# Results of Proficiency Test
## Vacuum Gasoil
## November 2013

**CONTENTS**

Appendices:

## 1    INTRODUCTION

On request of several participants, the Institute for Interlaboratory Studies decided to organise a new proficiency test for the analysis of Vacuum Gasoil (VGO) during the annual proficiency testing program 2013/2014. In this interlaboratory study 34 laboratories in 20 different countries have participated. See appendix 3 for the number of participants per country. In this report, the results of the 2013 VGO proficiency test are presented and discussed. This report is also electronically available through the iis internet site 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 #13236) that was purchased from a local supplier. The analyses for fit-for-use and homogeneity were subcontracted. Participants were requested 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 on IEC/ISO17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

### 2.2    PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organization, Statistics and Evaluation' of January 2010 (iis-protocol, version 3.2), which can be downloaded from 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 75 litre bulk material was transferred after homogenizing into 61 brown glass bottles of 1 litre (labelled #13236). The homogeneity of the subsamples #13236 was checked by determination of Density @15°C in accordance with ASTM D4052:11 on 8 stratified randomly selected samples.

|  | Density @ 15 °C in $kg/m^3$ |
|---|---|
| Sample #13236-1 | 930.9 |
| Sample #13236-2 | 930.9 |
| Sample #13236-3 | 930.9 |
| Sample #13236-4 | 930.9 |
| Sample #13236-5 | 930.9 |
| Sample #13236-6 | 930.9 |
| Sample #13236-7 | 930.9 |
| Sample #13236-8 | 930.9 |

Table 1: homogeneity test results of subsamples #13236

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 @ 15 °C in $kg/m^3$ |
|---|---|
| r (sample #13236) | 0.00 |
| reference test | ISO12185:96 |
| 0.3 x R(reference test) | 0.15 |

Table 2: evaluation of the repeatabilities of the subsamples #13236

The calculated repeatability is less than 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneity of the subsamples #13236 was assumed.

To each of the participating laboratories, 1 sample of 1 L in a brown glass bottle (labelled #13236) was sent on Nov 13, 2013.

## 2.5   ANALYSES

The participants were requested to determine on sample #13236: Acid Number (Total), Aniline Point, Asphaltenes, Density @ 15°C, Distillation (IBP, 10% rec, 30% rec, 50% rec, 70% rec, 90% rec and FBP), Flash Point PMcc, Kinematic Viscosity @ 50°C and @ 100°C, Micro Carbon Residue, Nitrogen, Total Sulphur Content and Metals (Arsenic, Calcium, Copper, Nickel, Iron, Silicon, Sodium, Vanadium).
To get comparable results a detailed report form, on which the units were prescribed as well as some of the required standards and a letter of instructions were prepared and made available for download on the iis website (www.iisnl.com). A SDS and a form to confirm receipt of the samples were added to the sample package.

## 3    RESULTS

During four weeks after sample despatch, the results of the individual laboratories weregathered. The original data are tabulated per determination in the appendix of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder fax was sent to those laboratories that had not reported results at that moment.

Shortly after the deadline, the available 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 results. Additional or corrected results are 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, version 3.2) of January 2010. 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.  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 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value, the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. 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.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

## 3.2   GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported 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 "x". Accepted data are represented as a triangle. Furthermore, Kernel Density Graphs were made. The Kernel Density is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms (see appendix 4; nos.13 and 14).

## 3.3   Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This target standard deviation was calculated from the literature reproducibility 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 the fit-for-useness of the reported test result.
The z-scor es were calculated according to:

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

The $z_{(target)}$ scores are listed in the result tables in appendix 1.

Absolute values for z<2 are very common and absolute values for z>3 are very rare. Therefore, the usual interpretation of z-scores is as follows:

$$|z| < 1 \quad \text{good}$$
$$1 < |z| < 2 \quad \text{satisfactory}$$
$$2 < |z| < 3 \quad \text{questionable}$$
$$3 < |z| \quad \text{unsatisfactory}$$

## 4    EVALUATION

In this interlaboratory study, some problems were encountered with the dispatch of the samples to laboratories in United States of America and Russia. Seven participants reported the test results after the final reporting date and one participant did not report any test results at all. Not all laboratories were able to report all analyses requested. In total 32 participants reported 474 test results. Observed were 12 outlying results, which is 2.5% of the numerical results. In proficiency studies, outlier percentages of 3% - 7.5% are quite normal.

Not all original data sets proved to have a normal distribution. Non-Gaussian distributions were found for the following determinations: Density @15°C, Flash Point PMcc, Nickel and Silicon. In these cases the statistical evaluation should be used with due care.

## 4.1    EVALUATION PER TEST

In this section, the results are discussed per test. The methods, which are used by the various laboratories, are taken into account for explaining the observed differences when possible and applicable. These methods are also in the tables together with the original data. The abbreviations, used in these tables, are listed in appendix 3.
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 intented for residual fuel oil and blending components. 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. The precision data of this method will be determined in 2014.

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

Aniline Point:      This determination was very problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements of ASTM D611:12.

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.

Density @15ºC:    This determination was not problematic. No statistical outliers were observed and the calculated reproducibility is in full 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:13.

Kin.Visco.@ 50°C: This determination may be 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 D445:12 for residual oils.

Kin.Visco.@ 100°C: This determination may be 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 D445:12 for residual oils.

Micro Carbon Residue: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with ASTM D4530:11 and ISO10370:93. It should be noted that the precision data for both methods has been obtained for the 2 ml capacity vials. Differences in vial size and/or sample intake may (partically) be the cause of the large spread.

Nitrogen: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with ASTM D5762:12.

Total Sulphur: 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 D4294:10.

**Metals:**

Arsenic (As): Only two participants reported a numerical result. Therefore no significant conclusions were drawn.

Calcium (Ca): 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 requirements of IP501:05.

Copper (Cu): All results, except one, were near or below the application range of method IP-PM-CW (0.1 – 10 mg/kg). Therefore no statistical conclusions were drawn.

Nickel (Ni): This determination may not be problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of IP501:05.

Iron (Fe): This determination may not be problematic. One statistical outlier was observed. However, the calculated reproducibility after rejection of the statistical outlier is in good agreement with the requirements of IP501:05.

Silicon (Si):      The consensus value was far below the application range of method IP501:05 (10 – 250 mg/kg). Therefore no statistical conclusions were drawn.

Sodium (Na):      This determination may not be 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.

Vanadium (V):      This determination may not be problematic. No statistical outliers were observed and the calculated reproducibility is in good agreement with the requirements of IP501:05.

Sim. Dist.      The Simulated Distillation according to ASTM D6352:12 was partly problematic. No statistical outliers were observed. The calculated reproducibilities were in agreement for IBP, 10% rec, 90% rec and FBP with the requirements of ASTM D6352:12. For 30% rec, 50% rec and 70% the calculated reproducibilities were not in agreement with the requirements of ASTM D6352:12.

Distillation
Acc. D1160      The distillation according to ASTM D1160:06 was not problematic. No statistical outliers were observed. The calculated reproducibilities were in agreement for 10% rec, 30% rec, 50% rec, 70% rec, 90% rec and FBP with the requirements of ASTM D1160:06. Only for IBP the calculated reproducibility was not at all in agreement with the requirements of ASTM D1160:06.
Note: Care should be taken in pressure and cooling temperature.

**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 | 23 | 1.26 | 0.18 | 0.32 |
| Aniline Point | $^{o}$C | 18 | 81.06 | 3.44 | 1.00 |
| Asphaltenes | %M/M | 16 | 0.097 | 0.156 | (0.019) |
| Density @ 15 °C | kg/m$^3$ | 31 | 931.2 | 1.46 | 1.50 |
| Flash Point PMcc | $^{o}$C | 22 | 203.7 | 7.6 | 10.0 |
| Kinematic Viscosity @ 50 °C | cSt | 30 | 68.48 | 1.31 | 5.07 |
| Kinematic Viscosity @ 100 °C | cSt | 25 | 10.30 | 0.20 | 0.73 |
| Micro Carbon Residue | %M/M | 23 | 0.45 | 0.22 | 0.16 |
| Nitrogen | %M/M | 20 | 0.17 | 0.04 | 0.05 |
| Total Sulphur Content | %M/M | 31 | 0.69 | 0.05 | 0.06 |
| Metals | | | | | |
| -Arsenic (As) | mg/kg | 5 | <1 | n.a | n.a |
| -Calcium (Ca) | mg/kg | 13 | 0.32 | 0.48 | 0.30 |
| -Copper (Cu) | mg/kg | 15 | <0.1 | n.a | n.a |
| -Nickel (Ni) | mg/kg | 17 | 0.16 | 0.23 | 0.61 |
| -Iron (Fe) | mg/kg | 16 | 0.41 | 0.34 | 0.57 |
| -Silicon (Si) | mg/kg | 12 | 1.00 | 2.60 | (0.33) |
| -Sodium (Na) | mg/kg | 17 | 0.57 | 0.83 | 0.78 |
| -Vanadium (V) | mg/kg | 16 | 0.37 | 0.25 | 0.93 |
| Distillation D6352 | | | | | |
| -IBP | $^{o}$C | 10 | 282.0 | 27.2 | 49.1 |
| -10% rec | $^{o}$C | 10 | 381.5 | 6.1 | 7.1 |
| -30% rec | $^{o}$C | 10 | 423.1 | 7.4 | 5.9 |
| -50% rec | $^{o}$C | 10 | 453.2 | 8.5 | 6.4 |
| -70% rec | $^{o}$C | 10 | 485.1 | 8.7 | 7.2 |
| -90% rec | $^{o}$C | 10 | 525.2 | 7.6 | 10.5 |
| -FBP | $^{o}$C | 10 | 590.2 | 29.9 | 38.1 |
| Distillation D1160 | | | | | |
| -IBP | $^{o}$C | 6 | 310.6 | 73.0 | 49.4 |
| -10% rec | $^{o}$C | 6 | 400.3 | 12.0 | 16.4 |
| -30% rec | $^{o}$C | 6 | 431.1 | 6.1 | 10.7 |
| -50% rec | $^{o}$C | 6 | 456.8 | 8.6 | 10.5 |
| -70% rec | $^{o}$C | 6 | 484.8 | 8.6 | 9.0 |
| -90% rec | $^{o}$C | 6 | 525.8 | 7.3 | 22.5 |
| -FBP | $^{o}$C | 6 | 558.4 | 19.3 | 26.9 |

Table 3: reproducibilities of results of sample #13236
results between brackets should 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.
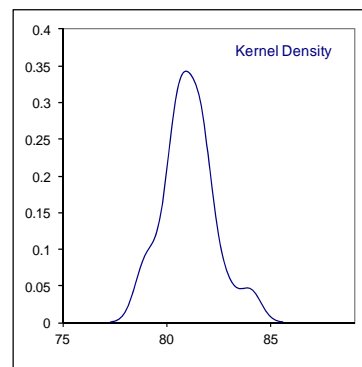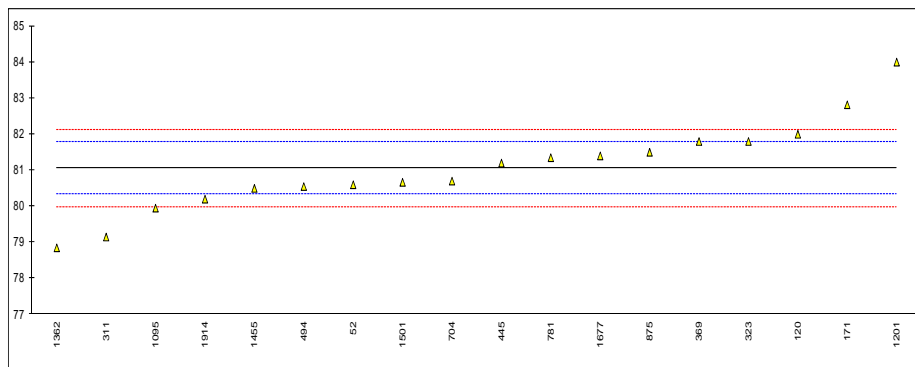
## APPENDIX 1

Determination of Acid Number (Total) on sample #13236; results in mg KOH/g

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | D664 | 1.3 | | 0.35 | |
| 62 | D664 | 1.27 | | 0.08 | |
| 120 | D664 | 1.191 | | -0.61 | |
| 131 | D664 | 1.18 | | -0.71 | |
| 171 | D664 | 1.24 | | -0.18 | |
| 311 | D664 | 1.34 | | 0.70 | |
| 313 | | ----- | | ----- | |
| 323 | D664 | 1.32 | | 0.52 | |
| 333 | | ----- | | ----- | |
| 334 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 369 | D664 | 1.198 | | -0.55 | |
| 445 | D664 | 1.327 | | 0.58 | |
| 494 | D664 | 1.315 | | 0.48 | |
| 657 | D664 | 1.38 | | 1.05 | |
| 704 | D664 | 1.19 | | -0.62 | |
| 781 | D664 | 1.315 | | 0.48 | |
| 785 | | ----- | | ----- | |
| 791 | | ----- | | ----- | |
| 875 | D664 | 1.22 | | -0.36 | |
| 966 | | ----- | | ----- | |
| 994 | D664 | 1.21 | | -0.44 | |
| 995 | D664 | 1.207 | | -0.47 | |
| 1065 | | ----- | | ----- | |
| 1066 | D664 | 1.23 | | -0.27 | |
| 1095 | D664 | 1.267 | | 0.06 | |
| 1201 | D664 | 1.35 | | 0.79 | |
| 1340 | | ----- | | ----- | |
| 1362 | D664 | 1.2195 | | -0.36 | |
| 1455 | D664 | 1.34 | | 0.70 | |
| 1501 | D664 | 1.180 | | -0.71 | |
| 1543 | | ----- | | ----- | |
| 1677 | D664 | 1.20 | | -0.53 | |
| 1914 | D664 | <0.05 | | ----- | |
| | normality | OK | | | |
| | n | 23 | | | |
| | outliers | 0 | | | |
| | mean (n) | 1.2604 | | | |
| | st.dev. (n) | 0.06429 | | | |
| | R(calc.) | 0.1800 | | | |
| | R(D664:11a) | 0.3187 | | | |





Kernel Density
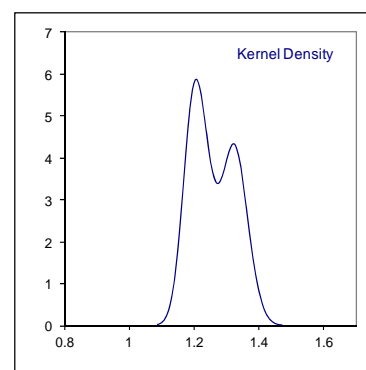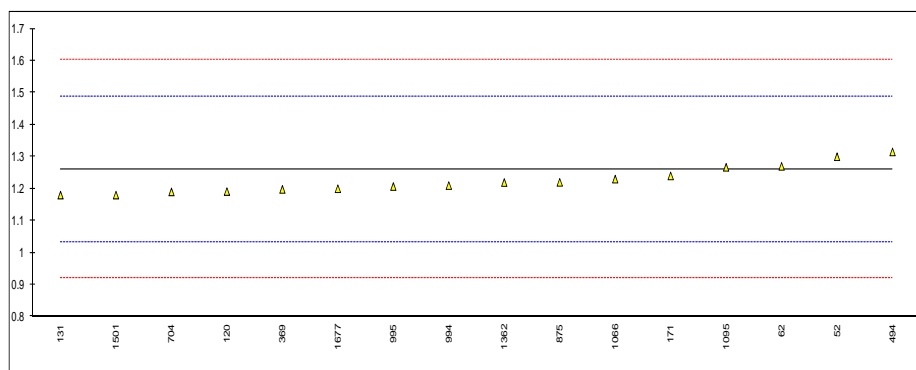
## Determination of Aniline Point on sample #13236; results in °C

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | D611 | 80.60 | | -1.28 | |
| 62 | | ----- | | ----- | |
| 120 | D611 | 82.0 | | 2.64 | |
| 131 | | ----- | | ----- | |
| 171 | D611 | 82.817 | | 4.93 | |
| 311 | D611 | 79.15 | | -5.34 | |
| 313 | | ----- | | ----- | |
| 323 | D611 | 81.8 | | 2.08 | |
| 333 | | ----- | | ----- | |
| 334 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 369 | D611 | 81.80 | | 2.08 | |
| 445 | IP2 | 81.20 | | 0.40 | |
| 494 | D611 | 80.55 | | -1.42 | |
| 657 | | ----- | | ----- | |
| 704 | D611 | 80.700 | | -1.00 | |
| 781 | D611 | 81.35 | | 0.82 | |
| 785 | | ----- | | ----- | |
| 791 | | ----- | | ----- | |
| 875 | D611 | 81.50 | | 1.24 | |
| 966 | | ----- | | ----- | |
| 994 | | ----- | | ----- | |
| 995 | | ----- | | ----- | |
| 1065 | | ----- | | ----- | |
| 1066 | | ----- | | ----- | |
| 1095 | D611 | 79.95 | | -3.10 | |
| 1201 | D611 | 84.0 | | 8.24 | |
| 1340 | | ----- | | ----- | |
| 1362 | D611 | 78.850 | | -6.18 | |
| 1455 | D611 | 80.50 | | -1.56 | |
| 1501 | D611 | 80.667 | | -1.09 | |
| 1543 | | ----- | | ----- | |
| 1677 | D611 | 81.4 | | 0.96 | |
| 1914 | D611 | 80.20 | | -2.40 | |

|  |  |
|---|---|
| normality | OK |
| n | 18 |
| outliers | 0 |
| mean (n) | 81.057 |
| st.dev. (n) | 1.2294 |
| R(calc.) | 3.442 |
| R(D611:12) | 1.000 |

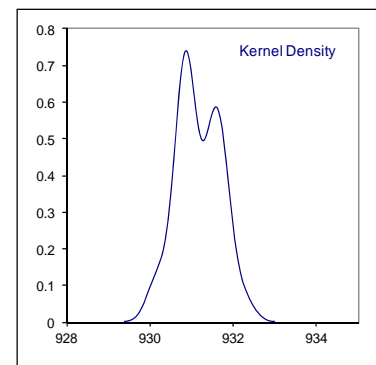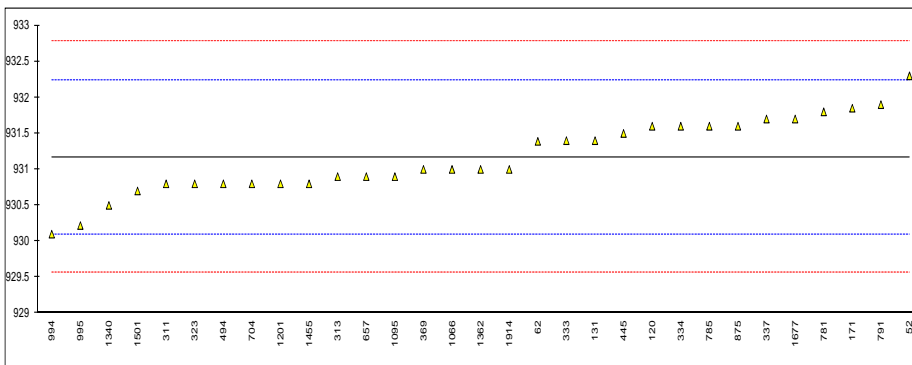Determination of Asphaltenes on sample #13236; results in %M/M

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | IP143 | 0.043 | | ----- | |
| 62 | | ----- | | ----- | |
| 120 | IP143 | 0.04 | | ----- | |
| 131 | | ----- | | ----- | |
| 171 | IP143 | 0.189 | | ----- | |
| 311 | | ----- | | ----- | |
| 313 | | ----- | | ----- | |
| 323 | IP143 | <0.50 | | ----- | |
| 333 | | ----- | | ----- | |
| 334 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 369 | | ----- | | ----- | |
| 445 | IP143 | 0.17 | | ----- | |
| 494 | IP143 | 0.055 | | ----- | |
| 657 | IP143 | 0.17 | | ----- | |
| 704 | IP143 | 0.099 | | ----- | |
| 781 | INH-642 | 0.0779 | | ----- | |
| 785 | | ----- | | ----- | |
| 791 | | ----- | | ----- | |
| 875 | | ----- | | ----- | |
| 966 | | ----- | | ----- | |
| 994 | IP143 | <0.5 | | ----- | |
| 995 | IP143 | <0.5 | | ----- | |
| 1065 | UOP614 | 0.0250 | | ----- | |
| 1066 | IP143 | 0.10 | | ----- | |
| 1095 | IP143 | 0.10 | | ----- | |
| 1201 | IP143 | 0.11 | | ----- | |
| 1340 | D6560 | 0.185 | | ----- | |
| 1362 | IP143 | 0.049 | | ----- | |
| 1455 | IP143 | 0.30 | G(0.05) | ----- | |
| 1501 | IP143 | 0.037 | | ----- | |
| 1543 | | ----- | | ----- | |
| 1677 | | ----- | W | ----- | result withdrawn, first reported: 1.62 |
| 1914 | IP143 | 0.10 | | ----- | |

| | | | |
|---|---|---|---|
| normality | OK | | |
| n | 16 | | |
| outliers | 1 | | |
| mean (n) | 0.0969 | | |
| st.dev. (n) | 0.05565 | | |
| R(calc.) | 0.1558 | | |
| R(IP143:04) | (0.0194) | | precision is applicable between 0.50% M/M and 30.0% M/M |
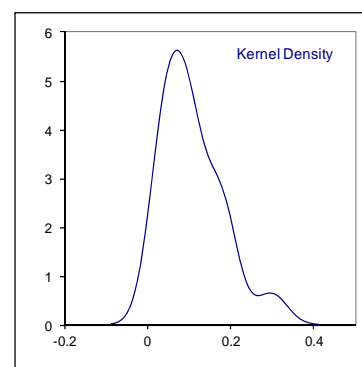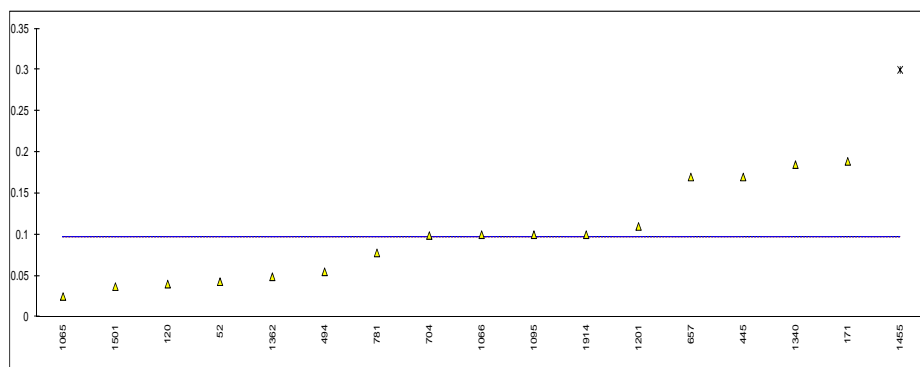
## Determination of Density @ 15 °C on sample #13236; results in kg/m$^3$

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 52 | D4052 | 932.3 | | 2.10 | |
| 62 | D4052 | 931.39 | | 0.41 | |
| 120 | ISO12185 | 931.6 | | 0.80 | |
| 131 | D4052 | 931.4 | | 0.42 | |
| 171 | D1298 | 931.85 | | 1.26 | |
| 311 | ISO12185 | 930.8 | | -0.70 | |
| 313 | ISO12185 | 930.9 | | -0.51 | |
| 323 | ISO12185 | 930.8 | | -0.70 | |
| 333 | ISO12185 | 931.4 | | 0.42 | |
| 334 | ISO12185 | 931.6 | | 0.80 | |
| 337 | ISO12185 | 931.7 | | 0.98 | |
| 369 | ISO12185 | 931.0 | | -0.32 | |
| 445 | IP365 | 931.5 | | 0.61 | |
| 494 | ISO12185 | 930.8 | | -0.70 | |
| 657 | ISO12185 | 930.9 | | -0.51 | |
| 704 | ISO12185 | 930.8 | | -0.70 | |
| 781 | ISO12185 | 931.8 | | 1.17 | |
| 785 | D1298 | 931.6 | | 0.80 | |
| 791 | D1298 | 931.9 | | 1.36 | |
| 875 | D1298 | 931.6 | | 0.80 | |
| 966 | | ----- | | ----- | |
| 994 | ISO12185 | 930.1 | | -2.00 | |
| 995 | ISO12185 | 930.22 | | -1.78 | |
| 1065 | | ----- | | ----- | |
| 1066 | ISO12185 | 931.0 | | -0.32 | |
| 1095 | ISO12185 | 930.9 | | -0.51 | |
| 1201 | ISO12185 | 930.8 | | -0.70 | |
| 1340 | ISO3675 | 930.5 | | -1.26 | |
| 1362 | ISO12185 | 931.00 | | -0.32 | |
| 1455 | ISO12185 | 930.8 | | -0.70 | |
| 1501 | ISO12185 | 930.7 | | -0.88 | |
| 1543 | | ----- | | ----- | |
| 1677 | D4052 | 931.7 | | 0.98 | |
| 1914 | ISO12185 | 931.0 | | -0.32 | |

| | |
|---|---|
| normality | not OK |
| n | 31 |
| outliers | 0 |
| mean (n) | 931.17 |
| st.dev. (n) | 0.520 |
| R(calc.) | 1.455 |
| R(ISO12185:96) | 1.500 |

Determination of Flash Point PMcc on sample #13236; results in °C

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | D93-B | 202.0 | | -0.49 | |
| 62 | D93-B | >150 | | ----- | |
| 120 | D93-B | 202 | | -0.49 | |
| 131 | | ----- | | ----- | |
| 171 | D93-B | 192.8 | G(0.05) | -3.06 | |
| 311 | D93 | >180.0 | | ----- | |
| 313 | D93 | >180.0 | | ----- | |
| 323 | D93-B | 151.0 | G(0.01) | -14.77 | |
| 333 | | ----- | | ----- | |
| 334 | | ----- | | ----- | |
| 337 | D93-A | 201.5 | | -0.63 | |
| 369 | D93-B | 200.0 | | -1.05 | |
| 445 | D93-A | 204.0 | | 0.07 | |
| 494 | D93-A | >200 | | ----- | |
| 657 | D93-B | 204.0 | | 0.07 | |
| 704 | D93-B | 198.7 | | -1.41 | |
| 781 | D93-B | 204.0 | | 0.07 | |
| 785 | D93-B | 205.0 | | 0.35 | |
| 791 | D93-B | 204.0 | | 0.07 | |
| 875 | D93-B | 204.5 | | 0.21 | |
| 966 | | ----- | | ----- | |
| 994 | D93-B | 204.0 | | 0.07 | |
| 995 | D93-B | 204.5 | | 0.21 | |
| 1065 | D93-A | >200 | | ----- | |
| 1066 | D93 | 205.0 | | 0.35 | |
| 1095 | D93-B | 210.0 | | 1.75 | |
| 1201 | D93-B | 205 | | 0.35 | |
| 1340 | D93-B | 205 | | 0.35 | |
| 1362 | D93 | 204.00 | | 0.07 | |
| 1455 | D93-A | 203.0 | | -0.21 | |
| 1501 | D93-B | 206.0 | | 0.63 | |
| 1543 | | ----- | | ----- | |
| 1677 | D93-A | 208.0 | | 1.19 | |
| 1914 | D93-B | 198.0 | | -1.61 | |

| | | | | | Only D93-A:13 data |
|---|---|---|---|---|---|
| normality | not OK | | | | OK |
| n | 22 | | | | 6 |
| outliers | 2 | | | | 0 |
| mean (n) | 203.74 | | | | 204.13 |
| st.dev. (n) | 2.711 | | | | 2.780 |
| R(calc.) | 7.59 | | | | 7.78 |
| R(D93-B:13) | 10.00 | | | | 14.00 |
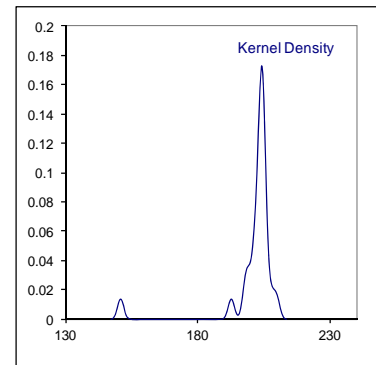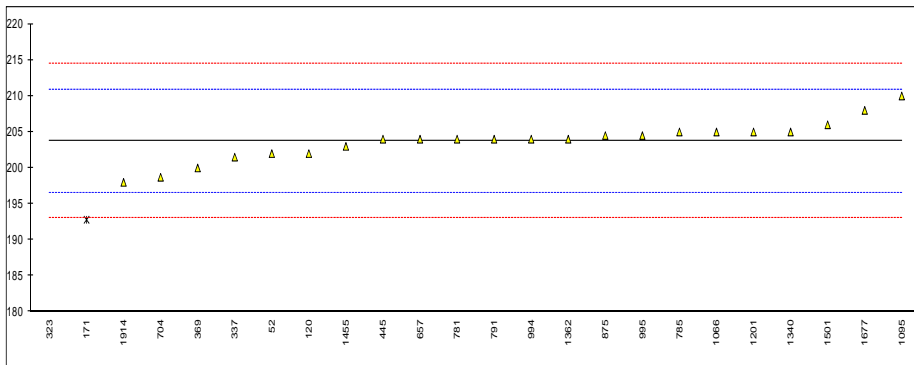
## Determination of Kinematic Viscosity @ 50°C on sample #13236; results in cSt

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | D445 | 68.62 | | 0.08 | |
| 62 | D445 | 67.904 | | -0.32 | |
| 120 | D445 | 68.55 | C | 0.04 | first reported: 63.25 |
| 131 | D445 | 68.09 | | -0.22 | |
| 171 | D445 | 68.65 | | 0.09 | |
| 311 | D445 | 68.48 | | 0.00 | |
| 313 | D445 | 68.43 | | -0.03 | |
| 323 | D445 | 68.12 | | -0.20 | |
| 333 | D445 | 68.19 | | -0.16 | |
| 334 | D445 | 68.409 | | -0.04 | |
| 337 | | ----- | | ----- | |
| 369 | D445 | 67.711 | | -0.43 | |
| 445 | D445 | 70.05 | | 0.87 | |
| 494 | D445 | 68.34 | | -0.08 | |
| 657 | D445 | 68.97 | | 0.27 | |
| 704 | D445 | 69.080 | | 0.33 | |
| 781 | D445 | 68.41 | | -0.04 | |
| 785 | D445 | 68.41 | | -0.04 | |
| 791 | D445 | 68.80 | | 0.18 | |
| 875 | D445 | 68.34 | | -0.08 | |
| 966 | | ----- | | ----- | |
| 994 | D445 | 68.47 | | -0.01 | |
| 995 | D445 | 68.36 | | -0.07 | |
| 1065 | D445 | 68.17 | | -0.17 | |
| 1066 | D445 | 68.89 | | 0.23 | |
| 1095 | D445 | 68.97 | | 0.27 | |
| 1201 | D445 | 67.98 | | -0.28 | |
| 1340 | ISO3104 | 67.735 | | -0.41 | |
| 1362 | D445 | 68.248 | | -0.13 | |
| 1455 | D445 | 68.30 | | -0.10 | |
| 1501 | D7042 | 65.840 | G(0.01) | -1.46 | |
| 1543 | | ----- | | ----- | |
| 1677 | D445 | 68.96 | | 0.26 | |
| 1914 | D445 | 68.84 | | 0.20 | |

| | |
|---|---|
| normality | OK |
| n | 30 |
| outliers | 1 |
| mean (n) | 68.483 |
| st.dev. (n) | 0.4674 |
| R(calc.) | 1.309 |
| R(D445:12) | 5.068 |
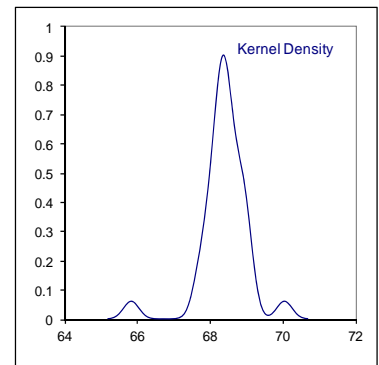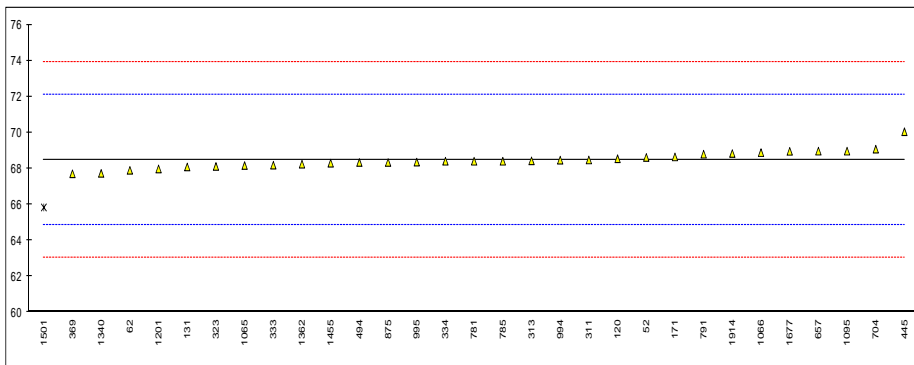




Kernel Density

Determination of Kinematic Viscosity @ 100$^o$C on sample #13236; results in cSt

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | D445 | 10.23 | | -0.27 | |
| 62 | | ----- | | ----- | |
| 120 | | ----- | | ----- | |
| 131 | D445 | 10.27 | | -0.11 | |
| 171 | D445 | 10.28 | | -0.07 | |
| 311 | D445 | 10.30 | | 0.00 | |
| 313 | D445 | 10.19 | | -0.42 | |
| 323 | D445 | 10.17 | | -0.50 | |
| 333 | D445 | 10.27 | | -0.11 | |
| 334 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 369 | D445 | 10.216 | | -0.32 | |
| 445 | D445 | 10.33 | | 0.12 | |
| 494 | D445 | 10.40 | | 0.38 | |
| 657 | D445 | 10.40 | | 0.38 | |
| 704 | D445 | 10.378 | | 0.30 | |
| 781 | D445 | 10.29 | | -0.04 | |
| 785 | D445 | 10.30 | | 0.00 | |
| 791 | | ----- | | ----- | |
| 875 | D445 | 10.31 | | 0.04 | |
| 966 | | ----- | | ----- | |
| 994 | D445 | 10.25 | | -0.19 | |
| 995 | D445 | 10.28 | | -0.07 | |
| 1065 | D445 | 10.28 | | -0.07 | |
| 1066 | D445 | 10.29 | | -0.04 | |
| 1095 | | ----- | | ----- | |
| 1201 | D445 | 10.255 | | -0.17 | |
| 1340 | ISO3104 | 10.339 | | 0.15 | |
| 1362 | | ----- | | ----- | |
| 1455 | D445 | 10.29 | | -0.04 | |
| 1501 | D7042 | 10.342 | | 0.16 | |
| 1543 | | ----- | | ----- | |
| 1677 | D445 | 10.50 | | 0.77 | |
| 1914 | D445 | 10.33 | | 0.12 | |

| | | |
|---|---|---|
| normality | OK | |
| n | 25 | |
| outliers | 0 | |
| mean (n) | 10.300 | |
| st.dev. (n) | 0.0707 | |
| R(calc.) | 0.198 | |
| R(D445:12) | 0.732 | |
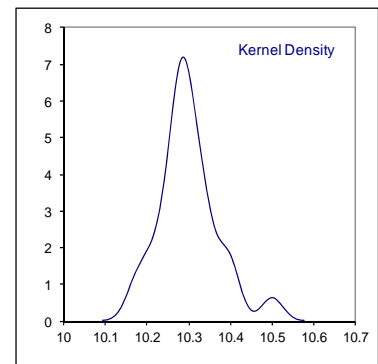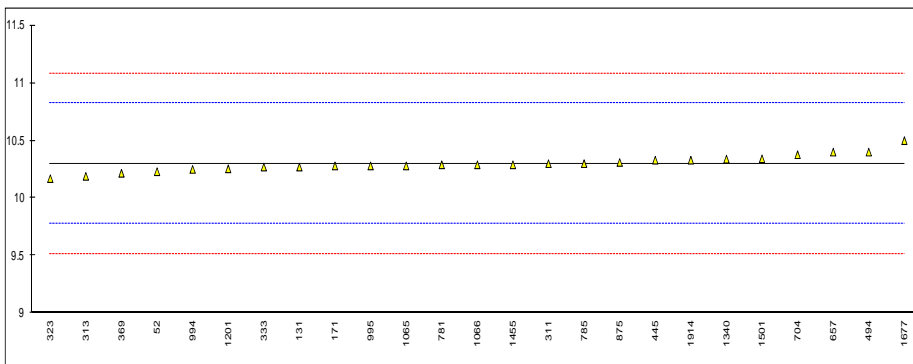
## Determination of Micro Carbon Residue on sample #13236; results in %M/M

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | D4530 | 0.40 | | -0.93 | |
| 62 | D4530 | 0.978 | G(0.01) | 9.08 | |
| 120 | D4530 | 0.44 | | -0.24 | |
| 131 | | ----- | | ----- | |
| 171 | D4530 | 0.3702 | | -1.45 | |
| 311 | D4530 | 0.39 | | -1.11 | |
| 313 | | ----- | | ----- | |
| 323 | D4530 | 0.43 | | -0.41 | |
| 333 | D4530 | 0.53 | | 1.32 | |
| 334 | ISO10370 | 0.372 | | -1.42 | |
| 337 | | ----- | | ----- | |
| 369 | D4530 | 0.546 | | 1.60 | |
| 445 | D4530 | 0.48 | | 0.45 | |
| 494 | D4530 | 0.548 | | 1.63 | |
| 657 | D4530 | 0.43 | | -0.41 | |
| 704 | D4530 | 0.567 | | 1.96 | |
| 781 | D4530 | 0.43 | | -0.41 | |
| 785 | | ----- | | ----- | |
| 791 | | ----- | | ----- | |
| 875 | D4530 | 0.41 | | -0.76 | |
| 966 | | ----- | | ----- | |
| 994 | | ----- | | ----- | |
| 995 | | ----- | | ----- | |
| 1065 | | ----- | | ----- | |
| 1066 | D4530 | 0.41 | | -0.76 | |
| 1095 | D4530 | 0.44 | | -0.24 | |
| 1201 | D4530 | 0.461 | | 0.12 | |
| 1340 | ISO10370 | 0.304 | | -2.60 | |
| 1362 | D4530 | 0.641 | | 3.24 | |
| 1455 | D4530 | 0.42 | | -0.59 | |
| 1501 | D4530 | 0.520 | | 1.15 | |
| 1543 | | ----- | | ----- | |
| 1677 | D4530 | 0.50 | | 0.80 | |
| 1914 | D4530 | 0.40 | | -0.93 | |

| | |
|---|---|
| normality | OK |
| n | 23 |
| outliers | 1 |
| mean (n) | 0.454 |
| st.dev. (n) | 0.0773 |
| R(calc.) | 0.216 |
| R(D4530:11) | 0.162          Compare R(ISO10370) = 0.145 |
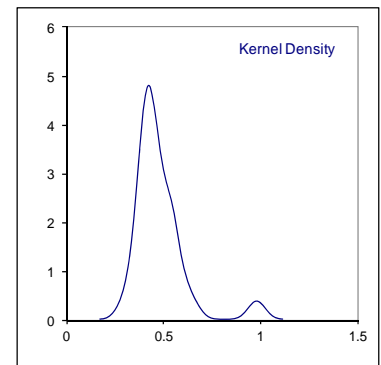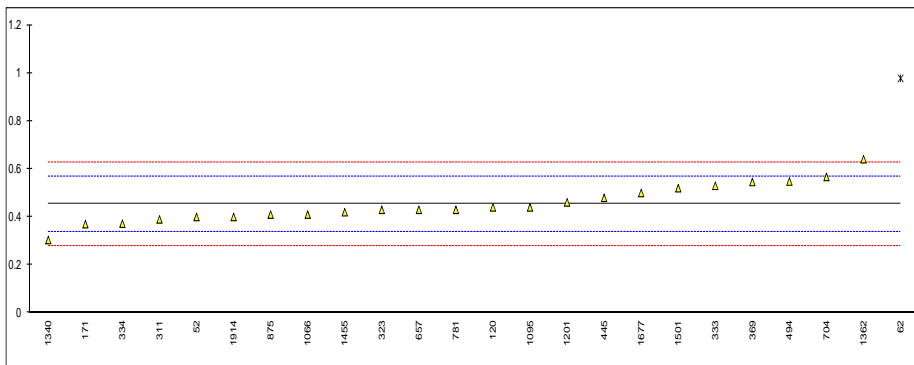
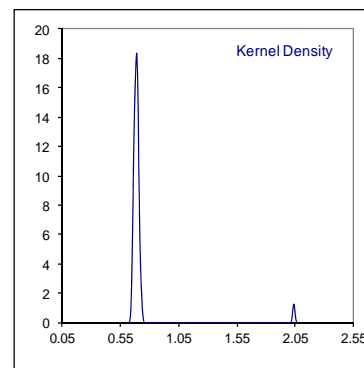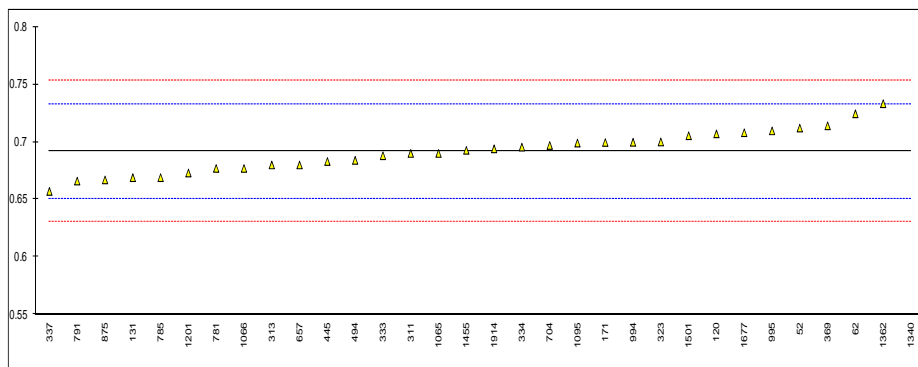## Determination of Nitrogen on sample #13236; results in %M/M

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | D5762 | 0.1720 | | -0.06 | |
| 62 | | ----- | | ----- | |
| 120 | D5762 | 0.1866 | | 0.83 | |
| 131 | D4629 | 0.1710 | C | -0.12 | first reported: 1710 %M/M |
| 171 | D5762 | 0.1551 | | -1.08 | |
| 311 | D5762 | 0.19 | | 1.04 | |
| 313 | | ----- | | ----- | |
| 323 | D5762 | 0.1705 | | -0.15 | |
| 333 | D5762 | 0.16 | | -0.79 | |
| 334 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 369 | D3228 | 0.173 | | 0.01 | |
| 445 | D5762 | 0.1929 | C | 1.22 | first reported: 1929 %M/M |
| 494 | D5762 | 0.1851 | | 0.74 | |
| 657 | D5762 | 0.1869 | | 0.85 | |
| 704 | D5762 | 0.1802 | | 0.44 | |
| 781 | D5762 | 0.16 | | -0.79 | |
| 785 | | ----- | | ----- | |
| 791 | | ----- | | ----- | |
| 875 | D5762 | 0.192 | | 1.16 | |
| 966 | | ----- | | ----- | |
| 994 | | ----- | | ----- | |
| 995 | | ----- | | ----- | |
| 1065 | D5762 | 0.145 | | -1.70 | |
| 1066 | D5762 | 0.178 | | 0.31 | |
| 1095 | D5762 | 0.169 | | -0.24 | |
| 1201 | D5762 | 0.2283 | G(0.01) | 3.37 | |
| 1340 | D5762 | 0.228072 | G(0.05) | 3.36 | reported 2280.72 mg/kg |
| 1362 | D3228 | 0.1518 | | -1.28 | |
| 1455 | | ----- | | ----- | |
| 1501 | | ----- | | ----- | |
| 1543 | | ----- | | ----- | |
| 1677 | D5762 | 0.163 | C | -0.60 | first reported: 1630 %M/M |
| 1914 | D5762 | 0.1760 | | 0.19 | |

| | | |
|---|---|---|
| normality | OK | |
| n | 20 | |
| outliers | 2 | |
| mean (n) | 0.173 | |
| st.dev. (n) | 0.0139 | |
| R(calc.) | 0.039 | |
| R(D5762:12) | 0.046 | |
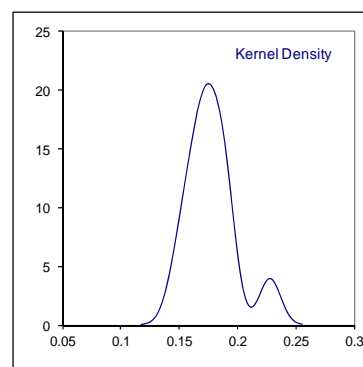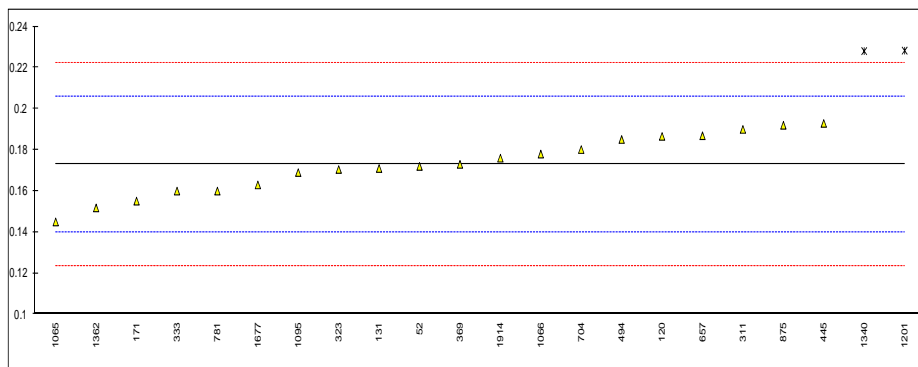
## Determination of Total Sulphur on sample #13236; results in %M/M

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | D4294 | 0.712 | | 0.99 | |
| 62 | D4294 | 0.72445 | | 1.60 | |
| 120 | D4294 | 0.707 | | 0.75 | |
| 131 | D4294 | 0.669 | | -1.11 | |
| 171 | D4294 | 0.6995 | | 0.38 | |
| 311 | D4294 | 0.69 | | -0.08 | |
| 313 | D4294 | 0.68 | | -0.57 | |
| 323 | D4294 | 0.70 | | 0.41 | |
| 333 | D2622 | 0.688 | | -0.18 | |
| 334 | D4294 | 0.6955 | | 0.19 | |
| 337 | D2622 | 0.657 | | -1.69 | |
| 369 | D2622 | 0.714 | | 1.09 | |
| 445 | IP336 | 0.683 | | -0.42 | |
| 494 | D4294 | 0.684 | | -0.37 | |
| 657 | D4294 | 0.680 | | -0.57 | |
| 704 | D4294 | 0.6969 | | 0.26 | |
| 781 | D4294 | 0.677 | | -0.72 | |
| 785 | D4294 | 0.669 | | -1.11 | |
| 791 | D4294 | 0.666 | | -1.25 | |
| 875 | D4294 | 0.667 | | -1.20 | |
| 966 | | ----- | | ----- | |
| 994 | D4294 | 0.6997 | | 0.39 | |
| 995 | D4294 | 0.7096 | | 0.88 | |
| 1065 | IP336 | 0.69 | | -0.08 | |
| 1066 | D4294 | 0.677 | | -0.72 | |
| 1095 | D4294 | 0.699 | | 0.36 | |
| 1201 | D4294 | 0.673 | | -0.91 | |
| 1340 | ISO8754 | 2.035 | G(0.01) | 65.65 | |
| 1362 | D4294 | 0.7332 | | 2.03 | |
| 1455 | D2622 | 0.6927 | | 0.05 | |
| 1501 | D4294 | 0.7054 | | 0.67 | |
| 1543 | | ----- | | ----- | |
| 1677 | D4294 | 0.708 | | 0.80 | |
| 1914 | D4294 | 0.694 | | 0.12 | |

| | | |
|---|---|---|
| normality | OK | |
| n | 31 | |
| outliers | 1 | |
| mean (n) | 0.692 | |
| st.dev. (n) | 0.0180 | |
| R(calc.) | 0.050 | |
| R(D4294:10) | 0.057 | |

Determination of Arsenic on sample #13236; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | | ----- | | ----- | |
| 62 | | ----- | | ----- | |
| 120 | | ----- | | ----- | |
| 131 | | ----- | | ----- | |
| 171 | D5708 | <0.10 | | ----- | |
| 311 | UOP986 | <0.05 | | ----- | |
| 313 | | ----- | | ----- | |
| 323 | | ----- | | ----- | |
| 333 | | ----- | | ----- | |
| 334 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 369 | | ----- | | ----- | |
| 445 | | ----- | | ----- | |
| 494 | | ----- | | ----- | |
| 657 | INH-0003 | <1 | | ----- | |
| 704 | | ----- | | ----- | |
| 781 | | ----- | | ----- | |
| 785 | | ----- | | ----- | |
| 791 | | ----- | | ----- | |
| 875 | | ----- | | ----- | |
| 966 | | ----- | | ----- | |
| 994 | | ----- | | ----- | |
| 995 | | ----- | | ----- | |
| 1065 | | ----- | | ----- | |
| 1066 | | ----- | | ----- | |
| 1095 | D5708Mod. | 0.05 | | ----- | |
| 1201 | | ----- | | ----- | |
| 1340 | | ----- | | ----- | |
| 1362 | | ----- | | ----- | |
| 1455 | | ----- | | ----- | |
| 1501 | | ----- | | ----- | |
| 1543 | | ----- | | ----- | |
| 1677 | | ----- | | ----- | |
| 1914 | in house | 0.27 | | ----- | |

| | | |
|---|---|---|
| normality | n.a | |
| n | 5 | |
| outliers | n.a | |
| mean (n) | <1 | |
| st.dev. (n) | n.a | |
| R(calc.) | n.a | |
| R(lit) | n.a | |

## Determination of Calcium on sample #13236; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | | ----- | | ----- | |
| 62 | IP470 | 0.3 | | -0.14 | |
| 120 | | ----- | | ----- | |
| 131 | | ----- | | ----- | |
| 171 | D5708 | <0.10 | | ----- | |
| 311 | D7111 | <0.5 | | ----- | |
| 313 | | ----- | | ----- | |
| 323 | INH-018 | <1 | | ----- | |
| 333 | | ----- | | ----- | |
| 334 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 369 | IP PM CW | 0.7 | | 3.54 | |
| 445 | ISO11885 | 0.24 | | -0.69 | |
| 494 | IP501 | 0.50 | | 1.70 | |
| 657 | INH-0003 | <1 | | ----- | |
| 704 | IP470 | 0.4 | | 0.78 | |
| 781 | IP PM CW | 0.2 | | -1.06 | |
| 785 | | ----- | | ----- | |
| 791 | | ----- | | ----- | |
| 875 | IP470 | 0.01 | | -2.81 | |
| 966 | | ----- | | ----- | |
| 994 | | ----- | | ----- | |
| 995 | | ----- | | ----- | |
| 1065 | | ----- | | ----- | |
| 1066 | | 0.14 | | -1.61 | |
| 1095 | D5708Mod. | 0.25 | | -0.60 | |
| 1201 | | 2.5 | G(0.01) | 20.11 | |
| 1340 | | ----- | | ----- | |
| 1362 | IP470 | 0.4 | | 0.78 | |
| 1455 | IP PM CW | 0.35 | | 0.32 | |
| 1501 | IP PM CW | 0.32 | | 0.04 | |
| 1543 | | ----- | | ----- | |
| 1677 | IP PM CW | 0.29 | | -0.23 | |
| 1914 | IP501 | <3 | C | ----- | first reported: 1 |

| | | | |
|---|---|---|---|
| normality | OK | | |
| n | 13 | | |
| outliers | 1 | | |
| mean (n) | 0.315 | | |
| st.dev. (n) | 0.1697 | | |
| R(calc.) | 0.475 | | |
| R(IP501:05) | 0.304 | Application range : 1 mg/kg - 70 mg/kg | |
| Compare | | | |
| R(IP470:05) | 3.539 | | |
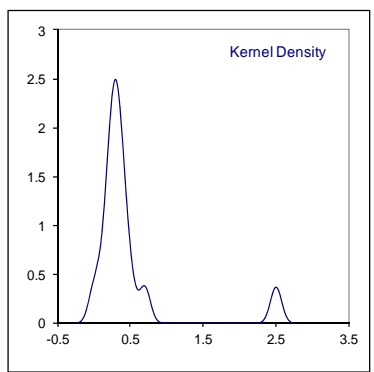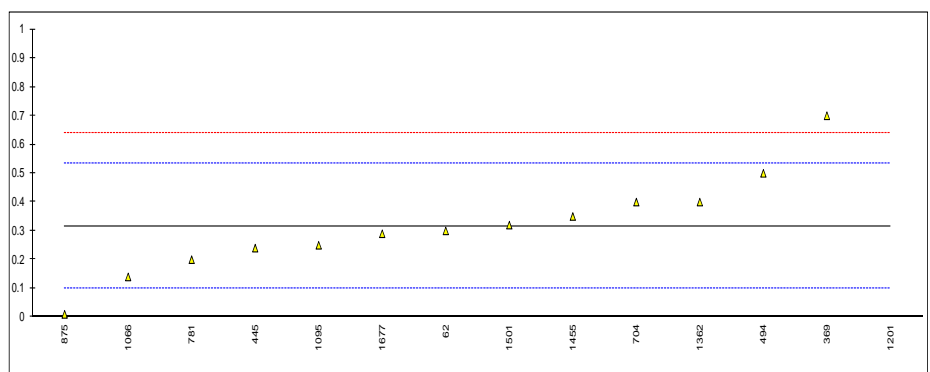
Determination of Copper on sample #13236; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | IP470 | <1 | | ----- | |
| 62 | | ----- | | ----- | |
| 120 | | ----- | | ----- | |
| 131 | | ----- | | ----- | |
| 171 | D5708 | <0.10 | | ----- | |
| 311 | D7111 | <0.5 | | ----- | |
| 313 | | ----- | | ----- | |
| 323 | INH-018 | <1 | | ----- | |
| 333 | | ----- | | ----- | |
| 334 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 369 | IP PM CW | <0.1 | | ----- | |
| 445 | IP PM CW | <0.1 | | ----- | |
| 494 | IP501 | <0.1 | | ----- | |
| 657 | INH-0003 | <1 | | ----- | |
| 704 | IP PM CW | 0.02 | | ----- | |
| 781 | IP PM CW | <0.1 | | ----- | |
| 785 | in house | 0.01 | | ----- | |
| 791 | in house | 0.01 | | ----- | |
| 875 | in house | 0.01 | | ----- | |
| 966 | | ----- | | ----- | |
| 994 | | ----- | | ----- | |
| 995 | | ----- | | ----- | |
| 1065 | | ----- | | ----- | |
| 1066 | | <0.10 | | ----- | |
| 1095 | D5708Mod. | 0.07 | | ----- | |
| 1201 | | 1.0 | | ----- | false positive result? |
| 1340 | | ----- | | ----- | |
| 1362 | in house | <1 | | ----- | |
| 1455 | IP PM CW | <0.1 | | ----- | |
| 1501 | IP PM CW | 0.01 | | ----- | |
| 1543 | | ----- | | ----- | |
| 1677 | IP PM CW | <0.1 | | ----- | |
| 1914 | IP PM CW | <0.1 | | ----- | |
| | normality | n.a | | | |
| | n | 15 | | | |
| | outliers | n.a | | | |
| | mean (n) | <0.1 | | | |
| | st.dev. (n) | n.a | | | |
| | R(calc.) | n.a | | | |
| | R(IP PM CW) | n.a | | | application range: 0.1 mg/kg -10 mg/kg |

## Determination of Nickel on sample #13236; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | IP470 | <1 | | ----- | |
| 62 | IP470 | 0.2 | | 0.19 | |
| 120 | | ----- | | ----- | |
| 131 | | ----- | | ----- | |
| 171 | D5708 | <0.10 | | ----- | |
| 311 | D7111 | <0.5 | | ----- | |
| 313 | | ----- | | ----- | |
| 323 | INH-018 | 0.4 | | 1.10 | |
| 333 | | ----- | | ----- | |
| 334 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 369 | IP PM CW | 0.2 | | 0.19 | |
| 445 | IP PM CW | 0.09 | | -0.32 | |
| 494 | IP501 | 0.13 | | -0.13 | |
| 657 | INH-0003 | <1 | | ----- | |
| 704 | IP PM CW | 0.12 | | -0.18 | |
| 781 | IP PM CW | 0.1 | | -0.27 | |
| 785 | IP470 | 0.13 | | -0.13 | |
| 791 | IP470 | 0.13 | | -0.13 | |
| 875 | IP470 | 0.17 | | 0.05 | |
| 966 | | ----- | | ----- | |
| 994 | | ----- | | ----- | |
| 995 | | ----- | | ----- | |
| 1065 | | ----- | | ----- | |
| 1066 | | 0.12 | | -0.18 | |
| 1095 | D5708Mod. | 0.16 | | 0.01 | |
| 1201 | | 1.5 | G(0.01) | 6.14 | |
| 1340 | | ----- | | ----- | |
| 1362 | IP470 | 0.3 | | 0.65 | |
| 1455 | IP PM CW | 0.15 | | -0.04 | |
| 1501 | IP PM CW | 0.09 | | -0.32 | |
| 1543 | | ----- | | ----- | |
| 1677 | IP PM CW | 0.11 | | -0.22 | |
| 1914 | IP PM CW | 0.1 | | -0.27 | |

| | | |
|---|---|---|
| normality | not OK | |
| n | 17 | |
| outliers | 1 | |
| mean (n) | 0.159 | |
| st.dev. (n) | 0.0814 | |
| R(calc.) | 0.228 | |
| R(IP501:05) | 0.611 | Application range : 1 mg/kg - 100 mg/kg |
| Compare | | |
| R(IP470:05) | 0.849 | |
| R(D5708:12 -A) | 0.098 | |
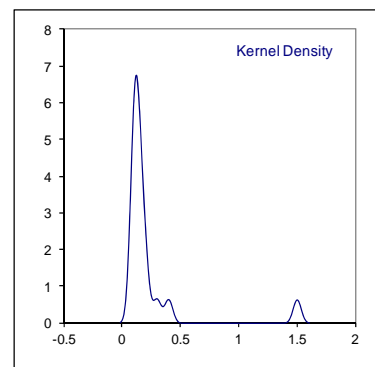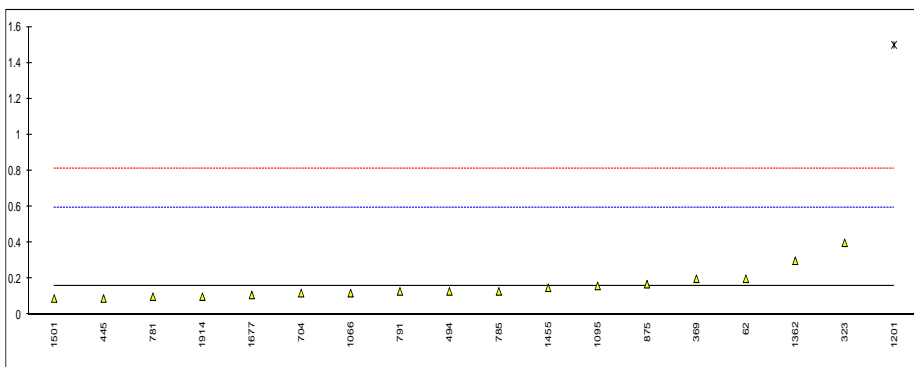
Determination of Iron on sample #13236; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | IP470 | <2 | | ----- | |
| 62 | IP470 | 0.3 | | -0.52 | |
| 120 | | ----- | | ----- | |
| 131 | | ----- | | ----- | |
| 171 | D5708 | <0.10 | | ----- | |
| 311 | D7111 | <0.5 | | ----- | |
| 313 | | ----- | | ----- | |
| 323 | INH-018 | <1 | | ----- | |
| 333 | | ----- | | ----- | |
| 334 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 369 | IP PM CW | 0.5 | | 0.46 | |
| 445 | IP PM CW | 0.47 | | 0.31 | |
| 494 | IP501 | 0.44 | | 0.16 | |
| 657 | INH-0003 | <1 | | ----- | |
| 704 | IP PM CW | 0.48 | | 0.36 | |
| 781 | IP PM CW | 0.5 | | 0.46 | |
| 785 | IP470 | 0.50 | | 0.46 | |
| 791 | IP470 | 0.19 | | -1.06 | |
| 875 | IP470 | 0.55 | | 0.70 | |
| 966 | | ----- | | ----- | |
| 994 | | ----- | | ----- | |
| 995 | | ----- | | ----- | |
| 1065 | | ----- | | ----- | |
| 1066 | | 0.41 | | 0.02 | |
| 1095 | D5708Mod. | 0.36 | | -0.23 | |
| 1201 | | 3.0 | G(0.01) | 12.70 | |
| 1340 | | ----- | | ----- | |
| 1362 | IP470 | 0.2 | | -1.01 | |
| 1455 | IP PM CW | 0.2 | | -1.01 | |
| 1501 | IP PM CW | 0.49 | | 0.41 | |
| 1543 | | ----- | | ----- | |
| 1677 | IP PM CW | 0.42 | | 0.06 | |
| 1914 | IP PM CW | 0.5 | | 0.46 | |

| | | |
|---|---|---|
| normality | OK | |
| n | 16 | |
| outliers | 1 | |
| mean (n) | 0.407 | |
| st.dev. (n) | 0.1208 | |
| R(calc.) | 0.338 | |
| R(IP501:05) | 0.572 | Application range : 2 mg/kg – 60 mg/kg |
| Compare | | |
| R (IP470:05) | 1.100 | |
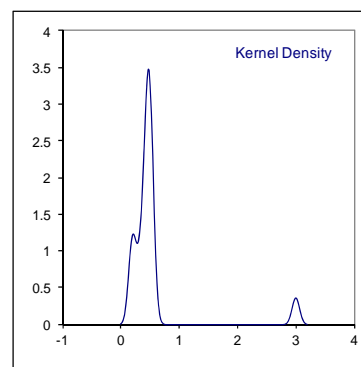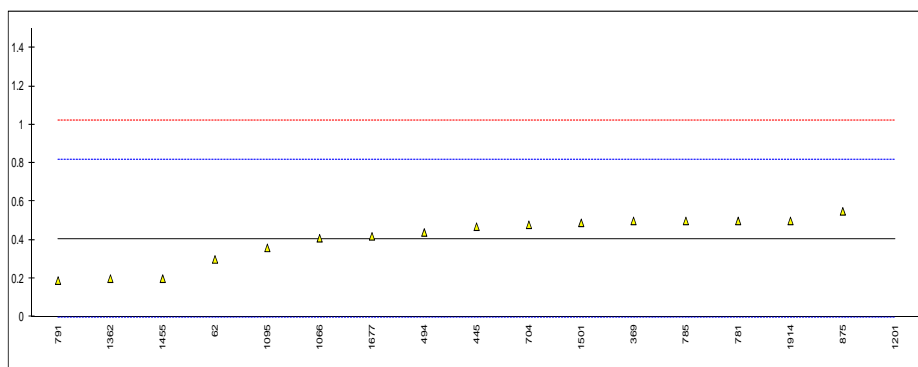
## Determination of Silicon on sample #13236; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | | ----- | | ----- | |
| 62 | IP470 | 2.6 | | ----- | |
| 120 | | ----- | | ----- | |
| 131 | | ----- | | ----- | |
| 171 | D5708 | 0.585 | | ----- | |
| 311 | UOP796 | <1 | | ----- | |
| 313 | | ----- | | ----- | |
| 323 | INH-018 | <1 | | ----- | |
| 333 | | ----- | | ----- | |
| 334 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 369 | D5184 | <5 | | ----- | |
| 445 | ISO11885 | <0.1 | | ----- | |
| 494 | IP501 | 0.87 | | ----- | |
| 657 | INH-0003 | <1 | | ----- | |
| 704 | IP470 | 0.6 | | ----- | |
| 781 | IP PM CW | 0.3 | | ----- | |
| 785 | IP470 | 0.69 | | ----- | |
| 791 | | ----- | | ----- | |
| 875 | IP470 | 0.49 | | ----- | |
| 966 | | ----- | | ----- | |
| 994 | | ----- | | ----- | |
| 995 | | ----- | | ----- | |
| 1065 | | ----- | | ----- | |
| 1066 | | <0.10 | | ----- | |
| 1095 | D5708Mod. | 0.13 | | ----- | |
| 1201 | | 3.2 | | ----- | |
| 1340 | | ----- | | ----- | |
| 1362 | IP470 | 1.0 | | ----- | |
| 1455 | | ----- | | ----- | |
| 1501 | | ----- | | ----- | |
| 1543 | | ----- | | ----- | |
| 1677 | IP501 | 0.64 | | ----- | |
| 1914 | IP501 | 1 | | ----- | |

| | | |
|---|---|---|
| normality | not OK | |
| n | 12 | |
| outliers | 0 | |
| mean (n) | 1.009 | |
| st.dev. (n) | 0.9284 | |
| R(calc.) | 2.599 | |
| R(IP501:05) | (0.335) | Application range : 10 mg/kg – 250 mg/kg |
| Compare | | |
| R(IP470:05) | 1.018 | |
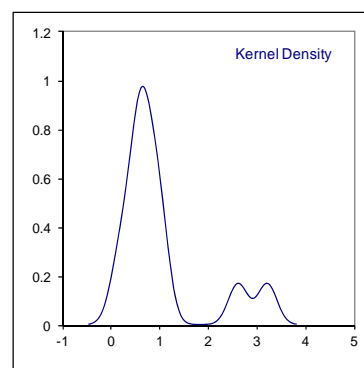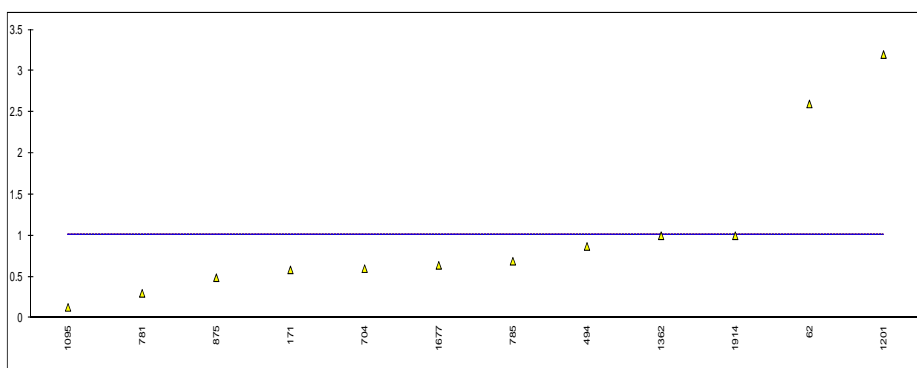
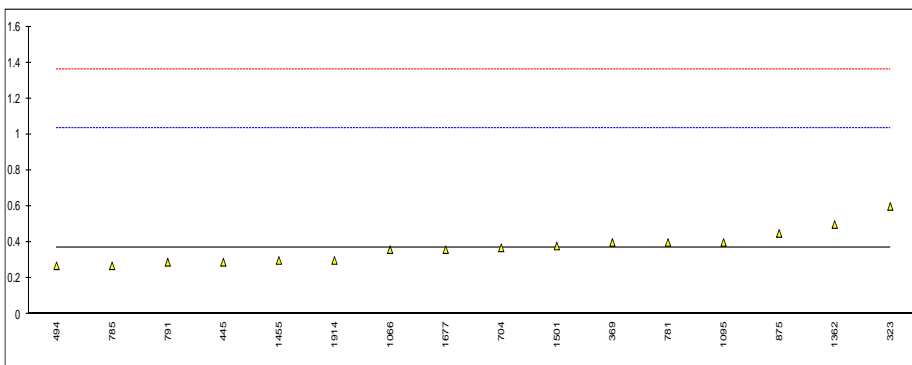## Determination of Sodium on sample #13236; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | IP470 | <1 | | ----- | |
| 62 | IP470 | 0.4 | | -0.60 | |
| 120 | | ----- | | ----- | |
| 131 | | ----- | | ----- | |
| 171 | D5708 | <0.10 | | ----- | |
| 311 | D7111 | <0.5 | | ----- | |
| 313 | | ----- | | ----- | |
| 323 | D5863 | 1.0 | | 1.54 | |
| 333 | | ----- | | ----- | |
| 334 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 369 | IP PM CW | 0.8 | | 0.83 | |
| 445 | IP PM CW | 0.54 | | -0.10 | |
| 494 | IP501 | 1.13 | | 2.01 | |
| 657 | INH-0003 | 1 | | 1.54 | |
| 704 | IP PM CW | 0.67 | | 0.36 | |
| 781 | IP PM CW | 0.4 | | -0.60 | |
| 785 | IP470 | 0.07 | | -1.79 | |
| 791 | IP470 | 0.16 | | -1.46 | |
| 875 | IP470 | 0.51 | | -0.21 | |
| 966 | | ----- | | ----- | |
| 994 | | ----- | | ----- | |
| 995 | | ----- | | ----- | |
| 1065 | | ----- | | ----- | |
| 1066 | | 0.39 | | -0.64 | |
| 1095 | D5708Mod. | 0.43 | | -0.50 | |
| 1201 | | 5.0 | G(0.01) | 15.86 | |
| 1340 | | ----- | | ----- | |
| 1362 | D5863 | <1 | | ----- | |
| 1455 | IP PM CW | 0.5 | | -0.25 | |
| 1501 | IP PM CW | 0.81 | | 0.86 | |
| 1543 | | ----- | | ----- | |
| 1677 | IP PM CW | 0.56 | | -0.03 | |
| 1914 | IP PM CW | 0.3 | | -0.96 | |

| | | |
|---|---|---|
| normality | OK | |
| n | 17 | |
| outliers | 1 | |
| mean (n) | 0.569 | |
| st.dev. (n) | 0.2973 | |
| R(calc.) | 0.832 | |
| R(IP501:05) | 0.782 | Application range : 1 mg/kg – 100 mg/kg |
| Compare | | |
| R(IP470:05) | 0.929 | |
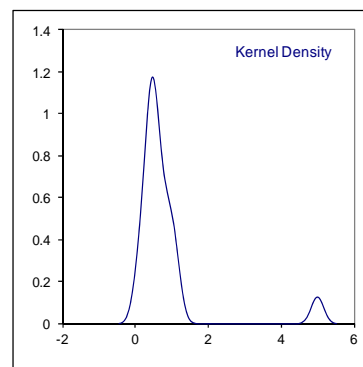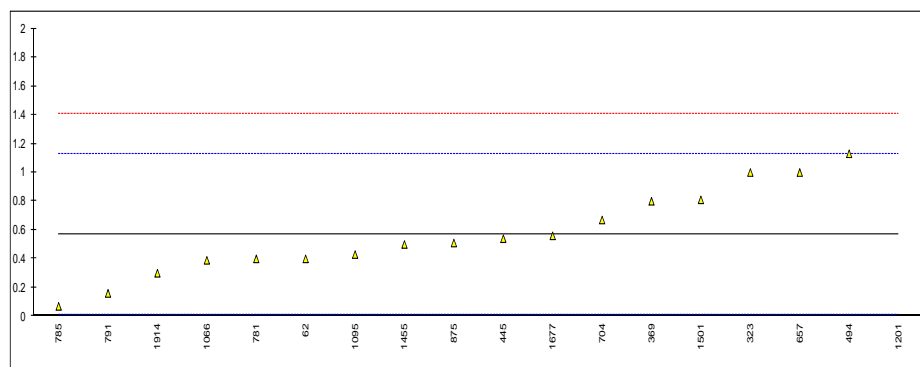
Determination of Vanadium on sample #13236; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 52 | IP470 | <1 | | ----- | |
| 62 | IP470 | <1 | | ----- | |
| 120 | | ----- | | ----- | |
| 131 | | ----- | | ----- | |
| 171 | D5708 | <0.10 | | ----- | |
| 311 | D7111 | <0.5 | | ----- | |
| 313 | | ----- | | ----- | |
| 323 | INH-018 | 0.6 | | 0.69 | |
| 333 | | ----- | | ----- | |
| 334 | | ----- | | ----- | |
| 337 | | ----- | | ----- | |
| 369 | IP PM CW | 0.4 | | 0.09 | |
| 445 | IP PM CW | 0.29 | | -0.25 | |
| 494 | IP501 | 0.27 | | -0.31 | |
| 657 | INH-0003 | <1 | | ----- | |
| 704 | IP PM CW | 0.37 | | 0.00 | |
| 781 | IP PM CW | 0.4 | | 0.09 | |
| 785 | IP470 | 0.27 | | -0.31 | |
| 791 | IP470 | 0.29 | | -0.25 | |
| 875 | IP470 | 0.45 | | 0.24 | |
| 966 | | ----- | | ----- | |
| 994 | | ----- | | ----- | |
| 995 | | ----- | | ----- | |
| 1065 | | ----- | | ----- | |
| 1066 | | 0.36 | | -0.03 | |
| 1095 | D5708Mod. | 0.40 | | 0.09 | |
| 1201 | | <0.5 | | ----- | |
| 1340 | | ----- | | ----- | |
| 1362 | IP470 | 0.5 | | 0.39 | |
| 1455 | IP PM CW | 0.3 | | -0.22 | |
| 1501 | IP PM CW | 0.38 | | 0.03 | |
| 1543 | | ----- | | ----- | |
| 1677 | IP PM CW | 0.36 | | -0.03 | |
| 1914 | IP PM CW | 0.3 | | -0.22 | |

| | | |
|---|---|---|
| normality | OK | |
| n | 16 | |
| outliers | 0 | |
| mean (n) | 0.371 | |
| st.dev. (n) | 0.0901 | |
| R(calc.) | 0.252 | |
| R(IP501:05) | 0.927 | Application range : 1 mg/kg – 400 mg/kg |
| Compare | | |
| R(IP470:05) | 1.986 | |
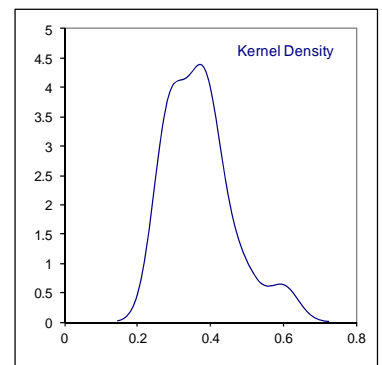
Determination of Sim. Dist. D6352 on sample #13236; result in °C

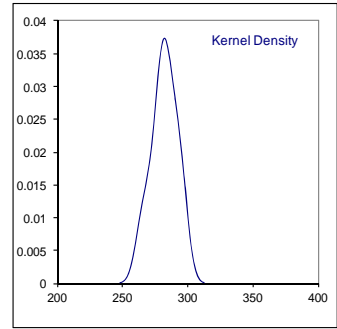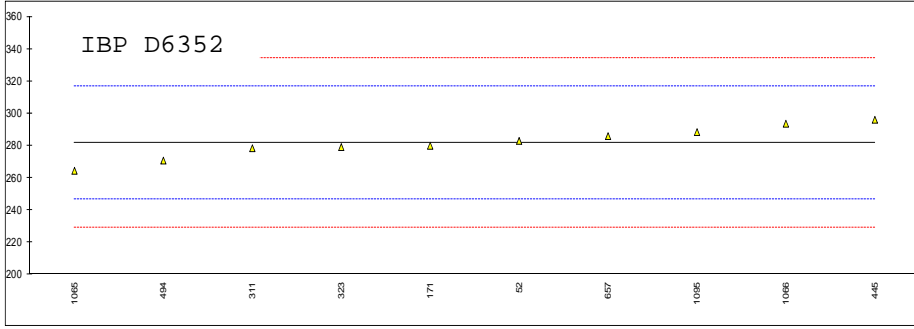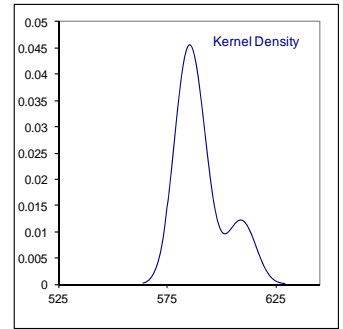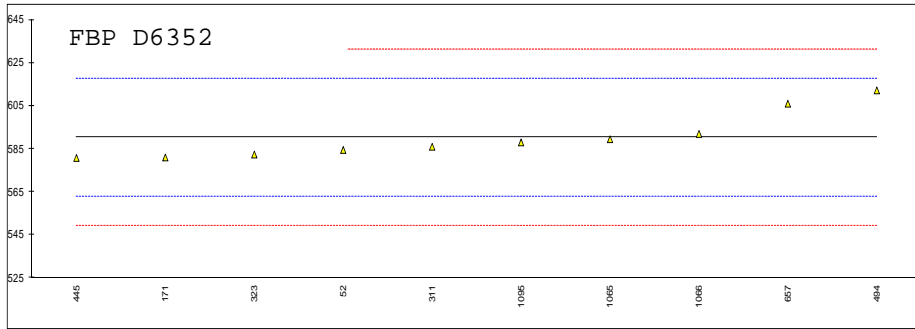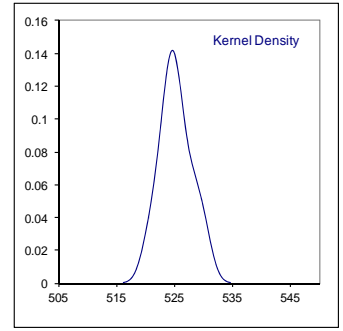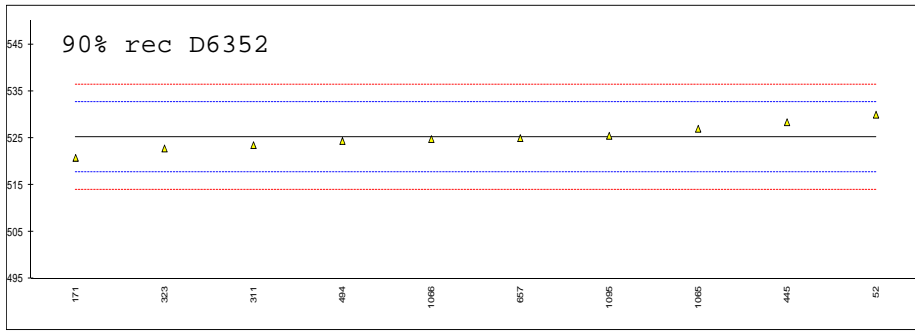| lab | method | IBP | 10% rec | 30% rec | 50% rec | 70% rec | 90% rec | FBP |
|-----|--------|-----|---------|---------|---------|---------|---------|-----|
| 52 | D7213 | <u>283.0</u> | 383.5 | 427.5 | 458.5 | 490.5 | <u>530.0</u> | 584.5 |
| 171 | D6352 | 280.0 | 378.2 | 419.6 | 449.2 | 480.6 | 520.8 | 581.0 |
| 311 | D6352 | 278.5 | 379.5 | 422.0 | 452.5 | 484.5 | 523.5 | 586.0 |
| 323 | D6352 | 279.2 | 379.0 | 421.2 | 450.9 | 482.5 | 522.8 | 582.4 |
| 445 | D2887 | 296.1 | 385.3 | 427.6 | 457.8 | 489.3 | 528.4 | 580.8 |
| 494 | D7213 | 270.8 | 381 | 422.8 | 453.2 | 485.2 | 524.4 | 612.2 |
| 657 | D6352 | 286.0 | 381.0 | 421.0 | 450.0 | 482.0 | 525.0 | 606.0 |
| 1065 | D6352 | 264.5 | 383.0 | 424.0 | 454.5 | 486.5 | 527.0 | 589.5 |
| 1066 | D6352 | 293.7 | 381.5 | 422.5 | 453.3 | 485.6 | 524.8 | 592.0 |
| 1095 | D7169 | 288.5 | 382.5 | 423.0 | 452.5 | 484.0 | 525.5 | 588.0 |
| | | | | | | | | |
| | normality | OK | OK | OK | OK | OK | OK | OK |
| | n | 10 | 10 | 10 | 10 | 10 | 10 | 10 |
| | outliers | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | mean (n) | 282.03 | 381.45 | 423.12 | 453.24 | 485.07 | 525.22 | 590.24 |
| | st.dev. (n) | 9.722 | 2.194 | 2.632 | 3.045 | 3.113 | 2.697 | 10.678 |
| | R(calc.) | 27.22 | 6.14 | 7.37 | 8.53 | 8.72 | 7.55 | 29.90 |
| | R(D6352:12) | 49.10 | 7.10 | 5.90 | 6.40 | 7.20 | 10.50 | 38.10 |

Underlined results are corrected:

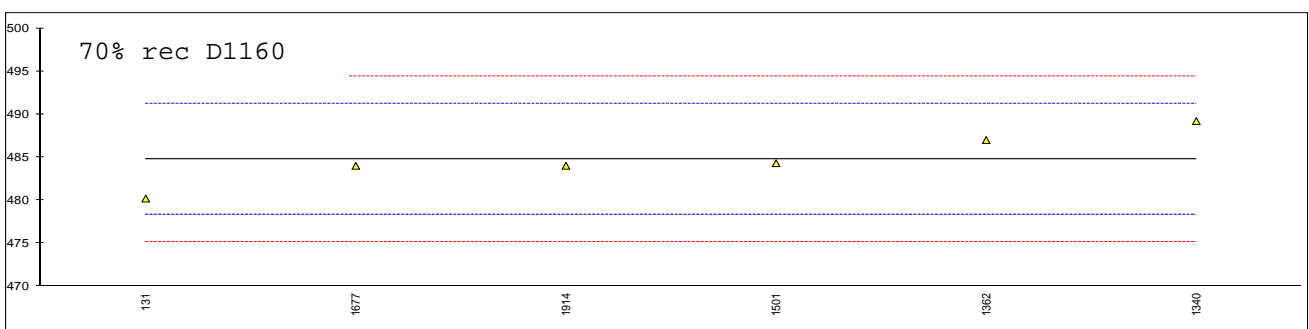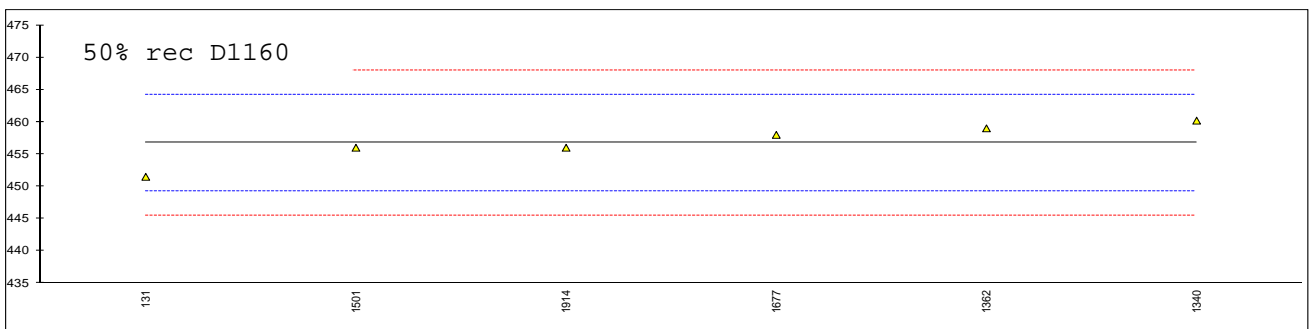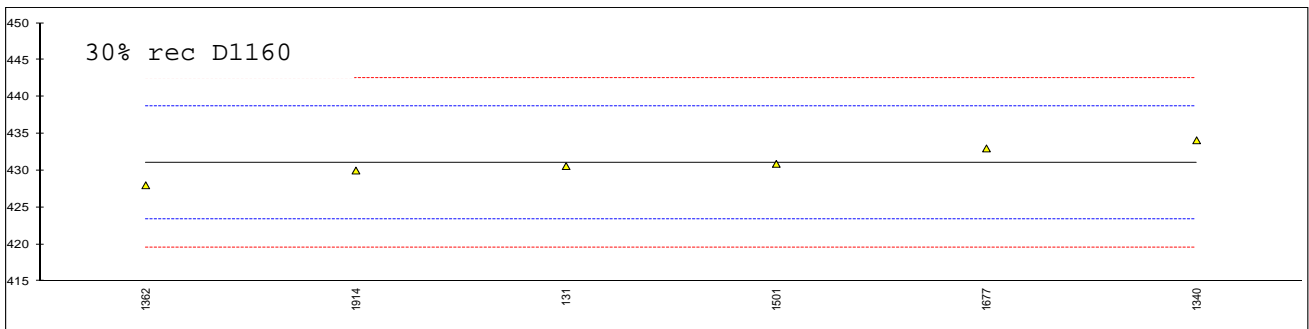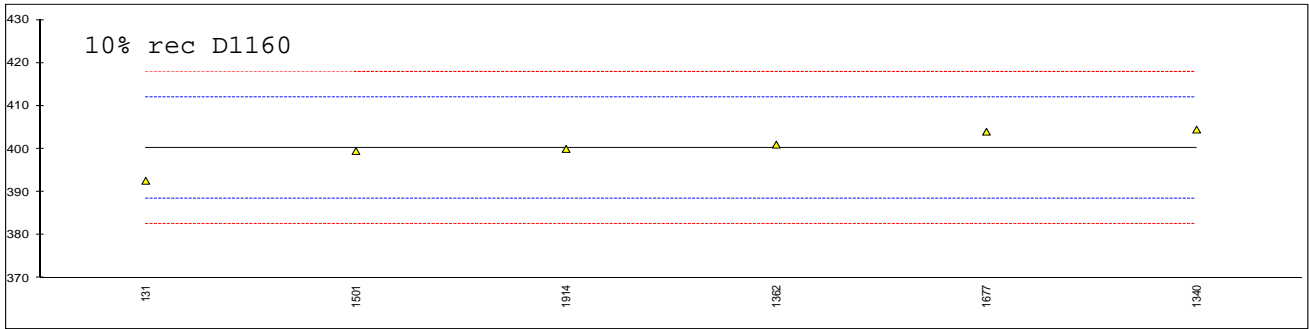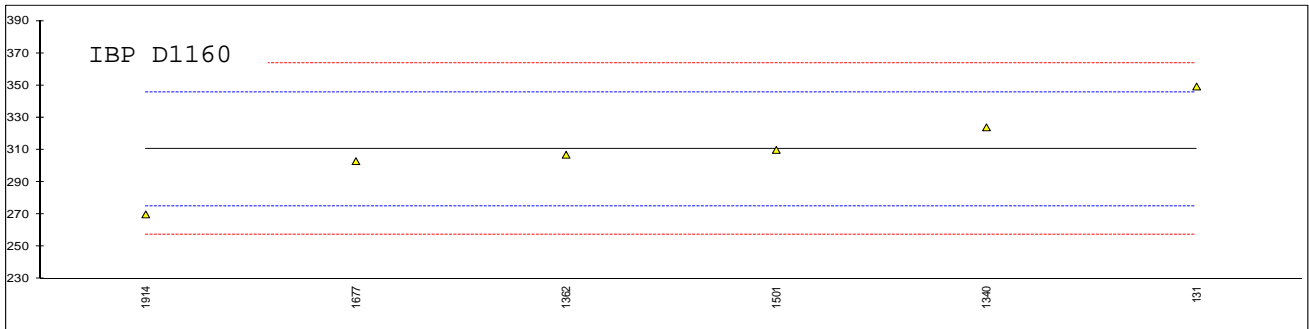Lab 52 IBP: first reported:187.5

     90% rec first reported: 535.5
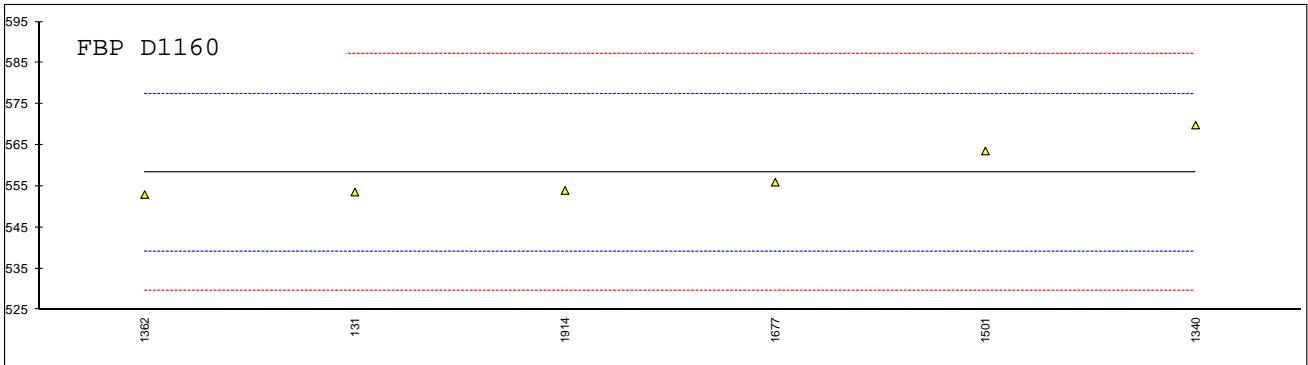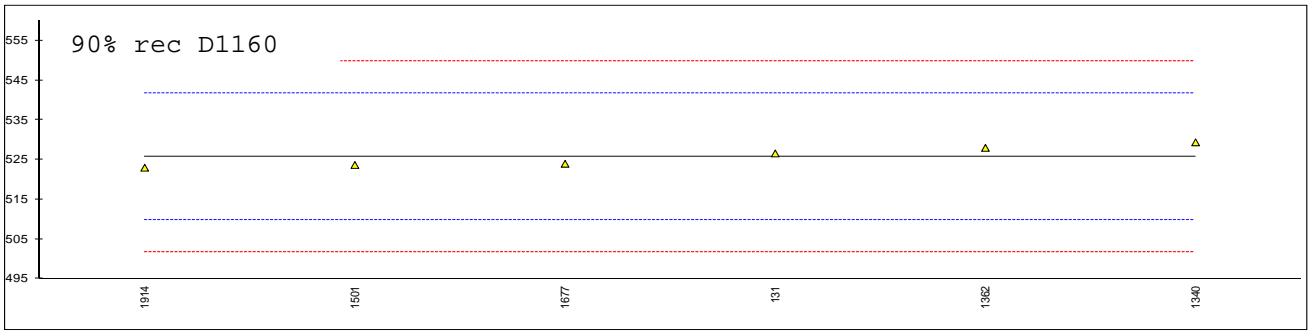
Determination of Distillation D1160 on sample #13236; result in °C

| lab | method | IBP | 10% rec | 30% rec | 50% rec | 70% rec | 90% rec | FBP |
|-----|--------|-----|---------|---------|---------|---------|---------|-----|
| 131 | D1160 | 349.3 | 392.6 | 430.6 | 451.5 | 480.2 | 526.6 | 553.6 |
| 1340 | D1160 | 324.0 | 404.5 | 434.1 | 460.2 | 489.2 | 529.4 | 569.9 |
| 1362 | D1160 | 307 | 401 | 428 | 459 | 487 | 528 | 553 |
| 1501 | D1160 | 310.0 | 399.5 | 430.9 | 456.0 | 484.3 | 523.7 | 563.6 |
| 1677 | D1160 | 303 | 404 | 433 | 458 | 484 | 524 | 556 |
| 1914 | D1160 | 270 | 400 | 430 | 456 | 484 | 523 | 554 |
| | | | | | | | | |
| | normality | OK | OK | OK | OK | OK | OK | OK |
| | n | 6 | 6 | 6 | 6 | 6 | 6 | 6 |
| | outliers | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| | mean (n) | 310.55 | 400.27 | 431.10 | 456.78 | 484.78 | 525.78 | 558.35 |
| | st.dev. (n) | 26.064 | 4.286 | 2.178 | 3.073 | 3.062 | 2.605 | 6.879 |
| | R(calc.) | 72.98 | 12.00 | 6.10 | 8.60 | 8.57 | 7.29 | 19.26 |
| | R(D1160:06) | 49.45 | 16.44 | 10.72 | 10.50 | 9.00 | 22.46 | 26.89 |

IBP D6352



Kernel Density



10% rec D6352



Kernel Density



30% rec D6352



Kernel Density



50% rec D6352



Kernel Density



70% rec D6352



Kernel Density

90% rec D6352



Kernel Density



FBP D6352



Kernel Density

IBP D1160

10% rec D1160

30% rec D1160

50% rec D1160

70% rec D1160
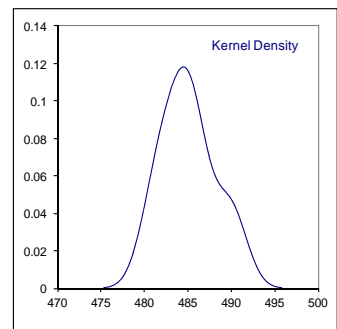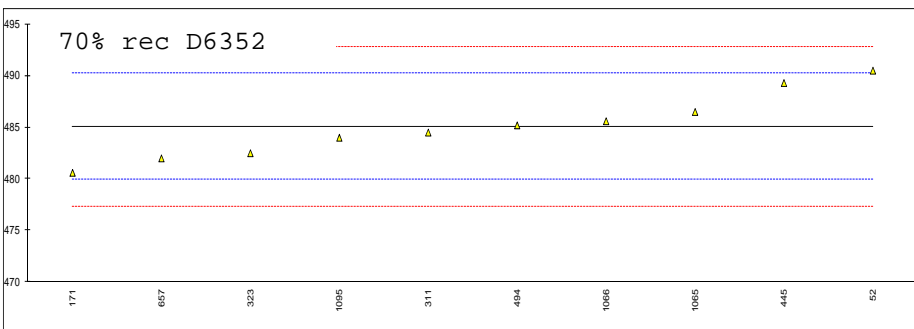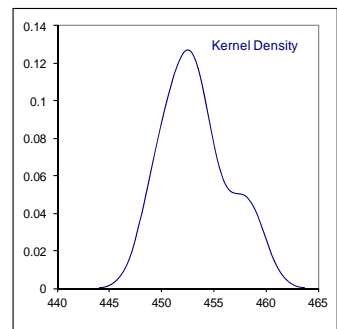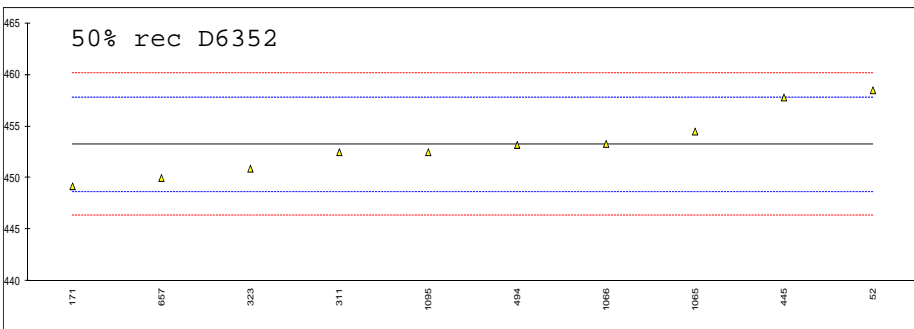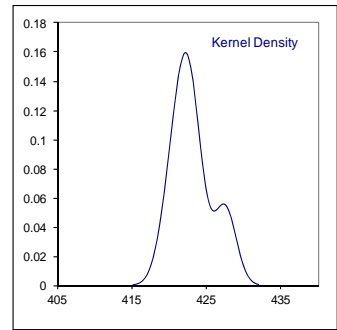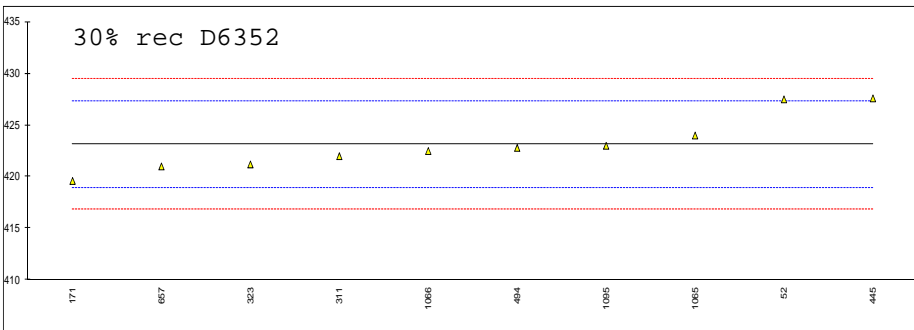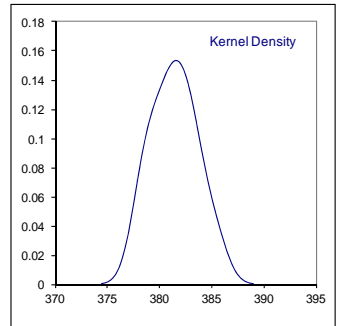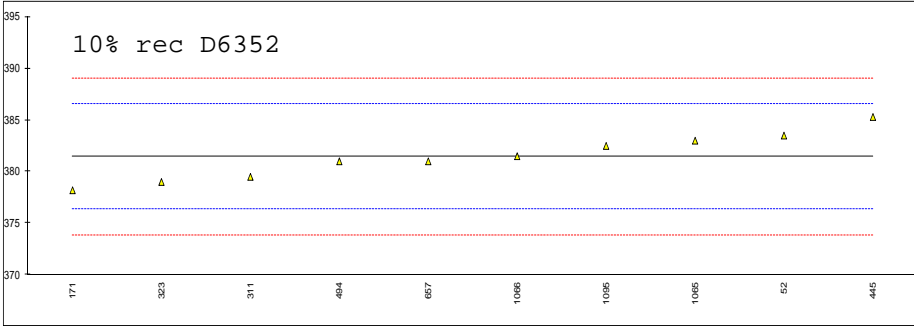
**APPENDIX 2**

z-scores Simulated Distillation according to ASTM D6352 on sample #13236

| lab | IBP | 10% | 30% | 50% | 70% | 90% | FBP |
|---|---|---|---|---|---|---|---|
| 52 | 0.06 | 0.81 | 2.08 | 2.30 | 2.11 | 1.27 | -0.42 |
| 171 | -0.12 | -1.28 | -1.67 | -1.77 | -1.74 | -1.18 | -0.68 |
| 311 | -0.20 | -0.77 | -0.53 | -0.32 | -0.22 | -0.46 | -0.31 |
| 323 | -0.16 | -0.97 | -0.91 | -1.02 | -1.00 | -0.65 | -0.58 |
| 445 | 0.80 | 1.52 | 2.13 | 1.99 | 1.64 | 0.85 | -0.69 |
| 494 | -0.64 | -0.18 | -0.15 | -0.02 | 0.05 | -0.22 | 1.61 |
| 657 | 0.23 | -0.18 | -1.01 | -1.42 | -1.19 | -0.06 | 1.16 |
| 1065 | -1.00 | 0.61 | 0.42 | 0.55 | 0.56 | 0.47 | -0.05 |
| 1066 | 0.67 | 0.02 | -0.29 | 0.03 | 0.21 | -0.11 | 0.13 |
| 1095 | 0.37 | 0.41 | -0.06 | -0.32 | -0.42 | 0.07 | -0.16 |

z-scores Distillation according to ASTM D1160 on sample #13236

| lab | IBP | 10% | 30% | 50% | 70% | 90% | FBP |
|---|---|---|---|---|---|---|---|
| 131 | 2.19 | -1.31 | -0.13 | -1.41 | -1.43 | 0.10 | -0.49 |
| 1340 | 0.76 | 0.72 | 0.78 | 0.91 | 1.37 | 0.45 | 1.20 |
| 1362 | -0.20 | 0.12 | -0.81 | 0.59 | 0.69 | 0.28 | -0.56 |
| 1501 | -0.03 | -0.13 | -0.05 | -0.21 | -0.15 | -0.26 | 0.55 |
| 1677 | -0.43 | 0.64 | 0.50 | 0.32 | -0.24 | -0.22 | -0.24 |
| 1914 | -2.30 | -0.05 | -0.29 | -0.21 | -0.24 | -0.35 | -0.45 |

**APPENDIX 3**

**Number of participants per country**

    1 lab in   AZERBAIJAN
    1 lab in   BELGIUM
    1 lab in   BOSNIA and HERZEGOVINA
  2 labs in  CANADA
    1 lab in   ESTONIA
  3 labs in  FRANCE
    1 lab in   GEORGIA
    1 lab in   GERMANY
    1 lab in   ISRAEL
  2 labs in  LATVIA
    1 lab in   LITHUANIA
  5 labs in  NETHERLANDS
    1 lab in   PORTUGAL
  5 labs in  RUSSIAN FEDERATION
    1 lab in   SAUDI ARABIA
    1 lab in   SINGAPORE
    1 lab in   SUDAN
    1 lab in   UKRAINE
    1 lab in   UNITED KINGDOM
  3 labs in  UNITED STATES OF AMERICA

### 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 |
| ex | = excluded from calculations |
| E | = error in calculations |
| n.a. | = not applicable |

**Literature**:

| | |
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