# Results of Proficiency Test
# n-Butyl Acrylate
# March 2019

Organised by: Institute for Interlaboratory Studies
Spijkenisse, the Netherlands

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**CONTENTS**

# 1 INTRODUCTION

Since 2004, the Institute for Interlaboratory Studies organizes a proficiency scheme for n-Butyl Acrylate every two year. During the annual proficiency testing program of 2018/2019, it was decided to continue the proficiency tests for the analysis of n-Butyl Acrylate.
In this interlaboratory study 25 laboratories in 20 different countries did register for participation. See appendix 2 for the number of participants per country.
In this report the results of the 2019 proficiency test for n-Butyl Acrylate 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 analyses for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory.
It was decided to send one sample of 0.5L of n-Butyl Acrylate, labelled #19042.
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 organisation 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 SAMPLE

The necessary bulk material of n-Butyl Acrylate was obtained from a local chemical supplier. After homogenisation, 35 amber glass bottles of 0.5L were filled and labelled #19042.
The homogeneity of the subsamples was checked by determination of Density at 20°C in accordance with ASTM D4052 on 8 stratified randomly selected samples.

| | Density at 20°C in kg/L |
|---|---|
| sample #19042-1 | 0.89905 |
| sample #19042-2 | 0.89905 |
| sample #19042-3 | 0.89905 |
| sample #19042-4 | 0.89903 |
| sample #19042-5 | 0.89905 |
| sample #19042-6 | 0.89905 |
| sample #19042-7 | 0.89906 |
| sample #19042-8 | 0.89906 |

Table 1: homogeneity test results of subsamples #19042

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) | 0.00003 |
| Reference test method | ISO12185:96 |
| 0.3*R (reference test method) | 0.00015 |

Table 2: evaluation of the repeatability of subsamples #19042

The calculated repeatability was in agreement with 0.3 times the corresponding reproducibility of the reference test method. Therefore, homogeneity of the subsamples #19042 was assumed.

To each of the participating laboratories one 0.5L bottle of n-Butyl Acrylate labelled #19042 was sent on March 13, 2019. An SDS was added to the sample package.

## 2.5 STABILITY OF THE SAMPLE

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

### 2.6 ANALYSES

The participants were requested to determine on sample #19042: Acidity as Acrylic Acid, Appearance, Color Pt/Co, Density at 20°C, Inhibitor as MEHQ, Purity by GC as received, Purity by GC on dry basis, n-Butanol, n-Butylacetate, n-Butylpropionate, di-n-Butylether, Isobutylacrylate, Isobutylpropionate, Other impurities, Total 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 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 reanalyses). 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 organisation 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 and/or 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. The Kernel Density Graph 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 reproducibilities, the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

This 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 result tables of 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:

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

## 4 EVALUATION

In this interlaboratory study, no major problems were encountered with the dispatch of the samples. All reporting participants reported before the dead line. One participant did not report any test results.
In total 24 participants reported 193 numerical test results. Observed were 8 outlying test results, which is 4.1% of the total of numerical test results. In proficiency studies, outlier percentages of 3% - 7.5% are normal.

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, 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 methods are also in the tables together with the original data. The abbreviations, used in these tables, are listed 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(2011)). In the results tables of appendix 1 only the method number and year of adoption or revision e.g. D1209:05 will be used.

**Sample #19042**

Acidity as Acrylic Acid: This determination was very problematic. No statistical outliers were observed. However, the calculated reproducibility is not at all in agreement with the requirements of ASTM D1613:17.

Appearance: No analytical problems were observed. All reporting labs agreed about the appearance of sample #19042, which is pass (bright, clear and free of suspended matter).

Color Pt/Co: 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 D1209:05(2011).

Density at 20°C: 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 ISO12185:96.

Inhibitor as MEHQ: This determination was not problematic. Two statistical outliers were observed. However, the calculated reproducibility after rejection of the statistical outliers is in agreement with the requirements of ASTM D3125:06(2012).

Purity as received and on dry basis: These determinations were not problematic. No statistical outliers were observed. The reported purities test results from one participant were marked with an “E” as the reported test result for purity “as received” was larger than the reported test result for purity “on dry basis”, which is not possible. Presumable the test results were mixed-up? However, it was decided not to exclude these test result as the difference between the two purities is relatively small and the number of test results low.

The calculated reproducibilities are both in good agreement with the requirements of ASTM D3362:05(2011). Please note: This test method was withdrawn with no replacement.

n-Butanol: 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 reproducibility estimated using the Horwitz equation.

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

n-Butylpropionate: 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 reproducibility estimated using the Horwitz equation.

di-n-Butylether: This determination may be problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the reproducibility estimated using the Horwitz equation.

Isobutylacrylate: 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 reproducibility estimated using the Horwitz equation.

Isobutylpropionate: No significant conclusions could be drawn as only two laboratories reported a test result.

Other Impurities: No significant conclusions could be drawn as only three laboratories reported a test result.

Total Impurities: This determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the calculated reproducibility estimated using the Horwitz equation (5 components).

Water: This determination was very problematic. No statistical outliers were observed. As the calculated reproducibility is not at all in agreement with the requirements of ASTM E1064:16, no z-scores were calculated. When evaluated over only E1064 test results the calculated reproducibility is still 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 results, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from literature reference test methods (in casu ASTM, EN test methods) or previous proficiency tests are presented in the next tables.

| Parameter | unit | n | average | 2.8 * sd | R (target) |
|---|---|---|---|---|---|
| Acidity as Acrylic Acid | mg/kg | 19 | 38.0 | 46.1 | 14 |
| Appearance | | 22 | Pass | n.a. | n.a. |
| Color Pt/Co | | 21 | 32.9 | 8.7 | 7 |
| Density at 20°C | kg/L | 21 | 0.8991 | 0.0002 | 0.0005 |
| Inhibitor as MEHQ | mg/kg | 21 | 14.8 | 1.9 | 2.2 |
| Purity as received | %M/M | 13 | 99.843 | 0.088 | 0.27 |
| Purity on dry basis | %M/M | 14 | 99.847 | 0.111 | 0.27 |
| n-Butanol | mg/kg | 10 | 124 | 36 | 27 |
| n-Butylacetate | mg/kg | 11 | 390 | 68 | 71 |
| n-Butylpropionate | mg/kg | 9 | 480 | 216 | 85 |
| di-n-Butylether | mg/kg | 9 | 118 | 32 | 26 |
| Isobutylacrylate | mg/kg | 8 | 87 | 39 | 20 |
| Isobutylpropionate | mg/kg | 2 | n.a. | n.a. | n.a. |
| Other impurities | mg/kg | 3 | n.a. | n.a. | n.a. |
| Total impurities | mg/kg | 6 | 1406 | 312 | 473 |
| Water | mg/kg | 23 | 81 | 66 | (13) |

Table 3: reproducibilities of test results of sample #19042

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

### 4.3 COMPARISON OF THE PROFICIENCY TEST OF MARCH 2019 WITH PREVIOUS PTS

| | March 2019 | April 2017 | June 2015 | May 2012 | April 2010 |
|---|---|---|---|---|---|
| Number of reporting labs | 24 | 16 | 13 | 14 | 17 |
| Number of results reported | 193 | 160 | 117 | 138 | 202 |
| Number of statistical outliers | 8 | 6 | 2 | 5 | 19 |
| Percentage outliers | 4.1% | 3.8% | 1.7% | 3.6% | 9.4% |

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

| | March 2019 | April 2017 | June 2015 | May 2012 | April 2010 |
|---|---|---|---|---|---|
| Acidity as Acrylic Acid | -- | + | ++ | ++ | - |
| Color Pt/Co | - | ++ | ++ | ++ | ++ |
| Density at 20°C | ++ | ++ | ++ | ++ | ++ |
| Inhibitor as MEHQ | + | - | ++ | ++ | +/- |
| Purity as received | ++ | ++ | ++ | ++ | ++ |
| Purity on dry basis | ++ | ++ | ++ | ++ | ++ |
| n-Butanol | - | - | ++ | +/- | -- |
| n-Butylacetate | +/- | + | ++ | ++ | + |
| n-Butylpropionate | -- | + | -- | +/- | +/- |
| di-n-Butylether | - | - | + | ++ | -- |
| Isobutylacrylate | -- | ++ | ++ | ++ | ++ |
| Isobutylpropionate | n.e. | n.e. | n.e. | n.e. | n.e. |
| Total impurities | + | - | ++ | n.e. | n.e. |
| Water | (--) | +/- | +/- | ++ | -- |

Table 5: comparison determinations against the reference test method

The following performance categories were used:

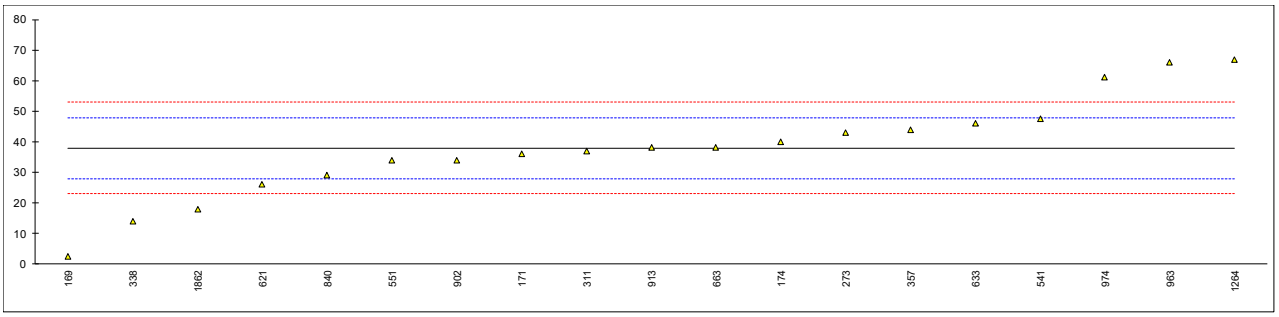
++: group performed much better than the reference test method
+ : group performed better than the reference test method
+/-: group performance similar to the reference test method
- : group performed worse than the reference test method
-- : group performed much worse than the reference test method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

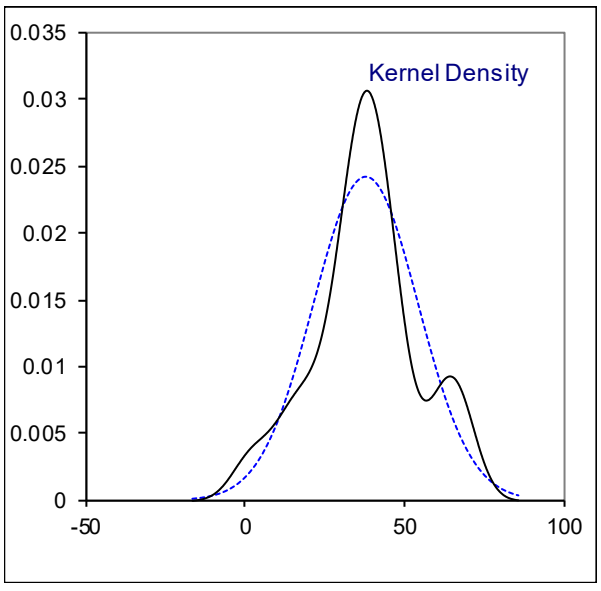
## APPENDIX 1

Determination of Acidity, as Acrylic Acid on sample #19042; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 169 | D1613 | 2.4 | | -7.11 | |
| 171 | D1613 | 36 | | -0.39 | |
| 174 | D1613 | 40 | | 0.41 | |
| 273 | D1613 | 43 | | 1.01 | |
| 311 | D1613 | 37 | | -0.19 | |
| 323 | | ----- | | ----- | |
| 338 | D1613 | 14 | | -4.79 | |
| 357 | D1613 | 44 | | 1.21 | |
| 522 | | ----- | | ----- | |
| 541 | D1613 | 47.4 | | 1.89 | |
| 551 | D1613 | 34 | | -0.79 | |
| 613 | | ----- | | ----- | |
| 621 | D1613 | 26 | | -2.39 | |
| 633 | D1613 | 46 | | 1.61 | |
| 663 | D1613 | 38.2 | | 0.05 | |
| 840 | D1613 | 29.1 | | -1.77 | |
| 872 | | ----- | | ----- | |
| 886 | | ----- | | ----- | |
| 902 | D1613 | 34 | | -0.79 | |
| 913 | D1613 | 38 | | 0.01 | |
| 963 | D1613 | 66 | | 5.61 | |
| 974 | D1613 | 61 | | 4.61 | |
| 1264 | D1613 | 67 | | 5.81 | |
| 1862 | D1613 | 18 | | -3.99 | |
| 9014 | | ----- | | ----- | |

| | |
|---|---|
| normality | OK |
| n | 19 |
| outliers | 0 |
| mean (n) | 37.953 |
| st.dev. (n) | 16.4569 |
| R(calc.) | 46.079 |
| st.dev.(D1613:17) | 5 |
| R(D1613:17) | 14 |



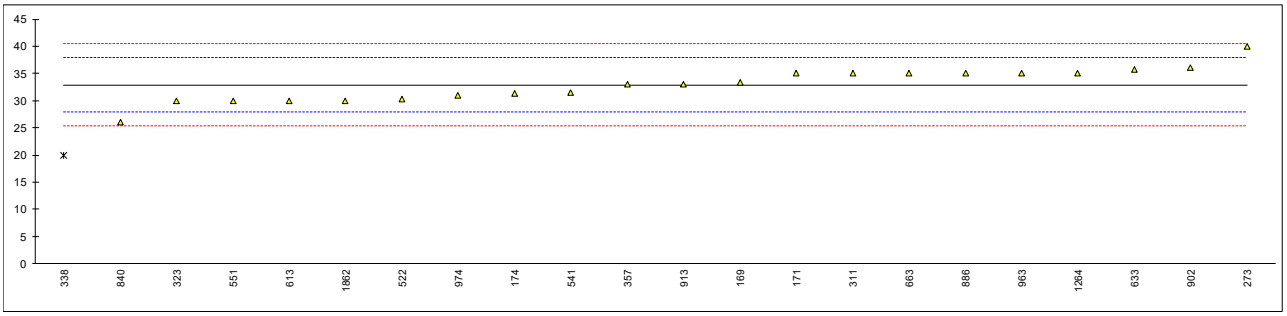
Determination of Appearance on sample #19042;

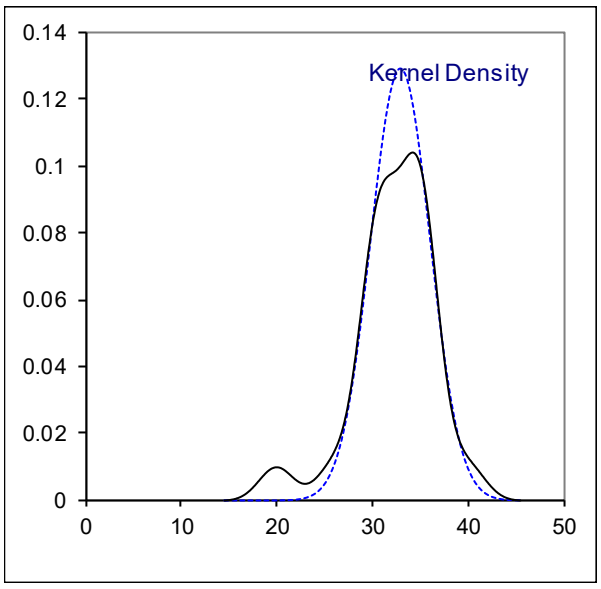
| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 169 | | CBFSM | | ----- | |
| 171 | E2680 | pass | | ----- | |
| 174 | Visual | Clear & Free | | ----- | |
| 273 | Visual | Pass | | ----- | |
| 311 | E2680 | pass | | ----- | |
| 323 | E2680 | clear&bright liquid | | ----- | |
| 338 | Visual | Clear and bright | | ----- | |
| 357 | E2680 | Pass | | ----- | |
| 522 | D4176 | PASS | | ----- | |
| 541 | E2680 | Pass | | ----- | |
| 551 | Visual | Pass | | ----- | |
| 613 | INH-101967 | PASS | | ----- | |
| 621 | D4176 | Clear & Bright | | ----- | |
| 633 | Visual | Clear & Bright | | ----- | |
| 663 | Visual | Clear & Bright | | ----- | |
| 840 | E2680 | Pass | | ----- | |
| 872 | | ----- | | ----- | |
| 886 | | ----- | | ----- | |
| 902 | E2680 | PASS | | ----- | |
| 913 | E2680 | Pass | | ----- | |
| 963 | E2680 | Pass | | ----- | |
| 974 | Visual | Clear & Brigth | | | |
| 1264 | Visual | Pass | | ----- | |
| 1862 | Visual | Pass | | ----- | |
| 9014 | | ----- | | ----- | |
| | n | 22 | | | |
| | mean (n) | Pass | | | |

## Determination of Color Pt/Co on sample #19042;

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 169 | D5386 | 33.3 | C | 0.15 | first reported <20.0 |
| 171 | D1209 | 35 | | 0.83 | |
| 174 | D5386 | 31.3 | | -0.65 | |
| 273 | D1209 | 40 | | 2.83 | |
| 311 | D1209 | 35 | | 0.83 | |
| 323 | D1209 | 30 | | -1.17 | |
| 338 | D1209 | 20 | D(0.01) | -5.17 | |
| 357 | D5386 | 33 | | 0.03 | |
| 522 | D1209 | 30.3 | | -1.05 | |
| 541 | D5386 | 31.5 | | -0.57 | |
| 551 | D1209 | 30 | | -1.17 | |
| 613 | D5386 | 30 | | -1.17 | |
| 621 | D1209 | Pass | | ----- | |
| 633 | D1209 | 35.8 | | 1.15 | |
| 663 | D1209 | 35 | | 0.83 | |
| 840 | D1209 | 26 | | -2.77 | |
| 872 | | ----- | | ----- | |
| 886 | D1209 | 35 | | 0.83 | |
| 902 | D5386 | 36 | | 1.23 | |
| 913 | D5386 | 33 | | 0.03 | |
| 963 | D1209 | 35 | | 0.83 | |
| 974 | D1209 | 31 | | -0.77 | |
| 1264 | D1209 | 35 | | 0.83 | |
| 1862 | D1209 | 30 | | -1.17 | |
| 9014 | | ----- | | ----- | |

| | |
|---|---|
| normality | OK |
| n | 21 |
| outliers | 1 |
| mean (n) | 32.91 |
| st.dev. (n) | 3.092 |
| R(calc.) | 8.66 |
| st.dev.(D1209:05) | 2.5 |
| R(D1209:05) | 7 |

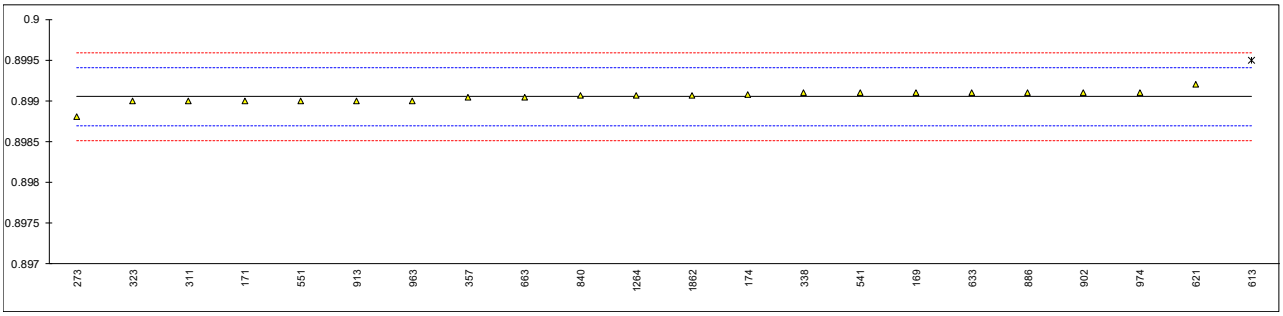


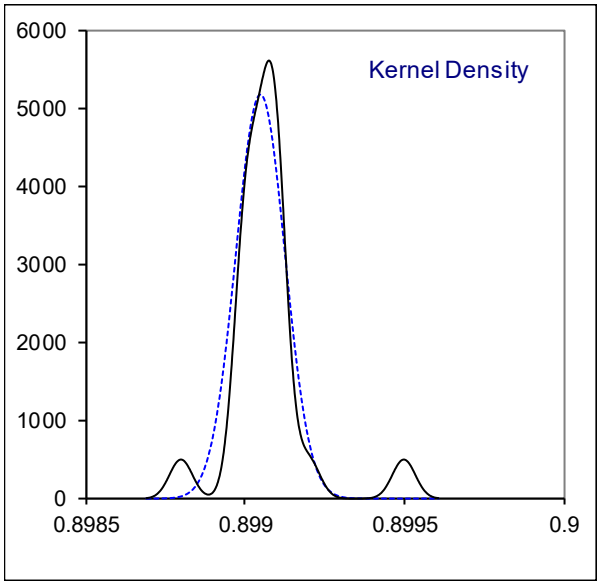



## Determination of Density at 20°C on sample #19042; results in kg/L

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 169 | D4052 | 0.8991 | C | 0.28 | first reported 899.1 kg/L |
| 171 | D4052 | 0.899 | | -0.28 | |
| 174 | D4052 | 0.89908 | | 0.17 | |
| 273 | D4052 | 0.8988 | | -1.40 | |
| 311 | D4052 | 0.8990 | | -0.28 | |
| 323 | D4052 | 0.8990 | | -0.28 | |
| 338 | ISO12185 | 0.8991 | | 0.28 | |
| 357 | D4052 | 0.89904 | | -0.05 | |
| 522 | | ----- | | ----- | |
| 541 | D4052 | 0.89910 | | 0.28 | |
| 551 | D4052 | 0.8990 | | -0.28 | |
| 613 | D4052 | 0.8995 | D(0.01) | 2.52 | |
| 621 | D4052 | 0.8992 | | 0.84 | |
| 633 | D4052 | 0.89910 | | 0.28 | |
| 663 | D4052 | 0.89904 | | -0.05 | |
| 840 | D4052 | 0.89906 | | 0.06 | |
| 872 | | ----- | | ----- | |
| 886 | D4052 | 0.8991 | | 0.28 | |
| 902 | ISO12185 | 0.8991 | | 0.28 | |
| 913 | ISO12185 | 0.8990 | | -0.28 | |
| 963 | ISO12185 | 0.8990 | | -0.28 | |
| 974 | D4052 | 0.8991 | | 0.28 | |
| 1264 | D4052 | 0.89906 | | 0.06 | |
| 1862 | D4052 | 0.89906 | | 0.06 | |
| 9014 | | ----- | | ----- | |

| | |
|---|---|
| normality | not OK |
| n | 21 |
| outliers | 1 |
| mean (n) | 0.89905 |
| st.dev. (n) | 0.000077 |
| R(calc.) | 0.00022 |
| st.dev.(ISO12185:96) | 0.000179 |
| R(ISO12185:96) | 0.0005 |

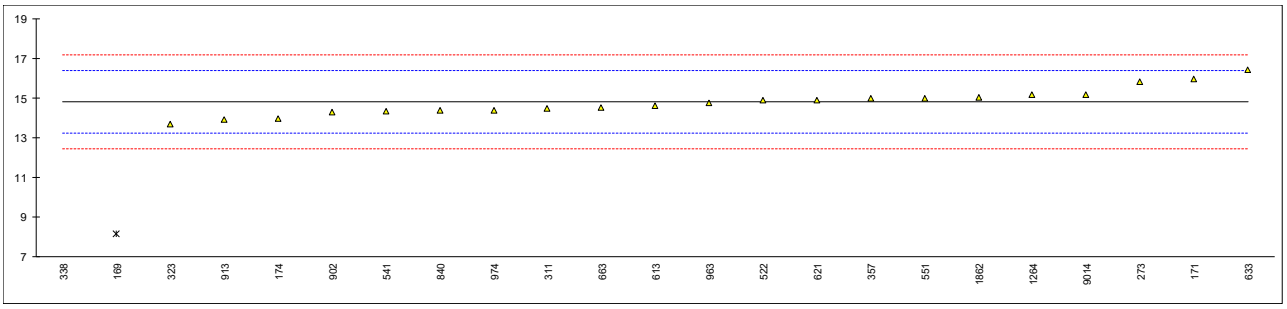


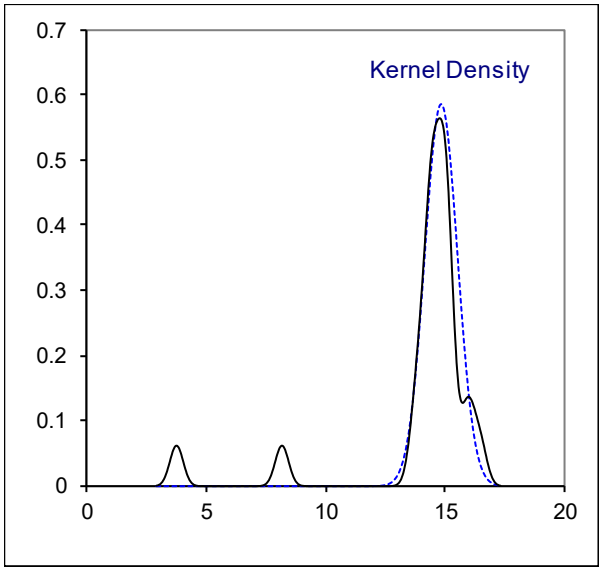



## Determination of Inhibitor as MEHQ on sample #19042; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 169 | D3125 | 8.19 | D(0.01) | -8.35 | |
| 171 | D3125 | 16.0 | | 1.49 | |
| 174 | D3125 | 14.0 | | -1.03 | |
| 273 | D3125 | 15.86 | | 1.31 | |
| 311 | D3125 | 14.5 | | -0.40 | |
| 323 | D3125 | 13.7 | | -1.41 | |
| 338 | D3125 | 3.8 | D(0.01) | -13.88 | |
| 357 | D3125 | 15.0 | | 0.23 | |
| 522 | D3125 | 14.9 | | 0.10 | |
| 541 | D3125 | 14.36 | | -0.58 | |
| 551 | D3125 | 15 | | 0.23 | |
| 613 | | 14.65 | | -0.21 | |
| 621 | D3125 | 14.9 | | 0.10 | |
| 633 | D3125 | 16.45 | | 2.05 | |
| 663 | D3125 | 14.54 | | -0.35 | |
| 840 | D3125 | 14.40 | | -0.53 | |
| 872 | | ----- | | ----- | |
| 886 | | ----- | | ----- | |
| 902 | D3125 | 14.3 | | -0.65 | |
| 913 | D3125 | 13.96 | | -1.08 | |
| 963 | D3125 | 14.8 | | -0.02 | |
| 974 | D3125 | 14.4 | | -0.53 | |
| 1264 | D3125 | 15.2 | | 0.48 | |
| 1862 | D3125 | 15.08 | | 0.33 | |
| 9014 | D3125 | 15.20 | | 0.48 | |

| | |
|---|---|
| normality | OK |
| n | 21 |
| outliers | 2 |
| mean (n) | 14.82 |
| st.dev. (n) | 0.681 |
| R(calc.) | 1.91 |
| st.dev.(D3125:06) | 0.794 |
| R(D3125:06) | 2.22 |

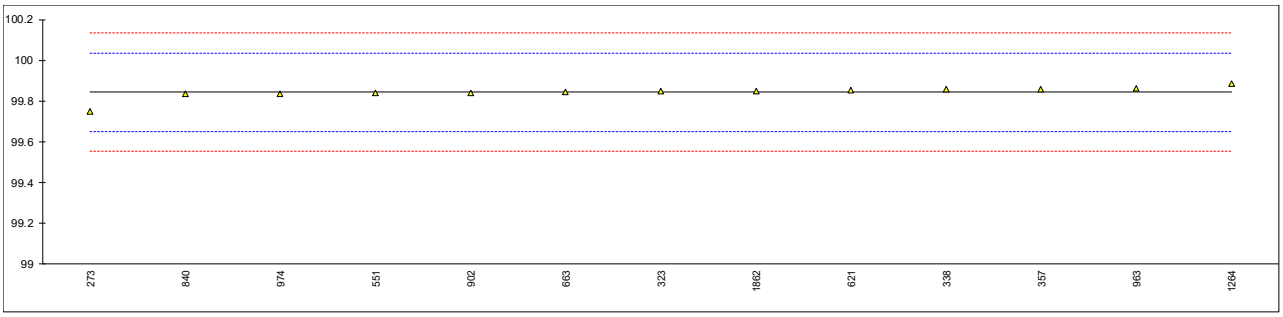


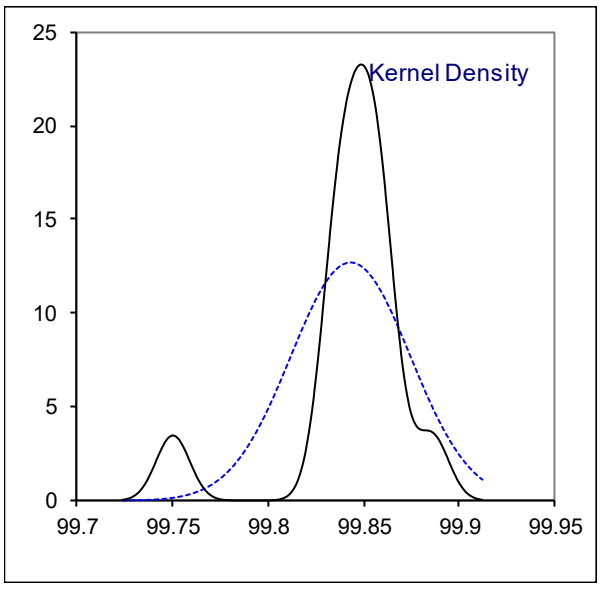



## Determination of Purity by GC as received on sample #19042; results in %M/M

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 169 | | ----- | | ----- | |
| 171 | | ----- | | ----- | |
| 174 | | ----- | | ----- | |
| 273 | D3362 | 99.75 | | -0.97 | |
| 311 | | ----- | | ----- | |
| 323 | D3362 | 99.85 | | 0.07 | |
| 338 | D3362 | 99.859 | | 0.16 | |
| 357 | D3362 | 99.859 | | 0.16 | |
| 522 | | ----- | | ----- | |
| 541 | | ----- | | ----- | |
| 551 | INH-3088 | 99.84 | | -0.03 | |
| 613 | | ----- | | ----- | |
| 621 | D3362 | 99.854 | E | 0.11 | E: test result “on dry basis” < test result “as received” |
| 633 | | ----- | | ----- | |
| 663 | INH-15 | 99.845 | | 0.02 | |
| 840 | INH-004 | 99.833 | | -0.11 | |
| 872 | | ----- | | ----- | |
| 886 | | ----- | | ----- | |
| 902 | INH-226 | 99.84 | | -0.03 | |
| 913 | | ----- | | ----- | |
| 963 | INH-102538 | 99.863 | | 0.20 | |
| 974 | INH-1B | 99.834 | | -0.10 | |
| 1264 | In house | 99.886 | | 0.44 | |
| 1862 | | 99.85 | | 0.07 | |
| 9014 | | ----- | | ----- | |

| | |
|---|---|
| normality | not OK |
| n | 13 |
| outliers | 0 |
| mean (n) | 99.8433 |
| st.dev. (n) | 0.03140 |
| R(calc.) | 0.0879 |
| st.dev.(D3362:05) | 0.09643 |
| R(D3362:05) | 0.27 |

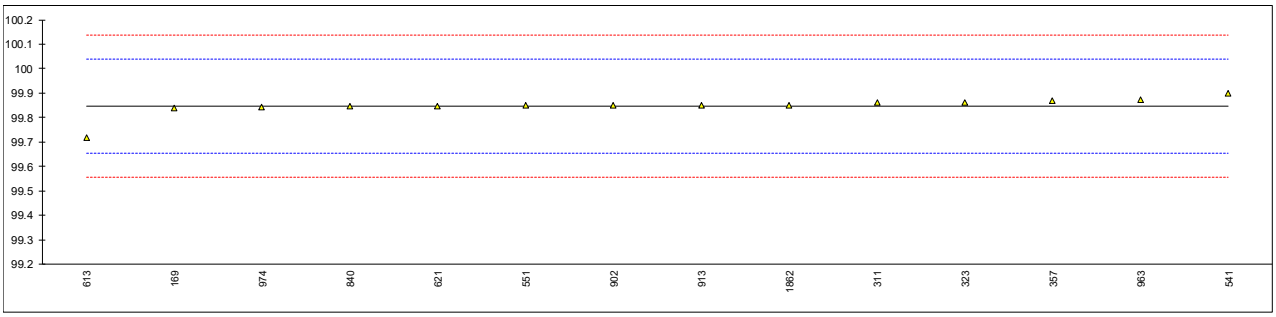


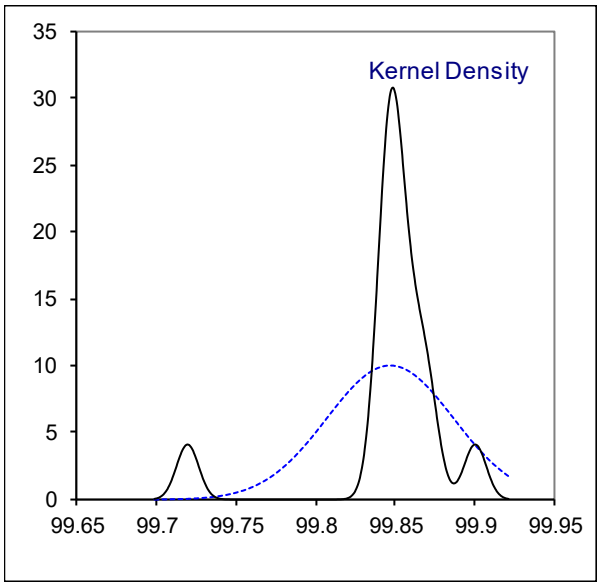



## Determination of Purity by GC on dry basis on sample #19042; results in %M/M

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 169 | D3362 | 99.84 | | -0.07 | |
| 171 | | ----- | | ----- | |
| 174 | | ----- | | ----- | |
| 273 | | ----- | | ----- | |
| 311 | INH-796 | 99.86 | | 0.14 | |
| 323 | D3362 | 99.86 | | 0.14 | |
| 338 | | ----- | | ----- | |
| 357 | D3362 | 99.868 | | 0.22 | |
| 522 | | ----- | | ----- | |
| 541 | INH-102538 | 99.900 | | 0.55 | |
| 551 | INH-3088 | 99.85 | | 0.03 | |
| 613 | INH-102538 | 99.72 | | -1.31 | |
| 621 | D3362 | 99.847 | E | 0.00 | E: test result “on dry basis” < test result “as received” |
| 633 | | ----- | | ----- | |
| 663 | | ----- | | ----- | |
| 840 | INH-004 | 99.845 | | -0.02 | |
| 872 | | ----- | | ----- | |
| 886 | | ----- | | ----- | |
| 902 | INH-226 | 99.85 | | 0.03 | |
| 913 | D3362 | 99.85 | | 0.03 | |
| 963 | INH-102538 | 99.872 | | 0.26 | |
| 974 | INH-1B | 99.843 | | -0.04 | |
| 1264 | | ----- | | ----- | |
| 1862 | | 99.85 | | 0.03 | |
| 9014 | | ----- | | ----- | |

| | |
|---|---|
| normality | not OK |
| n | 14 |
| outliers | 0 |
| mean (n) | 99.8468 |
| st.dev. (n) | 0.03966 |
| R(calc.) | 0.1110 |
| st.dev.(D3362:05) | 0.09643 |
| R(D3362:05) | 0.27 |

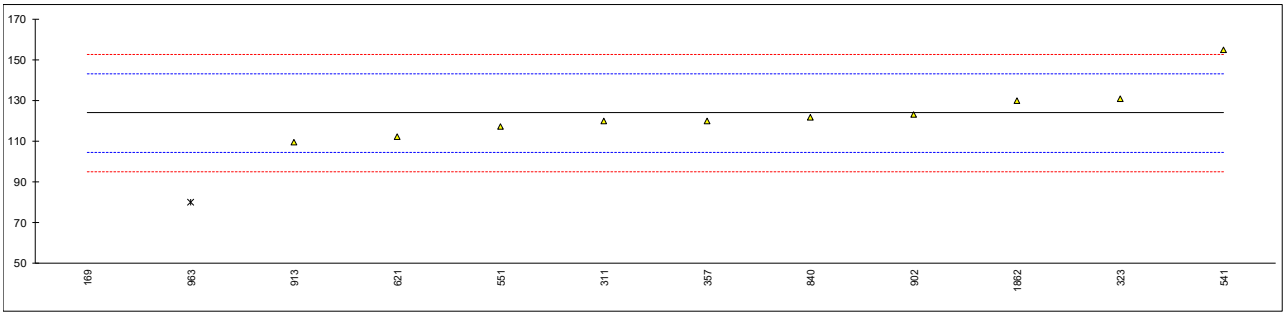


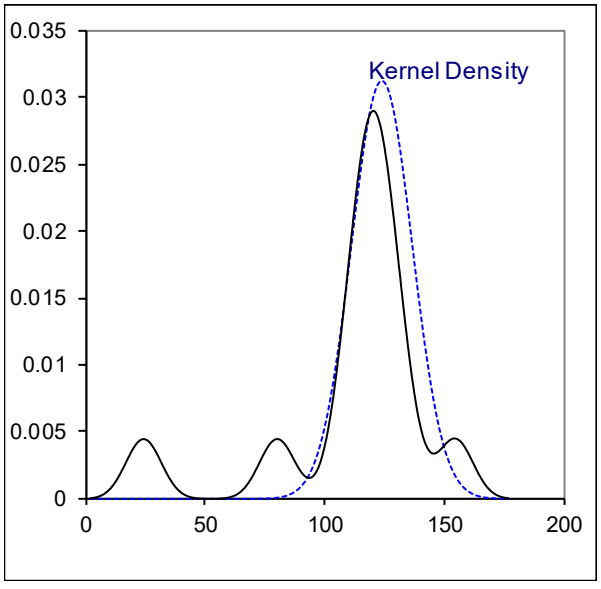



## Determination of n-Butanol on sample #19042; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 169 | D3362 | 24 | C,D(0.05) | -10.41 | first reported 2.4 |
| 171 | | ----- | | ----- | |
| 174 | | ----- | | ----- | |
| 273 | | ----- | | ----- | |
| 311 | INH-796 | 120 | | -0.40 | |
| 323 | D3362 | 131 | | 0.74 | |
| 338 | | ----- | | ----- | |
| 357 | D3362 | 120 | | -0.40 | |
| 522 | | ----- | | ----- | |
| 541 | INH-102538 | 154.6 | | 3.20 | |
| 551 | INH-3088 | 117 | | -0.71 | |
| 613 | INH-102538 | nd | C | ----- | first reported 1, possibly a false negative test result? |
| 621 | D3362 | 112 | | -1.24 | |
| 633 | | ----- | | ----- | |
| 663 | | ----- | | ----- | |
| 840 | INH-004 | 121.6 | | -0.24 | |
| 872 | | ----- | | ----- | |
| 886 | | ----- | | ----- | |
| 902 | INH-226 | 123 | | -0.09 | |
| 913 | D3362 | 109.4 | C | -1.51 | first reported 194 |
| 963 | INH-102538 | 80 | D(0.05) | -4.57 | |
| 974 | | ----- | | ----- | |
| 1264 | | ----- | | ----- | |
| 1862 | | 130 | | 0.64 | |
| 9014 | | ----- | | ----- | |

| | |
|---|---|
| normality | not OK |
| n | 10 |
| outliers | 2 |
| mean (n) | 123.86 |
| st.dev. (n) | 12.763 |
| R(calc.) | 35.74 |
| st.dev.(Horwitz) | 9.595 |
| R(Horwitz) | 26.87 |

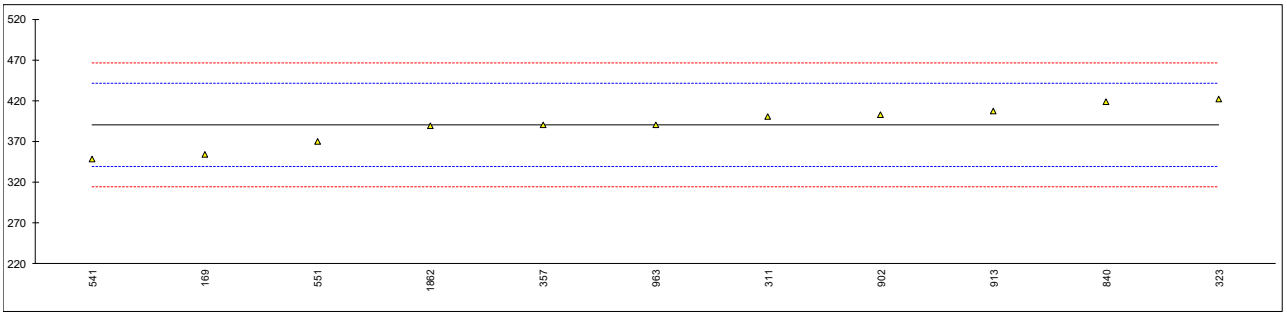


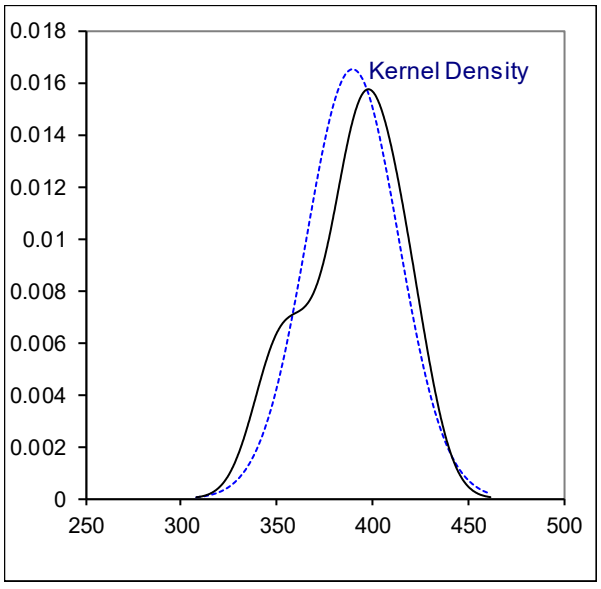



## Determination of n-Butylacetate on sample #19042; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 169 | D3362 | 353.6 | | -1.44 | |
| 171 | | ----- | | ----- | |
| 174 | | ----- | | ----- | |
| 273 | | ----- | | ----- | |
| 311 | INH-796 | 400 | | 0.39 | |
| 323 | D3362 | 422 | | 1.25 | |
| 338 | | ----- | | ----- | |
| 357 | D3362 | 390 | | -0.01 | |
| 522 | | ----- | | ----- | |
| 541 | INH-102538 | 348.7 | | -1.63 | |
| 551 | INH-3088 | 370 | | -0.79 | |
| 613 | INH-102538 | <1 | C | < -15.30 | first reported 186.5, possibly a false negative test result? |
| 621 | | ----- | | ----- | |
| 633 | | ----- | | ----- | |
| 663 | | ----- | | ----- | |
| 840 | INH-004 | 418.5 | | 1.11 | |
| 872 | | ----- | | ----- | |
| 886 | | ----- | | ----- | |
| 902 | INH-226 | 403 | | 0.50 | |
| 913 | D3362 | 407 | | 0.66 | |
| 963 | INH-102538 | 390 | | -0.01 | |
| 974 | | ----- | | ----- | |
| 1264 | | ----- | | ----- | |
| 1862 | | 389 | | -0.05 | |
| 9014 | | ----- | | ----- | |

| | |
|---|---|
| normality | OK |
| n | 11 |
| outliers | 0 |
| mean (n) | 390.16 |
| st.dev. (n) | 24.136 |
| R(calc.) | 67.58 |
| st.dev.(Horwitz) | 25.430 |
| R(Horwitz) | 71.20 |

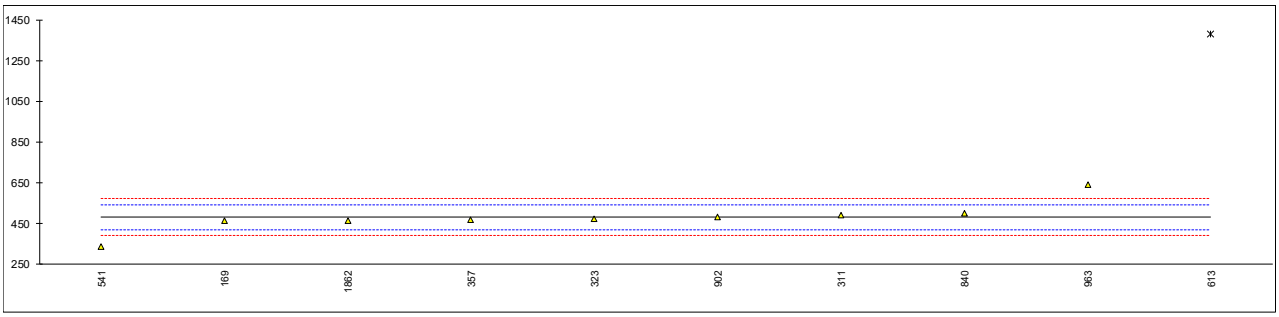


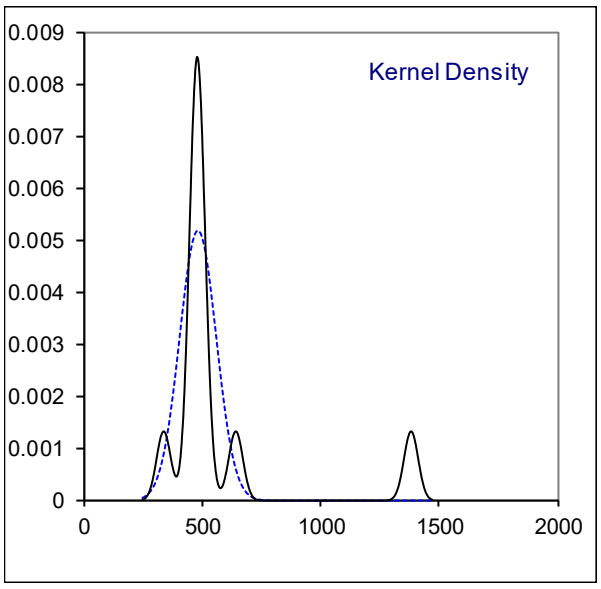



## Determination of n-Butylpropionate on sample #19042; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 169 | D3362 | 463.1 | | -0.57 | |
| 171 | | ----- | | ----- | |
| 174 | | ----- | | ----- | |
| 273 | | ----- | | ----- | |
| 311 | INH-796 | 490 | | 0.32 | |
| 323 | D3362 | 474 | | -0.21 | |
| 338 | | ----- | | ----- | |
| 357 | D3362 | 470 | | -0.34 | |
| 522 | | ----- | | ----- | |
| 541 | INH-102538 | 335.7 | | -4.76 | |
| 551 | | ----- | | ----- | |
| 613 | INH-102538 | 1380.0 | D(0.01) | 29.66 | |
| 621 | | ----- | | ----- | |
| 633 | | ----- | | ----- | |
| 663 | | ----- | | ----- | |
| 840 | INH-004 | 501.4 | | 0.70 | |
| 872 | | ----- | | ----- | |
| 886 | | ----- | | ----- | |
| 902 | INH-226 | 482 | | 0.06 | |
| 913 | | ----- | | ----- | |
| 963 | INH-102538 | 640 | | 5.27 | |
| 974 | | ----- | | ----- | |
| 1264 | | ----- | | ----- | |
| 1862 | | 466 | | -0.47 | |
| 9014 | | ----- | | ----- | |

| | |
|---|---|
| normality | not OK |
| n | 9 |
| outliers | 1 |
| mean (n) | 480.24 |
| st.dev. (n) | 77.137 |
| R(calc.) | 215.98 |
| st.dev.(Horwitz) | 30.338 |
| R(Horwitz) | 84.95 |

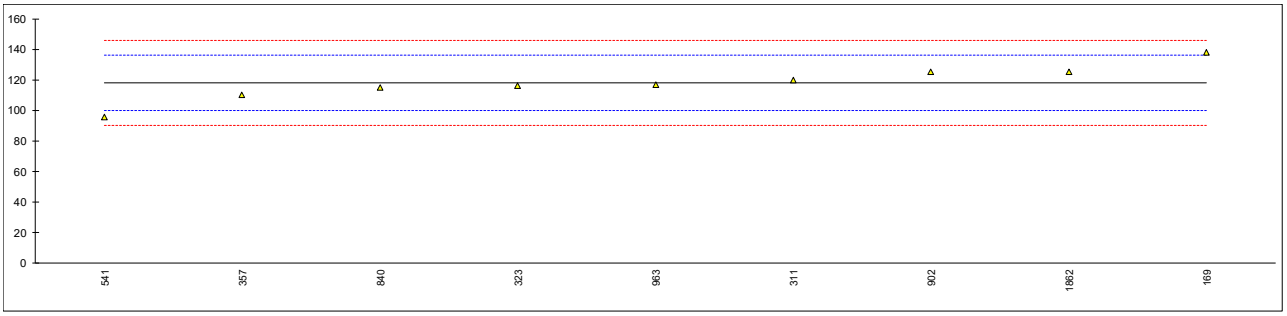


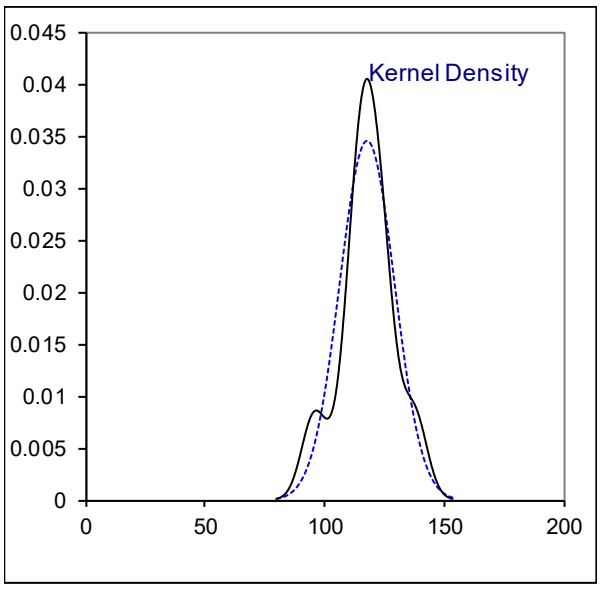



## Determination of di-n-Butylether on sample #19042; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 169 | D3362 | 137.8 | | 2.15 | |
| 171 | | ----- | | ----- | |
| 174 | | ----- | | ----- | |
| 273 | | ----- | | ----- | |
| 311 | INH-796 | 120 | | 0.22 | |
| 323 | D3362 | 116 | | -0.22 | |
| 338 | | ----- | | ----- | |
| 357 | D3362 | 110 | | -0.87 | |
| 522 | | ----- | | ----- | |
| 541 | INH-102538 | 95.9 | | -2.40 | |
| 551 | | ----- | | ----- | |
| 613 | | ----- | | ----- | |
| 621 | | ----- | | ----- | |
| 633 | | ----- | | ----- | |
| 663 | | ----- | | ----- | |
| 840 | INH-004 | 115.2 | | -0.30 | |
| 872 | | ----- | | ----- | |
| 886 | | ----- | | ----- | |
| 902 | INH-226 | 125 | | 0.76 | |
| 913 | | ----- | | ----- | |
| 963 | INH-102538 | 117 | | -0.11 | |
| 974 | | ----- | | ----- | |
| 1264 | | ----- | | ----- | |
| 1862 | | 125 | | 0.76 | |
| 9014 | | ----- | | ----- | |

| | |
|---|---|
| normality | suspect |
| n | 9 |
| outliers | 0 |
| mean (n) | 117.99 |
| st.dev. (n) | 11.507 |
| R(calc.) | 32.22 |
| st.dev.(Horwitz) | 9.207 |
| R(Horwitz) | 25.78 |

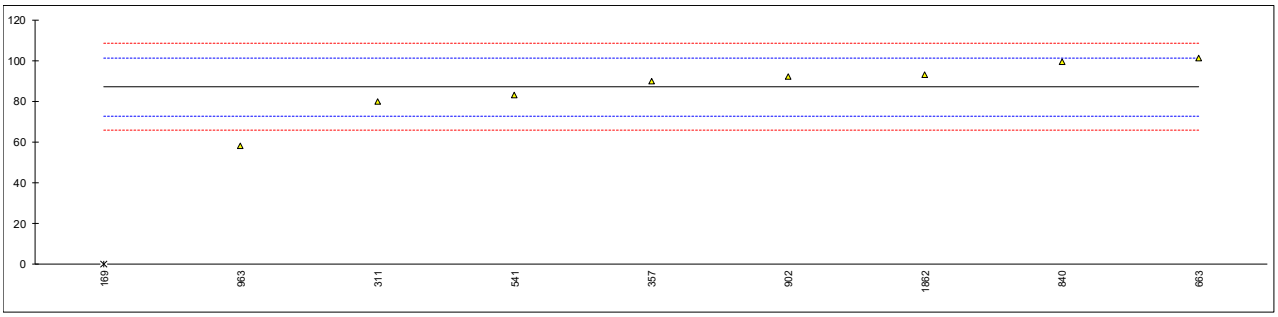


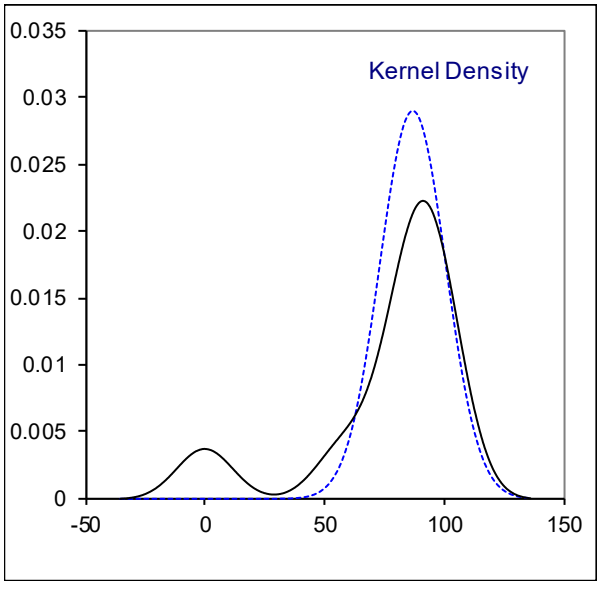



## Determination of Isobutylacrylate on sample #19042; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 169 | D3362 | 0 | D(0.05) | -12.24 | |
| 171 | | ----- | | ----- | |
| 174 | | ----- | | ----- | |
| 273 | | ----- | | ----- | |
| 311 | INH-796 | 80 | | -0.99 | |
| 323 | D3362 | <100 | | ----- | |
| 338 | | ----- | | ----- | |
| 357 | D3362 | 90 | | 0.41 | |
| 522 | | ----- | | ----- | |
| 541 | INH-102538 | 83.1 | | -0.56 | |
| 551 | | ----- | | ----- | |
| 613 | | ----- | | ----- | |
| 621 | | ----- | | ----- | |
| 633 | | ----- | | ----- | |
| 663 | INH-15 | 101.0 | | 1.96 | |
| 840 | INH-004 | 99.4 | | 1.73 | |
| 872 | | ----- | | ----- | |
| 886 | | ----- | | ----- | |
| 902 | INH-226 | 92 | | 0.69 | |
| 913 | | ----- | | ----- | |
| 963 | INH-102538 | 58 | | -4.09 | |
| 974 | | ----- | | ----- | |
| 1264 | | ----- | | ----- | |
| 1862 | | 93 | | 0.83 | |
| 9014 | | ----- | | ----- | |

| | |
|---|---|
| normality | not OK |
| n | 8 |
| outliers | 1 |
| mean (n) | 87.06 |
| st.dev. (n) | 13.758 |
| R(calc.) | 38.52 |
| st.dev.(Horwitz) | 7.112 |
| R(Horwitz) | 19.91 |



Determination of Isobutylpropionate on sample #19042; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 169 | D3362 | 0 | | ----- | |
| 171 | | ----- | | ----- | |
| 174 | | ----- | | ----- | |
| 273 | | ----- | | ----- | |
| 311 | | ----- | | ----- | |
| 323 | | ----- | | ----- | |
| 338 | | ----- | | ----- | |
| 357 | | ----- | | ----- | |
| 522 | | ----- | | ----- | |
| 541 | | ----- | | ----- | |
| 551 | | ----- | | ----- | |
| 613 | | ----- | | ----- | |
| 621 | | ----- | | ----- | |
| 633 | | ----- | | ----- | |
| 663 | | ----- | | ----- | |
| 840 | INH-004 | <10 | | ----- | |
| 872 | | ----- | | ----- | |
| 886 | | ----- | | ----- | |
| 902 | | ----- | | ----- | |
| 913 | | ----- | | ----- | |
| 963 | | ----- | | ----- | |
| 974 | | ----- | | ----- | |
| 1264 | | ----- | | ----- | |
| 1862 | | ----- | | ----- | |
| 9014 | | ----- | | ----- | |
| | n | 2 | | | |
| | mean (n) | <10 | | | |

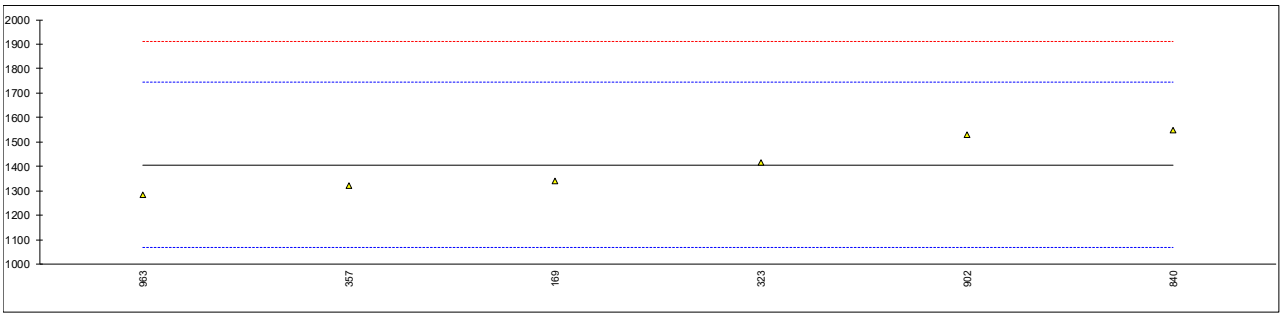
Determination of Other Impurities on sample #19042; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 169 | | ----- | | ----- | |
| 171 | | ----- | | ----- | |
| 174 | | ----- | | ----- | |
| 273 | | ----- | | ----- | |
| 311 | | ----- | | ----- | |
| 323 | D3362 | 182 | | ----- | |
| 338 | | ----- | | ----- | |
| 357 | | ----- | | ----- | |
| 522 | | ----- | | ----- | |
| 541 | | ----- | | ----- | |
| 551 | | ----- | | ----- | |
| 613 | | ----- | | ----- | |
| 621 | | ----- | | ----- | |
| 633 | | ----- | | ----- | |
| 663 | | ----- | | ----- | |
| 840 | INH-004 | 293.1 | | ----- | |
| 872 | | ----- | | ----- | |
| 886 | | ----- | | ----- | |
| 902 | INH-226 | 303 | | ----- | |
| 913 | | ----- | | ----- | |
| 963 | | ----- | | ----- | |
| 974 | | ----- | | ----- | |
| 1264 | | ----- | | ----- | |
| 1862 | | ----- | | ----- | |
| 9014 | | ----- | | ----- | |
| | n | 3 | | | |
| | mean (n) | (259.37) | | | |

## Determination of Total Impurities on sample #19042; results in mg/kg

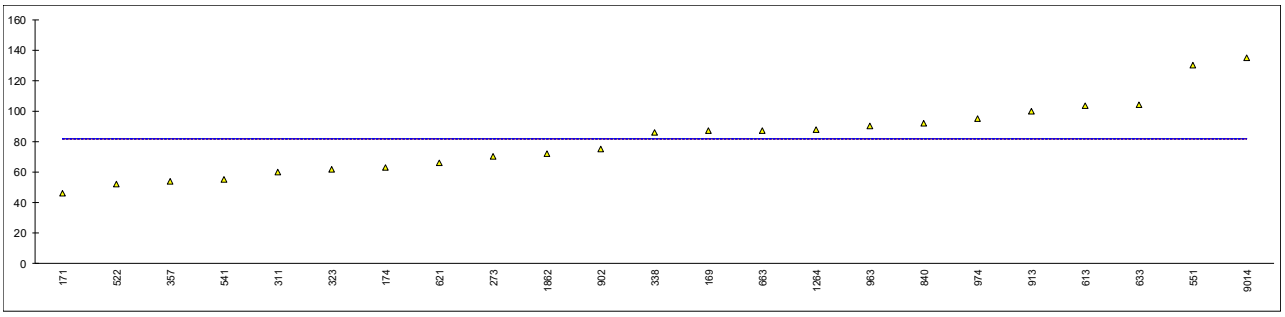
| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 169 | D3362 | 1338.96 | C | -0.40 | first reported 170.98 |
| 171 | | ----- | | ----- | |
| 174 | | ----- | | ----- | |
| 273 | | ----- | | ----- | |
| 311 | | ----- | | ----- | |
| 323 | D3362 | 1415 | | 0.05 | |
| 338 | | ----- | | ----- | |
| 357 | D3362 | 1320 | | -0.51 | |
| 522 | | ----- | | ----- | |
| 541 | | ----- | | ----- | |
| 551 | | ----- | | ----- | |
| 613 | | ----- | | ----- | |
| 621 | | ----- | | ----- | |
| 633 | | ----- | | ----- | |
| 663 | | ----- | | ----- | |
| 840 | INH-004 | 1549.1 | | 0.85 | |
| 872 | | ----- | | ----- | |
| 886 | | ----- | | ----- | |
| 902 | INH-226 | 1528 | | 0.72 | |
| 913 | | ----- | | ----- | |
| 963 | INH-102538 | 1285 | C | -0.72 | first reported 0.1285 |
| 974 | | ----- | | ----- | |
| 1264 | | ----- | | ----- | |
| 1862 | | ----- | | ----- | |
| 9014 | | ----- | | ----- | |

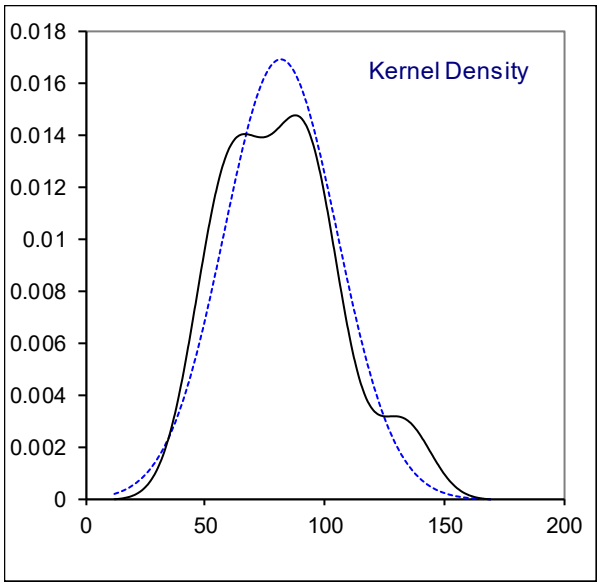
| | |
|---|---|
| normality | unknown |
| n | 6 |
| outliers | 0 |
| mean (n) | 1406.01 |
| st.dev. (n) | 111.332 |
| R(calc.) | 311.73 |
| st.dev.(Horwitz 5 comp) | 168.956 |
| R(Horwitz 5 comp) | 473.08 |



## Determination of Water on sample #19042; results in mg/kg

| lab | method | value | mark | z(targ) | remarks |
|---|---|---|---|---|---|
| 169 | E1064 | 87 | | ----- | |
| 171 | D1364 | 46 | | ----- | |
| 174 | E203 | 63 | | ----- | |
| 273 | E203 | 70 | | ----- | |
| 311 | E1064 | 60 | | ----- | |
| 323 | E1064 | 62 | | ----- | |
| 338 | E1064 | 86 | | ----- | |
| 357 | E1064 | 54 | | ----- | |
| 522 | E203 | 52.21 | C | ----- | first reported 0.052 mg/kg |
| 541 | E1064 | 55.0 | | ----- | |
| 551 | E1064 | 130 | | ----- | |
| 613 | E203 | 103.5 | | ----- | |
| 621 | E1064 | 66 | | ----- | |
| 633 | E1064 | 104 | C | ----- | first reported 0.0104 mg/kg |
| 663 | E1064 | 87.0 | | ----- | |
| 840 | E1064 | 92 | | ----- | |
| 872 | | ----- | | ----- | |
| 886 | | ----- | | ----- | |
| 902 | D1364 | 75 | | ----- | |
| 913 | E1064 | 100 | | ----- | |
| 963 | E1064 | 90 | | ----- | |
| 974 | E1064 | 95 | | ----- | |
| 1264 | E203 | 88 | | ----- | |
| 1862 | E1064 | 72 | | ----- | |
| 9014 | E203 | 135 | | ----- | |
| | | | | | Only E1064 |
| | normality | OK | | | OK |
| | n | 23 | | | 15 |
| | outliers | 0 | | | 0 |
| | mean (n) | 81.42 | | | 82.67 |
| | st.dev. (n) | 23.646 | | | 21.141 |
| | R(calc.) | 66.21 | | | 59.20 |
| | st.dev.(E1064:16) | (4.624) | | | 4.694 |
| | R(E1064:16) | (12.95) | | | 13.144 |
| | Compare | | | | |
| | R(E203) | (780) | | | |



## APPENDIX 2

### Number of participants per country

1 lab in ARGENTINA

1 lab in AUSTRALIA

1 lab in BELGIUM

1 lab in BRAZIL

1 lab in FINLAND

1 lab in FRANCE

1 lab in INDIA

1 lab in INDONESIA

1 lab in MEXICO

1 lab in NETHERLANDS

1 lab in PHILIPPINES

2 labs in RUSSIAN FEDERATION

1 lab in SAUDI ARABIA

1 lab in SOUTH AFRICA

1 lab in TAIWAN

1 lab in THAILAND

2 labs in TURKEY

2 labs in UNITED ARAB EMIRATES

3 labs in UNITED STATES OF AMERICA

1 lab in VIETNAM

**APPENDIX 3**

**Abbreviations**:

| | |
|---|---|
| C | = final 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 |
| fr. | = first reported |
| SDS | = Safety Data Sheet |

**Literature**: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