PE100+ ASSOCIATION

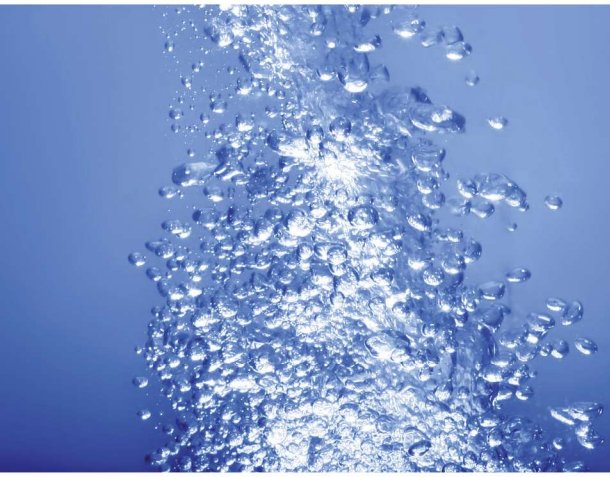
Round Robin Test 2019

LC 18841-2a
22 October 2020

Interlaboratory comparison

Accelerated Notched Pipe Test (aNPT)

ISO 13479



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Interlaboratory comparison

Accelerated Notched Pipe Test (aNPT)

ISO 13479

Colophon

Title	Interlaboratory comparison of accelerated notched pipe test
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Preface

Upon request of the Management Board of the PE100+ Association, Kiwa Nederland, as being the Administrator of the Association, has performed an interlaboratory comparison (round robin test) on the characteristic of the accelerated notched pipe test.

The resistance to slow crack growth test (SCG) is performed as an accelerated notched pipe test (aNPT) in accordance with the following International standard as base for the testing works:

ISO 13479	Polyolefin pipes for the conveyance of fluids - Determination of resistance to crack propagation - Test method for slow crack growth on notched pipes (ISO 13479: 2009)

Contents

1 Set up of the round robin test

The participants of the round robin test on the resistance internal pressure were selected by the Administrator of the PE100+ Association in consultation with the PE100+ Management Board.

The following groups were invited for participation:

- All members of the PE100+ Association with their own (internal) laboratory;
- All PE100+ listed laboratories on the PE100+ website;
- Other invited laboratories.

Samples were sent for testing to 9 laboratories:

- 3 labs did not deliver results as they had not performed the test.

To all laboratories 2 sets of 3 samples of 110mm/SDR11 PE pipes were sent. Each pipe sample had a total length of around 1 meter. Samples were made from 2 (assigned) different PE 100-RC materials. For information: one with C4 and one with C6 technology production. All samples were pre-notched by the same laboratory. As the round robin was not intended for differentiation in tested materials this aspect has not been further investigated in this report.

The labs were asked to perform the test in accordance with (EN) ISO 13479 with the modification that the testing environment was a 2% Water-in-nonylphenol (Arkopal N100) solution instead of water. Guidance could be obtained from the draft version of Annex A to ISO 13479 (which describes the aNPT test).

The request was to test 3 pipe samples.

The used test parameters were as follows:

Material	**Pipe Material A**	**Pipe Material B**
Test method	ISO 13479	ISO 13479
Test temperature	80°C	80°C
Test environment	2% Water-in-nonylphenol (Arkopal N100) in water	2% Water-in-nonylphenol (Arkopal N100) in water
Test pressure	9,2 bar	9,2 bar

The laboratories are made anonymously in this report by a random unique lab code number.

2 Test results

The test results as reported by the participants are given below. Each single lab has been given a single and unique Lab number for this round robin test. The request was to test 3 pipe samples.

Table 1: Reported test results for Material A

Laboratory	M 1	M 2	M 3
Lab 47	720,0	512,0	726,0
Lab 48	942,4	895,3	986,5
Lab 49	1247,0	709,2	1065,5
Lab 50	667,1	615,4	694,4
Lab 51	780,2	715,9	737,8
Lab 52	583,8	816,7	1027,8

Table 2: Reported test results for Material B

Laboratory	M 1	M 2	M 3
Lab 47	1562,0	1385,0	1483,0
Lab 48	3195,5*	1961,4	2060,6
Lab 49	655,5	1603,2	1175,1
Lab 50	1271,6	1389,0	1301,8
Lab 51	1117,0	1464,0	1008,0
Lab 52	1592,0	1473,5	1810,6

Remarks to these tables:

- Results are the (failure) testing times in hours (h)
- M1, M2, etc. are the repeated measurements on the 3 sent pipe samples;
- The designation “>” before a value designates that this is either an interrupted or aborted test at the given test time;
- If no value is given, no results were reported.
- * Grubb’s outlier (maximum value)

3 Evaluation of results

3.1 General assesment

The obtained test results have been evaluated in accordance with the International standard:

ISO 13528:2005	Statistical methods for use in proficiency testing by interlaboratory comparisons (ISO 13528: 2005 + C1: 2016)

The statistical evaluation has been performed by the certified software package ProLab which is in compliance with above standard.

The used algorithms are the A + S algorithms and the assessment is based on the evaluation of an absolute Z-score being smaller than 2.

On the complete data set for material A and B the Grubb's Outlier test has been performed.
For the material A no outliers were determined.
For the material B an outlier on the maximum value was determined. This single value (the highest result) is reported as an outlier.

3.2 Terms and definitions

The term and definitions as used in this report can be found in the International standards ISO 13528 and ISO 5725-1.

Additional explanations for the obtained evaluation results are the following:

3.2.1 Grubb's outlier test

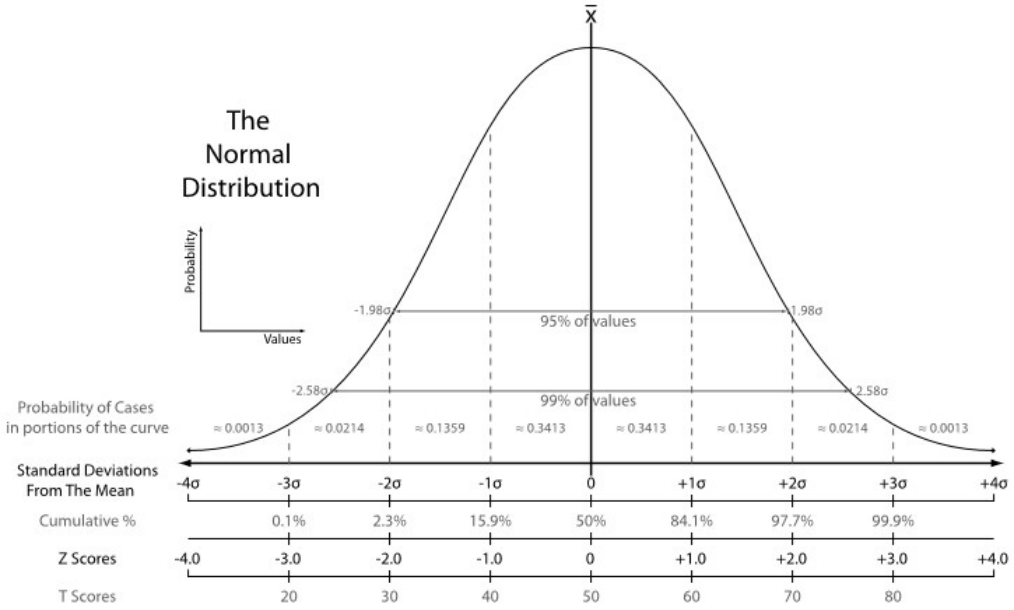
A statistical test used to detect outliers in a univariate data set assumed to come from a normally distributed population.

3.2.2 Z-Score

Standardised measure of laboratory performance on the bias, calculated using the assigned value and the standard deviation for proficiency assessment.

General classification of Z-scores are applied to this evaluation, where the absolute Z-score is taken:

- $|Z| \leq 1$: satisfactory
- $1 < |Z| \leq 2$: reasonable
- $2 < |Z| \leq 3$: questionable
- $|Z| > 3$: unsatisfactory



3.2.3 Mandel's h and k statistics

Mandel's h statistics and Mandel's k statistics present measures for graphically surveying the consistency of the data. They are helpful for laboratory assessment as well as for describing the variability of a (harmonised) measurement method.

Mandel's h statistics is used to determine if there are differences between the mean values of the laboratories, while Mandel's k statistics is used to evaluate the variance of each laboratory compared to the variances of the other laboratories.

The examination of the plots of Mandel's h and k statistics may indicate that specific laboratories exhibit patterns of results that are markedly different from the others. This is indicated by consistently high or low variation (compared to the other laboratories) and/or extreme (whether high or low) mean values.

3.2.4 Youden plot

The Youden plot window is a graphic of the results of two sample-measurand combinations. Such a presentation allows identifying systematic effects in the laboratory-specific deviations.

3.2.5 Repeatability

Precision under conditions where independent test results are obtained with the same method on identical test items in the same laboratory by the same operator using the same equipment within short intervals of time

3.2.6 Reproducibility

Precision under conditions where test results are obtained with the same method on identical test items in different laboratories with different operators using different equipment.

4 Material A

4.1 Basis statistic results material A

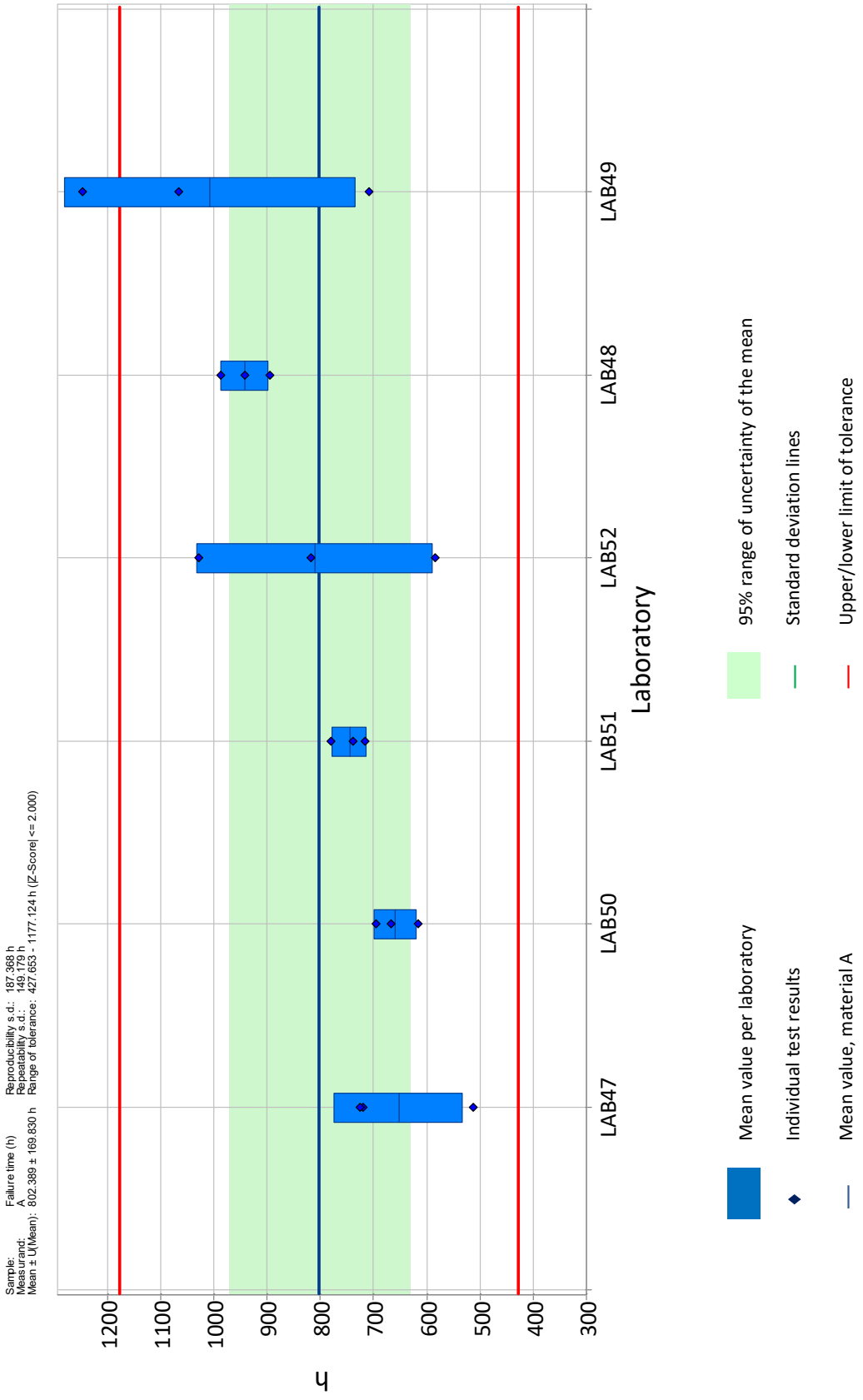
Table 3: Basis statistic results for Material A

Laboratory	Mean	Standard deviation	Z- Score
Lab 47	652,7	121,9	-0,799
Lab 48	941,4	45,6	0,742
Lab 49	1007,2	273,6	1,093
Lab 50	659,0	40,1	-0,765
Lab 51	744,6	32,7	-0,308
Lab 52	809,4	222,1	0,038

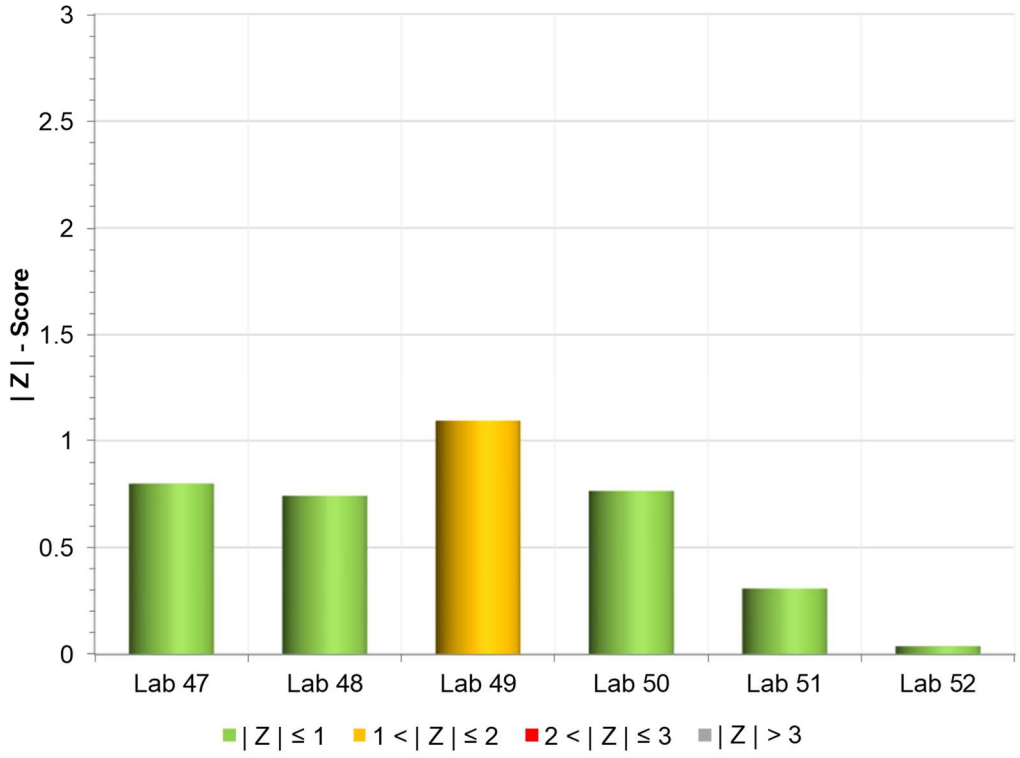
Remarks to this table:

- All Labs: the values are based on the obtained failure times (3).

4.2 Mean + standard deviation graph material A



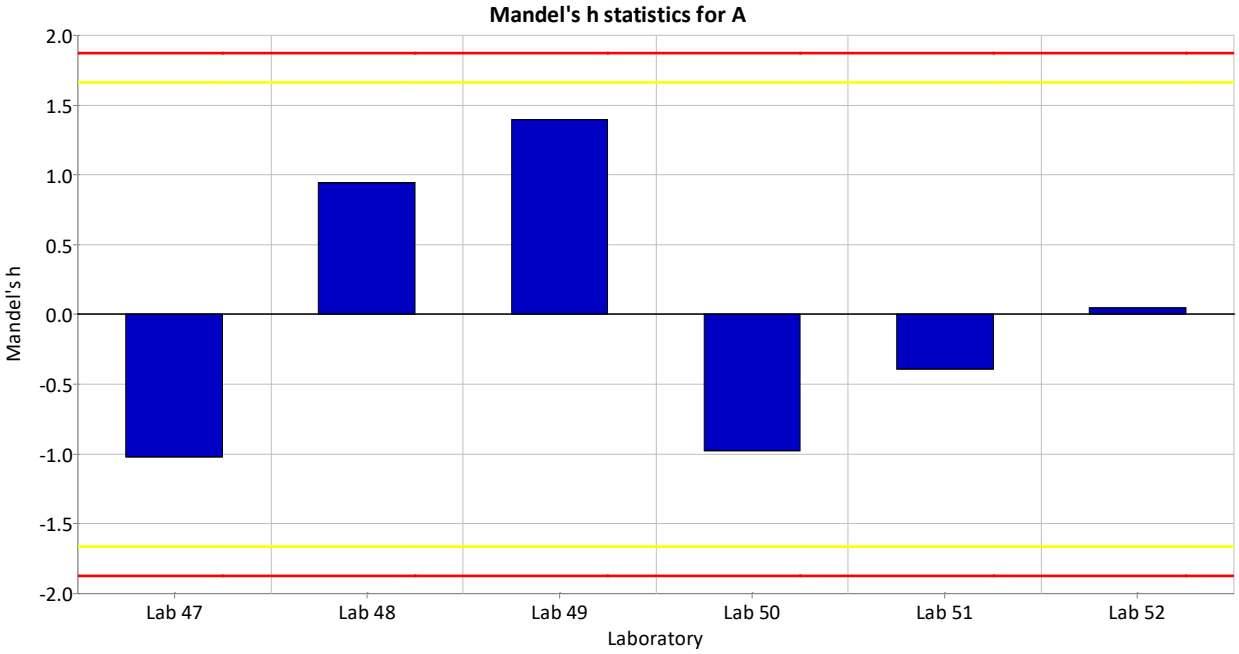
4.3 Z- Scores graph material A

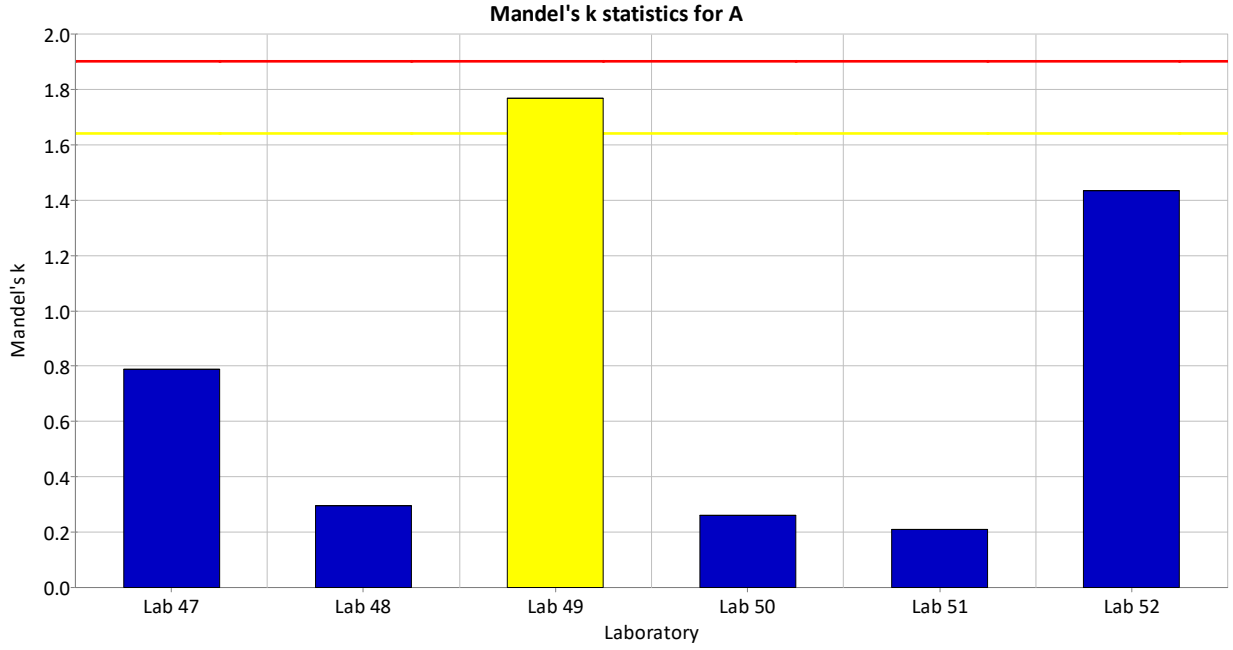


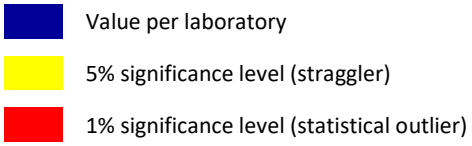
4.4 Mandel's h + k statistics material A

Table 3: Mandel's statistics for Material A

Laboratory	h statistics	Remarks	k statistics	Remarks
Lab 47	-1,020	-	0,787	-
Lab 48	0,947	-	0,295	-
Lab 49	1,396	-	1,767	Straggler
Lab 50	-0,977	-	0,259	-
Lab 51	-0,394	-	0,211	-
Lab 52	0,048	-	1,435	-







4.5 Detailed statistic calculation results material A

Table 4: Detailed statistic calculation results for Material A

Description	Result
Unit	h
Assessment	|Z|<=2,000
No. of laboratories that submitted results	6
No. of participants (according to design)	6
No. of laboratories with quantitative values	6
Minimal value	512
Maximal value	1247
Range	735
Median of number of measurement repetitions	3
95% range of uncertainty of the mean	±169,830
95% range of uncertainty of the mean, relative	21,17%
Median	777,25
Assigned value	802,389
Mean	802,389
SDPA	187,368
Reproducibility s.d.	187,368
Repeatability s.d.	149,179
Reprod. s.d. / Repeatability s.d. ratio	1,256
Rel. SDPA	23,35%
Rel. reproducibility s.d.	23,35%
Rel. repeatability s.d.	18,59%
Shapiro-Wilk test	0,436
Standard uncertainty of assigned value / SDPA	0,453
Lower limit of tolerance	427,653
Upper limit of tolerance	1177,124
Standard error	84,915
Rel. standard error	10,58%
Lower confidence limit	632,559
Upper confidence limit	972,219
No. of measurement values and states	18
No. of measurement values	18

5 Material B

5.1 Basis statistic results material B

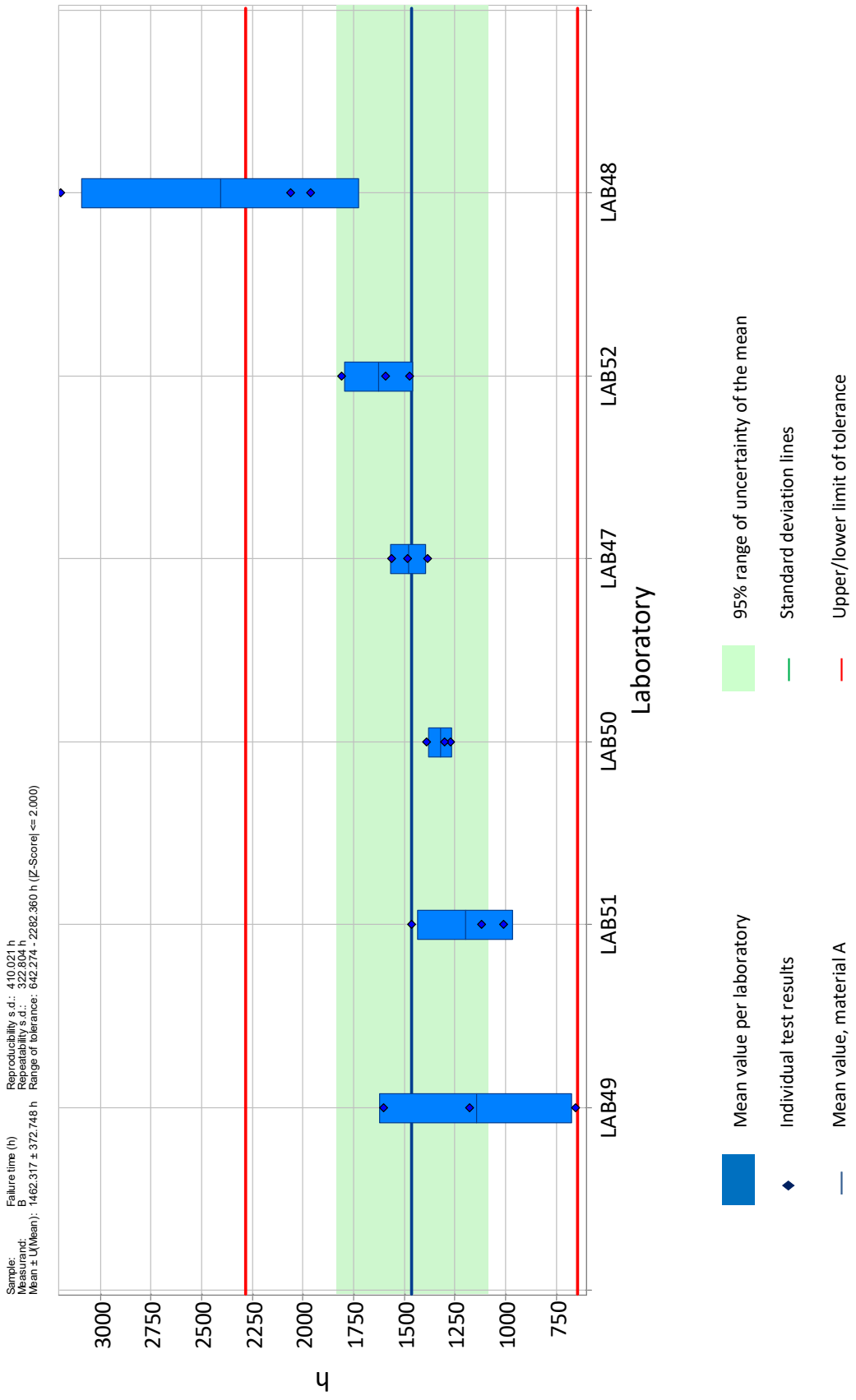
Table 4: Basis statistic results for Material B

Laboratory	Mean	Standard deviation	Z- Score
Lab 47	1476,7	88,7	0,035
Lab 48	2405,8	685,7	2,301
Lab 49	1144,6	474,6	-0,775
Lab 50	1320,8	61,0	-0,345
Lab 51	1196,3	238,1	-0,649
Lab 52	1625,4	171,0	0,398

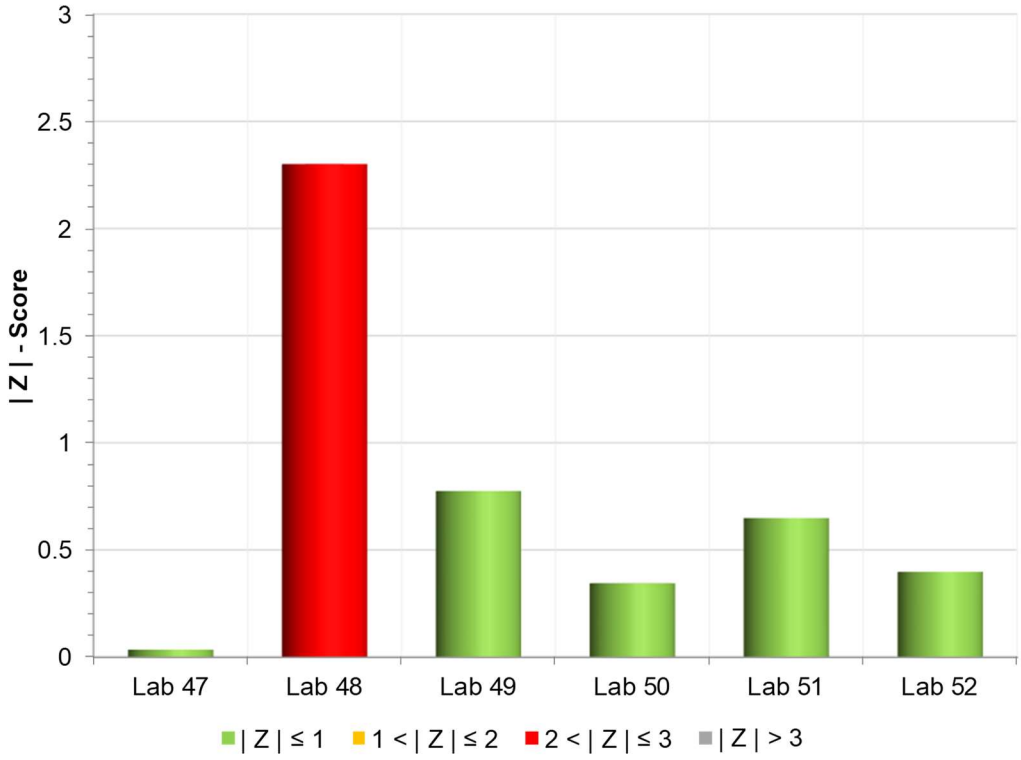
Remarks to this table:

- All Labs: the values are based on the obtained failure times (3).

5.2 Mean + standard deviation graph material B



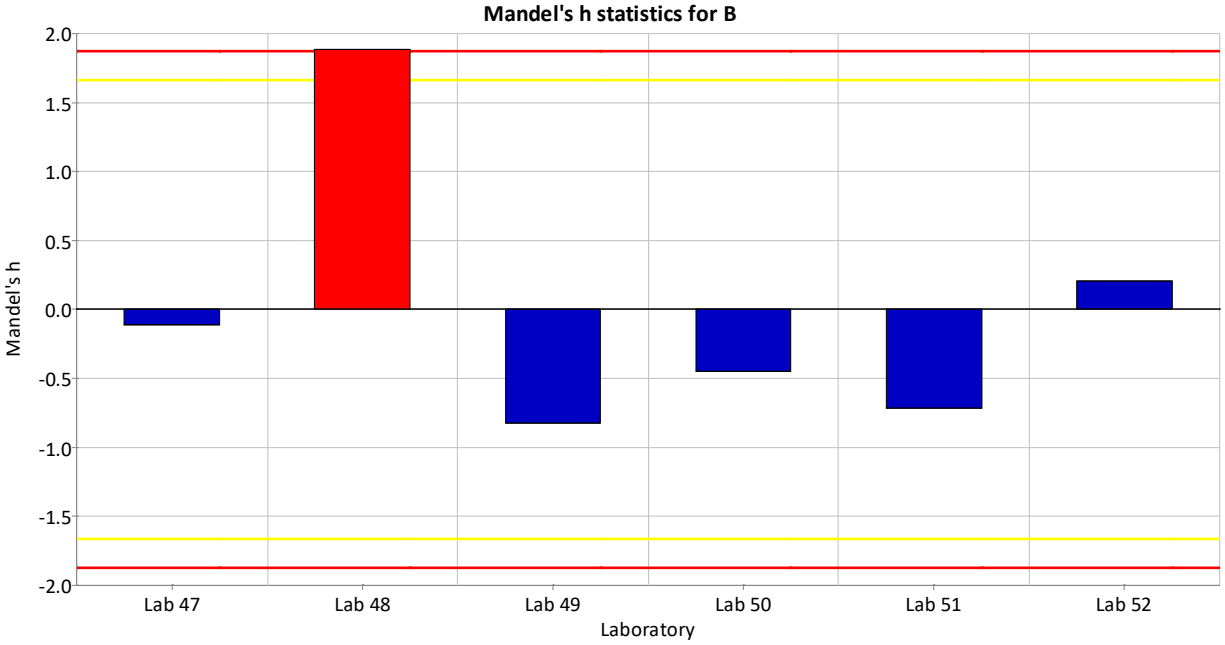
5.3 Z- Scores graph material B

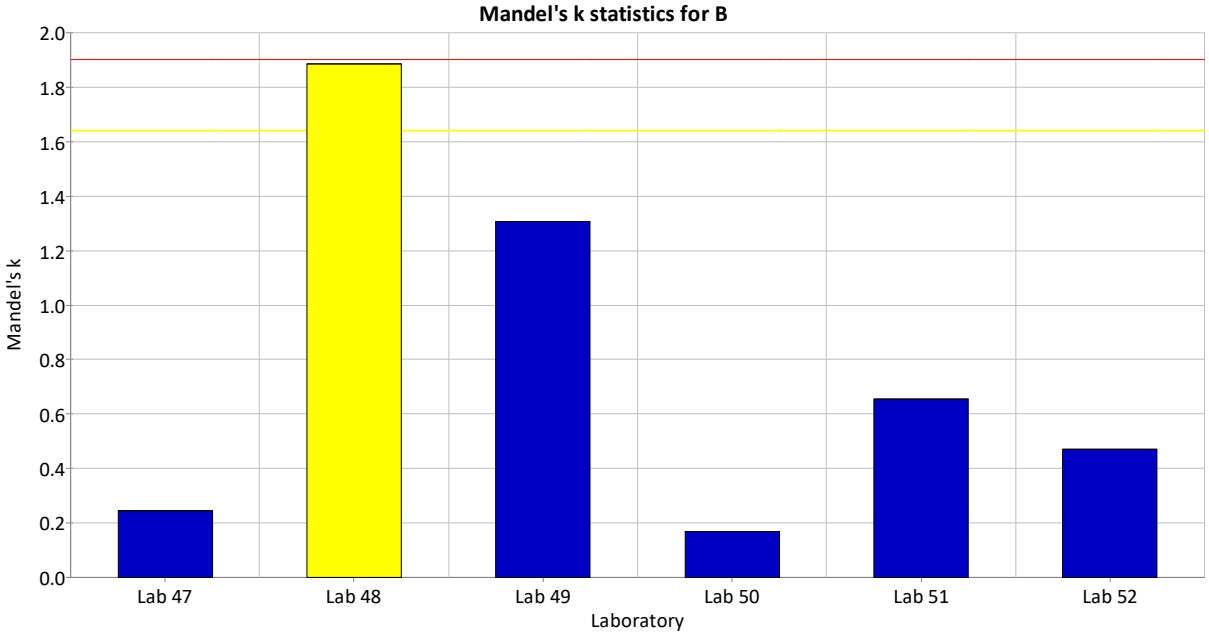


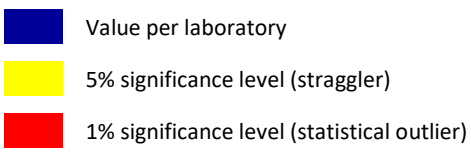
5.4 Mandel's h + k statistics

Table 3: Mandel's statistics for Material A

Laboratory	h statistics	Remarks	k statistics	Remarks
Lab 47	-0,111	-	0,244	-
Lab 48	1,886	Outlier	1,886	Straggler
Lab 49	-0,825	-	1,306	-
Lab 50	-0,446	-	0,168	-
Lab 51	-0,713	-	0,655	-
Lab 52	0,209	-	0,470	-







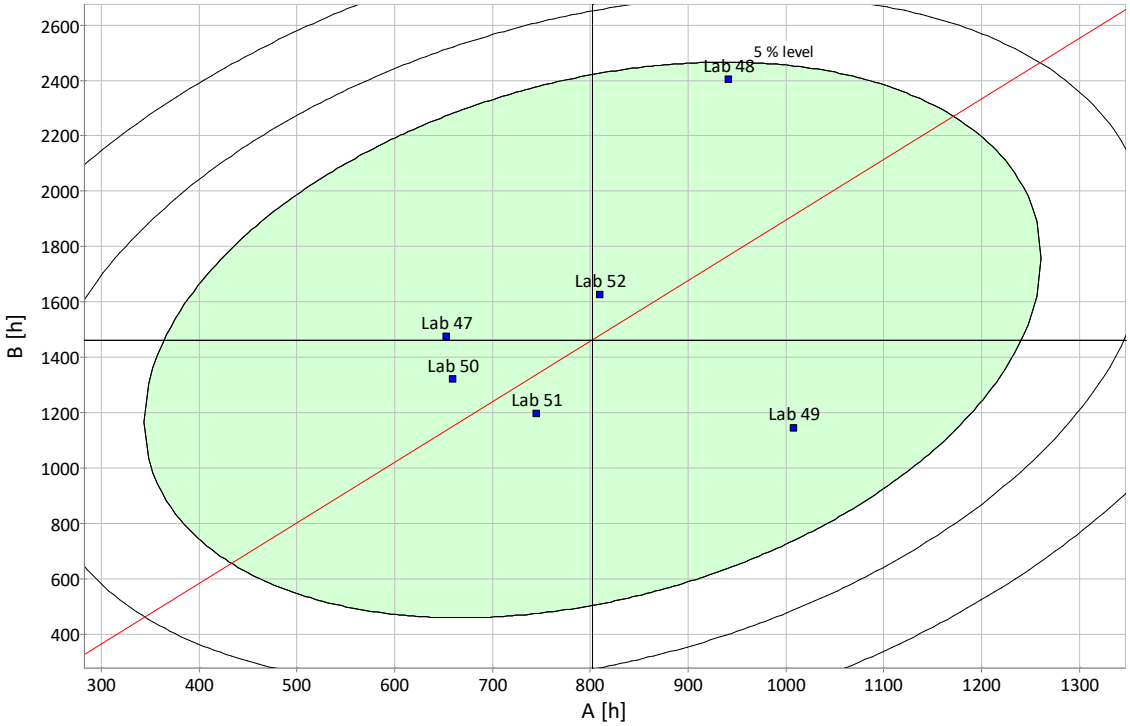
5.5 Detailed statistic calculation results material B

Table 4: Detailed statistic calculation results for Material B

Description	Result
Unit	h
Assessment	|Z|<=2,000
No. of laboratories that submitted results	6
No. of participants (according to design)	6
No. of laboratories with quantitative values	6
Minimal value	655,5
Maximal value	3195,5
Range	2540
Median of number of measurement repetitions	3
95% range of uncertainty of the mean	±372,748
95% range of uncertainty of the mean, relative	25,49%
Median	1392,4
Assigned value	1462,317
Mean	1462,317
SDPA	410,021
Reproducibility s.d.	410,021
Repeatability s.d.	322,804
Reprod. s.d. / Repeatability s.d. ratio	1,27
Rel. SDPA	28,04%
Rel. reproducibility s.d.	28,04%
Rel. repeatability s.d.	22,07%
Shapiro-Wilk test	0,098
Standard uncertainty of assigned value / SDPA	0,455
Lower limit of tolerance	642,274
Upper limit of tolerance	2282,36
Standard error	186,374
Rel. standard error	12,75%
Lower confidence limit	1089,569
Upper confidence limit	1835,065
No. of measurement values and states	18
No. of measurement values	18

6 Combined evaluation

6.1 Youden Plot



7 Conclusions

Conclusions for improvement are based on an additional questionnaire sent out to all participants. As the number of participants is low and identification of the individual laboratories could be made based on the individual (anonymous) answers the details are not mentioned in this report and only general test details are mentioned.

7.1.1 Experience of the laboratory

Based on the provided information no (statistical) conclusions can be drawn on the years of testing experience or number of tests performed by the laboratory in relation to their results in this round robin test.

7.1.2 Detergent solution preparation

Based on the provided information it is seen that

- the solution has been prepared based on both weight percentage as on volume percentage;
- the solution has been prepared both at 23°C and at 80°C;
- the aging time at 80°C of the solution varied between 72h and 336h.

In general based on the provided information no (statistical and) significant conclusions can be drawn on the above mentioned differences between the labs.

It is concluded that not all laboratories followed the same procedure for preparing the detergent solution.
It is recommended to specify the detergent preparation process more unambiguous and clearly in the test standard. In special on

- the concentration calculations;
- preparation temperature;
- aging time.

7.1.3 Set up of the test equipment and performance

Based on the provided information it is seen that

- Water tank capacities and circulation flows show large differences between the participants;
- Connections to both a single pressure station and manifold were used for this round robin test and resulted in difference in failure times with the samples put on manifold connections to the pressure station, resulting in general lower failure times in this round robin test;
- The position of the samples did show some difference in failure times with the horizontal position to obtain in this round robin test lower failure times.

In general based on the provided information no (statistical and) significant conclusions can be drawn on the above mentioned differences between the labs.

However it has to noted that the use of single pressure stations is the preferred option to determine actual failure times of pipe samples.

7.2 Overall conclusion

Based on the obtained results in combination with the additional questionnaire and the statistical evaluation no significant conclusions can be drawn on certain specific aspects of the test standard ISO 13479 with the aNPT modification in relation to the results. It should be noted that the number of participants was relative low in this round robin test.

Not all participants precisely followed the ISO 13479 draft Annex for the aNPT standard or had the same process of the performance but their results are not classified as statistically outliers. The process and preparation works are clearly not in line amongst all laboratories and this is recommended to be improved in the test standard ISO 13479.

Compared to the previous PE100+ round robin tests performed on the ISO 13479 standard for the “regular” notched pipe test (report LC 11251), this round robin test on the aNPT shows smaller variations in the results and better values for the reproducibility and the repeatability.