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MAKING THE ACCELERATED NOTCH PIPE TEST (ANPT) FUTURE-PROOF

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SHORT SUMMARY

This paper describes a study that compared the horizontal and vertical test positions in the Accelerated Notch Pipe Test. The study also explored the use of Dehyton® PL as an accelerating detergent to replace Arkopal® N100.

KEYWORDS

polyethylene, detergents, Arkopal, Dehyton, accelerated NPT

ABSTRACT

The latest generation of polyethylene grades (PE 100-RC and PE 4710 PLUS) have an increased resistance to slow crack growth. Such cracks can be initiated by either a scratch during installation or a rock indentation during use. The Accelerated Notch Pipe Test (ANPT) was standardized in ISO 13479 in 2022 to allow the long-term behavior of pipes to be evaluated in a fast and reliable way. This test method is specified in the standard for PE piping systems for the supply of gaseous fuels in Europe (EN 1555 series) and its inclusion in the international ISO 4437 series has been proposed.

In the ANPT method, four longitudinal notches are machined into the outer surface of the pipe. The notches are evenly distributed around the circumference. The pipe is pressurized hydrostatically and submerged in a detergent solution at 80 °C to accelerate failure.

A previous study showed that the test efficiency can be optimized by creating small containers for each individual pipe to minimize the amount of detergent [1]. The pipes are placed vertically rather than horizontally to further improve the test efficiency. The orientation of the pipes is not standardized in ISO 13479. This study investigated the differences between these two orientations. The results can be used to improve the standard in future.

This study also explored the use of a new detergent. ISO 13479 currently prescribes the use of Arkopal® N100 as the detergent for the test. However, this detergent is a *nonylphenol ethoxylate that is currently restricted by the REACH regulation published by the European Chemicals Agency [2], because it is very toxic to aquatic life; it has long-lasting effects and is endocrine disrupting [3]. This means the detergent cannot be imported, distributed or sold within Europe. While there is an exception for laboratory use, no importer has been found by any of the European test labs to date. This means a new detergent for the ANPT must be found as soon as possible to be able to determine whether the PE 100-RC pipes indeed meet the minimum requirements specified in the EN 1555 series. This study included a comparison between the failure times of pipes that were tested in Arkopal® N100 and in a new detergent, Dehyton®.*

INTRODUCTION

Scratches may result on the outside of a polyethylene (PE) pipe during trenchless installation. These scratches can slowly grow through the entire pipe wall. The Notch Pipe Test specified in ISO 13479 simulates this process. However, with this test, failure times will be over one year [1] for PE 100-RC. A correlated accelerated method, the accelerated notch pipe test (ANPT), was therefore developed. This method is described in an annex to ISO 13479 first published in 2022.

The ANPT is very similar to the standard NPT, but with one important difference: the notches are exposed to an external detergent solution instead of water to reduce the time to failure. Kiwa Technology uses smaller containers for each pipe rather than a large bath to minimize the required detergent quantities. This eco-friendly version was described in a previous publication [2].

ISO 13479 describes how a hydrostatic pressure test can be carried out on the notched pipes in accordance with ISO 1167-1. Neither ISO 13479 nor ISO 1167-1 prescribe the orientation of the test pieces (horizontal or vertical). ISO 1167-1 only states that supports or hangers are required to allow the test pieces to be placed in the tank. The standard also states that the test pieces must be immersed in a water bath without specifying the orientation of the test pipe.
Because Kiwa acquired new equipment (purchased from the same manufacturer) to allow more tests to be carried out simultaneously, it was decided to switch from horizontal to vertical placement of the test pipes to save space. This paper describes the results of the comparison between these two orientations.

Furthermore, the current annex to ISO 13479 specifies that the ANPT should be performed with 2% nonylphenol ethoxylate. This is better known by its trade name, Arkopal® N100.
However, the use of nonylphenol ethoxylate is currently restricted in Europe by the REACH regulation published by the European Chemicals Agency [3] because it is very toxic to aquatic life; it has long-lasting effects and is endocrine disrupting [4]. This means it cannot be manufactured, imported, distributed or sold on the market in Europe for a wide variety of purposes. While there is an exception for laboratory use, in practice it means that there is no importer in Europe. This makes it impossible for European test labs to obtain Arkopal® N100.

The European standards for polyethylene piping systems for the supply of gaseous fuels (the EN 1555 series) contain minimum requirements for PE 100-RC pipes that are to be tested using the ANPT with Arkopal® N100. A new detergent for the ANPT must be found as soon as possible to be able to determine whether the PE 100-RC pipes indeed meet these requirements. One alternative detergent, which is also used for the Accelerated Full Notch Creep Test (AFNCT) specified in ISO 16770, is lauramine oxide. This detergent is commercially available as Dehyton® PL. This paper describes comparative tests between Arkopal® N100 and Dehyton® PL performed using the ANPT.

EXPERIMENTAL

Three PE types were used in this study:

- A low performing high-density PE grade, which is expected to have a failure time of about 90 h when tested using the ANPT with Arkopal® N100 as the detergent. This material is referred to as “PE-HD”.
- Two PE 100-RC grades: one with butene (C_4H_8) and one with hexene (C_6H_{12}) used for the co-polymerization. These grades are expected to have failure times >300 h when tested using the ANPT with Arkopal® N100 as the detergent. These materials are referred to as “PE 100-RC A” and “PE 100-RC B”.
  Please note: the study was not intended to differentiate between the tested PE 100-RC grades. Any differences in outcome were not investigated further.

The tests were performed in accordance with ISO 13479. All tested PE pipes had a nominal outside diameter of 110 mm (4”) with SDR 11. Four notches were machined at equal distances around the PE pipe circumference to such a depth that a pipe wall ligament thickness of between 0.78 and 0.82 times the minimum wall thickness remained. The length of each notch was 110 mm (4”) at full depth. The pipe was filled with demineralized water. End caps were then installed on the test pipe, with one end cap connected to pressurizing equipment.

The test pipe was placed in a small container with the detergent solution (2 wt% active component*). Special containers were used for each alignment, i.e., horizontally or vertically (figure 1 and figure 2).
The horizontal containers had a V-shaped base containing a perforated steel tube. The detergent solution entered through the perforated steel tube. One end was connected to a circulation pump in the upper part of the container. The test pipe rested on the end caps to leave enough space for proper circulation of the detergent solution around the test pipe. Special tests with colorant confirmed the homogeneous distribution of the detergent solution throughout the container [2].
The test pipe in the vertical container rested on one end cap. The other end cap contained the connection to the pressurizing equipment. A perforated ring was initially created at

* 2 wt% Arkopal® N100, which contains between 90 and 100 wt% nonylphenol polyglycol ether
or 6.67 wt% Dehyton® PL, which contains between 29.0 and 31.0 wt% lauramine oxide.

Figure 1. Placement of the test pipe in the horizontal container (still without detergent solution).

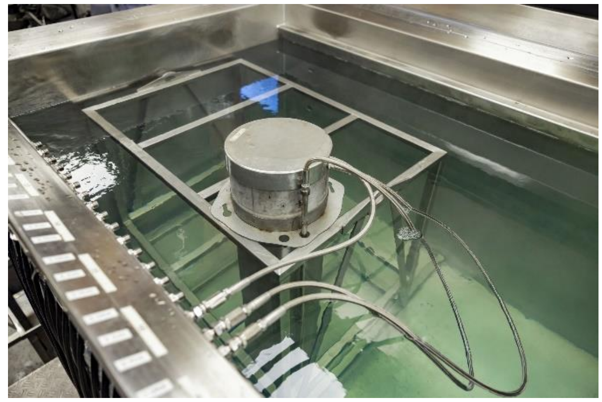

Figure 2. The vertical container, which contains the test pipe and the detergent solution, inside the water tank.

the top of the container. This was the inlet for the detergent solution. However, due to strange initial results (which eventually appeared to be the result of an incorrect calibration and not of the test setup), the setup was changed to use a vertical perforated tube at the side of the container. The outlet was located at the base of the container.

The containers were placed in a large water tank at an elevated temperature. The temperature within the smaller containers was kept at 80 °C (176 °F). The solution was circulated immediately after placing the containers in the water tank to prevent any separation of the detergent solution. The pipes were not immediately pressurized, but were first conditioned for either 6 h or 24 h. A conditioning time of 6 h is specified in the previous version of ISO 13479 (version 2009), while the current version (version 2022) specifies 24 h.
Following the conditioning time, the pressure was increased progressively and smoothly to 9.2 bar (133 psi), the starting point for the test (t = 0). A rapid drop in pressure was registered as failure, which marked the end of the test. The time to failure had to exceed 300 h for PE 100-RC pipes in the Arkopal® solution to meet the standard.

RESULTS AND DISCUSSION

Horizontal vs vertical

The results are given in table 1 and displayed graphically in figure 3. 58 test pipes made from PE-HD were tested in the vertical containers to determine the statistical uncertainty caused by the equipment. Fewer pipes were used for the other tests.

It is clear that the failure times for the samples in the horizontal containers were shorter than for those in the vertical containers. The same result was also found in the interlaboratory comparison of the ANPT as performed by the PE100+ Association. The report states that "*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*." [5].

Table 1. ANPT test failure times (average and standard deviation) in the horizontal and vertical test containers using three different materials and two detergents. The numbers of pipes tested are shown in brackets.

Material	**Detergent**	**Conditioning time** (h)	**Horizontal** Avg. ± St. Dev. (h) (number tested)	**Vertical** Avg. ± St. Dev. (h) (number tested)
PE-HD	Arkopal®	6	44.1 ± 6.4 (8)	71.5 ± 5.9 (58)
PE-HD	Dehyton®	6	11.6 ± 1.8 (2)	13.9 ± 0.5 (2)
PE 100-RC A	Dehyton®	6	165.0 ± 16.6 (3)	176.2 ± 15.2 (2)
PE 100-RC B	Dehyton®	6	588.9 ± 91.1 (3)	1012.2 ± 350.9 (2)

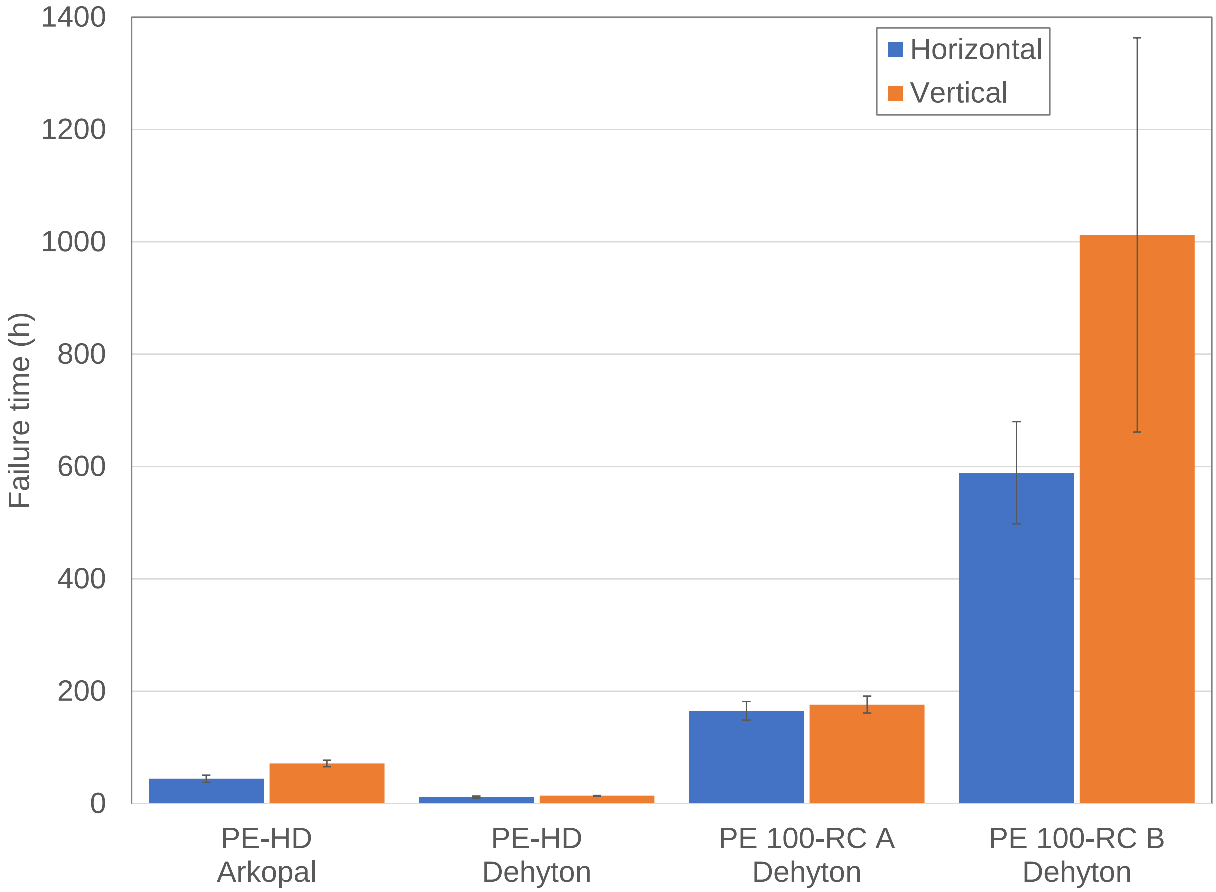

Figure 3. ANPT test failure times (average and standard deviation) in the horizontal and vertical test containers using three different materials and two detergents.

The cause of this difference is the most pertinent question. It is very likely that it is related to the detergent flow inside the container. For example, the vertical containers are round, while the horizontal containers are not, which means the flow will differ between the two types of containers.
A perforated ring was created initially when developing the vertical containers. A vertical perforated tube at the side of the container was used later. While the vast majority of tests were performed with the vertical perforated tube at the side of the container, one test using PE-HD in Arkopal® was carried out with the perforated ring. The failure time of 69.6 h was completely within the range of the remaining results (71.5 ± 5.9 h), which suggests that there is no difference in failure time when using either the perforated ring or the perforated tube.
This seems to indicate that the exact flow within the container is not particularly important. There is however another reason that could explain the differences between the horizontal and vertical containers. This is explained below.

One of the most noticeable results observed for the tests in the horizontal position is that the failure *always* occurred in the notches at the top of the pipe.
The pipes were always placed in the same way in the container, with notches 1 and 4 on the top and notches 3 and 4 on the bottom near the perforated tube (see figure 4 and figure 5). It is expected that the highest flow rate was at the base of the container. This implies that the detergent solution around notches 2 and 3 was refreshed most rapidly.
However, all pipes that were tested in the horizontal containers (including tests not described in this paper) failed at notch 1 or 4 (see figure 6). The other notch often started to fail as well (see figure 7), as described in the previous paper [2].
The pipes tested in the vertical containers were also always placed in the container in the same way. However, only one notch failed in these tests. This could have been any of the four notches; there was no higher rate of failure for a particular notch. The notches that did not fail did not show any signs of the start of a crack or indentation on the inner pipe wall.

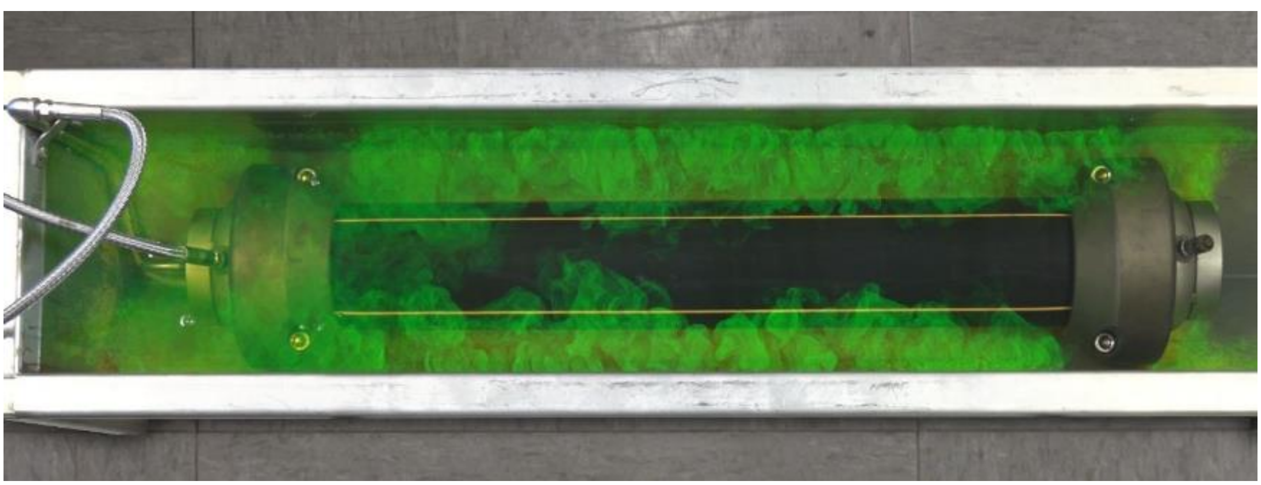

Figure 4. Photo of a test to check the flow in the horizontal container using a green colorant. While this test pipe has no notches, notches 1 and 4 would be positioned at the top of the pipe close to the yellow stripes in a normal test.

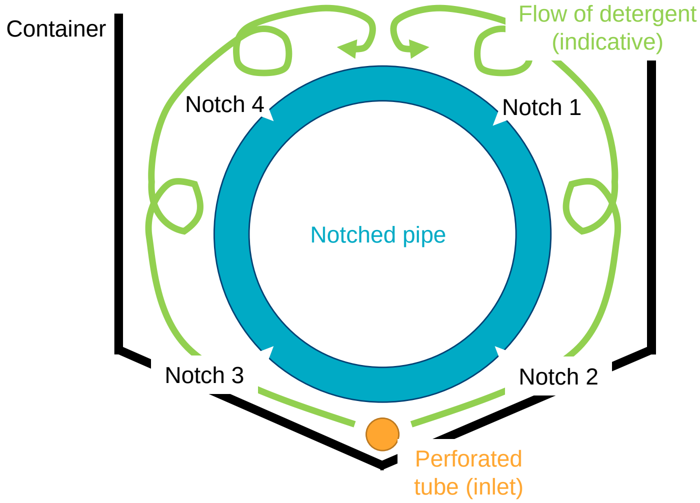

Figure 5. Schematic cross section of the notched pipe placed in the horizontal container. The pipe "floats" above the bottom of the container because it rests on the end caps.

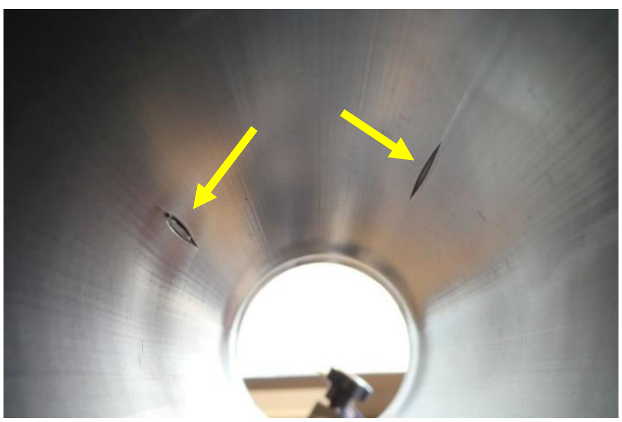

Figure 6. Pipe failure after testing in the horizontal container. Two cracks are visible at the top of the pipe (yellow arrows), although only one has failed.

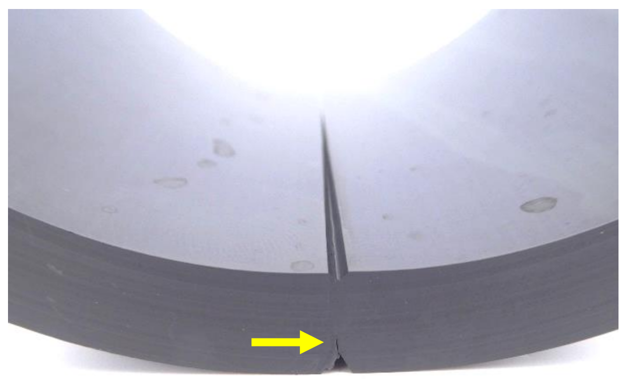

Figure 7. A non-failed notch at the top after testing a pipe in the horizontal container. A crack has started at the base of the notch (yellow arrow) and an indentation has formed on the inner pipe wall.

These results seem to indicate that gravity and a lack of refreshment of the detergent solution around notches 1 and 4, affected the failure time of these notches. The local environment around the notch tips of notches 1 and 4 seemed to be more aggressive. This may be because the Dehyton® concentration was higher at these locations. This resulted in earlier failure than with notches 2 and 3 in the horizontal position, or with any of the notches in the vertical position.

Arkopal® vs Dehyton®
The results are given in table 2 and displayed graphically in figure 8.

Table 2. ANPT test failure times (average and standard deviation) for Arkopal® N100 and Dehyton® PL using three different materials in the horizontal and vertical test containers. The numbers of pipes tested are shown in brackets.

Material	**Position**	**Conditioning time** (h)	**Arkopal®** Avg. ± St. Dev. (h) (number tested)	**Dehyton®** Avg. ± St. Dev. (h) (number tested)
PE-HD	Horizontal	6	44.1 ± 6.4 (8)	11.6 ± 1.8 (2)
PE-HD	Vertical	24	57.2 ± 1.7 (3)	14.0 ± 1.9 (5)
PE 100-RC A	Horizontal	6	659.0 ± 40.1 (3)	165.0 ± 16.6 (3)
PE 100-RC B	Horizontal	6	1320.8 ± 60.9 (3)	588.9 ± 91.1 (3)

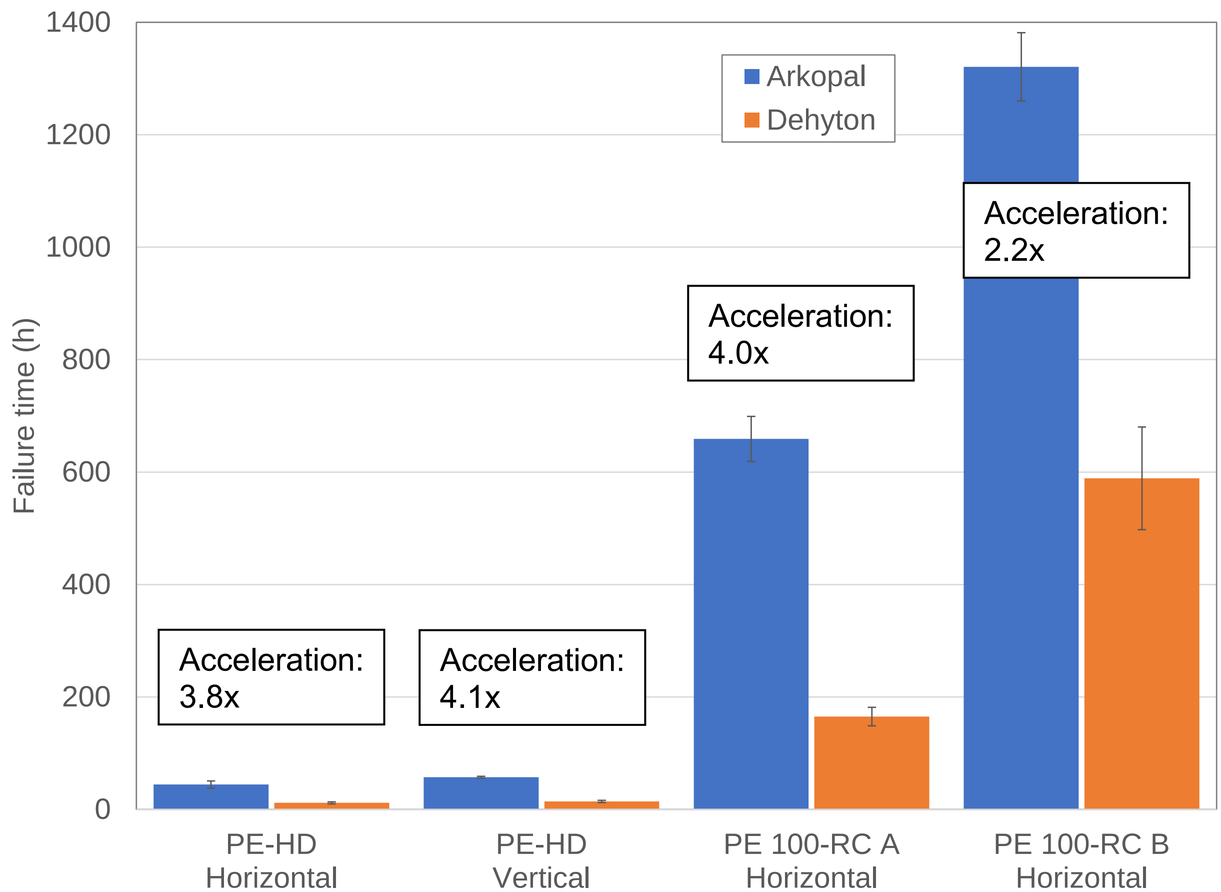

Figure 8. ANPT test failure times (average and standard deviation) for Arkopal® N100 and Dehyton® PL using three different materials in the horizontal and vertical test containers.

The acceleration factor ranges between 2.2 for PE 100-RC B and about 4 for PE-HD and PE 100-RC A.TGM published results of comparative tests with Arkopal® N100 and Dehyton® PL in ISO/TC138/SC5/WG20 [6]. They identified an acceleration factor of 2 for six different PE 100-RC grades. The acceleration varied somewhat more for five different PE 100 grades, which resulted in an acceleration factor between 2 and 8.

The current requirement specified in the EN 1555 series (*PE piping systems for the supply of gaseous fuels*) for the ANPT is based on results from TGM. Their initial tests showed an acceleration factor of around 5 between the normal NPT (without detergent) and the ANPT (with Arkopal® N100) [7]. However, after accumulating over ten years of experience, they identified a minimum acceleration factor of 30 [1]. It was therefore suggested that the NPT requirement of 8760 h in water could be obtained in 300 h using the ANPT with Arkopal®. This is currently specified in the EN 1555 series and its inclusion in the international version (ISO 4437) is also proposed.

This requirement can be adjusted due to the difference in the acceleration factors of Dehyton® and Arkopal®. Using the identified acceleration factor, a minimum failure time of 150 h may be possible if the most conservative acceleration factor of 2 is used. Both PE 100-RC grades tested by Kiwa meet this 150 h requirement for Dehyton®, although it should be noted that both these two grades more than met the 300 h requirement for Arkopal®. Moreover, the conditioning time was 6 h in these tests, while the current version of ISO 13479 requires a conditioning time of 24 h. Increasing the conditioning time also seems to decrease the failure time; PE-HD had an average failure time of 71.5 h with a 6 h conditioning time (see table 1) and an average failure time of 57.2 h with a 24 h conditioning time (see table 2). All these results were for tests using Arkopal® in the vertical containers.
A round robin should therefore be used to determine the final requirement for PE 100-RC grades tested with the ANPT using Dehyton®.

CONCLUSIONS

ISO 13479 does not prescribe the orientation of the test pieces (horizontal or vertical). However, the tests presented in this paper demonstrate that the orientation of the test pipe does affect the failure time: horizontal orientation leads to a shorter failure time. This may be due to the existence of a more aggressive environment inside the notches on the top side of the test pipe, which may arise because the Dehyton® concentration is higher at these locations. We therefore suggest that the orientation of the test pipes should be included in the test report as a minimum. The necessity of specifying the orientation for the ANPT in ISO 13479 to minimize scatter between test laboratories should also be considered.

As Arkopal® N100 cannot be imported, distributed or sold within Europe, a new detergent must be found as soon as possible. Dehyton® PL seems to be a good candidate, as it accelerates the ANPT even further. An acceleration factor of at least 2 between the ANPT with Arkopal® and Dehyton® was found. This could lead to a new requirement for PE 100-RC grades of 150 h.

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