# Results of Proficiency Test
# Isopropanol
# (Isopropyl alcohol)
# December 2019

**CONTENTS**

Appendices:

# 1      INTRODUCTION

Since 2003, the Institute for Interlaboratory Studies (iis) organizes a proficiency test for the analyzes of Isopropanol once every two years. During the annual program 2019/2020 it was decided to continue the round robin for the analyzes of Isopropanol according to the latest version of specification ASTM D770.

In this interlaboratory study, 16 laboratories in 10 different countries registered for participation. See appendix 2 for the number of participants per country.
In this report the results of the 2019 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 one 0.5 L glass bottle with Isopropanol labelled #19260 to the participants. Participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

## 2.1   QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. 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

A batch of approximately 15 liters of Isopropanol were obtained from a local chemical supplier and was spiked with NaCl and with Ethanol. After homogenization 30 amber glass bottles of 0.5 L were filled and labelled #19260. The homogeneity of the subsamples #19260 was checked by determination of the Density in accordance with ISO12185 on 8 stratified randomly selected samples.

|  | Density at 20°C in kg/L |
|---|---|
| sample #19260-1 | 0.78504 |
| sample #19260-2 | 0.78504 |
| sample #19260-3 | 0.78504 |
| sample #19260-4 | 0.78504 |
| sample #19260-5 | 0.78504 |
| sample #19260-6 | 0.78504 |
| sample #19260-7 | 0.78504 |
| sample #19260-8 | 0.78505 |

Table 1: homogeneity test results of subsamples #19260

From the above test results the repeatability was calculated and compared with 0.3 times the corresponding 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) | 0.00001 |
| reference test method | ISO12185:96 |
| 0.3 * R (ref. test method) | 0.00015 |

Table 2: evaluation of the repeatability of subsamples #19260

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

To each of the participating laboratories one 0.5-liter bottle labelled #19260 was sent on November 6, 2019. An SDS was added to the sample package.

## 2.5 STABILITY OF THE SAMPLES

The stability of Isopropanol packed in an amber glass bottle 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 #19260: Acidity as Acetic acid, Appearance, Inorganic Chloride, Color Pt/Co, Density at 20°C, Specific Gravity at 20/20°C, Distillation at 760 mmHg (IBP, 50% recovered & DP), Nonvolatile matter, Purity by GC on dry basis, Ethanol, n-Propanol, n-Butanol and Other Impurities, Water.

It was explicitly requested to treat the sample as if it was a routine sample 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 calculations.

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

## 3 RESULTS

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

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

## 3.1 STATISTICS

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

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

According to ISO5725 the original test results per determination were submitted to Dixon's, Grubbs' and/or Rosner's outlier tests. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, by G(0.05) or DG(0.05) for the Grubbs' test and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

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

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

## 3.2 GRAPHICS

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

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

## 3.3 Z-SCORES

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

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used. In some cases, a reproducibility based on former iis proficiency tests could be used.

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

The z-scores were calculated according to:

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

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

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

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

## 4    EVALUATION

In this proficiency test no problems were encountered with dispatch of the samples.
One participant did not receive the sample in time due to problems with custom clearance and could not report test results on time. Finally, 15 reporting laboratories submitted 148 numerical test results. Observed were 3 outlying test results, which is 2.0 %. In proficiency studies outlier percentages of 3 % - 7.5 % are quite normal.

### 4.1   EVALUATION PER TEST

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

In the iis PT reports, ASTM methods are referred to with a number (e.g. D1209) and an added designation for the year that the method was adopted or revised (e.g. D1209:05). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D1209:05(2019)). In the results tables of appendix 1 only the method number and year of adoption or revision e.g. D1209:05 are used.

Unfortunately, a suitable reference test method providing the precision data is not available for all determinations. For the tests that have no available precision data the calculated reproducibility was compared against the reproducibility estimated from the Horwitz equation.

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

Acidity as Acetic Acid: 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 D1613:17.

Appearance:          No analytical problems were observed. All reporting participants agreed about the appearance of sample #19260 to be 'clear and bright' or 'pass'.

Chloride, inorganic: This determination may be problematic. No statistical outliers were observed, but one test result was excluded. However, the calculated reproducibility after rejection of the suspect data is not in agreement with the requirements of IMPCA002:98.

Color Pt/Co:         This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D1209:05(2019).

Density at 20°C:     This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ISO12185:96.

Specific Gravity at 20/20°C: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ISO12185:96.

Distillation at 760 mmHg: This determination was not problematic. No statistical outliers were observed. All three calculated reproducibilities are in good agreement with the requirements of ASTM D1078:11 for the automated and the manual modes.

Nonvolatile matter: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D1353:13.

Purity on dry basis: Regretfully, the methods used do not provide any reproducibility limit. Therefore, no z-scores were calculated. No statistical outliers were observed. In comparison with the previous proficiency test (iis17C18) of December 2017, the calculated reproducibility of the 2019 PT is small.

Ethanol:             This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the estimated reproducibility using the Horwitz equation.

n-Propanol: This determination may be problematic. No statistical outliers were observed, but one test result was excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the estimated reproducibility using the Horwitz equation.

n-Butanol: All reported test results were near or below the detection limit. Therefore, no z-scores were calculated.

Other impurities: This determination may be problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the estimated reproducibility calculated using the Horwitz equation for 4 components.

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.

## 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

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

| Parameter | unit | n | average | 2.8 * sd | R (lit) |
|---|---|---|---|---|---|
| Acidity as Acetic Acid | mg/kg | 13 | 8.0 | 8.1 | 14 |
| Appearance | | 14 | pass | n.e. | n.e. |
| Chloride as Cl, Inorganic | mg/kg | 4 | 1.5 | 0.6 | 0.3 |
| Color Pt/Co | | 9 | 1.8 | 4.4 | 7 |
| Density at 20°C | kg/L | 14 | 0.7850 | 0.0002 | 0.0005 |
| Specific Gravity at 20/20°C | | 14 | 0.7865 | 0.0002 | 0.0005 |
| Initial Boiling Point | °C | 12 | 82.2 | 0.2 | 1.3 |
| 50% recovered | °C | 12 | 82.3 | 0.3 | 0.6 |
| Dry Point | °C | 12 | 82.4 | 0.4 | 0.9 |
| Nonvolatile matter | mg/100mL | 7 | 0.6 | 0.6 | 2.1 |
| Purity by GC on dry basis | %M/M | 12 | 99.974 | 0.014 | n.a. |
| Ethanol | mg/kg | 8 | 137 | 20 | 29 |
| n-Propanol | mg/kg | 6 | 8.0 | 5.6 | 2.6 |
| n-Butanol | mg/kg | 8 | <10 | n.e. | n.e. |
| Other impurities | mg/kg | 6 | 103 | 57 | 46 |
| Water | mg/kg | 11 | 196 | 61 | 31 |

Table 3: performance evaluation sample #19260

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

**4.3    COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2019 WITH THE PREVIOUS PTS.**

|  | December 2019 | December 2017 | December 2015 | November 2013 | November 2011 |
|---|---|---|---|---|---|
| Number of reporting labs | 15 | 17 | 17 | 16 | 13 |
| Number of results reported | 148 | 157 | 192 | 168 | 143 |
| Number of statistical outliers | 3 | 5 | 8 | 7 | 10 |
| Percentage outliers | 2.0% | 3.2% | 4.2% | 4.2% | 7.0% |

Table 4: comparison with previous proficiency tests

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

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

| Determination | December 2019 | December 2017 | December 2015 | November 2013 | November 2011 |
|---|---|---|---|---|---|
| Acidity as Acetic Acid | + | ++ | ++ | ++ | ++ |
| Chloride as Cl, Inorganic | -- | ++ | n.e. | n.e. | n.e. |
| Color Pt/Co | + | ++ | ++ | ++ | ++ |
| Density at 20°C | ++ | ++ | ++ | + | ++ |
| Specific Gravity at 20/20°C | ++ | ++ | ++ | + | ++ |
| Distillation at 760 mmHg | ++ | ++ | ++ | ++ | ++ |
| Nonvolatile matter | ++ | ++ | ++ | ++ | ++ |
| Purity by GC on dry basis | n.e. | n.e. | n.e. | (++) | (++) |
| Ethanol | + | - | +/- | n.e. | - |
| n-Propanol | -- | - | + | - | +/- |
| n-Butanol | n.e. | n.e. | n.e. | n.e. | n.e. |
| Other impurities | - | n.e. | -- | -- | + |
| Water | - | - | +/- | +/- | -- |

Table 5: comparison determinations against the reference test methods

Results between brackets are compared with reproducibility of the previous round robin, due to the lack of target data.

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 Acidity as Acetic Acid on sample #19260; results in mg/kg.

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 150 | D1613 | 8 | | 0.00 | |
| 171 | D1613 | 8 | C | 0.00 | First reported 0.0008 mg/kg |
| 173 | D1613 | 8 | | 0.00 | |
| 311 | D1613 | 8 | | 0.00 | |
| 323 | D1613 | 7 | | -0.20 | |
| 343 | D1613 | 8 | | 0.00 | |
| 445 | D1613 | 3 | | -1.00 | |
| 446 | D1613 | 7.6 | | -0.08 | |
| 551 | | ----- | | ----- | |
| 902 | D1613 | 8.6 | | 0.12 | |
| 913 | D1613 | 4.0 | | -0.80 | |
| 994 | D1613 | 15 | | 1.40 | |
| 1016 | D1613 | 7.6 | C | -0.08 | Reported 0.00076 mg/kg |
| 1438 | | ----- | | ----- | |
| 6201 | D1613 | 11 | | 0.60 | |
| 6262 | D1613 | 31 | G(0.01) | 4.60 | |

| | |
|---|---|
| normality | not OK |
| n | 13 |
| outliers | 1 |
| mean (n) | 7.98 |
| st.dev. (n) | 2.897 |
| R(calc.) | 8.11 |
| st.dev.(D1613:17) | 5 |
| R(D1613:17) | 14 |





Kernel Density
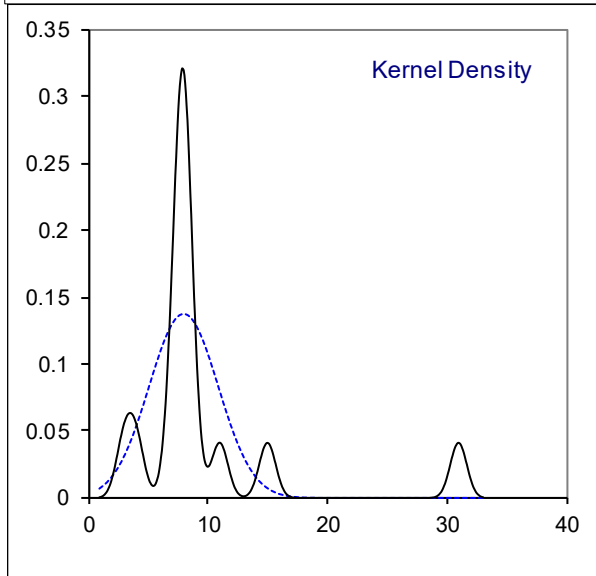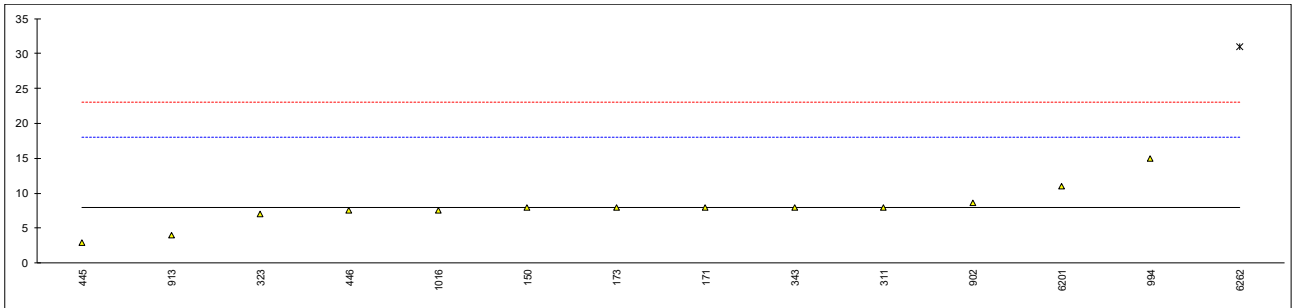
## Determination of Appearance on sample #19260;

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 150 | E2680 | Pass | | ----- | |
| 171 | E2680 | Pass | | ----- | |
| 173 | E2680 | Pass | | ----- | |
| 311 | E2680 | pass | | ----- | |
| 323 | D4176 | clear & bright liquid | | ----- | |
| 343 | E2680 | PASS | | ----- | |
| 445 | E2680 | Pass | | ----- | |
| 446 | E2680 | PASS | | ----- | |
| 551 | | ----- | | ----- | |
| 902 | E2680 | PASS | | ----- | |
| 913 | E2680 | Pass | | ----- | |
| 994 | E2680 | pass | | ----- | |
| 1016 | Visual | Pass | | ----- | |
| 1438 | | ----- | | ----- | |
| 6201 | Visual | B&C | | ----- | |
| 6262 | Visual | Bright and clear | | ----- | |

|  |  |
|--|--|
| n | 14 |
| mean (n) | Pass (bright&clear) |

## Determination of Chloride as Cl, Inorganic on sample #19260; results in mg/kg.

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 150 | IMPCA002 | 1.5 | | 0.23 | |
| 171 | IMPCA002 | 1.7 | | 2.10 | |
| 173 | | ----- | | ----- | |
| 311 | INH-158 | 1.5 | | 0.23 | |
| 323 | E2469 | 1 | ex | -4.43 | Test result excluded, rounded too much |
| 343 | | ----- | | ----- | |
| 445 | | ----- | | ----- | |
| 446 | | ----- | | ----- | |
| 551 | | ----- | | ----- | |
| 902 | | ----- | | ----- | |
| 913 | | ----- | | ----- | |
| 994 | | ----- | | ----- | |
| 1016 | | ----- | | ----- | |
| 1438 | | ----- | | ----- | |
| 6201 | IMPCA002 | 1.2 | | -2.57 | |
| 6262 | | ----- | | ----- | |

|  |  |
|--|--|
| normality | unknown |
| n | 4 |
| outliers | 0 (+1ex) |
| mean (n) | 1.47 |
| st.dev. (n) | 0.206 |
| R(calc.) | 0.58 |
| st.dev.(IMPCA002:98) | 0.107 |
| R(IMPCA002:98) | 0.3 |
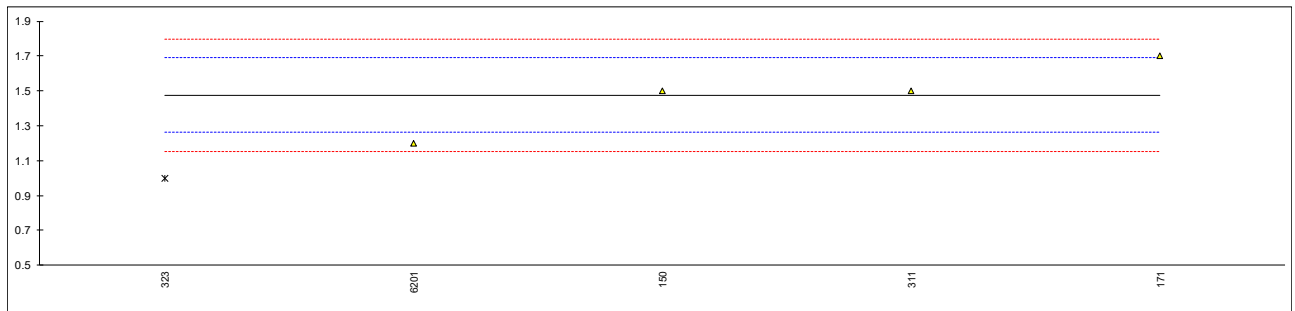
## Determination of Color Pt/Co on sample #19260;

| lab | method | value | mark | z(targ) | remarks |
|-----:|--------|-------|------|---------|---------|
| 150 | D5386 | 2 | | 0.09 | |
| 171 | D1209 | 5 | | 1.29 | |
| 173 | D5386 | 3 | | 0.49 | |
| 311 | D1209 | <5 | | ----- | |
| 323 | D1209 | <5 | | ----- | |
| 343 | D5386 | 2 | | 0.09 | |
| 445 | D5386 | 0 | | -0.71 | |
| 446 | D1209 | <5 | | ----- | |
| 551 | | ----- | | ----- | |
| 902 | D5386 | 2 | | 0.09 | |
| 913 | D5386 | 1 | | -0.31 | |
| 994 | D1209 | <5 | | ----- | |
| 1016 | D1209 | 1 | | -0.31 | |
| 1438 | | ----- | | ----- | |
| 6201 | D1209 | <5 | | ----- | |
| 6262 | D1209 | 0 | | -0.71 | |

|  |  |
|---|---|
| normality | suspect |
| n | 9 |
| outliers | 0 |
| mean (n) | 1.78 |
| st.dev. (n) | 1.563 |
| R(calc.) | 4.38 |
| st.dev.(D1209:05) | 2.5 |
| R(D1209:05) | 7 |





Kernel Density
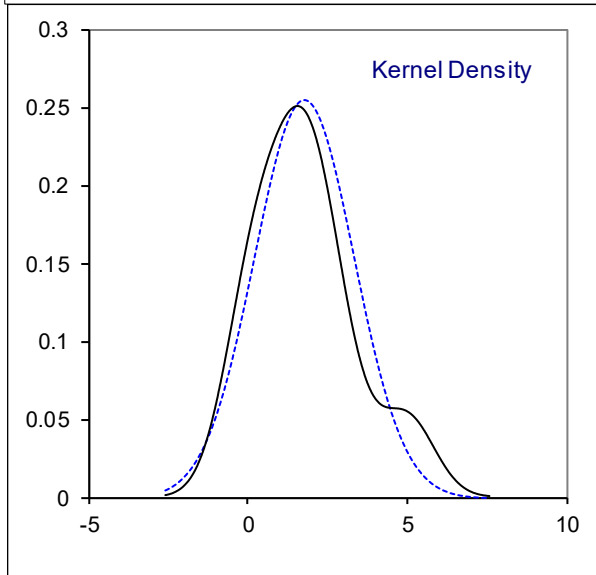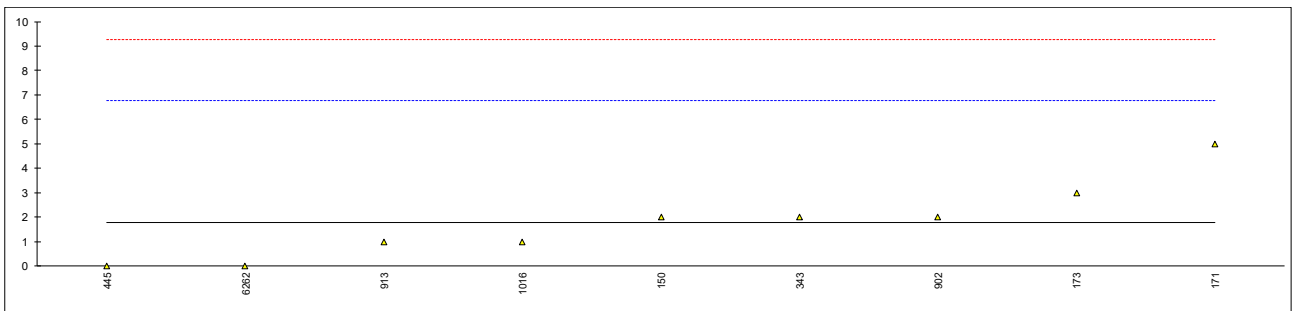
Determination of Density at 20°C on sample #19260; results in kg/L.

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 150 | D4052 | 0.7850 | | -0.22 | |
| 171 | D4052 | 0.7851 | | 0.34 | |
| 173 | D4052 | 0.7850 | | -0.22 | |
| 311 | D4052 | 0.7850 | | -0.22 | |
| 323 | ISO12185 | 0.7849 | | -0.78 | |
| 343 | D4052 | 0.7851 | | 0.34 | |
| 445 | D4052 | 0.7849 | | -0.78 | |
| 446 | D4052 | 0.7851 | | 0.34 | |
| 551 | | ----- | | ----- | |
| 902 | D4052 | 0.78505 | | 0.06 | |
| 913 | D4052 | 0.7850 | | -0.22 | |
| 994 | ISO12185 | 0.7851 | | 0.34 | |
| 1016 | D4052 | 0.7851 | | 0.34 | |
| 1438 | | ----- | | ----- | |
| 6201 | ISO12185 | 0.7851 | | 0.34 | |
| 6262 | ISO12185 | 0.7851 | | 0.34 | |

|  |  |
|--|--|
| normality | OK |
| n | 14 |
| outliers | 0 |
| mean (n) | 0.78504 |
| st.dev. (n) | 0.000078 |
| R(calc.) | 0.00021 |
| st.dev.(ISO12185:96) | 0.000179 |
| R(ISO12185:96) | 0.0005 |





Kernel Density
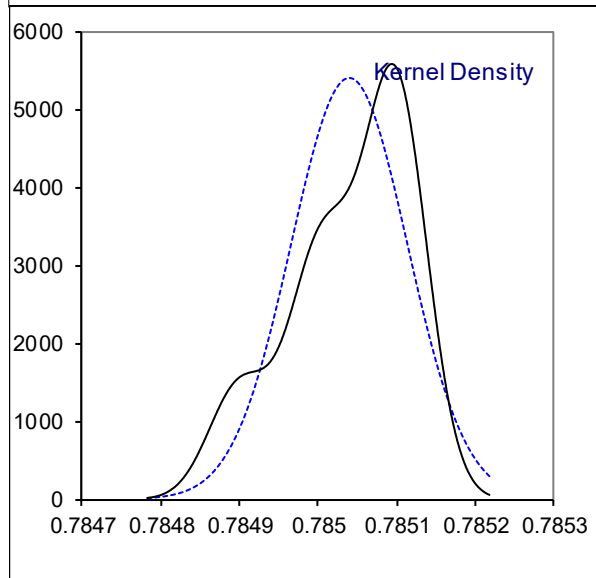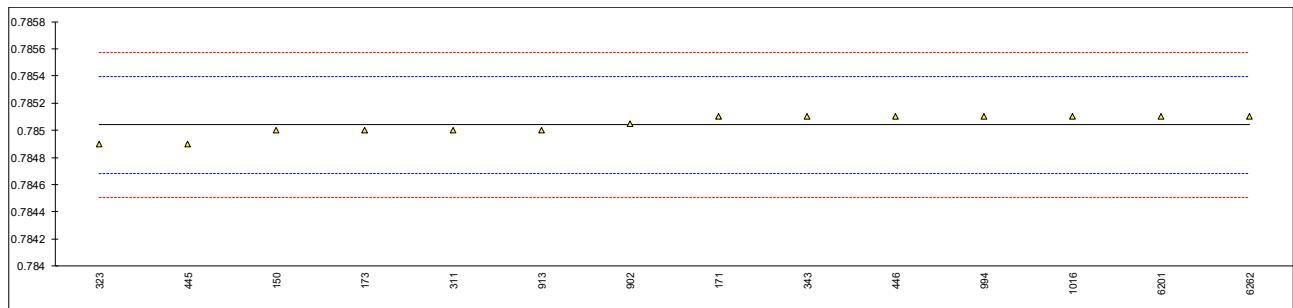
## Determination of Specific Gravity at 20/20°C on sample #19260;

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 150 | D4052 | 0.7864 | | -0.26 | |
| 171 | D4052 | 0.7865 | | 0.30 | |
| 173 | D4052 | 0.7864 | | -0.26 | |
| 311 | D4052 | 0.7864 | | -0.26 | |
| 323 | ISO12185 | 0.7865 | | 0.30 | |
| 343 | D4052 | 0.7865 | | 0.30 | |
| 445 | D4052 | 0.78629 | | -0.88 | |
| 446 | D4052 | 0.7865 | | 0.30 | |
| 551 | | ----- | | ----- | |
| 902 | D4052 | 0.78647 | | 0.13 | |
| 913 | D4052 | 0.7864 | | -0.26 | |
| 994 | ISO12185 | 0.7865 | | 0.30 | |
| 1016 | D4052 | 0.7864 | | -0.26 | |
| 1438 | | ----- | | ----- | |
| 6201 | ISO12185 | 0.7865 | | 0.30 | |
| 6262 | ISO12185 | 0.7865 | | 0.30 | |

|  |  |
|--|--|
| normality | OK |
| n | 14 |
| outliers | 0 |
| mean (n) | 0.78645 |
| st.dev. (n) | 0.000066 |
| R(calc.) | 0.00018 |
| st.dev.(ISO12185:96) | 0.000179 |
| R(ISO12185:96) | 0.0005 |





Kernel Density
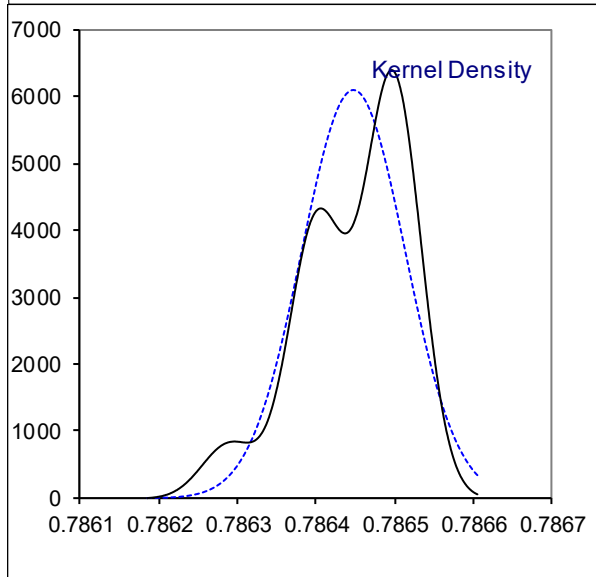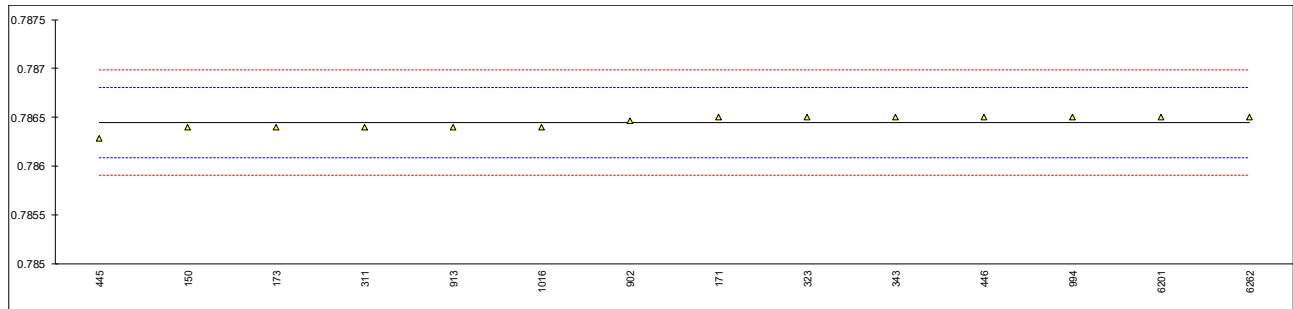
## Determination of Distillation at 760 mmHg on sample #19260; results in °C.

| lab | method | IBP | mark | z(targ) | 50%rec | mark | z(targ) | DP | mark | z(targ) |
|-----|--------|-----|------|---------|--------|------|---------|-----|------|---------|
| 150 | D1078-automated | 82.2 | | 0.05 | 82.3 | | 0.04 | 82.3 | | -0.26 |
| 171 | D1078-automated | 82.2 | | 0.05 | 82.2 | | -0.46 | 82.3 | | -0.26 |
| 173 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 311 | D1078-automated | 82.2 | | 0.05 | 82.3 | | 0.04 | 82.6 | | 0.69 |
| 323 | D1078-manual | 82.2 | | 0.05 | 82.3 | | 0.04 | 82.4 | | 0.05 |
| 343 | D1078-automated | 82.3 | | 0.27 | 82.4 | | 0.54 | 82.4 | | 0.05 |
| 445 | D1078-automated | 82.2 | | 0.05 | 82.3 | | 0.04 | 82.3 | | -0.26 |
| 446 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 551 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 902 | D1078-automated | 82.1 | | -0.16 | 82.3 | | 0.04 | 82.3 | | -0.26 |
| 913 | D1078-manual | 82.2 | | 0.05 | 82.3 | | 0.04 | 82.5 | | 0.37 |
| 994 | D1078-manual | 82.2 | | 0.05 | 82.4 | | 0.54 | 82.6 | | 0.69 |
| 1016 | | 82.0 | | -0.38 | 82.1 | | -0.95 | 82.2 | | -0.58 |
| 1438 | | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 6201 | D1078-manual | 82.2 | | 0.05 | 82.4 | | 0.54 | 82.5 | | 0.37 |
| 6262 | D1078-automated | 82.1 | | -0.16 | 82.2 | | -0.46 | 82.2 | | -0.58 |

|  |  |  |  |  |
|--|--|--|--|--|
| | normality | not OK | OK | OK |
| | n | 12 | 12 | 12 |
| | outliers | 0 | 0 | 0 |
| | mean (n) | 82.18 | 82.29 | 82.38 |
| | st.dev. (n) | 0.075 | 0.090 | 0.140 |
| | R(calc.) | 0.21 | 0.25 | 0.39 |
| | st.dev.(D1078-A:11) | 0.458 | 0.201 | 0.315 |
| | R(D1078-A:11) | 1.28 | 0.56 | 0.88 |
| Compare | | | | |
| | R(D1078-M:11) | 0.88 | 0.53 | 1.07 |



Initial Boiling Point



50% recovered



Dry Point
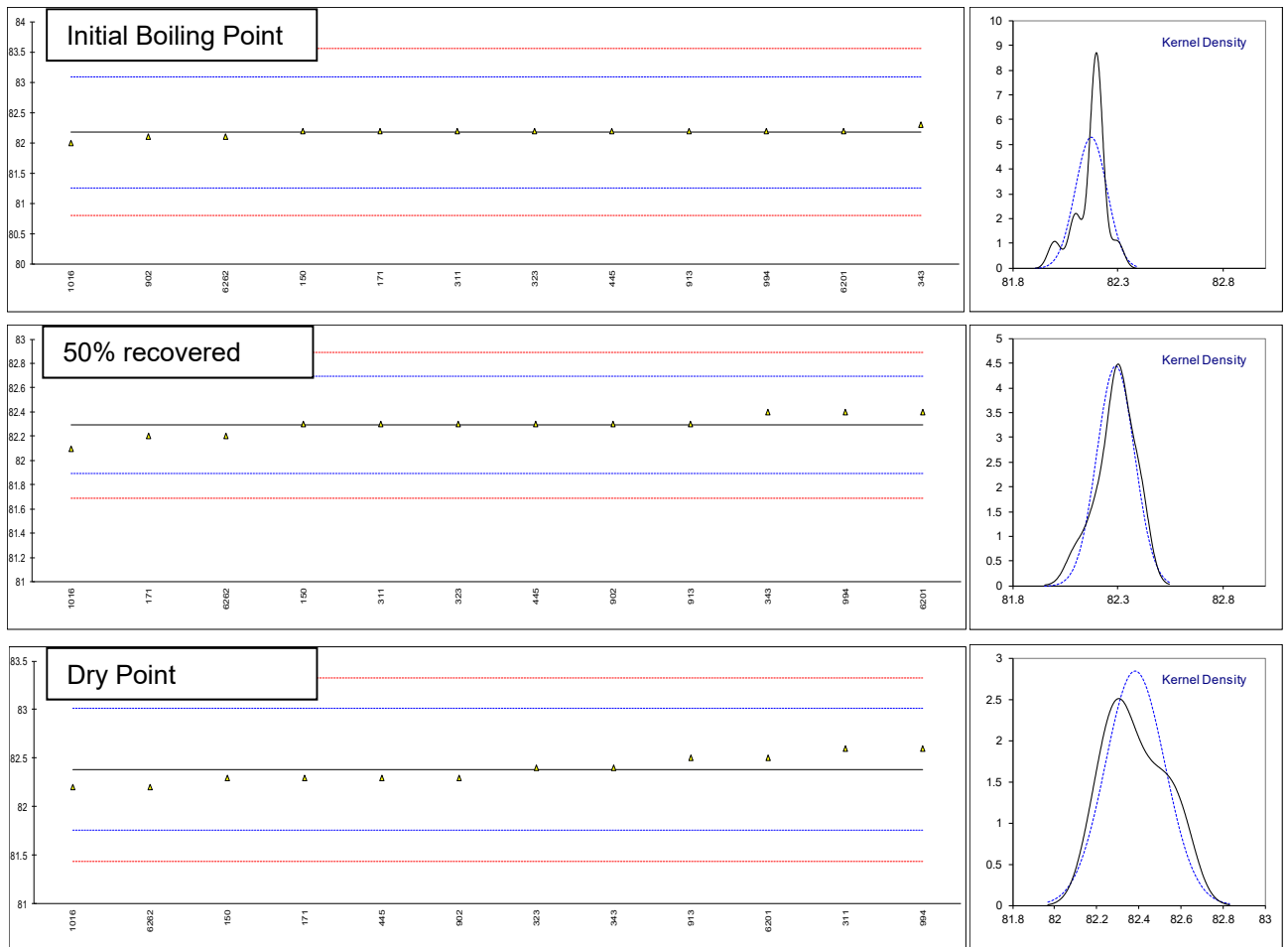
Determination of Nonvolatile matter on sample #19260; results in mg/100mL.

| lab | method | Value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 150 | D1353 | 0.3 | | -0.36 | |
| 171 | D1353 | <1 | | ----- | |
| 173 | D1353 | <0.1 | | ----- | |
| 311 | D1353 | <1 | | ----- | |
| 323 | D1353 | <1 | | ----- | |
| 343 | D1353 | 0.4 | | -0.23 | |
| 445 | D1353 | 1.0 | | 0.57 | |
| 446 | D1353 | 0.6 | | 0.04 | |
| 551 | | ----- | | ----- | |
| 902 | D1353 | 0.5 | | -0.09 | |
| 913 | D1353 | <1 | | ----- | |
| 994 | D1353 | <1 | | ----- | |
| 1016 | D1353 | 0.7 | | 0.17 | |
| 1438 | | ----- | | ----- | |
| 6201 | | ----- | | ----- | |
| 6262 | D1353 | 0.5 | | -0.09 | |

| | |
|---|---|
| normality | unknown |
| n | 7 |
| outliers | 0 |
| mean (n) | 0.57 |
| st.dev. (n) | 0.229 |
| R(calc.) | 0.64 |
| st.dev.(D1353:13) | 0.754 |
| R(D1353:13) | 2.11 |
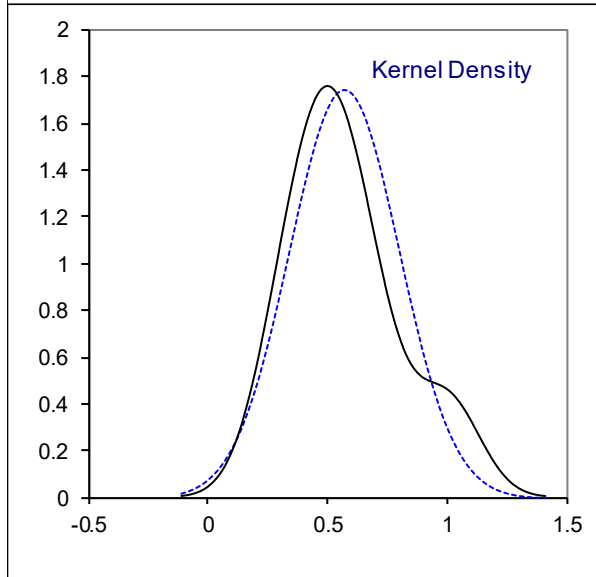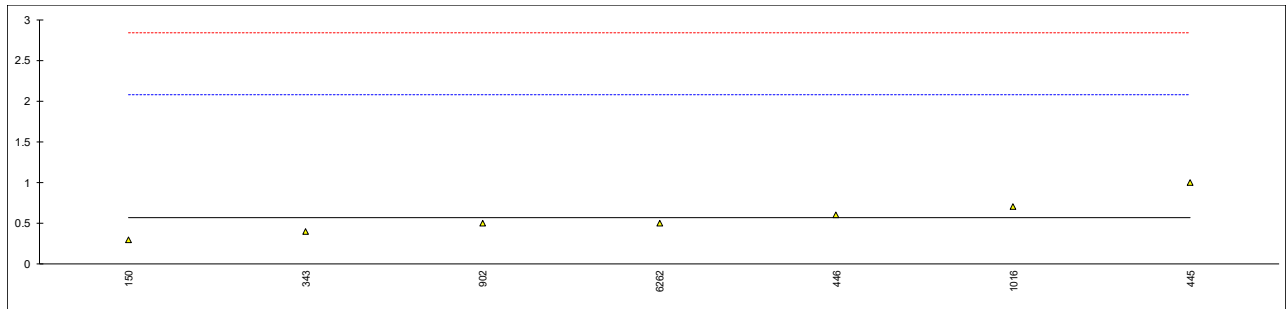
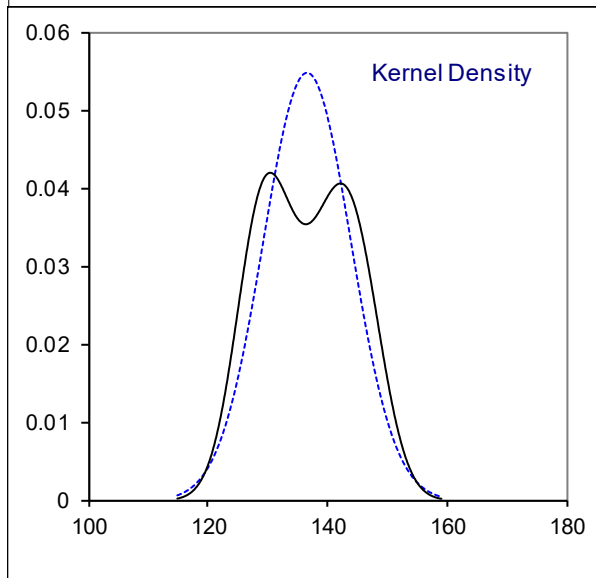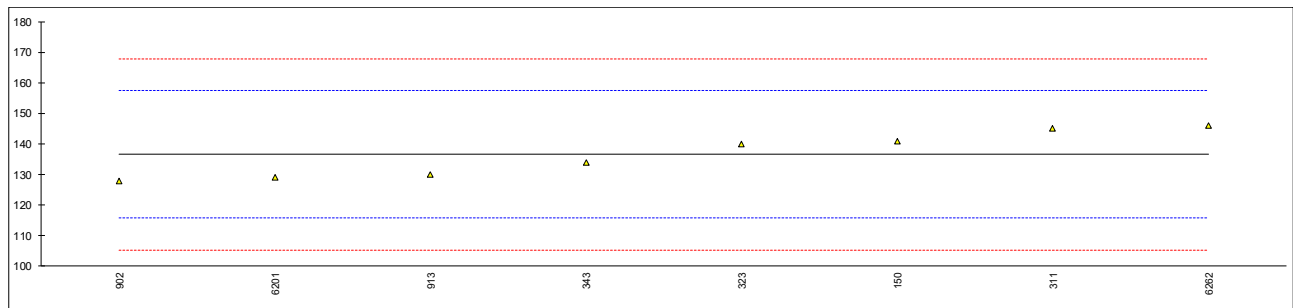Determination of Purity by GC on dry basis on sample #19260, results in %M/M.

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 150 | INH-5290 | 99.98 | | ----- | |
| 171 | | ----- | | ----- | |
| 173 | INH-6012 | 99.98 | | ----- | |
| 311 | INH-082 | 99.97 | | ----- | |
| 323 | INH-060 | 99.97 | | ----- | |
| 343 | DIN55685 | 99.97 | C | ----- | First reported 100.0 |
| 445 | | ----- | | ----- | |
| 446 | INH-CM | 99.97 | | ----- | |
| 551 | | ----- | | ----- | |
| 902 | INH-129 | 99.98 | | ----- | |
| 913 | INH-0002 | 99.98 | | ----- | |
| 994 | INH-015 | 99.969 | | ----- | |
| 1016 | | ----- | | ----- | |
| 1438 | | 99.97 | | ----- | |
| 6201 | In house | 99.98 | | ----- | |
| 6262 | | 99.973 | | ----- | |

| | | |
|---|---|---|
| | normality | OK |
| | n | 12 |
| | outliers | 0 |
| | mean (n) | 99.9743 |
| | st.dev. (n) | 0.00509 |
| | R(calc.) | 0.0142 |
| | st.dev.(lit.) | n.a. |
| | R(lit.) | n.a. |
| Compare | | |
| | R(iis17C18) | 0.019 |
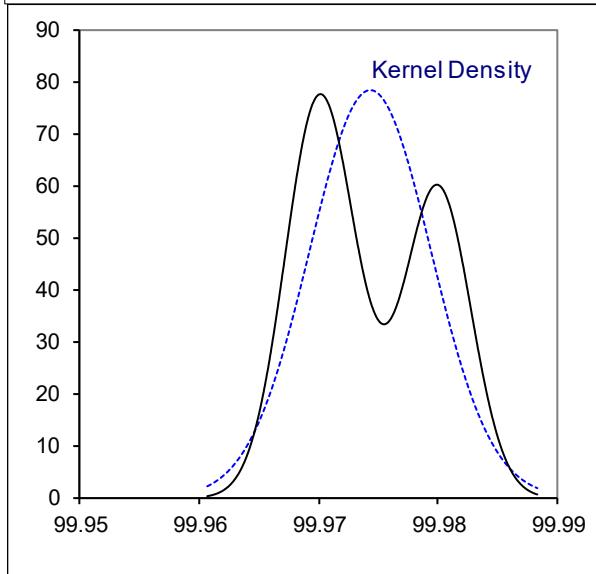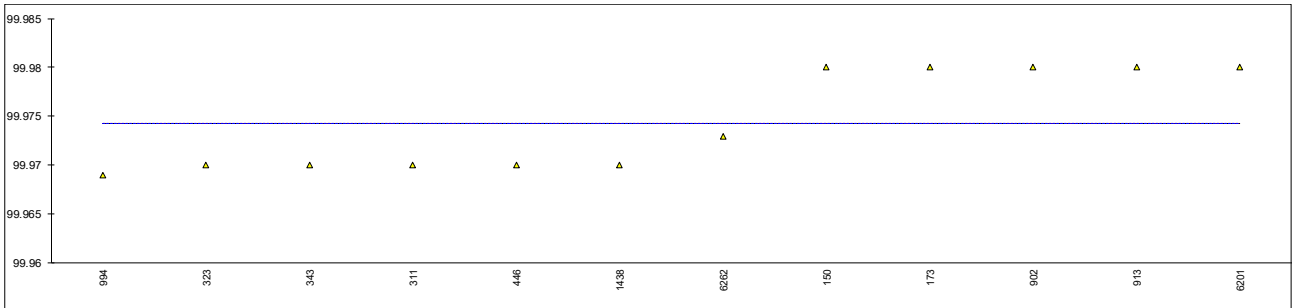
Determination of Ethanol on sample #19260; results in mg/kg.

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 150 | INH-5290 | 141 | | 0.42 | |
| 171 | | ----- | | ----- | |
| 173 | | ----- | | ----- | |
| 311 | INH-082 | 145 | | 0.80 | |
| 323 | INH-060 | 140 | | 0.32 | |
| 343 | DIN55685 | 134 | | -0.25 | |
| 445 | | ----- | | ----- | |
| 446 | | ----- | | ----- | |
| 551 | | ----- | | ----- | |
| 902 | INH-129 | 128 | | -0.83 | |
| 913 | INH-0002 | 130 | | -0.64 | |
| 994 | | ----- | | ----- | |
| 1016 | | ----- | | ----- | |
| 1438 | | ----- | | ----- | |
| 6201 | In house | 129 | | -0.73 | |
| 6262 | | 146 | | 0.90 | |

|  |  |
|--|--|
| normality | unknown |
| n | 8 |
| outliers | 0 |
| mean (n) | 136.63 |
| st.dev. (n) | 7.289 |
| R(calc.) | 20.41 |
| st.dev.(Horwitz) | 10.428 |
| R(Horwitz) | 29.20 |





Kernel Density

## Determination of n-Propanol on sample #19260; results in mg/kg.

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 150 | INH-5290 | 0 | ex | -8.55 | Test results excluded as zero is not a real result |
| 171 | | ----- | | ----- | |
| 173 | | ----- | | ----- | |
| 311 | INH-082 | 7 | | -1.07 | |
| 323 | INH-060 | <10 | | ----- | |
| 343 | DIN55685 | 9 | | 1.07 | |
| 445 | | ----- | | ----- | |
| 446 | | ----- | | ----- | |
| 551 | | ----- | | ----- | |
| 902 | INH-129 | 5 | | -3.21 | |
| 913 | INH-0002 | 10 | | 2.14 | |
| 994 | | ----- | | ----- | |
| 1016 | | ----- | | ----- | |
| 1438 | | ----- | | ----- | |
| 6201 | In house | 7 | | -1.07 | |
| 6262 | | 10 | | 2.14 | |

|  |  |
|--|--|
| normality | unknown |
| n | 6 |
| outliers | 0 (+1ex) |
| mean (n) | 8.00 |
| st.dev. (n) | 2.000 |
| R(calc.) | 5.60 |
| st.dev.(Horwitz) | 0.936 |
| R(Horwitz) | 2.62 |





Kernel Density
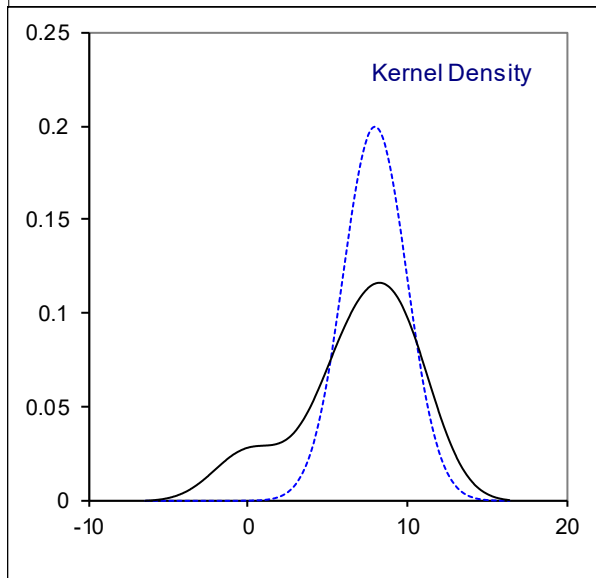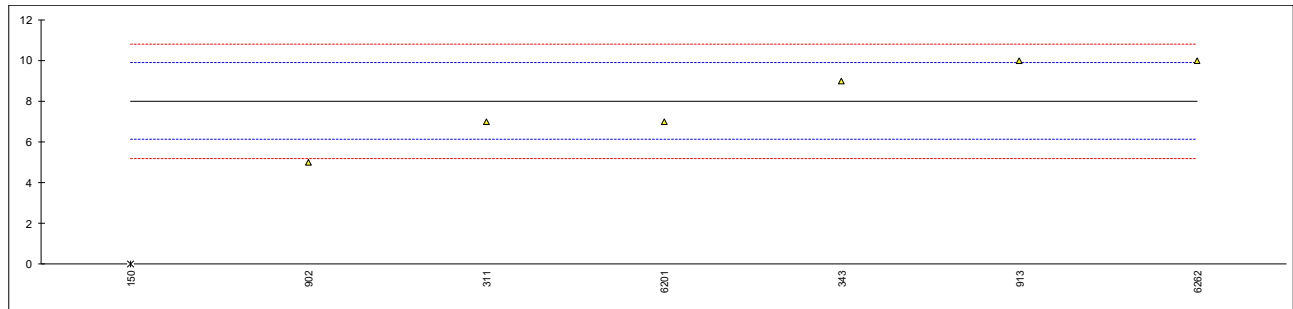
Determination of n-Butanol on sample #19260; results in mg/kg.

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 150 | INH-5290 | 0 | | ----- | |
| 171 | | ----- | | ----- | |
| 173 | | ----- | | ----- | |
| 311 | INH-082 | <1 | | ----- | |
| 323 | INH-060 | <10 | | ----- | |
| 343 | DIN55685 | 0 | | ----- | |
| 445 | | ----- | | ----- | |
| 446 | | ----- | | ----- | |
| 551 | | ----- | | ----- | |
| 902 | INH-129 | <5 | | ----- | |
| 913 | INH-0002 | <5 | | ----- | |
| 994 | | ----- | | ----- | |
| 1016 | | ----- | | ----- | |
| 1438 | | ----- | | ----- | |
| 6201 | In house | <5 | | ----- | |
| 6262 | | 0 | | ----- | |

|  |  |  |
|---|---|---|
| n | 8 | |
| mean (n) | <10 | |

Determination of Other impurities on sample #19260; results in mg/kg.

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 150 | | ----- | | ----- | |
| 171 | | ----- | | ----- | |
| 173 | | ----- | | ----- | |
| 311 | INH-082 | 115 | | 0.71 | |
| 323 | INH-060 | 118 | | 0.89 | |
| 343 | DIN55685 | 4 | C,G(0.05) | -6.04 | First reported 0 |
| 445 | | ----- | | ----- | |
| 446 | | ----- | | ----- | |
| 551 | | ----- | | ----- | |
| 902 | INH-129 | 68 | | -2.15 | |
| 913 | INH-0002 | 100 | | -0.20 | |
| 994 | | ----- | | ----- | |
| 1016 | | ----- | | ----- | |
| 1438 | | ----- | | ----- | |
| 6201 | In house | 95 | | -0.51 | |
| 6262 | | 124 | | 1.26 | |

| | |
|---|---|
| normality | unknown |
| n | 6 |
| outliers | 1 |
| mean (n) | 103.33 |
| st.dev. (n) | 20.530 |
| R(calc.) | 57.48 |
| st.dev.(Horwitz (4)) | 16.452 |
| R(Horwitz (4)) | 46.07 |





Kernel Density
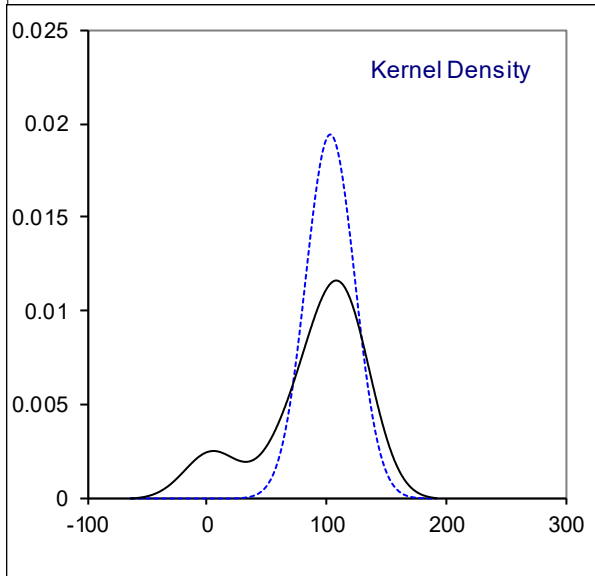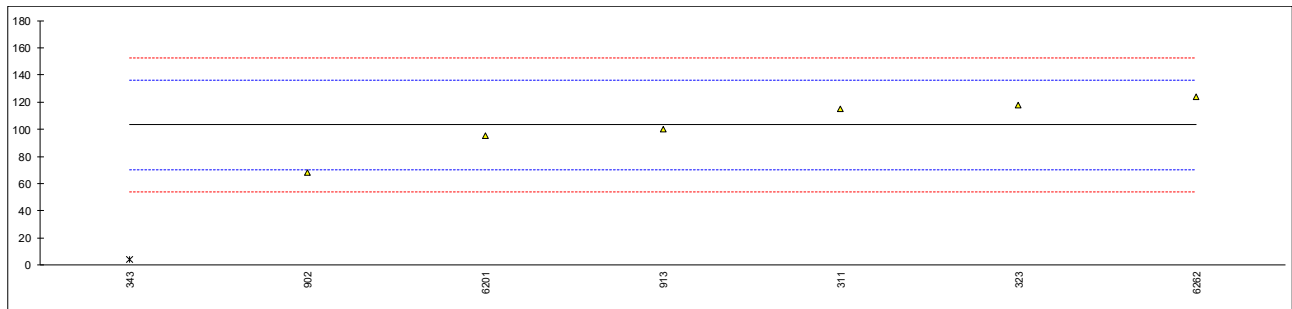
Determination of Water on sample #19260; results in mg/kg.

| lab | method | value | mark | z(targ) | remarks |
|-----|--------|-------|------|---------|---------|
| 150 | E1064 | 190 | | -0.54 | |
| 171 | E1064 | 179 | | -1.53 | |
| 173 | E1064 | 190 | | -0.54 | |
| 311 | | ----- | | ----- | |
| 323 | E1064 | 190 | | -0.54 | |
| 343 | E1064 | 220 | C | 2.15 | First reported 0.022 mg/kg |
| 445 | E1064 | 183.3 | | -1.14 | |
| 446 | E203 | 170 | | -2.34 | |
| 551 | | ----- | | ----- | |
| 902 | E1064 | 187 | | -0.81 | |
| 913 | E1064 | 215 | | 1.70 | |
| 994 | E1064 | 245 | | 4.40 | |
| 1016 | D1364 | 530 | G(0.01) | 30.00 | |
| 1438 | | ----- | | ----- | |
| 6201 | | ----- | W | ----- | Test result withdrawn, reported 478 |
| 6262 | E1064 | 187 | | -0.81 | |

|  |  |
|--|--|
| normality | not OK |
| n | 11 |
| outliers | 1 |
| mean (n) | 196.03 |
| st.dev. (n) | 21.758 |
| R(calc.) | 60.92 |
| st.dev.(E1064:16) | 11.132 |
| R(E1064:16) | 31.17 |
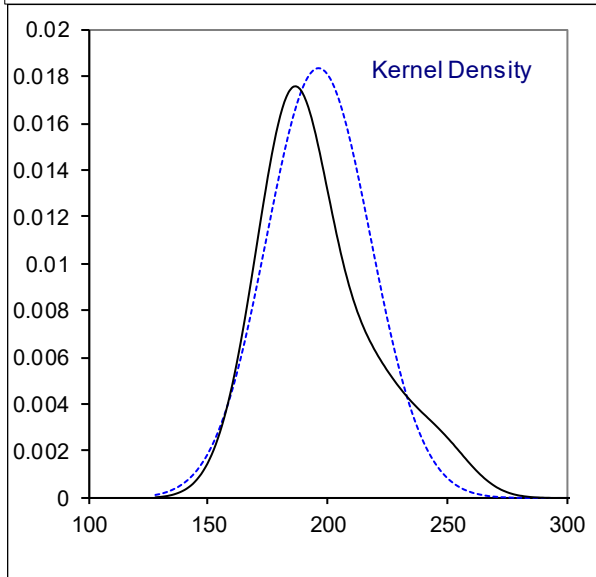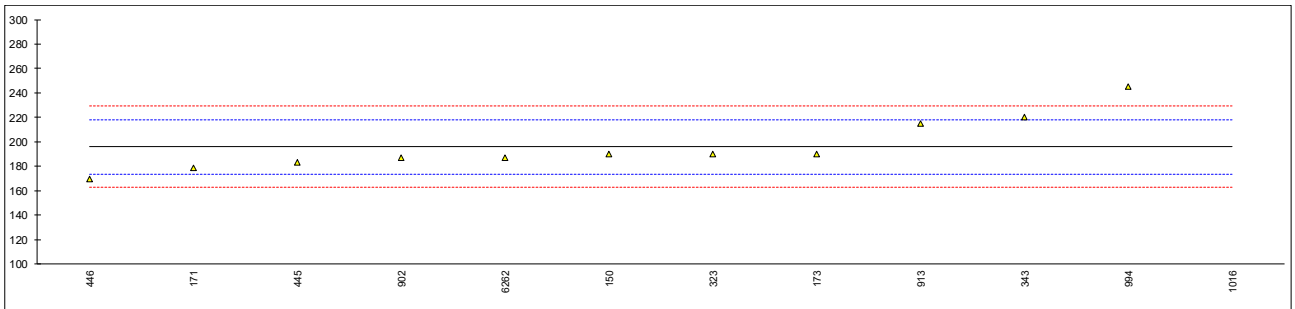
**APPENDIX 2**

**Number of participants per country**

    1 lab in AZERBAIJAN
2 labs in BELGIUM
  1 lab in BRAZIL
  1 lab in INDIA
  1 lab in ISRAEL
3 labs in NETHERLANDS
  1 lab in SPAIN
  1 lab in TURKEY
2 labs in UNITED KINGDOM
3 labs in UNITED STATES OF AMERICA

**APPENDIX 3**

**Abbreviations**

C             = final test result after checking of first reported suspect test result
D(0.01)    = outlier in Dixon's outlier test
D(0.05)    = straggler in Dixon's outlier test
G(0.01)    = outlier in Grubbs' outlier test
G(0.05)    = straggler in Grubbs' outlier test
DG(0.01)  = outlier in Double Grubbs' outlier test
DG(0.05)  = straggler in Double Grubbs' outlier test
R(0.01)    = outlier in Rosner's outlier test
R(0.05)    = straggler in Rosner's outlier test
E             = possibly an error in calculations
W            = test result withdrawn on request of participant
ex           = test result excluded from statistical evaluation
n.a.         = not applicable
n.e.         = not evaluated
n.d.         = not detected
SDS         = Safety Data Sheet

**Literature**

1       iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2       ASTM E178:02
3       ASTM E1301:03
4       ISO13528:05
5       ISO5725:86
6       ISO5725, parts 1-6, 1994
7       M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
8       W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
9       IP 367:84
10     DIN 38402 T41/42
11     P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
12     J.N. Miller, Analyst, 118, 455, (1993)
13     Analytical Methods Committee, Technical Brief, No 4, January 2001
14     P.J. Lowthian and M. Thompson, The Royal Society of Chemistry 2002, Analyst, 127, 1359-1364, (2002)
15     Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2), 165-172, (1983)
16     Horwitz, R. Albert, J. AOAC Int, 79, 3, 589, (1996)