**Institute for
Interlaboratory Studies**

# Results of Proficiency Test
# Benzene
# March 2023

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

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**Report:**   **iis23C01**

**April 2023**

**CONTENTS**

Appendices:

**1      INTRODUCTION**

Since 1999 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Benzene based on the latest version of ASTM D2359 every year. During the annual proficiency testing program 2022/2023 it was decided to continue the round robin for the analysis of Benzene.

In this interlaboratory study 49 laboratories in 22 countries registered for participation, see appendix 2 for the number of participants per country. In this report the results of the Benzene proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

**2      SET UP**

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.
It was decided to send two different samples of Benzene, one glass bottle of 1 L labelled #23020 for regular analyzes and one glass bottle of 30 mL labelled #23023 for determination of Total and Organic Chlorides only.
The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for the statistical evaluation.

**2.1    ACCREDITATION**

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC17043:2010 (R007), since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

**2.2    PROTOCOL**

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

**2.3    CONFIDENTIALITY STATEMENT**

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

## 2.4  SAMPLES

For the preparation of the sample for the regular analyzes in Benzene a batch of approximately 80 liters of Benzene was obtained from a local supplier. The Benzene was spiked with Toluene and Thiophene. After homogenization 80 amber glass bottles of 1 L were filled and labelled #23020.
The homogeneity of the subsamples was checked by the determination of Density at 20 ˚C in accordance with ASTM D4052 on 8 stratified randomly selected subsamples.

|  | Density at 20 °C in kg/L |
| --- | --- |
| sample #23020-1 | 0.87906 |
| sample #23020-2 | 0.87904 |
| sample #23020-3 | 0.87905 |
| sample #23020-4 | 0.87906 |
| sample #23020-5 | 0.87904 |
| sample #23020-6 | 0.87904 |
| sample #23020-7 | 0.87906 |
| sample #23020-8 | 0.87904 |

Table 1: homogeneity test results of subsamples #23020

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

|  | Density at 20 °C in kg/L |
| --- | --- |
| r (observed) | 0.00003 |
| reference test method | ISO12185:96 |
| 0.3 x R (reference test method) | 0.00015 |

Table 2: evaluation of the repeatability of subsamples #23020

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

For the preparation of the sample for the determination of Chlorides in Benzene a batch of approximately 5 liters of Benzene was obtained from a local supplier. The Benzene was spiked with o-Chlorotoluene. After homogenization 80 amber glass bottles of 30 mL were filled and labelled #23023.
The homogeneity of the subsamples was checked by the determination of Density at 20 ˚C in accordance with ASTM D4052 on 8 stratified randomly selected subsamples.

|                  | Density at 20 °C in kg/L |
|------------------|--------------------------|
| sample #23023-1  | 0.87896                  |
| sample #23023-2  | 0.87894                  |
| sample #23023-3  | 0.87895                  |
| sample #23023-4  | 0.87897                  |
| sample #23023-5  | 0.87894                  |
| sample #23023-6  | 0.87898                  |
| sample #23023-7  | 0.87895                  |
| sample #23023-8  | 0.87896                  |

Table 3: homogeneity test results of subsamples #23023

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

|                                     | Density at 20 °C in kg/L |
|-------------------------------------|--------------------------|
| r (observed)                        | 0.00004                  |
| reference test method               | ISO12185:96              |
| 0.3 x R (reference test method)     | 0.00015                  |

Table 4: evaluation of the repeatability of subsamples #23023

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

To each of the participating laboratories one 1 L bottle Benzene labelled #23020 and one 30 mL bottle Benzene labelled #23023 was sent on February 1, 2023. An SDS was added to the sample package.

## 2.5   STABILITY OF THE SAMPLES

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

## 2.6   ANALYZES

The participants were requested to determine on sample #23020: Acid Wash Color, Acidity, Appearance, Bromine Index, Color Pt/Co, Density at 20 °C, Distillation (IBP, 50% recovered, Dry Point, Distillation Range), Total Nitrogen, Purity by GC, Methylcyclohexane, Toluene, Nonaromatics, 1,4-Dioxane, Total Impurities, Solidification Point (anhydrous basis), Sulfur, Thiophene and Water.
On sample #23023 it was requested to determine Total Chlorides and Organic Chlorides.

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

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

## 3     RESULTS

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

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the result tables in appendix 1. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

## 3.1     STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5).
For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<…' or '>…' were not used in the statistical evaluation.

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

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

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

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

## 3.2  GRAPHICS

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

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

## 3.3  Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements (derived from e.g. ISO or ASTM test methods), the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

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

The z-scores were calculated according to:

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

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

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

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

## 4 EVALUATION

Some problems were encountered with the dispatch of the samples. Therefore, the reporting time on the data entry portal was extended with another week. Four participants reported test results after the extended reporting date and ten other participants did not report any test results. Not all participants were able to report all tests requested.
In total 39 participants reported 491 numerical test results. Observed were 15 outlying test results, which is 3.1%. In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

Not all data sets proved to have a normal Gaussian distribution. These are referred to as "not OK" or "suspect". The statistical evaluation of these data sets should be used with due care, see also paragraph 3.1.

### 4.1 EVALUATION PER SAMPLE AND PER TEST

In this section the reported test results are discussed per sample and per test. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables together with the original data in appendix 1. The abbreviations, used in these tables, are explained in appendix 3.

Unfortunately, a suitable reference test method, providing the precision data, is not available for all determinations. For these tests the calculated reproducibility was compared against the estimated reproducibility calculated with the Horwitz equation.

In the iis PT reports ASTM test methods are referred to with a number (e.g. D1209) and an added designation for the year that the test method was adopted or revised (e.g. D1209:05). When a method has been reapproved an "R" will be added and the year of approval (e.g. D1209:05R19).

**sample #23020**

Acid Wash Color: This determination was not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is in agreement with the requirements of ASTM D848:23.

Acidity: This determination was not problematic. Almost all laboratories reported "No free acid" or "Pass".

Appearance: This determination was not problematic. All laboratories agreed on the appearance of the sample, which was Clear and Bright (Pass).

Bromine Index: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D5776:21.

Color Pt/Co: This determination was not problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D5386:16 and ASTM D1209:05R19.

Density at 20 °C: This determination was not problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ISO12185:96.

Distillation: This determination was not problematic. In total two statistical outliers were observed over three parameters. The calculated reproducibilities of IBP, 50% recovered and Dry Point after rejection of the statistical outliers are in agreement with the requirements of ASTM D850:21 for both automated and manual mode.

Total Nitrogen: This determination was problematic depending on the test method used. One statistical outlier was observed. The calculated reproducibility is in agreement with the requirements of ASTM D4629:17 but is not in agreement with the requirements of ASTM D7184:20.

Purity by GC: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D7504:21.

Methylcyclohexane: This determination may be problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the estimated reproducibility calculated with the Horwitz equation.

Toluene: This determination was problematic. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM D7504:21.

Nonaromatics: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D7504:21.

1,4-Dioxane: This determination was not problematic. All reporting participants agreed on a value near or below the detection limit. Therefore, no z-scores are calculated.

Total Impurities: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility calculated with the Horwitz equation based on 3 components.

Solidification Point (anhydrous basis): This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM D852:20.

Sulfur: This determination was very problematic. No statistical outliers were observed. The calculated reproducibility is not at all in agreement with the requirements of ASTM D7183:18aR23 and not in agreement with the less strict ASTM D5453:19. Although more participants reported to use ASTM D5453:19 for this determination it was decided to evaluate with the stricter reproducibility of ASTM D7183 because this test method is mentioned in specification ASTM D2359.
When the test results of ASTM D7183 were evaluated separately the calculated reproducibility is still not in agreement.

Thiophene: This determination was very problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not at all in agreement with the requirements of ASTM D7011:15R19.

Water: This determination was problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the requirements of ASTM E1064:16.
When the test results of ASTM E1064 were evaluated separately the calculated reproducibility is still not in agreement.

**sample #23023**

Total Chlorides: This determination may be problematic. Two statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is not in agreement with the requirements of ASTM D5194:18.

Organic Chlorides: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D5808:20.

## 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from reference methods are presented in the next table.

| Parameter | unit | n | average | 2.8 * sd | R(lit) |
|---|---|---|---|---|---|
| Acid Wash Color | | 24 | 1.0 | 1.3 | 2.2 |
| Acidity | | 20 | No free acid | n.a. | n.a. |
| Appearance | | 38 | C&B (Pass) | n.a. | n.a. |
| Bromine Index | mg Br/100g | 34 | 4.6 | 4.9 | 4.3 |
| Color Pt/Co | | 17 | 0.9 | 4.0 | 4.7 |
| Density at 20 °C | kg/L | 34 | 0.8791 | 0.0002 | 0.0005 |
| Distillation, IBP | °C | 25 | 79.6 | 0.4 | 0.6 |
| Distillation, 50% rec. | °C | 24 | 80.1 | 0.1 | 0.2 |
| Distillation, DP | °C | 25 | 80.2 | 0.3 | 0.5 |
| Total Nitrogen | mg/kg | 17 | 0.31 | 0.49 | 0.55 |
| Purity by GC | %M/M | 37 | 99.982 | 0.012 | 0.025 |
| Methylcyclohexane | mg/kg | 22 | 12.4 | 4.9 | 3.8 |
| Toluene | mg/kg | 37 | 79.7 | 15.9 | 10.2 |
| Nonaromatics | mg/kg | 36 | 88.0 | 55.6 | 71.4 |
| 1,4-Dioxane | mg/kg | 22 | <10 | n.e. | n.e. |
| Total Impurities | mg/kg | 27 | 183 | 63 | 65 |
| Solidification Point *) | °C | 22 | 5.48 | 0.07 | 0.05 |
| Sulfur | mg/kg | 35 | 1.88 | 1.14 | 0.44 |
| Thiophene | mg/kg | 7 | 3.63 | 3.23 | 1.22 |
| Water | mg/kg | 33 | 173 | 32 | 28 |

Table 5: reproducibilities of tests on sample #23020

*) anhydrous basis

| Parameter | unit | n | average | 2.8 * sd | R(lit) |
|---|---|---|---|---|---|
| Total Chlorides | mg/kg | 6 | 1.3 | 1.1 | 0.9 |
| Organic Chlorides | mg/kg | 13 | 1.2 | 1.0 | 1.3 |

Table 6: reproducibilities of tests on sample #23023

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

**4.3   COMPARISON OF THE PROFICIENCY TEST OF MARCH 2023 WITH PREVIOUS PTS**

| | March 2023 | February 2022 | February 2021 | February 2020 | February 2019 |
|---|---|---|---|---|---|
| Number of reporting laboratories | 39 | 39 | 55 | 34 | 50 |
| Number of test results | 491 | 423 | 722 | 400 | 532 |
| Number of statistical outliers | 15 | 9 | 33 | 12 | 17 |
| Percentage of statistical outliers | 3.1% | 2.1% | 4.6% | 3.0% | 3.2% |

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 to the requirements of the reference test methods. The conclusions are given in the following table.

| | March 2023 | February 2022 | February 2021 | February 2020 | February 2019 |
|---|---|---|---|---|---|
| Acid Wash Color | + | ++ | ++ | ++ | ++ |
| Bromine Index | - | - | ++ | ++ | ++ |
| Color Pt/Co | + | + | + | + | + |
| Density at 20°C | ++ | ++ | ++ | ++ | ++ |
| Distillation | + | + | + | - | ++ |
| Total Nitrogen | + | +/- | -- | n.e. | - |
| Purity by GC | ++ | ++ | +/- | -- | ++ |
| Methylcyclohexane | - | n.e. | +/- | -- | - |
| Toluene | - | - | - | - | n.e. |
| Nonaromatics | + | - | + | + | ++ |
| 1,4-Dioxane | n.e. | n.e. | n.e. | n.e. | n.e. |
| Total Impurities | +/- | - | + | +/- | n.e. |
| Solidification Point *) | - | +/- | -- | -- | + |
| Sulfur | -- | n.e. | - | n.e. | +/- |
| Thiophene | -- | n.e. | - | n.e. | n.e. |
| Water | - | - | ++ | - | - |
| Total Chlorides | - | + | +/- | + | +/- |
| Organic Chlorides | + | + | +/- | + | ++ |

Table 8: comparison of determinations to the reference test methods

*) anhydrous basis

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

**APPENDIX 1**

Determination of Acid Wash Color on sample #23020;

| lab | method | Reported test value | iis conversion *) | mark | z(targ) | remarks |
|-----|--------|--------------------|--------------------|------|---------|---------|
| 52 | D848 | 1- | 0.75 | | -0.31 | |
| 150 | D848 | 1+ | 1.25 | | 0.34 | |
| 171 | D848 | Pass | Pass | | ----- | |
| 315 | D848 | 2 | 2 | | 1.31 | |
| 317 | | n | ----- | | ----- | |
| 323 | D848 | 1 | 1 | | 0.01 | |
| 334 | | n | ----- | | ----- | |
| 347 | | n | ----- | | ----- | |
| 444 | | n | ----- | | ----- | |
| 445 | | n | ----- | | ----- | |
| 551 | D848 | 1- | 0.75 | | -0.31 | |
| 555 | D848 | 1- | 0.75 | | -0.31 | |
| 657 | D848 | 1- | 0.75 | | -0.31 | |
| 663 | D848 | No.1 | 1 | | 0.01 | |
| 734 | D848 | 1+ | 1.25 | | 0.34 | |
| 823 | D848 | 1- | 0.75 | | -0.31 | |
| 852 | | n | ----- | | ----- | |
| 855 | | n | ----- | | ----- | |
| 862 | | n | ----- | | ----- | |
| 864 | D848 | No. 1- | 0.75 | | -0.31 | |
| 866 | | n | ----- | | ----- | |
| 868 | | n | ----- | | ----- | |
| 870 | | n | ----- | | ----- | |
| 877 | | n | ----- | | ----- | |
| 912 | | n | ----- | | ----- | |
| 913 | | n | ----- | | ----- | |
| 962 | D848 | 1 | 1 | | 0.01 | |
| 963 | D848 | 1- | 0.75 | | -0.31 | |
| 970 | | n | ----- | | ----- | |
| 995 | D848 | 1 | 1 | | 0.01 | |
| 997 | D848 | 1 | 1 | | 0.01 | |
| 1041 | D848 | 1- / 1- | 0.75 | | -0.31 | |
| 1069 | | n | ----- | | ----- | |
| 1081 | | n | ----- | | ----- | |
| 1117 | | n | ----- | | ----- | |
| 1264 | D848 | 2 | 2 | | 1.31 | |
| 1320 | D848 | 1+ | 1.25 | | 0.34 | |
| 1357 | D848 | 1- | 0.75 | | -0.31 | |
| 1429 | D848 | 2 | 2 | | 1.31 | |
| 1530 | | n | ----- | | ----- | |
| 1728 | D848 | 3- | 2.75 | R(0.05) | 2.29 | |
| 1812 | | n | ----- | | ----- | |
| 1823 | | n | ----- | | ----- | |
| 1880 | D848 | <1 | 0.75 | | -0.31 | |
| 6134 | D848 | 0 | 0 | | -1.29 | |
| 6198 | | n | ----- | | ----- | |
| 6203 | | n | ----- | | ----- | |
| 6262 | D848 | 1- | 0.75 | | -0.31 | |
| 9008 | D848 | <1.0 | 0.75 | | -0.31 | |

| | | |
|---|---|---|
| normality | suspect | |
| n | 24 | |
| outliers | 1 | |
| mean (n) | 0.990 | |
| st.dev. (n) | 0.4632 | |
| R(calc.) | 1.297 | |
| st.dev.(D848:23) | 0.7693 | |
| R(D848:23) | 2.154 | |

*) In the calculation of the mean, standard deviation, reproducibility and in the graphs, a reported value of 'y-', '-y' or '<y' is changed into y-0.25 (for example 1- into 0.75) and 'y+' is changed into y+0.25 (for example 0+ into 0.25).
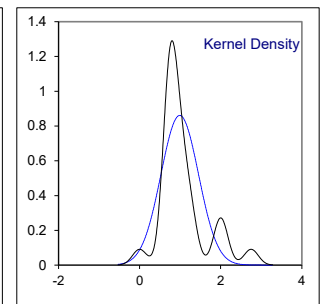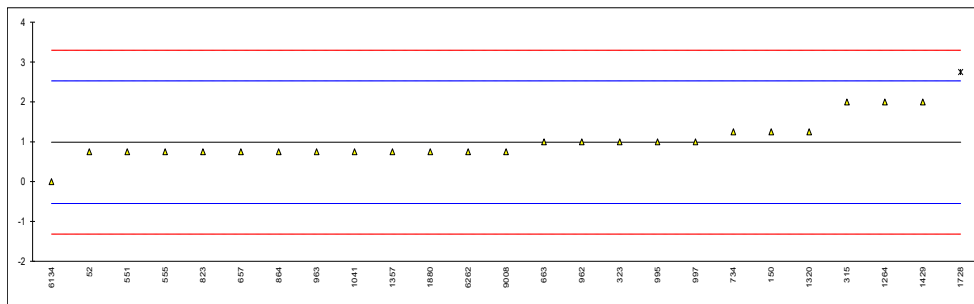
Determination of Acidity on sample #23020;

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | D847 | Nil | | ----- | |
| 150 | | ----- | | ----- | |
| 171 | D847 | No Free Acid | | ----- | |
| 315 | D847 | pass | | ----- | |
| 317 | | ----- | | ----- | |
| 323 | D847 | NFANEOA | | ----- | |
| 334 | D847 | PASS | | ----- | |
| 347 | | ----- | | ----- | |
| 444 | | ----- | | ----- | |
| 445 | | ----- | | ----- | |
| 551 | D847 | Pass | | ----- | |
| 555 | D847 | No free acid | | ----- | |
| 657 | D847 | Pass | | ----- | |
| 663 | D847 | Pass | | ----- | |
| 734 | | ----- | | ----- | |
| 823 | D847 | Pass | | ----- | |
| 852 | | ----- | | ----- | |
| 855 | | ----- | | ----- | |
| 862 | | ----- | | ----- | |
| 864 | | ----- | | ----- | |
| 866 | | ----- | | ----- | |
| 868 | | ----- | | ----- | |
| 870 | | ----- | | ----- | |
| 877 | | ----- | | ----- | |
| 912 | | ----- | | ----- | |
| 913 | | ----- | | ----- | |
| 962 | D847 | No free acid | | ----- | |
| 963 | D847 | No free acid | | ----- | |
| 970 | | ----- | | ----- | |
| 995 | | ----- | | ----- | |
| 997 | | ----- | | ----- | |
| 1041 | | ----- | | ----- | |
| 1069 | | ----- | | ----- | |
| 1081 | D847 | Pass | | ----- | |
| 1117 | D847 | 0.352 | | ----- | |
| 1264 | D847 | NO FREE ACID | | ----- | |
| 1320 | D847 | pass | | ----- | |
| 1357 | D847 | Free of Acid | | ----- | |
| 1429 | | ----- | | ----- | |
| 1530 | | ----- | | ----- | |
| 1728 | D847 | ABSENT | | ----- | |
| 1812 | | ----- | | ----- | |
| 1823 | D847 | Pass | | ----- | |
| 1880 | D847 | NFA | | ----- | |
| 6134 | D847 | No free acid | | ----- | |
| 6198 | | ----- | | ----- | |
| 6203 | | ----- | | ----- | |
| 6262 | D847 | PASS | | ----- | |
| 9008 | | ----- | | ----- | |
| | n | 20 | | | |
| | mean (n) | No free acid (Pass) | | | |

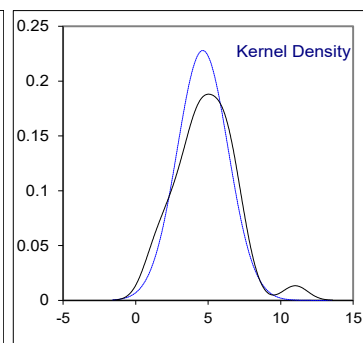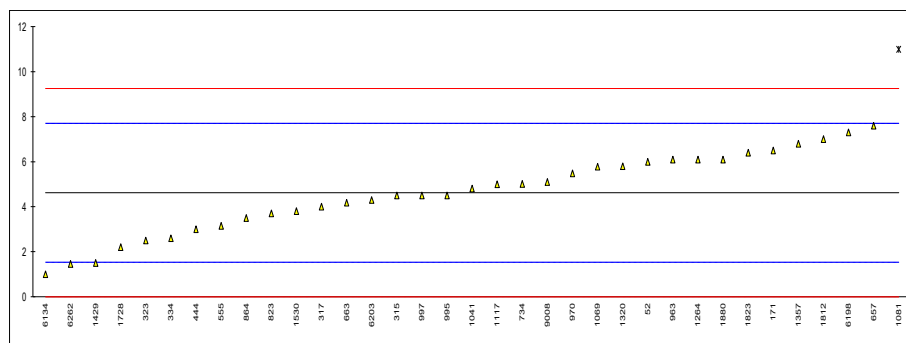## Determination of Appearance on sample #23020;

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | E2680 | pass | | ----- | |
| 150 | E2680 | Pass | | ----- | |
| 171 | Visual | Clear and free from suspended matter | | ----- | |
| 315 | E2680 | pass | | ----- | |
| 317 | D4176 | Pass | | ----- | |
| 323 | E2680 | C&B | | ----- | |
| 334 | EN15769 | clear and bright | | ----- | |
| 347 | E2680 | Pass | | ----- | |
| 444 | E2680 | Pass | | ----- | |
| 445 | | ----- | | ----- | |
| 551 | E2680 | Pass | | ----- | |
| 555 | E2680 | Pass | | ----- | |
| 657 | Visual | Pass | | ----- | |
| 663 | Visual | Pass | | ----- | |
| 734 | E2680 | Cl&Br | | ----- | |
| 823 | E2680 | Pass | | ----- | |
| 852 | | ----- | | ----- | |
| 855 | | ----- | | ----- | |
| 862 | | ----- | | ----- | |
| 864 | E2680 | Clear&Bright | | ----- | |
| 866 | | ----- | | ----- | |
| 868 | | ----- | | ----- | |
| 870 | | ----- | | ----- | |
| 877 | | ----- | | ----- | |
| 912 | | ----- | | ----- | |
| 913 | | ----- | | ----- | |
| 962 | D4176 | Clear & Bright | | ----- | |
| 963 | Visual | Clear | | ----- | |
| 970 | Visual | Clear & Bright | | ----- | |
| 995 | D4176 | C&B | | ----- | |
| 997 | E2680 | Pass C&B | | ----- | |
| 1041 | Visual | CBFSM | | ----- | |
| 1069 | Visual | Clear & bright | | ----- | |
| 1081 | Visual | B/C | | ----- | |
| 1117 | D4176 | Pass | | ----- | |
| 1264 | Visual | CLEAR & BRIGHT | | ----- | |
| 1320 | D4176 | C & B | | ----- | |
| 1357 | D4176 | Pass [C & B] | | ----- | |
| 1429 | Visual | Clear and Bright | | ----- | |
| 1530 | Visual | C&B | | ----- | |
| 1728 | Visual | CLEAR | | ----- | |
| 1812 | | ----- | | ----- | |
| 1823 | D4176 | Clear & FFSM & No Free Water | | ----- | |
| 1880 | Visual | Pass | | ----- | |
| 6134 | E2680 | Clear & Bright | | ----- | |
| 6198 | D4176 | Pass | | ----- | |
| 6203 | Visual | C&B | | ----- | |
| 6262 | Visual | bright and clear | | ----- | |
| 9008 | Visual | Clear | | ----- | |
| | n | 38 | | | |
| | mean (n) | Clear & Bright (Pass) | | | |

# Determination of Bromine Index on sample #23020; results in mg Br/100 g

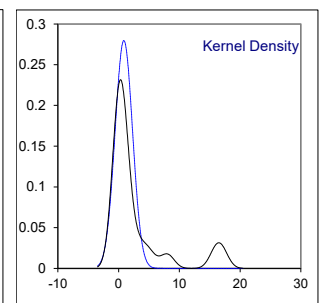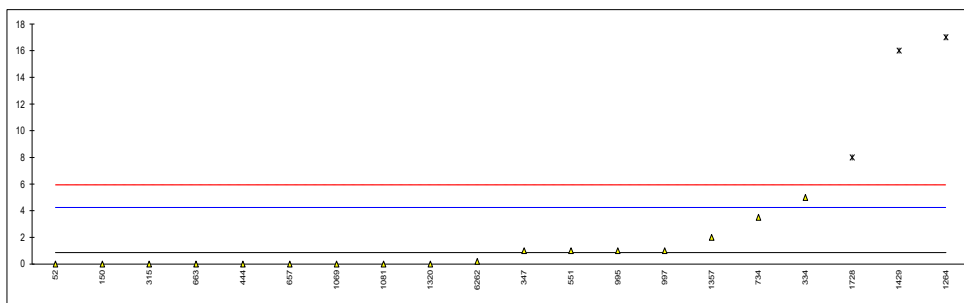| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 52 | D1492 | 6 | | 0.89 | |
| 150 | | ----- | | ----- | |
| 171 | D5776 | 6.5 | | 1.21 | |
| 315 | D5776 | 4.5 | | -0.08 | |
| 317 | D5776 | 4.0 | | -0.41 | |
| 323 | D5776 | 2.5 | | -1.38 | |
| 334 | D5776 | 2.6 | | -1.31 | |
| 347 | | ----- | | ----- | |
| 444 | D5776 | 3.0 | | -1.05 | |
| 445 | | ----- | | ----- | |
| 551 | | ----- | | ----- | |
| 555 | D5776 | 3.15 | | -0.96 | |
| 657 | D1492 | 7.595 | | 1.92 | |
| 663 | D5776 | 4.175 | | -0.29 | |
| 734 | D5776 | 5.01 | | 0.25 | |
| 823 | D1492 | 3.7 | | -0.60 | |
| 852 | | ----- | | ----- | |
| 855 | | ----- | | ----- | |
| 862 | | ----- | | ----- | |
| 864 | D5776 | 3.5 | | -0.73 | |
| 866 | | ----- | | ----- | |
| 868 | | ----- | | ----- | |
| 870 | | ----- | | ----- | |
| 877 | | ----- | | ----- | |
| 912 | | ----- | | ----- | |
| 913 | | ----- | | ----- | |
| 962 | | ----- | | ----- | |
| 963 | D1492 | 6.1 | | 0.96 | |
| 970 | D1492 | 5.48 | | 0.55 | |
| 995 | D5776 | 4.5 | | -0.08 | |
| 997 | D5776 | 4.5 | | -0.08 | |
| 1041 | DIN51774 | 4.81 | | 0.12 | |
| 1069 | D5776 | 5.775 | | 0.74 | |
| 1081 | D1492 | 11 | R(0.05) | 4.13 | |
| 1117 | D1492 | 5.0 | | 0.24 | |
| 1264 | D1492 | 6.1 | | 0.96 | |
| 1320 | D1492 | 5.8 | | 0.76 | |
| 1357 | D1492 | 6.8 | | 1.41 | |
| 1429 | D2710 | 1.50 | | -2.02 | |
| 1530 | DIN51774 | 3.805 | | -0.53 | |
| 1728 | D5776 | 2.2 | | -1.57 | |
| 1812 | D1492 | 7.0 | | 1.54 | |
| 1823 | D1492 | 6.4 | | 1.15 | |
| 1880 | D1492 | 6.1 | | 0.96 | |
| 6134 | D5776 | 1.0 | | -2.35 | |
| 6198 | D1492 | 7.3 | | 1.73 | |
| 6203 | D5776 | 4.3 | | -0.21 | |
| 6262 | D5776 | 1.455 | | -2.05 | |
| 9008 | D5776 | 5.1 | | 0.31 | |

|  |  |
|---|---|
| normality | OK |
| n | 34 |
| outliers | 1 |
| mean (n) | 4.625 |
| st.dev. (n) | 1.7514 |
| R(calc.) | 4.904 |
| st.dev.(D5776:21) | 1.5435 |
| R(D5776:21) | 4.322 |

## Determination of Color Pt/Co on sample #23020;

| lab | method | value | mark | z(targ) | remarks |
|---:|---|---|---|---:|---|
| 52 | D5386 | 0 | | -0.51 | |
| 150 | D5386 | 0 | | -0.51 | |
| 171 | D5386 | <5 | | ----- | |
| 315 | D5386 | 0 | | -0.51 | |
| 317 | D5386 | <5 | | ----- | |
| 323 | | off hue | | ----- | |
| 334 | D1209 | 5 | | 2.45 | |
| 347 | D5386 | 1 | | 0.08 | |
| 444 | D5386 | 0 | | -0.51 | |
| 445 | | ----- | | ----- | |
| 551 | D5386 | 1 | | 0.08 | |
| 555 | | ----- | | ----- | |
| 657 | D5386 | 0 | | -0.51 | |
| 663 | D5386 | 0 | | -0.51 | |
| 734 | D1209 | 3.5 | | 1.56 | |
| 823 | D5386 | <1 | | ----- | |
| 852 | | ----- | | ----- | |
| 855 | | ----- | | ----- | |
| 862 | | ----- | | ----- | |
| 864 | D1209 | <10 | | ----- | |
| 866 | | ----- | | ----- | |
| 868 | | ----- | | ----- | |
| 870 | | ----- | | ----- | |
| 877 | | ----- | | ----- | |
| 912 | | ----- | | ----- | |
| 913 | | ----- | | ----- | |
| 962 | | ----- | | ----- | |
| 963 | | ----- | | ----- | |
| 970 | D1209 | <5 | | ----- | |
| 995 | D5386 | 1 | | 0.08 | |
| 997 | D1209 | 1 | | 0.08 | |
| 1041 | ISO6271 | <5 | | ----- | |
| 1069 | D1209 | 0 | | -0.51 | |
| 1081 | D5386 | 0 | | -0.51 | |
| 1117 | D1209 | off hue | | ----- | |
| 1264 | D1209 | 17 | C,R(0.01) | 9.54 | first reported 16 |
| 1320 | D5386 | 0 | | -0.51 | |
| 1357 | D1209 | 2.0 | | 0.67 | |
| 1429 | D1209 | 16 | R(0.01) | 8.95 | |
| 1530 | D1209 | <3 | | ----- | |
| 1728 | D1209 | 8 | C,R(0.01) | 4.22 | first reported 12 |
| 1812 | | ----- | | ----- | |
| 1823 | D5386 | ND | | ----- | |
| 1880 | | ----- | | ----- | |
| 6134 | | ----- | | ----- | |
| 6198 | | ----- | | ----- | |
| 6203 | D1209 | <0 | | ----- | |
| 6262 | D1209 | 0.2 | | -0.39 | |
| 9008 | D5386 | <0.0 | | ----- | |

| | | | |
|---|---|---|---|
| | normality | not OK | |
| | n | 17 | |
| | outliers | 3 | |
| | mean (n) | 0.86 | |
| | st.dev. (n) | 1.427 | |
| | R(calc.) | 4.00 | |
| | st.dev.(D5386:16) | 1.691 | |
| | R(D5386:16) | 4.73 | |
| Compare | | | |
| | R(D1209:05R19) | 7 | |

# Determination of Density at 20 °C on sample #23020; results in kg/L

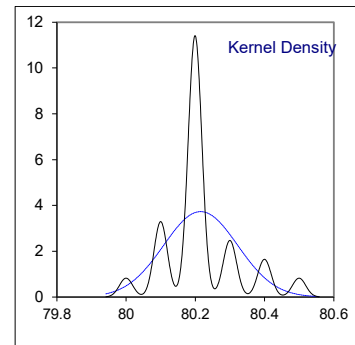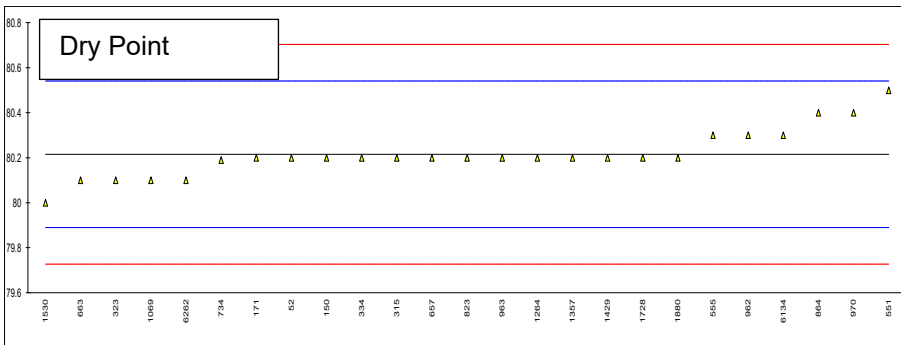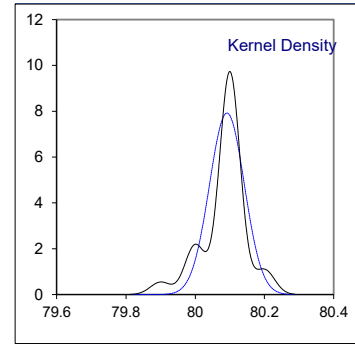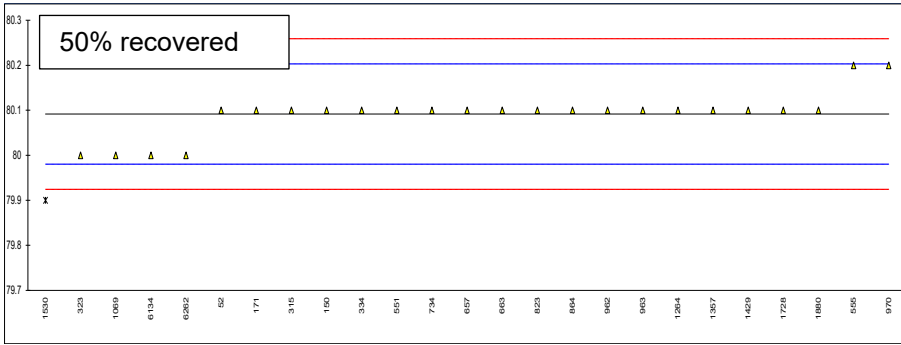| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | D4052 | 0.8790 | | -0.44 | |
| 150 | ISO12185 | 0.8790 | | -0.44 | |
| 171 | ISO12185 | 0.8791 | | 0.12 | |
| 315 | D4052 | 0.8790 | | -0.44 | |
| 317 | ISO12185 | 0.8791 | | 0.12 | |
| 323 | D4052 | 0.8790 | | -0.44 | |
| 334 | ISO12185 | 0.8844 | R(0.01) | 29.80 | |
| 347 | D4052 | 0.8790 | C | -0.44 | first reported 0.8843 at 15 °C |
| 444 | D4052 | 0.8791 | | 0.12 | |
| 445 | | ----- | | ----- | |
| 551 | D4052 | 0.8792 | | 0.68 | |
| 555 | D4052 | 0.87901 | | -0.39 | |
| 657 | D4052 | 0.8791 | | 0.12 | |
| 663 | D4052 | 0.87902 | | -0.33 | |
| 734 | D4052 | 0.8792 | | 0.68 | |
| 823 | D4052 | 0.8791 | | 0.12 | |
| 852 | | ----- | | ----- | |
| 855 | | ----- | | ----- | |
| 862 | | ----- | | ----- | |
| 864 | D4052 | 0.8791 | | 0.12 | |
| 866 | | ----- | | ----- | |
| 868 | | ----- | | ----- | |
| 870 | | ----- | | ----- | |
| 877 | | ----- | | ----- | |
| 912 | | ----- | | ----- | |
| 913 | | ----- | | ----- | |
| 962 | D4052 | 0.8791 | | 0.12 | |
| 963 | ISO12185 | 0.8791 | | 0.12 | |
| 970 | D4052 | 0.8791 | | 0.12 | |
| 995 | ISO12185 | 0.8791 | | 0.12 | |
| 997 | ISO12185 | 0.87903 | | -0.27 | |
| 1041 | | ----- | | ----- | |
| 1069 | D4052 | 0.87875 | R(0.05) | -1.84 | |
| 1081 | | ----- | | ----- | |
| 1117 | D4052 | 0.8789 | | -1.00 | |
| 1264 | D4052 | 0.8791 | | 0.12 | |
| 1320 | ISO12185 | 0.879 | | -0.44 | |
| 1357 | D4052 | 0.8791 | | 0.12 | |
| 1429 | D4052 | 0.8793 | | 1.24 | |
| 1530 | ISO12185 | 0.87927 | | 1.07 | |
| 1728 | ISO12185 | 0.87902 | | -0.33 | |
| 1812 | ISO12185 | 0.8790 | | -0.44 | |
| 1823 | D4052 | 0.8791 | | 0.12 | |
| 1880 | D4052 | 0.8790 | | -0.44 | |
| 6134 | | ----- | | ----- | |
| 6198 | D4052 | 0.87905 | | -0.16 | |
| 6203 | ISO12185 | 0.87918 | | 0.57 | |
| 6262 | D4052 | 0.8791 | | 0.12 | |
| 9008 | D4052 | 0.8791 | | 0.12 | |

| | | | |
|---|---|---|---|
| | normality | OK | |
| | n | 34 | |
| | outliers | 2 | |
| | mean (n) | 0.87908 | |
| | st.dev. (n) | 0.000083 | |
| | R(calc.) | 0.00023 | |
| | st.dev.(ISO12185:96) | 0.000179 | |
| | R(ISO12185:96) | 0.00050 | |
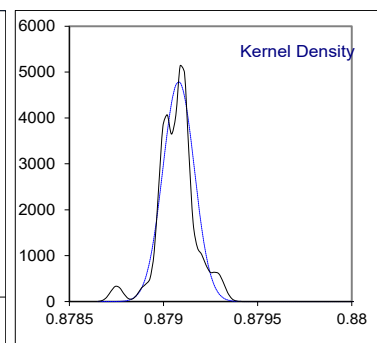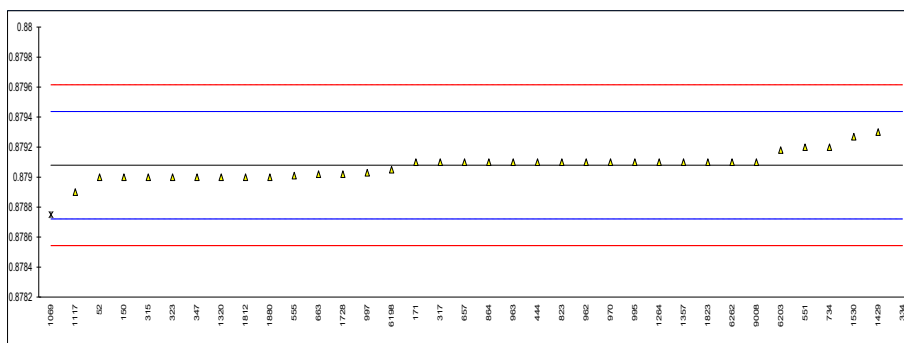
## Determination of Distillation on sample #23020; results in °C

| Lab | method | IBP | mark | z(targ) | 50% | mark | z(targ) | DP | mark | z(targ) | range | mark |
|-----|--------|-----|------|---------|-----|------|---------|-----|------|---------|-------|------|
| 52 | D850-automated | 79.7 | | 0.25 | 80.1 | | 0.15 | 80.2 | | -0.10 | 0.5 | |
| 150 | D850-automated | 79.6 | | -0.23 | 80.1 | | 0.15 | 80.2 | | -0.10 | ----- | |
| 171 | D850-automated | 79.8 | | 0.74 | 80.1 | | 0.15 | 80.2 | | -0.10 | 0.4 | |
| 315 | D850-automated | 79.7 | | 0.25 | 80.1 | | 0.15 | 80.2 | | -0.10 | 0.5 | |
| 317 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | |
| 323 | D850-automated | 79.8 | | 0.74 | 80.0 | | -1.65 | 80.1 | | -0.71 | 0.3 | |
| 334 | D850-automated | 79.7 | | 0.25 | 80.1 | | 0.15 | 80.2 | | -0.10 | 0.5 | |
| 347 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | |
| 444 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | |
| 445 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | |
| 551 | D850-automated | 79.7 | | 0.25 | 80.1 | | 0.15 | 80.5 | | 1.75 | 0.8 | |
| 555 | D850-manual | 79.8 | | 0.74 | 80.2 | | 1.94 | 80.3 | | 0.52 | 0.50 | |
| 657 | D850-automated | 79.7 | | 0.25 | 80.1 | | 0.15 | 80.2 | | -0.10 | 0.5 | |
| 663 | D850-automated | 79.6 | | -0.23 | 80.1 | | 0.15 | 80.1 | | -0.71 | 0.5 | |
| 734 | D850-automated | 79.68 | | 0.16 | 80.10 | | 0.15 | 80.19 | | -0.16 | 0.51 | |
| 823 | D850-automated | 79.4 | | -1.19 | 80.1 | | 0.15 | 80.2 | | -0.10 | 0.8 | |
| 852 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | |
| 855 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | |
| 862 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | |
| 864 | D850 | 79.8 | | 0.74 | 80.1 | | 0.15 | 80.4 | | 1.13 | 0.6 | |
| 866 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | |
| 868 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | |
| 870 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | |
| 877 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | |
| 912 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | |
| 913 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | |
| 962 | D850-automated | 79.3 | | -1.67 | 80.1 | | 0.15 | 80.3 | | 0.52 | 1.0 | |
| 963 | D850-automated | 79.4 | C | -1.19 | 80.1 | | 0.15 | 80.2 | | -0.10 | 1.0 | |
| 970 | D850 | 79.6 | | -0.23 | 80.2 | | 1.94 | 80.4 | | 1.13 | ----- | |
| 995 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | |
| 997 | D850-manual | 79.40 | | -1.19 | ----- | | ----- | ----- | | ----- | ----- | |
| 1041 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | |
| 1069 | D850-automated | 79.1 | R(0.05) | -2.64 | 80.0 | | -1.65 | 80.1 | | -0.71 | 79,1-80,1 | |
| 1081 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | |
| 1117 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | |
| 1264 | D850-automated | 79.6 | | -0.23 | 80.1 | | 0.15 | 80.2 | | -0.10 | 0.8 | |
| 1320 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | |
| 1357 | D850-automated | 79.7 | | 0.25 | 80.1 | | 0.15 | 80.2 | | -0.10 | 0.5 | |
| 1429 | D850-automated | 79.7 | | 0.25 | 80.1 | | 0.15 | 80.2 | | -0.10 | 0.5 | |
| 1530 | D850-automated | 79.70 | | 0.25 | 79.90 | R(0.05) | -3.44 | 80.00 | | -1.32 | 0.30 | |
| 1728 | D850-manual | 79.8 | | 0.74 | 80.1 | | 0.15 | 80.2 | | -0.10 | 0.4 | |
| 1812 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | |
| 1823 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | |
| 1880 | D850-automated | 79.6 | | -0.23 | 80.1 | | 0.15 | 80.2 | | -0.10 | 0.6 | |
| 6134 | D850-automated | 79.6 | | -0.23 | 80.0 | | -1.65 | 80.3 | | 0.52 | 0.7 | |
| 6198 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | |
| 6203 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | |
| 6262 | D850-automated | 79.8 | | 0.74 | 80.0 | | -1.65 | 80.1 | | -0.71 | 0.3 | |
| 9008 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | |
| | normality | OK | | | suspect | | | suspect | | | | |
| | n | 25 | | | 24 | | | 25 | | | | |
| | outliers | 1 | | | 1 | | | 0 | | | | |
| | mean (n) | 79.65 | | | 80.09 | | | 80.22 | | | | |
| | st.dev. (n) | 0.142 | | | 0.050 | | | 0.107 | | | | |
| | R(calc.) | 0.40 | | | 0.14 | | | 0.30 | | | | |
| | st.dev.(D850-A:21) | 0.208 | | | 0.056 | | | 0.163 | | | | |
| | R(D850-A:21) | 0.58 | | | 0.16 | | | 0.46 | | | | |
| Compare | | | | | | | | | | | | |
| | R(D850-M:21) | 0.41 | | | 0.65 | | | 0.65 | | | | |

Lab 963 first reported 79.2
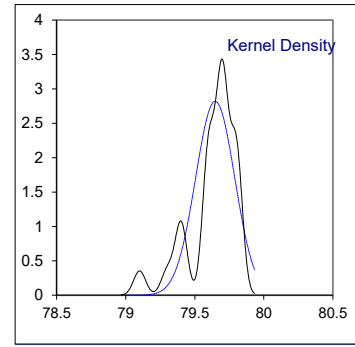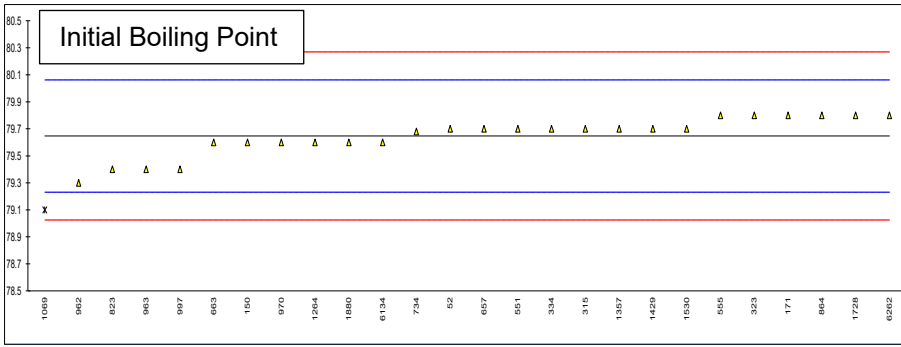
## Determination of Total Nitrogen on sample #23020; results in mg/kg

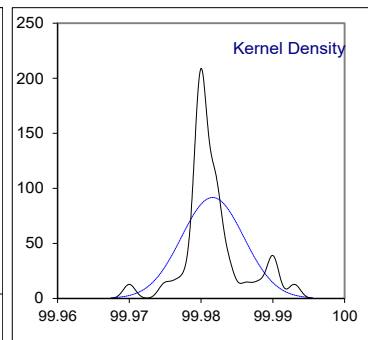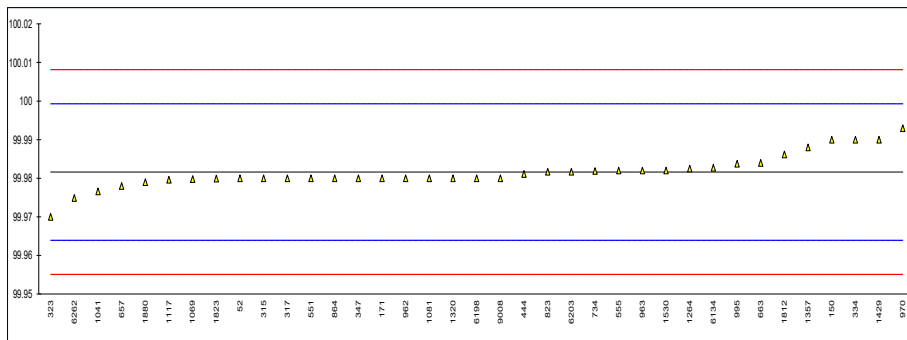| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | D7184 | 0.12 | | -0.95 | |
| 150 | D7184 | <0.10 | | ----- | |
| 171 | | ----- | | ----- | |
| 315 | D7184 | 0.18 | | -0.65 | |
| 317 | D4629 | 0.4 | | 0.48 | |
| 323 | D6069 | <1 | | ----- | |
| 334 | D4629 | <0.3 | | ----- | |
| 347 | D4629 | <1 | | ----- | |
| 444 | D4629 | 0.32 | | 0.07 | |
| 445 | | ----- | | ----- | |
| 551 | D4629 | <1 | | ----- | |
| 555 | | ----- | | ----- | |
| 657 | D4629 | 3.307 | C,G(0.01) | 15.34 | first reported 4.8867 |
| 663 | D4629 | <0.3 | | ----- | |
| 734 | D7184 | 0.67 | | 1.86 | |
| 823 | D7184 | 0.28 | | -0.13 | |
| 852 | | ----- | | ----- | |
| 855 | | ----- | | ----- | |
| 862 | | ----- | | ----- | |
| 864 | SH/T 0657 | <1 | | ----- | |
| 866 | | ----- | | ----- | |
| 868 | | ----- | | ----- | |
| 870 | | ----- | | ----- | |
| 877 | | ----- | | ----- | |
| 912 | | ----- | | ----- | |
| 913 | | ----- | | ----- | |
| 962 | D7184 | 0.2 | | -0.54 | |
| 963 | D7184 | <0.3 | | ----- | |
| 970 | | ----- | | ----- | |
| 995 | | ----- | | ----- | |
| 997 | | ----- | | ----- | |
| 1041 | D6069 | <0.2 | | ----- | |
| 1069 | D4629 | 0.528 | | 1.13 | |
| 1081 | D6069 | 0.4 | | 0.48 | |
| 1117 | D7184 | 0.0817 | C | -1.15 | first reported 0.093 mg/L |
| 1264 | D4629 | 0.45 | | 0.73 | |
| 1320 | D4629 | 0.17 | | -0.70 | |
| 1357 | D4629 | <0.3 | | ----- | |
| 1429 | D4629 | <0.5 | | ----- | |
| 1530 | D4629 | <1 | | ----- | |
| 1728 | | ----- | | ----- | |
| 1812 | D6069 | 0.120 | | -0.95 | |
| 1823 | D7184 | <0.20 | | ----- | |
| 1880 | D6069 | <0.1 | | ----- | |
| 6134 | D4629 | 0.35 | | 0.22 | |
| 6198 | D4629 | 0.23 | | -0.39 | |
| 6203 | D4629 | 0.56 | | 1.30 | |
| 6262 | | ----- | | ----- | |
| 9008 | D7184 | 0.1466 | | -0.82 | |

| | | | | |
|---|---|---|---|---|
| | normality | OK | | |
| | n | 17 | | |
| | outliers | 1 | | |
| | mean (n) | 0.306 | | |
| | st.dev. (n) | 0.1739 | | |
| | R(calc.) | 0.487 | | |
| | st.dev.(D4629:17) | 0.1956 | | |
| | R(D4629:17) | 0.548 | | application range 0.3 – 100 mg/kg |
| Compare | | | | |
| | R(D7184:20) | 0.236 | | application range 0.13 – 1.2 mg/kg |
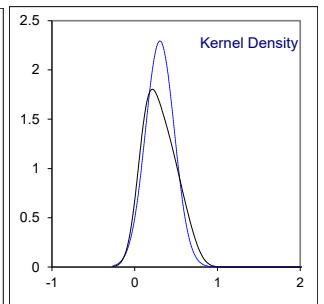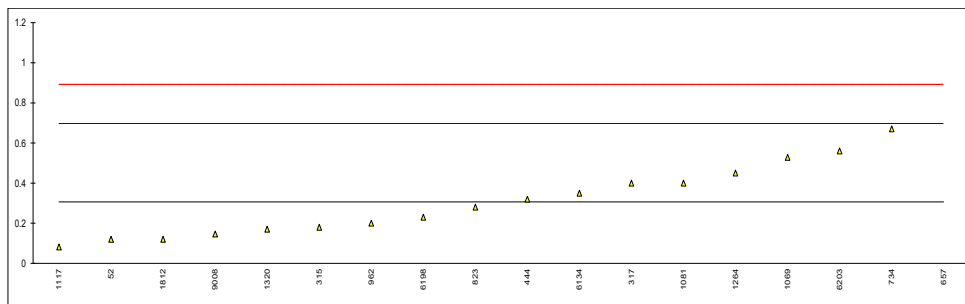
# Determination of Purity by GC on sample #23020; results in %M/M

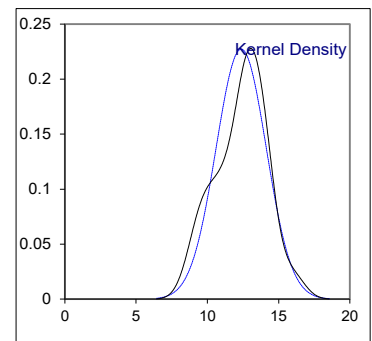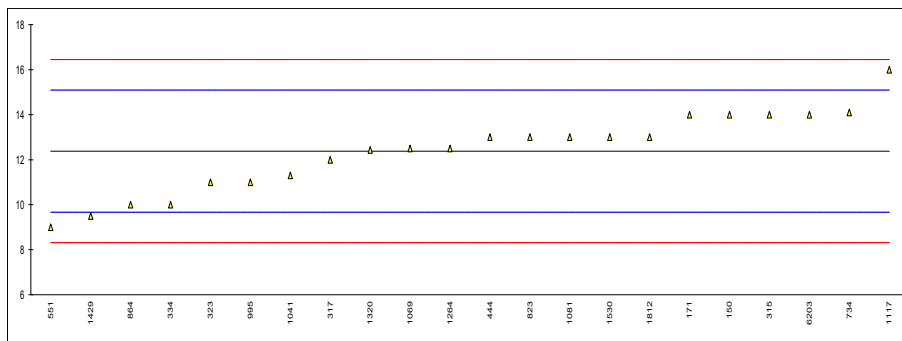| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | D7504 | 99.98 | | -0.18 | |
| 150 | D7504 | 99.99 | | 0.95 | |
| 171 | D7504 | 99.98 | | -0.18 | |
| 315 | D7504 | 99.98 | | -0.18 | |
| 317 | D7504 | 99.98 | | -0.18 | |
| 323 | D7504 | 99.97 | | -1.31 | |
| 334 | D4492 | 99.99 | | 0.95 | |
| 347 | D4492 | 99.98 | | -0.18 | |
| 444 | D4492 | 99.9811 | | -0.06 | |
| 445 | | ----- | | ----- | |
| 551 | D7504 | 99.98 | | -0.18 | |
| 555 | D7504 | 99.982 | | 0.04 | |
| 657 | D7504 | 99.9780 | | -0.41 | |
| 663 | D7504 | 99.984 | | 0.27 | |
| 734 | D7504 | 99.98187 | | 0.03 | |
| 823 | D7504 | 99.9817 | | 0.01 | |
| 852 | | ----- | | ----- | |
| 855 | | ----- | | ----- | |
| 862 | | ----- | | ----- | |
| 864 | D7504 | 99.98 | | -0.18 | |
| 866 | | ----- | | ----- | |
| 868 | | ----- | | ----- | |
| 870 | | ----- | | ----- | |
| 877 | | ----- | | ----- | |
| 912 | | ----- | | ----- | |
| 913 | | ----- | | ----- | |
| 962 | D7504 | 99.98 | | -0.18 | |
| 963 | D7504 | 99.982 | | 0.04 | |
| 970 | D7504 | 99.993 | | 1.29 | |
| 995 | D7504 | 99.9838 | | 0.25 | |
| 997 | | ----- | | ----- | |
| 1041 | | 99.9766 | | -0.57 | |
| 1069 | D4492 | 99.9798 | | -0.21 | |
| 1081 | D4492 | 99.98 | | -0.18 | |
| 1117 | D4492 | 99.9796 | | -0.23 | |
| 1264 | D7504 | 99.9825 | | 0.10 | |
| 1320 | D7504 | 99.98 | C | -0.18 | first reported 99.99 |
| 1357 | D7504 | 99.988 | | 0.72 | |
| 1429 | D7504 | 99.99 | | 0.95 | |
| 1530 | D7504 | 99.982 | | 0.04 | |
| 1728 | | ----- | | ----- | |
| 1812 | | 99.9862 | | 0.52 | |
| 1823 | D7504 | 99.9799 | | -0.19 | |
| 1880 | D4492 | 99.979 | | -0.30 | |
| 6134 | D7504 | 99.9827 | | 0.12 | |
| 6198 | D7504 | 99.98 | | -0.18 | |
| 6203 | D7504 | 99.9817 | | 0.01 | |
| 6262 | D7504 | 99.9749 | | -0.76 | |
| 9008 | D7504 | 99.98 | | -0.18 | |

| | | |
|---|---|---|
| normality | suspect | |
| n | 37 | |
| outliers | 0 | |
| mean (n) | 99.98161 | |
| st.dev. (n) | 0.004352 | |
| R(calc.) | 0.01219 | |
| st.dev.(D7504:21) | 0.008835 | |
| R(D7504:21) | 0.02474 | |





Kernel Density

## Determination of Methylcyclohexane on sample #23020 in mg/kg

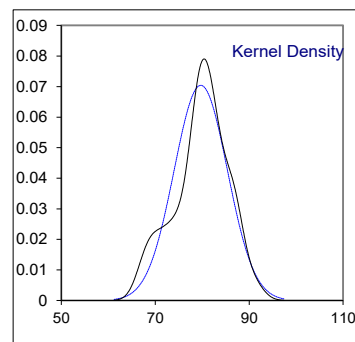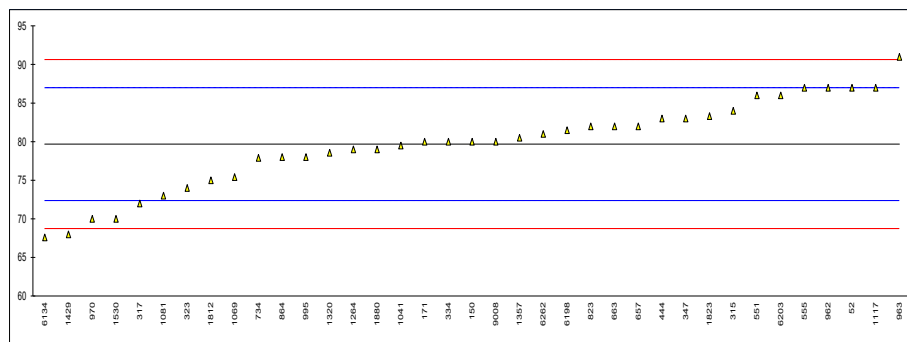| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | | ----- | | ----- | |
| 150 | D7504 | 14 | | 1.20 | |
| 171 | D7504 | 14 | | 1.20 | |
| 315 | D5713 | 14 | | 1.20 | |
| 317 | D5713 | 12 | | -0.28 | |
| 323 | D7504 | 11 | | -1.02 | |
| 334 | D4492 | 10 | | -1.75 | |
| 347 | | ----- | | ----- | |
| 444 | D5713 | 13 | | 0.46 | |
| 445 | | ----- | | ----- | |
| 551 | D7504 | 9 | C | -2.49 | first reported 19 |
| 555 | | ----- | | ----- | |
| 657 | | ----- | | ----- | |
| 663 | | ----- | | ----- | |
| 734 | D7360 | 14.1 | | 1.27 | |
| 823 | D5713 | 13 | | 0.46 | |
| 852 | | ----- | | ----- | |
| 855 | | ----- | | ----- | |
| 862 | | ----- | | ----- | |
| 864 | D7504 | 10 | | -1.75 | |
| 866 | | ----- | | ----- | |
| 868 | | ----- | | ----- | |
| 870 | | ----- | | ----- | |
| 877 | | ----- | | ----- | |
| 912 | | ----- | | ----- | |
| 913 | | ----- | | ----- | |
| 962 | | ----- | | ----- | |
| 963 | | ----- | | ----- | |
| 970 | | ----- | | ----- | |
| 995 | D7504 | 11 | | -1.02 | |
| 997 | | ----- | | ----- | |
| 1041 | | 11.3 | | -0.80 | |
| 1069 | D4492 | 12.5 | | 0.09 | |
| 1081 | | 13 | | 0.46 | |
| 1117 | D4492 | 16 | C | 2.67 | first reported 0.0016 mg/kg |
| 1264 | D7504 | 12.5 | | 0.09 | |
| 1320 | D7504 | 12.44 | C | 0.04 | first reported 2.77 |
| 1357 | D7504 | <10 | | ----- | |
| 1429 | D7504 | 9.5 | | -2.12 | |
| 1530 | D7504 | 13.0 | | 0.46 | |
| 1728 | | ----- | | ----- | |
| 1812 | | 13 | C | 0.46 | first reported 18 |
| 1823 | | ----- | | ----- | |
| 1880 | | ----- | | ----- | |
| 6134 | | ----- | | ----- | |
| 6198 | | ----- | | ----- | |
| 6203 | D7504 | 14 | | 1.20 | |
| 6262 | | ----- | | ----- | |
| 9008 | | ----- | | ----- | |

| | | | |
|---|---|---|---|
| | normality | OK | |
| | n | 22 | |
| | outliers | 0 | |
| | mean (n) | 12.38 | |
| | st.dev. (n) | 1.755 | |
| | R(calc.) | 4.91 | |
| | st.dev.(Horwitz) | 1.356 | |
| | R(Horwitz) | 3.80 | |

## Determination of Toluene on sample #23020; results in mg/kg

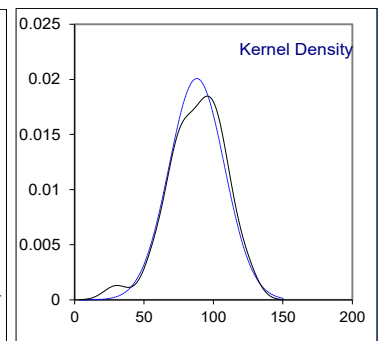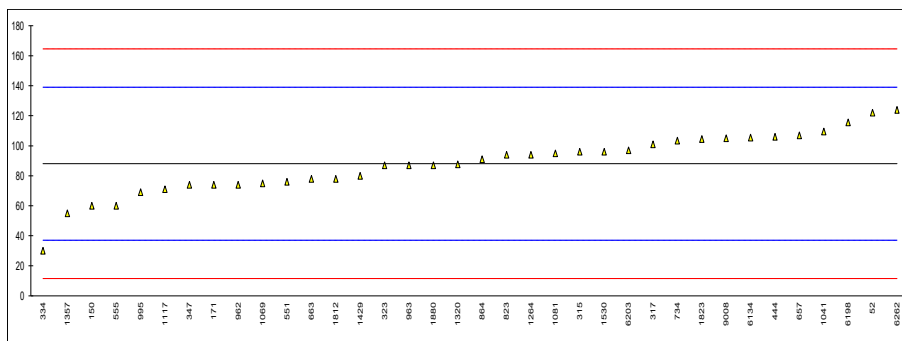| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | D7504 | 87 | | 2.00 | |
| 150 | D7504 | 80 | C | 0.09 | first reported 0.008 mg/kg |
| 171 | D7504 | 80 | | 0.09 | |
| 315 | D7504 | 84 | | 1.18 | |
| 317 | D7504 | 72 | | -2.10 | |
| 323 | D7504 | 74 | | -1.56 | |
| 334 | D4492 | 80 | | 0.09 | |
| 347 | D4492 | 83 | | 0.91 | |
| 444 | D5713 | 83 | | 0.91 | |
| 445 | | ----- | | ----- | |
| 551 | D7504 | 86 | | 1.73 | |
| 555 | D7504 | 87 | | 2.00 | |
| 657 | D7504 | 82 | | 0.63 | |
| 663 | D7504 | 82 | | 0.63 | |
| 734 | D7504 | 77.9 | | -0.49 | |
| 823 | D7504 | 82 | | 0.63 | |
| 852 | | ----- | | ----- | |
| 855 | | ----- | | ----- | |
| 862 | | ----- | | ----- | |
| 864 | D7504 | 78 | | -0.46 | |
| 866 | | ----- | | ----- | |
| 868 | | ----- | | ----- | |
| 870 | | ----- | | ----- | |
| 877 | | ----- | | ----- | |
| 912 | | ----- | | ----- | |
| 913 | | ----- | | ----- | |
| 962 | D7504 | 87 | | 2.00 | |
| 963 | D7504 | 91 | | 3.10 | |
| 970 | D7504 | 70 | | -2.65 | |
| 995 | D7504 | 78 | | -0.46 | |
| 997 | | ----- | | ----- | |
| 1041 | | 79.5 | | -0.05 | |
| 1069 | D4492 | 75.4 | | -1.17 | |
| 1081 | D4492 | 73 | | -1.83 | |
| 1117 | D4492 | 87 | C | 2.00 | first reported 0.0087 mg/kg |
| 1264 | D7504 | 79 | | -0.19 | |
| 1320 | D7504 | 78.56 | C | -0.31 | first reported 25.57 |
| 1357 | D7504 | 80.5 | | 0.22 | |
| 1429 | D7504 | 68 | | -3.20 | |
| 1530 | D7504 | 70 | C | -2.65 | first reported <10 |
| 1728 | | ----- | | ----- | |
| 1812 | | 75 | | -1.28 | |
| 1823 | D7504 | 83.31 | | 0.99 | |
| 1880 | D4492 | 79 | | -0.19 | |
| 6134 | D7504 | 67.6 | | -3.31 | |
| 6198 | D7504 | 81.5 | | 0.50 | |
| 6203 | D7504 | 86 | | 1.73 | |
| 6262 | D7504 | 81 | | 0.36 | |
| 9008 | D7504 | 80 | | 0.09 | |

| | | |
|---|---|---|
| normality | OK | |
| n | 37 | |
| outliers | 0 | |
| mean (n) | 79.68 | |
| st.dev. (n) | 5.665 | |
| R(calc.) | 15.86 | |
| st.dev.(D7504:21) | 3.653 | |
| R(D7504:21) | 10.23 | |

## Determination of Nonaromatics on sample #23020; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 52 | D7504 | 122 | | 1.33 | |
| 150 | D7504 | 60 | C | -1.10 | first reported 0.006 mg/kg |
| 171 | D7504 | 74 | | -0.55 | |
| 315 | D7504 | 96 | | 0.31 | |
| 317 | D7504 | 101 | | 0.51 | |
| 323 | D7504 | 87 | | -0.04 | |
| 334 | D4492 | 30 | | -2.28 | |
| 347 | D4492 | 74 | | -0.55 | |
| 444 | D4492 | 106 | | 0.70 | |
| 445 | | ----- | | ----- | |
| 551 | D7504 | 76 | | -0.47 | |
| 555 | D7504 | 60 | | -1.10 | |
| 657 | D7504 | 107 | | 0.74 | |
| 663 | D7504 | 78 | | -0.39 | |
| 734 | D7504 | 103.4 | | 0.60 | |
| 823 | D7504 | 94 | | 0.23 | |
| 852 | | ----- | | ----- | |
| 855 | | ----- | | ----- | |
| 862 | | ----- | | ----- | |
| 864 | D7504 | 91 | | 0.12 | |
| 866 | | ----- | | ----- | |
| 868 | | ----- | | ----- | |
| 870 | | ----- | | ----- | |
| 877 | | ----- | | ----- | |
| 912 | | ----- | | ----- | |
| 913 | | ----- | | ----- | |
| 962 | D7504 | 74 | | -0.55 | |
| 963 | D7504 | 87 | | -0.04 | |
| 970 | | ----- | | ----- | |
| 995 | D7504 | 69 | | -0.75 | |
| 997 | | ----- | | ----- | |
| 1041 | | 109.5 | | 0.84 | |
| 1069 | D4492 | 75.0 | | -0.51 | |
| 1081 | D4492 | 95 | | 0.27 | |
| 1117 | D4492 | 71 | C | -0.67 | first reported 0.0071 mg/kg |
| 1264 | D7504 | 94 | | 0.23 | |
| 1320 | D7504 | 87.64 | C | -0.02 | first reported 26.86 |
| 1357 | D7504 | 55 | | -1.30 | |
| 1429 | D7504 | 80 | | -0.31 | |
| 1530 | D7504 | 96 | C | 0.31 | first reported 166.4 |
| 1728 | | ----- | | ----- | |
| 1812 | | 78 | | -0.39 | |
| 1823 | D7504 | 104.56 | | 0.65 | |
| 1880 | D4492 | 87 | | -0.04 | |
| 6134 | D7504 | 105.5 | | 0.68 | |
| 6198 | D7504 | 115.6 | | 1.08 | |
| 6203 | D7504 | 97 | | 0.35 | |
| 6262 | D7504 | 124 | C | 1.41 | first reported 149 |
| 9008 | D7504 | 105 | | 0.67 | |

| | | |
|---|---|---|
| | normality | OK |
| | n | 36 |
| | outliers | 0 |
| | mean (n) | 88.03 |
| | st.dev. (n) | 19.867 |
| | R(calc.) | 55.63 |
| | st.dev.(D7504:21) | 25.508 |
| | R(D7504:21) | 71.42 |

Determination of 1,4-Dioxane on sample #23020; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | D7504 | <2 | | ----- | |
| 150 | | ----- | | ----- | |
| 171 | D7504 | <2 | | ----- | |
| 315 | D7504 | <2 | | ----- | |
| 317 | D7504 | <2 | | ----- | |
| 323 | D7504 | <2 | | ----- | |
| 334 | | ----- | | ----- | |
| 347 | | ----- | | ----- | |
| 444 | | ----- | | ----- | |
| 445 | | ----- | | ----- | |
| 551 | D7504 | <10 | | ----- | |
| 555 | D7504 | <10.0 | | ----- | |
| 657 | D7504 | <2 | | ----- | |
| 663 | UOP921-92 | <1 | | ----- | |
| 734 | D7504 | 0 | | ----- | |
| 823 | D7504 | <2 | | ----- | |
| 852 | | ----- | | ----- | |
| 855 | | ----- | | ----- | |
| 862 | | ----- | | ----- | |
| 864 | D7504 | <10 | | ----- | |
| 866 | | ----- | | ----- | |
| 868 | | ----- | | ----- | |
| 870 | | ----- | | ----- | |
| 877 | | ----- | | ----- | |
| 912 | | ----- | | ----- | |
| 913 | | ----- | | ----- | |
| 962 | D7504 | <10 | | ----- | |
| 963 | D7504 | <10 | | ----- | |
| 970 | | ----- | | ----- | |
| 995 | D7504 | 0 | | ----- | |
| 997 | | ----- | | ----- | |
| 1041 | | ----- | | ----- | |
| 1069 | | ----- | | ----- | |
| 1081 | | ----- | | ----- | |
| 1117 | | ----- | | ----- | |
| 1264 | D7504 | <10 | | ----- | |
| 1320 | | ----- | | ----- | |
| 1357 | D7504 | <10 | | ----- | |
| 1429 | | ----- | | ----- | |
| 1530 | D7504 | <10 | | ----- | |
| 1728 | | ----- | | ----- | |
| 1812 | | ----- | | ----- | |
| 1823 | D7504 | <2 | | ----- | |
| 1880 | | ----- | | ----- | |
| 6134 | | ----- | | ----- | |
| 6198 | D7504 | <2 | | ----- | |
| 6203 | D7504 | 0 | | ----- | |
| 6262 | D7504 | 0 | | ----- | |
| 9008 | | ----- | | ----- | |
| | n | 22 | | | |
| | mean (n) | <10 | | | |

## Determination of Total Impurities on sample #23020; results in mg/kg

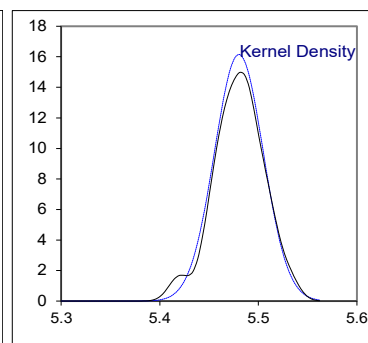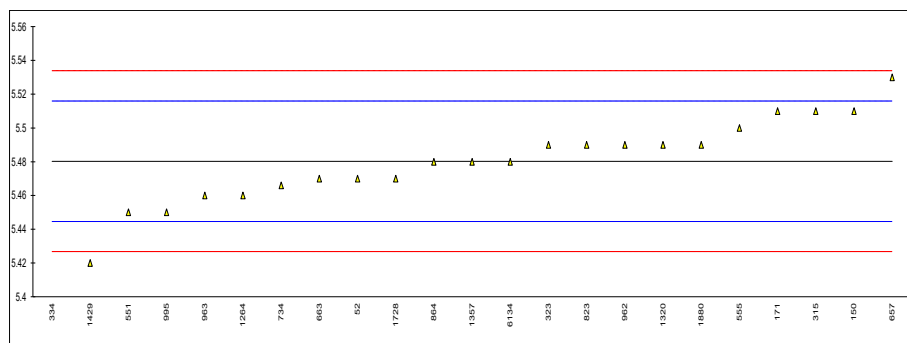| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | D7504 | 200 | | 0.72 | |
| 150 | | ----- | | ----- | |
| 171 | D7504 | 168 | | -0.66 | |
| 315 | D7504 | 200 | | 0.72 | |
| 317 | D7504 | 188 | | 0.21 | |
| 323 | D7504 | 161 | | -0.96 | |
| 334 | D4492 | 140 | | -1.87 | |
| 347 | D4492 | 157 | | -1.13 | |
| 444 | D4492 | 189 | | 0.25 | |
| 445 | | ----- | | ----- | |
| 551 | D7504 | 205 | | 0.94 | |
| 555 | | ----- | | ----- | |
| 657 | | ----- | | ----- | |
| 663 | D7504 | 160 | | -1.00 | |
| 734 | D7504 | 181.3 | | -0.08 | |
| 823 | D7504 | 183 | | -0.01 | |
| 852 | | ----- | | ----- | |
| 855 | | ----- | | ----- | |
| 862 | | ----- | | ----- | |
| 864 | D7504 | 191 | | 0.34 | |
| 866 | | ----- | | ----- | |
| 868 | | ----- | | ----- | |
| 870 | | ----- | | ----- | |
| 877 | | ----- | | ----- | |
| 912 | | ----- | | ----- | |
| 913 | | ----- | | ----- | |
| 962 | D7504 | 161 | | -0.96 | |
| 963 | D7504 | 178 | | -0.23 | |
| 970 | | ----- | | ----- | |
| 995 | D7504 | 160 | | -1.00 | |
| 997 | | ----- | | ----- | |
| 1041 | | ----- | | ----- | |
| 1069 | D4492 | 207.5 | | 1.05 | |
| 1081 | | ----- | | ----- | |
| 1117 | D4492 | 204 | C | 0.90 | first reported 0.0204 mg/kg |
| 1264 | D7504 | 185 | | 0.08 | |
| 1320 | D7504 | 178.64 | C | -0.20 | first reported 55.20 |
| 1357 | D7504 | n.a | | ----- | |
| 1429 | | ----- | | ----- | |
| 1530 | D7504 | 181.0 | | -0.10 | |
| 1728 | | ----- | | ----- | |
| 1812 | | 138 | | -1.95 | |
| 1823 | D7504 | 201.29 | | 0.78 | |
| 1880 | | ----- | | ----- | |
| 6134 | | ----- | | ----- | |
| 6198 | D7504 | 215.6 | | 1.40 | |
| 6203 | D7504 | 197 | | 0.59 | |
| 6262 | D7504 | 232 | C | 2.10 | first reported 0.0251 mg/kg |
| 9008 | D7504 | 185 | | 0.08 | |

| | | |
|---|---|---|
| normality | OK | |
| n | 27 | |
| outliers | 0 | |
| mean (n) | 183.23 | |
| st.dev. (n) | 22.430 | |
| R(calc.) | 62.80 | |
| st.dev.(Horwitz 3 comp) | 23.178 | |
| R(Horwitz 3 comp) | 64.90 | |
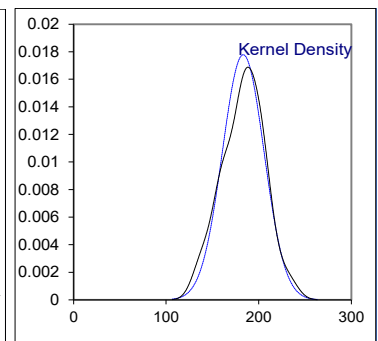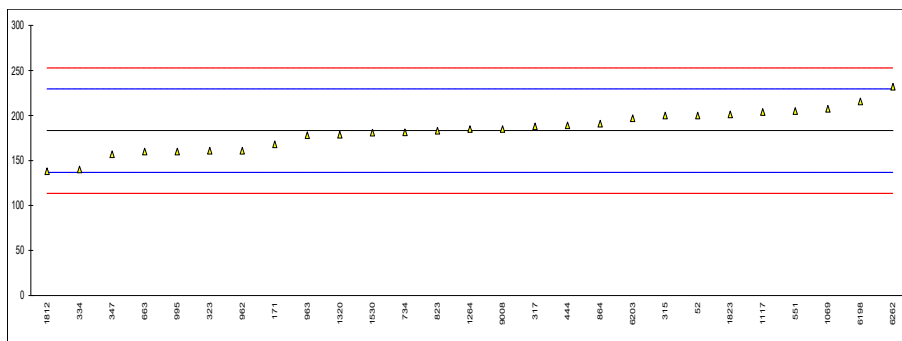
## Determination of Solidification Point (anhydrous basis) on sample #23020; results in °C

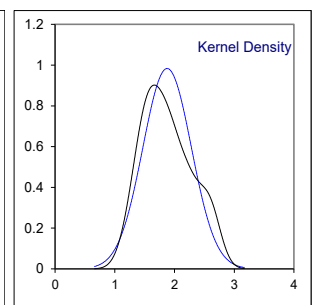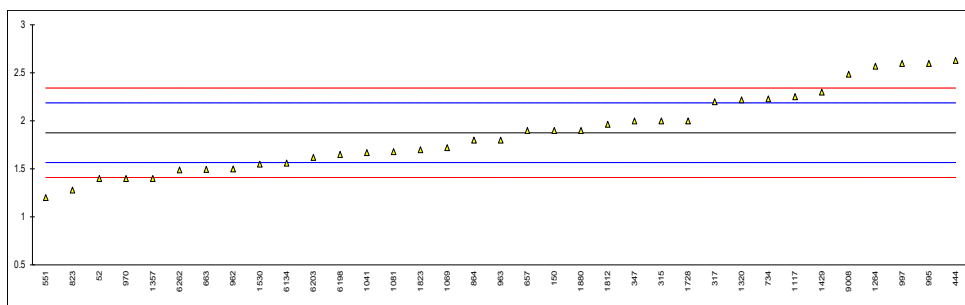| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | D852 | 5.47 | | -0.58 | |
| 150 | D852 | 5.51 | | 1.66 | |
| 171 | D852 | 5.51 | | 1.66 | |
| 315 | D852 | 5.51 | | 1.66 | |
| 317 | | ----- | | ----- | |
| 323 | D852 | 5.49 | | 0.54 | |
| 334 | D852 | 4.38 | R(0.01) | -61.62 | |
| 347 | | ----- | | ----- | |
| 444 | | ----- | | ----- | |
| 445 | | ----- | | ----- | |
| 551 | D852 | 5.45 | | -1.70 | |
| 555 | D852 | 5.50 | | 1.10 | |
| 657 | D852 | 5.53 | | 2.78 | |
| 663 | D852 | 5.47 | | -0.58 | |
| 734 | D852 | 5.466 | | -0.80 | |
| 823 | D852 | 5.49 | | 0.54 | |
| 852 | | ----- | | ----- | |
| 855 | | ----- | | ----- | |
| 862 | | ----- | | ----- | |
| 864 | D852 | 5.48 | | -0.02 | |
| 866 | | ----- | | ----- | |
| 868 | | ----- | | ----- | |
| 870 | | ----- | | ----- | |
| 877 | | ----- | | ----- | |
| 912 | | ----- | | ----- | |
| 913 | | ----- | | ----- | |
| 962 | D852 | 5.49 | | 0.54 | |
| 963 | D852 | 5.46 | | -1.14 | |
| 970 | | ----- | | ----- | |
| 995 | D852 | 5.45 | | -1.70 | |
| 997 | | ----- | | ----- | |
| 1041 | | ----- | | ----- | |
| 1069 | | ----- | | ----- | |
| 1081 | | ----- | | ----- | |
| 1117 | | ----- | | ----- | |
| 1264 | D852 | 5.46 | | -1.14 | |
| 1320 | D852 | 5.49 | | 0.54 | |
| 1357 | D852 | 5.48 | | -0.02 | |
| 1429 | D852 | 5.42 | | -3.38 | |
| 1530 | | ----- | | ----- | |
| 1728 | D852 | 5.47 | | -0.58 | |
| 1812 | | ----- | | ----- | |
| 1823 | | ----- | | ----- | |
| 1880 | D852 | 5.49 | | 0.54 | |
| 6134 | D852 | 5.48 | | -0.02 | |
| 6198 | | ----- | | ----- | |
| 6203 | | ----- | | ----- | |
| 6262 | | ----- | W | ----- | test result withdrawn, reported 5.4 |
| 9008 | | ----- | | ----- | |

| | | |
|---|---|---|
| normality | OK | |
| n | 22 | |
| outliers | 1 | |
| mean (n) | 5.480 | |
| st.dev. (n) | 0.0247 | |
| R(calc.) | 0.069 | |
| st.dev.(D852:20) | 0.0179 | |
| R(D852:20) | 0.05 | |

## Determination of Sulfur on sample #23020; results in mg/kg

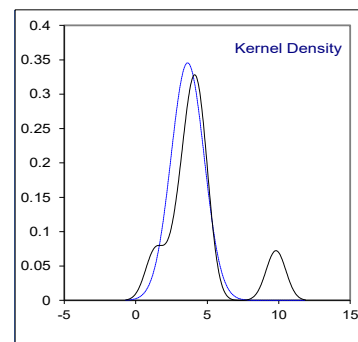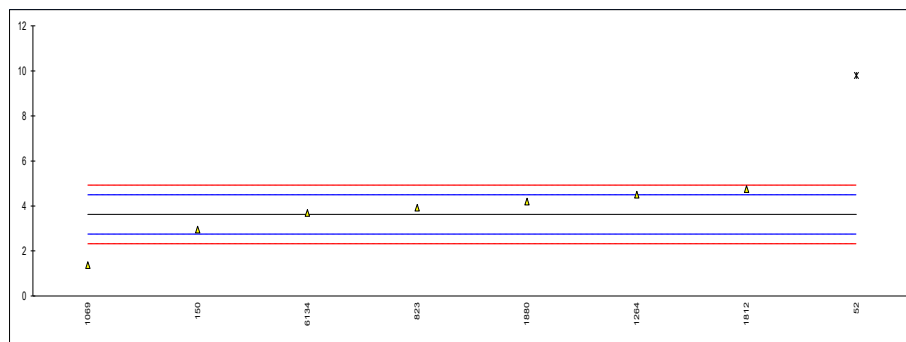| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | D7183 | 1.4 | | -3.06 | |
| 150 | D7183 | 1.9 | | 0.15 | |
| 171 | | ----- | | ----- | |
| 315 | D7183 | 2.0 | | 0.79 | |
| 317 | D5453 | 2.2 | | 2.08 | |
| 323 | D5453 | <1 | | <-5.63 | Possibly a false negative test result? |
| 334 | | ----- | | ----- | |
| 347 | D5453 | 2 | | 0.79 | |
| 444 | D5453 | 2.63 | | 4.84 | |
| 445 | | ----- | | ----- | |
| 551 | D5453 | 1.2 | | -4.35 | |
| 555 | | ----- | | ----- | |
| 657 | D5453 | 1.9 | | 0.15 | |
| 663 | D5453 | 1.495 | | -2.45 | |
| 734 | D5453 | 2.23 | | 2.27 | |
| 823 | D7183 | 1.28 | | -3.83 | |
| 852 | | ----- | | ----- | |
| 855 | | ----- | | ----- | |
| 862 | | ----- | | ----- | |
| 864 | D7183 | 1.8 | | -0.49 | |
| 866 | | ----- | | ----- | |
| 868 | | ----- | | ----- | |
| 870 | | ----- | | ----- | |
| 877 | | ----- | | ----- | |
| 912 | | ----- | | ----- | |
| 913 | | ----- | | ----- | |
| 962 | D7183 | 1.5 | | -2.42 | |
| 963 | D7183 | 1.8 | | -0.49 | |
| 970 | D5453 | 1.4 | | -3.06 | |
| 995 | D7183 | 2.6 | | 4.65 | |
| 997 | D7183 | 2.6 | | 4.65 | |
| 1041 | D5453 | 1.67 | | -1.33 | |
| 1069 | D7183 | 1.722 | | -0.99 | |
| 1081 | D7183 | 1.68 | | -1.26 | |
| 1117 | D5453 | 2.252 | | 2.41 | |
| 1264 | D5453 | 2.57 | | 4.46 | |
| 1320 | ISO20846 | 2.22 | | 2.21 | |
| 1357 | D5453 | 1.4 | | -3.06 | |
| 1429 | D5453 | 2.3 | | 2.72 | |
| 1530 | D5453 | 1.55 | | -2.10 | |
| 1728 | D5453 | 2 | | 0.79 | |
| 1812 | ISO20846 | 1.965 | | 0.57 | |
| 1823 | D5453 | 1.70 | | -1.13 | |
| 1880 | D5453 | 1.9 | | 0.15 | |
| 6134 | D7183 | 1.56 | | -2.03 | |
| 6198 | D5453 | 1.65 | | -1.45 | |
| 6203 | D5453 | 1.62 | | -1.65 | |
| 6262 | D5453 | 1.49 | | -2.48 | |
| 9008 | D7183 | 2.4866 | | 3.92 | |

|  |  |  | D7183 only |
|---|---|---|---|
| normality | OK | | OK |
| n | 35 | | 13 |
| outliers | 0 | | 0 |
| mean (n) | 1.876 | | 1.8714 |
| st.dev. (n) | 0.4056 | | 0.4410 |
| R(calc.) | 1.136 | | 1.2347 |
| st.dev.(D7183:18aR23) | 0.1556 | | 0.1553 |
| R(D7183:18aR23) | 0.436 | | 0.435 |
| Compare | | | |
| R(D5453:19) | 0.929 | | |

## Determination of Thiophene on sample #23020; results in mg/kg

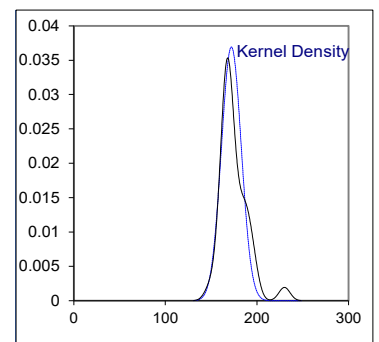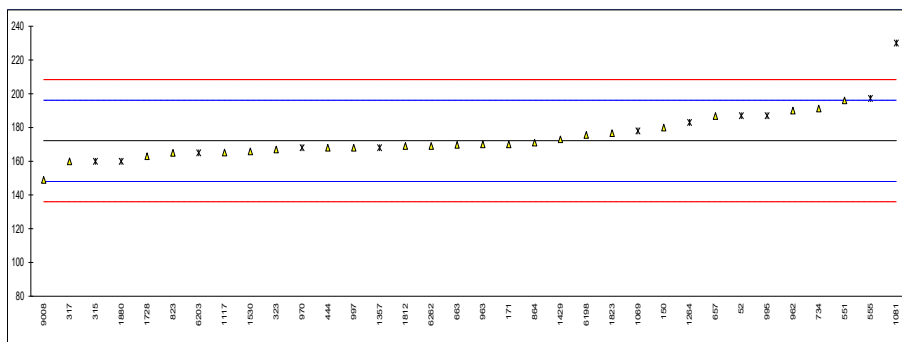| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | D4735 | 9.8 | C,G(0.05) | 14.21 | first reported <0.80 |
| 150 | | 2.95 | C | -1.56 | first reported <0.80 |
| 171 | D7011 | <0.1 | | <-8.12 | possibly a false negative test result? |
| 315 | | ----- | | ----- | |
| 317 | | ----- | | ----- | |
| 323 | INH-306 | <0.1 | | <-8.12 | possibly a false negative test result? |
| 334 | | ----- | | ----- | |
| 347 | | ----- | | ----- | |
| 444 | | ----- | | ----- | |
| 445 | | ----- | | ----- | |
| 551 | | ----- | | ----- | |
| 555 | | ----- | | ----- | |
| 657 | | ----- | | ----- | |
| 663 | | ----- | | ----- | |
| 734 | | ----- | | ----- | |
| 823 | D4735 | 3.92 | | 0.68 | |
| 852 | | ----- | | ----- | |
| 855 | | ----- | | ----- | |
| 862 | | ----- | | ----- | |
| 864 | | ----- | | ----- | |
| 866 | | ----- | | ----- | |
| 868 | | ----- | | ----- | |
| 870 | | ----- | | ----- | |
| 877 | | ----- | | ----- | |
| 912 | | ----- | | ----- | |
| 913 | | ----- | | ----- | |
| 962 | | ----- | | ----- | |
| 963 | | ----- | | ----- | |
| 970 | | ----- | | ----- | |
| 995 | | ----- | | ----- | |
| 997 | | ----- | | ----- | |
| 1041 | | ----- | | ----- | |
| 1069 | In house | 1.37 | | -5.19 | |
| 1081 | | ----- | | ----- | |
| 1117 | | ----- | | ----- | |
| 1264 | D7011 | 4.5 | | 2.01 | |
| 1320 | | ----- | | ----- | |
| 1357 | D7011 | >1.0 | C | ----- | first reported <1.0 |
| 1429 | | ----- | | ----- | |
| 1530 | | ----- | | ----- | |
| 1728 | | ----- | | ----- | |
| 1812 | | 4.75 | | 2.59 | |
| 1823 | | ----- | | ----- | |
| 1880 | D4735 | 4.2 | | 1.32 | |
| 6134 | D7011 | 3.69 | | 0.15 | |
| 6198 | | ----- | | ----- | |
| 6203 | | ----- | | ----- | |
| 6262 | | ----- | | ----- | |
| 9008 | | ----- | | ----- | |

| | |
|---|---|
| normality | unknown |
| n | 7 |
| outliers | 1 |
| mean (n) | 3.626 |
| st.dev. (n) | 1.1543 |
| R(calc.) | 3.232 |
| st.dev.(D7011:15R19) | 0.4344 |
| R(D7011:15R19) | 1.216 |

## Determination of Water on sample #23020; results in mg/kg

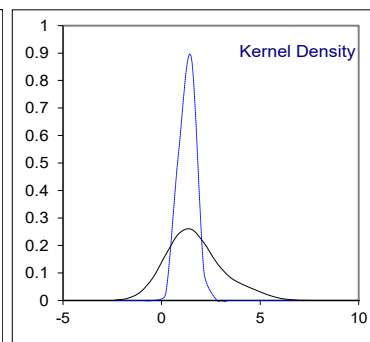| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | D6304 | 187 | C | 1.41 | first reported 230 |
| 150 | E1064 | 180 | | 0.70 | |
| 171 | E1064 | 170 | | -0.31 | |
| 315 | D7375 | 160 | | -1.33 | |
| 317 | E1064 | 160 | | -1.33 | |
| 323 | E1064 | 167 | | -0.62 | |
| 334 | | ----- | | ----- | |
| 347 | | ----- | | ----- | |
| 444 | E1064 | 168 | | -0.52 | |
| 445 | | ----- | | ----- | |
| 551 | E1064 | 196 | | 2.33 | |
| 555 | D4017 | 197.219 | | 2.45 | |
| 657 | E1064 | 186.8 | | 1.39 | |
| 663 | E1064 | 169.7 | | -0.35 | |
| 734 | E1064 | 191.2 | | 1.84 | |
| 823 | E1064 | 165 | | -0.82 | |
| 852 | | ----- | | ----- | |
| 855 | | ----- | | ----- | |
| 862 | | ----- | | ----- | |
| 864 | E1064 | 171 | | -0.21 | |
| 866 | | ----- | | ----- | |
| 868 | | ----- | | ----- | |
| 870 | | ----- | | ----- | |
| 877 | | ----- | | ----- | |
| 912 | | ----- | | ----- | |
| 913 | | ----- | | ----- | |
| 962 | E1064 | 190 | | 1.72 | |
| 963 | E1064 | 170 | | -0.31 | |
| 970 | D6304 | 168 | | -0.52 | |
| 995 | D6304 | 187 | | 1.41 | |
| 997 | E1064 | 168 | | -0.52 | |
| 1041 | | ----- | | ----- | |
| 1069 | D1364 | 178 | | 0.50 | |
| 1081 | D6304 | 230 | R(0.01) | 5.79 | |
| 1117 | E1064 | 165.2 | | -0.80 | |
| 1264 | D6304 | 183 | | 1.01 | |
| 1320 | | ----- | | ----- | |
| 1357 | D6304 | 168 | | -0.52 | |
| 1429 | E1064 | 173 | | -0.01 | |
| 1530 | E1064 | 165.8 | | -0.74 | |
| 1728 | E1064 | 163 | | -1.03 | |
| 1812 | | 169 | | -0.42 | |
| 1823 | E1064 | 176.67 | | 0.36 | |
| 1880 | D6304 | 160 | | -1.33 | |
| 6134 | | ----- | | ----- | |
| 6198 | E1064 | 175.6 | | 0.25 | |
| 6203 | ISO12937 | 165 | | -0.82 | |
| 6262 | E1064 | 169 | | -0.42 | |
| 9008 | E1064 | 148.9 | | -2.46 | |

|  |  |  | E1064 only |
|--|--|--|-----------|
| normality | OK | | OK |
| n | 33 | | 23 |
| outliers | 1 | | 0 |
| mean (n) | 173.1 | | 172.1 |
| st.dev. (n) | 11.38 | | 10.81 |
| R(calc.) | 31.9 | | 30.3 |
| st.dev.(E1064:16) | 9.83 | | 9.77 |
| R(E1064:16) | 27.5 | | 27.4 |

## Determination of Total Chlorides on sample #23023; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | D7536 | 159.7 | C,G(0.01) | 492.78 | first reported 158.2 |
| 150 | D7359 | 1.7 | | 1.22 | |
| 171 | | ----- | | ----- | |
| 315 | | ----- | | ----- | |
| 317 | | ----- | | ----- | |
| 323 | D7359 | 4 | G(0.05) | 8.38 | |
| 334 | | ----- | | ----- | |
| 347 | | ----- | | ----- | |
| 444 | | ----- | | ----- | |
| 445 | | ----- | | ----- | |
| 551 | | ----- | | ----- | |
| 555 | | ----- | | ----- | |
| 657 | | ----- | | ----- | |
| 663 | | ----- | | ----- | |
| 734 | | ----- | | ----- | |
| 823 | | ----- | | ----- | |
| 852 | | ----- | | ----- | |
| 855 | | ----- | | ----- | |
| 862 | | ----- | | ----- | |
| 864 | | ----- | | ----- | |
| 866 | | ----- | | ----- | |
| 868 | | ----- | | ----- | |
| 870 | | ----- | | ----- | |
| 877 | | ----- | | ----- | |
| 912 | | ----- | | ----- | |
| 913 | | ----- | | ----- | |
| 962 | | ----- | | ----- | |
| 963 | | ----- | | ----- | |
| 970 | | ----- | | ----- | |
| 995 | | ----- | | ----- | |
| 997 | | ----- | | ----- | |
| 1041 | | ----- | | ----- | |
| 1069 | D7359 | 1.0064 | | -0.94 | |
| 1081 | | ----- | | ----- | |
| 1117 | | ----- | | ----- | |
| 1264 | D5194 | 1.6 | C | 0.91 | first reported 346 |
| 1320 | | ----- | | ----- | |
| 1357 | UOP779 | 1.7 | | 1.22 | |
| 1429 | | ----- | | ----- | |
| 1530 | | ----- | | ----- | |
| 1728 | | ----- | | ----- | |
| 1812 | | 0.89 | | -1.30 | |
| 1823 | | ----- | | ----- | |
| 1880 | | ----- | | ----- | |
| 6134 | | ----- | | ----- | |
| 6198 | | ----- | | ----- | |
| 6203 | | ----- | | ----- | |
| 6262 | | ----- | | ----- | |
| 9008 | D5808 | 0.95 | | -1.11 | |

| | | |
|---|---|---|
| normality | unknown | |
| n | 6 | |
| outliers | 2 | |
| mean (n) | 1.308 | |
| st.dev. (n) | 0.3966 | |
| R(calc.) | 1.110 | |
| st.dev.(D5194:18) | 0.3214 | |
| R(D5194:18) | 0.9 | |
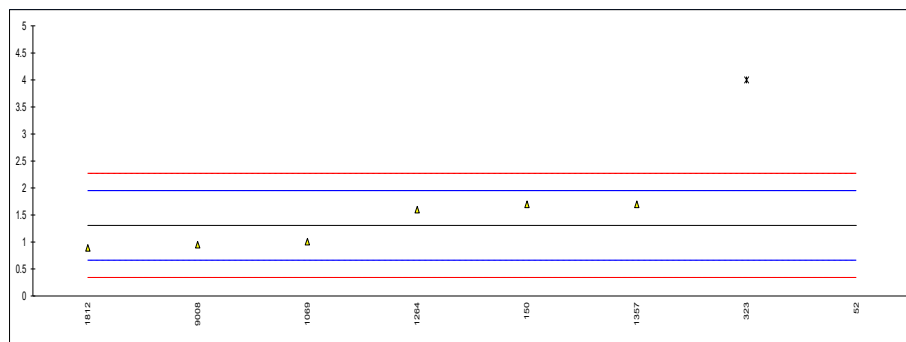
## Determination of Organic Chlorides on sample #23023; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 52 | | ----- | | ----- | |
| 150 | D7359 | 1.7 | | 1.02 | |
| 171 | D5808 | <1.0 | | ----- | |
| 315 | D5808 | 1.1 | | -0.27 | |
| 317 | UOP779 | 1.2 | | -0.06 | |
| 323 | | ----- | | ----- | |
| 334 | | ----- | | ----- | |
| 347 | | ----- | | ----- | |
| 444 | IP510 | 1.3 | | 0.16 | |
| 445 | | ----- | | ----- | |
| 551 | | ----- | | ----- | |
| 555 | | ----- | | ----- | |
| 657 | D5808 | 1.35 | | 0.26 | |
| 663 | D5808 | <0.7 | | ----- | |
| 734 | | ----- | | ----- | |
| 823 | | ----- | | ----- | |
| 852 | | ----- | | ----- | |
| 855 | | ----- | | ----- | |
| 862 | | ----- | | ----- | |
| 864 | | ----- | | ----- | |
| 866 | | ----- | | ----- | |
| 868 | | ----- | | ----- | |
| 870 | | ----- | | ----- | |
| 877 | | ----- | | ----- | |
| 912 | | ----- | | ----- | |
| 913 | | ----- | | ----- | |
| 962 | D5808 | 1.5 | | 0.59 | |
| 963 | D5808 | 1.9 | | 1.45 | |
| 970 | | ----- | | ----- | |
| 995 | | ----- | | ----- | |
| 997 | | ----- | | ----- | |
| 1041 | | ----- | | ----- | |
| 1069 | | ----- | | ----- | |
| 1081 | D5808 | <0.5 | | ----- | |
| 1117 | D7359 | 0.817 | | -0.88 | |
| 1264 | D5808 | 0.78 | | -0.96 | |
| 1320 | EN14077 | 1.01 | | -0.47 | |
| 1357 | D5808 | n.a | | ----- | |
| 1429 | | ----- | | ----- | |
| 1530 | | ----- | | ----- | |
| 1728 | | ----- | | ----- | |
| 1812 | | ----- | | ----- | |
| 1823 | | ----- | | ----- | |
| 1880 | D5808 | 0.95 | | -0.60 | |
| 6134 | D4929 | 1.595 | | 0.79 | |
| 6198 | D5808 | 0.75 | | -1.03 | |
| 6203 | D5808 | <0.7 | | ----- | |
| 6262 | | ----- | | ----- | |
| 9008 | | ----- | | ----- | |

| | | |
|---|---|---|
| normality | OK | |
| n | 13 | |
| outliers | 0 | |
| mean (n) | 1.227 | |
| st.dev. (n) | 0.3700 | |
| R(calc.) | 1.036 | |
| st.dev.(D5808:20) | 0.4643 | |
| R(D5808:20) | 1.3 | |
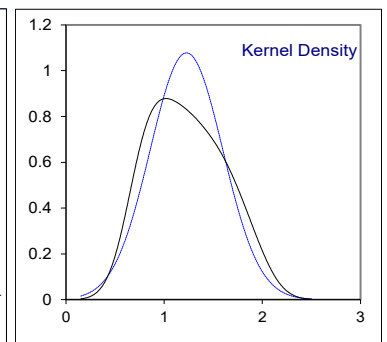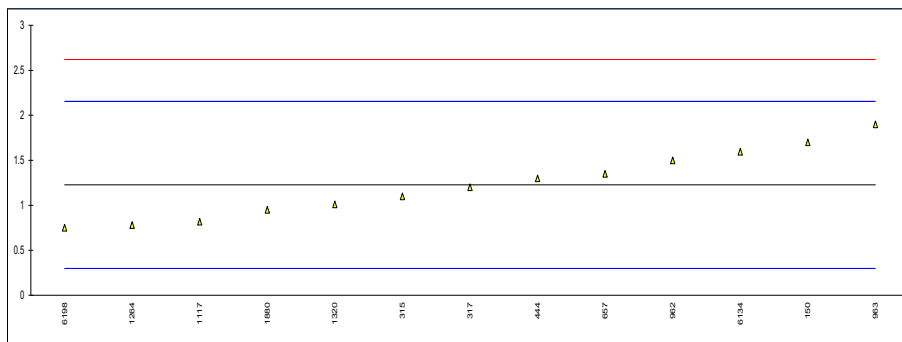
**APPENDIX 2**

**Number of participants per country**

  2 labs in BELGIUM
  2 labs in BRAZIL
  1 lab in CANADA
 10 labs in CHINA, People's Republic
  1 lab in FINLAND
  1 lab in FRANCE
  2 labs in GEORGIA
  3 labs in GERMANY
  3 labs in INDIA
  1 lab in KAZAKHSTAN
  1 lab in KOREA, Republic of
  2 labs in KUWAIT
  4 labs in NETHERLANDS
  2 labs in OMAN
  2 labs in ROMANIA
  3 labs in SAUDI ARABIA
  1 lab in SINGAPORE
  1 lab in SLOVAKIA
  1 lab in SPAIN
  1 lab in THAILAND
  3 labs in UNITED KINGDOM
  2 labs in UNITED STATES OF AMERICA

**APPENDIX 3**

**Abbreviations**

| | |
|---|---|
| C | = final test result after checking of first reported suspect test result |
| D(0.01) | = outlier in Dixon's outlier test |
| D(0.05) | = straggler in Dixon's outlier test |
| G(0.01) | = outlier in Grubbs' outlier test |
| G(0.05) | = straggler in Grubbs' outlier test |
| DG(0.01) | = outlier in Double Grubbs' outlier test |
| DG(0.05) | = straggler in Double Grubbs' outlier test |
| R(0.01) | = outlier in Rosner's outlier test |
| R(0.05) | = straggler in Rosner's outlier test |
| E | = calculation difference between reported test result and result calculated by iis |
| W | = test result withdrawn on request of participant |
| ex | = test result excluded from statistical evaluation |
| n.a. | = not applicable |
| n.e. | = not evaluated |
| n.d. | = not detected |
| fr. | = first reported |
| f+? | = possibly a false positive test result? |
| f-? | = possibly a false negative test result? |
| SDS | = Safety Data Sheet |

**Literature**

1      iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018

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3      ISO5725 parts 1-6:94

4      ISO13528:05

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6      W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)

7      P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)

8      J.N. Miller, Analyst, 118, 455, (1993)

9      Analytical Methods Committee, Technical Brief, No 4, January 2001

10     P.J. Lowthian and M. Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)

11     W. Horwitz and R. Albert, J. AOAC Int, 79.3, 589-621, (1996)

12     Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)