

Results of Proficiency Test
Ethanol (REN & Food)
November 2014

Organised by: Institute for Interlaboratory Studies (iis)
Spijkenisse, the Netherlands

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1. INTRODUCTION

Since 2007, a proficiency test for REN/Food grade Ethanol is organised every year by the Institute for Interlaboratory Studies. During the planning of the annual proficiency testing program 2014/2015, it was decided to continue the round robin for the analysis of REN/Food grade Ethanol.

In this interlaboratory study, 28 laboratories in 17 different countries have participated. See appendix 2 for the number of participants per country. In this report, the results of the 2014 proficiency test are presented and discussed. This report is also electronically available through the iis internet site www.iisnl.com.

2 SET-UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organiser of this proficiency test. Analysis for fit-for-use and homogeneity testing were subcontracted. It was decided to send one sample (1* 0.5 L of 95% REN/Food grade Ethanol, labelled #14232). Participants were requested to report rounded and unrounded results. The unrounded results were preferably used for statistical evaluation.

2.1 ACCREDITATION

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, is accredited in agreement with ISO/IEC 17043:10, (R007) since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This PT falls under the accredited scope. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Also customer's satisfaction is measured on 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 April 2014 (iis-protocol, version 3.3). The protocol can be downloaded from iis website <http://www.iisnl.com>.

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

The necessary bulk material for sample #14232 was obtained from a local trader. The approximately 50 litre bulk sample was, after homogenisation in a precleaned drum, divided over 50 amber glass bottles of 0.5 L and labelled #14232. The homogeneity of these subsamples was checked by determination of Density in accordance with ASTM D4052 and Water in accordance with ASTM D1364 on 8 stratified randomly selected samples.

| Sample | Density @ 20°C in kg/L | Water in %M/M |
|-----------------|------------------------|---------------|
| Sample #14232-1 | 0.80594 | 5.605 |
| Sample #14232-2 | 0.80594 | 5.638 |
| Sample #14232-3 | 0.80594 | 5.644 |
| Sample #14232-4 | 0.80594 | 5.644 |
| Sample #14232-5 | 0.80594 | 5.649 |
| Sample #14232-6 | 0.80594 | 5.634 |
| Sample #14232-7 | 0.80594 | 5.648 |
| Sample #14232-8 | 0.80595 | 5.652 |

table 1: Homogeneity test results of subsamples #14232

From the test results of table 1, the repeatabilities were calculated and compared with 0.3 times the corresponding target reproducibility in agreement with the procedure of ISO 13528, Annex B2 in the next table:

| | Density @ 20°C in kg/L | Water in %M/M |
|-----------------------|------------------------|---------------|
| r (Observed) | 0.00001 | 0.042 |
| reference method | ISO12185:96 | ASTM D1364:12 |
| 0.3 * R (ref. method) | 0.00015 | 0.043 |

table 2: Repeatability of subsamples #14232

The repeatabilities of the results from the homogeneity test were equal or less than the requirements of the respective standards. Therefore, homogeneity of all the prepared subsamples was assumed.

To each of the participating laboratories 1*0.5 L bottle of sample #14232 was sent on November 12, 2014.

2.5 STABILITY OF THE SAMPLES

The stability of Ethanol, 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 asked to determine on sample #14232: Density at 20°C, Non-volatile matter, Permanganate Time Test, pHe, Purity on dry basis, Strength (in %M/M and %V/V), Water (titrimetric) and UV transmittance at 300, 270, 260, 250, 240, 230 and 220nm.

To get comparable results a detailed report form, on which the units were prescribed as well as the required standards and a letter of instructions were prepared and made available on the data entry portal www.kpmd.co.uk/sgs-iis/. The detailed report form was also made available for download on the iis website www.iisnl.com.

A SDS and a form to confirm receipt of the samples were added to the sample package.

3 RESULTS

During four weeks after sample despatch, the results of the individual laboratories were received. The original reported results are tabulated per determination in appendix 1 of this report. The laboratories are presented by their code numbers.

Directly after deadline, a reminder fax was sent to those laboratories that had not yet reported any results at that moment.

Shortly after the deadline, the available results were screened for suspect data. A 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 results. Additional or corrected results are used for data analysis and original results are placed under 'Remarks' in the result tables in appendix 1.

3.1 STATISTICS

Statistical calculations were performed as described in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' (iis-protocol, version 3.3) of April 2014.

For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded results. 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. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation of the results should be used with due care.

According to ISO 5725 the original 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 (ref. 15). 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. When the uncertainty passed the evaluation, no remarks are made in the report. However, when the uncertainty failed the evaluation it is mentioned in the report and it will have consequences for the evaluation of the test results.

Finally, the reproducibilities were calculated from the standard deviations by multiplying these 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 analysis 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 standard. 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 (see appendix 3, nos.13-14). 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 spread of this interlaboratory study. The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used. In some cases, literature repeatability is available; in other cases, a reproducibility of a former iis proficiency test could be used and the Horwitz equation can be used to estimate target reproducibility.

The z-scores were calculated according to:

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

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 |

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, this in order to evaluate whether the reported test result is fit-for-use.

4. EVALUATION

In this proficiency test, some problems were encountered with the despatch of the samples. Four participants reported results after the final reporting date and three participants did not report any results at all. Not all laboratories were able to perform all analysis requested.

In total 25 laboratories reported 210 numerical results. Observed were 13 outlying results, which is 6.2%. In proficiency studies, outlier percentages of 3% - 7.5% are normal.

4.1 EVALUATION PER TEST

In this section, the results are discussed per test.

The methods, which were used by the various laboratories, are 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. D1363) and an added designation for the year that the method was adopted or revised (e.g. D1363:06). If applicable, a designation in parentheses is added to designate the year of reapproval (e.g. D1363:06(2011)). In the results tables of Appendix 1 only the method number and year of adoption or revision e.g. D1363:06 will be used.

All original data sets proved to have a normal Gaussian distribution

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

- Nonvolatile matter: Six participants reported a “less than” result and eleven a numerical value. The determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the requirements of ASTM D1353:13.
- Permanganate Time Test: This determination was problematic. No statistical outliers were observed. However, the calculated reproducibility is not in agreement with the requirements of ASTM D1363:06(2011).
- pHe: This determination was very problematic. No statistical outliers were observed. However, the calculated reproducibility was not at all in agreement with the requirements of ASTM D6423:08. Looking at the Kernel Density graph, the data appear to have a bimodal distribution. When both sets of data are evaluated separately the spread becomes smaller.
- Purity on dry basis: Regretfully, no standard test method with precision data exists. Therefore no significant conclusions could be drawn. No statistical outliers were observed. The calculated reproducibility is large in comparison to the calculated reproducibility of the previous proficiency test iis13C13 of November 2013 (0.018 vs 0.012).
- Strength(%M/M): This determination may not be problematic. One statistical outlier was observed. Regretfully, no standard test method with precision data exists. The calculated reproducibility, after rejection of the statistical outlier, is small in comparison to the calculated reproducibility in the previous proficiency test iis13C13 of November 2013 (0.053 vs 0.075).
- Strength (%V/V): This determination may not be problematic. Two statistical outlier were observed. However, the calculated reproducibility after rejection of the statistical outlier is in agreement with the reproducibility derived from the OIML table and ISO12185:96. The spread found is small in comparison to the calculated reproducibility of the previous proficiency test iis13C13 of November 2013 (0.028 vs. 0.041).
- Water: This determination was problematic. One statistical outlier was observed. The calculated reproducibility, after rejection of the statistical outlier, is not in agreement with the requirements of ASTM D1364:02(2012).
- UV absorbance: Regretfully, no standard test method with precision data exists. Therefore no significant conclusions were drawn.

Laboratory 357 and 859 reported results obtained with a 50 mm cell, these were converted to results of a 10 mm cell by dividing them by 5. Test results from laboratories 551, 1242 and 1835 were outliers for a number of wavelengths. Since the test results are not independent, the other values were excluded. In total 9 outlying results were found and 10 other test results were excluded.

For 270 nm, 230 nm and 220 nm, the calculated reproducibilities are small in comparison with the calculated reproducibilities from the previous proficiency test iis13C13 of November 2013. For 300 nm and 240 nm, the calculated reproducibilities are larger than in the PT of 2013.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the relevant standard and the reproducibility as found for the group of participating laboratories. The average results per sample, calculated reproducibilities and reproducibilities derived from literature standards (in casu ASTM, EN standards) or previous proficiency tests are compared in the next table.

| Parameter | unit | n | average | 2.8 *sd _R | R (lit) |
|------------------------|----------|----|---------|----------------------|---------|
| Density @ 20°C | kg/L | 22 | 0.8060 | 0.0001 | 0.0005 |
| Nonvolatile matter | mg/100mL | 11 | 0.8 | 1.2 | 2.1 |
| Permanganate Time Test | min. | 13 | 22.9 | 10.6 | 5.8 |
| pHe | | 11 | 6.94 | 1.35 | 0.52 |
| Purity on dry basis | %M/M | 9 | 99.994 | 0.018 | (0.012) |
| Strength | %M/M | 13 | 94.36 | 0.05 | (0.08) |
| Strength | %V/V | 17 | 96.36 | 0.03 | 0.06 |
| Water | %M/M | 14 | 5.589 | 0.232 | 0.142 |
| UV-absorbance 300 nm | | 11 | 0.001 | 0.003 | (0.002) |
| UV-absorbance 270 nm | | 11 | 0.019 | 0.005 | (0.018) |
| UV-absorbance 260 nm | | 10 | 0.021 | 0.007 | n.a. |
| UV-absorbance 250 nm | | 11 | 0.044 | 0.009 | n.a. |
| UV-absorbance 240 nm | | 12 | 0.115 | 0.016 | (0.011) |
| UV-absorbance 230 nm | | 12 | 0.213 | 0.020 | (0.035) |
| UV-absorbance 220 nm | | 12 | 0.306 | 0.036 | (0.456) |

Table 3: Reproducibilities of sample #14232

() Results between brackets are compared with the observed reproducibility of the previous proficiency test.

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2014 WITH PREVIOUS PTs

| | <i>November 2014</i> | <i>November 2013</i> | <i>November 2012</i> | <i>November 2011</i> | <i>November 2010</i> |
|--------------------------------|----------------------|----------------------|----------------------|----------------------|----------------------|
| Number of reporting labs | 25 | 24 | 24 | 23 | 28 |
| Number of results reported | 210 | 160 | 169 | 151 | 189 |
| Number of statistical outliers | 13 | 9 | 5 | 9 | 13 |
| Percentage outliers | 6.2% | 5.6% | 3.0% | 6.0% | 6.9% |

Table 4: comparison with previous proficiency tests.

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

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

| Parameter | <i>November 2014</i> | <i>November 2013</i> | <i>November 2012</i> | <i>November 2011</i> | <i>November 2010</i> |
|------------------------|----------------------|----------------------|----------------------|----------------------|----------------------|
| Density @ 20°C | ++ | ++ | ++ | ++ | ++ |
| Nonvolatile matter | ++ | n.e. | ++ | n.e. | ++ |
| Permanganate Time Test | - | + | -- | (--) | (--) |
| pHe | -- | n.e. | n.e. | n.e. | n.e. |
| Purity on dry basis | (-) | (-) | (+) | (+) | (--) |
| Strength %M/M | (+) | (+) | (--) | ++ | (--) |
| Strength %V/V | + | + | -- | ++ | ++ |
| Water | - | -- | -- | +/- | ++ |
| UV-absorbance 300 nm | (-) | (++) | (-) | (--) | (++) |
| UV-absorbance 270 nm | (++) | (+/-) | (--) | (-) | (++) |
| UV-absorbance 240 nm | (-) | (++) | (+/-) | (-) | (++) |
| UV-absorbance 230 nm | (+) | (-) | (++) | (-) | (+) |
| UV-absorbance 220 nm | (++) | (--) | (--) | (-) | (-) |

Table 5: comparison determinations of sample #14232 against the standard

() results between brackets are compared with the observed reproducibility of the previous round robin

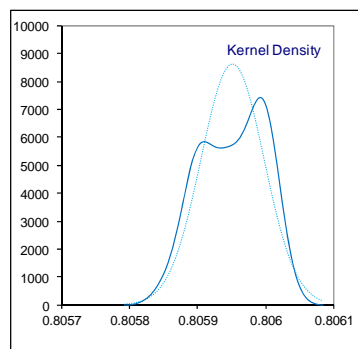
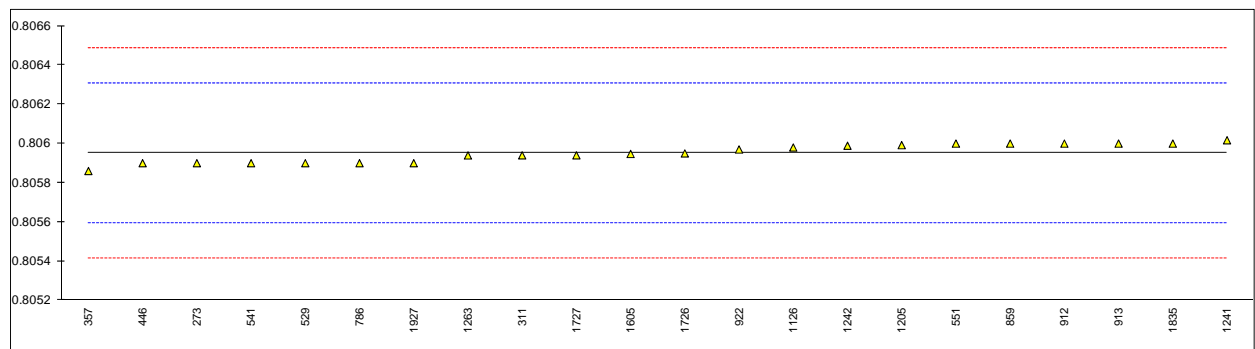
The performance of the determinations against the requirements of the respective standards is listed in the above table. The following performance categories were used:

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

APPENDIX 1

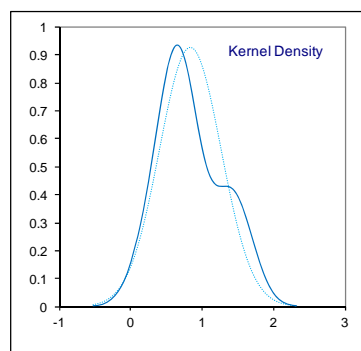
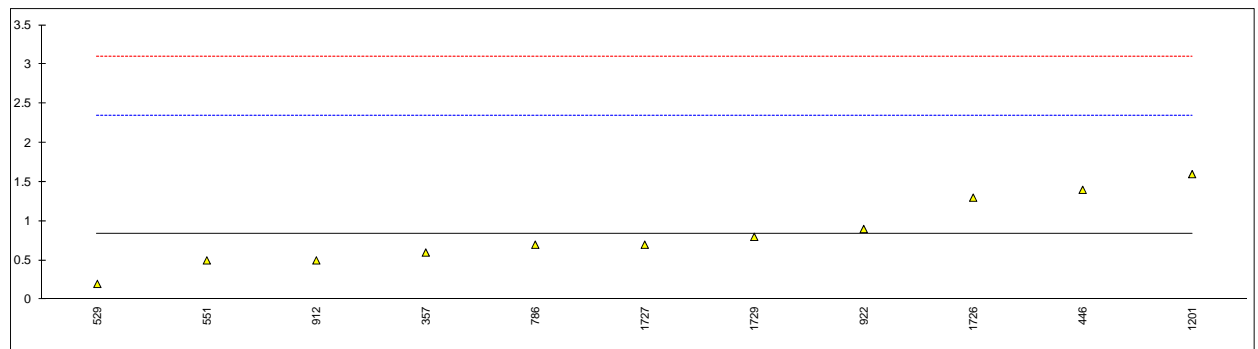
Determination of Density at 20°C on sample #14232; results in kg/L

| lab | method | value | mark | z(targ) | remarks |
|----------------|----------|-----------|------|---------|------------------------|
| 273 | D4052 | 0.8059 | | -0.29 | |
| 311 | D4052 | 0.80594 | | -0.06 | |
| 357 | D4052 | 0.80586 | | -0.51 | |
| 446 | D4052 | 0.8059 | | -0.29 | |
| 522 | | ----- | | ----- | |
| 529 | D4052 | 0.8059 | | -0.29 | |
| 541 | D4052 | 0.8059 | | -0.29 | |
| 551 | D4052 | 0.80600 | | 0.27 | |
| 559 | | ----- | | ----- | |
| 786 | D4052 | 0.8059 | | -0.29 | |
| 859 | D4052 | 0.8060 | | 0.27 | |
| 912 | D4052 | 0.8060 | | 0.27 | |
| 913 | D4052 | 0.8060 | | 0.27 | |
| 922 | D4052 | 0.80597 | | 0.11 | |
| 1126 | D4052 | 0.80598 | C | 0.16 | First reported: 805.98 |
| 1201 | | ----- | | ----- | |
| 1205 | in house | 0.805992 | | 0.23 | |
| 1241 | INH-50 | 0.806017 | | 0.37 | |
| 1242 | D4052 | 0.805989 | | 0.21 | |
| 1263 | ISO12185 | 0.8059395 | | -0.07 | |
| 1574 | | ----- | | ----- | |
| 1605 | D4052 | 0.805947 | | -0.02 | |
| 1726 | D4052 | 0.80595 | | -0.01 | |
| 1727 | D4052 | 0.80594 | | -0.06 | |
| 1729 | | ----- | | ----- | |
| 1835 | D4052 | 0.8060 | | 0.27 | |
| 1927 | INH-4052 | 0.8059 | | -0.29 | |
| 1933 | | ----- | | ----- | |
| normality | | OK | | | |
| n | | 22 | | | |
| outliers | | 0 | | | |
| mean (n) | | 0.805951 | | | |
| st.dev. (n) | | 0.0000463 | | | |
| R(calc.) | | 0.000130 | | | |
| R(ISO12185:96) | | 0.000500 | | | |



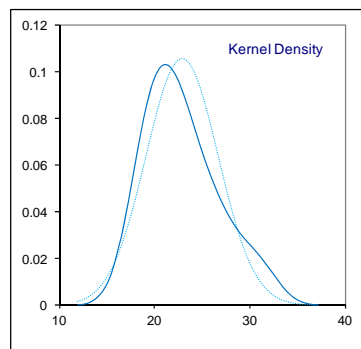
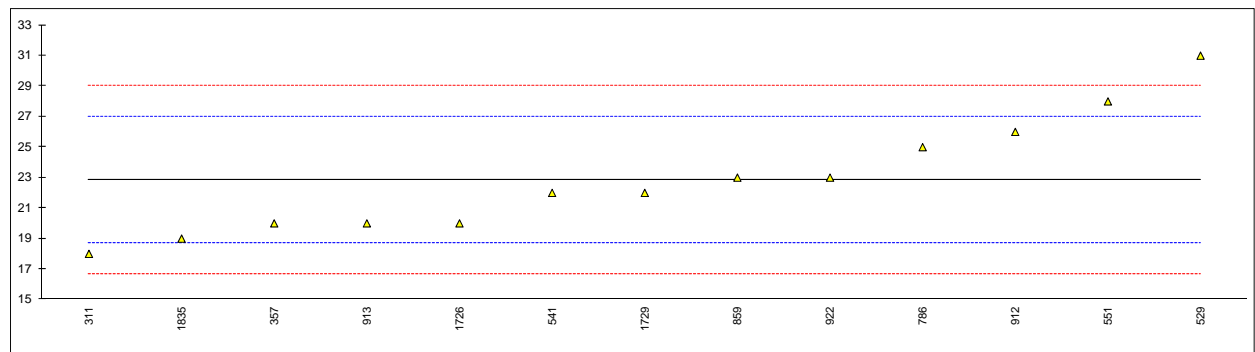
Determination of Nonvolatile matter on sample #14232; results in mg/100mL

| lab | method | value | mark | z(targ) | remarks |
|-------------|---------|-------|------|---------|---------|
| 273 | | ---- | | ---- | |
| 311 | D1353 | <10 | | ---- | |
| 357 | D1353 | 0.6 | | -0.31 | |
| 446 | D1353 | 1.4 | | 0.75 | |
| 522 | | ---- | | ---- | |
| 529 | D1353 | 0.2 | | -0.84 | |
| 541 | D1353 | <1 | | ---- | |
| 551 | D1353 | 0.5 | | -0.45 | |
| 559 | | ---- | | ---- | |
| 786 | D1353 | 0.70 | | -0.18 | |
| 859 | D1353 | <1 | | ---- | |
| 912 | D1353 | 0.5 | | -0.45 | |
| 913 | D1353 | <1.0 | | ---- | |
| 922 | D1353 | 0.9 | | 0.08 | |
| 1126 | | ---- | | ---- | |
| 1201 | D1353 | 1.6 | | 1.01 | |
| 1205 | | ---- | | ---- | |
| 1241 | | ---- | | ---- | |
| 1242 | | ---- | | ---- | |
| 1263 | D1353 | <1.0 | | ---- | |
| 1574 | | ---- | | ---- | |
| 1605 | | ---- | | ---- | |
| 1726 | EN15691 | 1.30 | | 0.62 | |
| 1727 | | 0.7 | | -0.18 | |
| 1729 | D1353 | 0.80 | | -0.05 | |
| 1835 | EN15691 | <10 | | ---- | |
| 1927 | | ---- | | ---- | |
| 1933 | | ---- | | ---- | |
| normality | | OK | | | |
| n | | 11 | | | |
| outliers | | 0 | | | |
| mean (n) | | 0.84 | | | |
| st.dev. (n) | | 0.430 | | | |
| R(calc.) | | 1.20 | | | |
| R(D1353:13) | | 2.11 | | | |



Determination of Permanganate Time Test @ 15 °C on sample #14232; results in minutes

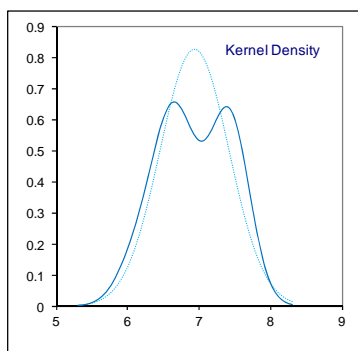
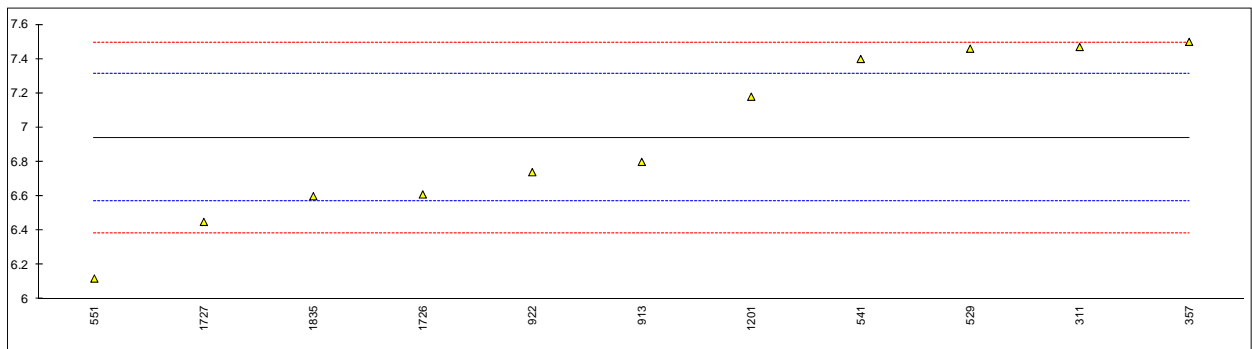
| lab | method | value | mark | z(targ) | remarks |
|-------------|--------|-------|------|---------|---------|
| 273 | | ---- | | ---- | |
| 311 | D1363 | 18 | | -2.36 | |
| 357 | D1363 | 20 | | -1.38 | |
| 446 | | ---- | | ---- | |
| 522 | | ---- | | ---- | |
| 529 | D1363 | 31.0 | | 3.97 | |
| 541 | D1363 | 22 | | -0.41 | |
| 551 | D1363 | 28 | | 2.51 | |
| 559 | | ---- | | ---- | |
| 786 | D1363 | 25 | | 1.05 | |
| 859 | D1363 | 23 | | 0.07 | |
| 912 | D1363 | 26 | | 1.53 | |
| 913 | D1363 | 20 | | -1.38 | |
| 922 | D1363 | 23 | | 0.07 | |
| 1126 | | ---- | | ---- | |
| 1201 | | ---- | | ---- | |
| 1205 | | ---- | | ---- | |
| 1241 | | ---- | | ---- | |
| 1242 | | ---- | | ---- | |
| 1263 | | ---- | | ---- | |
| 1574 | | ---- | | ---- | |
| 1605 | | ---- | | ---- | |
| 1726 | D1363 | 20 | | -1.38 | |
| 1727 | | ---- | | ---- | |
| 1729 | D1363 | 22 | | -0.41 | |
| 1835 | D1363 | 19 | | -1.87 | |
| 1927 | | ---- | | ---- | |
| 1933 | | ---- | | ---- | |
| normality | | OK | | | |
| n | | 13 | | | |
| outliers | | 0 | | | |
| mean (n) | | 22.85 | | | |
| st.dev. (n) | | 3.783 | | | |
| R(calc.) | | 10.59 | | | |
| R(D1363:06) | | 5.76 | | | |



Determination of pHe on sample #14232;

| lab | method | value | mark | z(targ) | remarks |
|------|---------|-------|------|---------|---------|
| 273 | | ---- | | ---- | |
| 311 | D6423 | 7.47 | | 2.86 | |
| 357 | D6423 | 7.5 | | 3.02 | |
| 446 | | ---- | | ---- | |
| 522 | | ---- | | ---- | |
| 529 | D6423 | 7.46 | | 2.80 | |
| 541 | D6423 | 7.40 | | 2.48 | |
| 551 | D6423 | 6.12 | | -4.41 | |
| 559 | | ---- | | ---- | |
| 786 | | ---- | | ---- | |
| 859 | | ---- | | ---- | |
| 912 | | ---- | | ---- | |
| 913 | D6423 | 6.8 | | -0.75 | |
| 922 | D6423 | 6.74 | | -1.07 | |
| 1126 | | ---- | | ---- | |
| 1201 | EN15490 | 7.18 | | 1.30 | |
| 1205 | | ---- | | ---- | |
| 1241 | | ---- | | ---- | |
| 1242 | | ---- | | ---- | |
| 1263 | | ---- | | ---- | |
| 1574 | | ---- | | ---- | |
| 1605 | | ---- | | ---- | |
| 1726 | EN15490 | 6.61 | | -1.77 | |
| 1727 | | 6.45 | | -2.63 | |
| 1729 | | ---- | | ---- | |
| 1835 | EN15490 | 6.6 | | -1.83 | |
| 1927 | | ---- | | ---- | |
| 1933 | | ---- | | ---- | |

| | | | | |
|-------------|--------|--|---------|---------|
| | | | Group 1 | Group 2 |
| normality | OK | | | |
| n | 11 | | 6 | 5 |
| outliers | 0 | | 0 | 0 |
| mean (n) | 6.939 | | 6.553 | 7.402 |
| st.dev. (n) | 0.4828 | | 0.2448 | 0.1293 |
| R(calc.) | 1.352 | | 0.685 | 0.3621 |
| R(D6423:08) | 0.520 | | 0.520 | 0.520 |

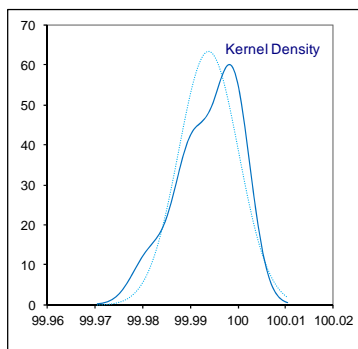
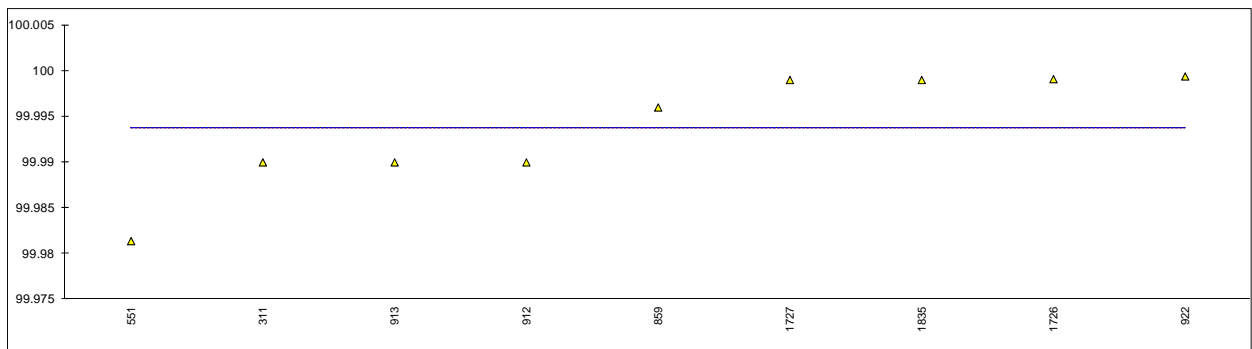


Determination of Purity of Ethanol on dry basis on sample #14232; results in %M/M

| lab | method | value | mark | z(targ) | remarks |
|------|----------|----------|------|---------|---------|
| 273 | | ---- | | ---- | |
| 311 | INH-529 | 99.99 | | ---- | |
| 357 | EN15721 | >99.99 | | ---- | |
| 446 | | ---- | | ---- | |
| 522 | | ---- | | ---- | |
| 529 | | ---- | | ---- | |
| 541 | | ---- | | ---- | |
| 551 | INH-1313 | 99.98139 | | ---- | |
| 559 | | ---- | | ---- | |
| 786 | | ---- | | ---- | |
| 859 | | 99.996 | | ---- | |
| 912 | INH-0001 | 99.99 | | ---- | |
| 913 | D5501 | 99.99 | | ---- | |
| 922 | INH-0001 | 99.9994 | | ---- | |
| 1126 | | ---- | | ---- | |
| 1201 | | ---- | | ---- | |
| 1205 | | ---- | | ---- | |
| 1241 | | ---- | | ---- | |
| 1242 | | ---- | | ---- | |
| 1263 | | ---- | | ---- | |
| 1574 | | ---- | | ---- | |
| 1605 | | ---- | | ---- | |
| 1726 | EN15721 | 99.9991 | | ---- | |
| 1727 | | 99.999 | | ---- | |
| 1729 | | ---- | | ---- | |
| 1835 | in house | 99.999 | | ---- | |
| 1927 | | ---- | | ---- | |
| 1933 | | ---- | | ---- | |

normality OK
n 9
outliers 0
mean (n) 99.9938
st.dev. (n) 0.00628
R(calc.) 0.0176
R(lit.) unknown

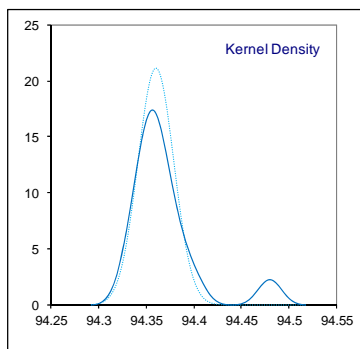
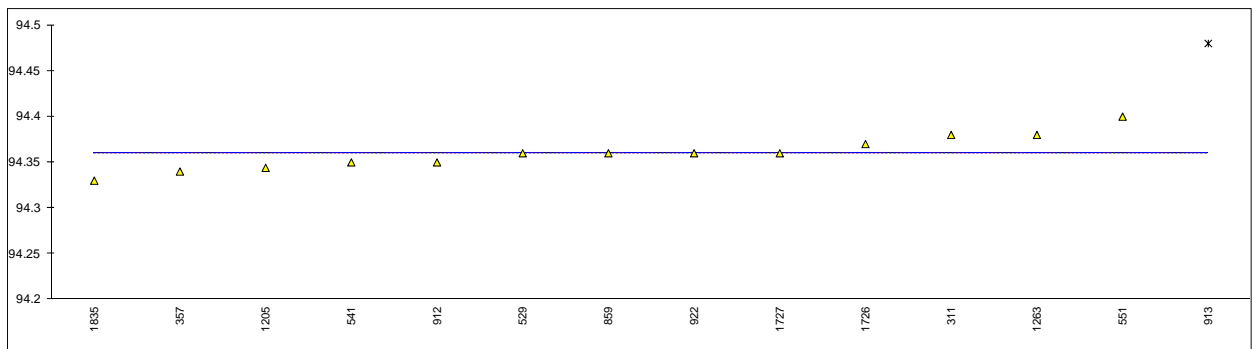
Compare R(iis13C13) = 0.0123



Determination of Strength on sample #14232; results in %M/M

| lab | method | value | mark | z(targ) | remarks |
|-------------|------------|---------|---------|---------|---------|
| 273 | | ---- | | ---- | |
| 311 | Table OIML | 94.38 | | ---- | |
| 357 | Table OIML | 94.34 | | ---- | |
| 446 | | ---- | | ---- | |
| 522 | | ---- | | ---- | |
| 529 | D4052 | 94.36 | | ---- | |
| 541 | Table OIML | 94.35 | | ---- | |
| 551 | INH-15639 | 94.4 | | ---- | |
| 559 | | ---- | | ---- | |
| 786 | | ---- | | ---- | |
| 859 | Table OIML | 94.36 | | ---- | |
| 912 | Table OIML | 94.35 | | ---- | |
| 913 | D5501 | 94.48 | G(0.01) | ---- | |
| 922 | Table OIML | 94.36 | | ---- | |
| 1126 | | ---- | | ---- | |
| 1201 | | ---- | | ---- | |
| 1205 | Table OIML | 94.344 | | ---- | |
| 1241 | | ---- | | ---- | |
| 1242 | | ---- | | ---- | |
| 1263 | Table OIML | 94.38 | | ---- | |
| 1574 | | ---- | | ---- | |
| 1605 | | ---- | | ---- | |
| 1726 | Table OIML | 94.37 | | ---- | |
| 1727 | Table OIML | 94.36 | | ---- | |
| 1729 | | ---- | | ---- | |
| 1835 | Table OIML | 94.33 | | ---- | |
| 1927 | | ---- | | ---- | |
| 1933 | | ---- | | ---- | |
| normality | | OK | | | |
| n | | 13 | | | |
| outliers | | 1 | | | |
| mean (n) | | 94.360 | | | |
| st.dev. (n) | | 0.0188 | | | |
| R(calc.) | | 0.053 | | | |
| R(lit.) | | unknown | | | |

Compare R(iis13C13) = 0.075

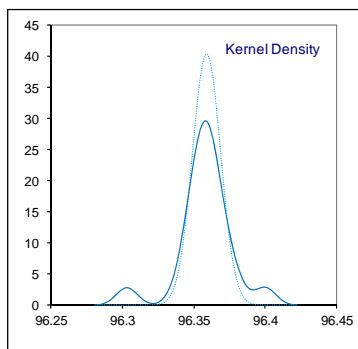
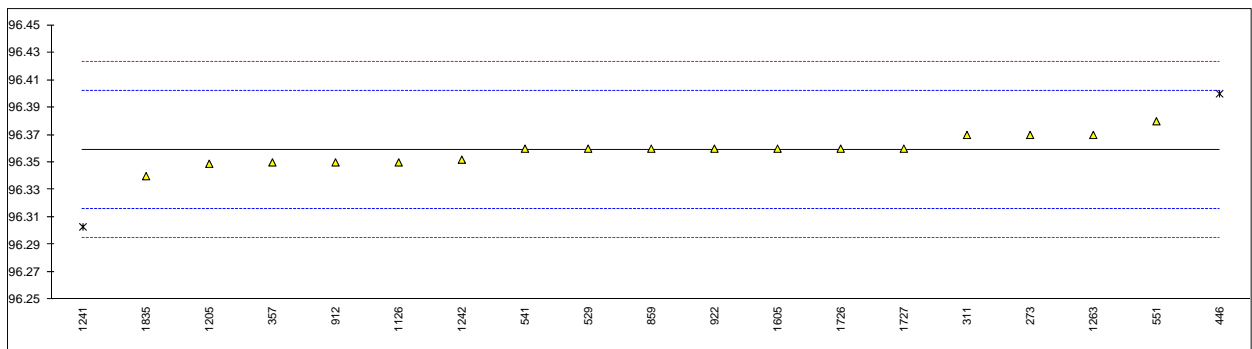


Determination of Strength on sample #14232; results in %V/V

| lab | method | value | mark | z(target) | remarks |
|------|------------|--------|---------|-----------|---------|
| 273 | Table OIML | 96.37 | | 0.52 | |
| 311 | Table OIML | 96.37 | | 0.52 | |
| 357 | Table OIML | 96.35 | | -0.41 | |
| 446 | Table OIML | 96.40 | G(0.05) | 1.92 | |
| 522 | | ----- | | ----- | |
| 529 | D4052 | 96.36 | | 0.05 | |
| 541 | Table OIML | 96.36 | | 0.05 | |
| 551 | INH-15639 | 96.38 | | 0.99 | |
| 559 | | ----- | | ----- | |
| 786 | | ----- | | ----- | |
| 859 | Table OIML | 96.36 | | 0.05 | |
| 912 | Table OIML | 96.35 | | -0.41 | |
| 913 | | ----- | | ----- | |
| 922 | Table OIML | 96.36 | | 0.05 | |
| 1126 | Table OIML | 96.35 | | -0.41 | |
| 1201 | | ----- | | ----- | |
| 1205 | Table OIML | 96.349 | | -0.46 | |
| 1241 | INH-50 | 96.303 | G(0.05) | -2.61 | |
| 1242 | Table OIML | 96.352 | | -0.32 | |
| 1263 | Table OIML | 96.37 | | 0.52 | |
| 1574 | | ----- | | ----- | |
| 1605 | Table OIML | 96.360 | | 0.05 | |
| 1726 | Table OIML | 96.36 | | 0.05 | |
| 1727 | Table OIML | 96.36 | | 0.05 | |
| 1729 | | ----- | | ----- | |
| 1835 | Table OIML | 96.34 | | -0.88 | |
| 1927 | | ----- | | ----- | |
| 1933 | | ----- | | ----- | |

normality OK
n 17
outliers 2
mean (n) 96.359
st.dev. (n) 0.0099
R(calc.) 0.028
R(OIML table) 0.060

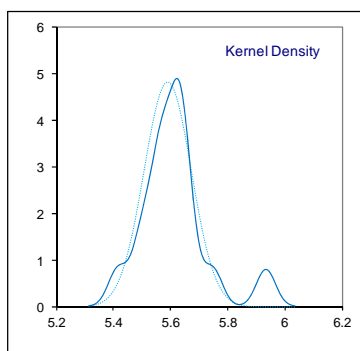
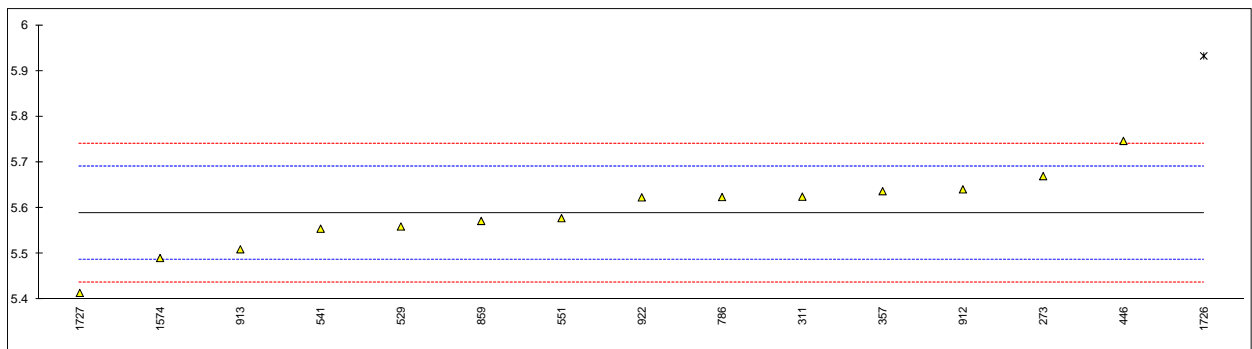
Compare R(iis13C13) = 0.041



Determination of Water (Titrimetric) on sample #14232; results in %M/M

| lab | method | value | mark | z(targ) | remarks |
|------|--------|--------|---------|---------|-----------------------|
| 273 | E203 | 5.670 | | 1.60 | |
| 311 | D1364 | 5.625 | | 0.71 | |
| 357 | E203 | 5.637 | | 0.94 | |
| 446 | E203 | 5.747 | | 3.12 | |
| 522 | | ----- | | ----- | |
| 529 | D1364 | 5.5598 | | -0.58 | |
| 541 | E1064 | 5.555 | | -0.67 | |
| 551 | D1364 | 5.578 | | -0.22 | |
| 559 | | ----- | | ----- | |
| 786 | D1364 | 5.6242 | C | 0.69 | First reported: 6.099 |
| 859 | D1364 | 5.572 | | -0.34 | |
| 912 | E203 | 5.641 | | 1.02 | |
| 913 | D1364 | 5.51 | | -1.56 | |
| 922 | E203 | 5.6235 | | 0.68 | |
| 1126 | | ----- | | ----- | |
| 1201 | | ----- | | ----- | |
| 1205 | | ----- | | ----- | |
| 1241 | | ----- | | ----- | |
| 1242 | | ----- | | ----- | |
| 1263 | | ----- | | ----- | |
| 1574 | INH-76 | 5.4909 | | -1.94 | |
| 1605 | | ----- | | ----- | |
| 1726 | D1364 | 5.9328 | G(0.05) | 6.78 | |
| 1727 | | 5.4146 | | -3.45 | |
| 1729 | | ----- | | ----- | |
| 1835 | | ----- | | ----- | |
| 1927 | | ----- | | ----- | |
| 1933 | | ----- | | ----- | |

normality OK
n 14
outliers 1
mean (n) 5.5891
st.dev. (n) 0.08272
R(calc.) 0.2316
R(D1364:02) 0.1418

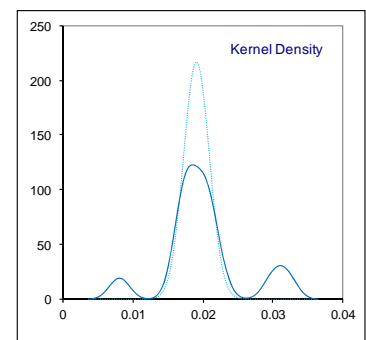
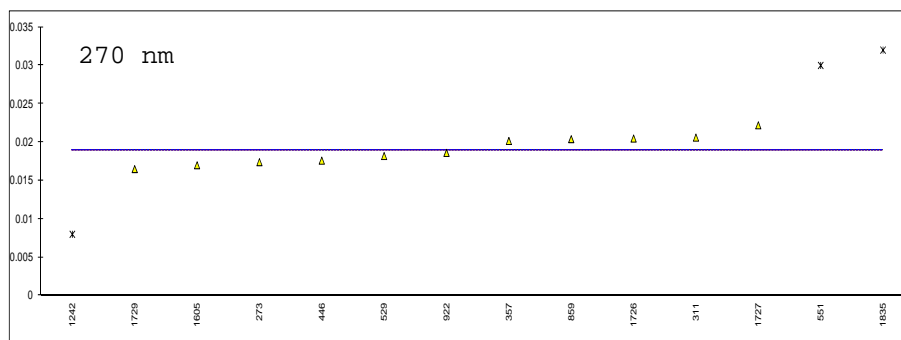
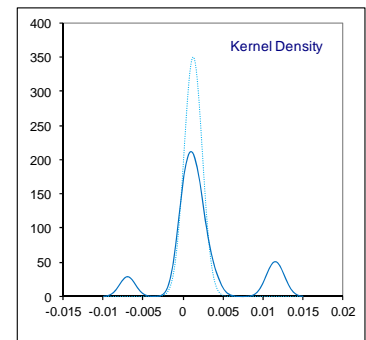
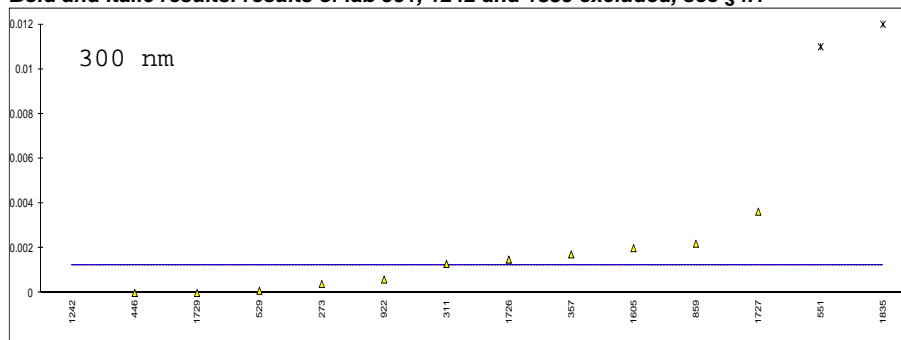


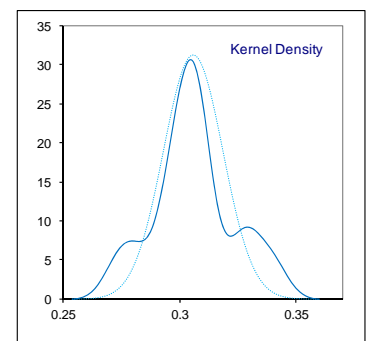
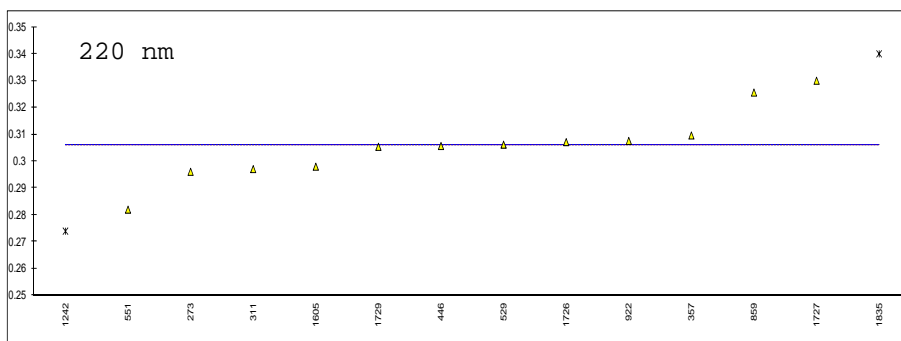
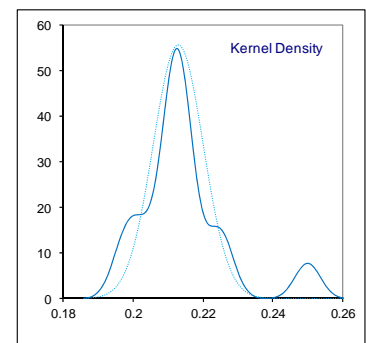
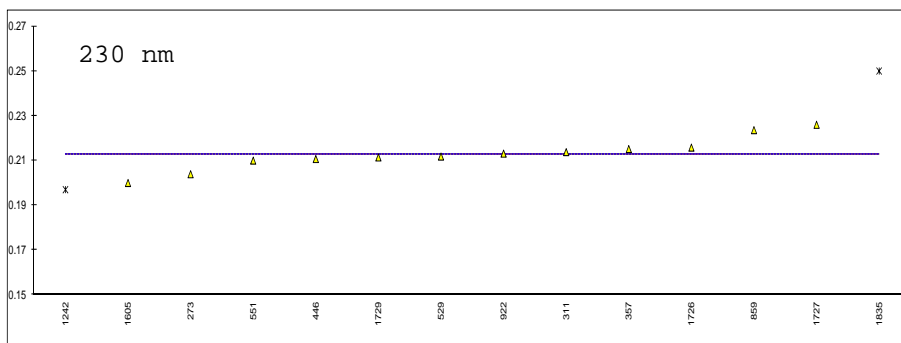
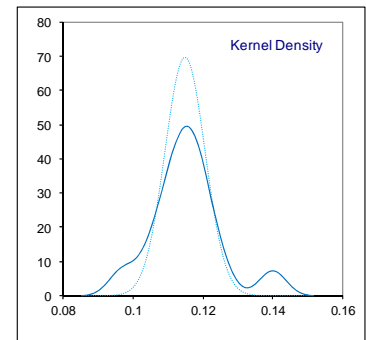
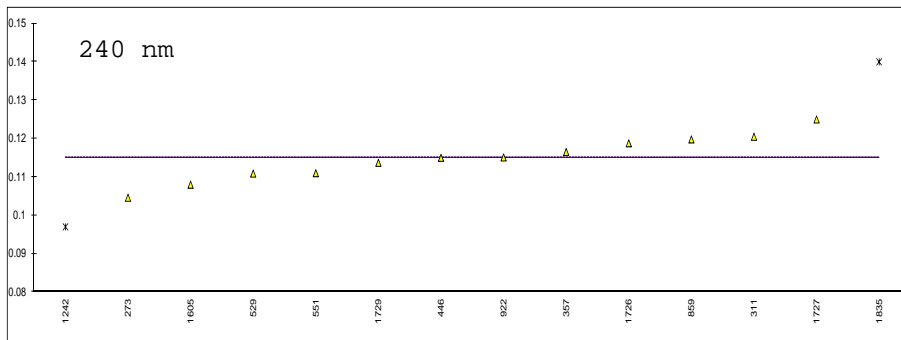
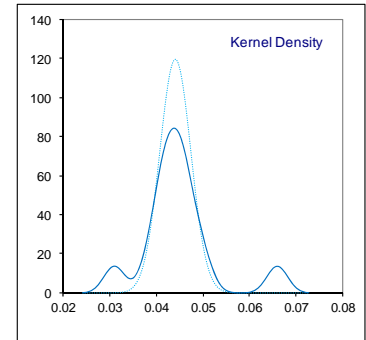
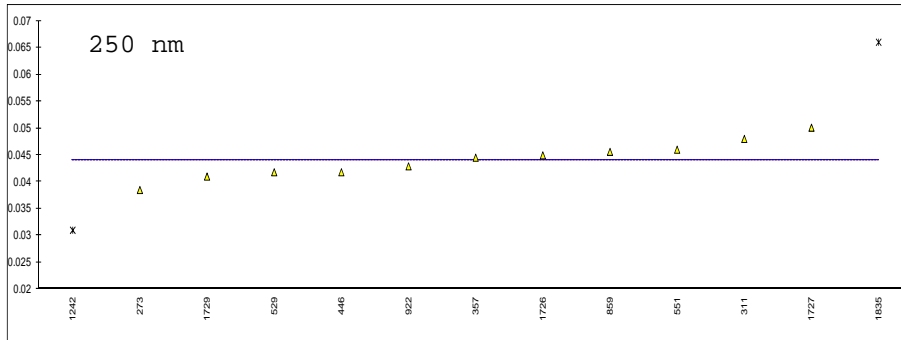
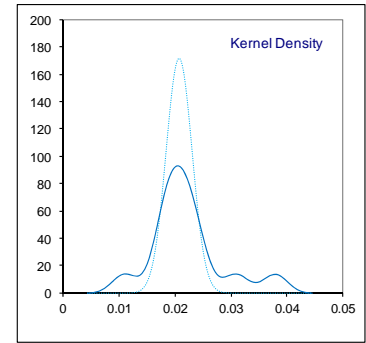
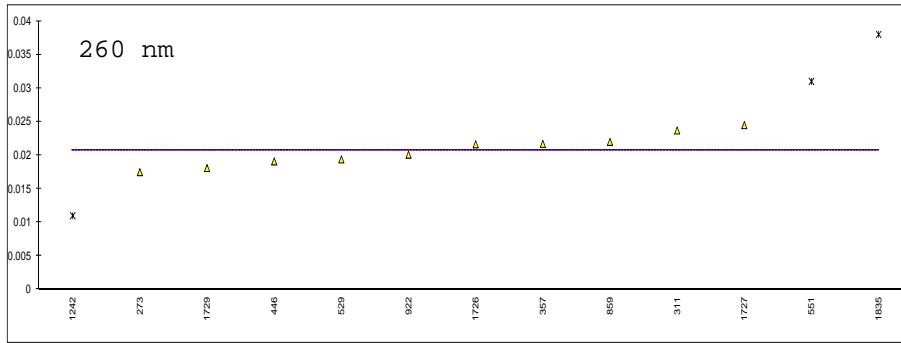
Determination of UV absorbance on sample #14232;

| lab | method | cuvette | 300nm | 270nm | 260nm | 250nm | 240nm | 230nm | 220nm | Pass/Fail |
|-------------|----------|---------|---------------|--------------|--------------|--------------|--------------|--------------|--------------|-------------|
| 273 | IMPCA004 | ---- | 0.0004 | 0.0174 | 0.0175 | 0.0385 | 0.1046 | 0.2039 | 0.2961 | ---- |
| 311 | INH-094 | 10 mm | 0.0013 | 0.0206 | 0.0237 | 0.0480 | 0.1205 | 0.2138 | 0.2971 | Fail |
| 357 | INH-13 | 50 mm | 0.0017* | 0.0202* | 0.0217* | 0.0445* | 0.1165* | 0.2152* | 0.3096* | Fail |
| 446 | INH-CM13 | 10 mm | 0 | 0.0176 | 0.0191 | 0.0418 | 0.1150 | 0.2107 | 0.3057 | Fail |
| 522 | | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- |
| 529 | | 10 mm | 0.0001 | 0.0182 | 0.0194 | 0.0418 | 0.1109 | 0.2118 | 0.3062 | Pass |
| 541 | | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- |
| 551 | INH-3063 | 10 mm | 0.011 | 0.030 | 0.031 | 0.046 | 0.111 | 0.210 | 0.282 | Fail |
| 559 | | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- |
| 786 | | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- |
| 859 | | 50 mm | 0.0022* | 0.020* | 0.022* | 0.0456* | 0.1198* | 0.2236* | 0.3256* | Fail |
| 912 | | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- |
| 913 | | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- |
| 922 | INH-UV | 10 mm | 0.0006 | 0.0186 | 0.0201 | 0.0429 | 0.1151 | 0.2131 | 0.3076 | Fail |
| 1126 | | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- |
| 1201 | | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- |
| 1205 | | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- |
| 1241 | | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- |
| 1242 | | 10 mm | -0.007 | 0.008 | 0.011 | 0.031 | 0.097 | 0.197 | 0.274 | Pass |
| 1263 | | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- |
| 1574 | | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- |
| 1605 | | 10 mm | 0.002 | 0.017 | ---- | ---- | 0.108 | 0.200 | 0.298 | ---- |
| 1726 | in house | 10 mm | 0.00149 | 0.02048 | 0.02165 | 0.04495 | 0.11880 | 0.21580 | 0.30716 | Pass |
| 1727 | | 10 mm | 0.00363 | 0.02220 | 0.02450 | 0.05010 | 0.125 | 0.226 | 0.33 | Pass |
| 1729 | in house | 10 mm | 0.000 | 0.0165 | 0.0181 | 0.0410 | 0.1137 | 0.2114 | 0.3054 | ---- |
| 1835 | | 10mm | 0.012 | 0.032 | 0.038 | 0.066 | 0.140 | 0.250 | 0.340 | Pass |
| 1927 | | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- |
| 1933 | | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- |
| normality | | | OK | OK | OK | OK | OK | OK | OK | |
| n | | | 11 | 11 | 10 | 11 | 12 | 12 | 12 | 11 |
| outliers | | | 3 | 0 (+3ex) | 3 | 2 | 0 (+2ex) | 1 (+1ex) | 0 (+2ex) | 0 (+2ex) |
| mean (n) | | | 0.0012 | 0.0190 | 0.0208 | 0.0441 | 0.1149 | 0.2129 | 0.3059 | |
| st.dev. (n) | | | 0.00114 | 0.00184 | 0.00232 | 0.00333 | 0.00573 | 0.00716 | 0.01277 | Fail: 6 |
| R(calc.) | | | 0.0032 | 0.0052 | 0.0065 | 0.0093 | 0.0161 | 0.0201 | 0.0358 | Pass:3 |
| R(lit) | | | unknown | unknown | unknown | unknown | unknown | unknown | unknown | (+2ex) |
| R(iis13C13) | | | 0.0023 | 0.0183 | | | 0.0105 | 0.0350 | 0.4560 | |

*) Lab 357 and 859 used a 50 mm cell. The reported values were divided by 5 for evaluation.
 Lab 357 reported: 0.0086 – 0.1008 – 0.1085 – 0.2226 – 0.5826 – 1.0758 – 1.5481
 Lab 859 reported: 0.011 – 0.102 – 0.110 – 0.228 – 0.599 – 1.118 – 1.628

Bold and underlined results: outliers according to Dixon/Grubbs
Bold and italic results: results of lab 551, 1242 and 1835 excluded, see §4.1





APPENDIX 2

Number of participants per country

- 1 lab in ARGENTINA
- 1 lab in AUSTRIA
- 2 labs in BELGIUM
- 2 labs in BRAZIL
- 1 lab in CHINA, People's Republic
- 1 lab in FINLAND
- 1 lab in FRANCE
- 1 lab in HONG KONG
- 2 labs in INDIA
- 2 labs in MEXICO
- 5 labs in NETHERLANDS
- 2 labs in PAKISTAN
- 1 lab in RUSSIAN FEDERATION
- 1 lab in SOUTH AFRICA
- 3 labs in SPAIN
- 1 lab in THAILAND
- 1 lab in UNITED KINGDOM

APPENDIX 3

Abbreviations:

| | |
|----------|--|
| C | = final result after checking of first reported suspect 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 outlier test |
| R(0.05) | = straggler in Rosner outlier test |
| E | = error in calculations |
| ex | = excluded from calculations |
| n.a. | = not applicable |
| OILM | = International Organization of Legal Metrology |
| U | = unit error |
| SDS | = safety data sheet |

Literature:

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- 4 ASTM E1301-03
- 5 ISO13528-05
- 5 ISO 5725-86
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- 15 Bernard Rosner, Percentage Points for a Generalized ESD Many-Outlier Procedure, *Technometrics*, 25(2), pp. 165-172, (1983)