# Results of Proficiency Test
# Vinyl Acetate Monomer (VAM)
# February 2021

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

**Author:** Mrs. E.R. Montenij-Bos
**Correctors:** ing. A.S. Noordman-de Neef & ing. R.J. Starink
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**CONTENTS**

# 1 INTRODUCTION

Since 2007 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the analysis of Vinyl Acetate Monomer (VAM) in accordance with the latest version of the specification ASTM D2190 every year. During the annual proficiency testing program 2020/2021 it was decided to continue the round robin for the analysis of Vinyl Acetate Monomer (VAM).

In this interlaboratory study 22 laboratories in 17 different countries registered for participation. See appendix 2 for the number of participants per country.
In this report the results of the Vinyl Acetate Monomer (VAM) 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 sample of 0.5 liter Vinyl Acetate Monomer (VAM) labelled #21001.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 a 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 25 liters of Vinyl Acetate Monomer (VAM) was obtained from a participating laboratory. After homogenization 48 amber glass bottles of 0.5 liter were filled and labelled #21001.
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 #21001-1 | 0.93216 |
| sample #21001-2 | 0.93216 |
| sample #21001-3 | 0.93216 |
| sample #21001-4 | 0.93215 |
| sample #21001-5 | 0.93215 |
| sample #21001-6 | 0.93215 |
| sample #21001-7 | 0.93215 |
| sample #21001-8 | 0.93216 |

Table 1: homogeneity test results of subsamples #21001

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) | 0.00001 |
| reference test method | ISO12185:96 |
| 0.3 x R (reference test method) | 0.00015 |

Table 2: evaluation of the repeatability of subsamples #21001

The calculated repeatability was 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 bottle of 0.5 liter Vinyl Acetate Monomer (VAM) labelled #21001 was sent on January 13, 2021. An SDS was added to the sample package.

## 2.5 STABILITY OF THE SAMPLES

The stability of Vinyl Acetate Monomer (VAM) 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 without Nitrogen purge, Apparent Specific Gravity 20/20°C, Color Pt/Co, Density at 20°C, Distillation (IBP, 50% recovered, Dry Point, Distillation Range), Inhibitor as Hydroquinone, Purity by GC and Impurities (Acetaldehyde, Acetone, Ethyl Acetate, Methyl Acetate) 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 reanalysis). Additional or corrected test results are used for data analysis and the 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.

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 ISO 13528 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 test 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, 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, like Horwitz

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

Some problems were encountered with the dispatch of the samples due to COVID-19 pandemic. Therefore, the reporting time on the data entry portal was extended with one week. Two participants did not report any test results at all and two participants reported test results after the extended final reporting date. Not all participants were able to report test results for all tests requested.
In total 20 participants reported 225 numerical test results. Observed were 11 outlying test results, which is 4.9%. In proficiency studies, 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. The abbreviations, used in these tables, are explained in appendix 3.

In the iis PT reports, ASTM test methods are referred to with a number and if appropriate an indication of sub test method (e.g. D1209) and an added designation for the year that the test 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 test result tables of appendix 1 only the test method number (sub) and year of adoption or revision (e.g. D1209:05) will be used.

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

**Sample #21001**

Acidity without Nitrogen purge: 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 D2086:08(2012). Test method is withdrawn with no replacement in 2021.

Apparent Specific Gravity 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.

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. 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 for all distillation parameters; IBP, 50% recovered, Dry Point and Distillation Range. No statistical outliers were observed. The calculated reproducibilities are in good agreement with the requirements of ASTM D1078:11(2019) (automated and manual mode).

Inhibitor as Hydroquinone: 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 ASTM D2193:06(2012). Test method is withdrawn with no replacement in 2021.

Purity by GC: Regretfully, no reference test method with precision data exists for this determination. Therefore, no z-scores were calculated. No statistical outliers were observed. The calculated reproducibility (0.0232) was in line with the previous PT (0.0247 in iis20C01).

Acetaldehyde: This determination was not problematic. Three statistical outliers were observed. The calculated reproducibility after rejection of the statistical outliers is in agreement with the estimated reproducibility calculated with the Horwitz equation.

Acetone: This determination was not problematic. Most participants agreed on a test result less than 10 mg/kg. Therefore, no z-scores were calculated.

Ethyl Acetate: 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 estimated reproducibility calculated with the Horwitz equation.

Methyl Acetate: 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 with the Horwitz equation.

Water: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in good agreement with the requirements of ASTM D1364:02(2012).

### 4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method or as declared by the estimated target reproducibility calculated with the Horwitz equation 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 literature reference test methods (in casu ASTM, EN and ISO test methods) or estimated using the Horwitz equation are presented in the next table.

| Parameter | unit | n | average | 2.8 * sd | R(lit) |
|---|---|---|---|---|---|
| Acidity without $N_2$ purge | mg/kg | 13 | 28.1 | 11.5 | 6 |
| Apparent Specific Gravity 20/20°C | | 19 | 0.9339 | 0.0002 | 0.0005 |
| Color Pt/Co | | 15 | 3.2 | 3.6 | 7 |
| Density at 20°C | kg/L | 17 | 0.9322 | 0.0001 | 0.0005 |
| Initial Boiling Point | °C | 16 | 72.4 | 0.3 | 1.1 |
| 50% recovery | °C | 16 | 72.7 | 0.3 | 0.5 |
| Dry Point | °C | 16 | 72.8 | 0.3 | 0.8 |
| Range | °C | 16 | 0.4 | 0.3 | 0.7 |

| Parameter | unit | n | average | 2.8 * sd | R(lit) |
|---|---|---|---|---|---|
| Inhibitor as Hydroquinone | mg/kg | 18 | 2.6 | 0.5 | 1.0 |
| Purity by GC | %M/M | 18 | 99.972 | 0.023 | n.a. |
| Acetaldehyde | mg/kg | 12 | 21.7 | 5.7 | 6.1 |
| Acetone | mg/kg | 11 | <10 | n.e. | n.e. |
| Ethyl Acetate | mg/kg | 12 | 163 | 56 | 34 |
| Methyl Acetate | mg/kg | 9 | 10.4 | 5.4 | 3.3 |
| Water | mg/kg | 17 | 105 | 42 | 61 |

Table 3: reproducibilities of tests on sample #21001

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 EVALUATION OF THE PROFICIENCY TEST OF FEBRUARY 2021 WITH PREVIOUS PTS

| | February 2021 | February 2020 | February 2019 | February 2018 | February 2017 |
|---|---|---|---|---|---|
| Number of reporting laboratories | 22 | 20 | 27 | 25 | 23 |
| Number of test results | 225 | 214 | 247 | 282 | 266 |
| Number of statistical outliers | 11 | 10 | 11 | 10 | 8 |
| Percentage statistical outliers | 4.9% | 4.7% | 4.5% | 3.5% | 3.0% |

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

| Parameter | February 2021 | February 2020 | February 2019 | February 2018 | February 2017 |
|---|---|---|---|---|---|
| Acidity without $N_2$ purge | -- | -- | (--) | - | -- |
| Apparent Specific Gravity 20/20°C | ++ | ++ | + | ++ | ++ |
| Color Pt/Co | ++ | ++ | + | ++ | ++ |
| Density at 20°C | ++ | ++ | + | ++ | ++ |
| Distillation | ++ | ++ | +/- | ++ | ++ |
| Inhibitor as Hydroquinone | + | -- | (--) | - | +/- |
| Purity by GC | + | ++ | -- | ++ | -- |
| Acetaldehyde | + | - | - | -- | - |
| Acetone | n.e. | n.e. | n.e. | n.e. | - |
| Ethyl Acetate | - | - | - | ++ | + |
| Methyl Acetate | - | - | - | + | - |
| Water | + | + | + | + | ++ |

Table 5: comparison determinations against the reference test methods
Results between brackets should be used with due care

In the table above the following performance categories were used:

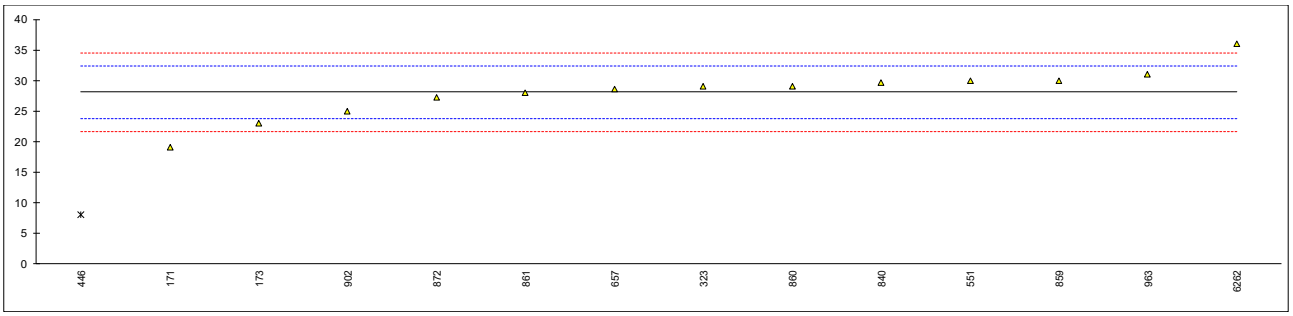
- ++ : group performed much better than the reference test method
- + : group performed better than the reference test method
- +/- : group performance equals the reference test method
- - : group performed worse than the reference test method
- -- : group performed much worse than the reference test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

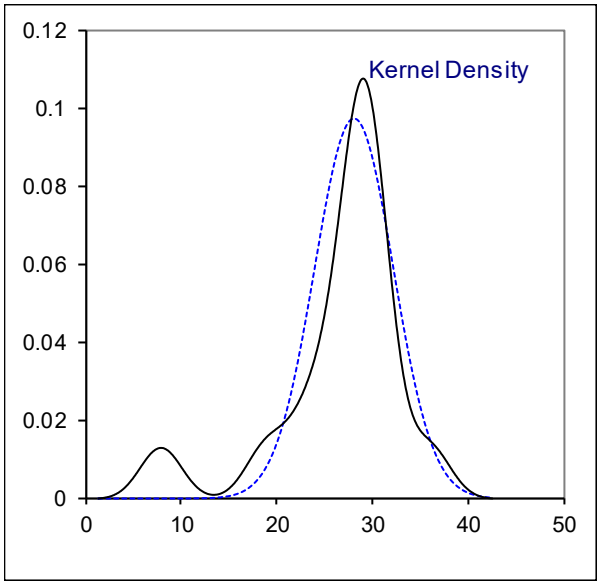
**APPENDIX 1**

Determination of Acidity without $N_2$ purge on sample #21001; results in mg/kg

| lab | method | value | mark | z(targ) | Remarks |
|---|---|---|---|---|---|
| 171 | D2086 | 19 | | -4.25 | |
| 173 | INH-44 | 23 | | -2.39 | |
| 273 | | ----- | | ----- | |
| 323 | D2086 | 29 | | 0.41 | |
| 347 | | ----- | | ----- | |
| 391 | | ----- | | ----- | |
| 446 | D2086 | 8 | G(0.01) | -9.39 | |
| 522 | | ----- | | ----- | |
| 541 | | ----- | | ----- | |
| 551 | D2086 | 30 | | 0.88 | |
| 657 | D2086 | 28.6 | | 0.23 | |
| 840 | D2086 | 29.7 | | 0.74 | |
| 859 | D2086 | 30 | | 0.88 | |
| 860 | D2086 | 29 | | 0.41 | |
| 861 | D2086 | 28 | | -0.05 | |
| 872 | D2086 | 27.2 | | -0.43 | |
| 902 | D2086 | 25 | C | -1.45 | first reported 17 |
| 913 | | ----- | | ----- | |
| 963 | D2086 | 31 | | 1.35 | |
| 974 | | ----- | | ----- | |
| 1429 | | ----- | | ----- | |
| 6262 | D2086 | 36 | | 3.68 | |

| | |
|---|---|
| normality | suspect |
| n | 13 |
| outliers | 1 |
| mean (n) | 28.12 |
| st.dev. (n) | 4.100 |
| R(calc.) | 11.48 |
| st.dev.(D2086:08) | 2.143 |
| R(D2086:08) | 6 |

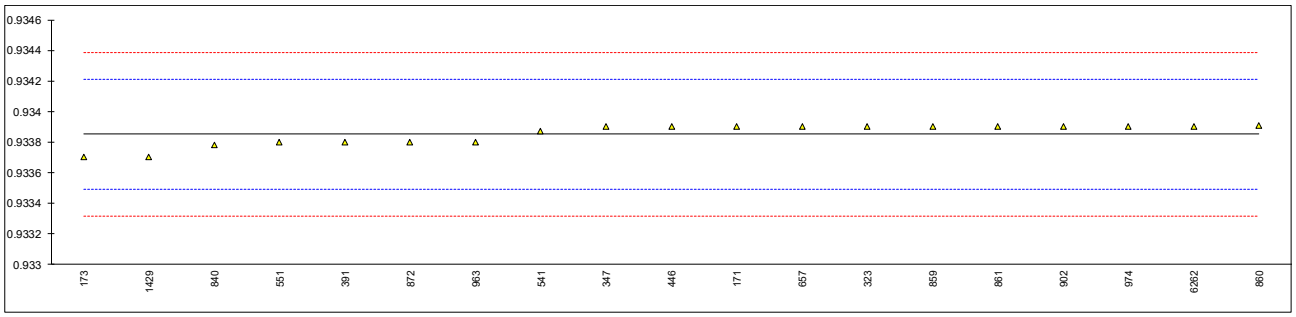


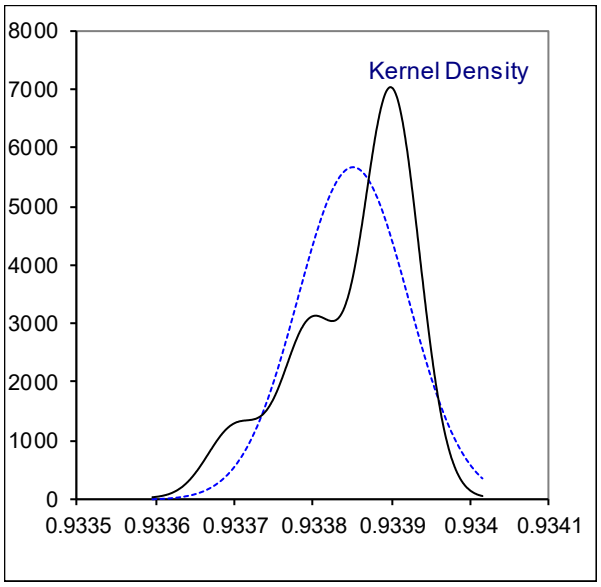



## Determination of Apparent Specific Gravity 20/20°C on sample #21001;

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 171 | D4052 | 0.9339 | | 0.28 | |
| 173 | D4052 | 0.9337 | | -0.84 | |
| 273 | | ----- | | ----- | |
| 323 | D4052 | 0.9339 | | 0.28 | |
| 347 | D4052 | 0.9339 | | 0.28 | |
| 391 | ISO12185 | 0.9338 | | -0.28 | |
| 446 | D4052 | 0.9339 | C | 0.28 | first reported 0.933 |
| 522 | | ----- | | ----- | |
| 541 | D4052 | 0.93387 | | 0.11 | |
| 551 | D4052 | 0.9338 | | -0.28 | |
| 657 | D4052 | 0.93390 | | 0.28 | |
| 840 | D4052 | 0.93378 | | -0.39 | |
| 859 | D4052 | 0.9339 | | 0.28 | |
| 860 | D4052 | 0.93391 | | 0.33 | |
| 861 | D4052 | 0.9339 | | 0.28 | |
| 872 | D4052 | 0.9338 | | -0.28 | |
| 902 | D4052 | 0.9339 | | 0.28 | |
| 913 | | ----- | | ----- | |
| 963 | ISO12185 | 0.9338 | | -0.28 | |
| 974 | D4052 | 0.9339 | | 0.28 | |
| 1429 | | 0.9337 | | -0.84 | |
| 6262 | D4052 | 0.9339 | | 0.28 | |

| | |
|---|---|
| normality | OK |
| n | 19 |
| outliers | 0 |
| mean (n) | 0.93385 |
| st.dev. (n) | 0.000070 |
| R(calc.) | 0.00020 |
| st.dev.(ISO12185:96) | 0.000179 |
| R(ISO12185:96) | 0.0005 |

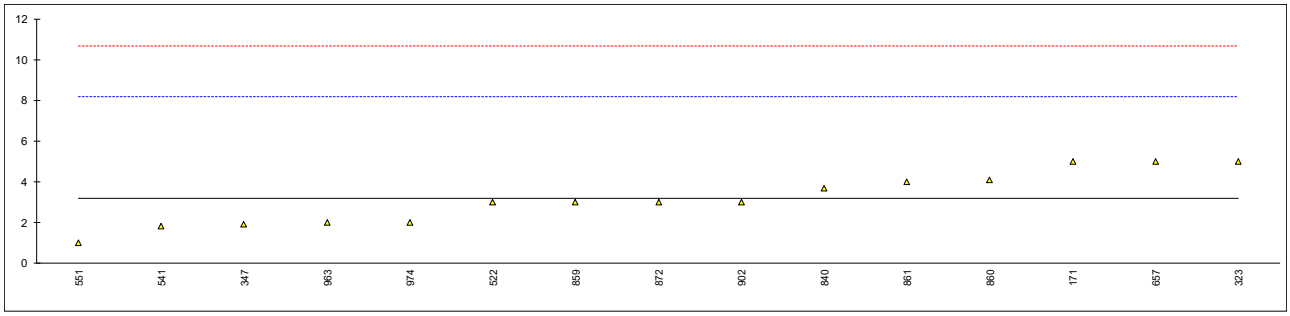


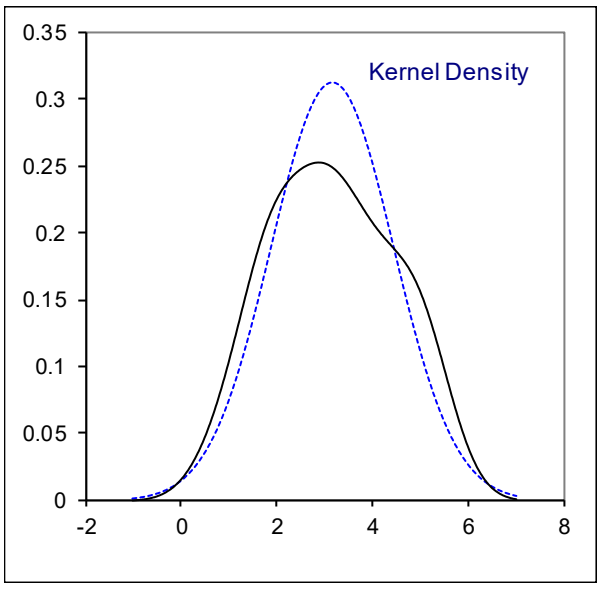



## Determination of Color Pt/Co on sample #21001;

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 171 | D1209 | 5 | | 0.73 | |
| 173 | D1209 | <5 | | ----- | |
| 273 | | ----- | | ----- | |
| 323 | D5386 | 5 | | 0.73 | |
| 347 | D5386 | 1.9 | | -0.51 | |
| 391 | D1209 | <5 | | ----- | |
| 446 | D1209 | <5 | | ----- | |
| 522 | | 3.0 | | -0.07 | |
| 541 | D5386 | 1.8 | | -0.55 | |
| 551 | D1209 | 1 | | -0.87 | |
| 657 | D1209 | 5 | | 0.73 | |
| 840 | D5386 | 3.7 | | 0.21 | |
| 859 | D1209 | 3 | | -0.07 | |
| 860 | D5386 | 4.1 | | 0.37 | |
| 861 | D5386 | 4 | | 0.33 | |
| 872 | D1209 | 3 | | -0.07 | |
| 902 | D5386 | 3 | | -0.07 | |
| 913 | | ----- | | ----- | |
| 963 | D1209 | 2 | | -0.47 | |
| 974 | D5386 | 2 | | -0.47 | |
| 1429 | | <5 | | ----- | |
| 6262 | D1209 | <5 | | ----- | |

| | |
|---|---|
| normality | OK |
| n | 15 |
| outliers | 0 |
| mean (n) | 3.17 |
| st.dev. (n) | 1.280 |
| R(calc.) | 3.58 |
| st.dev.(D1209:05) | 2.500 |
| R(D1209:05) | 7 |

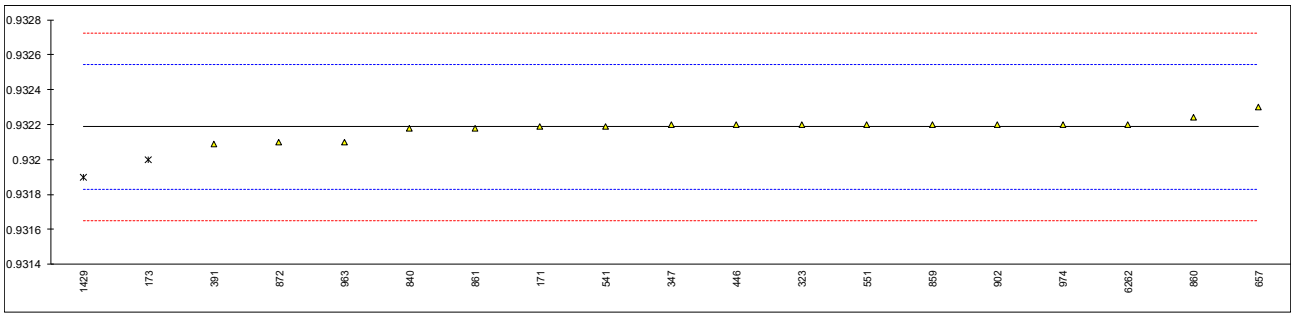


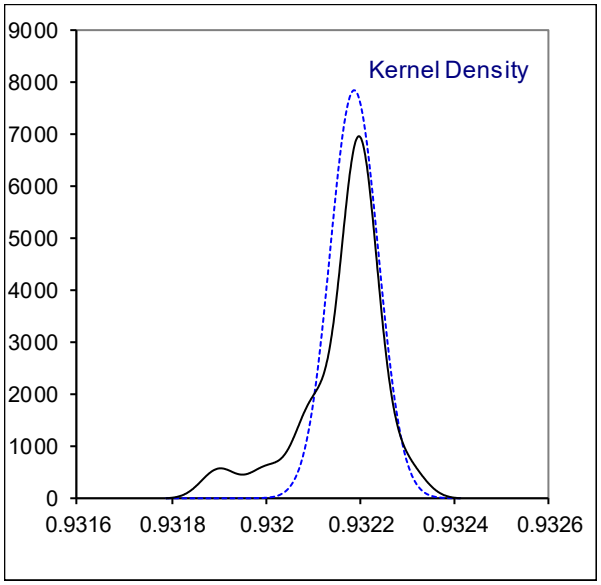



## Determination of Density at 20°C on sample #21001; results in kg/L

| lab | Method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 171 | D4052 | 0.93219 | | 0.02 | |
| 173 | D4052 | 0.9320 | G(0.05) | -1.04 | |
| 273 | | ----- | | ----- | |
| 323 | D4052 | 0.9322 | | 0.08 | |
| 347 | D4052 | 0.9322 | | 0.08 | |
| 391 | ISO12185 | 0.93209 | | -0.54 | |
| 446 | D4052 | 0.9322 | C | 0.08 | first reported 0.9313 |
| 522 | | ----- | | ----- | |
| 541 | D4052 | 0.93219 | | 0.02 | |
| 551 | D4052 | 0.9322 | | 0.08 | |
| 657 | D4052 | 0.9323 | | 0.64 | |
| 840 | D4052 | 0.93218 | | -0.04 | |
| 859 | D4052 | 0.9322 | | 0.08 | |
| 860 | D4052 | 0.93224 | | 0.30 | |
| 861 | D4052 | 0.93218 | | -0.04 | |
| 872 | D4052 | 0.9321 | | -0.48 | |
| 902 | D4052 | 0.9322 | | 0.08 | |
| 913 | | ----- | | ----- | |
| 963 | ISO12185 | 0.9321 | | -0.48 | |
| 974 | D4052 | 0.9322 | | 0.08 | |
| 1429 | | 0.9319 | G(0.05) | -1.60 | |
| 6262 | D4052 | 0.9322 | | 0.08 | |

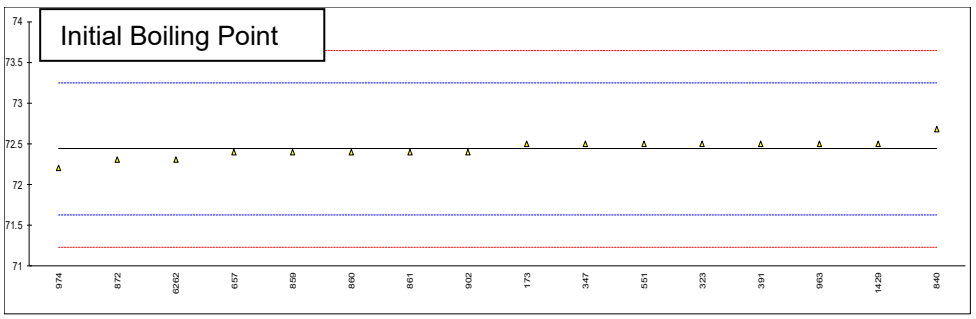
| | |
|---|---|
| Normality | suspect |
| N | 17 |
| Outliers | 2 |
| mean (n) | 0.93219 |
| st.dev. (n) | 0.000051 |
| R(calc.) | 0.00014 |
| st.dev.(ISO12185:96) | 0.000179 |
| R(ISO12185:96) | 0.0005 |

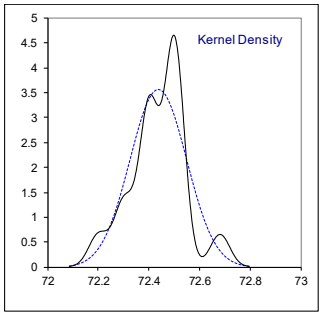


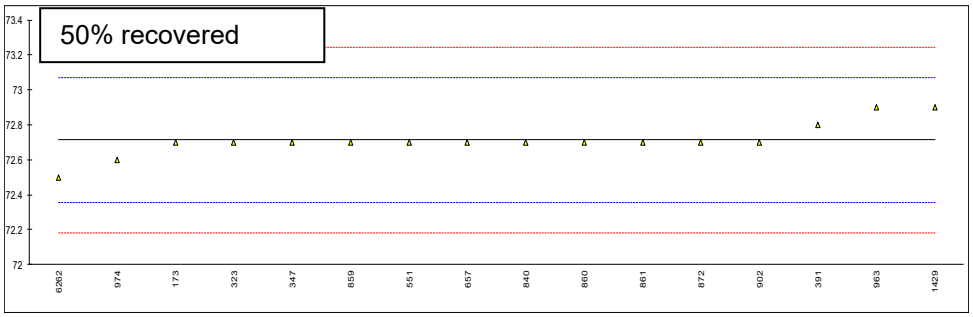



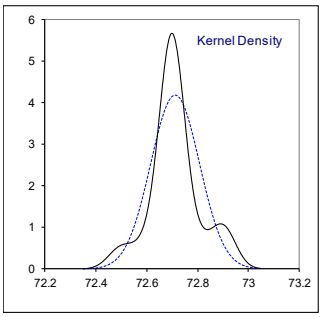
## Determination of Distillation on sample #21001; results in °C

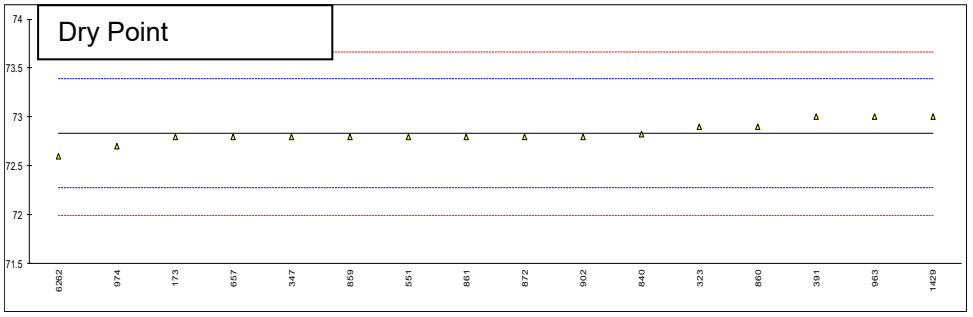
| lab | method | IBP | mark | z(targ) | 50%rec | mark | z(targ) | DP | mark | z(targ) | range | mark | z(targ) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 171 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 173 | D1078-A | 72.5 | | 0.16 | 72.7 | | -0.07 | 72.8 | | -0.12 | 0.3 | | -0.39 |
| 273 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 323 | D1078-A | 72.5 | | 0.16 | 72.7 | | -0.07 | 72.9 | | 0.24 | 0.4 | | 0.02 |
| 347 | D1078-A | 72.5 | | 0.16 | 72.7 | | -0.07 | 72.8 | | -0.12 | 0.3 | | -0.39 |
| 391 | D1078-A | 72.5 | | 0.16 | 72.8 | | 0.49 | 73.0 | | 0.60 | 0.5 | | 0.42 |
| 446 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 522 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 541 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 551 | D1078 | 72.5 | | 0.16 | 72.7 | | -0.07 | 72.8 | | -0.12 | 0.3 | | -0.39 |
| 657 | D1078-M | 72.4 | | -0.09 | 72.7 | | -0.07 | 72.8 | | -0.12 | 0.4 | | 0.02 |
| 840 | D1078-A | 72.68 | | 0.60 | 72.70 | | -0.07 | 72.82 | | -0.04 | 0.14 | | -1.03 |
| 859 | D1078-M | 72.4 | | -0.09 | 72.7 | | -0.07 | 72.8 | | -0.12 | 0.4 | | 0.02 |
| 860 | D1078-M | 72.4 | | -0.09 | 72.7 | | -0.07 | 72.9 | | 0.24 | 0.5 | | 0.42 |
| 861 | D1078-M | 72.4 | | -0.09 | 72.7 | | -0.07 | 72.8 | | -0.12 | 0.4 | | 0.02 |
| 872 | D1078-M | 72.3 | | -0.34 | 72.7 | | -0.07 | 72.8 | | -0.12 | 0.5 | | 0.42 |
| 902 | D1078-A | 72.4 | | -0.09 | 72.7 | | -0.07 | 72.8 | | -0.12 | 0.4 | | 0.02 |
| 913 | | ----- | | ----- | ----- | | ----- | ----- | | ----- | ----- | | ----- |
| 963 | D1078-A | 72.5 | | 0.16 | 72.9 | | 1.06 | 73.0 | | 0.60 | 0.5 | | 0.42 |
| 974 | D1078-A | 72.2 | | -0.59 | 72.6 | | -0.63 | 72.7 | | -0.48 | 0.5 | | 0.42 |
| 1429 | | 72.5 | | 0.16 | 72.9 | | 1.06 | 73.0 | | 0.60 | 0.5 | | 0.42 |
| 6262 | D1078-A | 72.3 | | -0.34 | 72.5 | | -1.20 | 72.6 | | -0.84 | 0.3 | | -0.39 |
| | | | | | | | | | | | | | |
| normality | | suspect | | | suspect | | | OK | | | OK | | |
| n | | 16 | | | 16 | | | 16 | | | 16 | | |
| outliers | | 0 | | | 0 | | | 0 | | | 0 | | |
| mean (n) | | 72.44 | | | 72.71 | | | 72.83 | | | 0.40 | | |
| st.dev. (n) | | 0.117 | | | 0.096 | | | 0.108 | | | 0.106 | | |
| R(calc.) | | 0.31 | | | 0.27 | | | 0.30 | | | 0.30 | | |
| st.dev.(D1078-A:11) | | 0.404 | | | 0.177 | | | 0.278 | | | 0.249 | | |
| R(D1078-A:11) | | 1.13 | | | 0.50 | | | 0.78 | | | 0.70 | | |
| Compare | | | | | | | | | | | | | |
| R(D1078-M:11) | | 0.78 | | | 0.47 | | | 0.95 | | | 0.63 | | |

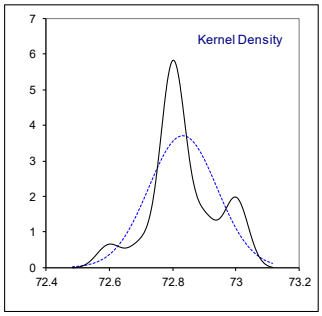

Initial Boiling Point
974 872 6262 657 859 860 861 902 173 347 551 323 391 963 1429 840

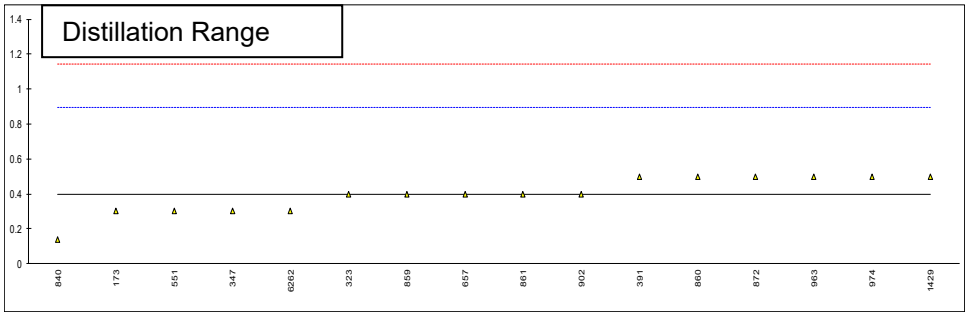

Kernel Density

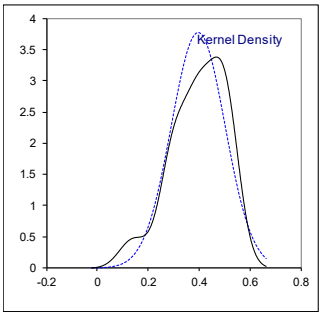

50% recovered
6262 974 173 323 347 859 551 657 840 860 861 872 902 391 963 1429


Kernel Density


Dry Point
6262 974 173 657 347 859 551 861 872 902 840 323 860 391 963 1429


Kernel Density

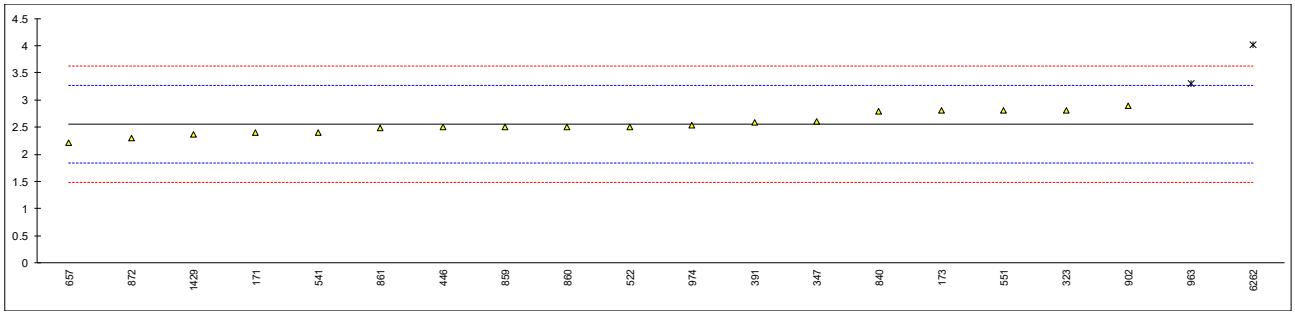

Distillation Range
840 173 551 347 6262 323 859 657 861 902 391 860 872 963 974 1429

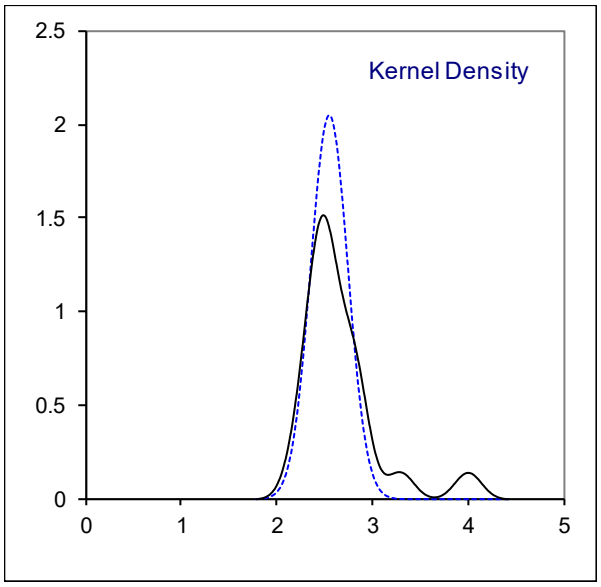

Kernel Density

## Determination of Inhibitor as Hydroquinone on sample #21001; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 171 | D2193 | 2.4 | | -0.43 | |
| 173 | D2193 | 2.8 | | 0.69 | |
| 273 | | ----- | | ----- | |
| 323 | D2193 | 2.8 | | 0.69 | |
| 347 | INH-097 | 2.6 | C | 0.13 | first reported 3.8 |
| 391 | D2193 | 2.58 | | 0.07 | |
| 446 | D2193 | 2.5 | | -0.15 | |
| 522 | | 2.51 | | -0.12 | |
| 541 | D2193 | 2.40 | | -0.43 | |
| 551 | D2193 | 2.8 | | 0.69 | |
| 657 | D2193 | 2.21 | | -0.96 | |
| 840 | D2193 | 2.79 | | 0.66 | |
| 859 | D2193 | 2.5 | | -0.15 | |
| 860 | D2193 | 2.5 | | -0.15 | |
| 861 | D2193 | 2.48 | | -0.21 | |
| 872 | D2193 | 2.3 | | -0.71 | |
| 902 | D2193 | 2.9 | C | 0.97 | first reported 2,,9 |
| 913 | | ----- | | ----- | |
| 963 | D2193 | 3.3 | G(0.05) | 2.09 | |
| 974 | D2193 | 2.54 | | -0.04 | |
| 1429 | | 2.36 | | -0.54 | |
| 6262 | D2193 | 4.01 | G(0.01) | 4.08 | |

| | |
|---|---|
| normality | OK |
| n | 18 |
| outliers | 2 |
| mean (n) | 2.55 |
| st.dev. (n) | 0.195 |
| R(calc.) | 0.54 |
| st.dev.(D2193:06) | 0.357 |
| R(D2193:06) | 1.0 |

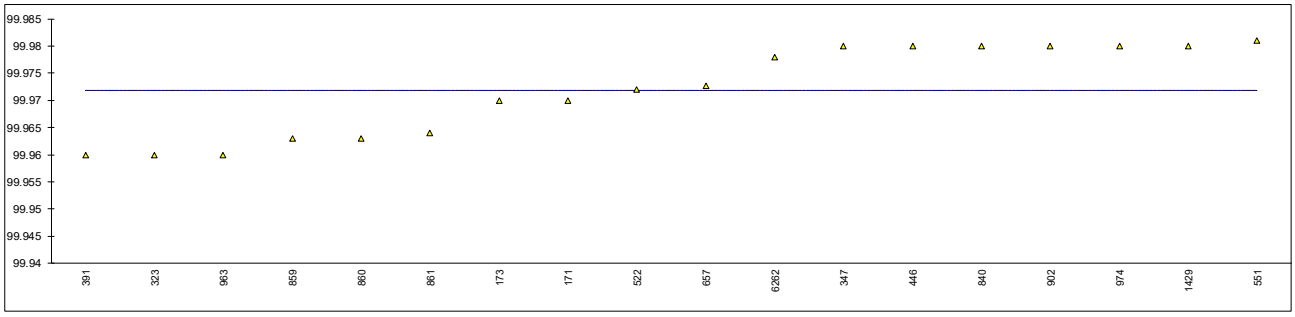


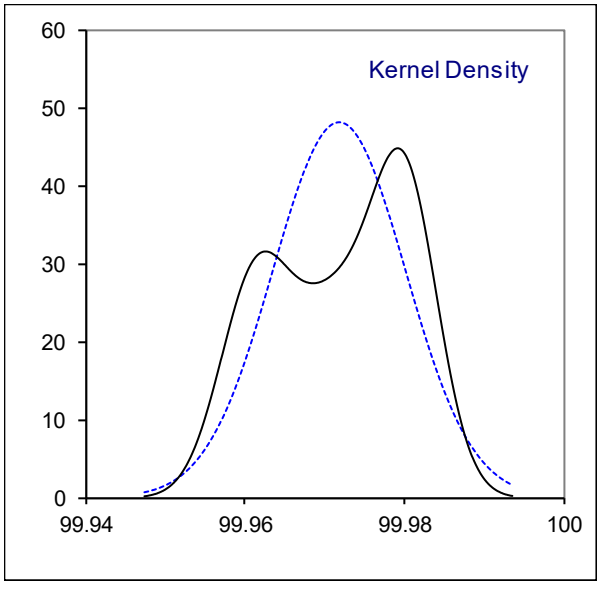



## Determination of Purity by GC on sample #21001; results in %M/M

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 171 | INH-001 | 99.97 | | ----- | |
| 173 | INH-257 | 99.97 | | ----- | |
| 273 | | ----- | | ----- | |
| 323 | INH-067 | 99.96 | | ----- | |
| 347 | INH-096 | 99.98 | | ----- | |
| 391 | INH-257 | 99.96 | | ----- | |
| 446 | INH-582 | 99.98 | | ----- | |
| 522 | DOWN102582 | 99.972 | | ----- | |
| 541 | | ----- | | ----- | |
| 551 | INH-1355 | 99.981 | | ----- | |
| 657 | INH-047 | 99.9727 | | ----- | |
| 840 | D3545 | 99.980 | | ----- | |
| 859 | INH-1628.2 | 99.963 | | ----- | |
| 860 | INH-1628.2 | 99.963 | | ----- | |
| 861 | INH-1628.2 | 99.964 | | ----- | |
| 872 | | ----- | | ----- | |
| 902 | INH-22 | 99.98 | | ----- | |
| 913 | | ----- | | ----- | |
| 963 | INH-8124 | 99.96 | | ----- | |
| 974 | INH-582 | 99.98 | | ----- | |
| 1429 | | 99.98 | | ----- | |
| 6262 | | 99.9779 | | ----- | |

| | | |
|---|---|---|
| | normality | OK |
| | n | 18 |
| | outliers | 0 |
| | mean (n) | 99.9719 |
| | st.dev. (n) | 0.00828 |
| | R(calc.) | 0.0232 |
| | st.dev.(lit) | n.a. |
| | R(lit) | n.a. |
| Compare | | |
| | R(iis20C01) | 0.0247 |

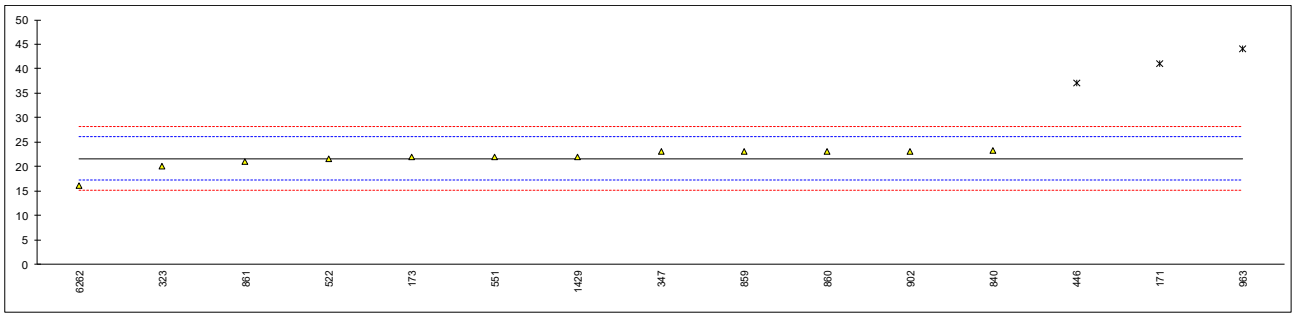


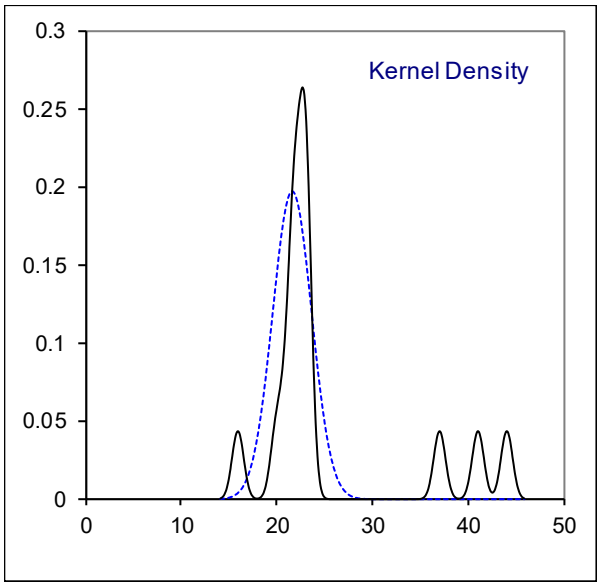



## Determination of Acetaldehyde on sample #21001; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 171 | INH-001 | 41 | DG(0.05) | 8.87 | |
| 173 | INH-47 | 22 | | 0.16 | |
| 273 | | ----- | | ----- | |
| 323 | INH-067 | 20 | | -0.76 | |
| 347 | INH-096 | 23 | | 0.62 | |
| 391 | | ----- | | ----- | |
| 446 | INH-582 | 37 | G(0.01) | 7.04 | |
| 522 | DOWN102582 | 21.64 | | -0.01 | |
| 541 | | ----- | | ----- | |
| 551 | INH-1355 | 22 | | 0.16 | |
| 657 | | ----- | | ----- | |
| 840 | D3545 | 23.2 | | 0.71 | |
| 859 | INH-1628.2 | 23 | | 0.62 | |
| 860 | INH-1628.2 | 23 | | 0.62 | |
| 861 | INH-1628.2 | 21 | | -0.30 | |
| 872 | | ----- | | ----- | |
| 902 | INH-22 | 23 | | 0.62 | |
| 913 | | ----- | | ----- | |
| 963 | INH-8124 | 44 | DG(0.05) | 10.25 | |
| 974 | | ----- | | ----- | |
| 1429 | | 22 | | 0.16 | |
| 6262 | | 16 | | -2.59 | |

| | |
|---|---|
| normality | not OK |
| n | 12 |
| outliers | 3 |
| mean (n) | 21.65 |
| st.dev. (n) | 2.025 |
| R(calc.) | 5.67 |
| st.dev.(Horwitz) | 2.181 |
| R(Horwitz) | 6.11 |



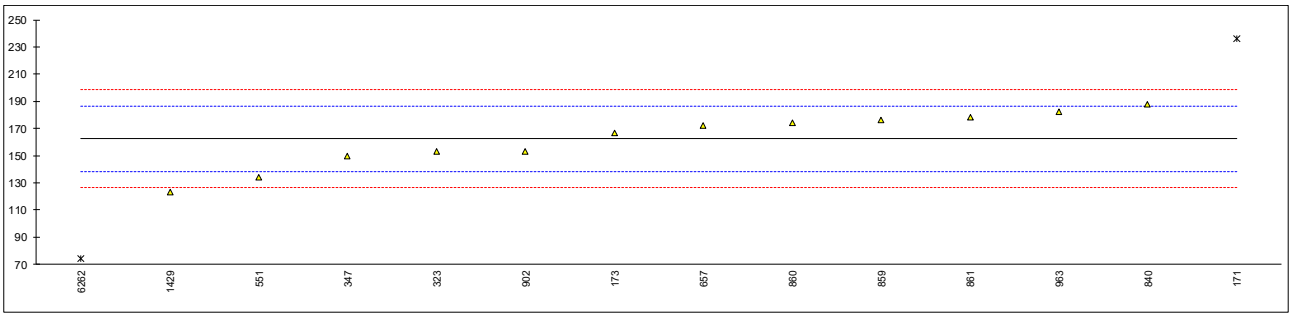
Determination of Acetone on sample #21001; results in mg/kg

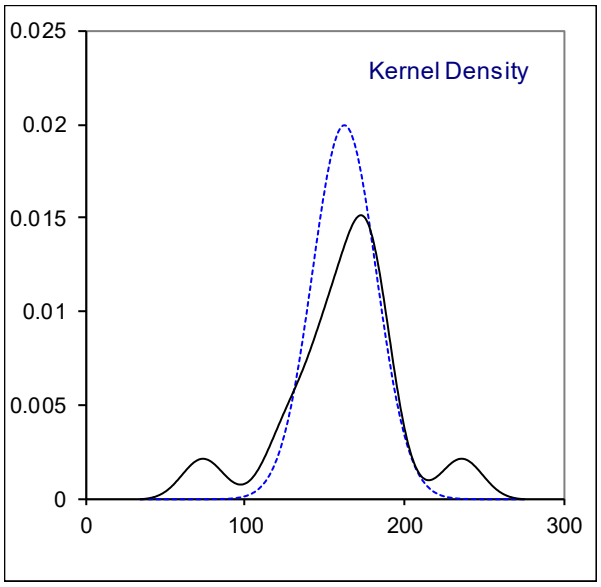
| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 171 | INH-001 | <10 | | ----- | |
| 173 | | ----- | | ----- | |
| 273 | | ----- | | ----- | |
| 323 | | <10 | | ----- | |
| 347 | | ----- | | ----- | |
| 391 | | ----- | | ----- | |
| 446 | | ----- | | ----- | |
| 522 | | ----- | | ----- | |
| 541 | | ----- | | ----- | |
| 551 | INH-1355 | <10 | | ----- | |
| 657 | INH-047 | < 10 | | ----- | |
| 840 | D3545 | <10 | | ----- | |
| 859 | INH-1628.2 | <10 | | ----- | |
| 860 | INH-1628.2 | <10 | | ----- | |
| 861 | INH-1628.2 | <10 | | ----- | |
| 872 | | ----- | | ----- | |
| 902 | INH-22 | <10 | | ----- | |
| 913 | | ----- | | ----- | |
| 963 | | ----- | | ----- | |
| 974 | | ----- | | ----- | |
| 1429 | | <1 | | ----- | |
| 6262 | | <10 | | ----- | |
| | n | 11 | | | |
| | mean (n) | <10 | | | |

## Determination of Ethyl Acetate on sample #21001; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 171 | INH-001 | 236 | DG(0.05) | 6.08 | |
| 173 | INH-47 | 167 | | 0.37 | |
| 273 | | ----- | | ----- | |
| 323 | INH-067 | 153 | | -0.79 | |
| 347 | INH-096 | 150 | | -1.04 | |
| 391 | | ----- | | ----- | |
| 446 | | ----- | | ----- | |
| 522 | | ----- | | ----- | |
| 541 | | ----- | | ----- | |
| 551 | INH-1355 | 134 | | -2.36 | |
| 657 | INH-047 | 172.4 | | 0.82 | |
| 840 | D3545 | 187.9 | | 2.10 | |
| 859 | INH-1628.2 | 176 | | 1.11 | |
| 860 | INH-1628.2 | 174 | | 0.95 | |
| 861 | INH-1628.2 | 178 | | 1.28 | |
| 872 | | ----- | | ----- | |
| 902 | INH-22 | 153 | | -0.79 | |
| 913 | | ----- | | ----- | |
| 963 | INH-8124 | 182 | | 1.61 | |
| 974 | | ----- | | ----- | |
| 1429 | | 123 | | -3.27 | |
| 6262 | | 74 | DG(0.05) | -7.32 | |

| | |
|---|---|
| normality | OK |
| n | 12 |
| outliers | 2 |
| mean (n) | 162.53 |
| st.dev. (n) | 20.017 |
| R(calc.) | 56.06 |
| st.dev.(Horwitz) | 12.085 |
| R(Horwitz) | 33.84 |

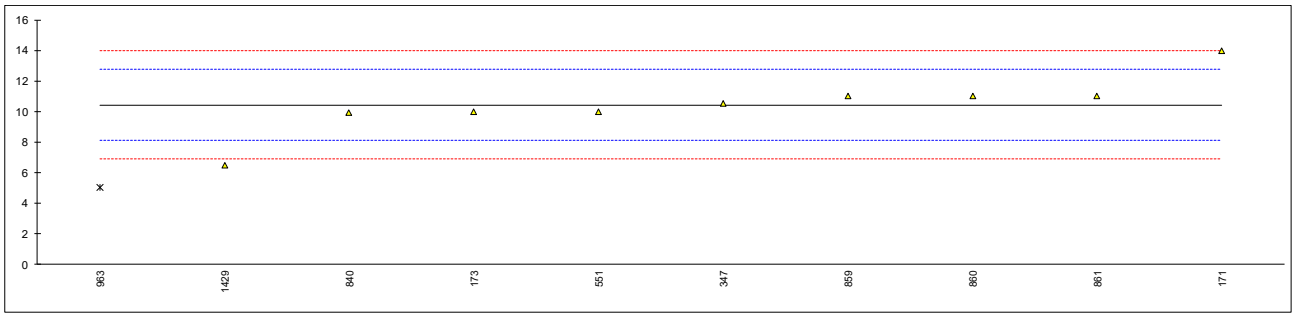


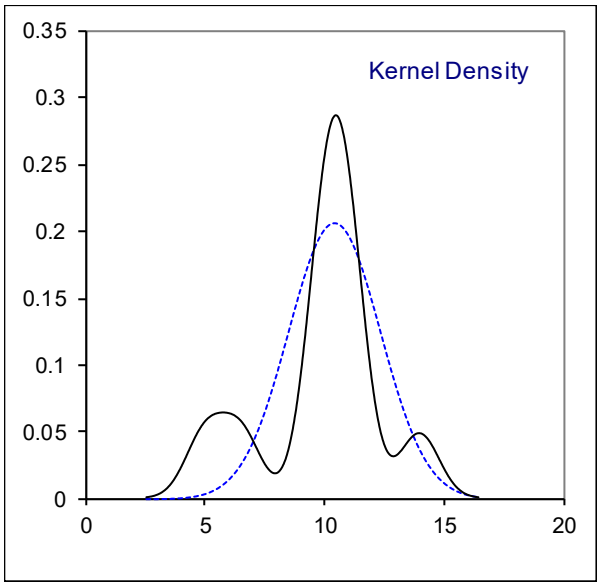



## Determination of Methyl Acetate on sample #21001; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 171 | INH-001 | 14 | | 3.04 | |
| 173 | INH-47 | 10 | | -0.37 | |
| 273 | | ----- | | ----- | |
| 323 | INH-067 | <10 | | ----- | |
| 347 | INH-096 | 10.5 | C | 0.06 | first reported 13 |
| 391 | | ----- | | ----- | |
| 446 | | ----- | | ----- | |
| 522 | | ----- | | ----- | |
| 541 | | ----- | | ----- | |
| 551 | INH-1355 | 10 | | -0.37 | |
| 657 | INH-047 | < 10 | | ----- | |
| 840 | D3545 | 9.9 | | -0.45 | |
| 859 | INH-1628.2 | 11 | | 0.48 | |
| 860 | INH-1628.2 | 11 | | 0.48 | |
| 861 | INH-1628.2 | 11 | | 0.48 | |
| 872 | | ----- | | ----- | |
| 902 | INH-22 | <10 | | ----- | |
| 913 | | ----- | | ----- | |
| 963 | INH-8124 | 5 | D(0.05) | -4.63 | |
| 974 | | ----- | | ----- | |
| 1429 | | 6.5 | | -3.35 | |
| 6262 | | <10 | | ----- | |

| | |
|---|---|
| normality | not OK |
| n | 9 |
| outliers | 1 |
| mean (n) | 10.43 |
| st.dev. (n) | 1.931 |
| R(calc.) | 5.41 |
| st.dev.(Horwitz) | 1.173 |
| R(Horwitz) | 3.28 |

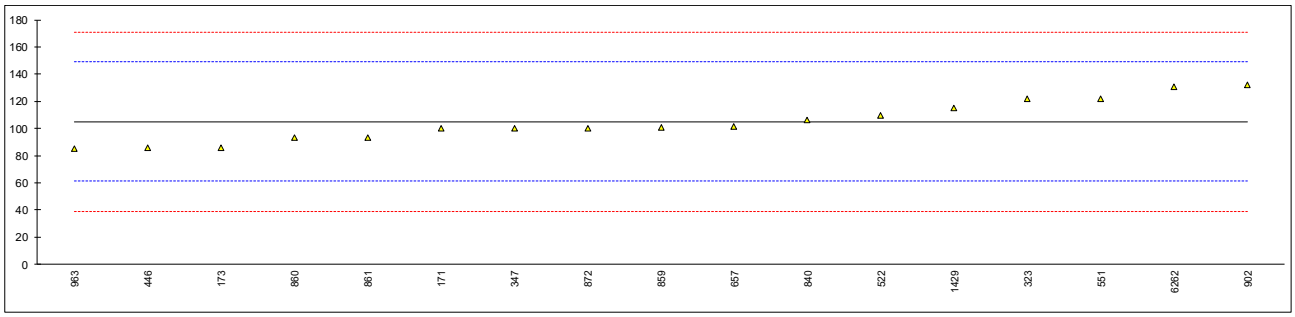


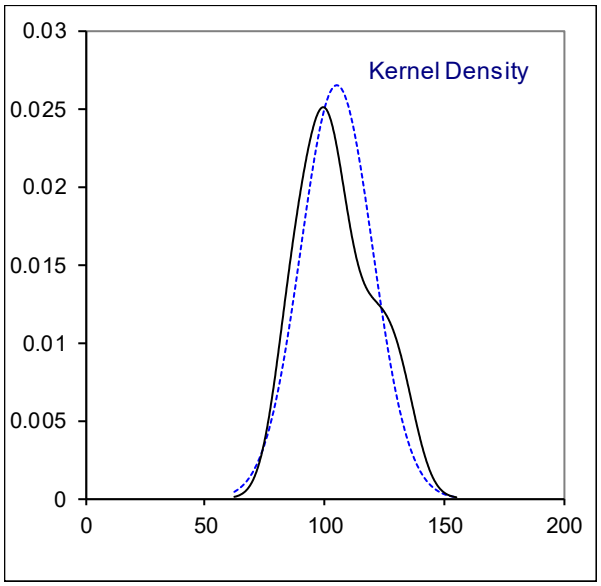



## Determination of Water on sample #21001; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 171 | D1364 | 100 | | -0.22 | |
| 173 | E203 | 86 | | -0.86 | |
| 273 | | ----- | | ----- | |
| 323 | D1364 | 122 | | 0.78 | |
| 347 | D1364 | 100 | | -0.22 | |
| 391 | | ----- | | ----- | |
| 446 | D1364 | 86 | | -0.86 | |
| 522 | E203 | 109.7 | | 0.22 | |
| 541 | | ----- | | ----- | |
| 551 | E1364 | 122 | | 0.78 | |
| 657 | E1064 | 101.6 | | -0.15 | |
| 840 | D1364 | 106 | | 0.05 | |
| 859 | D1364 | 101 | | -0.18 | |
| 860 | D1364 | 93 | | -0.54 | |
| 861 | D1364 | 93 | | -0.54 | |
| 872 | D1364 | 100 | | -0.22 | |
| 902 | D1364 | 132 | | 1.23 | |
| 913 | | ----- | | ----- | |
| 963 | D1364 | 85 | | -0.91 | |
| 974 | | ----- | | ----- | |
| 1429 | | 115 | | 0.46 | |
| 6262 | D1364 | 131 | | 1.19 | |

| | |
|---|---|
| normality | OK |
| n | 17 |
| outliers | 0 |
| mean (n) | 104.90 |
| st.dev. (n) | 15.038 |
| R(calc.) | 42.11 |
| st.dev.(D1364:02) | 21.947 |
| R(D1364:02) | 61.45 |



## APPENDIX 2

### Number of participants per country

| | |
|---|---|
| 1 lab in | ARGENTINA |
| 2 labs in | BELGIUM |
| 1 lab in | BRAZIL |
| 3 labs in | CHINA, People's Republic |
| 1 lab in | INDIA |
| 1 lab in | ITALY |
| 1 lab in | MEXICO |
| 1 lab in | RUSSIAN FEDERATION |
| 1 lab in | SAUDI ARABIA |
| 1 lab in | SINGAPORE |
| 1 lab in | SOUTH AFRICA |
| 1 lab in | SPAIN |
| 1 lab in | TURKEY |
| 1 lab in | UNITED ARAB EMIRATES |
| 2 labs in | UNITED KINGDOM |
| 2 labs in | UNITED STATES OF AMERICA |
| 1 lab in | VIETNAM |

**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 |
| SDS | = Safety Data Sheet |

**Literature**


1 iis Interlaboratory Studies, Protocol for the Organisation, Statistics & Evaluation, June 2018
2 Horwitz, R. Albert, J. AOAC Int, 79, 3, 589, (1996)
3 ASTM E178:08
4 ASTM E1301:03
5 ISO5725:86
6 ISO5725, parts 1-6:94
7 ISO13528:05
8 M. Thompson and R. Wood, J. AOAC Int, 76, 926, (1993)
9 W.J. Youden and E.H. Steiner, Statistical Manual of the AOAC, (1975)
10 IP367:84
11 DIN38402 T41/42
12 P.L. Davies, Fr. Z. Anal. Chem, 331, 513, (1988)
13 J.N. Miller, Analyst, 118, 455, (1993)
14 Analytical Methods Committee, Technical brief No 4, January 2001
15 P.J. Lowthian and M.Thompson, The Royal Society of Chemistry, Analyst, 127, 1359-1364, (2002)
16 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, Technometrics, 25(2),165-172, (1983)