |         | -----   |                                               |
| 463                                 |          | -----   |         | -----   |                                               |
| 494                                 | IP501    | 0.4     |         | -----   |                                               |
| 621                                 |          | -----   |         | -----   |                                               |
| 657                                 | IP501    | 1       |         | -----   |                                               |
| 663                                 | IP501    | 0.0     |         | -----   |                                               |
| 704                                 | IP470    | <10     |         | -----   |                                               |
| 732                                 |          | -----   |         | -----   |                                               |
| 743                                 |          | -----   |         | -----   |                                               |
| 781                                 |          | -----   |         | -----   |                                               |
| 784                                 |          | -----   |         | -----   |                                               |
| 785                                 | IP470    | 1.05    |         | -----   |                                               |
| 791                                 | IP470    | 1       |         | -----   |                                               |
| 874                                 | IP501    | 0.9     |         | -----   |                                               |
| 875                                 | IP501    | <1      |         | -----   |                                               |
| 994                                 | IP501    | <10     |         | -----   |                                               |
| 995                                 | IP470    | 1.1     |         | -----   |                                               |
| 1065                                |          | -----   |         | -----   |                                               |
| 1134                                | IP PM CW | 0       |         | -----   |                                               |
| 1297                                |          | -----   |         | -----   |                                               |
| 1365                                | IP470    | 0.5     |         | -----   |                                               |
| 1379                                |          | -----   |         | -----   |                                               |
| 1397                                |          | -----   |         | -----   |                                               |
| 1455                                |          | -----   |         | -----   |                                               |
| 1510                                |          | -----   |         | -----   |                                               |
| 1543                                |          | -----   |         | -----   |                                               |
| 1556                                |          | -----   |         | -----   |                                               |
| 1635                                | D5185    | <0.2    |         | -----   |                                               |
| 1677                                | IP501    | <10     |         | -----   |                                               |
| 1857                                | IP501    | 0.5     |         | -----   |                                               |
| 1881                                | IP470    | 1.41    |         | -----   |                                               |
| 1949                                | IP PM CW | 0.68    |         | -----   |                                               |
| 1986                                | IP470    | 0.21    |         | -----   |                                               |
| 1995                                |          | -----   |         | -----   |                                               |
| 6020                                |          | -----   |         | -----   |                                               |
| 6021                                | IP501    | 1.0     |         | -----   |                                               |
| <b><u>Results of IP501 only</u></b> |          |         |         |         |                                               |
| normality                           | OK       | OK      |         |         | Application range IP501/IP470: 10 – 250 mg/kg |
| n                                   | 16       | 6       |         |         | Application range IP PM CW/04: 0.1 – 10 mg/kg |
| outliers                            | 1        | 1       |         |         |                                               |
| mean (n)                            | 0.6456   | 0.6333  |         |         |                                               |
| st.dev. (n)                         | 0.43021  | 0.40332 |         |         |                                               |
| R(calc.)                            | 1.2046   | 1.1293  |         |         |                                               |
| R(IP501:05)                         | 0.2143   | 0.2143  |         |         | Compare R(IP470:05) = 1.0353                  |



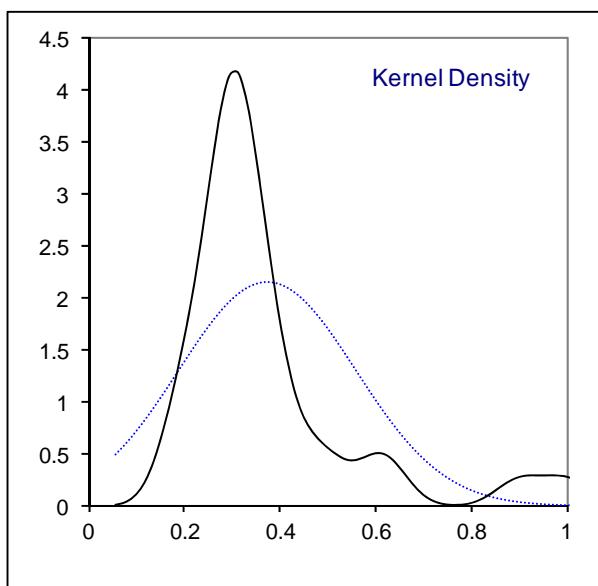
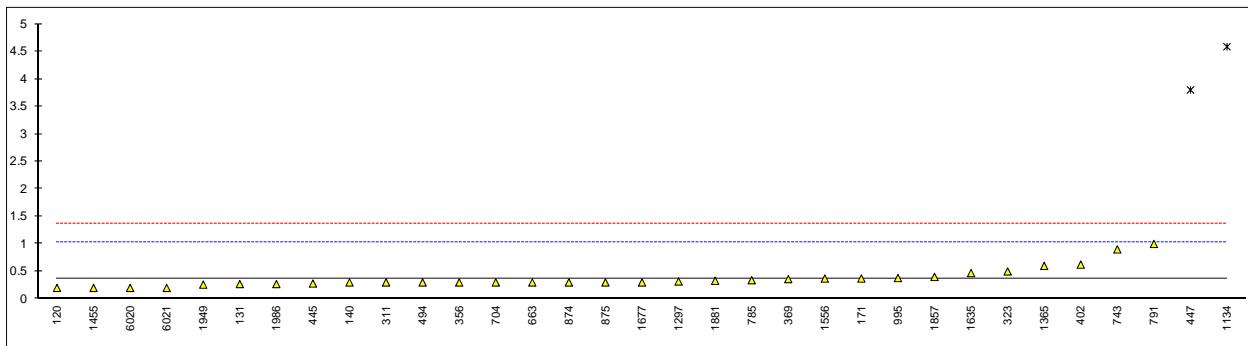
## Determination of Sodium on sample #15250; results in mg/kg

| lab                                           | method     | value | mark | z(targ) | remarks           |
|-----------------------------------------------|------------|-------|------|---------|-------------------|
| 52                                            | IP470      | 1.4   |      | 1.32    |                   |
| 62                                            |            | ----- |      | -----   |                   |
| 120                                           | IP501      | 0.7   |      | -0.60   |                   |
| 131                                           |            | ----- |      | -----   |                   |
| 133                                           |            | ----- |      | -----   |                   |
| 140                                           | D5708      | 1.12  |      | 0.55    |                   |
| 150                                           | D5708      | <1    |      | -----   |                   |
| 158                                           |            | ----- |      | -----   |                   |
| 171                                           | D5708      | 1.4   |      | 1.32    |                   |
| 228                                           |            | ----- |      | -----   |                   |
| 311                                           | IP PM CW   | 0.8   |      | -0.33   |                   |
| 313                                           |            | ----- |      | -----   |                   |
| 317                                           | IP501      | 1     | C    | 0.22    | First reported: 2 |
| 323                                           | D4752BMod. | 0.7   |      | -0.60   |                   |
| 333                                           |            | ----- |      | -----   |                   |
| 334                                           |            | ----- |      | -----   |                   |
| 336                                           |            | ----- |      | -----   |                   |
| 337                                           |            | ----- |      | -----   |                   |
| 340                                           | IP501      | 1.0   |      | 0.22    |                   |
| 356                                           | IP PM CW   | 0.8   |      | -0.33   |                   |
| 369                                           | IP PM CW   | 0.78  |      | -0.38   |                   |
| 402                                           | IP501      | 0.682 |      | -0.65   |                   |
| 445                                           |            | ----- |      | -----   |                   |
| 447                                           | IP PM CW   | 1.9   |      | 2.70    |                   |
| 463                                           |            | ----- |      | -----   |                   |
| 494                                           | IP501      | 1.5   |      | 1.60    |                   |
| 621                                           |            | ----- |      | -----   |                   |
| 657                                           | IP501      | <1    |      | -----   |                   |
| 663                                           | IP501      | 0.0   |      | -2.53   |                   |
| 704                                           | IP PM CW   | 0.97  |      | 0.14    |                   |
| 732                                           |            | ----- |      | -----   |                   |
| 743                                           | IP470      | 0.8   |      | -0.33   |                   |
| 781                                           |            | ----- |      | -----   |                   |
| 784                                           |            | ----- |      | -----   |                   |
| 785                                           | IP470      | 0.86  |      | -0.16   |                   |
| 791                                           | IP470      | 1     |      | 0.22    |                   |
| 874                                           | IP501      | 0.9   |      | -0.05   |                   |
| 875                                           | IP501      | <1    |      | -----   |                   |
| 994                                           |            | ----- |      | -----   |                   |
| 995                                           | D5863      | 0.89  |      | -0.08   |                   |
| 1065                                          |            | ----- |      | -----   |                   |
| 1134                                          | IP PM CW   | 0.166 |      | -2.07   |                   |
| 1297                                          |            | ----- |      | -----   |                   |
| 1365                                          | IP470      | 0.5   |      | -1.15   |                   |
| 1379                                          |            | ----- |      | -----   |                   |
| 1397                                          |            | ----- |      | -----   |                   |
| 1455                                          | IP PM CW   | 0.8   |      | -0.33   |                   |
| 1510                                          |            | ----- |      | -----   |                   |
| 1543                                          |            | ----- |      | -----   |                   |
| 1556                                          | in house   | 0.57  |      | -0.96   |                   |
| 1635                                          | D5185      | <1    |      | -----   |                   |
| 1677                                          | IP PM CW   | 0.93  |      | 0.03    |                   |
| 1857                                          | IP501      | 1     |      | 0.22    |                   |
| 1881                                          | IP PM CW   | 0.71  |      | -0.58   |                   |
| 1949                                          | IP PM CW   | 1.14  |      | 0.61    |                   |
| 1986                                          | IP PM CW   | 1.00  |      | 0.22    |                   |
| 1995                                          |            | ----- |      | -----   |                   |
| 6020                                          | IP470      | 1.48  |      | 1.54    |                   |
| 6021                                          | IP501      | 1.0   |      | 0.22    |                   |
| normality                                     |            |       |      |         |                   |
| n                                             |            |       |      |         |                   |
| outliers                                      |            |       |      |         |                   |
| mean (n)                                      |            |       |      |         |                   |
| st.dev. (n)                                   |            |       |      |         |                   |
| R(calc.)                                      |            |       |      |         |                   |
| R(IP501:05)                                   |            |       |      |         |                   |
| suspect                                       |            |       |      |         |                   |
| 31                                            |            |       |      |         |                   |
| 0                                             |            |       |      |         |                   |
| 0.9193                                        |            |       |      |         |                   |
| 0.37390                                       |            |       |      |         |                   |
| 1.0469                                        |            |       |      |         |                   |
| 1.0185                                        |            |       |      |         |                   |
| Application range IP501/IP470: 1 – 100 mg/kg  |            |       |      |         |                   |
| Application range IP PM CW/04: 0.1 – 10 mg/kg |            |       |      |         |                   |
| Compare R(IP470:05) = 1.2388                  |            |       |      |         |                   |



## Determination of Vanadium on sample #15250; results in mg/kg

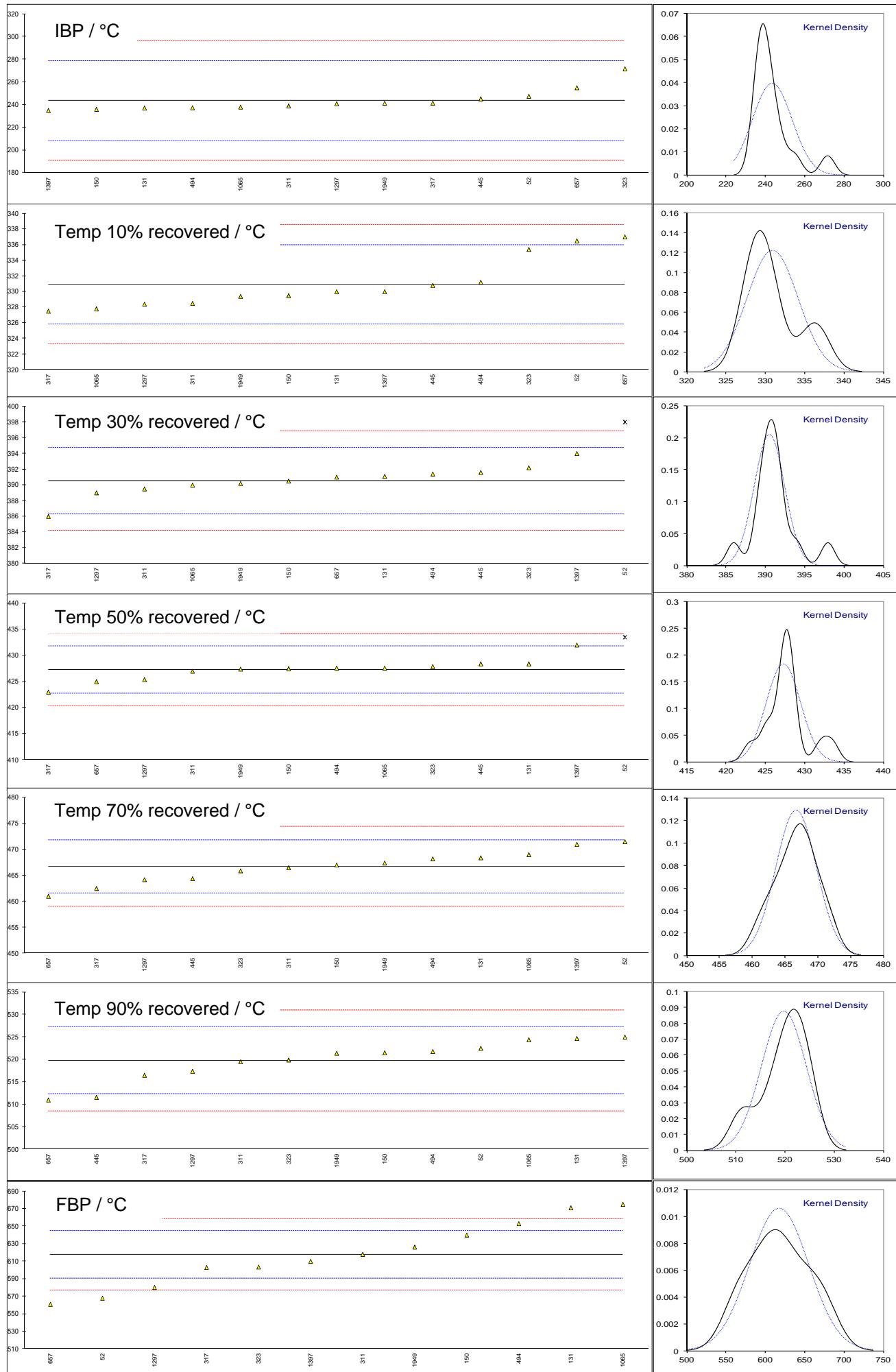
| lab                                           | method   | value | mark    | z(targ) | remarks |
|-----------------------------------------------|----------|-------|---------|---------|---------|
| 52                                            | IP470    | <1    |         | ----    |         |
| 62                                            |          | ----  |         | ----    |         |
| 120                                           | IP501    | 0.2   |         | -0.52   |         |
| 131                                           | IP501    | 0.27  |         | -0.31   |         |
| 133                                           |          | ----  |         | ----    |         |
| 140                                           | D5708    | 0.30  |         | -0.22   |         |
| 150                                           | D5708    | <1    |         | ----    |         |
| 158                                           |          | ----  |         | ----    |         |
| 171                                           | D5708    | 0.371 |         | 0.00    |         |
| 228                                           |          | ----  |         | ----    |         |
| 311                                           | IP PM CW | 0.3   |         | -0.22   |         |
| 313                                           |          | ----  |         | ----    |         |
| 317                                           | IP501    | <1    |         | ----    |         |
| 323                                           | INH-018  | 0.5   |         | 0.38    |         |
| 333                                           |          | ----  |         | ----    |         |
| 334                                           |          | ----  |         | ----    |         |
| 336                                           |          | ----  |         | ----    |         |
| 337                                           |          | ----  |         | ----    |         |
| 340                                           | IP501    | <1    |         | ----    |         |
| 356                                           | IP PM CW | 0.3   |         | -0.22   |         |
| 369                                           | IP PM CW | 0.36  |         | -0.04   |         |
| 402                                           | IP501    | 0.624 |         | 0.76    |         |
| 445                                           | IP PM CW | 0.28  |         | -0.28   |         |
| 447                                           | IP PM CW | 3.8   | R(0.01) | 10.34   |         |
| 463                                           |          | ----  |         | ----    |         |
| 494                                           | IP501    | 0.3   |         | -0.22   |         |
| 621                                           |          | ----  |         | ----    |         |
| 657                                           | IP501    | <1    |         | ----    |         |
| 663                                           | IP501    | 0.3   |         | -0.22   |         |
| 704                                           | IP PM CW | 0.30  |         | -0.22   |         |
| 732                                           |          | ----  |         | ----    |         |
| 743                                           | IP470    | 0.9   |         | 1.59    |         |
| 781                                           |          | ----  |         | ----    |         |
| 784                                           |          | ----  |         | ----    |         |
| 785                                           | IP470    | 0.34  |         | -0.10   |         |
| 791                                           | IP470    | 1     |         | 1.89    |         |
| 874                                           | IP501    | 0.3   |         | -0.22   |         |
| 875                                           | IP501    | 0.30  |         | -0.22   |         |
| 994                                           | IP501    | <1    |         | ----    |         |
| 995                                           | D5863    | 0.38  |         | 0.02    |         |
| 1065                                          |          | ----  |         | ----    |         |
| 1134                                          | IP PM CW | 4.586 | R(0.01) | 12.71   |         |
| 1297                                          | D5708    | 0.317 |         | -0.17   |         |
| 1365                                          | IP470    | 0.6   |         | 0.69    |         |
| 1379                                          |          | ----  |         | ----    |         |
| 1397                                          |          | ----  |         | ----    |         |
| 1455                                          | IP PM CW | 0.2   |         | -0.52   |         |
| 1510                                          |          | ----  |         | ----    |         |
| 1543                                          |          | ----  |         | ----    |         |
| 1556                                          | in house | 0.37  |         | -0.01   |         |
| 1635                                          | D5185    | 0.47  |         | 0.29    |         |
| 1677                                          | IP PM CW | 0.30  |         | -0.22   |         |
| 1857                                          | IP501    | 0.4   |         | 0.08    |         |
| 1881                                          | IP PM CW | 0.33  |         | -0.13   |         |
| 1949                                          | IP PM CW | 0.26  |         | -0.34   |         |
| 1986                                          | IP PM CW | 0.27  |         | -0.31   |         |
| 1995                                          |          | ----  |         | ----    |         |
| 6020                                          | IP470    | 0.2   |         | -0.52   |         |
| 6021                                          | IP501    | 0.2   |         | -0.52   |         |
| normality                                     |          |       |         |         |         |
| n                                             |          |       |         |         |         |
| outliers                                      |          |       |         |         |         |
| mean (n)                                      |          |       |         |         |         |
| st.dev. (n)                                   |          |       |         |         |         |
| R(calc.)                                      |          |       |         |         |         |
| R(IP501:05)                                   |          |       |         |         |         |
| Application range IP501/IP470: 1 – 400 mg/kg  |          |       |         |         |         |
| Application range IP PM CW/04: 0.1 – 10 mg/kg |          |       |         |         |         |
| Compare R(IP470:05) = 1.989                   |          |       |         |         |         |



## Determination of Simulated Distillation acc. to ASTM D6352 on sample #15250; result in °C

| lab         | method      | IBP    | 10% rec | 30% rec              | 50% rec              | 70% rec | 90% rec | FBP    |
|-------------|-------------|--------|---------|----------------------|----------------------|---------|---------|--------|
| 52          | D7213       | 247.5  | 336.5   | <u>398.0 D(0.05)</u> | <u>433.5 D(0.05)</u> | 471.5   | 522.5   | 568.0  |
| 62          |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 120         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 131         | D6352       | 237.2  | 330     | 391.1                | 428.4                | 468.4   | 524.7   | 671    |
| 133         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 140         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 150         | D7169       | 236.0  | 329.5   | 390.5                | 427.5                | 467.0   | 521.5   | 640.0  |
| 158         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 171         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 228         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 311         | D6352       | 239.0  | 328.5   | 389.5                | 427.0                | 466.5   | 519.5   | 618.0  |
| 313         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 317         | D6352       | 241.5  | 327.5   | 386.0                | 423.0                | 462.5   | 516.5   | 603.0  |
| 323         | D6352       | 271.7  | 335.4   | 392.2                | 427.9                | 465.9   | 519.9   | 603.5  |
| 333         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 334         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 336         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 337         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 340         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 356         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 369         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 402         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 445         | D2887       | 245.2  | 330.8   | 391.6                | 428.4                | 464.4   | 511.6   | >538   |
| 447         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 463         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 494         | D6352       | 237.4  | 331.2   | 391.4                | 427.6                | 468.2   | 521.8   | 653.0  |
| 621         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 657         | D6352       | 255.0  | 337.0   | 391.0                | 425.0                | 461.0   | 511.0   | 561.0  |
| 663         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 704         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 732         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 743         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 781         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 784         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 785         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 791         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 874         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 875         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 994         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 995         |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 1065        | D6352       | 238.0  | 327.8   | 390.0                | 427.6                | 469.0   | 524.4   | 675.0  |
| 1134        |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 1297        | D7213       | 241.0  | 328.4   | 389.0                | 425.4                | 464.2   | 517.4   | 580.2  |
| 1365        |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 1379        |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 1397        | D6352       | 235.0  | 330.0   | 394.0                | 432.0                | 471.0   | 525.0   | 610.0  |
| 1455        |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 1510        |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 1543        |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 1556        |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 1635        |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 1677        |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 1857        |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 1881        |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 1949        | combination | 241.4  | 329.4   | 390.2                | 427.4                | 467.4   | 521.4   | 626.2  |
| 1986        |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 1995        |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 6020        |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| 6021        |             | ----   | ----    | ----                 | ----                 | ----    | ----    | ----   |
| normality   |             | not OK | suspect | not OK               | not OK               | OK      | OK      | OK     |
| n           |             | 13     | 13      | 12                   | 12                   | 13      | 13      | 12     |
| outliers    |             | 0      | 0       | 1                    | 1                    | 0       | 0       | 0      |
| mean (n)    |             | 243.53 | 330.92  | 390.54               | 427.27               | 466.69  | 519.78  | 617.41 |
| st.dev. (n) |             | 10.070 | 3.265   | 1.944                | 2.186                | 3.094   | 4.568   | 37.546 |
| R(calc.)    |             | 28.20  | 9.14    | 5.44                 | 6.12                 | 8.66    | 12.79   | 105.13 |
| R(D6352:15) |             | 49.10  | 7.10    | 5.90                 | 6.40                 | 7.20    | 10.50   | 38.10  |

Lab 1949 reported to use a combination of methods: D7169/D7500/D6352



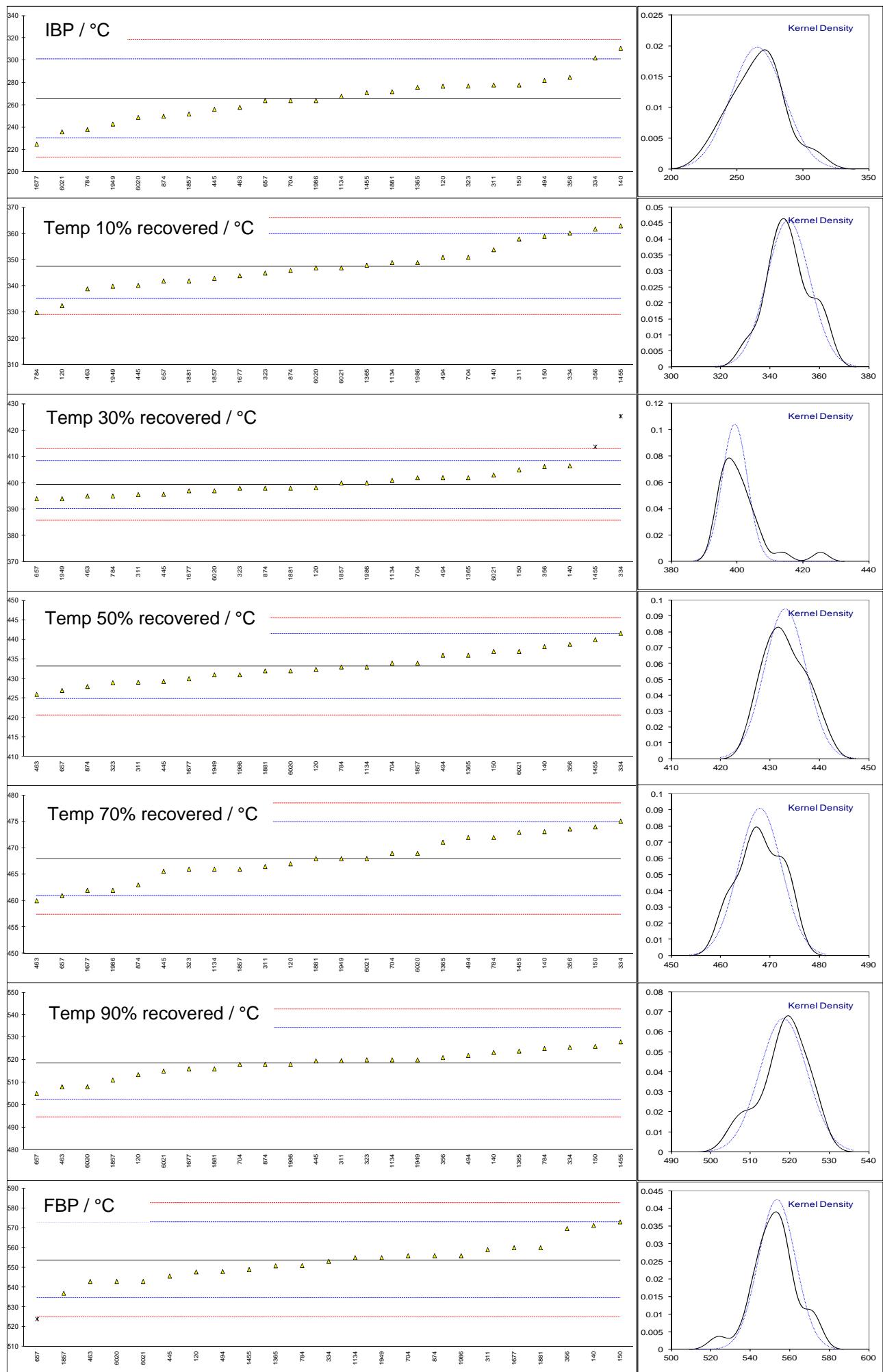
## Determination of Distillation acc. to ASTM D1160 on sample #15250; result in °C

| lab         | method | IBP    | 10% rec | 30% rec                | 50% rec | 70% rec | 90% rec | FBP           |
|-------------|--------|--------|---------|------------------------|---------|---------|---------|---------------|
| 52          |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 62          |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 120         | D1160  | 276.9  | 332.6   | 398.2                  | 432.4   | 467.0   | 513.4   | 547.8         |
| 131         |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 133         |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 140         | D1160  | 310.9  | 353.9   | 406.5                  | 438.2   | 473.1   | 523.3   | 571.3         |
| 150         | D1160  | 278    | 359     | 405                    | 437     | 474     | 526     | 573           |
| 158         |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 171         |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 228         |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 311         | D1160  | 278.0  | 358.0   | 395.6                  | 429.1   | 466.5   | 519.6   | 559.1         |
| 313         |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 317         |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 323         | D1160  | 277    | 345     | 398                    | 429     | 466     | 520     | ----          |
| 333         |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 334         | D1160  | 302.2  | 360.3   | <b>425.3 R(0.01)</b>   | 441.6   | 475.1   | 525.6   | 553.2         |
| 336         |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 337         |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 340         |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 356         | D1160  | 284.9  | 361.8   | 406.2                  | 438.8   | 473.6   | 521.0   | 569.7         |
| 369         |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 402         |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 445         | D1160  | 256.2  | 340.3   | 395.7                  | 429.3   | 465.6   | 519.5   | 545.7         |
| 447         |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 463         | D1160  | 258    | 339     | 395                    | 426     | 460     | 508     | 543           |
| 494         | D1160  | 282    | 351     | 402                    | 436     | 472     | 522     | 548           |
| 621         |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 657         | D1160  | 264    | 342     | 394                    | 427     | 461     | 505     | <b>524 ex</b> |
| 663         |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 704         | D1160  | 264    | 351     | 402                    | 434     | 469     | 518     | 556           |
| 732         |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 743         |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 781         |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 784         | D1160  | 238.0  | 330.0   | 395.0                  | 433.0   | 472.0   | 525.0   | 551.0         |
| 785         |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 791         |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 874         | D1160  | 250    | 346     | 398                    | 428     | 463     | 518     | 556           |
| 875         |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 994         |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 995         |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 1065        |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 1134        | D1160  | 268    | 349     | 401                    | 433     | 466     | 520     | 555           |
| 1297        |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 1365        | D1160  | 276.0  | 348.0   | 402.0                  | 436.0   | 471.1   | 523.9   | 550.9         |
| 1379        |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 1397        |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 1455        | D1160  | 271    | 363     | <b>413.7 C.R(0.05)</b> | 440     | 473     | 528     | 549           |
| 1510        |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 1543        |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 1556        |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 1635        |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 1677        | D1160  | 225    | 344     | 397                    | 430     | 462     | 516     | 560           |
| 1857        | D1160  | 252    | 343     | 400                    | 434     | 466     | 511     | 537           |
| 1881        | D1160  | 272    | 342     | 398                    | 432     | 468     | 516     | 560           |
| 1949        | D1160  | 243    | 340     | 394                    | 431     | 468     | 520     | 555           |
| 1986        | D1160  | 264    | 349     | 400                    | 431     | 462     | 518     | 556           |
| 1995        |        | ----   | ----    | ----                   | ----    | ----    | ----    | ----          |
| 6020        | D1160  | 249    | 347     | 397                    | 432     | 469     | 508     | 543           |
| 6021        | D1160  | 236    | 347     | 403                    | 437     | 468     | 515     | 543           |
| normality   | OK     | OK     | OK      | OK                     | OK      | OK      | OK      | OK            |
| n           | 24     | 24     | 22      | 24                     | 24      | 24      | 24      | 22            |
| outliers    | 0      | 0      | 2       | 0                      | 0       | 0       | 0       | 0+1ex         |
| mean (n)    | 265.67 | 347.58 | 399.24  | 433.14                 | 467.96  | 518.35  | 553.76  |               |
| st.dev. (n) | 20.256 | 8.677  | 3.829   | 4.219                  | 4.390   | 6.003   | 9.404   |               |
| R(calc.)    | 56.72  | 24.30  | 10.72   | 11.81                  | 12.29   | 16.81   | 26.33   |               |
| R(D1160:15) | 49.45  | 17.14  | 12.70   | 11.61                  | 9.81    | 22.42   | 26.89   |               |

Lab 657 the reported value for FBP excluded, has stopped the determination

Lab 1455 first reported: 414

Lab 1949 also reported values for manual distillation: 273; 348; 404; 434; 469; 519; 542 respectively



**APPENDIX 2**

z-scores of the determination of Simulated Distillation acc. to ASTM D6352 on sample #15250

| lab  | IBP   | 10%   | 30%                | 50%                 | 70%   | 90%   | FBP   |
|------|-------|-------|--------------------|---------------------|-------|-------|-------|
| 52   | 0.23  | 2.20  | <b><u>3.54</u></b> | <b><u>2.73</u></b>  | 1.87  | 0.72  | -3.63 |
| 62   | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 120  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 131  | -0.36 | -0.36 | 0.26               | 0.50                | 0.66  | 1.31  | 3.94  |
| 133  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 140  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 150  | -0.43 | -0.56 | -0.02              | 0.10                | 0.12  | 0.46  | 1.66  |
| 158  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 171  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 228  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 311  | -0.26 | -0.96 | -0.49              | -0.12               | -0.07 | -0.08 | 0.04  |
| 313  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 317  | -0.12 | -1.35 | -2.16              | <b><u>-1.87</u></b> | -1.63 | -0.88 | -1.06 |
| 323  | 1.61  | 1.77  | 0.79               | 0.28                | -0.31 | 0.03  | -1.02 |
| 333  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 334  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 336  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 337  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 340  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 356  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 369  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 402  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 445  | 0.10  | -0.05 | 0.50               | 0.50                | -0.89 | -2.18 | ----  |
| 447  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 463  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 494  | -0.35 | 0.11  | 0.41               | 0.15                | 0.59  | 0.54  | 2.62  |
| 621  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 657  | 0.65  | 2.40  | 0.22               | -0.99               | -2.21 | -2.34 | -4.15 |
| 663  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 704  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 732  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 743  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 781  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 784  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 785  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 791  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 874  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 875  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 994  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 995  | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 1065 | -0.32 | -1.23 | -0.26              | 0.15                | 0.90  | 1.23  | 4.23  |
| 1134 | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 1297 | -0.14 | -1.00 | -0.73              | -0.82               | -0.97 | -0.64 | -2.73 |
| 1365 | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 1379 | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 1397 | -0.49 | -0.36 | 1.64               | 2.07                | 1.68  | 1.39  | -0.54 |
| 1455 | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 1510 | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 1543 | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 1556 | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 1635 | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 1677 | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 1857 | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 1881 | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 1949 | -0.12 | -0.60 | -0.16              | 0.06                | 0.28  | 0.43  | 0.65  |
| 1986 | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 1995 | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 6020 | ---   | ----  | ----               | ----                | ----  | ----  | ----  |
| 6021 | ---   | ----  | ----               | ----                | ----  | ----  | ----  |

z-scores underlined and bold belong to the statistical outliers acc. to Dixon outlier test.

## z-scores of the determination of Distillation according to ASTM D1160 on sample #15250

| lab  | IBP   | 10%   | 30%         | 50%   | 70%   | 90%   | FBP   |
|------|-------|-------|-------------|-------|-------|-------|-------|
| 52   | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 62   | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 120  | 0.64  | -2.45 | -0.23       | -0.18 | -0.27 | -0.62 | -0.62 |
| 131  | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 133  | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 140  | 2.56  | 1.03  | 1.60        | 1.22  | 1.47  | 0.62  | 1.83  |
| 150  | 0.70  | 1.87  | 1.27        | 0.93  | 1.72  | 0.96  | 2.00  |
| 158  | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 171  | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 228  | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 311  | 0.70  | 1.70  | -0.80       | -0.97 | -0.42 | 0.16  | 0.56  |
| 313  | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 317  | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 323  | 0.64  | -0.42 | -0.27       | -1.00 | -0.56 | 0.21  | ----  |
| 333  | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 334  | 2.07  | 2.08  | <b>5.75</b> | 2.04  | 2.04  | 0.91  | -0.06 |
| 336  | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 337  | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 340  | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 356  | 1.09  | 2.32  | 1.54        | 1.36  | 1.61  | 0.33  | 1.66  |
| 369  | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 402  | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 445  | -0.54 | -1.19 | -0.78       | -0.93 | -0.67 | 0.14  | -0.84 |
| 447  | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 463  | -0.43 | -1.40 | -0.93       | -1.72 | -2.27 | -1.29 | -1.12 |
| 494  | 0.92  | 0.56  | 0.61        | 0.69  | 1.15  | 0.46  | -0.60 |
| 621  | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 657  | -0.09 | -0.91 | -1.15       | -1.48 | -1.99 | -1.67 | -3.10 |
| 663  | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 704  | -0.09 | 0.56  | 0.61        | 0.21  | 0.30  | -0.04 | 0.23  |
| 732  | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 743  | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 781  | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 784  | -1.57 | -2.87 | -0.93       | -0.03 | 1.15  | 0.83  | -0.29 |
| 785  | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 791  | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 874  | -0.89 | -0.26 | -0.27       | -1.24 | -1.42 | -0.04 | 0.23  |
| 875  | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 994  | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 995  | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 1065 | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 1134 | 0.13  | 0.23  | 0.39        | -0.03 | -0.56 | 0.21  | 0.13  |
| 1297 | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 1365 | 0.58  | 0.07  | 0.61        | 0.69  | 0.90  | 0.69  | -0.30 |
| 1379 | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 1397 | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 1455 | 0.30  | 2.52  | <b>3.19</b> | 1.65  | 1.44  | 1.21  | -0.50 |
| 1510 | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 1543 | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 1556 | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 1635 | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 1677 | -2.30 | -0.58 | -0.49       | -0.76 | -1.70 | -0.29 | 0.65  |
| 1857 | -0.77 | -0.75 | 0.17        | 0.21  | -0.56 | -0.92 | -1.74 |
| 1881 | 0.36  | -0.91 | -0.27       | -0.28 | 0.01  | -0.29 | 0.65  |
| 1949 | -1.28 | -1.24 | -1.15       | -0.52 | 0.01  | 0.21  | 0.13  |
| 1986 | -0.09 | 0.23  | 0.17        | -0.52 | -1.70 | -0.04 | 0.23  |
| 1995 | ----  | ----  | ----        | ----  | ----  | ----  | ----  |
| 6020 | -0.94 | -0.09 | -0.49       | -0.28 | 0.30  | -1.29 | -1.12 |
| 6021 | -1.68 | -0.09 | 0.83        | 0.93  | 0.01  | -0.42 | -1.12 |

z-scores underlined and bold belong to the statistical outliers acc. to Rosner outlier test.

**APPENDIX 3****Number of participants per country**

2 labs in CANADA  
7 labs in UNITED STATES OF AMERICA  
1 lab in TOGO  
4 labs in NETHERLANDS  
1 lab in BELGIUM  
5 labs in FRANCE  
1 lab in MALTA  
1 lab in LATVIA  
1 lab in ROMANIA  
4 labs in UNITED KINGDOM  
2 labs in SWEDEN  
1 lab in GERMANY  
1 lab in INDONESIA  
1 lab in SINGAPORE  
1 lab in THAILAND  
2 labs in UKRAINE  
1 lab in KAZAKHSTAN  
12 labs in RUSSIAN FEDERATION  
1 lab in AZERBAIJAN  
2 labs in GEORGIA  
1 lab in ISRAEL  
1 lab in GREECE  
2 labs in CROATIA  
1 lab in SUDAN  
1 lab in ESTONIA  
1 lab in LITHUANIA  
1 lab in EGYPT

**APPENDIX 4****Abbreviations**

|          |                                                                |
|----------|----------------------------------------------------------------|
| C        | = final result after checking of first reported suspect 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                           |
| ex       | = test result excluded from calculations                       |
| E        | = probably error in calculations                               |
| W        | = withdrawn result on request of participant                   |
| n.a.     | = not applicable                                               |
| SDS      | = Safety Data Sheet                                            |

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