



Institute for
Interlaboratory Studies

Results of Proficiency Test Isopropanol (IPA) December 2023

Organized by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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1 INTRODUCTION

Since 2003 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Isopropanol (IPA) based on the latest version of ASTM D770 once every two years. During the annual proficiency testing program of 2023 it was decided to continue the round robin for the analysis of Isopropanol (IPA).

In this interlaboratory study 17 laboratories in 13 countries registered for participation, see appendix 2 for the number of participants per country. In this report the results of the Isopropanol (IPA) 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 a laboratory that has performed the tests in accordance with for ISO/IEC17043 relevant requirements of ISO/IEC17025.

It was decided to send one sample Isopropanol (IPA) in a 0.5 liter bottle labelled #23270. The 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 was obtained from a local chemical supplier. After homogenization 30 amber glass bottles of 0.5 L were filled and labelled #23270.

The homogeneity of the subsamples was checked by 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 #23270-1 | 0.78509 |
| sample #23270-2 | 0.78509 |
| sample #23270-3 | 0.78509 |
| sample #23270-4 | 0.78508 |
| sample #23270-5 | 0.78511 |
| sample #23270-6 | 0.78509 |
| sample #23270-7 | 0.78509 |
| sample #23270-8 | 0.78508 |

Table 1: homogeneity test results of subsamples #23270

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 #23270

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 0.5 L bottle of Isopropanol labelled #23270 was sent on November 15, 2023. An SDS was added to the sample package.

2.5 STABILITY OF THE SAMPLES

The stability of Isopropanol 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: Acidity as Acetic acid, Appearance, Inorganic Chloride as Cl, Color Pt/Co, Density at 20 °C, Specific Gravity 20/20 °C, Distillation (IBP, 50% recovered & DP), Nonvolatile matter, Purity by GC on dry basis, Ethanol, n-Propanol, n-Butanol, Other impurities and 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 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:

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

The $z_{(\text{target})}$ 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

In this proficiency test no problems were encountered with the dispatch of the samples. Three participants reported test results after the final reporting date. Not all participants were able to report all tests requested.

In total 17 participants submitted 146 numerical test results. Observed were 6 outlying test results, which is 4.1%. 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 TEST

In this section the reported test 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 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).

Acidity as Acetic Acid: The group of participants met the target requirements. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D1613:17R23.

Appearance: All reporting participants agreed on a test result as Pass or Clear and Bright.

Inorganic Chloride as Cl: Only four participants reported a test result. They agreed on a level of <1 mg/kg. Therefore, no z-scores are calculated.

Color Pt/Co: The group of participants met the target requirements. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D1209:05R19.

Density at 20 °C: The group of participants met the target requirements. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ISO12185:96.

Specific Gravity at 20/20 °C: The group of participants met the target requirements. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ISO12185:96.

Distillation at 760 mmHg: The group of participants met the target requirements. No statistical outliers were observed. All three calculated reproducibilities are in agreement with the requirements of ASTM D1078:11R19 for the automated as well as the manual mode.

Nonvolatile matter: All reporting participants agreed on a level of <1 mg/100 mL. Therefore, no z-scores are calculated.

Purity by GC on dry basis: Regretfully, the methods used do not provide any reproducibility limit. Therefore, no z-scores are calculated. Two statistical outliers were observed.

Ethanol: The group of participants may have had difficulty to meet the target requirements. No statistical outliers were observed. Due to the large variation in test results, no z-scores are calculated.

n-Propanol: The group of participants may have had difficulty to meet the target requirements. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outlier is not in agreement with the estimated reproducibility calculated with the Horwitz equation.

n-Butanol: Most reporting participants agreed on a level of <10 mg/kg. Therefore, no z-scores are calculated.

Other impurities: Only four participants reported a test result. They agreed on a level of <50 mg/kg. Therefore, no z-scores are calculated.

Water: The group of participants had difficulty to meet the target requirements. No statistical outliers were observed. The calculated reproducibility is not in agreement with the requirements of ASTM E1064:24.

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) |
|------------------------------|-----------|----|------------|----------|--------|
| Acidity as Acetic Acid | mg/kg | 11 | 6.9 | 4.9 | 14 |
| Appearance | | 15 | Pass (C&B) | n.a. | n.a. |
| Inorganic Chloride as Cl | mg/kg | 4 | <1 | n.e. | n.e. |
| Color Pt/Co | | 13 | 2.2 | 4.5 | 7 |
| Density at 20 °C | kg/L | 15 | 0.7851 | 0.0002 | 0.0005 |
| Specific Gravity at 20/20 °C | | 13 | 0.7865 | 0.0002 | 0.0005 |
| Initial Boiling Point | °C | 13 | 82.1 | 0.4 | 1.3 |
| 50% recovered | °C | 13 | 82.3 | 0.4 | 0.6 |
| Dry Point | °C | 13 | 82.4 | 0.5 | 0.9 |
| Nonvolatile matter | mg/100 mL | 10 | <1 | n.e. | n.e. |
| Purity by GC on dry basis | %M/M | 10 | 99.985 | 0.018 | n.a. |
| Ethanol | mg/kg | 6 | 62 | 57 | (15) |
| n-Propanol | mg/kg | 7 | 42 | 15 | 11 |

| Parameter | unit | n | average | 2.8 * sd | R(lit) |
|------------------|-------|----|---------|----------|--------|
| n-Butanol | mg/kg | 7 | <10 | n.e. | n.e. |
| Other impurities | mg/kg | 3 | <50 | n.e. | n.e. |
| Water | mg/kg | 16 | 442 | 182 | 70 |

Table 3: reproducibilities of tests on sample #23270

For results between brackets no z-scores are calculated.

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 DECEMBER 2023 WITH PREVIOUS PTS

| | December 2023 | December 2021 | December 2019 | December 2017 | December 2015 |
|------------------------------------|---------------|---------------|---------------|---------------|---------------|
| Number of reporting laboratories | 17 | 13 | 15 | 17 | 17 |
| Number of test results | 146 | 133 | 148 | 157 | 192 |
| Number of statistical outliers | 6 | 2 | 3 | 5 | 8 |
| Percentage of statistical outliers | 4.1% | 1.5% | 2.0% | 3.2% | 4.2% |

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

| Parameter | December 2023 | December 2021 | December 2019 | December 2017 | December 2015 |
|------------------------------|---------------|---------------|---------------|---------------|---------------|
| Acidity as Acetic Acid | ++ | ++ | + | ++ | ++ |
| Inorganic Chloride as Cl | 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 | n.e. | n.e. | ++ | ++ | ++ |
| Ethanol | (--) | (--) | + | - | +/- |
| n-Propanol | - | (--) | -- | - | + |
| n-Butanol | n.e. | (--) | n.e. | n.e. | n.e. |
| Other impurities | n.e. | n.e. | - | n.e. | -- |
| Water | -- | -- | - | - | +/- |

Table 5: comparison of determinations to the reference test methods

For results between brackets no z-scores are calculated.

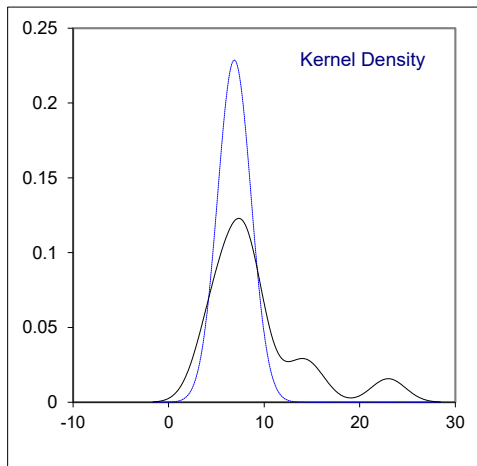
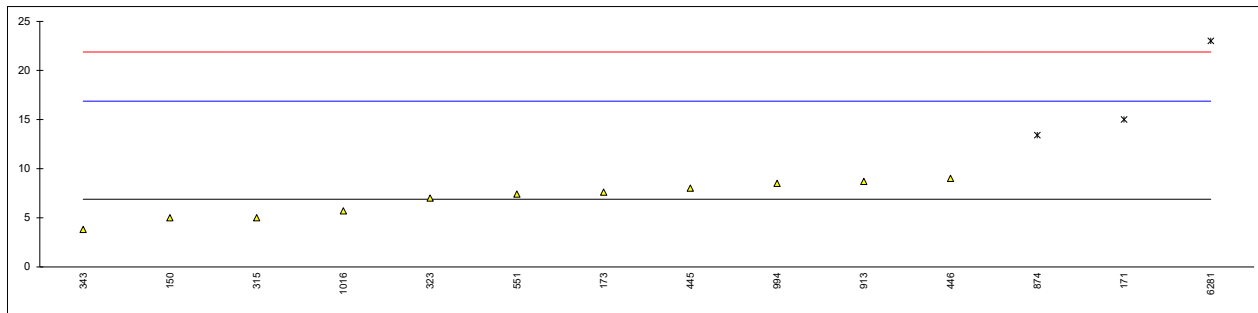
The following performance categories were used:

- ++ : 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 #23270; results in mg/kg.

| lab | method | value | mark | z(targ) | remarks |
|----------------------|--------|-------|------------|---------|-----------------------------|
| 150 | D1613 | 5 | | -0.38 | |
| 171 | D1613 | 15 | C,DG(0.05) | 1.62 | first reported 0.0015 mg/kg |
| 173 | D1613 | 7.6 | | 0.14 | |
| 315 | D1613 | 5 | | -0.38 | |
| 323 | D1613 | 7 | | 0.02 | |
| 343 | D1613 | 3.8 | | -0.62 | |
| 396 | | ----- | | ----- | |
| 445 | D1613 | 8 | C | 0.22 | first reported 0.0008 mg/kg |
| 446 | D1613 | 9 | | 0.42 | |
| 551 | D1613 | 7.4 | | 0.10 | |
| 874 | D1613 | 13.4 | DG(0.05) | 1.30 | |
| 913 | D1613 | 8.7 | | 0.36 | |
| 994 | D1613 | 8.5 | | 0.32 | |
| 1016 | D1613 | 5.7 | | -0.24 | |
| 1438 | | ----- | | ----- | |
| 2458 | | ----- | | ----- | |
| 6281 | D1613 | 23 | G(0.01) | 3.22 | |
| normality | | OK | | | |
| n | | 11 | | | |
| outliers | | 3 | | | |
| mean (n) | | 6.88 | | | |
| st.dev. (n) | | 1.745 | | | |
| R(calc.) | | 4.88 | | | |
| st.dev.(D1613:17R23) | | 5 | | | |
| R(D1613:17R23) | | 14 | | | |



Determination of Appearance on sample #23270;

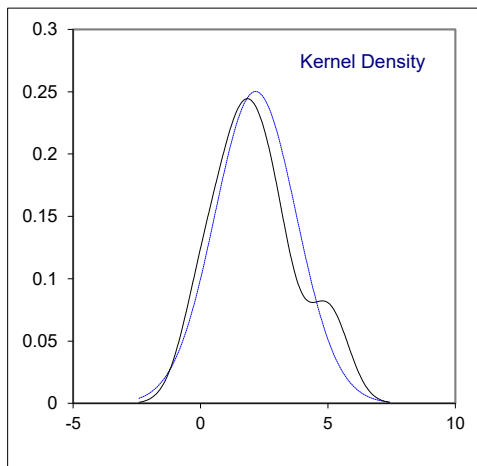
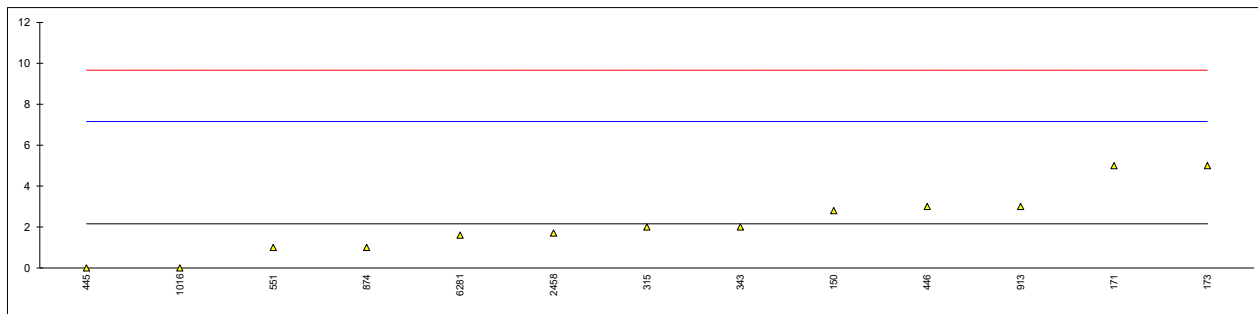
| lab | method | value | mark | z(targ) | remarks |
|----------|--------|---|------|---------|---------|
| 150 | Visual | clear, bright, free from solid matter and water | | ---- | |
| 171 | E2680 | Clear | | ---- | |
| 173 | E2680 | Pass | | ---- | |
| 315 | E2680 | pass | | ---- | |
| 323 | Visual | PASS | | ---- | |
| 343 | E2680 | Pass | | ---- | |
| 396 | Visual | Clear & bright | | ---- | |
| 445 | E2680 | Pass C&B | | ---- | |
| 446 | E2680 | PASS | | ---- | |
| 551 | E2680 | Pass | | ---- | |
| 874 | E2680 | Pass | | ---- | |
| 913 | E2680 | Clear and Bright | | ---- | |
| 994 | Visual | pass | | ---- | |
| 1016 | Visual | pass | | ---- | |
| 1438 | | ---- | | ---- | |
| 2458 | | ---- | | ---- | |
| 6281 | Visual | Clear&Bright | | ---- | |
| n | | 15 | | | |
| mean (n) | | Pass (Clear and Bright) | | | |

Determination of Inorganic Chloride as Cl on sample #23270; results in mg/kg.

| lab | method | value | mark | z(targ) | remarks |
|------|----------|-------|------|---------|---------|
| 150 | | ---- | | ---- | |
| 171 | | ---- | | ---- | |
| 173 | | ---- | | ---- | |
| 315 | INH-158 | <0.2 | | ---- | |
| 323 | IMPCA002 | <0.3 | | ---- | |
| 343 | | ---- | | ---- | |
| 396 | | ---- | | ---- | |
| 445 | | ---- | | ---- | |
| 446 | | ---- | | ---- | |
| 551 | | ---- | | ---- | |
| 874 | IMPCA002 | <0.25 | | ---- | |
| 913 | In-House | <1 | | ---- | |
| 994 | | ---- | | ---- | |
| 1016 | | ---- | | ---- | |
| 1438 | | ---- | | ---- | |
| 2458 | | ---- | | ---- | |
| 6281 | | ---- | | ---- | |
| | n | 4 | | | |
| | mean (n) | <1 | | | |

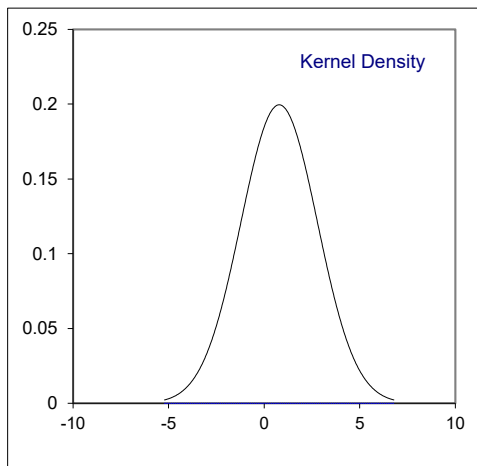
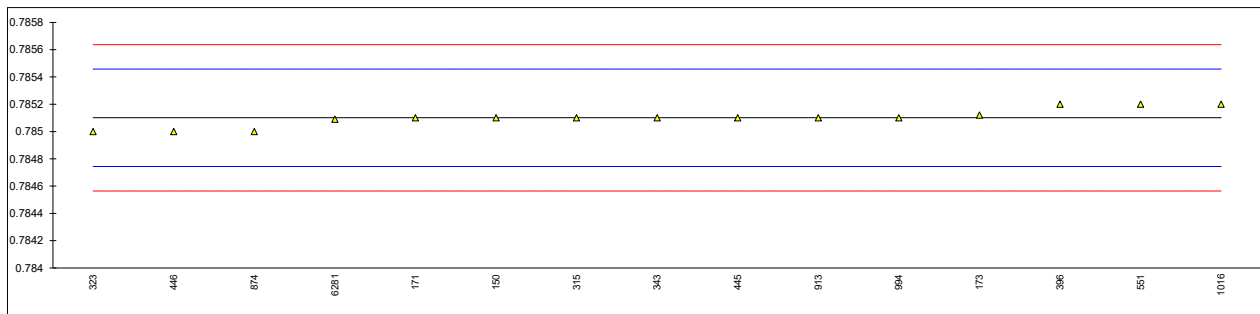
Determination of Color Pt/Co on sample #23270;

| lab | method | value | mark | z(targ) | remarks |
|----------------------|---------|-------|------|---------|---------|
| 150 | D1209 | 2.8 | | 0.26 | |
| 171 | D1209 | 5 | | 1.14 | |
| 173 | D1209 | 5 | | 1.14 | |
| 315 | D5386 | 2 | | -0.06 | |
| 323 | D1209 | <5 | | ---- | |
| 343 | D5386 | 2 | | -0.06 | |
| 396 | | ---- | | ---- | |
| 445 | D1209 | 0 | | -0.86 | |
| 446 | D5386 | 3 | | 0.34 | |
| 551 | D1209 | 1 | | -0.46 | |
| 874 | D1209 | 1 | | -0.46 | |
| 913 | D5386 | 3 | | 0.34 | |
| 994 | D1209 | <5 | | ---- | |
| 1016 | D1209 | 0 | | -0.86 | |
| 1438 | | ---- | | ---- | |
| 2458 | ISO6271 | 1.7 | | -0.18 | |
| 6281 | D1209 | 1.6 | | -0.22 | |
| normality | | OK | | | |
| n | | 13 | | | |
| outliers | | 0 | | | |
| mean (n) | | 2.16 | | | |
| st.dev. (n) | | 1.596 | | | |
| R(calc.) | | 4.47 | | | |
| st.dev.(D1209:05R19) | | 2.5 | | | |
| R(D1209:05R19) | | 7 | | | |



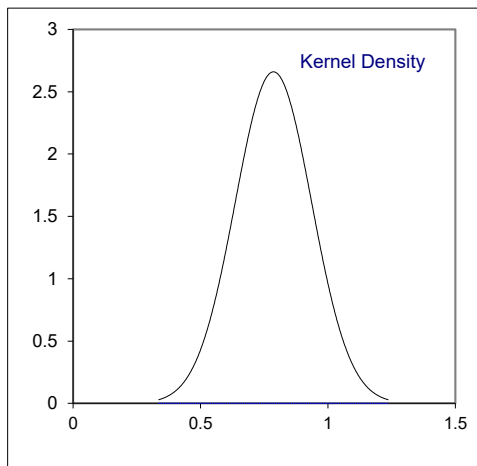
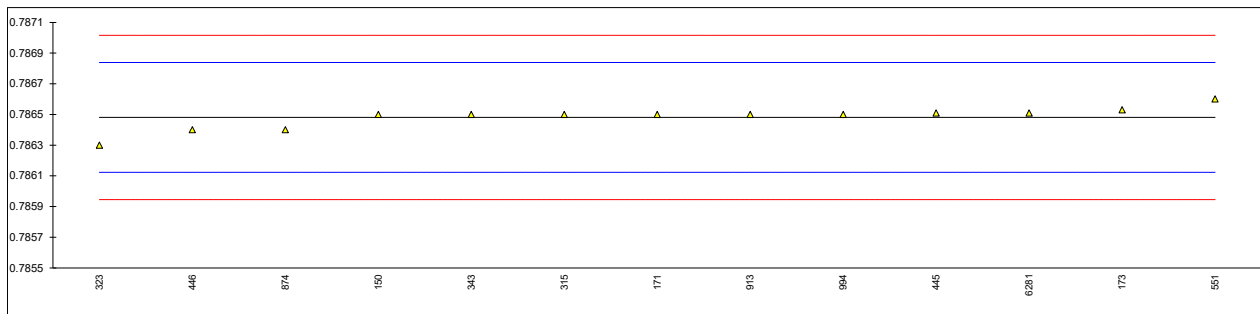
Determination of Density at 20 °C on sample #23270; results in kg/L.

| lab | method | value | mark | z(targ) | remarks |
|----------------------|----------|----------|------|---------|---------|
| 150 | D4052 | 0.7851 | | 0.00 | |
| 171 | ISO12185 | 0.7851 | | 0.00 | |
| 173 | D4052 | 0.78512 | | 0.11 | |
| 315 | D4052 | 0.7851 | | 0.00 | |
| 323 | D4052 | 0.7850 | | -0.56 | |
| 343 | D4052 | 0.7851 | | 0.00 | |
| 396 | D4052 | 0.7852 | | 0.56 | |
| 445 | ISO12185 | 0.7851 | | 0.00 | |
| 446 | D4052 | 0.7850 | | -0.56 | |
| 551 | D4052 | 0.7852 | | 0.56 | |
| 874 | ISO12185 | 0.7850 | | -0.56 | |
| 913 | D4052 | 0.7851 | | 0.00 | |
| 994 | ISO12185 | 0.7851 | | 0.00 | |
| 1016 | D4052 | 0.7852 | | 0.56 | |
| 1438 | | ---- | | ---- | |
| 2458 | | ---- | | ---- | |
| 6281 | D4052 | 0.78509 | | -0.06 | |
| normality | | OK | | | |
| n | | 15 | | | |
| outliers | | 0 | | | |
| mean (n) | | 0.78510 | | | |
| st.dev. (n) | | 0.000066 | | | |
| R(calc.) | | 0.00018 | | | |
| st.dev.(ISO12185:96) | | 0.000179 | | | |
| R(ISO12185:96) | | 0.0005 | | | |



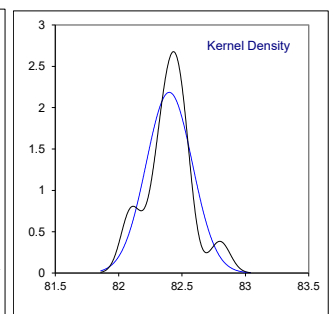
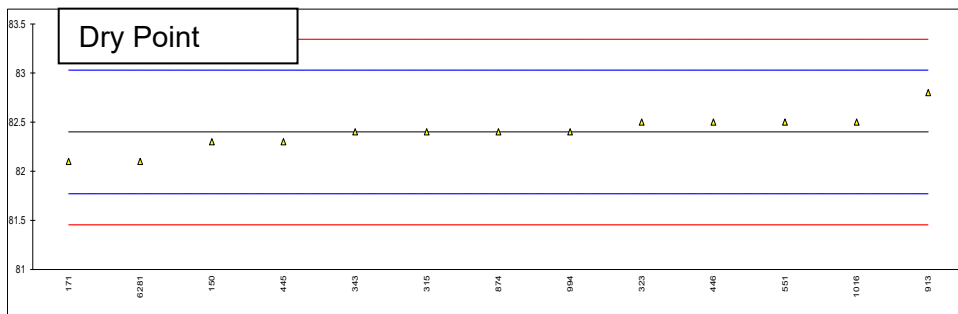
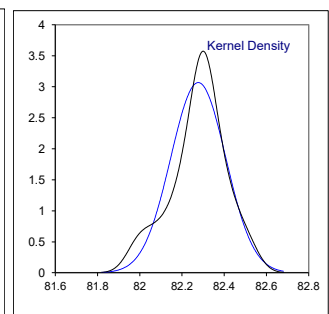
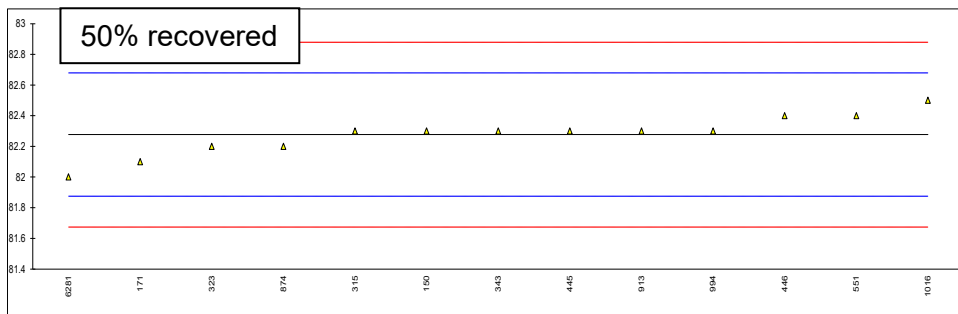
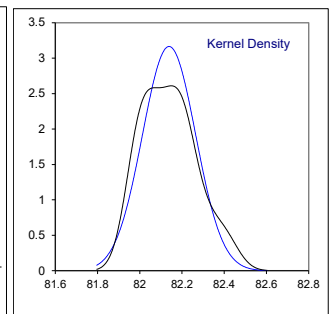
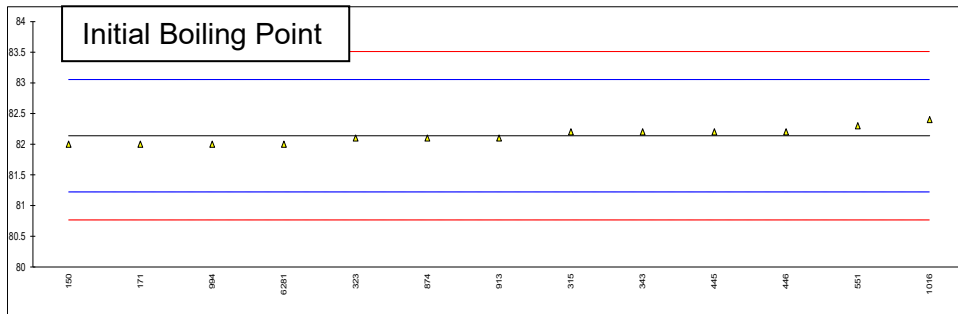
Determination of Specific Gravity at 20/20 °C on sample #23270;

| lab | method | value | mark | z(targ) | remarks |
|----------------------|----------|----------|------|---------|---------|
| 150 | D4052 | 0.7865 | | 0.11 | |
| 171 | ISO12185 | 0.7865 | | 0.11 | |
| 173 | D4052 | 0.78653 | | 0.28 | |
| 315 | D4052 | 0.7865 | | 0.11 | |
| 323 | D4052 | 0.7863 | | -1.01 | |
| 343 | D4052 | 0.7865 | | 0.11 | |
| 396 | | ----- | | ----- | |
| 445 | ISO12185 | 0.78651 | | 0.16 | |
| 446 | D4052 | 0.7864 | | -0.45 | |
| 551 | D4052 | 0.7866 | | 0.67 | |
| 874 | ISO12185 | 0.7864 | | -0.45 | |
| 913 | D4052 | 0.7865 | | 0.11 | |
| 994 | ISO12185 | 0.7865 | | 0.11 | |
| 1016 | | ----- | | ----- | |
| 1438 | | ----- | | ----- | |
| 2458 | | ----- | | ----- | |
| 6281 | D4052 | 0.78651 | | 0.16 | |
| normality | | not OK | | | |
| n | | 13 | | | |
| outliers | | 0 | | | |
| mean (n) | | 0.78648 | | | |
| st.dev. (n) | | 0.000074 | | | |
| R(calc.) | | 0.00021 | | | |
| st.dev.(ISO12185:96) | | 0.000179 | | | |
| R(ISO12185:96) | | 0.0005 | | | |



Determination of Distillation at 760 mmHg on sample #23270; results in °C.

| lab | method | IBP | mark | z(targ) | 50%rec | mark | z(targ) | DP | mark | z(targ) |
|------------------------|-----------------|-------|------|---------|--------|------|---------|---------|------|---------|
| 150 | D1078-automated | 82.0 | | -0.30 | 82.3 | | 0.11 | 82.3 | | -0.32 |
| 171 | D1078-automated | 82.0 | | -0.30 | 82.1 | | -0.88 | 82.1 | | -0.95 |
| 173 | | ---- | | ---- | | | | | | ---- |
| 315 | D1078-automated | 82.2 | | 0.13 | 82.3 | | 0.11 | 82.4 | | 0.00 |
| 323 | D1078-automated | 82.1 | | -0.08 | 82.2 | | -0.38 | 82.5 | | 0.32 |
| 343 | D1078-automated | 82.2 | | 0.13 | 82.3 | | 0.11 | 82.4 | | 0.00 |
| 396 | | ---- | | ---- | | | | | | ---- |
| 445 | D1078-manual | 82.2 | | 0.13 | 82.3 | | 0.11 | 82.3 | | -0.32 |
| 446 | D1078-automated | 82.2 | | 0.13 | 82.4 | | 0.61 | 82.5 | | 0.32 |
| 551 | D1078-automated | 82.3 | | 0.35 | 82.4 | | 0.61 | 82.5 | | 0.32 |
| 874 | D1078-manual | 82.1 | | -0.08 | 82.2 | | -0.38 | 82.4 | | 0.00 |
| 913 | D1078-manual | 82.1 | | -0.08 | 82.3 | | 0.11 | 82.8 | | 1.27 |
| 994 | D1078-manual | 82.0 | | -0.30 | 82.3 | | 0.11 | 82.4 | | 0.00 |
| 1016 | D1078-automated | 82.4 | | 0.57 | 82.5 | | 1.11 | 82.5 | | 0.32 |
| 1438 | | ---- | | ---- | | | | | | ---- |
| 2458 | | ---- | | ---- | | | | | | ---- |
| 6281 | D1078-automated | 82.0 | | -0.30 | 82.0 | | -1.38 | 82.1 | | -0.95 |
| normality | | OK | | | OK | | | suspect | | |
| n | | 13 | | | 13 | | | 13 | | |
| outliers | | 0 | | | 0 | | | 0 | | |
| mean (n) | | 82.14 | | | 82.28 | | | 82.40 | | |
| st.dev. (n) | | 0.126 | | | 0.130 | | | 0.183 | | |
| R(calc.) | | 0.35 | | | 0.36 | | | 0.51 | | |
| st.dev.(D1078-A:11R19) | | 0.458 | | | 0.201 | | | 0.315 | | |
| R(D1078-A:11R19) | | 1.28 | | | 0.56 | | | 0.88 | | |
| Compare: | | | | | | | | | | |
| R(D1078-M:11R19) | | 0.88 | | | 0.53 | | | 1.07 | | |

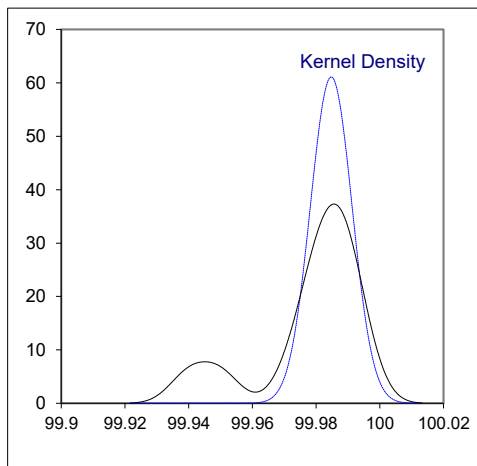
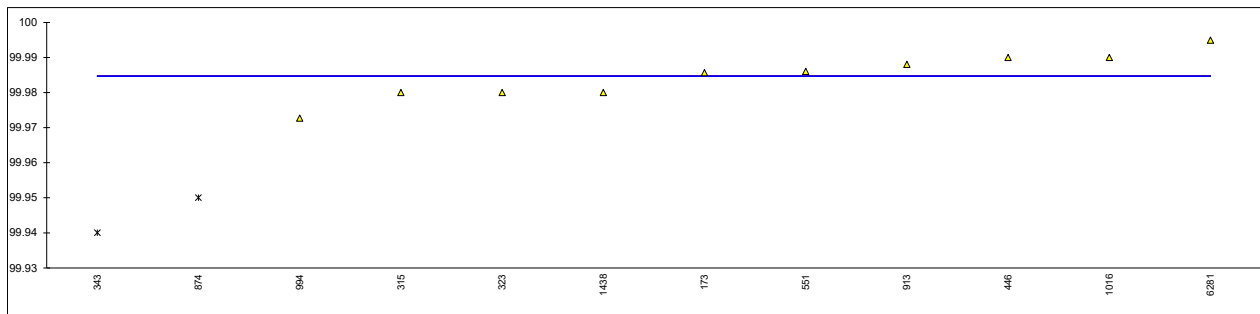


Determination of Nonvolatile matter on sample #23270; results in mg/100 mL.

| lab | method | value | mark | z(targ) | remarks |
|------|----------|-------|------|---------|---------|
| 150 | D1353 | 0.0 | | ---- | |
| 171 | D1353 | 0.0 | | ---- | |
| 173 | | ---- | | ---- | |
| 315 | D1353 | <1 | | ---- | |
| 323 | D1353 | <1 | | ---- | |
| 343 | D1353 | <1 | | ---- | |
| 396 | | ---- | | ---- | |
| 445 | D1353 | <1 | | ---- | |
| 446 | D1353 | 0 | | ---- | |
| 551 | D1353 | 0.2 | | ---- | |
| 874 | D1353 | <1.0 | | ---- | |
| 913 | D1353 | 0.6 | | ---- | |
| 994 | | ---- | | ---- | |
| 1016 | | ---- | | ---- | |
| 1438 | | ---- | | ---- | |
| 2458 | | ---- | | ---- | |
| 6281 | | ---- | | ---- | |
| | n | 10 | | | |
| | mean (n) | <1 | | | |

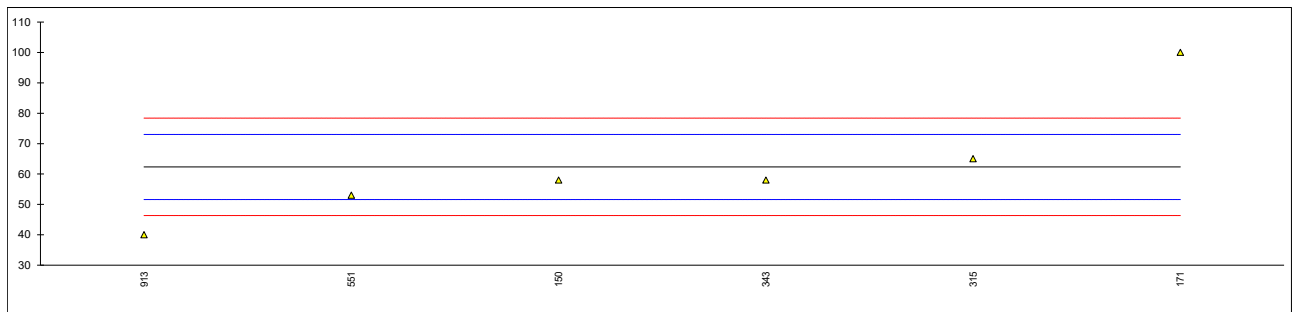
Determination of Purity by GC on dry basis on sample #23270, results in %M/M.

| lab | method | value | mark | z(targ) | remarks |
|---------------|-----------|-----------|----------|---------|---------|
| 150 | | ---- | | ---- | |
| 171 | | ---- | | ---- | |
| 173 | INH-6012 | 99.985634 | | ---- | |
| 315 | INH-082 | 99.98 | | ---- | |
| 323 | INH-060 | 99.98 | | ---- | |
| 343 | DIN55685 | 99.94 | DG(0.01) | ---- | |
| 396 | | ---- | | ---- | |
| 445 | | ---- | | ---- | |
| 446 | INH-595 | 99.99 | | ---- | |
| 551 | INH-3214 | 99.986 | | ---- | |
| 874 | GOST9805 | 99.95 | DG(0.01) | ---- | |
| 913 | D3545 | 99.988 | | ---- | |
| 994 | INH-15-12 | 99.9727 | | ---- | |
| 1016 | DIN55685 | 99.99 | | ---- | |
| 1438 | | 99.98 | | ---- | |
| 2458 | | ---- | | ---- | |
| 6281 | | 99.9949 | | ---- | |
| normality | | OK | | | |
| n | | 10 | | | |
| outliers | | 2 | | | |
| mean (n) | | 99.9847 | | | |
| st.dev. (n) | | 0.00653 | | | |
| R(calc.) | | 0.0183 | | | |
| st.dev.(lit.) | | n.a. | | | |
| R(lit.) | | n.a. | | | |



Determination of Ethanol on sample #23270; results in mg/kg.

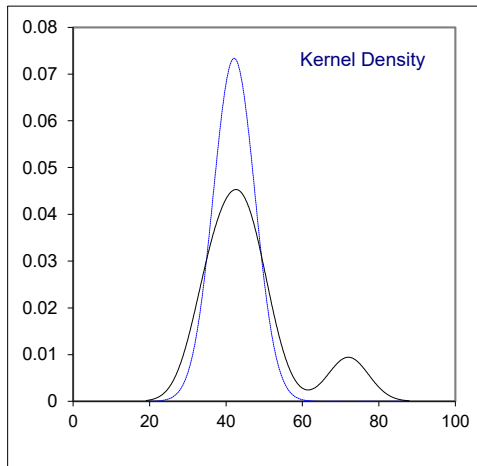
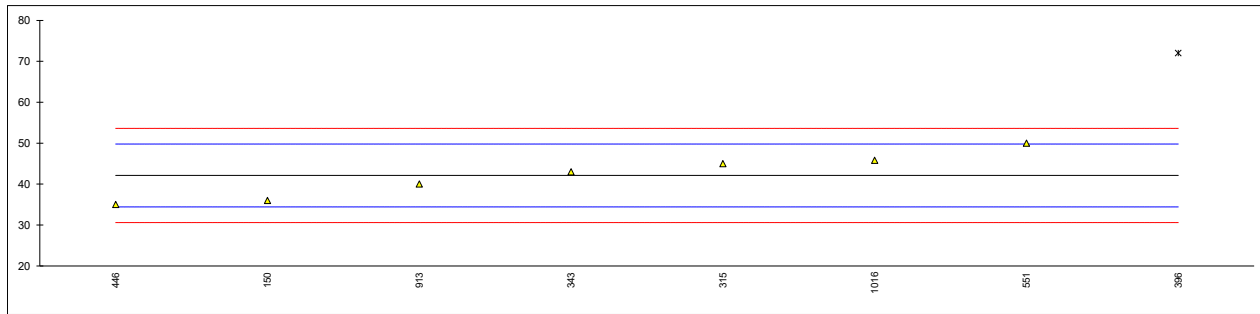
| lab | method | value | mark | z(targ) | remarks |
|------------------|-------------|---------|------|---------|--|
| 150 | BRCP5290 | 58 | | ---- | |
| 171 | INH-67-63-0 | 100 | C | ---- | first reported 0.01 mg/kg |
| 173 | | ---- | | ---- | |
| 315 | INH-082 | 65 | | ---- | |
| 323 | INH-060 | <100 | C | ---- | first reported <0.01 mg/kg |
| 343 | DIN55685 | 58 | | ---- | |
| 396 | | ---- | | ---- | |
| 445 | | ---- | | ---- | |
| 446 | INH-595 | <10 | | ---- | possibly a false negative test result? |
| 551 | INH-3214 | 53 | | ---- | |
| 874 | | ---- | | ---- | |
| 913 | D3545 | 40 | | ---- | |
| 994 | | ---- | | ---- | |
| 1016 | | ---- | | ---- | |
| 1438 | | ---- | | ---- | |
| 2458 | | ---- | | ---- | |
| 6281 | | ---- | | ---- | |
| normality | | unknown | | | |
| n | | 6 | | | |
| outliers | | 0 | | | |
| mean (n) | | 62.33 | | | |
| st.dev. (n) | | 20.245 | | | |
| R(calc.) | | 56.69 | | | |
| st.dev.(Horwitz) | | (5.354) | | | |
| R(Horwitz) | | (14.99) | | | |



Determination of n-Propanol on sample #23270; results in mg/kg.

| lab | method | value | mark | z(targ) | remarks |
|------|-------------|-------|---------|---------|----------------------------|
| 150 | BRCP5290 | 36 | | -1.59 | |
| 171 | INH-67-63-0 | <100 | C | ---- | first reported <0.01 mg/kg |
| 173 | | ---- | | ---- | |
| 315 | INH-082 | 45 | | 0.75 | |
| 323 | INH-060 | <100 | C | ---- | first reported <0.01 mg/kg |
| 343 | DIN55685 | 43 | | 0.23 | |
| 396 | INH-595 | 72 | G(0.05) | 7.79 | |
| 445 | | ---- | | ---- | |
| 446 | INH-595 | 35 | | -1.85 | |
| 551 | INH-3214 | 50 | | 2.05 | |
| 874 | | ---- | | ---- | |
| 913 | D3545 | 40 | | -0.55 | |
| 994 | | ---- | | ---- | |
| 1016 | DIN55685 | 45.8 | | 0.96 | |
| 1438 | | ---- | | ---- | |
| 2458 | | ---- | | ---- | |
| 6281 | | ---- | | ---- | |

normality unknown
n 7
outliers 1
mean (n) 42.11
st.dev. (n) 5.437
R(calc.) 15.22
st.dev.(Horwitz) 3.838
R(Horwitz) 10.75



Determination of n-Butanol on sample #23270; results in mg/kg.

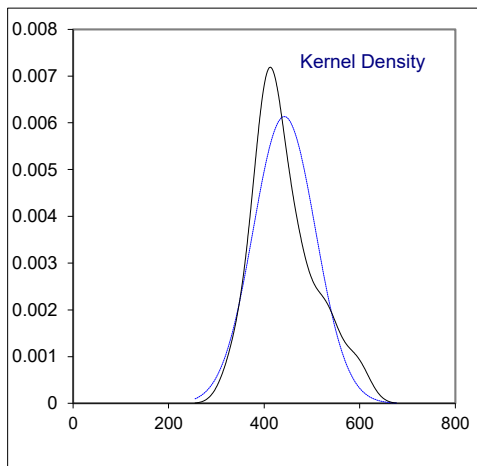
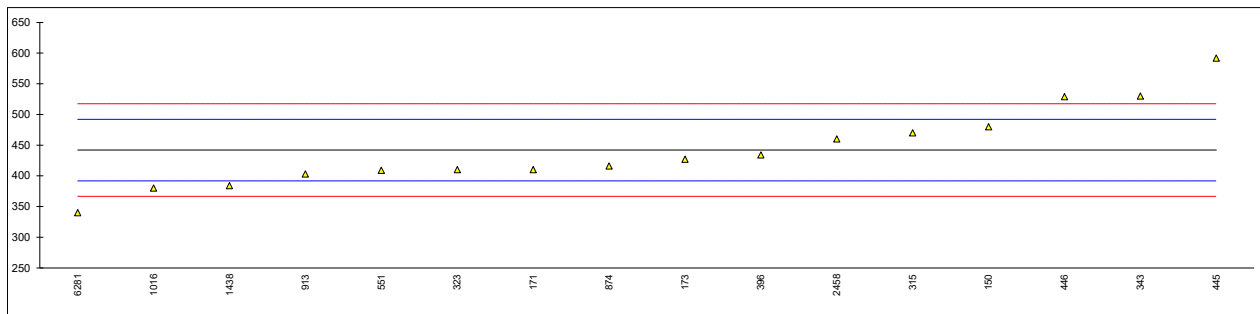
| lab | method | value | mark | z(targ) | remarks |
|----------|-------------|-------|------|---------|----------------------------|
| 150 | | ---- | | ---- | |
| 171 | INH-67-63-0 | <100 | C | ---- | first reported <0.01 mg/kg |
| 173 | | ---- | | ---- | |
| 315 | INH-082 | <5 | | ---- | |
| 323 | INH-060 | <100 | C | ---- | first reported <0.01 mg/kg |
| 343 | DIN55685 | 0 | | ---- | |
| 396 | INH-595 | 3 | | ---- | |
| 445 | | ---- | | ---- | |
| 446 | INH-595 | <10 | | ---- | |
| 551 | INH-3214 | <10 | | ---- | |
| 874 | | ---- | | ---- | |
| 913 | D3545 | <5 | | ---- | |
| 994 | | ---- | | ---- | |
| 1016 | DIN55685 | 0 | | ---- | |
| 1438 | | ---- | | ---- | |
| 2458 | | ---- | | ---- | |
| 6281 | | ---- | | ---- | |
| n | | 7 | | | |
| mean (n) | | <10 | | | |

Determination of Other impurities on sample #23270; results in mg/kg.

| lab | method | value | mark | z(targ) | remarks |
|------|----------|-------|------|---------|----------------------------|
| 150 | | ---- | | ---- | |
| 171 | | ---- | | ---- | |
| 173 | | ---- | | ---- | |
| 315 | | ---- | | ---- | |
| 323 | INH-060 | <100 | C | ---- | first reported <0.01 mg/kg |
| 343 | DIN55685 | 30 | | ---- | |
| 396 | | ---- | | ---- | |
| 445 | | ---- | | ---- | |
| 446 | INH-595 | <50 | | ---- | |
| 551 | INH-3214 | 39 | | ---- | |
| 874 | | ---- | | ---- | |
| 913 | | ---- | | ---- | |
| 994 | | ---- | | ---- | |
| 1016 | | ---- | | ---- | |
| 1438 | | ---- | | ---- | |
| 2458 | | ---- | | ---- | |
| 6281 | | ---- | | ---- | |
| | n | 3 | | | |
| | mean (n) | <50 | | | |

Determination of Water on sample #23270; results in mg/kg.

| lab | method | value | mark | z(targ) | remarks |
|-------------------|----------|--------|------|---------|----------------------------|
| 150 | E1064 | 480 | | 1.51 | |
| 171 | E1064 | 410 | | -1.28 | |
| 173 | E1064 | 427 | | -0.60 | |
| 315 | E1064 | 470 | | 1.11 | |
| 323 | E1064 | 410 | | -1.28 | |
| 343 | E1064 | 530 | C | 3.50 | first reported 0.053 mg/kg |
| 396 | D1364 | 434 | | -0.32 | |
| 445 | E1064 | 591.7 | | 5.96 | |
| 446 | D1364 | 529 | | 3.46 | |
| 551 | E1064 | 409 | | -1.32 | |
| 874 | E1064 | 416.04 | | -1.04 | |
| 913 | E1064 | 403 | | -1.56 | |
| 994 | | ----- | | ----- | |
| 1016 | D1364 | 380 | | -2.47 | |
| 1438 | E1064 | 384 | | -2.31 | |
| 2458 | ISO13267 | 460 | | 0.71 | |
| 6281 | D1364 | 340 | | -4.07 | |
| normality | | OK | | | |
| n | | 16 | | | |
| outliers | | 0 | | | |
| mean (n) | | 442.11 | | | |
| st.dev. (n) | | 65.068 | | | |
| R(calc.) | | 182.19 | | | |
| st.dev.(E1064:24) | | 25.105 | | | |
| R(E1064:24) | | 70.30 | | | |



APPENDIX 2

Number of participants per country

1 lab in AZERBAIJAN
1 lab in BELGIUM
1 lab in BRAZIL
1 lab in GERMANY
1 lab in INDIA
1 lab in ISRAEL
1 lab in ITALY
2 labs in NETHERLANDS
1 lab in RUSSIAN FEDERATION
1 lab in SPAIN
1 lab in THAILAND
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 | = 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

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- 4 ISO13528:05
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- 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)

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