

Results of Proficiency Test  
Fuel/Bio-ethanol  
December 2009

Organised by: Institute for Interlaboratory Studies (iis)  
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## 1. INTRODUCTION

Since 1995, a proficiency test for Ethanol was organised every year by the Institute for Interlaboratory Studies. During the annual proficiency testing program 2009/2010, it was decided to continue the round robin for the analysis of Fuel/Bio-ethanol. In the international interlaboratory study for Fuel/Bio-ethanol, 47 laboratories in 24 different countries have participated. See appendix 2 for a list of participants in alphabetical country order. In this report, the results of the proficiency test are presented and discussed.

## 2 SET-UP

The Institute for Interlaboratory Studies (i.i.s.) in Spijkenisse, The Netherlands, was the organiser of this proficiency test. It was decided to send 2 samples of Ethanol (1\* 1 L of Fuel Ethanol, labelled #0992 and 1\* 0.25 L of Fuel Ethanol labelled #0993, especially for gaschromatography purpose). Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

### 2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, The Netherlands, is accredited in accordance with the ISO-guide G13:2007, (R007) since January 2000 by the Dutch Accreditation Council RvA (Raad voor Accreditatie). This ensures 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

### 2.2 PROTOCOL

The protocol followed in the organisation of this proficiency test was the one as described for proficiency testing in the report 'i.i.s. Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' (i.i.s.-protocol, version 3.2) of January 2010.

### 2.3 CONFIDENTIALITY STATEMENT

All data present 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 for the samples #0992 and #0993 was obtained from a local trader. Approximately 75 litre was delivered and was split for preparation for both samples.

Approximately 60 litre of the bulk material was transferred into a precleaned drum and was homogenised. After homogenisation, 60 subsamples were transferred to 1 litre amber glass bottles, and labelled #0992. The homogeneity of the subsamples #0992 was checked by determination of Density in accordance with ASTM D4052:09 and Water in accordance with EN15489:07 on 8 stratified random selected samples.

| Sample  | Density @ 20°C in kg/L | Water in %M/M |
|---------|------------------------|---------------|
| #0992-1 | 0.78986                | 0.172         |
| #0992-2 | 0.78986                | 0.171         |
| #0992-3 | 0.78986                | 0.171         |
| #0992-4 | 0.78986                | 0.171         |
| #0992-5 | 0.78986                | 0.173         |
| #0992-6 | 0.78986                | 0.172         |
| #0992-7 | 0.78986                | 0.172         |
| #0992-8 | 0.78986                | 0.173         |

table 1: Homogeneity tests of subsamples #0992

From the test results of table 1, the repeatabilities were calculated and compared with 0.3 times the corresponding target reproducibility in agreement with the procedure of ISO 13528, Annex B2 in the next table:

| Sample                | Density @ 20°C in kg/L | Water in %M/M |
|-----------------------|------------------------|---------------|
| r (Observed)          | 0.00000                | 0.0023        |
| reference method      | D4052:09               | EN15489:07    |
| 0.3 * R (ref. method) | 0.00015                | 0.0066        |

table 2: Repeatability of subsamples #0992

The calculated repeatability for Density and Water was equal or less than 0.3 times the corresponding reproducibility of respectively ASTM D4052:09 and EN15489:07. Therefore, homogeneity of the subsamples #0992 was assumed.

Approximately 13 litre of the bulk material was transferred into in a precleaned can and was spiked up to 15.5 mg/kg with Benzene, and up to 10.7 mg/kg with Isopropanol. After thorough homogenisation, 64 subsamples were transferred to 0.25 litre amber glass bottles, and labelled #0993. The homogeneity of the subsamples #0993 was checked by determination of Density in accordance with ASTM D4052:09, Benzene and Isopropanol in accordance with the strict requirements of the Horwitz equation on 7 stratified random selected samples.

| Sample  | Density @ 20°C in kg/L | Benzene in mg/kg | IPA in mg/kg |
|---------|------------------------|------------------|--------------|
| #0993-1 | 0.78990                | 14               | 14           |
| #0993-2 | 0.78989                | 14               | 14           |
| #0993-3 | 0.78989                | 15               | 14           |
| #0993-4 | 0.78989                | 15               | 13           |
| #0993-5 | 0.78989                | 15               | 13           |
| #0993-6 | 0.78991                | 14               | 13           |
| #0993-7 | 0.78989                | 14               | 13           |

table 3: Homogeneity tests of subsamples #0993

From the test results of table 3, the repeatabilities were calculated and compared with 0.3 times the corresponding target reproducibility in agreement with the procedure of ISO 13528, Annex B2 in the next table:

| Sample                | Density @ 20°C in kg/L | Benzene in mg/kg | IPA in mg/kg |
|-----------------------|------------------------|------------------|--------------|
| r (Observed)          | 0.00002                | 1.5              | 1.5          |
| reference method      | D4052:09               | Horwitz          | Horwitz      |
| 0.3 * R (ref. method) | 0.00015                | 1.3              | 1.2          |

table 4: Repeatability of subsamples #0993

The calculated repeatability for Density, is in agreement with the 0.3 times the reproducibility of ASTM D4052:09.

The calculated reproducibility of Benzene and Isopropanol does not appear to be in agreement with 0.3 times the corresponding reproducibility the strict requirements of the Horwitz equation. However, this is very likely caused by rounding of the raw data (see table 3).

Therefore the homogeneity of the subsamples #0993 was assumed.

To each of the participating laboratories 1 \* 1 L bottle (labelled #0992) and 1 \* 0.25 L bottle (labelled #0993) were sent on November 4, 2009.

## 2.5 STABILITY OF THE SAMPLES

The stability of Ethanol, packed in the amber glass bottles, was checked. The material was found sufficiently stable for the period of the proficiency test.

## 2.6 ANALYSES

The participants were asked to determine on sample #0992: Acidity as Acetic Acid, Aldehydes as Acetaldehyde, Anorganic Chloride as Cl, Appearance, Density @20°C, Nonvolatile Matter, Organic Chloride, pH, Sulphate, Total Sulphur, Water (coulometric and titrimetric), Copper and Phosphorous.

On sample #0993 was asked to determine: Purity on dry basis, Acetaldehyde, Acetal, Acetone, Benzene, Cyclohexane, Crotonaldehyde, DEG, Dioxane, Ethylacetate, Iso-Amylalcohol, Iso-Butanol, Iso-propanol, MEG, Methanol, n-Amylalcohol, n-Butanol, n-Propanol, sec-Amylalcohol, sec-Butanol, tert-Amylalcohol, tert-Butanol and Total Impurities by GC.

To get comparable results a detailed report form, on which the units were printed, was sent together with each set of samples. In addition, a letter of instructions and a SDS were added to the package.

### 3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were received. The original 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 fax was sent to those laboratories that had not yet reported any 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 'i.i.s. Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' (i.i.s.-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 should be used with due care.

In accordance with 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. Finally, the reproducibilities were calculated from the standard deviations by multiplying these 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 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 (see appendix 3, nr.14-15).

### 3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements, e.g. ASTM reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the spread of this interlaboratory study. The target standard deviation was calculated from the literature reproducibility by division with 2.8.

In case no literature reproducibility was available, other target values were used. In some cases, literature repeatability is available; in other cases a reproducibility of a former iis proficiency test could be used and also the Horwitz equation can be used to estimate target reproducibility.

The z-scores were calculated according to:

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

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

|               |                |
|---------------|----------------|
| $ z  < 1$     | good           |
| $1 <  z  < 2$ | satisfactory   |
| $2 <  z  < 3$ | questionable   |
| $3 <  z $     | unsatisfactory |

## 4. EVALUATION

In this proficiency test major problems were encountered with despatch of the samples. Several laboratories in Brazil, Canada, Pakistan and Turkey received their samples late or not at all.

Ten participants reported the results after the final reporting date and three participants did not report any results at all.

Not all laboratories were able to perform all analysis requested. The 44 reporting laboratories did send in 616 (numerical) results. Observed were 44 outlying results, which is 7.1%. In proficiency studies, outlier percentages of 3 % - 7.5 % are normal.

The concentrations of some GC-impurities were low and sometimes even below the detection limit. Consequently, many participants reported 'less than' values for these components. For these components no significant conclusions could be drawn.

### 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.

Not normal distributions were found for the following determinations: Density @20°C, Copper, Acetal, Crotonaldehyde, DEG, Dioxane, sec-Amyl alcohol, sec-Butanol, tert-Amyl alcohol, and tert-butanol. In these cases the statistical evaluation should be used with due care. One can see that this is justified from the Kernel Density Graphs.

#### **Sample #0992**

Acidity: This determination not problematic. Only one statistical outlier was detected. The calculated reproducibility is, after rejection of the statistical outlier, almost in agreement with the requirements of ASTM 1613:06.

Aldehydes: This determination may be problematic. This parameter was analysed by a variety of techniques (e.g. spectrophotometric and chromatographic). Because only precision data for ASTM E441 was available, it was decided to use the Horwitz equation to estimate the target reproducibility.  
Three statistical outliers were observed. The reproducibility is, after rejection of the statistical outliers, not in agreement with the strict estimated reproducibility calculated using the Horwitz equation.

Anorg.Chloride: This determination is not problematic. No statistical outliers were detected. The calculated reproducibility is in full agreement with the requirements of EN15484:07.  
In several previous PT's for Fuel/Bio-ethanol it appeared that turbidimetric analyses are problematic with lower chloride concentrations. The turbidimetric results proved to have a tendency to

be reported as false negatives. Results for anorganic chlorides by turbidimetric analysis were hence excluded.

- Appearance: This determination is not problematic. All participants agree about the appearance of sample #0992. Uniformity of reporting can be improved. A new standardized method is available for Appearance since 2009, being ASTM E2680. According this method the appearance should be reported as 'pass' (or 'fail').
- Density @20°C: This determination is not at all problematic. Three statistical outliers were observed. However, the calculated reproducibility is, after rejection of the statistical outliers, in full agreement with the requirements of ASTM D4052:09.
- N.V.M.: This determination is problematic. Three statistical outliers were detected. The calculated reproducibility is, after rejection of the statistical outliers, not in agreement with the requirements of ASTM D1353:09.
- Organic Chloride: This concentration is below the application range of ASTM D4929:07-B, therefore no conclusions were drawn for this parameter.
- pHe: This determination is very problematic. The test results may be bimodally divided. As the procedure of EN15490:07 is significantly different from ASTM D6423:04 (e.g. stirring/stabilization time before measurement), its results are excluded from calculations. After exclusion of the EN15490 results, only one statistical outlier was observed. However, the calculated reproducibility is, after rejection of the statistical outlier, not at all in agreement with the requirements of ASTM D6423:04. When all results are evaluated (including EN15490 results), a slightly different mean and calculated repeatability are observed. The calculated reproducibility is again not at all in agreement with the requirements of ASTM D6423:04, nor with EN15490:07.
- Copper: Only two participants reported a numerical result. Therefore no significant conclusions were drawn.
- Phosphorus: This concentration is below the application range of EN15487:07. Therefore no significant conclusions were drawn.
- Sulphate: This concentration may be near or below the LOD of the methods used. Therefore no significant conclusions were drawn.
- Total Sulphur: This determination is not problematic. Two statistical outliers were detected. The calculated reproducibility is, after rejection of the statistical outliers, in good agreement with the requirements of EN15485:07.

Water: This determination is not problematic for either the coulometric or the titrimetric mode. Only for the coulometric mode three statistical outliers were detected. Both calculated reproducibilities are, after rejection of the statistical outliers, in full agreement with the requirements of respectively EN15489:07 (coulometric) and ASTM E203:08 (titrimetric).

### **Sample #0993 (GC- Impurities)**

Purity on db: This determination is not problematic. Five statistical outliers were detected. The calculated reproducibility is, after rejection of the statistical outlier, in full agreement with the requirements of EN15721:07.

Acetaldehyde: This determination may be very problematic. Only one statistical outlier, was observed, and two false negatives were reported. However, the calculated reproducibility is, after rejection of the statistical outlier, not at all in agreement with the strict estimated reproducibility calculated using the Horwitz equation.

Acetal: This determination may be problematic. Only one statistical outlier was observed, and one false negative was reported. However, the calculated reproducibility is, after rejection of the statistical outlier, not in agreement with the strict estimated reproducibility calculated using the Horwitz equation.

Acetone: This concentration is probably near or below the limit of detection (LOD), hence no significant conclusions were drawn. For this test two false positives were observed.

Benzene: This determination may not be problematic. One statistical outlier was observed, and three false negatives were reported. The calculated reproducibility is, after rejection of the statistical outlier, in agreement with the strict estimated reproducibility calculated using the Horwitz equation.  
The average recovery of Benzene and the theoretical increment of 15.5 mg/kg may be good (<95%). The actual blank Benzene content is unknown.

Cyclohexane: This parameter is probably near or below the limit of detection (LOD), hence no significant conclusions were drawn.

Ethylacetate: This determination may be problematic. Two statistical outliers were observed. The calculated reproducibility is, after rejection of the statistical outliers, not in agreement with the strict estimated reproducibility calculated using the Horwitz equation.

- Iso Amylalcohol: This determination is very problematic. Two statistical outliers were observed. Also, the group was found to be bimodally divided. The calculated reproducibility is, after rejection of the statistical outliers, not at all in agreement with the strict estimated reproducibility calculated using the Horwitz equation. When the two groups are evaluated individually, only the calculated reproducibility of group 2 is in agreement with the strict estimated reproducibility calculated using the Horwitz equation.
- Isobutanol: This determination may be problematic. Two statistical outliers were detected. The calculated reproducibility is, after rejection of the statistical outliers, not in agreement with the strict estimated reproducibility calculated using the Horwitz equation.
- Isopropanol: This determination may be problematic. Only one statistical outlier was observed. However, the calculated reproducibility is, after rejection of the statistical outlier, not in agreement with the strict estimated reproducibility calculated using the Horwitz equation. The average recovery of Isopropanol and the theoretical increment of 10.7 mg/kg may be good (<110%). The actual blank Isopropanol content is unknown.
- MEG: This determination may be problematic. One statistical outlier was detected. The calculated reproducibility is, after rejection of the statistical outlier, not at all in agreement with the strict estimated reproducibility calculated using the Horwitz equation. However, the low number of results may partially explain the large spread observed.
- Methanol: This determination is problematic for five laboratories. Five statistical outliers were detected. However, the calculated reproducibility is, after rejection of the statistical outliers, in full agreement with the requirements according to EN15721:07 and ASTM D5501:09.
- n-Butanol: This concentration is probably near or below the limit of detection, hence no significant conclusions were drawn.
- n-Propanol: This determination may be problematic. Three statistical outliers were observed. The calculated reproducibility is, after rejection of the statistical outlier, not in agreement with the strict estimated reproducibility calculated using the Horwitz equation.
- Sec-Butanol: This concentration is probably near or below the limit of detection, hence no significant conclusions were drawn.
- Total Impurities: This determination may not be problematic. Two statistical outliers were observed. The calculated reproducibility is, after rejection of the

statistical outliers, in good agreement with the estimated reproducibility calculated using the Horwitz equation.

For Crotonaldehyde, DEG, Dioxane, n-Amylalcohol, sec-Amylalcohol, tert-Amylalcohol and tert-Butanol only few numerical results were reported. Therefore no significant conclusions were drawn for these parameters.

#### 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. The average results per sample, calculated reproducibilities and reproducibilities derived from literature standards (in casu ASTM standards) or the Horwitz equation are compared in the next table.

| Parameter                | unit      | n  | average | 2.8 *sd <sub>R</sub> | R (lit) |
|--------------------------|-----------|----|---------|----------------------|---------|
| Acidity as Acetic acid   | mg/kg     | 38 | 31.05   | 15.00                | 14.00   |
| Aldehyde as Acetaldehyde | mg/kg     | 17 | 141.5   | 104.2                | 60.16   |
| Anorganic Chloride as Cl | mg/kg     | 18 | 0.44    | 0.46                 | 1.26    |
| Density @ 20°C           | kg/L      | 39 | 0.78993 | 0.00020              | 0.00050 |
| Nonvolatile Matter       | mg/100 mL | 20 | 1.52    | 3.53                 | 2.40    |
| Organic Chloride         | mg/kg     | 5  | 0.51    | 0.63                 | n.a.    |
| pHe                      |           | 24 | 6.37    | 1.47                 | 0.52    |
| Copper                   | mg/kg     | 2  | n.a.    | n.a.                 | n.a.    |
| Phosphorous              | mg/kg     | 7  | 0.011   | 0.014                | n.a.    |
| Sulphate                 | mg/kg     | 5  | 0.68    | 1.57                 | n.a.    |
| Total Sulphur            | mg/kg     | 26 | 8.56    | 2.89                 | 3.86    |
| Water (Coulometric)      | % M/M     | 29 | 0.1716  | 0.0148               | 0.0220  |
| Water (Titrimetric)      | % M/M     | 26 | 0.1690  | 0.0215               | 0.0780  |

Table 5: Reproducibilities of sample #0992

| Parameter           | unit  | n  | average | 2.8 *sd <sub>R</sub> | R (lit) |
|---------------------|-------|----|---------|----------------------|---------|
| Purity on dry basis | %M/M  | 24 | 99.851  | 0.061                | 0.067   |
| Acetaldehyde        | mg/kg | 23 | 127.0   | 111.1                | 27.4    |
| Acetal              | mg/kg | 23 | 200.0   | 53.6                 | 40.4    |
| Acetone             | mg/kg | 15 | 6.76    | 5.12                 | (2.27)  |
| Benzene             | mg/kg | 18 | 14.77   | 4.41                 | 4.41    |
| Cyclohexane         | mg/kg | 12 | 5.45    | 4.92                 | (1.89)  |
| Crotonaldehyde      | mg/kg | 2  | n.a.    | n.a.                 | n.a.    |
| DEG                 | mg/kg | 0  | n.a.    | n.a.                 | n.a.    |
| Dioxane             | mg/kg | 0  | n.a.    | n.a.                 | n.a.    |
| Ethylacetate        | mg/kg | 27 | 123.3   | 30.8                 | 26.8    |
| iso-Amylalcohol     | mg/kg | 21 | 89.18   | 20.06                | (20.32) |
| iso-Butanol         | mg/kg | 24 | 253.4   | 59.7                 | 49.4    |
| iso-Propanol        | mg/kg | 20 | 12.06   | 5.36                 | 3.71    |
| MEG                 | mg/kg | 5  | 20.26   | 14.80                | 5.77    |
| Methanol            | mg/kg | 25 | 60.26   | 16.68                | 20.17   |
| n-Amylalcohol       | mg/kg | 4  | n.a.    | n.a.                 | n.a.    |
| n-Butanol           | mg/kg | 7  | 2.35    | 1.27                 | n.a.    |
| n-Propanol          | mg/kg | 27 | 388.3   | 86.8                 | 70.9    |
| sec-Amylalcohol     | mg/kg | 2  | n.a.    | n.a.                 | n.a.    |
| sec-Butanol         | mg/kg | 6  | 0.93    | 0.35                 | n.a.    |
| tert-Amylalcohol    | mg/kg | 3  | n.a.    | n.a.                 | n.a.    |
| tert-Butanol        | mg/kg | 3  | n.a.    | n.a.                 | n.a.    |
| Total impurities    | mg/kg | 18 | 1398.6  | 504.4                | 631.9   |

Table 6: Reproducibilities of sample #0993

Results between brackets should be used with care, as the average is near or below the application range

Without further statistical calculations, it can be concluded that for most of the tests there is a not much compliance of the group of participating laboratories with the relative standards. The tests, that are problematic, have been discussed in paragraph 4.1.

#### 4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2009 WITH PREVIOUS PTS

|                            | <i>December 2009</i> | <i>December 2008</i> | <i>December 2007</i> | <i>December 2006</i> |
|----------------------------|----------------------|----------------------|----------------------|----------------------|
| Number of reporting labs   | 44                   | 53                   | 40                   | 35                   |
| Number of results reported | 616                  | 557                  | 595                  | 567                  |
| Statistical outliers       | 44                   | 40                   | 30                   | 50                   |
| Percentage outliers        | 7.1%                 | 7.2%                 | 5.0%                 | 8.8%                 |

table 7: comparison with previous proficiency tests.

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

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

| Determination            | December 2009 | December 2008 | December 2007 | December 2006 |
|--------------------------|---------------|---------------|---------------|---------------|
| Acidity as Acetic Acid   | +/-           | +/-           | +/-           | ++            |
| Aldehyde as Acetaldehyde | --            | --            | --            | --            |
| Anorganic Chloride as Cl | ++            | -             | +             | ++            |
| Density @ 20°C           | ++            | ++            | ++            | ++            |
| Nonvolatile Matter       | --            | ++            | ++            | n.e.          |
| Organic Chloride         | n.e.          | -             | n.e.          | ++            |
| pHe                      | --            | --            | --            | --            |
| Copper as Cu             | n.e.          |               |               |               |
| Phosphorus as P          | n.e.          |               |               |               |
| Sulphate                 | n.e.          | --            | n.e.          | n.e.          |
| Total Sulphur            | ++            | +             | -             | --            |
| Water coulometric        | ++            | ++            | ++            | ++            |
| Water titrimetric        | ++            | ++            | --            | --            |
| Purity on dry basis      | +             | (++)          | (--)          | (++)          |
| Acetaldehyde             | --            | n.e.          | n.e.          | n.e.          |
| Acetal                   | -             | n.e.          | n.e.          | n.e.          |
| Acetone                  | n.e.          | (--)          | --            | -             |
| Benzene                  | +             | n.e.          | +/-           | ++            |
| Cyclohexane              | n.e.          | n.e.          | n.e.          | n.e.          |
| Crotonaldehyde           | n.e.          | n.e.          | n.e.          | n.e.          |
| DEG                      | n.e.          | n.e.          | n.e.          | n.e.          |
| Dioxane                  | n.e.          | n.e.          | n.e.          | n.e.          |
| Ethylacetate             | -             | n.e.          | n.e.          | n.e.          |
| iso-Amylalcohol          | --            | n.e.          | --            | n.e.          |
| iso-Butanol              | -             | (-)           | --            | n.e.          |
| iso-Propanol             | --            | n.e.          | n.e.          | +/-           |
| MEG                      | --            | n.e.          | n.e.          | n.e.          |
| Methanol                 | ++            | (--)          | --            | --            |
| n-Amylalcohol            | n.e.          | n.e.          | n.e.          | n.e.          |
| n-Butanol                | n.e.          | n.e.          | n.e.          | n.e.          |
| n-Propanol               | -             | (+/-)         | --            | n.e.          |
| sec-Amylalcohol          | n.e.          | n.e.          | n.e.          | n.e.          |
| sec-Butanol              | n.e.          | (+/-)         | n.e.          | n.e.          |
| tert-Amylalcohol         | n.e.          | n.e.          | n.e.          | n.e.          |
| tert-Butanol             | n.e.          | n.e.          | n.e.          | n.e.          |
| Total Impurities         | ++            | n.e.          | n.e.          | n.e.          |

table 8: comparison determinations against the standard results between brackets are compared with the spread of the previous round robin

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

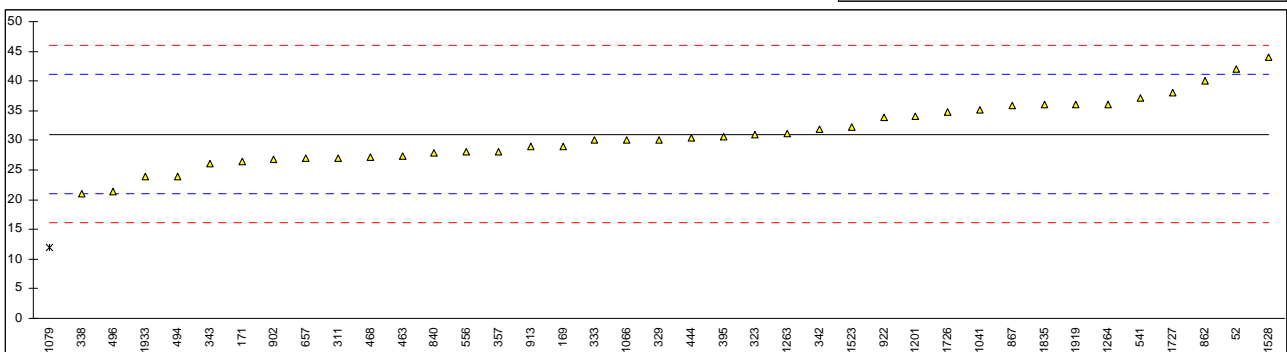
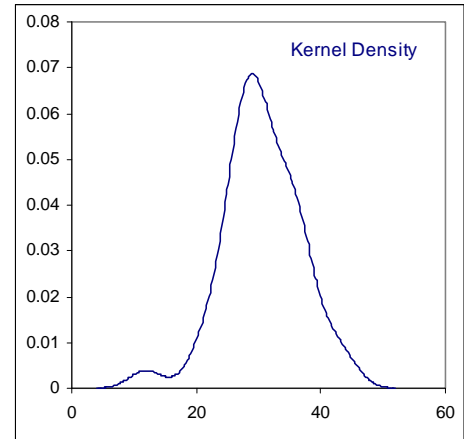
- ++: group performed much better than the standard
- + : group performed better than the standard
- +/-: group performance equals the standard
- : group performed worse than the standard
- : group performed much worse than the standard
- n.e.: not evaluated

**APPENDIX 1**

**Determination of Acidity as Acetic Acid on sample #0992; results in mg/kg**

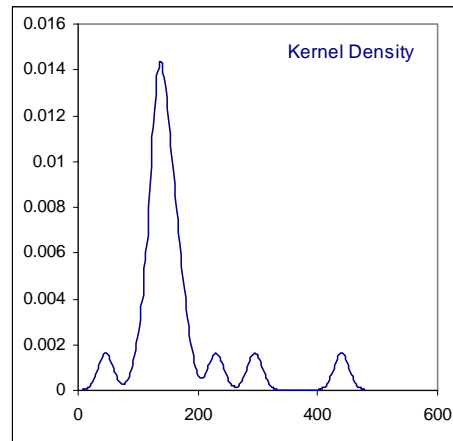
| lab  | method  | value  | mark    | z(targ) | remarks                                      |
|------|---------|--------|---------|---------|--|
| 52   | D1613   | 42     |         | 2.19    |  |
| 150  |         | ----   |         | ----    |  |
| 169  | D1613   | 29     | C       | -0.41   | First reported in deviating unit 0.0029 %M/M |
| 171  | D1613   | 26.4   |         | -0.93   |  |
| 311  | D1613   | 27     |         | -0.81   |  |
| 323  | D1613   | 31     |         | -0.01   |  |
| 329  | D1613   | 30     |         | -0.21   |  |
| 333  | D1613   | 30     | C       | -0.21   | First reported in deviating unit 0.003 %M/M  |
| 338  | D1613   | 21     |         | -2.01   |  |
| 342  | D1613   | 31.8   |         | 0.15    |  |
| 343  | EN15491 | 26     | C       | -1.01   | First reported in deviating unit 0.0026 %M/M |
| 357  | D1613   | 28     |         | -0.61   |  |
| 395  | D1613   | 30.65  |         | -0.08   |  |
| 444  | D1613   | 30.4   |         | -0.13   |  |
| 463  | D1613   | 27.345 |         | -0.74   |  |
| 468  | D1613   | 27.23  |         | -0.76   |  |
| 494  | D1613   | 24     |         | -1.41   |  |
| 496  | D1613   | 21.32  |         | -1.95   |  |
| 541  | D1613   | 37.21  |         | 1.23    |  |
| 551  |         | ----   |         | ----    |  |
| 556  | D1613   | 28     |         | -0.61   |  |
| 559  |         | ----   |         | ----    |  |
| 657  | D1613   | 27     |         | -0.81   |  |
| 840  | D1613   | 27.9   |         | -0.63   |  |
| 862  | D1613   | 40     |         | 1.79    |  |
| 867  | D1613   | 35.8   |         | 0.95    |  |
| 902  | D1613   | 26.76  |         | -0.86   |  |
| 913  | D1613   | 29.0   |         | -0.41   |  |
| 922  | D1613   | 33.95  |         | 0.58    |  |
| 1041 | EN14591 | 35.2   |         | 0.83    |  |
| 1059 |         | ----   |         | ----    |  |
| 1066 | D1613   | 30     |         | -0.21   |  |
| 1079 | D1613   | 12     | G(0.05) | -3.81   |  |
| 1154 |         | ----   |         | ----    |  |
| 1161 |         | ----   |         | ----    |  |
| 1201 | D1613   | 34     |         | 0.59    |  |
| 1263 | D1613   | 31.1   |         | 0.01    |  |
| 1264 | D1613   | 36     | U       | 0.99    | Reported in deviating unit (%M/M)            |
| 1521 |         | ----   |         | ----    |  |
| 1523 | ISO1388 | 32.3   |         | 0.25    |  |
| 1528 | EN15491 | 44     |         | 2.59    |  |
| 1605 |         | ----   |         | ----    |  |
| 1726 | D1613   | 34.7   |         | 0.73    |  |
| 1727 | D1613   | 38     |         | 1.39    |  |
| 1835 | D1613   | 36     |         | 0.99    |  |
| 1919 | D1613   | 36     |         | 0.99    |  |
| 1933 | EN15491 | 24     |         | -1.41   |  |

normality OK  
n 38  
outliers 1  
mean (n) 31.05  
st.dev. (n) 5.358  
R(calc.) 15.00  
R(D1613:06) 14.00

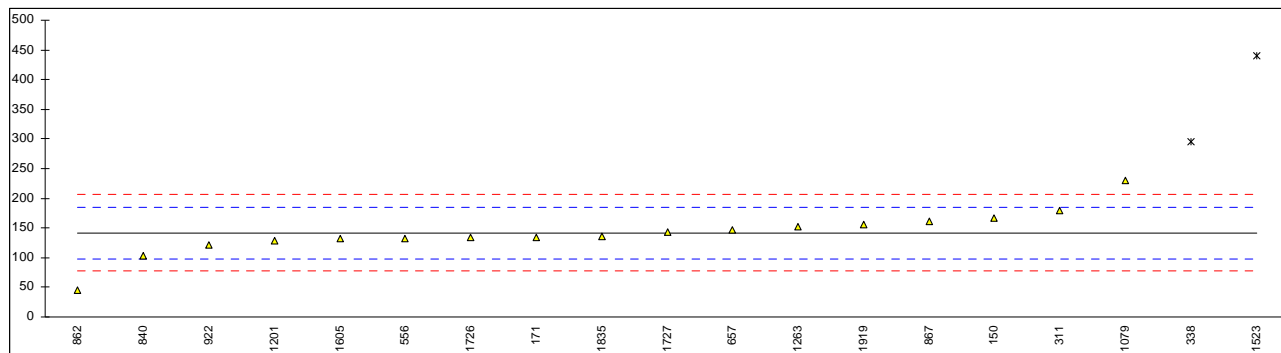


Determination of Aldehydes as Acetaldehyde on sample #0992; results in mg/kg

| lab  | method      | value   | mark      | z(targ) | remarks           |
|------|-------------|---------|-----------|---------|-------------------|
| 52   |             | ----    |           | ----    |                   |
| 150  | INH-ETOH    | 167.0   |           | 1.19    |                   |
| 169  |             | ----    |           | ----    |                   |
| 171  | D5501       | 134.6   |           | -0.32   |                   |
| 311  | INH-529     | 180     |           | 1.79    |                   |
| 323  |             | ----    |           | ----    |                   |
| 329  |             | ----    |           | ----    |                   |
| 333  |             | ----    |           | ----    |                   |
| 338  | INH-GC      | 295     | C,G(0.05) | 7.15    | First reported 86 |
| 342  |             | ----    |           | ----    |                   |
| 343  |             | ----    |           | ----    |                   |
| 357  |             | ----    |           | ----    |                   |
| 395  |             | ----    |           | ----    |                   |
| 444  |             | ----    |           | ----    |                   |
| 463  |             | ----    |           | ----    |                   |
| 468  |             | ----    |           | ----    |                   |
| 494  |             | ----    |           | ----    |                   |
| 496  |             | ----    |           | ----    |                   |
| 541  |             | ----    |           | ----    |                   |
| 551  |             | ----    |           | ----    |                   |
| 556  | GLC         | 132.911 |           | -0.40   |                   |
| 559  |             | ----    |           | ----    |                   |
| 657  | INH-01      | 145.9   |           | 0.21    |                   |
| 840  | INH-0001    | 104     |           | -1.74   |                   |
| 862  | INH-01/07   | 46      |           | -4.44   |                   |
| 867  | INH-394.2   | 162.0   |           | 0.96    |                   |
| 902  |             | ----    |           | ----    |                   |
| 913  |             | ----    |           | ----    |                   |
| 922  | INH-0001    | 120.8   |           | -0.96   |                   |
| 1041 |             | ----    |           | ----    |                   |
| 1059 |             | ----    |           | ----    |                   |
| 1066 |             | ----    |           | ----    |                   |
| 1079 | E411        | 230     |           | 4.12    |                   |
| 1154 |             | ----    |           | ----    |                   |
| 1161 |             | ----    |           | ----    |                   |
| 1201 | E411        | 128     |           | -0.63   |                   |
| 1263 | ISO1388     | 152.57  |           | 0.52    |                   |
| 1264 |             | ----    |           | ----    |                   |
| 1521 |             | ----    |           | ----    |                   |
| 1523 | D5501       | 439.5   | G(0.01)   | 13.87   |                   |
| 1528 | EC2870      | <10     |           | <-6.12  |                   |
| 1605 | in house    | 132.3   |           | -0.43   |                   |
| 1726 | in house    | 134     |           | -0.35   |                   |
| 1727 |             | 143     |           | 0.07    |                   |
| 1835 | INH-GC      | 136     |           | -0.25   |                   |
| 1919 |             | 156     |           | 0.68    |                   |
| 1933 |             | ----    |           | ----    |                   |
|      | normality   | OK      |           |         |                   |
|      | n           | 17      |           |         |                   |
|      | outliers    | 2       |           |         |                   |
|      | mean (n)    | 141.48  |           |         |                   |
|      | st.dev. (n) | 37.223  |           |         |                   |
|      | R(calc.)    | 104.23  |           |         |                   |
|      | R(Horwitz)  | 60.16   |           |         |                   |

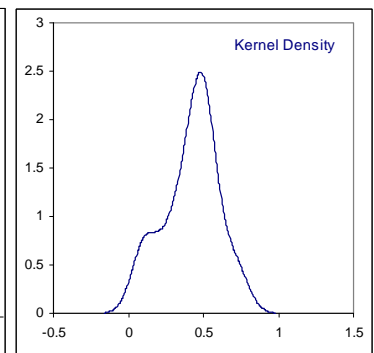
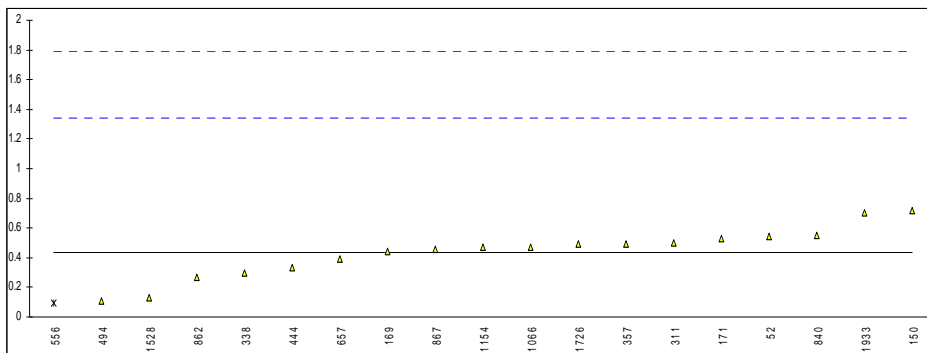


Compare with R(E411:05) = 24.05



Determination of Anorganic Chlorides as Cl<sup>-</sup> on sample #0992; results in mg/kg

| lab  | method        | value  | mark        | z(targ) | remarks   |
|------|---------------|--------|-------------|---------|---|
| 52   | IMACA002      | 0.54   |             | 0.23    |   |
| 150  | D7328         | 0.72   |             | 0.62    |   |
| 169  | D7319         | 0.440  |             | 0.00    |   |
| 171  | D7328         | 0.53   |             | 0.20    |   |
| 311  | EN15484       | 0.5    |             | 0.14    |   |
| 323  | EN15484       | <5.0   |             | ----    |   |
| 329  | EN15484       | <5.0   |             | ----    |   |
| 333  | EN15484       | <5     |             | ----    |   |
| 338  | IMPCA002      | 0.3    |             | -0.31   |   |
| 342  |               | ----   |             | ----    |   |
| 343  |               | ----   |             | ----    |   |
| 357  | IMPCA002      | 0.49   |             | 0.11    |   |
| 395  |               | ----   |             | ----    |   |
| 444  | EN15492       | 0.33   |             | -0.24   |   |
| 463  | EN15484       | <4     |             | ----    |   |
| 468  | EN15484       | <4     |             | ----    |   |
| 494  | EN15484       | 0.11   |             | -0.73   |   |
| 496  |               | ----   |             | ----    |   |
| 541  |               | ----   |             | ----    |   |
| 551  |               | ----   |             | ----    |   |
| 556  | Turbidimetric | 0.0968 | ex          | -0.76   | Result is excluded as test method gives false negative? |
| 559  |               | ----   |             | ----    |   |
| 657  | D7328         | 0.39   |             | -0.11   |   |
| 840  | IMPCA002      | 0.55   |             | 0.25    |   |
| 862  | IMPCA002      | 0.27   |             | -0.37   |   |
| 867  | IMPCA002      | 0.46   |             | 0.05    |   |
| 902  |               | ----   |             | ----    |   |
| 913  |               | ----   |             | ----    |   |
| 922  |               | ----   |             | ----    |   |
| 1041 |               | ----   |             | ----    |   |
| 1059 |               | ----   |             | ----    |   |
| 1066 | EN15484       | 0.47   |             | 0.07    |   |
| 1079 | EN15484       | <1     |             | ----    |   |
| 1154 | EN15492       | 0.47   |             | 0.07    |   |
| 1161 |               | ----   |             | ----    |   |
| 1201 | EN15484       | <1     |             | ----    |   |
| 1263 |               | ----   |             | ----    |   |
| 1264 |               | ----   |             | ----    |   |
| 1521 |               | ----   |             | ----    |   |
| 1523 |               | ----   |             | ----    |   |
| 1528 | EN15484       | 0.13   |             | -0.68   |   |
| 1605 |               | ----   |             | ----    |   |
| 1726 | in house      | 0.49   |             | 0.11    |   |
| 1727 | EN15484       | <0.2   |             | ----    |   |
| 1835 | Turbidimetric | <1     | ex          | ----    | Result is excluded as test method gives false negative? |
| 1919 |               | ----   |             | ----    |   |
| 1933 | EN15484       | 0.7    |             | 0.58    |   |
|      | normality     | OK     |             |         |   |
|      | n             | 18     |             |         |   |
|      | outliers      | 1      |             |         |   |
|      | mean (n)      | 0.44   |             |         |   |
|      | st.dev. (n)   | 0.164  |             |         |   |
|      | R(calc.)      | 0.46   |             |         |   |
|      | R(EN15484:07) | 1.26   | (1.60 mg/L) |         |   |



## Determination of Appearance on sample #0992;

| lab  | method      | value  | mark | z(targ) | remarks |
|------|-------------|--------|------|---------|---------|
| 52   | D4806       | CBFSM  |      | ----    |         |
| 150  | Visual      | C&F    |      | ----    |         |
| 169  |             | ----   |      | ----    |         |
| 171  | E2680       | PASS   |      | ----    |         |
| 311  | E2680       | PASS   |      | ----    |         |
| 323  | INH-001     | CFFSM  |      | ----    |         |
| 329  | INH-001     | CFFSM  |      | ----    |         |
| 333  |             | ----   |      | ----    |         |
| 338  | Visual      | CBFFSM |      | ----    |         |
| 342  | E2680       | PASS   |      | ----    |         |
| 343  | INH-1008    | C&B    |      | ----    |         |
| 357  | E2680       | PASS   |      | ----    |         |
| 395  | E2680       | PASS   |      | ----    |         |
| 444  | E2680       | PASS   |      | ----    |         |
| 463  | D4176       | PASS   |      | ----    |         |
| 468  | E2680       | PASS   |      | ----    |         |
| 494  | E2680       | C&B    |      | ----    |         |
| 496  | E2680       | PASS   |      | ----    |         |
| 541  | E2680       | CFFSM  |      | ----    |         |
| 551  |             | ----   |      | ----    |         |
| 556  | Visual      | CLEAR  |      | ----    |         |
| 559  |             | ----   |      | ----    |         |
| 657  | E2680       | PASS   |      | ----    |         |
| 840  | E2680       | PASS   |      | ----    |         |
| 862  | E2680       | PASS   |      | ----    |         |
| 867  | E2680       | C&B    |      | ----    |         |
| 902  | E2680       | PASS   |      | ----    |         |
| 913  | E2680       | CFFSM  |      | ----    |         |
| 922  | Visual      | CLFSM  |      | ----    |         |
| 1041 | Visual      | C&F    |      | ----    |         |
| 1059 |             | ----   |      | ----    |         |
| 1066 |             | ----   |      | ----    |         |
| 1079 | Visual      | B&C    |      | ----    |         |
| 1154 |             | ----   |      | ----    |         |
| 1161 |             | ----   |      | ----    |         |
| 1201 | Visual      | B&C    |      | ----    |         |
| 1263 |             | ----   |      | ----    |         |
| 1264 | Visual      | CLEAR  |      | ----    |         |
| 1521 | EN15769     | T&C    |      | ----    |         |
| 1523 |             | ----   |      | ----    |         |
| 1528 |             | ----   |      | ----    |         |
| 1605 |             | ----   |      | ----    |         |
| 1726 | E2680       | B&C    |      | ----    |         |
| 1727 | E2680       | B&C    |      | ----    |         |
| 1835 | EN15769     | T&C    |      | ----    |         |
| 1919 |             | ----   |      | ----    |         |
| 1933 | Visual      | CLEAR  |      | ----    |         |
|      | normality   | n.a.   |      |         |         |
|      | n           | 34     |      |         |         |
|      | outliers    | 0      |      |         |         |
|      | mean (n)    | n.a.   |      |         |         |
|      | st.dev. (n) | n.a.   |      |         |         |
|      | R(calc.)    | n.a.   |      |         |         |
|      | R()         | n.a.   |      |         |         |

CFFSM = clear &amp; free from suspended matter

B&amp;C = clear &amp; bright

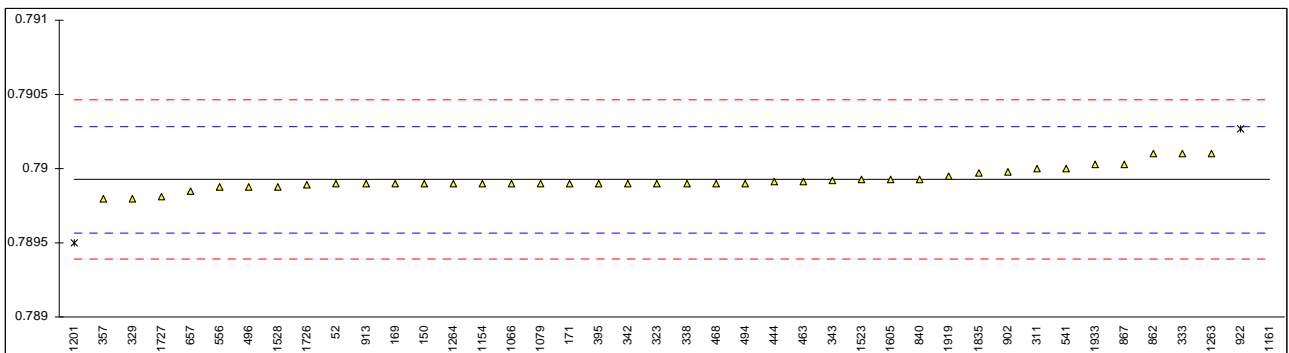
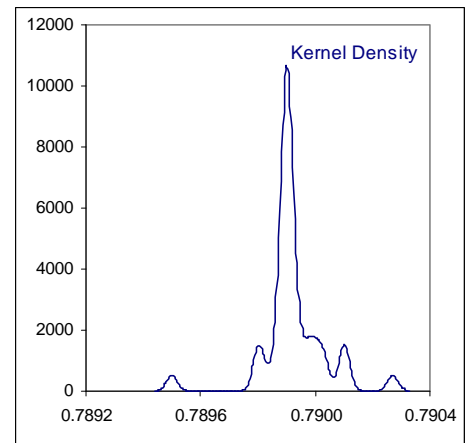
C = clear

C&amp;F = clear &amp; free

T&amp;C = Transparent &amp; colourless

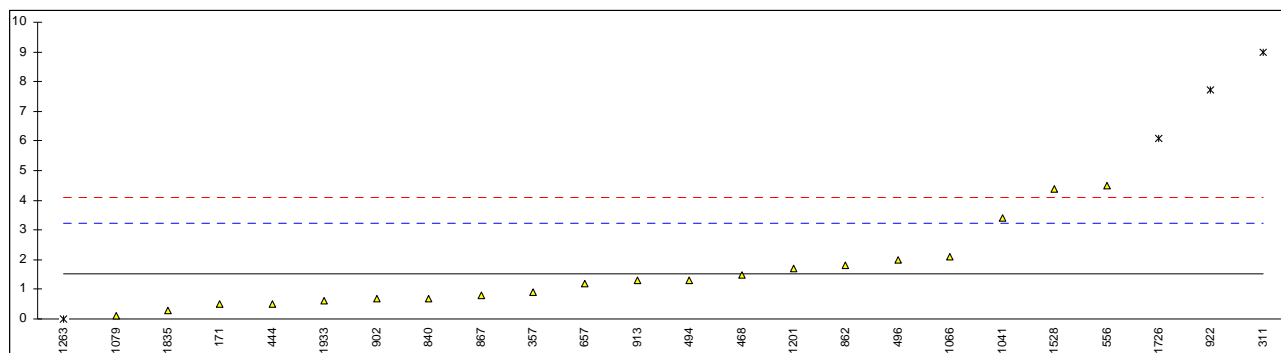
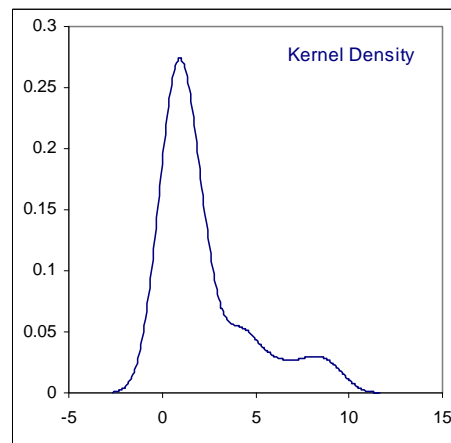
Determination of Density @ 20°C on sample #0992; results in kg/L

| lab  | method      | value    | mark    | z(targ) | remarks  |
|------|-------------|----------|---------|---------|--|
| 52   | D4052       | 0.7899   |         | -0.14   |  |
| 150  | D4052       | 0.7899   |         | -0.14   |  |
| 169  | D4052       | 0.7899   |         | -0.14   |  |
| 171  | D4052       | 0.7899   |         | -0.14   |  |
| 311  | D4052       | 0.7900   |         | 0.42    |  |
| 323  | D4052       | 0.7899   |         | -0.14   |  |
| 329  | D4052       | 0.7898   |         | -0.70   |  |
| 333  | D4052       | 0.7901   |         | 0.98    |  |
| 338  | ISO12185    | 0.7899   |         | -0.14   |  |
| 342  | D4052       | 0.7899   |         | -0.14   |  |
| 343  | D4052       | 0.78992  |         | -0.03   |  |
| 357  | D4052       | 0.7898   |         | -0.70   |  |
| 395  | D4052       | 0.7899   |         | -0.14   |  |
| 444  | D4052       | 0.78991  |         | -0.08   |  |
| 463  | D4052       | 0.78991  |         | -0.08   |  |
| 468  | D4052       | 0.7899   | C       | -0.14   | First reported in deviating unit 789.9 kg/m <sup>3</sup> |
| 494  | D4052       | 0.7899   |         | -0.14   |  |
| 496  | D4052       | 0.78988  |         | -0.25   |  |
| 541  | D4052       | 0.7900   |         | 0.42    |  |
| 551  |             | ----     |         | ----    |  |
| 556  | D4052       | 0.78988  |         | -0.25   |  |
| 559  |             | ----     |         | ----    |  |
| 657  | D4052       | 0.78985  |         | -0.42   |  |
| 840  | D4052       | 0.78993  |         | 0.03    |  |
| 862  | D4052       | 0.7901   |         | 0.98    |  |
| 867  | D4052       | 0.79003  |         | 0.59    |  |
| 902  | D4052       | 0.78998  |         | 0.31    |  |
| 913  | D1298       | 0.7899   |         | -0.14   |  |
| 922  | D4052       | 0.79027  | G(0.05) | 1.93    |  |
| 1041 |             | ----     |         | ----    |  |
| 1059 |             | ----     |         | ----    |  |
| 1066 | D4052       | 0.7899   |         | -0.14   |  |
| 1079 | D4052       | 0.7899   |         | -0.14   |  |
| 1154 | ISO12185    | 0.7899   |         | -0.14   |  |
| 1161 | ISO12185    | 0.798    | G(0.01) | 45.22   |  |
| 1201 | D4052       | 0.7895   | G(0.01) | -2.38   |  |
| 1263 | ISO12185    | 0.790104 |         | 1.00    |  |
| 1264 | D4052       | 0.7899   |         | -0.14   |  |
| 1521 |             | ----     |         | ----    |  |
| 1523 | D4052       | 0.789926 |         | 0.01    |  |
| 1528 | D4052       | 0.78988  |         | -0.25   |  |
| 1605 | D4052       | 0.78993  |         | 0.03    |  |
| 1726 | D4052       | 0.78989  |         | -0.20   |  |
| 1727 | D4052       | 0.78981  |         | -0.64   |  |
| 1835 | D4052       | 0.78997  |         | 0.25    |  |
| 1919 | D4052       | 0.78995  |         | 0.14    |  |
| 1933 | EN12185     | 0.79003  |         | 0.59    |  |
|      | normality   | not OK   |         |         |  |
|      | n           | 39       |         |         |  |
|      | outliers    | 3        |         |         |  |
|      | mean (n)    | 0.78993  |         |         |  |
|      | st.dev. (n) | 0.000073 |         |         |  |
|      | R(calc.)    | 0.00020  |         |         |  |
|      | R(D4052:09) | 0.00050  |         |         |  |



Determination of Nonvolatile Matter on sample #0992; results in mg/100mL

| lab  | method      | value | mark     | z(targ) | remarks                  |
|------|-------------|-------|----------|---------|--------------------------|
| 52   |             | ----  |          | ----    |                          |
| 150  | D1353       | <1    |          | <-0.61  |                          |
| 169  |             | ----  |          | ----    |                          |
| 171  | D1353       | 0.5   |          | -1.18   |                          |
| 311  | D1353       | 9     | DG(0.01) | 8.73    |                          |
| 323  |             | ----  |          | ----    |                          |
| 329  |             | ----  |          | ----    |                          |
| 333  |             | ----  |          | ----    |                          |
| 338  |             | ----  |          | ----    |                          |
| 342  |             | ----  |          | ----    |                          |
| 343  | EC-2870     | <10   |          | ----    |                          |
| 357  | D1353       | 0.9   |          | -0.72   |                          |
| 395  |             | ----  |          | ----    |                          |
| 444  | D1353       | 0.52  |          | -1.16   |                          |
| 463  |             | ----  |          | ----    |                          |
| 468  | D1353       | 1.5   |          | -0.02   |                          |
| 494  | D1353       | 1.3   |          | -0.25   |                          |
| 496  | D1353       | 2.0   |          | 0.57    |                          |
| 541  |             | ----  |          | ----    |                          |
| 551  |             | ----  |          | ----    |                          |
| 556  | D1353       | 4.5   |          | 3.48    |                          |
| 559  |             | ----  |          | ----    |                          |
| 657  | D1353       | 1.2   |          | -0.37   |                          |
| 840  | D1353       | 0.7   |          | -0.95   |                          |
| 862  | D1353       | 1.8   |          | 0.33    |                          |
| 867  | D1353       | 0.8   |          | -0.83   |                          |
| 902  | D1353       | 0.68  |          | -0.97   |                          |
| 913  | D1353       | 1.3   |          | -0.25   |                          |
| 922  | D1353       | 7.7   | DG(0.01) | 7.22    |                          |
| 1041 | EN15691     | 3.4   |          | 2.20    |                          |
| 1059 |             | ----  |          | ----    |                          |
| 1066 | D1353       | 2.1   |          | 0.68    |                          |
| 1079 | D1353       | 0.1   |          | -1.65   |                          |
| 1154 |             | ----  |          | ----    |                          |
| 1161 |             | ----  |          | ----    |                          |
| 1201 | D1353       | 1.7   |          | 0.22    |                          |
| 1263 | D1353       | 0.0   | ex       | -1.77   | Zero is not a real value |
| 1264 |             | ----  |          | ----    |                          |
| 1521 |             | ----  |          | ----    |                          |
| 1523 |             | ----  |          | ----    |                          |
| 1528 | EC2870      | 4.4   |          | 3.37    |                          |
| 1605 |             | ----  |          | ----    |                          |
| 1726 | D1353       | 6.1   | G(0.05)  | 5.35    |                          |
| 1727 |             | ----  |          | ----    |                          |
| 1835 | EN15691     | 0.3   |          | -1.42   |                          |
| 1919 |             | ----  |          | ----    |                          |
| 1933 | D1353       | 0.6   |          | -1.07   |                          |
|      | normality   | OK    |          |         |                          |
|      | n           | 20    |          |         |                          |
|      | outliers    | 3     |          |         |                          |
|      | mean (n)    | 1.52  |          |         |                          |
|      | st.dev. (n) | 1.262 |          |         |                          |
|      | R(calc.)    | 3.53  |          |         |                          |
|      | R(D1353:09) | 2.40  |          |         |                          |

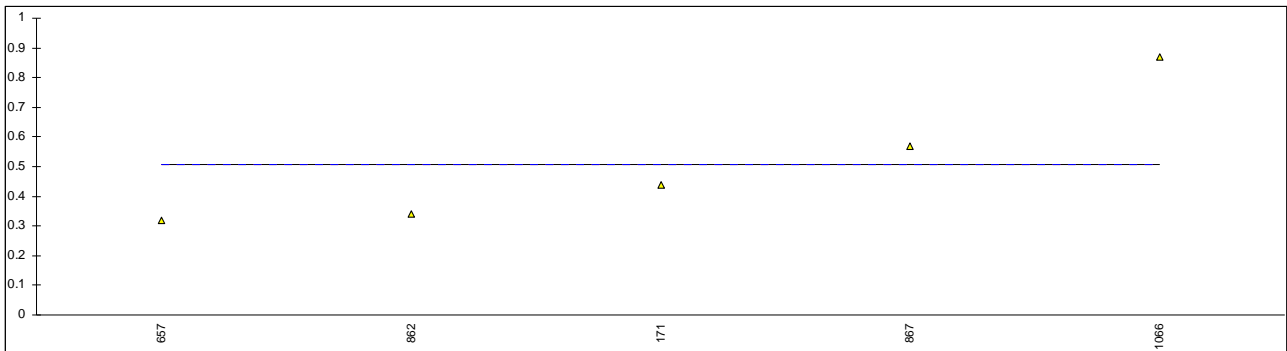


Determination of Organic Chloride on sample #0992; results in mg/kg

| lab  | method  | value | mark | z(targ) | remarks |
|------|---------|-------|------|---------|---------|
| 52   |         | ----  |      | ----    |         |
| 150  |         | ----  |      | ----    |         |
| 169  |         | ----  |      | ----    |         |
| 171  | D5808   | 0.44  |      | ----    |         |
| 311  | D4929-B | <1    |      | ----    |         |
| 323  |         | ----  |      | ----    |         |
| 329  |         | ----  |      | ----    |         |
| 333  |         | ----  |      | ----    |         |
| 338  |         | ----  |      | ----    |         |
| 342  |         | ----  |      | ----    |         |
| 343  |         | ----  |      | ----    |         |
| 357  | D5808   | <1    |      | ----    |         |
| 395  |         | ----  |      | ----    |         |
| 444  |         | ----  |      | ----    |         |
| 463  |         | ----  |      | ----    |         |
| 468  |         | ----  |      | ----    |         |
| 494  | EN14077 | <1    |      | ----    |         |
| 496  | D4929-B | <1    |      | ----    |         |
| 541  |         | ----  |      | ----    |         |
| 551  |         | ----  |      | ----    |         |
| 556  |         | ----  |      | ----    |         |
| 559  |         | ----  |      | ----    |         |
| 657  | D4929-B | 0.32  |      | ----    |         |
| 840  |         | ----  |      | ----    |         |
| 862  | D4929-B | 0.34  |      | ----    |         |
| 867  | D5808   | 0.57  |      | ----    |         |
| 902  |         | ----  |      | ----    |         |
| 913  |         | ----  |      | ----    |         |
| 922  |         | ----  |      | ----    |         |
| 1041 |         | ----  |      | ----    |         |
| 1059 |         | ----  |      | ----    |         |
| 1066 | D4929-B | 0.87  |      | ----    |         |
| 1079 | D5808   | <1    |      | ----    |         |
| 1154 |         | ----  |      | ----    |         |
| 1161 |         | ----  |      | ----    |         |
| 1201 | D4929-B | <1    |      | ----    |         |
| 1263 |         | ----  |      | ----    |         |
| 1264 |         | ----  |      | ----    |         |
| 1521 |         | ----  |      | ----    |         |
| 1523 |         | ----  |      | ----    |         |
| 1528 |         | ----  |      | ----    |         |
| 1605 |         | ----  |      | ----    |         |
| 1726 |         | ----  |      | ----    |         |
| 1727 |         | ----  |      | ----    |         |
| 1835 |         | ----  |      | ----    |         |
| 1919 |         | ----  |      | ----    |         |
| 1933 |         | ----  |      | ----    |         |

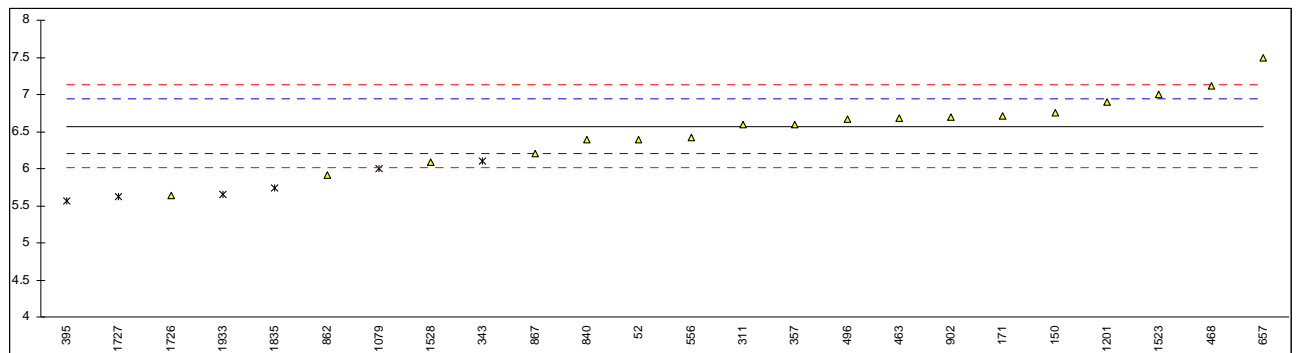
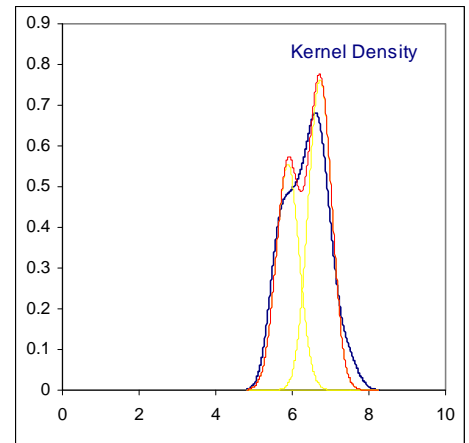
normality OK  
n 5  
outliers 0  
mean (n) 0.51  
st.dev. (n) 0.225  
R(calc.) 0.63  
R(D4929:07B) n.a.

Application range >1 mg/kg



Determination of pHe on sample #0992;

| lab  | method      | value  | mark    | z(targ)         | remarks                        |
|------|-------------|--------|---------|-----------------|--------------------------------|
| 52   | D6423       | 6.39   |         | -0.97           |                                |
| 150  | D6423       | 6.76   |         | 1.02            |                                |
| 169  |             | ----   |         | ----            |                                |
| 171  | D6423       | 6.71   |         | 0.76            |                                |
| 311  | D6423       | 6.6    |         | 0.16            |                                |
| 323  |             | ----   |         | ----            |                                |
| 329  |             | ----   |         | ----            |                                |
| 333  |             | ----   |         | ----            |                                |
| 338  |             | ----   |         | ----            |                                |
| 342  |             | ----   |         | ----            |                                |
| 343  | EN15490     | 6.1    | ex      | -2.53           | See §4.1                       |
| 357  | D6423       | 6.6    |         | 0.16            |                                |
| 395  | D6423       | 5.56   | G(0,05) | -5.44           |                                |
| 444  |             | ----   |         | ----            |                                |
| 463  | D6423       | 6.6745 |         | 0.56            |                                |
| 468  | D6423       | 7.12   |         | 2.96            |                                |
| 494  |             | ----   |         | ----            |                                |
| 496  | D6423       | 6.66   |         | 0.49            |                                |
| 541  |             | ----   |         | ----            |                                |
| 551  |             | ----   |         | ----            |                                |
| 556  | D6423       | 6.42   |         | -0.81           |                                |
| 559  |             | ----   |         | ----            |                                |
| 657  | D6423       | 7.49   |         | 4.96            |                                |
| 840  | D6423       | 6.39   |         | -0.97           |                                |
| 862  | D6423       | 5.92   |         | -3.50           |                                |
| 867  | D6423       | 6.20   |         | -1.99           |                                |
| 902  | D6423       | 6.7    |         | 0.70            |                                |
| 913  |             | ----   |         | ----            |                                |
| 922  |             | ----   |         | ----            |                                |
| 1041 |             | ----   |         | ----            |                                |
| 1059 |             | ----   |         | ----            |                                |
| 1066 |             | ----   |         | ----            |                                |
| 1079 | EN15490     | 6.0    | ex      | -3.07           | See §4.1                       |
| 1154 |             | ----   |         | ----            |                                |
| 1161 |             | ----   |         | ----            |                                |
| 1201 | D6423       | 6.9    |         | 1.78            |                                |
| 1263 |             | ----   |         | ----            |                                |
| 1264 |             | ----   |         | ----            |                                |
| 1521 |             | ----   |         | ----            |                                |
| 1523 | D6423       | 7.00   |         | 2.32            |                                |
| 1528 | D6423       | 6.08   |         | -2.64           |                                |
| 1605 |             | ----   |         | ----            |                                |
| 1726 | D6423       | 5.64   |         | -5.01           |                                |
| 1727 | EN15490     | 5.62   | ex      | -5.11           | See §4.1                       |
| 1835 | EN15490     | 5.74   | ex      | -4.47           | See §4.1                       |
| 1919 |             | ----   |         | ----            |                                |
| 1933 | EN15490     | 5.65   | ex      | -4.95           | See §4.1                       |
|      |             |        |         | <u>All data</u> |                                |
|      | normality   | OK     |         | OK              |                                |
|      | n           | 18     |         | 24              |                                |
|      | outliers    | 1      |         | 0               |                                |
|      | mean (n)    | 6.57   |         | 6.37            |                                |
|      | st.dev. (n) | 0.440  |         | 0.525           |                                |
|      | R(calc.)    | 1.23   |         | 1.47            |                                |
|      | R(D6423:08) | 0.52   |         | 0.52            | Compare with R(EN15490) = 0.61 |



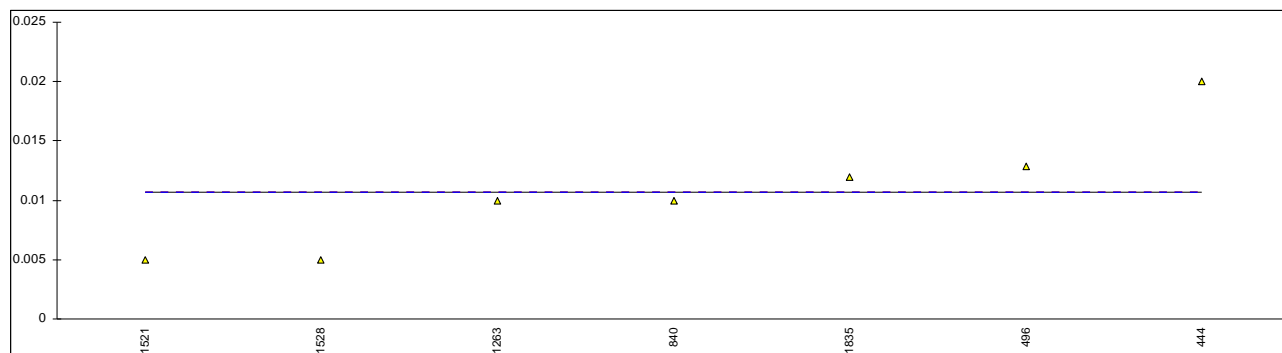
## Determination of Copper (Cu) on sample #0992; results in mg/kg

| lab  | method        | value  | mark | z(targ) | remarks |
|------|---------------|--------|------|---------|---------|
| 52   | D1688(mod)    | <0.05  |      | ----    |         |
| 150  | D1688         | <0.1   |      | ----    |         |
| 169  | D5863         | <0.04  |      | ----    |         |
| 171  | D1688         | <0.1   |      | ----    |         |
| 311  | INH-110       | <0.05  |      | ----    |         |
| 323  | EN15488       | <0.07  |      | ----    |         |
| 329  |               | ----   |      | ----    |         |
| 333  | EN15488       | <0.07  |      | ----    |         |
| 338  |               | ----   |      | ----    |         |
| 342  |               | ----   |      | ----    |         |
| 343  | EN15488       | <0.07  |      | ----    |         |
| 357  | INH-SP        | <0.02  |      | ----    |         |
| 395  |               | ----   |      | ----    |         |
| 444  |               | ----   |      | ----    |         |
| 463  |               | ----   |      | ----    |         |
| 468  | EN15488       | <0.1   |      | ----    |         |
| 494  |               | ----   |      | ----    |         |
| 496  |               | ----   |      | ----    |         |
| 541  | AAS           | <0.5   |      | ----    |         |
| 551  |               | ----   |      | ----    |         |
| 556  |               | ----   |      | ----    |         |
| 559  |               | ----   |      | ----    |         |
| 657  |               | ----   |      | ----    |         |
| 840  | ICP           | 0.004  |      | ----    |         |
| 862  | GB18350       | <0.01  |      | ----    |         |
| 867  |               | ----   |      | ----    |         |
| 902  |               | ----   |      | ----    |         |
| 913  |               | ----   |      | ----    |         |
| 922  |               | ----   |      | ----    |         |
| 1041 |               | ----   |      | ----    |         |
| 1059 |               | ----   |      | ----    |         |
| 1066 | EN15488       | <0.002 |      | ----    |         |
| 1079 | ICP           | <0.01  |      | ----    |         |
| 1154 | EN15837       | <0.030 |      | ----    |         |
| 1161 |               | ----   |      | ----    |         |
| 1201 | EN15488       | <0.1   |      | ----    |         |
| 1263 | INH-113       | 0.0048 |      | ----    |         |
| 1264 |               | ----   |      | ----    |         |
| 1521 | in house      | <0.02  |      | ----    |         |
| 1523 |               | ----   |      | ----    |         |
| 1528 | EN15488       | <0.01  |      | ----    |         |
| 1605 |               | ----   |      | ----    |         |
| 1726 |               | ----   |      | ----    |         |
| 1727 |               | ----   |      | ----    |         |
| 1835 |               | ----   |      | ----    |         |
| 1919 |               | ----   |      | ----    |         |
| 1933 | ISO11885      | <0.01  |      | ----    |         |
|      | normality     | n.a.   |      |         |         |
|      | n             | 2      |      |         |         |
|      | outliers      | 0      |      |         |         |
|      | mean (n)      | n.a.   |      |         |         |
|      | st.dev. (n)   | n.a.   |      |         |         |
|      | R(calc.)      | n.a.   |      |         |         |
|      | R(EN15488:07) | n.a.   |      |         |         |

Determination of Phosphorus (P) on sample #0992; results in mg/kg

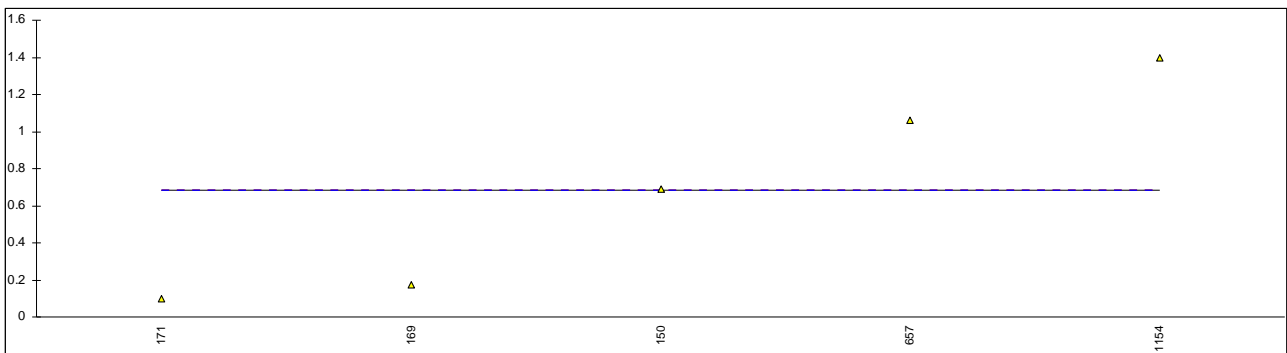
| lab  | method        | value  | mark | z(targ) | remarks |
|------|---------------|--------|------|---------|---------|
| 52   |               | ----   |      | ----    |         |
| 150  |               | ----   |      | ----    |         |
| 169  |               | ----   |      | ----    |         |
| 171  | D3231         | <0.01  |      | ----    |         |
| 311  | INH-110       | <0.2   |      | ----    |         |
| 323  | EN15487       | <0.19  |      | ----    |         |
| 329  | EN15487       | <0.19  |      | ----    |         |
| 333  | EN15487       | <0.19  |      | ----    |         |
| 338  |               | ----   |      | ----    |         |
| 342  |               | ----   |      | ----    |         |
| 343  | EN15487       | <0.15  |      | ----    |         |
| 357  | EN15487       | <0.15  |      | ----    |         |
| 395  |               | ----   |      | ----    |         |
| 444  | IP555         | 0.020  |      | ----    |         |
| 463  |               | ----   |      | ----    |         |
| 468  | EN15487       | <0.15  |      | ----    |         |
| 494  |               | ----   |      | ----    |         |
| 496  | EN15487       | 0.0129 |      | ----    |         |
| 541  |               | ----   |      | ----    |         |
| 551  |               | ----   |      | ----    |         |
| 556  |               | ----   |      | ----    |         |
| 559  |               | ----   |      | ----    |         |
| 657  |               | ----   |      | ----    |         |
| 840  | IP500(mod)    | 0.01   |      | ----    |         |
| 862  |               | ----   |      | ----    |         |
| 867  |               | ----   |      | ----    |         |
| 902  |               | ----   |      | ----    |         |
| 913  |               | ----   |      | ----    |         |
| 922  |               | ----   |      | ----    |         |
| 1041 |               | ----   |      | ----    |         |
| 1059 |               | ----   |      | ----    |         |
| 1066 |               | ----   |      | ----    |         |
| 1079 | ICP           | <0.1   |      | ----    |         |
| 1154 | EN15837       | <0.10  |      | ----    |         |
| 1161 |               | ----   |      | ----    |         |
| 1201 | EN15487       | <0.1   |      | ----    |         |
| 1263 | EN15487       | 0.010  |      | ----    |         |
| 1264 |               | ----   |      | ----    |         |
| 1521 | EN15487       | 0.005  |      | ----    |         |
| 1523 |               | ----   |      | ----    |         |
| 1528 | EN15487       | 0.005  |      | ----    |         |
| 1605 |               | ----   |      | ----    |         |
| 1726 | EN15487       | <0.15  |      | ----    |         |
| 1727 | EN15487       | <0.2   |      | ----    |         |
| 1835 | EN15487       | 0.012  |      | ----    |         |
| 1919 |               | ----   |      | ----    |         |
| 1933 | ISO11885      | <0.1   |      | ----    |         |
|      | normality     | OK     |      |         |         |
|      | n             | 7      |      |         |         |
|      | outliers      | 0      |      |         |         |
|      | mean (n)      | 0.011  |      |         |         |
|      | st.dev. (n)   | 0.0051 |      |         |         |
|      | R(calc.)      | 0.014  |      |         |         |
|      | R(EN15487:07) | n.a.   |      |         |         |

Range = 0.15 – 1.50 mg/L (0.118 – 1.18 mg/kg)



Determination of Sulphate on sample #0992; results in mg/kg

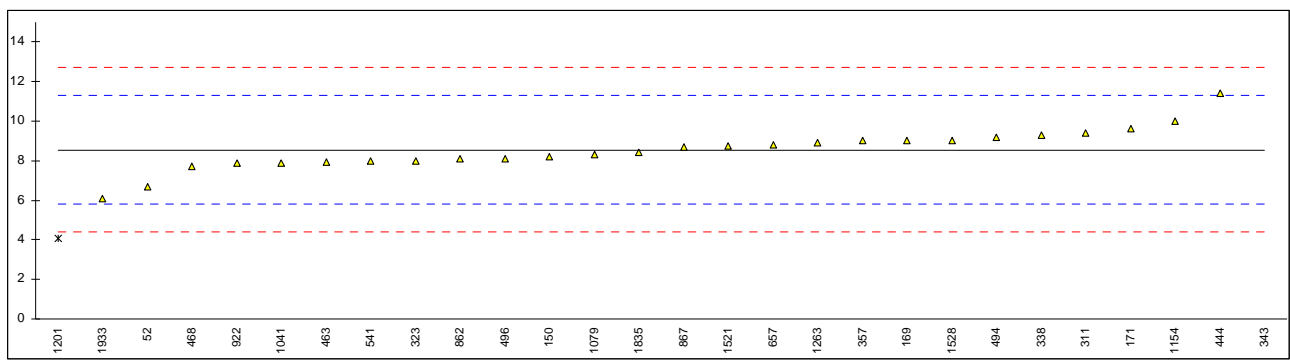
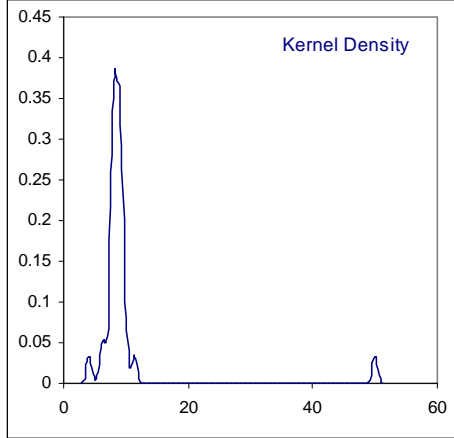
| lab  | method        | value | mark | z(targ) | remarks |
|------|---------------|-------|------|---------|---------|
| 52   | D7318         | <1    |      | ----    |         |
| 150  | D7328         | 0.69  |      | ----    |         |
| 169  | D7319         | 0.172 |      | ----    |         |
| 171  | D7328         | 0.1   |      | ----    |         |
| 311  | INH-518       | <10   |      | ----    |         |
| 323  |               | ----  |      | ----    |         |
| 329  |               | ----  |      | ----    |         |
| 333  |               | ----  |      | ----    |         |
| 338  |               | ----  |      | ----    |         |
| 342  |               | ----  |      | ----    |         |
| 343  | EN15492       | <1.1  |      | ----    |         |
| 357  |               | ----  |      | ----    |         |
| 395  |               | ----  |      | ----    |         |
| 444  |               | ----  |      | ----    |         |
| 463  |               | ----  |      | ----    |         |
| 468  |               | ----  |      | ----    |         |
| 494  |               | ----  |      | ----    |         |
| 496  |               | ----  |      | ----    |         |
| 541  |               | ----  |      | ----    |         |
| 551  |               | ----  |      | ----    |         |
| 556  | Turbidimetric | <1.0  |      | ----    |         |
| 559  |               | ----  |      | ----    |         |
| 657  | D7328         | 1.06  |      | ----    |         |
| 840  |               | ----  |      | ----    |         |
| 862  |               | ----  |      | ----    |         |
| 867  |               | ----  |      | ----    |         |
| 902  |               | ----  |      | ----    |         |
| 913  |               | ----  |      | ----    |         |
| 922  |               | ----  |      | ----    |         |
| 1041 |               | ----  |      | ----    |         |
| 1059 |               | ----  |      | ----    |         |
| 1066 |               | ----  |      | ----    |         |
| 1079 | EN15492       | <1    |      | ----    |         |
| 1154 | EN15492       | 1.40  |      | ----    |         |
| 1161 |               | ----  |      | ----    |         |
| 1201 |               | ----  |      | ----    |         |
| 1263 |               | ----  |      | ----    |         |
| 1264 |               | ----  |      | ----    |         |
| 1521 |               | ----  |      | ----    |         |
| 1523 |               | ----  |      | ----    |         |
| 1528 |               | ----  |      | ----    |         |
| 1605 |               | ----  |      | ----    |         |
| 1726 |               | ----  |      | ----    |         |
| 1727 |               | ----  |      | ----    |         |
| 1835 |               | ----  |      | ----    |         |
| 1919 |               | ----  |      | ----    |         |
| 1933 | in house      | <0.1  |      | ----    |         |
|      | normality     | OK    |      |         |         |
|      | n             | 5     |      |         |         |
|      | outliers      | 0     |      |         |         |
|      | mean (n)      | 0.68  |      |         |         |
|      | st.dev. (n)   | 0.561 |      |         |         |
|      | R(calc.)      | 1.57  |      |         |         |
|      | R(EN15492:07) | n.a.  |      |         |         |



Determination of Total Sulphur on sample #0992; results in mg/kg

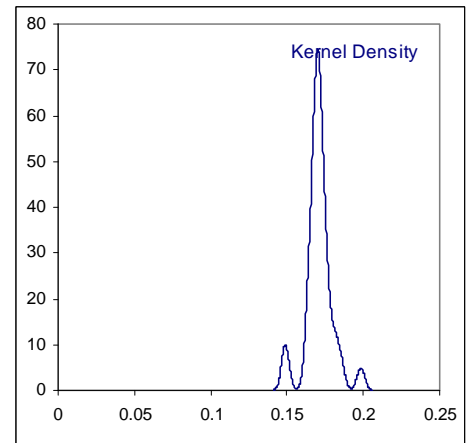
| lab  | method        | value | mark    | z(targ) | remarks                      |
|------|---------------|-------|---------|---------|------------------------------|
| 52   | D5453         | 6.7   |         | -1.35   |                              |
| 150  | D5453         | 8.2   |         | -0.26   |                              |
| 169  | D5453         | 9.029 |         | 0.34    |                              |
| 171  | D5453         | 9.63  |         | 0.78    |                              |
| 311  | D5453         | 9.4   |         | 0.61    |                              |
| 323  | EN15486       | 8     |         | -0.41   |                              |
| 329  |               | ----  |         | ----    |                              |
| 333  |               | ----  |         | ----    |                              |
| 338  | ISO20884      | 9.3   |         | 0.54    |                              |
| 342  |               | ----  |         | ----    |                              |
| 343  | EN15837       | 50.0  | G(0.01) | 30.07   |                              |
| 357  | D5453         | 9.0   |         | 0.32    |                              |
| 395  |               | ----  |         | ----    |                              |
| 444  | IP554         | 11.39 |         | 2.05    |                              |
| 463  | D5453         | 7.93  | C       | -0.46   | First reported 3.35          |
| 468  | EN15485       | 7.7   | C       | -0.62   | First reported 4.1           |
| 494  | D5453         | 9.2   |         | 0.47    |                              |
| 496  | EN15485       | 8.1   |         | -0.33   |                              |
| 541  | D5453         | 8     |         | -0.41   |                              |
| 551  |               | ----  |         | ----    |                              |
| 556  |               | ----  |         | ----    |                              |
| 559  |               | ----  |         | ----    |                              |
| 657  | D5453         | 8.8   |         | 0.18    |                              |
| 840  |               | ----  |         | ----    |                              |
| 862  | D5453         | 8.1   |         | -0.33   |                              |
| 867  | D3120         | 8.7   |         | 0.10    |                              |
| 902  |               | ----  |         | ----    |                              |
| 913  |               | ----  |         | ----    |                              |
| 922  | D5453         | 7.88  |         | -0.49   |                              |
| 1041 | EN20846       | 7.90  |         | -0.48   |                              |
| 1059 |               | ----  |         | ----    |                              |
| 1066 |               | ----  |         | ----    |                              |
| 1079 | D5453         | 8.3   |         | -0.19   |                              |
| 1154 | ISO20846      | 10.0  |         | 1.05    |                              |
| 1161 |               | ----  |         | ----    |                              |
| 1201 | EN15485       | 4.1   | G(0.01) | -3.23   |                              |
| 1263 | ISO20846      | 8.940 |         | 0.28    |                              |
| 1264 |               | ----  |         | ----    |                              |
| 1521 | EN15486       | 8.77  |         | 0.15    | Also reported 9.50 (EN15485) |
| 1523 |               | ----  |         | ----    |                              |
| 1528 | EN15486       | 9.04  |         | 0.35    |                              |
| 1605 |               | ----  |         | ----    |                              |
| 1726 |               | ----  |         | ----    |                              |
| 1727 |               | ----  |         | ----    |                              |
| 1835 | EN15485       | 8.4   |         | -0.11   |                              |
| 1919 |               | ----  |         | ----    |                              |
| 1933 | EN15486       | 6.11  |         | -1.78   |                              |
|      | normality     | OK    |         |         |                              |
|      | n             | 26    |         |         |                              |
|      | outliers      | 2     |         |         |                              |
|      | mean (n)      | 8.56  |         |         |                              |
|      | st.dev. (n)   | 1.031 |         |         |                              |
|      | R(calc.)      | 2.89  |         |         |                              |
|      | R(EN15485:07) | 3.86  |         |         |                              |

Compare with R(D5453) = 2.91

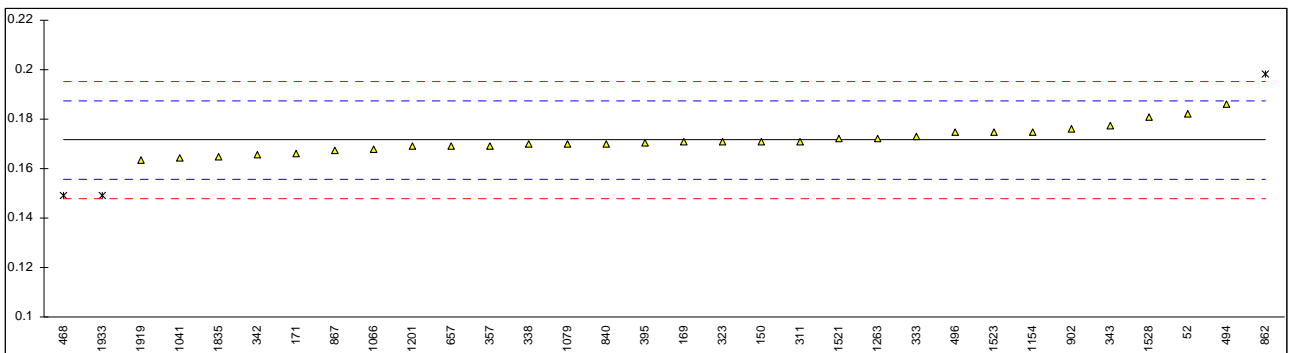


Determination of Water on sample #0992; results in %M/M == Coulometric ==

| lab  | method        | value   | mark     | z(targ) | remarks                                     |
|------|---------------|---------|----------|---------|---|
| 52   | E1064         | 0.182   |          | 1.33    |   |
| 150  | E1064         | 0.171   |          | -0.07   |   |
| 169  | E1064         | 0.1708  |          | -0.10   |   |
| 171  | E1064         | 0.1663  |          | -0.67   |   |
| 311  | E1064         | 0.171   |          | -0.07   |   |
| 323  | EN15489       | 0.171   |          | -0.07   |   |
| 329  |               | ----    |          | ----    |   |
| 333  | EN15489       | 0.173   |          | 0.18    |   |
| 338  | ISO12937      | 0.170   | C        | -0.20   | First reported in deviating unit 1700 mg/kg |
| 342  | E1064         | 0.1657  |          | -0.74   |   |
| 343  | EN15489       | 0.1775  |          | 0.76    |   |
| 357  | E1064         | 0.169   |          | -0.33   |   |
| 395  | EN15489       | 0.1704  |          | -0.15   |   |
| 444  |               | ----    |          | ----    |   |
| 463  |               | ----    |          | ----    |   |
| 468  | EN15489       | 0.149   | DG(0.01) | -2.87   |   |
| 494  | EN15489       | 0.1859  |          | 1.82    |   |
| 496  | EN15489       | 0.1746  |          | 0.39    |   |
| 541  |               | ----    |          | ----    |   |
| 551  |               | ----    |          | ----    |   |
| 556  |               | ----    |          | ----    |   |
| 559  |               | ----    |          | ----    |   |
| 657  | E1064         | 0.169   |          | -0.33   |   |
| 840  | E1064         | 0.1701  |          | -0.19   |   |
| 862  | E1064         | 0.1984  | G(0.05)  | 3.41    |   |
| 867  | E1064         | 0.1672  |          | -0.55   |   |
| 902  | E1064         | 0.176   |          | 0.56    |   |
| 913  |               | ----    |          | ----    |   |
| 922  |               | ----    |          | ----    |   |
| 1041 | EN15489       | 0.1643  | C        | -0.92   | First reported in deviating unit 1643 mg/kg |
| 1059 |               | ----    |          | ----    |   |
| 1066 | EN15489       | 0.168   |          | -0.45   |   |
| 1079 | EN15489       | 0.170   |          | -0.20   |   |
| 1154 | ISO12937      | 0.175   |          | 0.44    |   |
| 1161 |               | ----    |          | ----    |   |
| 1201 | EN15489       | 0.169   |          | -0.33   |   |
| 1263 | ISO12937      | 0.17222 |          | 0.08    |   |
| 1264 |               | ----    |          | ----    |   |
| 1521 | EN15489       | 0.172   |          | 0.06    |   |
| 1523 | E1064         | 0.175   |          | 0.44    |   |
| 1528 | EN15489       | 0.181   |          | 1.20    |   |
| 1605 |               | ----    |          | ----    |   |
| 1726 |               | ----    |          | ----    |   |
| 1727 |               | ----    |          | ----    |   |
| 1835 | EN15489       | 0.1647  |          | -0.87   |   |
| 1919 | EN15489       | 0.16344 |          | -1.03   |   |
| 1933 | EN15489       | 0.149   | DG(0.01) | -2.87   |   |
|      | normality     | OK      |          |         |   |
|      | n             | 29      |          |         |   |
|      | outliers      | 3       |          |         |   |
|      | mean (n)      | 0.1716  |          |         |   |
|      | st.dev. (n)   | 0.00529 |          |         |   |
|      | R(calc.)      | 0.0148  |          |         |   |
|      | R(EN15489:07) | 0.0220  |          |         |   |

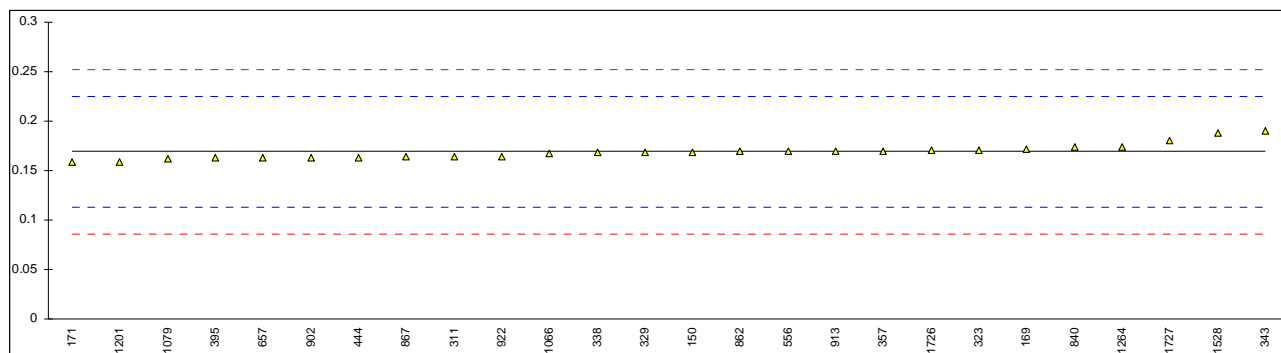
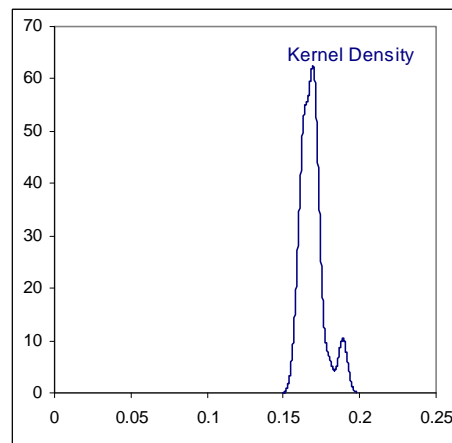


Compare with R(E1064) = 0.0292



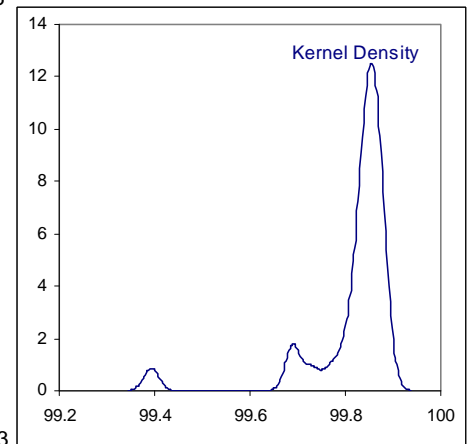
Determination of Water on sample #0992; results in %M/M == Titrimetric ==

| lab  | method      | value   | mark | z(targ) | remarks                                     |
|------|-------------|---------|------|---------|---|
| 52   |             | ----    |      | ----    |   |
| 150  | E203        | 0.169   |      | 0.00    |   |
| 169  | E203        | 0.1719  |      | 0.10    |   |
| 171  | E203        | 0.1583  |      | -0.39   |   |
| 311  | E203        | 0.164   |      | -0.18   |   |
| 323  | E203        | 0.171   |      | 0.07    |   |
| 329  | E203        | 0.169   |      | 0.00    |   |
| 333  |             | ----    |      | ----    |   |
| 338  | ISO6296     | 0.168   | C    | -0.04   | First reported in deviating unit 1680 mg/kg |
| 342  |             | ----    |      | ----    |   |
| 343  | E203        | 0.19    |      | 0.75    |   |
| 357  | E203        | 0.170   |      | 0.03    |   |
| 395  | E203        | 0.1630  |      | -0.22   |   |
| 444  | E203        | 0.1635  |      | -0.20   |   |
| 463  |             | ----    |      | ----    |   |
| 468  |             | ----    |      | ----    |   |
| 494  |             | ----    |      | ----    |   |
| 496  |             | ----    |      | ----    |   |
| 541  |             | ----    |      | ----    |   |
| 551  |             | ----    |      | ----    |   |
| 556  | E203        | 0.1696  |      | 0.02    |   |
| 559  |             | ----    |      | ----    |   |
| 657  | E203        | 0.163   |      | -0.22   |   |
| 840  | E203        | 0.1734  |      | 0.16    |   |
| 862  | E203        | 0.1692  |      | 0.01    |   |
| 867  | E203        | 0.1639  |      | -0.18   |   |
| 902  | E203        | 0.163   |      | -0.22   |   |
| 913  | E203        | 0.1699  |      | 0.03    |   |
| 922  | E203        | 0.1643  |      | -0.17   |   |
| 1041 |             | ----    |      | ----    |   |
| 1059 |             | ----    |      | ----    |   |
| 1066 | E203        | 0.167   |      | -0.07   |   |
| 1079 | E203        | 0.162   |      | -0.25   |   |
| 1154 |             | ----    |      | ----    |   |
| 1161 |             | ----    |      | ----    |   |
| 1201 | E203        | 0.159   |      | -0.36   |   |
| 1263 |             | ----    |      | ----    |   |
| 1264 | E203        | 0.1744  |      | 0.19    |   |
| 1521 |             | ----    |      | ----    |   |
| 1523 |             | ----    |      | ----    |   |
| 1528 | E203        | 0.188   |      | 0.68    |   |
| 1605 |             | ----    |      | ----    |   |
| 1726 | E203        | 0.1708  |      | 0.06    |   |
| 1727 | E203        | 0.18    |      | 0.39    |   |
| 1835 |             | ----    |      | ----    |   |
| 1919 |             | ----    |      | ----    |   |
| 1933 |             | ----    |      | ----    |   |
|      | normality   | OK      |      |         |   |
|      | n           | 26      |      |         |   |
|      | outliers    | 0       |      |         |   |
|      | mean (n)    | 0.1690  |      |         |   |
|      | st.dev. (n) | 0.00767 |      |         |   |
|      | R(calc.)    | 0.0215  |      |         |   |
|      | R(E203:08)  | 0.0780  |      |         |   |

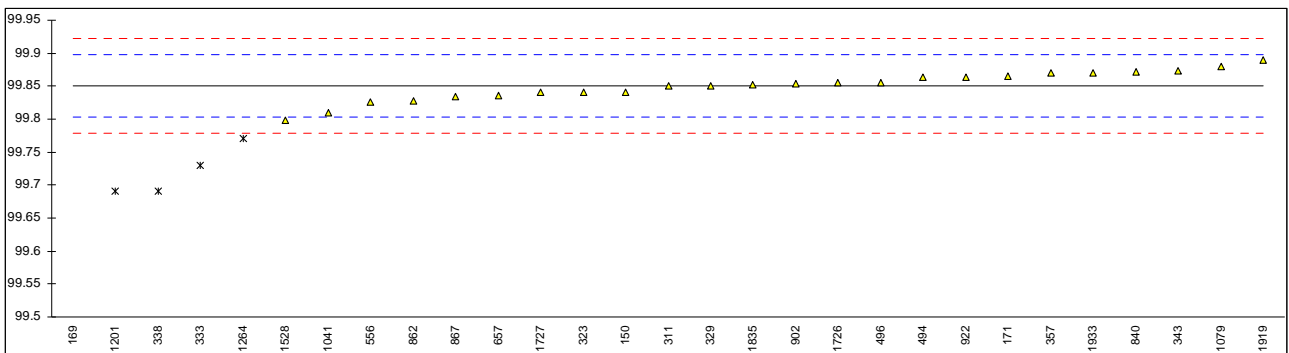


Determination of Purity on dry basis on sample #0993; results in %M/M

| lab  | method        | value   | mark       | z(targ) | remarks                         |
|------|---------------|---------|------------|---------|---------------------------------|
| 52   |               | ----    |            | ----    |                                 |
| 150  | INH-ETOH      | 99.84   |            | -0.45   |                                 |
| 169  | D5501         | 99.393  | G(0.01)    | -19.14  |                                 |
| 171  | D5501         | 99.866  |            | 0.64    |                                 |
| 311  | INH-529       | 99.85   |            | -0.03   |                                 |
| 323  | INH-155       | 99.84   |            | -0.45   |                                 |
| 329  | INH-155       | 99.85   |            | -0.03   |                                 |
| 333  | INH-2870      | 99.73   | G(0.01)    | -5.05   |                                 |
| 338  | GC            | 99.69   | C,DG(0.01) | -6.72   | First reported 99.67            |
| 342  |               | ----    |            | ----    |                                 |
| 343  | INH-0001      | 99.874  |            | 0.98    |                                 |
| 357  | INH-012       | 99.87   |            | 0.81    |                                 |
| 395  |               | ----    |            | ----    |                                 |
| 444  |               | ----    |            | ----    |                                 |
| 463  |               | ----    |            | ----    |                                 |
| 468  |               | ----    |            | ----    |                                 |
| 494  | EN15721       | 99.863  |            | 0.52    |                                 |
| 496  | EN15721       | 99.8561 |            | 0.23    |                                 |
| 541  |               | ----    |            | ----    |                                 |
| 551  |               | ----    |            | ----    |                                 |
| 556  | GLC           | 99.8261 |            | -1.03   |                                 |
| 559  |               | ----    |            | ----    |                                 |
| 657  | INH-01/07     | 99.8354 |            | -0.64   |                                 |
| 840  | INH-0001      | 99.871  |            | 0.85    |                                 |
| 862  | INH-01/07     | 99.827  |            | -0.99   |                                 |
| 867  | INH-0001      | 99.835  |            | -0.66   |                                 |
| 902  | INH-01/07     | 99.854  |            | 0.14    |                                 |
| 913  |               | ----    |            | ----    |                                 |
| 922  | INH-0001      | 99.863  |            | 0.52    |                                 |
| 1041 | EN15721       | 99.81   |            | -1.70   |                                 |
| 1059 |               | ----    |            | ----    |                                 |
| 1066 |               | ----    |            | ----    |                                 |
| 1079 | EN15721       | 99.88   |            | 1.23    |                                 |
| 1154 |               | ----    |            | ----    |                                 |
| 1161 |               | ----    |            | ----    |                                 |
| 1201 |               | 99.69   | DG(0.01)   | -6.72   |                                 |
| 1263 |               | ----    | W          | ----    | Withdrawn, first reported 99.73 |
| 1264 | D5501         | 99.77   | G(0.05)    | -3.37   |                                 |
| 1521 |               | ----    |            | ----    |                                 |
| 1523 |               | ----    |            | ----    |                                 |
| 1528 | EC2870        | 99.799  |            | -2.16   |                                 |
| 1605 |               | ----    |            | ----    |                                 |
| 1726 | in house      | 99.855  |            | 0.18    |                                 |
| 1727 | GC-FID        | 99.84   |            | -0.45   |                                 |
| 1835 | in house      | 99.8523 |            | 0.07    |                                 |
| 1919 |               | 99.89   |            | 1.64    |                                 |
| 1933 |               | 99.87   |            | 0.81    |                                 |
|      | normality     | OK      |            |         |                                 |
|      | n             | 24      |            |         |                                 |
|      | outliers      | 5       |            |         |                                 |
|      | mean (n)      | 99.851  |            |         |                                 |
|      | st.dev. (n)   | 0.0219  |            |         |                                 |
|      | R(calc.)      | 0.061   |            |         |                                 |
|      | R(EN15721:07) | 0.067   |            |         |                                 |

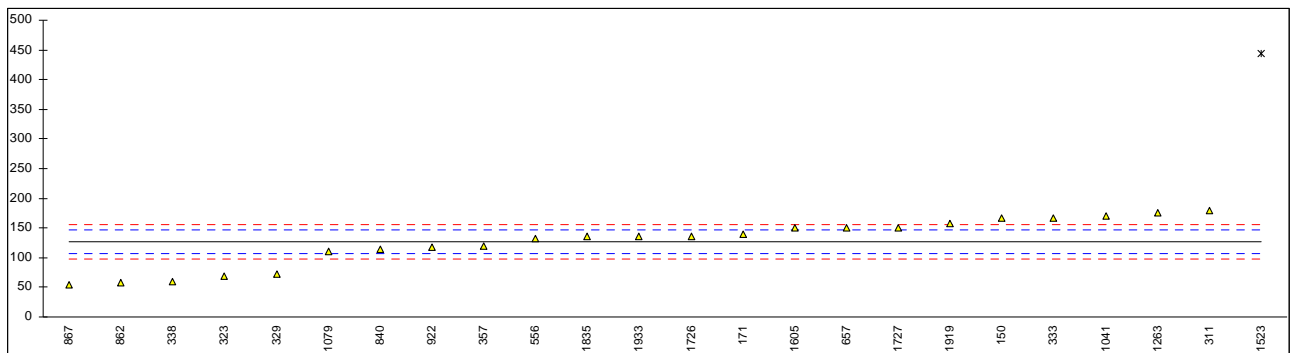
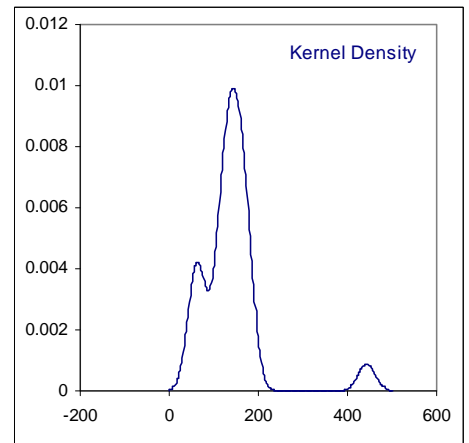


Compare with R(D5501) = 0.53



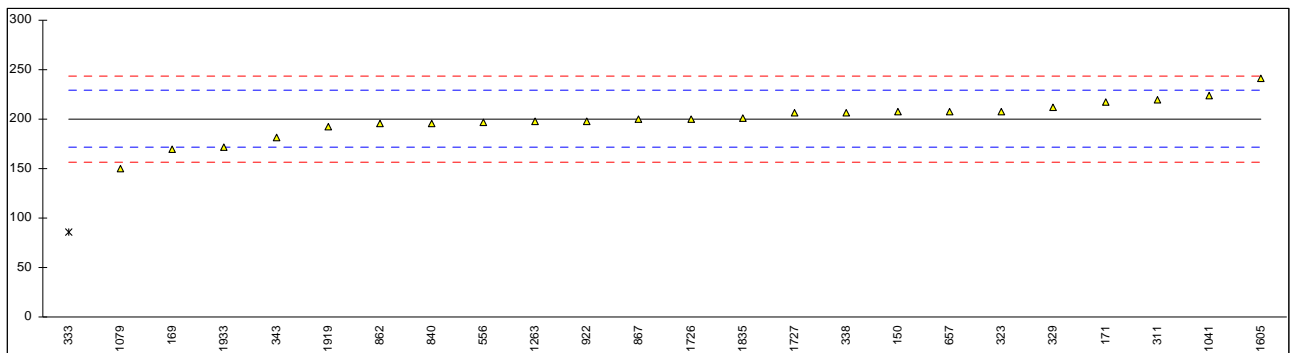
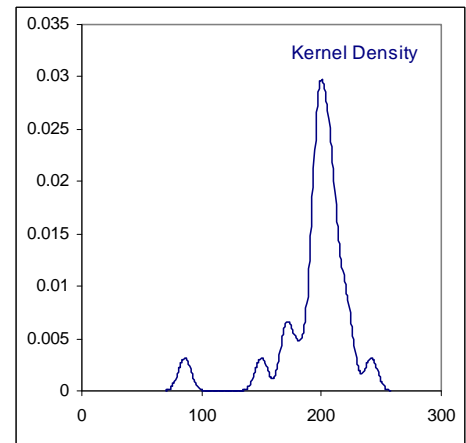
Determination of Acetaldehyde on sample #0993; results in mg/kg

| lab  | method      | value   | mark    | z(targ) | remarks         |
|------|-------------|---------|---------|---------|-----------------|
| 52   |             | ----    |         | ----    |                 |
| 150  | INH-ETOH    | 167.0   |         | 4.08    |                 |
| 169  |             | ----    |         | ----    |                 |
| 171  | D5501       | 140.1   |         | 1.33    |                 |
| 311  | INH-529     | 180     |         | 5.40    |                 |
| 323  | INH-155     | 68      |         | -6.02   |                 |
| 329  | INH-155     | 73      |         | -5.51   |                 |
| 333  | INH-2870    | 167     |         | 4.08    |                 |
| 338  | GC          | 59      |         | -6.94   |                 |
| 342  |             | ----    |         | ----    |                 |
| 343  |             | ----    |         | ----    |                 |
| 357  | INH-012     | 120     |         | -0.72   |                 |
| 395  |             | ----    |         | ----    |                 |
| 444  |             | ----    |         | ----    |                 |
| 463  |             | ----    |         | ----    |                 |
| 468  |             | ----    |         | ----    |                 |
| 494  |             | ----    |         | ----    |                 |
| 496  |             | ----    |         | ----    |                 |
| 541  |             | ----    |         | ----    |                 |
| 551  |             | ----    |         | ----    |                 |
| 556  | GLC         | 132.911 |         | 0.60    |                 |
| 559  |             | ----    |         | ----    |                 |
| 657  | INH-01/07   | 149.8   |         | 2.32    |                 |
| 840  | INH-0001    | 115     |         | -1.23   |                 |
| 862  | INH-01/07   | 58      |         | -7.04   |                 |
| 867  | INH-0001    | 55.1    |         | -7.34   |                 |
| 902  |             | ----    |         | ----    |                 |
| 913  |             | ----    |         | ----    |                 |
| 922  | INH-0001    | 118.3   |         | -0.89   |                 |
| 1041 | EN15721     | 171.1   |         | 4.49    |                 |
| 1059 |             | ----    |         | ----    |                 |
| 1066 |             | ----    |         | ----    |                 |
| 1079 | EN15721     | 110     |         | -1.74   |                 |
| 1154 |             | ----    |         | ----    |                 |
| 1161 |             | ----    |         | ----    |                 |
| 1201 |             | <10     |         | <-11.94 | False negative? |
| 1263 | D5501       | 174.9   |         | 4.88    |                 |
| 1264 |             | ----    |         | ----    |                 |
| 1521 |             | ----    |         | ----    |                 |
| 1523 | D5501       | 443.4   | G(0.01) | 32.27   |                 |
| 1528 | EC2870      | <10     |         | <-11.94 | False negative? |
| 1605 | in house    | 149.6   |         | 2.30    |                 |
| 1726 | in house    | 136     |         | 0.91    |                 |
| 1727 | GC-FID      | 150     |         | 2.34    |                 |
| 1835 | in house    | 135     |         | 0.81    |                 |
| 1919 |             | 157     |         | 3.06    |                 |
| 1933 |             | 135     |         | 0.81    |                 |
|      | normality   | OK      |         |         |                 |
|      | n           | 23      |         |         |                 |
|      | outliers    | 1       |         |         |                 |
|      | mean (n)    | 127.0   |         |         |                 |
|      | st.dev. (n) | 39.68   |         |         |                 |
|      | R(calc.)    | 111.1   |         |         |                 |
|      | R(Horwitz)  | 27.4    |         |         |                 |



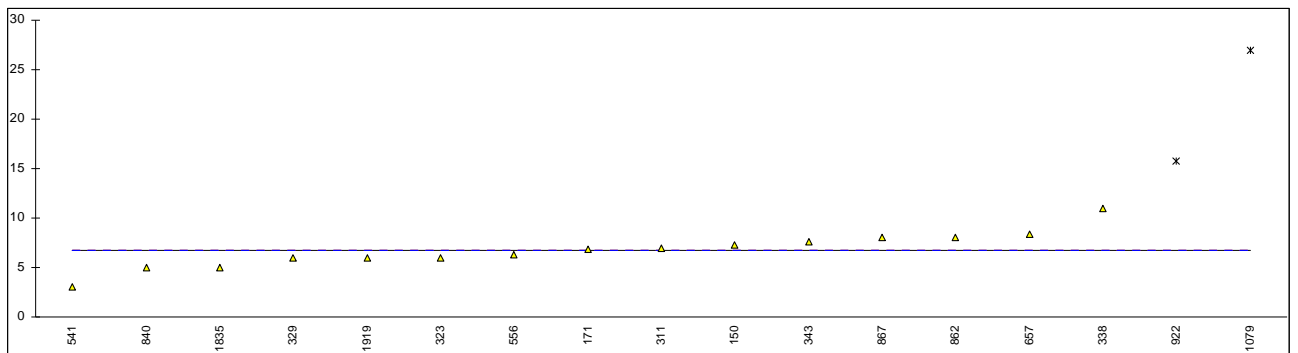
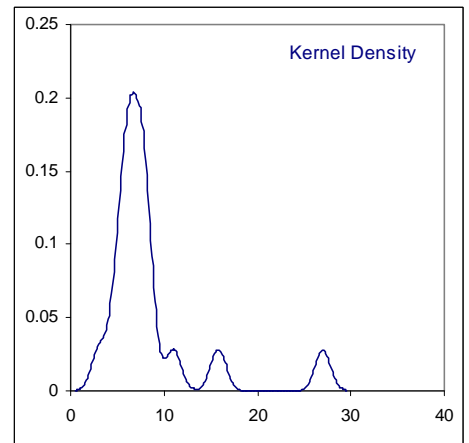
Determination of Acetal on sample #0993; results in mg/kg

| lab  | method      | value   | mark    | z(targ) | remarks            |
|------|-------------|---------|---------|---------|--------------------|
| 52   |             | ----    |         | ----    |                    |
| 150  | INH-ETOH    | 207.4   |         | 0.51    |                    |
| 169  | D5501       | 170     |         | -2.08   |                    |
| 171  | D5501       | 217.2   |         | 1.19    |                    |
| 311  | INH-529     | 220     |         | 1.38    |                    |
| 323  | INH-155     | 208     |         | 0.55    |                    |
| 329  | INH-155     | 212     |         | 0.83    |                    |
| 333  | INH-2870    | 86      | G(0.01) | -7.91   |                    |
| 338  | GC          | 207     | C       | 0.48    | First reported 260 |
| 342  |             | ----    |         | ----    |                    |
| 343  | EN15721     | 181.7   |         | -1.27   |                    |
| 357  |             | ----    |         | ----    |                    |
| 395  |             | ----    |         | ----    |                    |
| 444  |             | ----    |         | ----    |                    |
| 463  |             | ----    |         | ----    |                    |
| 468  |             | ----    |         | ----    |                    |
| 494  |             | ----    |         | ----    |                    |
| 496  |             | ----    |         | ----    |                    |
| 541  |             | ----    |         | ----    |                    |
| 551  |             | ----    |         | ----    |                    |
| 556  | GLC         | 196.202 |         | -0.27   |                    |
| 559  |             | ----    |         | ----    |                    |
| 657  | INH-01/07   | 207.6   |         | 0.52    |                    |
| 840  | INH-0001    | 196     |         | -0.28   |                    |
| 862  | INH-01/07   | 196     |         | -0.28   |                    |
| 867  | INH-0001    | 199.9   |         | -0.01   |                    |
| 902  |             | ----    |         | ----    |                    |
| 913  |             | ----    |         | ----    |                    |
| 922  | INH-0001    | 198.1   |         | -0.13   |                    |
| 1041 | EN15721     | 223.8   |         | 1.65    |                    |
| 1059 |             | ----    |         | ----    |                    |
| 1066 |             | ----    |         | ----    |                    |
| 1079 | EN15721     | 150     |         | -3.47   |                    |
| 1154 |             | ----    |         | ----    |                    |
| 1161 |             | ----    |         | ----    |                    |
| 1201 |             | <10     |         | <-13.18 | False negative?    |
| 1263 | D5501       | 197.3   |         | -0.19   |                    |
| 1264 |             | ----    |         | ----    |                    |
| 1521 |             | ----    |         | ----    |                    |
| 1523 |             | ----    |         | ----    |                    |
| 1528 |             | ----    |         | ----    |                    |
| 1605 | in house    | 241.7   |         | 2.89    |                    |
| 1726 | in house    | 200     |         | 0.00    |                    |
| 1727 | GC-FID      | 206     |         | 0.41    |                    |
| 1835 | in house    | 201     |         | 0.07    |                    |
| 1919 |             | 192     |         | -0.56   |                    |
| 1933 |             | 172     |         | -1.94   |                    |
|      | normality   | not OK  |         |         |                    |
|      | n           | 23      |         |         |                    |
|      | outliers    | 1       |         |         |                    |
|      | mean (n)    | 200.0   |         |         |                    |
|      | st.dev. (n) | 19.13   |         |         |                    |
|      | R(calc.)    | 53.6    |         |         |                    |
|      | R(Horwitz)  | 40.4    |         |         |                    |



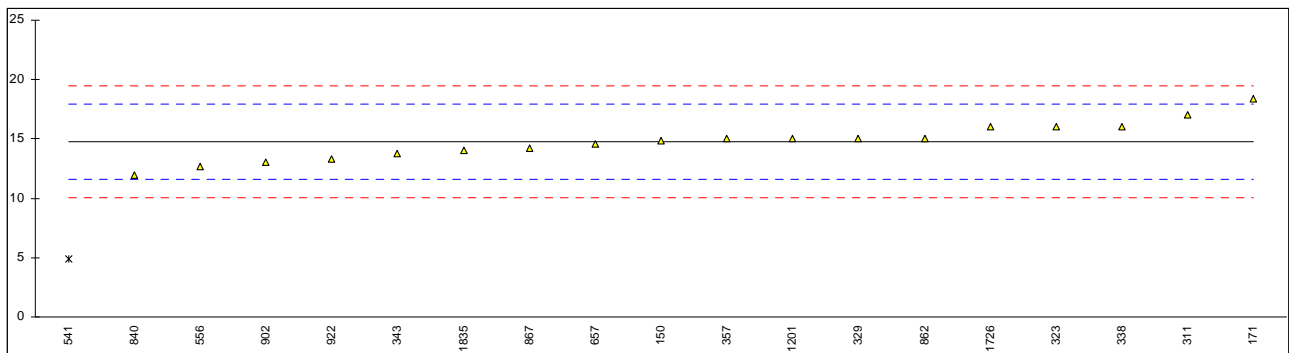
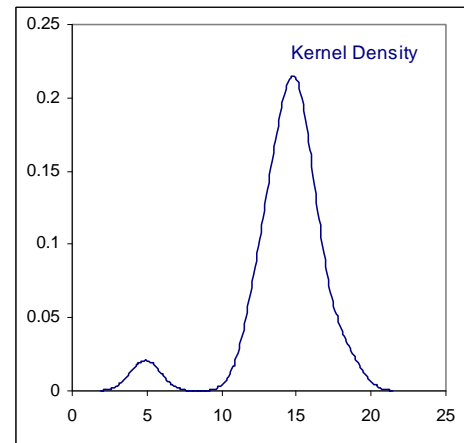
Determination of Acetone on sample #0993; results in mg/kg

| lab  | method      | value  | mark    | z(targ) | remarks              |
|------|-------------|--------|---------|---------|----------------------|
| 52   |             | ----   |         | ----    |                      |
| 150  | INH-ETOH    | 7.3    |         | ----    |                      |
| 169  |             | ----   |         | ----    |                      |
| 171  | D5501       | 6.8    |         | ----    |                      |
| 311  | INH-529     | 7      |         | ----    |                      |
| 323  | INH-155     | 6      |         | ----    |                      |
| 329  | INH-155     | 6      |         | ----    |                      |
| 333  |             | ----   |         | ----    |                      |
| 338  | GC          | 11     |         | ----    |                      |
| 342  |             | ----   |         | ----    |                      |
| 343  | INH-001     | 7.6    |         | ----    |                      |
| 357  | INH-012     | <10    |         | ----    |                      |
| 395  |             | ----   |         | ----    |                      |
| 444  |             | ----   |         | ----    |                      |
| 463  |             | ----   |         | ----    |                      |
| 468  |             | ----   |         | ----    |                      |
| 494  |             | ----   |         | ----    |                      |
| 496  |             | ----   |         | ----    |                      |
| 541  | INH-0001    | 3.03   |         | ----    |                      |
| 551  |             | ----   |         | ----    |                      |
| 556  | GLC         | 6.329  |         | ----    |                      |
| 559  |             | ----   |         | ----    |                      |
| 657  | INH-01/07   | 8.4    |         | ----    |                      |
| 840  | INH-0001    | 5      |         | ----    |                      |
| 862  | INH-01/07   | 8      |         | ----    |                      |
| 867  | INH-0001    | 8.0    |         | ----    |                      |
| 902  | INH-01/07   | <10    |         | ----    |                      |
| 913  |             | ----   |         | ----    |                      |
| 922  | INH-0001    | 15.8   | G(0.01) | ----    | False positive?      |
| 1041 | EN15721     | <10    | C       | ----    | First reported 568.6 |
| 1059 |             | ----   |         | ----    |                      |
| 1066 |             | ----   |         | ----    |                      |
| 1079 | INH-GC      | 27     | G(0.01) | ----    | False positive?      |
| 1154 |             | ----   |         | ----    |                      |
| 1161 |             | ----   |         | ----    |                      |
| 1201 |             | <10    |         | ----    |                      |
| 1263 | D5501       | <5     |         | ----    |                      |
| 1264 | D5501       | N.D.   |         | ----    |                      |
| 1521 |             | ----   |         | ----    |                      |
| 1523 |             | ----   |         | ----    |                      |
| 1528 |             | ----   |         | ----    |                      |
| 1605 |             | ----   |         | ----    |                      |
| 1726 | in house    | N.D.   |         | ----    |                      |
| 1727 | GC-FID      | <10    |         | ----    |                      |
| 1835 | in house    | 5      |         | ----    |                      |
| 1919 |             | 6      |         | ----    |                      |
| 1933 |             | <7     |         | ----    |                      |
|      | normality   | OK     |         |         |                      |
|      | n           | 15     |         |         |                      |
|      | outliers    | 2      |         |         |                      |
|      | mean (n)    | 6.76   |         |         |                      |
|      | st.dev. (n) | 1.829  |         |         |                      |
|      | R(calc.)    | 5.12   |         |         |                      |
|      | R(Horwitz)  | (2.27) |         |         |                      |



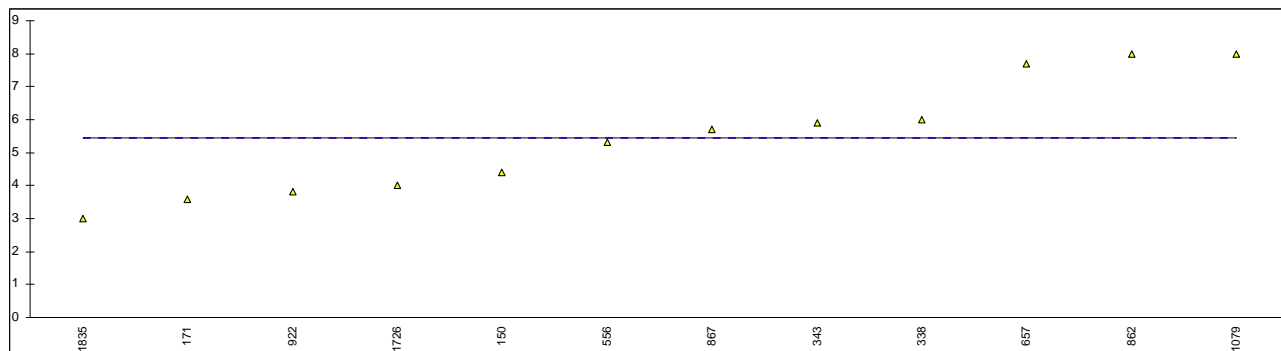
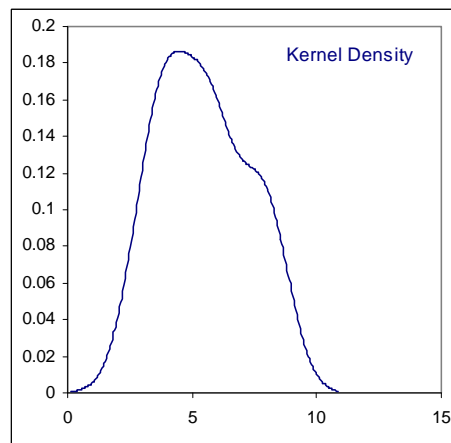
Determination of Benzene on sample #0993; results in mg/kg

| lab         | method    | value  | mark      | z(targ)      | remarks             |
|-------------|-----------|--------|-----------|--------------|---------------------|
| 52          |           | ----   |           | ----         |                     |
| 150         | INH-ETOH  | 14.9   |           | 0.08         |                     |
| 169         |           | ----   |           | ----         |                     |
| 171         | D5501     | 18.4   |           | 2.30         |                     |
| 311         | INH-529   | 17     |           | 1.42         |                     |
| 323         | INH-155   | 16     |           | 0.78         |                     |
| 329         | INH-155   | 15     |           | 0.15         |                     |
| 333         |           | ----   |           | ----         |                     |
| 338         | GC        | 16     | C         | 0.78         | First reported 25   |
| 342         |           | ----   |           | ----         |                     |
| 343         | INH-001   | 13.8   |           | -0.62        |                     |
| 357         | INH-012   | 15     |           | 0.15         |                     |
| 395         |           | ----   |           | ----         |                     |
| 444         |           | ----   |           | ----         |                     |
| 463         |           | ----   |           | ----         |                     |
| 468         |           | ----   |           | ----         |                     |
| 494         |           | ----   |           | ----         |                     |
| 496         |           | ----   |           | ----         |                     |
| 541         | INH-0001  | 4.90   | C,G(0.01) | -6.26        | First reported 5.60 |
| 551         |           | ----   |           | ----         |                     |
| 556         | GLC       | 12.658 |           | -1.34        |                     |
| 559         |           | ----   |           | ----         |                     |
| 657         | INH-01/07 | 14.6   |           | -0.11        |                     |
| 840         | INH-0001  | 12     |           | -1.76        |                     |
| 862         | INH-01/07 | 15     |           | 0.15         |                     |
| 867         | INH-0001  | 14.2   |           | -0.36        |                     |
| 902         | INH-01/07 | 13     |           | -1.12        |                     |
| 913         |           | ----   |           | ----         |                     |
| 922         | INH-0001  | 13.3   |           | -0.93        |                     |
| 1041        | EN15721   | <10    |           | <-3.03       | False negative?     |
| 1059        |           | ----   |           | ----         |                     |
| 1066        |           | ----   |           | ----         |                     |
| 1079        | INH-GC    | <1     |           | <-8.74       | False negative?     |
| 1154        |           | ----   |           | ----         |                     |
| 1161        |           | ----   |           | ----         |                     |
| 1201        |           | 15     |           | 0.15         |                     |
| 1263        |           | ----   |           | ----         |                     |
| 1264        |           | ----   |           | ----         |                     |
| 1521        |           | ----   |           | ----         |                     |
| 1523        |           | ----   |           | ----         |                     |
| 1528        |           | ----   |           | ----         |                     |
| 1605        |           | ----   |           | ----         |                     |
| 1726        | in house  | 16     |           | 0.78         |                     |
| 1727        |           | ----   |           | ----         |                     |
| 1835        | in house  | 14     |           | -0.49        |                     |
| 1919        |           | ----   |           | ----         |                     |
| 1933        |           | <7     |           | <-4.93       | False negative?     |
| normality   | OK        |        |           |              |                     |
| n           | 18        |        |           |              |                     |
| outliers    | 1         |        |           |              |                     |
| mean (n)    | 14.77     |        |           |              |                     |
| st.dev. (n) | 1.576     |        |           |              |                     |
| R(calc.)    | 4.41      |        |           |              |                     |
| R(Horwitz)  | 4.41      |        |           |              |                     |
|             |           |        |           | <u>Spike</u> |                     |
|             |           |        |           | 15.5         | Recovery = <95%     |



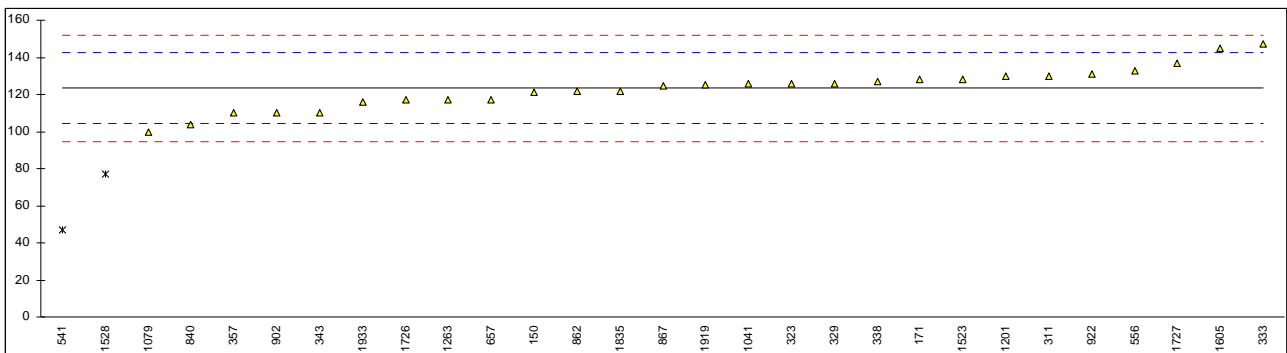
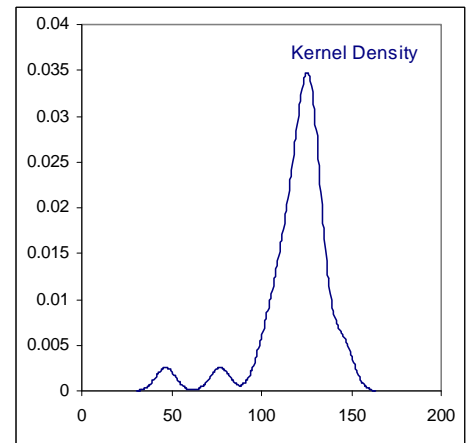
Determination of Cyclohexane on sample #0993; results in mg/kg

| lab  | method      | value  | mark | z(targ) | remarks |
|------|-------------|--------|------|---------|---------|
| 52   |             | ----   |      | ----    |         |
| 150  | INH-ETOH    | 4.4    |      | ----    |         |
| 169  |             | ----   |      | ----    |         |
| 171  | D5501       | 3.6    |      | ----    |         |
| 311  | INH-529     | <5     |      | ----    |         |
| 323  | INH-155     | <5     |      | ----    |         |
| 329  | INH-155     | <5     |      | ----    |         |
| 333  |             | ----   |      | ----    |         |
| 338  | GC          | 6      |      | ----    |         |
| 342  |             | ----   |      | ----    |         |
| 343  | INH-001     | 5.9    |      | ----    |         |
| 357  | INH-012     | <10    |      | ----    |         |
| 395  |             | ----   |      | ----    |         |
| 444  |             | ----   |      | ----    |         |
| 463  |             | ----   |      | ----    |         |
| 468  |             | ----   |      | ----    |         |
| 494  |             | ----   |      | ----    |         |
| 496  |             | ----   |      | ----    |         |
| 541  |             | ----   |      | ----    |         |
| 551  |             | ----   |      | ----    |         |
| 556  | GLC         | 5.3063 |      | ----    |         |
| 559  |             | ----   |      | ----    |         |
| 657  | INH-01/07   | 7.7    |      | ----    |         |
| 840  | INH-0001    | <1     |      | ----    |         |
| 862  | INH-01/07   | 8      |      | ----    |         |
| 867  | INH-0001    | 5.7    |      | ----    |         |
| 902  |             | ----   |      | ----    |         |
| 913  |             | ----   |      | ----    |         |
| 922  | INH-0001    | 3.8    |      | ----    |         |
| 1041 |             | ----   |      | ----    |         |
| 1059 |             | ----   |      | ----    |         |
| 1066 |             | ----   |      | ----    |         |
| 1079 | INH-GC      | 8      |      | ----    |         |
| 1154 |             | ----   |      | ----    |         |
| 1161 |             | ----   |      | ----    |         |
| 1201 |             | <10    |      | ----    |         |
| 1263 |             | ----   |      | ----    |         |
| 1264 |             | ----   |      | ----    |         |
| 1521 |             | ----   |      | ----    |         |
| 1523 |             | ----   |      | ----    |         |
| 1528 |             | ----   |      | ----    |         |
| 1605 |             | ----   |      | ----    |         |
| 1726 | in house    | 4      |      | ----    |         |
| 1727 |             | ----   |      | ----    |         |
| 1835 | in house    | 3      |      | ----    |         |
| 1919 |             | ----   |      | ----    |         |
| 1933 |             | <7     |      | ----    |         |
|      | normality   | OK     |      |         |         |
|      | n           | 12     |      |         |         |
|      | outliers    | 0      |      |         |         |
|      | mean (n)    | 5.45   |      |         |         |
|      | st.dev. (n) | 1.758  |      |         |         |
|      | R(calc.)    | 4.92   |      |         |         |
|      | R(Horwitz)  | (1.89) |      |         |         |



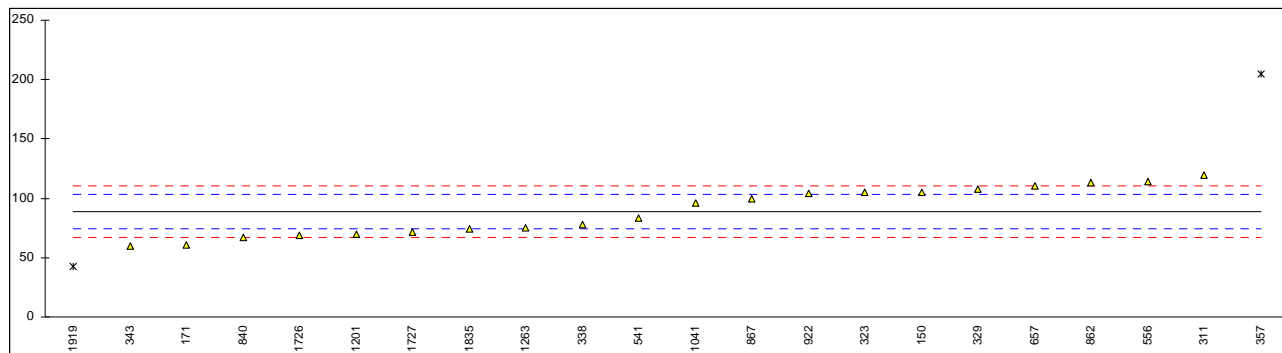
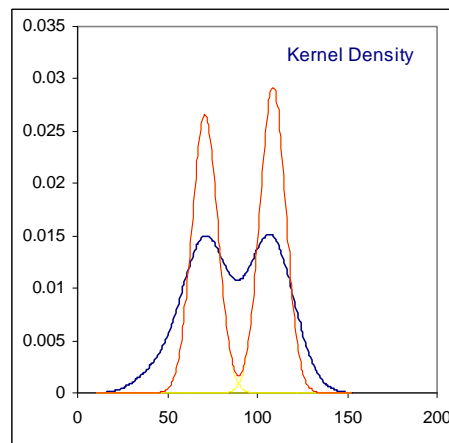
Determination of Ethylacetate on sample #0993; results in mg/kg

| lab  | method      | value   | mark      | z(targ) | remarks              |
|------|-------------|---------|-----------|---------|----------------------|
| 52   |             | ----    |           | ----    |                      |
| 150  | INH-ETOH    | 121.0   |           | -0.24   |                      |
| 169  |             | ----    |           | ----    |                      |
| 171  | D5501       | 127.9   |           | 0.48    |                      |
| 311  | INH-529     | 130     |           | 0.70    |                      |
| 323  | INH-155     | 126     |           | 0.28    |                      |
| 329  | INH-155     | 126     |           | 0.28    |                      |
| 333  | INH-2870    | 147     |           | 2.48    |                      |
| 338  | GC          | 127     | C         | 0.39    | First reported 184   |
| 342  |             | ----    |           | ----    |                      |
| 343  | EN15721     | 110.3   |           | -1.36   |                      |
| 357  | INH-012     | 110     |           | -1.39   |                      |
| 395  |             | ----    |           | ----    |                      |
| 444  |             | ----    |           | ----    |                      |
| 463  |             | ----    |           | ----    |                      |
| 468  |             | ----    |           | ----    |                      |
| 494  |             | ----    |           | ----    |                      |
| 496  |             | ----    |           | ----    |                      |
| 541  | INH-0001    | 46.76   | C,G(0.01) | -8.01   | First reported 51.40 |
| 551  |             | ----    |           | ----    |                      |
| 556  | GLC         | 132.911 |           | 1.00    |                      |
| 559  |             | ----    |           | ----    |                      |
| 657  | INH-01/07   | 117.3   |           | -0.63   |                      |
| 840  | INH-0001    | 104     |           | -2.02   |                      |
| 862  | INH-01/07   | 122     |           | -0.14   |                      |
| 867  | INH-0001    | 124.5   |           | 0.12    |                      |
| 902  | INH-01/07   | 110     |           | -1.39   |                      |
| 913  |             | ----    |           | ----    |                      |
| 922  | INH-0001    | 131.1   |           | 0.81    |                      |
| 1041 | EN15721     | 125.7   |           | 0.25    |                      |
| 1059 |             | ----    |           | ----    |                      |
| 1066 |             | ----    |           | ----    |                      |
| 1079 | EN15721     | 100     |           | -2.44   |                      |
| 1154 |             | ----    |           | ----    |                      |
| 1161 |             | ----    |           | ----    |                      |
| 1201 |             | 130     |           | 0.70    |                      |
| 1263 | D5501       | 117.0   |           | -0.66   |                      |
| 1264 |             | ----    |           | ----    |                      |
| 1521 |             | ----    |           | ----    |                      |
| 1523 | D5501       | 128.1   |           | 0.50    |                      |
| 1528 | EC2870      | 77      | G(0.01)   | -4.85   |                      |
| 1605 | in house    | 144.7   |           | 2.24    |                      |
| 1726 | in house    | 117     |           | -0.66   |                      |
| 1727 | GC-FID      | 137     |           | 1.43    |                      |
| 1835 | in house    | 122     |           | -0.14   |                      |
| 1919 |             | 125     |           | 0.18    |                      |
| 1933 |             | 116     |           | -0.77   |                      |
|      | normality   | OK      |           |         |                      |
|      | n           | 27      |           |         |                      |
|      | outliers    | 2       |           |         |                      |
|      | mean (n)    | 123.3   |           |         |                      |
|      | st.dev. (n) | 11.00   |           |         |                      |
|      | R(calc.)    | 30.8    |           |         |                      |
|      | R(Horwitz)  | 26.8    |           |         |                      |



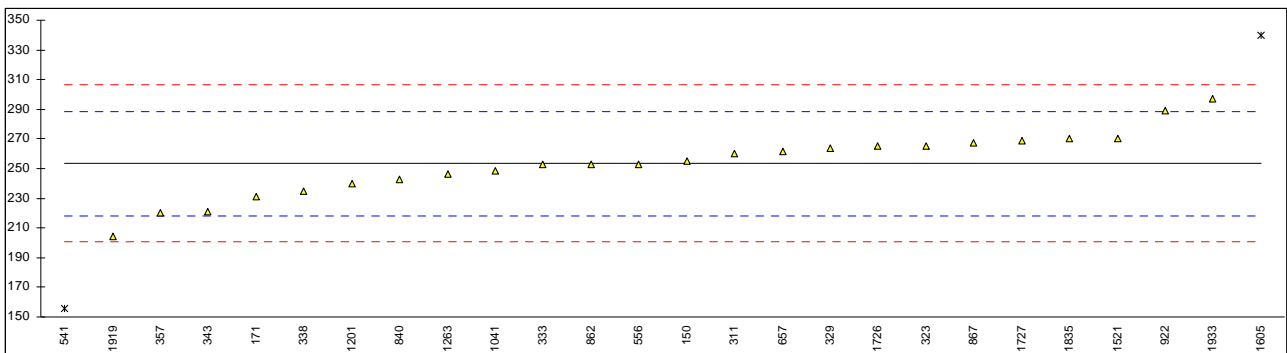
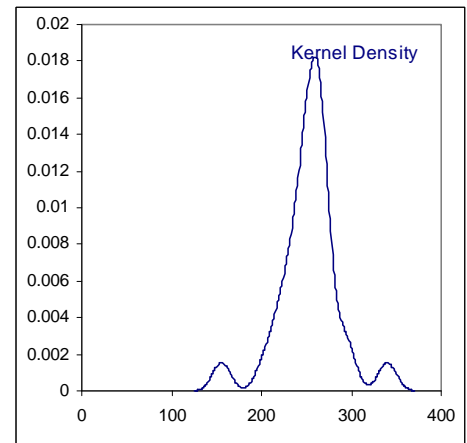
Determination of Iso Amyl Alcohol on sample #0993; results in mg/kg

| lab         | method    | value   | mark    | z(targ)        | remarks         |
|-------------|-----------|---------|---------|----------------|-----------------|
| 52          |           | ----    |         | ----           |                 |
| 150         | INH-ETOH  | 105.1   |         | ----           |                 |
| 169         |           | ----    |         | ----           |                 |
| 171         | D5501     | 60.6    |         | ----           |                 |
| 311         | INH-529   | 120     |         | ----           |                 |
| 323         | INH-155   | 105     |         | ----           |                 |
| 329         | INH-155   | 108     |         | ----           |                 |
| 333         |           | ----    |         | ----           |                 |
| 338         | GC        | 78      |         | ----           |                 |
| 342         |           | ----    |         | ----           |                 |
| 343         | EN15721   | 59.8    |         | ----           |                 |
| 357         | INH-012   | 205     | G(0.01) | ----           | false positive? |
| 395         |           | ----    |         | ----           |                 |
| 444         |           | ----    |         | ----           |                 |
| 463         |           | ----    |         | ----           |                 |
| 468         |           | ----    |         | ----           |                 |
| 494         |           | ----    |         | ----           |                 |
| 496         |           | ----    |         | ----           |                 |
| 541         | INH-0001  | 83.07   |         | ----           |                 |
| 551         |           | ----    |         | ----           |                 |
| 556         | GLC       | 113.924 |         | ----           |                 |
| 559         |           | ----    |         | ----           |                 |
| 657         | INH-01/07 | 110.2   |         | ----           |                 |
| 840         | INH-0001  | 67      |         | ----           |                 |
| 862         | INH-01/07 | 113     |         | ----           |                 |
| 867         | INH-0001  | 99.4    |         | ----           |                 |
| 902         |           | ----    |         | ----           |                 |
| 913         |           | ----    |         | ----           |                 |
| 922         | INH-0001  | 104.2   |         | ----           |                 |
| 1041        | EN15721   | 96.3    |         | ----           |                 |
| 1059        |           | ----    |         | ----           |                 |
| 1066        |           | ----    |         | ----           |                 |
| 1079        |           | ----    |         | ----           |                 |
| 1154        |           | ----    |         | ----           |                 |
| 1161        |           | ----    |         | ----           |                 |
| 1201        |           | 70      |         | ----           |                 |
| 1263        | D5501     | 75.0    |         | ----           |                 |
| 1264        |           | ----    |         | ----           |                 |
| 1521        | EN15721   | <100    |         | ----           |                 |
| 1523        |           | ----    |         | ----           |                 |
| 1528        |           | ----    |         | ----           |                 |
| 1605        |           | ----    |         | ----           |                 |
| 1726        | in house  | 69      |         | ----           |                 |
| 1727        | GC-FID    | 72      |         | ----           |                 |
| 1835        | in house  | 74      |         | ----           |                 |
| 1919        |           | 43      | G(0.05) | ----           |                 |
| 1933        |           | ----    |         | ----           |                 |
|             |           |         |         | <u>Group 1</u> | <u>Group 2</u>  |
| normality   | OK        |         |         | OK             | OK              |
| n           | 20        |         |         | 10             | 10              |
| outliers    | 2         |         |         | 1              | 1               |
| mean (n)    | 89.18     |         |         | 70.85          | 107.52          |
| st.dev. (n) | 20.061    |         |         | 7.265          | 7.059           |
| R(calc.)    | 56.17     |         |         | 20.34          | 19.77           |
| R(Horwitz)  | (20.32)   |         |         | 16.72          | 23.82           |



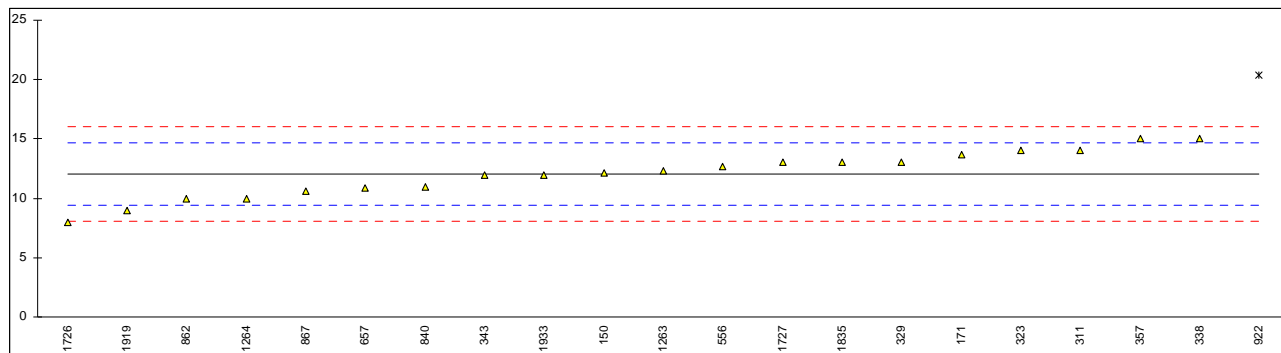
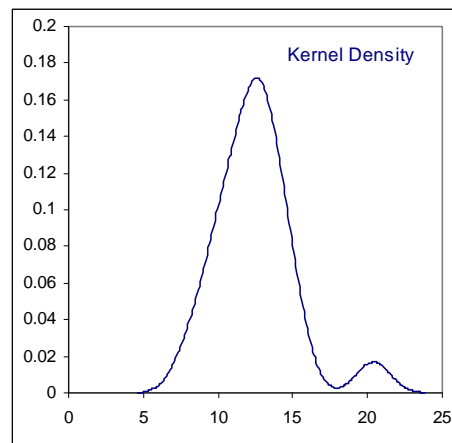
Determination of Isobutanol on sample #0993; results in mg/kg

| lab  | method      | value   | mark      | z(targ) | remarks               |
|------|-------------|---------|-----------|---------|-----------------------|
| 52   |             | ----    |           | ----    |                       |
| 150  | INH-ETOH    | 255.4   |           | 0.11    |                       |
| 169  |             | ----    |           | ----    |                       |
| 171  | D5501       | 231.3   |           | -1.25   |                       |
| 311  | INH-529     | 260     |           | 0.37    |                       |
| 323  | INH-155     | 265     |           | 0.66    |                       |
| 329  | INH-155     | 264     |           | 0.60    |                       |
| 333  | INH-2870    | 253     |           | -0.02   |                       |
| 338  | GC          | 235     | C         | -1.04   | First reported 260    |
| 342  |             | ----    |           | ----    |                       |
| 343  | EN15721     | 221.3   |           | -1.82   |                       |
| 357  | INH-012     | 220     |           | -1.90   |                       |
| 395  |             | ----    |           | ----    |                       |
| 444  |             | ----    |           | ----    |                       |
| 463  |             | ----    |           | ----    |                       |
| 468  |             | ----    |           | ----    |                       |
| 494  |             | ----    |           | ----    |                       |
| 496  |             | ----    |           | ----    |                       |
| 541  | INH-0001    | 155.76  | C,G(0.05) | -5.54   | First reported 180.09 |
| 551  |             | ----    |           | ----    |                       |
| 556  | GLC         | 253.164 |           | -0.01   |                       |
| 559  |             | ----    |           | ----    |                       |
| 657  | INH-01/07   | 261.7   |           | 0.47    |                       |
| 840  | INH-0001    | 243     |           | -0.59   |                       |
| 862  | INH-01/07   | 253     |           | -0.02   |                       |
| 867  | INH-0001    | 267.2   |           | 0.78    |                       |
| 902  |             | ----    |           | ----    |                       |
| 913  |             | ----    |           | ----    |                       |
| 922  | INH-0001    | 289.2   |           | 2.03    |                       |
| 1041 | EN15721     | 248.7   |           | -0.27   |                       |
| 1059 |             | ----    |           | ----    |                       |
| 1066 |             | ----    |           | ----    |                       |
| 1079 |             | ----    |           | ----    |                       |
| 1154 |             | ----    |           | ----    |                       |
| 1161 |             | ----    |           | ----    |                       |
| 1201 |             | 240     |           | -0.76   |                       |
| 1263 | D5501       | 246.1   |           | -0.42   |                       |
| 1264 |             | ----    |           | ----    |                       |
| 1521 | EN15721     | 270     |           | 0.94    |                       |
| 1523 |             | ----    |           | ----    |                       |
| 1528 |             | ----    |           | ----    |                       |
| 1605 | in house    | 340.1   | G(0.05)   | 4.92    |                       |
| 1726 | in house    | 265     |           | 0.66    |                       |
| 1727 | GC-FID      | 269     |           | 0.88    |                       |
| 1835 | in house    | 270     |           | 0.94    |                       |
| 1919 |             | 204     |           | -2.80   |                       |
| 1933 |             | 297     |           | 2.47    |                       |
|      | normality   | OK      |           |         |                       |
|      | n           | 24      |           |         |                       |
|      | outliers    | 2       |           |         |                       |
|      | mean (n)    | 253.4   |           |         |                       |
|      | st.dev. (n) | 21.31   |           |         |                       |
|      | R(calc.)    | 59.7    |           |         |                       |
|      | R(Horwitz)  | 49.4    |           |         |                       |



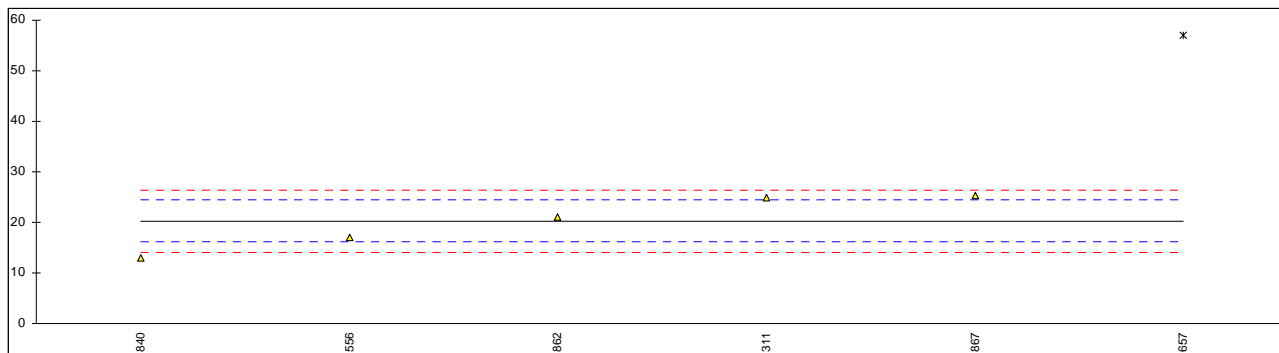
Determination of Isopropanol on sample #0993; results in mg/kg

| lab  | method      | value  | mark    | z(targ)      | remarks           |
|------|-------------|--------|---------|--------------|-------------------|
| 52   |             | ----   |         | ----         |                   |
| 150  | INH-ETOH    | 12.1   |         | 0.03         |                   |
| 169  |             | ----   |         | ----         |                   |
| 171  | D5501       | 13.7   |         | 1.23         |                   |
| 311  | INH-529     | 14     |         | 1.46         |                   |
| 323  | INH-155     | 14     |         | 1.46         |                   |
| 329  | INH-155     | 13     |         | 0.71         |                   |
| 333  |             | ----   |         | ----         |                   |
| 338  | GC          | 15     | C       | 2.21         | First reported 9  |
| 342  |             | ----   |         | ----         |                   |
| 343  | INH-001     | 12.0   |         | -0.05        |                   |
| 357  | INH-012     | 15     |         | 2.21         |                   |
| 395  |             | ----   |         | ----         |                   |
| 444  |             | ----   |         | ----         |                   |
| 463  |             | ----   |         | ----         |                   |
| 468  |             | ----   |         | ----         |                   |
| 494  |             | ----   |         | ----         |                   |
| 496  |             | ----   |         | ----         |                   |
| 541  |             | ----   |         | ----         |                   |
| 551  |             | ----   |         | ----         |                   |
| 556  | GLC         | 12.658 |         | 0.45         |                   |
| 559  |             | ----   |         | ----         |                   |
| 657  | INH-01/07   | 10.9   |         | -0.88        |                   |
| 840  | INH-0001    | 11     |         | -0.80        |                   |
| 862  | INH-01/07   | 10     |         | -1.55        |                   |
| 867  | INH-0001    | 10.6   |         | -1.10        |                   |
| 902  | INH-01/07   | <10    |         | <-1.55       |                   |
| 913  |             | ----   |         | ----         |                   |
| 922  | INH-0001    | 20.4   | G(0.01) | 6.28         |                   |
| 1041 | EN15721     | <10    |         | <-1.55       |                   |
| 1059 |             | ----   |         | ----         |                   |
| 1066 |             | ----   |         | ----         |                   |
| 1079 |             | ----   |         | ----         |                   |
| 1154 |             | ----   |         | ----         |                   |
| 1161 |             | ----   |         | ----         |                   |
| 1201 |             | <10    |         | <-1.55       |                   |
| 1263 | D5501       | 12.3   |         | 0.18         |                   |
| 1264 | D5501       | 10     |         | -1.55        |                   |
| 1521 |             | ----   |         | ----         |                   |
| 1523 |             | ----   |         | ----         |                   |
| 1528 |             | ----   |         | ----         |                   |
| 1605 |             | ----   |         | ----         |                   |
| 1726 | in house    | 8      | C       | -3.06        | First reported 18 |
| 1727 | GC-FID      | 13     |         | 0.71         |                   |
| 1835 | in house    | 13     |         | 0.71         |                   |
| 1919 |             | 9      |         | -2.31        |                   |
| 1933 |             | 12     |         | -0.05        |                   |
|      | normality   | OK     |         |              |                   |
|      | n           | 20     |         |              |                   |
|      | outliers    | 1      |         |              |                   |
|      | mean (n)    | 12.06  |         |              |                   |
|      | st.dev. (n) | 1.913  |         |              |                   |
|      | R(calc.)    | 5.36   |         |              |                   |
|      | R(Horwitz)  | 3.71   |         |              |                   |
|      |             |        |         | <u>Spike</u> | Recovery = <110%  |



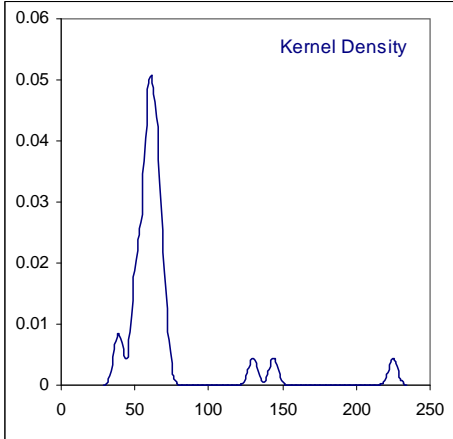
Determination of MEG on sample #0993; results in mg/kg

| lab         | method    | value | mark    | z(targ) | remarks |
|-------------|-----------|-------|---------|---------|---------|
| 52          |           | ----  |         | ----    |         |
| 150         |           | ----  |         | ----    |         |
| 169         |           | ----  |         | ----    |         |
| 171         |           | ----  |         | ----    |         |
| 311         | INH-270   | 25    |         | 2.30    |         |
| 323         |           | ----  |         | ----    |         |
| 329         |           | ----  |         | ----    |         |
| 333         |           | ----  |         | ----    |         |
| 338         |           | ----  |         | ----    |         |
| 342         |           | ----  |         | ----    |         |
| 343         |           | ----  |         | ----    |         |
| 357         |           | ----  |         | ----    |         |
| 395         |           | ----  |         | ----    |         |
| 444         |           | ----  |         | ----    |         |
| 463         |           | ----  |         | ----    |         |
| 468         |           | ----  |         | ----    |         |
| 494         |           | ----  |         | ----    |         |
| 496         |           | ----  |         | ----    |         |
| 541         |           | ----  |         | ----    |         |
| 551         |           | ----  |         | ----    |         |
| 556         | GLC       | 17    |         | -1.58   |         |
| 559         |           | ----  |         | ----    |         |
| 657         | INH-01/07 | 57.1  | G(0.05) | 17.87   |         |
| 840         | INH-0001  | 13    |         | -3.52   |         |
| 862         | INH-01/07 | 21    |         | 0.36    |         |
| 867         | INH-0001  | 25.3  |         | 2.45    |         |
| 902         |           | ----  |         | ----    |         |
| 913         |           | ----  |         | ----    |         |
| 922         |           | ----  |         | ----    |         |
| 1041        |           | ----  |         | ----    |         |
| 1059        |           | ----  |         | ----    |         |
| 1066        |           | ----  |         | ----    |         |
| 1079        |           | ----  |         | ----    |         |
| 1154        |           | ----  |         | ----    |         |
| 1161        |           | ----  |         | ----    |         |
| 1201        |           | ----  |         | ----    |         |
| 1263        |           | ----  |         | ----    |         |
| 1264        |           | ----  |         | ----    |         |
| 1521        |           | ----  |         | ----    |         |
| 1523        |           | ----  |         | ----    |         |
| 1528        |           | ----  |         | ----    |         |
| 1605        |           | ----  |         | ----    |         |
| 1726        | in house  | N.D.  |         | ----    |         |
| 1727        |           | ----  |         | ----    |         |
| 1835        |           | ----  |         | ----    |         |
| 1919        |           | ----  |         | ----    |         |
| 1933        |           | ----  |         | ----    |         |
| normality   |           | OK    |         |         |         |
| n           |           | 5     |         |         |         |
| outliers    |           | 1     |         |         |         |
| mean (n)    |           | 20.26 |         |         |         |
| st.dev. (n) |           | 5.286 |         |         |         |
| R(calc.)    |           | 14.80 |         |         |         |
| R(Horwitz)  |           | 5.77  |         |         |         |

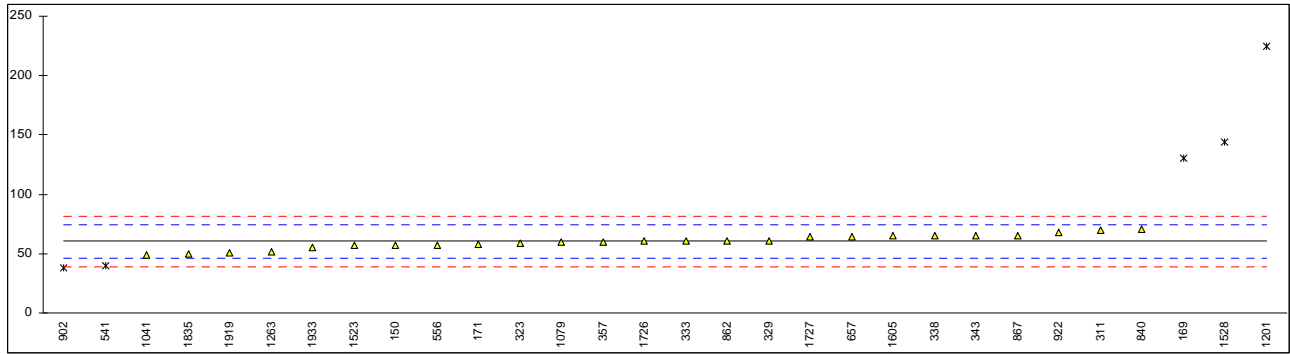


Determination of Methanol on sample #0993; results in mg/kg

| lab  | method        | value  | mark       | z(targ) | remarks             |
|------|---------------|--------|------------|---------|---------------------|
| 52   |               | ----   |            | ----    |                     |
| 150  | INH-ETOH      | 56.7   |            | -0.49   |                     |
| 169  | D5501         | 130    | G(0.01)    | 9.68    |                     |
| 171  | D5501         | 58.4   |            | -0.26   |                     |
| 311  | INH529        | 70     |            | 1.35    |                     |
| 323  | INH-155       | 59     |            | -0.18   |                     |
| 329  | INH-155       | 61     |            | 0.10    |                     |
| 333  | INH-2870      | 61     |            | 0.10    |                     |
| 338  | GC            | 65     | C          | 0.66    | First reported 94   |
| 342  |               | ----   |            | ----    |                     |
| 343  | INH-001       | 65     | C          | 0.66    | First reported 91.6 |
| 357  | INH-012       | 60     |            | -0.04   |                     |
| 395  |               | ----   |            | ----    |                     |
| 444  |               | ----   |            | ----    |                     |
| 463  |               | ----   |            | ----    |                     |
| 468  |               | ----   |            | ----    |                     |
| 494  |               | ----   |            | ----    |                     |
| 496  |               | ----   |            | ----    |                     |
| 541  | INH-0001      | 39.84  | DG(0.05)   | -2.84   |                     |
| 551  |               | ----   |            | ----    |                     |
| 556  | GLC           | 56.962 |            | -0.46   |                     |
| 559  |               | ----   |            | ----    |                     |
| 657  | INH-01/07     | 64.5   |            | 0.59    |                     |
| 840  | INH-0001      | 71     |            | 1.49    |                     |
| 862  | INH-01/07     | 61     |            | 0.10    |                     |
| 867  | INH-0001      | 65.4   |            | 0.71    |                     |
| 902  | INH-01/07     | 38     | C,DG(0.05) | -3.09   | First reported 27   |
| 913  |               | ----   |            | ----    |                     |
| 922  | INH-0001      | 68.1   |            | 1.09    |                     |
| 1041 | EN15721       | 49.3   |            | -1.52   |                     |
| 1059 |               | ----   |            | ----    |                     |
| 1066 |               | ----   |            | ----    |                     |
| 1079 | EN15721       | 60     |            | -0.04   |                     |
| 1154 |               | ----   |            | ----    |                     |
| 1161 |               | ----   |            | ----    |                     |
| 1201 |               | 225    | G(0.01)    | 22.87   |                     |
| 1263 | D5501         | 51.7   |            | -1.19   |                     |
| 1264 |               | ----   |            | ----    |                     |
| 1521 | EN79528       | <100   |            | ----    |                     |
| 1523 | D5501         | 56.7   |            | -0.49   |                     |
| 1528 | EC2870        | 144    | G(0.01)    | 11.62   |                     |
| 1605 | in house      | 64.8   |            | 0.63    |                     |
| 1726 | in house      | 61     |            | 0.10    |                     |
| 1727 | GC-FID        | 64     |            | 0.52    |                     |
| 1835 | in house      | 50     |            | -1.42   |                     |
| 1919 |               | 51     |            | -1.29   |                     |
| 1933 |               | 55     |            | -0.73   |                     |
|      | normality     | OK     |            |         |                     |
|      | n             | 25     |            |         |                     |
|      | outliers      | 5      |            |         |                     |
|      | mean (n)      | 60.26  |            |         |                     |
|      | st.dev. (n)   | 5.956  |            |         |                     |
|      | R(calc.)      | 16.68  |            |         |                     |
|      | R(EN15721:07) | 20.17  |            |         |                     |

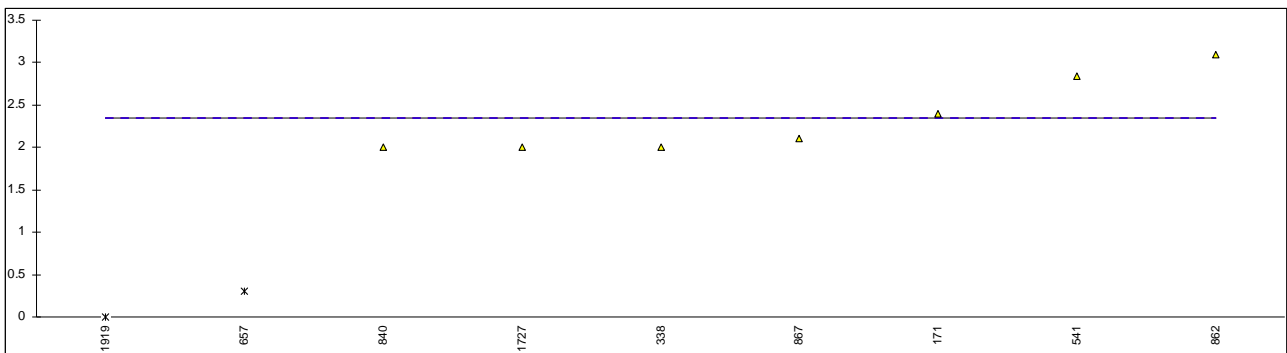
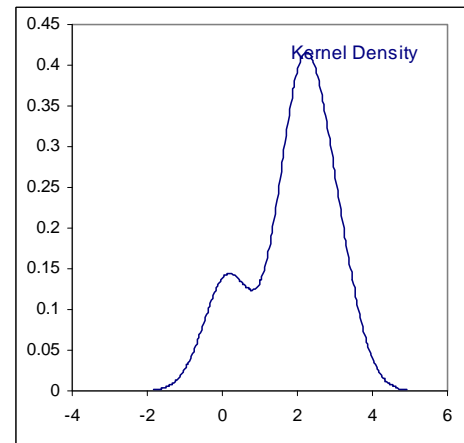


Compare with R(D5501:09) = 90.98



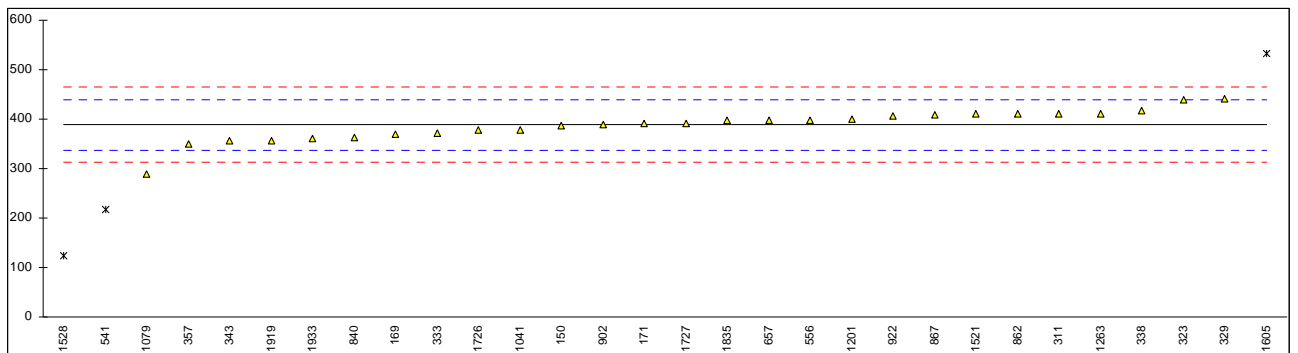
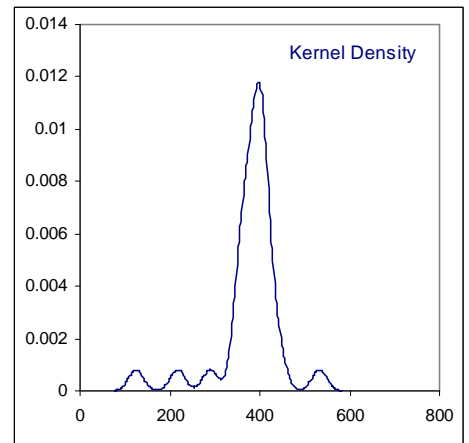
Determination of n-Butanol on sample #0993; results in mg/kg

| lab  | method      | value | mark    | z(targ) | remarks                  |
|------|-------------|-------|---------|---------|--------------------------|
| 52   |             | ----  |         | ----    |                          |
| 150  | INH-ETOH    | <1    |         | ----    |                          |
| 169  |             | ----  |         | ----    |                          |
| 171  | D5501       | 2.4   |         | ----    |                          |
| 311  | INH529      | <5    |         | ----    |                          |
| 323  | INH-155     | <5    |         | ----    |                          |
| 329  | INH-155     | <5    |         | ----    |                          |
| 333  |             | ----  |         | ----    |                          |
| 338  | GC          | 2     | C       | ----    | First reported 2.4       |
| 342  |             | ----  |         | ----    |                          |
| 343  | EN15721     | <10   |         | ----    |                          |
| 357  | INH-012     | <10   |         | ----    |                          |
| 395  |             | ----  |         | ----    |                          |
| 444  |             | ----  |         | ----    |                          |
| 463  |             | ----  |         | ----    |                          |
| 468  |             | ----  |         | ----    |                          |
| 494  |             | ----  |         | ----    |                          |
| 496  |             | ----  |         | ----    |                          |
| 541  | INH-0001    | 2.84  |         | ----    |                          |
| 551  |             | ----  |         | ----    |                          |
| 556  |             | ----  |         | ----    |                          |
| 559  |             | ----  |         | ----    |                          |
| 657  | INH-01/07   | 0.3   | G(0.05) | ----    |                          |
| 840  | INH-0001    | 2     |         | ----    |                          |
| 862  | INH-01/07   | 3.1   |         | ----    |                          |
| 867  | INH-0001    | 2.1   |         | ----    |                          |
| 902  |             | ----  |         | ----    |                          |
| 913  |             | ----  |         | ----    |                          |
| 922  |             | ----  |         | ----    |                          |
| 1041 | EN15721     | <10   |         | ----    |                          |
| 1059 |             | ----  |         | ----    |                          |
| 1066 |             | ----  |         | ----    |                          |
| 1079 | EN15721     | <10   |         | ----    |                          |
| 1154 |             | ----  |         | ----    |                          |
| 1161 |             | ----  |         | ----    |                          |
| 1201 |             | <10   |         | ----    |                          |
| 1263 | D5501       | <5    |         | ----    |                          |
| 1264 |             | ----  |         | ----    |                          |
| 1521 |             | ----  |         | ----    |                          |
| 1523 |             | ----  |         | ----    |                          |
| 1528 |             | ----  |         | ----    |                          |
| 1605 |             | ----  |         | ----    |                          |
| 1726 | in house    | N.D.  |         | ----    |                          |
| 1727 | GC-FID      | 2     |         | ----    |                          |
| 1835 | in house    | N.D.  |         | ----    |                          |
| 1919 |             | 0     | ex      | ----    | Zero is not a real value |
| 1933 |             | <7    |         | ----    |                          |
|      | normality   | OK    |         |         |                          |
|      | n           | 7     |         |         |                          |
|      | outliers    | 1     |         |         |                          |
|      | mean (n)    | 2.35  |         |         |                          |
|      | st.dev. (n) | 0.454 |         |         |                          |
|      | R(calc.)    | 1.27  |         |         |                          |
|      | R(lit)      | n.a.  |         |         |                          |



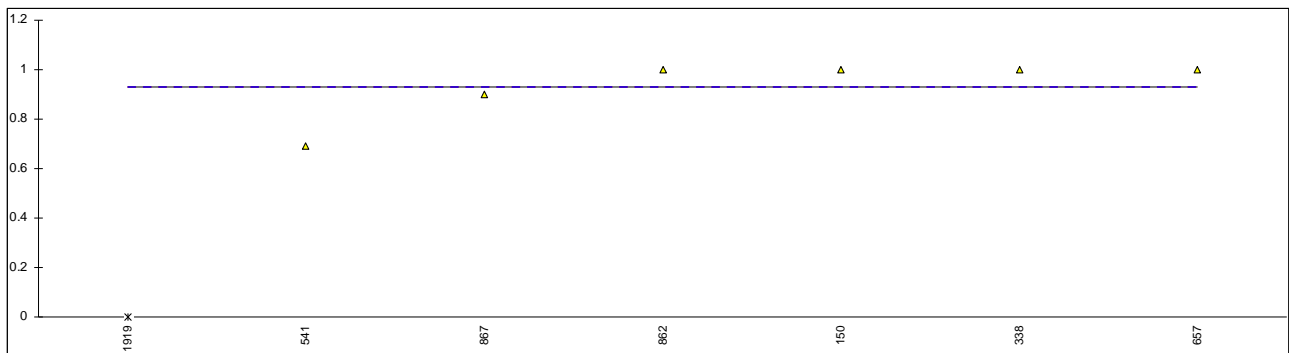
Determination of n-Propanol on sample #0993; results in mg/kg

| lab  | method      | value   | mark      | z(targ) | remarks               |
|------|-------------|---------|-----------|---------|-----------------------|
| 52   |             | ----    |           | ----    |                       |
| 150  | INH-ETOH    | 388.0   |           | -0.01   |                       |
| 169  | D5501       | 370     |           | -0.72   |                       |
| 171  | D5501       | 390.5   |           | 0.09    |                       |
| 311  | INH529      | 410     |           | 0.86    |                       |
| 323  | INH-155     | 440     |           | 2.04    |                       |
| 329  | INH-155     | 442     |           | 2.12    |                       |
| 333  | INH-2870    | 372     |           | -0.65   |                       |
| 338  | GC          | 417     | C         | 1.13    | First reported 492    |
| 342  |             | ----    |           | ----    |                       |
| 343  | EN15721     | 355.7   |           | -1.29   |                       |
| 357  | INH-012     | 350     |           | -1.51   |                       |
| 395  |             | ----    |           | ----    |                       |
| 444  |             | ----    |           | ----    |                       |
| 463  |             | ----    |           | ----    |                       |
| 468  |             | ----    |           | ----    |                       |
| 494  |             | ----    |           | ----    |                       |
| 496  |             | ----    |           | ----    |                       |
| 541  | INH-0001    | 217.50  | C,G(0.01) | -6.74   | First reported 260.17 |
| 551  |             | ----    |           | ----    |                       |
| 556  | GLC         | 398.734 |           | 0.41    |                       |
| 559  |             | ----    |           | ----    |                       |
| 657  | INH-01/07   | 398.0   |           | 0.38    |                       |
| 840  | INH-0001    | 362     |           | -1.04   |                       |
| 862  | INH-01/07   | 410     |           | 0.86    |                       |
| 867  | INH-0001    | 407.7   |           | 0.76    |                       |
| 902  | INH-01/07   | 390     |           | 0.07    |                       |
| 913  |             | ----    |           | ----    |                       |
| 922  | INH-0001    | 407.6   |           | 0.76    |                       |
| 1041 | EN15721     | 379.2   |           | -0.36   |                       |
| 1059 |             | ----    |           | ----    |                       |
| 1066 |             | ----    |           | ----    |                       |
| 1079 | EN15721     | 290     |           | -3.88   |                       |
| 1154 |             | ----    |           | ----    |                       |
| 1161 |             | ----    |           | ----    |                       |
| 1201 |             | 400     |           | 0.46    |                       |
| 1263 | D5501       | 411.8   |           | 0.93    |                       |
| 1264 |             | ----    |           | ----    |                       |
| 1521 | EN79521     | 410     |           | 0.86    |                       |
| 1523 |             | ----    |           | ----    |                       |
| 1528 | EC2870      | 125     | G(0.01)   | -10.40  |                       |
| 1605 | in house    | 533.0   | G(0.01)   | 5.71    |                       |
| 1726 | in house    | 379     |           | -0.37   |                       |
| 1727 | GC-FID      | 392     |           | 0.14    |                       |
| 1835 | in house    | 397     |           | 0.34    |                       |
| 1919 |             | 357     |           | -1.24   |                       |
| 1933 |             | 360     |           | -1.12   |                       |
|      | normality   | OK      |           |         |                       |
|      | n           | 27      |           |         |                       |
|      | outliers    | 3       |           |         |                       |
|      | mean (n)    | 388.3   |           |         |                       |
|      | st.dev. (n) | 30.99   |           |         |                       |
|      | R(calc.)    | 86.8    |           |         |                       |
|      | R(Horwitz)  | 70.9    |           |         |                       |



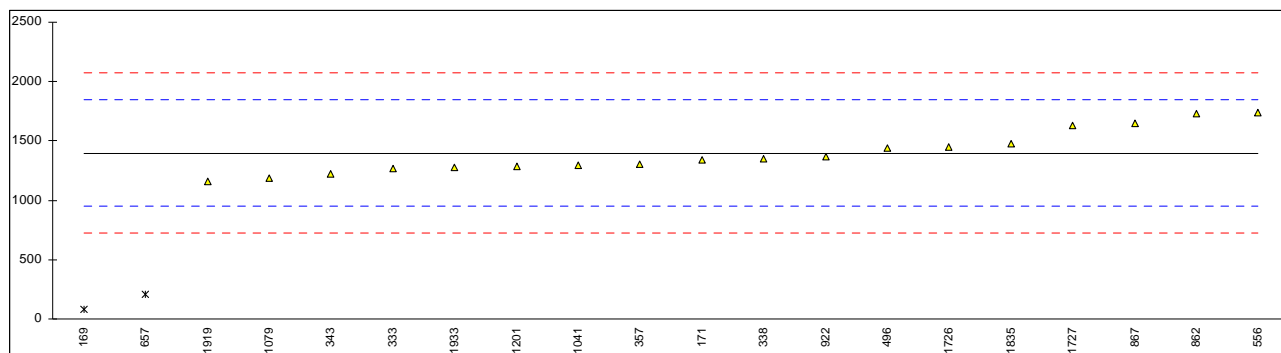
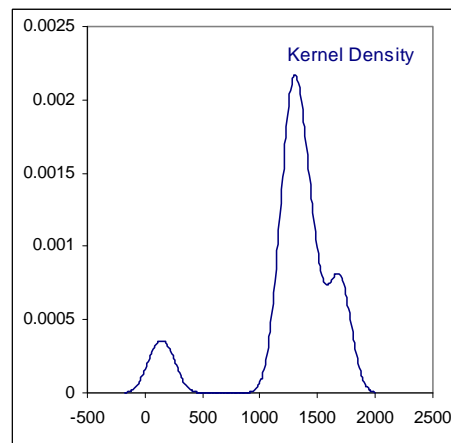
Determination of sec-Butanol on sample #0993; results in mg/kg

| lab  | method      | value  | mark | z(targ) | remarks                  |
|------|-------------|--------|------|---------|--------------------------|
| 52   |             | ----   |      | ----    |                          |
| 150  | INH-ETOH    | 1.0    |      | ----    |                          |
| 169  |             | ----   |      | ----    |                          |
| 171  | D5501       | <1     |      | ----    |                          |
| 311  | INH529      | <5     |      | ----    |                          |
| 323  | INH-155     | <5     |      | ----    |                          |
| 329  | INH-155     | <5     |      | ----    |                          |
| 333  |             | ----   |      | ----    |                          |
| 338  | GC          | 1      | C    | ----    | First reported 1.6       |
| 342  |             | ----   |      | ----    |                          |
| 343  | EN15721     | <10    |      | ----    |                          |
| 357  | INH-012     | <10    |      | ----    |                          |
| 395  |             | ----   |      | ----    |                          |
| 444  |             | ----   |      | ----    |                          |
| 463  |             | ----   |      | ----    |                          |
| 468  |             | ----   |      | ----    |                          |
| 494  |             | ----   |      | ----    |                          |
| 496  |             | ----   |      | ----    |                          |
| 541  | INH-0001    | 0.69   |      | ----    |                          |
| 551  |             | ----   |      | ----    |                          |
| 556  | GLC         | <6.33  |      | ----    |                          |
| 559  |             | ----   |      | ----    |                          |
| 657  | INH-01/07   | 1.0    |      | ----    |                          |
| 840  | INH-0001    | <2     |      | ----    |                          |
| 862  | INH-01/07   | 1      |      | ----    |                          |
| 867  | INH-0001    | 0.9    |      | ----    |                          |
| 902  |             | ----   |      | ----    |                          |
| 913  |             | ----   |      | ----    |                          |
| 922  |             | ----   |      | ----    |                          |
| 1041 | EN15721     | <10    |      | ----    |                          |
| 1059 |             | ----   |      | ----    |                          |
| 1066 |             | ----   |      | ----    |                          |
| 1079 | EN15721     | <10    |      | ----    |                          |
| 1154 |             | ----   |      | ----    |                          |
| 1161 |             | ----   |      | ----    |                          |
| 1201 |             | <10    |      | ----    |                          |
| 1263 | D5501       | <5     |      | ----    |                          |
| 1264 | D5501       | N.D.   |      | ----    |                          |
| 1521 | EN79521     | <100   |      | ----    |                          |
| 1523 |             | ----   |      | ----    |                          |
| 1528 |             | ----   |      | ----    |                          |
| 1605 |             | ----   |      | ----    |                          |
| 1726 | in house    | N.D.   |      | ----    |                          |
| 1727 |             | ----   |      | ----    |                          |
| 1835 | in house    | N.D.   |      | ----    |                          |
| 1919 |             | 0      | ex   | ----    | Zero is not a real value |
| 1933 |             | <7     |      | ----    |                          |
|      | normality   | not OK |      |         |                          |
|      | n           | 6      |      |         |                          |
|      | outliers    | 0      |      |         |                          |
|      | mean (n)    | 0.93   |      |         |                          |
|      | st.dev. (n) | 0.125  |      |         |                          |
|      | R(calc.)    | 0.35   |      |         |                          |
|      | R(lit)      | n.a.   |      |         |                          |



Determination of Total Impurities on sample #0993; results in mg/kg

| lab  | method      | value   | mark    | z(targ) | remarks                                     |
|------|-------------|---------|---------|---------|---|
| 52   |             | ----    |         | ----    |   |
| 150  |             | ----    |         | ----    |   |
| 169  | D5501       | 82      | G(0.05) | -5.83   |   |
| 171  | D5501       | 1337.9  |         | -0.27   |   |
| 311  |             | ----    |         | ----    |   |
| 323  |             | ----    |         | ----    |   |
| 329  |             | ----    |         | ----    |   |
| 333  | INH-2870    | 1270    |         | -0.57   |   |
| 338  | GC          | 1353    | C       | -0.20   | First reported 1592                         |
| 342  |             | ----    |         | ----    |   |
| 343  | INH-0001    | 1225    | C       | -0.77   | First reported in deviating unit 0.126 %M/M |
| 357  | INH-012     | 1300    |         | -0.44   |   |
| 395  |             | ----    |         | ----    |   |
| 444  |             | ----    |         | ----    |   |
| 463  |             | ----    |         | ----    |   |
| 468  |             | ----    |         | ----    |   |
| 494  |             | ----    |         | ----    |   |
| 496  | EN15721     | 1439    |         | 0.18    |   |
| 541  |             | ----    |         | ----    |   |
| 551  |             | ----    |         | ----    |   |
| 556  | GLC         | 1738.67 |         | 1.51    |   |
| 559  |             | ----    |         | ----    |   |
| 657  | INH-01/07   | 206.5   | G(0.01) | -5.28   |   |
| 840  |             | ----    |         | ----    |   |
| 862  | INH-01/07   | 1730    |         | 1.47    |   |
| 867  | INH-0001    | 1646    |         | 1.10    |   |
| 902  |             | ----    |         | ----    |   |
| 913  |             | ----    |         | ----    |   |
| 922  | Calculated  | 1369.9  |         | -0.13   |   |
| 1041 | EN15721     | 1294.1  | C       | -0.46   | First reported 1936.1                       |
| 1059 |             | ----    |         | ----    |   |
| 1066 |             | ----    |         | ----    |   |
| 1079 | EN15721     | 1190    |         | -0.92   |   |
| 1154 |             | ----    |         | ----    |   |
| 1161 |             | ----    |         | ----    |   |
| 1201 |             | 1285    |         | -0.50   |   |
| 1263 |             | ----    |         | ----    |   |
| 1264 |             | ----    |         | ----    |   |
| 1521 |             | ----    |         | ----    |   |
| 1523 |             | ----    |         | ----    |   |
| 1528 |             | ----    |         | ----    |   |
| 1605 |             | ----    |         | ----    |   |
| 1726 | in house    | 1450    |         | 0.23    |   |
| 1727 | GC-FID      | 1630    |         | 1.03    |   |
| 1835 | in house    | 1477    |         | 0.35    |   |
| 1919 |             | 1161    |         | -1.05   |   |
| 1933 |             | 1279    |         | -0.53   |   |
|      | normality   | OK      |         |         |   |
|      | n           | 18      |         |         |   |
|      | outliers    | 2       |         |         |   |
|      | mean (n)    | 1398.6  |         |         |   |
|      | st.dev. (n) | 180.15  |         |         |   |
|      | R(calc.)    | 504.4   |         |         |   |
|      | R(Horwitz)  | 631.9   |         |         |   |



## Determination of other components on sample #0993; results in mg/kg

| lab  | method      | Croton-<br>aldehyde | mark | DEG  | mark | Dioxane    | mark    | n-Amyl-<br>alcohol | mark      |
|------|-------------|---------------------|------|------|------|------------|---------|--------------------|-----------|
| 52   |             | ----                |      | ---- |      | ----       |         | ----               |           |
| 150  | INH-ETOH    | ----                |      | ---- |      | ----       |         | ----               |           |
| 169  |             | ----                |      | ---- |      | ----       |         | ----               |           |
| 171  | D5501       | <1                  |      | ---- |      | <b>8.2</b> | False + | <1                 |           |
| 311  | INH-529     | <5                  |      | ---- |      | ----       |         | <5                 |           |
| 323  | INH-155     | <5                  |      | ---- |      | <5         |         | <5                 |           |
| 329  | INH-155     | <5                  |      | ---- |      | <5         |         | <5                 |           |
| 333  | INH-2870    | ----                |      | ---- |      | ----       |         | ----               |           |
| 338  | GC          | ----                |      | ---- |      | ----       |         | 2                  | C; fr 2.4 |
| 342  |             | ----                |      | ---- |      | ----       |         | ----               |           |
| 343  |             | <5                  |      | ---- |      | ----       |         | <5                 |           |
| 357  | INH-012     | <10                 |      | ---- |      | ----       |         | <10                |           |
| 395  |             | ----                |      | ---- |      | ----       |         | ----               |           |
| 444  |             | ----                |      | ---- |      | ----       |         | ----               |           |
| 463  |             | ----                |      | ---- |      | ----       |         | ----               |           |
| 468  |             | ----                |      | ---- |      | ----       |         | ----               |           |
| 494  |             | ----                |      | ---- |      | ----       |         | ----               |           |
| 496  |             | ----                |      | ---- |      | ----       |         | ----               |           |
| 541  |             | ----                |      | ---- |      | ----       |         | ----               |           |
| 551  |             | ----                |      | ---- |      | ----       |         | ----               |           |
| 556  | GLC         | <0.5                |      | <1.0 |      | ----       |         | ----               |           |
| 559  |             | ----                |      | ---- |      | ----       |         | ----               |           |
| 657  | INH-01/07   | 2.1                 |      | N.D. |      | N.D.       |         | 2.2                |           |
| 840  | INH-0001    | <2                  |      | <2   |      | ----       |         | <2                 |           |
| 862  | INH-01/07   | <5                  |      | <5   |      | <5         |         | 2.7                |           |
| 867  | INH-0001    | 1.6                 |      | <5   |      | ----       |         | 1.3                |           |
| 902  |             | ----                |      | ---- |      | ----       |         | ----               |           |
| 913  |             | ----                |      | ---- |      | ----       |         | ----               |           |
| 922  | INH-0001    | ----                |      | ---- |      | ----       |         | <2                 |           |
| 1041 | EN15721     | ----                |      | ---- |      | ----       |         | <10                |           |
| 1059 |             | ----                |      | ---- |      | ----       |         | ----               |           |
| 1066 |             | ----                |      | ---- |      | ----       |         | ----               |           |
| 1079 | EN15721     | ----                |      | ---- |      | ----       |         | ----               |           |
| 1154 |             | ----                |      | ---- |      | ----       |         | ----               |           |
| 1161 |             | ----                |      | ---- |      | ----       |         | ----               |           |
| 1201 |             | <10                 |      | ---- |      | ----       |         | <10                |           |
| 1263 | D5501       | ----                |      | ---- |      | ----       |         | <5                 |           |
| 1264 |             | N.D.                |      | ---- |      | ----       |         | ----               |           |
| 1521 |             | ----                |      | ---- |      | ----       |         | <100               |           |
| 1523 | D5501       | ----                |      | ---- |      | ----       |         | ----               |           |
| 1528 | EC2870      | ----                |      | ---- |      | ----       |         | <10                |           |
| 1605 | in house    | ----                |      | ---- |      | ----       |         | ----               |           |
| 1726 | in house    | N.D.                |      | N.D. |      | N.D.       |         | N.D.               |           |
| 1727 | GC-FID      | ----                |      | ---- |      | ----       |         | ----               |           |
| 1835 | in house    | ----                |      | ---- |      | ----       |         | ----               |           |
| 1919 |             | ----                |      | ---- |      | ----       |         | 0                  |           |
| 1933 |             | ----                |      | ---- |      | ----       |         | <7                 |           |
|      | normality   | n.a.                |      | n.a. |      | n.a.       |         | n.a.               |           |
|      | n           | 2                   |      | 0    |      | 0          |         | 4                  |           |
|      | outliers    | 0                   |      | 0    |      | 0          |         | 0                  |           |
|      | mean (n)    | n.a.                |      | n.a. |      | n.a.       |         | n.a.               |           |
|      | st.dev. (n) | n.a.                |      | n.a. |      | n.a.       |         | n.a.               |           |
|      | R(calc.)    | n.a.                |      | n.a. |      | n.a.       |         | n.a.               |           |
|      | R(Horwitz)  | n.a.                |      | n.a. |      | n.a.       |         | n.a.               |           |

## Determination of other components on sample #0993; results in mg/kg

| lab  | method      | sec-<br>Amyl-<br>alcohol | mark    | tert-<br>Amyl-<br>alcohol | mark | tert-<br>butanol | mark       |
|------|-------------|--------------------------|---------|---------------------------|------|------------------|------------|
| 52   |             | ----                     |         | ----                      |      | ----             |            |
| 150  | INH-ETOH    | ----                     |         | ----                      |      | ----             |            |
| 169  |             | ----                     |         | ----                      |      | ----             |            |
| 171  | D5501       | <1                       |         | <1                        |      | 3.0              |            |
| 311  | INH-529     | <5                       |         | <5                        |      | <5               |            |
| 323  | INH-155     | <5                       |         | <5                        |      | <5               |            |
| 329  | INH-155     | <5                       |         | <5                        |      | <5               |            |
| 333  | INH-2870    | ----                     |         | ----                      |      | ----             |            |
| 338  | GC          | <b>78</b>                | False + | ----                      |      | ----             |            |
| 342  |             | ----                     |         | ----                      |      | ----             |            |
| 343  |             | <5                       |         | <5                        |      | 11               | C; fr 21.1 |
| 357  | INH-012     | <10                      |         | <10                       |      | <10              |            |
| 395  |             | ----                     |         | ----                      |      | ----             |            |
| 444  |             | ----                     |         | ----                      |      | ----             |            |
| 463  |             | ----                     |         | ----                      |      | ----             |            |
| 468  |             | ----                     |         | ----                      |      | ----             |            |
| 494  |             | ----                     |         | ----                      |      | ----             |            |
| 496  |             | ----                     |         | ----                      |      | ----             |            |
| 541  |             | ----                     |         | 0.37                      |      | ----             |            |
| 551  |             | ----                     |         | ----                      |      | ----             |            |
| 556  | GLC         | ----                     |         | ----                      |      | ----             |            |
| 559  |             | ----                     |         | ----                      |      | ----             |            |
| 657  | INH-01/07   | 0.2                      |         | N.D.                      |      | N.D.             |            |
| 840  | INH-0001    | <2                       |         | 2                         |      | <2               |            |
| 862  | INH-01/07   | 2                        |         | 1                         |      | <5               |            |
| 867  | INH-0001    | 1.9                      |         | <5                        |      | 2.4              |            |
| 902  |             | ----                     |         | ----                      |      | ----             |            |
| 913  |             | ----                     |         | ----                      |      | ----             |            |
| 922  | INH-0001    | ----                     |         | ----                      |      | ----             |            |
| 1041 | EN15721     | <10                      |         | <10                       |      | <10              | C; fr 30.3 |
| 1059 |             | ----                     |         | ----                      |      | ----             |            |
| 1066 |             | ----                     |         | ----                      |      | ----             |            |
| 1079 | EN15721     | ----                     |         | ----                      |      | ----             |            |
| 1154 |             | ----                     |         | ----                      |      | ----             |            |
| 1161 |             | ----                     |         | ----                      |      | ----             |            |
| 1201 |             | <10                      |         | <10                       |      | <10              |            |
| 1263 | D5501       | <5                       |         | ----                      |      | ----             |            |
| 1264 |             | ----                     |         | ----                      |      | N.D.             |            |
| 1521 |             | ----                     |         | <100                      |      | ----             |            |
| 1523 | D5501       | ----                     |         | ----                      |      | ----             |            |
| 1528 | EC2870      | <10                      |         | ----                      |      | ----             |            |
| 1605 | in house    | ----                     |         | ----                      |      | ----             |            |
| 1726 | in house    | ----                     |         | <5                        |      | N.D.             |            |
| 1727 | GC-FID      | ----                     |         | ----                      |      | ----             |            |
| 1835 | in house    | ----                     |         | ----                      |      | ----             |            |
| 1919 |             | ----                     |         | 0                         |      | ----             |            |
| 1933 |             | ----                     |         | <7                        |      | ----             |            |
|      | normality   | n.a.                     |         | n.a.                      |      | n.a.             |            |
|      | n           | 2                        |         | 3                         |      | 3                |            |
|      | outliers    | 0                        |         | 0                         |      | 0                |            |
|      | mean (n)    | n.a.                     |         | n.a.                      |      | n.a.             |            |
|      | st.dev. (n) | n.a.                     |         | n.a.                      |      | n.a.             |            |
|      | R(calc.)    | n.a.                     |         | n.a.                      |      | n.a.             |            |
|      | R(Horwitz)  | n.a.                     |         | n.a.                      |      | n.a.             |            |

**APPENDIX 2****Participants:**

1 laboratory in ARGENTINA  
1 laboratory in AUSTRIA  
4 laboratories in BELGIUM  
3 laboratories in BRAZIL  
1 laboratory in CANADA  
1 laboratory in FINLAND  
4 laboratories in FRANCE  
3 laboratories in GERMANY  
1 laboratory in INDIA  
1 laboratory in ITALY  
2 laboratories in P.R. of CHINA  
1 laboratory in PAKISTAN  
1 laboratory in POLAND  
1 laboratory in ROMANIA  
1 laboratory in SINGAPORE  
1 laboratory in SLOVENIA  
5 laboratories in SPAIN  
3 laboratories in SWEDEN  
4 laboratories in THE NETHERLANDS  
2 laboratories in TURKEY  
1 laboratory in U.A.E.  
3 laboratories in U.S.A.  
1 laboratory in UNITED KINGDOM  
1 laboratory in VIETNAM

## APPENDIX 3

### 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                     |
| E        | = error in calculations  |
| ex       | = excluded from calculations                                   |
| n.a.     | = not applicable   |
| U        | = unit error   |
| SDS      | = Safety Data Sheet  |

### Literature:

- 1 i.i.s. Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, January 2010
- 2 W. Horwitz and R. Albert, J. AOAC Int., Vol. 79, 3, p. 589, (1996)
- 3 ASTM E178-02
- 4 ASTM E1301-03
- 5 ISO 5725-86
- 6 ISO 5725, parts 1-6, 1994
- 7 ISO 13528
- 8 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
- 9 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
- 10 IP 367/84
- 11 DIN 38402 T41/42
- 12 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
- 13 J.N. Miller, Analyst, 118, 455, (1993)
- 14 Analytical Methods Committee Technical brief, No 4. January 2001
- 15 The Royal Society of Chemistry 2002, Analyst, 2002, 127, page 1359-1364, P.J. Lowthian and M. Thompson. (see <http://www.rsc.org/suppdata/an/b2/b205600n/>)