Results of Proficiency Test REN/Food Ethanol November 2011

Institute for Interlaboratory Studies (iis) Organised by:

Spijkenisse, the Netherlands

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1. INTRODUCTION

Since 2007, a proficiency test for REN/Food Ethanol is organised every year by the Institute for Interlaboratory Studies. During the planning of the annual proficiency testing program 2011/2012, it was decided to continue the round robin for the analysis of REN/Food grade Ethanol.

In this interlaboratory study, 26 laboratories in 13 different countries have participated. See appendix 3 for the number of participants per country. In this report, the results of the proficiency test are presented and discussed.

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 #11122). 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 17043:10, (R007) since January 2000, by the Dutch Accreditation Council (Raad voor Accreditatie). This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentially 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' (iis-protocol, version 3.2) of January 2010.

2.3 CONFIDENTIALITY STATEMENT

All data present 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 #11122 was obtained from a local trader. The approximately 25 litre bulk sample was, after homogenisation in a precleaned can, divided over 48 amber glass bottles of 0.5 L and labelled #11122. The homogeneity of these subsamples was checked by determination of Density in accordance with ASTM D4052:11 and Water in accordance with ASTM D1364:07 on 8 stratified randomly selected samples.

| Sample | Density @ 20℃ in kg/L | Water in %M/M |
|-----------------|-----------------------|---------------|
| Sample #11122-1 | 0.80467 | 5.634 |
| Sample #11122-2 | 0.80471 | 5.634 |
| Sample #11122-3 | 0.80471 | 5.629 |
| Sample #11122-4 | 0.80471 | 5.629 |
| Sample #11122-5 | 0.80472 | 5.595 |
| Sample #11122-6 | 0.80472 | 5.615 |
| Sample #11122-7 | 0.80471 | 5.614 |
| Sample #11122-8 | 0.80471 | 5.627 |

table 1: Homogeneity tests of subsamples #11122

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) | 0.00004 | 0.037 |
| reference method | ASTM D4052:11 | ASTM D1364:07 |
| 0.3 * R (ref. method) | 0.00015 | 0.043 |

table 2: Repeatability of subsamples #11122

The repeatabilities of the results from the homogeneity test were in agreement with 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 #11122 was sent on December 2, 2011.

2.5 ANALYSES

The participants were asked to determine on sample #11122: Density @ 20°C, Nonvolatile matter, Permanganate Time Test, Purity on dry basis, Water (titrimetric), Strength (in %V/V and %M/M) and UV transmittance at 300, 270, 240, 230 and 220nm. To get comparable results a detailed report form, on which the units were printed, was sent together with each sample. In addition, a letter of instructions and a SDS were added to the 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 3.2) of January 2010.

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. After removal of outliers, this check was repeated. Not all data sets proved to have a normal distribution, in which cases the statistical evaluation should be used with due care.

In accordance with ISO 5725 (1986 and 1994) the original results per determination were submitted subsequently to Dixon and Grubbs outlier tests. Outliers are marked by D(0.01) for the Dixon test, by G(0.01) or DG(0.01) for the Grubbs test. Stragglers are marked by D(0.05) for the Dixon test, by G(0.05) or DG(0.05) for the Grubbs 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 under 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 4, nr.13-14).

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 - 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 used, this in order to evaluate the fit-for-useness of the reported test result.

4. EVALUATION

In this proficiency test, some problems were encountered with 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. Finally, the 23 reporting laboratories did send in 151 (numerical) results. Observed were 9 outlying results, which is 6.0%. 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 4.

On the registration form the participants were asked to fill out the analytical details regarding the strength determination and UV absorbance. Twenty laboratories answered the questions fully or partially (See Appendix 2). From the analytical details none of the participants did perform a distillation before the strength determination and all participants used the density meter for the strength determination.

A not normal distribution was found for the following determinations: Density, Purity and Strength. In this case the statistical evaluation should be used with due care.

<u>Density:</u> This determination was not problematic. One statistical outlier was

observed and the calculated reproducibility is in good agreement with

the requirements of ASTM D4052:11.

Nonvolatile matter: Almost all participants reported a "less then" result. Therefore no

significant conclusions were drawn.

<u>Water:</u> 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

D1364:07.

Permanganate Test: Regretfully, no precision data are given in ASTM D1363:06 for

Ethanol. Therefore, no conclusions were drawn. All participants

reported a time larger then 20 minutes.

Purity on dry basis: Regretfully, no standard test method with precision data exists.

Therefore no conclusions were drawn. No statistical outliers were observed. The calculated reproducibility is nearly equal to the

calculated reproducibility of the previous proficiency test (iis10C12b) of November 2010 (0.0111 vs 0.0124).

Strength (%V/V):

This determination is not problematic. One statistical outlier was observed. The calculated reproducibility after rejection of the statistical outliers is in good agreement with the reproducibility derived from the OIML table and ASTM D4052:11.

Strength(%M/M):

This determination may be not problematic. Regretfully, no standard test method with precision data exists.

The calculated reproducibility is small in comparison with the calculated reproducibility in the previous proficiency test (iis10C12b) of November 2010 (0.044 vs 0.095).

UV absorbance:

Regretfully, no standard test method with precision data exists.

Therefore no significant conclusions were drawn.

In total 5 statistical outliers were observed. The calculated reproducibilities are all large in comparison with the calculated reproducibilities in a previous proficiency test (iis10C12b) of November 2010.

From the analytical details, it is clear that all participants measured the UV absorbance against water and all used a 10 mm cuvette, except laboratories 312 and 1242. These participants used respectively a 5 mm and a 50 mm cuvette.

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.

| | unit | n | average | 2.8 *sd _R | R (lit) |
|------------------------|----------|----|---------|----------------------|----------|
| Density @ 20°C | kg/L | 22 | 0.80591 | 0.00014 | 0.00050 |
| Nonvolatile matter | mg/100mL | 4 | 0.131 | n.a | n.a |
| Water | %M/M | 11 | 5.594 | 0.142 | 0.168 |
| Permanganate Time Test | min. | 9 | 33.3 | 13.9 | (8.4) |
| Purity on dry basis | %M/M | 10 | 99.994 | 0.011 | (0.012) |
| Strength | %V/V | 22 | 96.373 | 0.038 | 0.100 |
| Strength | %M/M | 12 | 94.383 | 0.044 | (0.095) |
| UV-absorbance 300 nm | | 6 | 0.0010 | 0.0050 | (0.0016) |
| UV-absorbance 270 nm | | 7 | 0.0028 | 0.0079 | (0.0065) |
| UV-absorbance 240 nm | | 12 | 0.0380 | 0.0177 | (0.0093) |
| UV-absorbance 230 nm | | 14 | 0.0931 | 0.0354 | (0.0284) |
| UV-absorbance 220 nm | | 13 | 0.1912 | 0.0592 | (0.0344) |

Table 3: Reproducibilities of sample #11122

Results between brackets are compared with the spread of the previous PT or estimated from a target reproducibility

4.3 COMPARISON OF THE PROFICIENCY TEST OF NOVEMBER 2011 WITH PREVIOUS PT'S

| | November 2011 | November 2010 | December 2009 | December 2008 |
|--------------------------------|---------------|---------------|---------------|---------------|
| Number of reporting labs | 23 | 28 | 31 | 22 |
| Number of results reported | 151 | 189 | 299 | 153 |
| Number of statistical outliers | 9 | 13 | 34 | 8 |
| Percentage outliers | 6.0% | 6.9% | 11.4% | 5.2% |

table 4: comparison with previous proficiency tests.

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

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

| | November 2011 | November 2010 | December 2009 | December 2008 |
|------------------------|---------------|---------------|---------------|---------------|
| Density @ 20°C | ++ | ++ | ++ | ++ |
| Nonvolatile matter | n.e | ++ | n.e. | n.e. |
| Water | +/- | ++ | | |
| Permanganate Time Test | () | () | | |
| Purity on dry basis | (+) | () | (++) | (+/-) |
| Strength %V/V | ++ | ++ | - | (++) |
| Strength %M/M | ++ | () | (++) | () |
| 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 #11122 against the standard

results between brackets are compared with the spread 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

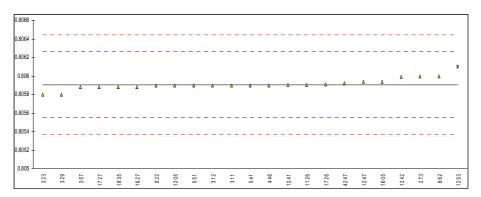
-- : group performed much worse than the standard

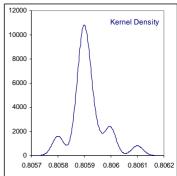
n.e.: not evaluated

APPENDIX 1

Determination of Density @ 20°C on sample #11122; results in kg/L

| lab | method | value | mark | z(targ) | remarks |
|------|-------------|----------|---------|---------|---------|
| 273 | D4052 | 0.8060 | | 0.53 | |
| 311 | D4052 | 0.8059 | | -0.03 | |
| 312 | D4052 | 0.8059 | | -0.03 | |
| 323 | D4052 | 0.8058 | | -0.59 | |
| 329 | D4052 | 0.8058 | | -0.59 | |
| 357 | D4052 | 0.80588 | | -0.14 | |
| 446 | D4052 | 0.8059 | | -0.03 | |
| 522 | | | | | |
| 541 | D4052 | 0.8059 | | -0.03 | |
| 551 | D4052 | 0.8059 | | -0.03 | |
| 556 | | | | | |
| 559 | | | | | |
| 862 | D4052 | 0.80600 | | 0.53 | |
| 922 | D4052 | 0.8059 | | -0.03 | |
| 1126 | D4052 | 0.805907 | | 0.01 | |
| 1205 | in house | 0.80590 | | -0.03 | |
| 1241 | D4052 | 0.805903 | | -0.02 | |
| 1242 | D4052 | 0.805990 | | 0.47 | |
| 1247 | INH-4500 | 0.80594 | | 0.19 | |
| 1253 | D4052 | 0.80610 | G(0.05) | 1.09 | |
| 1425 | | | , , | | |
| 1605 | D4052 | 0.80594 | | 0.19 | |
| 1627 | INH-79528 | 0.80588 | | -0.14 | |
| 1726 | D4052 | 0.80591 | | 0.02 | |
| 1727 | D4052 | 0.80588 | | -0.14 | |
| 1835 | D4052 | 0.80588 | | -0.14 | |
| 4247 | INH-4500 | 0.80592 | | 0.08 | |
| | | 0.00002 | | 0.00 | |
| | normality | not OK | | | |
| | n | 22 | | | |
| | outliers | 1 | | | |
| | mean (n) | 0.80591 | | | |
| | st.dev. (n) | 0.000501 | | | |
| | R(calc.) | 0.00014 | | | |
| | R(D4052:11) | 0.00050 | | | |
| | (0-1002.11) | 0.00000 | | | |



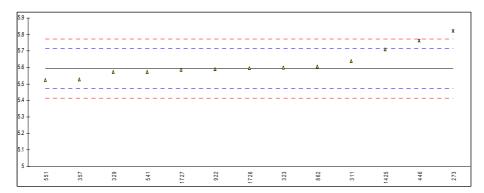


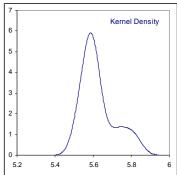
Determination of Nonvolatile matter on sample #11122; results in mg/100mL

| lab | method | value | mark z(ta | arg) | remarks |
|--------------|--------------------|--------------|-----------|------|---------|
| 273 | | | | | |
| 311 | D1353 | <1 | | | |
| 312 | | | | | |
| 323 | D1353 | <1 | | | |
| 329 | D1353 | <1 | | | |
| 357 | D1353 | <1 | • | | |
| 446 | INH-4524 | <10 | • | | |
| 522 | _ | | | | |
| 541 | D1353 | <0.1 | • | | |
| 551 | D1353 | 0.0 | | | |
| 556 | | | | | |
| 559 | | | | | |
| 862 | D1353 | <0.1 | | | |
| 922 | D1353 | 0.19 | • | | |
| 1126 | | | | | |
| 1205 | | | | | |
| 1241 | | | | | |
| 1242 | | | | | |
| 1247 | | | | | |
| 1253 | | | | | |
| 1425 | | | | | |
| 1605 | INIL 70500 | 0.025 | | | |
| 1627 1726 | INH-79529 D1353 | 0.035 <10 | | | |
| 1726 | D1353 | 0.3 | | | |
| 1835 | EN15691 | <10 | | | |
| 4247 | LINIOUSI | < 10 | | | |
| 4247 | | | | | |
| | normality | unknown | | | |
| | n | 4 | | | |
| | outliers | n.a | | | |
| | mean (n) | 0.131 | | | |
| | st.dev. (n) | n.a | | | |
| | R(calc.) | n.a | | | |
| | R(D1353:09) | n.a | | | |
| | (2 .000.00) | | | | |

Determination of Water (Titrimetric) on sample #11122; results in %M/M

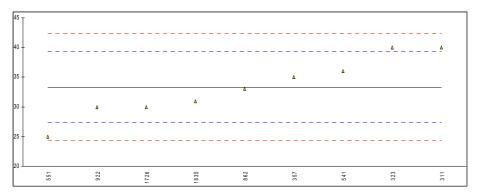
| lab | method | value | mark | z(targ) | remarks |
|------|-------------|---------|------------|---------|----------------------|
| 273 | E203 | 5.821 | C,DG(0.05) | 3.79 | first reported: 6.21 |
| 311 | D1364 | 5.64 | | 0.77 | |
| 312 | | | | | |
| 323 | D1364 | 5.600 | | 0.10 | |
| 329 | E203 | 5.573 | | -0.35 | |
| 357 | E203 | 5.529 | | -1.08 | |
| 446 | E203 | 5.7632 | DG(0.05) | 2.83 | |
| 522 | | | | | |
| 541 | E1064 | 5.575 | | -0.31 | |
| 551 | D1364 | 5.525 | | -1.15 | |
| 556 | | | | | |
| 559 | | | | | |
| 862 | D1364 | 5.606 | | 0.21 | |
| 922 | E203 | 5.59 | | -0.06 | |
| 1126 | | | | | |
| 1205 | | | | | |
| 1241 | | | | | |
| 1242 | | | | | |
| 1247 | | | | | |
| 1253 | | | | | |
| 1425 | in house | 5.71 | | 1.94 | |
| 1605 | | | | | |
| 1627 | | | | | |
| 1726 | D1364 | 5.5965 | | 0.05 | |
| 1727 | D1364 | 5.5863 | | -0.12 | |
| 1835 | | | | | |
| 4247 | | | | | |
| | 19. | 014 | | | |
| | normality | OK | | | |
| | n | 11 | | | |
| | outliers | 2 | | | |
| | mean (n) | 5.5937 | | | |
| | st.dev. (n) | 0.05062 | | | |
| | R(calc.) | 0.1417 | | | |
| | R(D1364:07) | 0.1678 | | | |

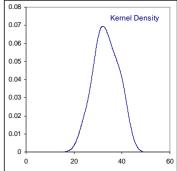




Determination of Permanganate Time Test @ 15 °C on sample #11122; results in minutes

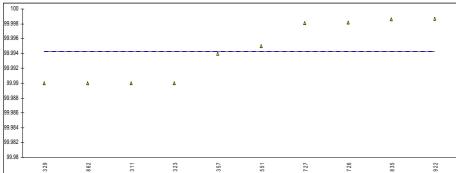
| lab | method | value | mark | z(targ) | remarks |
|--|-------------|--|------|---------|---------|
| 273 | | | | | |
| 311 | D1363 | 40 | | | |
| 312 | | | | | |
| 323 | D1363Mod. | 40 | | | |
| 329 | D1363 | >20 | | | |
| 357 | D1363 | 35 | | | |
| 446 | | | | | |
| 522 | | | | | |
| 541 | D1363 | 36 | | | |
| 551 | D1363 | 25 | | | |
| | | | | | |
| | | | | | |
| 862 | | | | | |
| | D1363 | 30 | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | D1363 | 30 | | | |
| | _ | | | | |
| 1835 | D1363 | 31 | | | |
| 4247 | | | | | |
| | | | | | |
| | | OK | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | st.dev. (n) | 4.95 | | | |
| | | | | | |
| | R(D1363:06) | | | | |
| 556 559 862 922 1126 1205 1241 1242 1247 1253 1425 1605 1627 1726 1727 1835 4247 | R(calc.) | 33 30 30 31 OK 9 0 33.3 | | | |

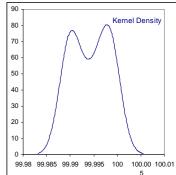




Determination of Purity on dry basis on sample #11122; results in %M/M

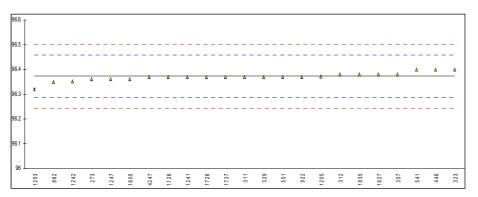
| lab | method | value | mark | z(targ) | remarks | |
|--------------|-------------|---------|------|---------|-----------------------------|----------------|
| 273 | | | | | | |
| 311 | | 99.99 | | | | |
| 312 | | | | | | |
| 323 | INH-001 | 99.99 | | | | |
| 329 | | 99.99 | | | | |
| 357 | EN15721 | 99.994 | | | | |
| 446 | INH-CM | >99.99 | | | | |
| 522 | | | | | | |
| 541 | | | | | | |
| 551 | INH-1313 | 99.995 | | | | |
| 556 | | | | | | |
| 559 | | | | | | |
| 862 | INH-0001 | 99.99 | | | | |
| 922 | INH-0001 | 99.9987 | | | | |
| 1126 | | | | | | |
| 1205 | | | | | | |
| 1241 | | | | | | |
| 1242 | | | | | | |
| 1247 | | | | | | |
| 1253 | | | | | | |
| 1425 | | | | | | |
| 1605 | | | | | | |
| 1627 | in harra | | | | | |
| 1726 | in house | 99.9982 | | | | |
| 1727 | in house | 99.9981 | | | | |
| 1835 4247 | in house | 99.9986 | | | | |
| 4247 | | | | | | |
| | normality | not OK | | | | |
| | n | 10 | | | | |
| | outliers | 0 | | | | |
| | mean (n) | 99.994 | | | | |
| | st.dev. (n) | 0.0040 | | | | |
| | R(calc.) | 0.011 | | | | |
| | R(Lit) | unknown | | | Compare R(iis10C12b)=0.0124 | |
| | () | | | | , , , , , , | |
| 100 T | | | | | | 90 |
| 99.998 | | | | | Δ Δ Δ | Kernel Density |
| 99.996 | | | | | | 80 - |
| | | | | Δ | | 70 - |
| 99.994 | | | Δ | | | 60 - |
| 99.992 | | | | | | 50 - |
| 99.99 | Δ | Δ Δ | | | | |
| 99.988 | | | | | | 40 - |

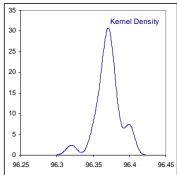




Determination of Strength on sample #11122; results in %V/V

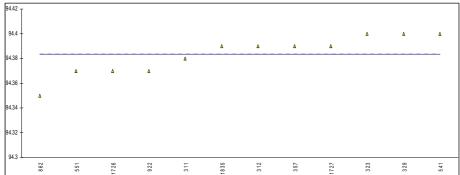
| lab | method | value | mark | z(targ) | remarks |
|------|--------------|--------|---------|---------|----------------------------|
| 273 | OIML | 96.36 | | -0.36 | |
| 311 | OIML | 96.37 | | -0.08 | |
| 312 | OIML | 96.38 | | 0.20 | |
| 323 | D4052 | 96.4 | | 0.76 | |
| 329 | OIML | 96.37 | | -0.08 | |
| 357 | OIML | 96.38 | | 0.20 | |
| 446 | OIML | 96.4 | | 0.76 | |
| 522 | | | | | |
| 541 | Dens.Det. | 96.4 | | 0.76 | |
| 551 | D4052 | 96.37 | | -0.08 | |
| 556 | | | | | |
| 559 | | | | | |
| 862 | Table | 96.35 | | -0.64 | |
| 922 | OIML | 96.37 | | -0.08 | |
| 1126 | | 96.37 | | -0.08 | |
| 1205 | in house | 96.372 | | -0.03 | |
| 1241 | | 96.37 | | -0.08 | |
| 1242 | | 96.352 | | -0.59 | |
| 1247 | in house | 96.36 | | -0.36 | |
| 1253 | | 96.32 | G(0.05) | -1.48 | |
| 1425 | | | | | |
| 1605 | | 96.36 | | -0.36 | |
| 1627 | INH-79528 | 96.38 | | 0.20 | |
| 1726 | OIML | 96.37 | | -0.08 | |
| 1727 | | 96.37 | | -0.08 | |
| 1835 | OIML | 96.38 | | 0.20 | |
| 4247 | in house | 96.37 | | -0.08 | |
| | normality | not OK | | | |
| | n | 22 | | | |
| | outliers | 1 | | | |
| | mean (n) | 96.373 | | | |
| | st.dev. (n) | 0.0137 | | | |
| | R(calc.) | 0.038 | | | |
| | R(see \$4.1) | 0.100 | | | Compare R(iis10C12b)=0.095 |
| | (+) | | | | |

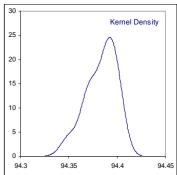




Determination of Strength on sample #11122; results in %M/M

| lab | method | value | mark | z(targ) | remarks | |
|---------|-------------|---------|------|---------|----------------------------|----|
| 273 | | | | | | |
| 311 | OIML | 94.38 | | | | |
| 312 | OIML | 94.39 | | | | |
| 323 | D4052 | 94.4 | | | | |
| 329 | | 94.40 | | | | |
| 357 | OIML | 94.39 | | | | |
| 446 | | | | | | |
| 522 | | | | | | |
| 541 | Dens.Det. | 94.4 | | | | |
| 551 | D4052 | 94.37 | | | | |
| 556 | | | | | | |
| 559 | | | | | | |
| 862 | Table | 94.35 | | | | |
| 922 | OIML | 94.37 | | | | |
| 1126 | | | | | | |
| 1205 | | | | | | |
| 1241 | | | | | | |
| 1242 | | | | | | |
| 1247 | | | | | | |
| 1253 | | | | | | |
| 1425 | | | | | | |
| 1605 | | | | | | |
| 1627 | | | | | | |
| 1726 | OIML | 94.37 | | | | |
| 1727 | | 94.39 | | | | |
| 1835 | OIML | 94.39 | | | | |
| 4247 | | | | | | |
| | normality | not OK | | | | |
| | n | 12 | | | | |
| | outliers | 0 | | | | |
| | mean (n) | 94.383 | | | | |
| | st.dev. (n) | 0.0156 | | | | |
| | R(calc.) | 0.044 | | | | |
| | R(Lit) | unknown | | | Compare R(iis10C12b)=0.095 | |
| | () | | | | 1 (| |
| | | | | | | |
| 94.42 T | | | | | | 30 |





Determination of UV absorbance on sample #11122;

| lab | method | 300nm | mark | 270nm | mark | 240nm | mark | 230nm | mark | 220nm | mark |
|------|--------------|----------|---------|----------|---------|----------|----------|----------|------|---------|-----------|
| 273 | IMPCA004 | <0.01 | | <0.01 | | 0.037 | | 0.086 | | 0.169 | |
| 311 | | < 0.005 | | < 0.005 | | 0.047 | | 0.107 | | 0.214 | |
| 312 | INH-094 | < 0.005 | | < 0.005 | С | 0.042 | С | 0.099 | С | 0.201 | С |
| 323 | INH-CM | < 0.001 | | < 0.001 | | 0.030 | | 0.083 | | 0.176 | |
| 329 | | < 0.001 | | 0.002 | | 0.040 | | 0.093 | | 0.192 | |
| 357 | | | | | | | | | | | |
| 446 | INH-CM | < 0.01 | | < 0.01 | | 0.039 | | 0.099 | | 0.211 | |
| 522 | | | | | | | | | | | |
| 541 | | | | | | | | | | | |
| 551 | INH-1519 | 0.0022 | | 0.0066 | | 0.04235 | | 0.09185 | | 0.1792 | |
| 556 | | | | | | | | | | | |
| 559 | | | | | | | | | | | |
| 862 | IMPCA004 | 0.004 | | 0.006 | | 0.045 | | 0.100 | | 0.193 | |
| 922 | | 0.0000 | | 0.0000 | | 0.0352 | | 0.0867 | | 0.1788 | |
| 1126 | | | | | | | | | | | |
| 1205 | | | | | | | | | | | |
| 1241 | | | | | | | | | | | |
| 1242 | | -0.0008 | | -0.0009 | | 0.027 | | 0.070 | | 0.099 | C,G(0.05) |
| 1247 | | | | | | | | | | | |
| 1253 | | | | | | | | | | | |
| 1425 | | | | | | | | | | | |
| 1605 | | < 0.05 | | < 0.05 | | 0.030 | | 0.079 | | 0.181 | |
| 1627 | | | _ | | _ | | | | _ | | |
| 1726 | | 0.000315 | C | 0.002457 | C | 0.065565 | DG(0.05) | 0.094381 | С | 0.24082 | |
| 1727 | | 0.01356 | G(0.01) | 0.02037 | G(0.01) | 0.06644 | DG(0.05) | 0.122 | | 0.164 | |
| 1835 | | 0.000325 | | 0.003516 | | 0.041456 | | 0.092277 | | 0.18537 | |
| 4247 | | | | | | | | | | | |
| | normality | OK | | OK | | OK | | OK | | OK | |
| | n | 6 | | 7 | | 12 | | 14 | | 13 | |
| | outliers | 1 | | 1 | | 2 | | 0 | | 1 | |
| | mean (n) | 0.00101 | | 0.00281 | | 0.0380 | | 0.0931 | | 0.1912 | |
| | st.dev. (n) | 0.001767 | | 0.002813 | | 0.00632 | | 0.01264 | | 0.02116 | |
| | R(calc.) | 0.00495 | | 0.00788 | | 0.0177 | | 0.0354 | | 0.0592 | |
| | R(Lit) | unknown | | unknown | | unknown | | unknown | | unknown | |
| | R(iis10C12b) | 0.00163 | | 0.00651 | | 0.0093 | | 0.0284 | | 0.0344 | |
| | (| 2.00.00 | | | | | | | | | |

C = Corrected

first reported test results:

lab 312,270 nm:0.007

lab 312,240 nm:0.209

lab 312,230 nm:0.497

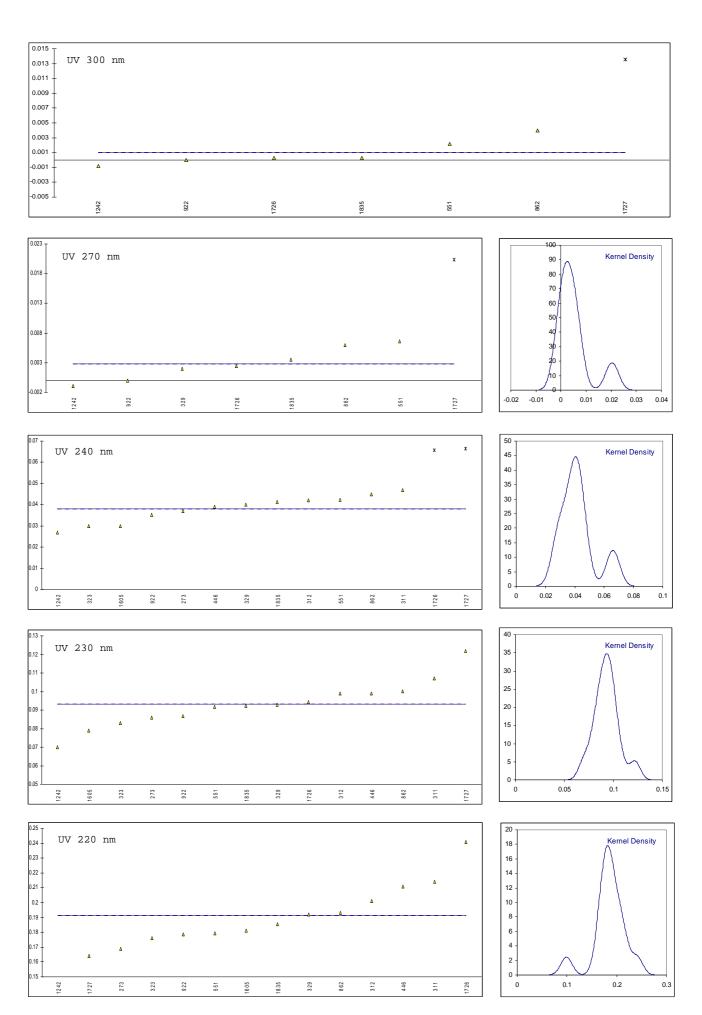
lab 312,220 nm:1.005

lab 1242, 220 nm:0.124

lab 1726, 300 nm: 0.024105

lab 1726, 270 nm:0.027316

lab 1726, 230 nm:0.13518



APPENDIX 2

Analytical details regarding Strength determination and UV absorbance.

| Analyt | icai actans | regarding Stre | ligili actori | illiation and | 1 0 4 403010 | aricc. | 1 |
|--------|--------------|-------------------------------|----------------------|--|---------------|------------------|--------------------------|
| Lab | | | Strength | | UV absorbance | | Other details |
| | Distillation | Equipment | used sample in ml | how much distillate was obtained in ml | cuvette(mm) | measured aginst: | |
| 273 | no | Density meter | | | 10 | H ₂ O | |
| 311 | no | | | | 10 | H ₂ O | |
| 312 | no | Density meter | | | 50 | H₂O | |
| 323 | | | | | 10 | H₂O | |
| 329 | no | | | | 10 | H₂O | |
| 357 | no | | | | | | |
| 446 | | | | | 10 | H₂O | |
| 541 | no | | | | | | |
| 551 | no | | | | 10 | H₂O | |
| 862 | no | Density meter & alcohol table | | | 10 | H₂O | |
| 922 | | | | | 10 | H ₂ O | |
| | no | Dichtheidsmeting* | | | | | |
| 1126 | yes | GC | 10 | 50 | | | * is used for evaluation |
| 1205 | no | | | | | | |
| 1241 | no | Density meter | | | | | |
| 1242 | no | | | | 5 | H ₂ O | |
| 1247 | no | | | | | | |
| 1605 | no | Density meter | | | 10 | H ₂ O | |
| 1726 | no | Density meter | | | 10 | H ₂ O | |
| 1727 | | | | | 10 | H ₂ O | |
| 1835 | | | | | 10 | H2O | |

APPENDIX 3

Number of participants per country

- 1 lab in ARGENTINA
- 4 labs in BELGIUM
- 3 labs in BRAZIL
- 1 lab in FINLAND
- 1 lab in HONG KONG
- 1 lab in MEXICO
- 1 lab in P.R. of CHINA
- 1 lab in PAKISTAN
- 1 lab in POLAND
- 1 lab in SOUTH AFRICA
- 3 labs in SPAIN
- 7 labs in THE NETHERLANDS
- 1 lab in UNITED KINGDOM

APPENDIX 4

Abbreviations:

C = final result after checking of first reported suspect result

 $\begin{array}{ll} D(0.01) &= \text{outlier in Dixon's outlier test} \\ D(0.05) &= \text{straggler in Dixon's outlier test} \\ G(0.01) &= \text{outlier in Grubbs' outlier test} \\ G(0.05) &= \text{straggler in Grubbs' outlier test} \\ DG(0.01) &= \text{outlier in Double Grubbs' outlier test} \end{array}$

DG(0.05) = straggler in Double Grubbs' 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

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