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FINDING THE RIGHT PIPE TEST FOR POLYETHYLENE WITH RAISED RESISTANCE TO SLOW CRACK GROWTH

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SHORT SUMMARY
This paper describes developments related to two pipe tests for PE 100-RC. The first is the point load test (PLT), for which improvements to the test equipment and the current status of an international study are described. The second is the accelerated notch pipe test (aNPT), for which an eco-friendly version is presented. To minimize the required detergent quantities, the test uses smaller containers for each pipe, rather than a large bath.

KEYWORDS
point load test (PLT), accelerated notch pipe test (aNPT), polyethylene with raised resistance to slow crack growth (PE 100-RC)

ABSTRACT
For PE 100-RC, two tests are still under development for pipes: the point load test (PLT) and the accelerated notch pipe test (aNPT).

The PLT was developed by a project group consisting of manufacturers and end users. The PLT determines the resistance to point loads as they can occur in practice. An earlier study showed that the average lifespan of PE 50 pipes under point loading can be estimated fairly accurately. A new study has determined that large scale failures in second-generation PE pipes due to point loads are not likely to occur in the coming decades.
In another study, the results for the PLT were promising but a clear comparison between the three different laboratories and a conclusion regarding the right detergent for proper acceleration of the test was lacking. In a new study, three different laboratories tested PE 100 pipe material from two manufacturers with Arkopal N100. These pipes were also used to experiment with new detergents to accelerate the test. The results of the investigations will be used for the new PLT standard (ISO/CD 22102) and the revision of PE pipe standards such as EN 1555 and ISO 4437.

In the aNPT, the crack growth resistance from an initial notch is measured. This test is performed in exactly the same way as in the standard NPT (ISO 13479), but with one important difference: instead of using water, the pipe is placed in a detergent solution. To keep the test eco-friendly, the amount of detergent is minimized by creating small containers for each individual pipe.
This test method is currently being standardized as an annex to ISO 13479. A round robin investigation was therefore initiated.

INTRODUCTION
With the latest generation of polyethylene grades having a raised resistance to slow crack growth (PE 100-RC), there is a high demand for suitable test methods. Three tests on the polymer are seen as suitable candidates for standardization purposes [1]. These are the strain hardening test (SHT), the cyclic cracked round bar test (CRB) and the accelerated full notch creep test (aFNCT).

The SHT has been standardized in ISO 18488 since 2015, and has quickly become the new standard for Batch Release Testing (BRT). It is a simple tensile test performed at 80°C on a thin specimen from a compression molded plaque [2,3]. It has been demonstrated that the resulting strain hardening modulus corresponds very closely to several environmental stress cracking test methods for high-density polyethylene [4-8].

The CRB test as specified in ISO 18489 is performed at room temperature. By dynamically loading a notched cylinder, the resistance to fatigue corresponds to the resistance to slow crack growth without the need for detergents. Test bars can be machined from compression molded plaques. CRB results are commonly available within several days, depending on the grade [9-13].

The FNCT as specified in ISO 16770 is performed on a notched specimen, which is exposed to a detergent solution while under a static tensile load at elevated temperature. The failure time at a reference tensile stress is calculated by means of interpolation.
The test is commonly performed at 80°C in a 2% solution of Arkopal N100 (CAS no. 9016-45-9). However, this method takes more than one year for PE 100-RC [14]. An accelerated version has therefore been developed using a 2% solution of lauramine oxide (6.67% Dehyton, CAS no. 85408-49-7) at 90°C [1]. This is known as the aFNCT. The aFNCT reduces the failure time to below 1000 hours at stresses around 4 MPa.

With these methods for the polymer available, there is a need for a pipe test. Piping materials made from PE 100-RC can be used for installations in which excavated soil is used as the embedding material, or for trenchless installation methods where more surface damage may be expected.
When the excavated soil is used as the embedding material, stone and rock indentations are likely to occur. A new, scratch-free pipe may therefore be locally deformed, leading to tensile stresses in the inner side of the pipe wall, which may eventually lead to premature failure. The Point Load Test (PLT) has been developed to simulate this failure behavior.
During trenchless installation, scratches may result on the outside of the pipe. These may slowly grow through the entire pipe wall. The Notch Pipe Test as specified in ISO 13479 simulates this process. However, for PE 100-RC, failure times will be over one year [15]. A correlated accelerated method, the accelerated notch pipe test (aNPT), has therefore been developed.

This paper describes the technical challenges in respect of the equipment for both tests. Moreover, preliminary results for the PLT are given to allow an accelerated version suitable for PE 100-RC to be identified.

POINT LOAD TEST (PLT)

Test method and equipment

The point load test method is based on the limited description given in PAS 1075. Three international laboratories therefore began redeveloping the test in 2014, in order to create an open test method.

An overview of the PLT has previously been published [3], and technical details are described in the international standard currently under development (ISO/CD 22102). The equipment and method are summarized below. Figure 1 gives a schematic overview and shows a photo of the test setup at Kiwa Technology.

The PLT consists of a normal water tank as used in the hydrostatic pressure test described in ISO 1167-1. The test pipe is supported or hung in the tank and can be pressurized using the pressurizing equipment.

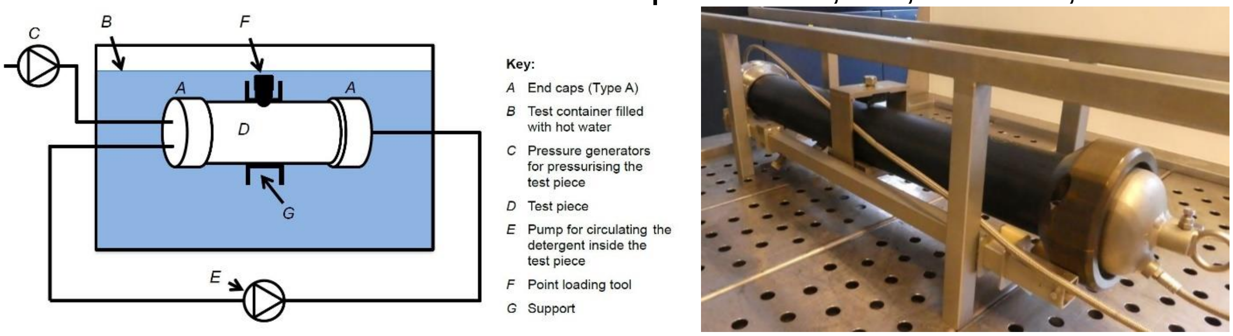

Figure 1. Left: Schematic overview of the point load test (PLT). Right: PE pipe deformed by a point load and filled with detergent solution, ready to be immersed in the water tank.

The following additional equipment is required for the PLT:

- Circulation equipment: the test pipe is filled with a detergent solution, which must be circulated inside the pipe (*E* in figure 1).
- Point loading tool and support: the tool, which has a hemispherical tip, introduces a fixed displacement in the radial direction of the pipe (*F* and *G* in figure 1).

The test was developed for PE 100-RC. This is a material considered suitable for alternative installation methods, such as trenchless pipe laying or where excavated soil is used as the embedding material (laying without sand beds), while maintaining a life expectancy of more than 100 years. The PLT simulates a pipe exposed to a point load. The temperature is raised to accelerate the failure time. Assuming Arrhenius to be valid, and using an activation energy of 66.1 kJ/mol, 1 year at 80°C corresponds to 100 years at 20°C [14]. The acceleration obtained by using a detergent (2% Arkopal N100) is used as a safety factor.

A previous study has shown that the average lifespan of PE 50 pipes under point loading can be estimated fairly accurately [16]. In this previous study, Dehyton was used as the detergent solution.

Improvements to the equipment

In 2018, the report of a DVGW research project became available. This presented the first results and the repeatability within each test laboratory [1]. During this project, various improvements to the equipment were identified.

The connections and pumps were one of the biggest challenges. At 90°C in particular, the detergent solution with Dehyton was very harmful to the rubber sealing rings made from Viton, a fluorocarbon rubber (FKM) (see figure 2). This led to many leaks in the equipment, making the point load test impossible to complete.

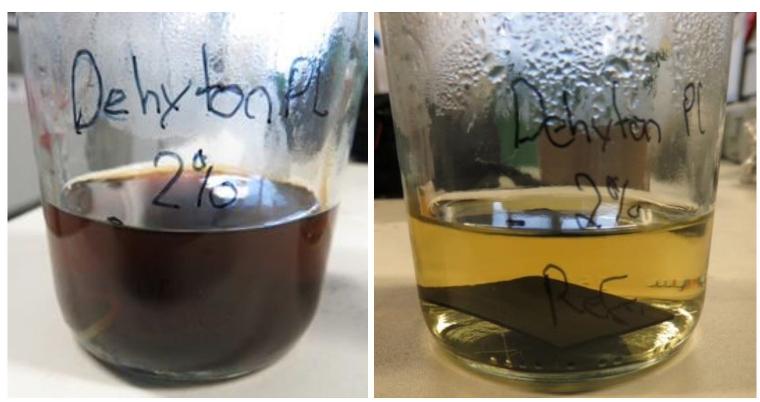

Figure 2. Seals made of FKM (left) and EPDM (right) kept in Dehyton PL at 90°C for 3 days. Image courtesy of SKZ.

The problem was solved in two main ways:

1. Avoiding the use of rubber sealing rings where possible. This means that most quick-connect fittings are replaced with fixed connections.
2. If rubber sealing is required (e.g. in the pumps), EPDM is used. EPDM appears to be much more resistant to the detergent solutions used.

If the pipes fail due to the point load, the detergent solution inside the pipe will flow into the water tank. It is expected that this will have no effect on the other pipes, because the tensile stress is found at the inner pipe wall, and this is the location where the crack starts. Detergent on the outside of the pipe should therefore not accelerate the failure time. Moreover, the water tank has an internal volume of 1800 liters, which means that the detergent concentration will be very low.

Nevertheless, after the failure of many pipes, the concentration of detergent will increase and will thus affect the rest of the equipment. A deposit was observed on the tank walls, and the float switch failed multiple times. This problem was solved by continuously refreshing the water in the tank at a slow rate. After failure of a pipe, the detergent concentration will increase. However, it will decrease again over time, because of the clean water that is continuously added.

Finally, there are brand-specific properties that are difficult to align between the laboratories. For instance, a turbulent flow in the detergent solution inside the pipe would be expected to give the shortest failure times. A turbulent flow prevents segregation of the detergent solution, and will lead to a better-defined concentration at the inside wall at the point loading tool. However, because of the use of different equipment brands at the three labs, the manner and the speed at which the detergent flows along this point on the inner wall of the pipe also differs.
To determine if a flow is turbulent, the Reynolds number (*Re*) can be calculated:

$$Re = \frac{\mathrm{v} \times D}{\upsilon}$$

where:

- v = flow speed (volume flow speed divided by the cross section of the pipe) (m/s)
- *D* = internal diameter of the pipe (m)
- ν = kinematic viscosity of the fluid (m^2/s)

The Reynolds number is about 1000 when using a volume flow of 1.5 l/min, an internal pipe diameter of 90 mm and a kinematic viscosity of 0.3643 mm^2/s (for water at 80°C [17]). This indicates that laminar flow occurs inside the PE pipe.
Many discussions took place at Kiwa Technology about how to adjust the flow, because this involves more than simply increasing the pump speed in the circulation system. For instance, increasing the flow may result in a sort of water jet from one end cap to the other end cap, without the desired turbulent flow. Moreover, these are expensive pumps, because they have to circulate the solution under pressure and withstand the detergent.
It was eventually decided to use an internal system to spray the detergent solution directly onto the inner side of the pipe wall. This system was already in use at one of the other two laboratories.

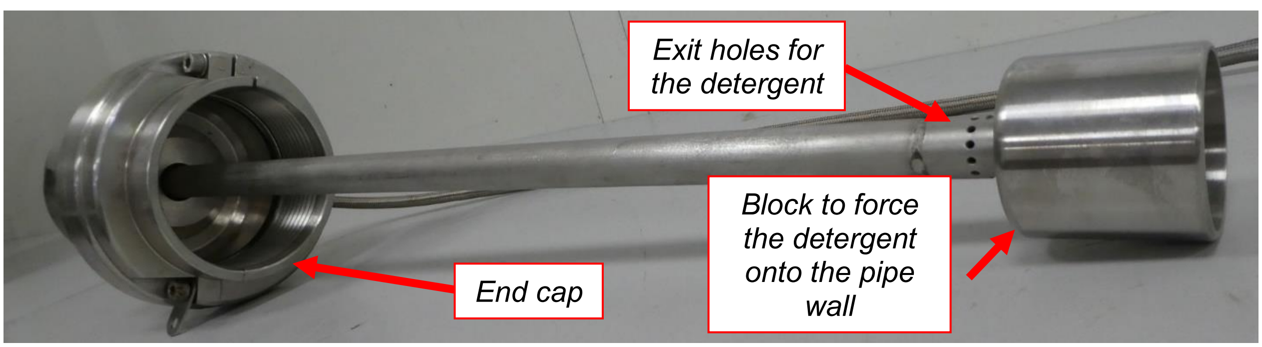

Figure 3. Internal system to spray the detergent solution directly onto the inner side of the pipe wall.

Current results

While a previous study has shown that the average lifespan of PE 50 pipes under point loading can be estimated fairly accurately [16], Dutch end users wished to know whether failures in second-generation PE pipes due to point loads can be expected in the coming decades. The results for all first-generation PE pipes tested with the PLT were therefore compared with the results for two HDPE pipes produced in 1987 and stored unused at Kiwa Technology. Note that the results for the first-generation PE pipes contain the results of multiple studies, in which different detergents, concentrations, SDR classes and penetration depths of the point load applied. The differences in results were only very minor. The data for 70 tested pipes has therefore been combined.

The results are given in figure 4 (left), where the data points are analyzed using the 3-parameter model specified in ISO 9080. The solid line are the LTHS and the dashed lines are the LPL. The predicted average lifespan using this data is a few years longer than was found in the previous study [16], but the same conclusions remain valid:

- The soil temperature is very important for the time to failure.
- Thin pipes fail sooner at the same point load and pressure than thick-walled pipes.
- The life expectancy of first-generation PE pipes can be prolonged if the pressure is decreased.

The results for the two second-generation HDPE pipes are given in figure 4 (right). Both pipes are treated as one material. It is clear that, in this case, the failure time is independent of the hoop stress (and thus the internal pressure). Note that no LPL could be calculated due to the limited number of samples.

A “generation factor” is calculated for different stresses and temperatures (see figure 5) using the 3-parameter model. Although the “generation factor” is lower at lower stresses, the life expectancy at lower stresses is much higher. The calculated life expectancies for second-generation PE operated at 2 – 4 bar(g) at 10 – 14°C thus exceed 100 years. Large scale failures in second-generation PE pipes due to point loads are therefore not expected in the coming decades, although incidents are still possible. It must be emphasized that these pipes are designed for installation in sand bedding. Only PE 100-RC pipes are designed for long-term resistance to point loads under all circumstances.

Details of the test results for PE 100 pipes with the new and publicly-available point load test method are given in a DVGW report [1], and were presented at Plastic Pipes in Infrastructure [18]. Tests on different PE grades showed good repeatability within a test laboratory environment. However, PE 80 materials in particular had much longer times to failure than expected. This may

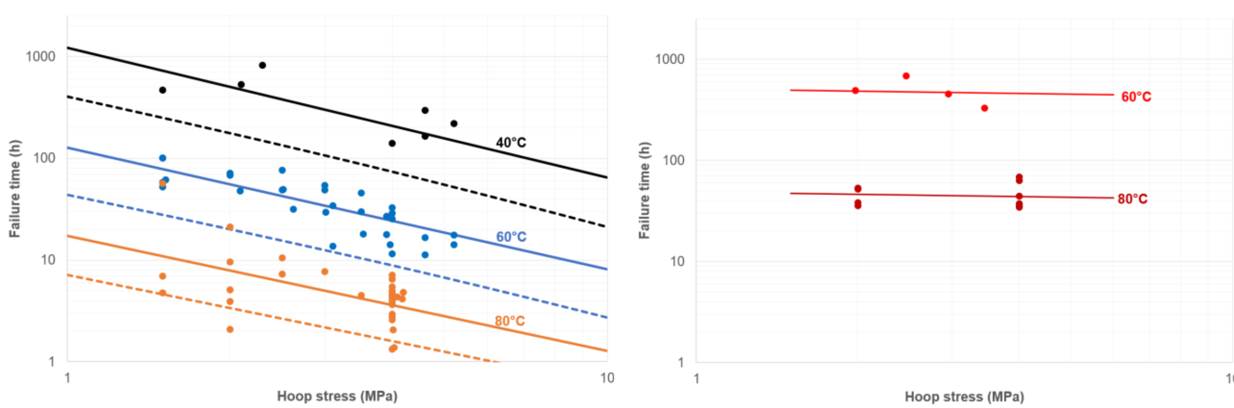

Figure 4. PLT results for first-generation HDPE (left) and second-generation HDPE (right).

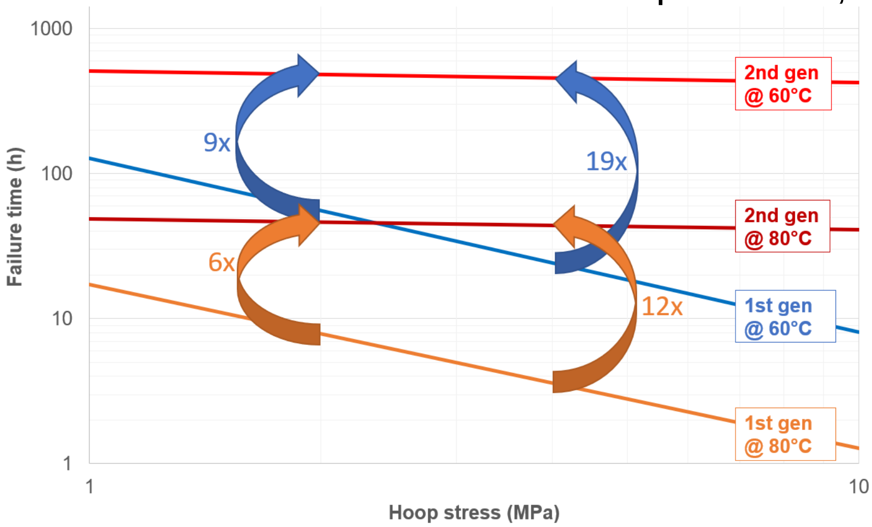

Figure 5. "Generation factor" for two temperatures (60 and 80°C) and two stresses (2 and 4 MPa).

be due to a higher degree of relaxation under the constant deformation of the point than is the case with PE 100 materials. Moreover, the point load probably led to significant local plastic deformation, which meant that the influence of the point load was less pronounced. Overall, no accelerated procedure could be derived from the point load test results. However, some PE 100 pipes showed promising results.

A second DVGW project has therefore been initiated, with multiple goals:

- To ensure the reproducibility of the PLT and comparability between different labs.
- To develop a new method to reduce the testing time of one year previously required.
- To determine a minimum requirement for the accelerated test method, i.e. a minimum service life to be achieved for pipes made from PE 100-RC under specified test conditions (temperature, wetting agent, internal pressure).

To determine the reproducibility, two new PE 100 pipes were produced. These were tested with 2% Arkopal N100 at three test laboratories (Kiwa, SKZ and TGM) at 4 MPa. The results are given in figure 7. At all three test laboratories, material *PE 2019-266* had a longer failure time than *PE 2019-268*. The scatter within the laboratory results is very good. There is some scatter between the laboratories, which is normal for such tests (e.g. hydrostatic pressure tests, notched pipe test). Two examples of the inner pipe wall under the point load of failed pipes are shown in figure 6. The failure mode is brittle for all laboratories. It is similar to the failure mode seen before [3]. However, there were some instances where the pipe failed outside the area of the point load.

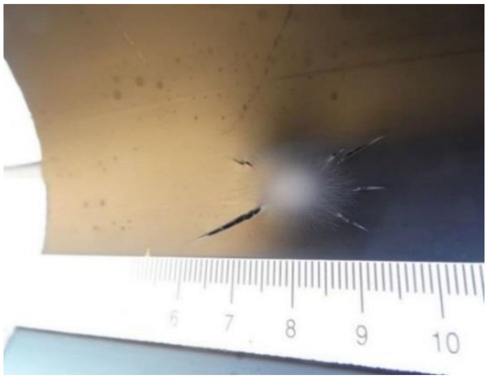

Figure 6. Inner pipe wall under the point load of a failed pipe, tested at lab1 (left) and lab2 (right).

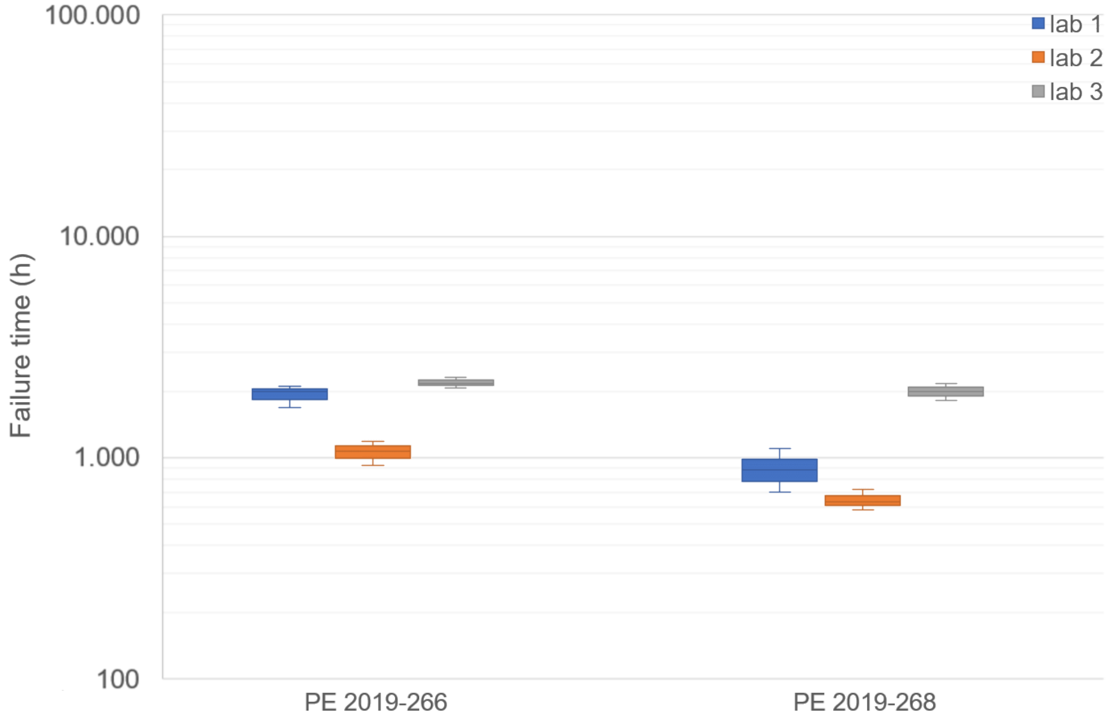

Figure 7. Failure times of two PE pipes tested at three laboratories with 2% Arkopal N100, 4 MPa at 80°C.

Based on these results, all three test labs will test the two pipe materials with another detergent to determine which detergent results in the best acceleration. These are:

- 4 MPa, 80°C and 90°C, 2% lauramine oxide (6.67% Dehyton) + 3% lauryl ether sulfate (5% Genapol LRO paste) + buffer
- 4 MPa, 90°C, 2% lauramine oxide (6.67% Dehyton)
- 4 MPa, 90°C, 2% benzenesulfonic acid (8% Disponil LDBS 25)

No results are available at the time of writing.

Following discussions in ISO/TC138/SC5/WG20 (March 4th, 2020, Delft) on the standardization of the test method in ISO/CD 22102, further improvements to the test procedure and equipment were made. For example, the tip of the point load should not turn. The sequence of the procedure is also aligned more effectively: the point load is first applied, the pipe is then filled with the detergent solution, and finally the pipe is immersed in the water tank.
These improvements will be used in the second round robin, in which each test institute will test the two PE 100 pipes with the best accelerated test conditions. No results are available at the time of writing.

ACCELERATED NOTCH PIPE TEST (ANPT)

Test method and equipment

The notch pipe test (NPT) is a well-known and much used test method. The first version of the standard (ISO 13479) was published in 1997. The method published by Kratochvilla et al [13,19] attracted new interest [15] in the search for new methods to test PE 100-RC pipes.

The aNPT is very similar to the standard NPT, but with one important difference: to reduce the time to failure, an external detergent solution is used in contact with the notches instead of water. An annex to ISO/TC138/SC5/WG20 containing more details about the test method is currently being drafted. For now, the aNPT is performed with only 2% Arkopal N100.

Improvements to the equipment

Although replacing water with a detergent solution may appear to be a minimal change, it has major consequences. If the water tank is used for normal NPT or hydrostatic pressure tests, detergents are absolutely forbidden. Even the use of detergents in the same room is highly discouraged, as a small quantity of detergent in otherwise clean water can have a major impact on the test results.

The separate tank with the detergent solution must withstand the new, aggressive environment. Because Arkopal N100 at 80°C is not as aggressive as Dehyton at 90°C, the environment is relatively mild, but one needs to be aware that leakages in the equipment may occur in the long run.
The tank is also often relatively large. For example, the water tank at Kiwa Technology for the PLT contains 1800 liters of water. A 2% detergent solution would require 36 kg of Arkopal N100.
Arkopal N100 is a nonylphenol ethoxylate (CAS no. 9016-45-9), which is described as persistent, bio-accumulative and toxic [20] by the ECHA. It is a substance of very high concern (SVHC), which requires authorization before it is used (Annex XIV of REACH). Some uses of this substance are restricted under Annex XVII of REACH.
This means that several hundred liters of Arkopal N100 are required annually, because reusing the detergent is not advisable [21-23] in view of its ageing. Kiwa Technology is therefore of the opinion that tests in Arkopal N100 should always be performed in a freshly-prepared solution.
The consequence is that a large quantity of Arkopal is needed each year, and the enormous volume of waste water (1800 liters each time) needs to be properly disposed of.

All these arguments led to the conclusion that filling the complete tank with a detergent solution is not a viable option for Kiwa Technology. Smaller tanks were designed to make the test eco-friendly. However, it is essential to have a good mixture to ensure a homogeneous temperature and detergent distribution. According to the information given in the ISO working group, a normal water tank would have sufficient flow to prevent segregation of the detergent in the solution. The circulation system of the PLT is used to create such a flow in the smaller tanks. The bottom of the tank has a V shape, in which a perforated pipe is placed. This is the inlet for the detergent solution. The outlet is placed at the top side of the tank. In this way, the Arkopal N100 is forced upwards, while it would sink if no flow were present (Figure 9). The circulation system was checked with a colorant. This demonstrated visually that the system works very well (Figure 8).

The new tank system reduces the volume of the detergent solution to only 30 liters per pipe, requiring only 600 grams of Arkopal N100 per test. Moreover, each pipe can be tested in a freshly made solution, so that each pipe is exposed to Arkopal of the same age.

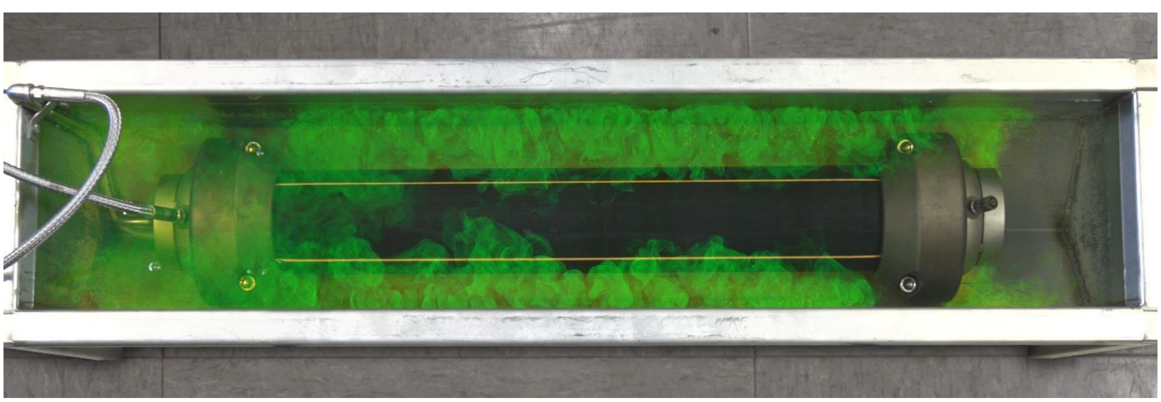

Figure 8. Checking the flow with a colorant in a smaller tank for the aNPT. A few seconds after this photo was taken, the green colorant was homogeneously distributed.

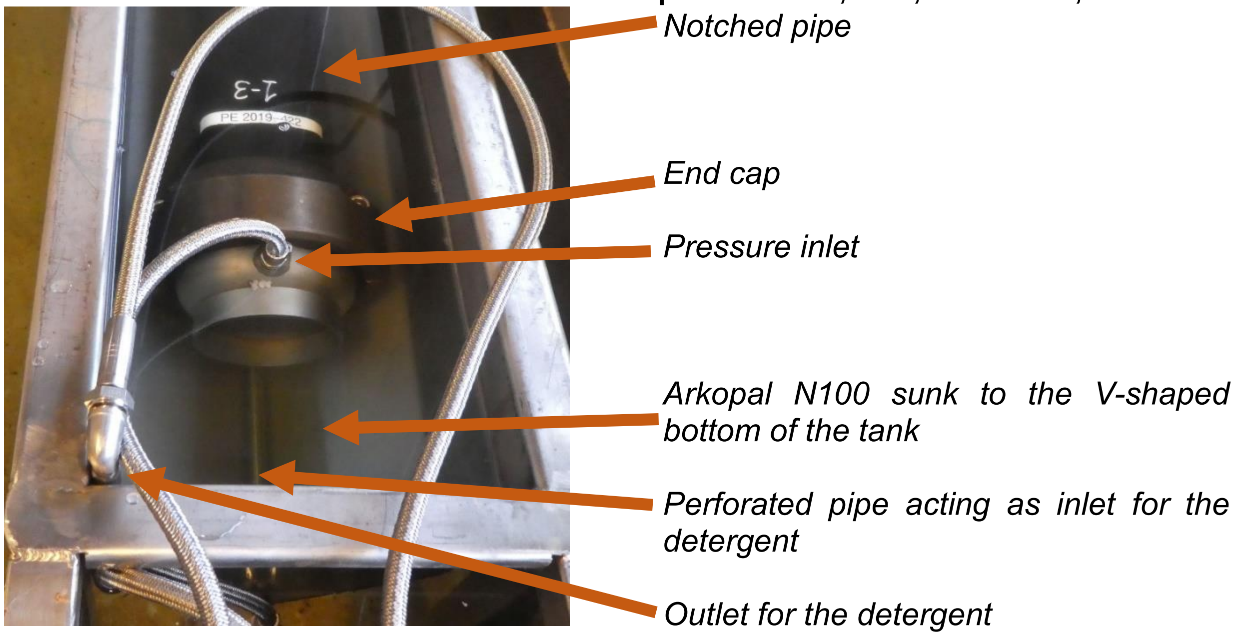

Figure 9. The smaller test setup at Kiwa Technology for the aNPT. In this photo, it is placed outside the water tank and the circulation is stopped, causing segregation of the Arkopal N100 solution.

Current results

Three pipes of two HDPE grades were tested with the equipment as described above during a round robin organized by the PE 100+ Association. After failure, multiple notches were visible on the inner pipe wall of all the pipes (see one example on the left of figure 10), although only one notch actually leaked. One pipe was cut through the notches. The cross sections are shown in figure 10 and figure 11. The failed notch had a ductile part on the inside of the notch (figure 10 right). Another notch that was also visible on the inner pipe wall did show a starting crack, and had an indentation on the inner pipe wall (figure 11 left). A notch that was not visible on the inner pipe wall did not show a crack, but a line is clearly visible (figure 11 right).

While this was not investigated further, a possible explanation is that failure starts with void formation, which results in a visible line in the HDPE. Starting at the notch tip, a crack develops from these voids. The residual tensile stresses cause a ductile flow of material on the other side. Eventually, the remaining ligament is so thin that it has a tendency to burst ductile through the crack. However, because of the small crack, it is unable to do so and therefore shrinks back to the inner pipe wall.

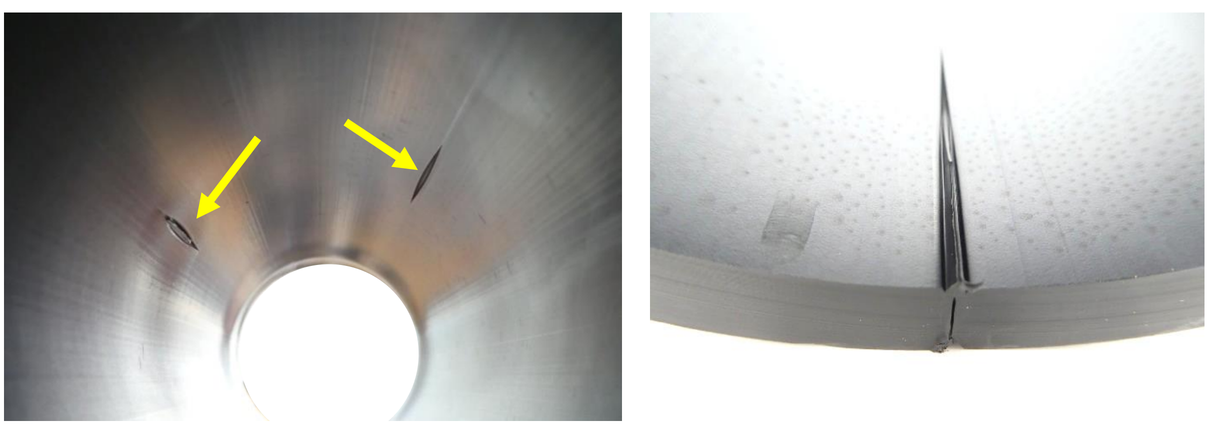

Figure 10. Pipe failure after the aNPT. Left: multiple notches are visible (yellow arrows), although only one failed. Right: the failed notch has a ductile part on the inner pipe wall.

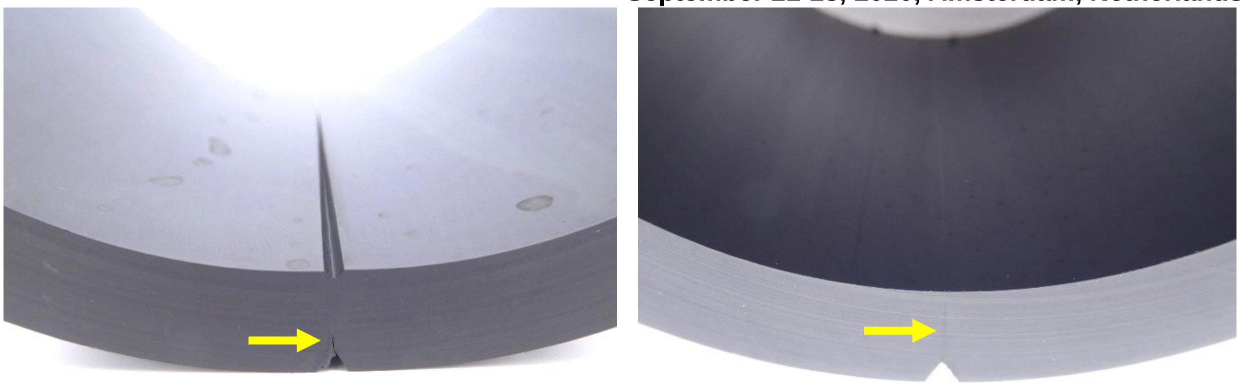

Figure 11. Pipe failure after the aNPT. Left: a non-failed notch has a starting crack (yellow arrow) and an indentation on the inner pipe wall. Right: a non-failed notch has no crack, but a fine line over the ligament is visible (yellow arrow).

CONCLUSIONS

Two pipe tests are under development: the point load test (PLT) and the accelerated notch pipe test (aNPT). With the improvements presented in this paper, these tests can be used to test PE 100-RC grades.

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